(19)国家知识产权局

(12)发明专利

(10)授权公告号 CN 113994496 B
(45)授权公告日 2025.06.06

(21)申请号 202180003824.2
(22)申请日 2021.03.11
(65)同一申请的已公布的文献号
申请公布号 CN 113994496 A
(43)申请公布日 2022.01.28
(30)优先权数据
10-2020-0030232 2020.03.11 KR
(85)PCT国际申请进入国家阶段日
2021.12.08
(86)PCT国际申请的申请数据
PCT/KR2021/003037 2021.03.11
(87)PCT国际申请的公布数据
WO2021/182893 KO 2021.09.16
(73)专利权人 株式会社LG化学
地址 韩国首尔
(72)发明人 金旼俊 李东勋 车龙范 徐尚德 金永锡 吴重锡 金曙渊
(74)专利代理机构 北京集佳知识产权代理有限公司 11227
专利代理师 冷永华 赵菲
(51)Int.Cl.
H10K 85/60(2023.01)
H10K 50/11(2023.01)
(56)对比文件
WO 2020013448 A1,2020.01.16
CN 110313078 A,2019.10.08
CN 110268036 A,2019.09.20
KR 20190005522 A,2019.01.16
审查员 柴莹莹

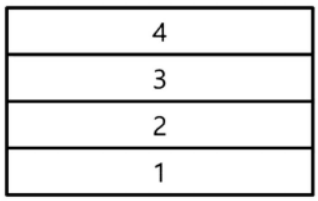
权利要求书200页 说明书310页 附图1页

(54)发明名称

有机发光器件

(57)摘要

本公开内容涉及具有改善的驱动电压、效率和寿命的有机发光器件。



CN 113994496 B

1.一种有机发光器件,包括:

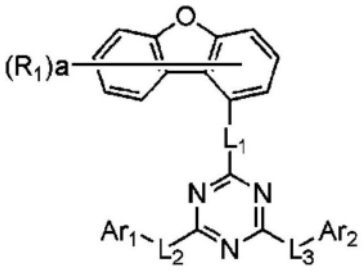
阳极;

阴极;以及

设置在所述阳极与所述阴极之间的发光层,

其中所述发光层包含由以下化学式1表示的化合物和由以下化学式2表示的化合物,

[化学式1]



其中在化学式1中,

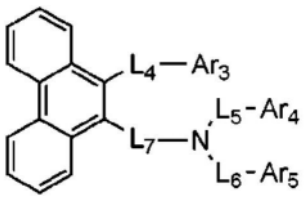
Ar_1和Ar_2各自独立地为经氘取代或未经取代的苯基、经氘取代或未经取代的联苯基、经氘取代或未经取代的三联苯基、经氘取代或未经取代的萘基、经氘取代或未经取代的菲基、二苯并呋喃基、或者二苯并噻吩基,

L_1至L_3各自独立地为单键、亚苯基、亚联苯基或亚萘基,并且L_1至L_3各自独立地未经取代或经氘取代,

R_1为氢、氘、经氘取代或未经取代的苯基、经氘取代或未经取代的联苯基、经氘取代或未经取代的三联苯基、经氘取代或未经取代的萘基、经氘取代或未经取代的菲基、三亚苯基、萘基苯基、苯基萘基、荧蒽基、二氢茚基、二苯并呋喃基、二苯并噻吩基、苯并萘并呋喃基、或者苯并萘并噻吩基,

a为0至7的整数,

[化学式2]



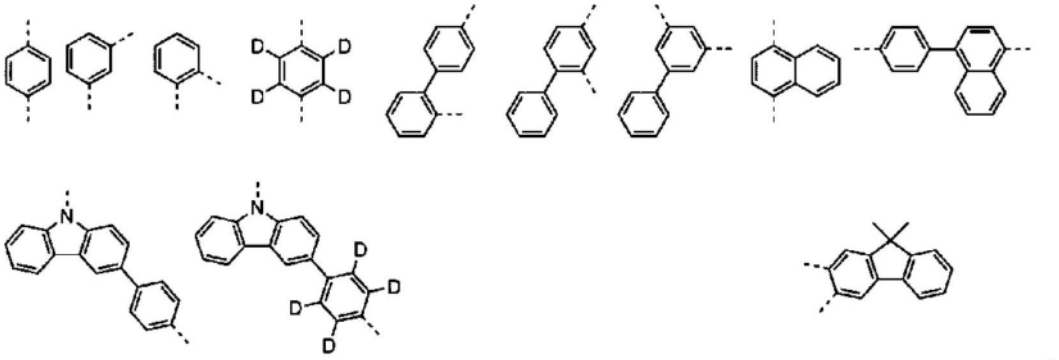
其中在化学式2中,

Ar_3为氢;或者经氘取代或未经取代的苯基,

Ar_4和Ar_5各自独立地为苯基、经5个氘取代的苯基、联苯基、经4个氘取代的联苯基、经9个氘取代的联苯基、三联苯基、经4个氘取代的三联苯基、四联苯基、萘基、菲基、三亚苯基、二甲基芴基、二苯基芴基、咔唑基、苯基咔唑基、或二苯并噻吩基,

L_4为单键、亚苯基、经4个氘取代的亚苯基、亚联苯基、亚萘基、苯基亚萘基、亚咔唑基、苯基亚咔唑基、经4个氘取代的苯基亚咔唑基、亚二苯并呋喃基、苯基亚二苯并呋喃基、经4个氘取代的苯基亚二苯并呋喃基、或二甲基亚芴基,

L_5和L_6各自独立地为单键或选自以下中的任一者:



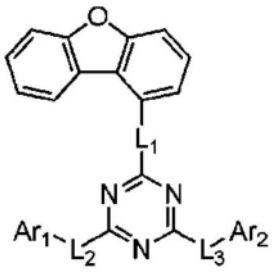
以及

L_7为亚苯基、亚联苯基、或者亚萘基，并且L_7未经取代或经氘取代。

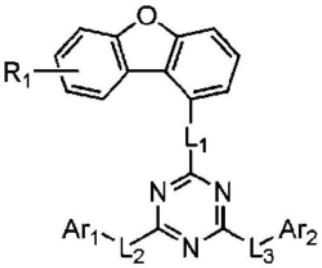
2.根据权利要求1所述的有机发光器件，

其中由化学式1表示的所述化合物由以下化学式1-1至1-3中的任一者表示：

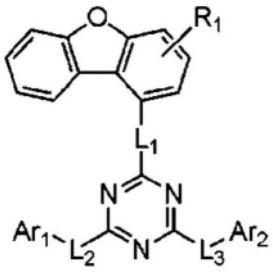
[化学式1-1]



[化学式1-2]



[化学式1-3]



其中在化学式1-1至1-3中，

Ar_1和Ar_2、以及L_1至L_3如权利要求1中所限定，以及

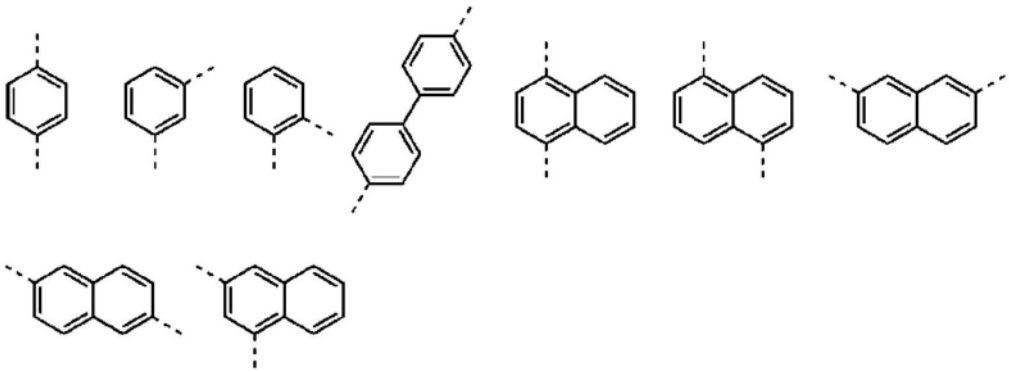
R_1为氘、经氘取代或未经取代的苯基、经氘取代或未经取代的联苯基、经氘取代或未经取代的三联苯基、经氘取代或未经取代的萘基、经氘取代或未经取代的菲基、三亚苯基、萘基苯基、苯基萘基、荧蒽基、二氢茚基、二苯并呋喃基、二苯并噻吩基、苯并萘并呋喃基、或者苯并萘并噻吩基。

3.根据权利要求1所述的有机发光器件，

其中Ar_1和Ar_2各自独立地为苯基、联苯基、三联苯基、萘基、菲基、二苯并呋喃基或二苯并噻吩基。

4.根据权利要求1所述的有机发光器件，

其中L_1至L_3各自独立地为单键或选自以下中的任一者：



5.根据权利要求1所述的有机发光器件，

其中每个R_1各自独立地为氢、氘、苯基、联苯基、三联苯基、萘基、菲基、三亚苯基、萘基苯基、苯基萘基、荧蒽基、二氢茚基、二苯并呋喃基、二苯并噻吩基、苯并萘并呋喃基或苯并萘并噻吩基。

6.根据权利要求1所述的有机发光器件，

其中Ar_1、Ar_2和R_1中的至少一者为萘基、菲基、二苯并呋喃基、或二苯并噻吩基，或者

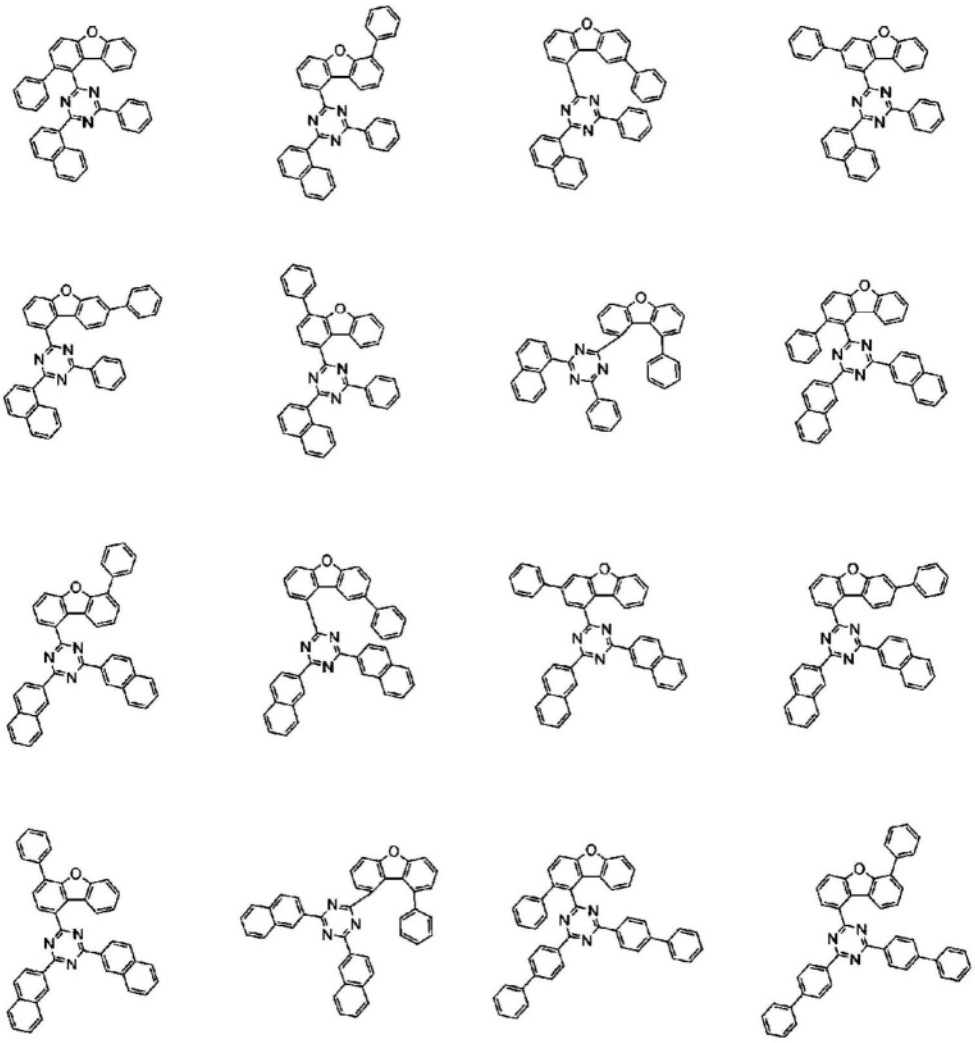
R_1为苯基萘基、萘基苯基、荧蒽基、苯并萘并呋喃基或苯并萘并噻吩基。

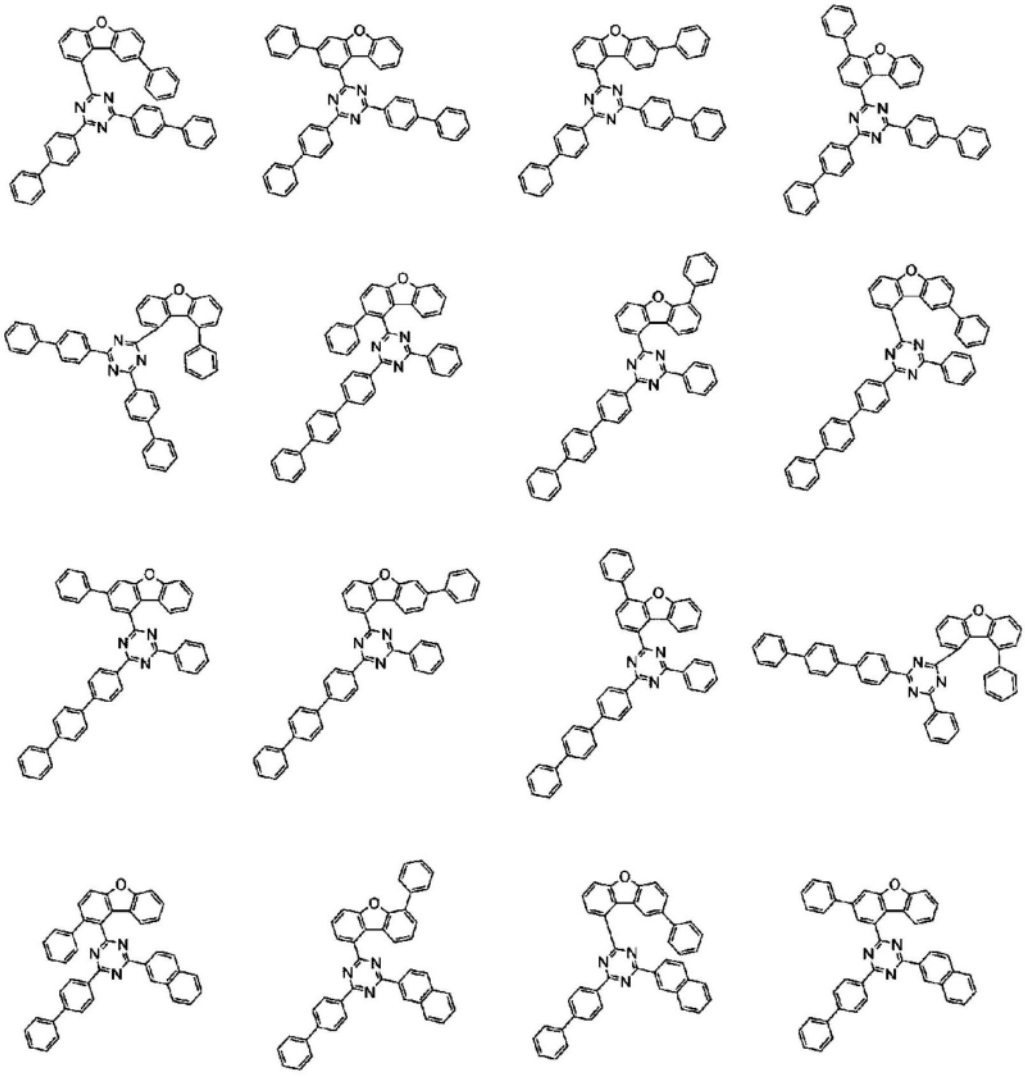
7.根据权利要求1所述的有机发光器件，

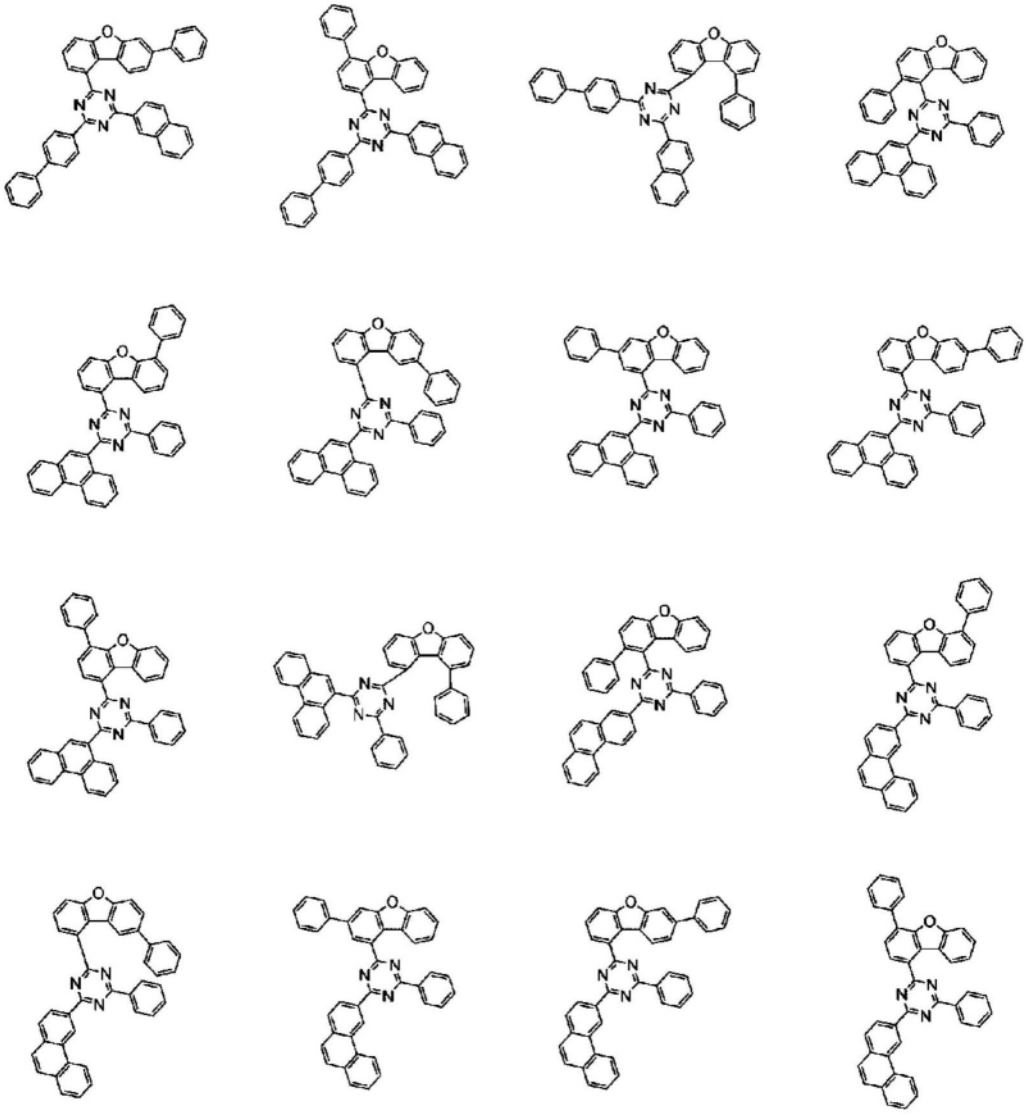
其中a为0或1。

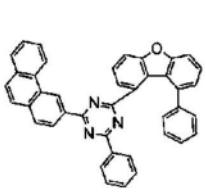
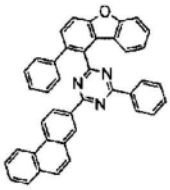
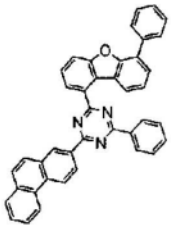
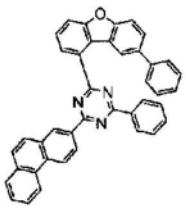
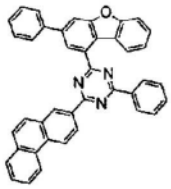
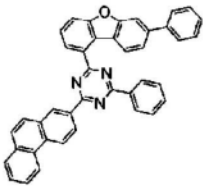
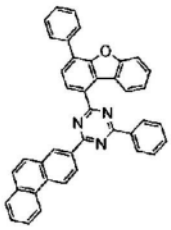
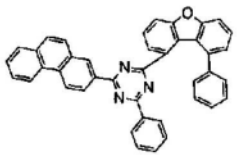
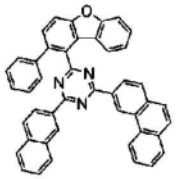
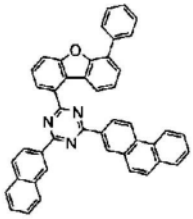
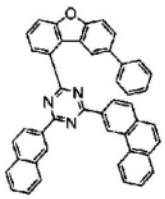
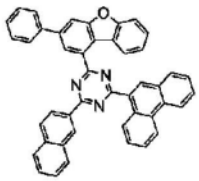
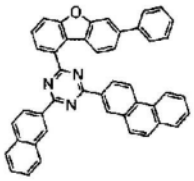
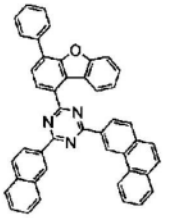
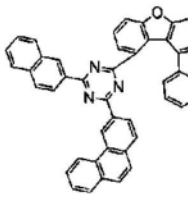
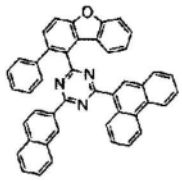
8.根据权利要求1所述的有机发光器件，

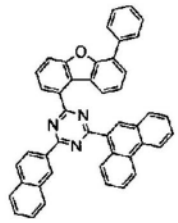
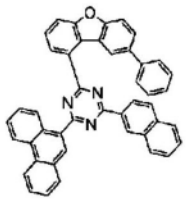
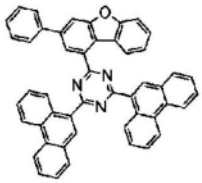
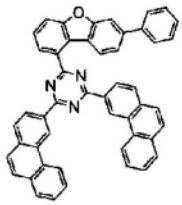
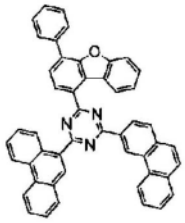
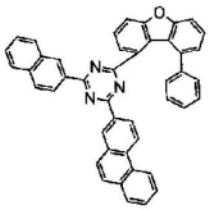
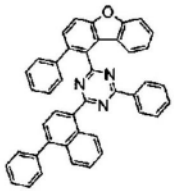
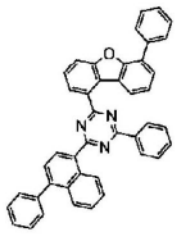
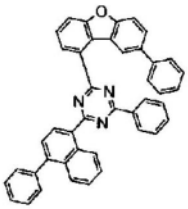
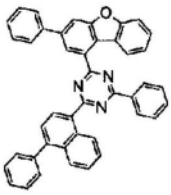
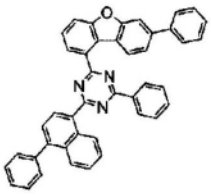
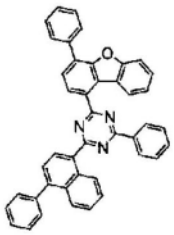
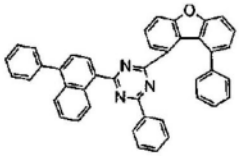
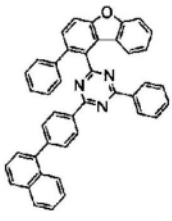
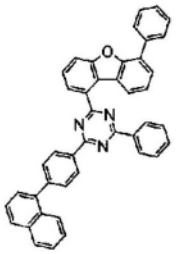
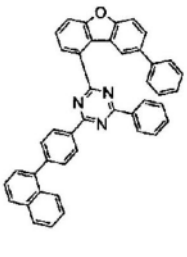
其中由化学式1表示的所述化合物为选自以下中的任一者：

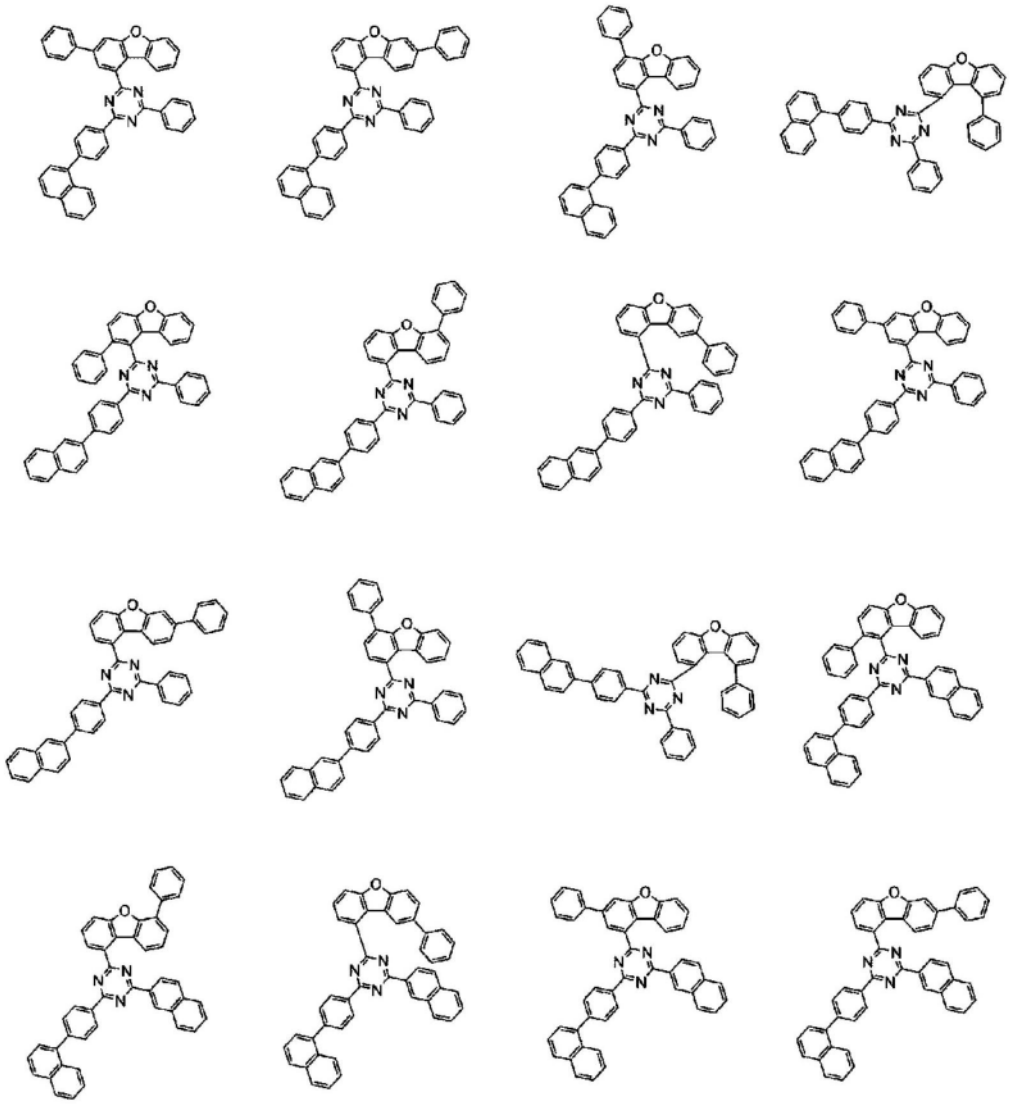


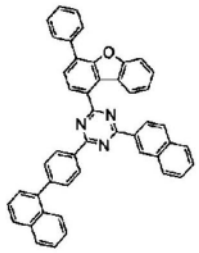
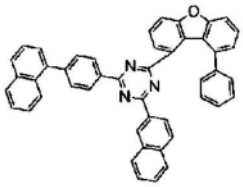
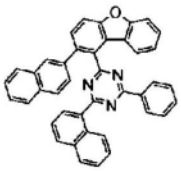
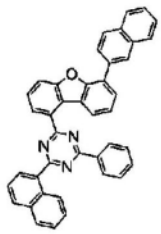
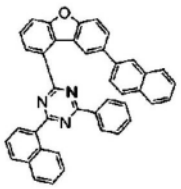
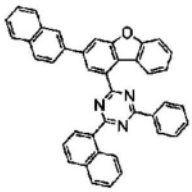
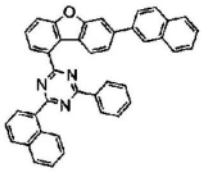
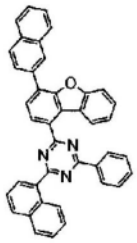
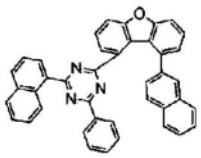
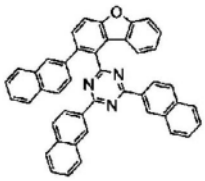
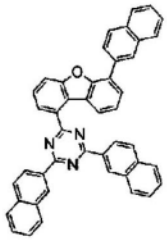
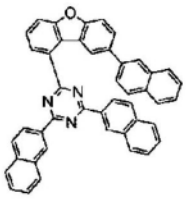
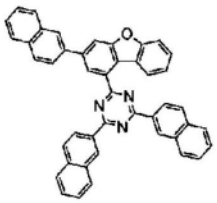
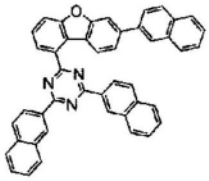
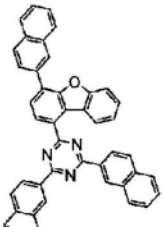
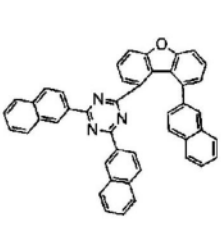


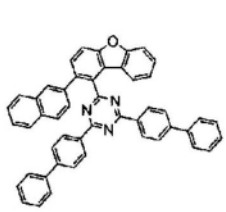
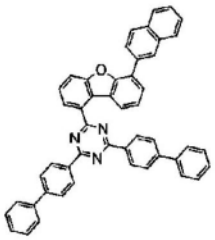
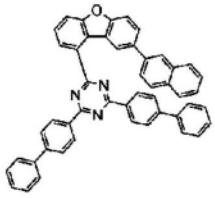
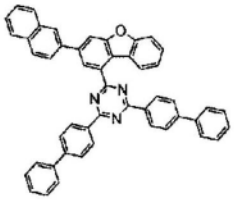
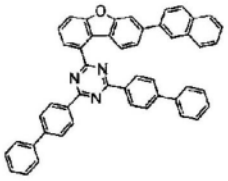
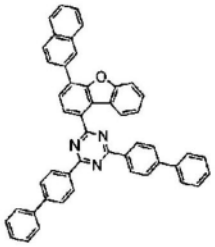
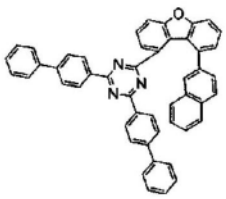
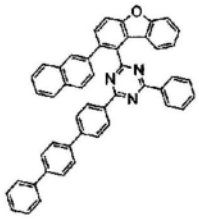
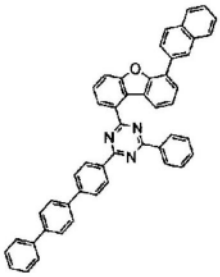
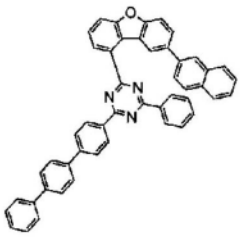
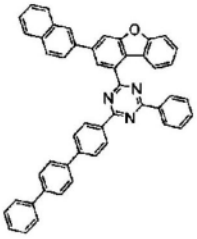
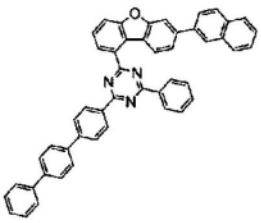
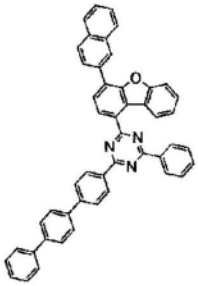
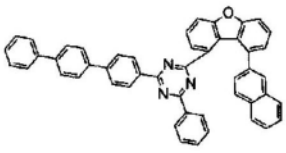
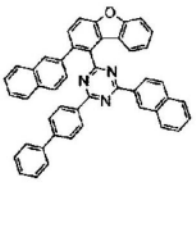
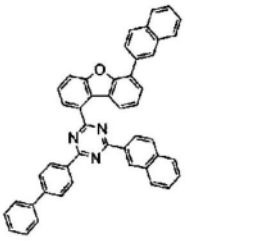


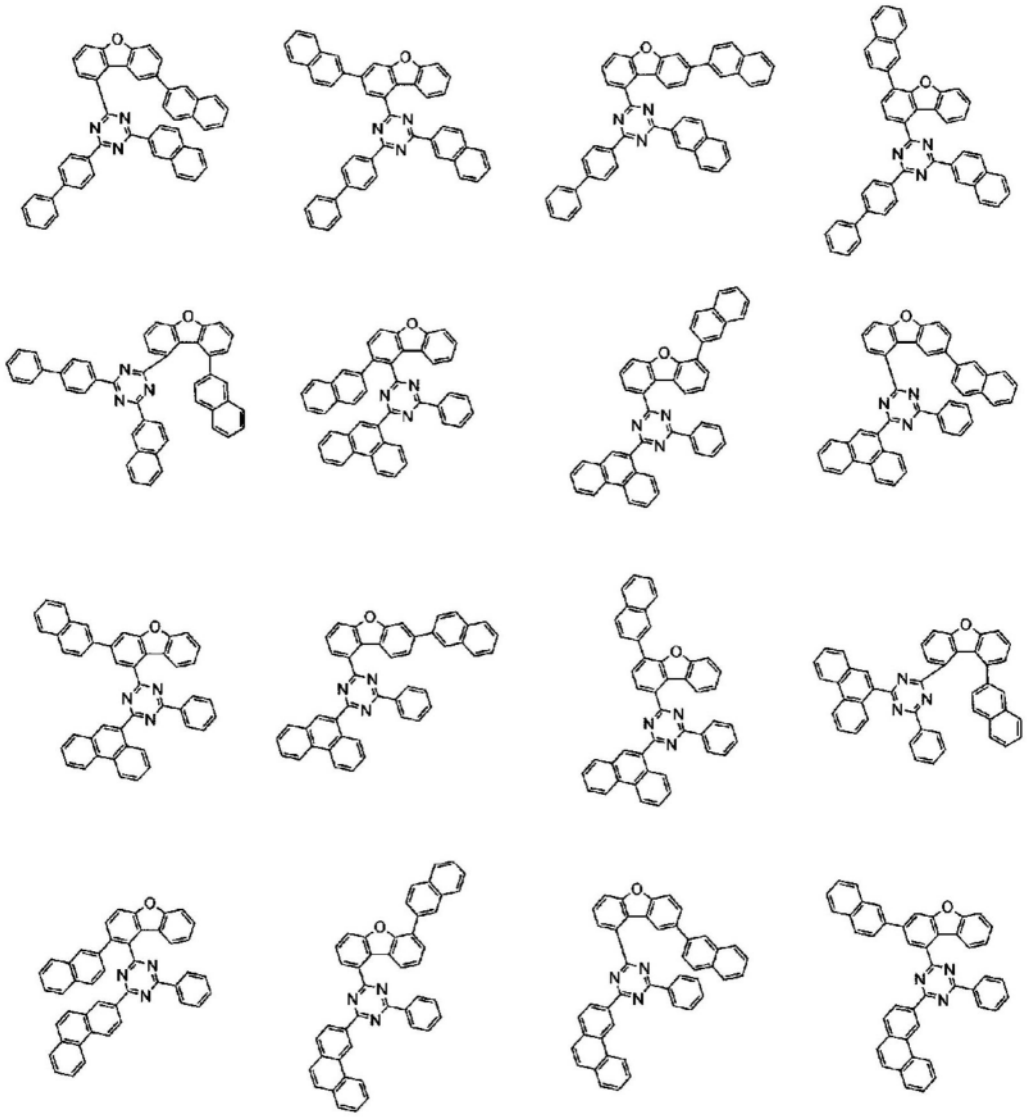

















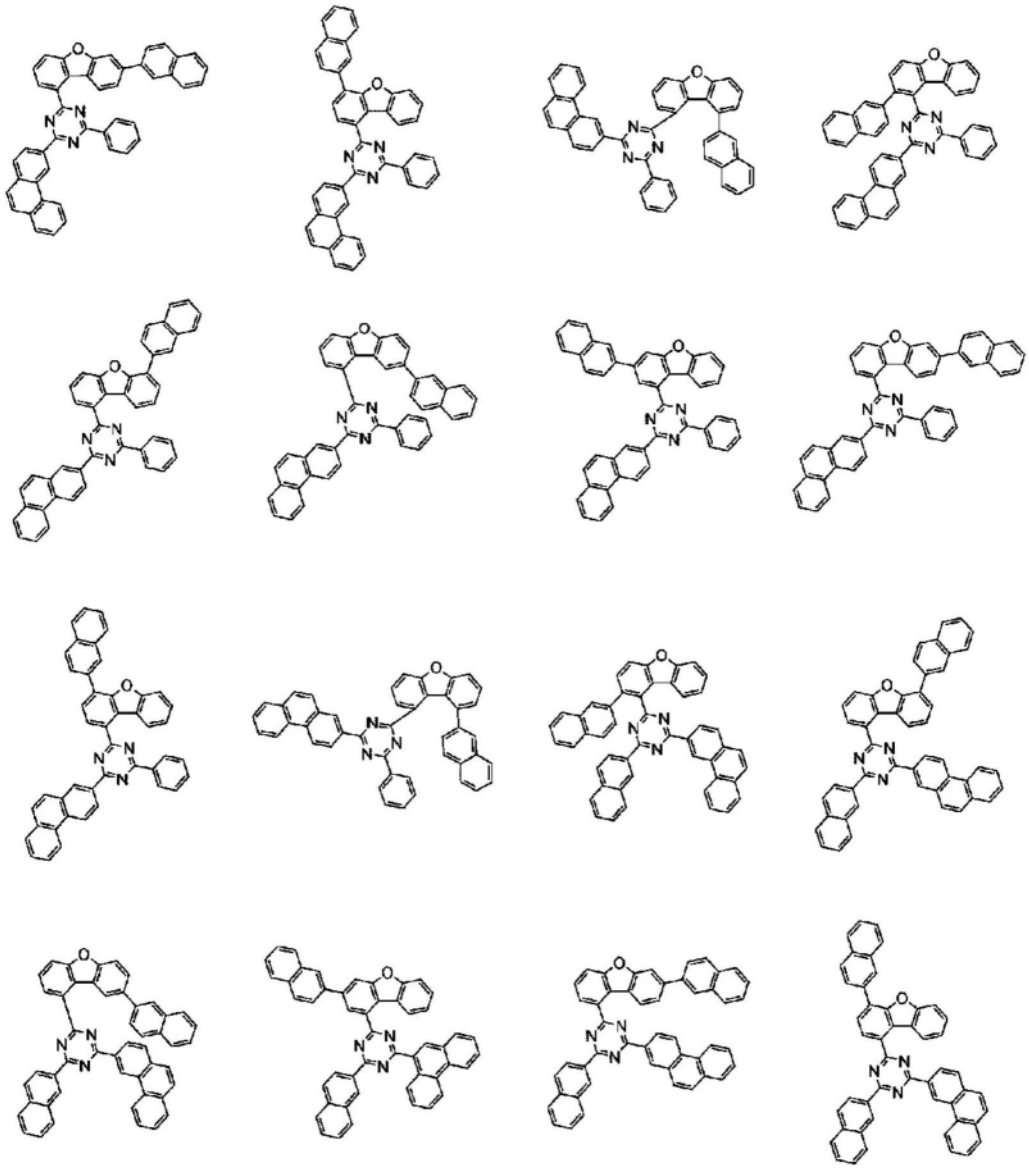

















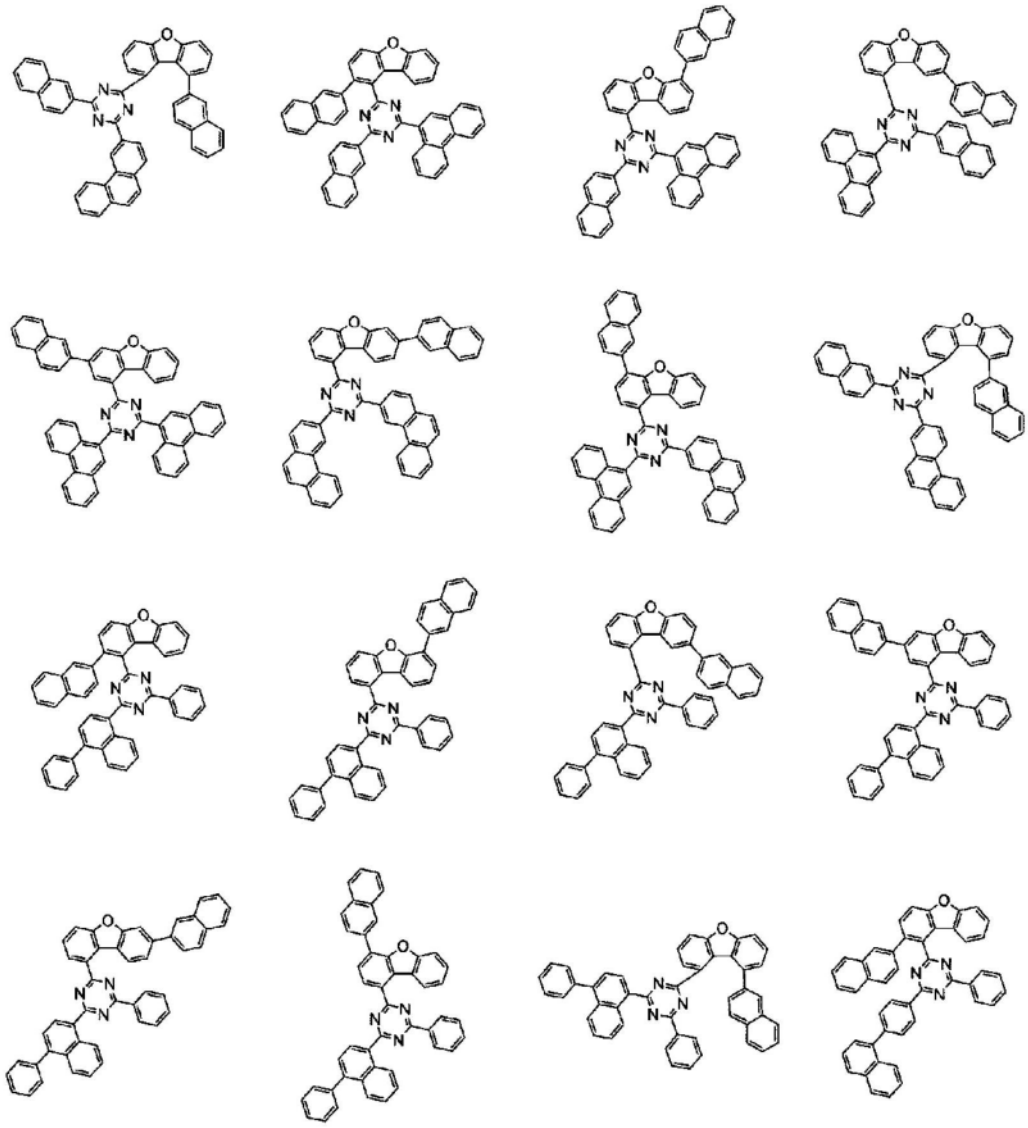


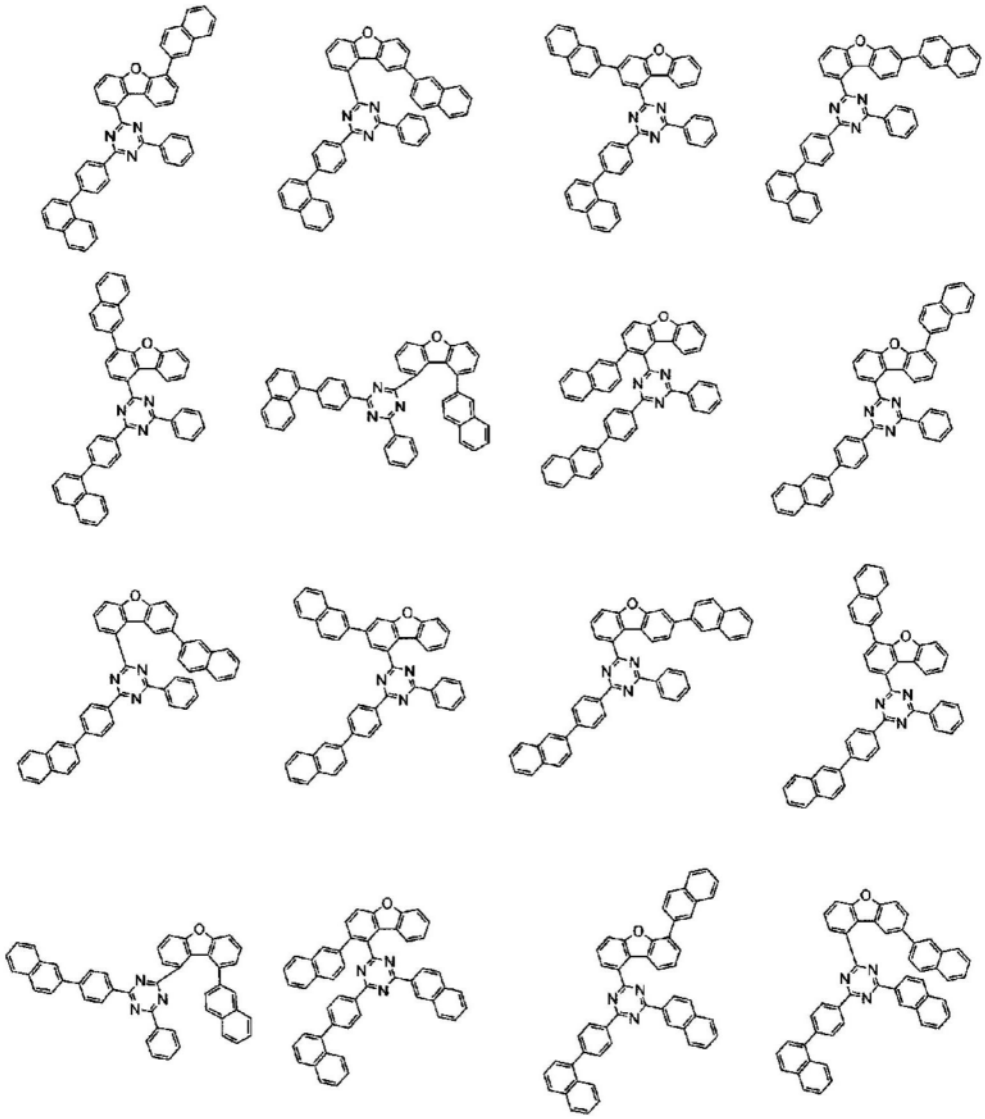

















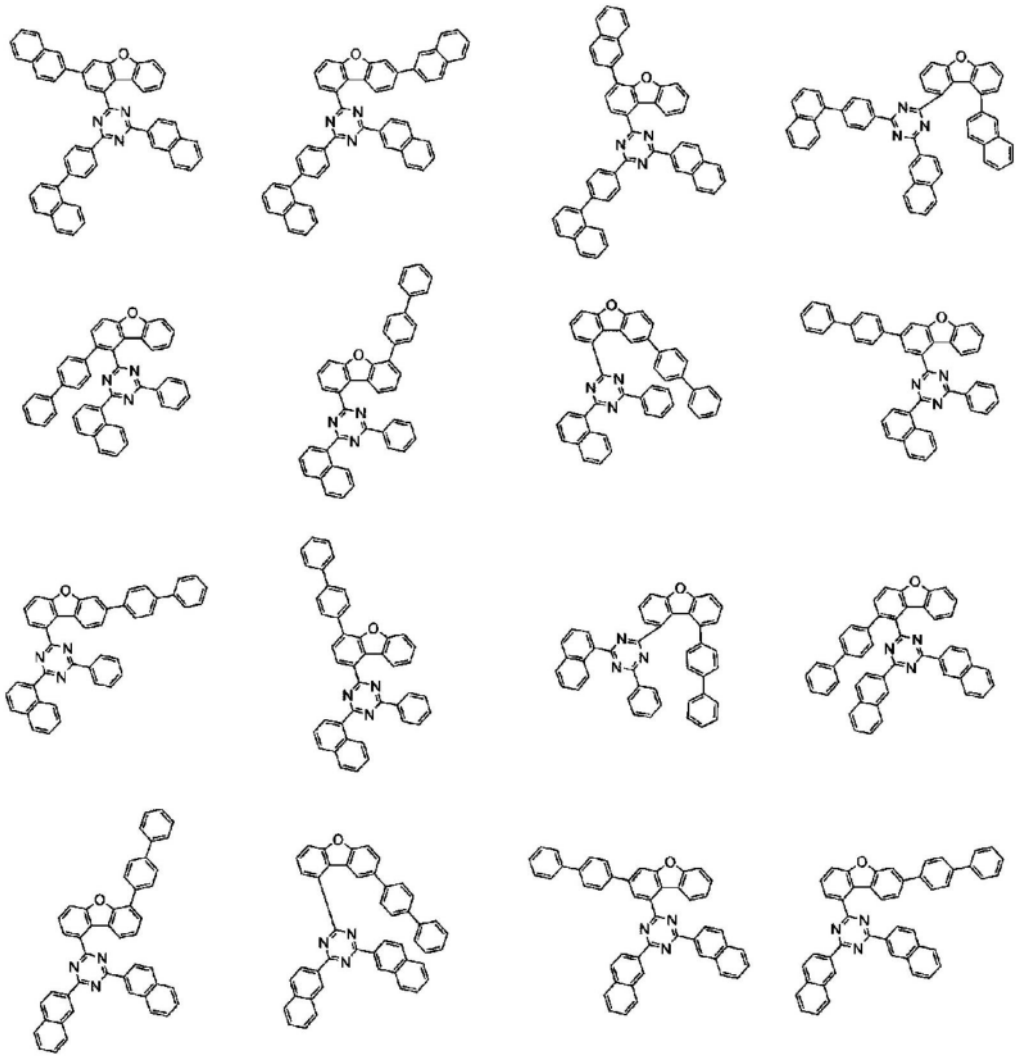

















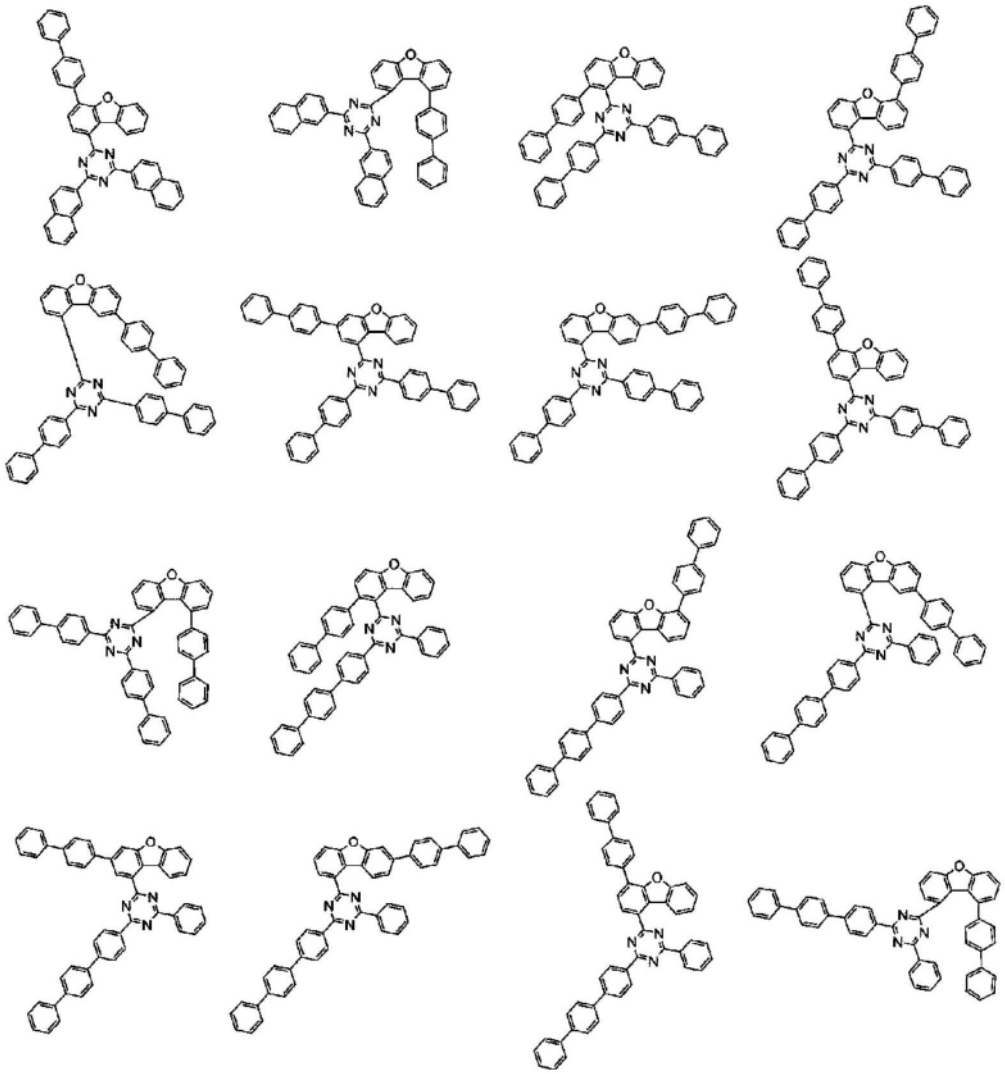


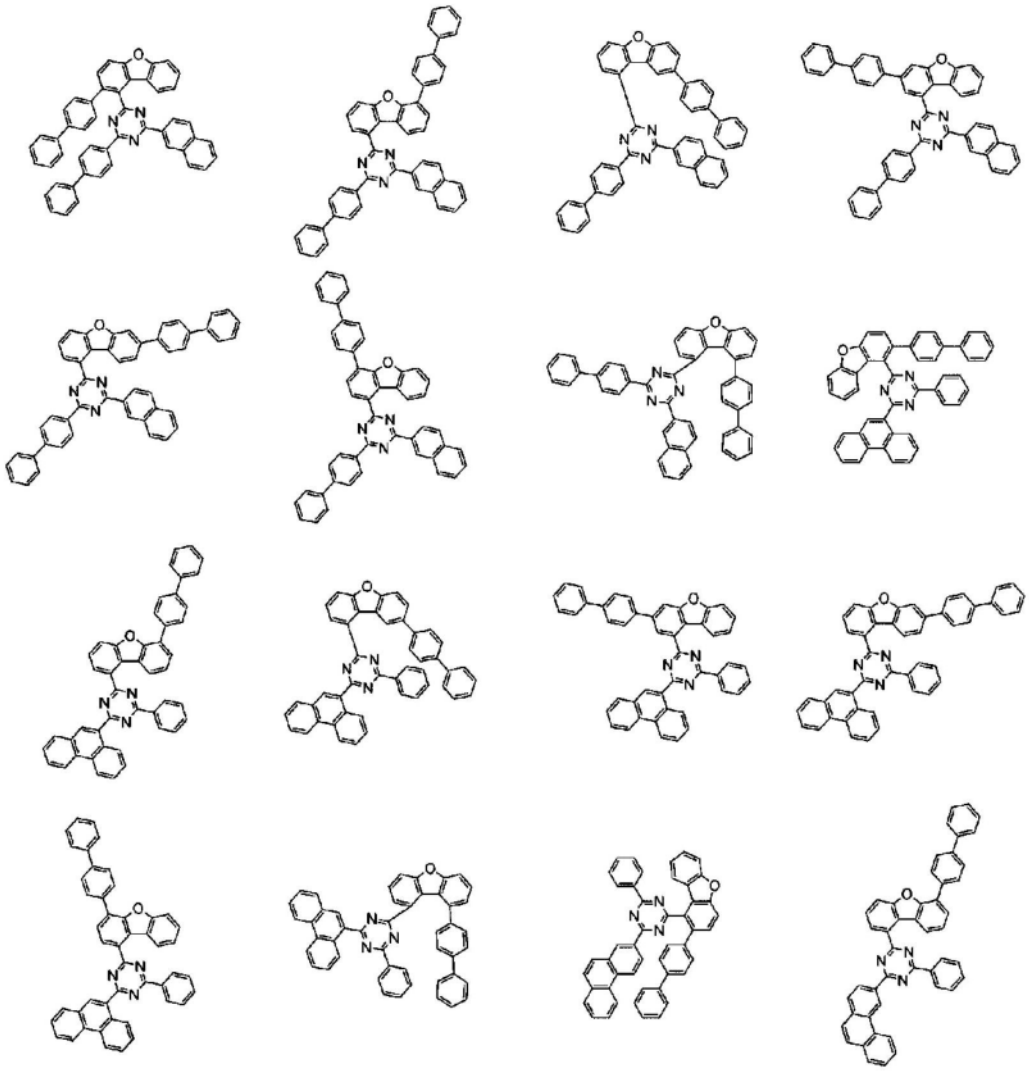


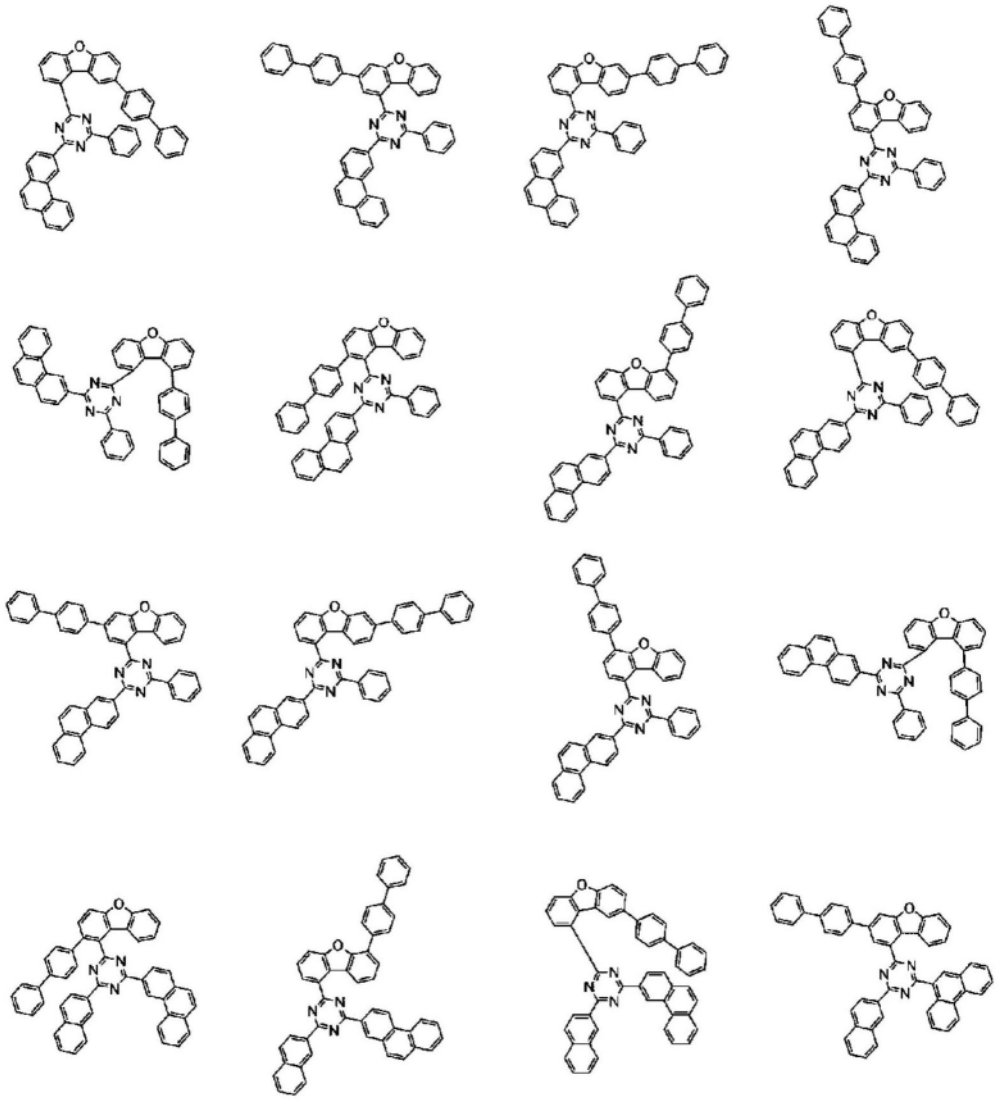


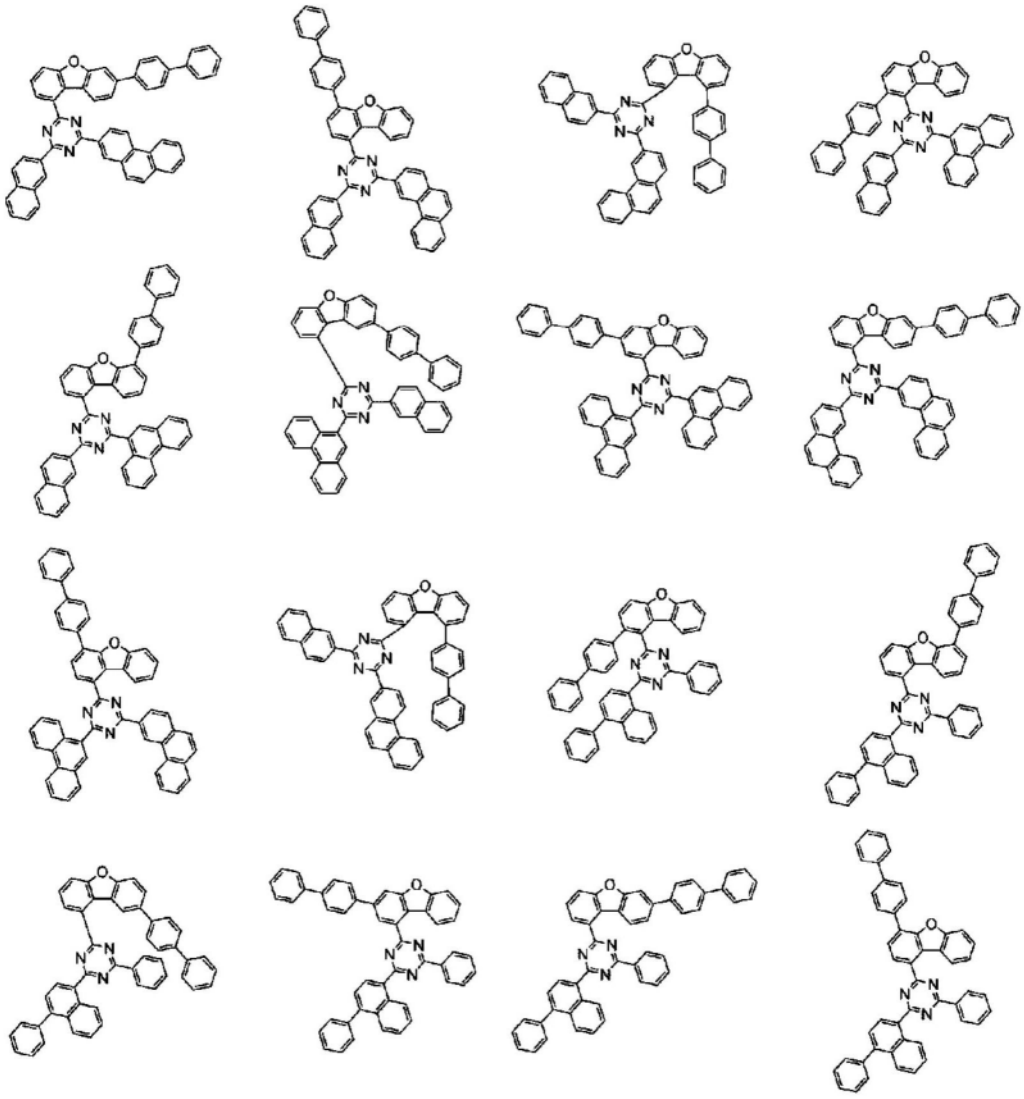


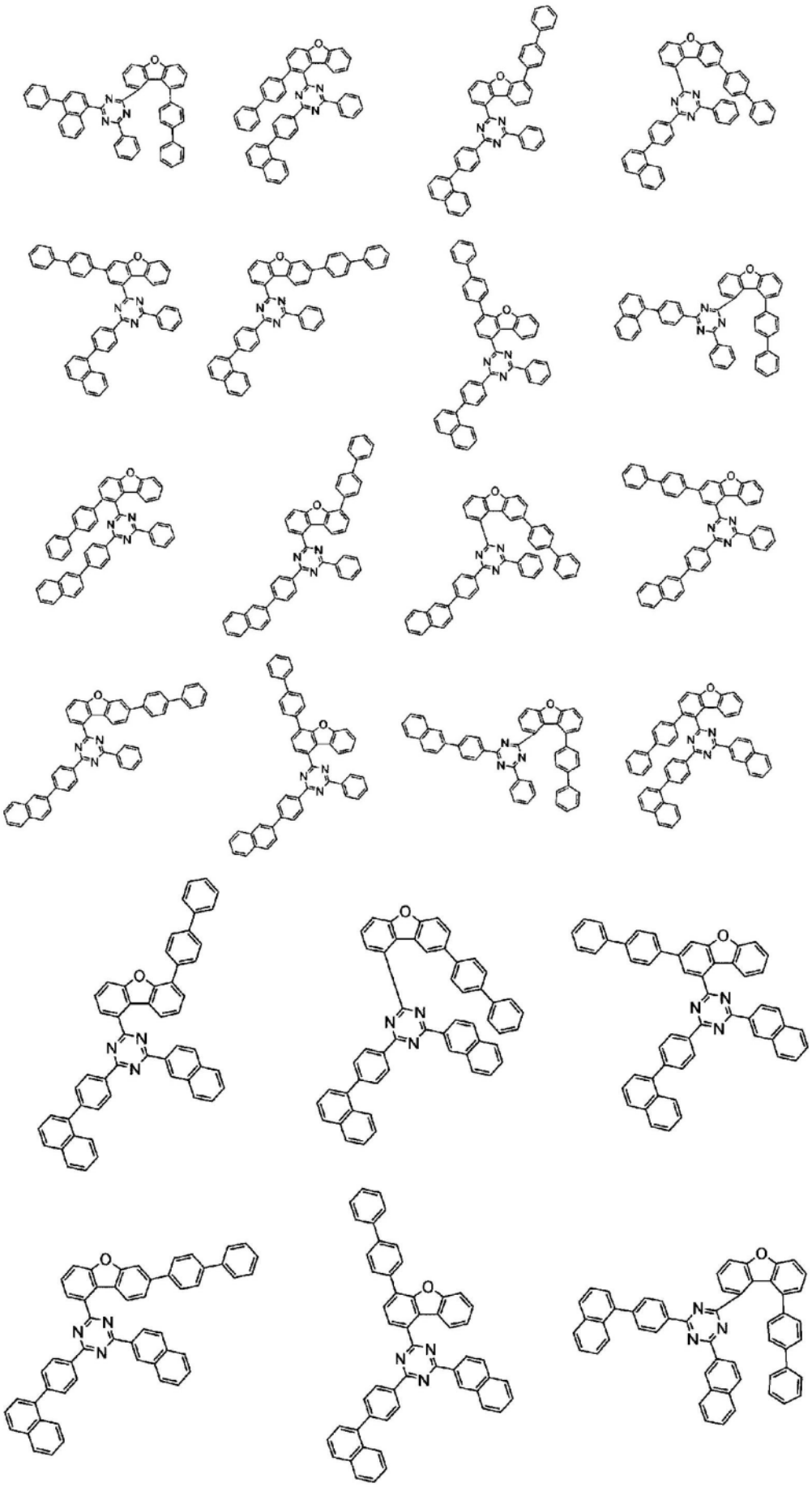


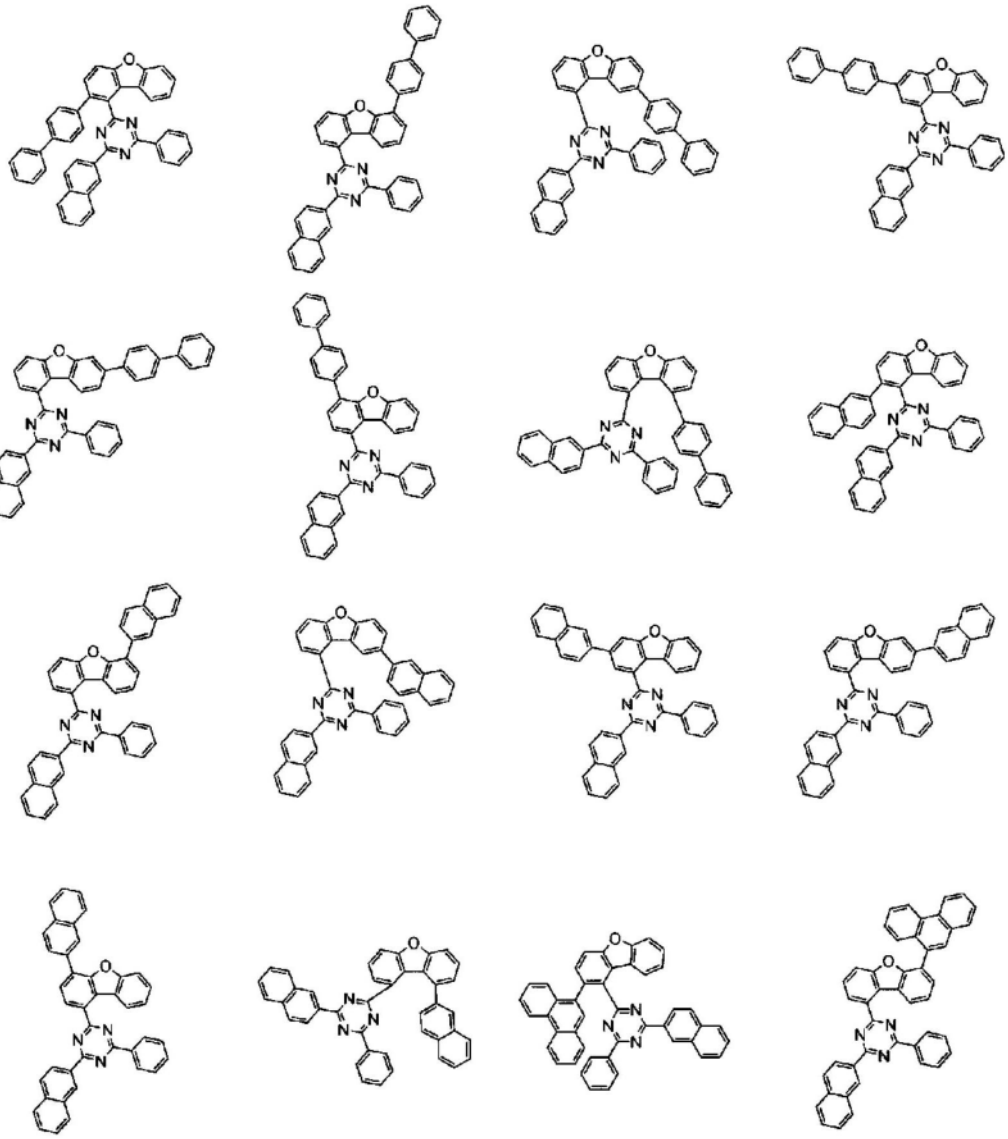


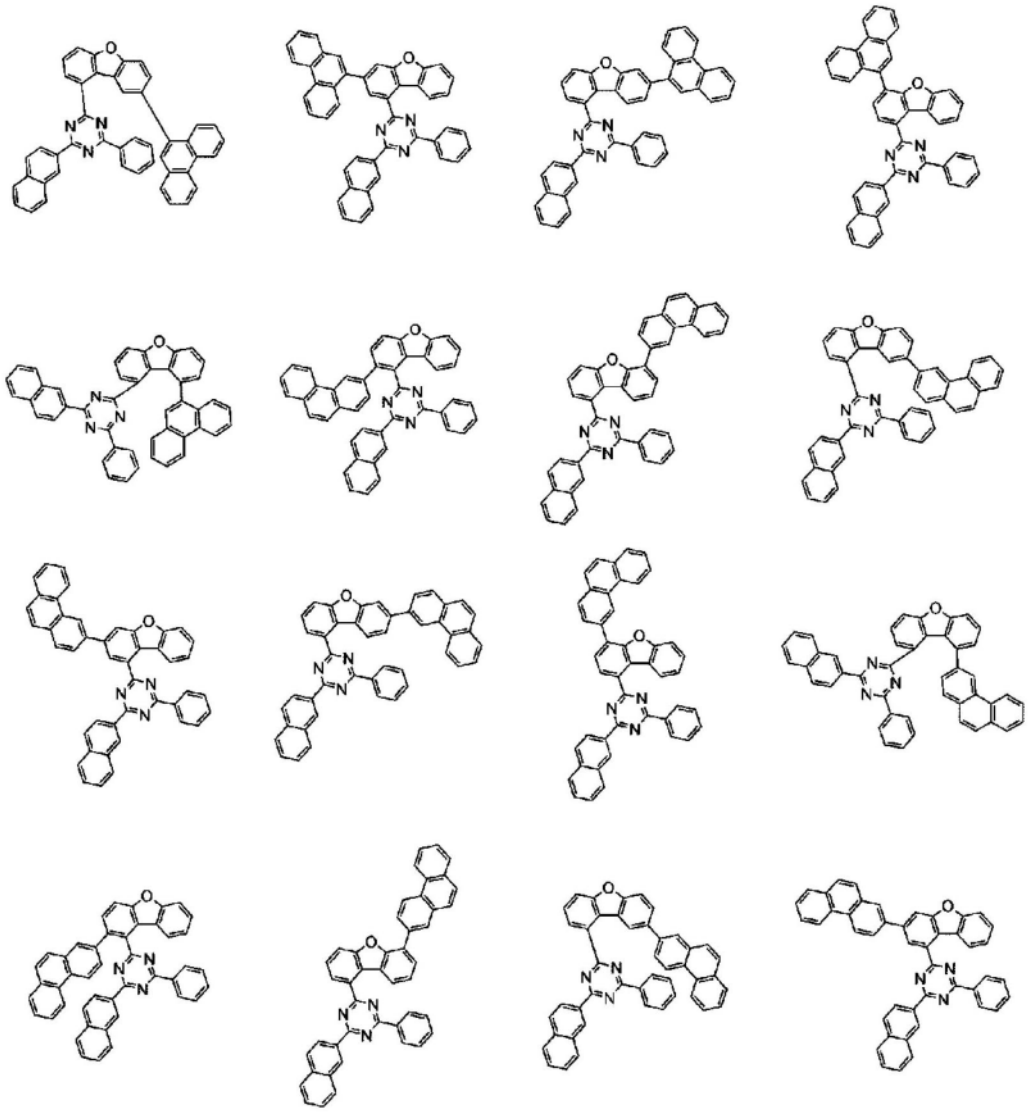


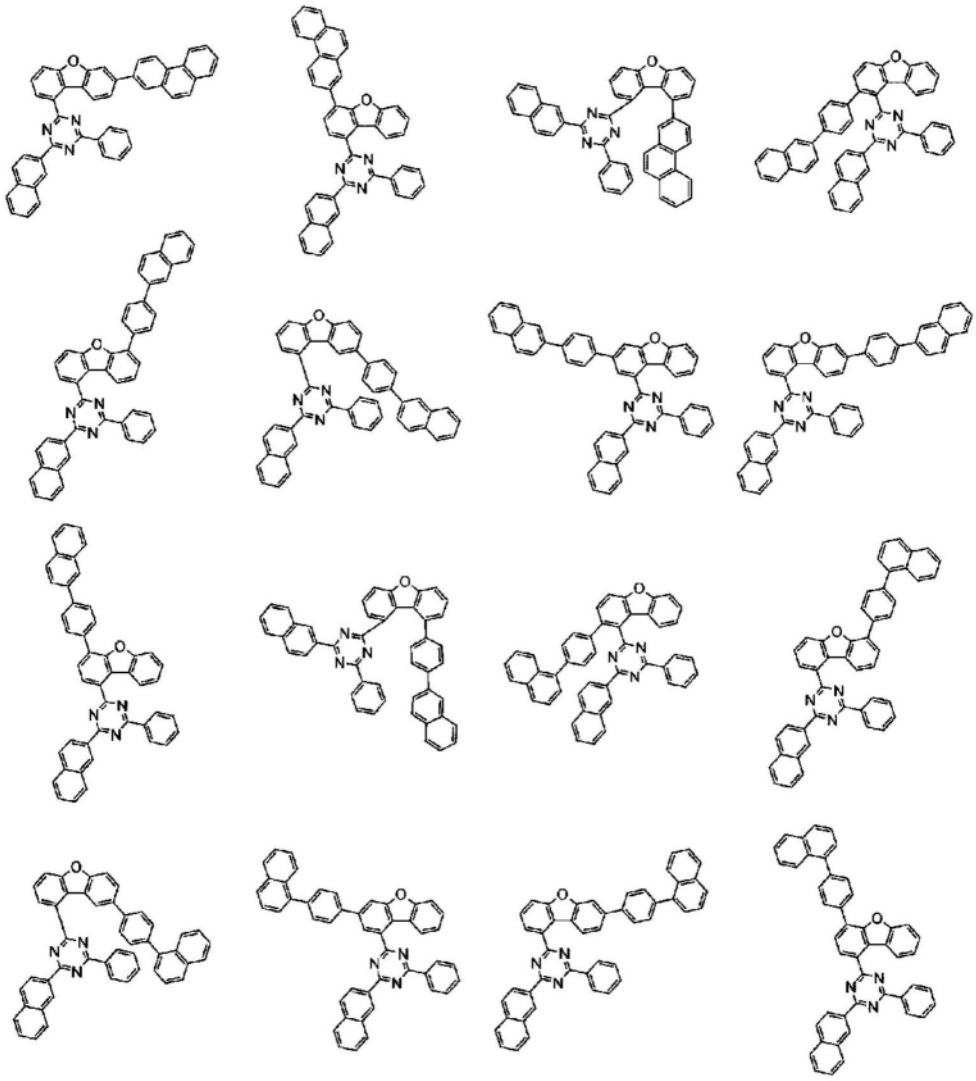


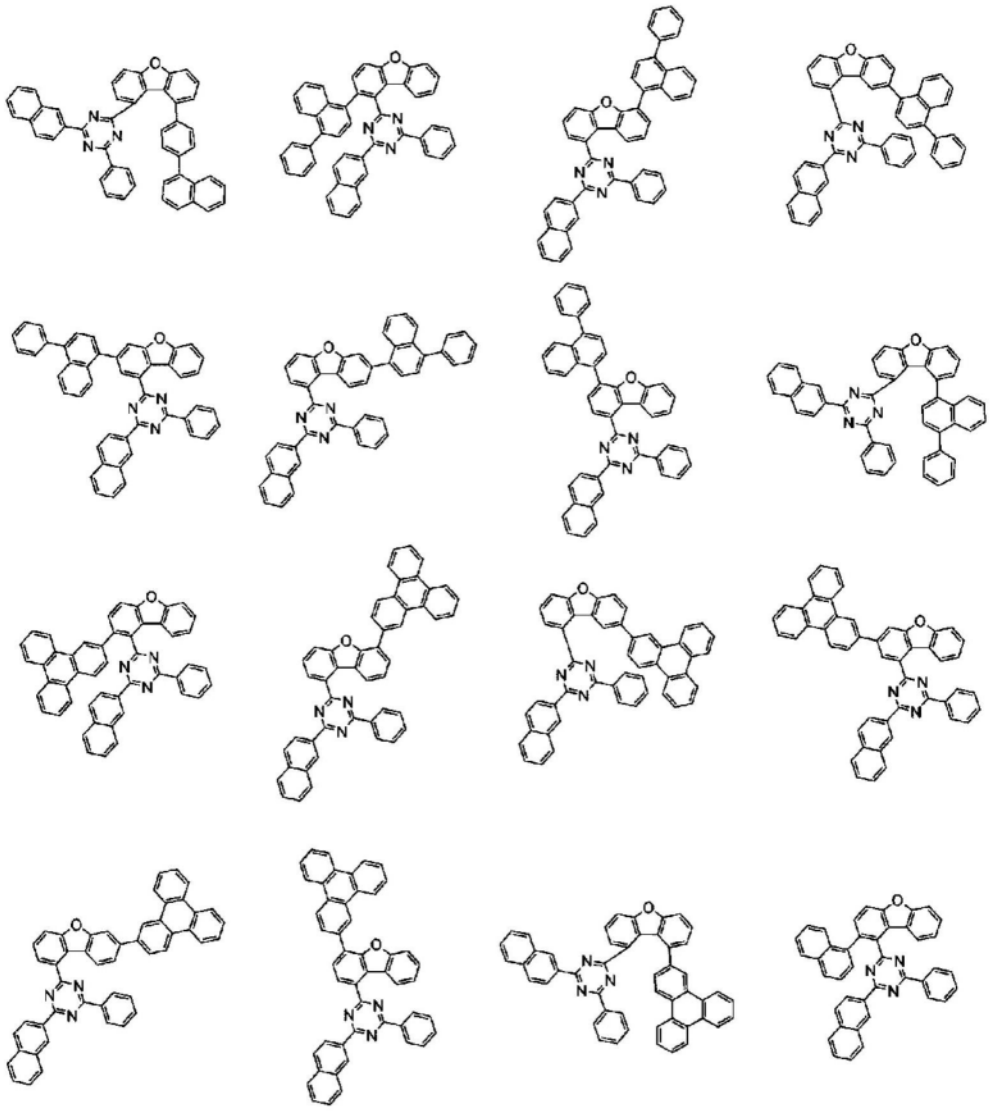


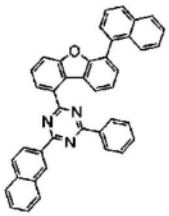
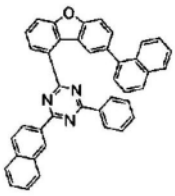
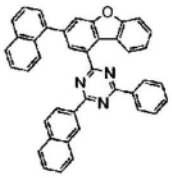
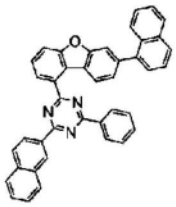
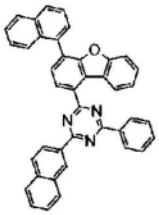
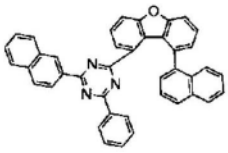
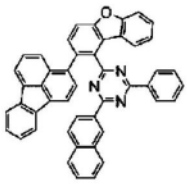
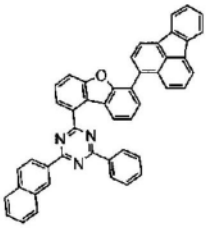
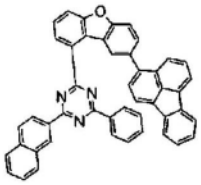
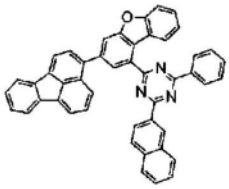
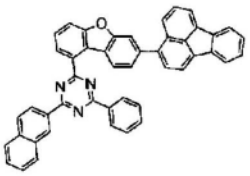
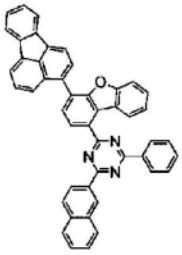
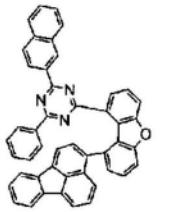
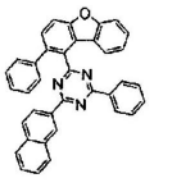
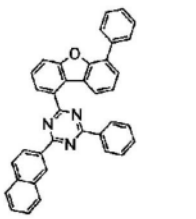
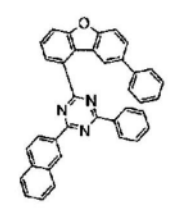


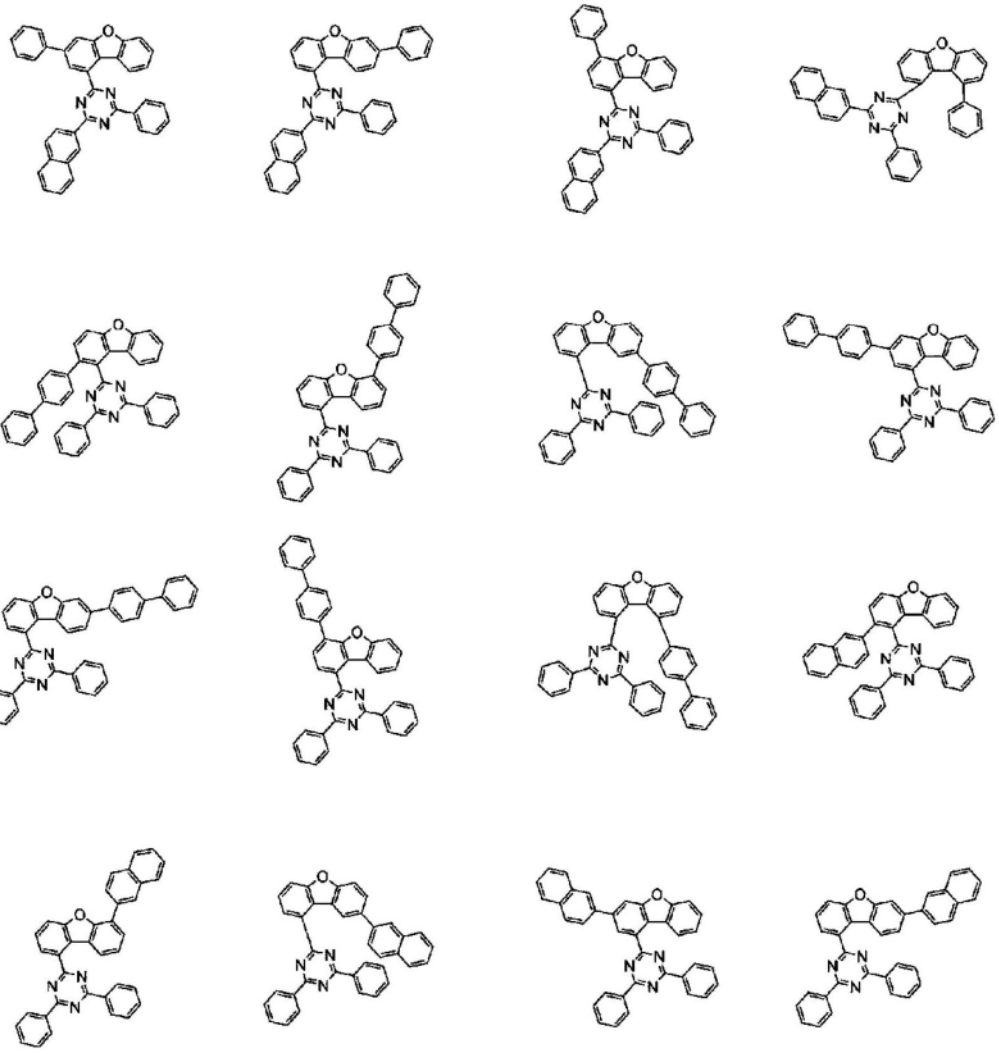


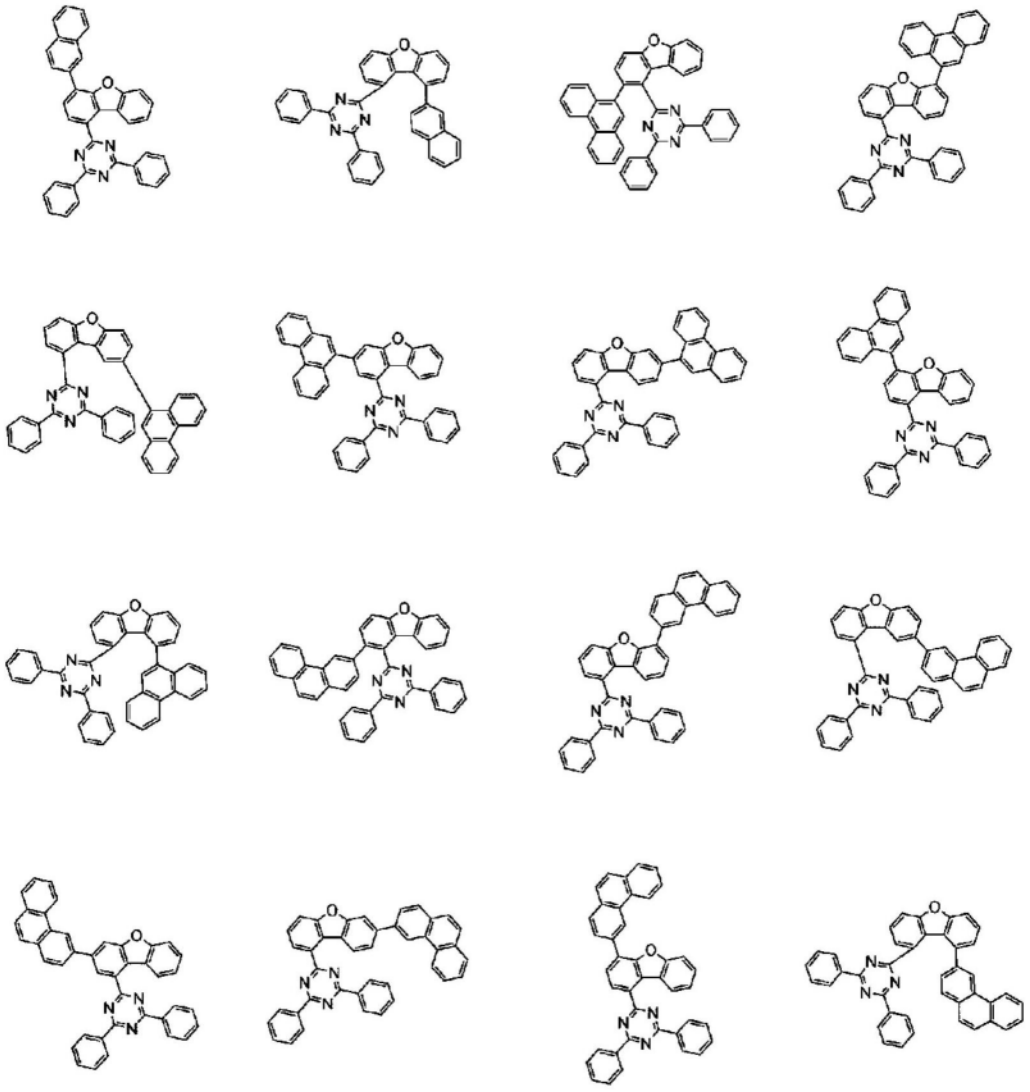


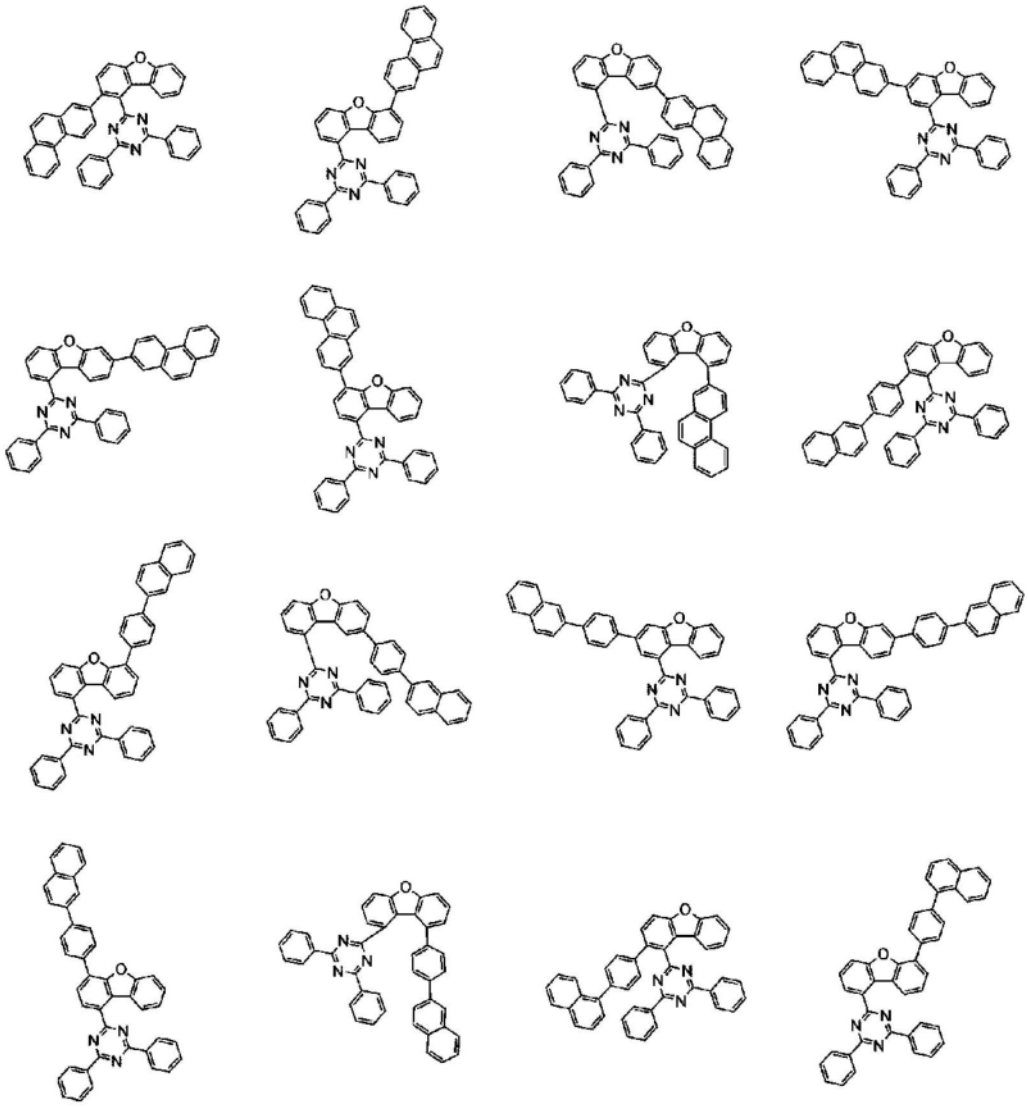


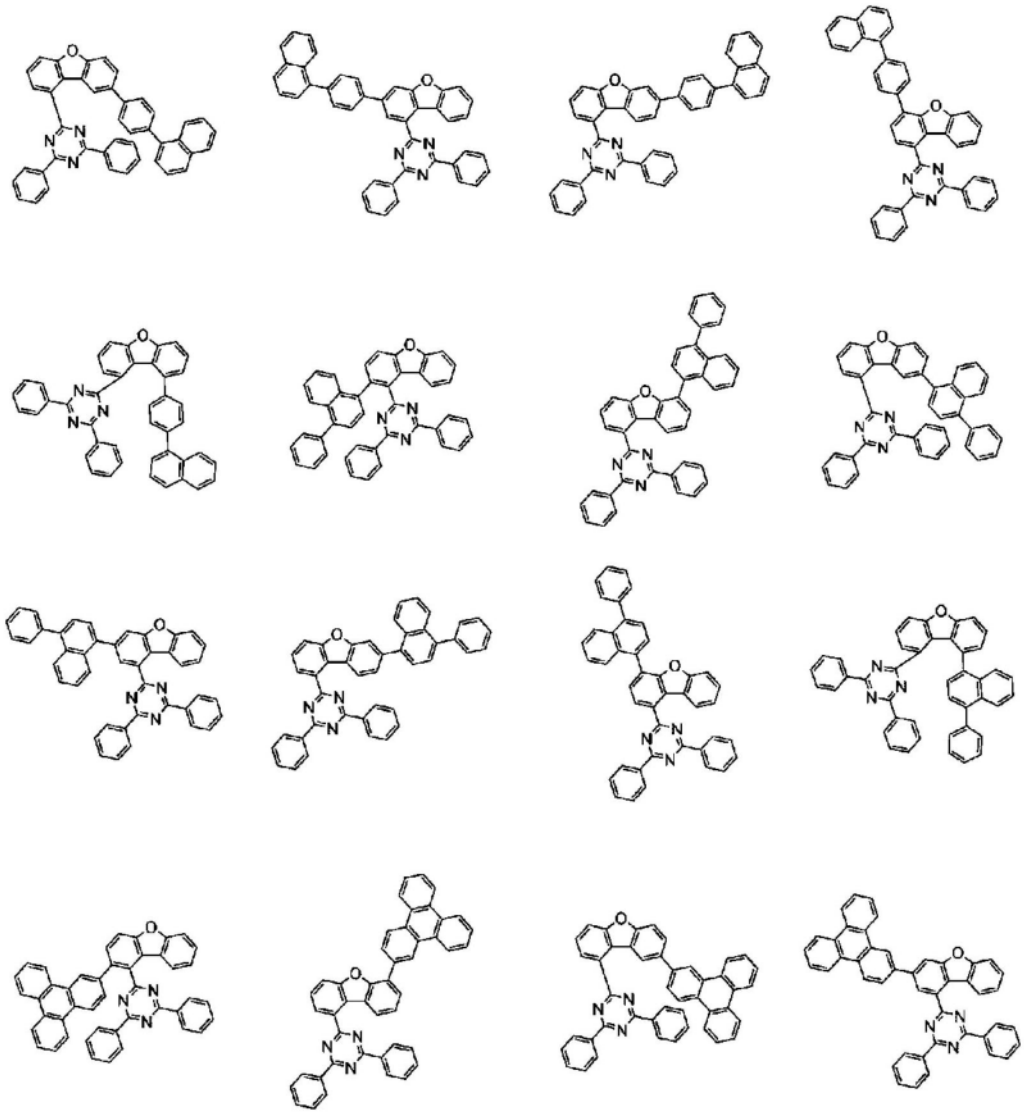


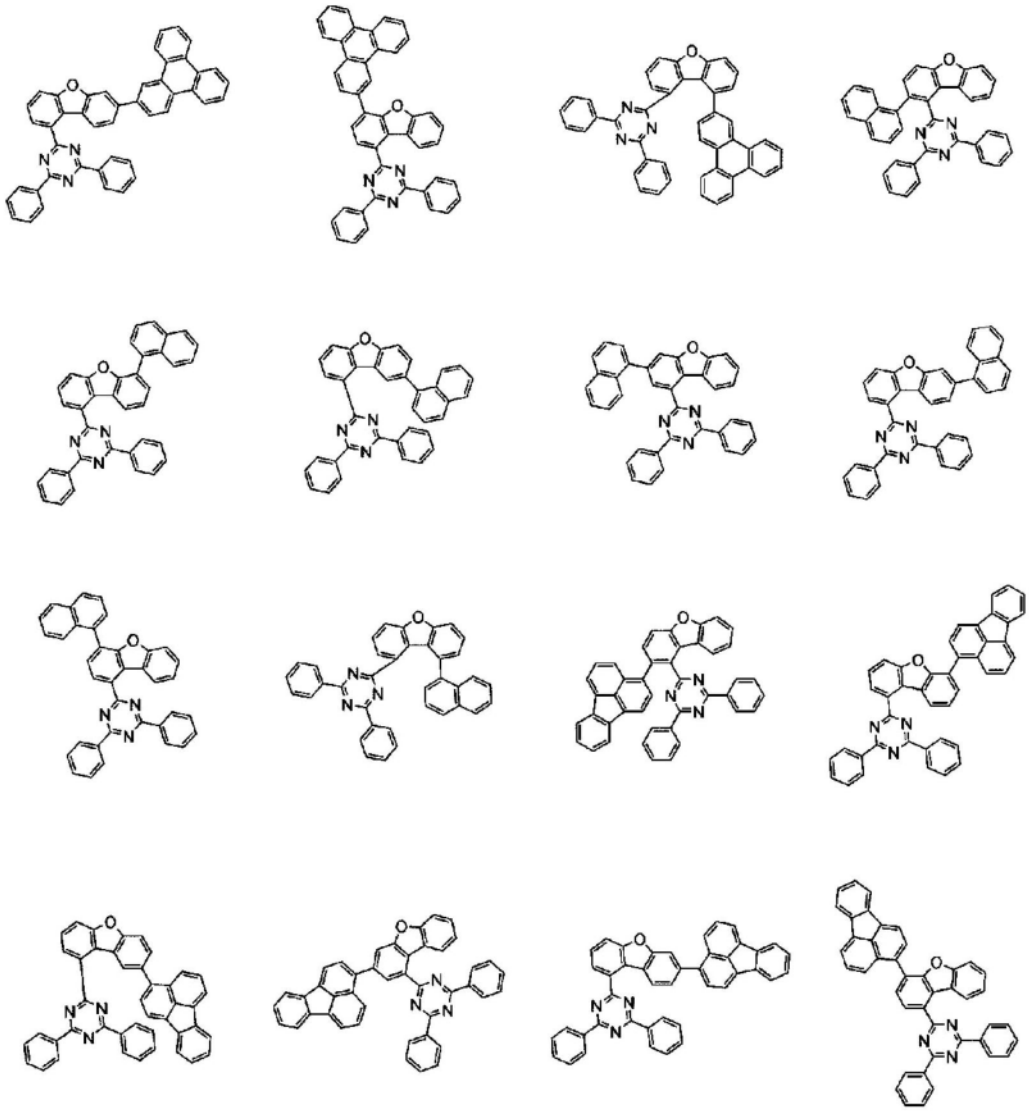

















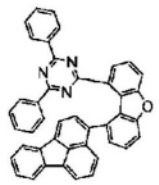
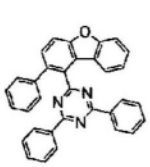
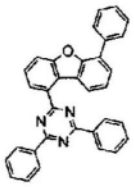
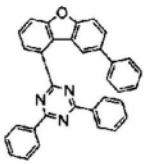
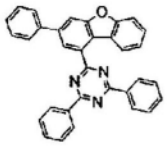
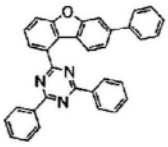
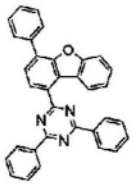
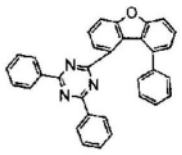
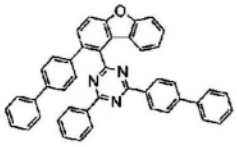
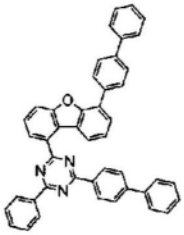
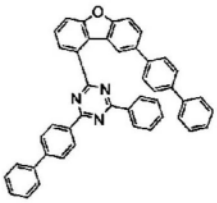
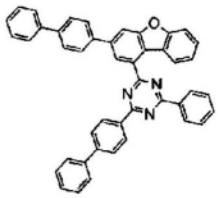
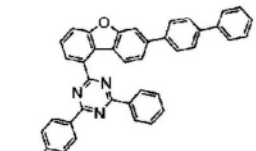
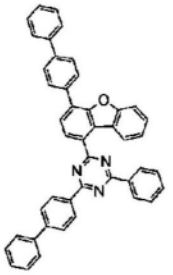
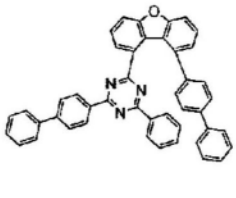
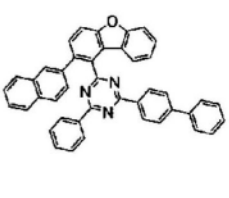


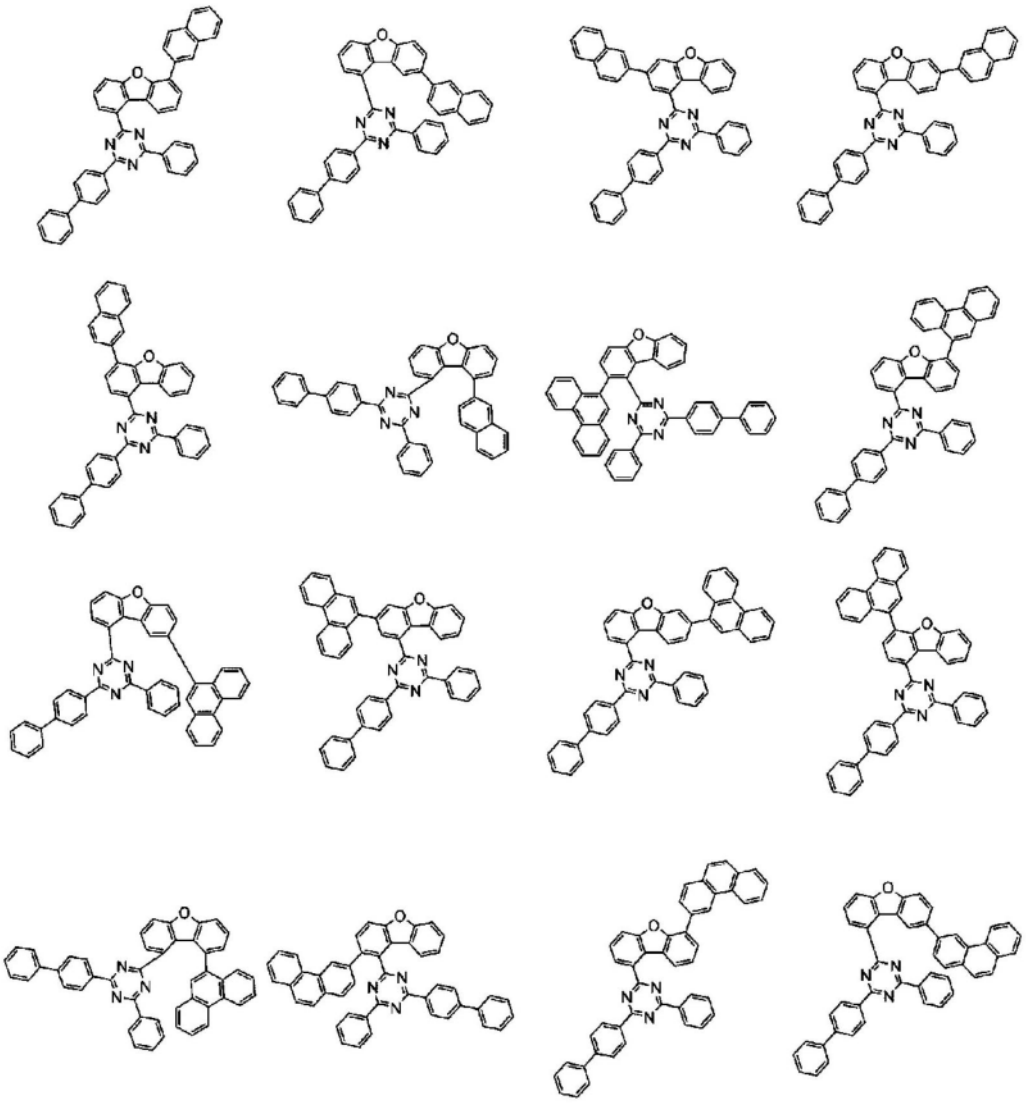


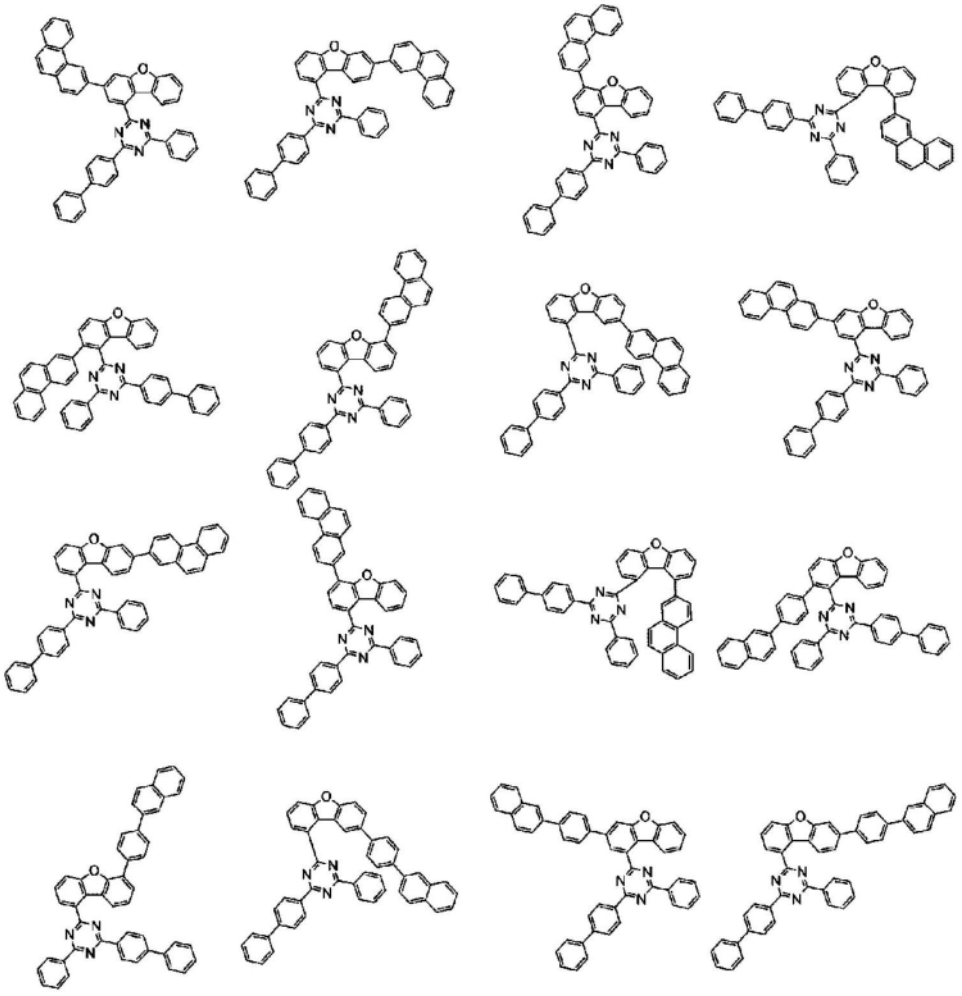


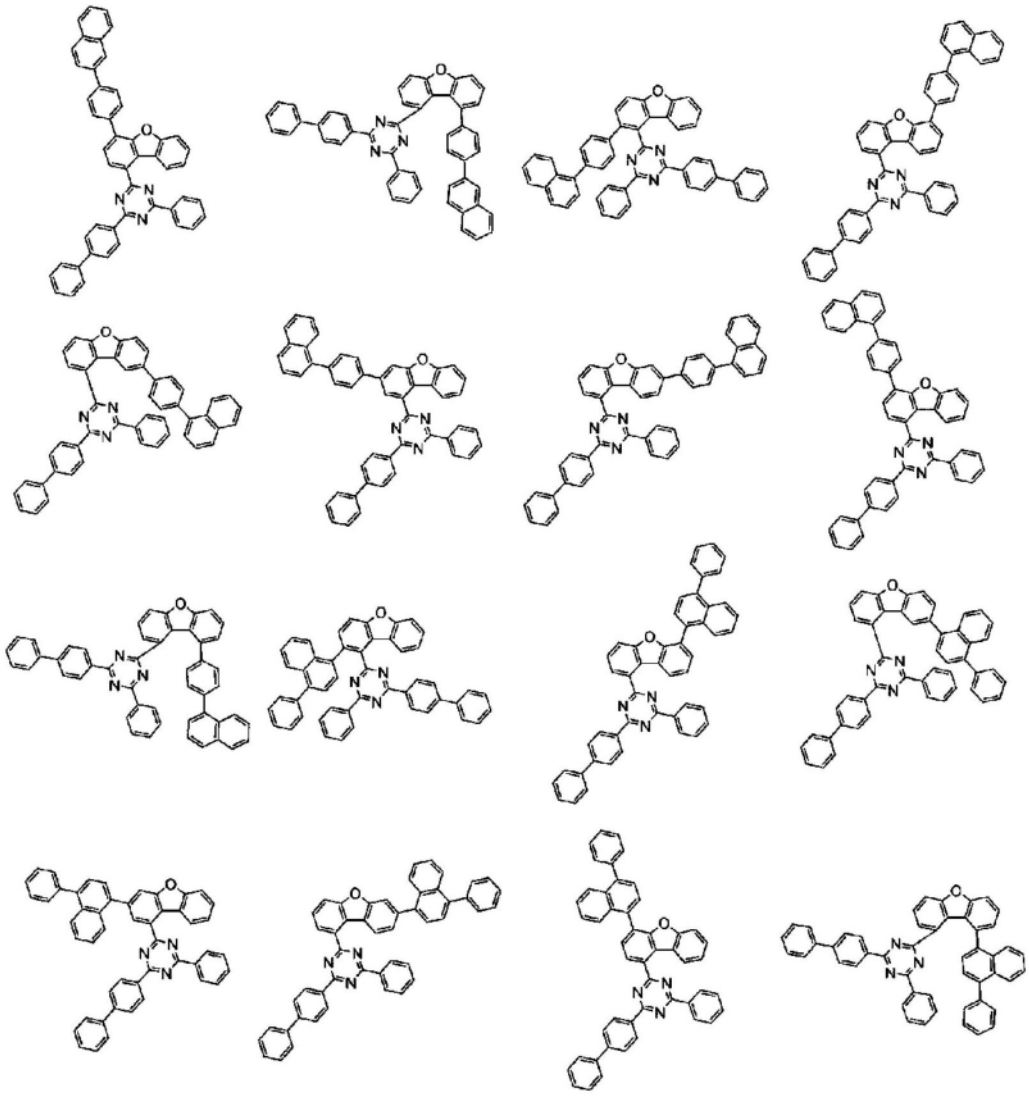


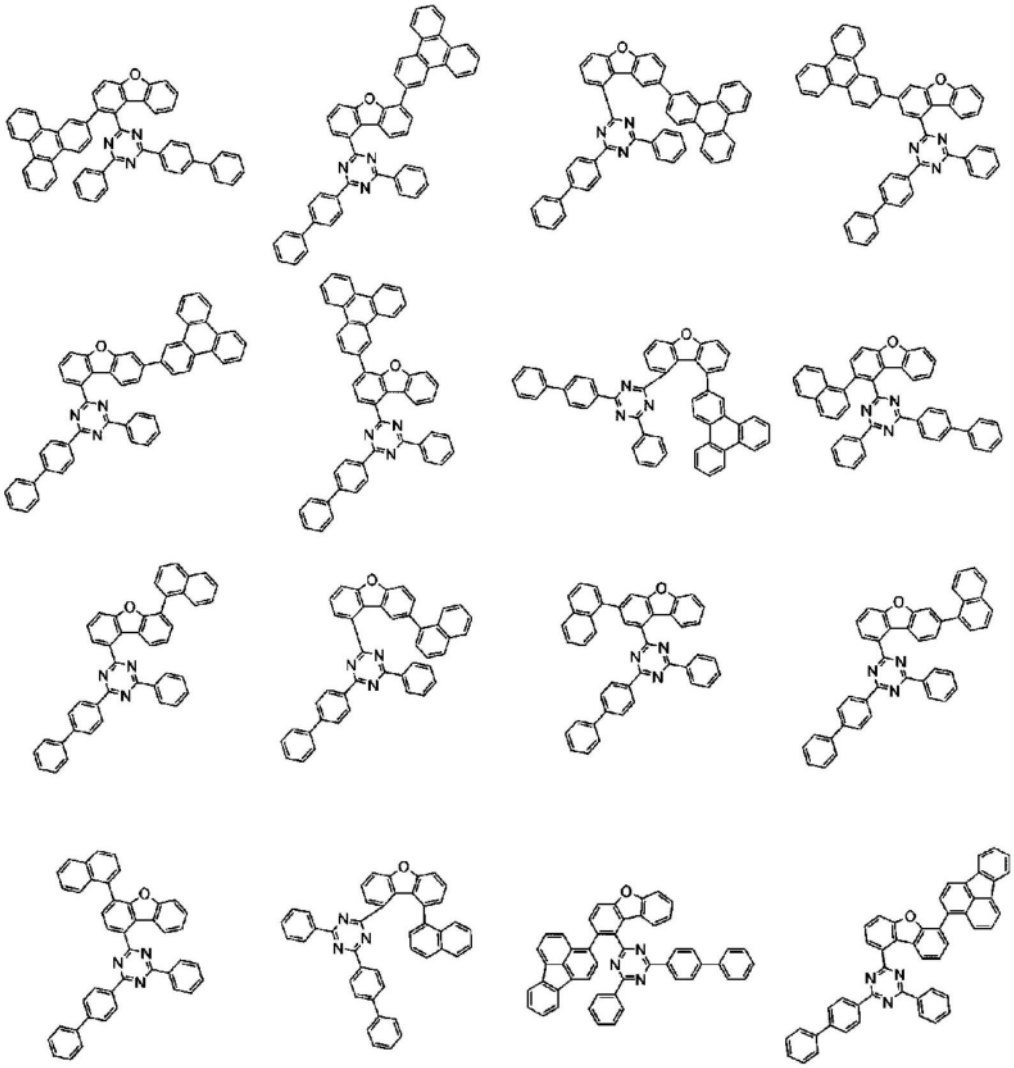


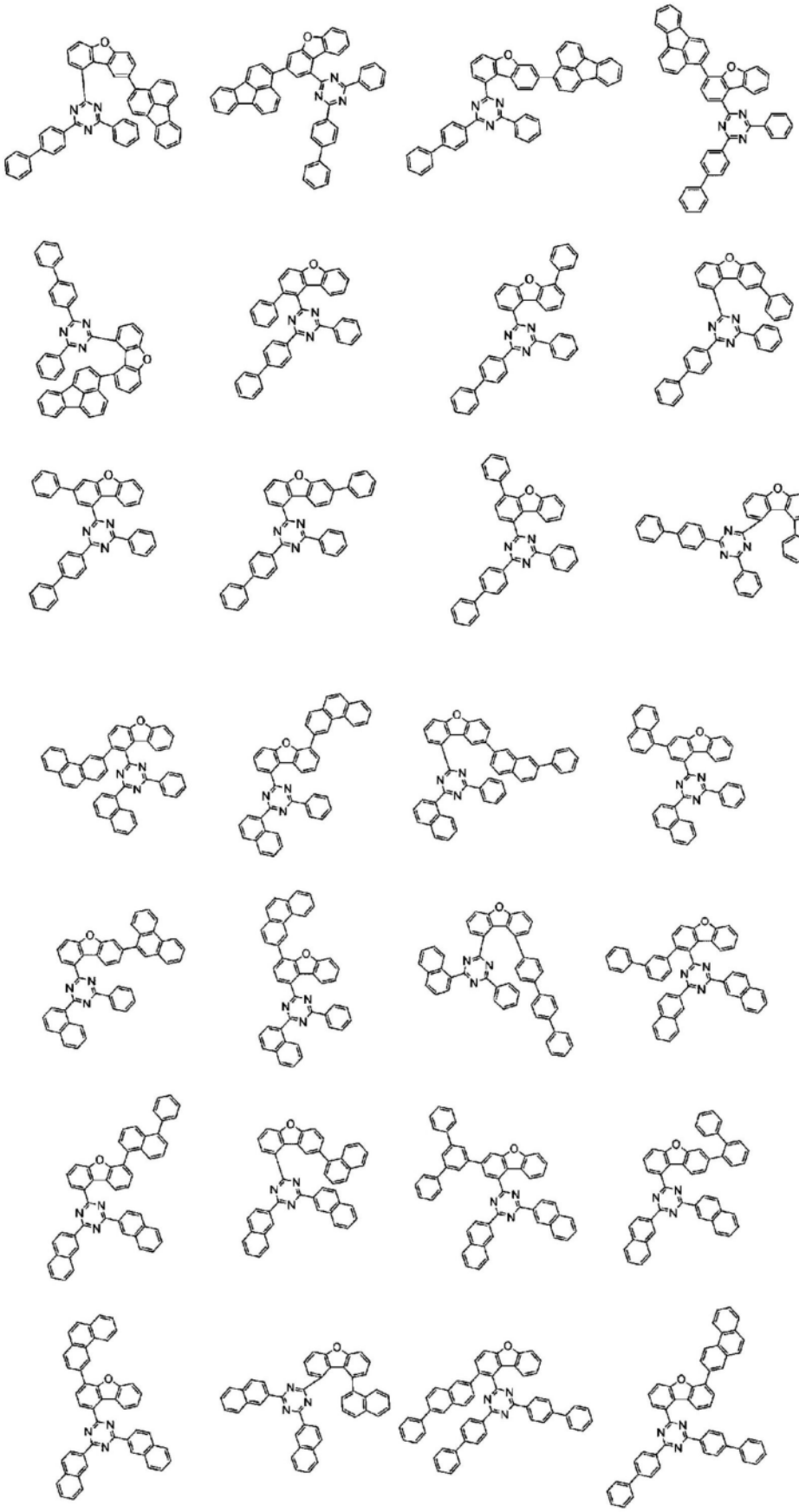

















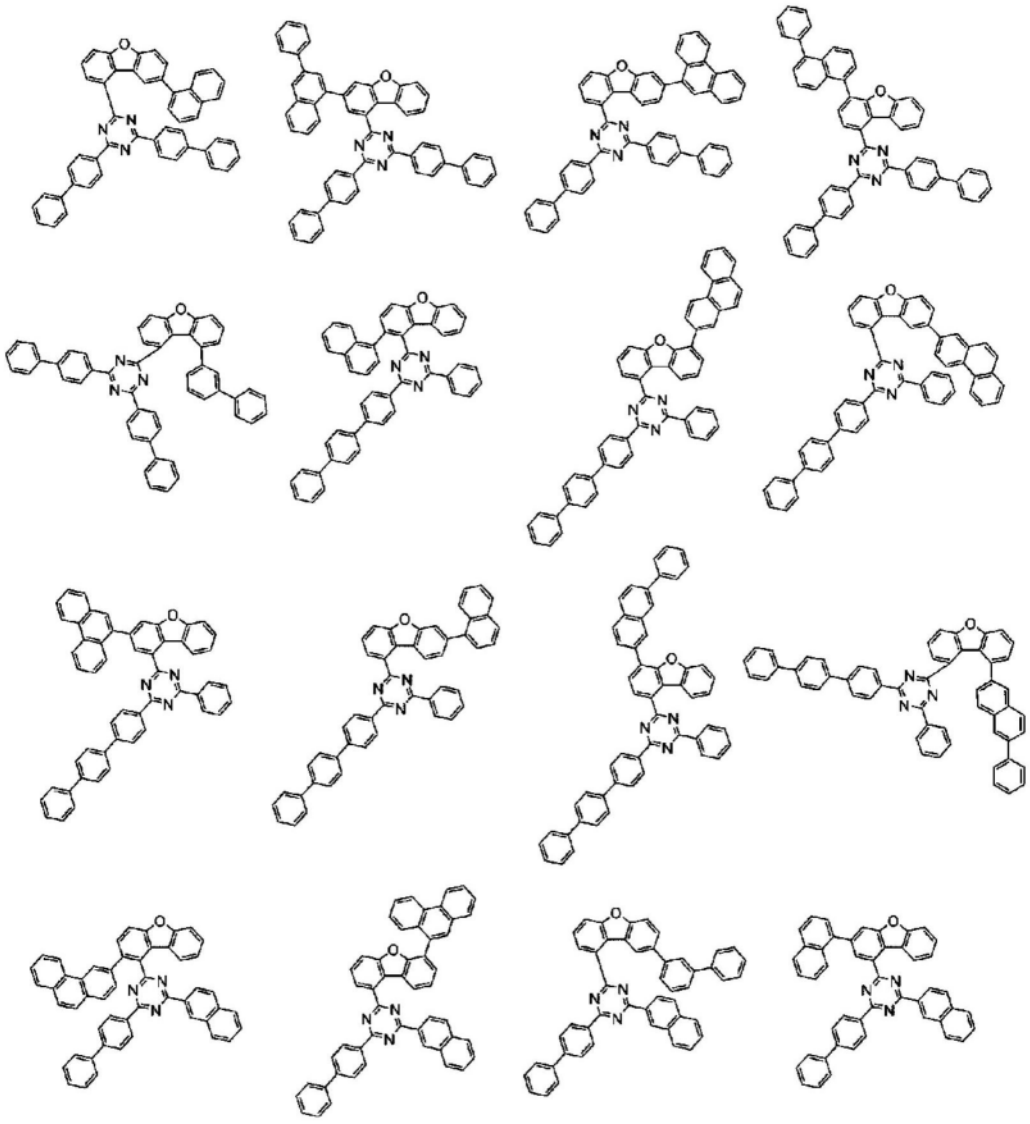


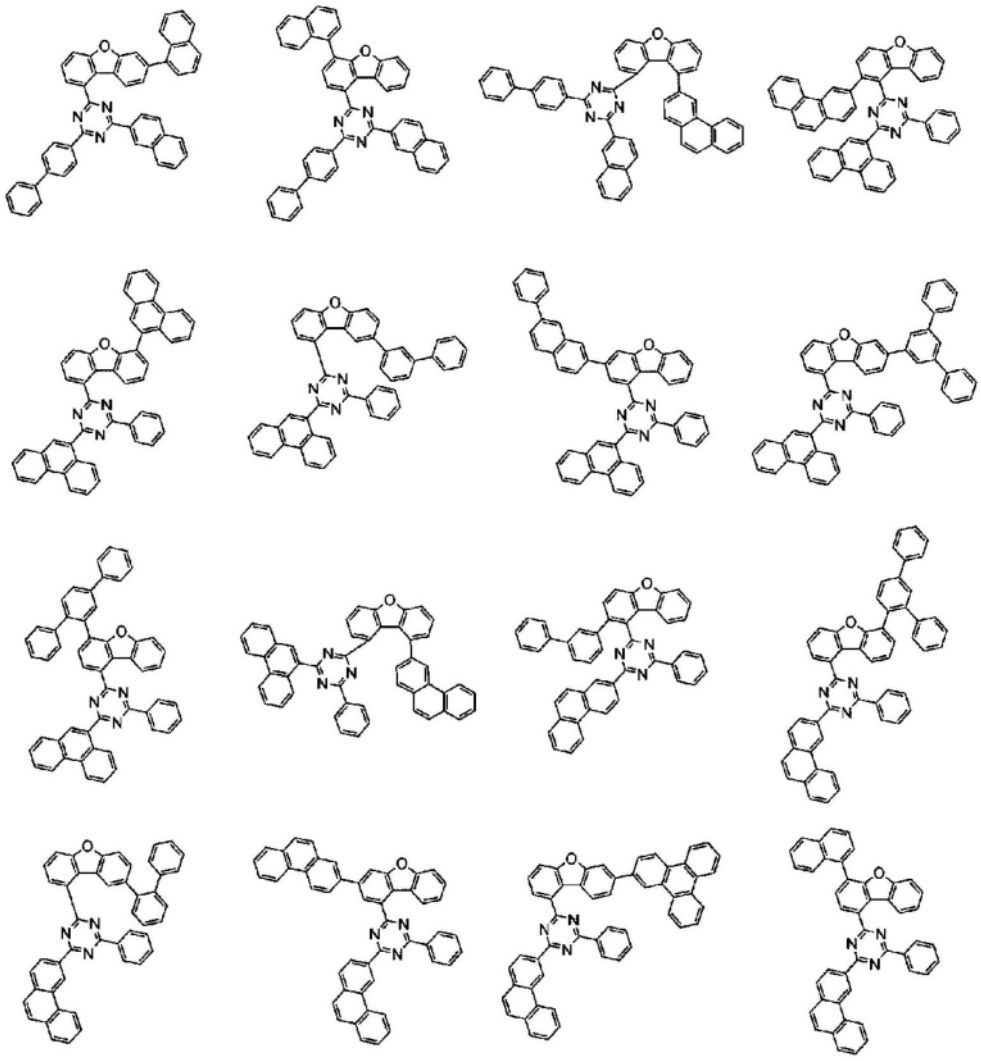


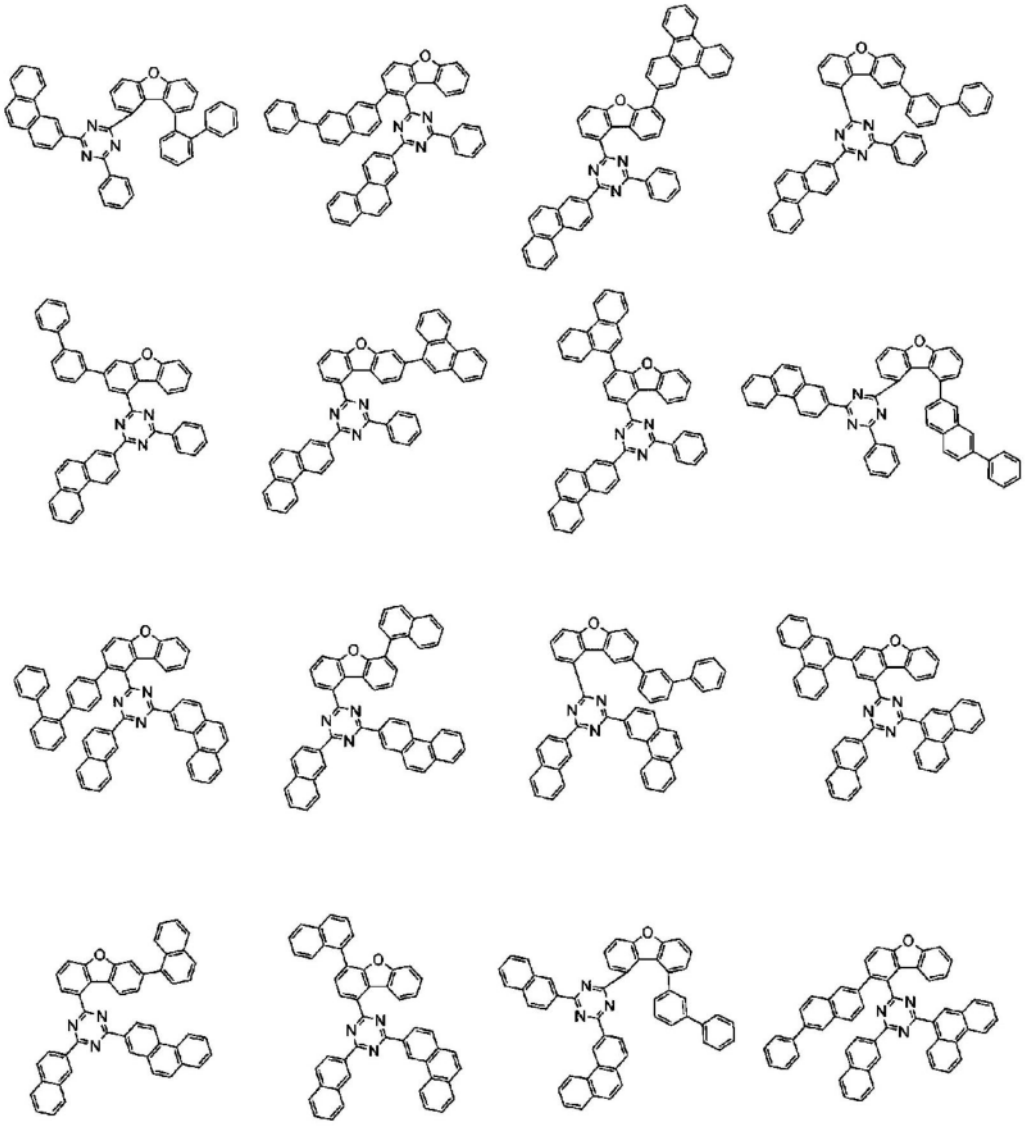


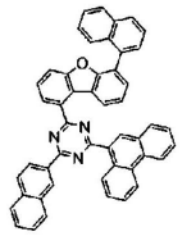
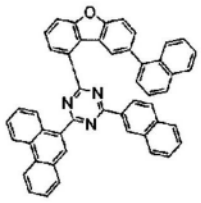
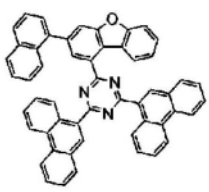
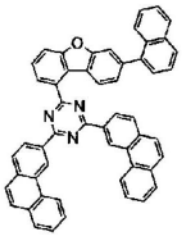
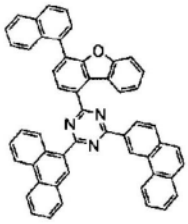
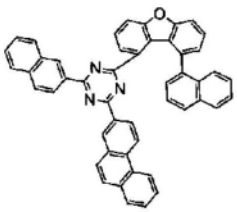
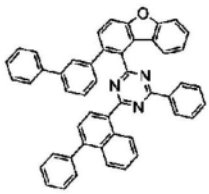
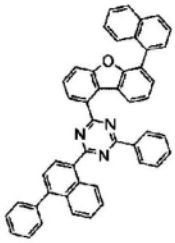
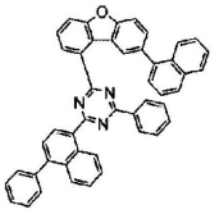
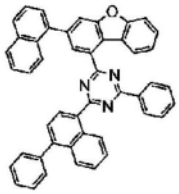
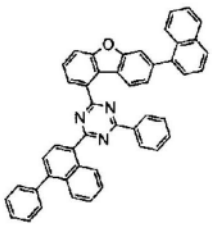
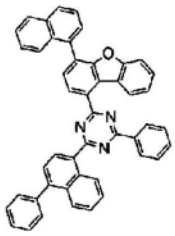
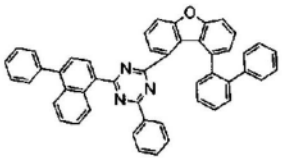
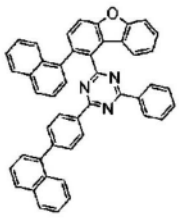
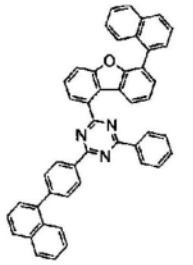


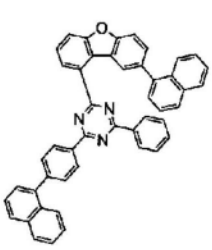


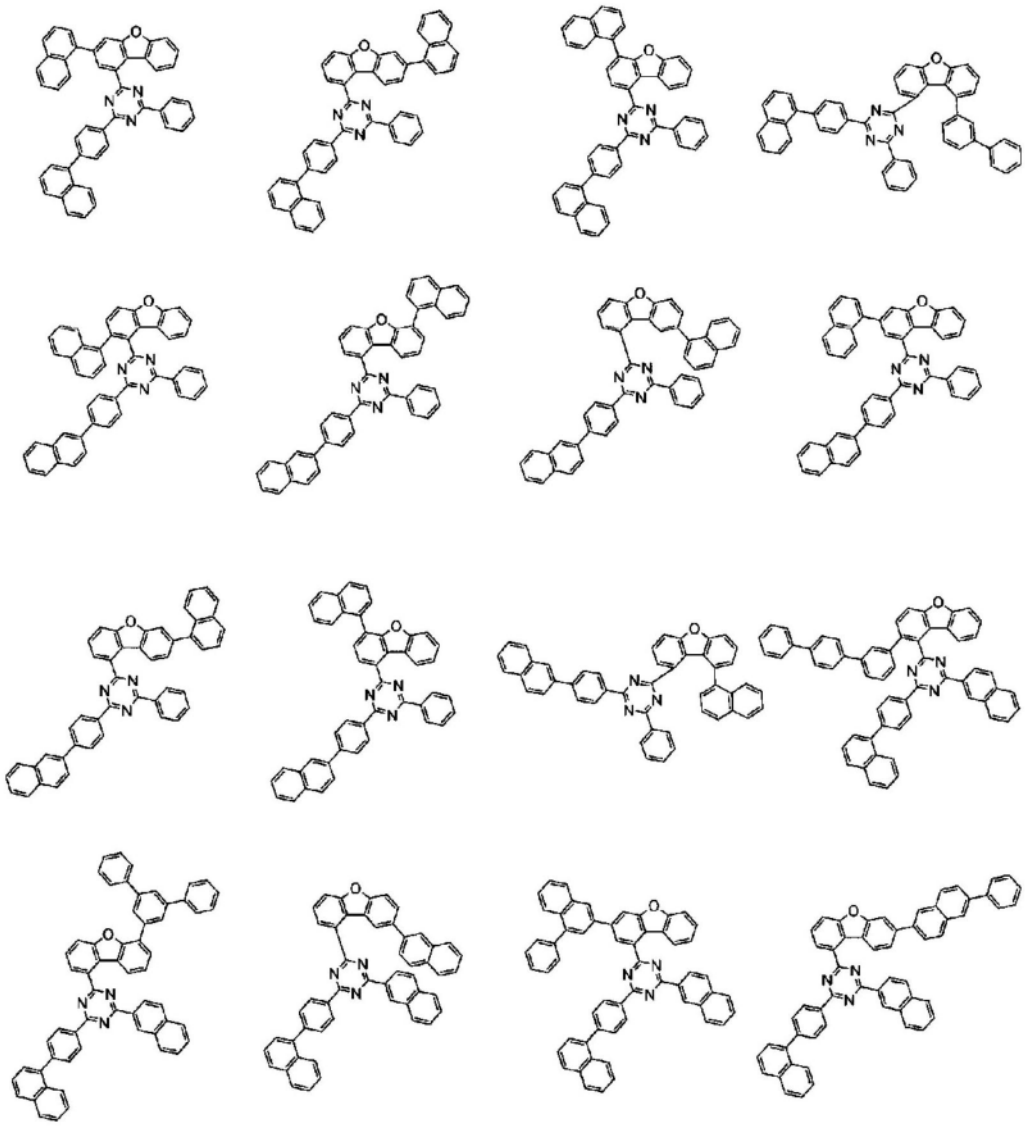


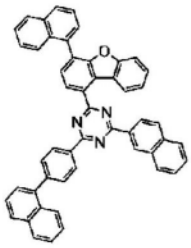
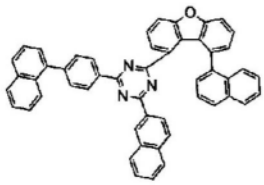
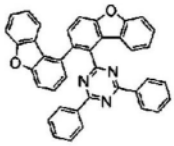
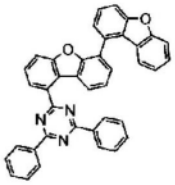
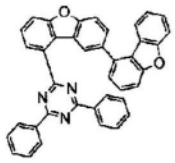
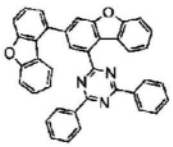
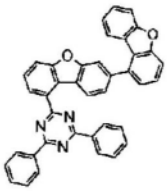
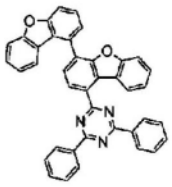
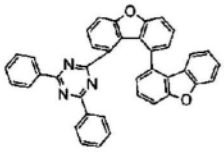
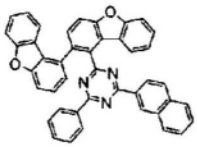
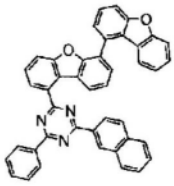
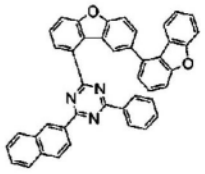
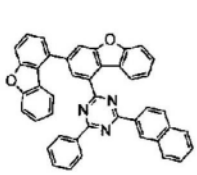
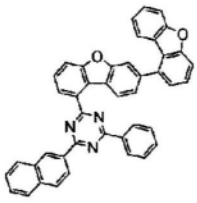
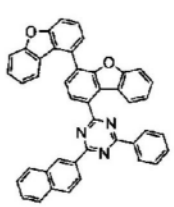
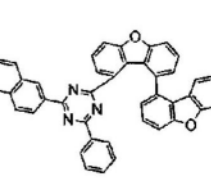


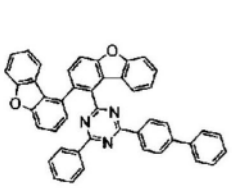
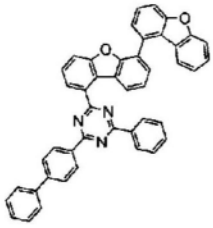
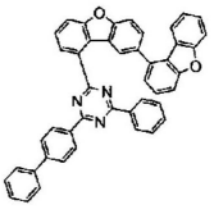
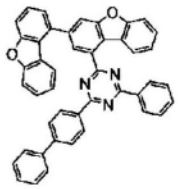
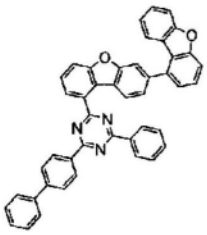
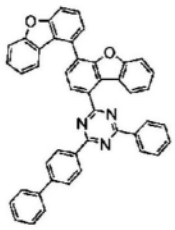
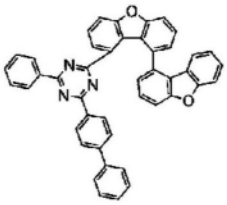
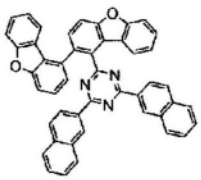
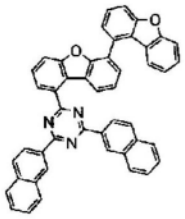
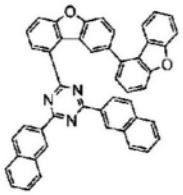
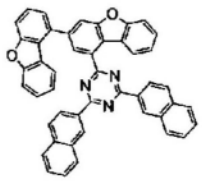
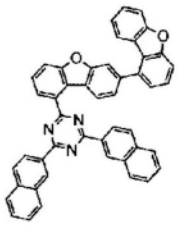
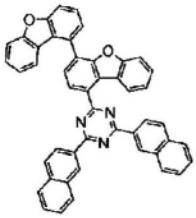
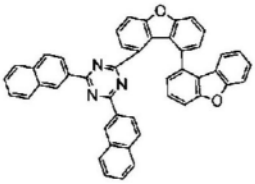
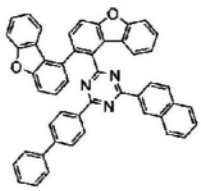
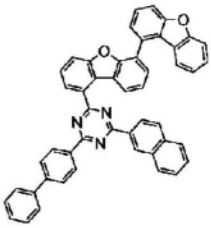


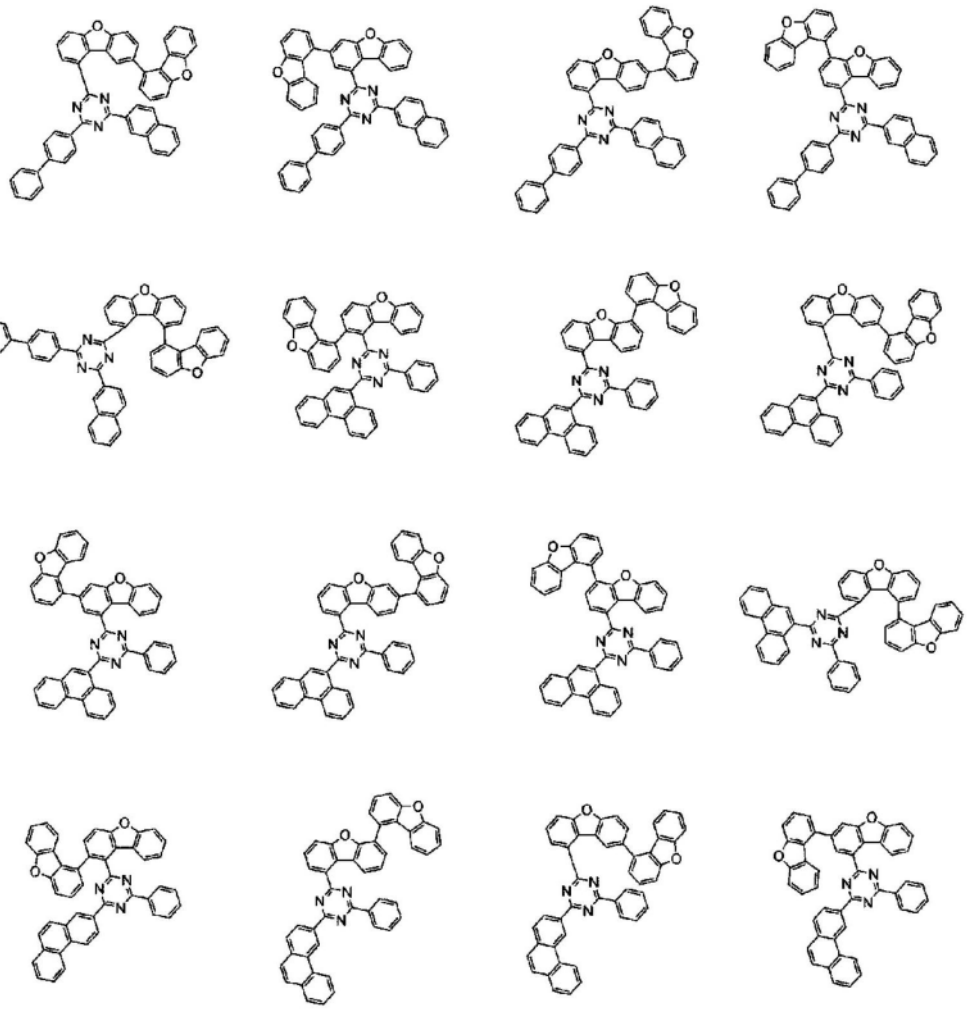
















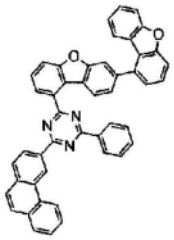
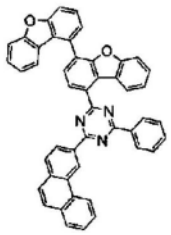
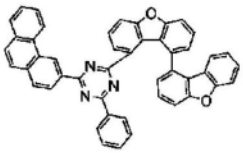
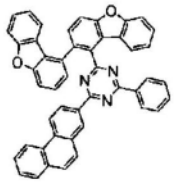
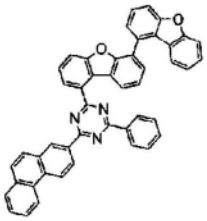
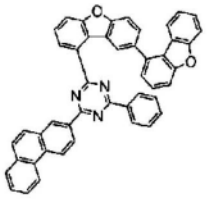
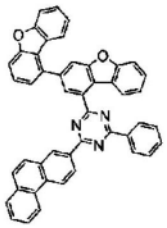
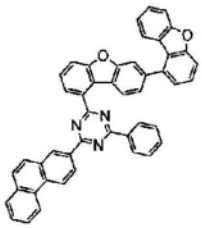
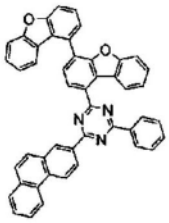
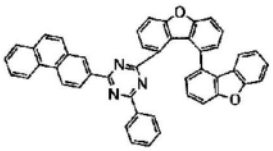
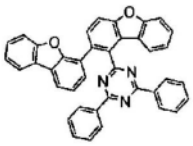
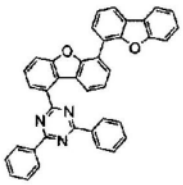
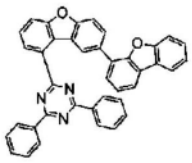
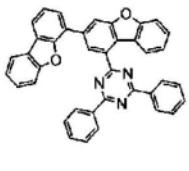
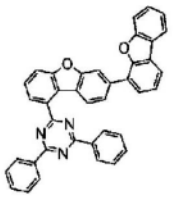
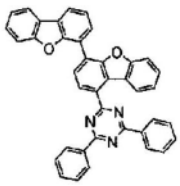


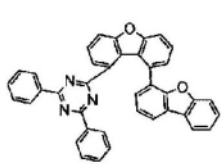
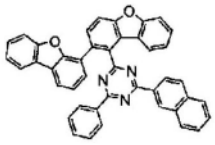
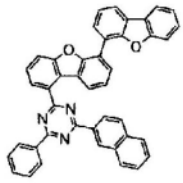
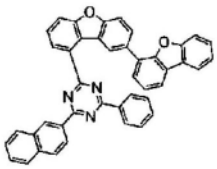
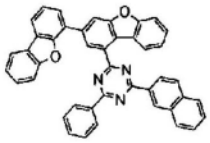
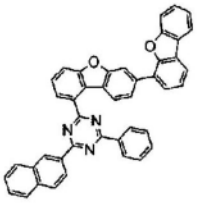
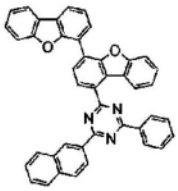
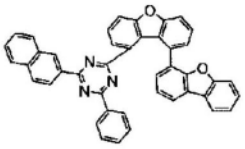
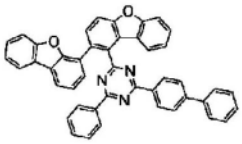
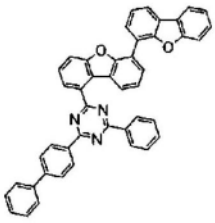
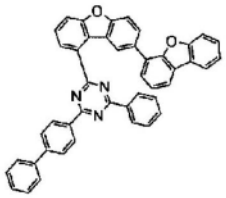
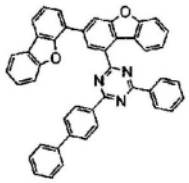
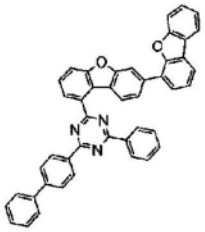
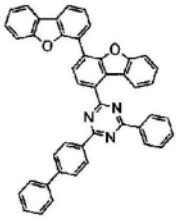
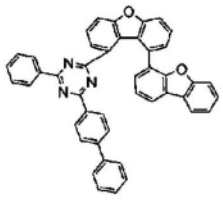
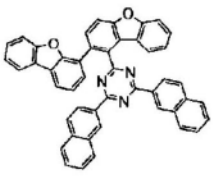


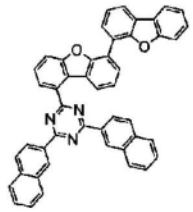
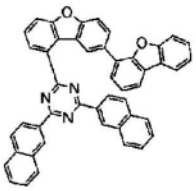
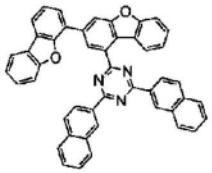
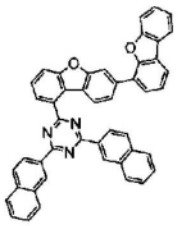
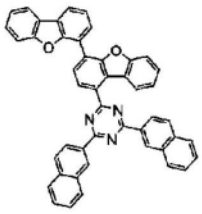
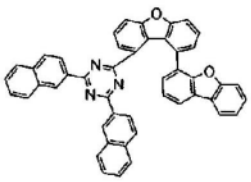
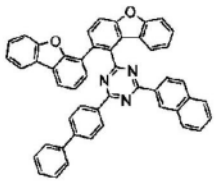
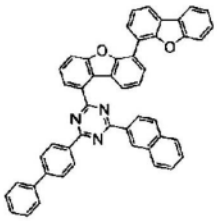
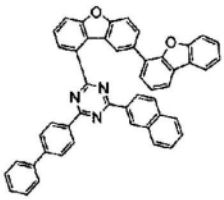
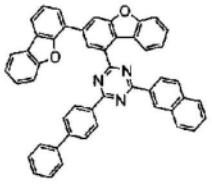
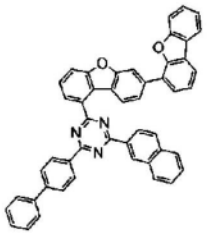
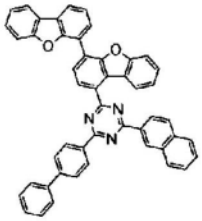
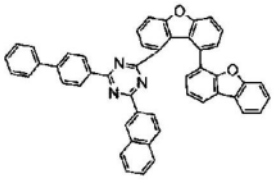
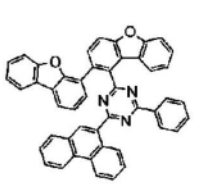
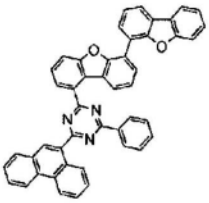
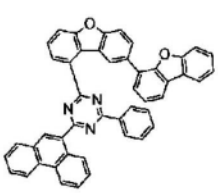

















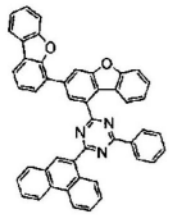
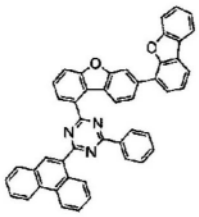
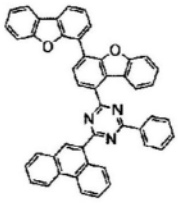
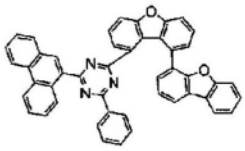
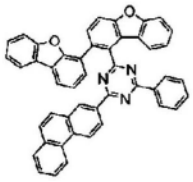
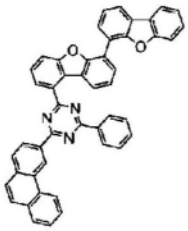
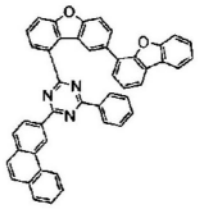
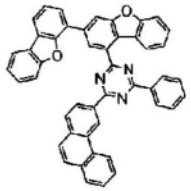
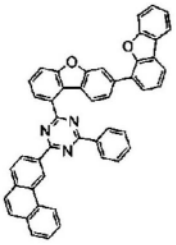
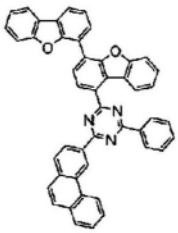
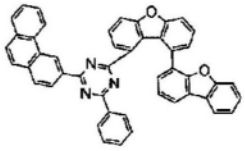
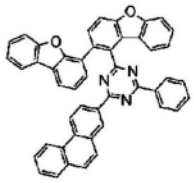
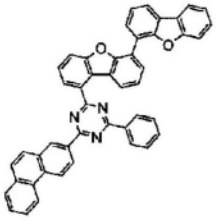
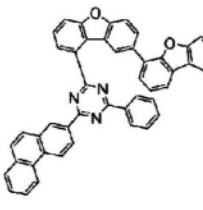
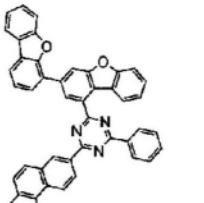
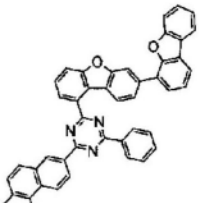

















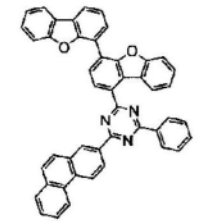
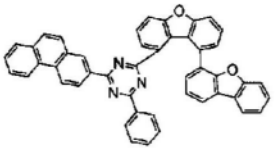
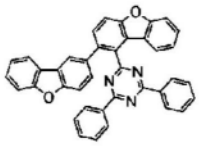
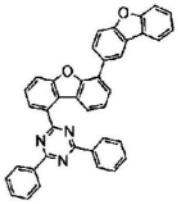
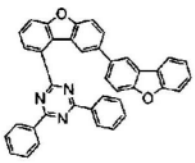
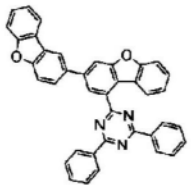
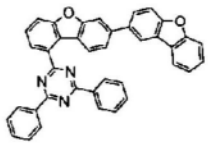
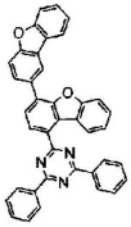
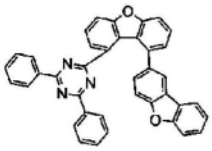
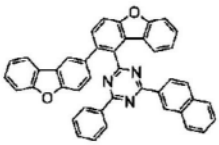
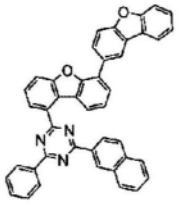
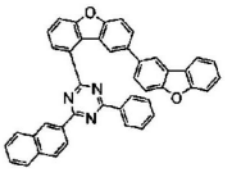
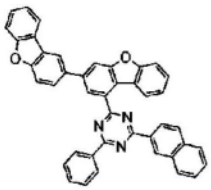
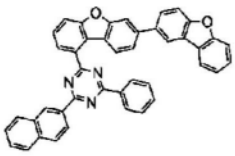
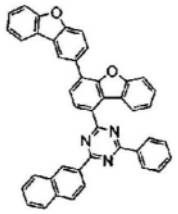
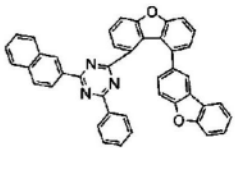


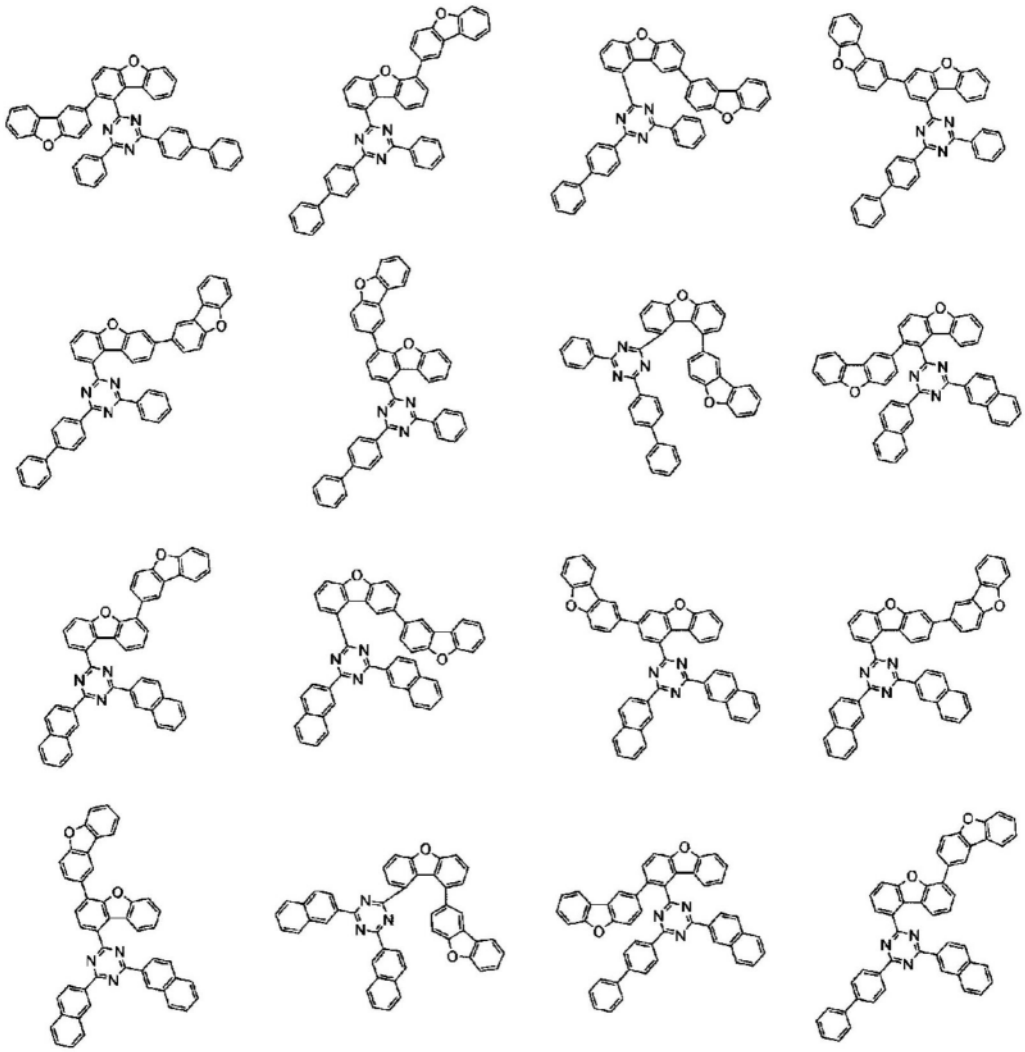

















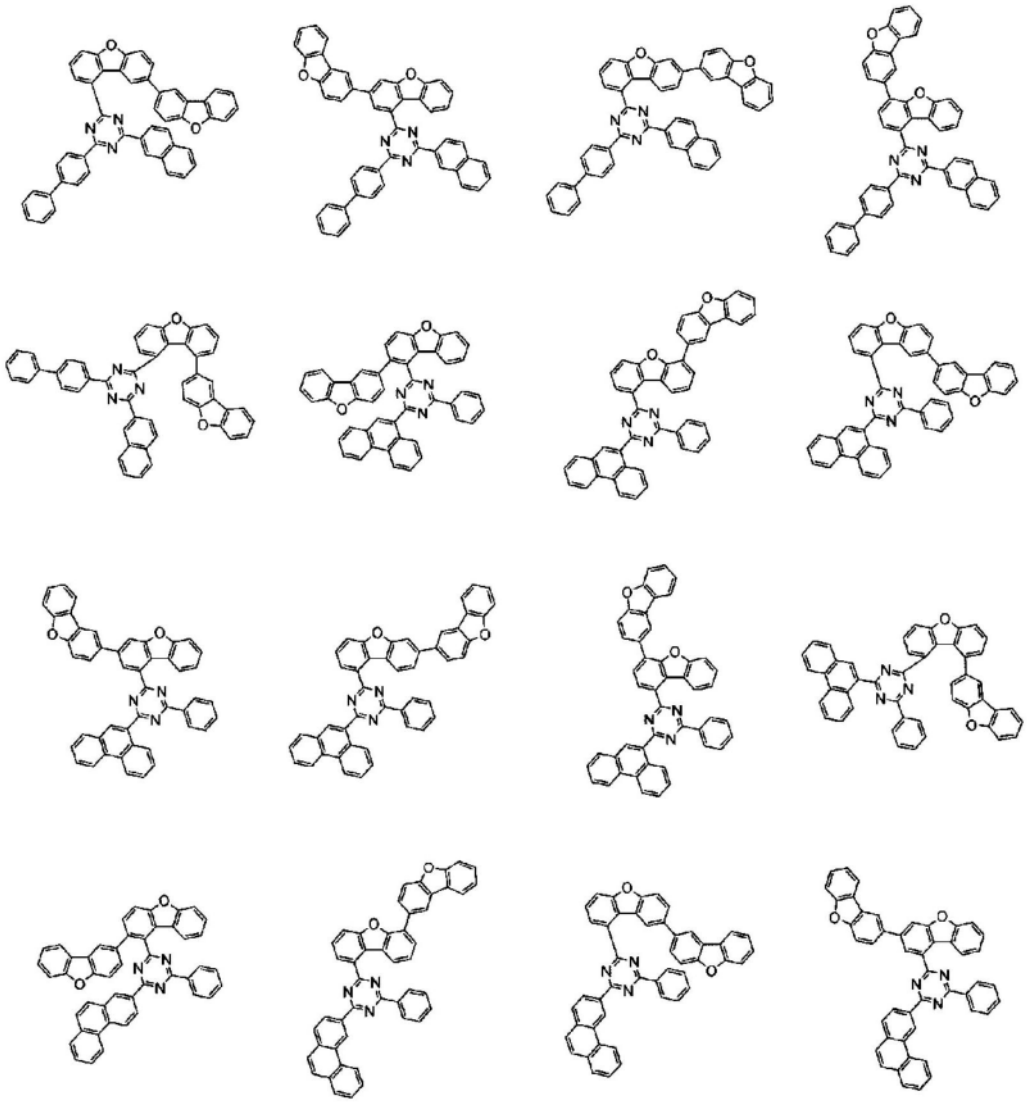

















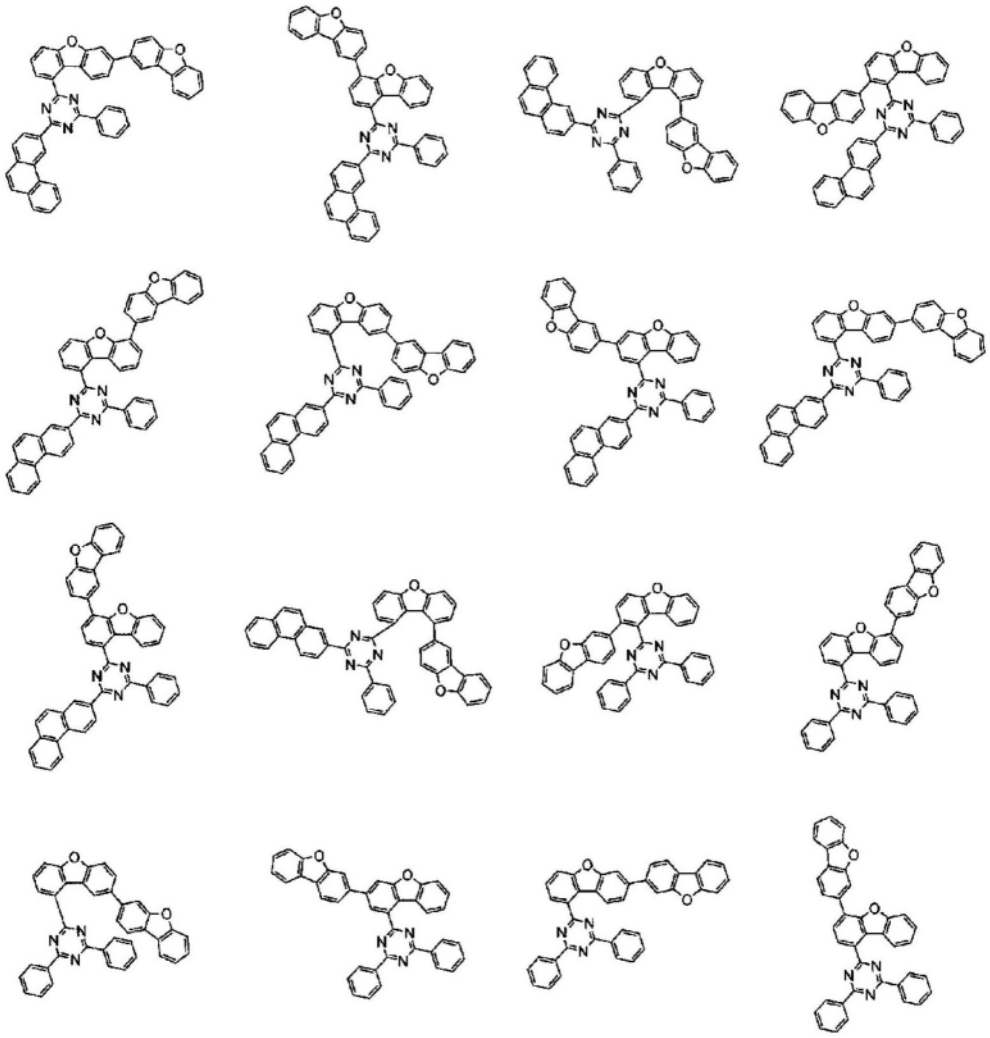

















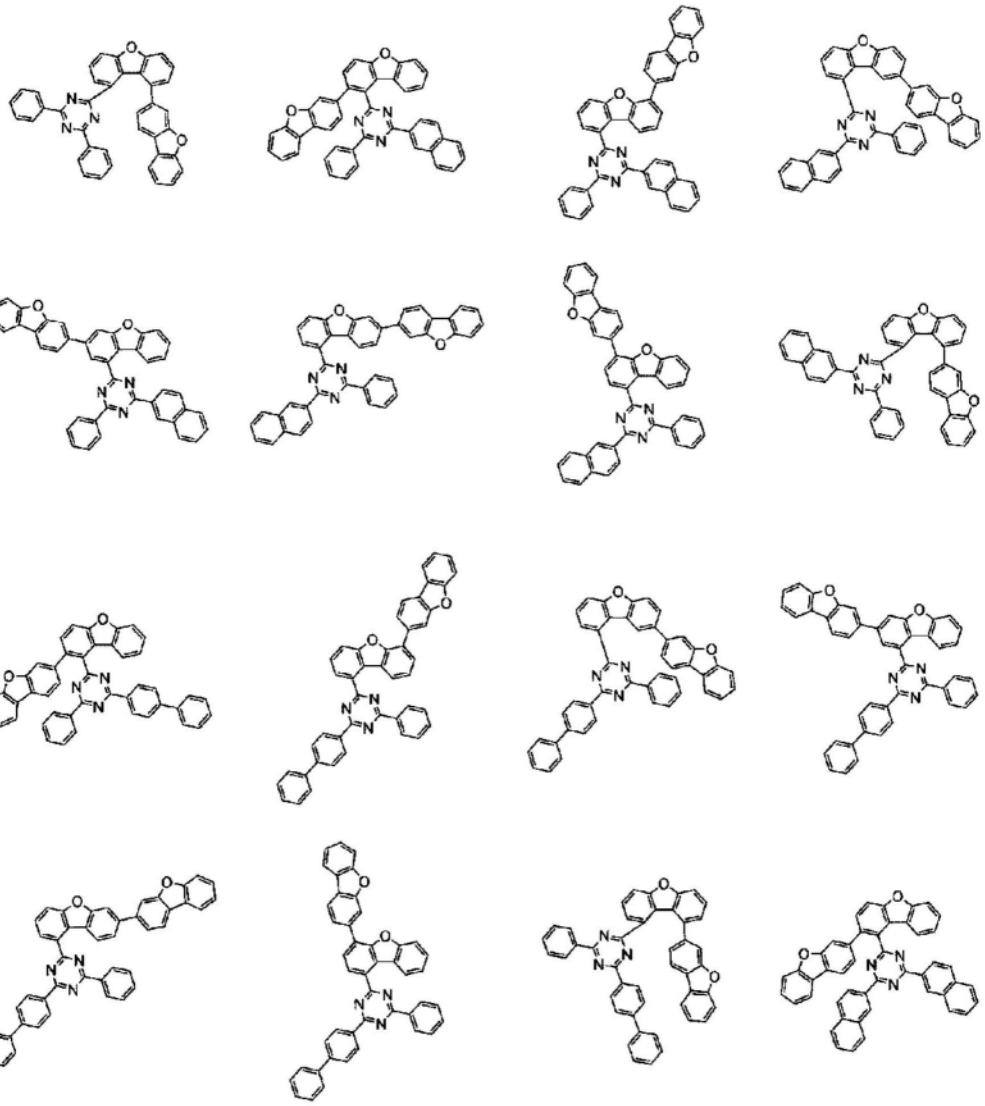

















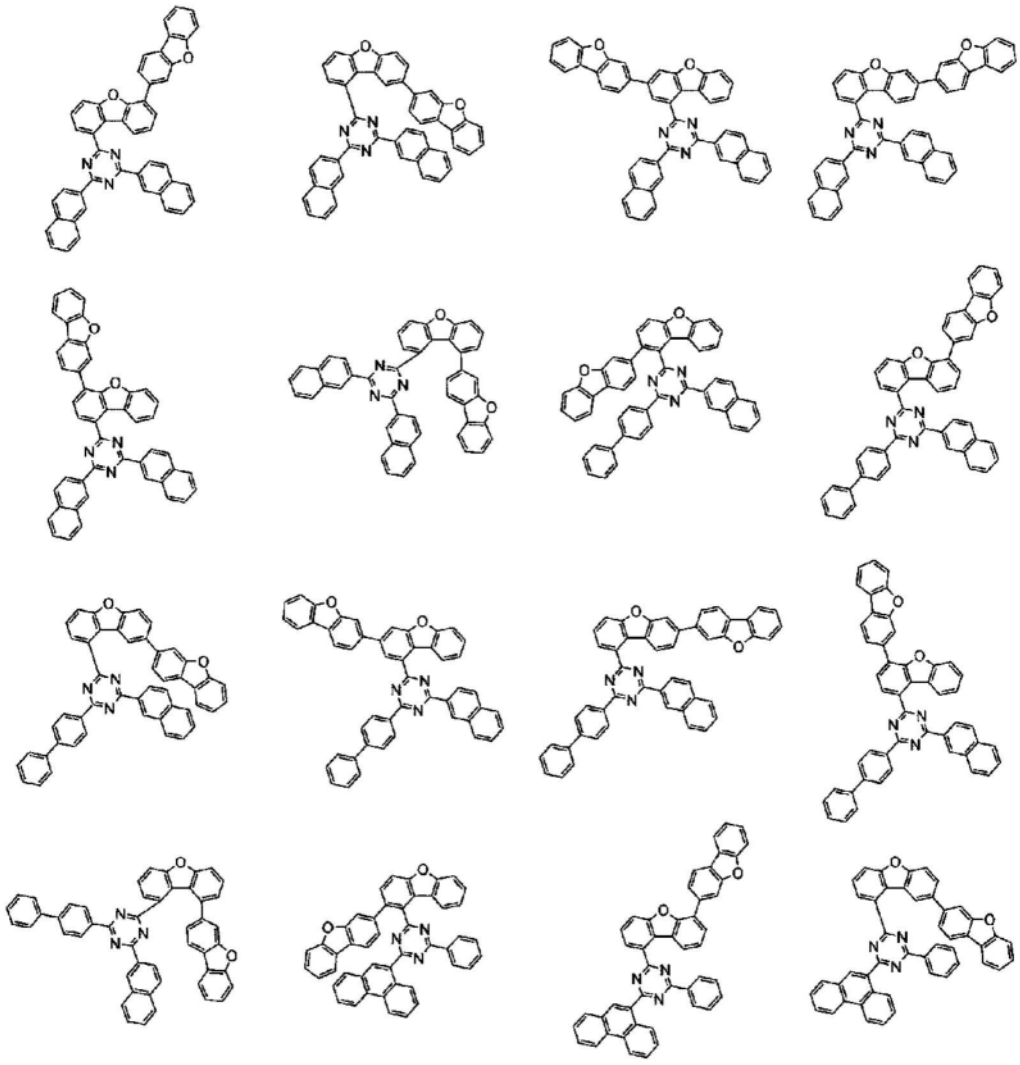

















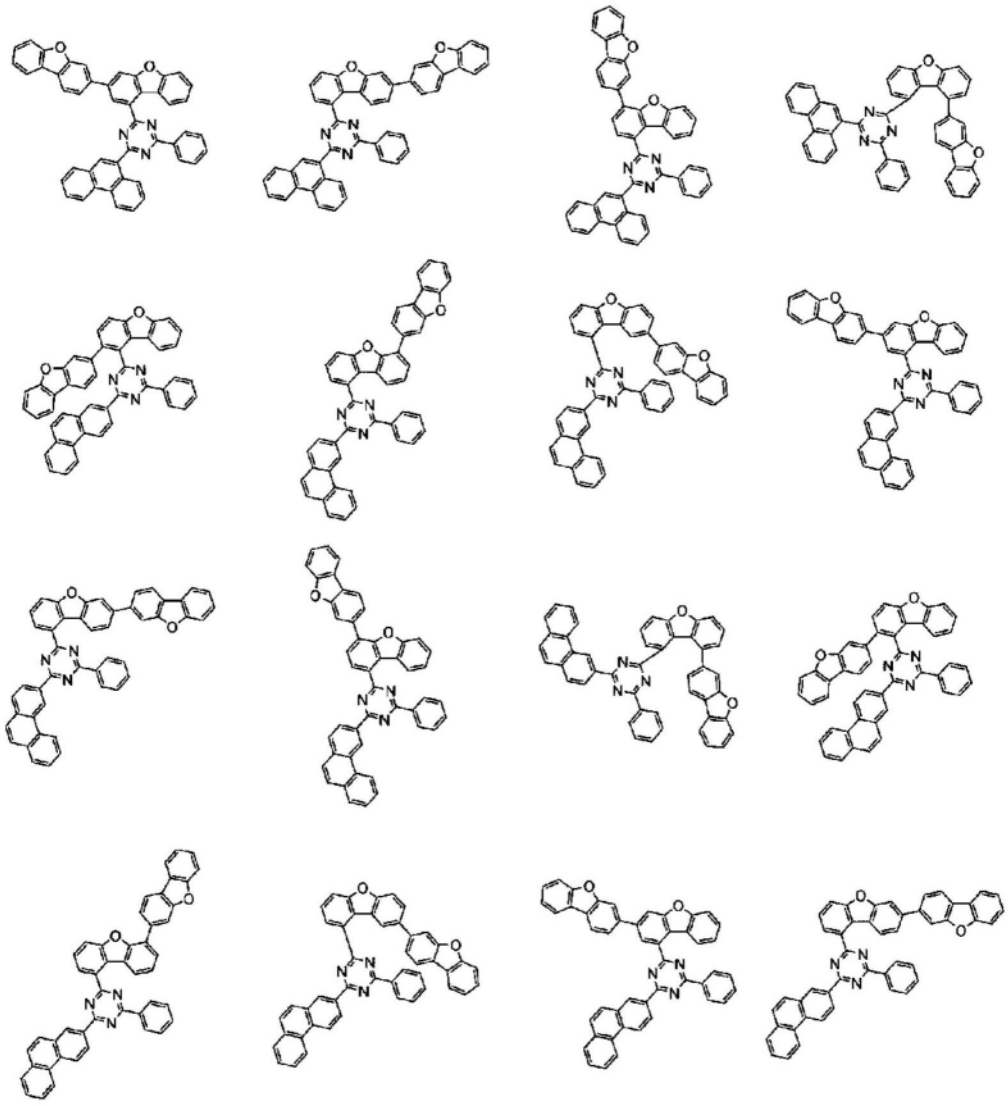


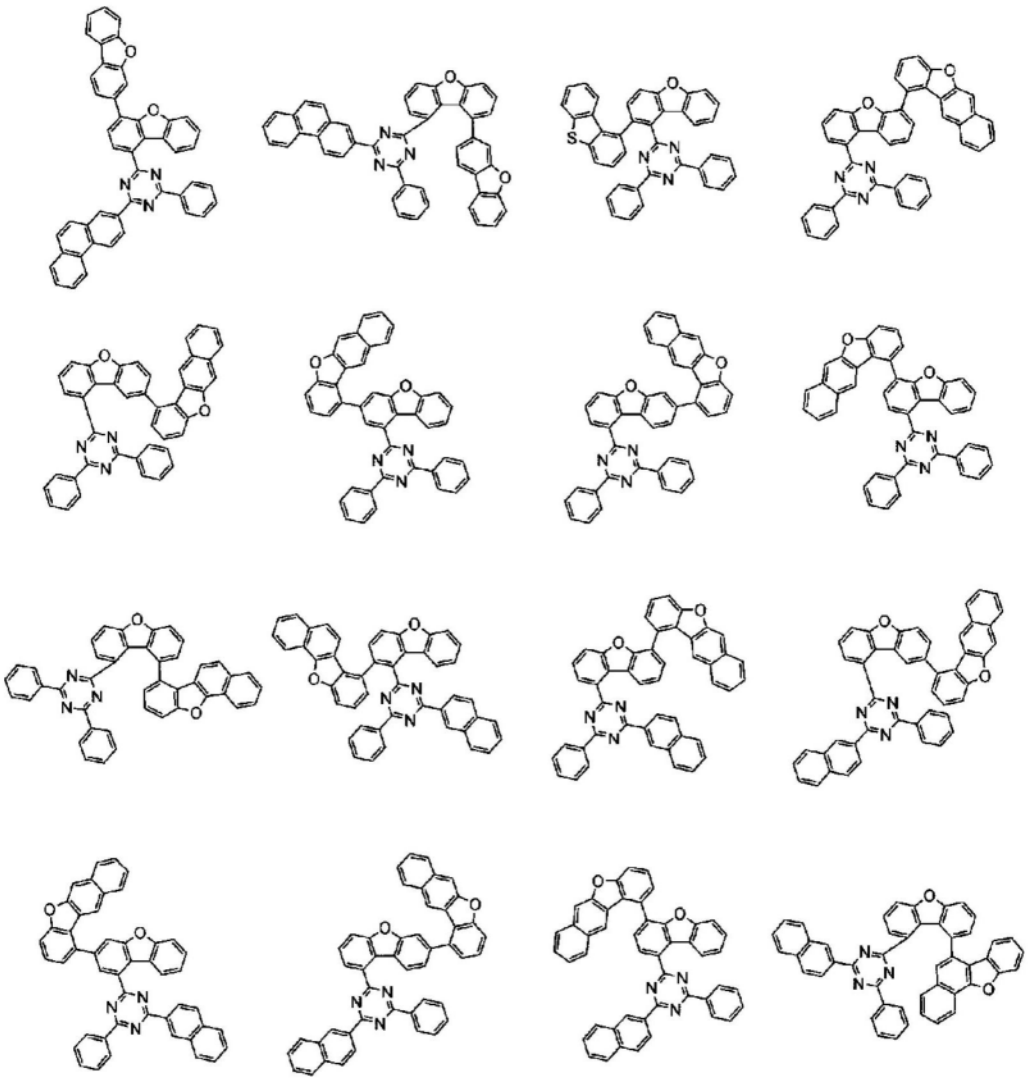


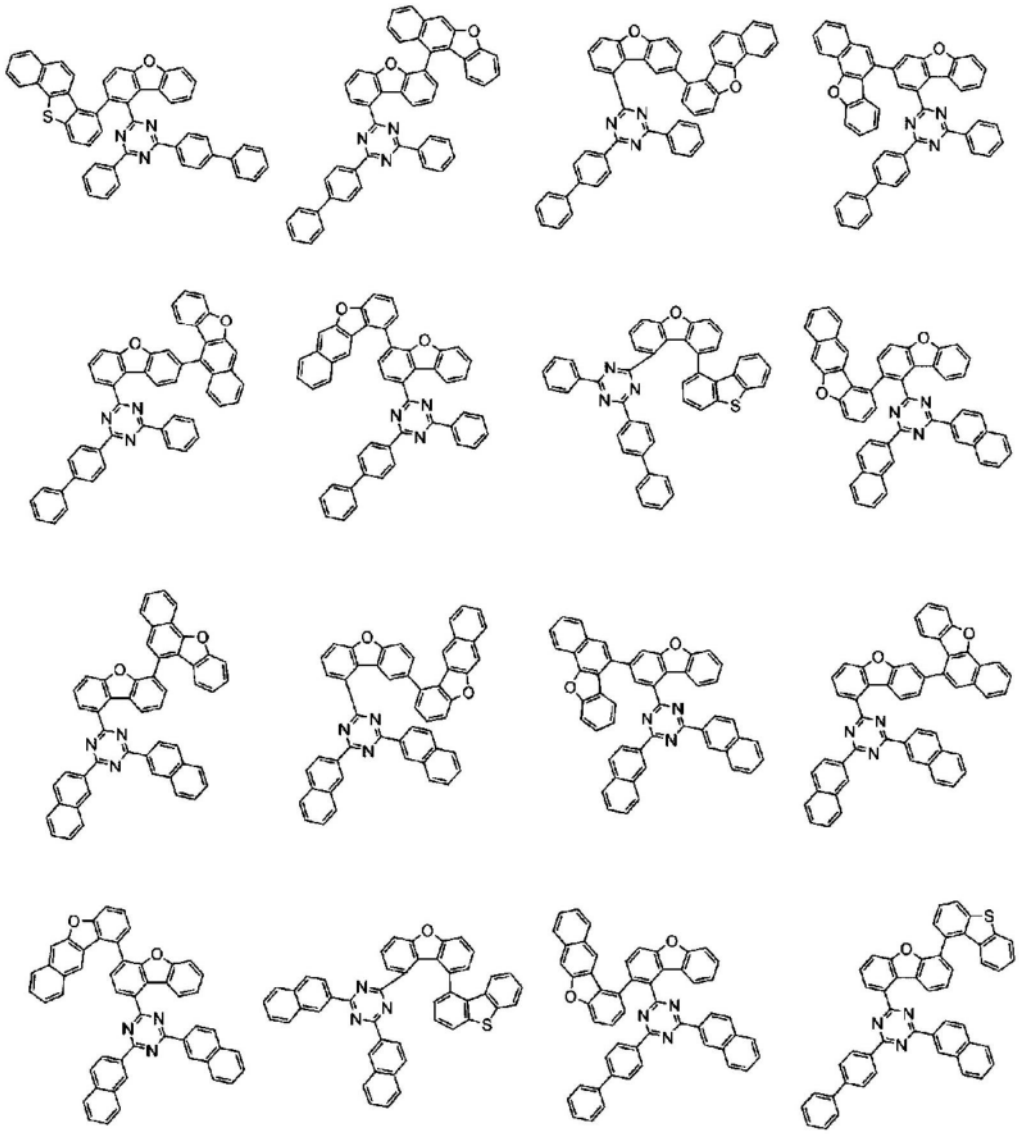


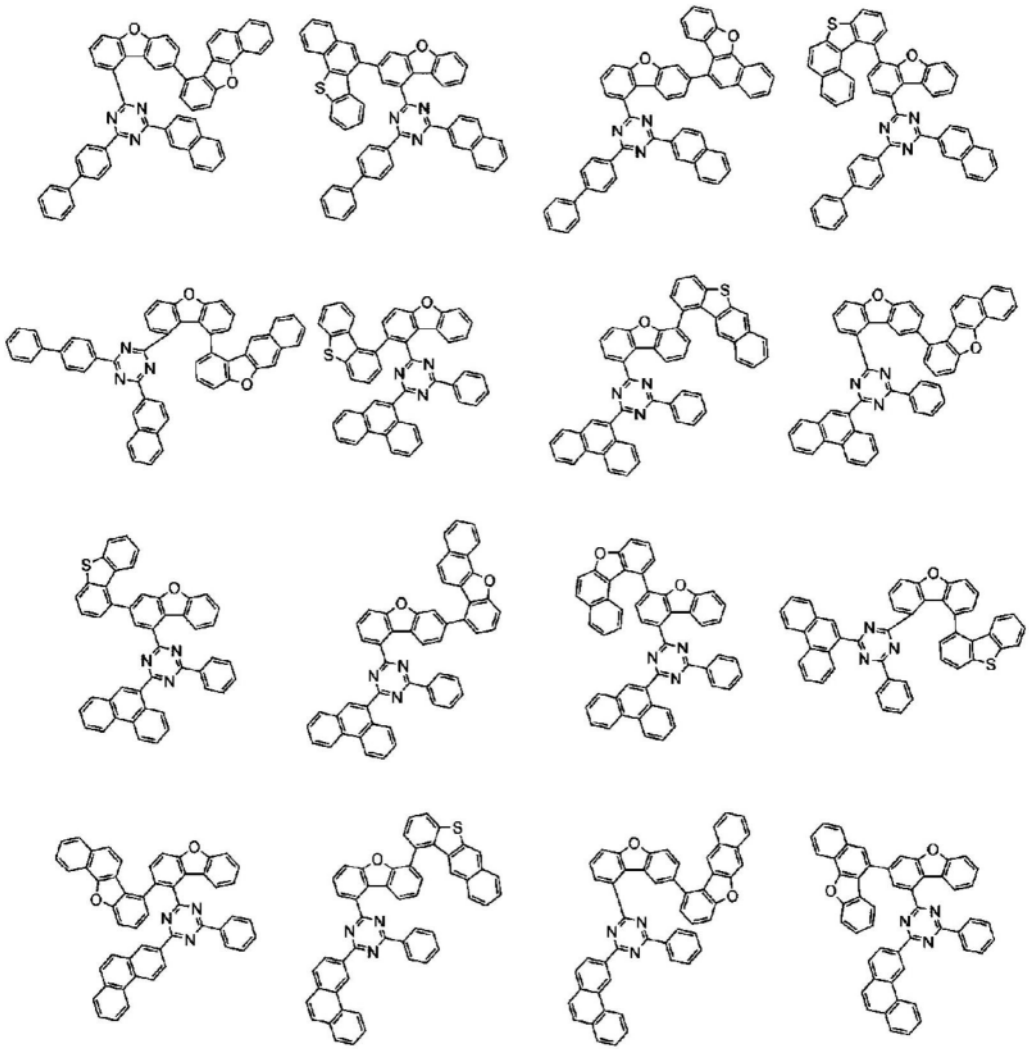


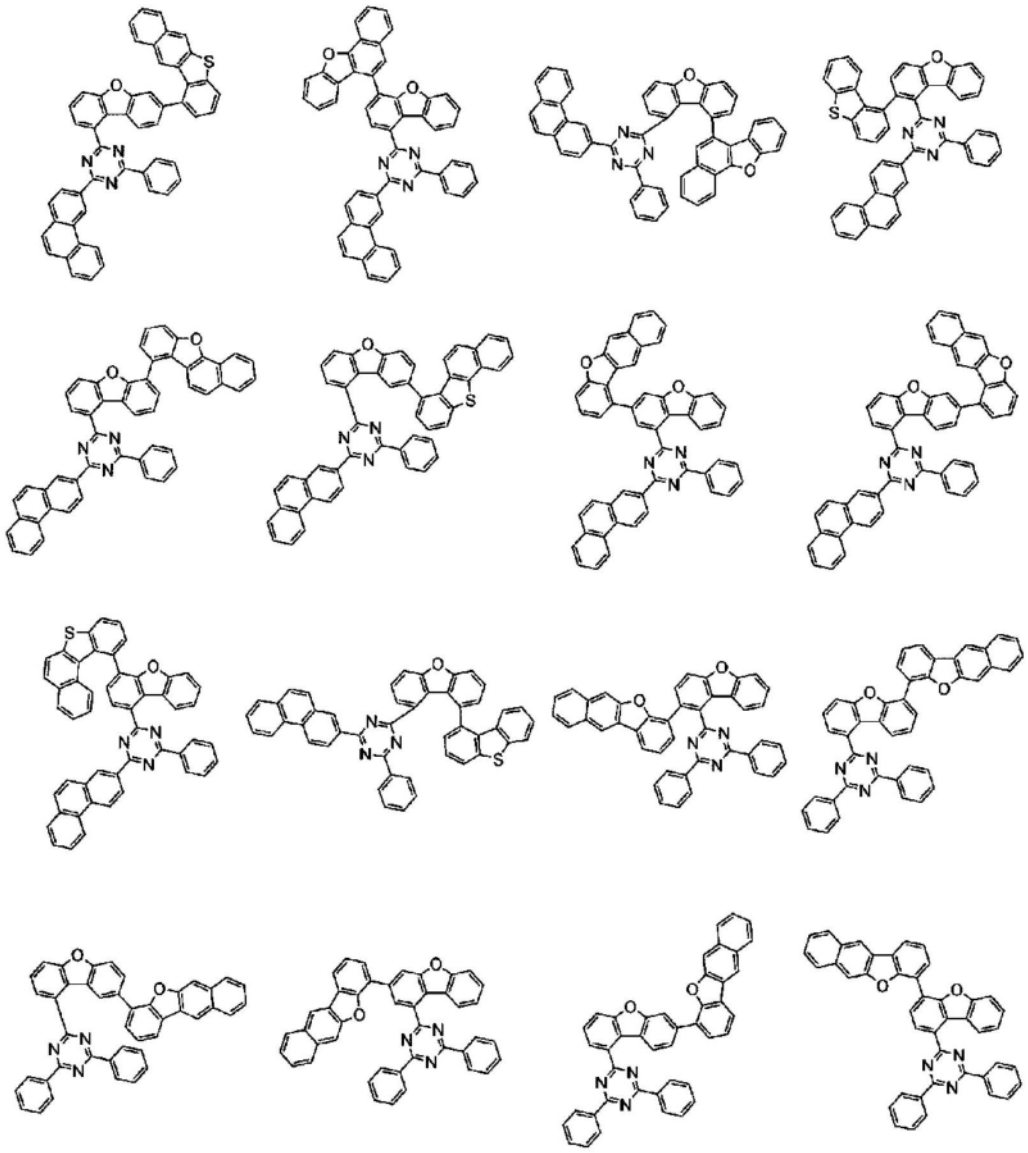


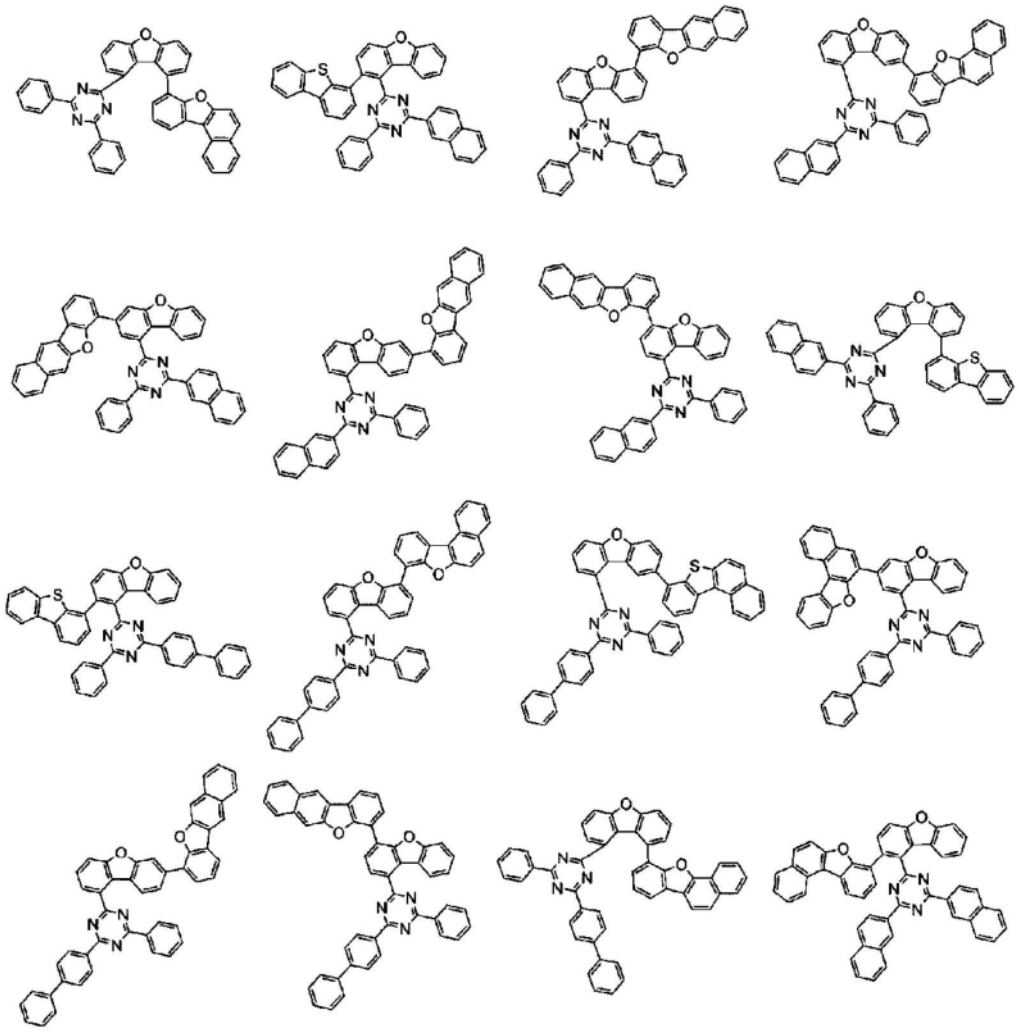


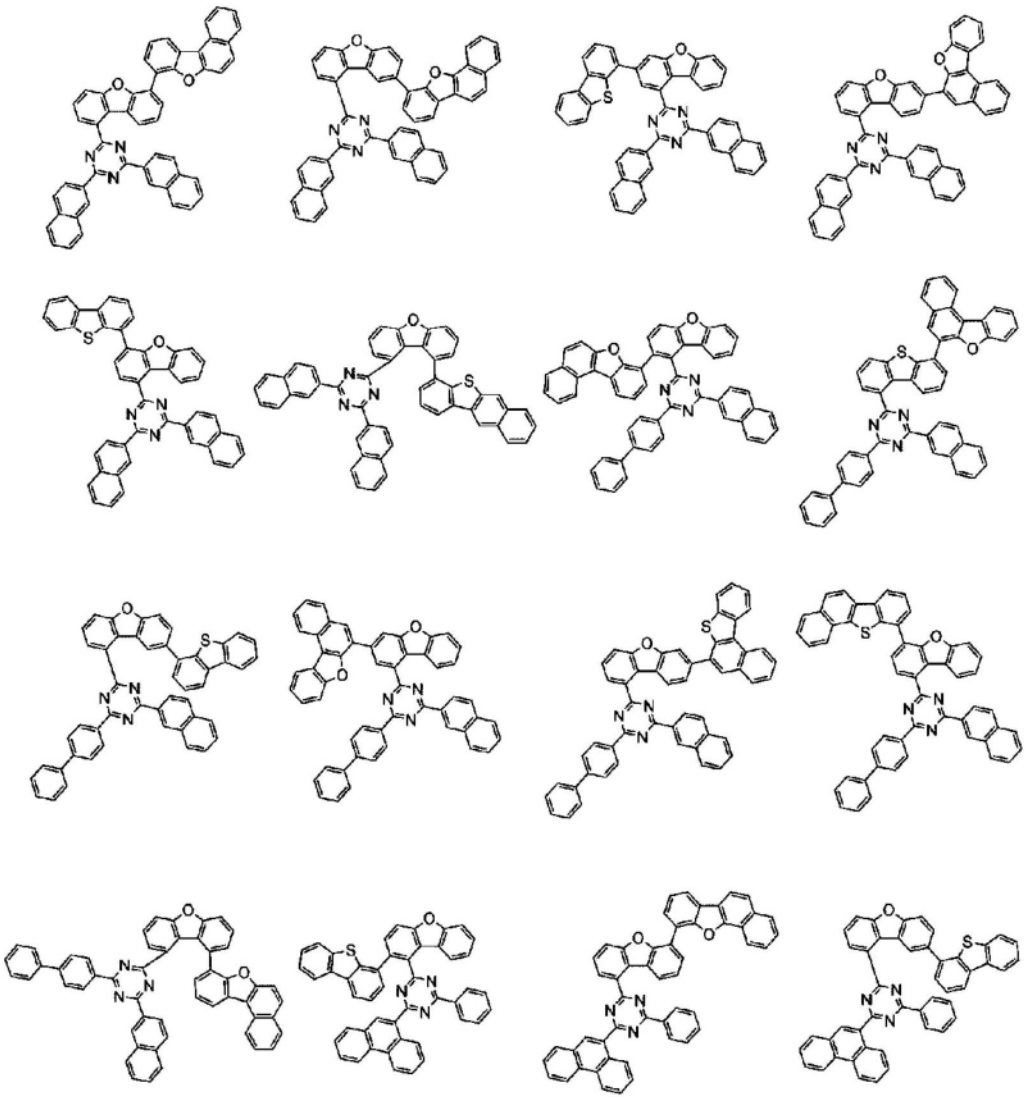


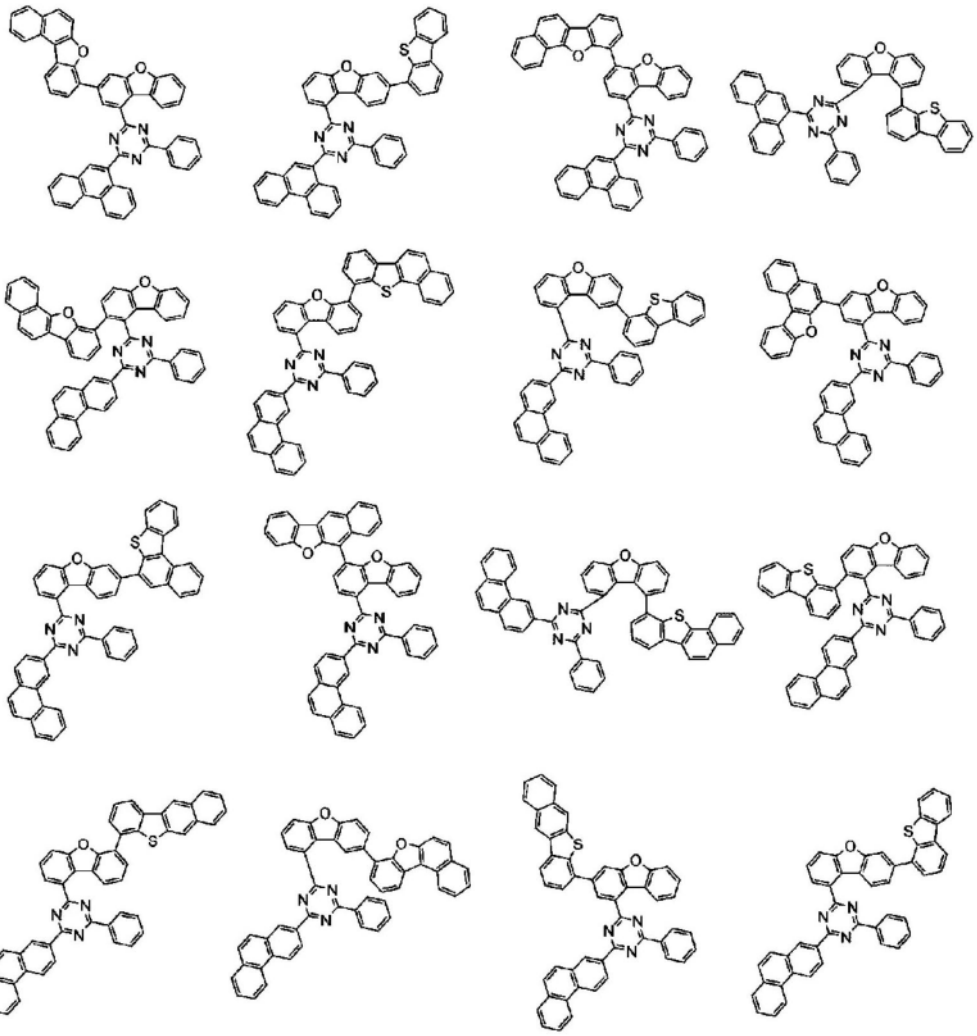


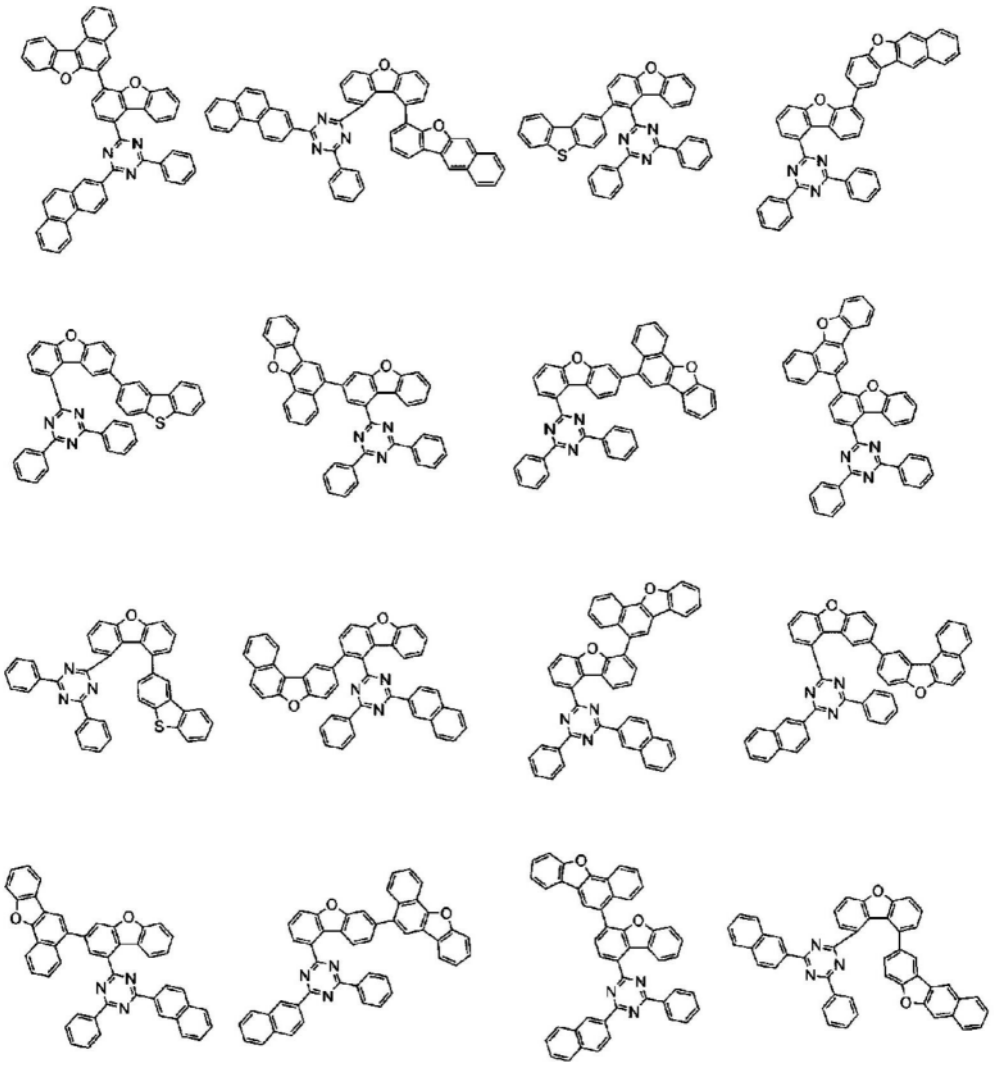


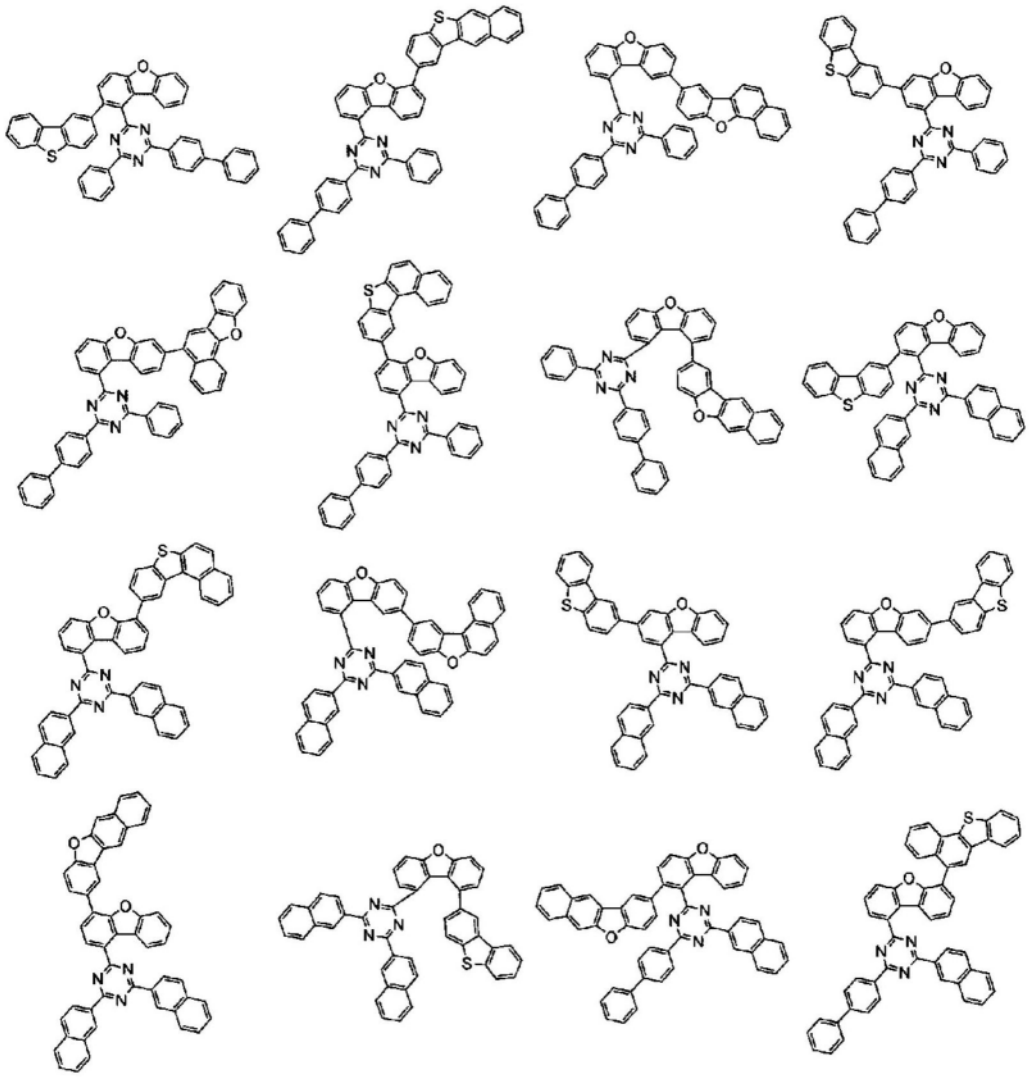


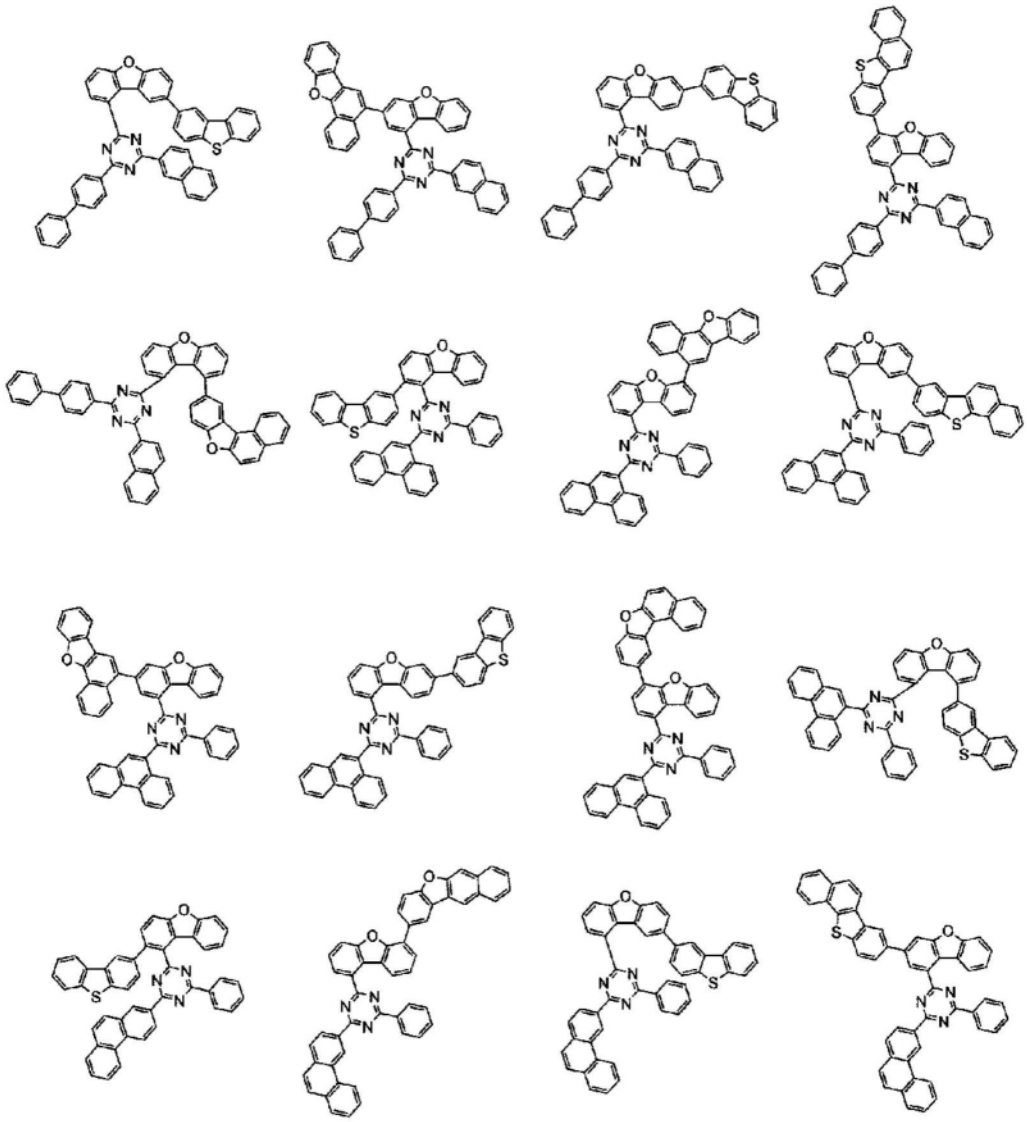


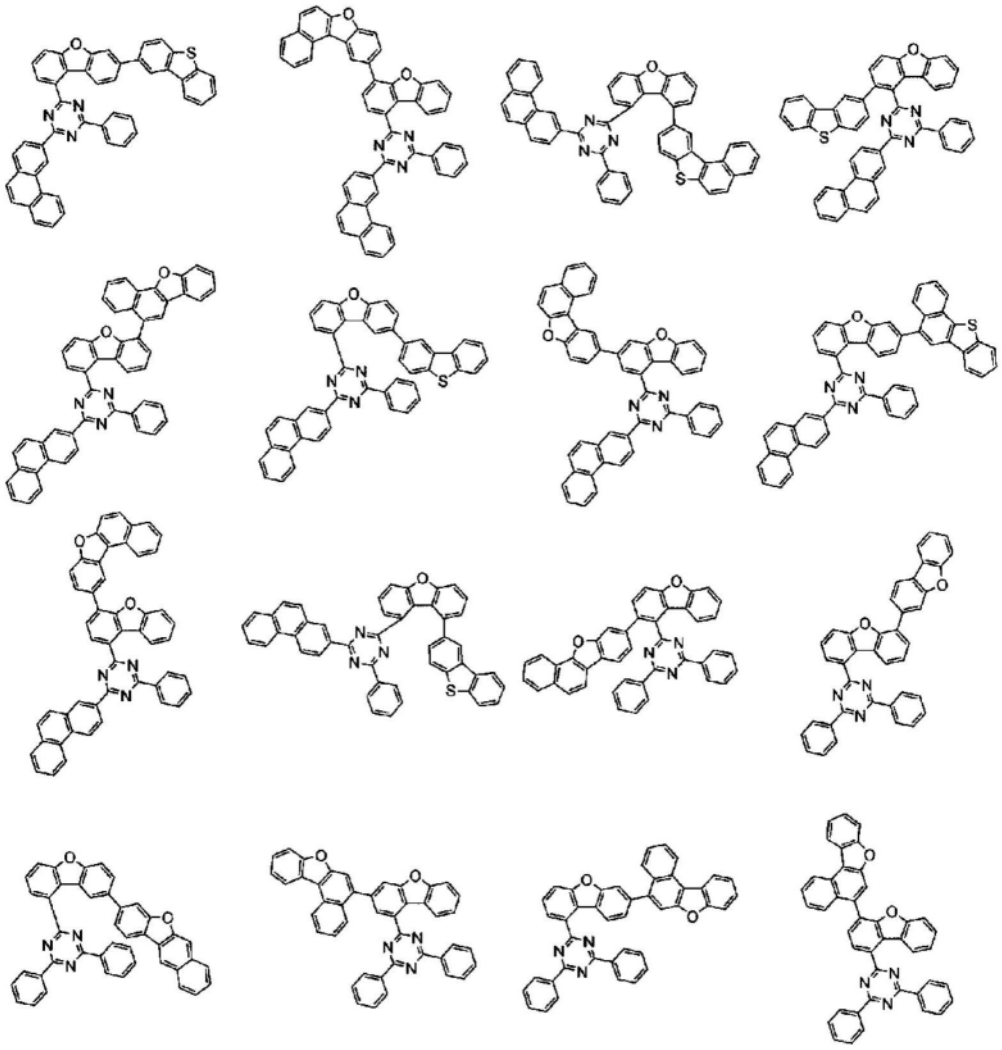


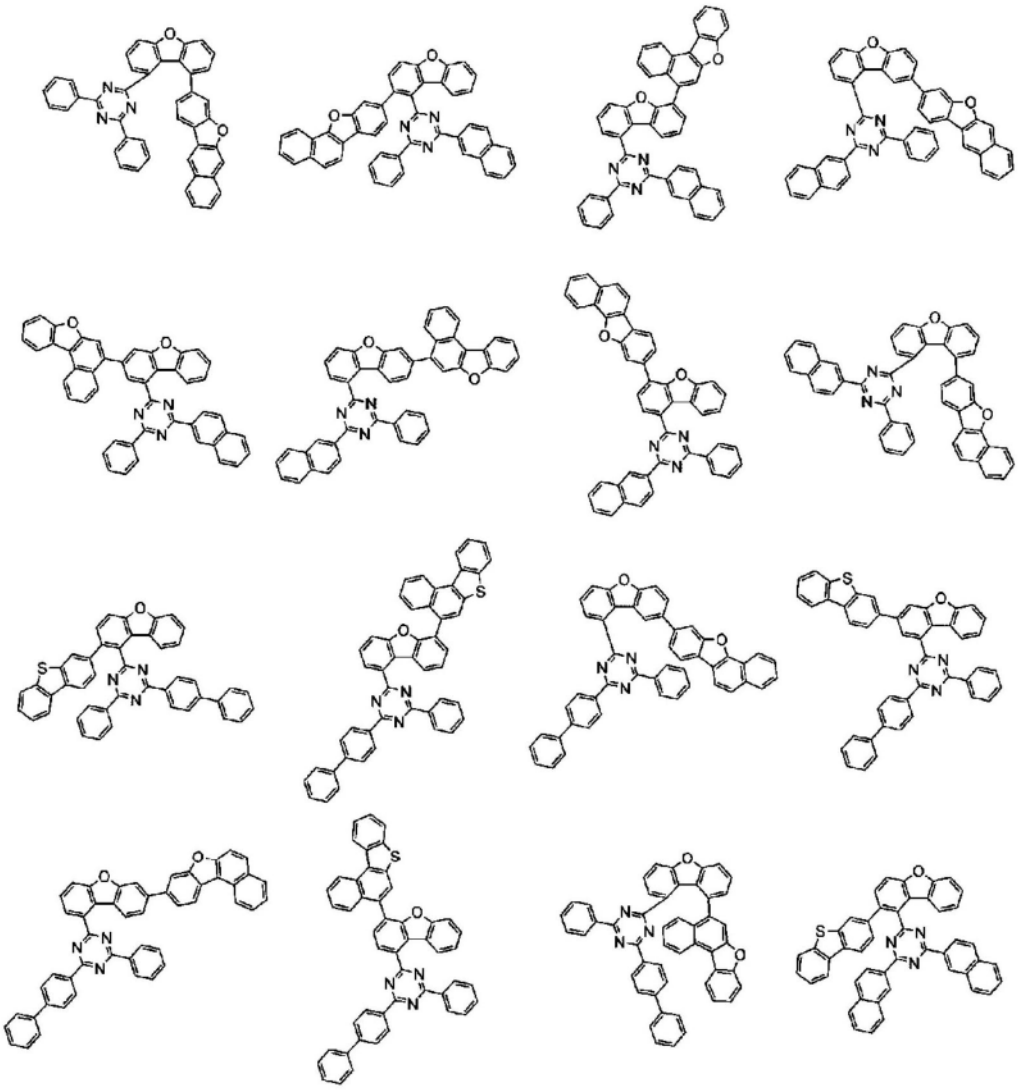


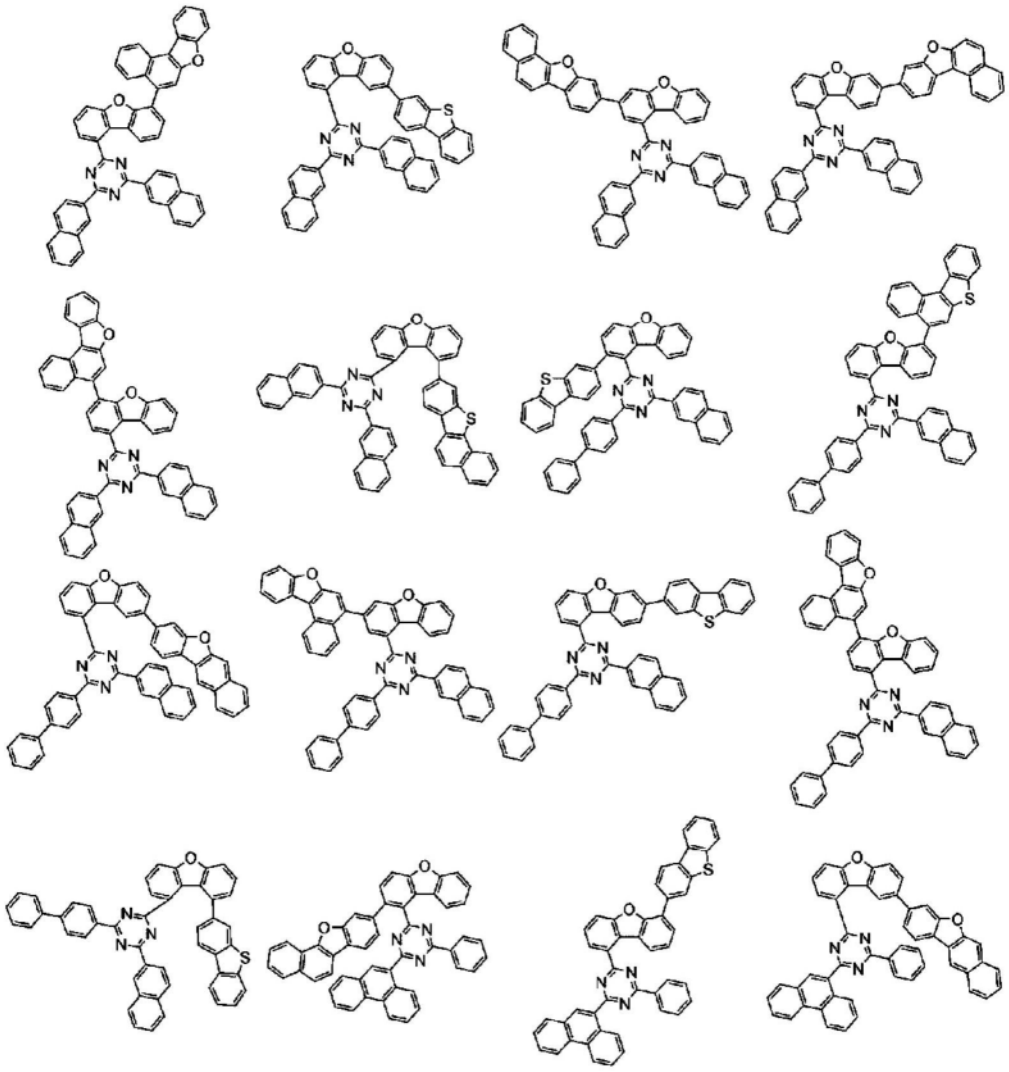


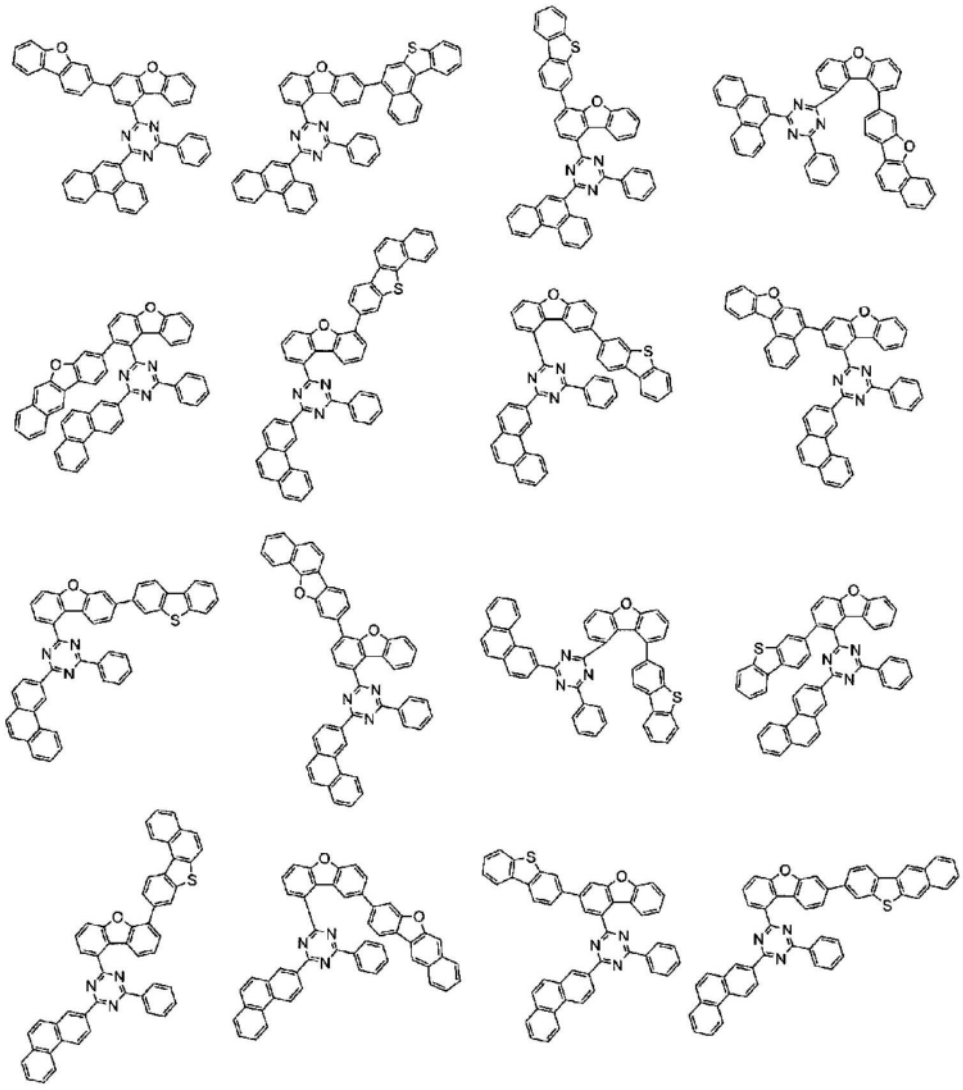


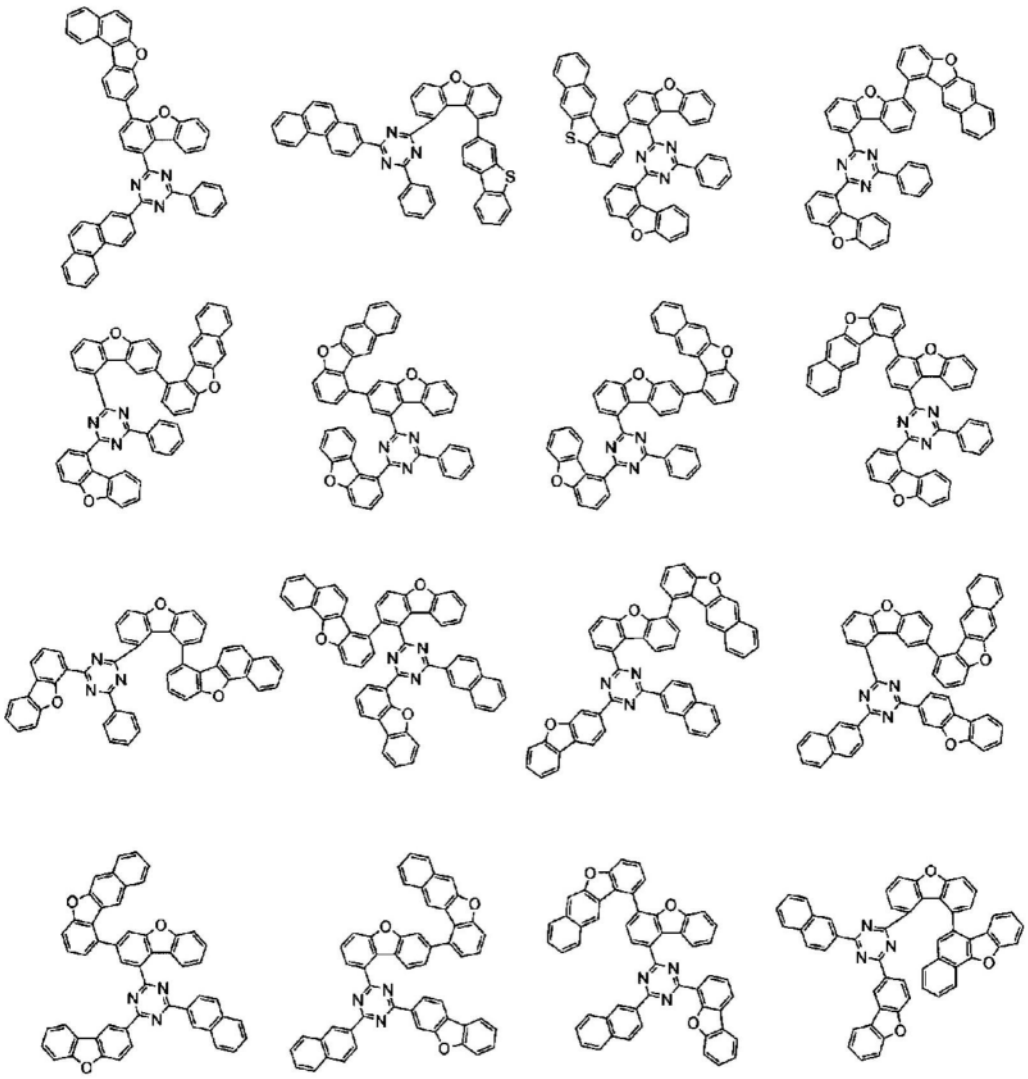


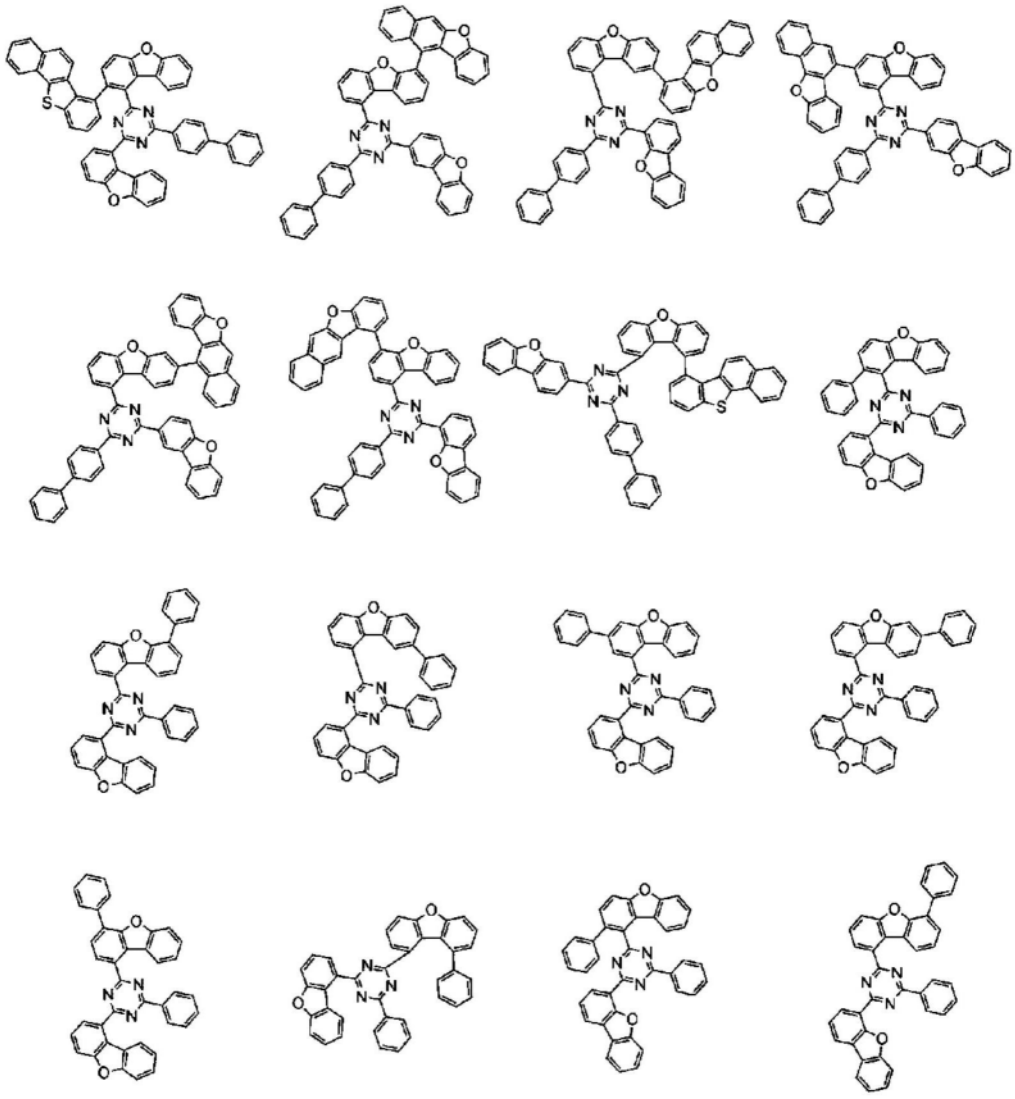


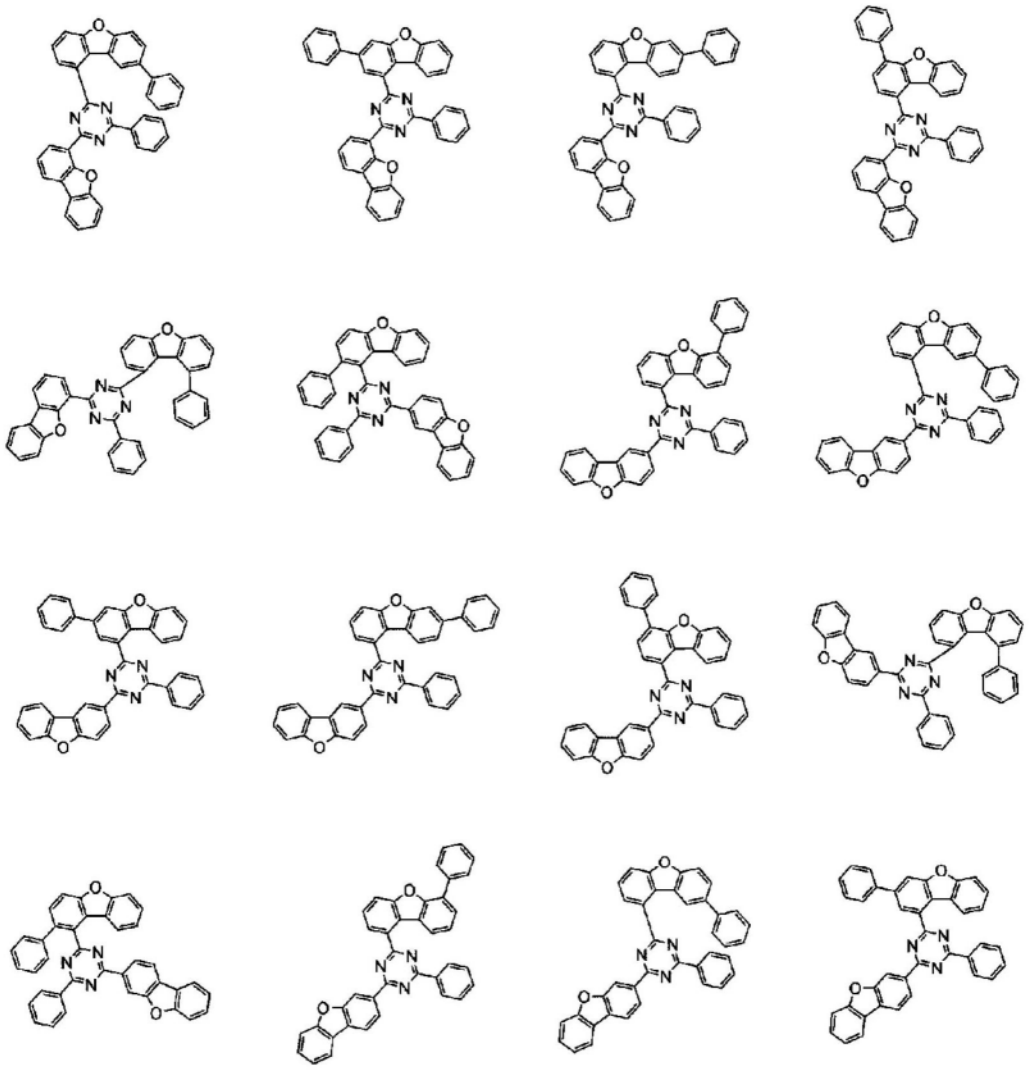


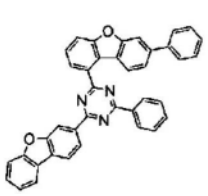
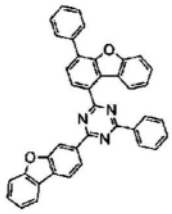
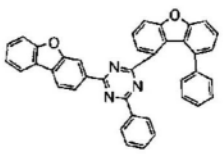
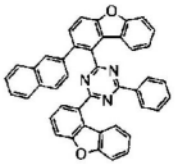
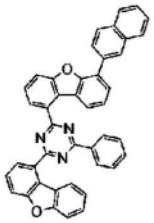
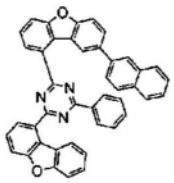
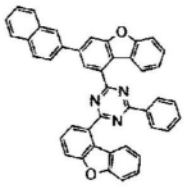
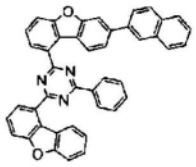
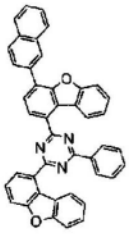
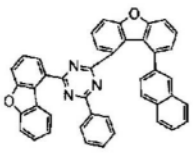
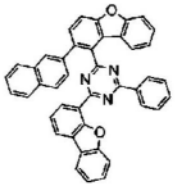
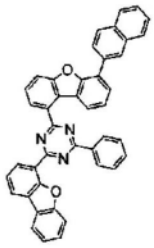
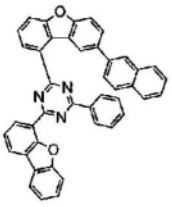
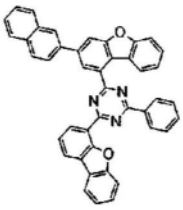
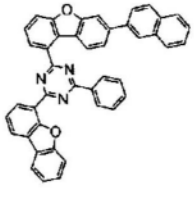
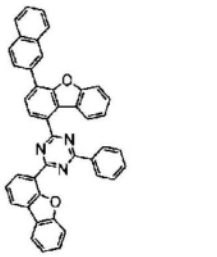


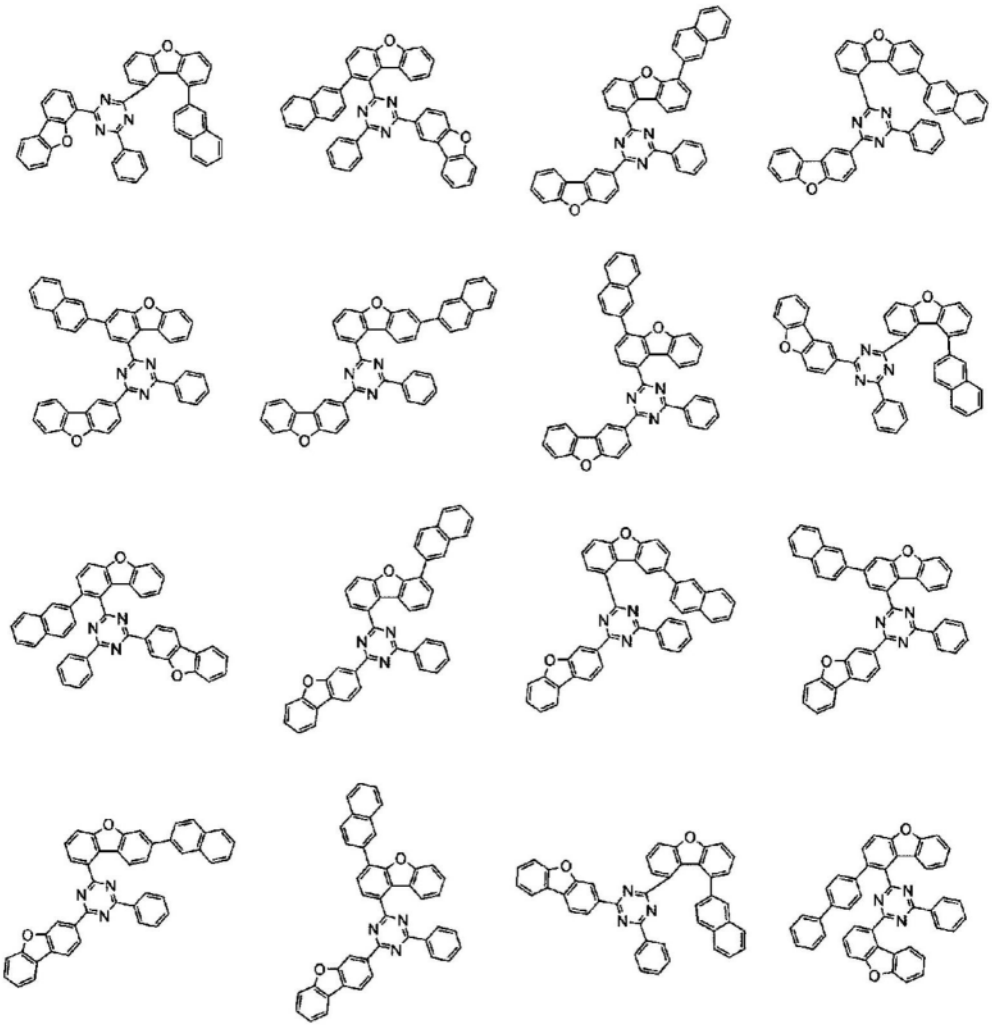


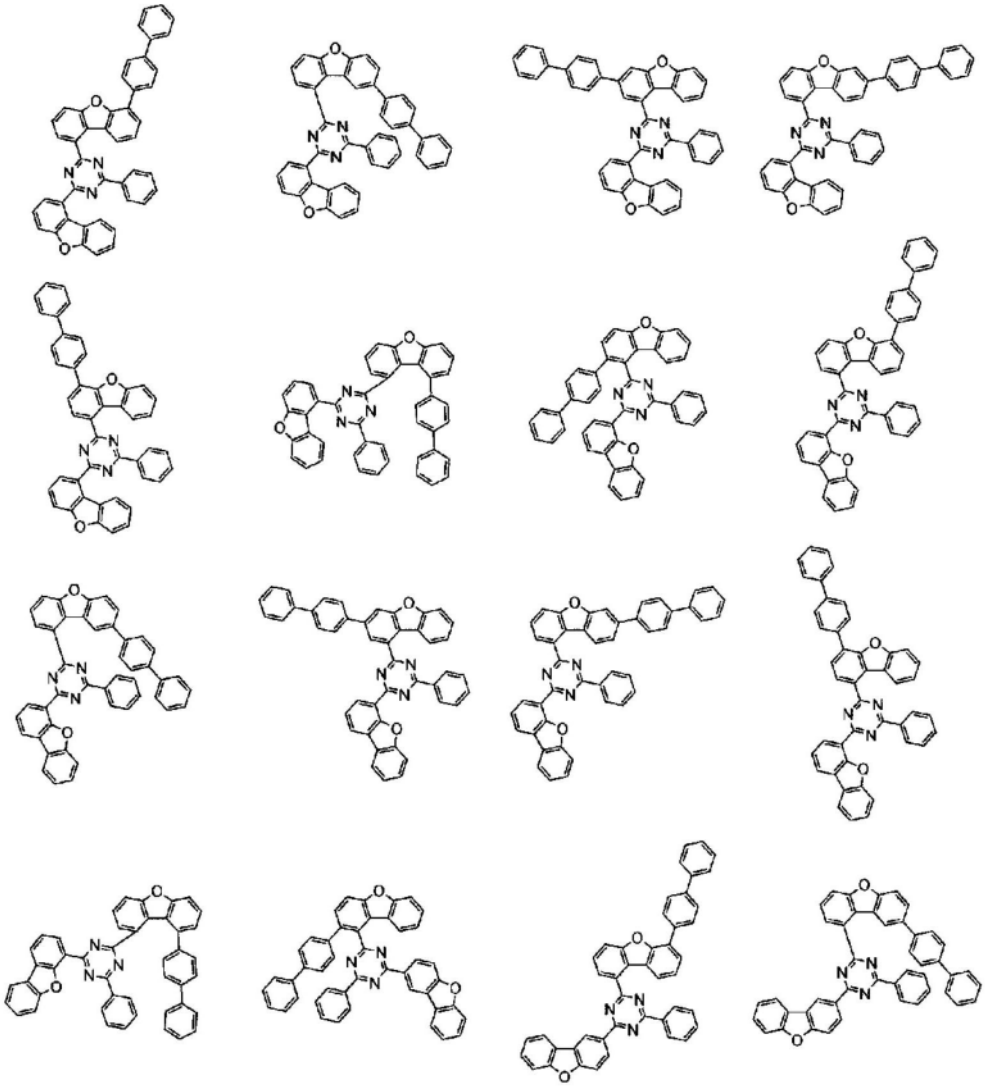


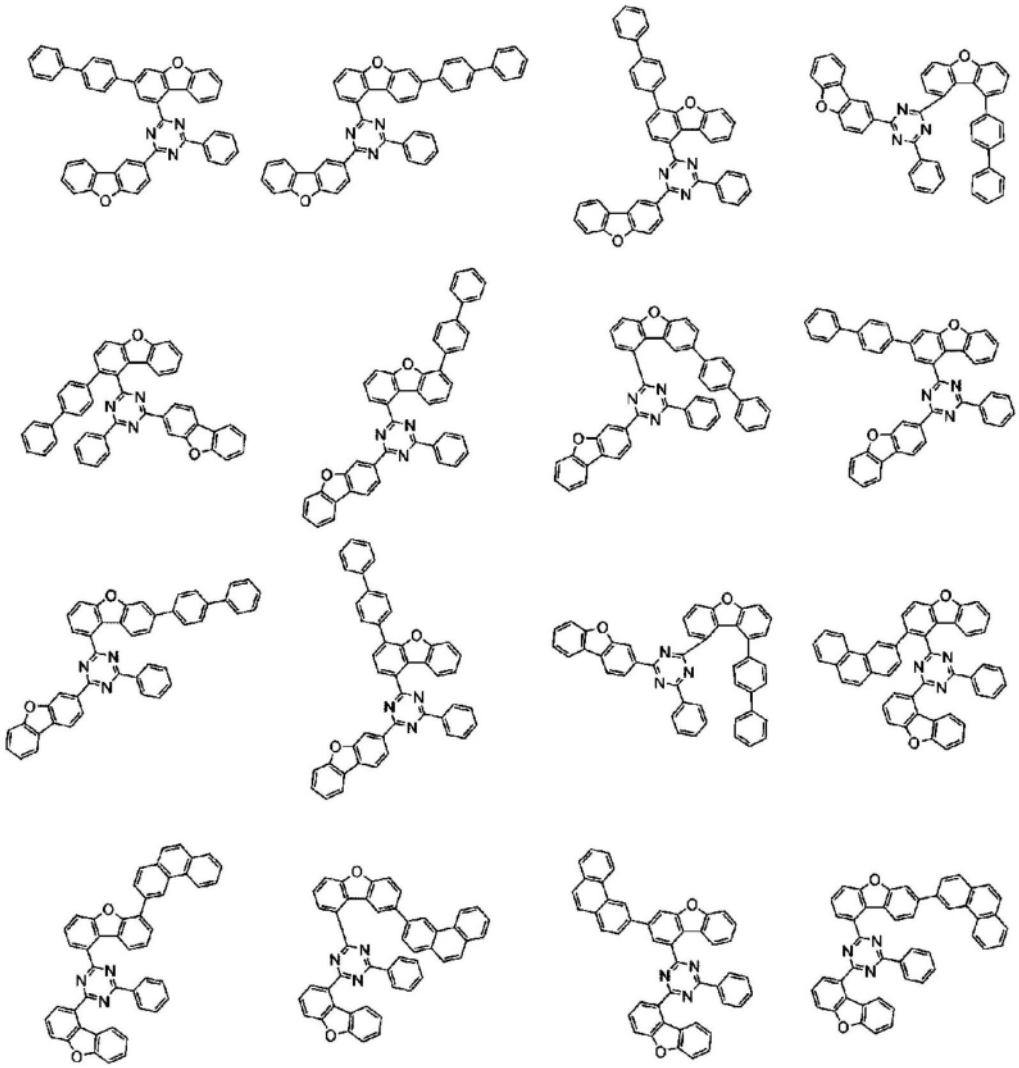


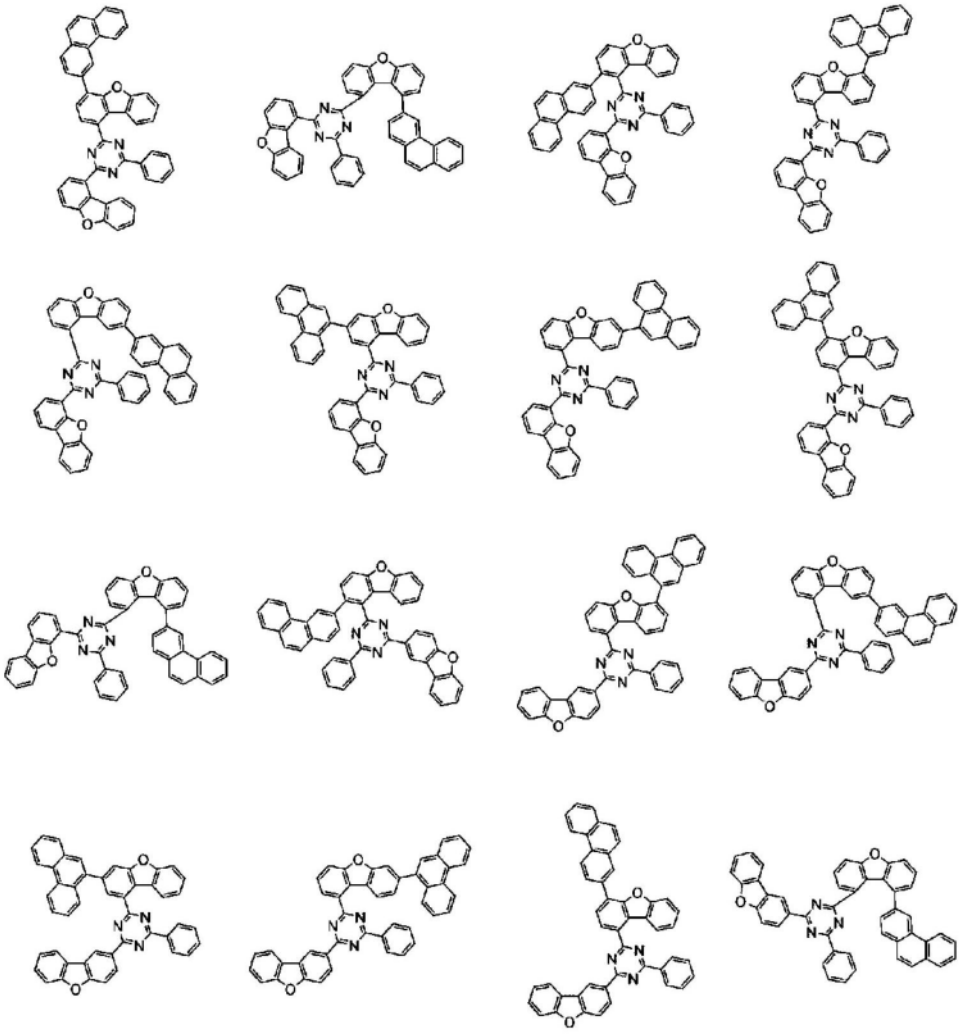


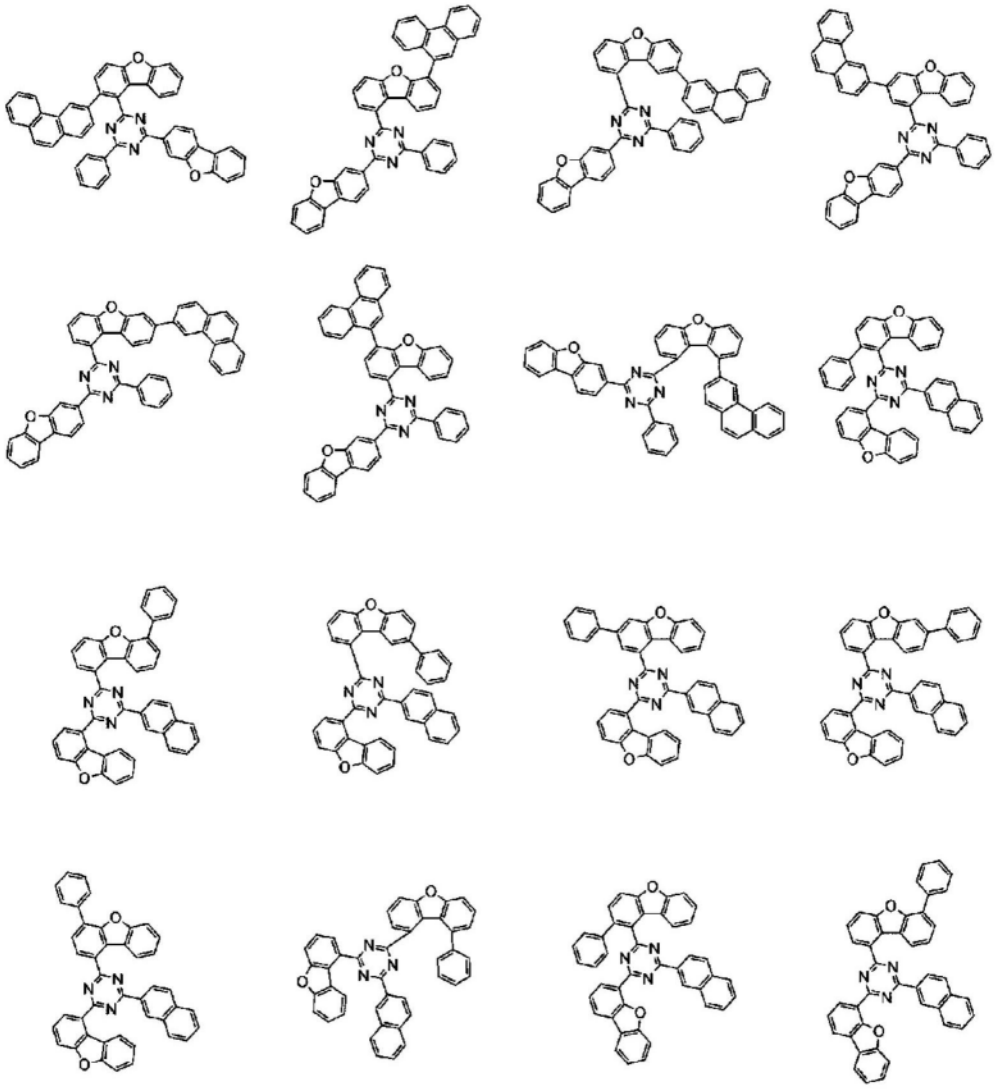

















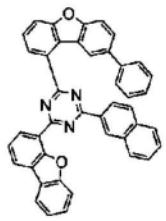
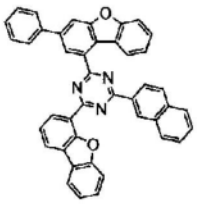
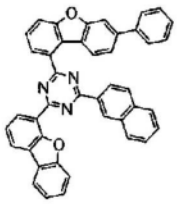
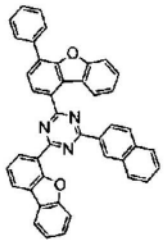
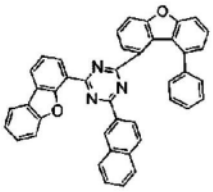
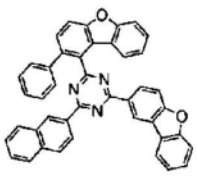
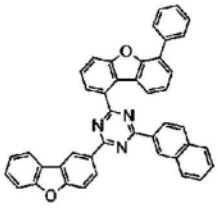
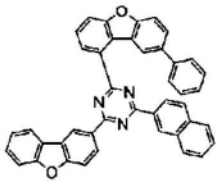
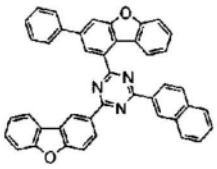
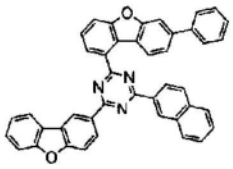
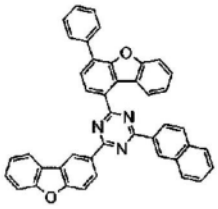
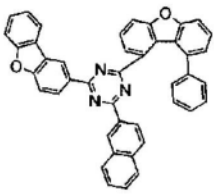
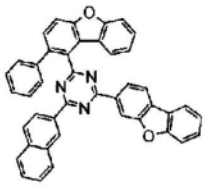
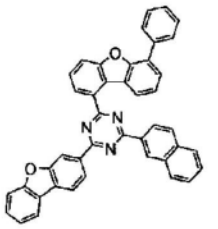
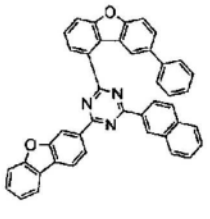
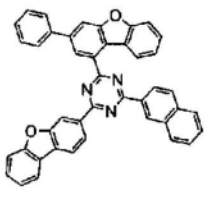


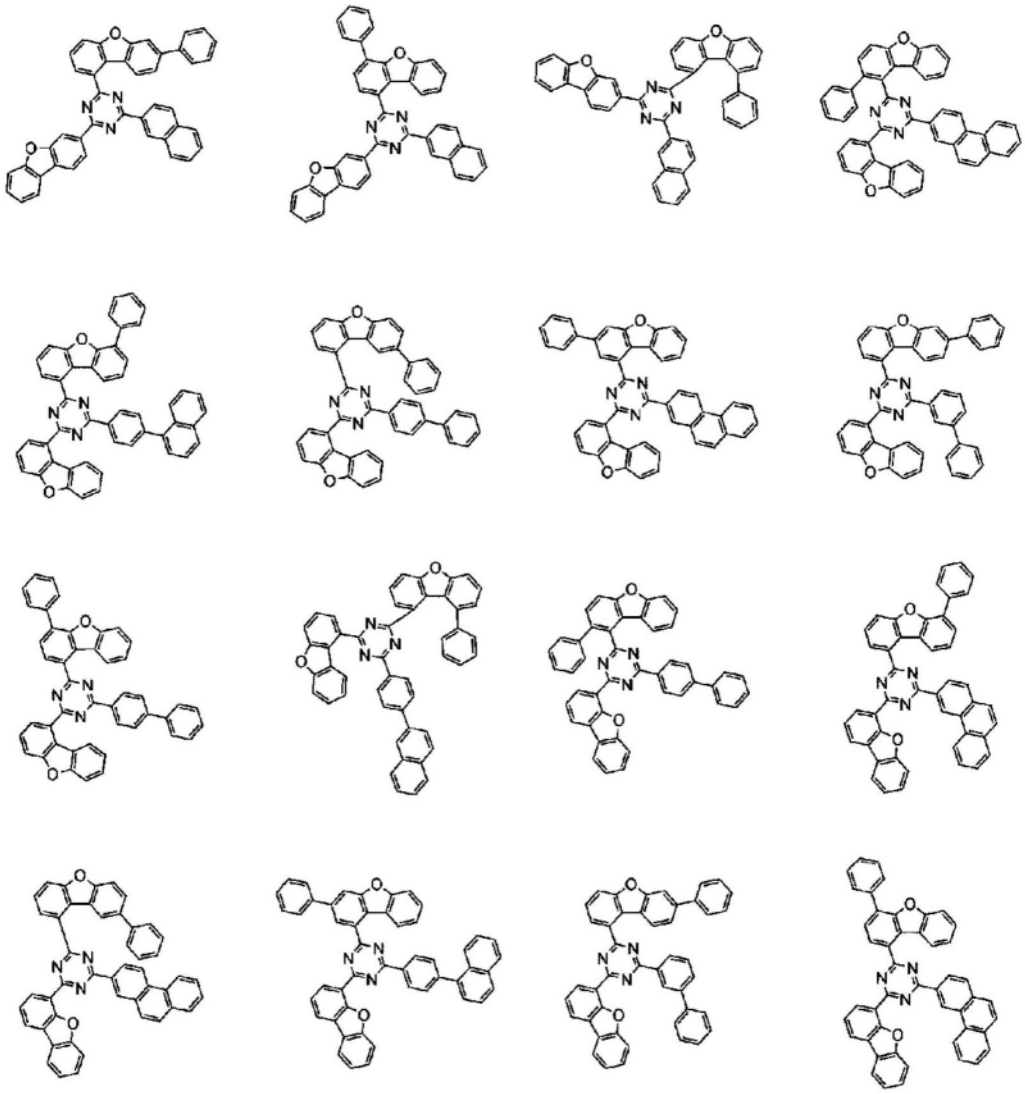


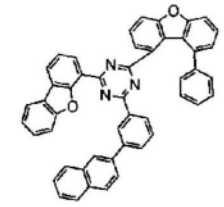
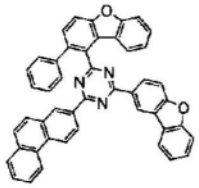
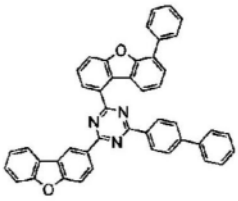
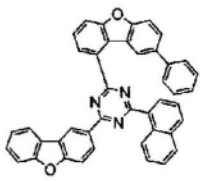
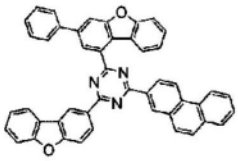
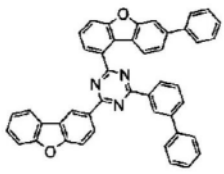
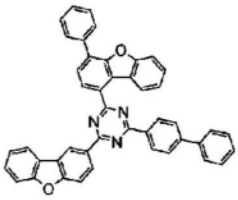
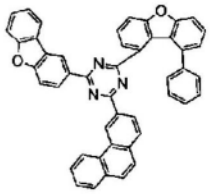
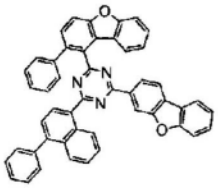
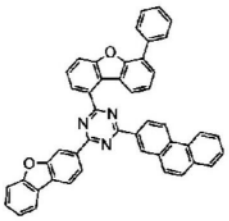
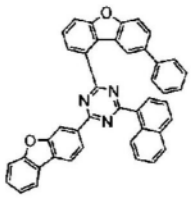
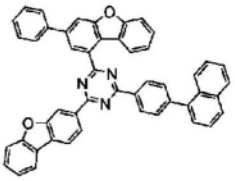
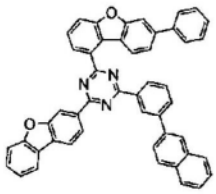
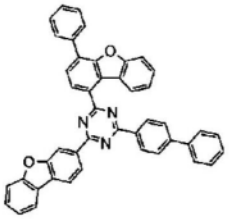
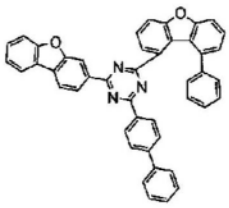
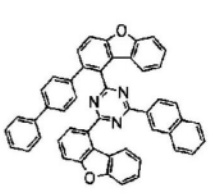


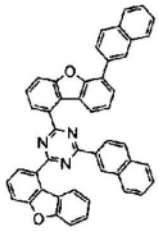
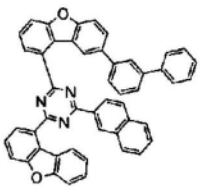
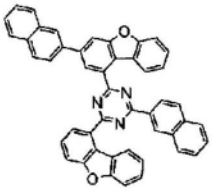


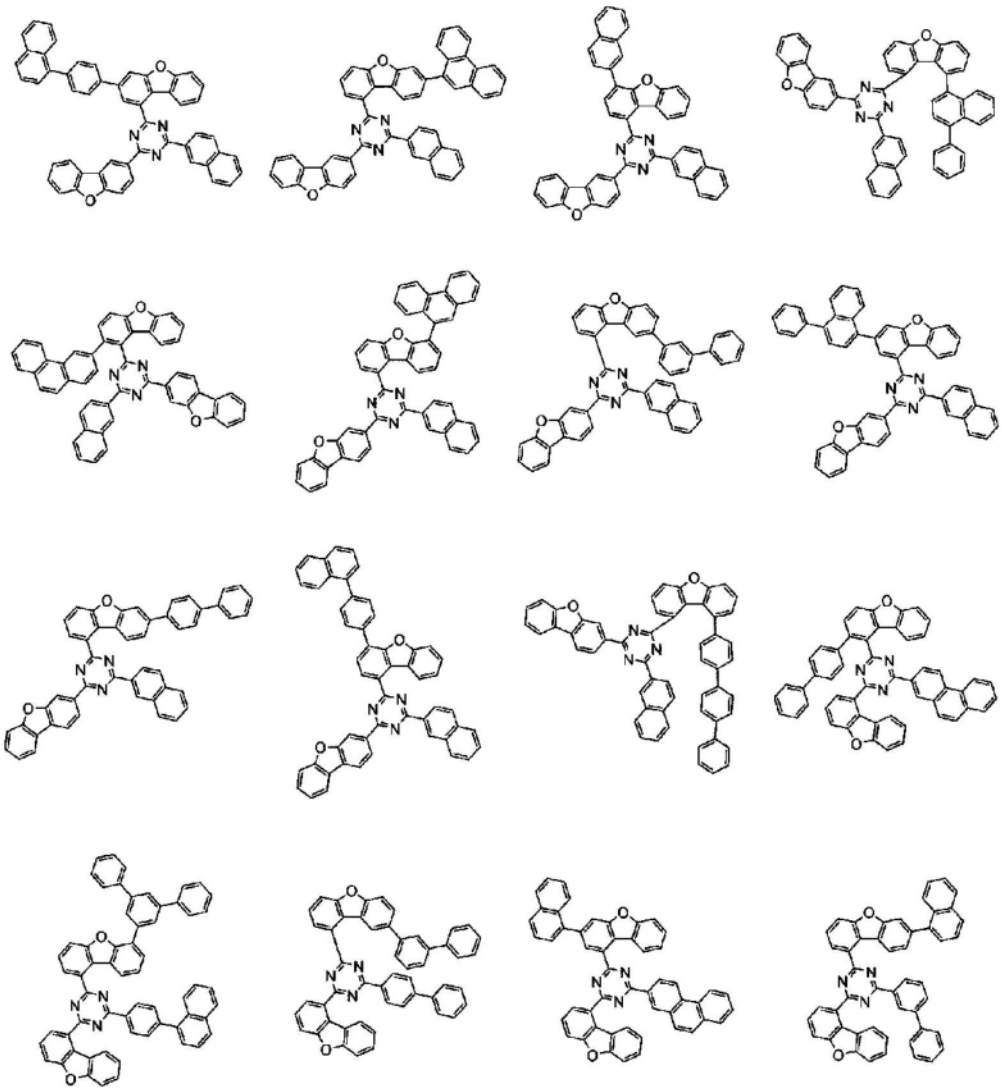


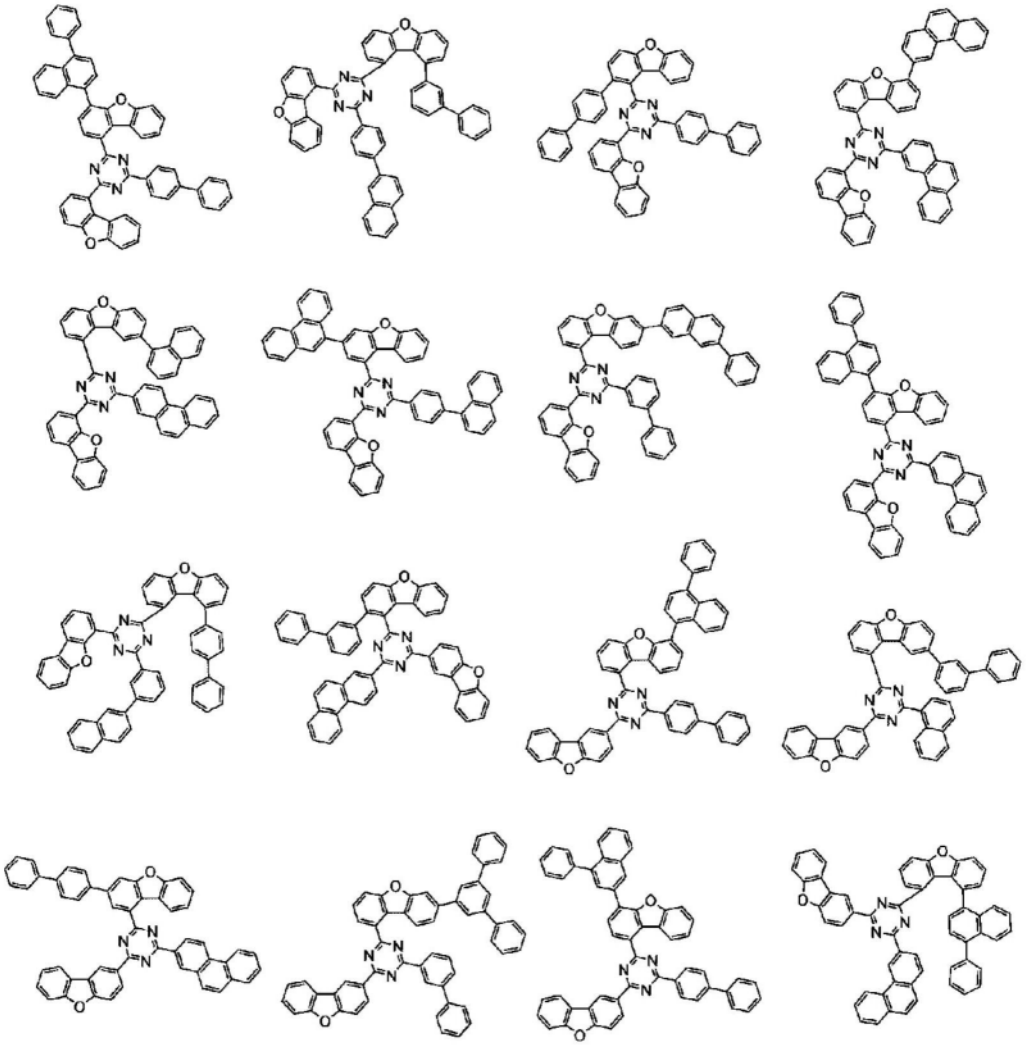

















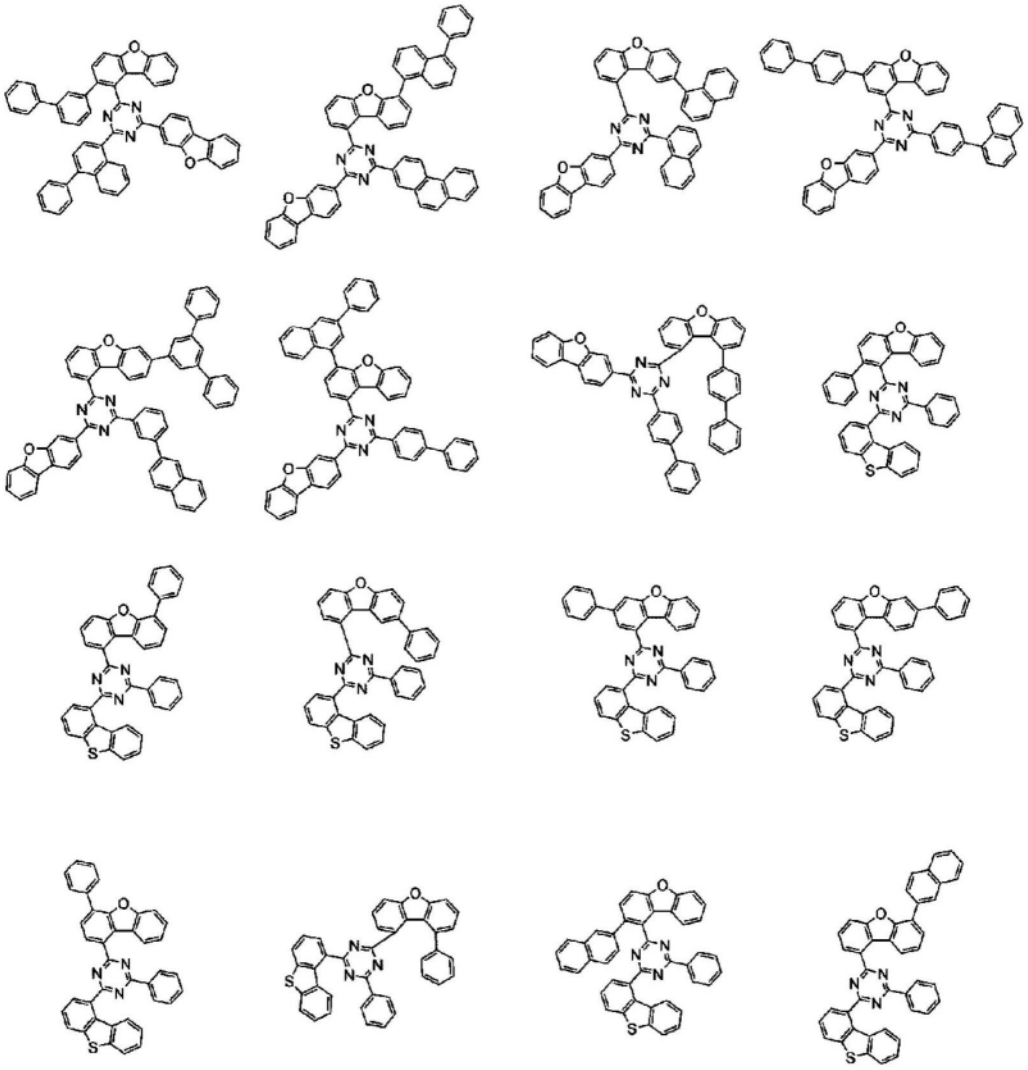


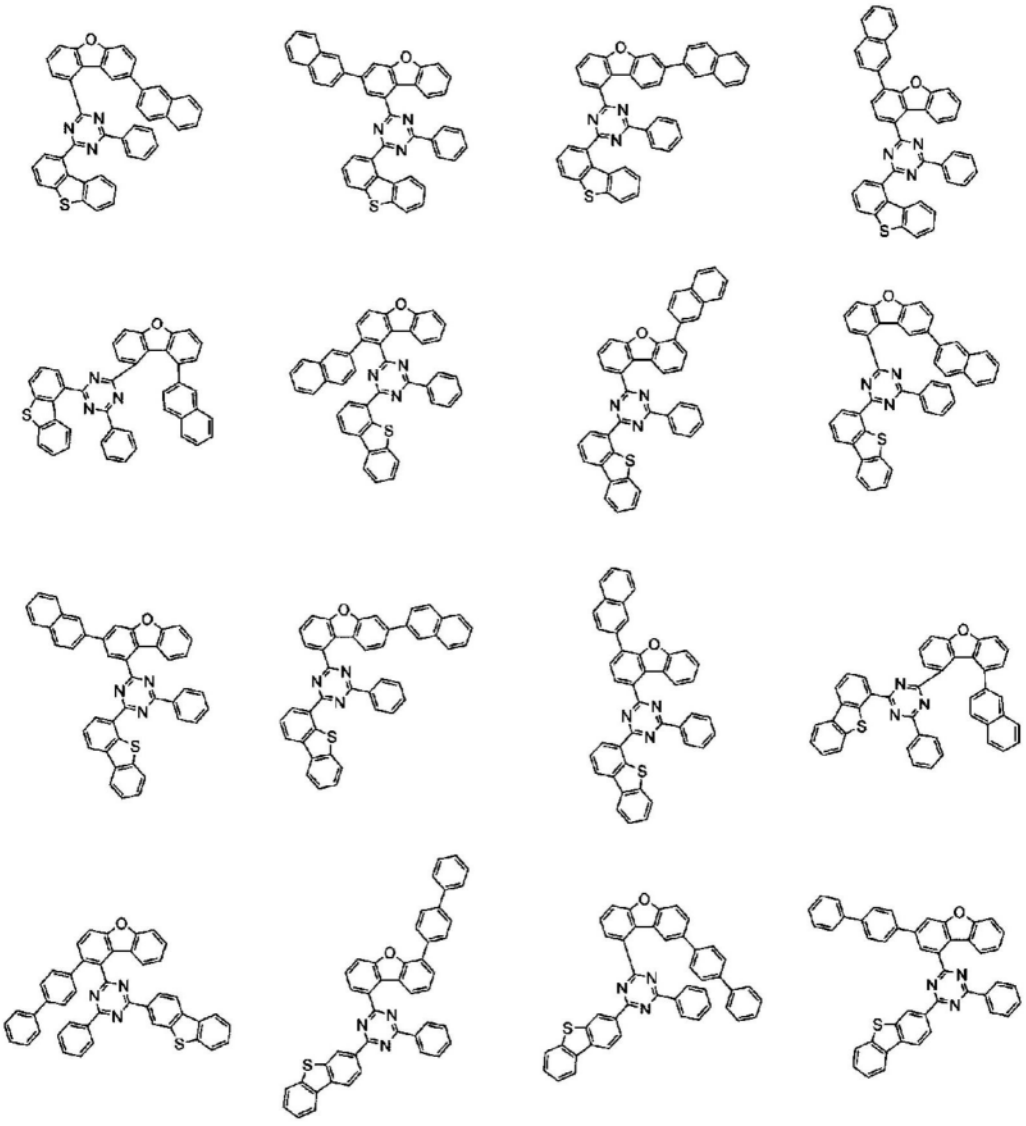

















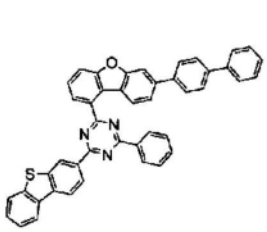
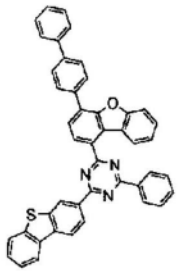
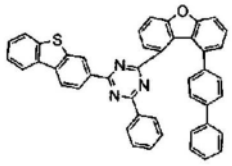
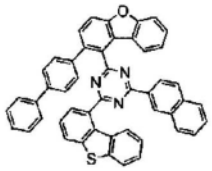
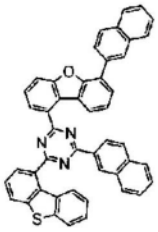
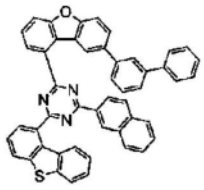
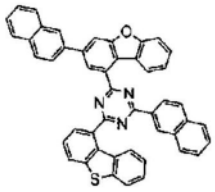
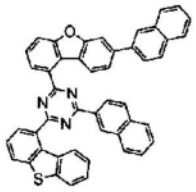
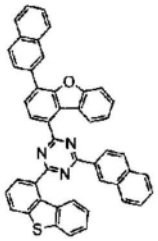
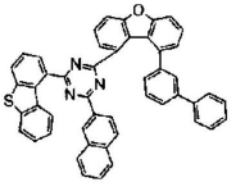
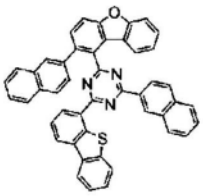
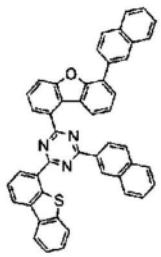
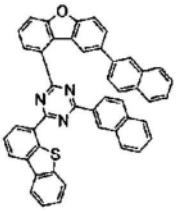
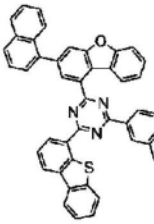
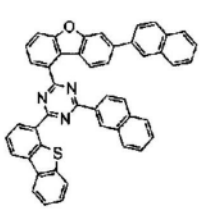
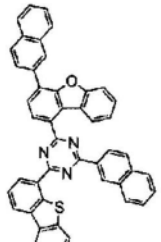




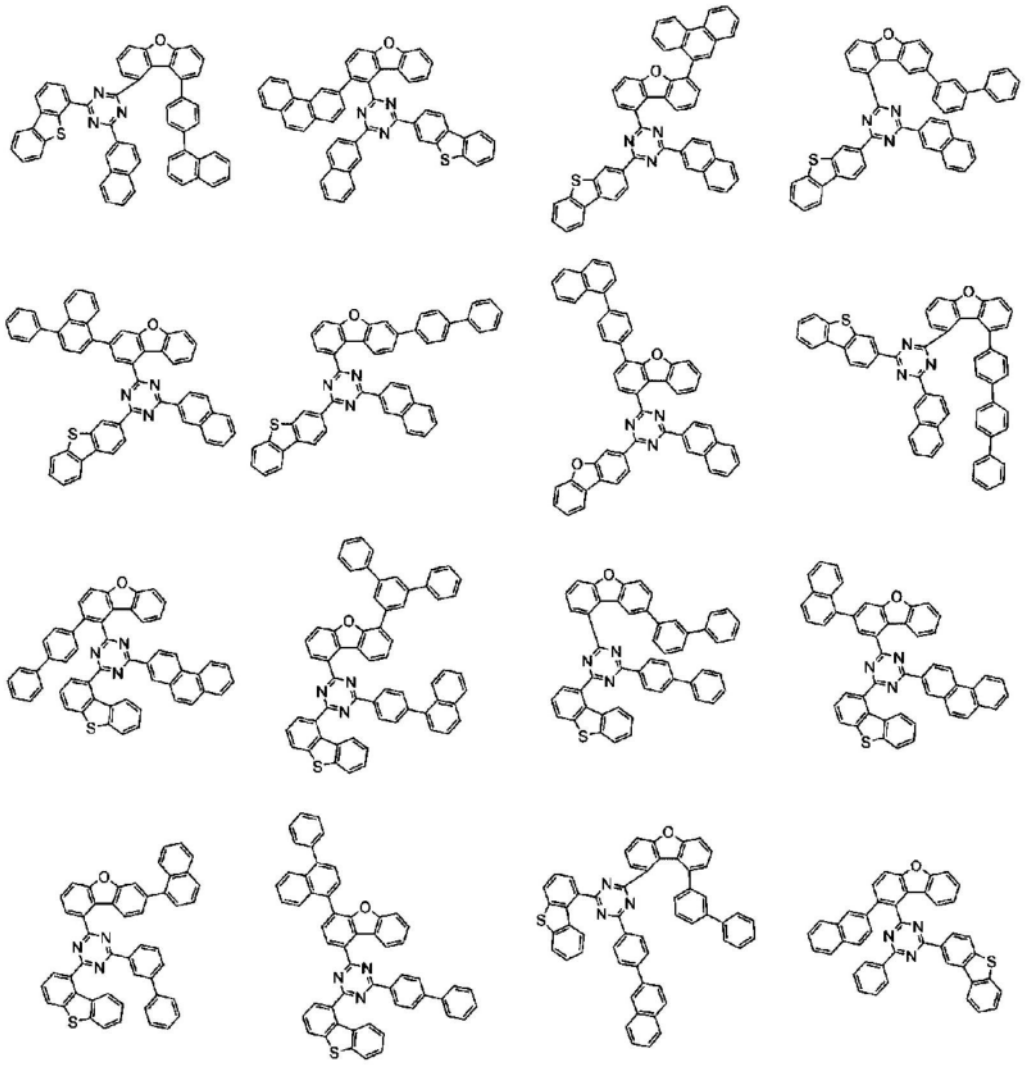


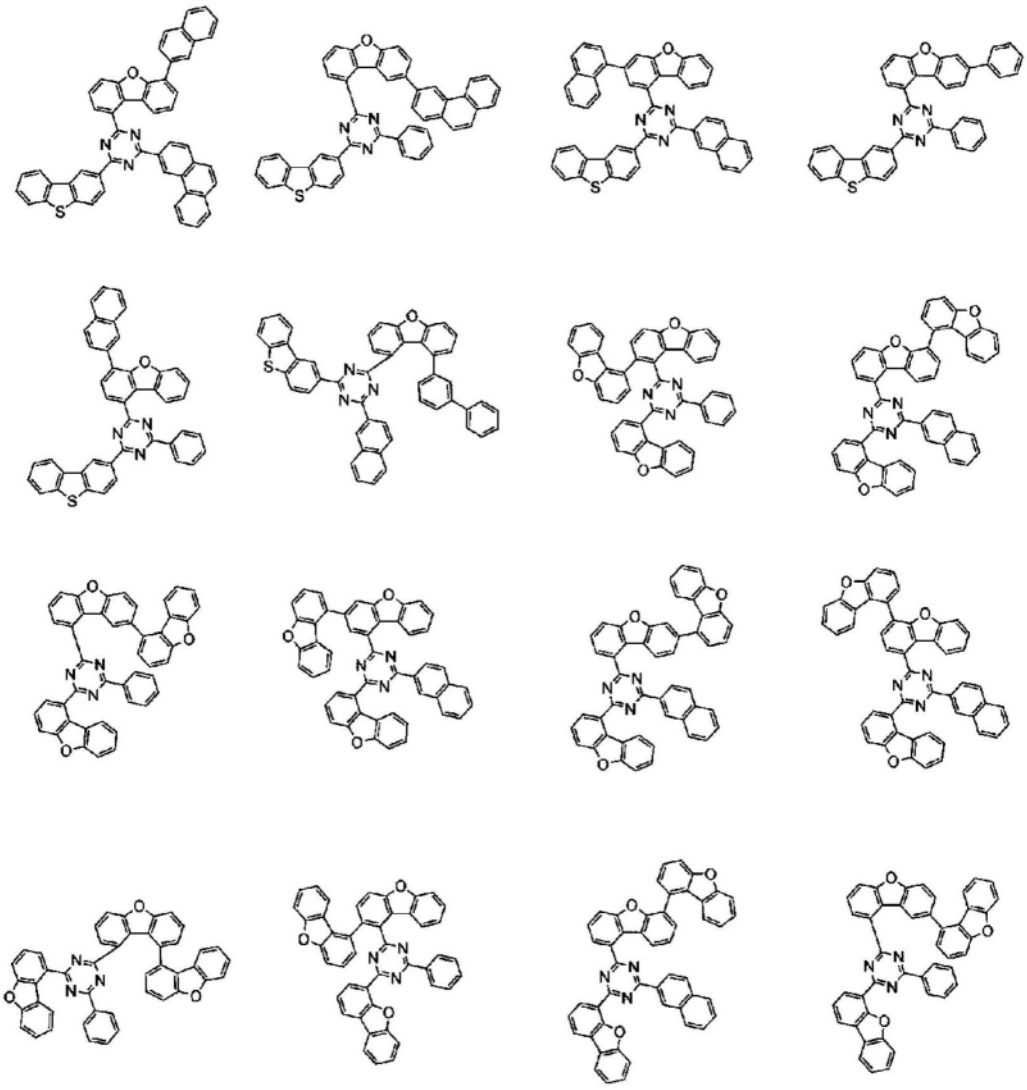


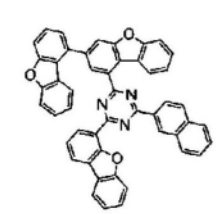
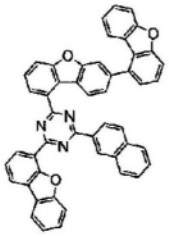
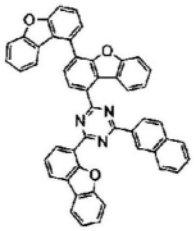
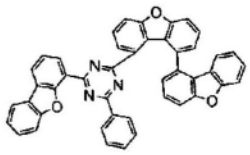
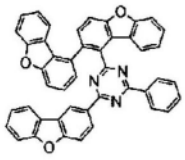
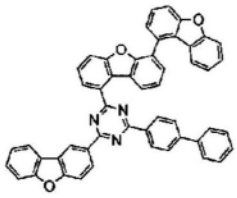
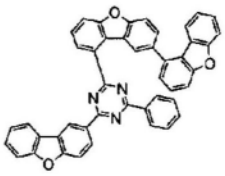
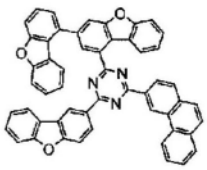
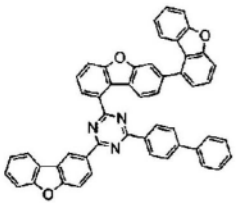
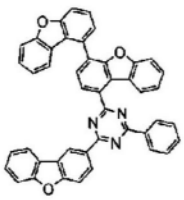
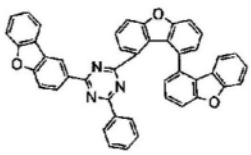
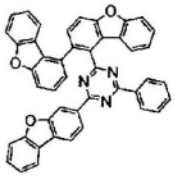
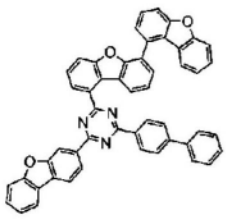
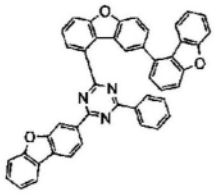
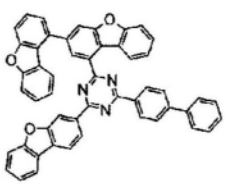
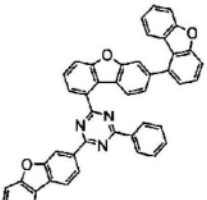


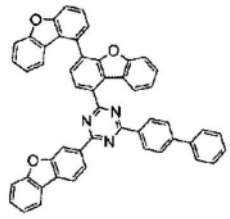
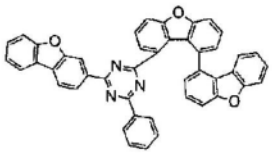
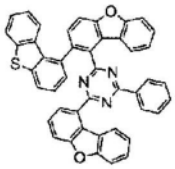
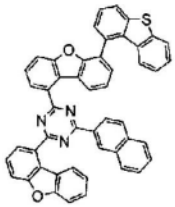
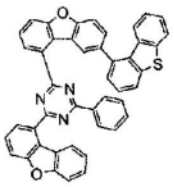
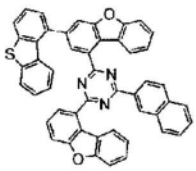
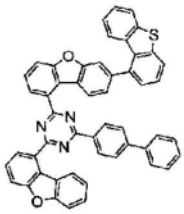
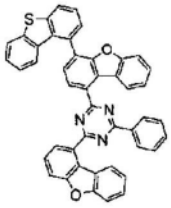
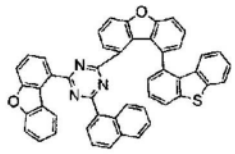
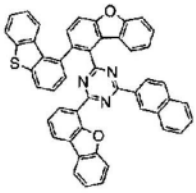
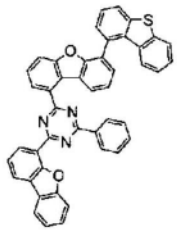
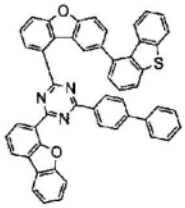
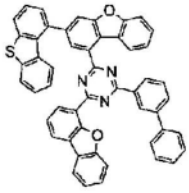
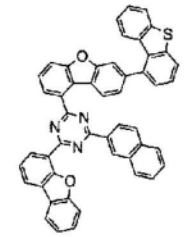
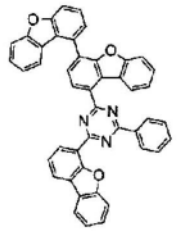
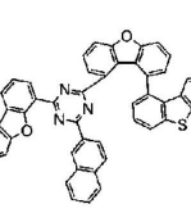


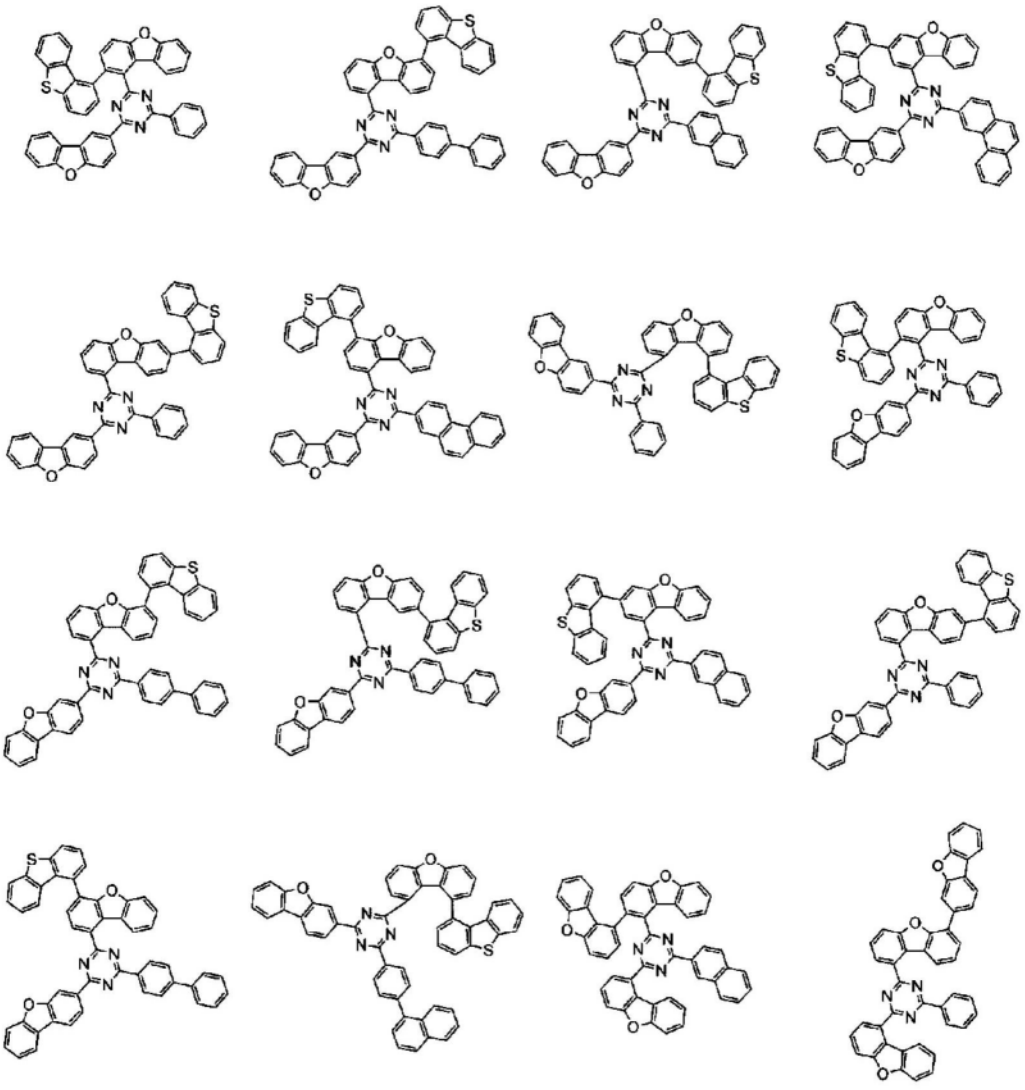

















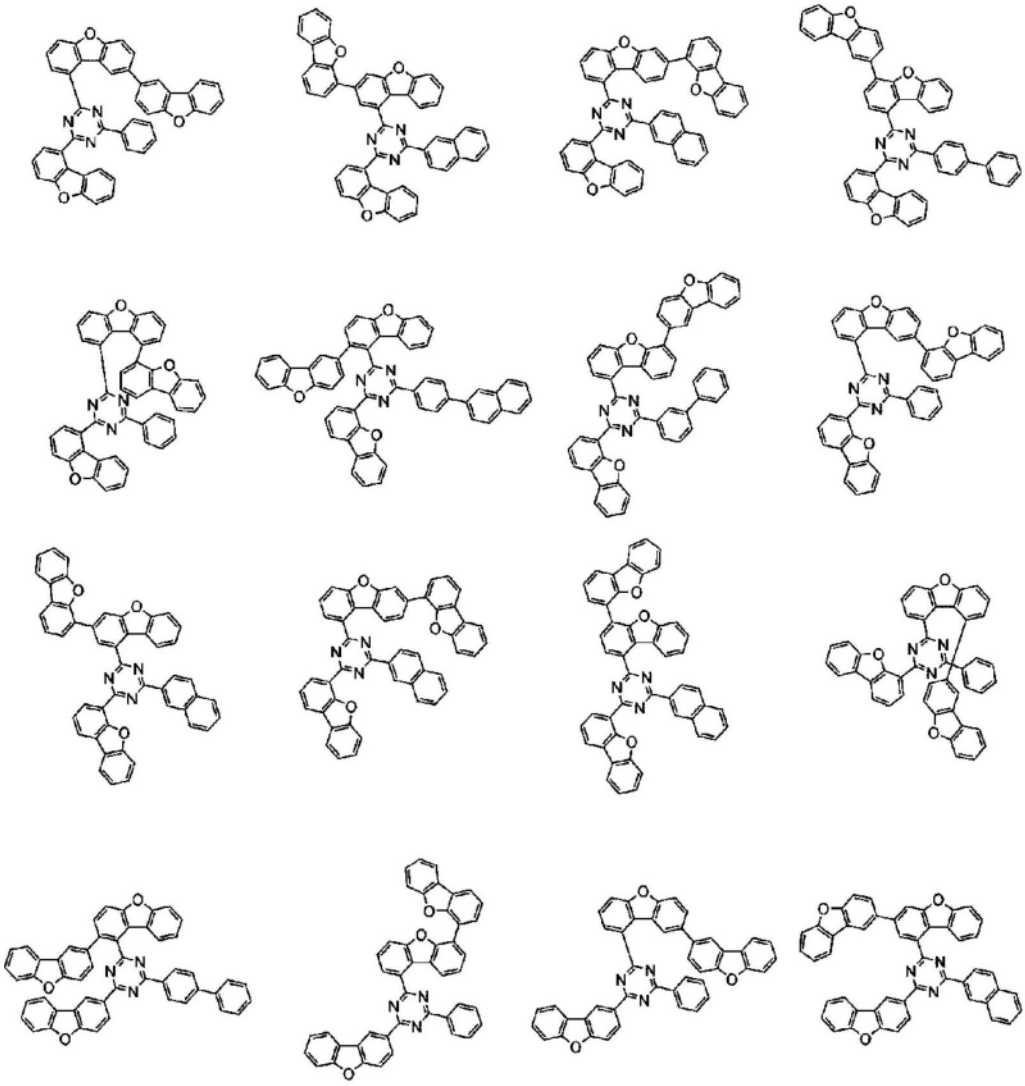


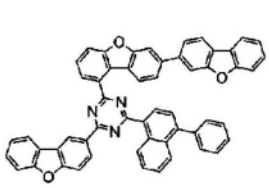
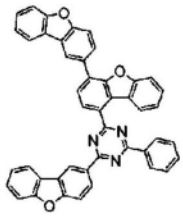
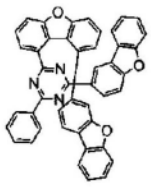
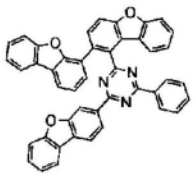
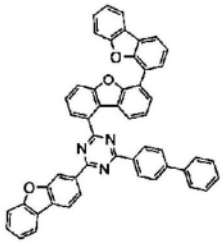
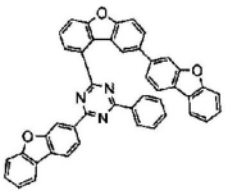
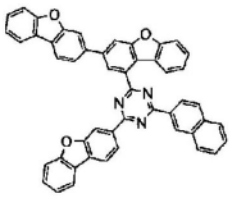
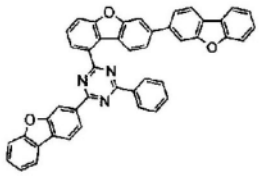
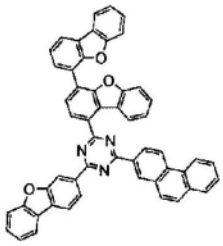
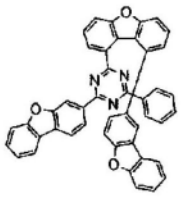
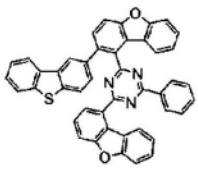
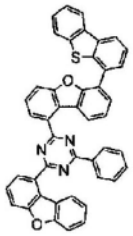
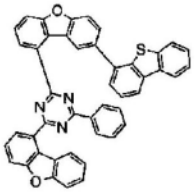
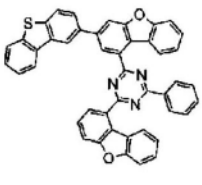
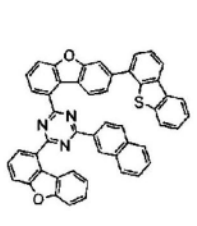
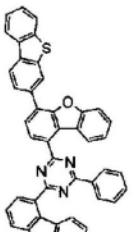


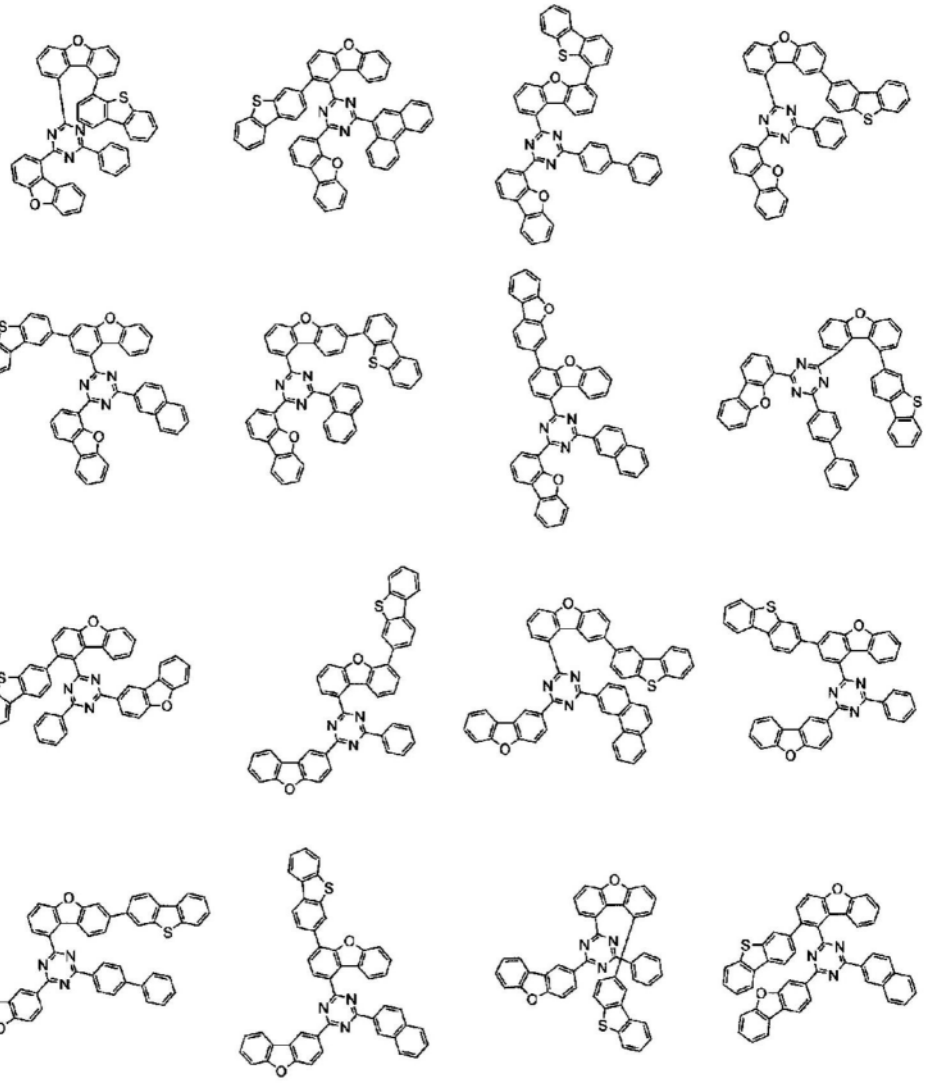

















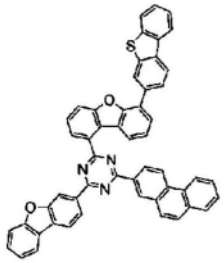
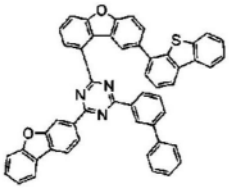
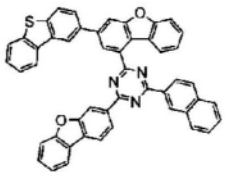
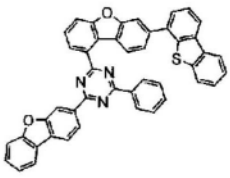
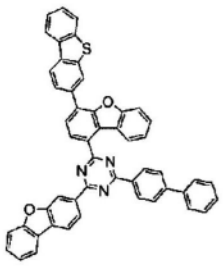
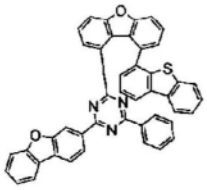
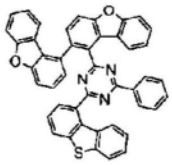
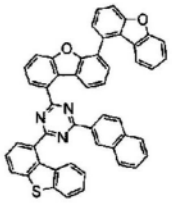
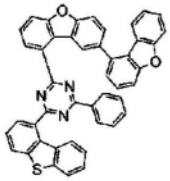
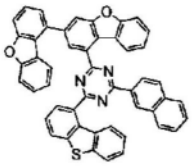
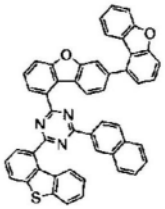
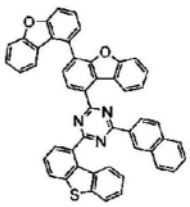
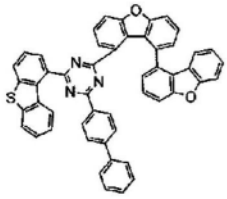
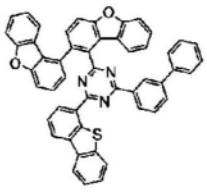
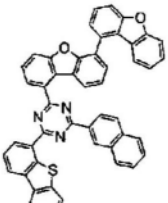
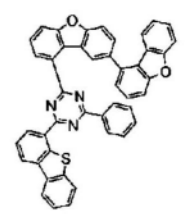

















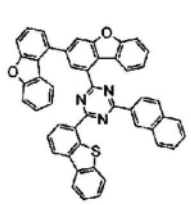
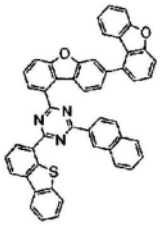
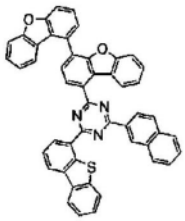
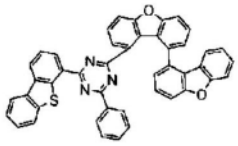
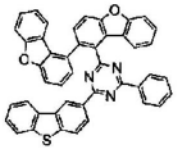
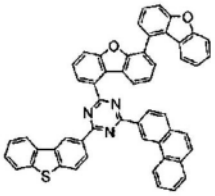
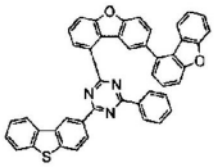
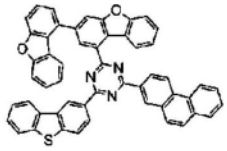
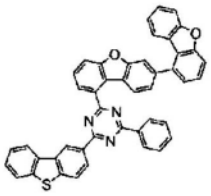
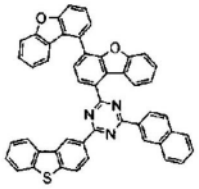
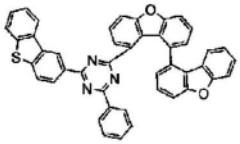
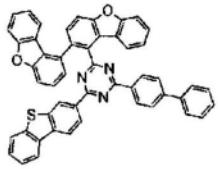
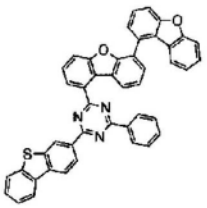
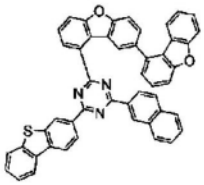
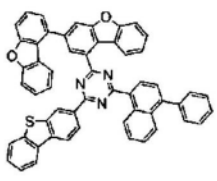
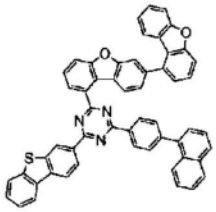


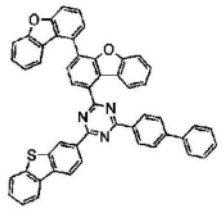
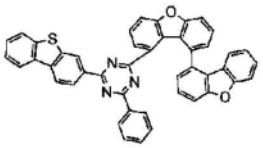
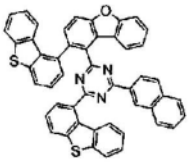
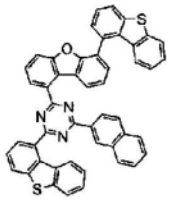
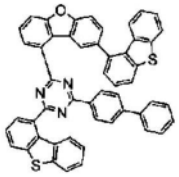
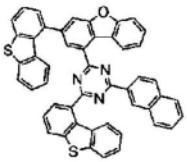
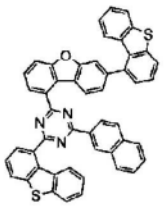
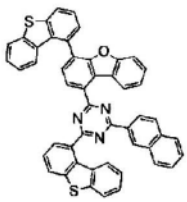
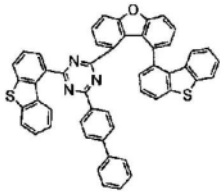
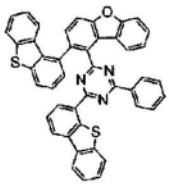
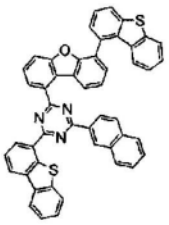
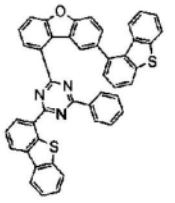
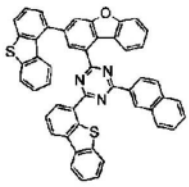
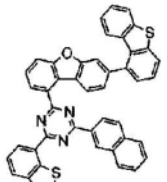
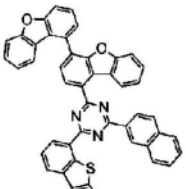


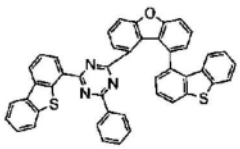

















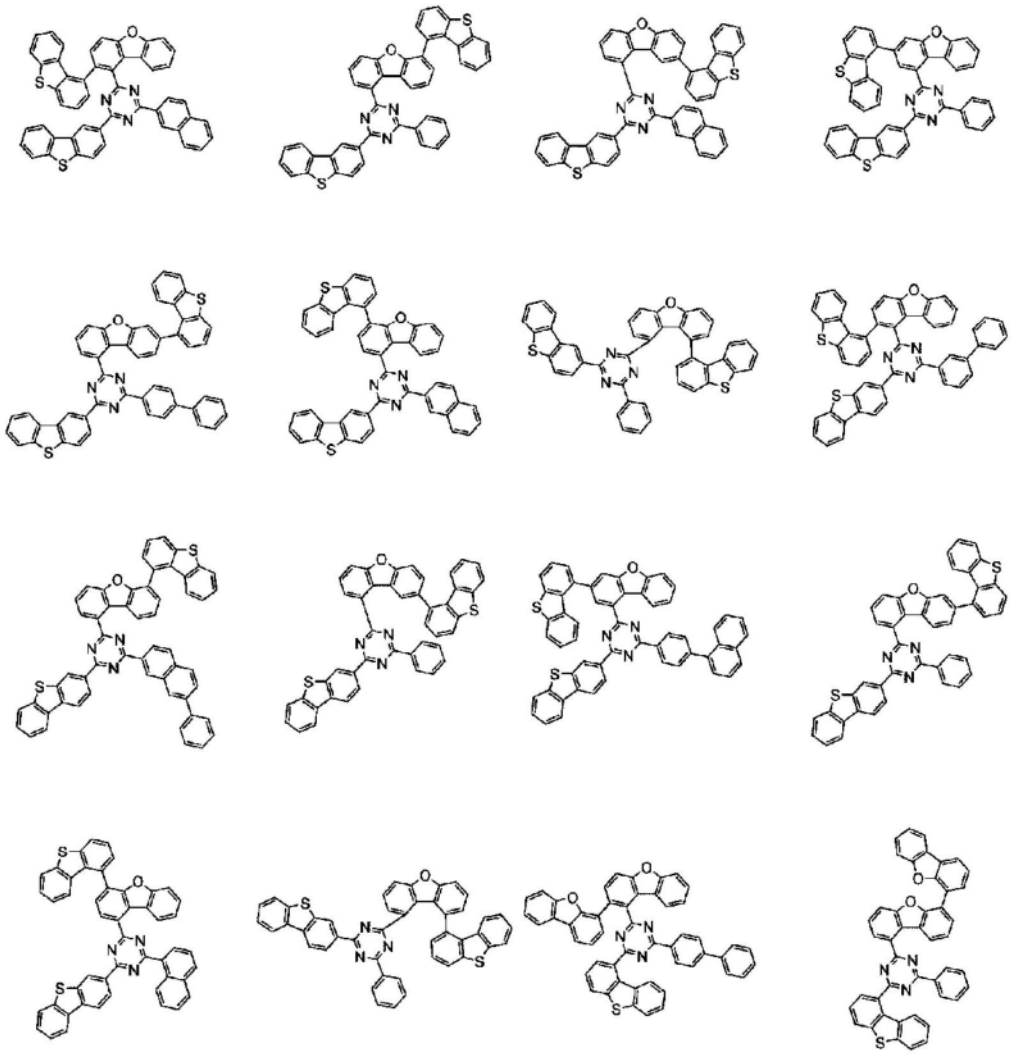


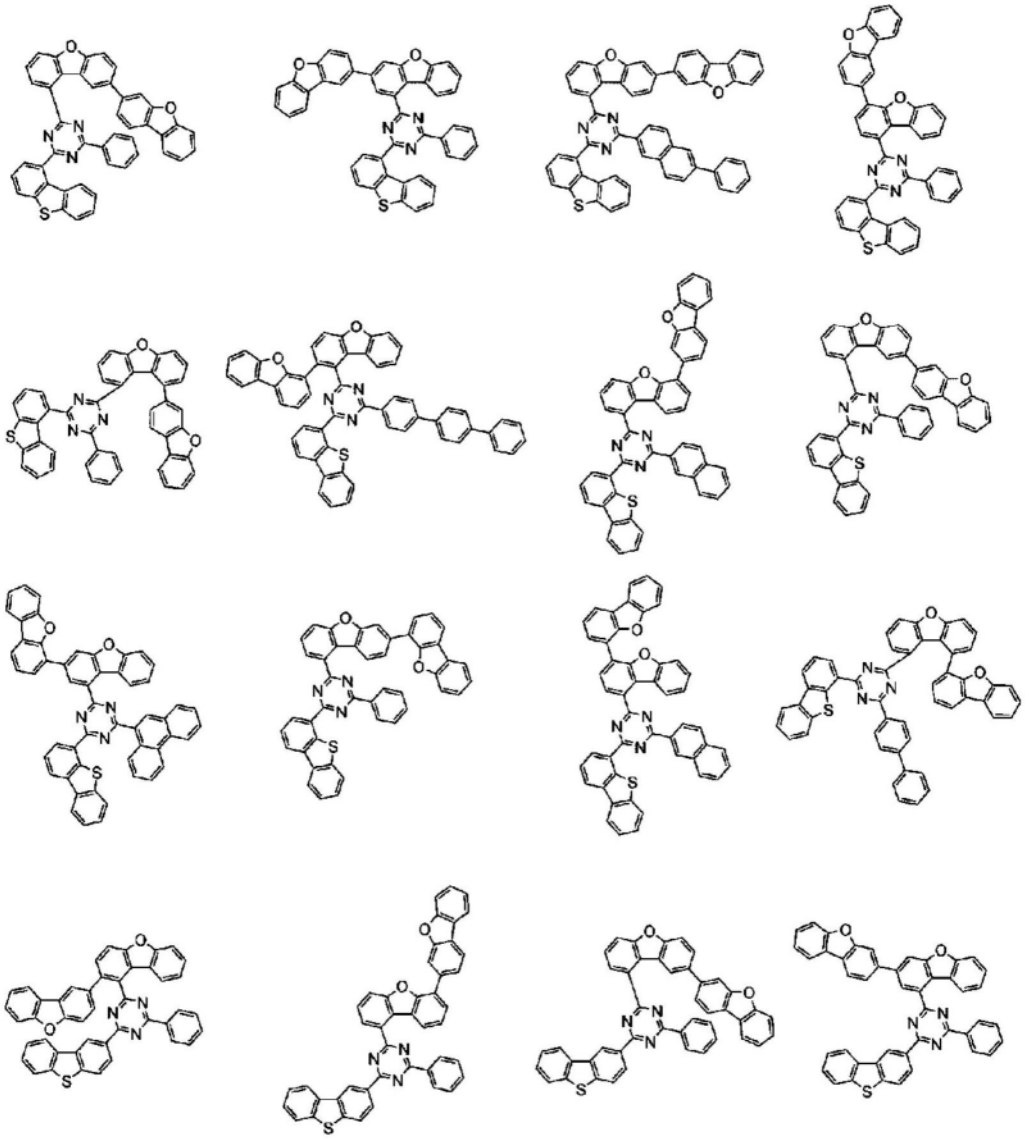

















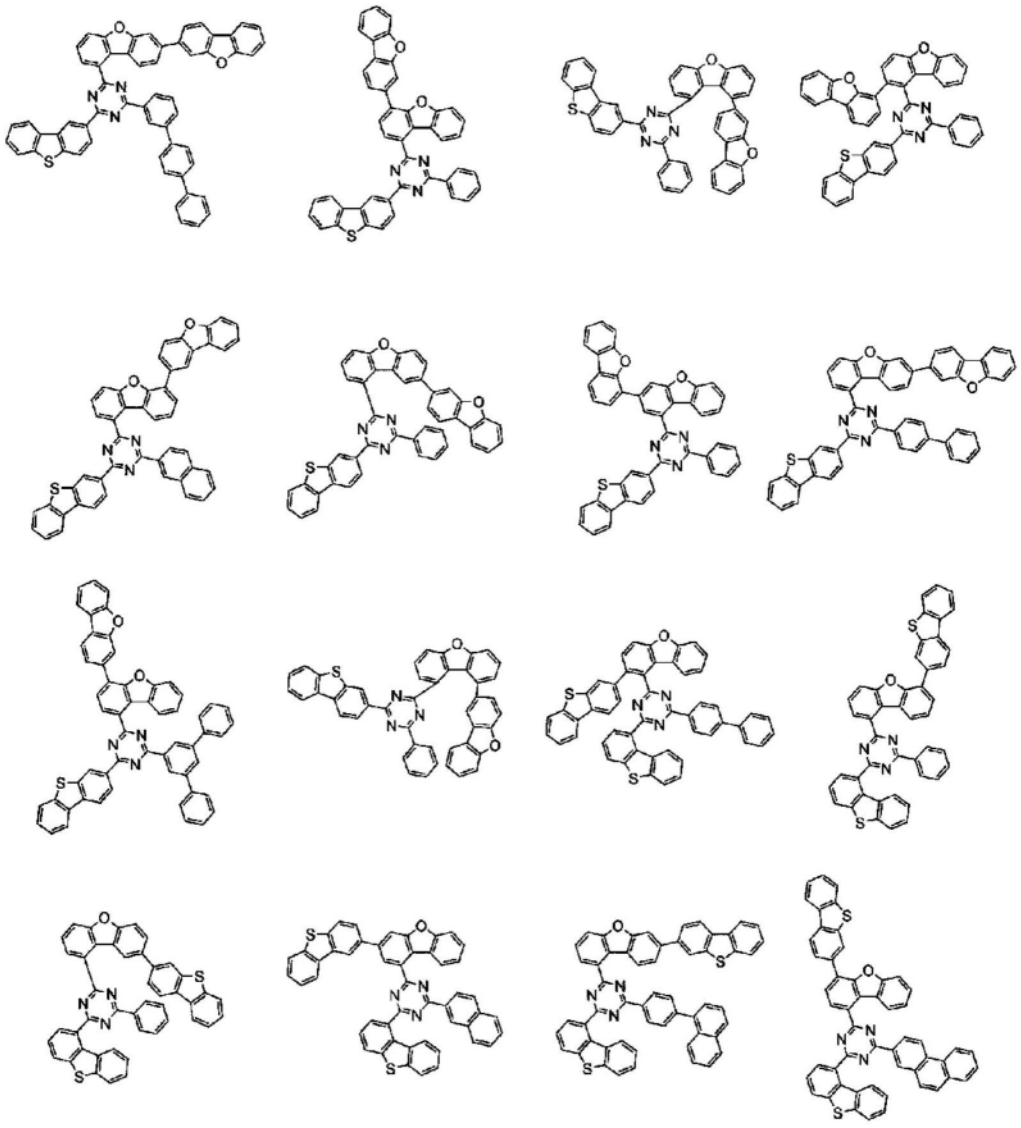

















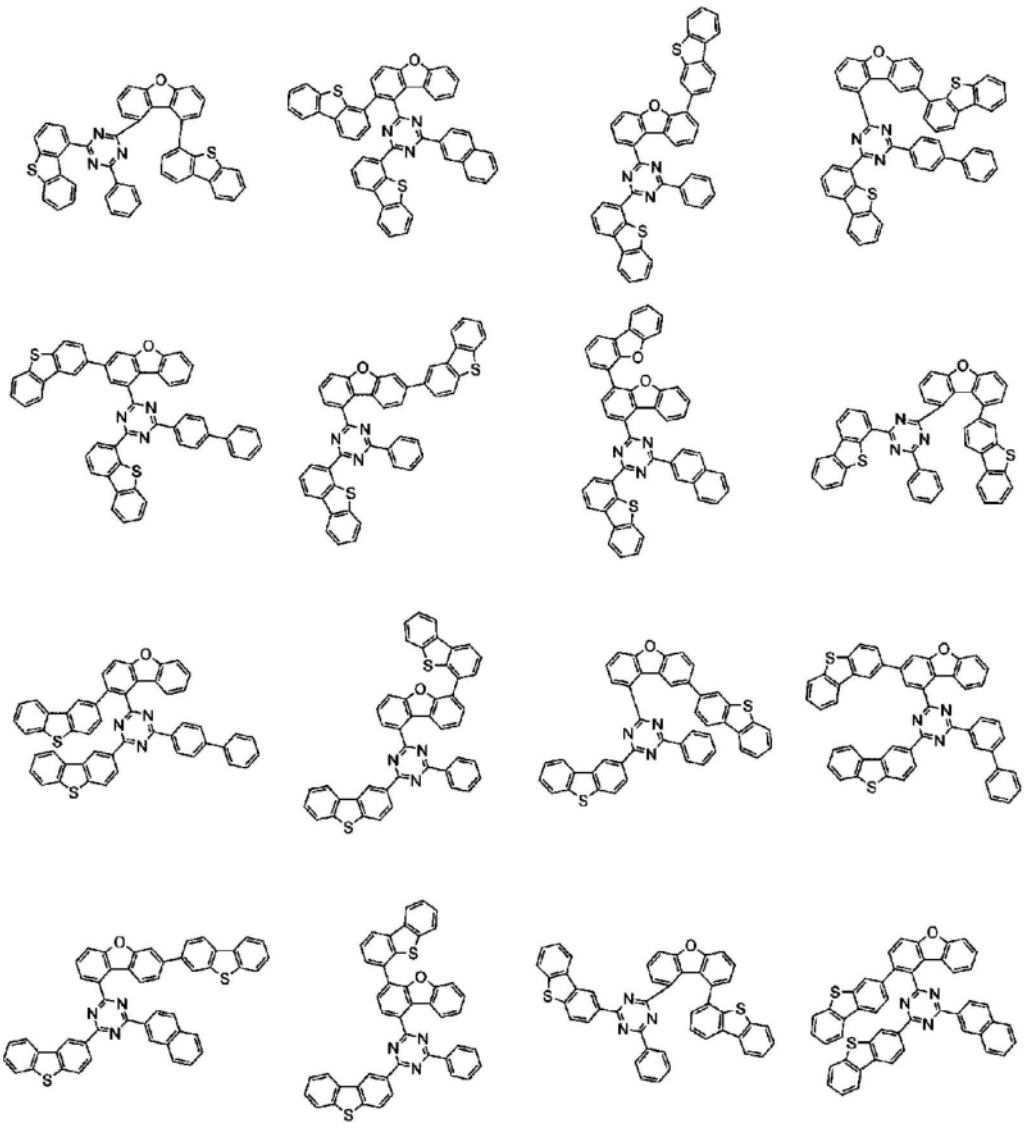
















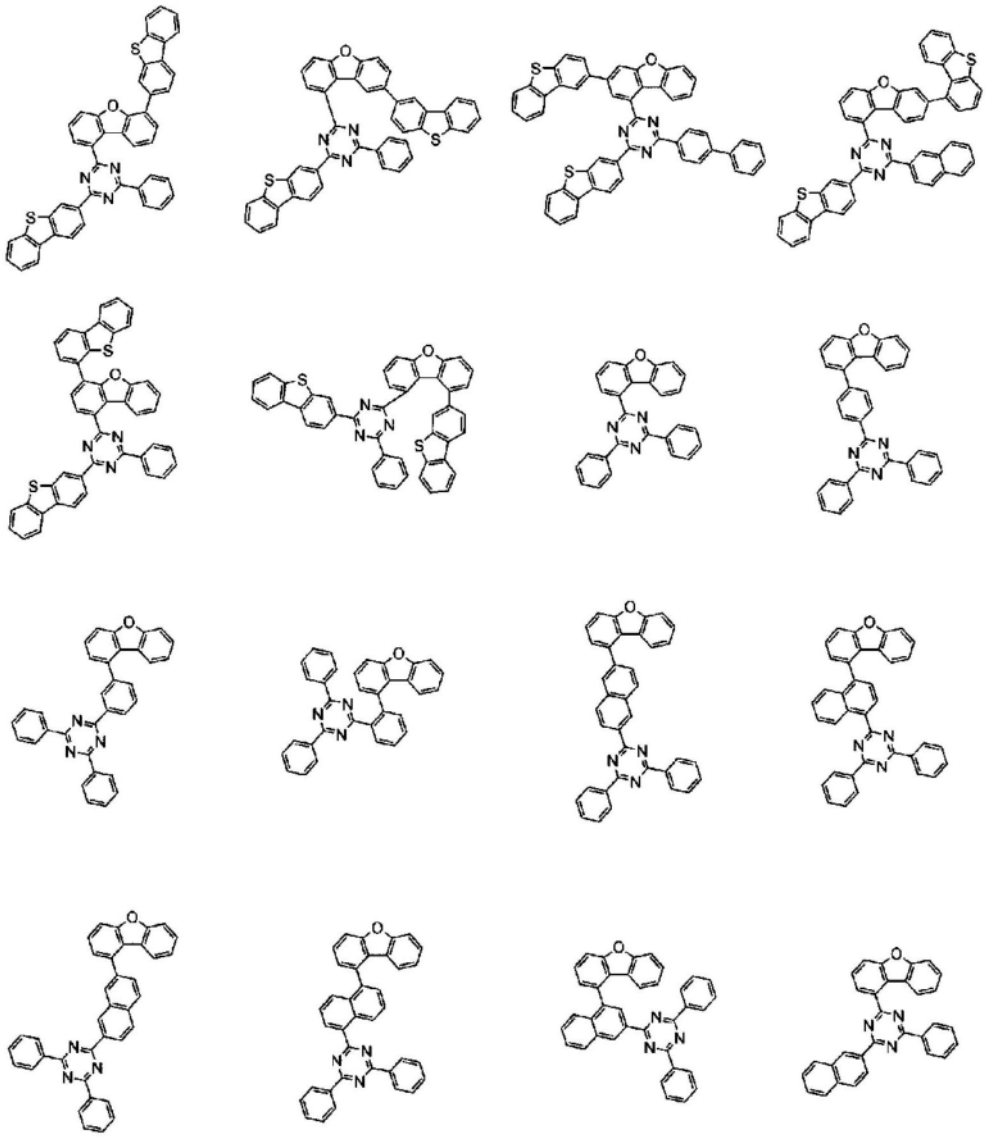


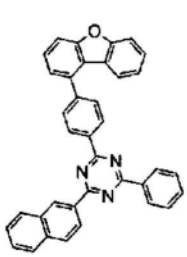
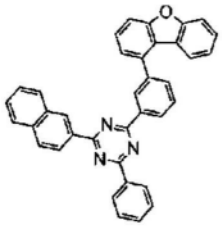
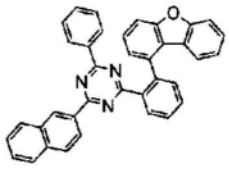
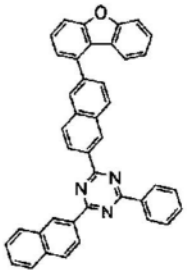
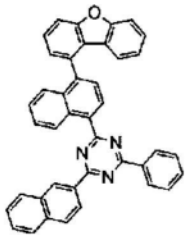
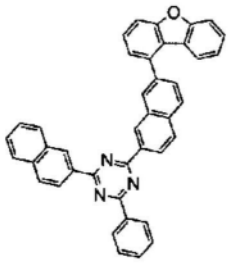
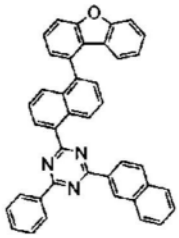
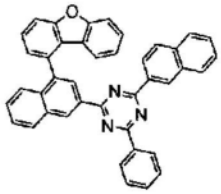
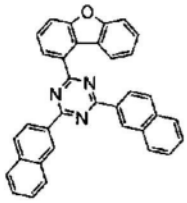
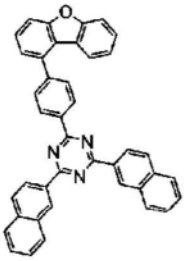
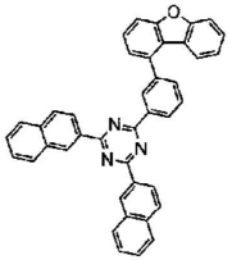
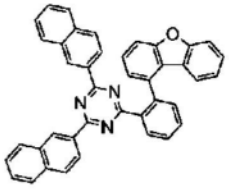
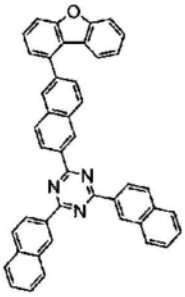
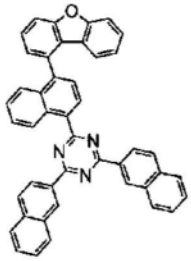
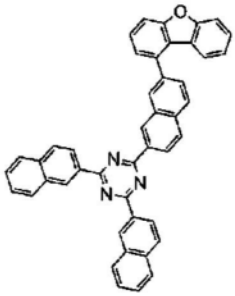
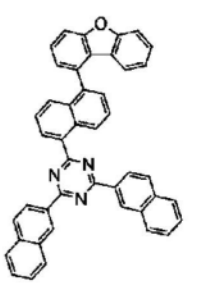


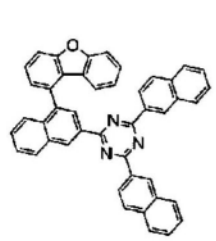
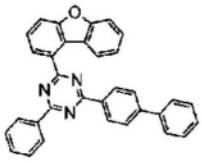
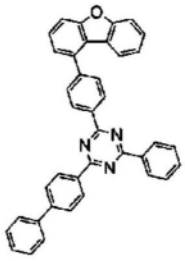
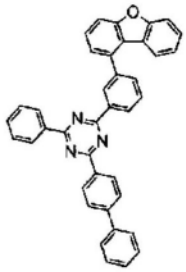
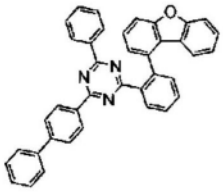
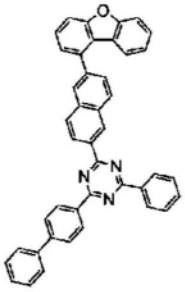
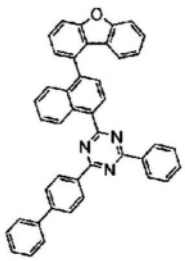
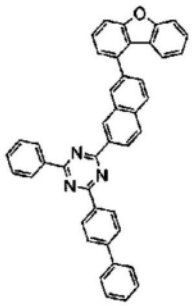
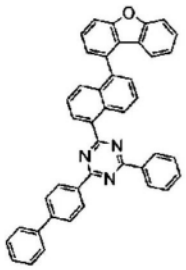
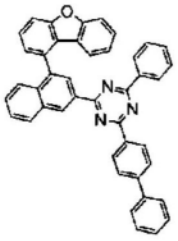
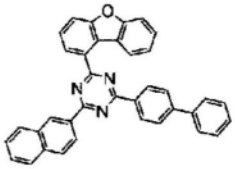
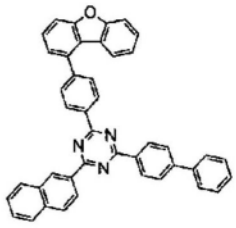
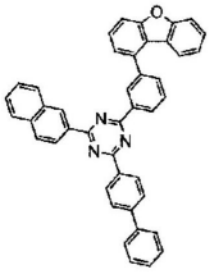
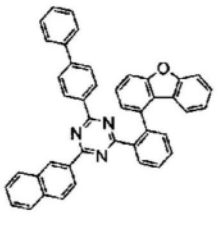
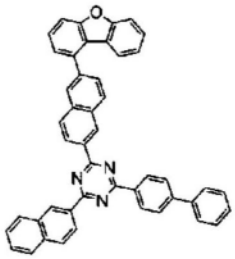
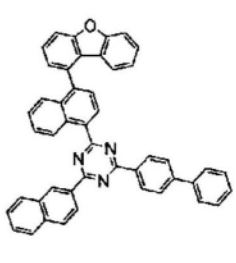


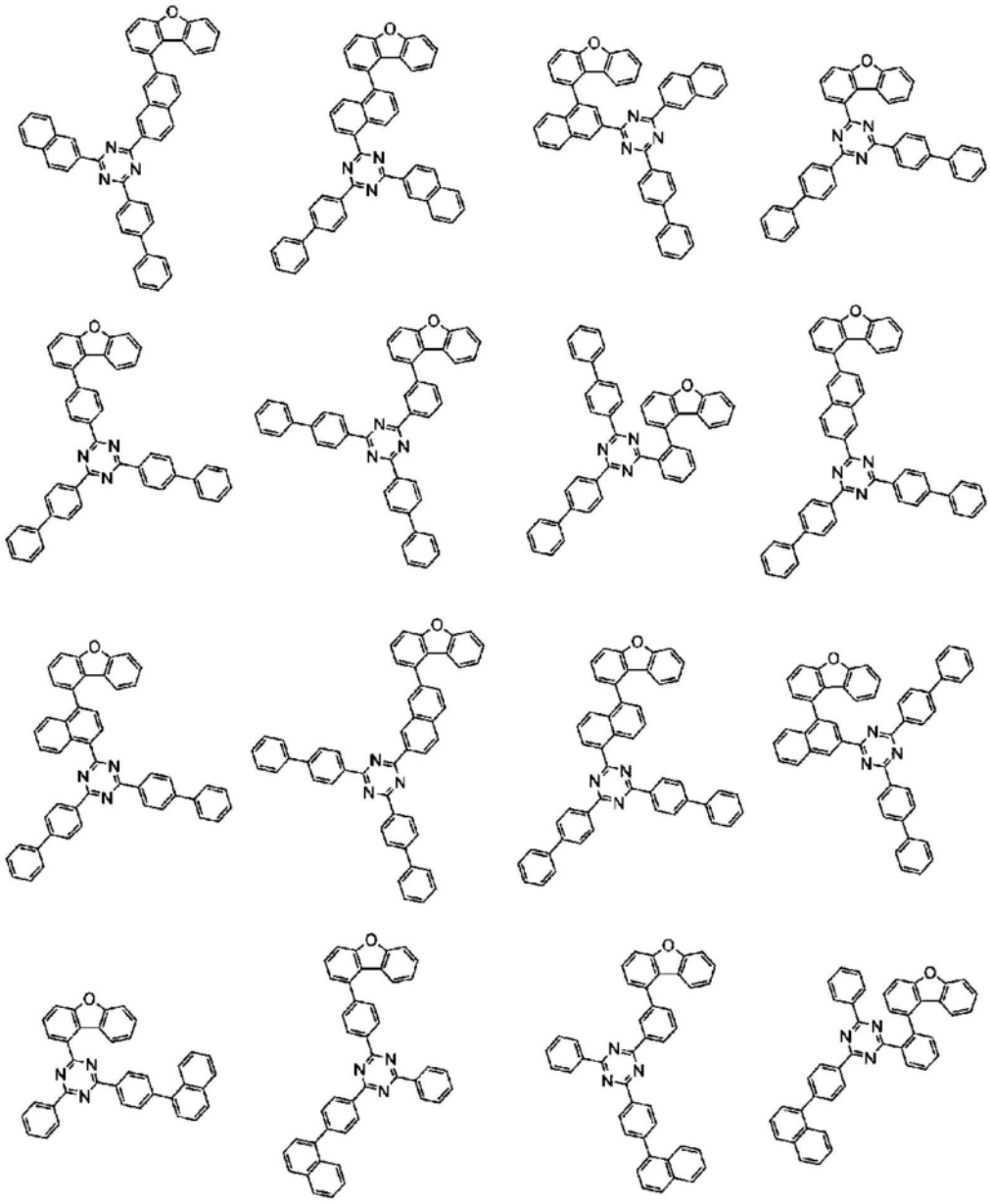


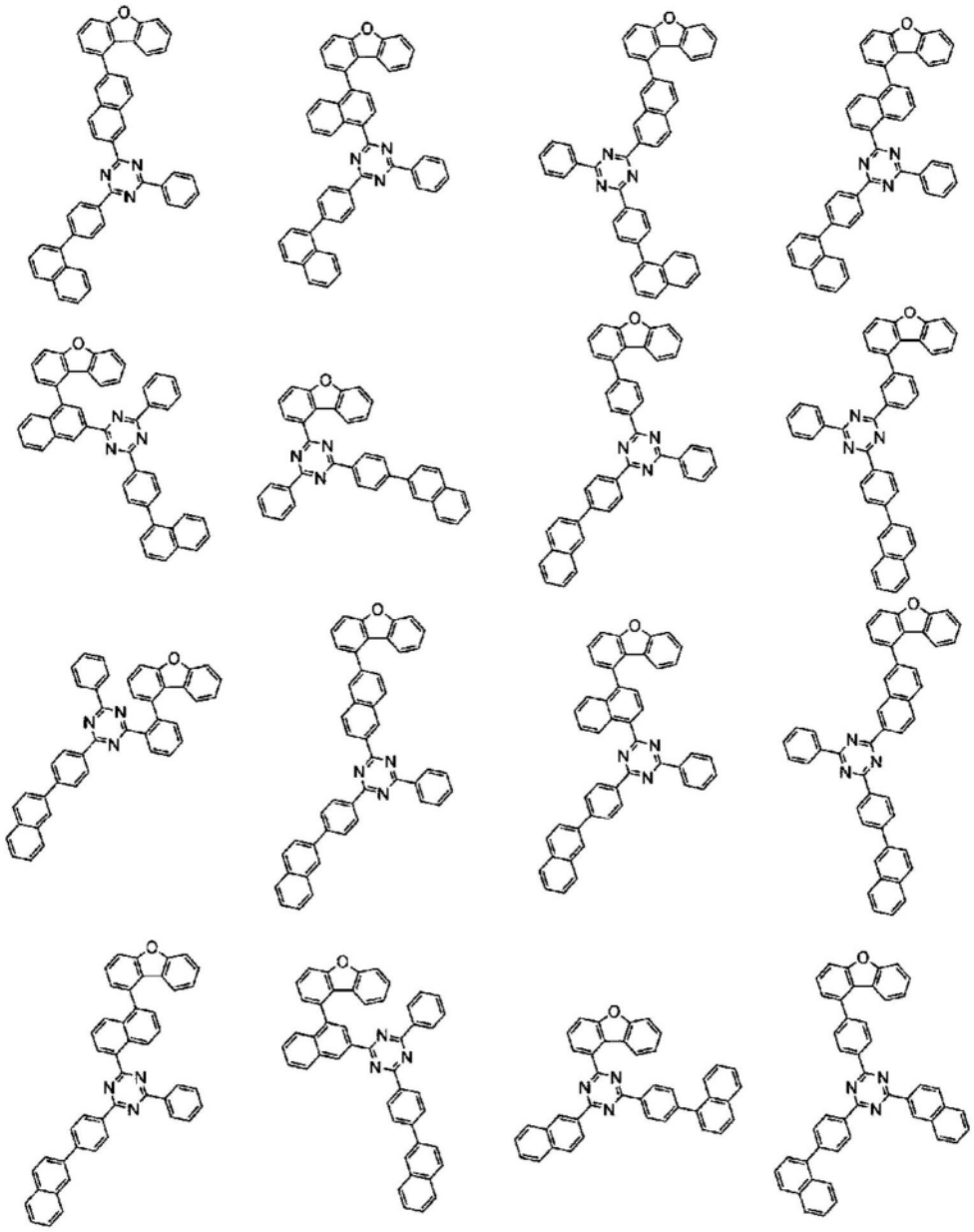


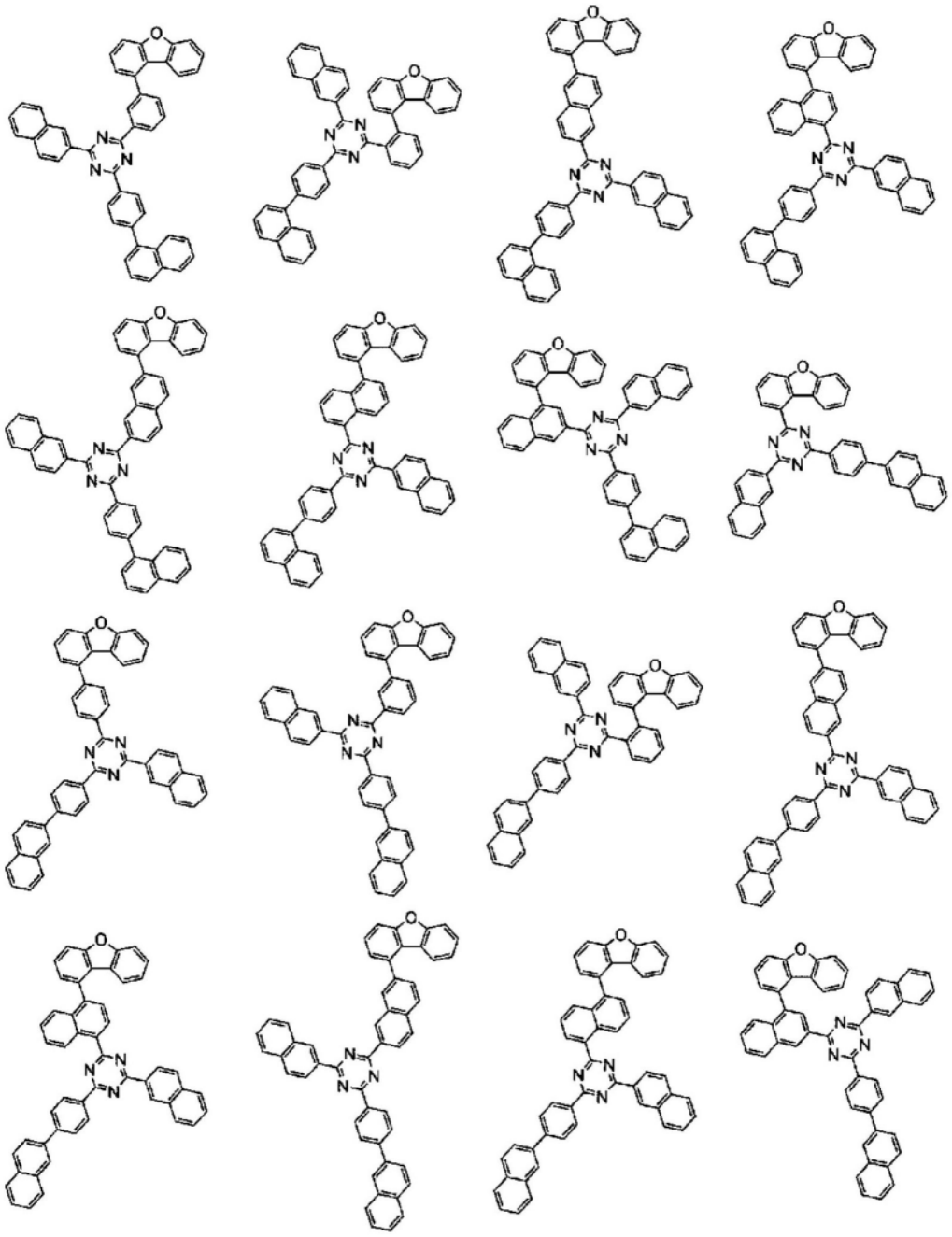


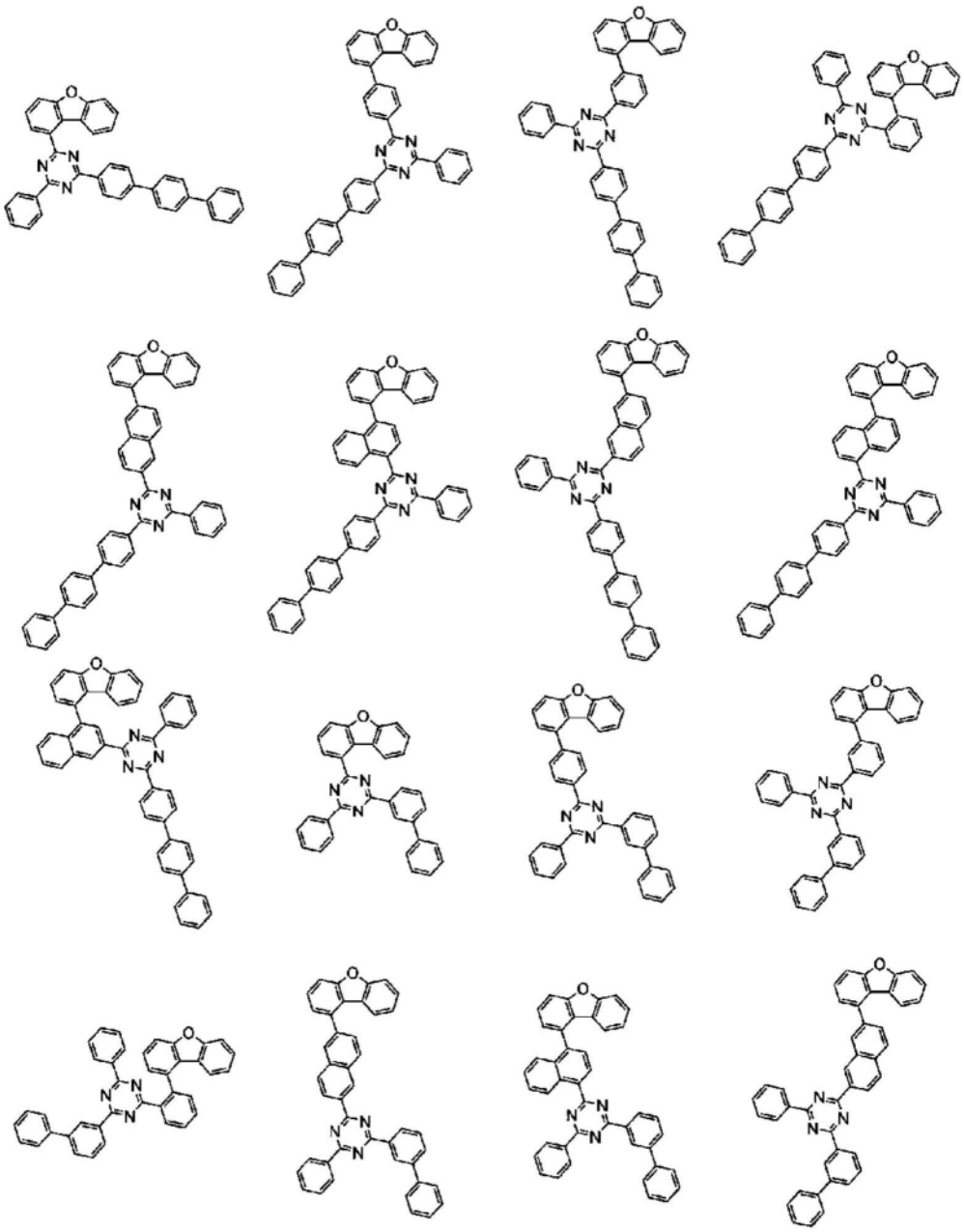

















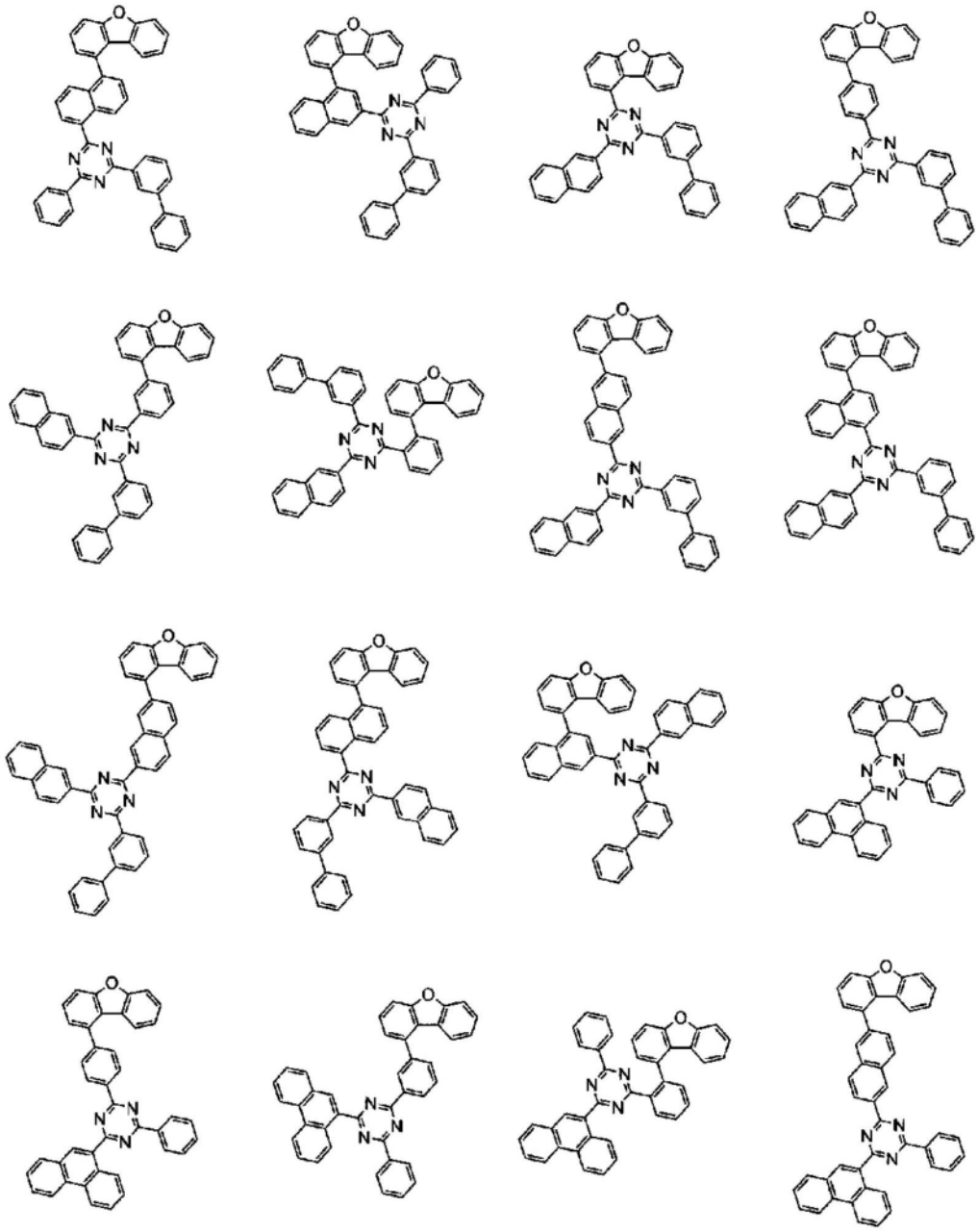

















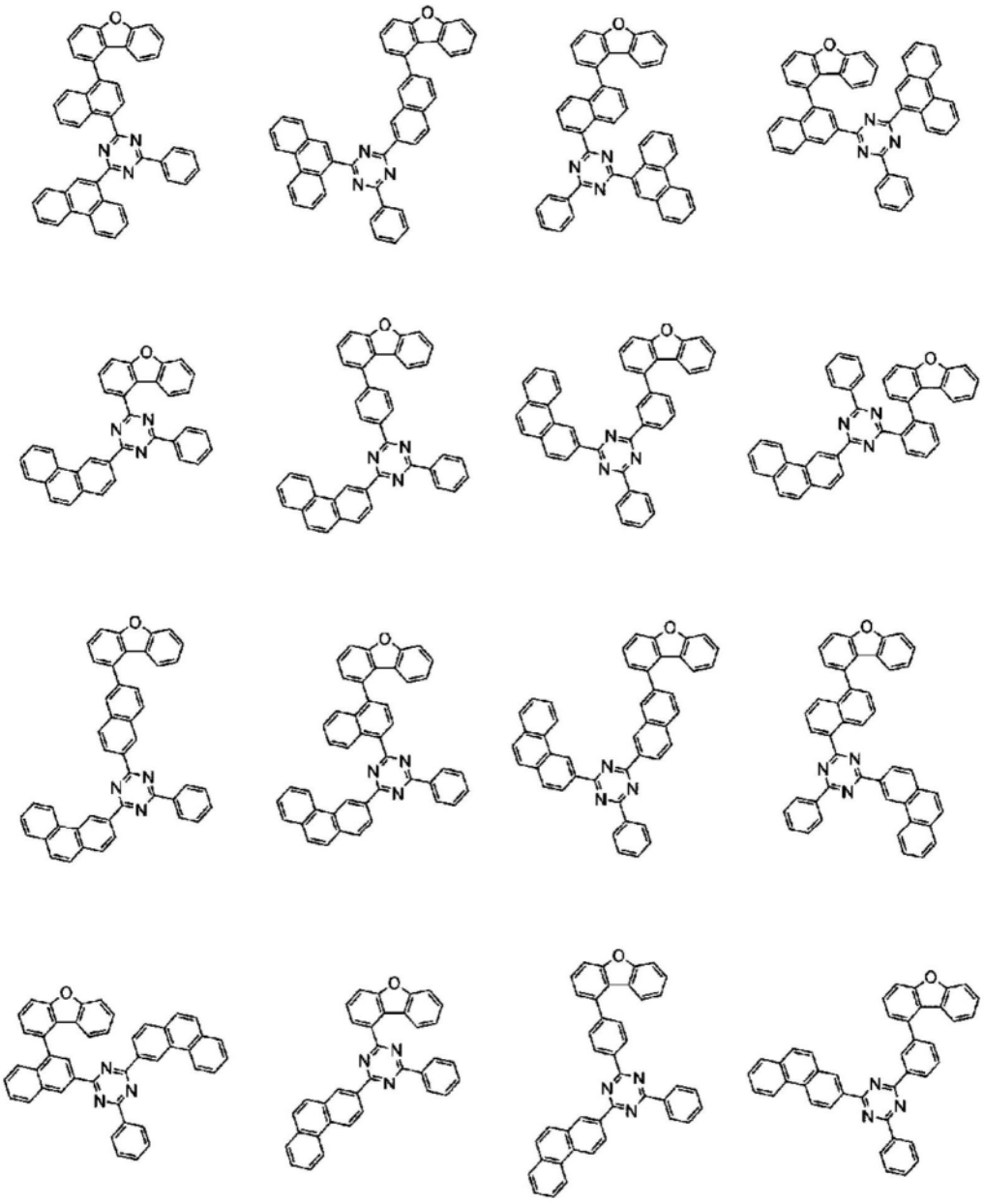


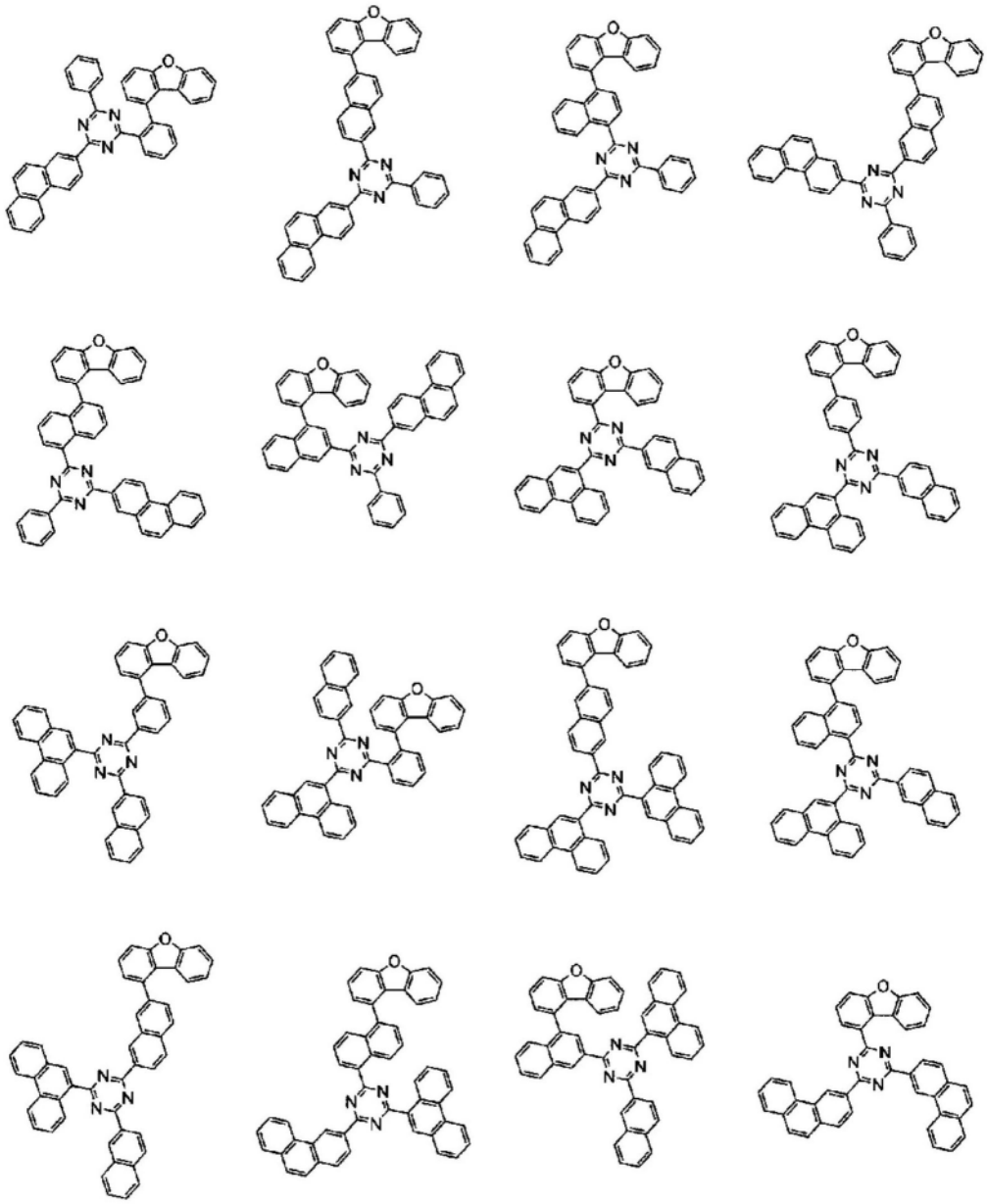


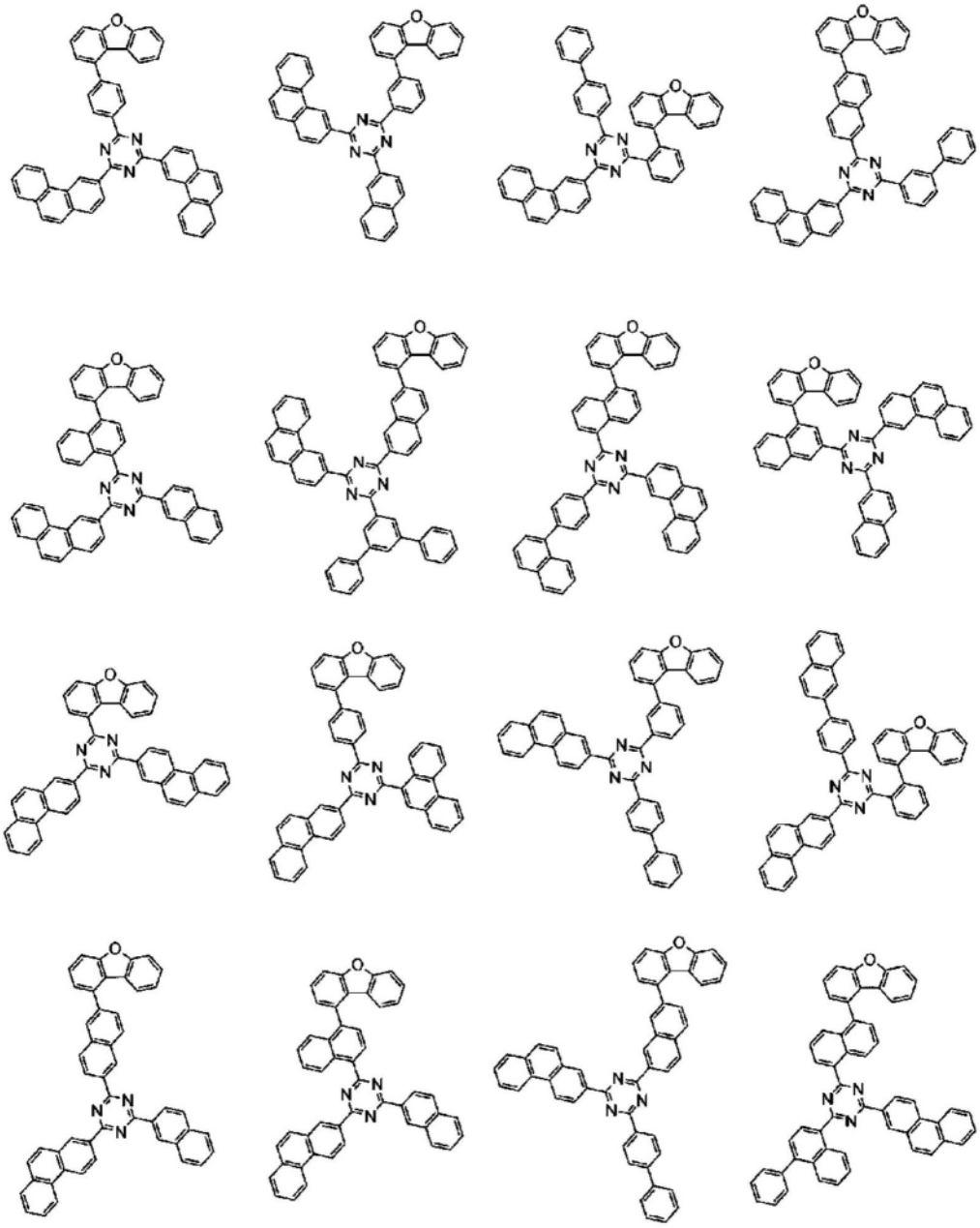


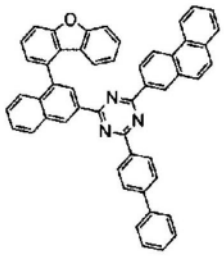
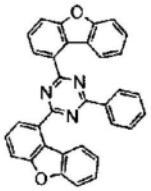
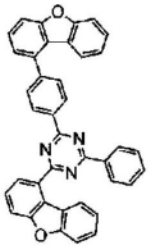
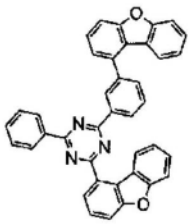
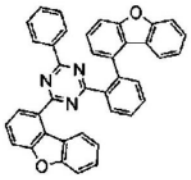
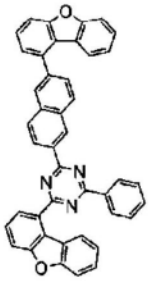
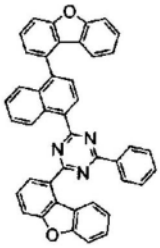
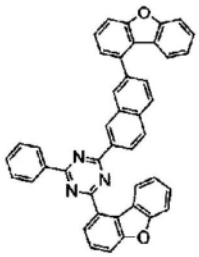
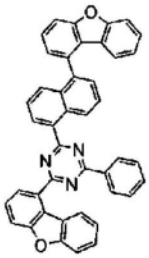
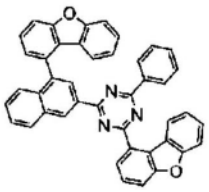
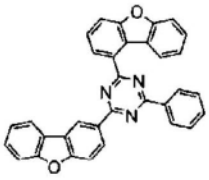
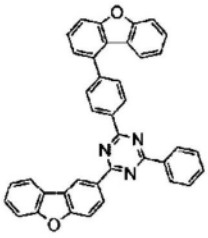
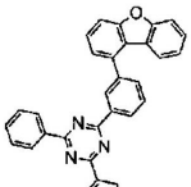
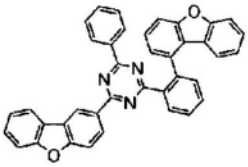
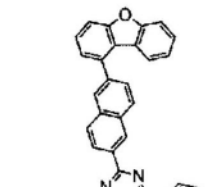
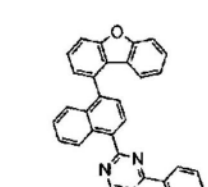


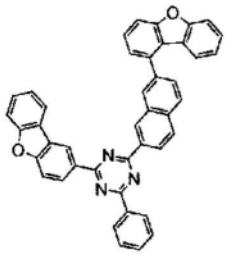
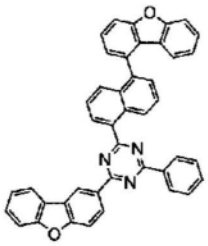
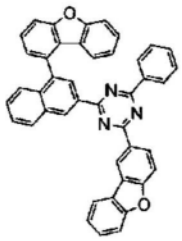
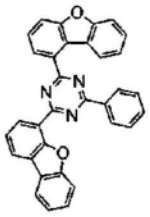
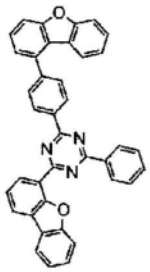
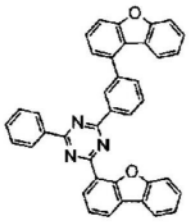
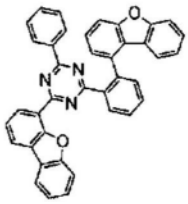
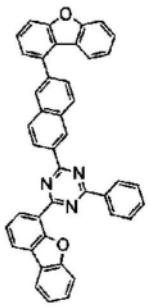
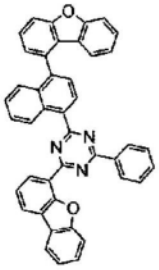
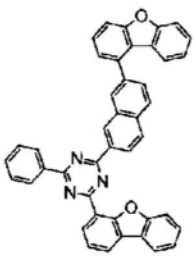
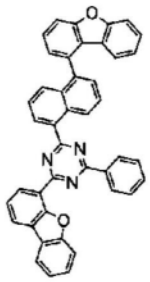
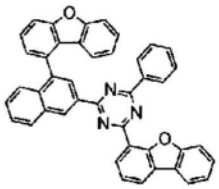
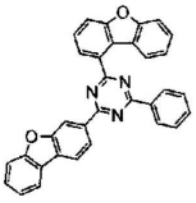
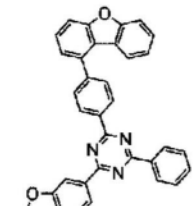
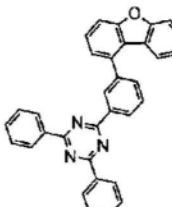
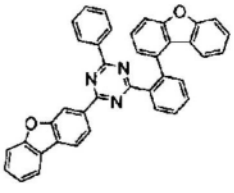


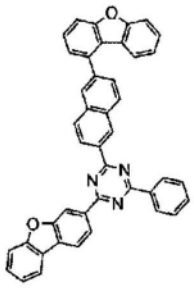
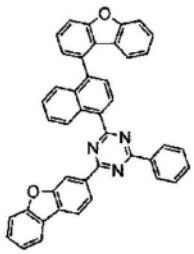
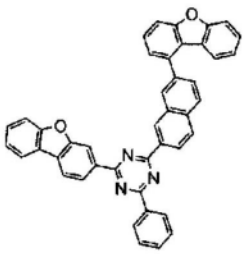
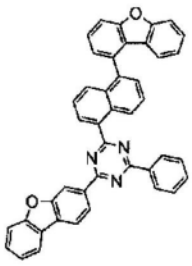
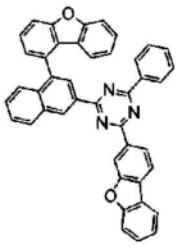
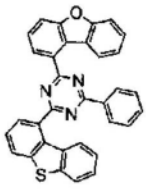
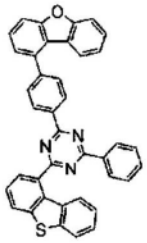
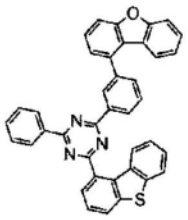
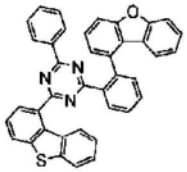
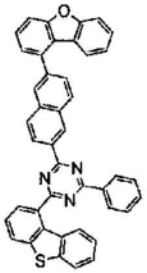
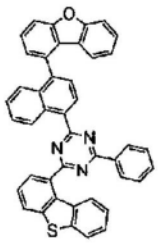
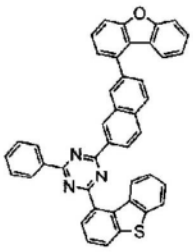
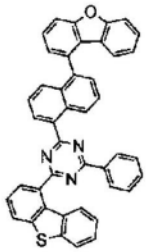
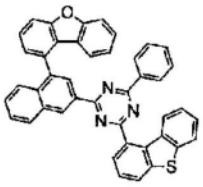
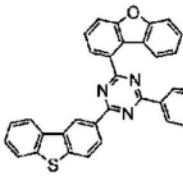
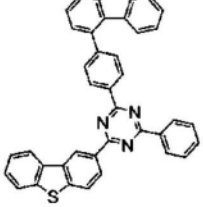


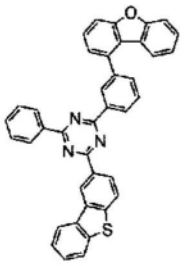
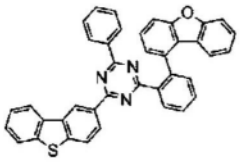
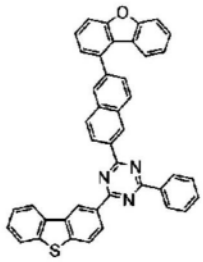
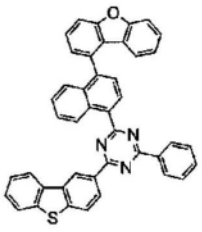
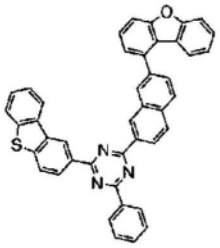
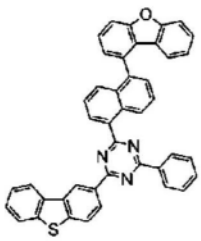
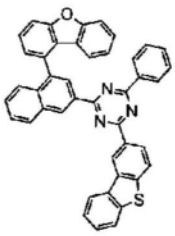
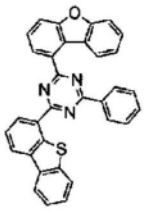
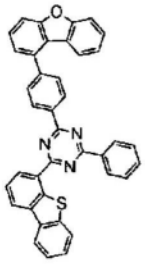
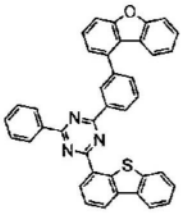
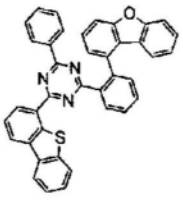
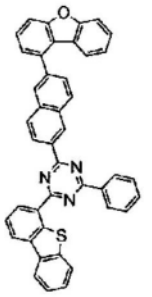
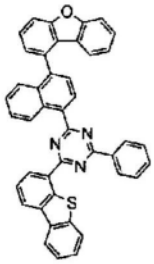
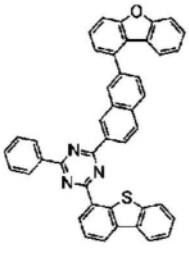
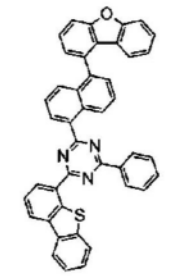
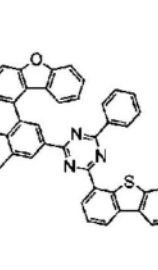


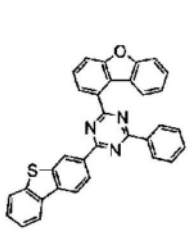
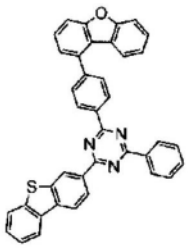
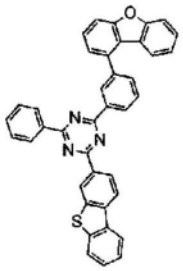
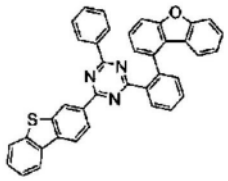
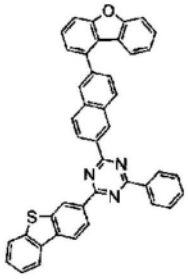
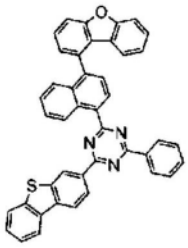
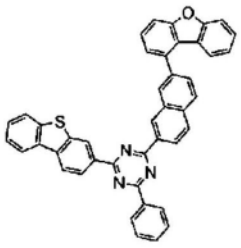
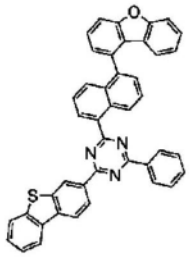
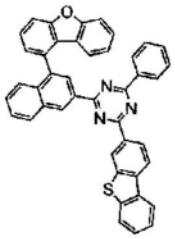
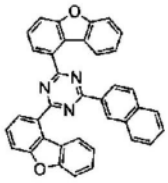
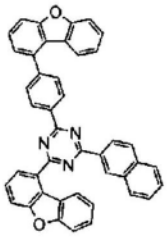
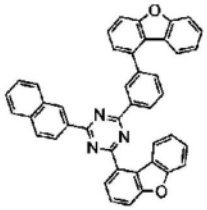
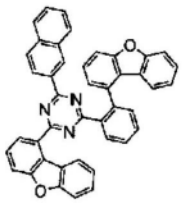
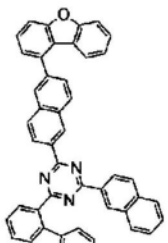
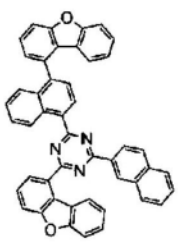
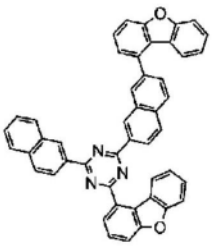


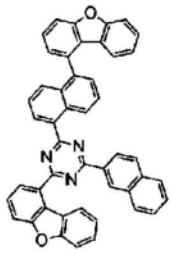
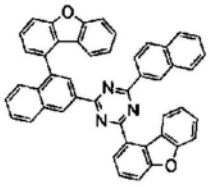
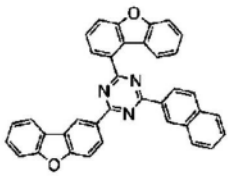
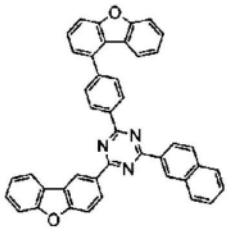
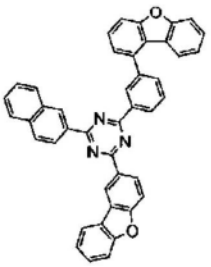
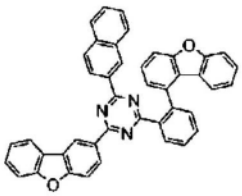
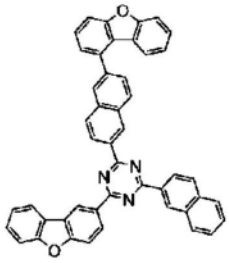
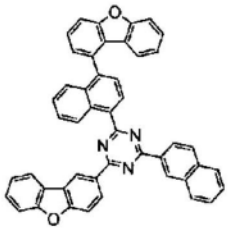
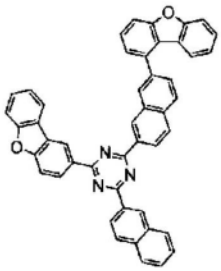
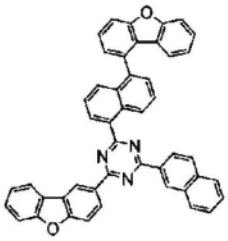
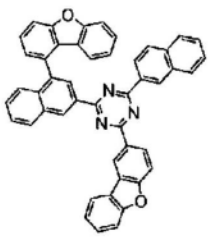
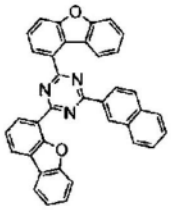
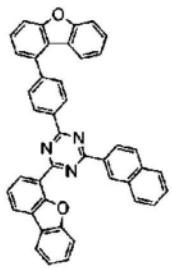
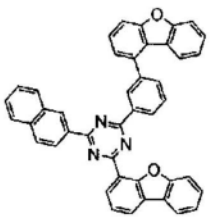
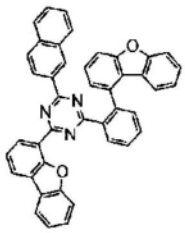
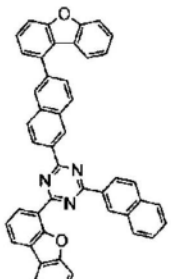

















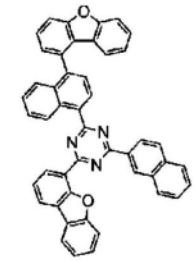
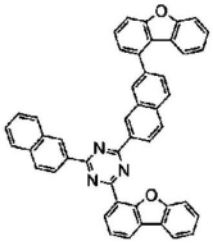
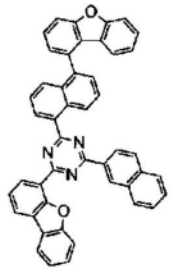
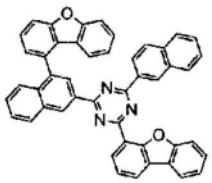
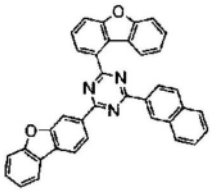
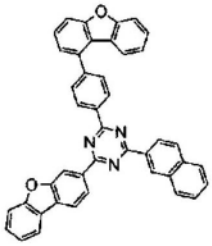
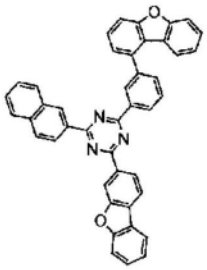
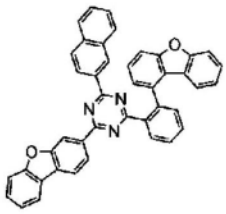
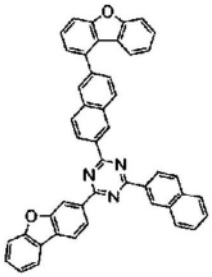
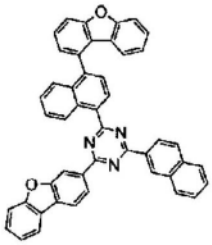
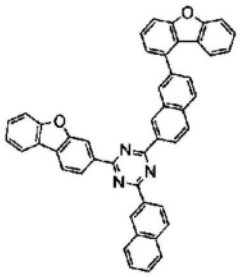
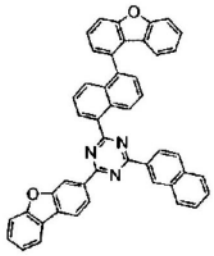
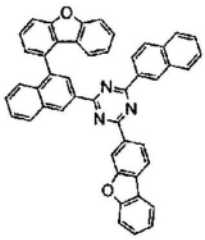
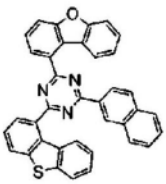
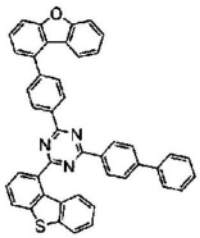
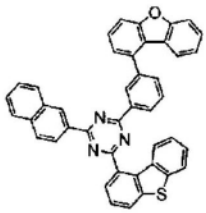

















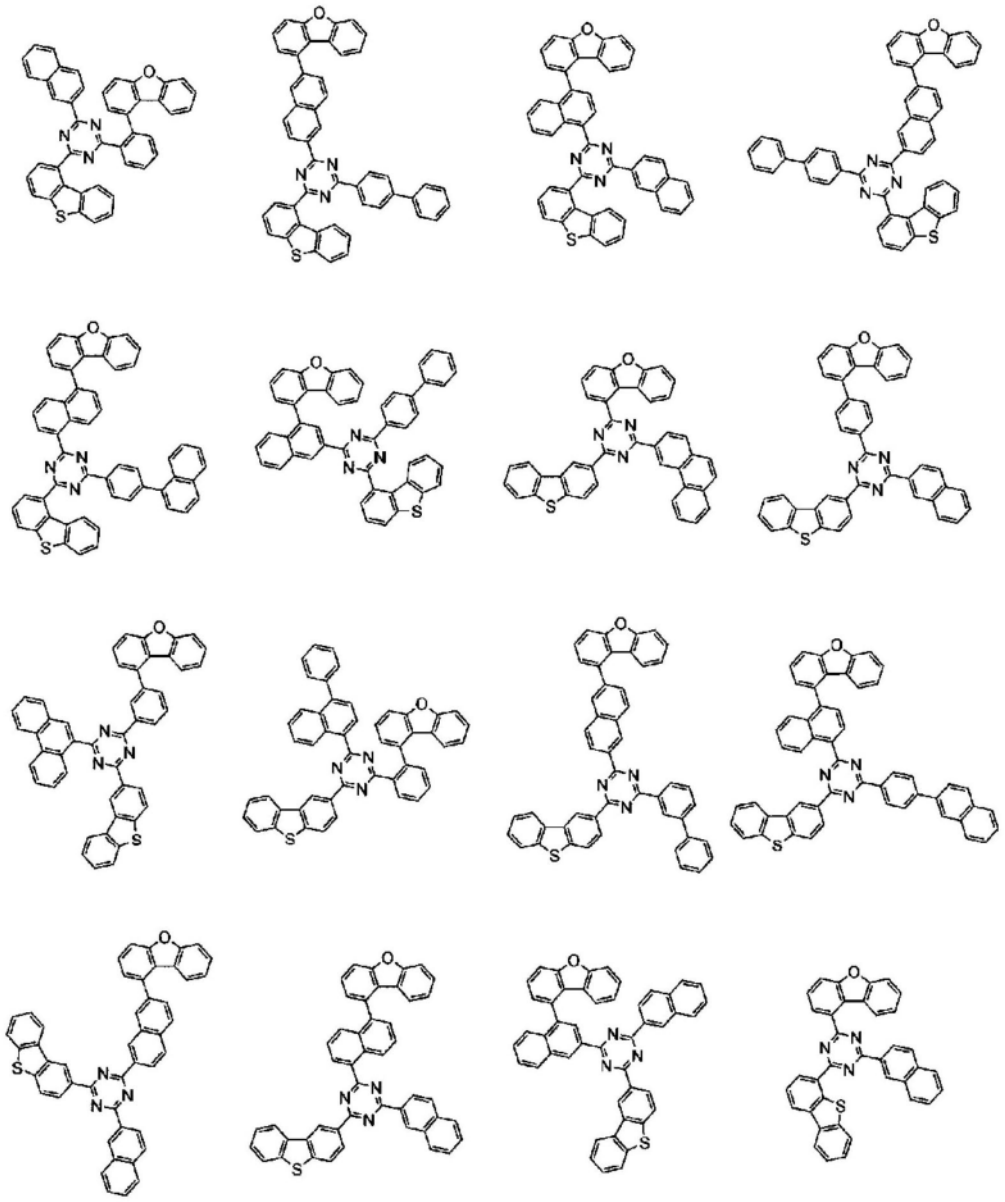

















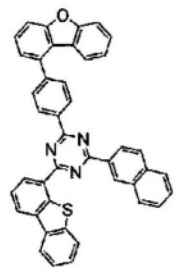
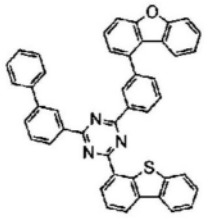
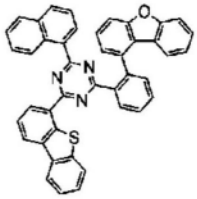
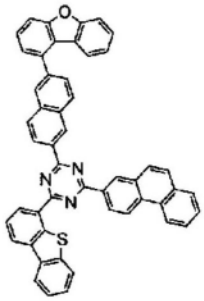
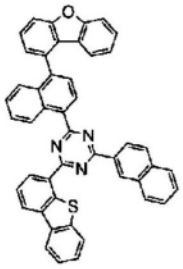
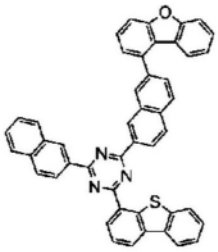
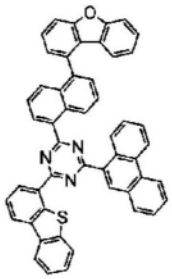
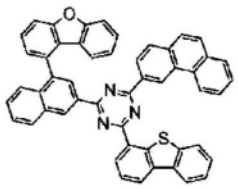
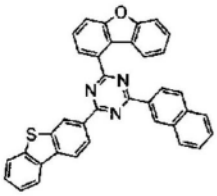
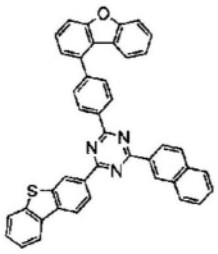
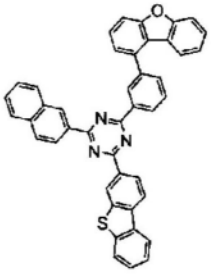
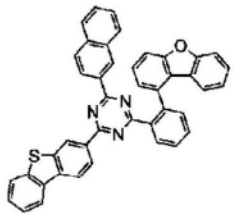
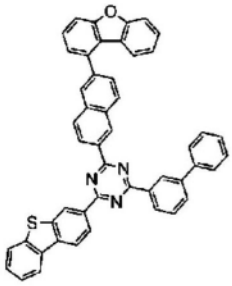
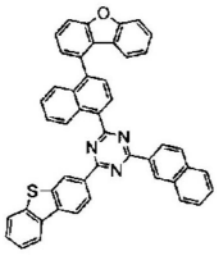
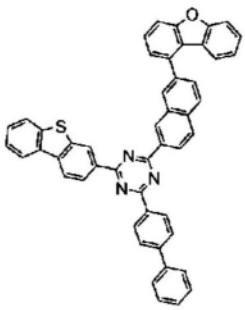
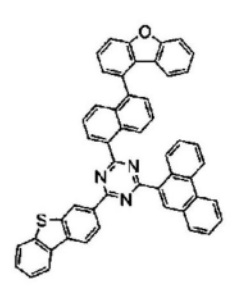

















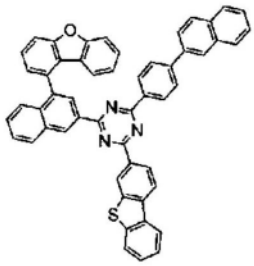
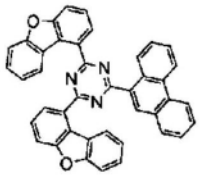
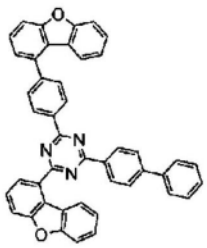
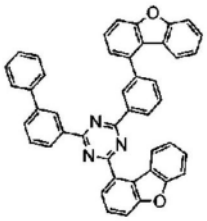
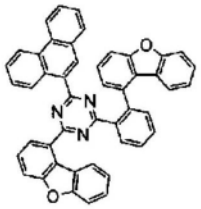
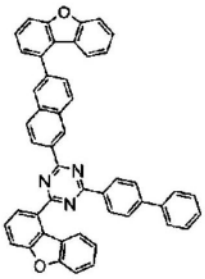
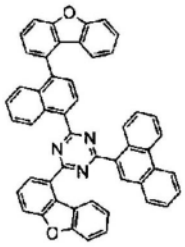
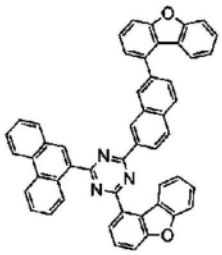
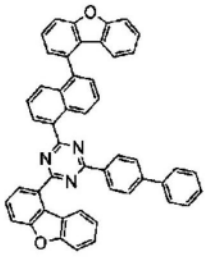
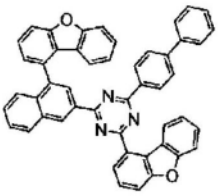
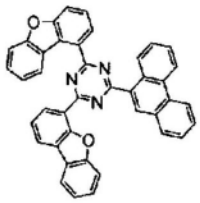
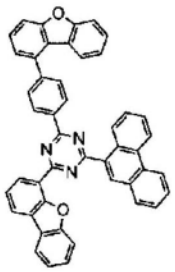
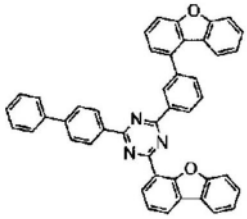
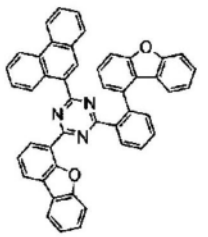
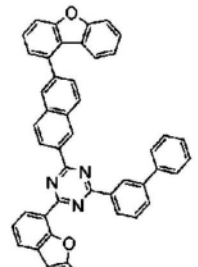
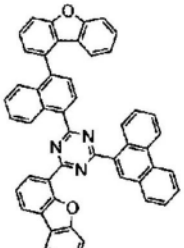

















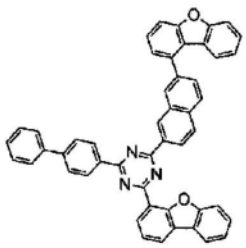
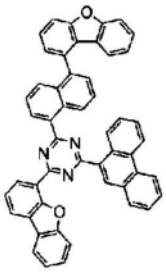
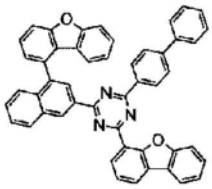
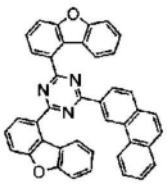
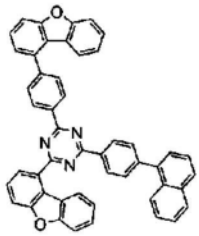
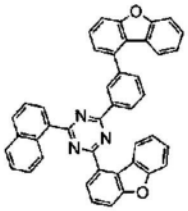
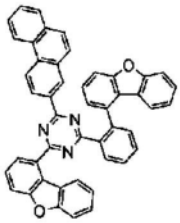
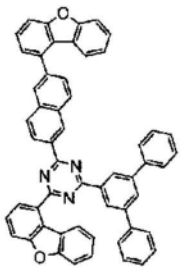
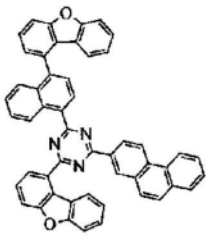
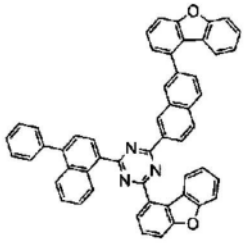
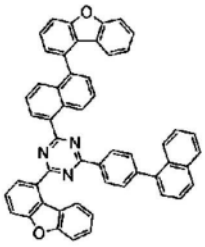
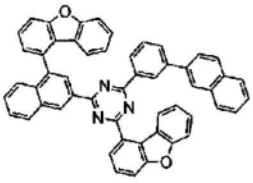
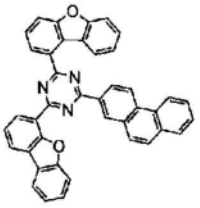
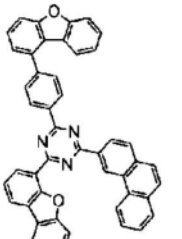
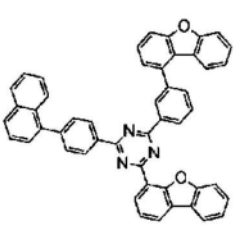

















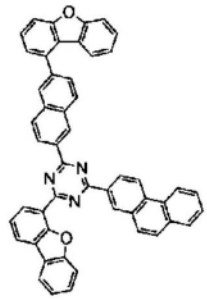
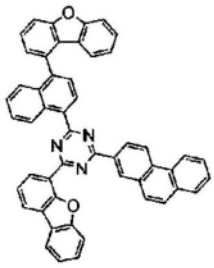
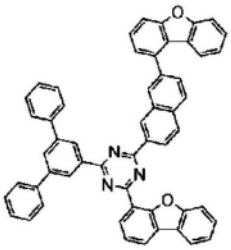
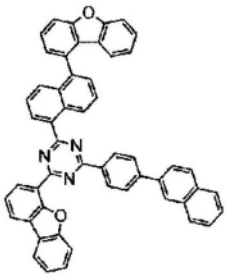
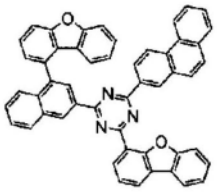
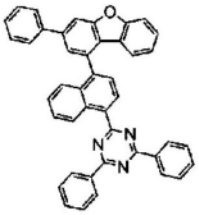
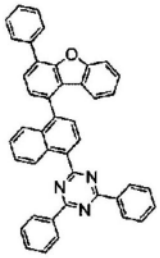
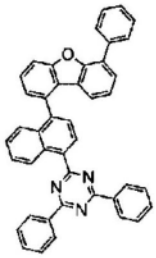
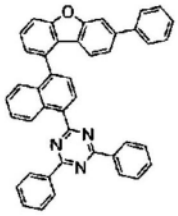
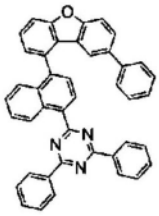
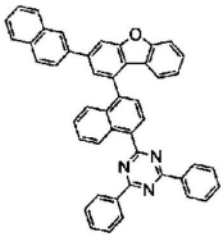
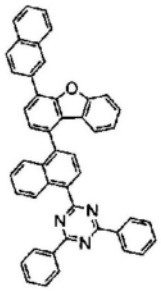
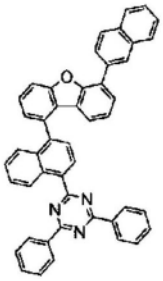
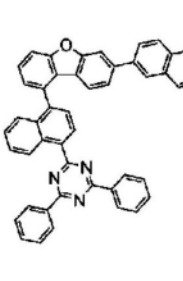
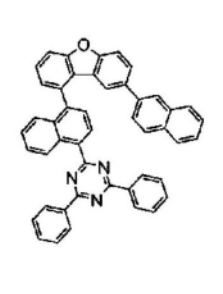
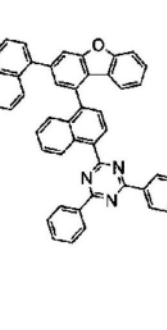

















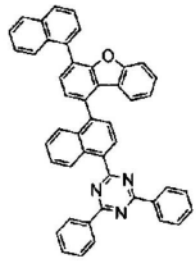
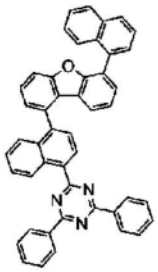
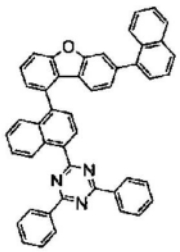
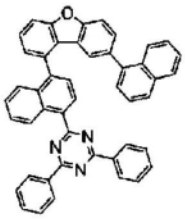
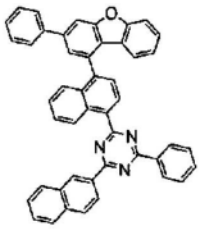
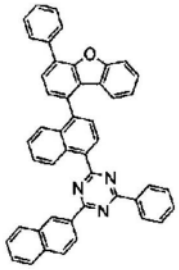
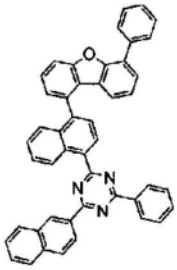
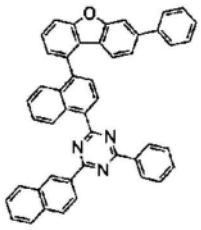
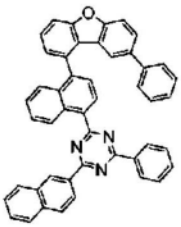
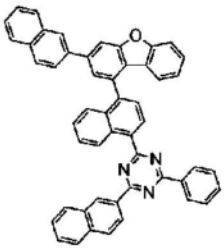
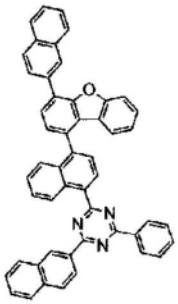
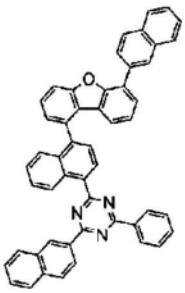
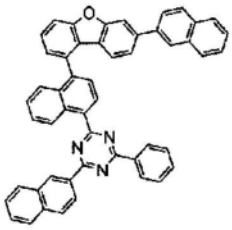
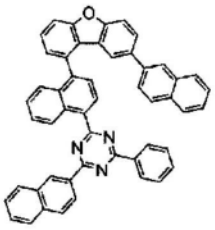
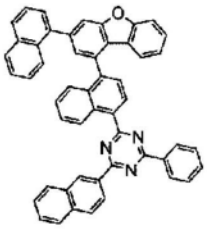
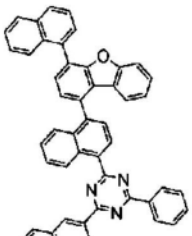


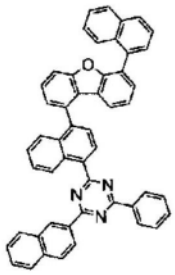
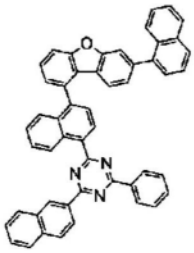
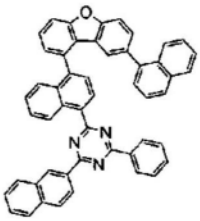
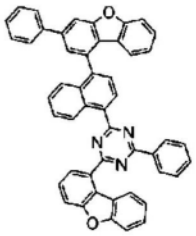
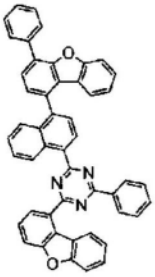
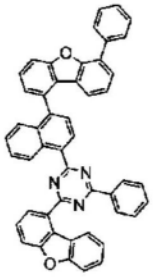
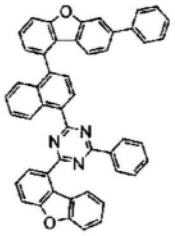
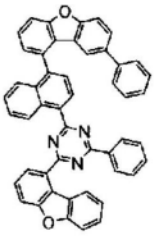
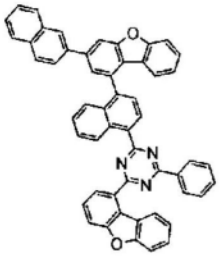
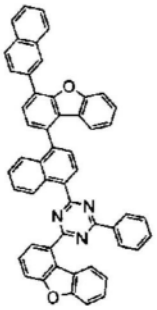
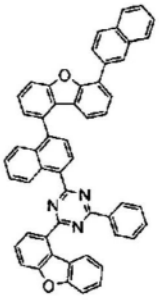
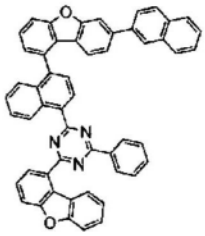
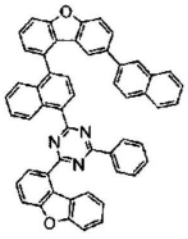
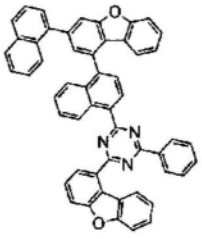
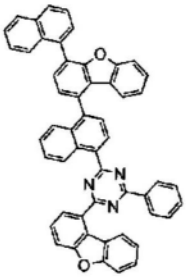
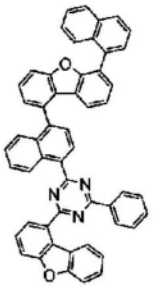

















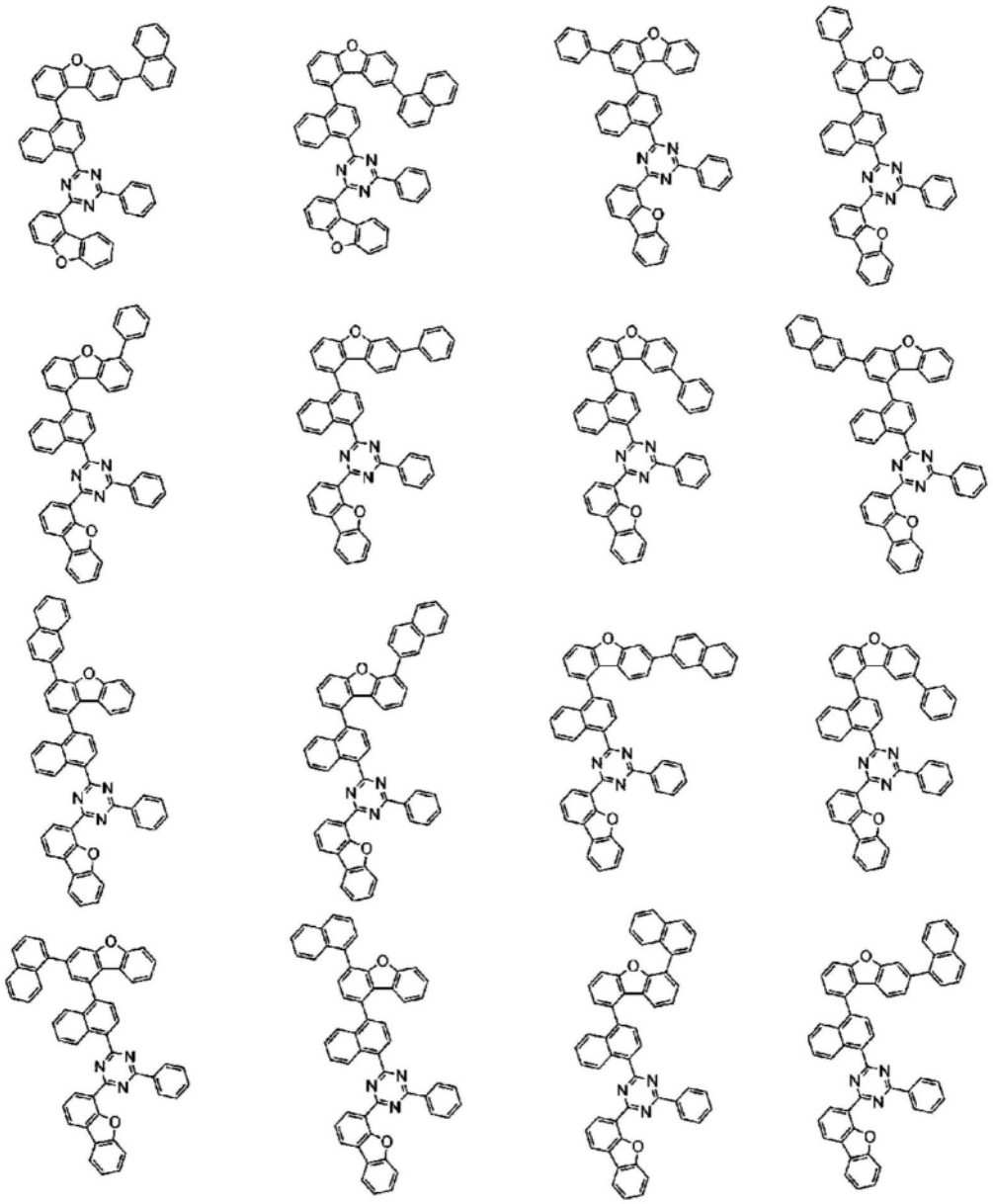

















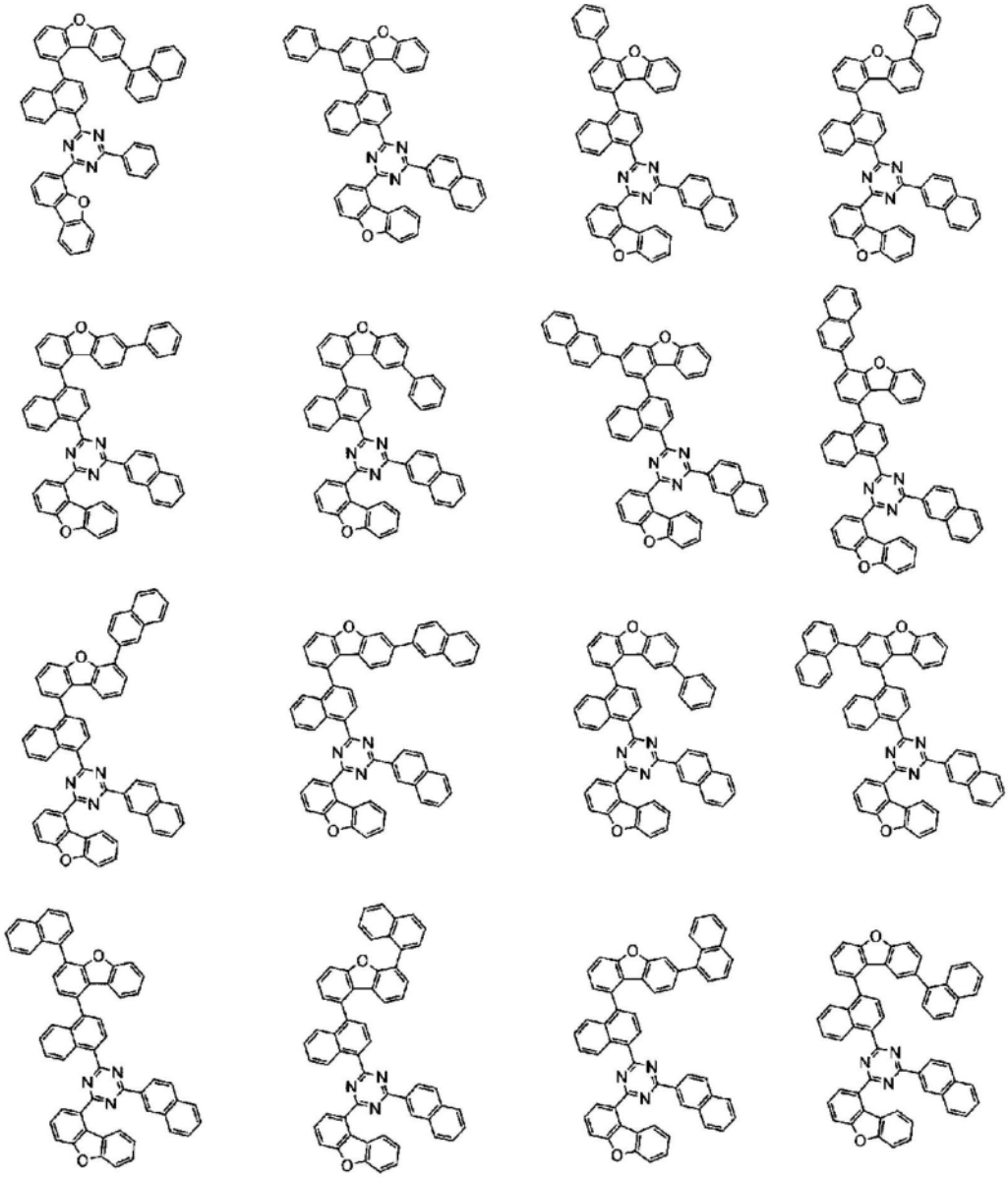
















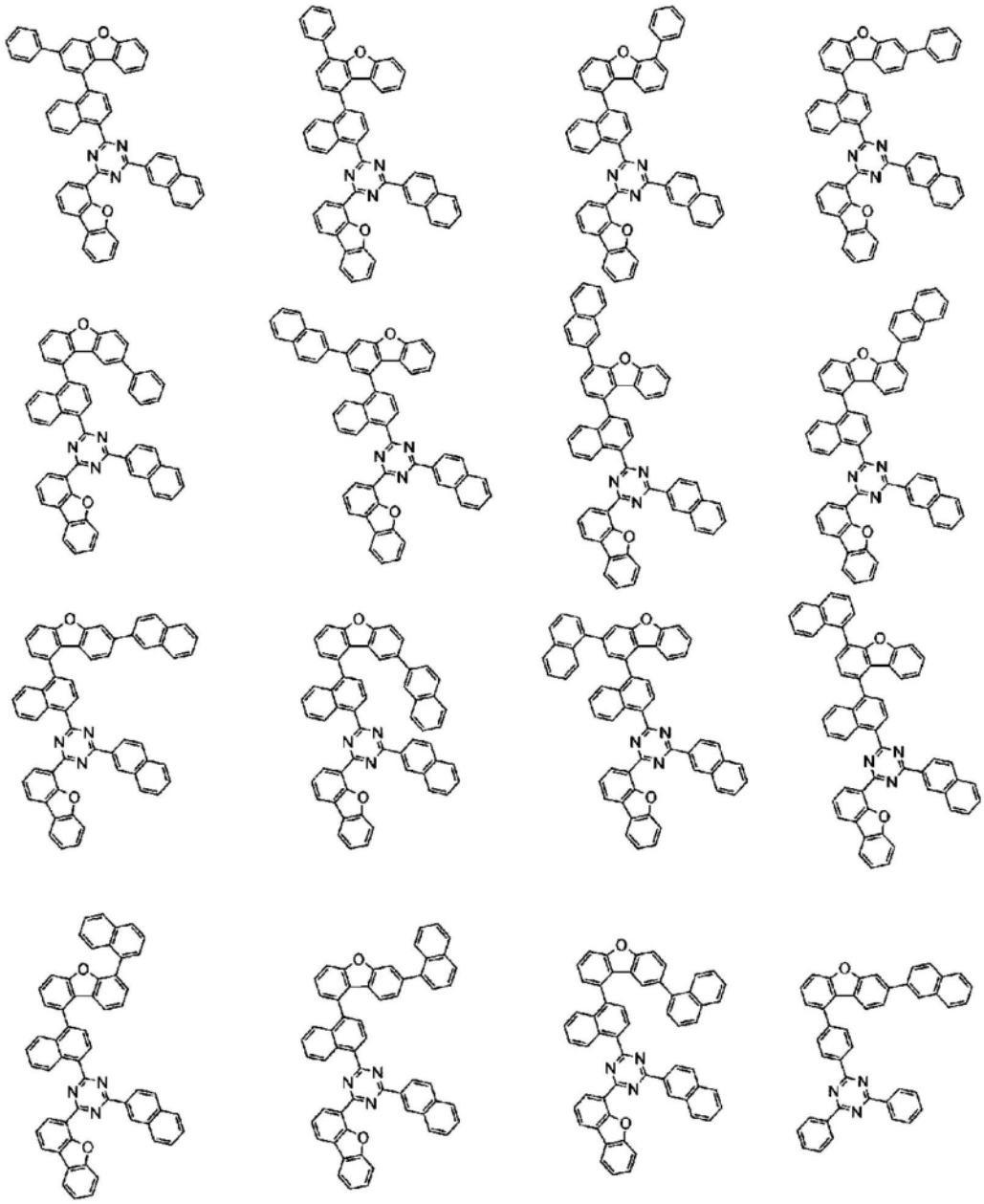

















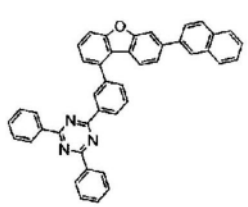
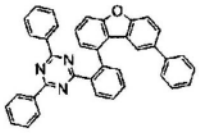
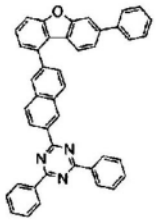
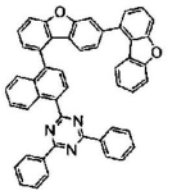
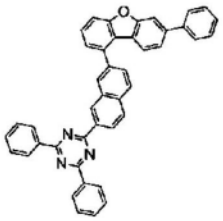
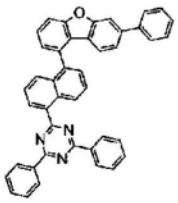
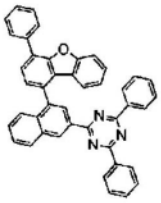
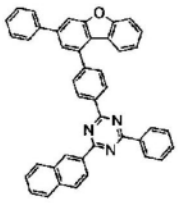
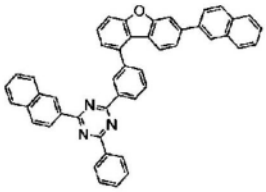
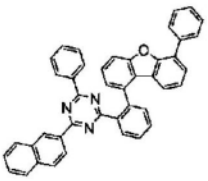
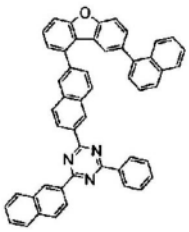
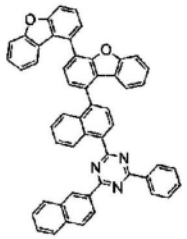
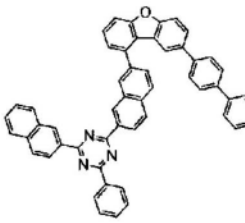
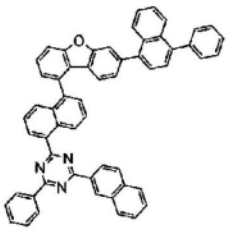
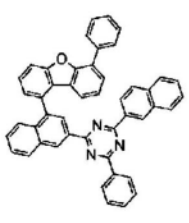
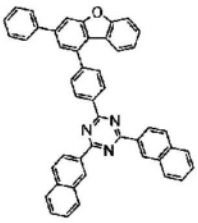

















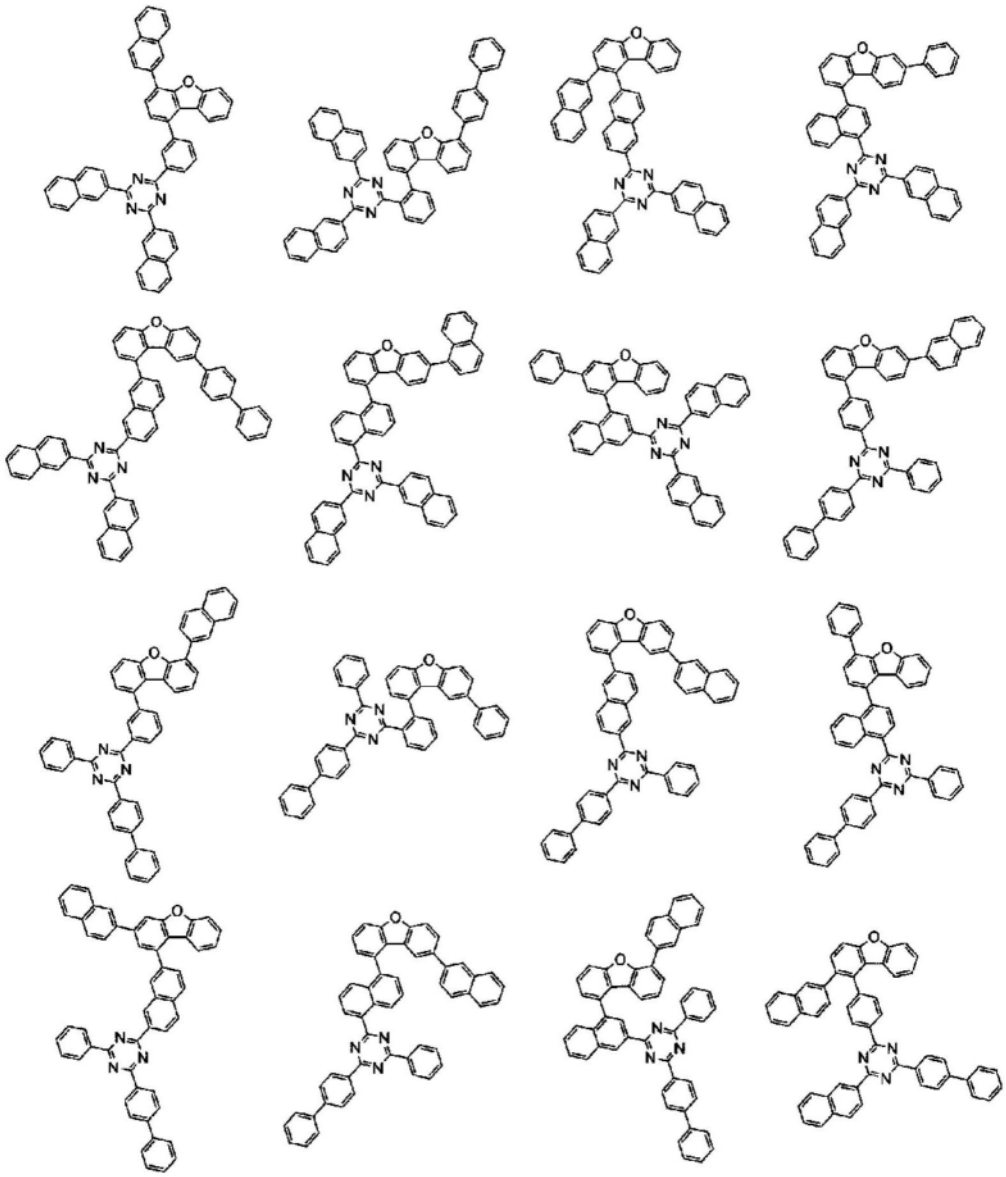

















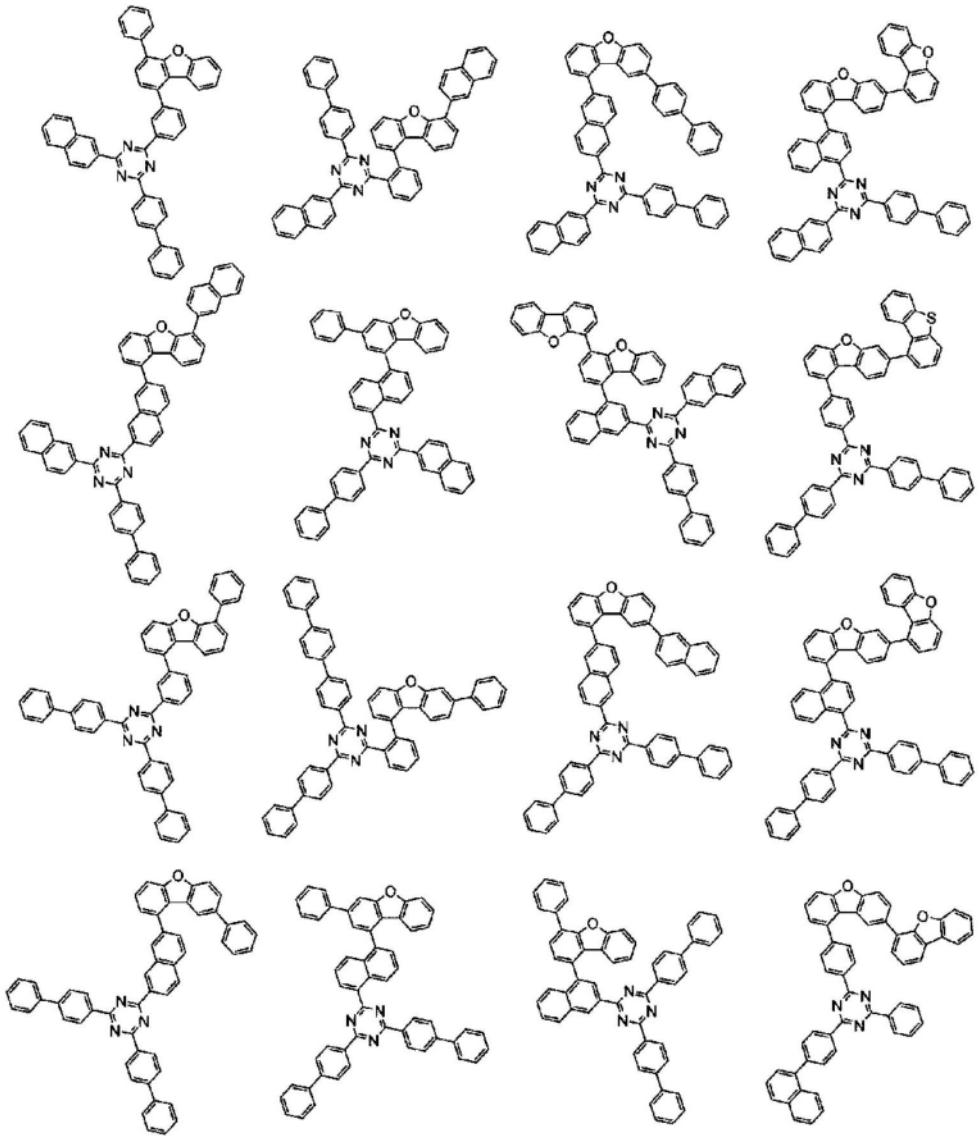


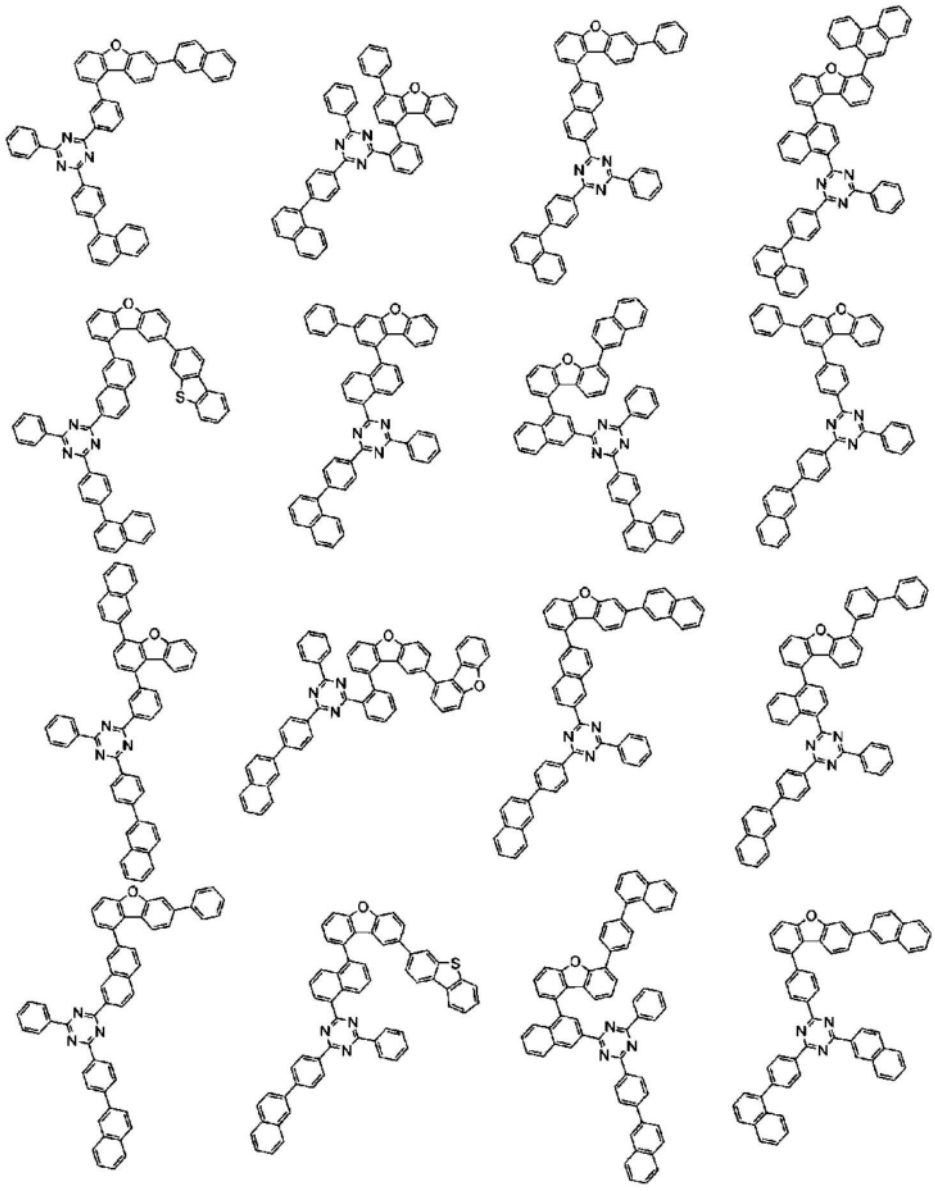


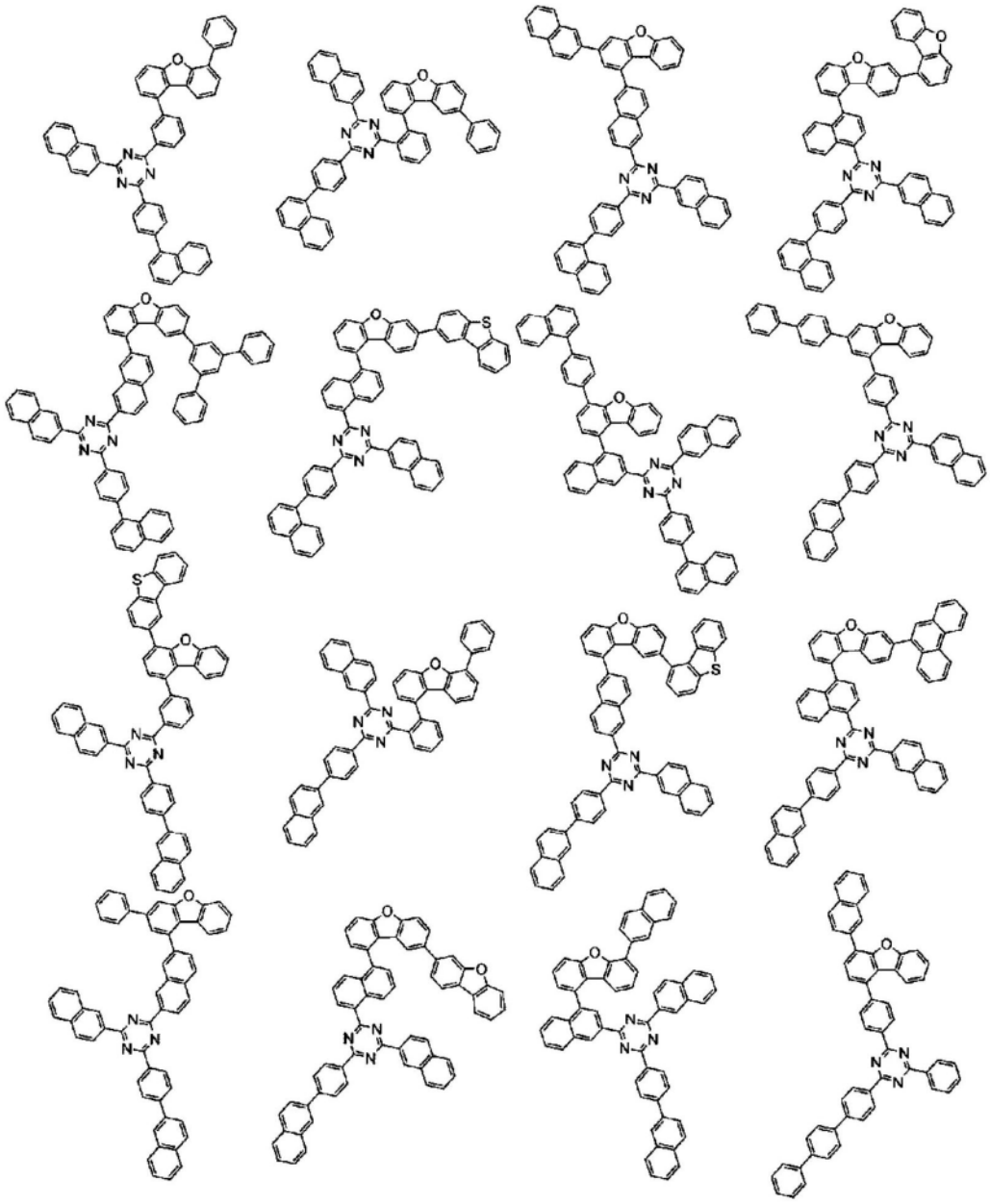


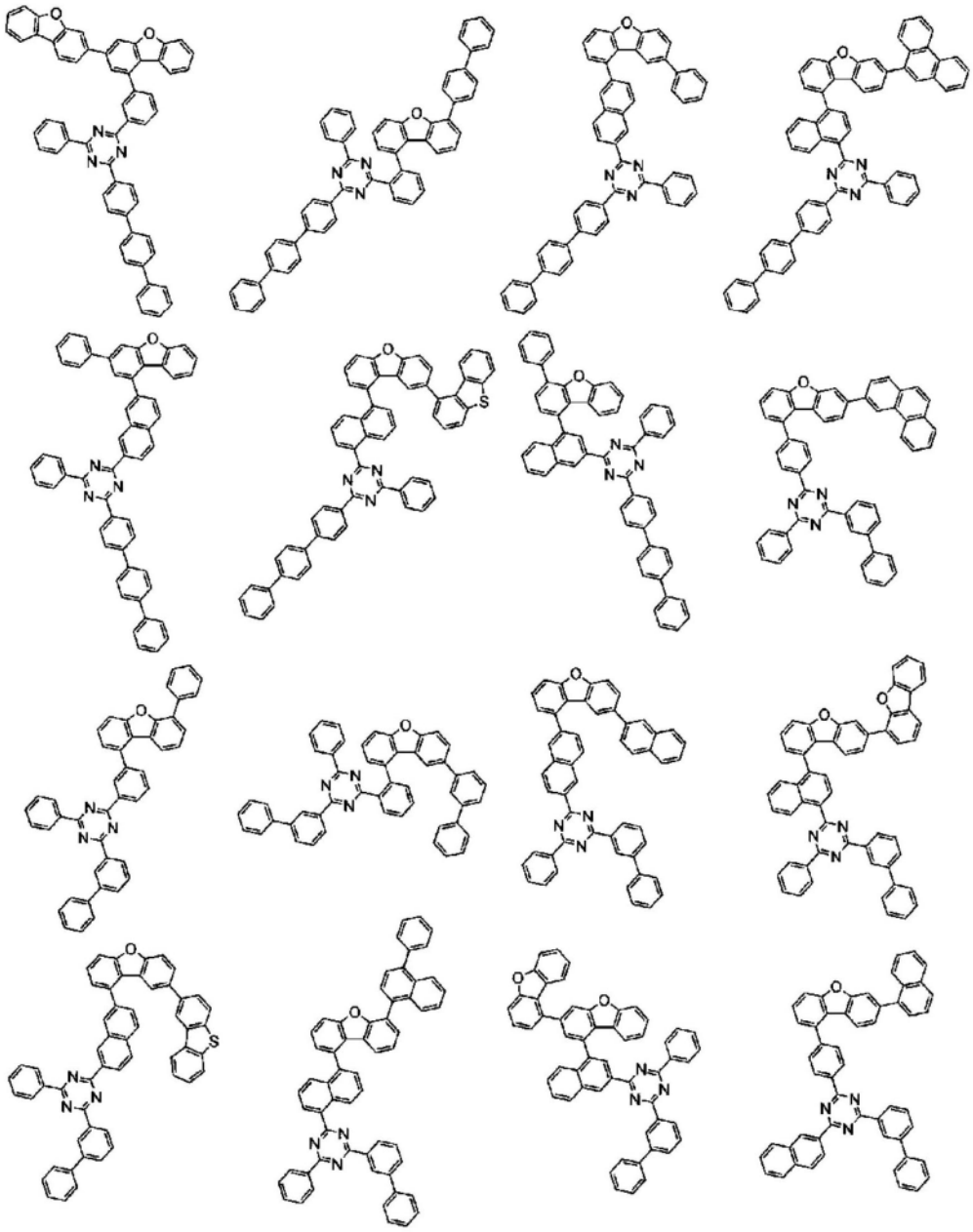

















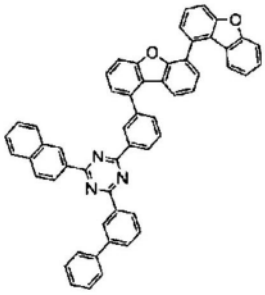
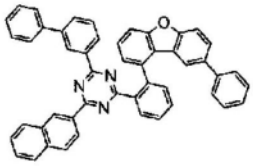
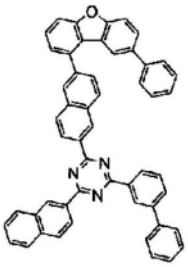
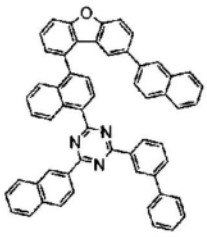
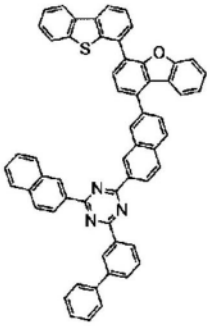
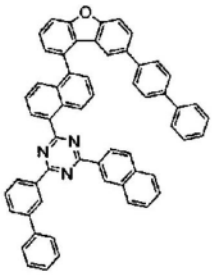
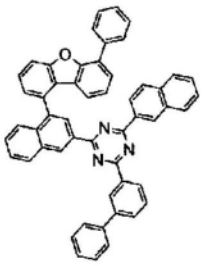
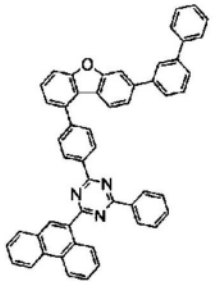
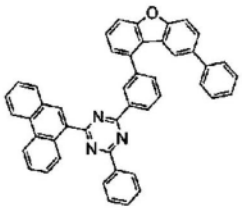
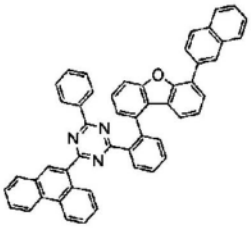
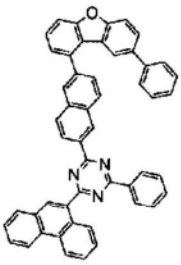
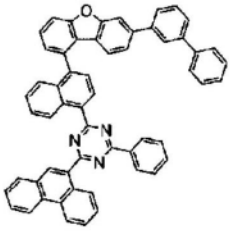
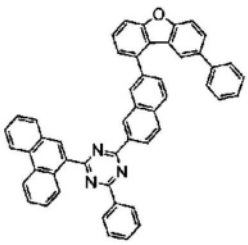
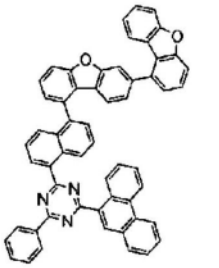
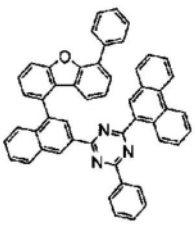
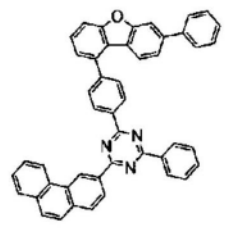


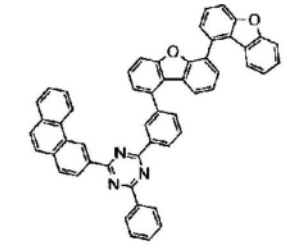
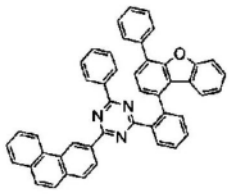
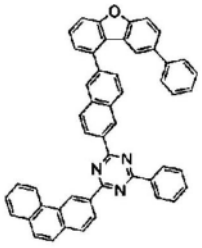
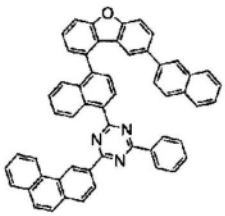
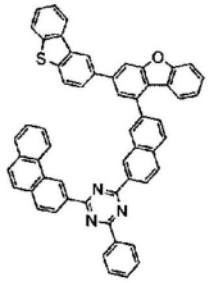
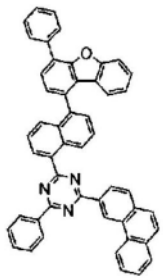
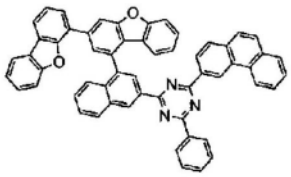
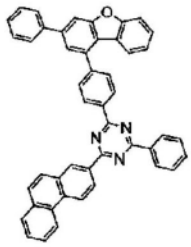
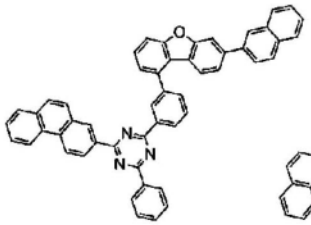
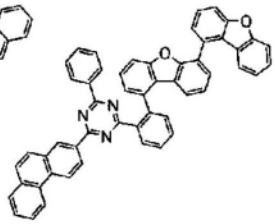
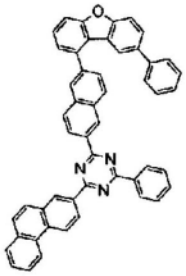
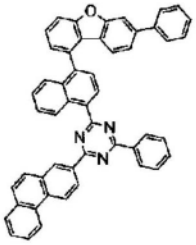
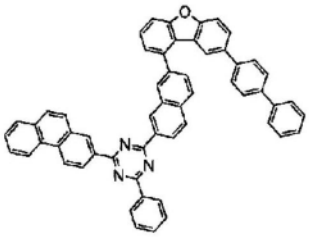
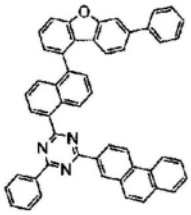
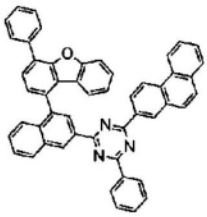
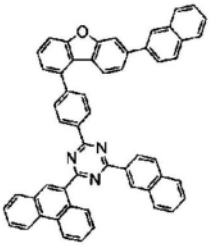


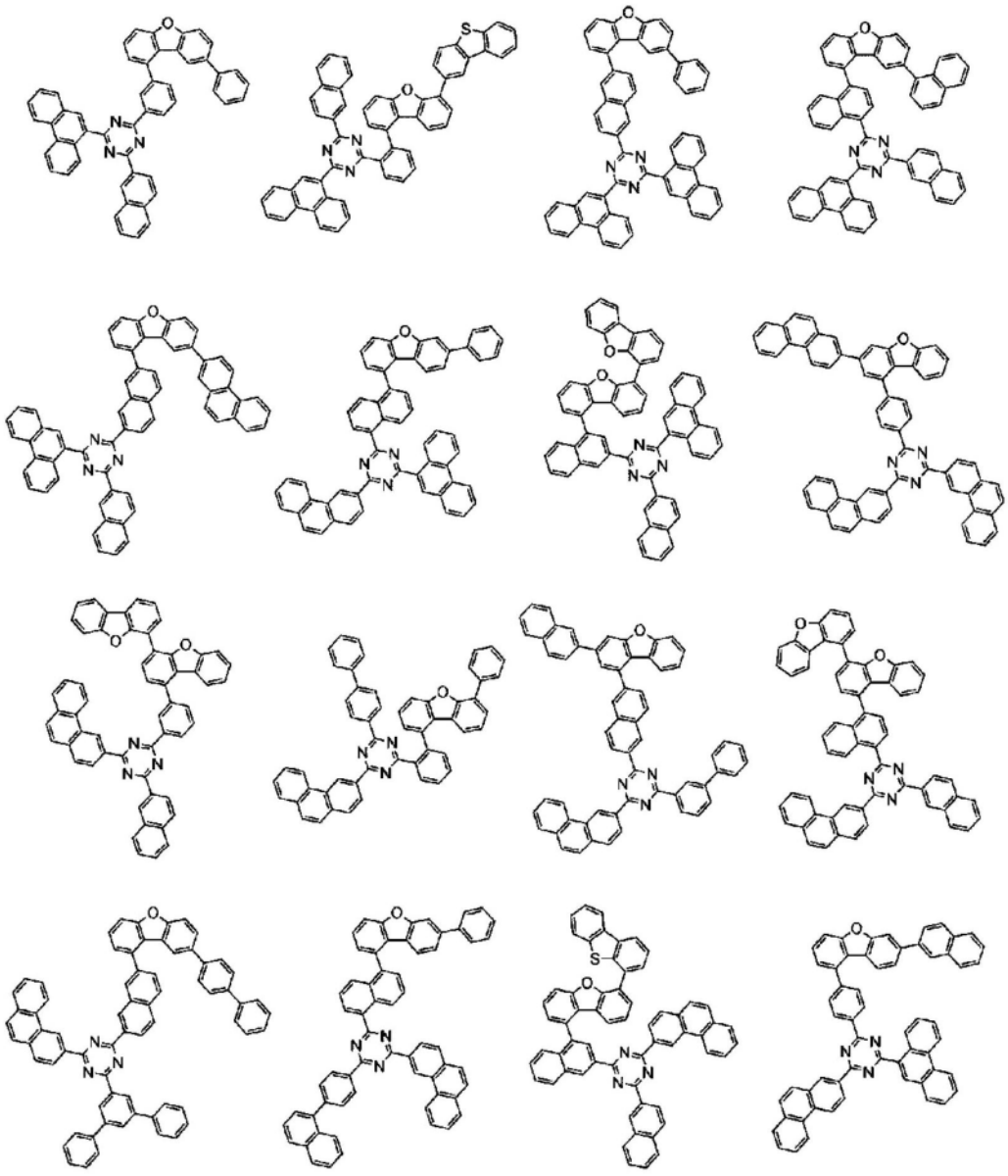


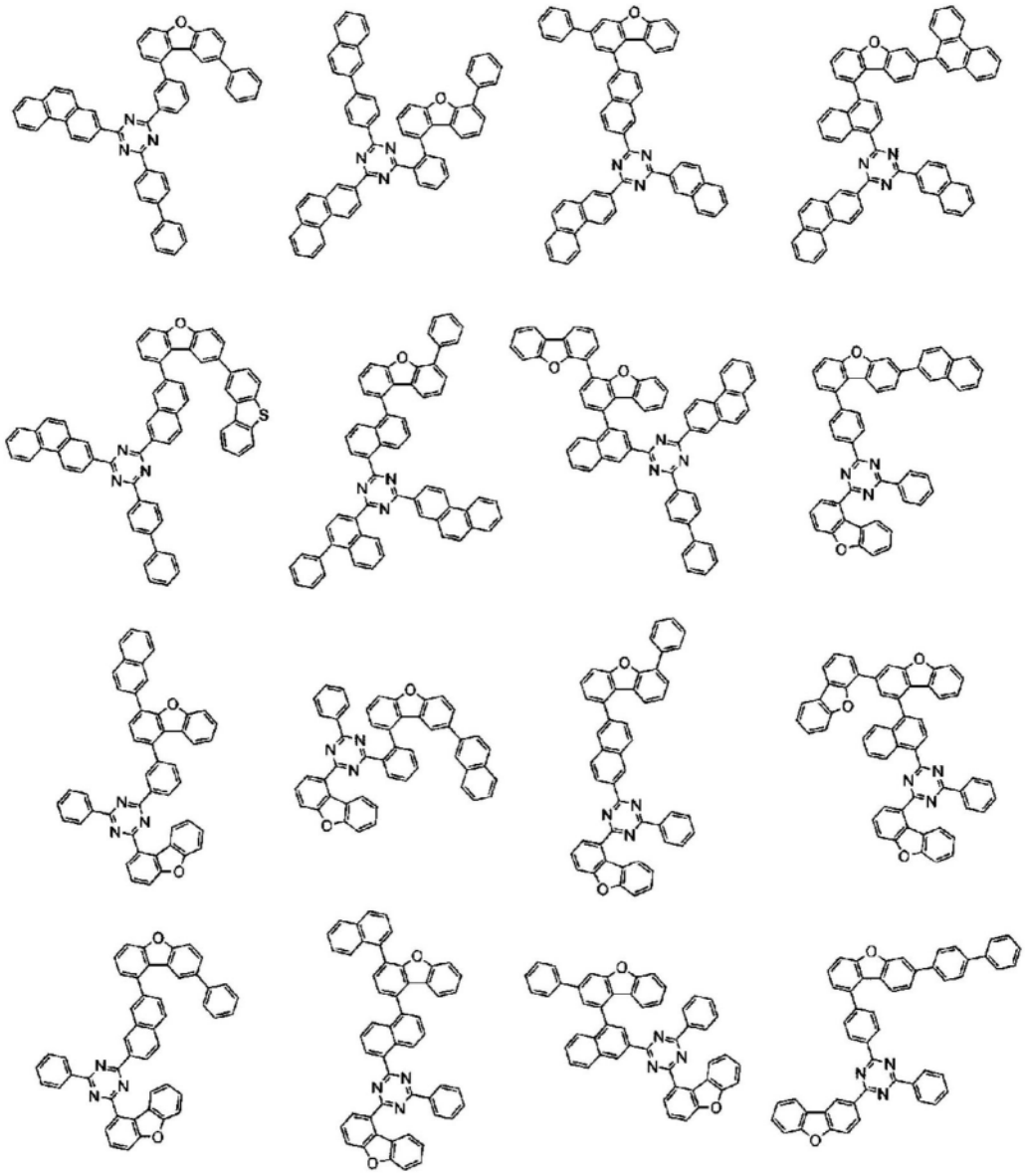


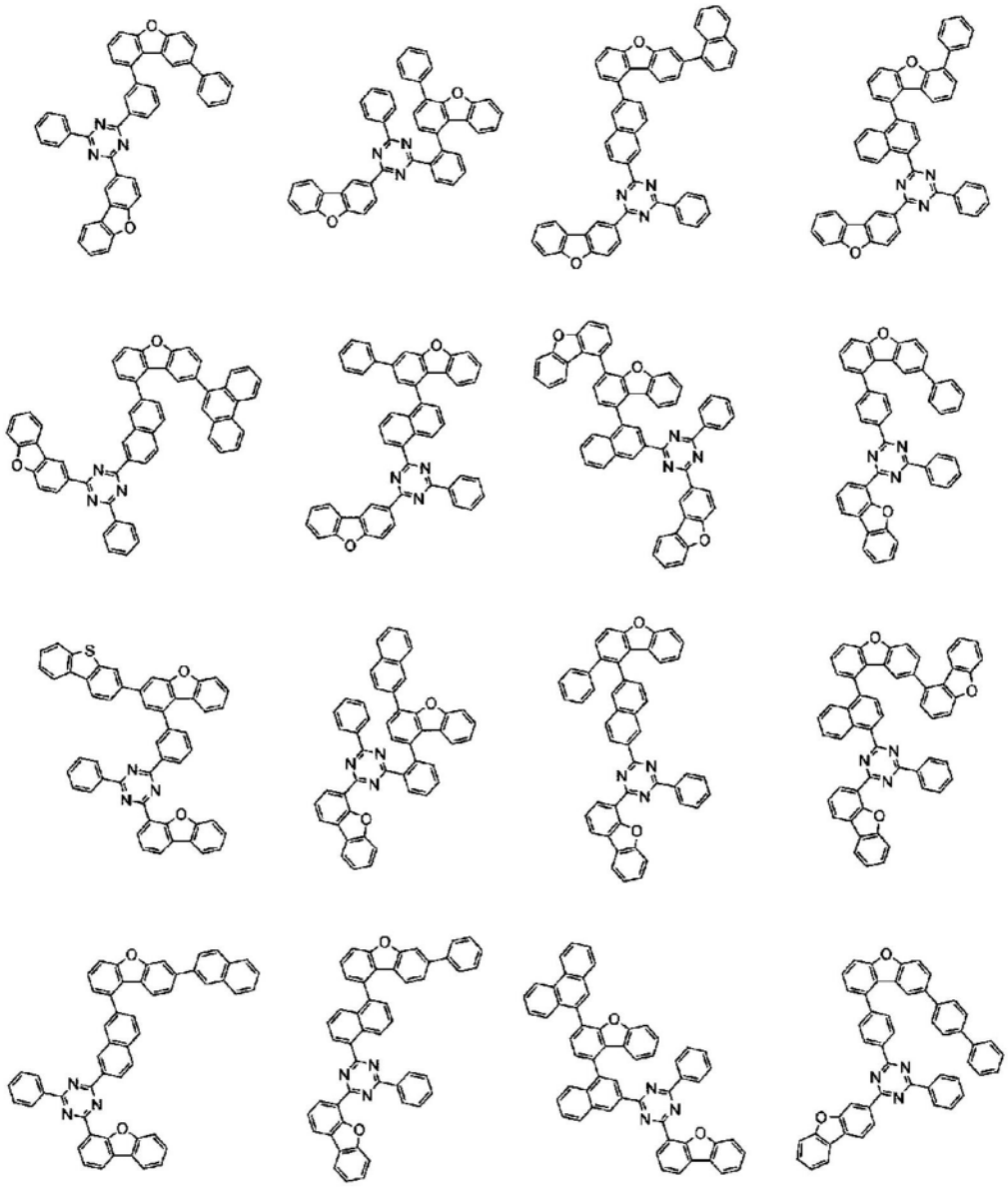


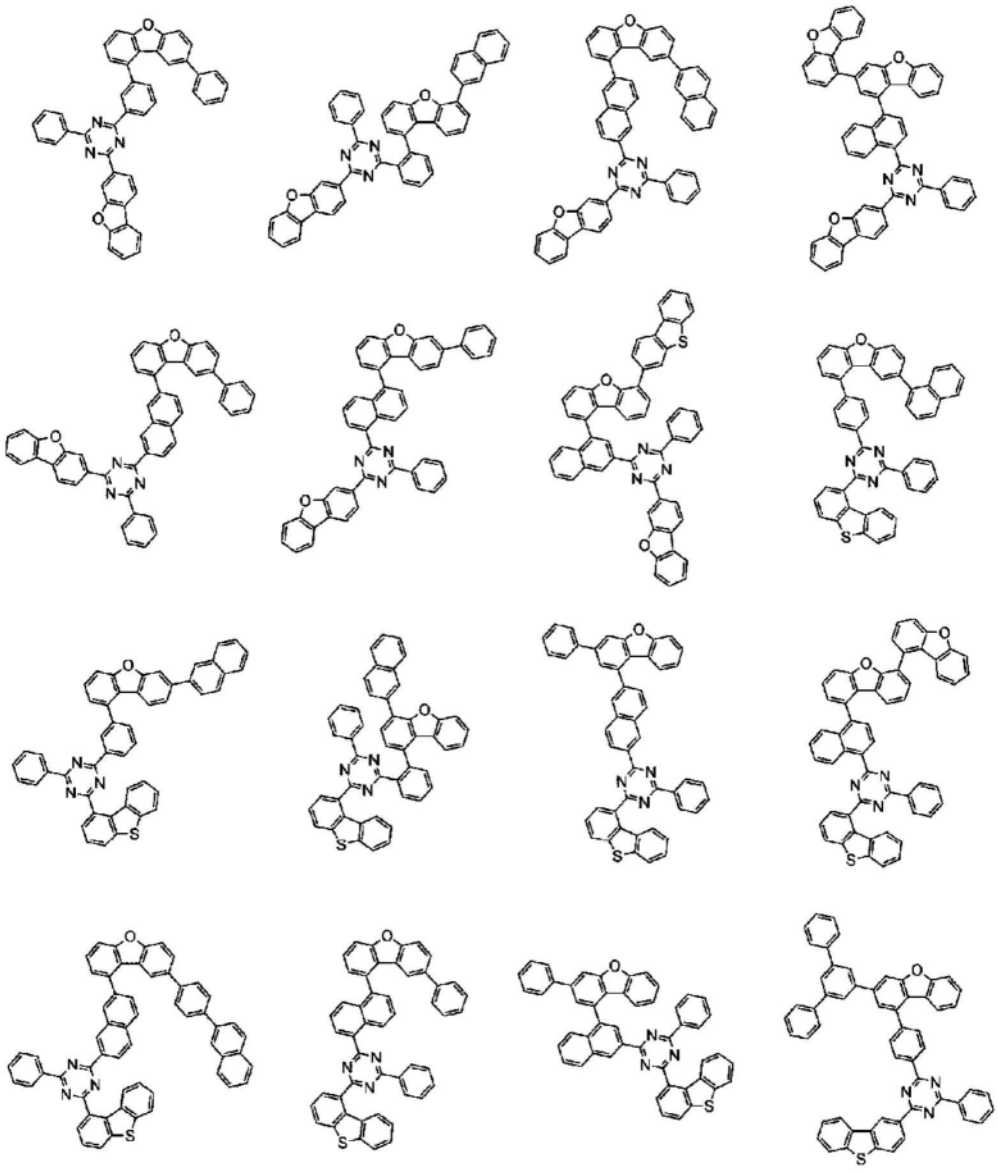

















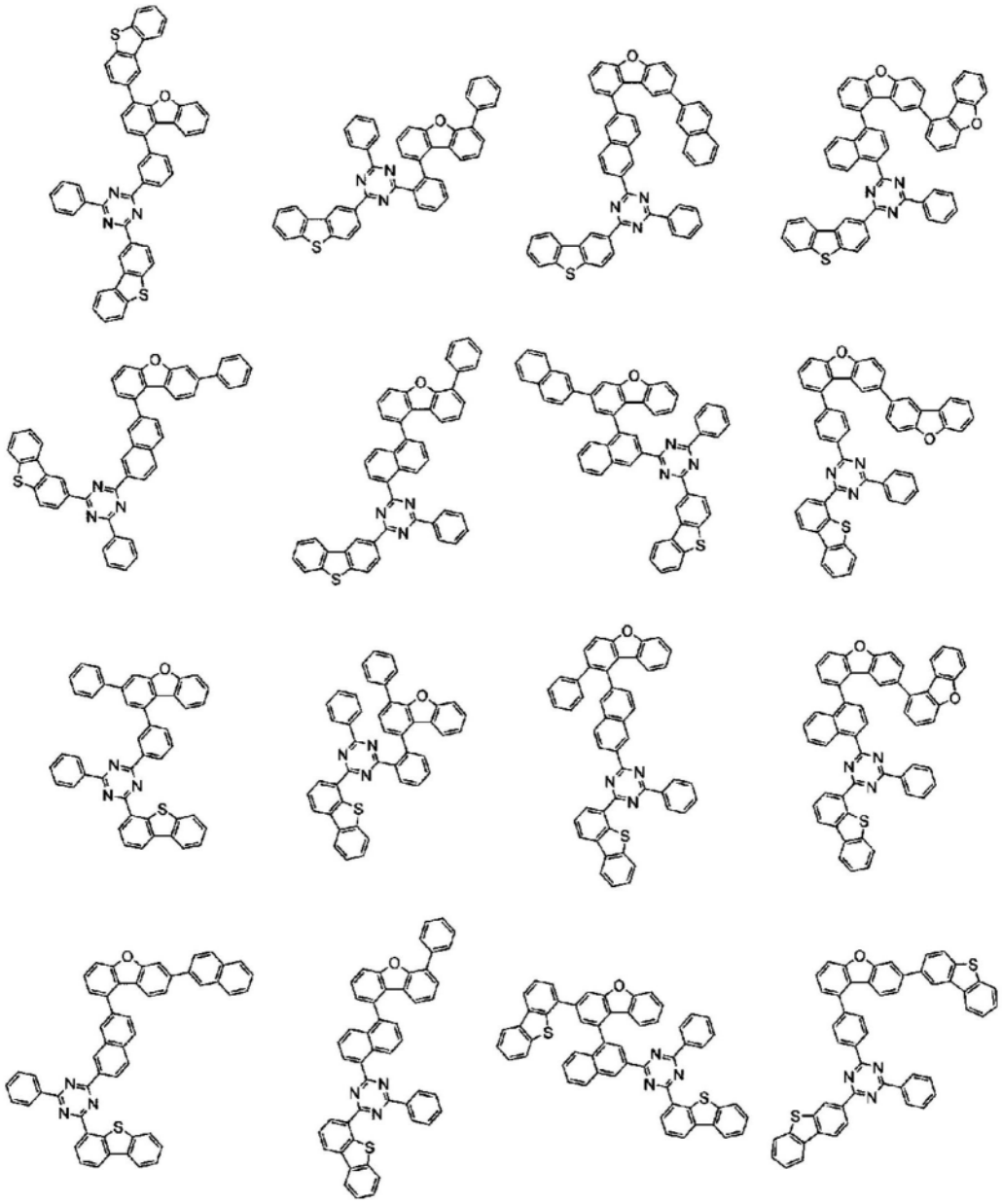

















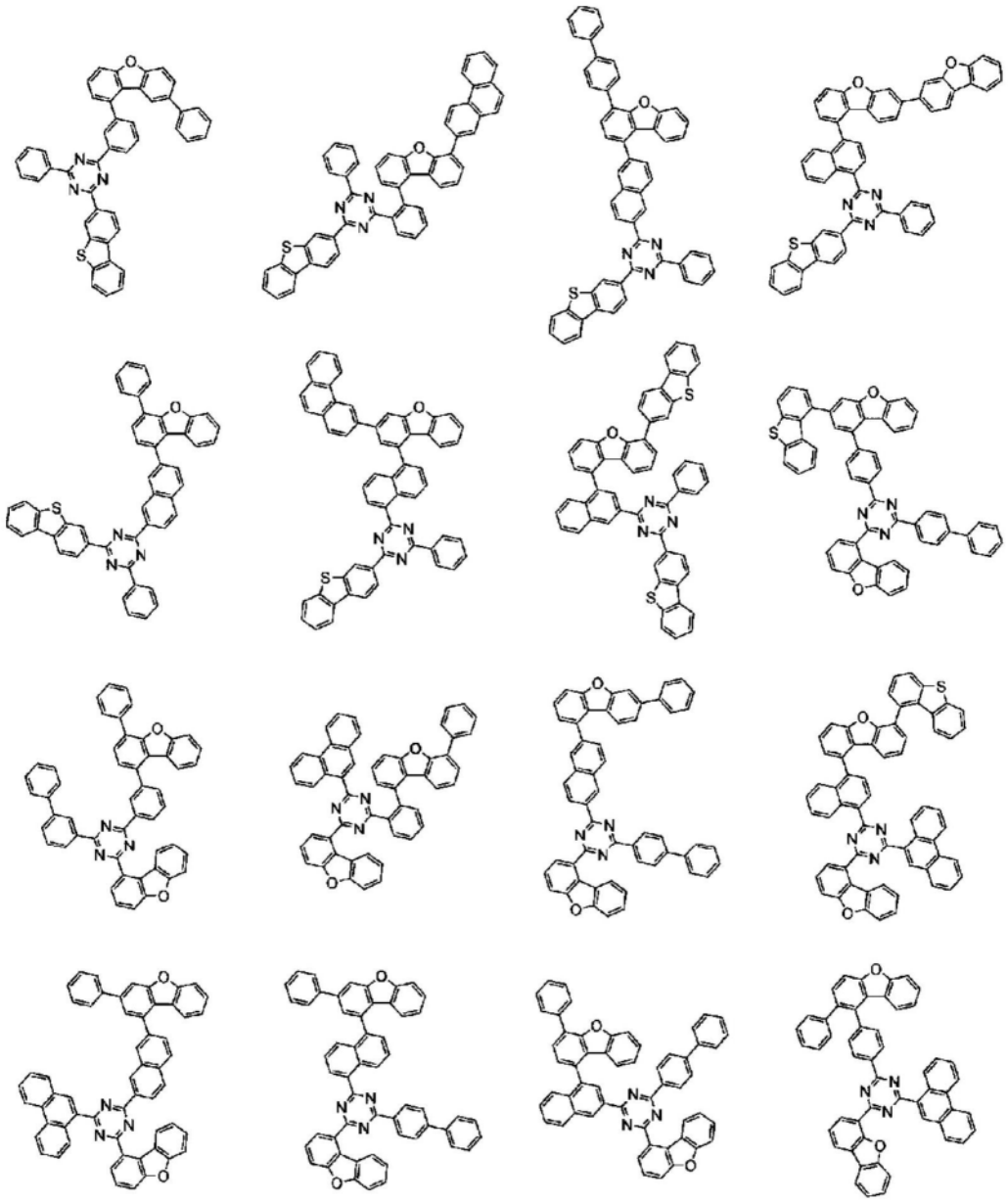


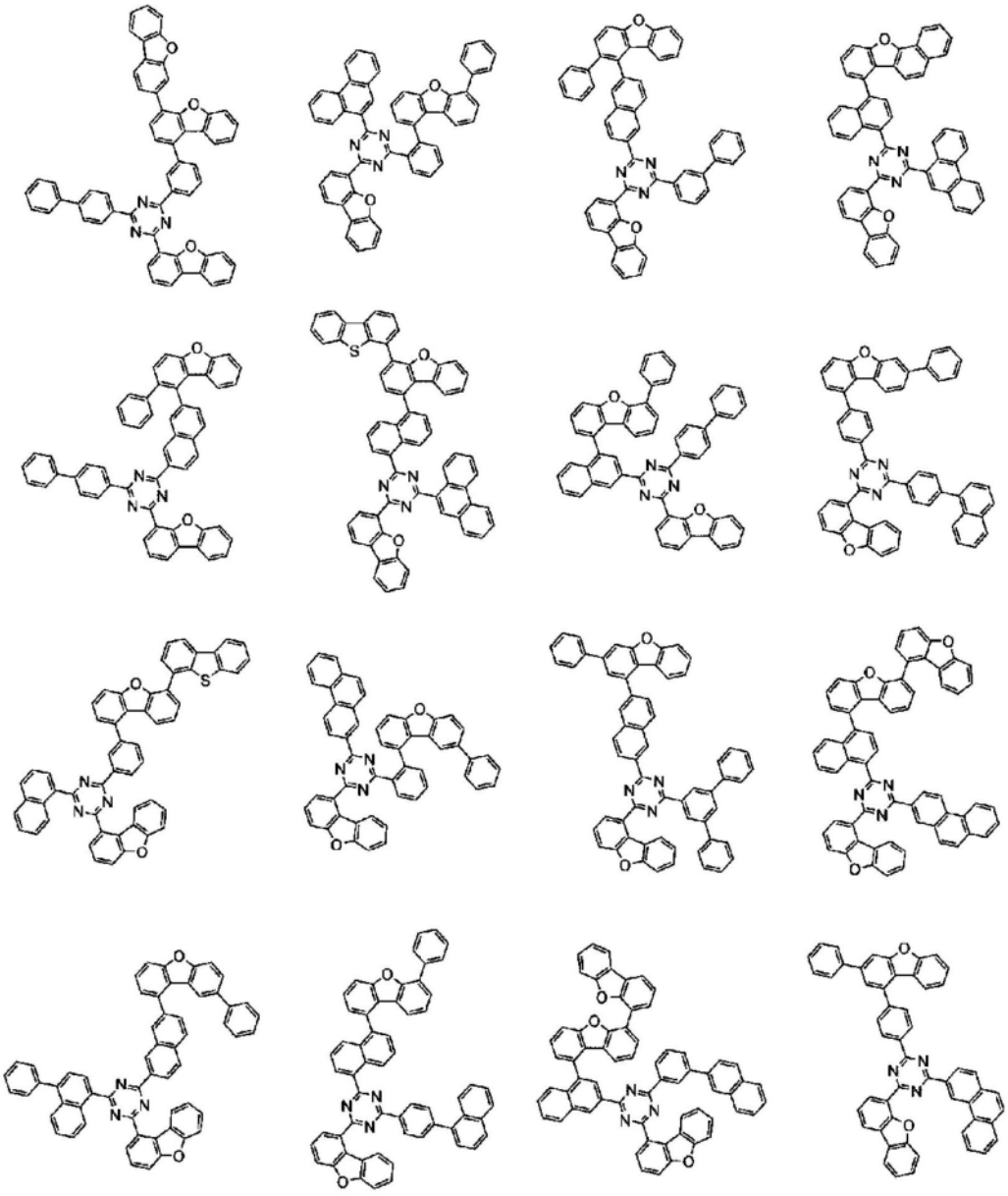


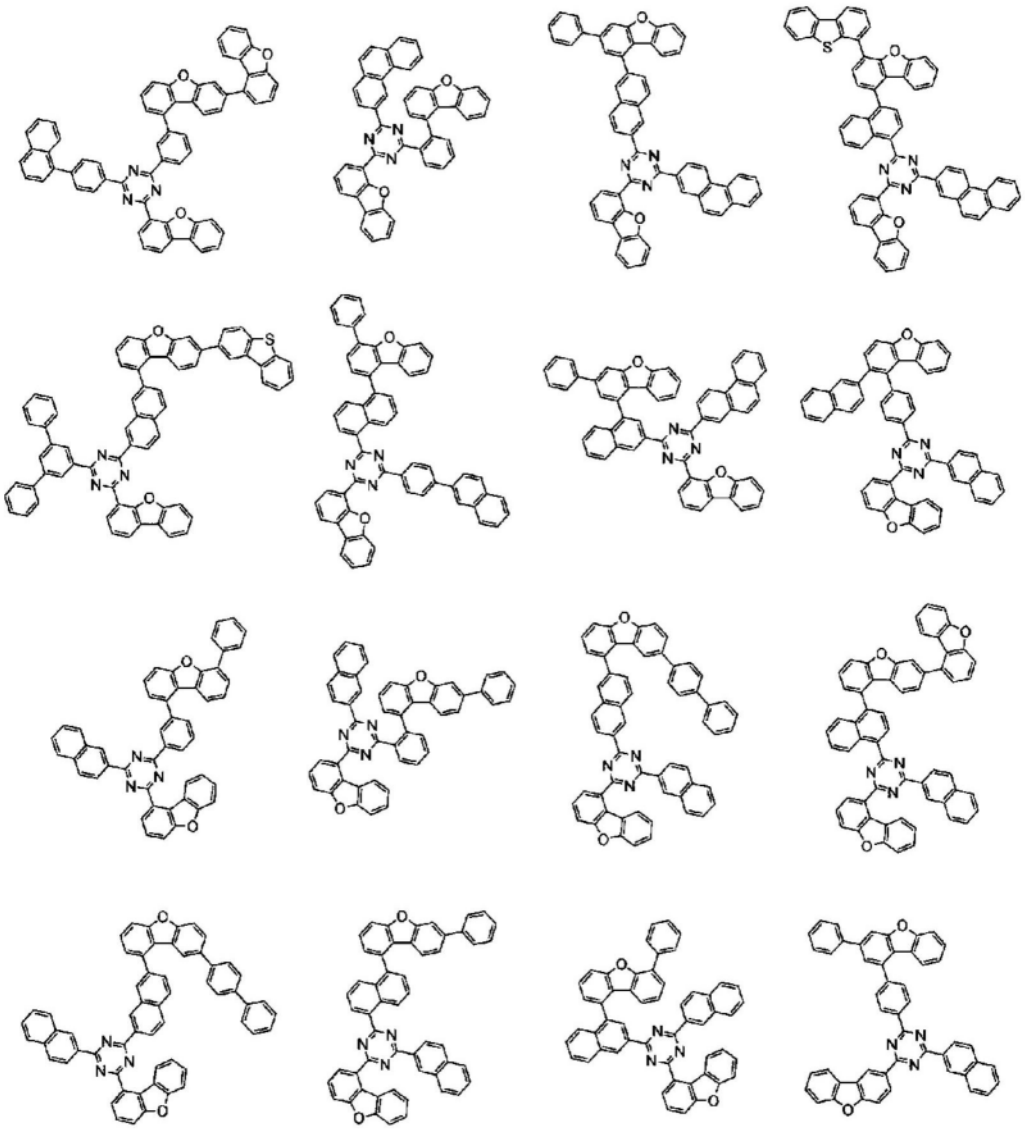


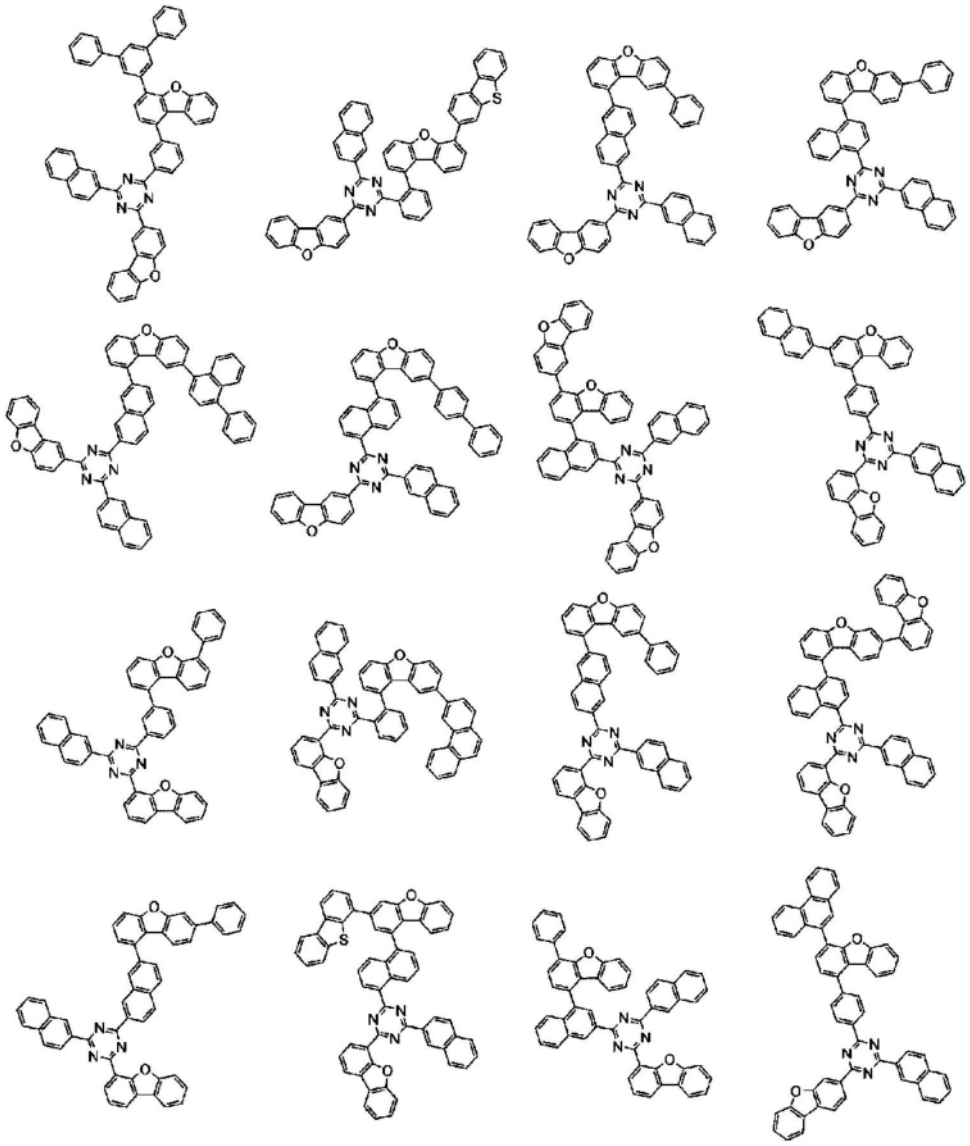


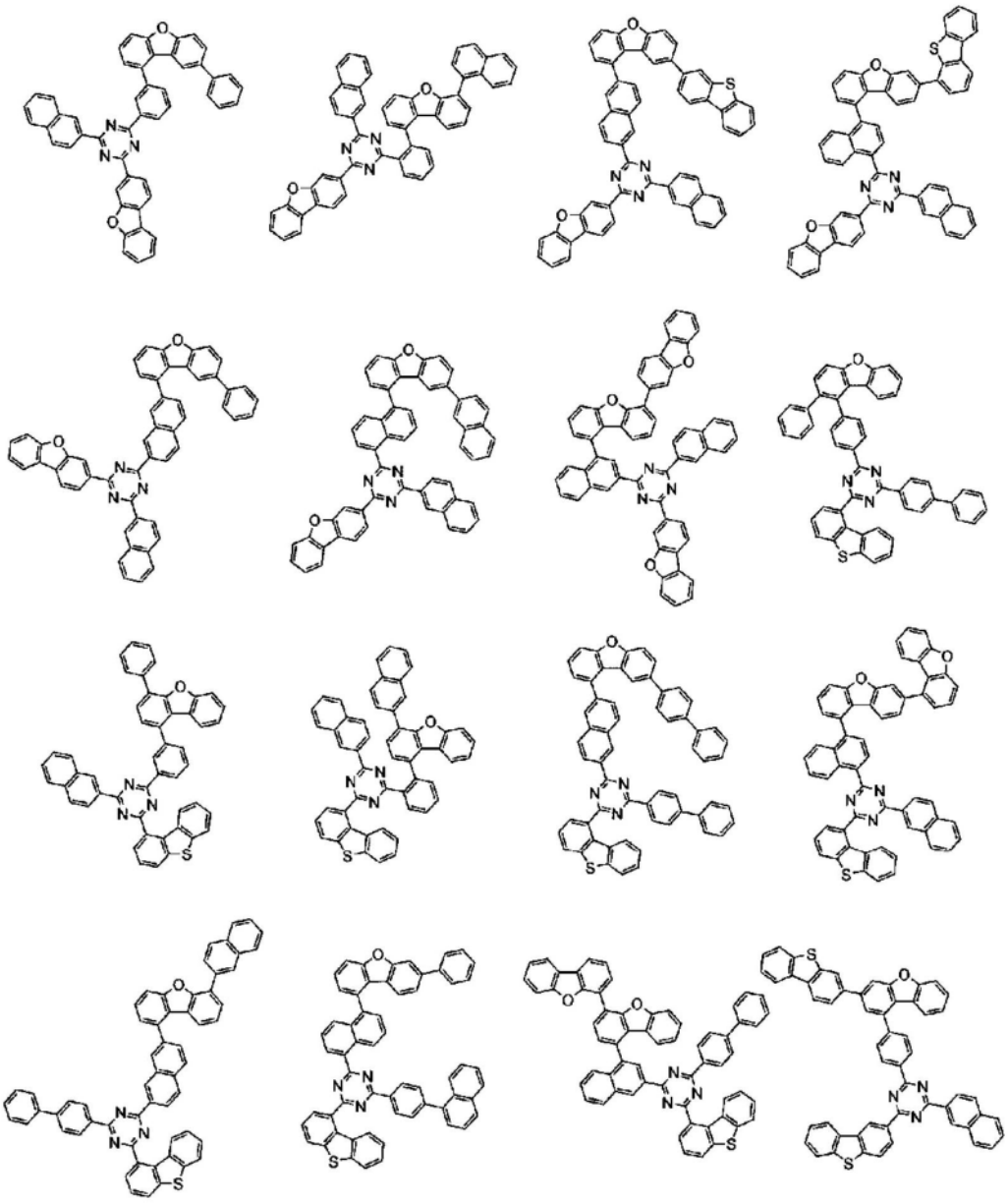


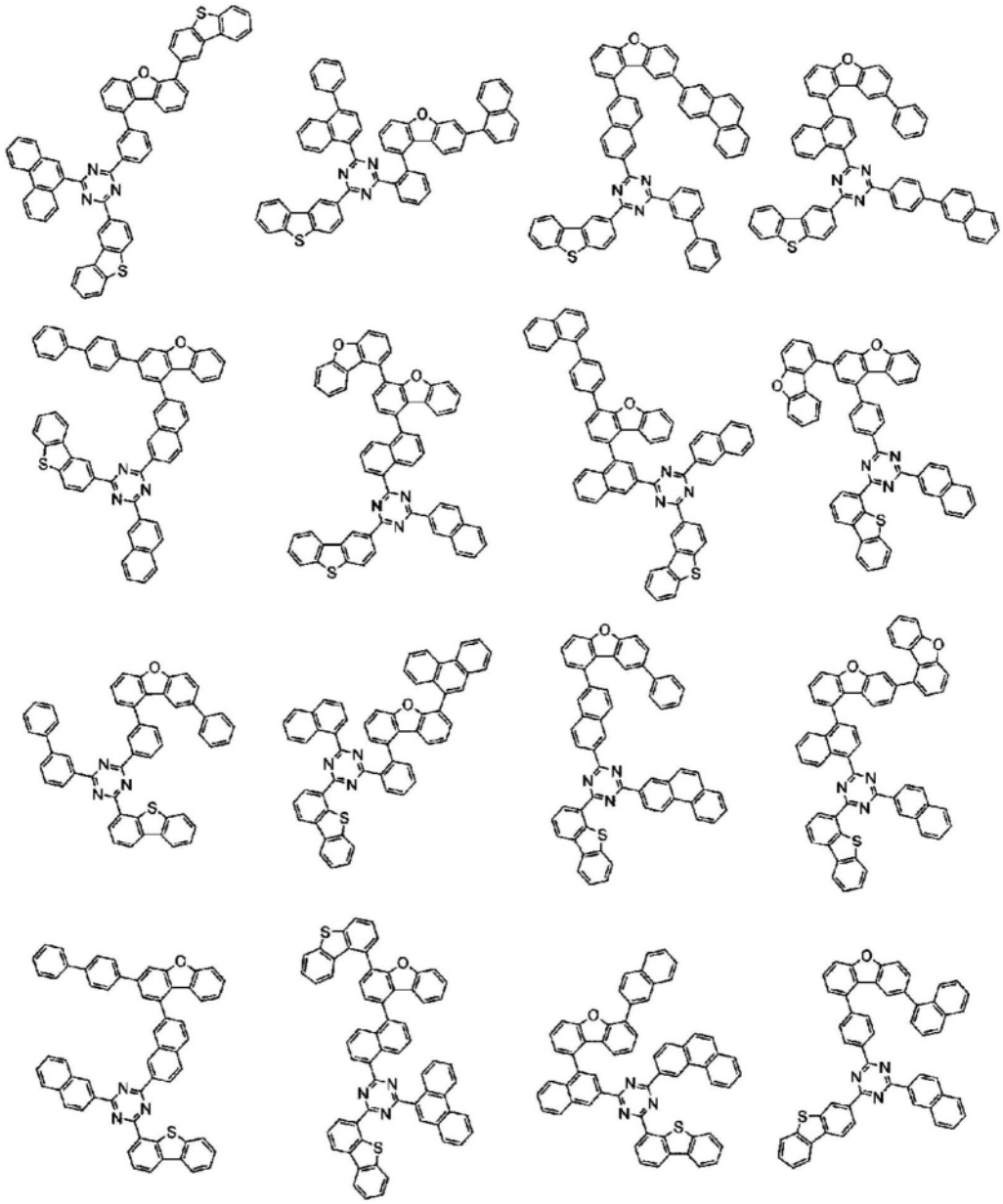


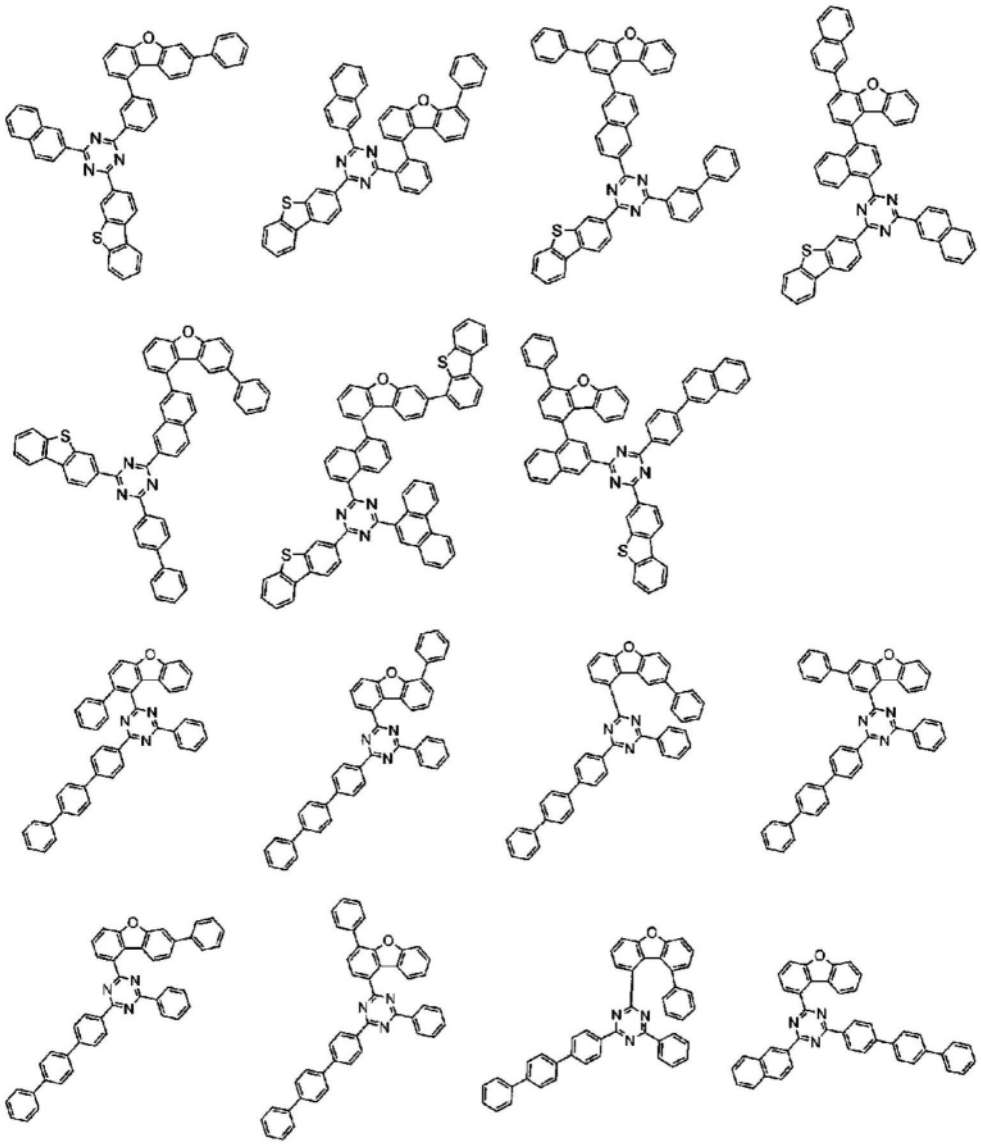


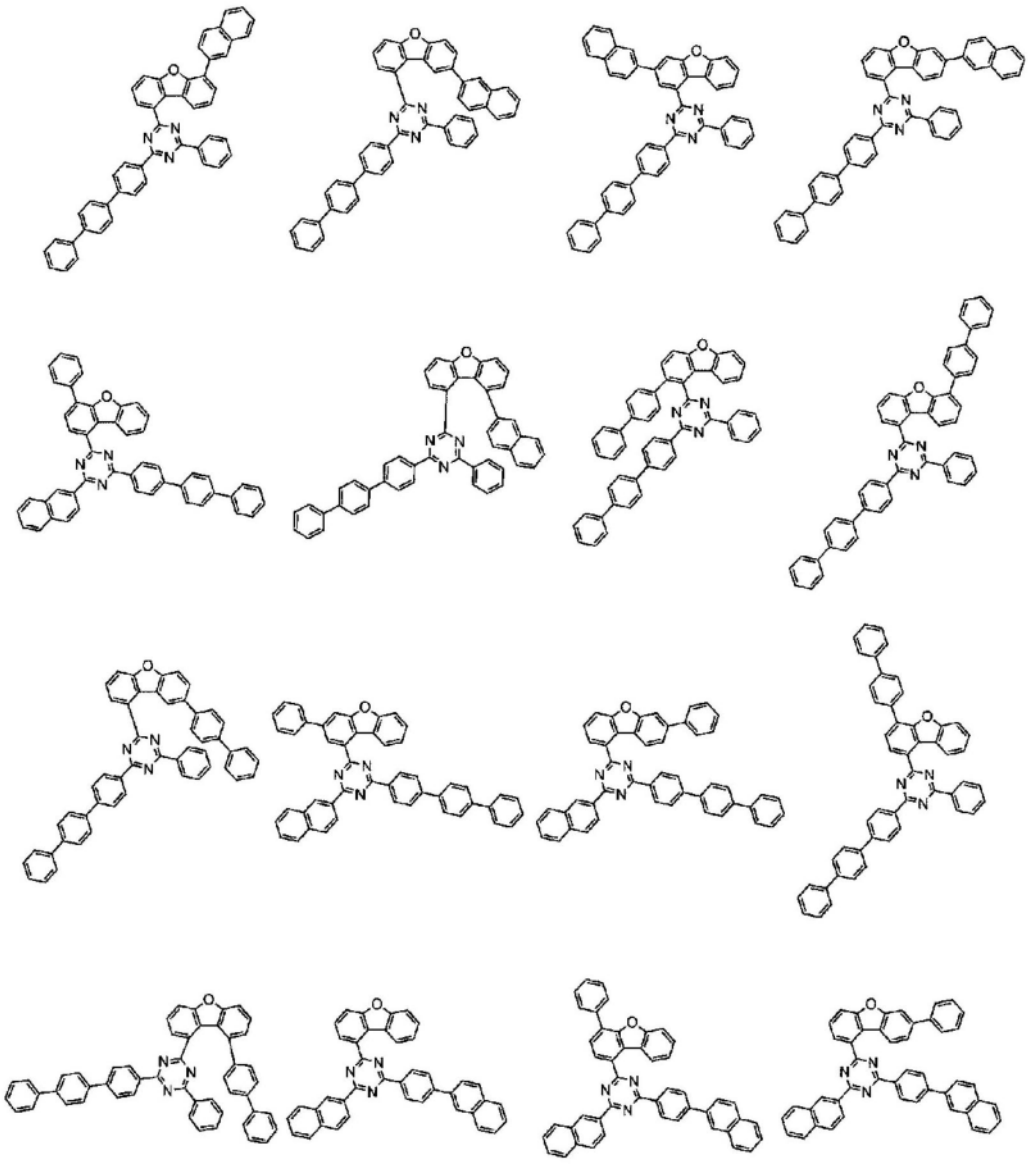


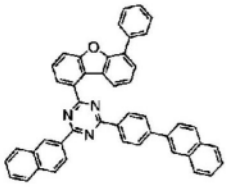
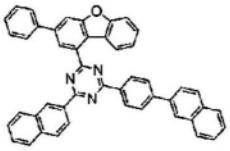
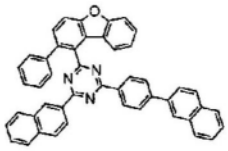
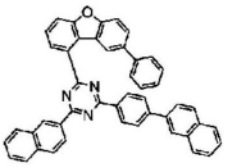
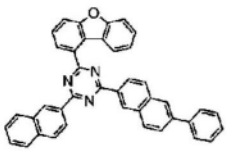
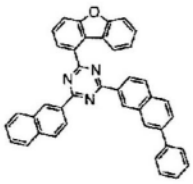
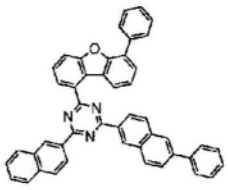
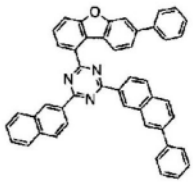
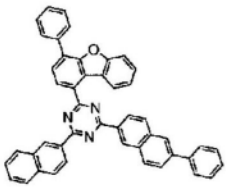
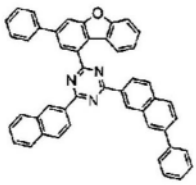
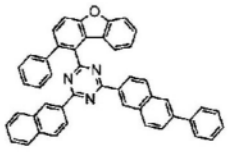
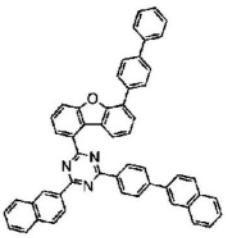
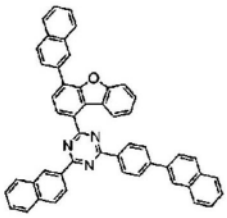
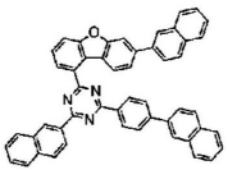
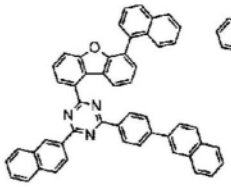
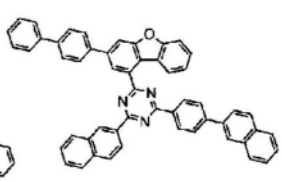


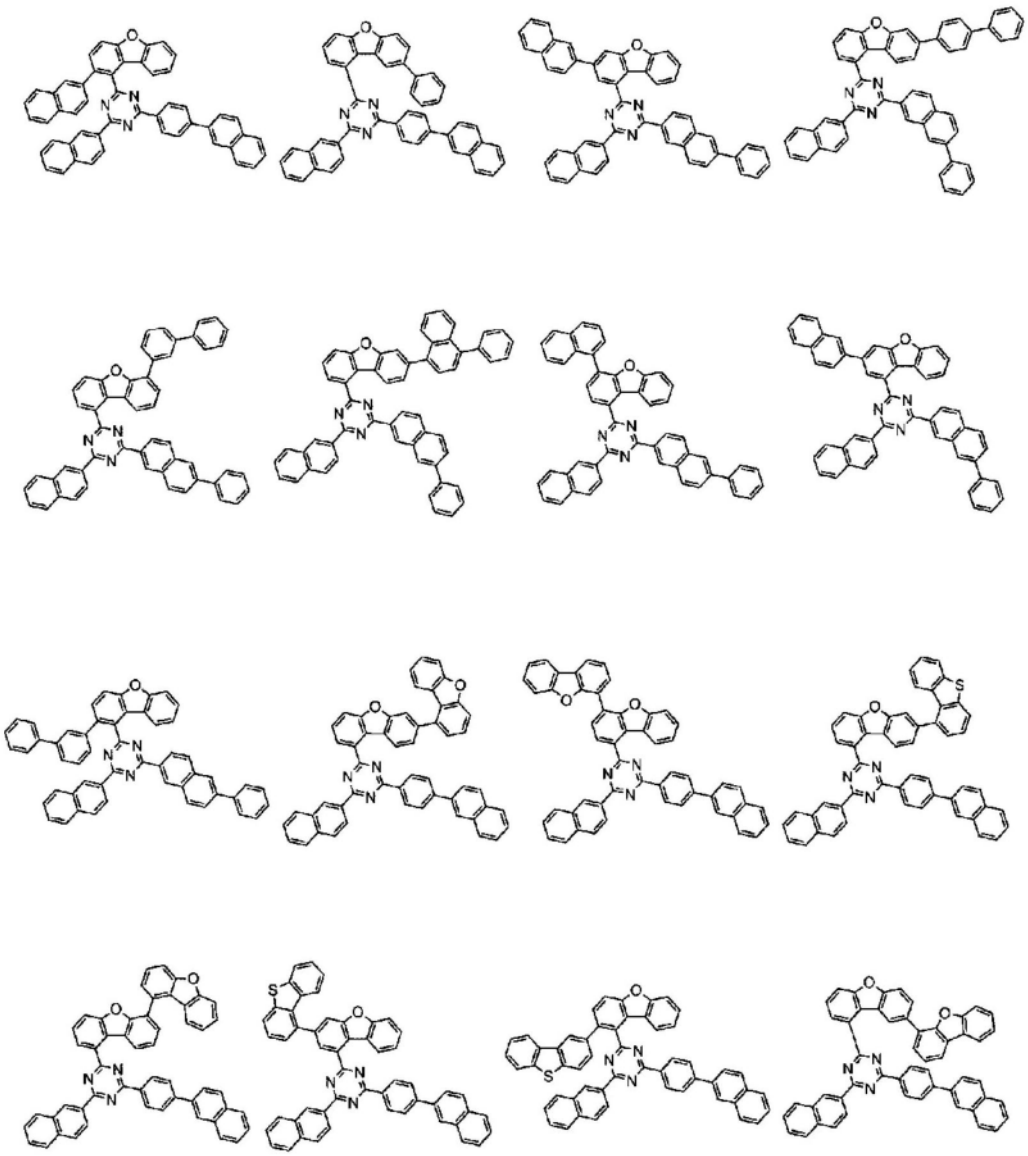


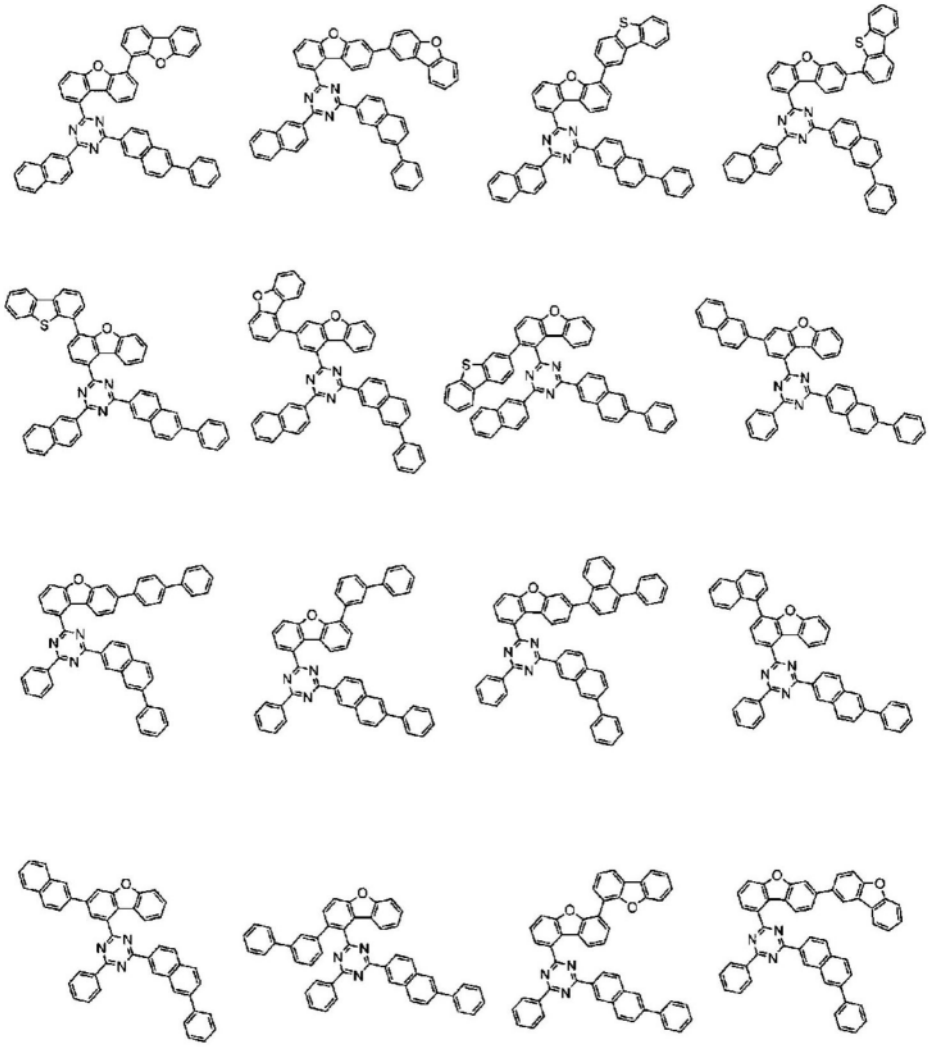


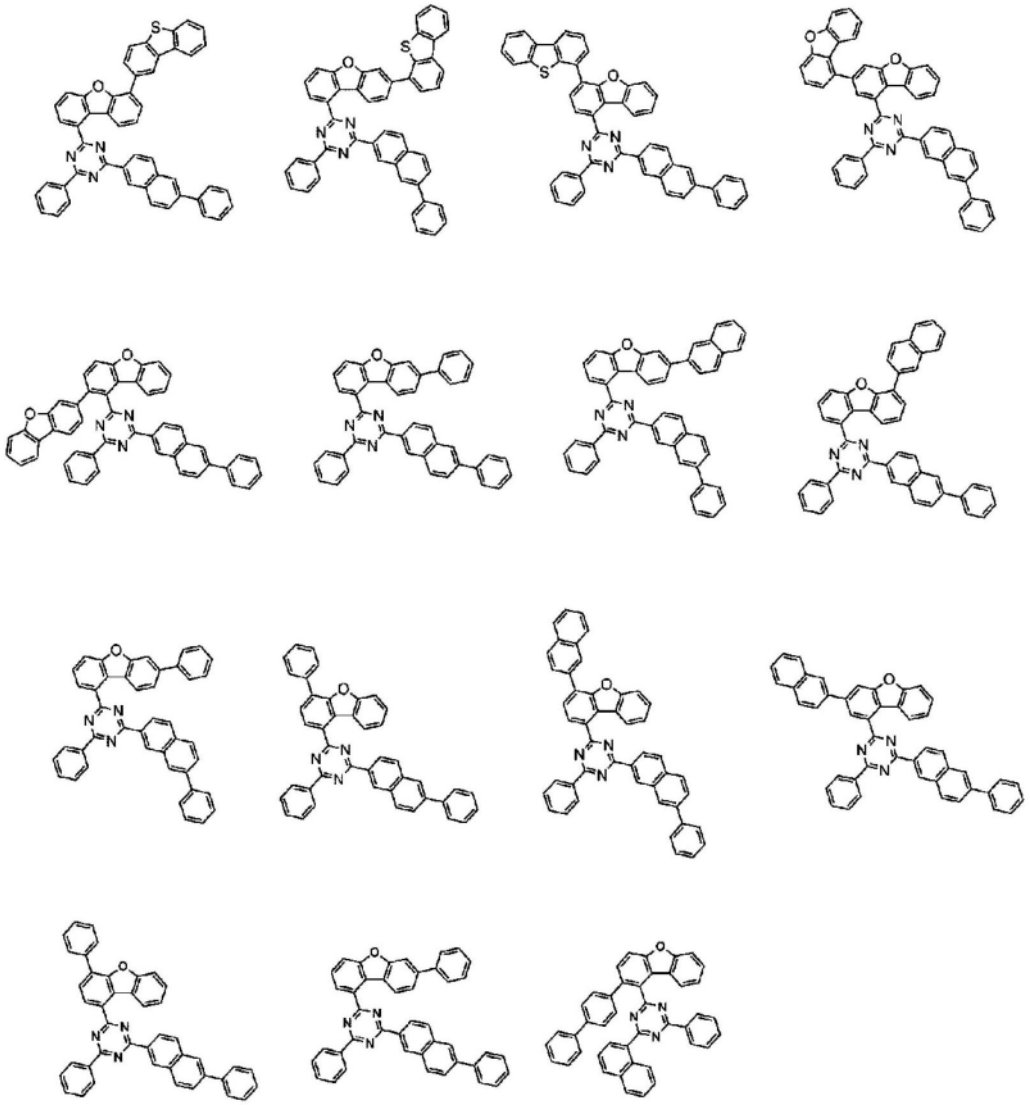








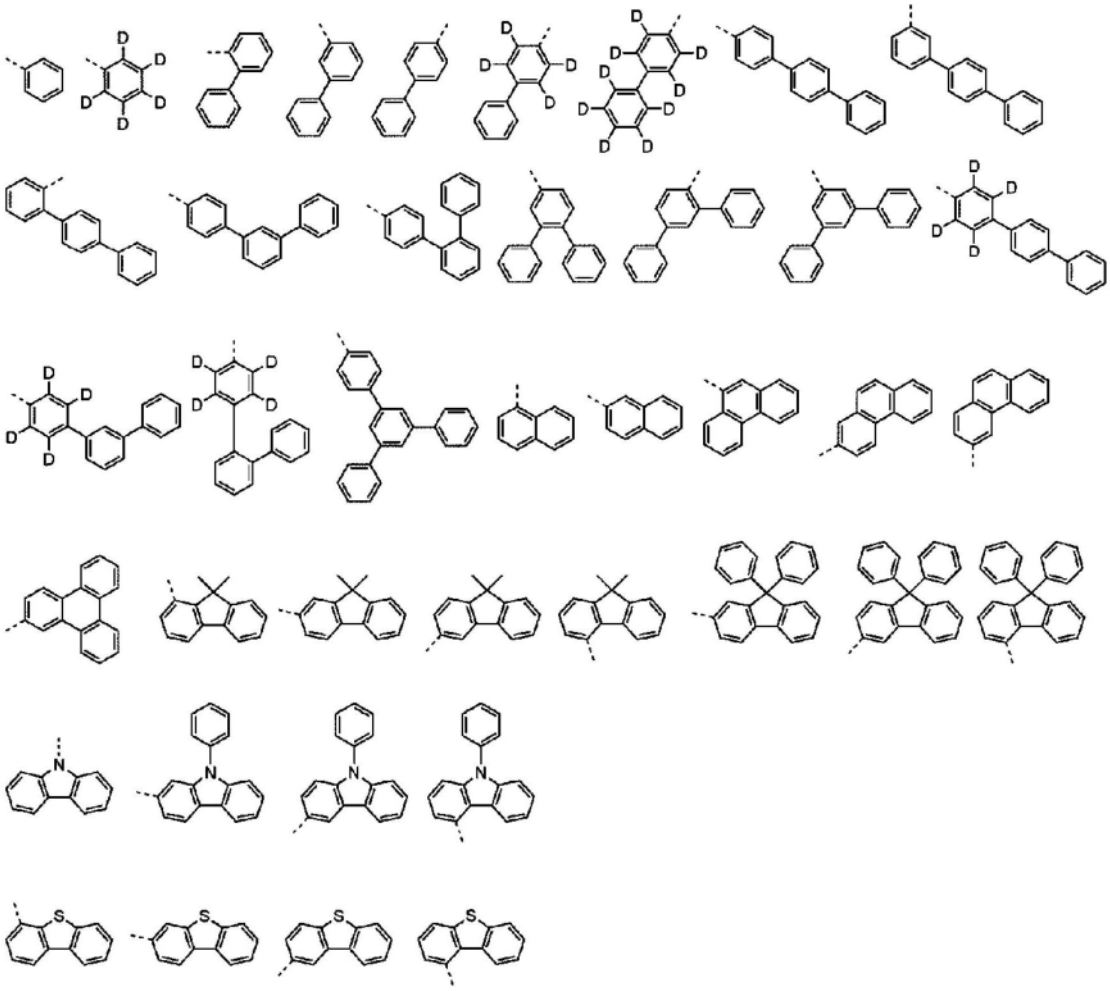


9.根据权利要求1所述的有机发光器件，

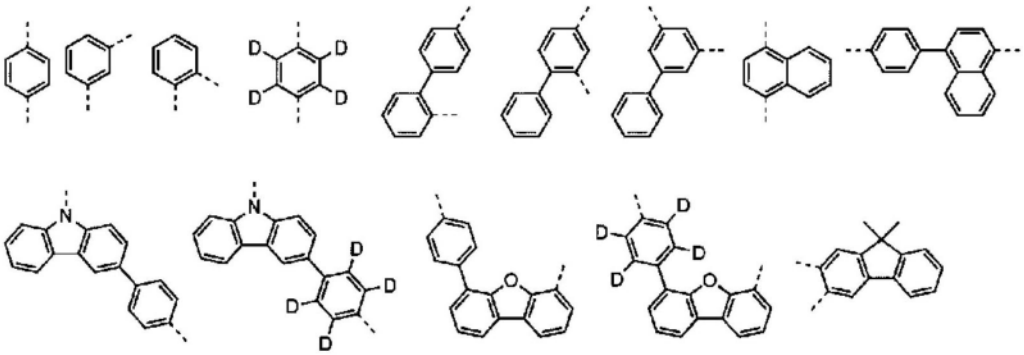
其中Ar_3为氢或苯基。

10.根据权利要求1所述的有机发光器件，

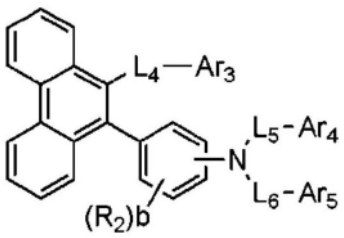
其中Ar_4和Ar_5各自独立地为选自以下中的任一者：



11.根据权利要求1所述的有机发光器件，
其中L_4为单键或选自以下中的任一者：



12.根据权利要求1所述的有机发光器件，
其中由化学式2表示的所述化合物由以下化学式2-1表示：
[化学式2-1]



其中在化学式2-1中，

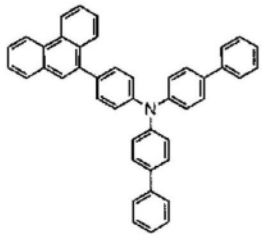
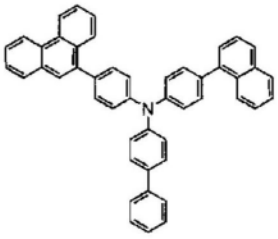
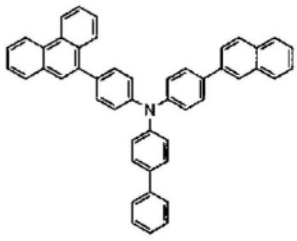
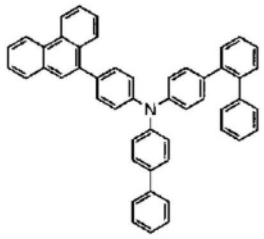
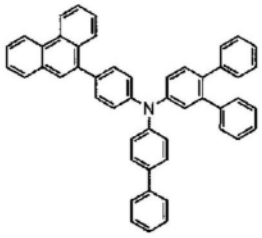
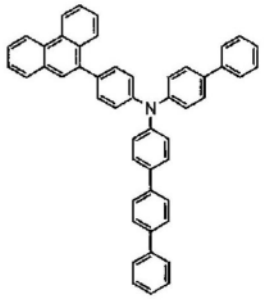
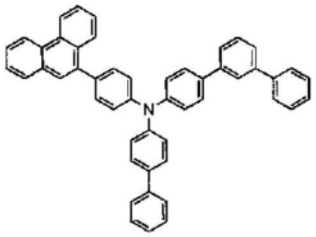
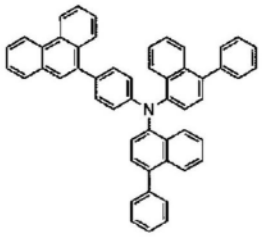
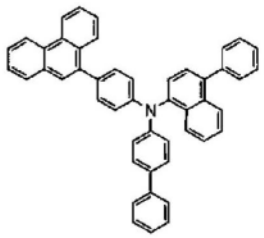
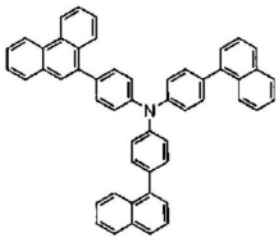
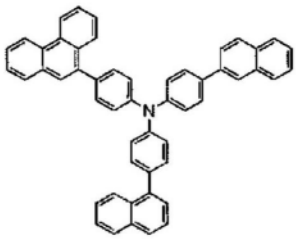
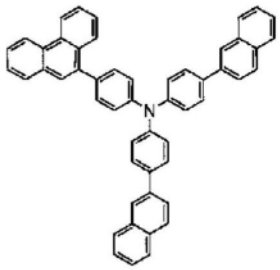
Ar_3至Ar_5和L_4至L_6如权利要求1中所限定，R_2为氢、或者氘，以及

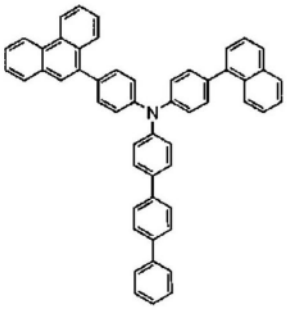
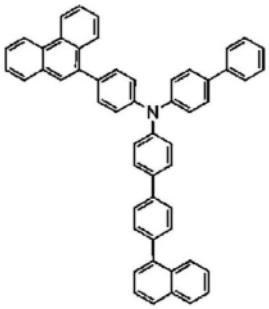
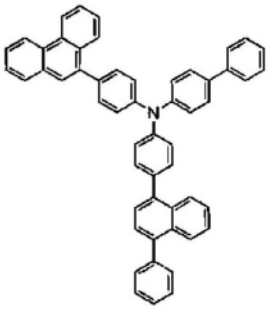
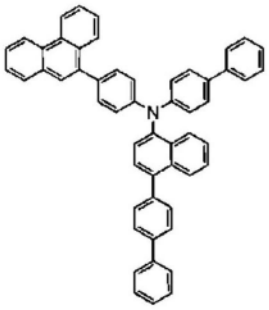
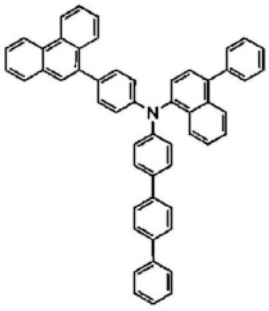
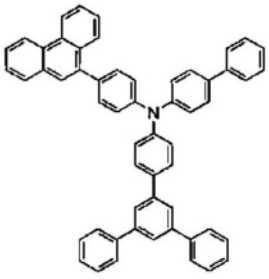
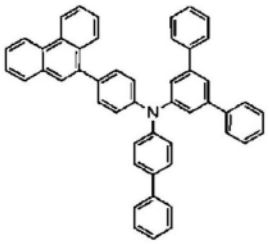
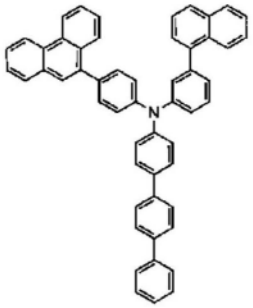
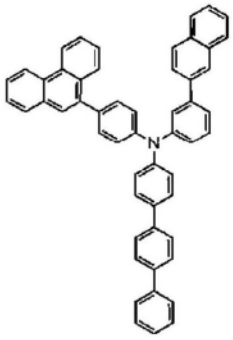
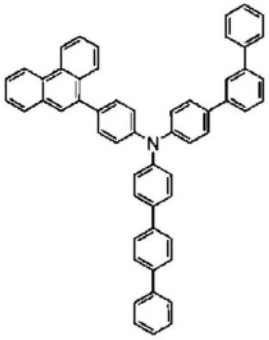
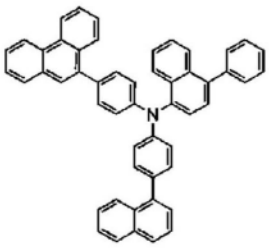
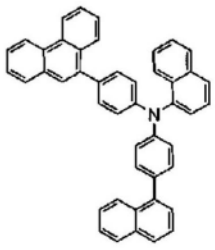
b为0至4的整数。

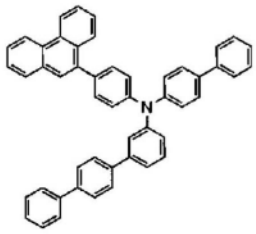
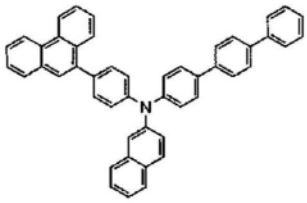
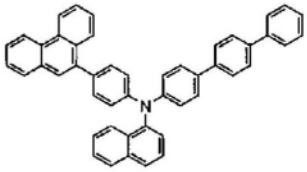
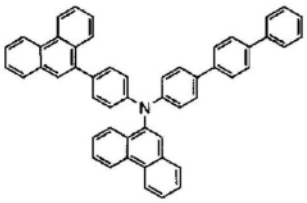
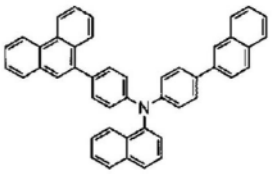
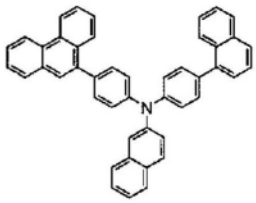
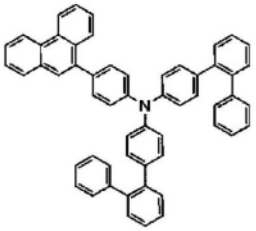
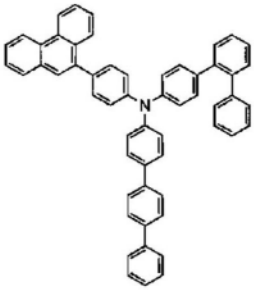
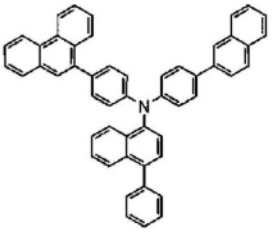
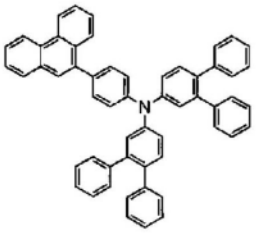
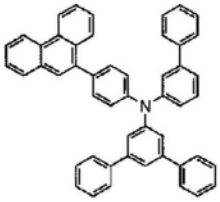
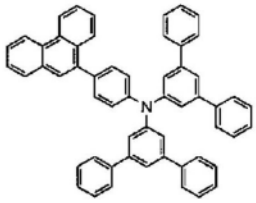
13.根据权利要求12所述的有机发光器件，其中R_2为氢。

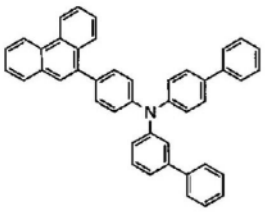
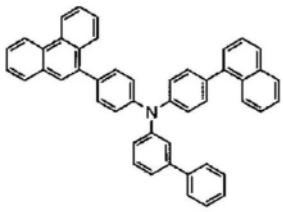
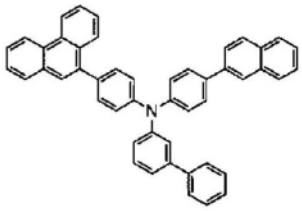
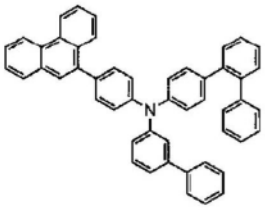
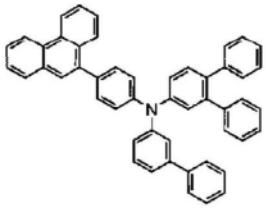
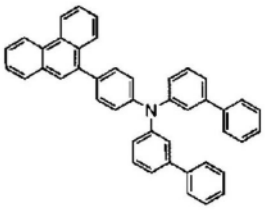
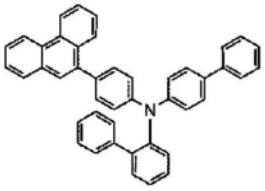
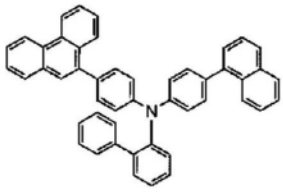
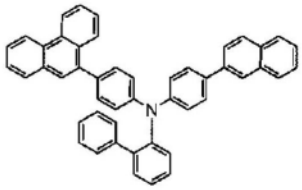
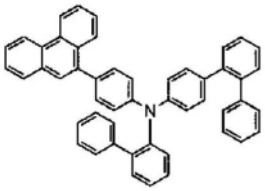
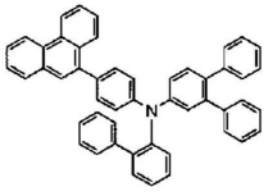
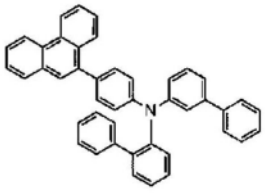
14.根据权利要求1所述的有机发光器件，

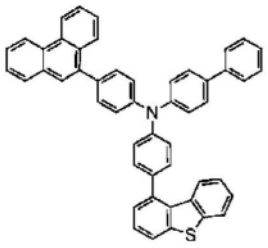
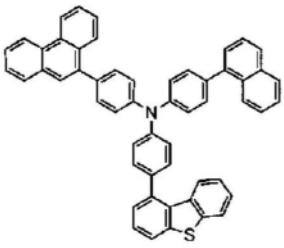
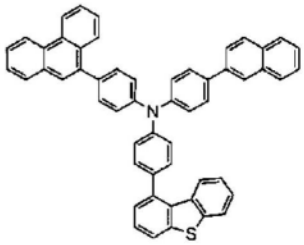
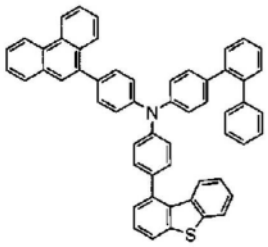
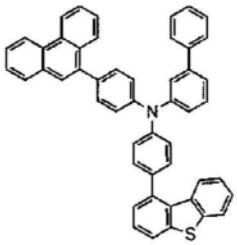
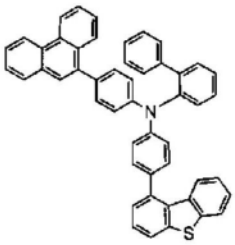
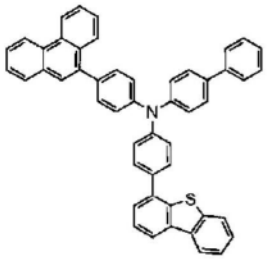
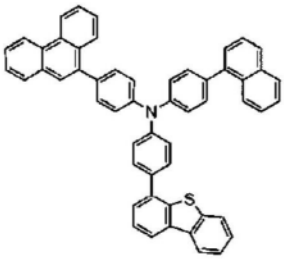
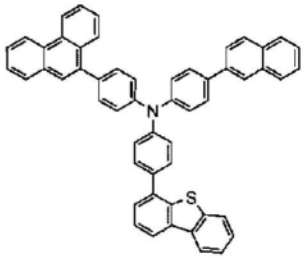
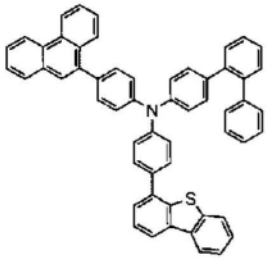
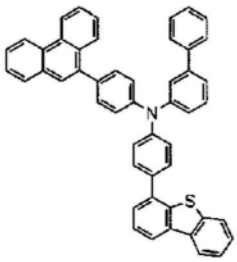
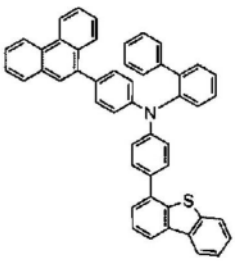
其中由化学式2表示的所述化合物选自以下：

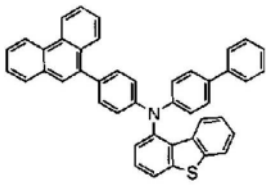
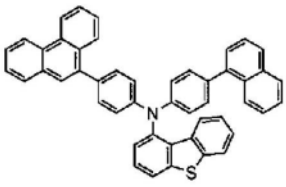
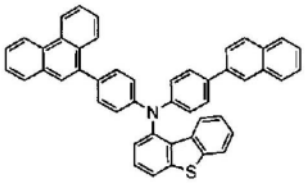
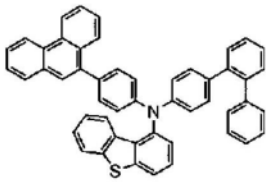
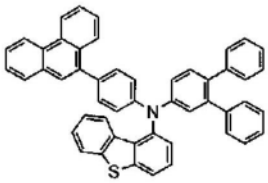
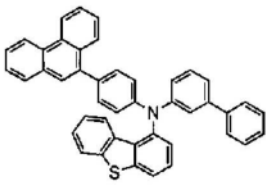
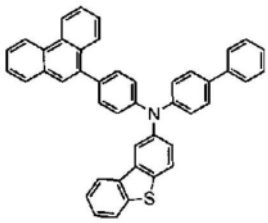
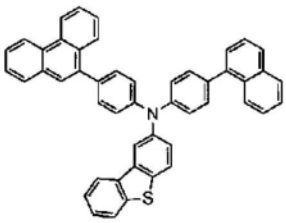
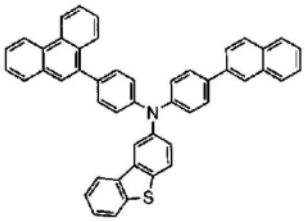
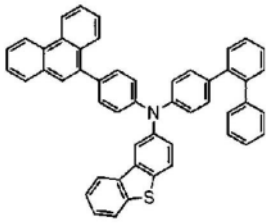
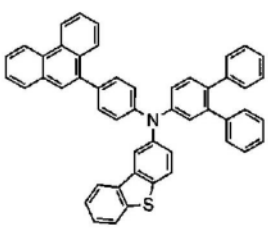
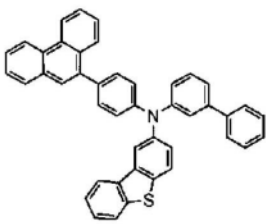













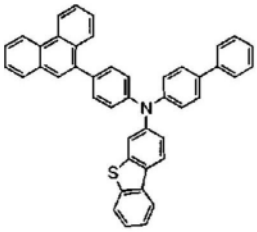
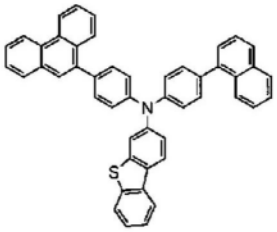
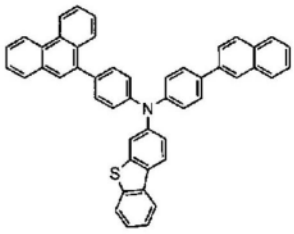
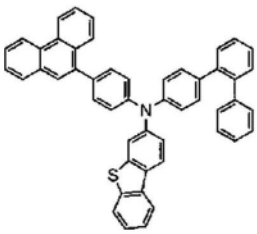
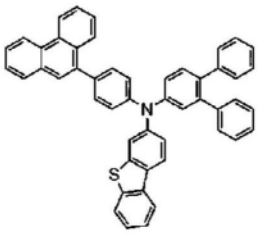
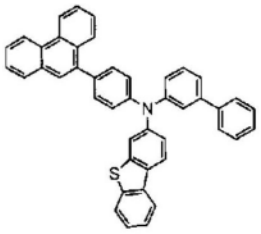
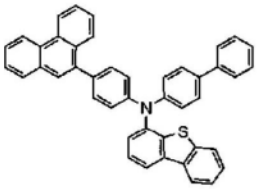
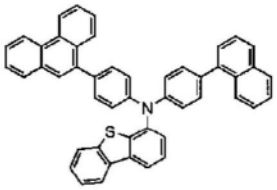
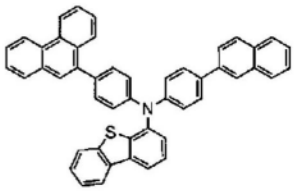
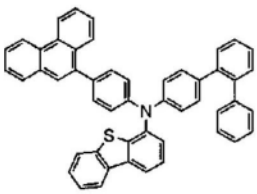
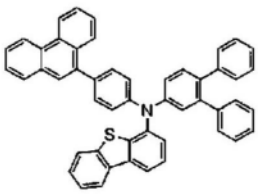
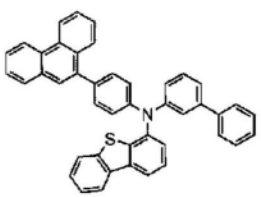













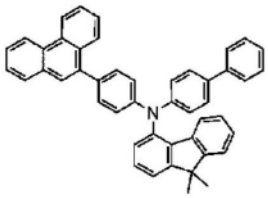
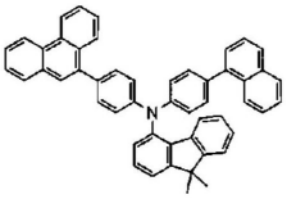
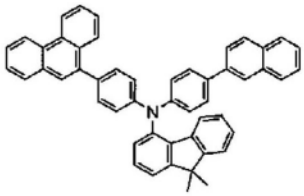
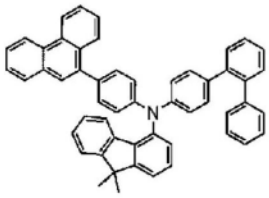
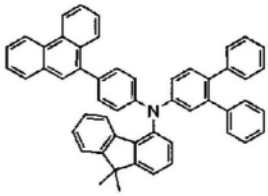
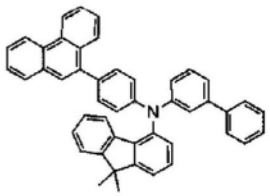
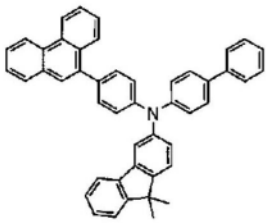
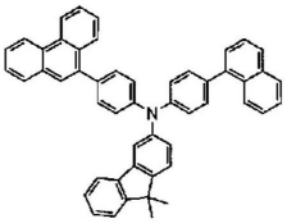
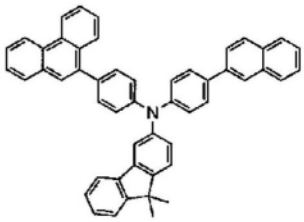
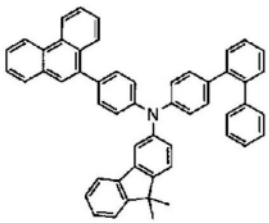
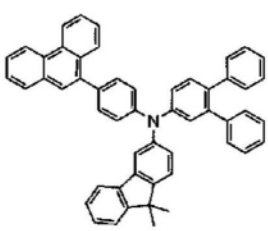
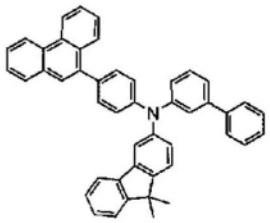













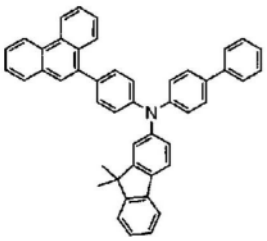
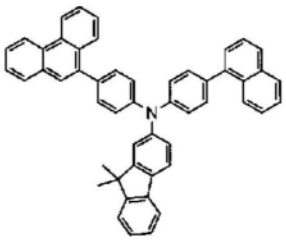
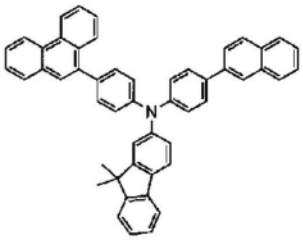
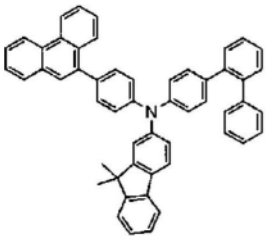
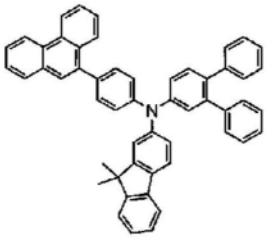
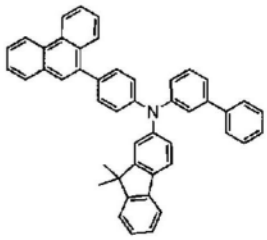
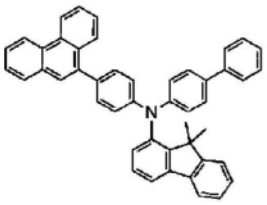
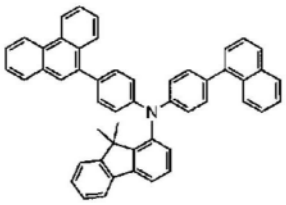
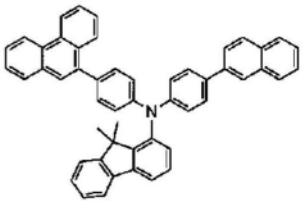
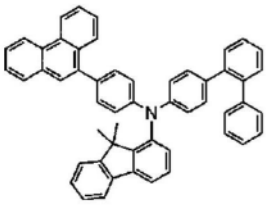
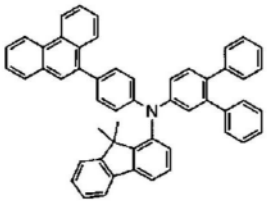
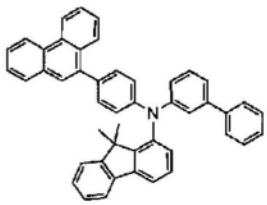













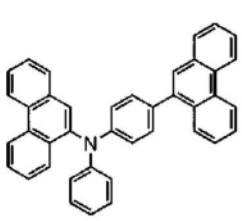
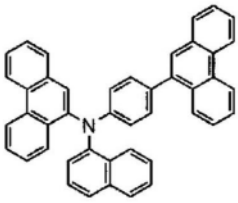
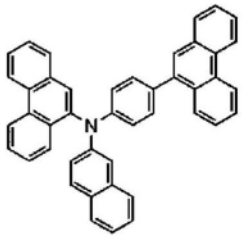
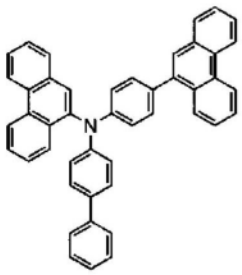
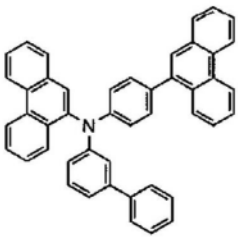
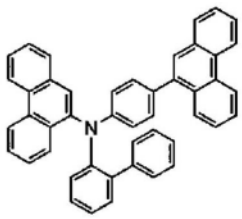
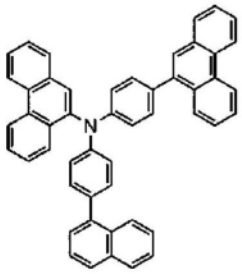
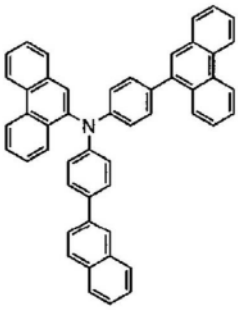
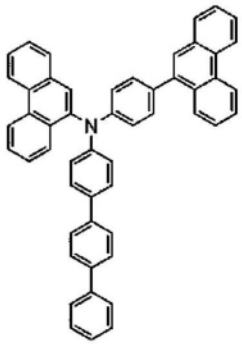
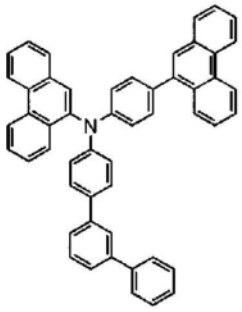
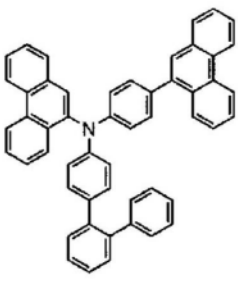
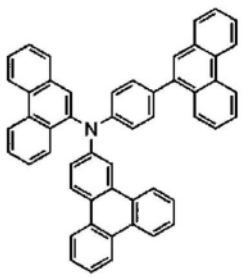













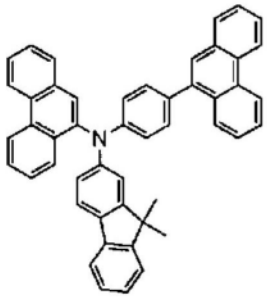













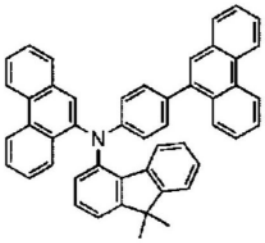













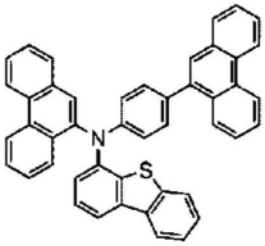













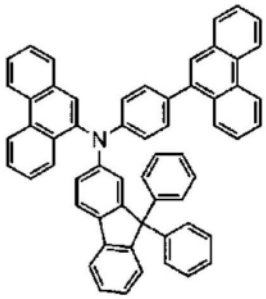













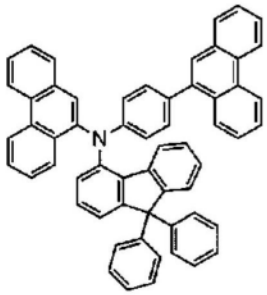













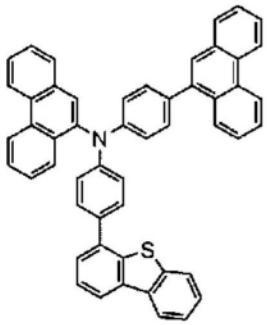


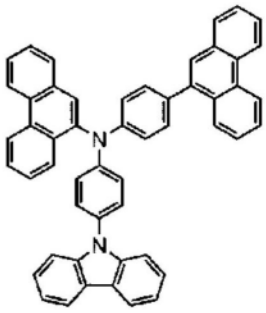


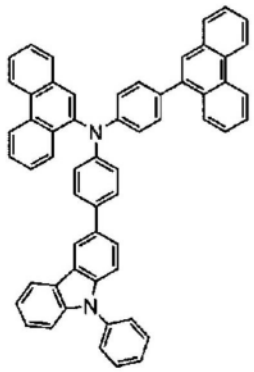
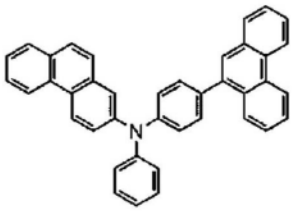
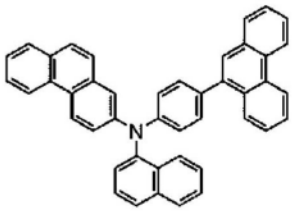
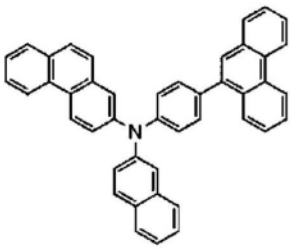
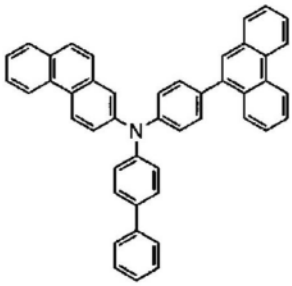
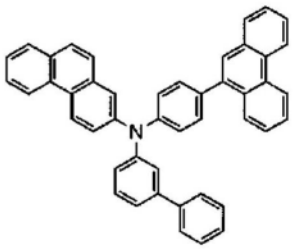
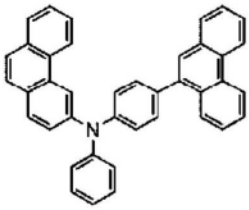
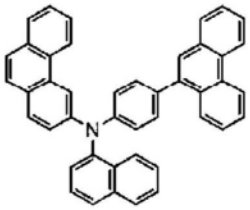
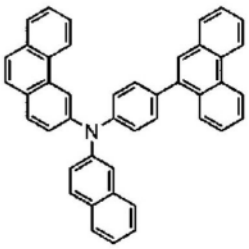
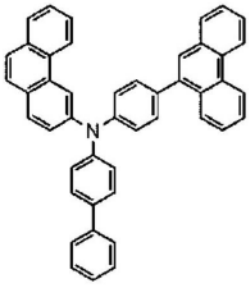
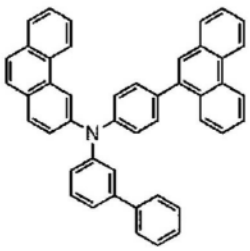
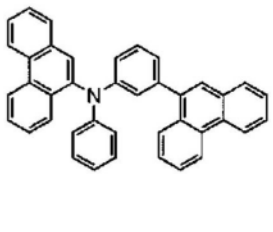


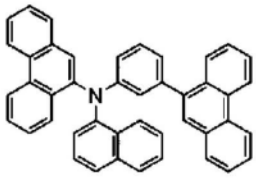
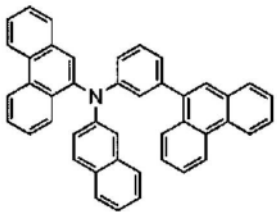
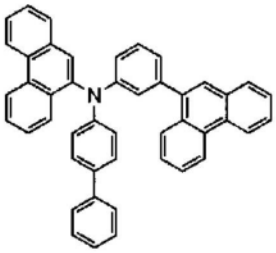
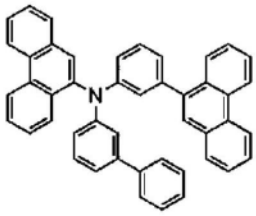
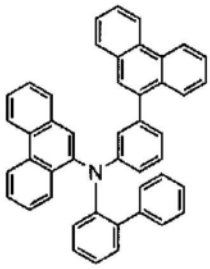
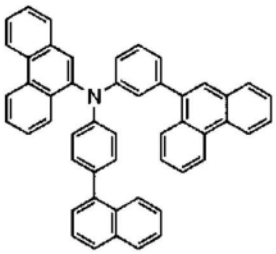
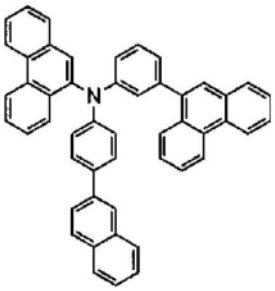
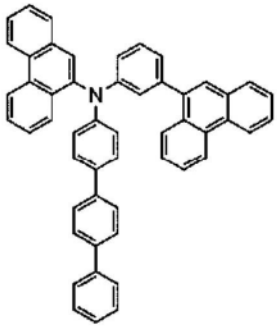
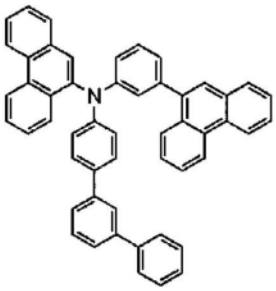
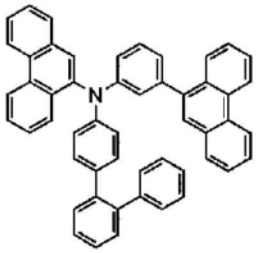
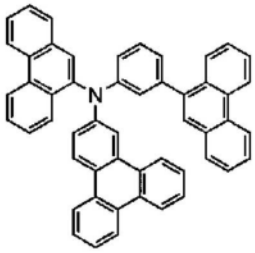
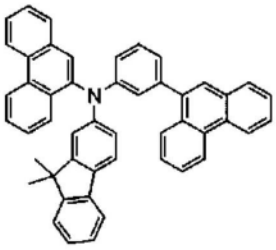


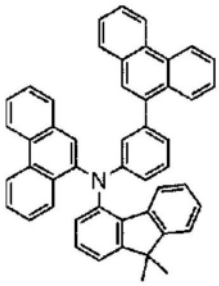


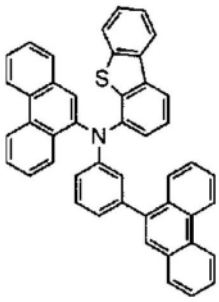


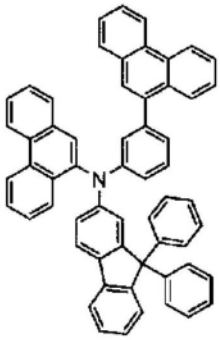


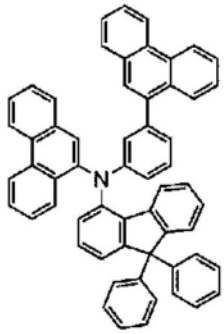













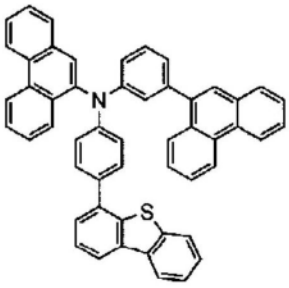













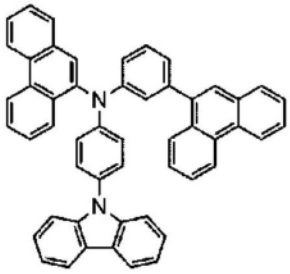


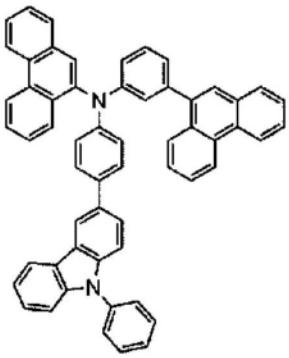


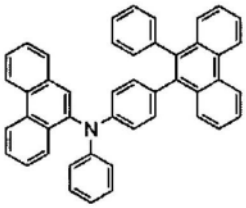
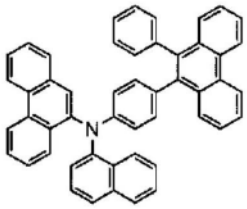
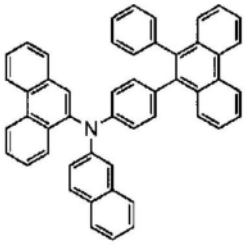
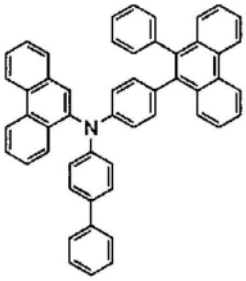
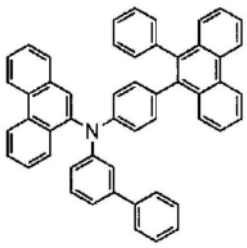
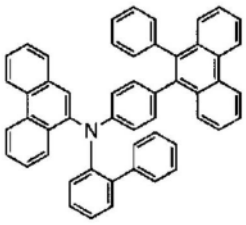
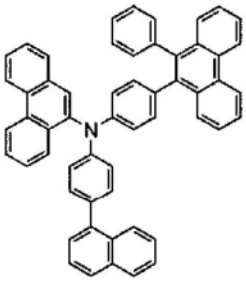
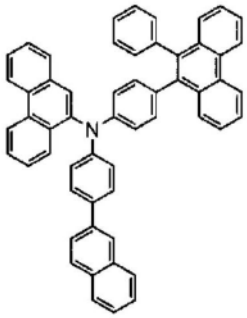
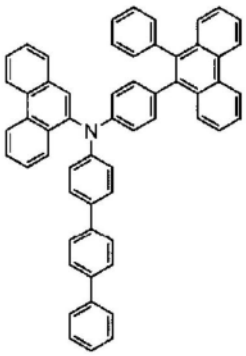
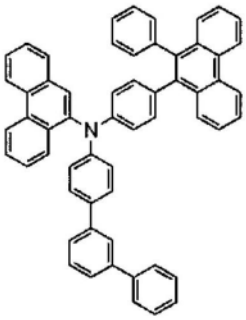
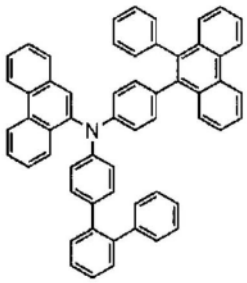
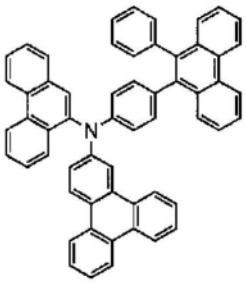


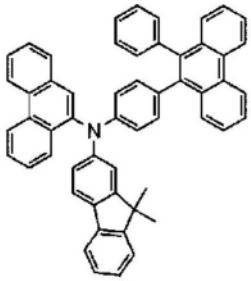


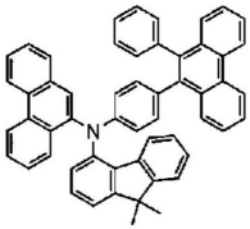


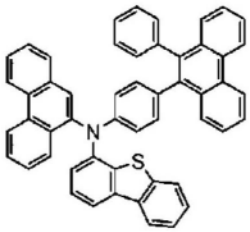


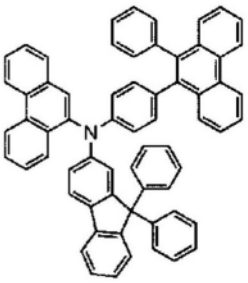


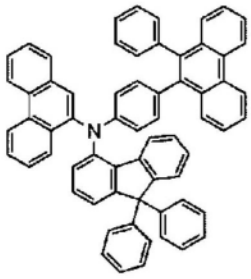













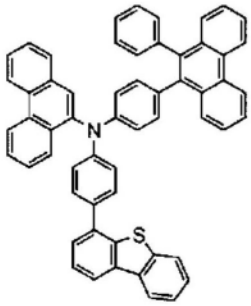


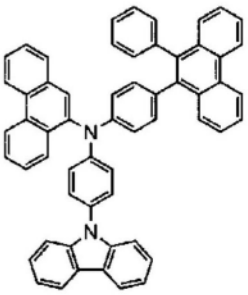


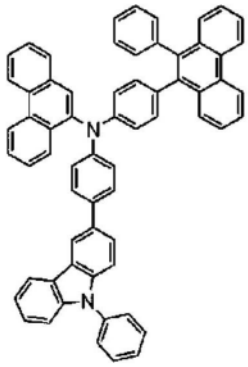
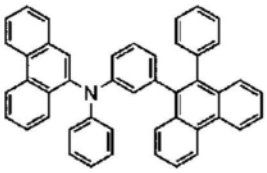
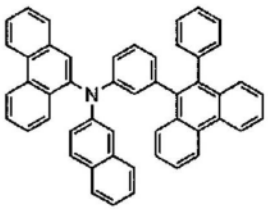
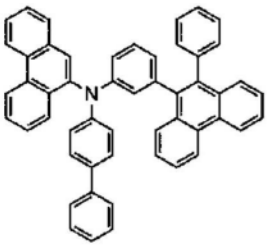
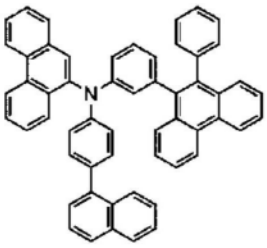
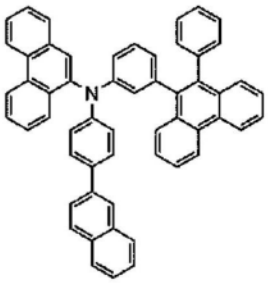
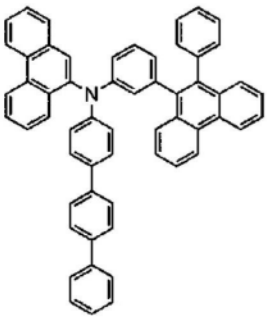
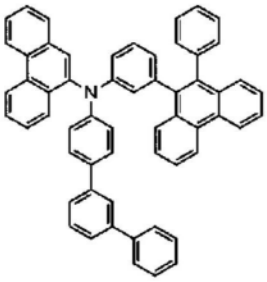
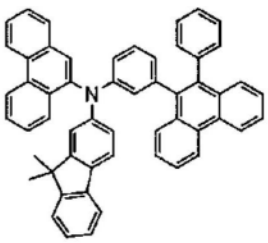
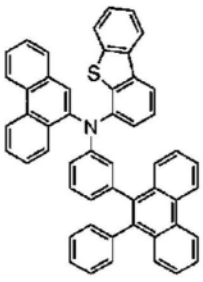


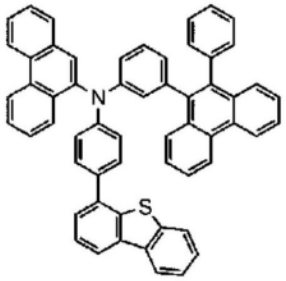
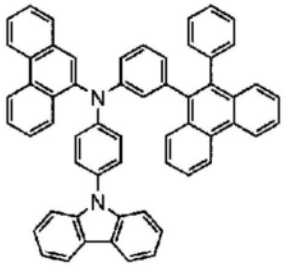
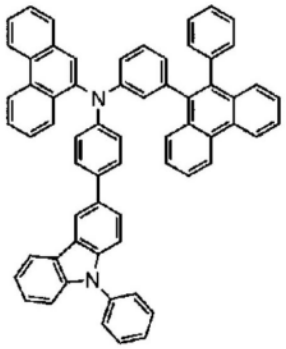
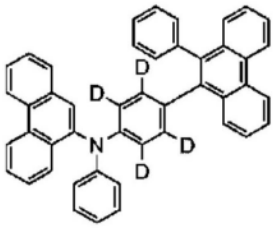
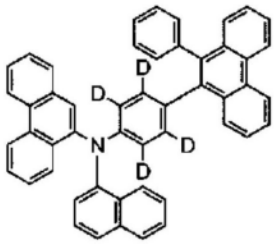
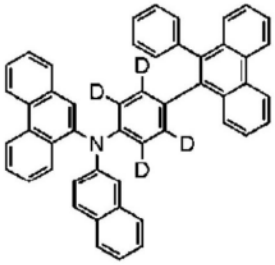
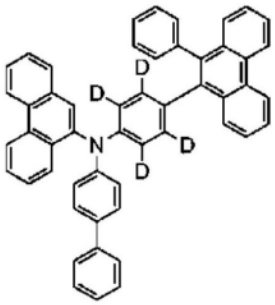
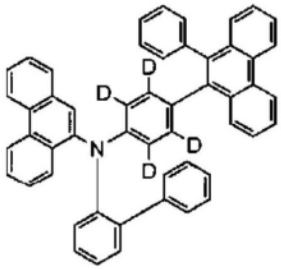
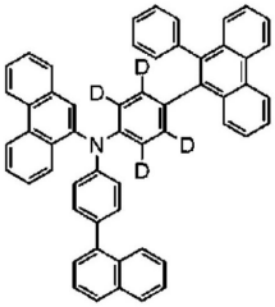
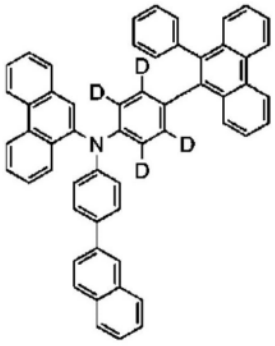
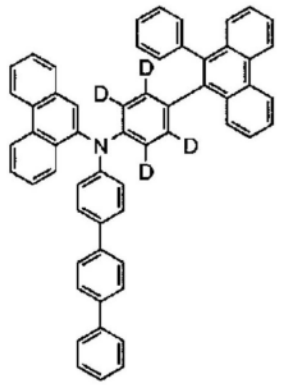


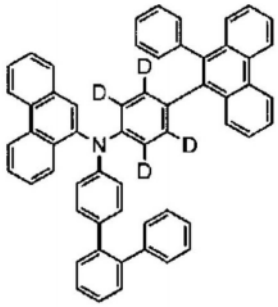
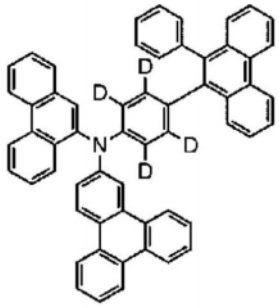
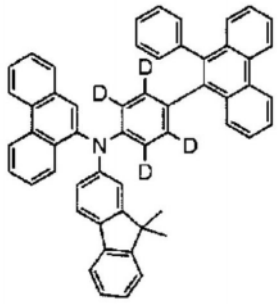
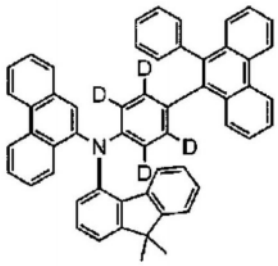
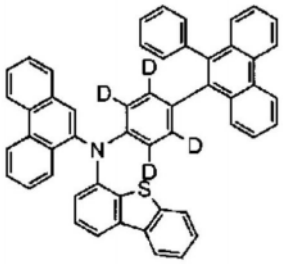
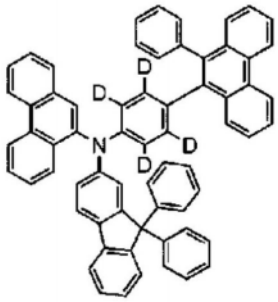
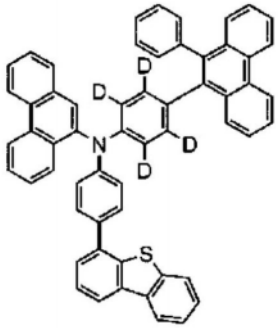
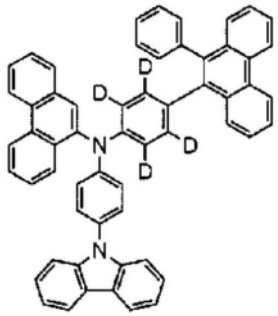


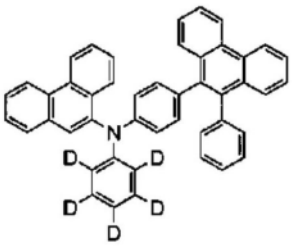
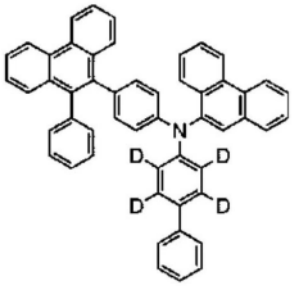
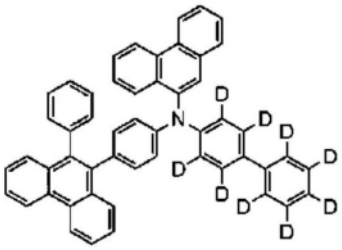
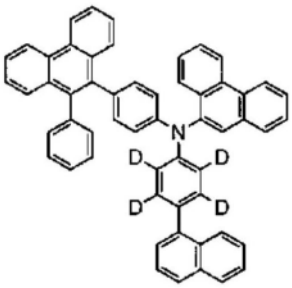
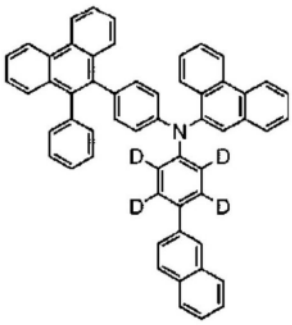
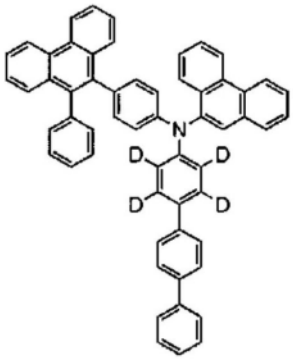
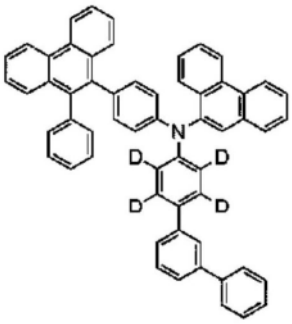
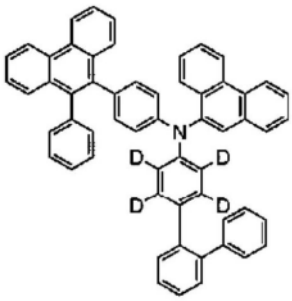
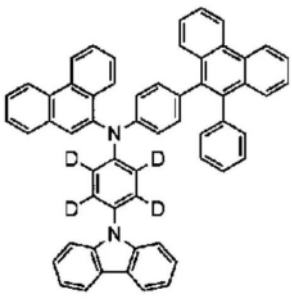
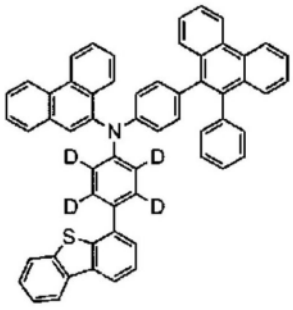


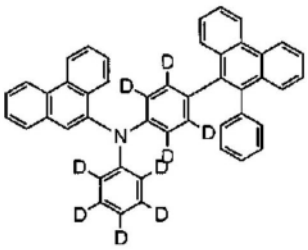
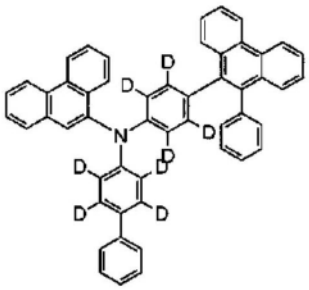
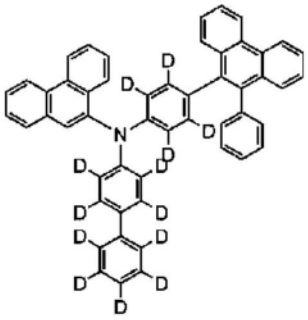
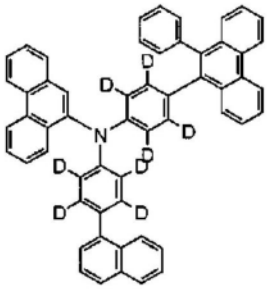
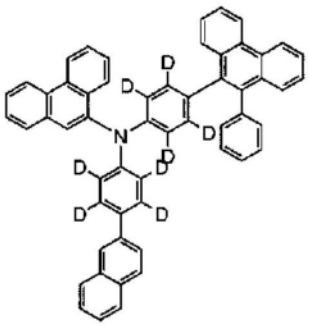
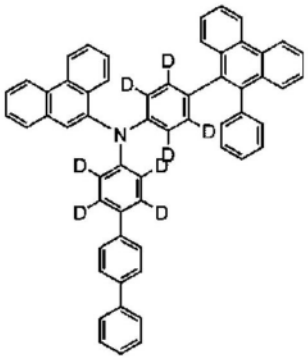
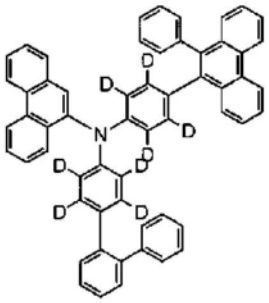
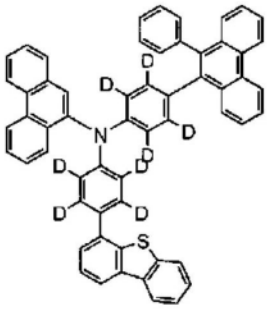
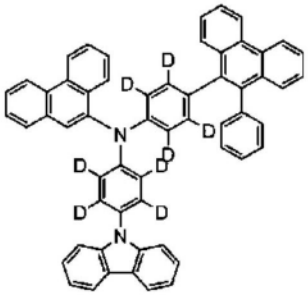


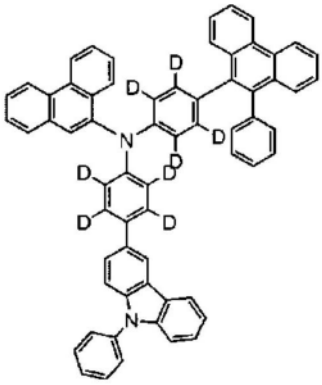
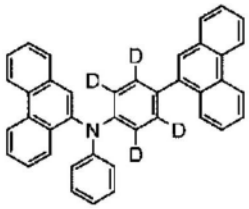
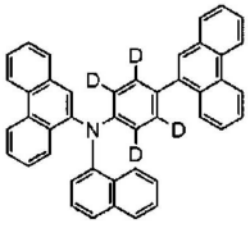
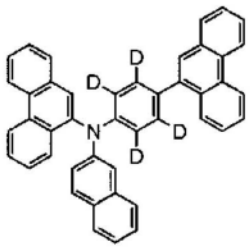
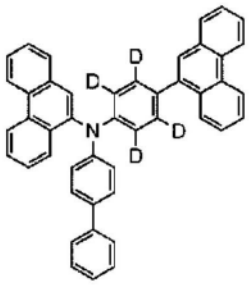
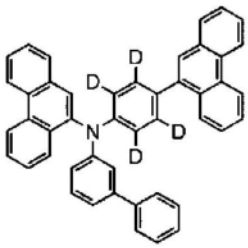
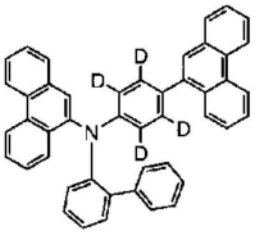
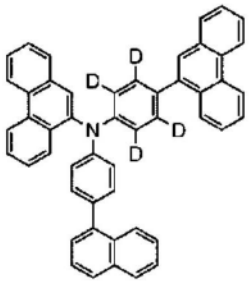
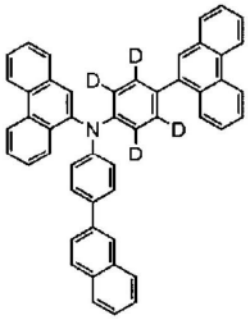
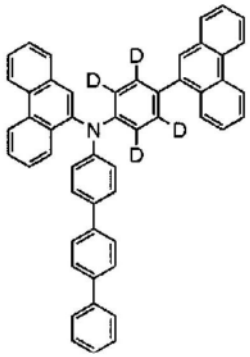
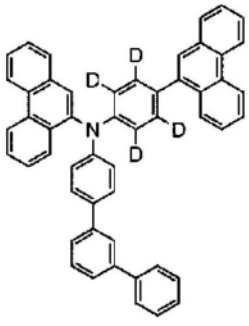
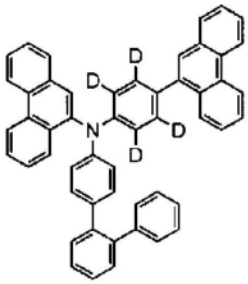











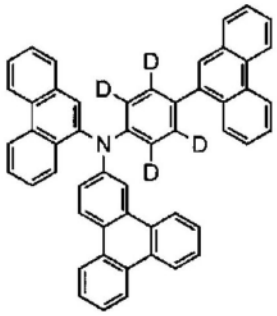












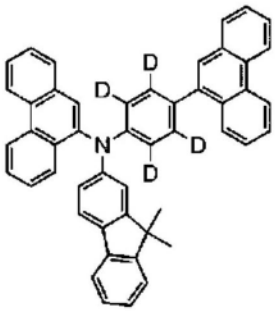









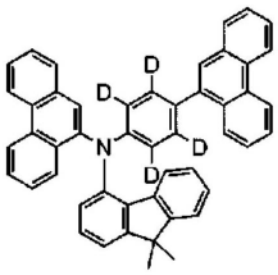











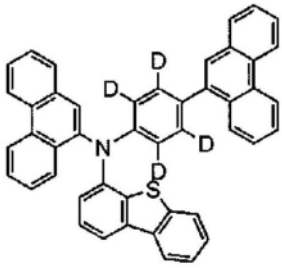










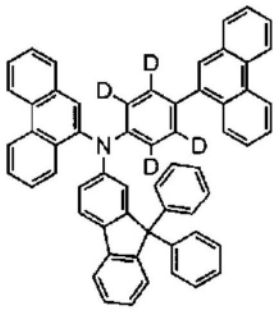













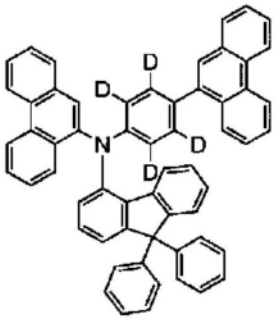


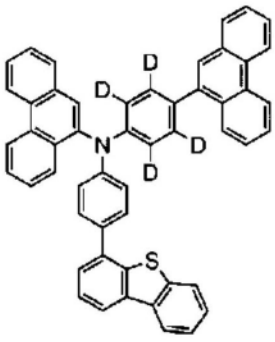


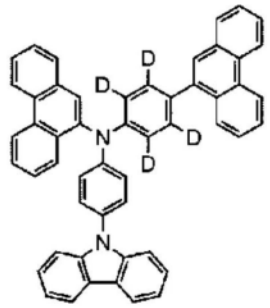
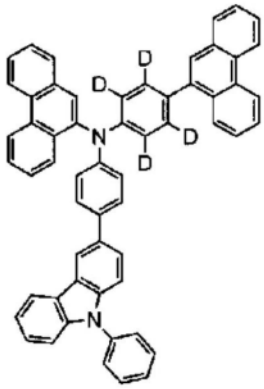
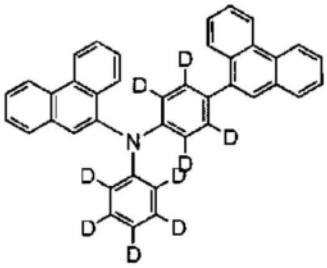
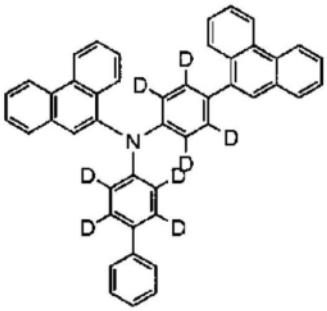
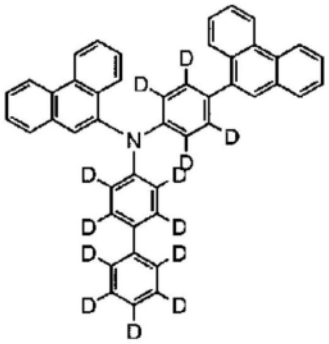
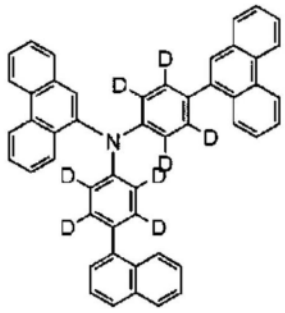
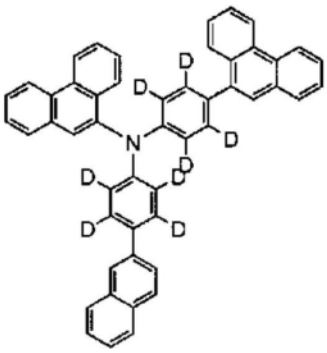
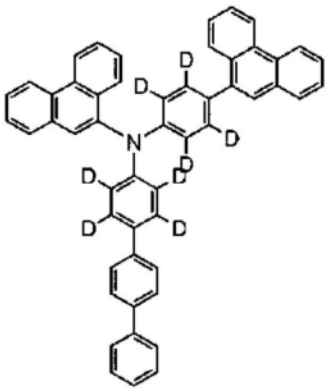
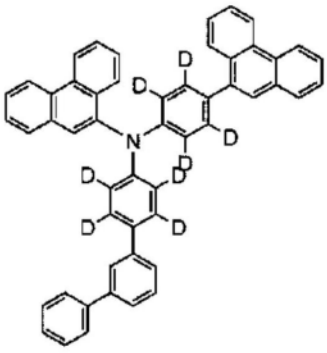
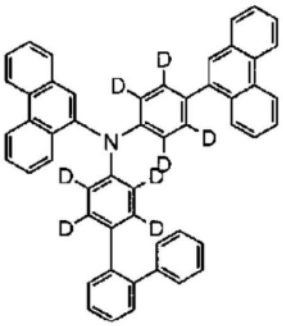


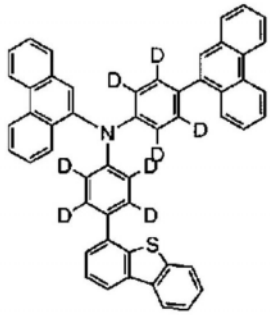
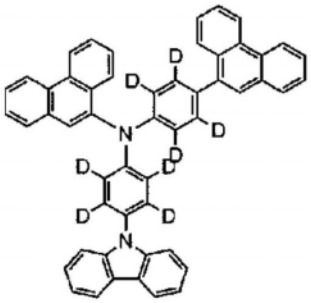
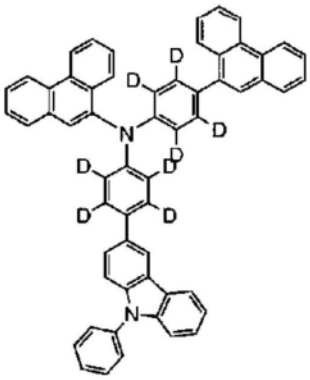
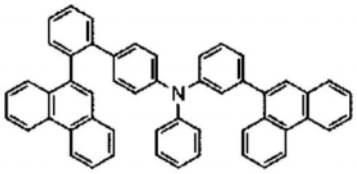
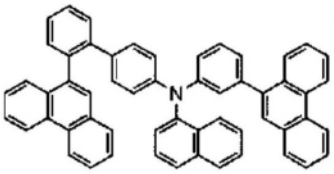
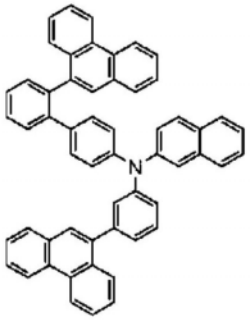
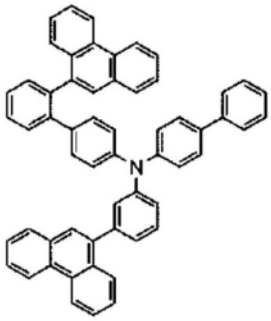
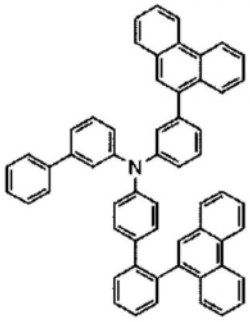
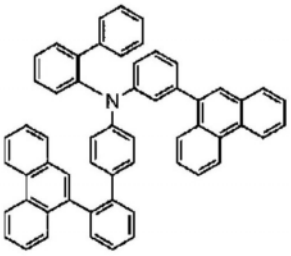
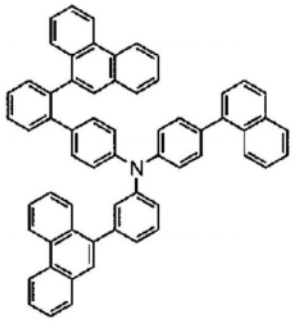
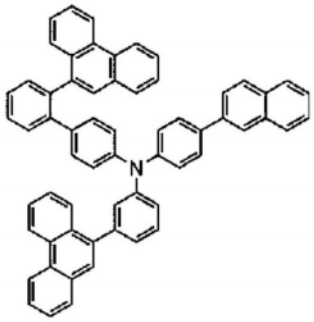


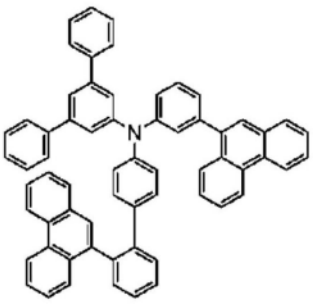
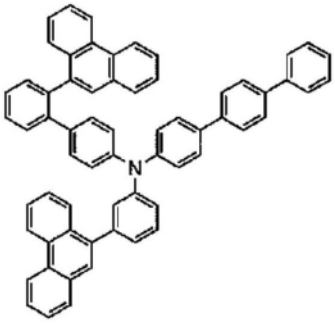
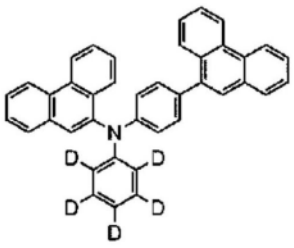
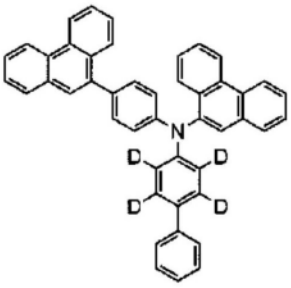
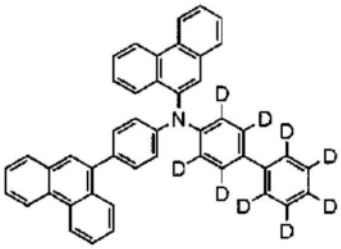
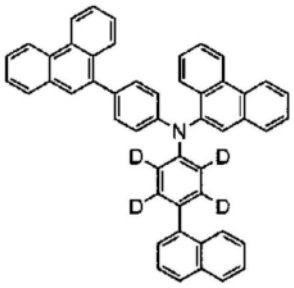
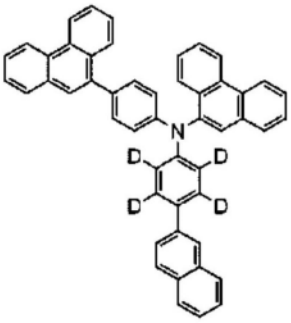
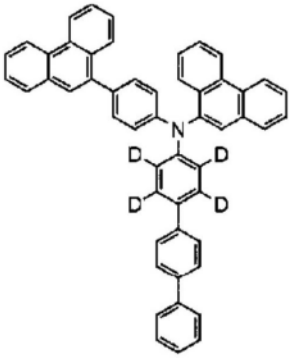
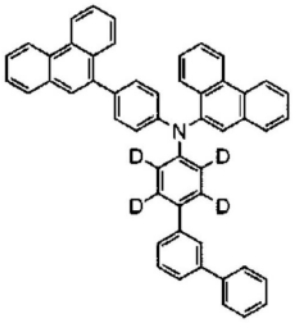
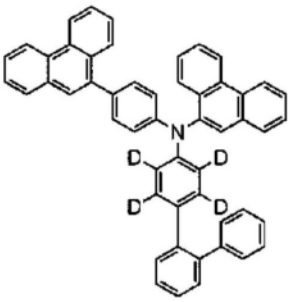


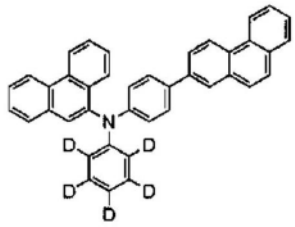
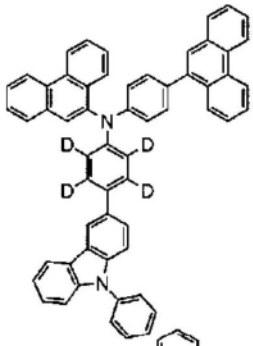
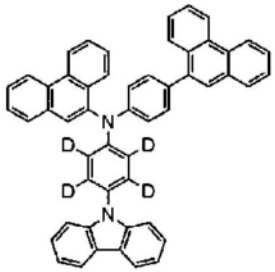
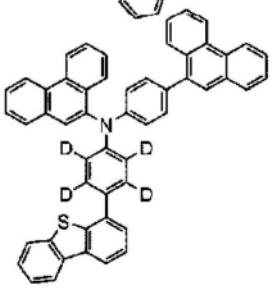
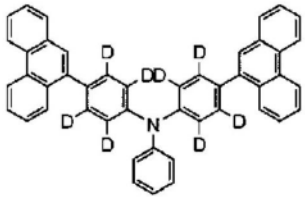
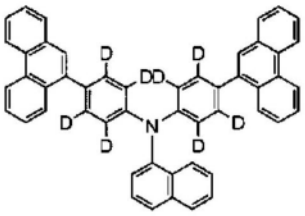
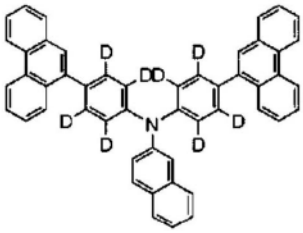
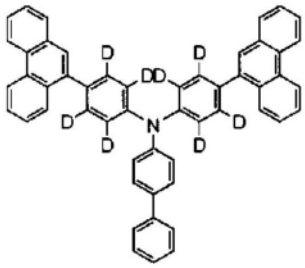
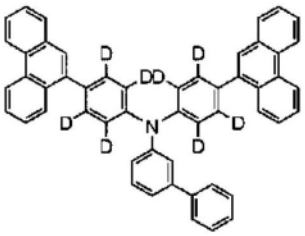
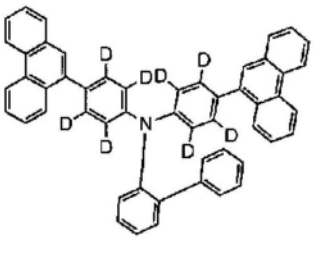
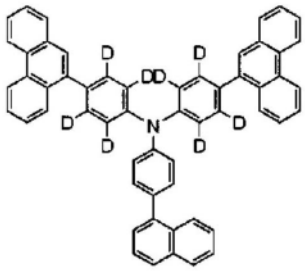


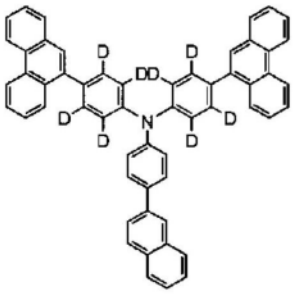
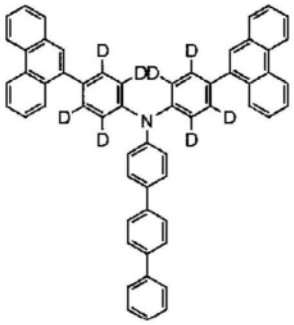
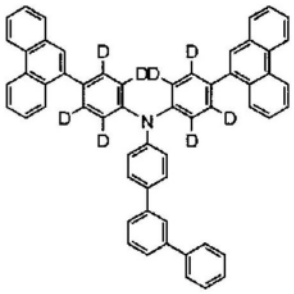
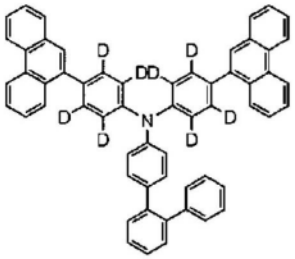
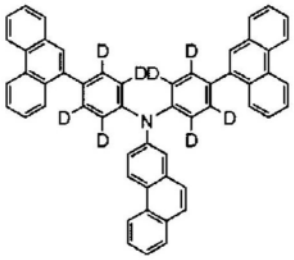
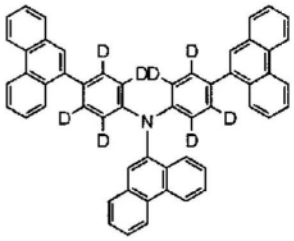
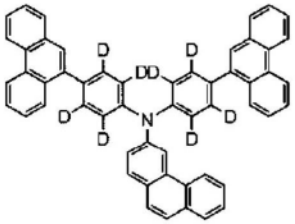
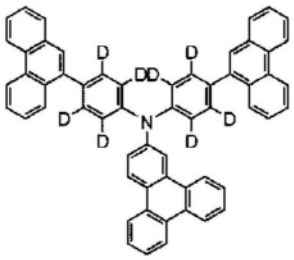
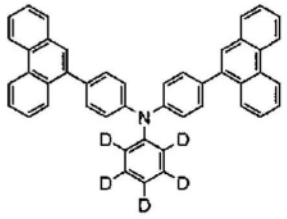
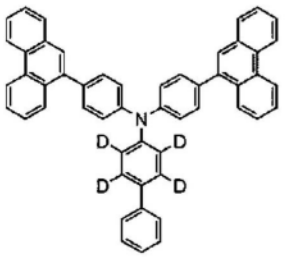
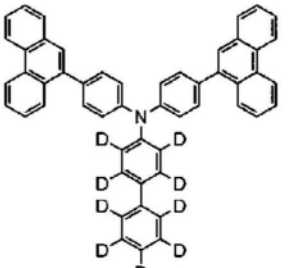
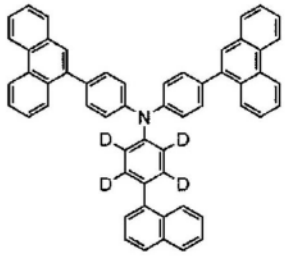


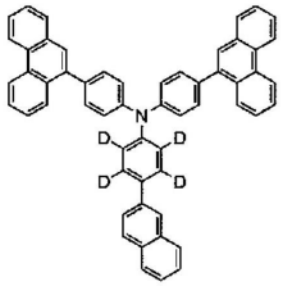
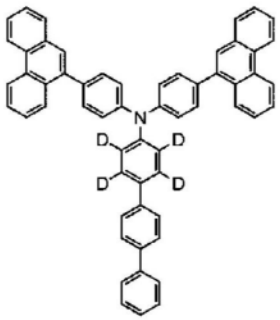
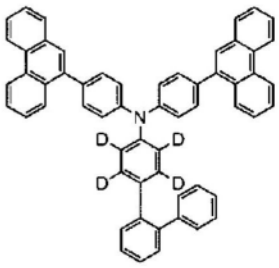
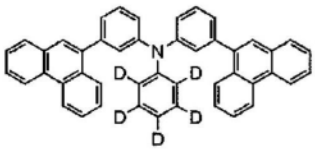
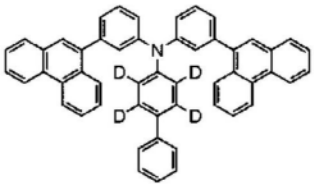
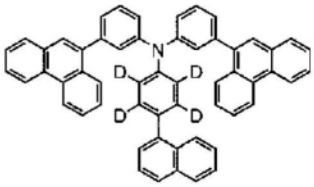
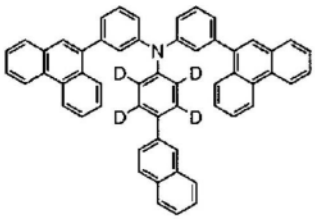
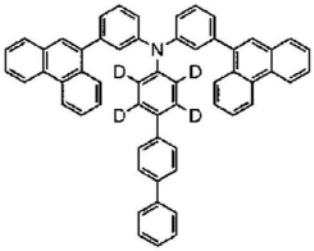
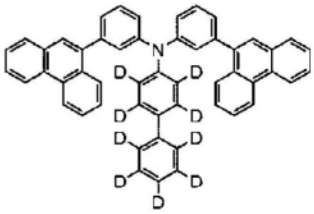
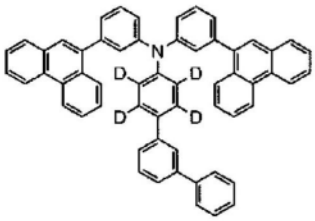
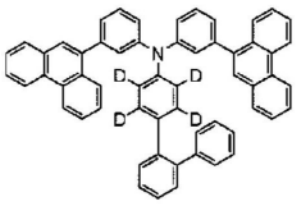
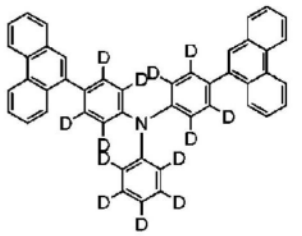











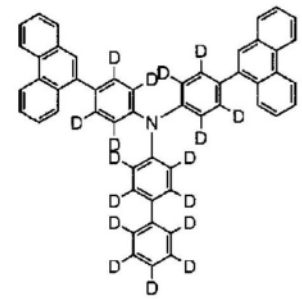
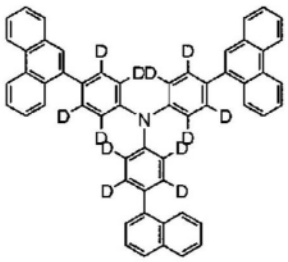
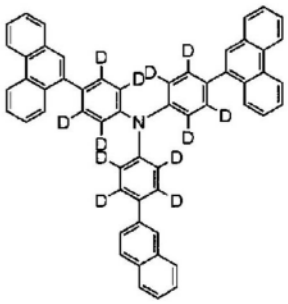
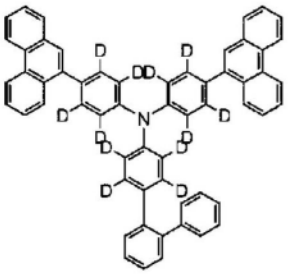
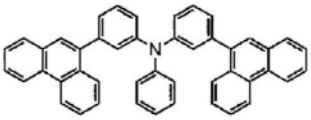
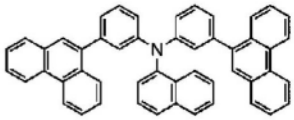
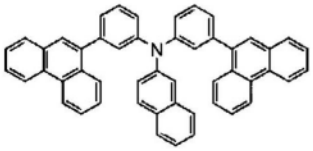
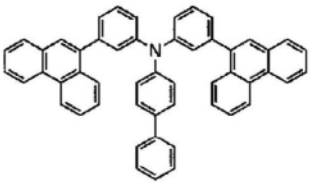
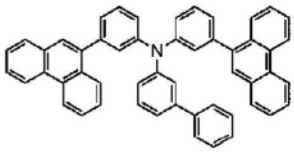
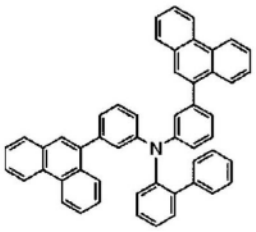
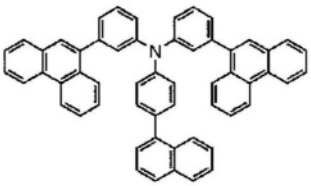
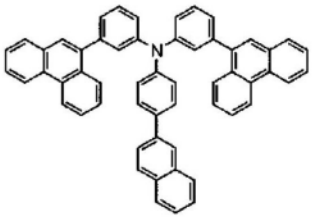












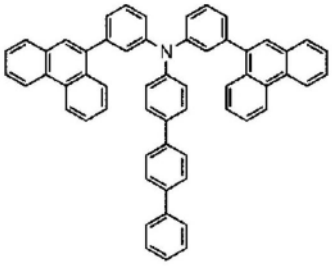
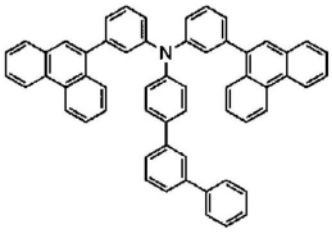
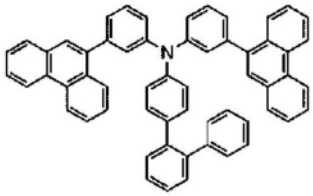
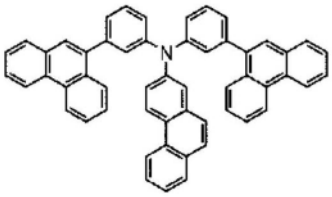
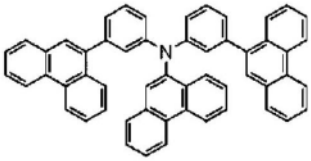
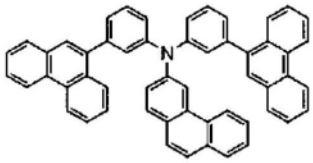
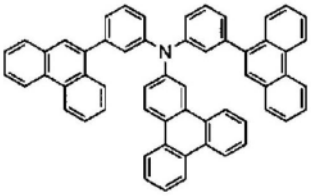
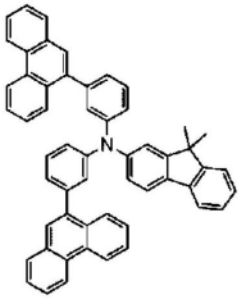
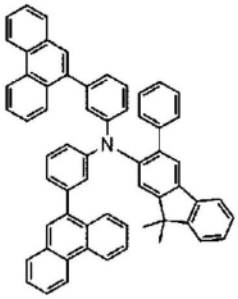
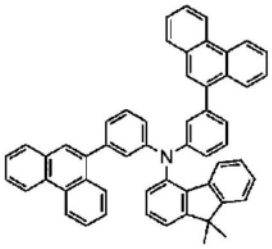











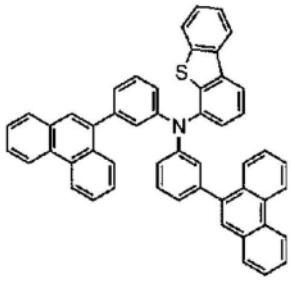
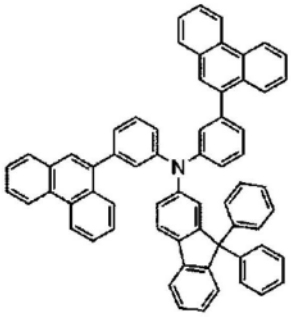
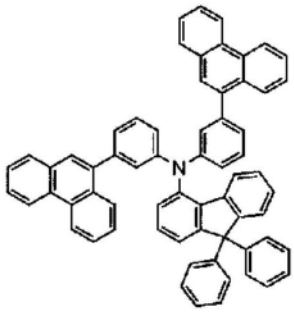
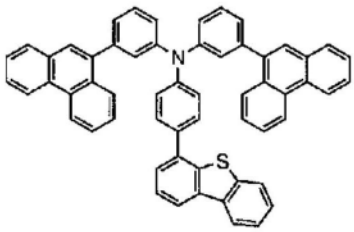
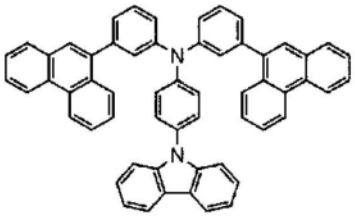
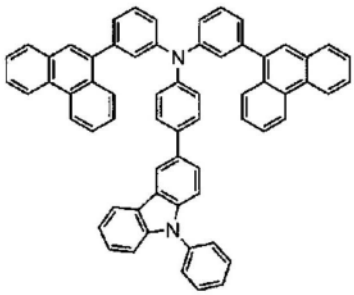
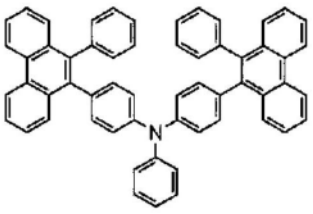
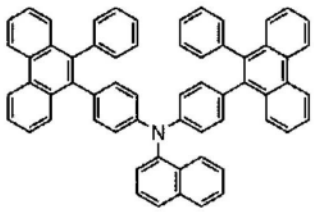
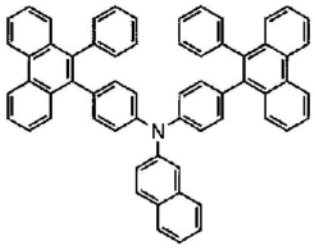












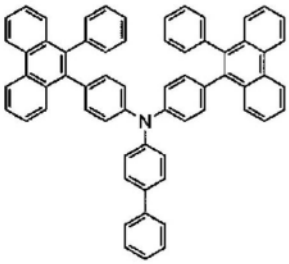
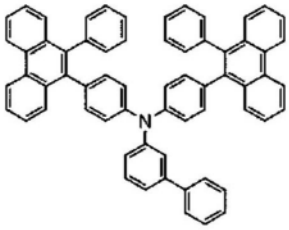
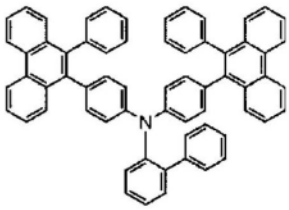
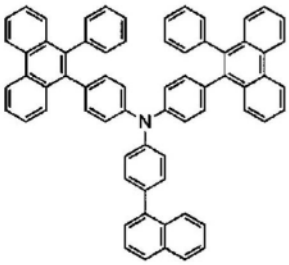
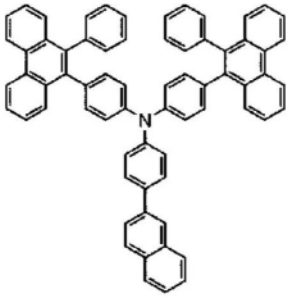
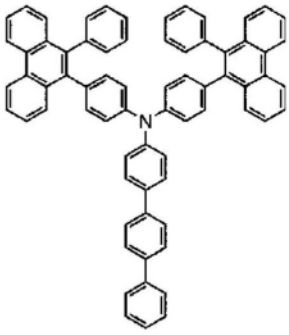
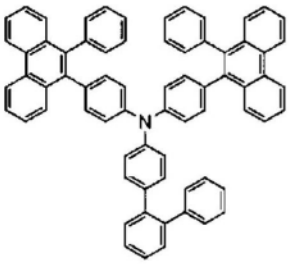
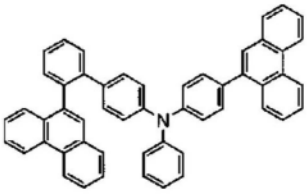
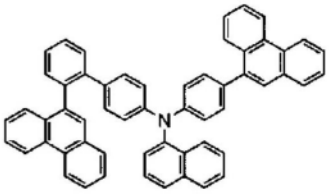
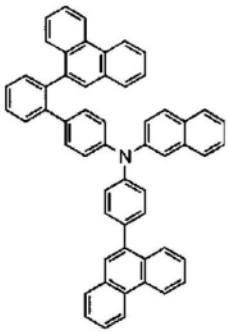
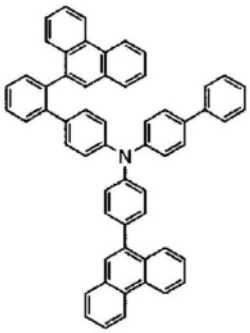
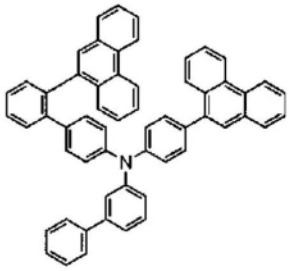













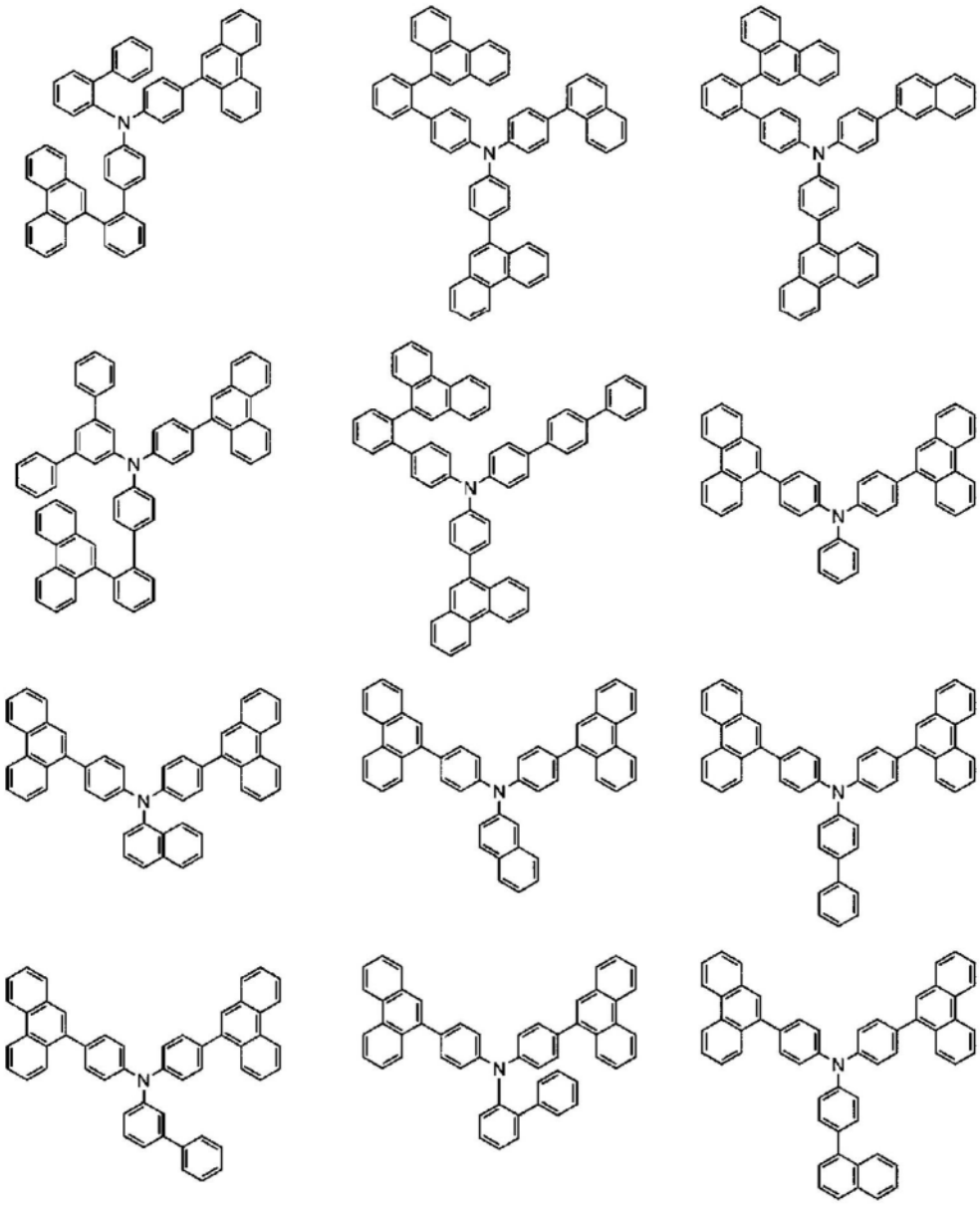













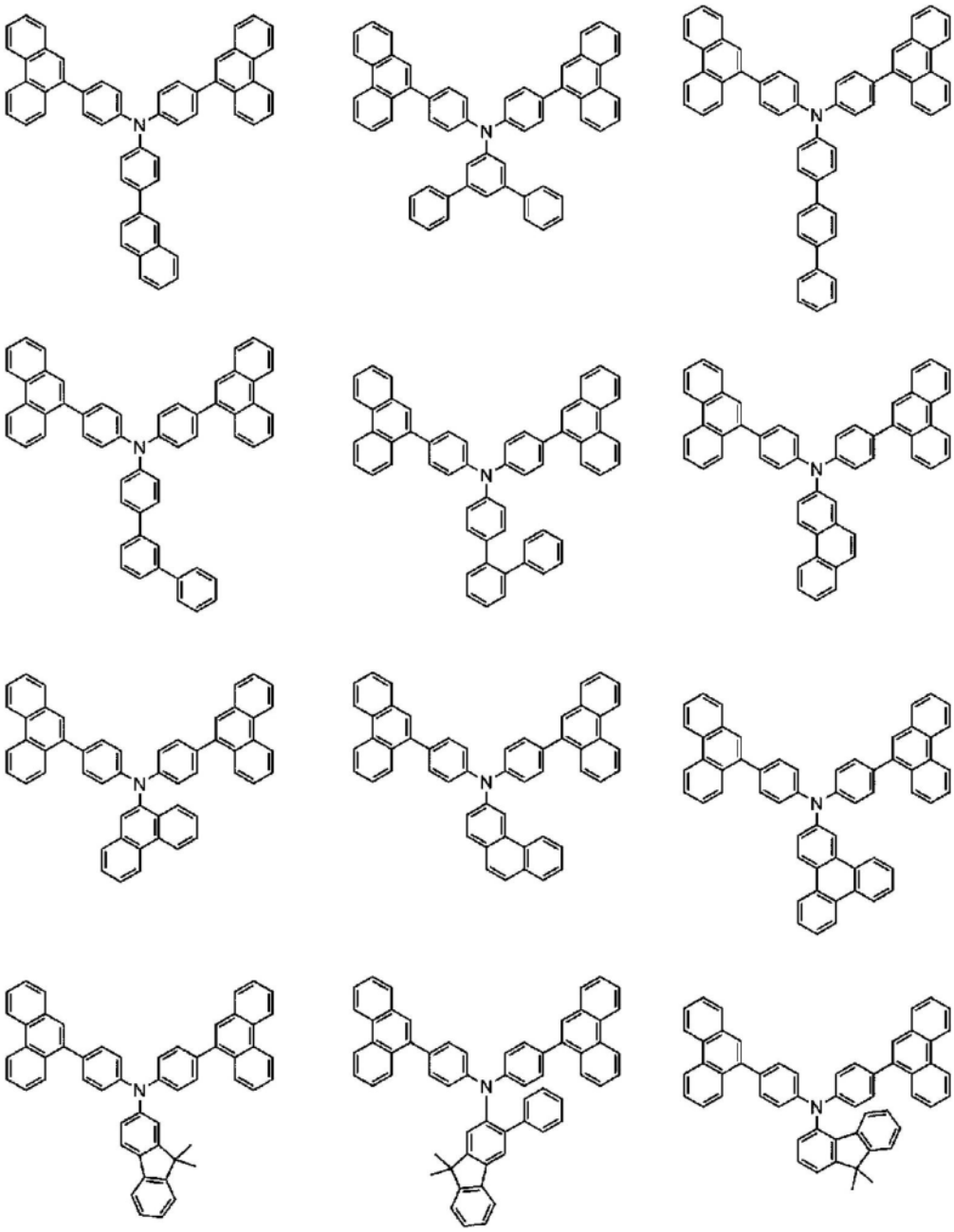













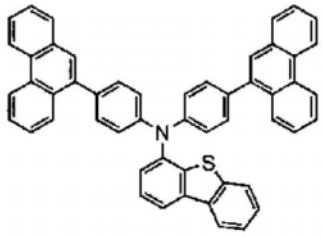











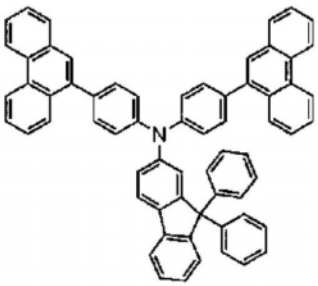










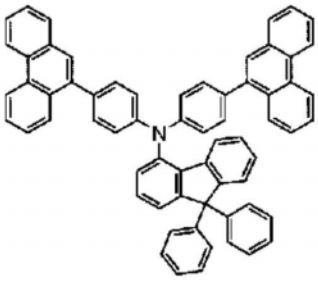













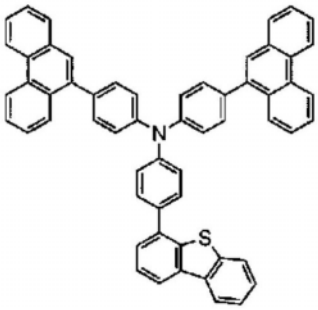


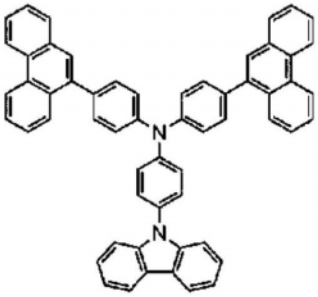


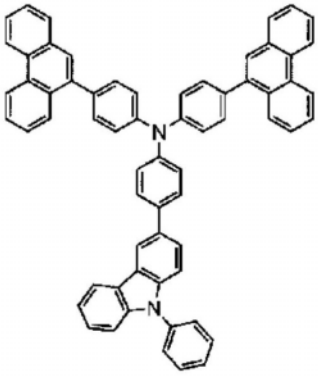


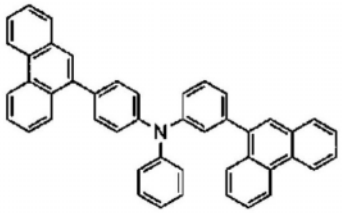


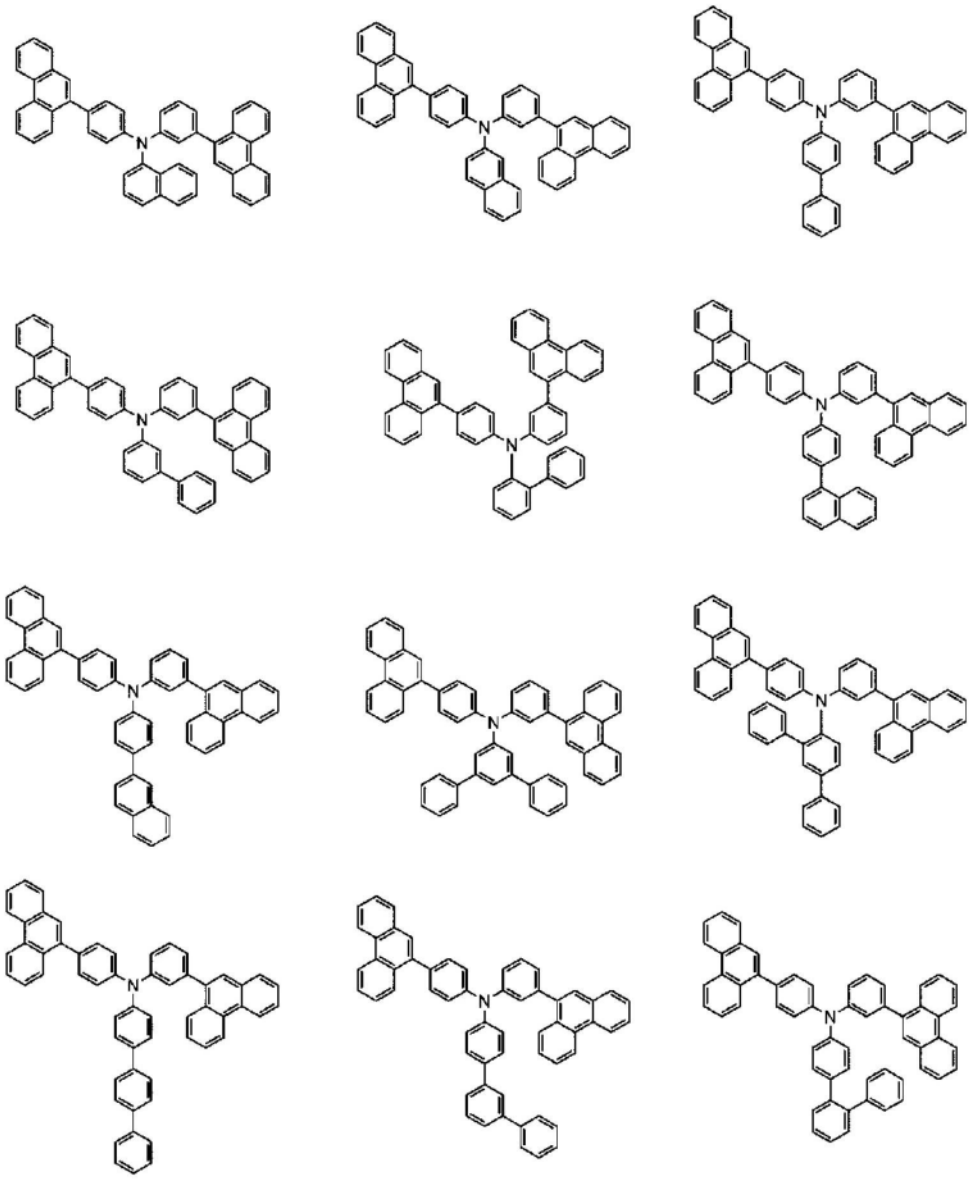


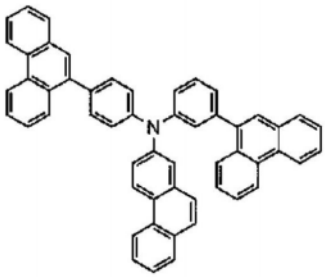
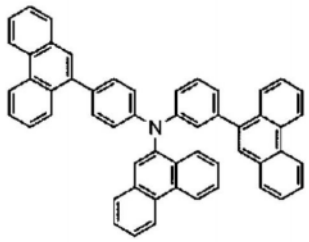
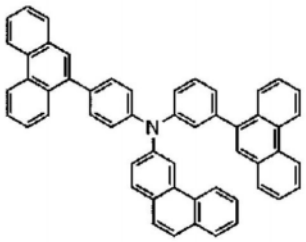
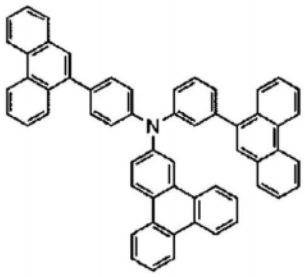
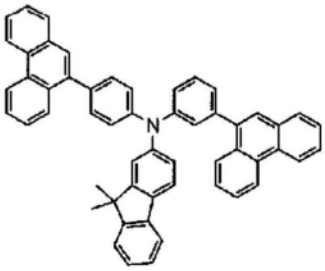
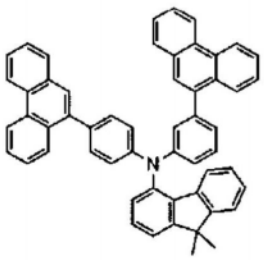
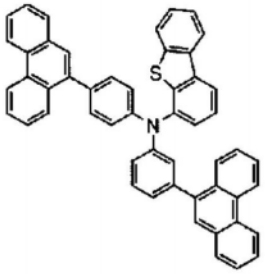
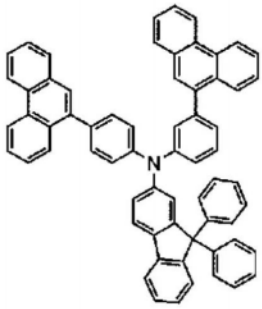
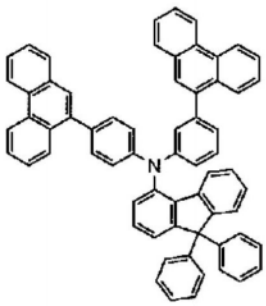


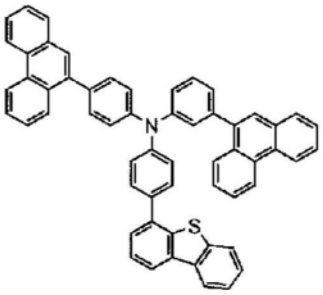


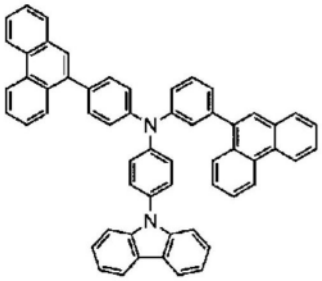


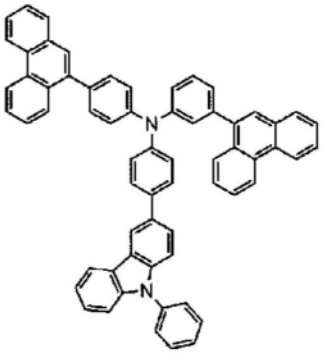


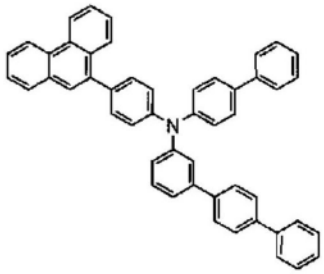


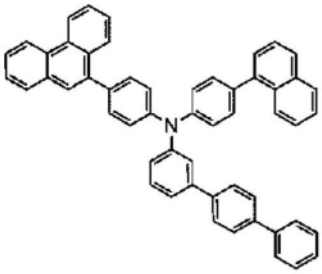










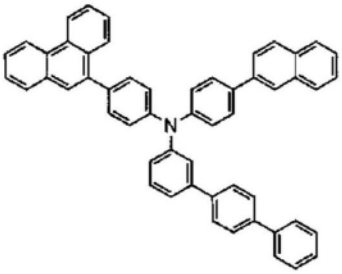


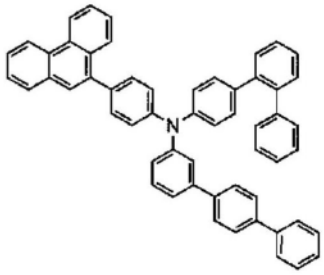


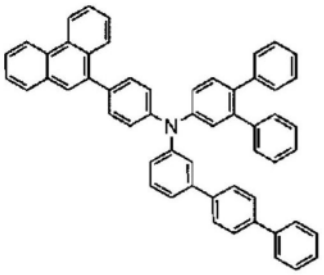


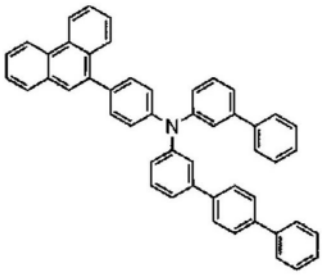


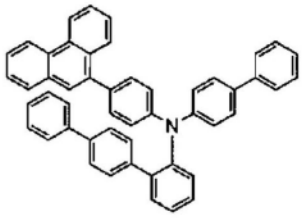


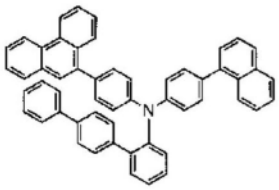
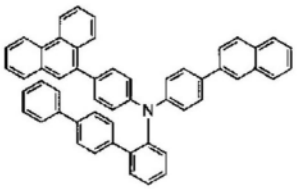
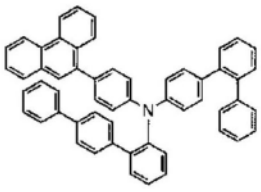
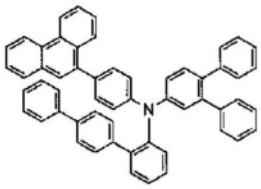
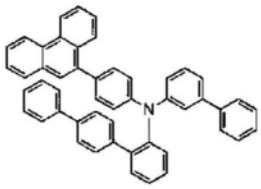
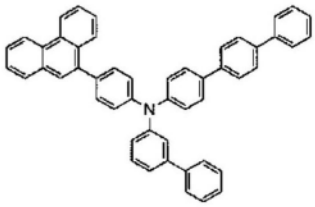
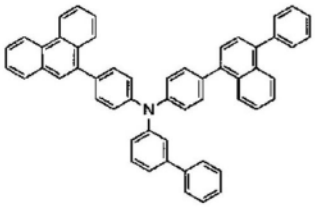
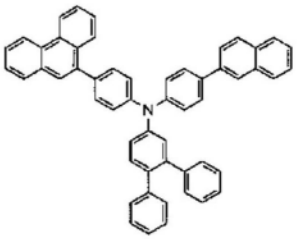
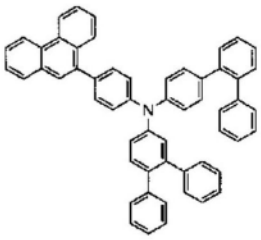
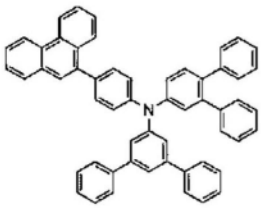
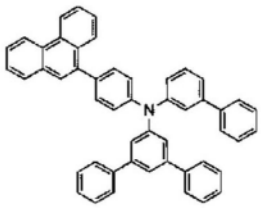
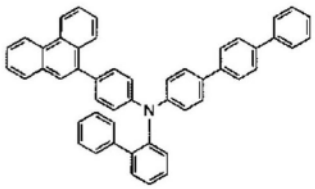
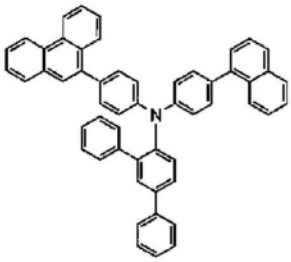
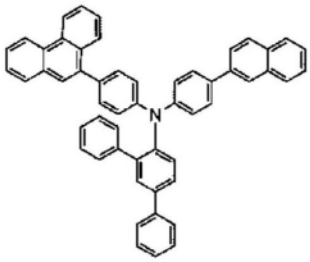
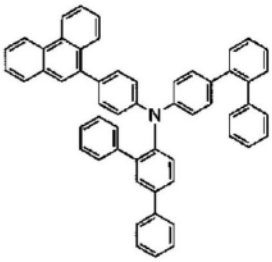
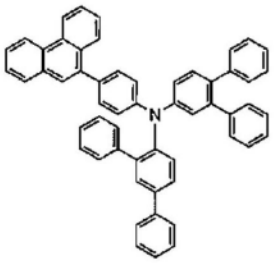
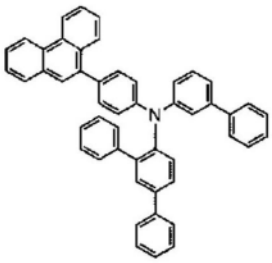


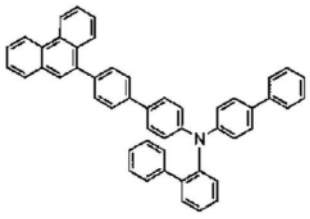


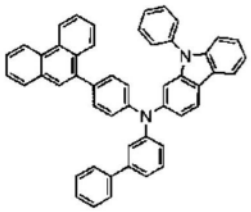


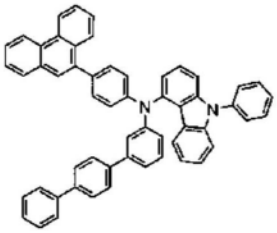


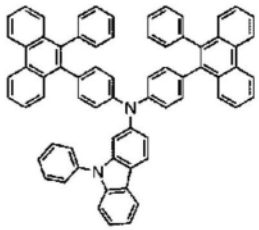


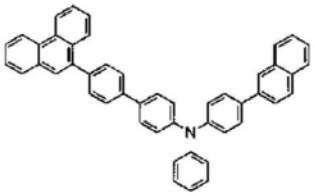


















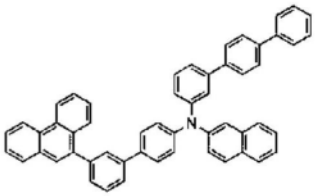


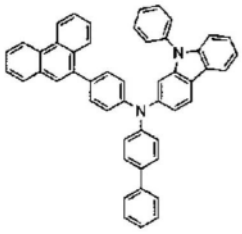


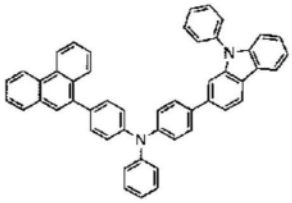


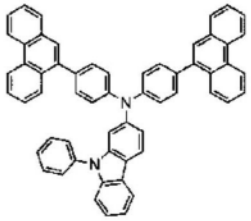


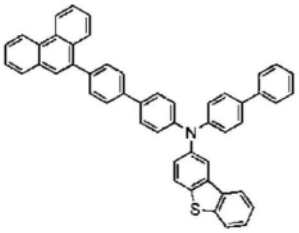


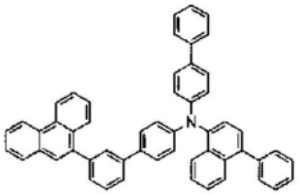


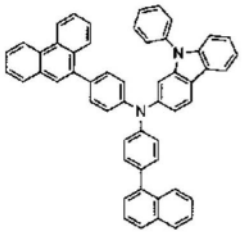


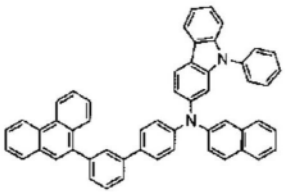


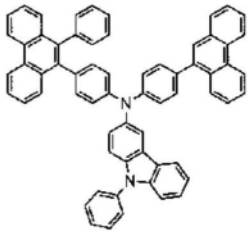


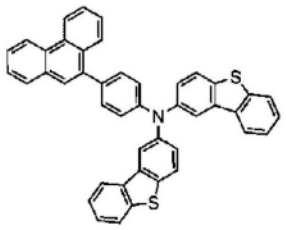
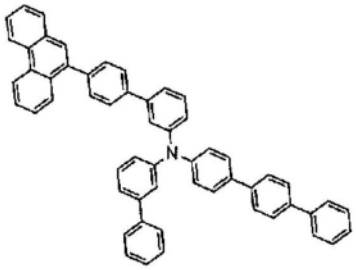
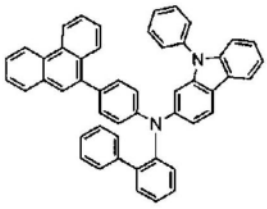
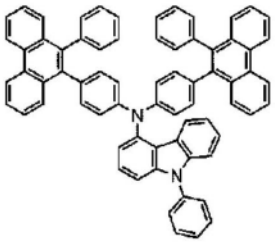
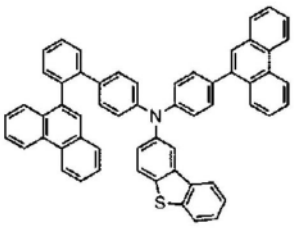
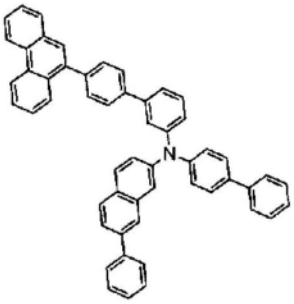
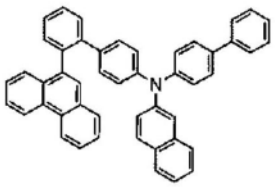
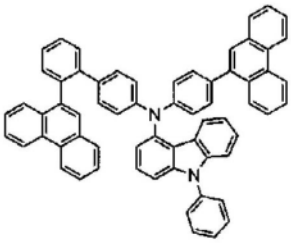
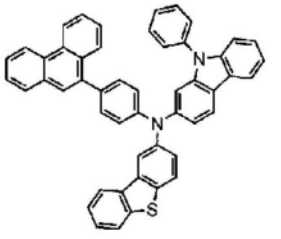
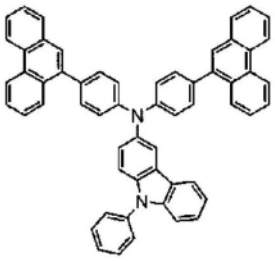


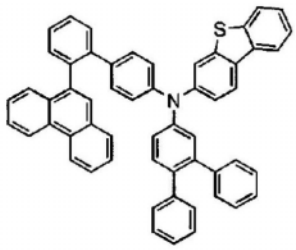


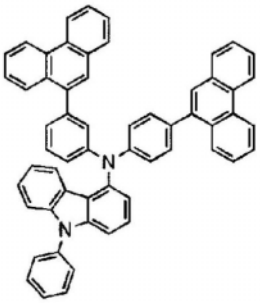


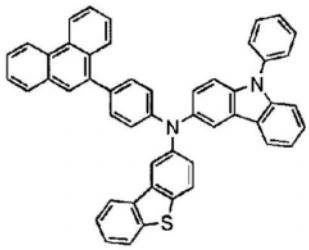










有机发光器件

技术领域

[0001] 本公开内容涉及具有改善的驱动电压、效率和寿命的有机发光器件。

背景技术

[0002] 通常,有机发光现象是指通过利用有机材料将电能转化成光能的现象。利用有机发光现象的有机发光器件具有诸如宽的视角,优异的对比度,快速的响应时间,优异的亮度、驱动电压和响应速度的特性,因此已进行了许多研究。

[0003] 有机发光器件通常具有包括阳极、阴极以及设置在阳极与阴极之间的有机材料层的结构。有机材料层通常具有包含不同材料的多层结构以提高有机发光器件的效率和稳定性,例如,有机材料层可以由空穴注入层、空穴传输层、发光层、电子传输层、电子注入层等形成。在有机发光器件的结构中,如果在两个电极之间施加电压,则空穴从阳极注入至有机材料层中并且电子从阴极注入至有机材料层中,并且当注入的空穴和电子彼此相遇时形成激子,并且当激子再次落至基态时发光。

[0004] 在如上所述的有机发光器件中,持续需要开发具有改善的驱动电压、效率和寿命的有机发光器件。

[0005] 现有技术文献

[0006] 专利文献

[0007] (专利文献1)韩国未审查专利公开第10-2000-0051826号

发明内容

[0008] 技术问题

[0009] 本公开内容涉及具有改善的驱动电压、效率和寿命的有机发光器件。

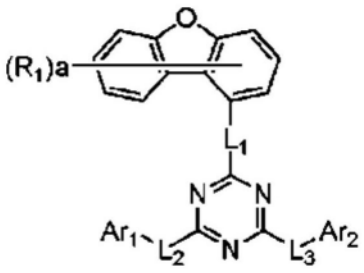
[0010] 技术方案

[0011] 在本文中提供了以下有机发光器件:

[0012] 有机发光器件包括:阳极、阴极以及设置在阳极与阴极之间的发光层,

[0013] 其中发光层包含由以下化学式1表示的化合物和由以下化学式2表示的化合物。

[0014] [化学式1]

[0015] 

[0016] 其中在化学式1中,

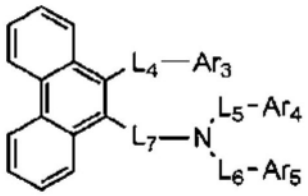
[0017] Ar_1和Ar_2各自独立地为经取代或未经取代的C_{6-60}芳基;或者经取代或未经取代的包含选自N、O和S中的一者或更多者的C_{2-60}杂芳基,

[0018] L_1至L_3各自独立地为单键、或者经取代或未经取代的C_{6-60}亚芳基，

[0019] R_1为氢；氘；经取代或未经取代的C_{6-60}芳基；或者经取代或未经取代的包含选自N、O和S中的一者或更多者的C_{2-60}杂芳基，

[0020] a为0至7的整数，

[0021] [化学式2]

[0022] 

[0023] 其中在化学式2中，

[0024] Ar_3为氢；经取代或未经取代的C_{6-60}芳基；或者经取代或未经取代的包含选自N、O和S中的一者或更多者的C_{2-60}杂芳基，

[0025] Ar_4和Ar_5各自独立地为经取代或未经取代的C_{6-60}芳基；或者经取代或未经取代的包含选自N、O和S中的一者或更多者的C_{2-60}杂芳基，

[0026] L_4至L_6各自独立地为单键；经取代或未经取代的C_{6-60}亚芳基；或者经取代或未经取代的包含选自N、O和S中的一者或更多者的C_{2-60}亚杂芳基，以及

[0027] L_7为经取代或未经取代的C_{6-60}亚芳基。

[0028] 有益效果

[0029] 上述有机发光器件可以通过在发光层中包含由化学式1表示的化合物和由化学式2表示的化合物而改善效率、实现低驱动电压和/或改善寿命特性。

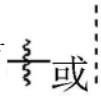
附图说明

[0030] 图1示出了包括基底1、阳极2、发光层3和阴极4的有机发光器件的一个实例。

[0031] 图2示出了包括基底1、阳极2、空穴注入层5、空穴传输层6、电子阻挡层7、发光层3、空穴阻挡层8、电子注入和传输层9以及阴极4的有机发光器件的一个实例。

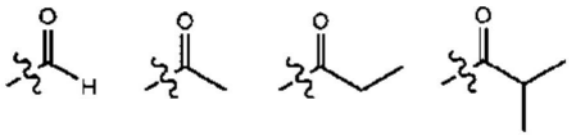
具体实施方式

[0032] 在下文中，将更详细地描述本公开内容的实施方案以便于理解本发明。

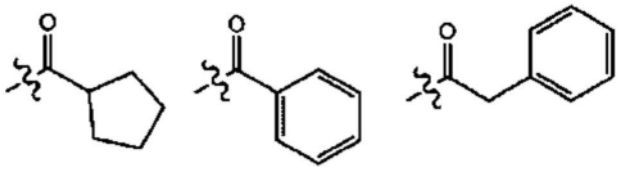
[0033] 如本文中所使用，符号  意指与另一个取代基连接的键。

[0034] 如本文中所使用，术语“经取代或未经取代的”意指未经取代或经选自以下中的一个或更多个取代基取代：氘；卤素基团；腈基；硝基；羟基；羰基；酯基；酰亚胺基；氨基；氧化膦基；烷氧基；芳氧基；烷基硫基；芳基硫基；烷基磺酰基；芳基磺酰基；甲硅烷基；硼基；烷基；环烷基；烯基；芳基；芳烷基；芳烯基；烷基芳基；烷基胺基；芳烷基胺基；杂芳基胺基；芳基胺基；芳基膦基；或者包含N、O和S原子中的至少一者的杂芳基，或者未经取代或经以上例示的取代基中的两个或更多个取代基连接的取代基取代。例如，“两个或更多个取代基连接的取代基”可以为联苯基。即，联苯基可以为芳基，或者其也可以被解释为两个苯基连接的取代基。

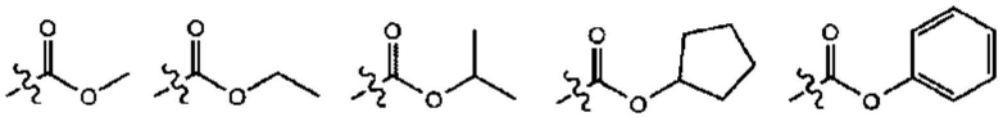
[0035] 在本公开内容中,羰基的碳数没有特别限制,但优选为1至40。具体地,羰基可以为具有以下结构式的基团,但不限于此。



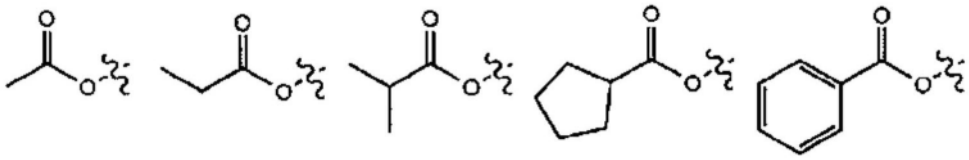
[0036]



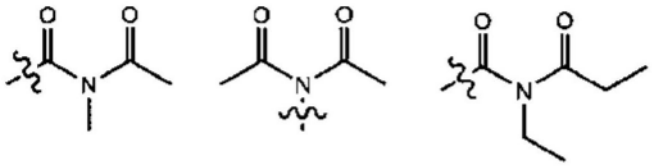
[0037] 在本公开内容中,酯基可以具有其中酯基的氧可以经具有1至25个碳原子的直链、支链或环状烷基,或者具有6至25个碳原子的芳基取代的结构。具体地,酯基可以为具有以下结构式的基团,但不限于此。



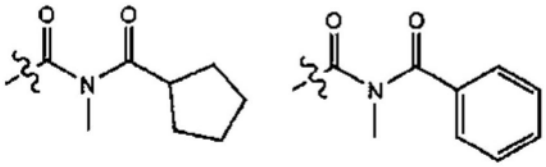
[0038]



[0039] 在本公开内容中,酰亚胺基的碳数没有特别限制,但优选为1至25。具体地,酰亚胺基可以为具有以下结构式的基团,但不限于此。



[0040]



[0041] 在本公开内容中,甲硅烷基具体地包括三甲基甲硅烷基、三乙基甲硅烷基、叔丁基二甲基甲硅烷基、乙烯基二甲基甲硅烷基、丙基二甲基甲硅烷基、三苯基甲硅烷基、二苯基甲硅烷基、苯基甲硅烷基等,但不限于此。

[0042] 在本公开内容中,硼基具体地包括三甲基硼基、三乙基硼基、叔丁基二甲基硼基、三苯基硼基和苯基硼基,但不限于此。

[0043] 在本公开内容中,卤素基团的实例包括氟、氯、溴或碘。

[0044] 在本公开内容中,烷基可以为直链或支链的,并且其碳数没有特别限制,但优选为1至40。根据一个实施方案,烷基的碳数为1至20。根据另一个实施方案,烷基的碳数为1至10。根据另一个实施方案,烷基的碳数为1至6。烷基的具体实例包括甲基、乙基、丙基、正丙基、异丙基、丁基、正丁基、异丁基、叔丁基、仲丁基、1-甲基-丁基、1-乙基-丁基、戊基、正戊基、异戊基、新戊基、叔戊基、己基、正己基、1-甲基戊基、2-甲基戊基、4-甲基-2-戊基、3,3-

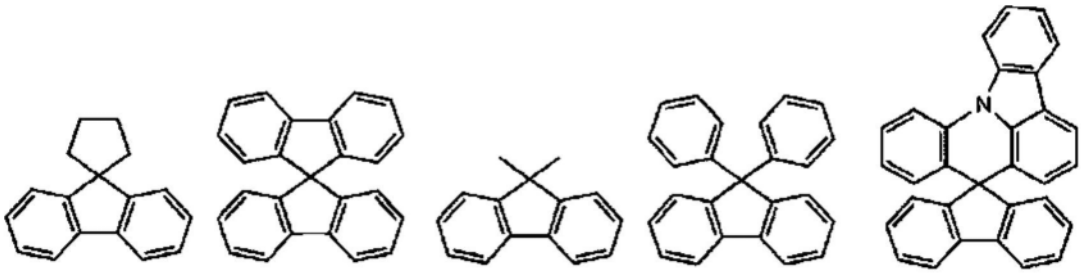
二甲基丁基、2-乙基丁基、庚基、正庚基、1-甲基己基、环戊基甲基、环己基甲基、辛基、正辛基、叔辛基、1-甲基庚基、2-乙基己基、2-丙基戊基、正壬基、2,2-二甲基庚基、1-乙基-丙基、1,1-二甲基-丙基、异己基、2-甲基戊基、4-甲基己基、5-甲基己基等,但不限于此。

[0045] 在本公开内容中,烯基可以为直链或支链的,并且其碳数没有特别限制,但优选为2至40。根据一个实施方案,烯基的碳数为2至20。根据另一个实施方案,烯基的碳数为2至10。根据又一个实施方案,烯基的碳数为2至6。其具体实例包括乙烯基、1-丙烯基、异丙烯基、1-丁烯基、2-丁烯基、3-丁烯基、1-戊烯基、2-戊烯基、3-戊烯基、3-甲基-1-丁烯基、1,3-丁二烯基、烯丙基、1-苯基乙烯基-1-基、2-苯基乙烯基-1-基、2,2-二苯基乙烯基-1-基、2-苯基-2-(萘基-1-基)乙烯基-1-基、2,2-双(二苯基-1-基)乙烯基-1-基、苊基、苯乙烯基等,但不限于此。

[0046] 在本公开内容中,环烷基没有特别限制,但其碳数优选为3至60。根据一个实施方案,环烷基的碳数为3至30。根据另一个实施方案,环烷基的碳数为3至20。根据又一个实施方案,环烷基的碳数为3至6。其具体实例包括环丙基、环丁基、环戊基、3-甲基环戊基、2,3-二甲基环戊基、环己基、3-甲基环己基、4-甲基环己基、2,3-二甲基环己基、3,4,5-三甲基环己基、4-叔丁基环己基、环庚基、环辛基等,但不限于此。

[0047] 在本公开内容中,芳基没有特别地限制,但其碳数优选为6至60,并且其可以为单环芳基或多环芳基。根据一个实施方案,芳基的碳数为6至30。根据一个实施方案,芳基的碳数为6至20。芳基可以为作为单环芳基的苯基、联苯基、三联苯基等,但不限于此。多环芳基包括萘基、蒽基、菲基、芘基、䓛基、䓛基等,但不限于此。

[0048] 在本公开内容中,芴基可以为经取代的,并且两个取代基可以彼此连接以形成螺环结构。在芴基为经取代的情况下,可以形成



等。然而,结构不限于此。

[0049] 在本公开内容中,杂环基为包含O、N、Si和S中的一者或更多者作为杂原子的杂环基,并且其碳数没有特别限制,但优选为2至60。杂环基的实例包括噻吩基、呋喃基、吡咯基、咪唑基、噻唑基、噁唑基、噁二唑基、三唑基、吡啶基、联吡啶基、嘧啶基、三嗪基、吖啶基、哒嗪基、吡嗪基、喹啉基、喹唑啉基、喹喔啉基、酞嗪基、吡啶并嘧啶基、吡啶并吡嗪基、吡嗪并吡嗪基、异喹啉基、吲哚基、咔唑基、苯并噁唑基、苯并咪唑基、苯并噻唑基、苯并咔唑基、苯并噻吩基、二苯并噻吩基、苯并呋喃基、菲咯啉基、异噁唑基、噻二唑基、吩噻嗪基、二苯并呋喃基等,但不限于此。

[0050] 在本公开内容中,芳烷基、芳烯基、烷基芳基和芳基胺基中的芳基与前述芳基的实例相同。在本公开内容中,芳烷基、烷基芳基和烷基胺基中的烷基与前述烷基的实例相同。在本公开内容中,杂芳基胺中的杂芳基可以应用前述杂环基的描述。在本公开内容中,芳烯

基中的烯基与前述烯基的实例相同。在本公开内容中,可以应用前述芳基的描述,不同之处在于亚芳基是二价基团。在本公开内容中,可以应用前述杂芳基的描述,不同之处在于亚杂芳基是二价基团。在本公开内容中,可以应用前述芳基或环烷基的描述,不同之处在于烃环不是一价基团而是通过使两个取代基结合而形成的。在本公开内容中,可以应用前述杂环基的描述,不同之处在于杂环不是一价基团而是通过使两个取代基结合而形成的。

[0051] 在下文中,将针对各配置详细地描述本公开内容。

[0052] 阳极和阴极

[0053] 本公开内容中使用的阳极和阴极意指有机发光器件中使用的电极。

[0054] 作为阳极材料,通常优选使用具有大功函数的材料,使得空穴可以顺利地注入到有机材料层中。阳极材料的具体实例包括:金属,例如钒、铬、铜、锌、和金,或其合金;金属氧化物,例如氧化锌、氧化铟、氧化铟锡(ITO)和氧化铟锌(IZO);金属和氧化物的组合,例如ZnO:Al或SnO_2:Sb;导电聚合物,例如聚(3-甲基噻吩)、聚[3,4-(亚乙基-1,2-二氧基)噻吩](PEDOT)、聚吡咯和聚苯胺;等等,但不限于此。

[0055] 作为阴极材料,通常优选使用具有小功函数的材料,使得电子可以容易地注入到有机材料层中。阴极材料的具体实例包括:金属,例如镁、钙、钠、钾、钛、铟、钇、锂、钆、铝、银、锡和铅,或其合金;多层结构材料,例如LiF/Al或LiO_2/Al;等等,但不限于此。

[0056] 空穴注入层

[0057] 如有必要,根据本公开内容的有机发光器件还可以包括在阳极上的空穴注入层。

[0058] 空穴注入层是注入来自电极的空穴的层,并且空穴注入材料优选为这样的化合物:其具有传输空穴的能力、在阳极中的空穴注入效应和优异的对发光层或发光材料的空穴注入效应,防止发光层中产生的激子移动至电子注入层或电子注入材料,并且具有优异的薄膜形成能力。优选的是,空穴注入材料的HOMO(最高占据分子轨道)在阳极材料的功函数与周围有机材料层的HOMO之间。

[0059] 空穴注入材料的具体实例包括金属卟啉,低聚噻吩,基于芳基胺的有机材料,基于六腈六氮杂苯并菲的有机材料,基于喹吖啶酮的有机材料,基于 的有机材料,基于蒽醌、聚苯胺和聚噻吩的导电聚合物等,但不限于此。

[0060] 空穴传输层

[0061] 如果必要,根据本公开内容的有机发光器件可以包括在阳极上(或者当存在空穴注入层时,在空穴注入层上)的空穴传输层。

[0062] 空穴传输层为接收来自阳极或空穴注入层的空穴并将空穴传输至发光层的层。空穴传输材料适当地为具有大的空穴迁移率的材料,其可以接收来自阳极或空穴注入层的空穴并将空穴传输至发光层。

[0063] 空穴传输材料的具体实例包括基于芳基胺的有机材料、导电聚合物、同时存在共轭部分和非共轭部分的嵌段共聚物等,但不限于此。

[0064] 电子阻挡层

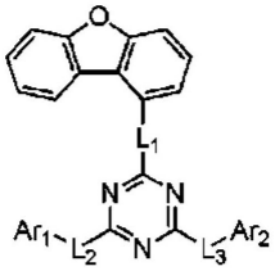
[0065] 电子阻挡层为设置在空穴传输层与发光层之间以防止注入阴极的电子被转移至空穴传输层而不在发光层中复合的层,其也可以称为电子抑制层或电子阻止层。电子阻挡层优选为具有比电子传输层更小的电子亲和势的材料。

[0066] 发光层

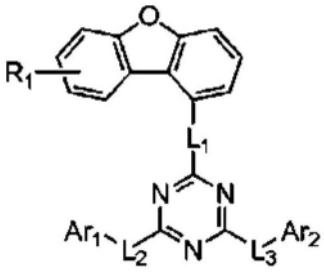
[0067] 本公开内容中使用的发光层意指可以通过使从阳极和阴极传输的空穴和电子结合而发出可见光区域中的光的层。通常,发光层包含主体材料和掺杂剂材料,并且在本公开内容中,包含由化学式1表示的化合物和由化学式2表示的化合物作为主体。

[0068] 优选地,由化学式1表示的化合物可以由以下化学式1-1至1-3中的任一者表示:

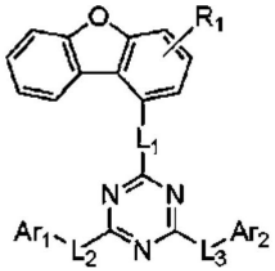
[0069] [化学式1-1]

[0070] 

[0071] [化学式1-2]

[0072] 

[0073] [化学式1-3]

[0074] 

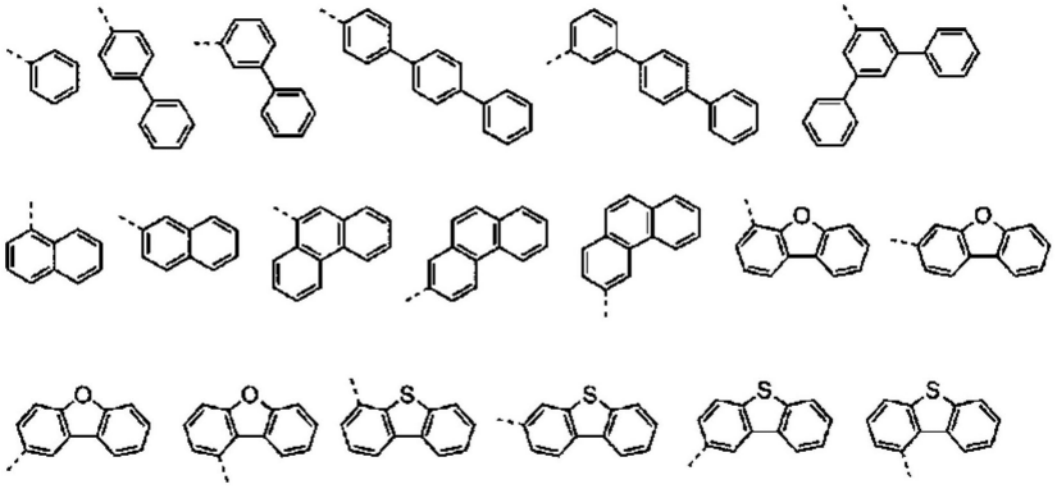
[0075] 其中在化学式1-1至1-3中,

[0076] Ar_1和Ar_2、L_1至L_3以及R_1如化学式1中所限定。

[0077] 优选地,Ar_1和Ar_2可以各自独立地为经取代或未经取代的C_{6-20}芳基;或者经取代或未经取代的包含选自N、O和S中的一者或更多者的C_{2-20}杂芳基,

[0078] 更优选地,Ar_1和Ar_2可以各自独立地为苯基、联苯基、三联苯基、萘基、菲基、二苯并呋喃基或二苯并噻吩基,以及

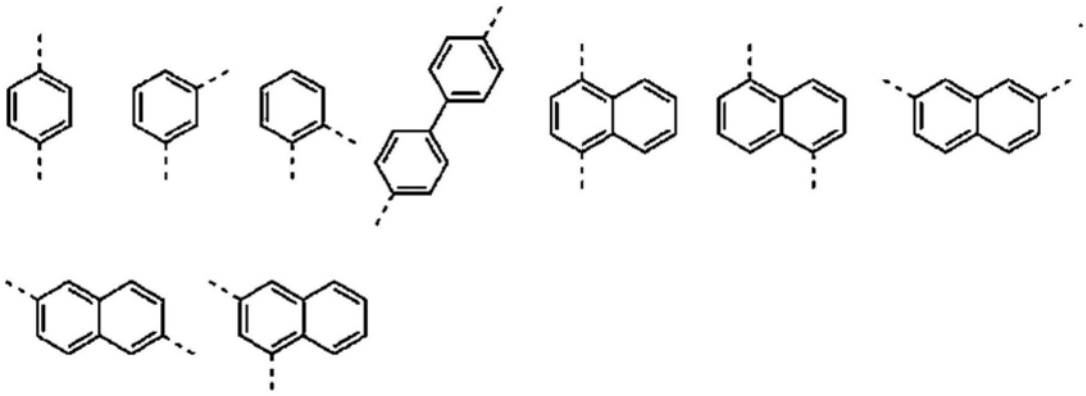
[0079] 最优选地,Ar_1和Ar_2可以各自独立地为选自以下中的任一者:

[0080] 

[0081] 优选地，L_1至L_3可以各自独立地为单键、或者经取代或未经取代的C_{6-20}亚芳基，

[0082] 更优选地，L_1至L_3可以各自独立地为单键、亚苯基、亚联苯基或亚萘基，以及

[0083] 最优选地，L_1至L_3可以各自独立地为单键或选自以下中的任一者：

[0084] 

[0085] 优选地，每个R_1可以独立地为氢；氘；经取代或未经取代的C_{6-20}芳基；或者经取代或未经取代的包含选自N、O和S中的一者或更多者的C_{2-20}杂芳基，以及

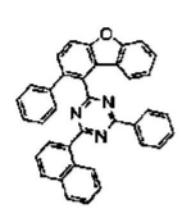
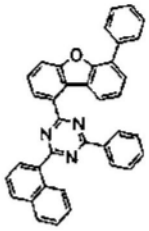
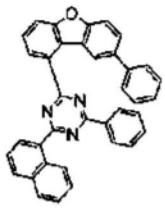
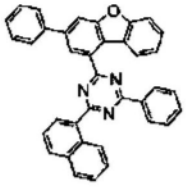
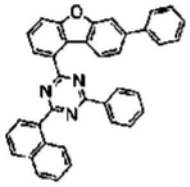
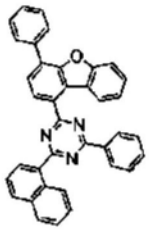
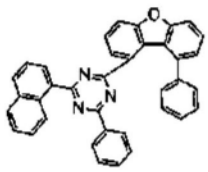
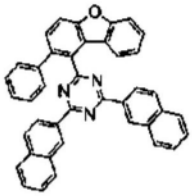
[0086] 更优选地，每个R_1可以独立地为氢、氘、苯基、联苯基、三联苯基、萘基、菲基、三亚苯基、萘基苯基、苯基萘基、荧蒽基、二氢茚基、二苯并呋喃基、二苯并噻吩基、苯并萘并呋喃基或苯并萘并噻吩基。

[0087] 优选地，a可以为0或1。更优选地，a可以为1。

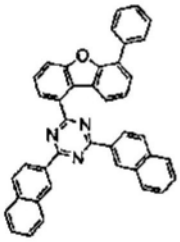
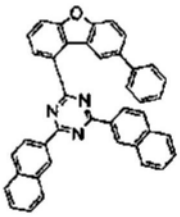
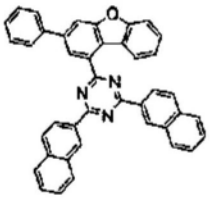
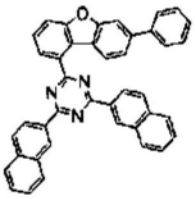
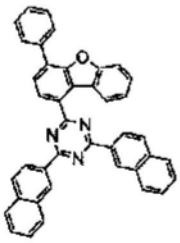
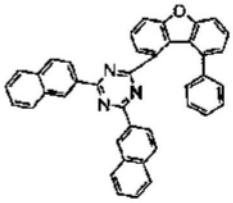
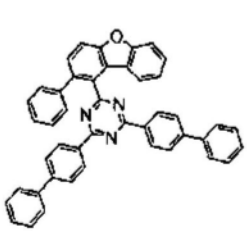
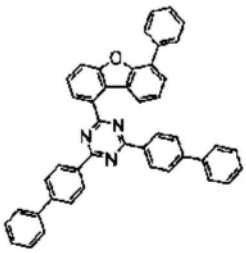
[0088] 优选地，Ar_1、Ar_2和R_1中的至少一者可以为萘基、苯基萘基、萘基苯基、菲基、荧蒽基、二苯并呋喃基、二苯并噻吩基、苯并萘并呋喃基或苯并萘并噻吩基。

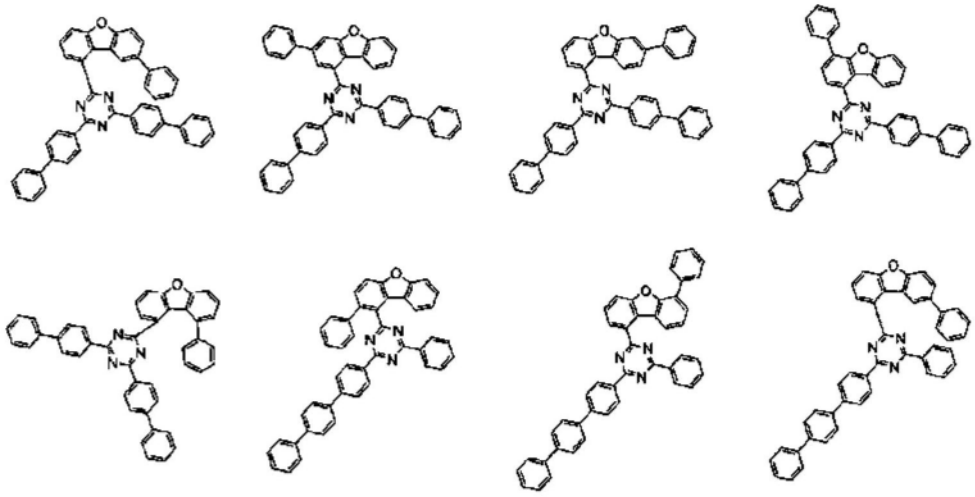
[0089] 更优选地，Ar_1、Ar_2和R_1中的至少一者可以为萘基、苯基萘基、萘基苯基、荧蒽基、二苯并呋喃基、苯并萘并呋喃基或苯并萘并噻吩基。

[0090] 由化学式1表示的化合物的代表性实例如下：

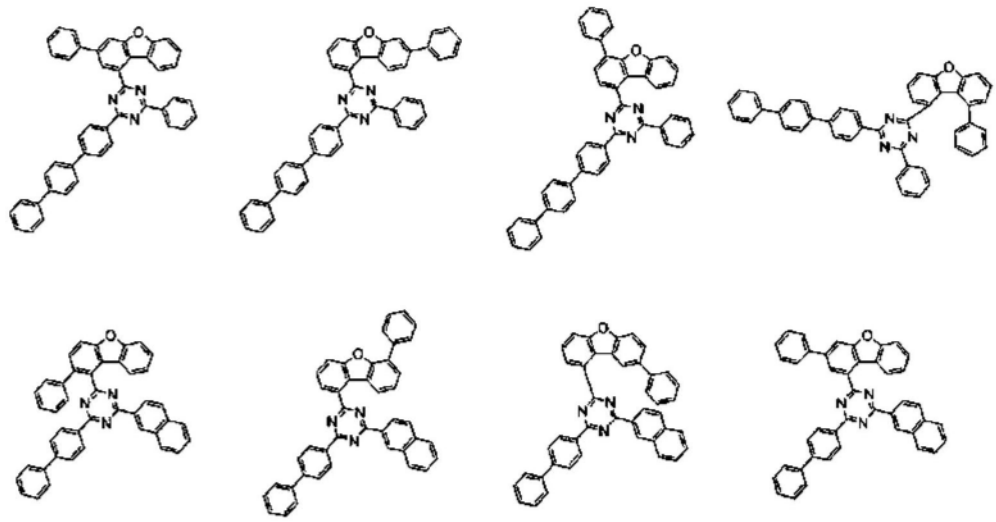









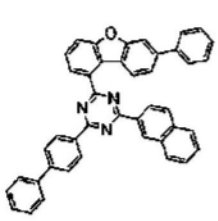
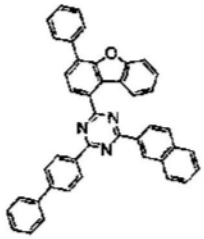
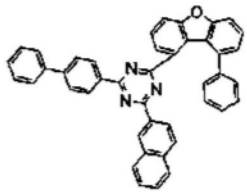
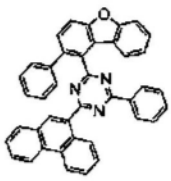
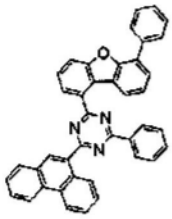
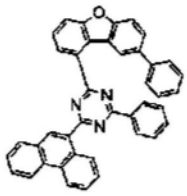
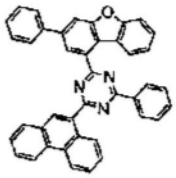
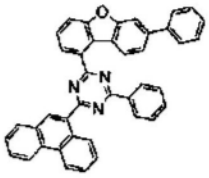
[0091]

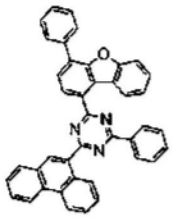
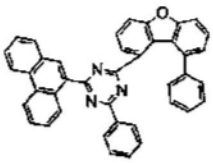
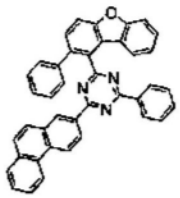
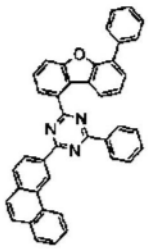
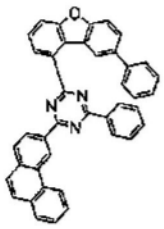
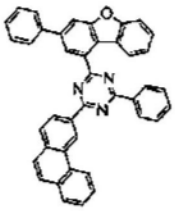
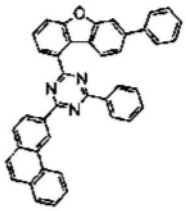
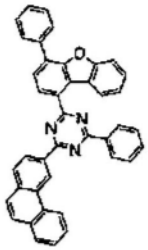


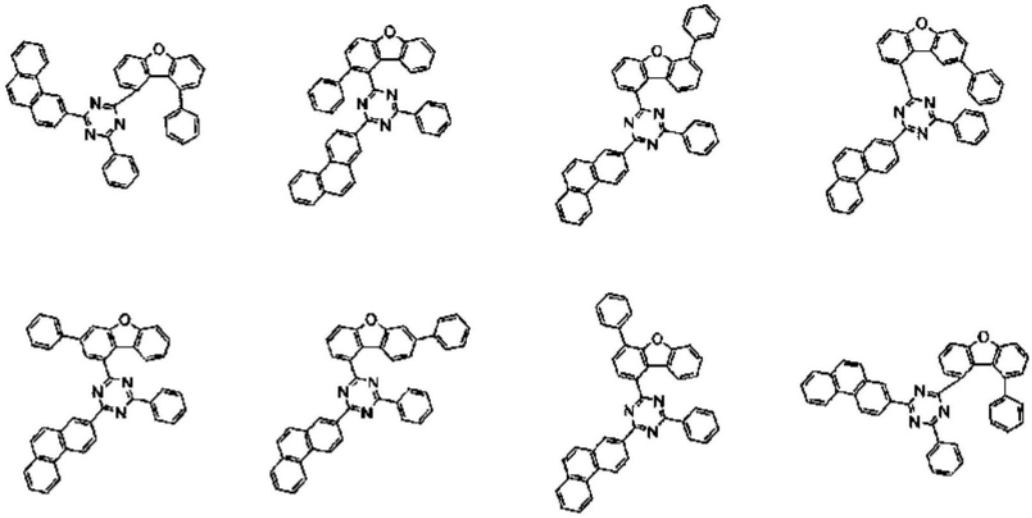
[0092]



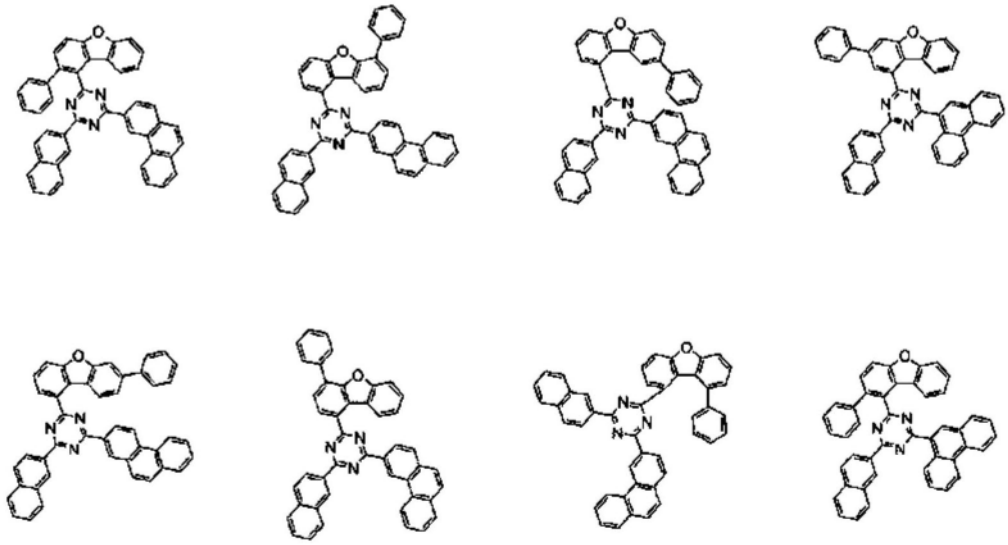









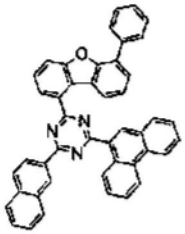
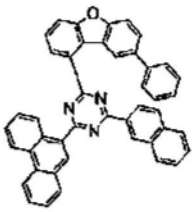
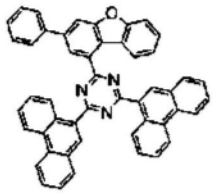
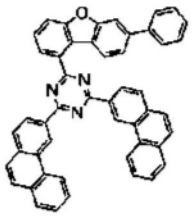
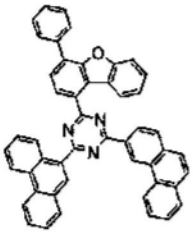
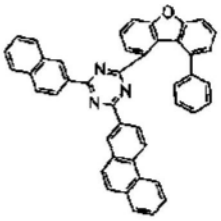
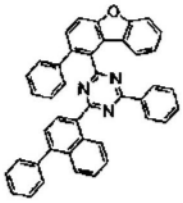
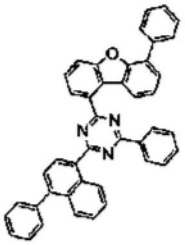
[0093]

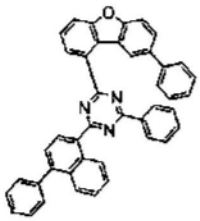
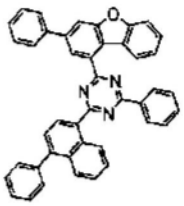
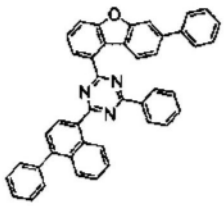
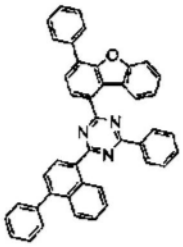
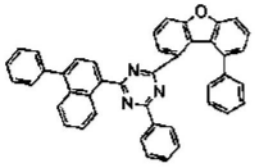
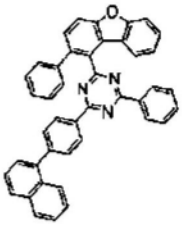
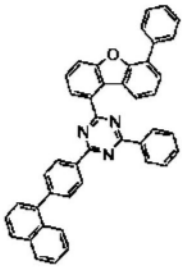
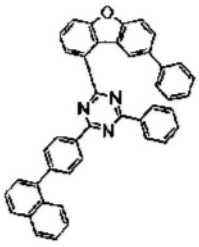


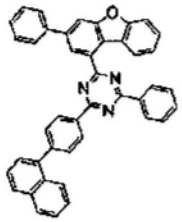
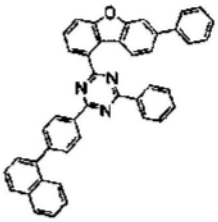
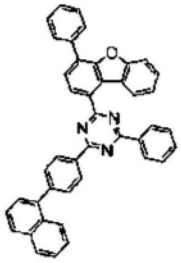
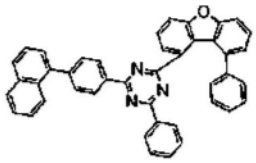
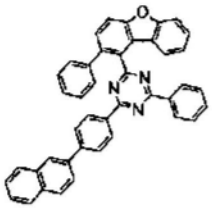
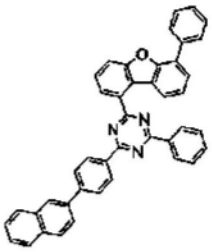
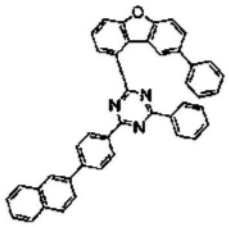
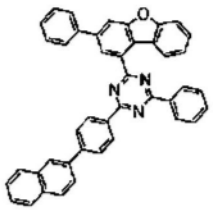
[0094]



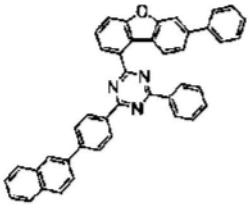
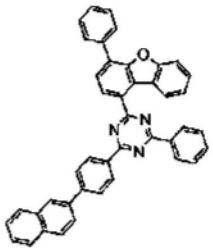
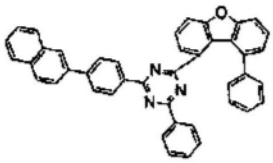
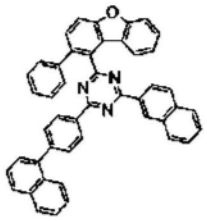
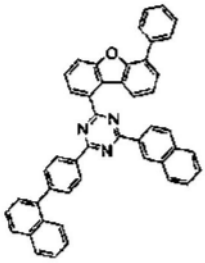
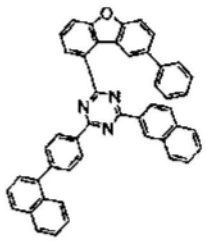
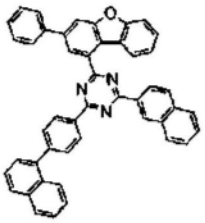
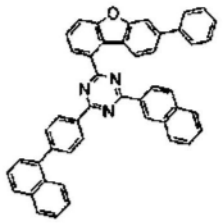









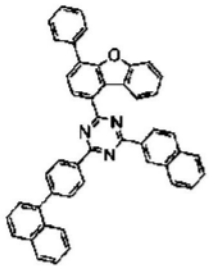
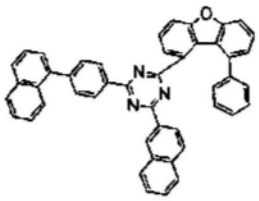
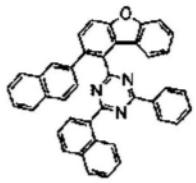
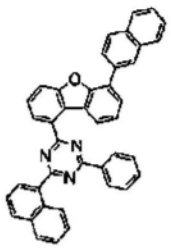
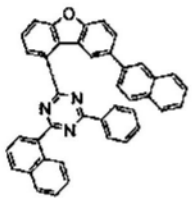
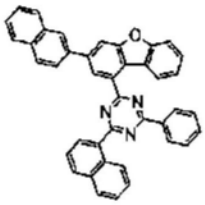
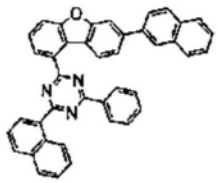
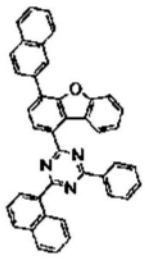
[0095]

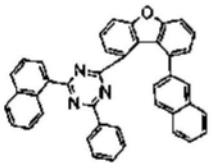
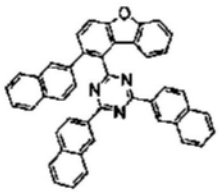
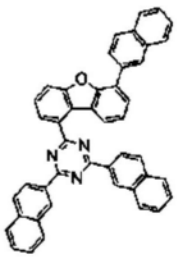
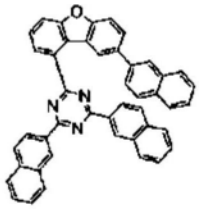
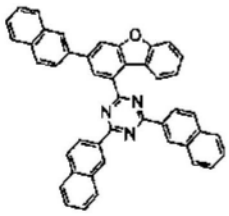
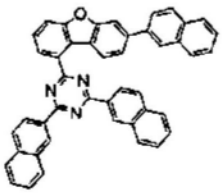
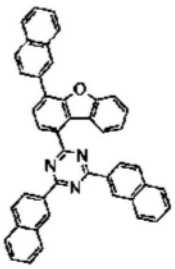
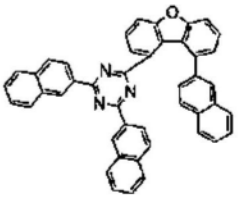









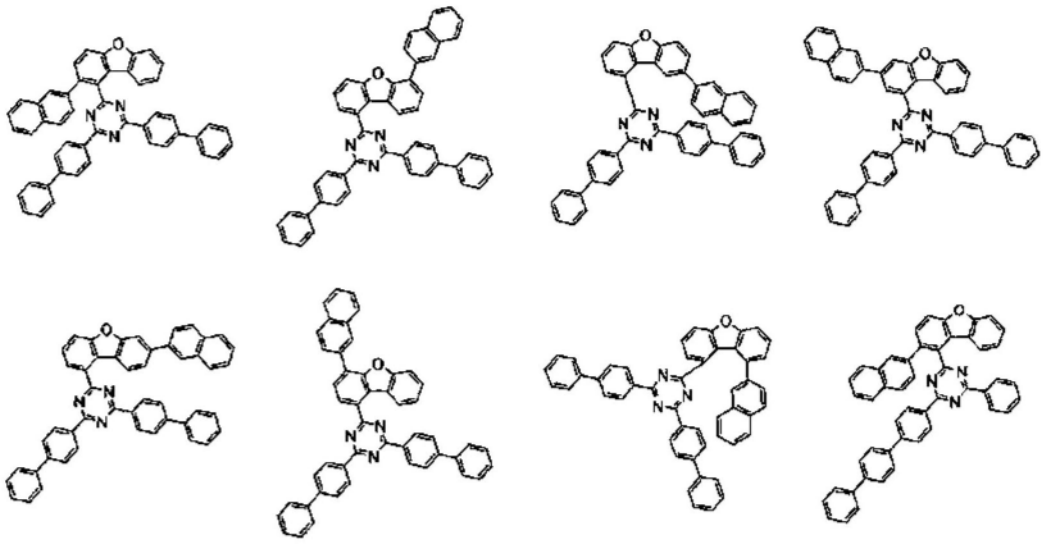
[0096]

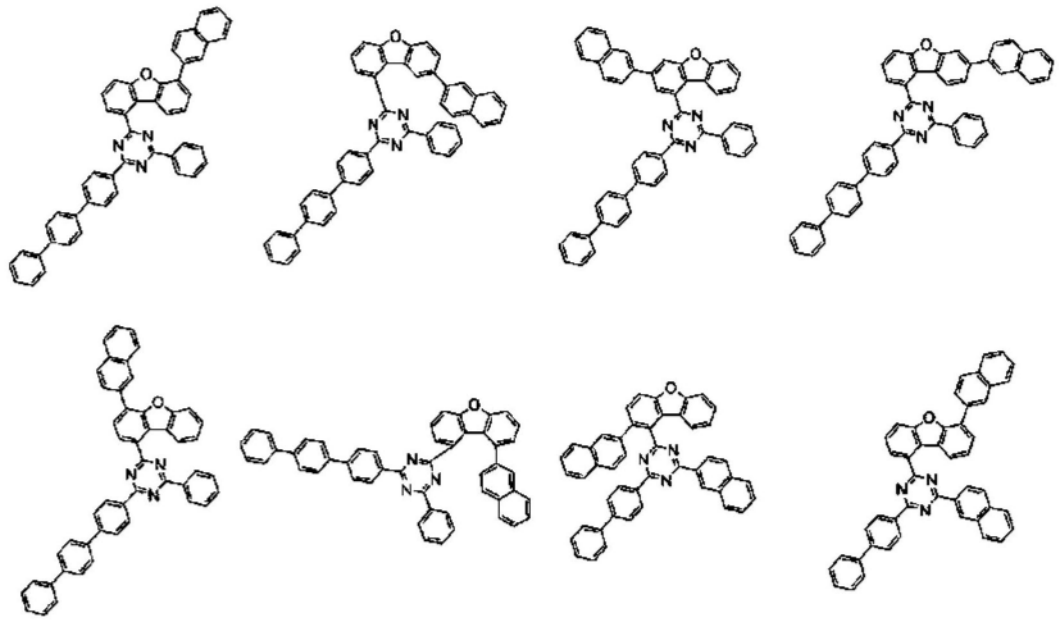









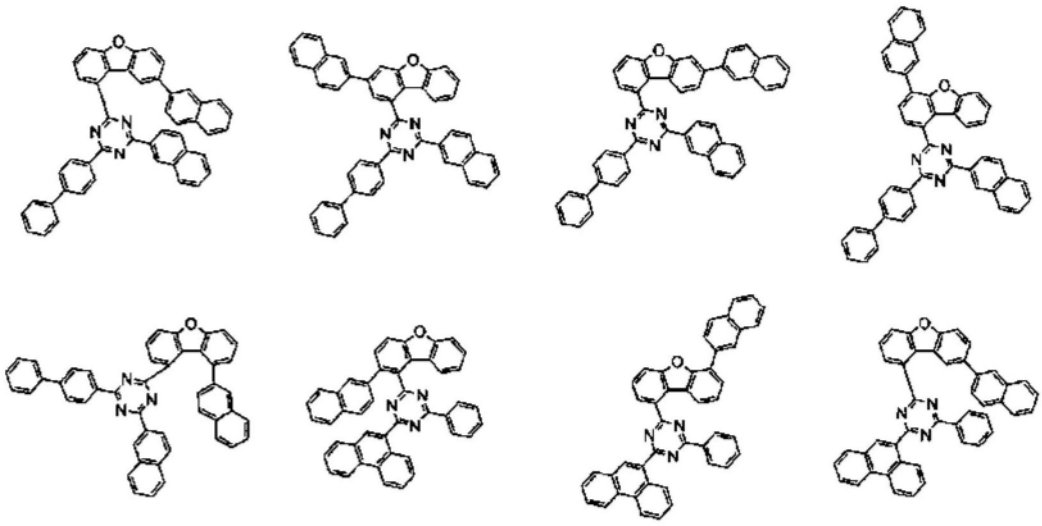
[0097]

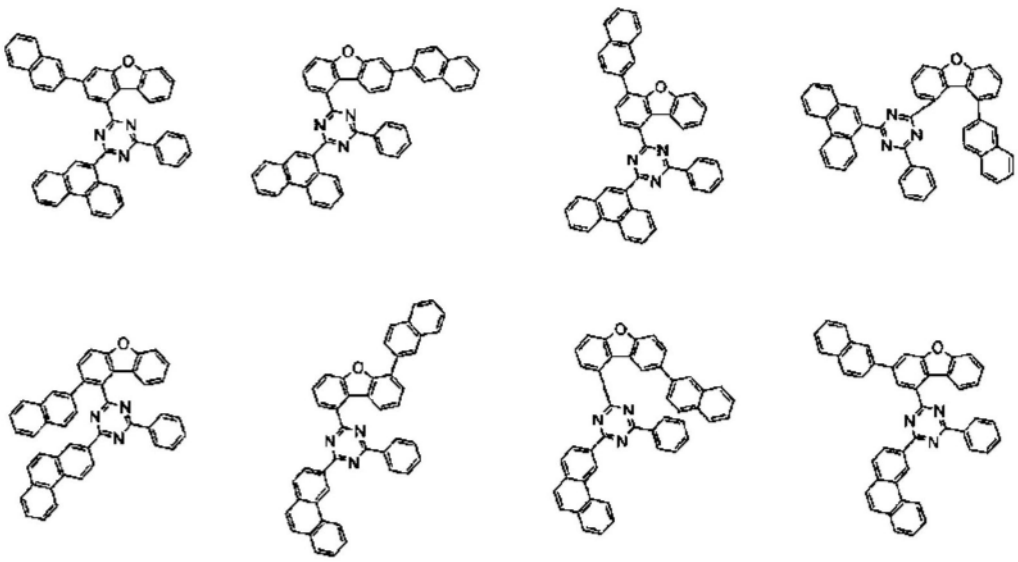


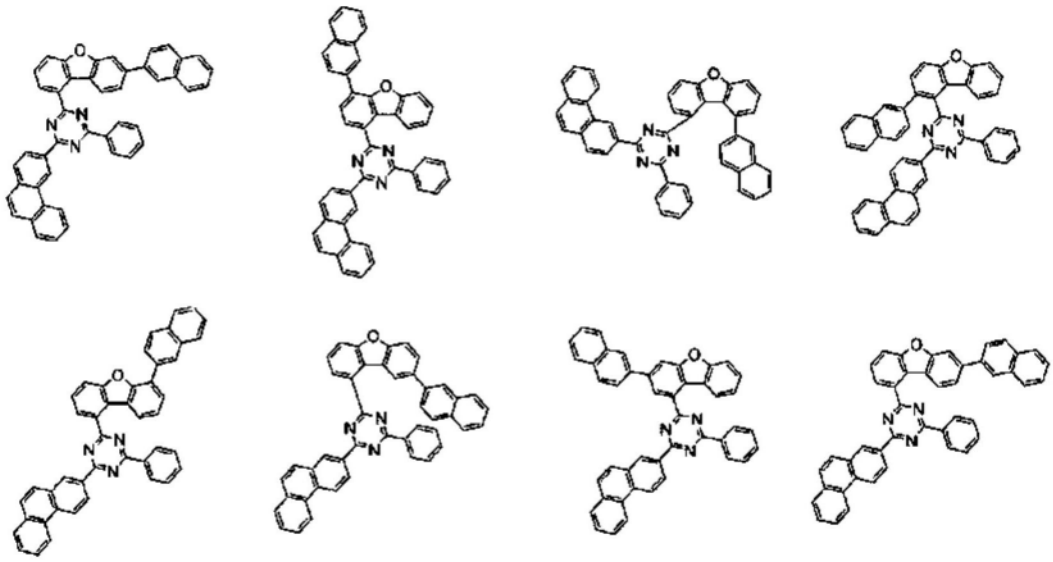
[0098]



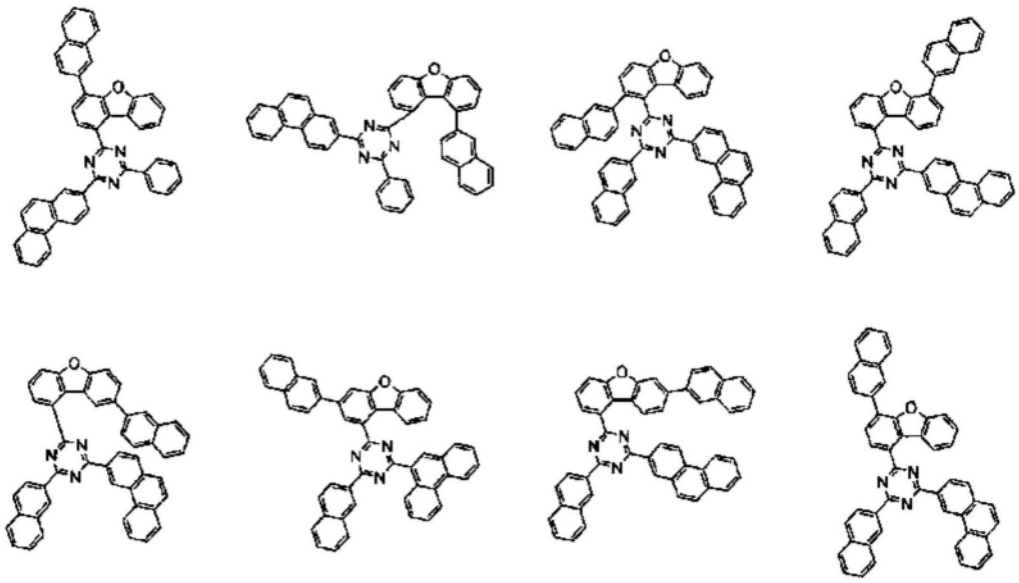


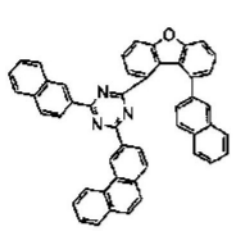
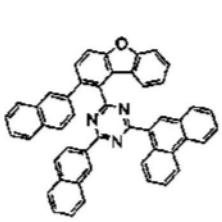
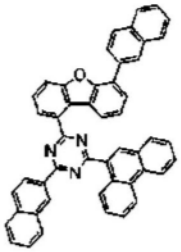
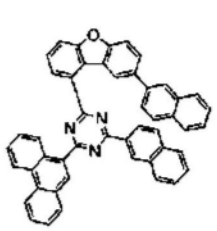
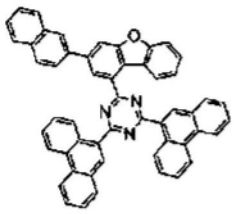
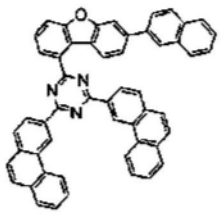
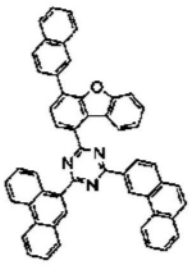
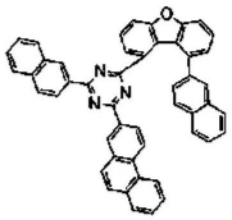
[0099]



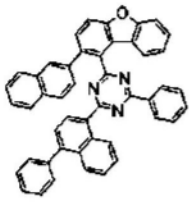
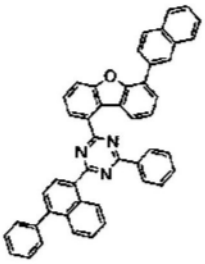
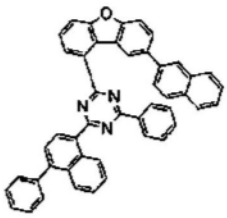
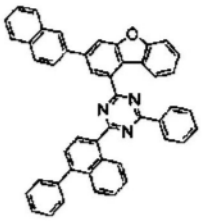
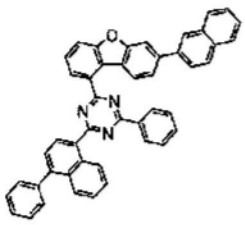
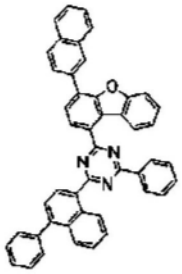
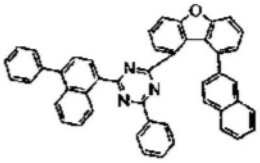
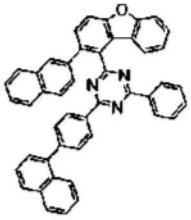


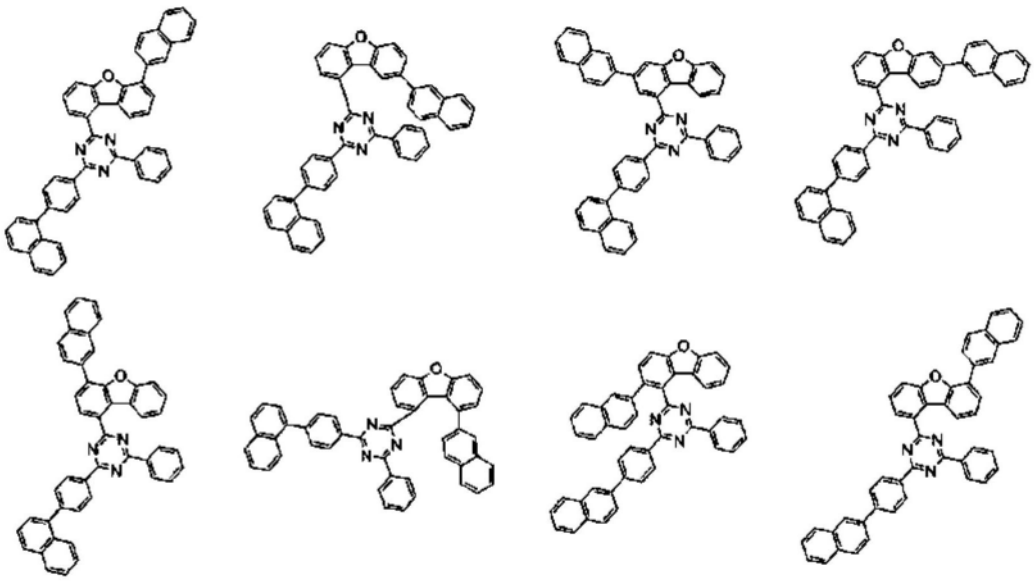
[0100]



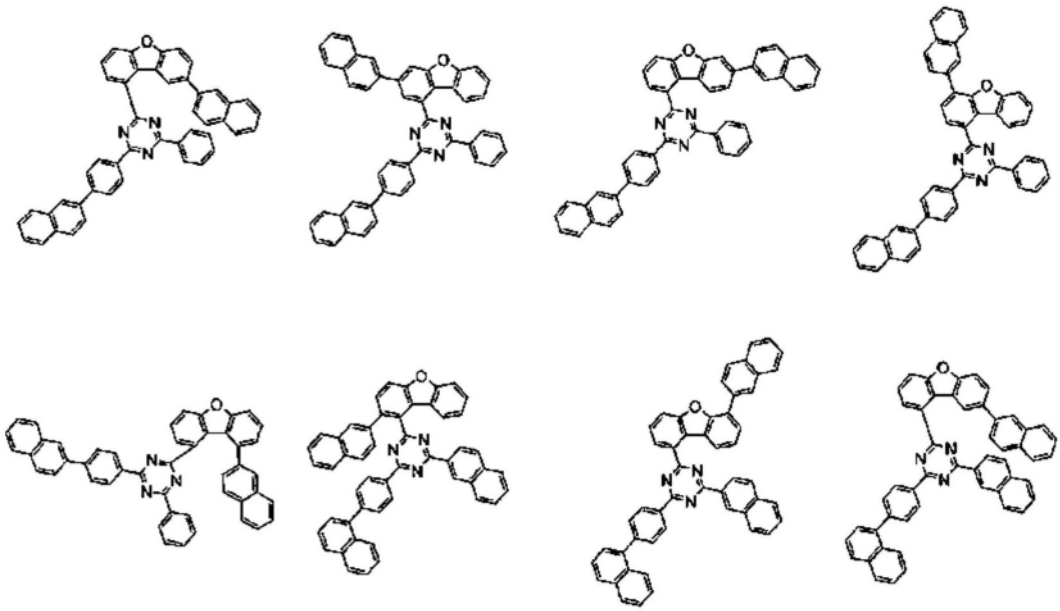









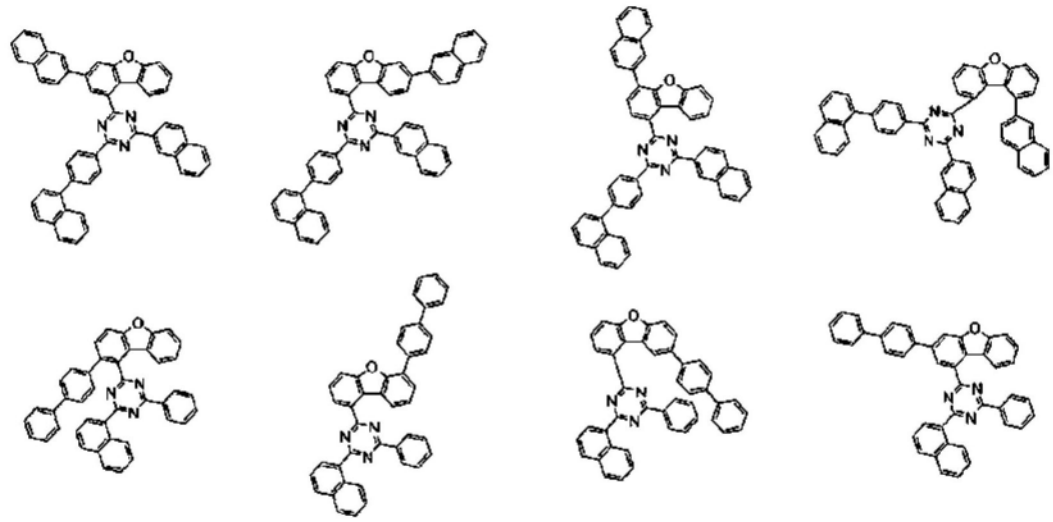
[0101]

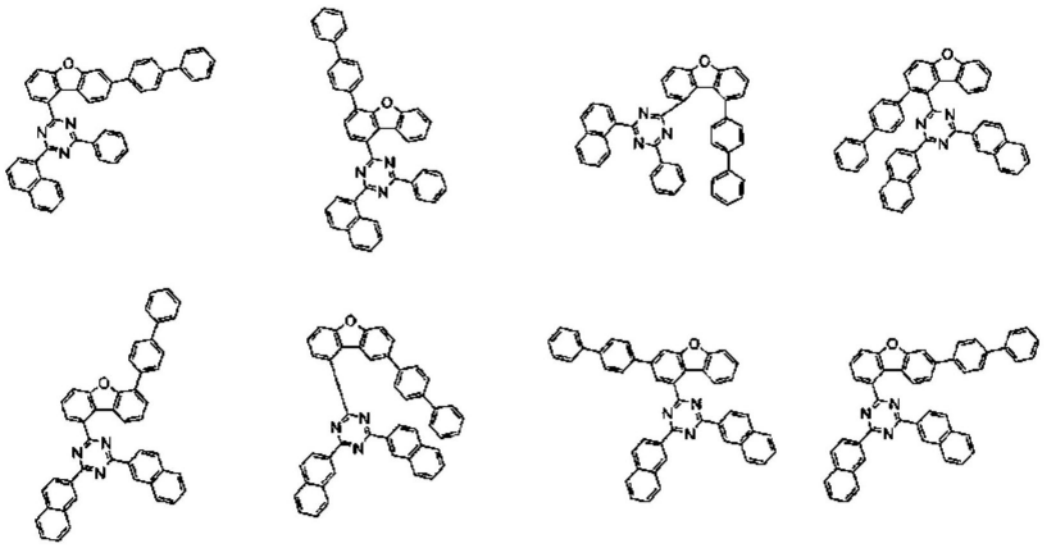


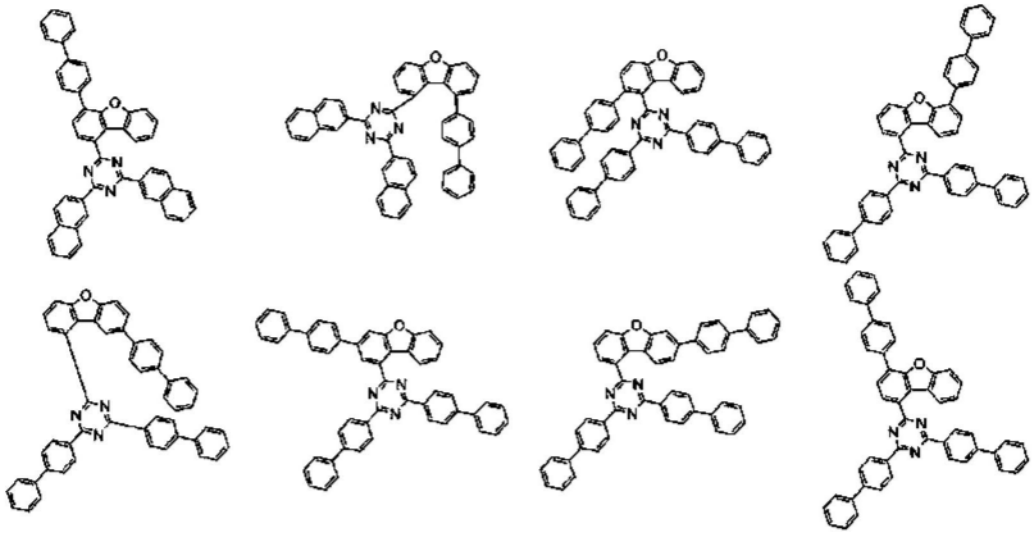
[0102]



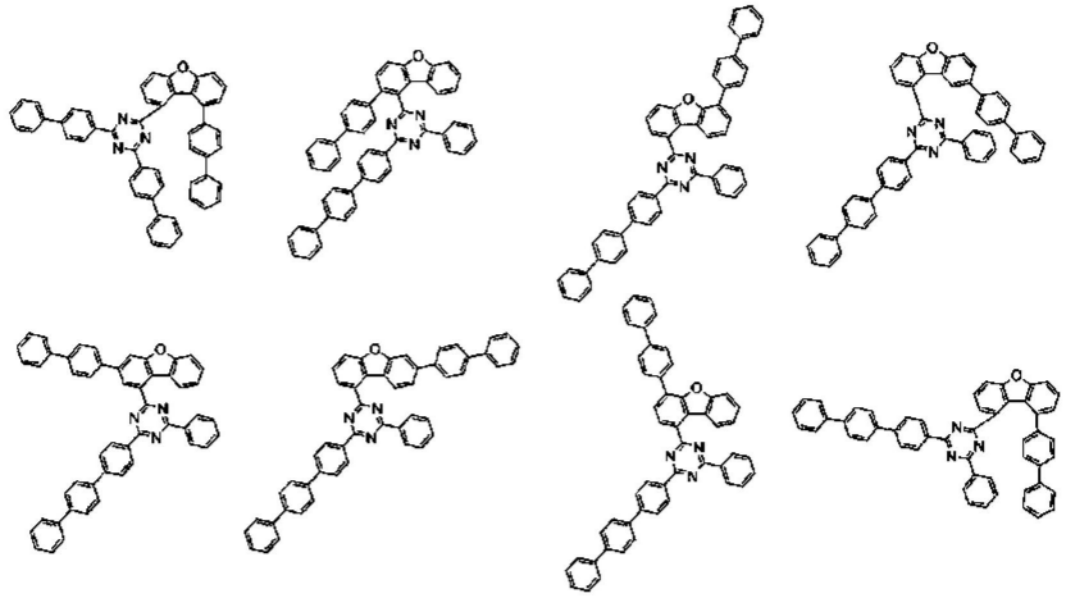


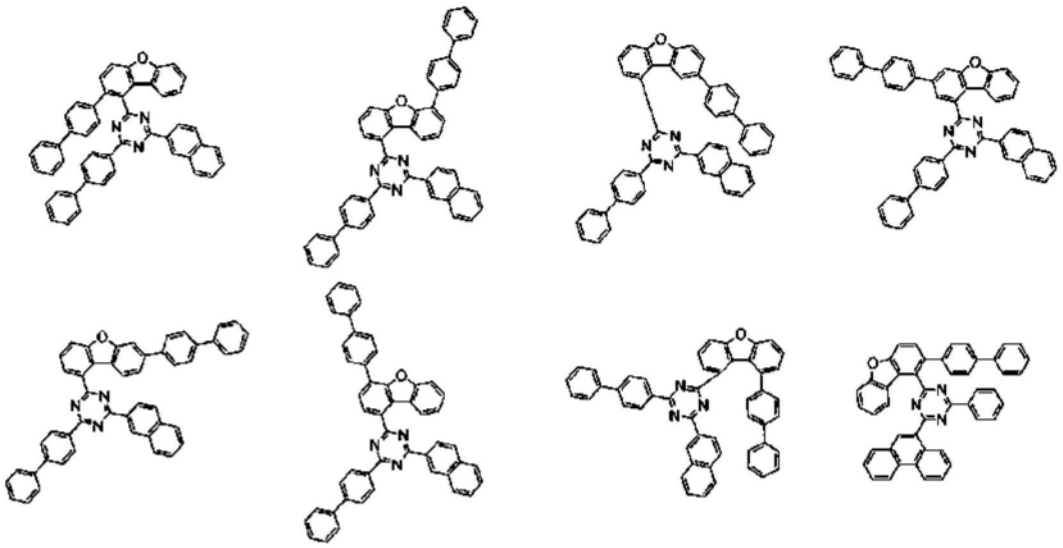
[0103]



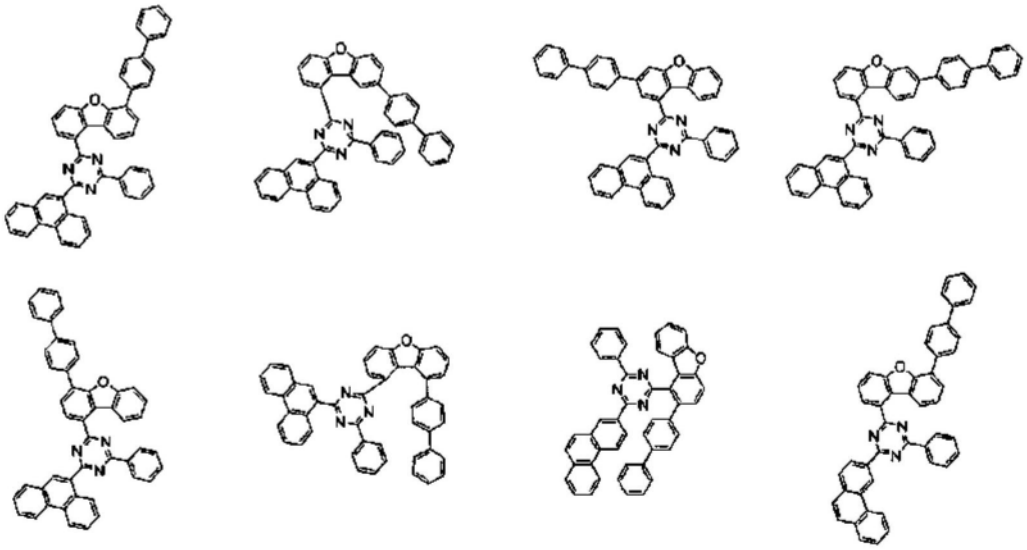


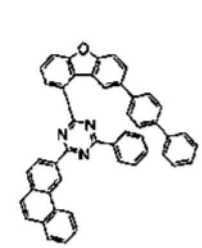
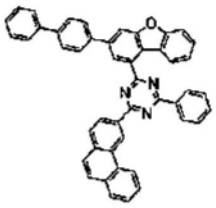
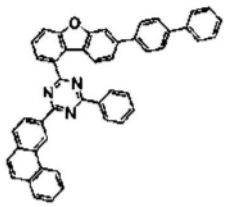
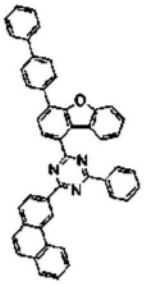
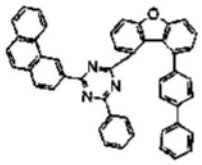
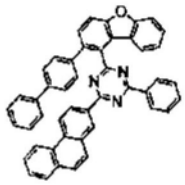
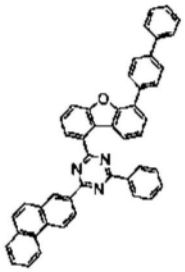
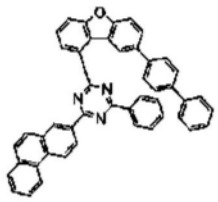
[0104]



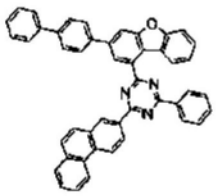
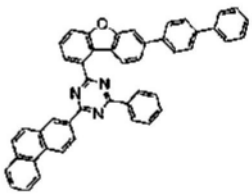
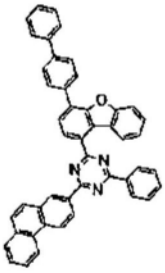
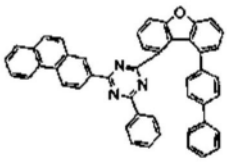
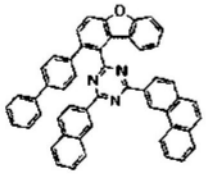
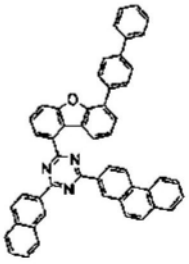
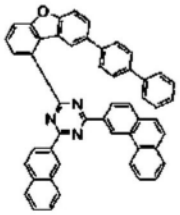
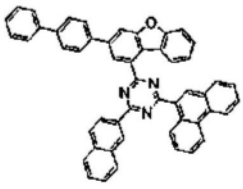


[0105]

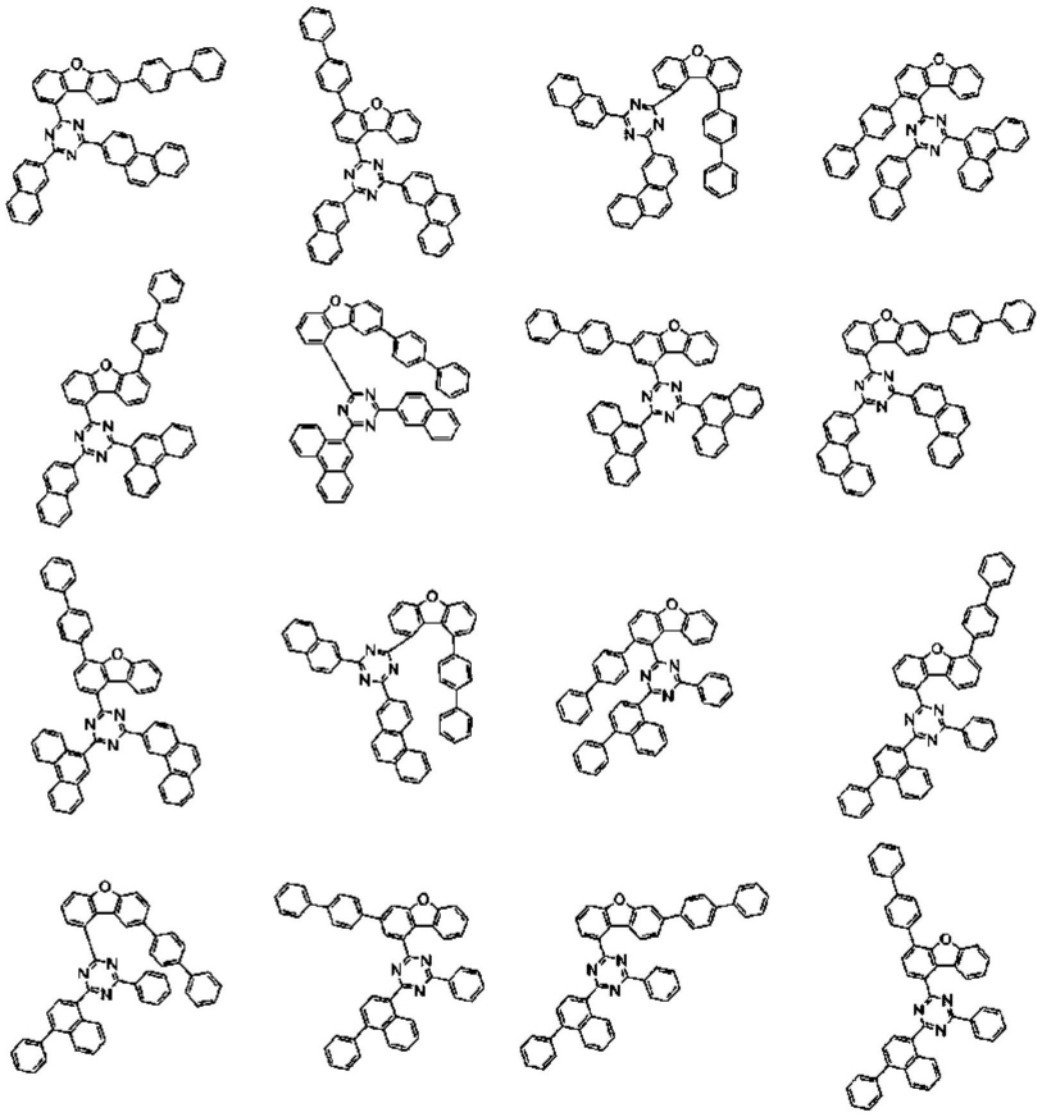


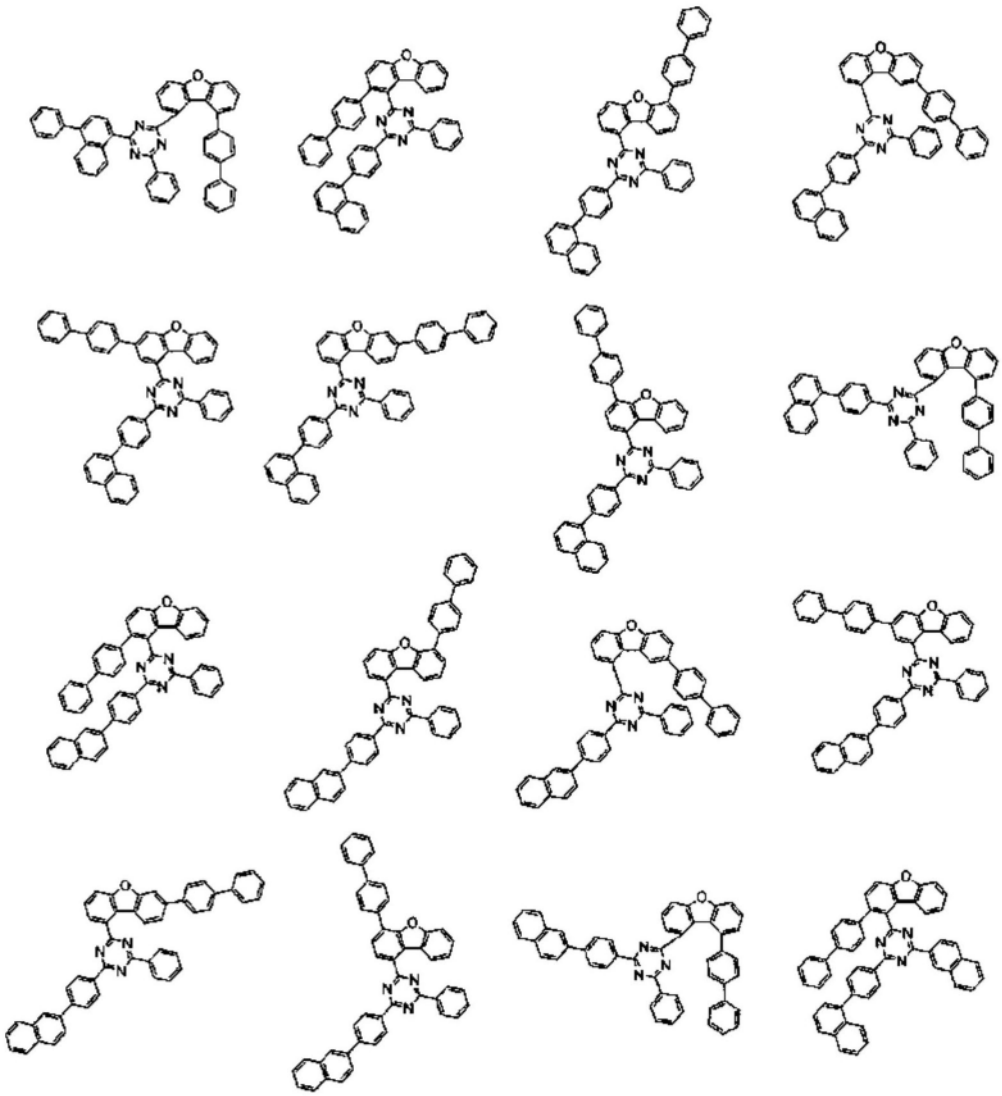
[0106]

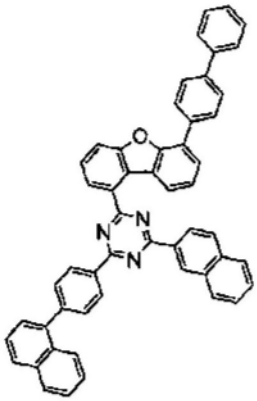
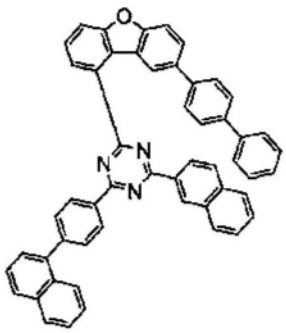
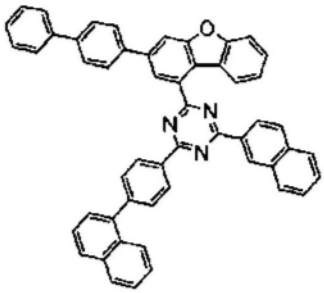
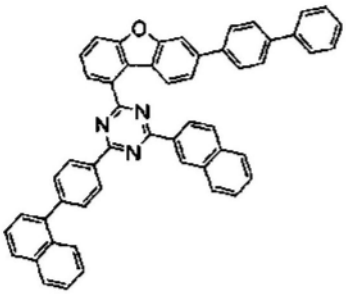
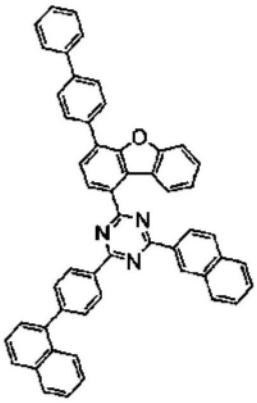
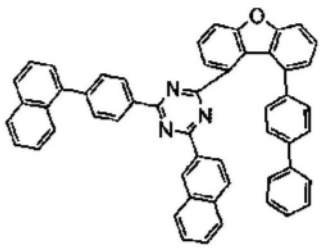
[0107]



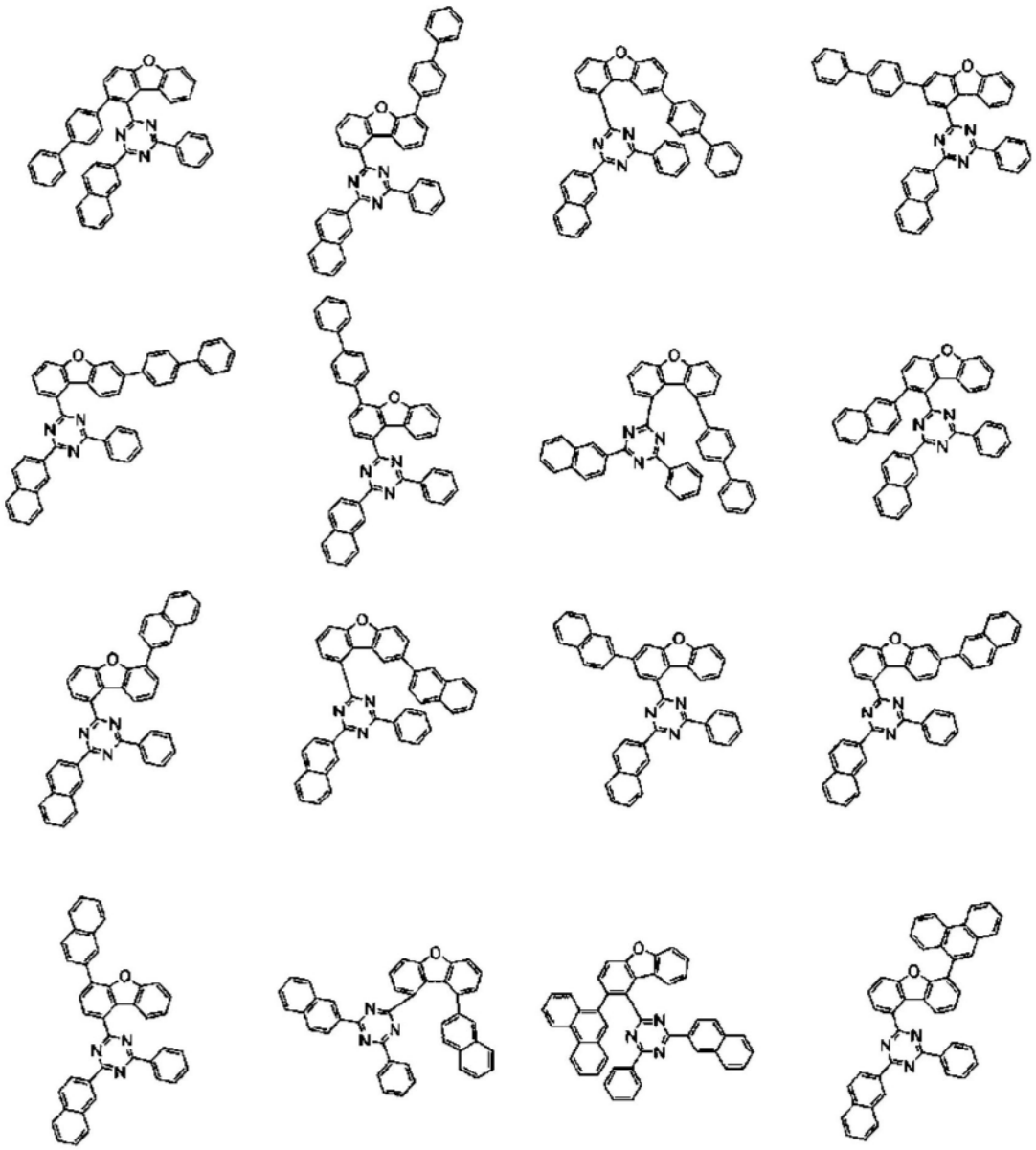
[0108]

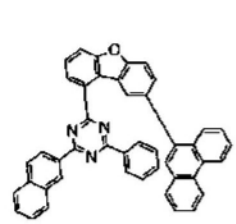
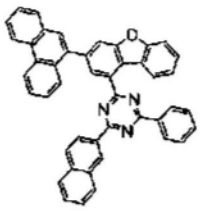
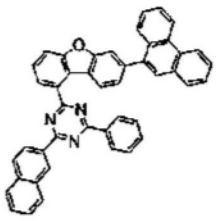
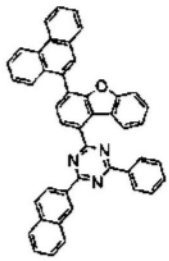
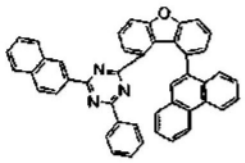
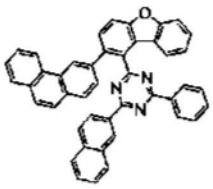
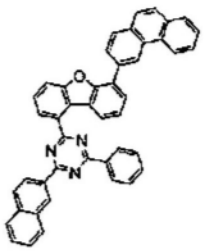
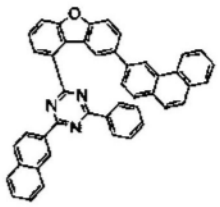


[0109]

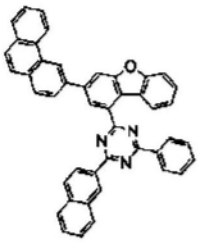
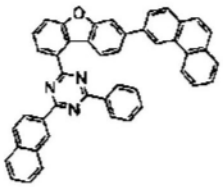
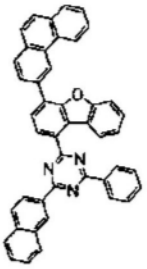
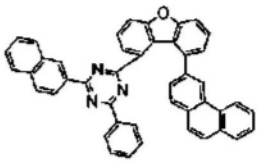
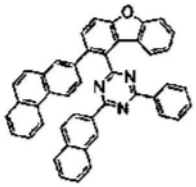
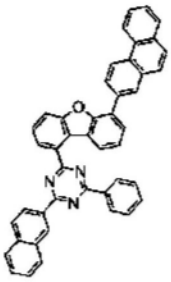
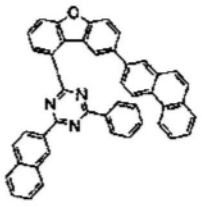
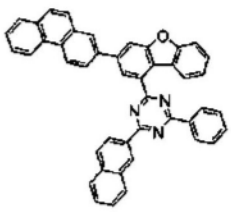







[0110]

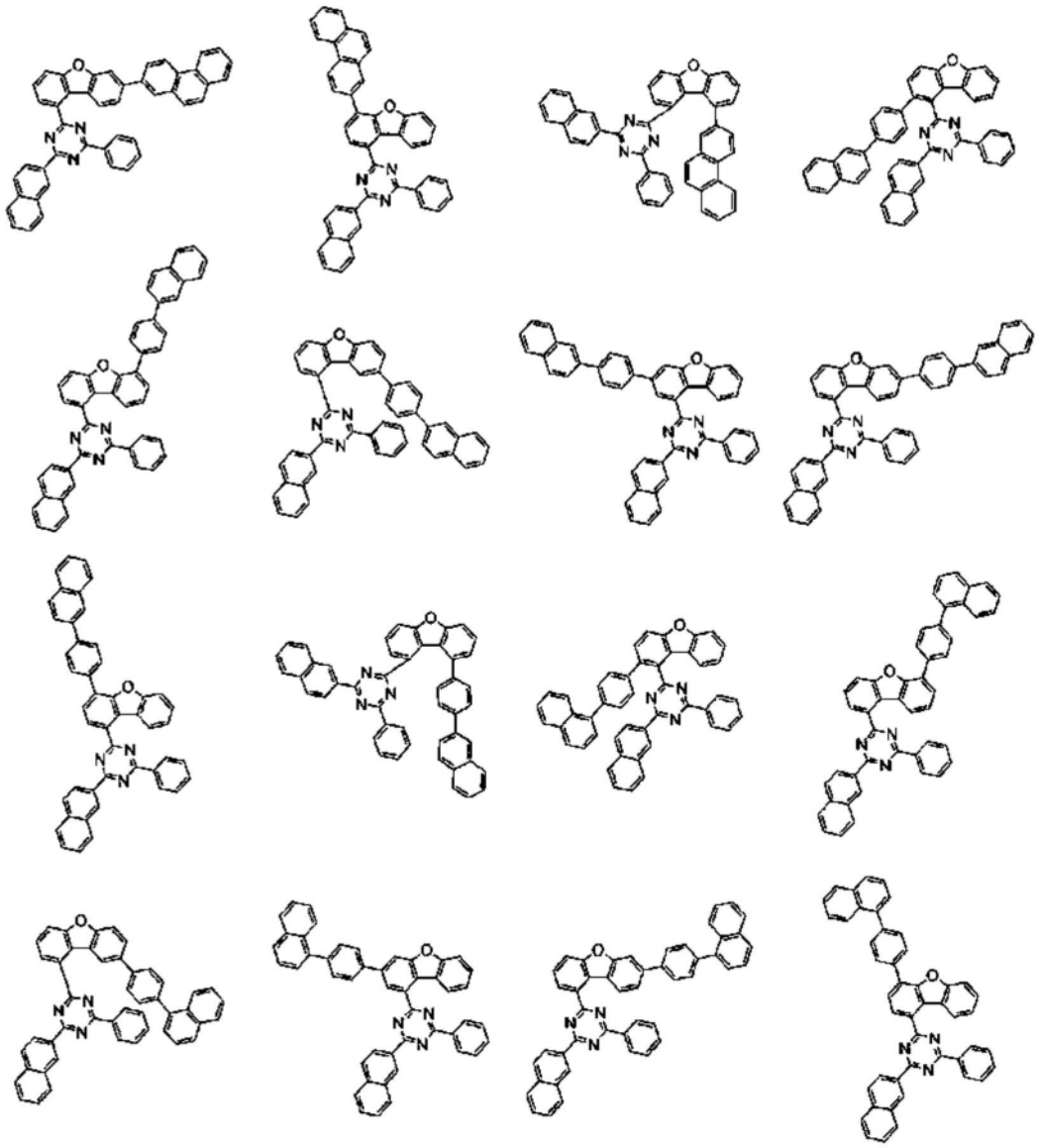


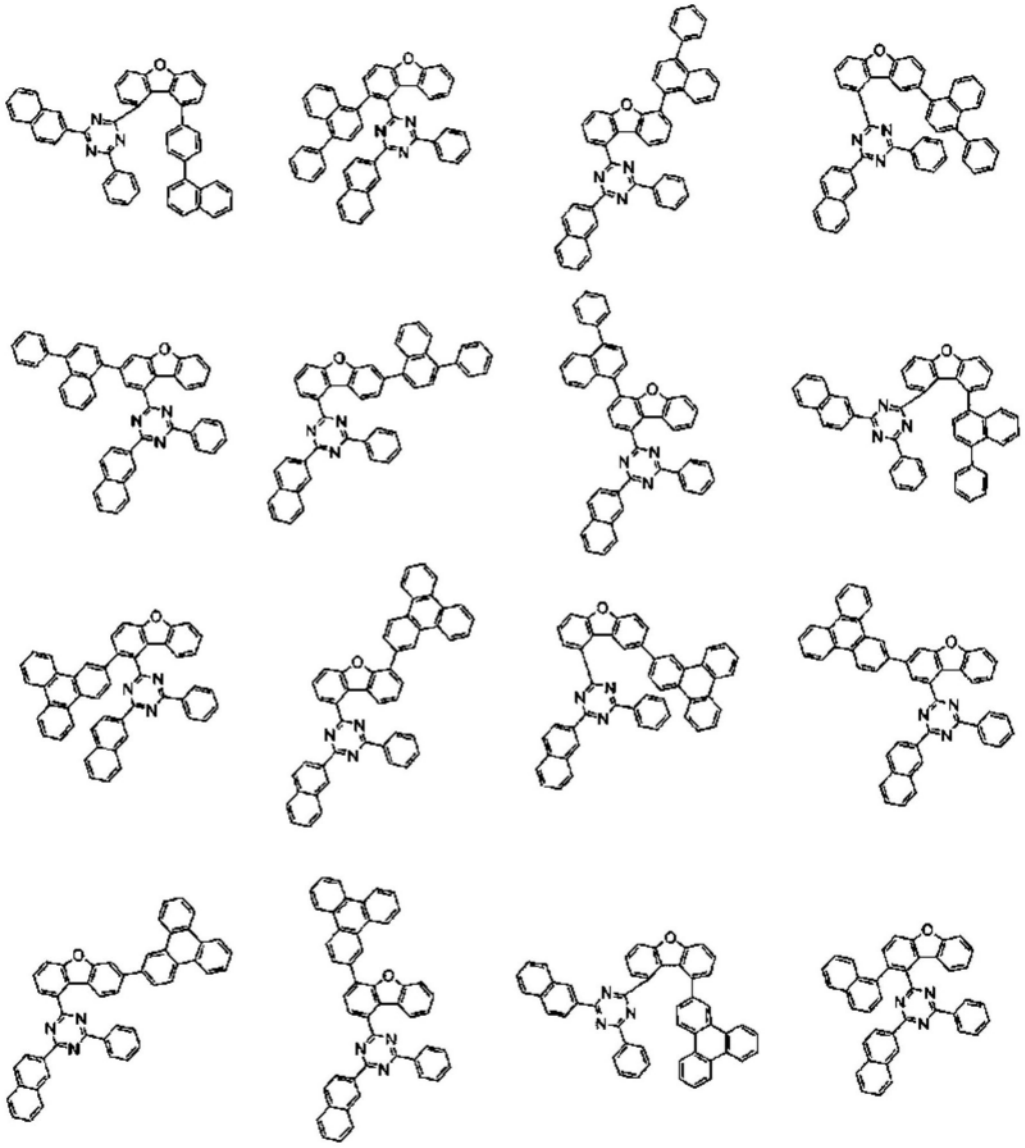
[0111]

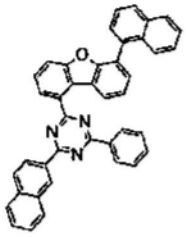
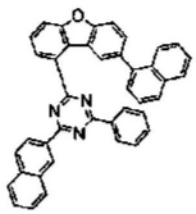
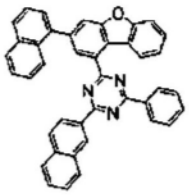
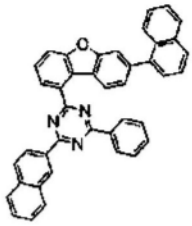
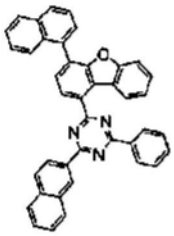
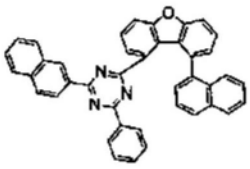
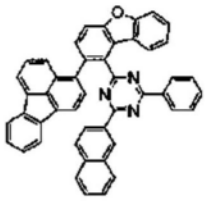
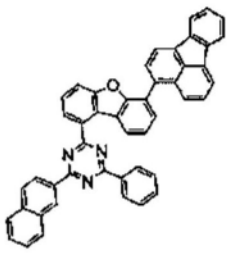









[0112]

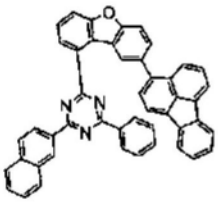
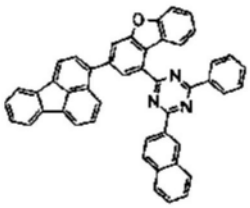
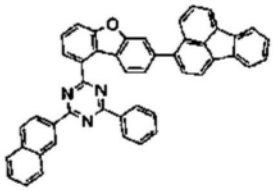
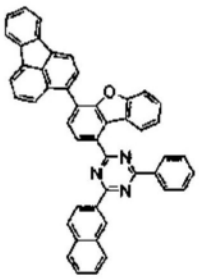
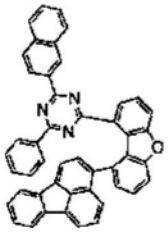
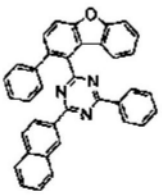
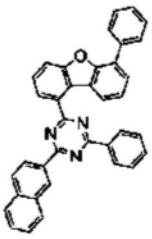
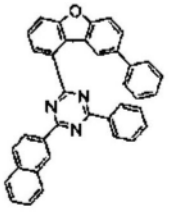


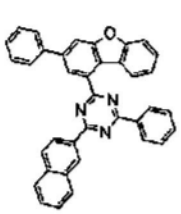
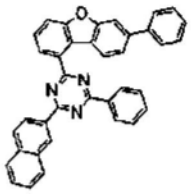
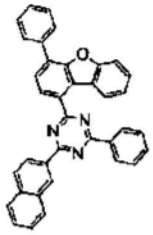
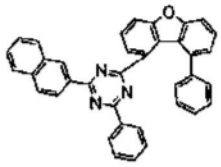
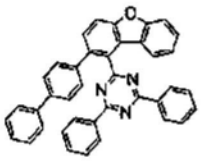
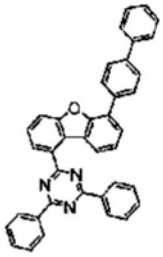
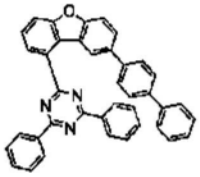
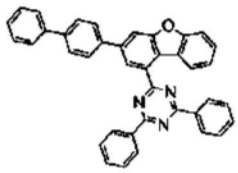
[0113]



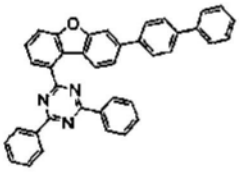
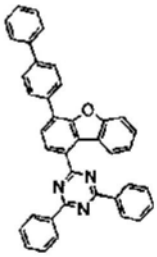
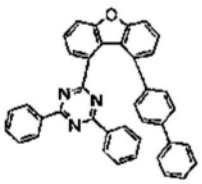
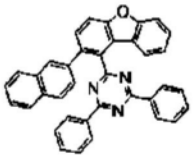
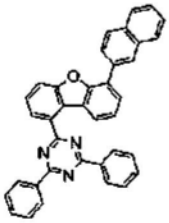
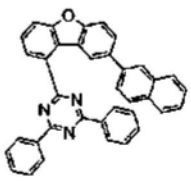
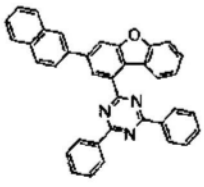
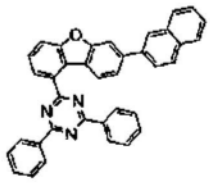









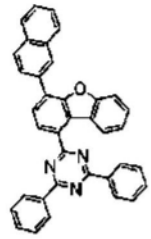
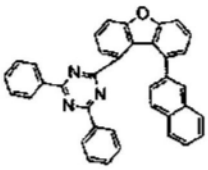
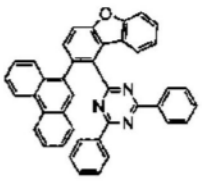
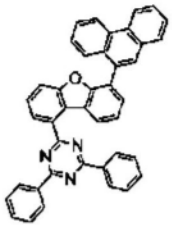
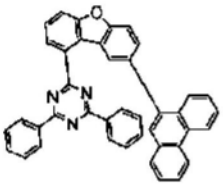
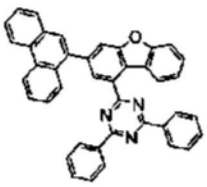
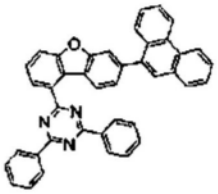
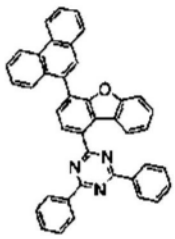
[0114]

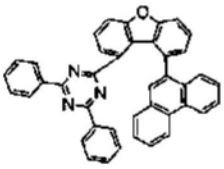
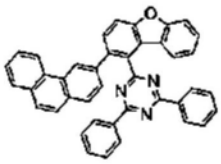
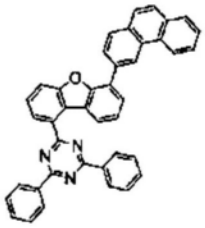
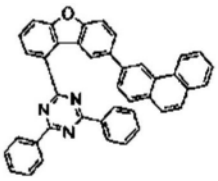
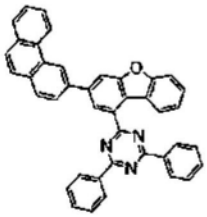
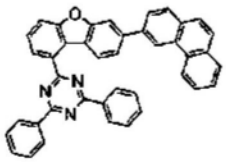
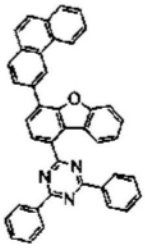
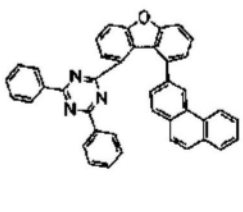









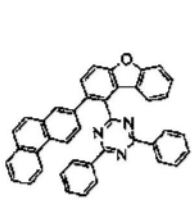
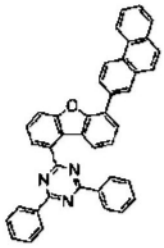
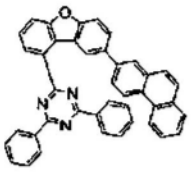
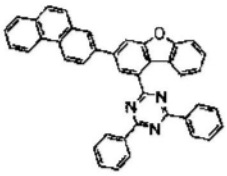
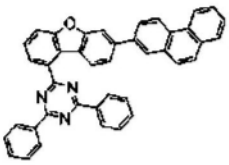
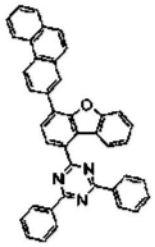
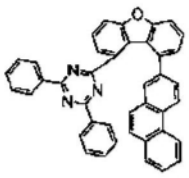
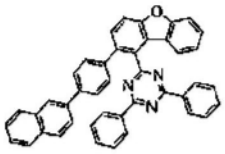
[0115]

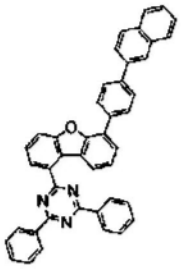
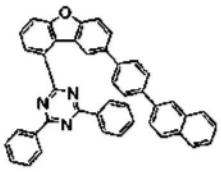
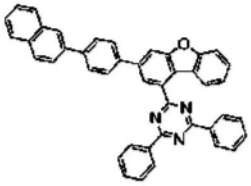
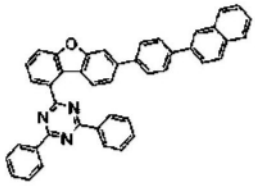
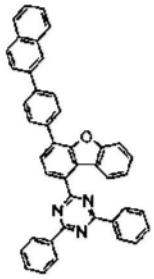
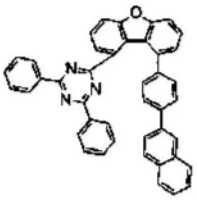
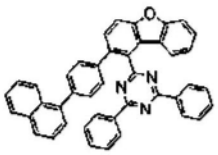
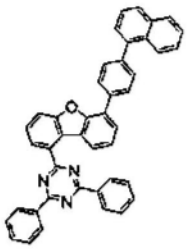









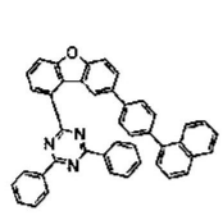
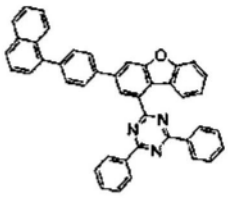
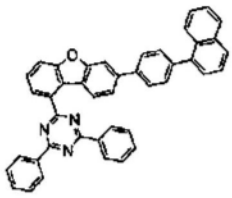
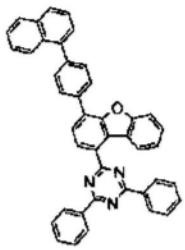
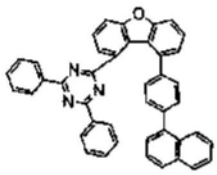
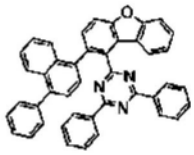
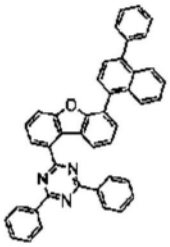
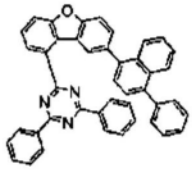
[0116]

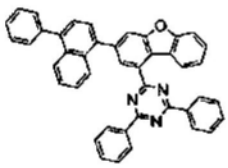
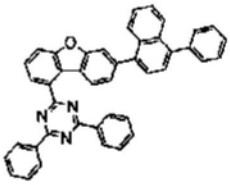
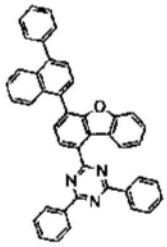
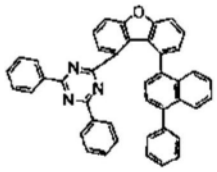
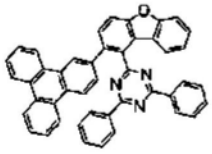
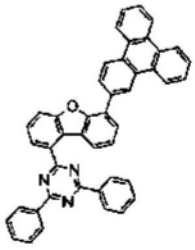
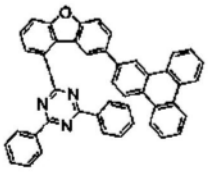
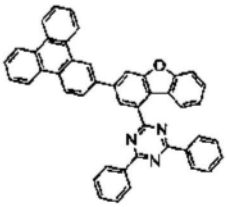









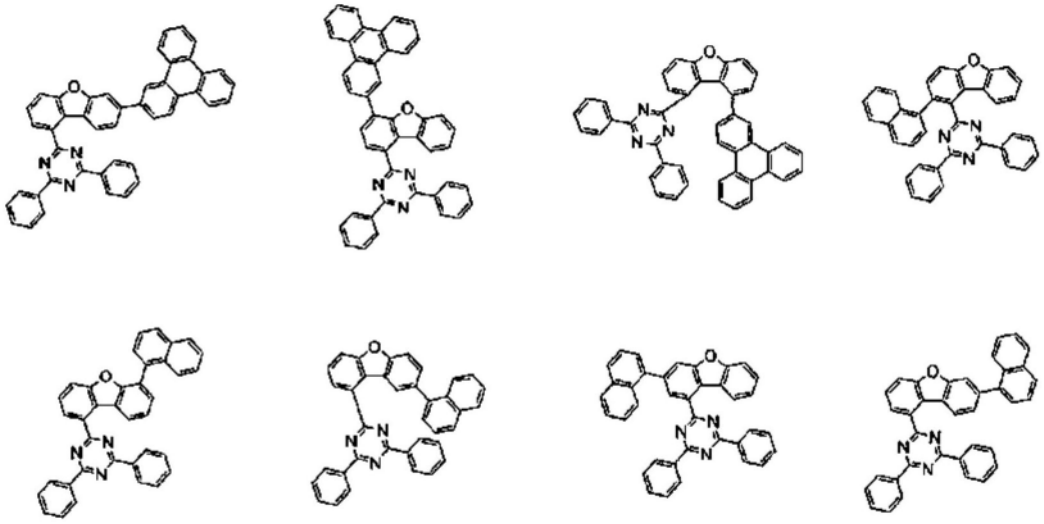
[0117]

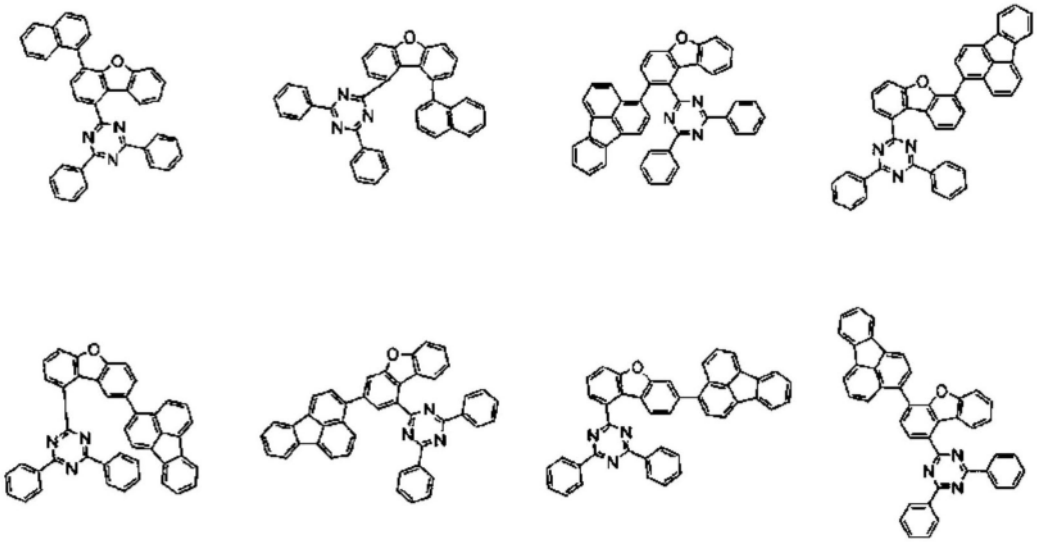









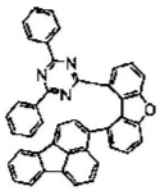
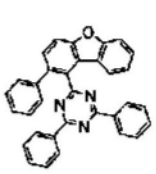
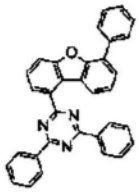
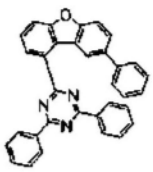
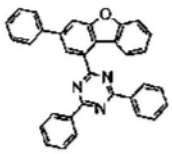
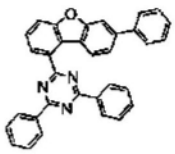
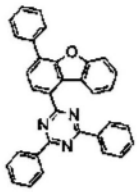
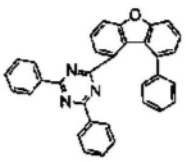
[0118]

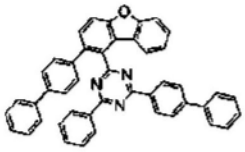
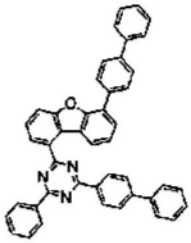
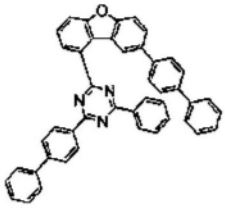
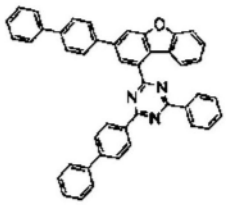
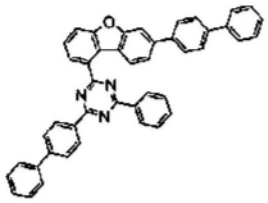
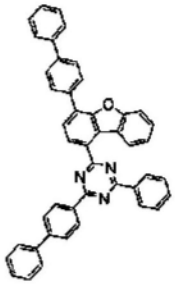
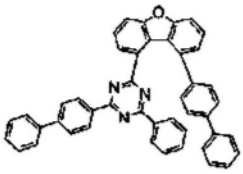
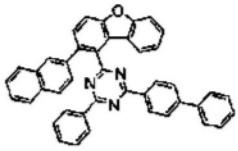


[0119]

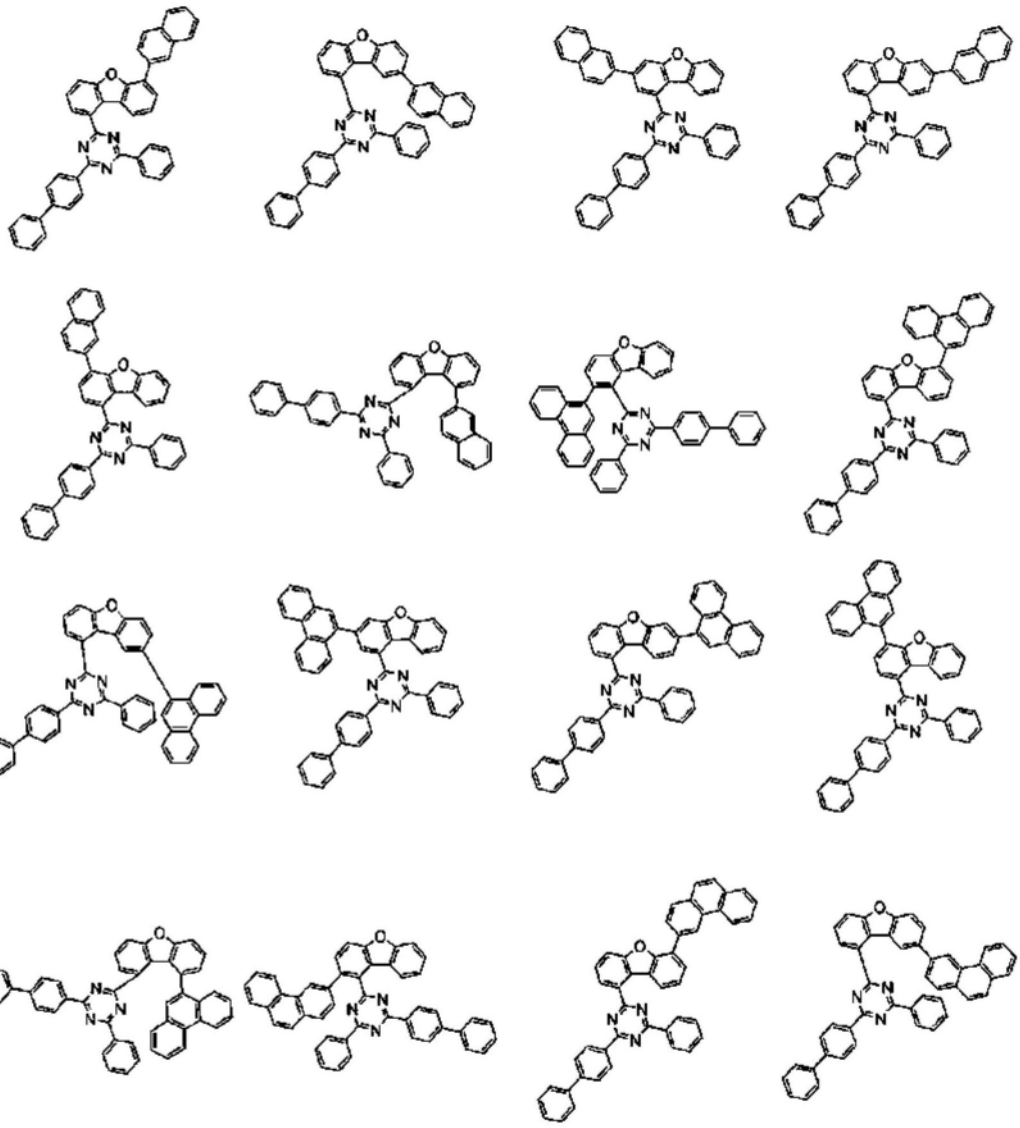


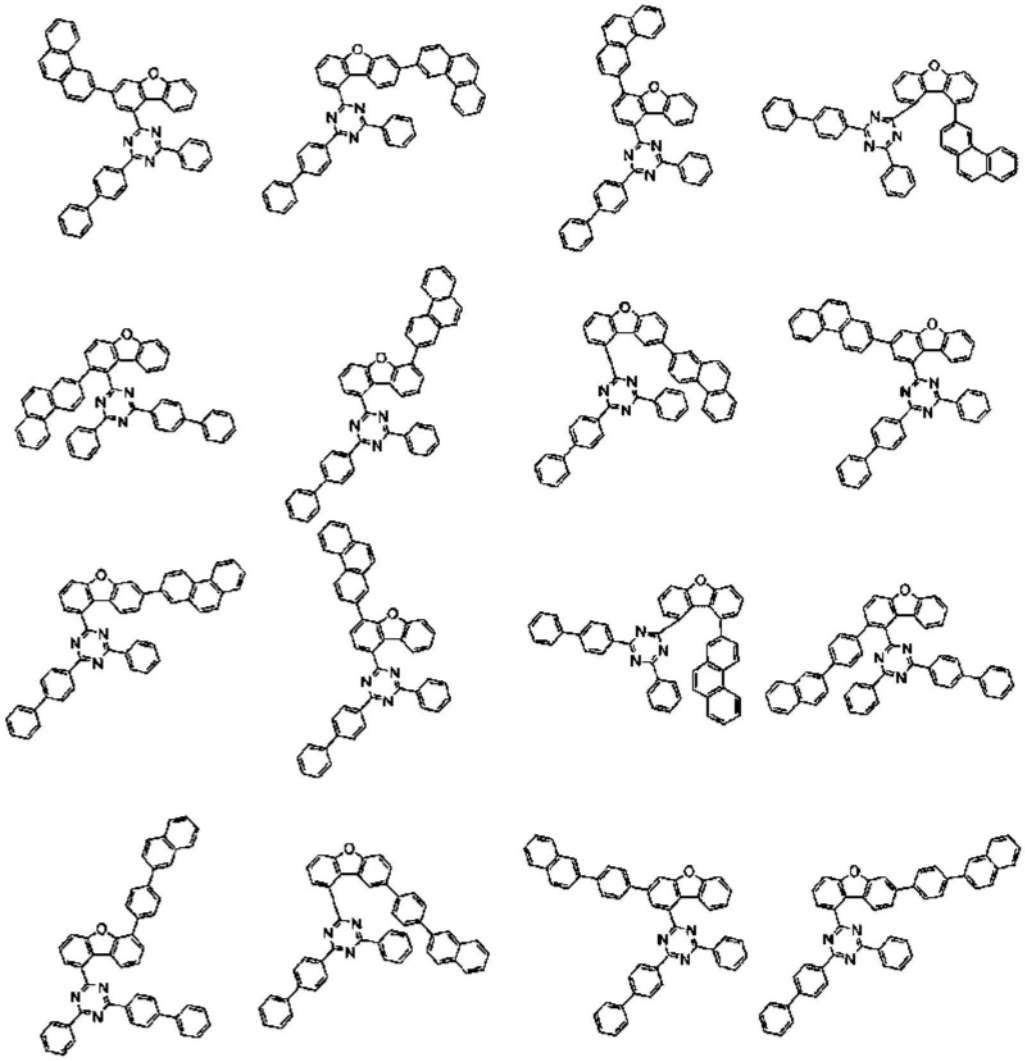
[0120]

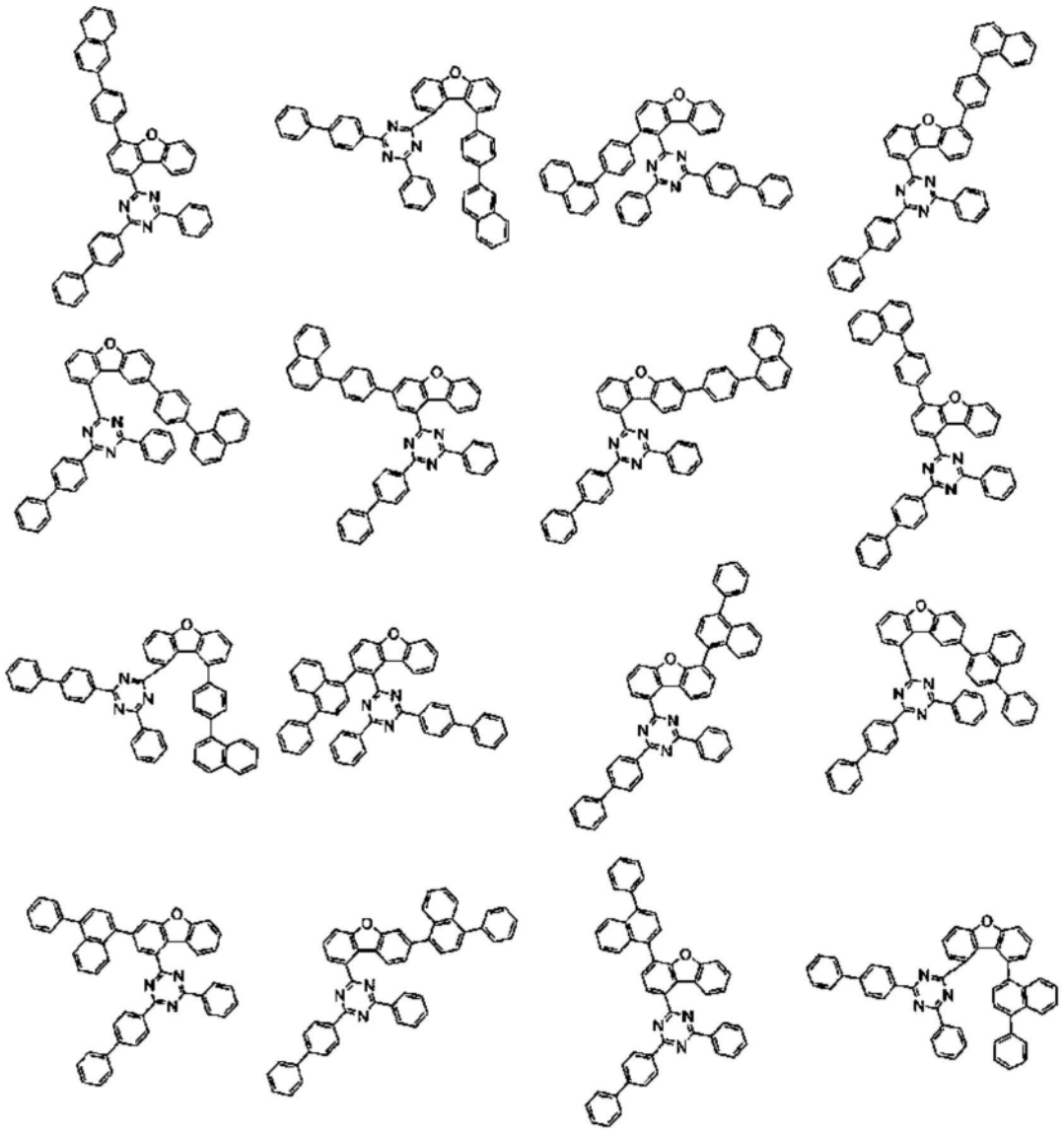
[0121]



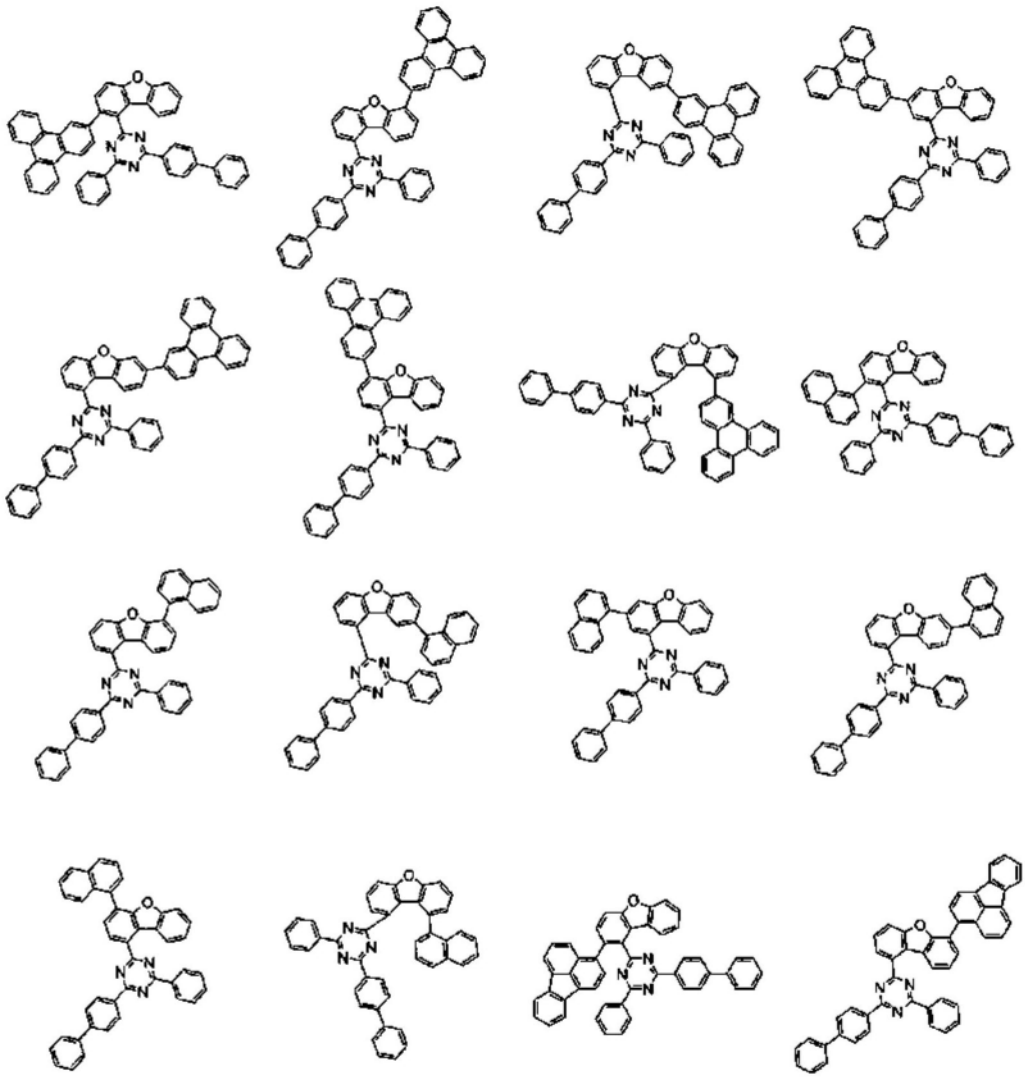
[0122]



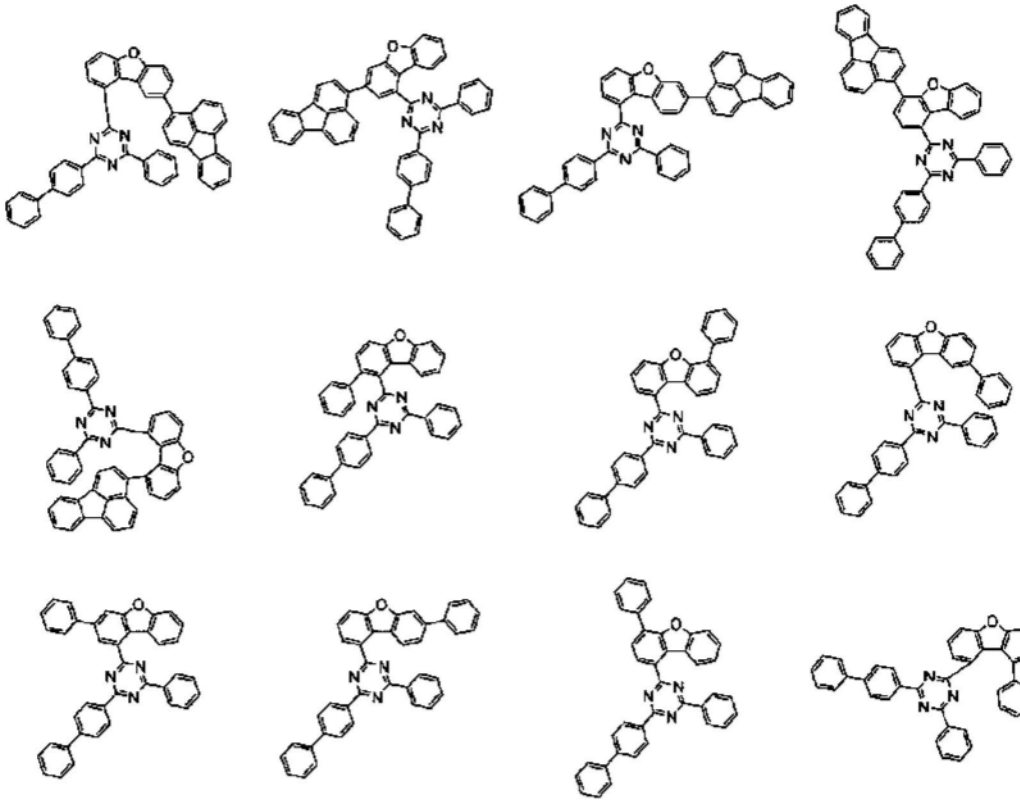
[0123]

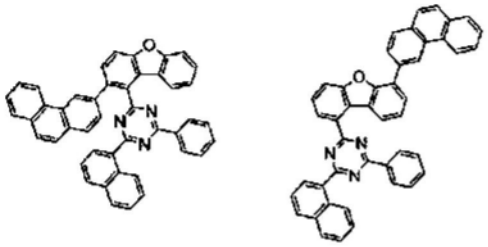


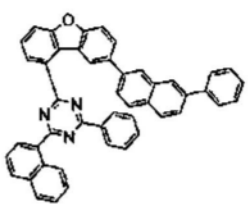
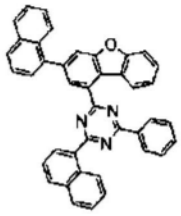
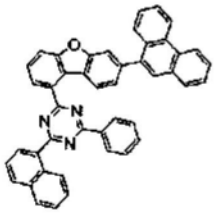
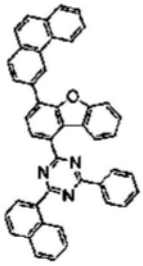
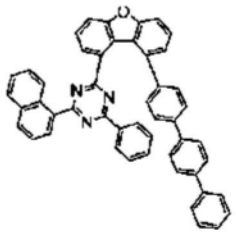
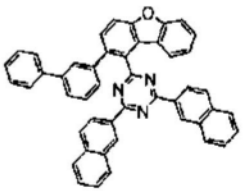
[0124]



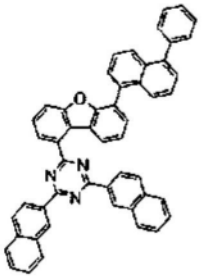
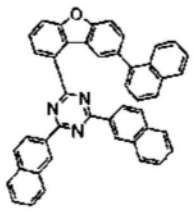
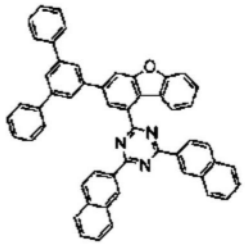
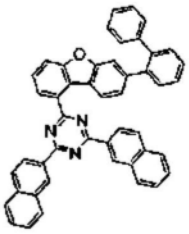
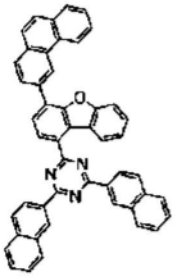
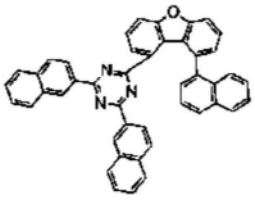
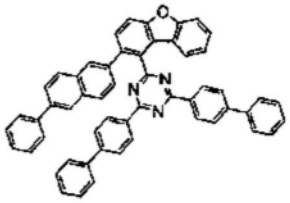
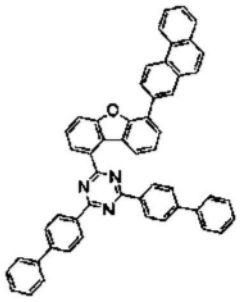
[0125]



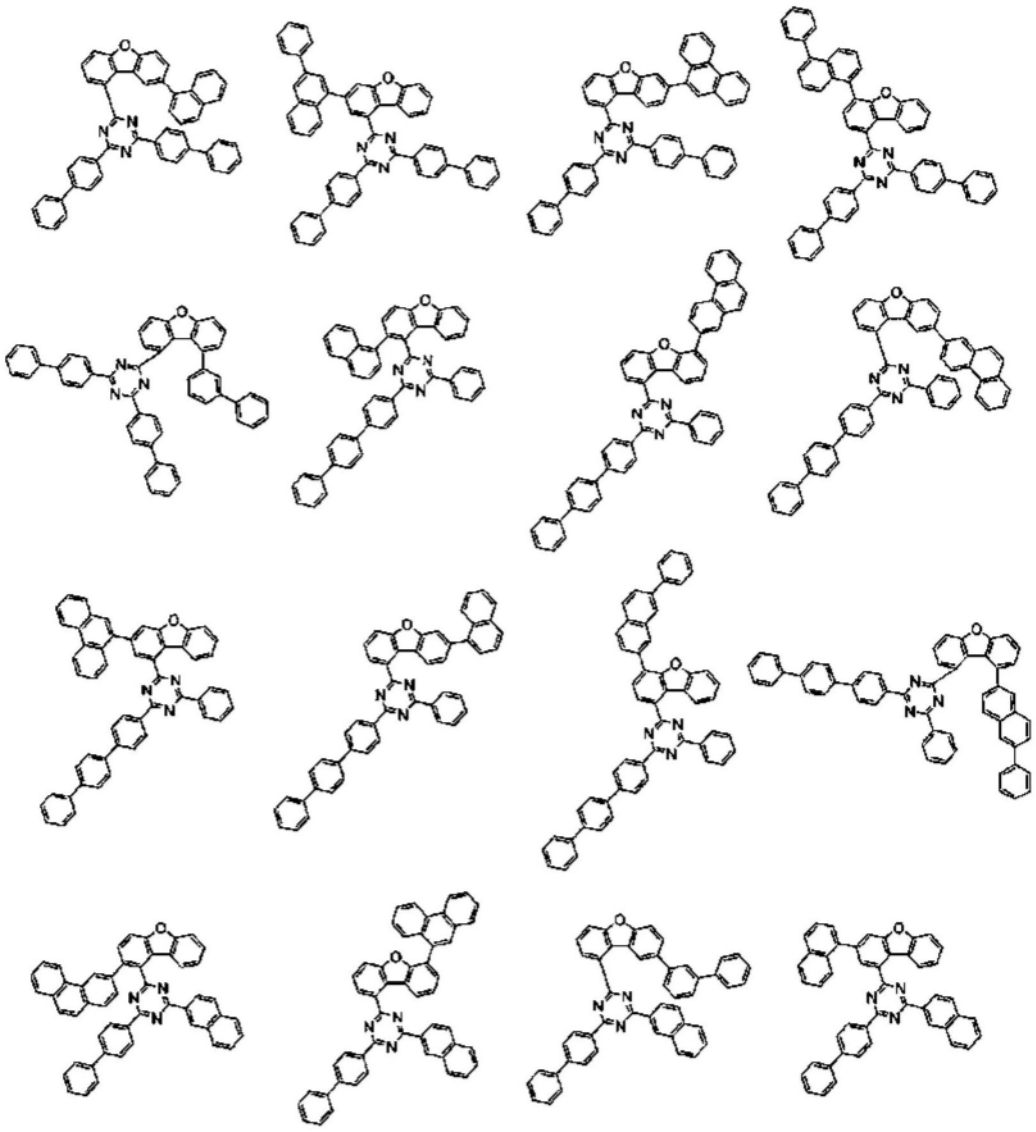


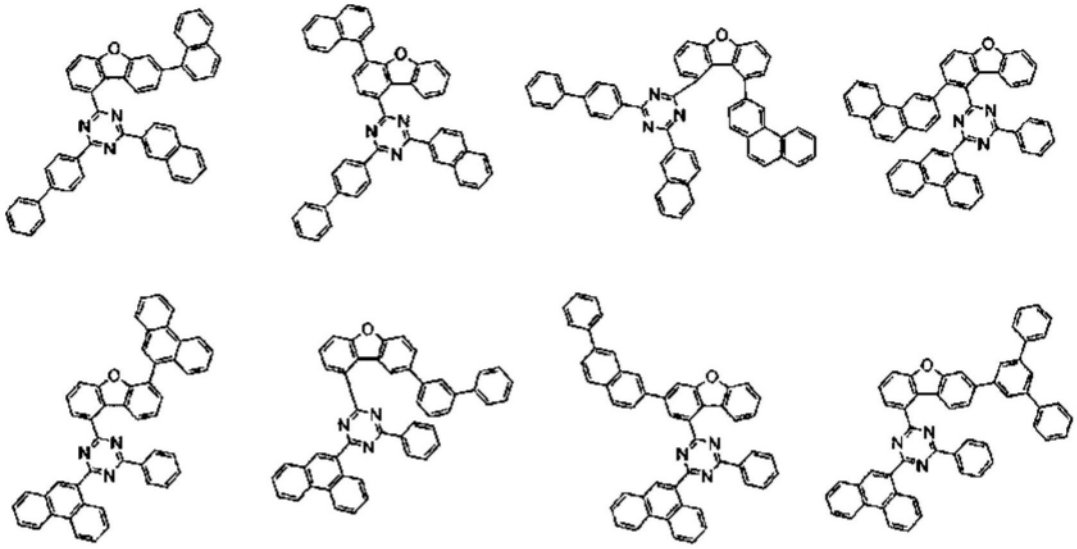







[0126]

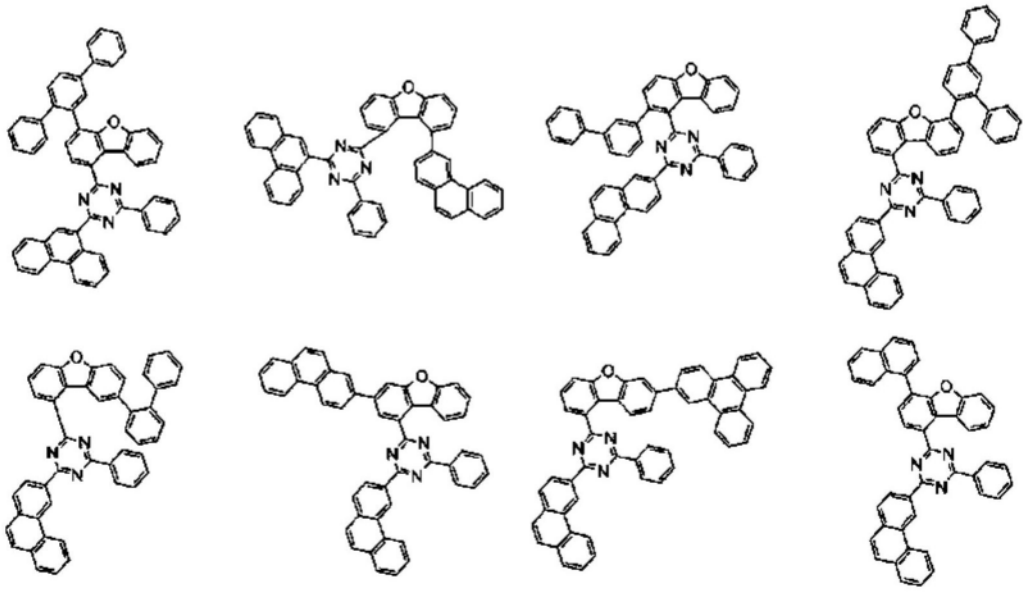









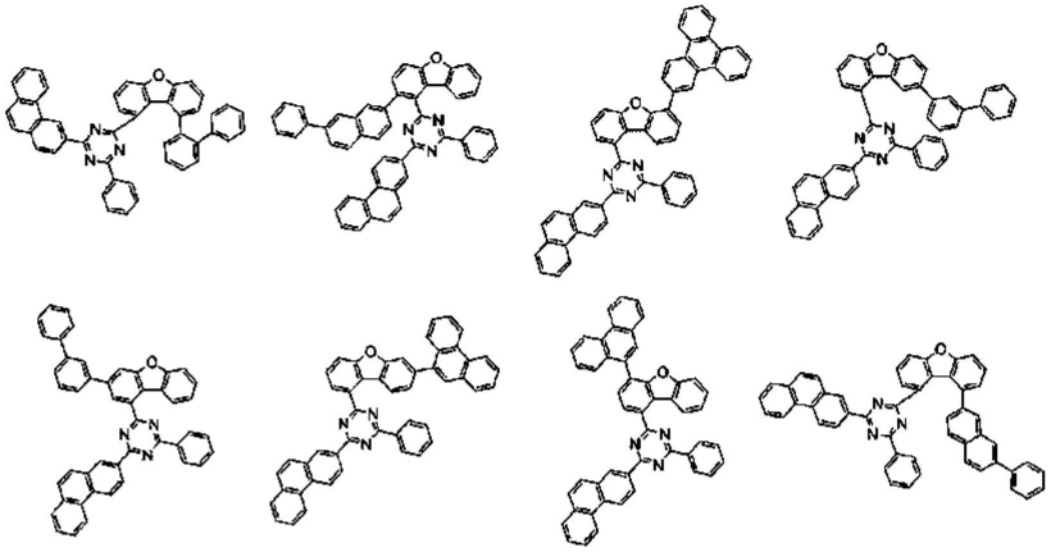
[0127]



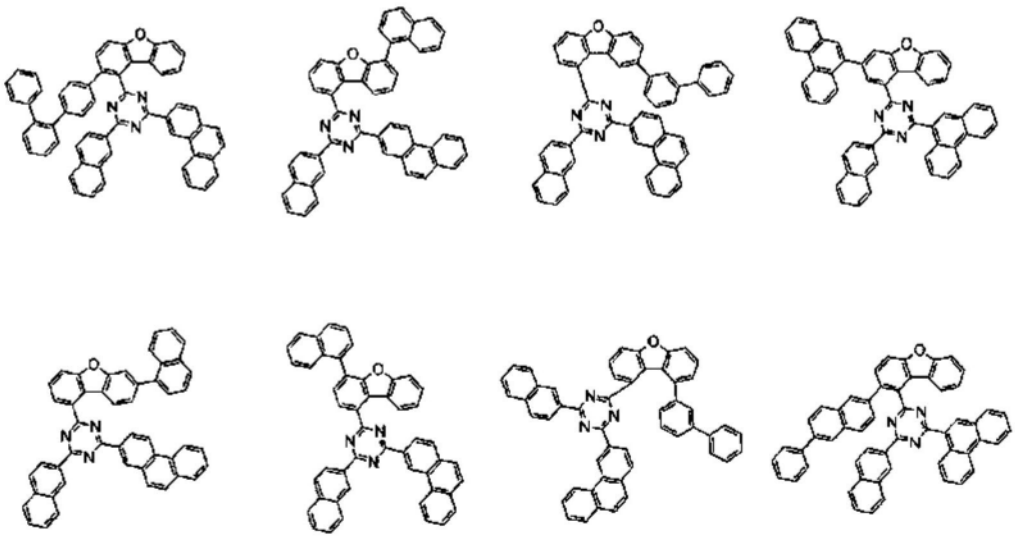


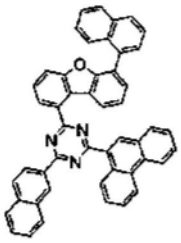
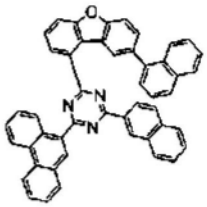
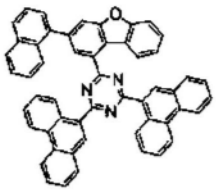
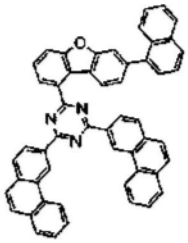
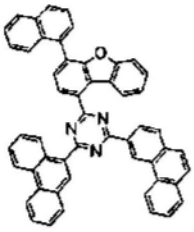
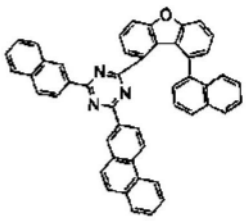
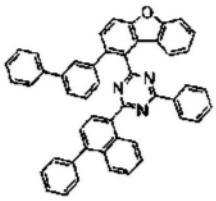
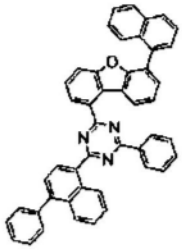
[0128]



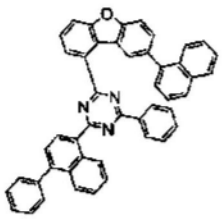
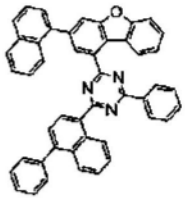
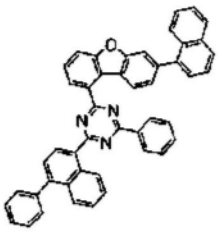
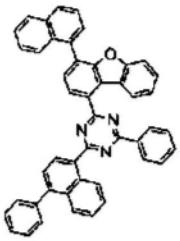
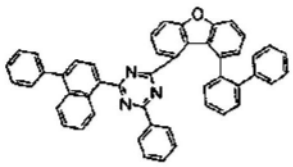
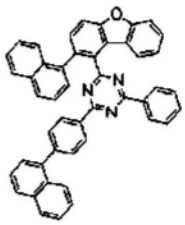
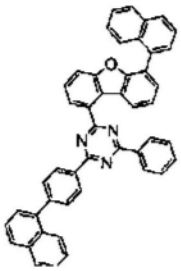
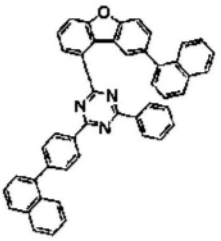


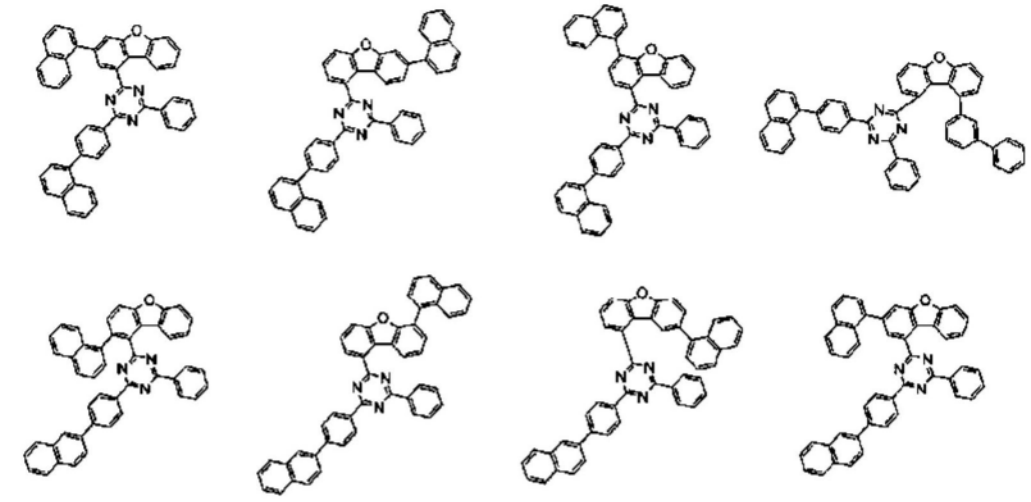
[0129]



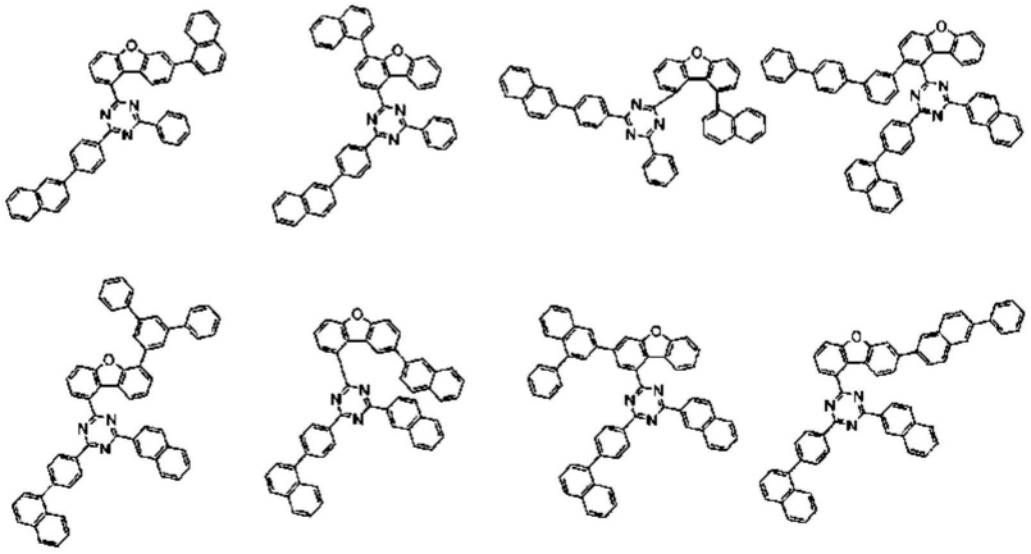









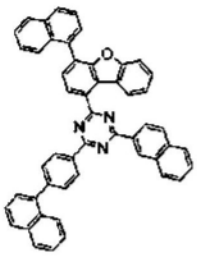
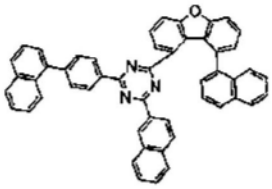
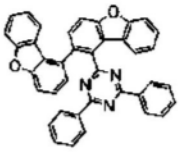
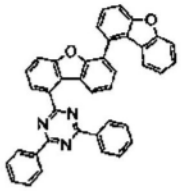
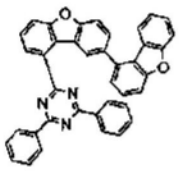
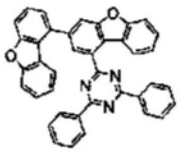
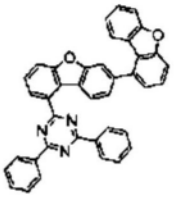
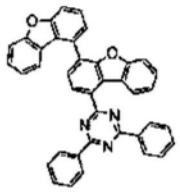
[0130]

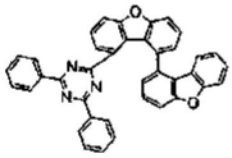
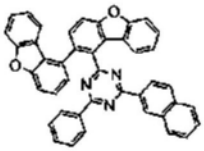
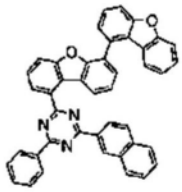
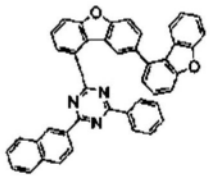
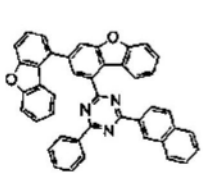
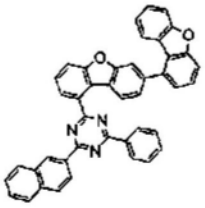
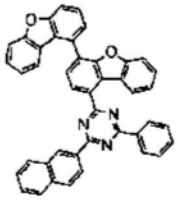
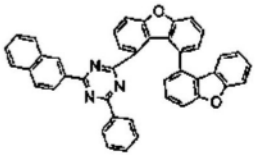


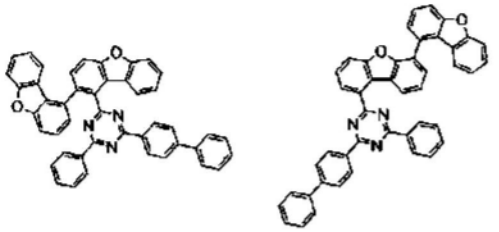
[0131]

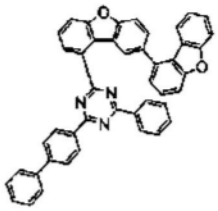
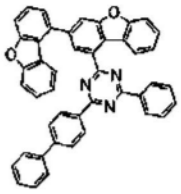
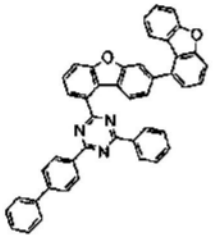
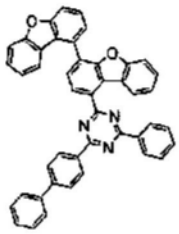
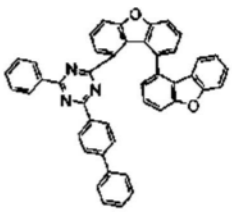
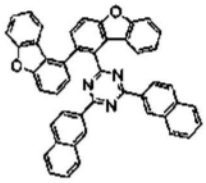


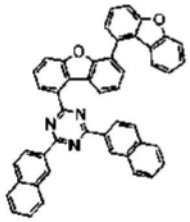
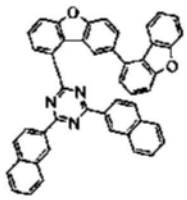
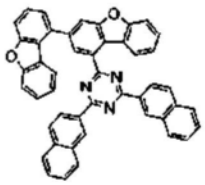
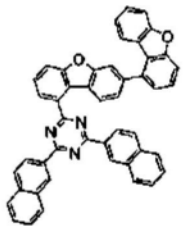
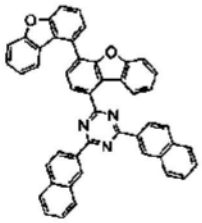
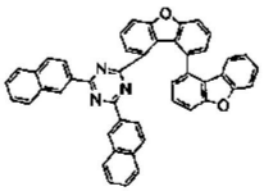
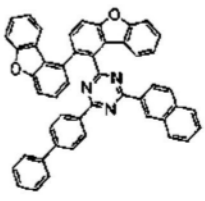
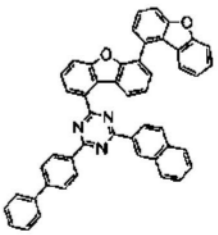
[0132]

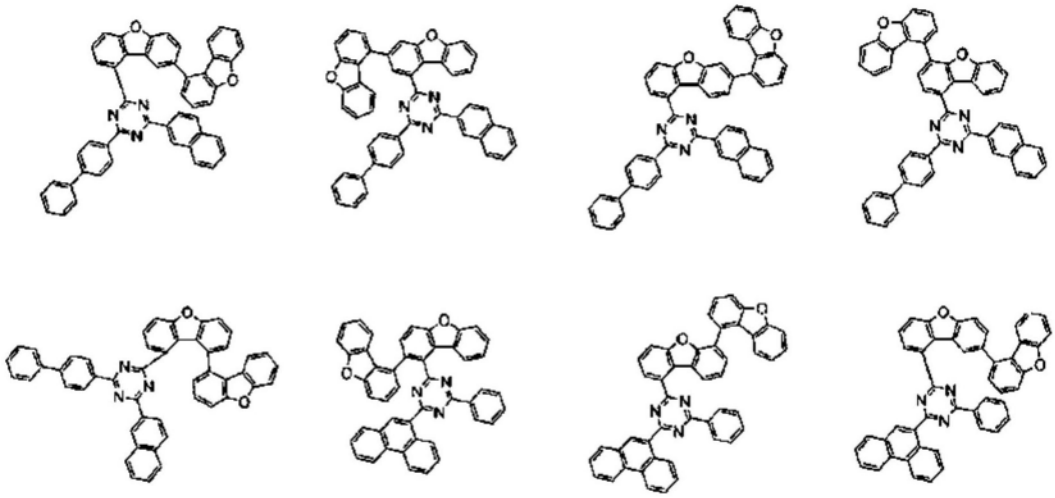











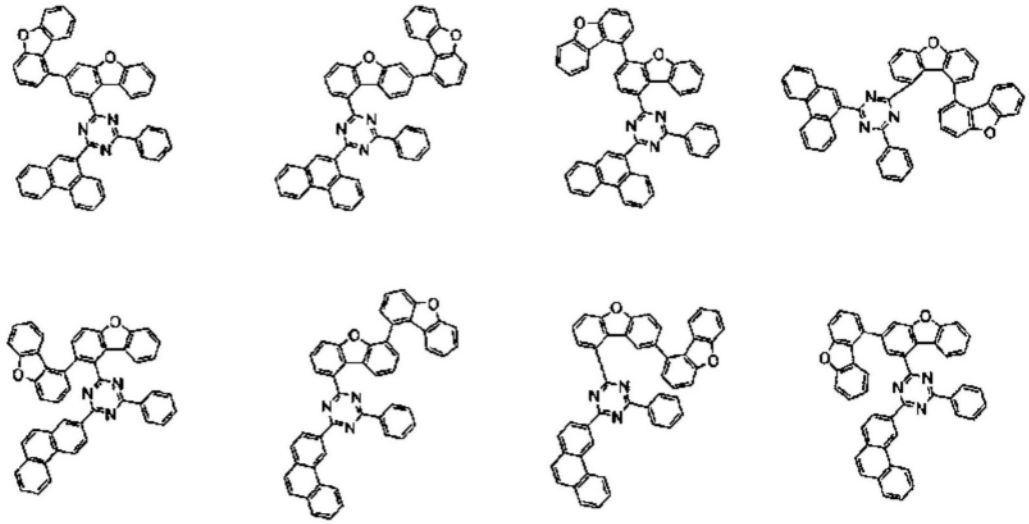







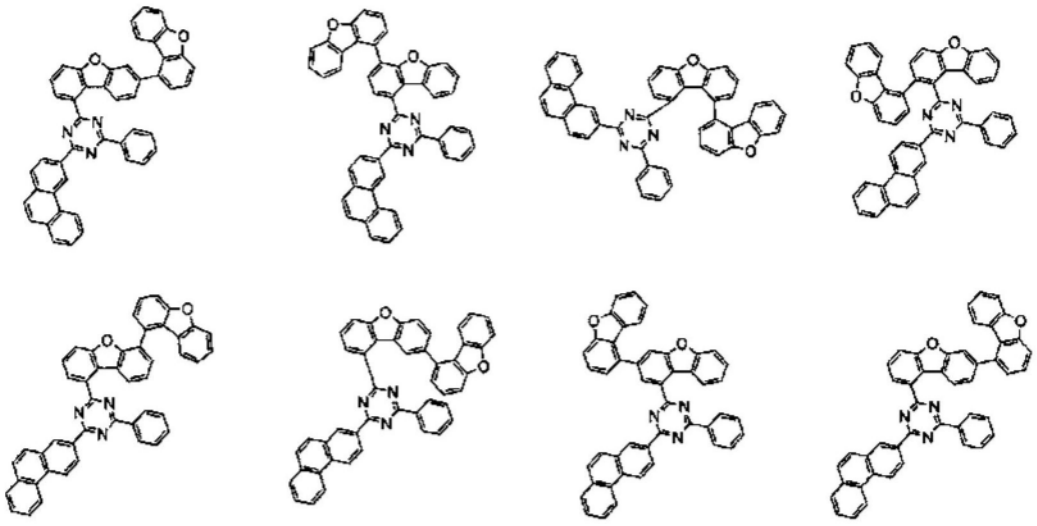
[0133]

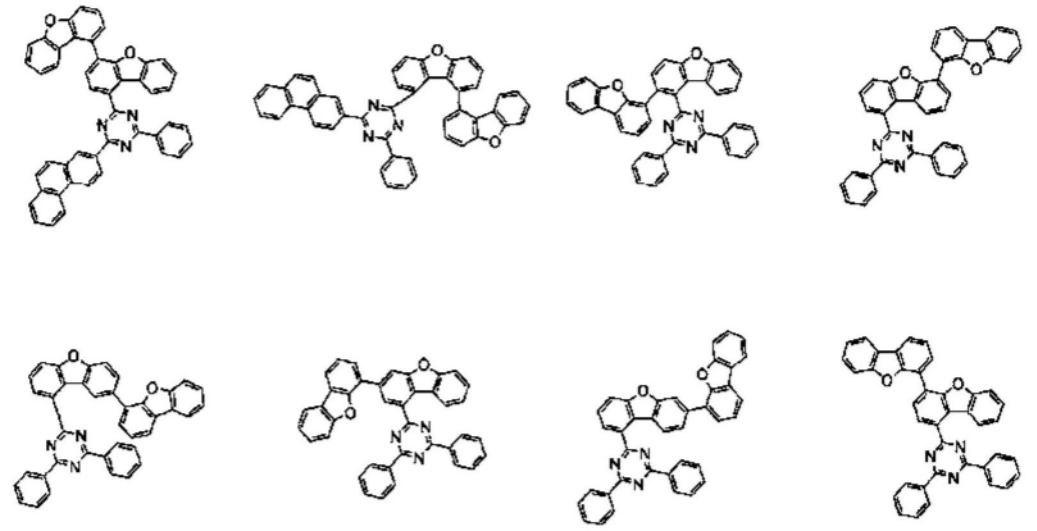


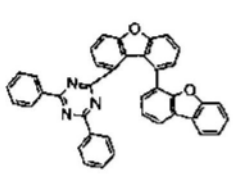
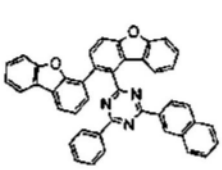
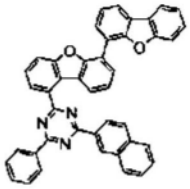
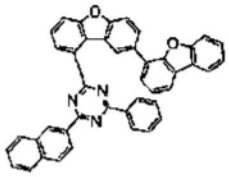
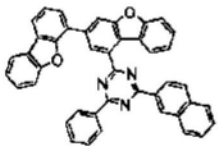
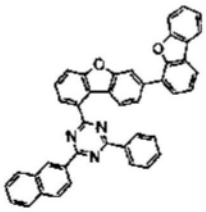
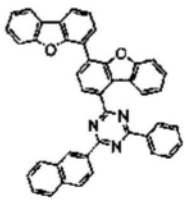
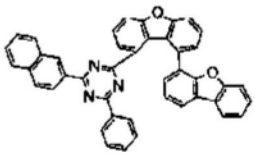
[0134]



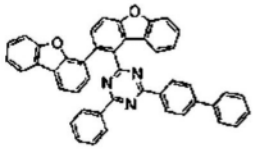
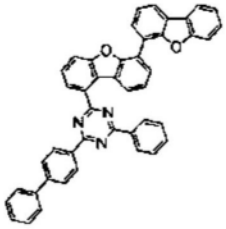
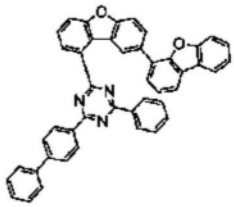
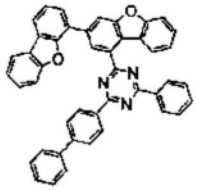
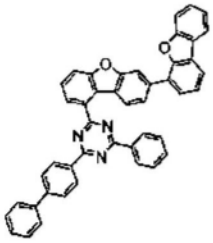
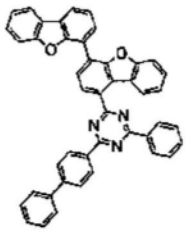
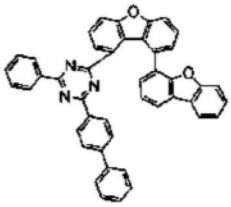
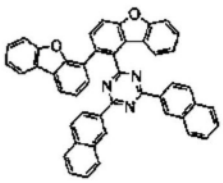


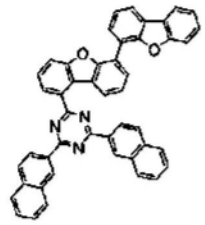
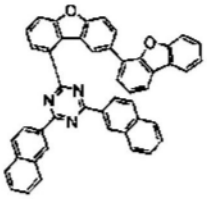
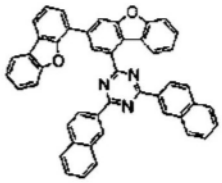
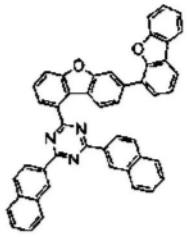
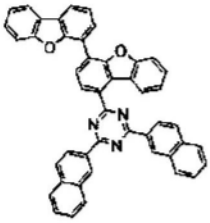
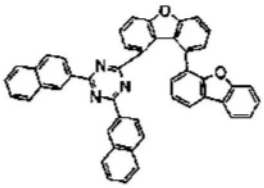
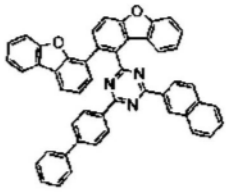
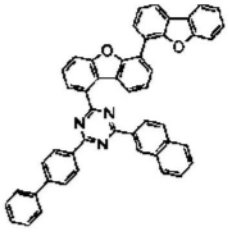
[0135]



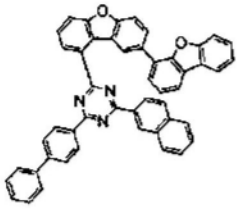
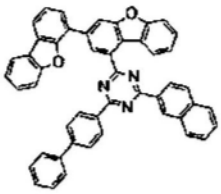
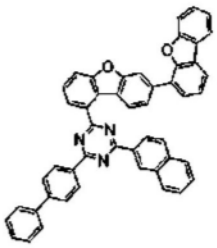
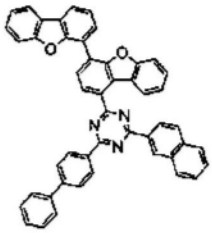
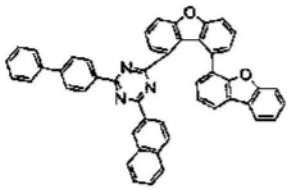
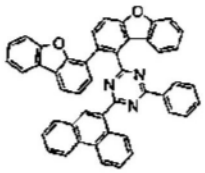
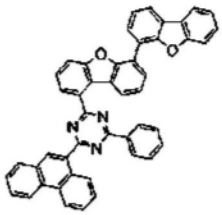
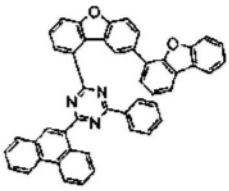









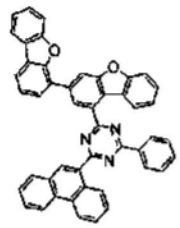
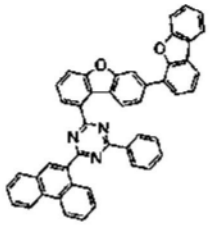
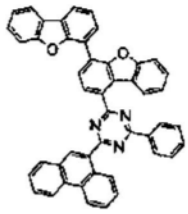
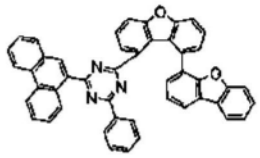
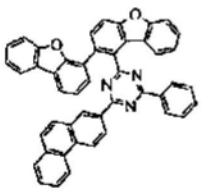
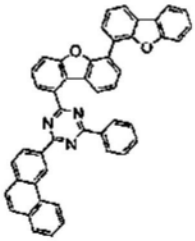
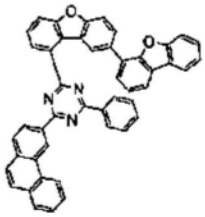
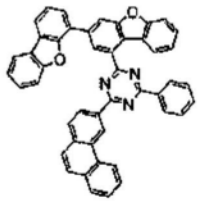
[0136]

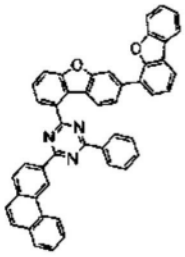
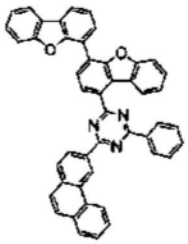
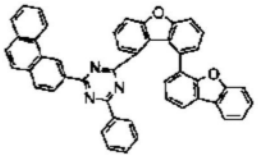
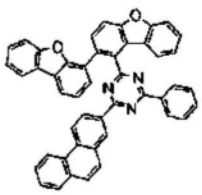
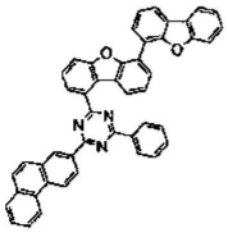
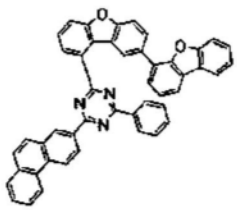
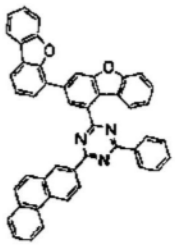
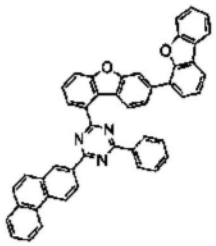









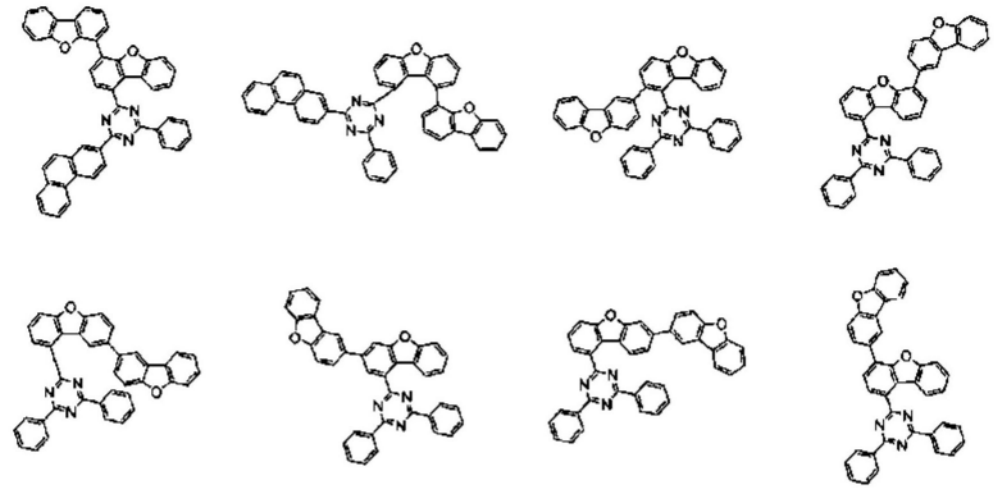
[0137]

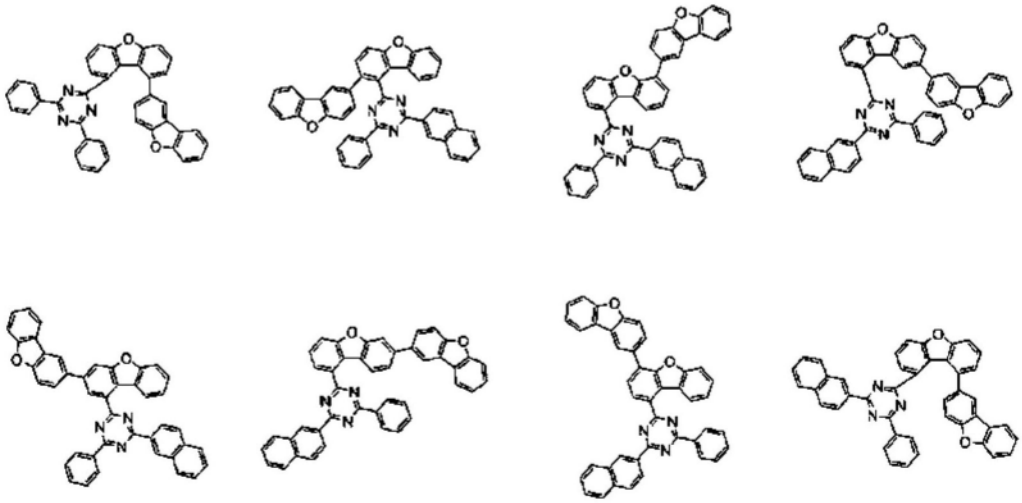









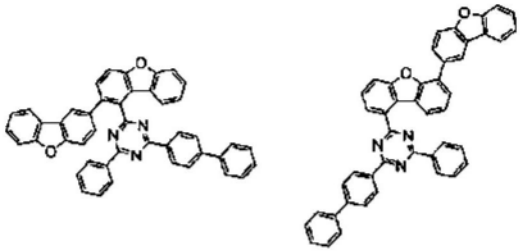
[0138]

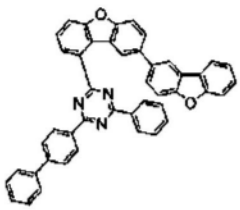
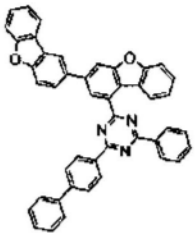
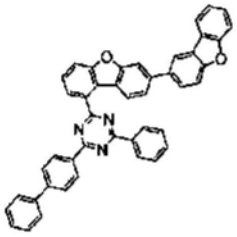
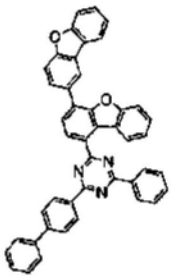
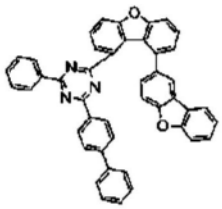
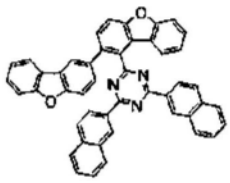











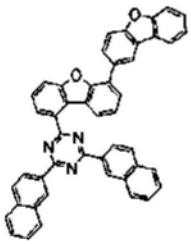
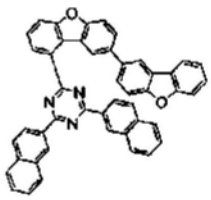
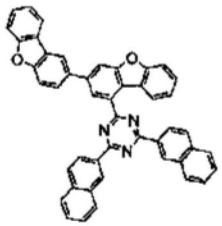
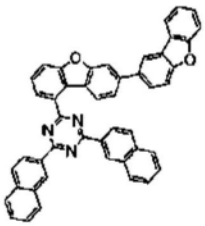
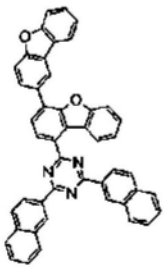
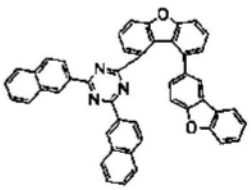
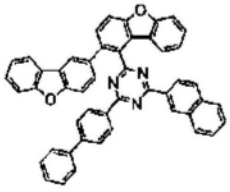
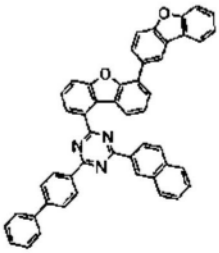
[0139]

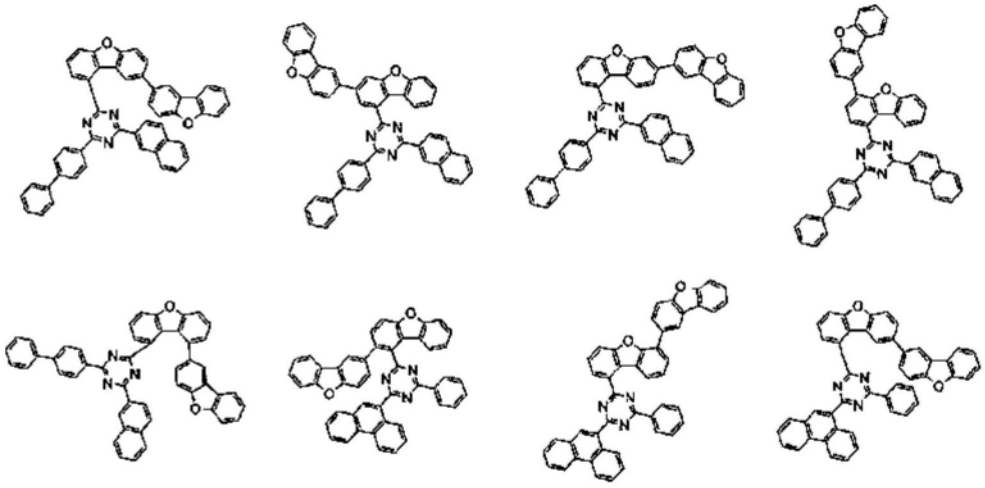




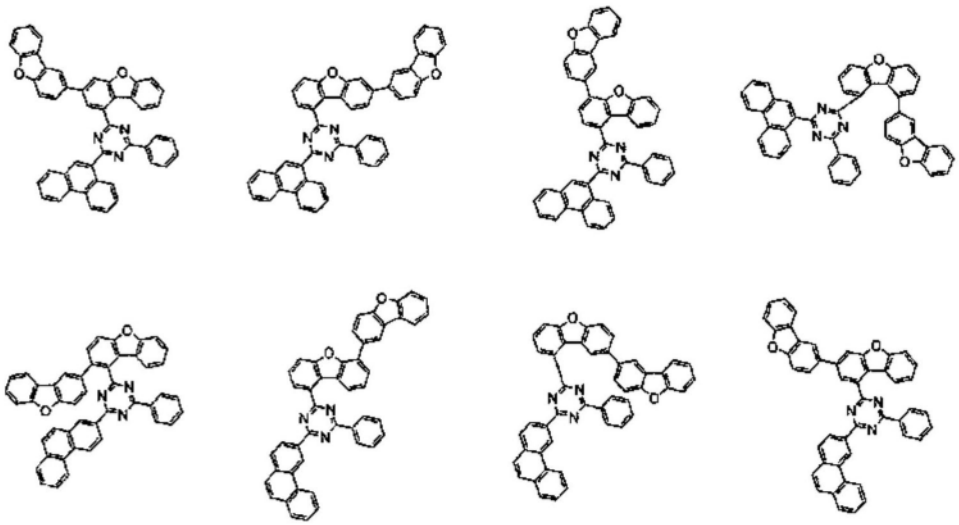







[0140]

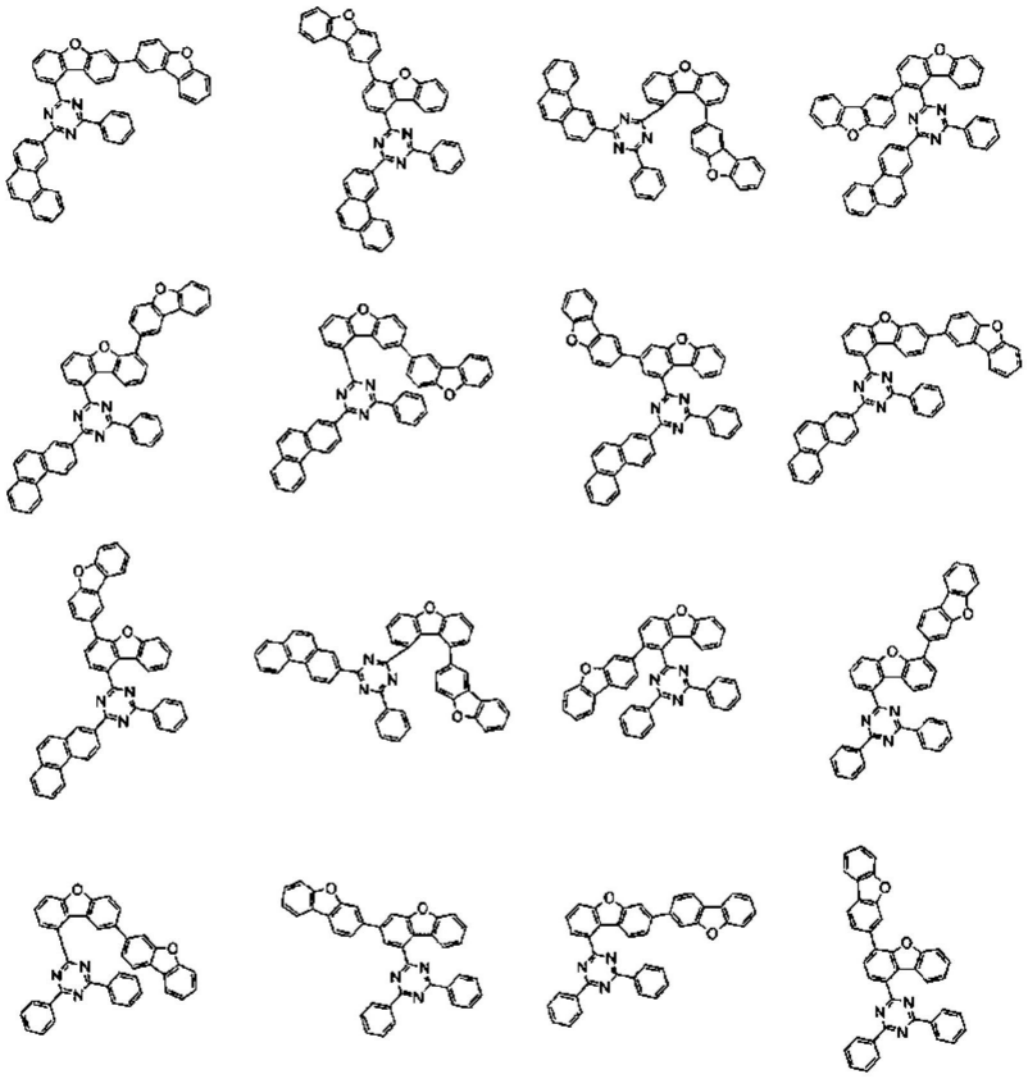









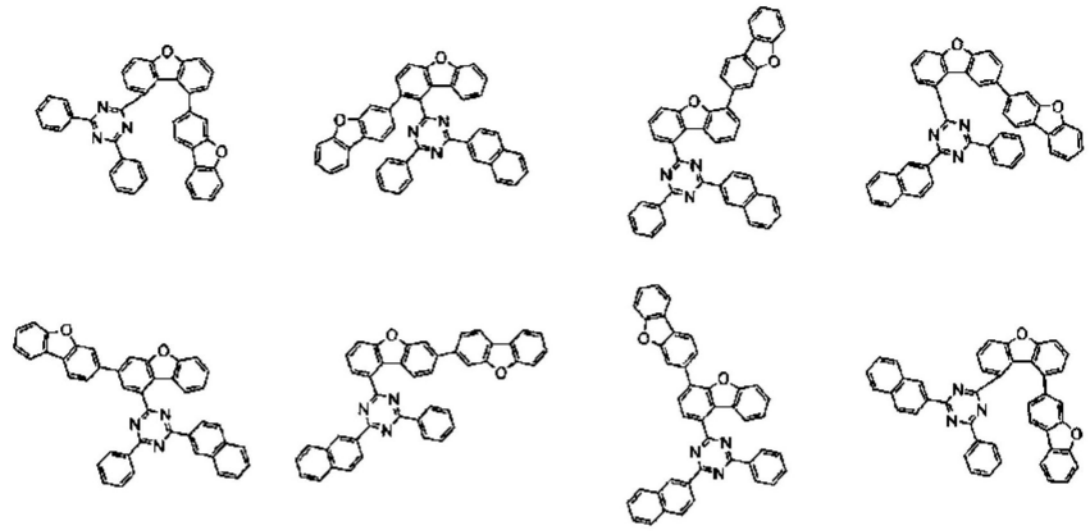


[0141]

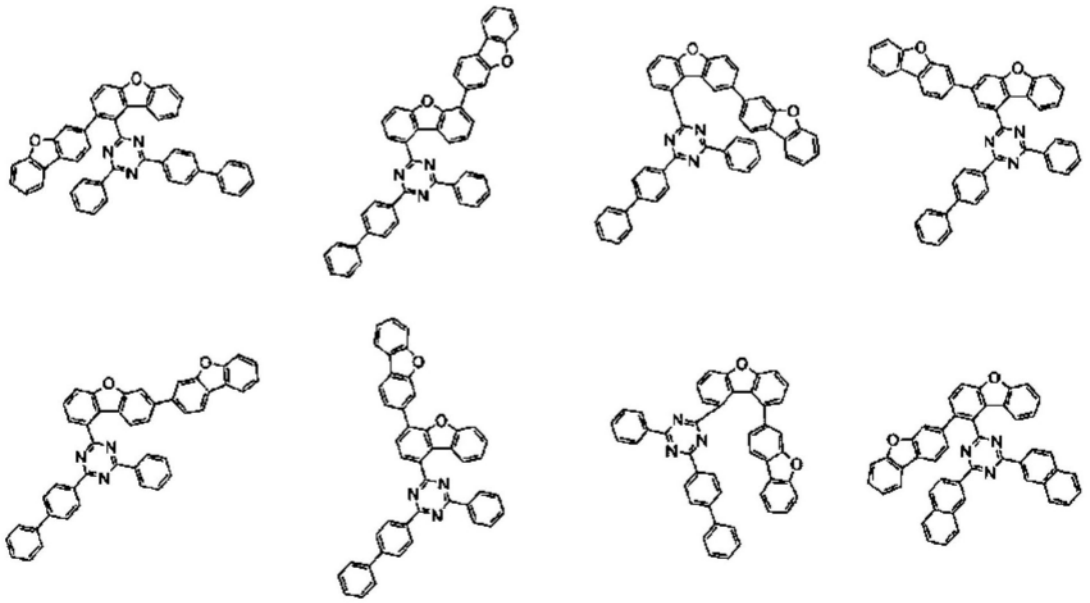


[0142]

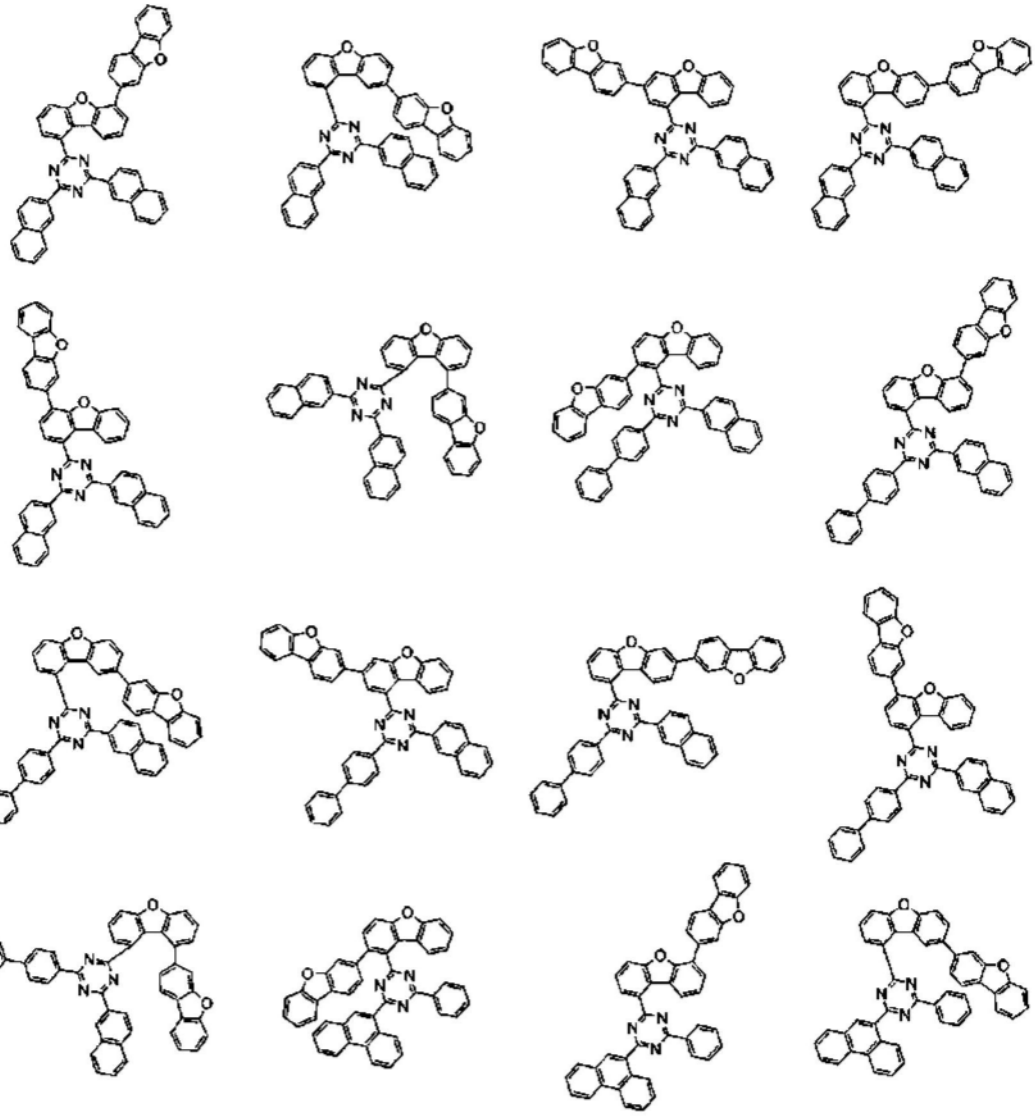




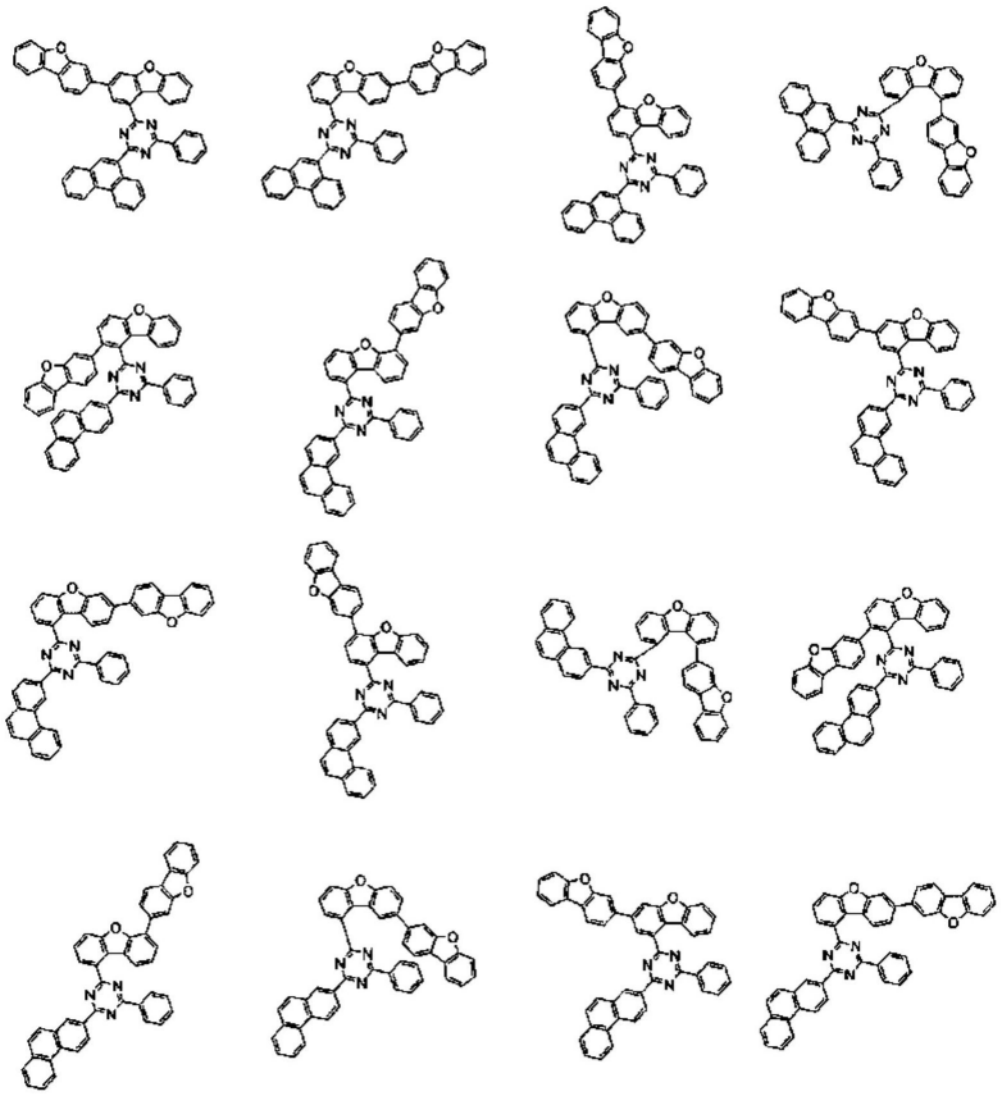
[0143]



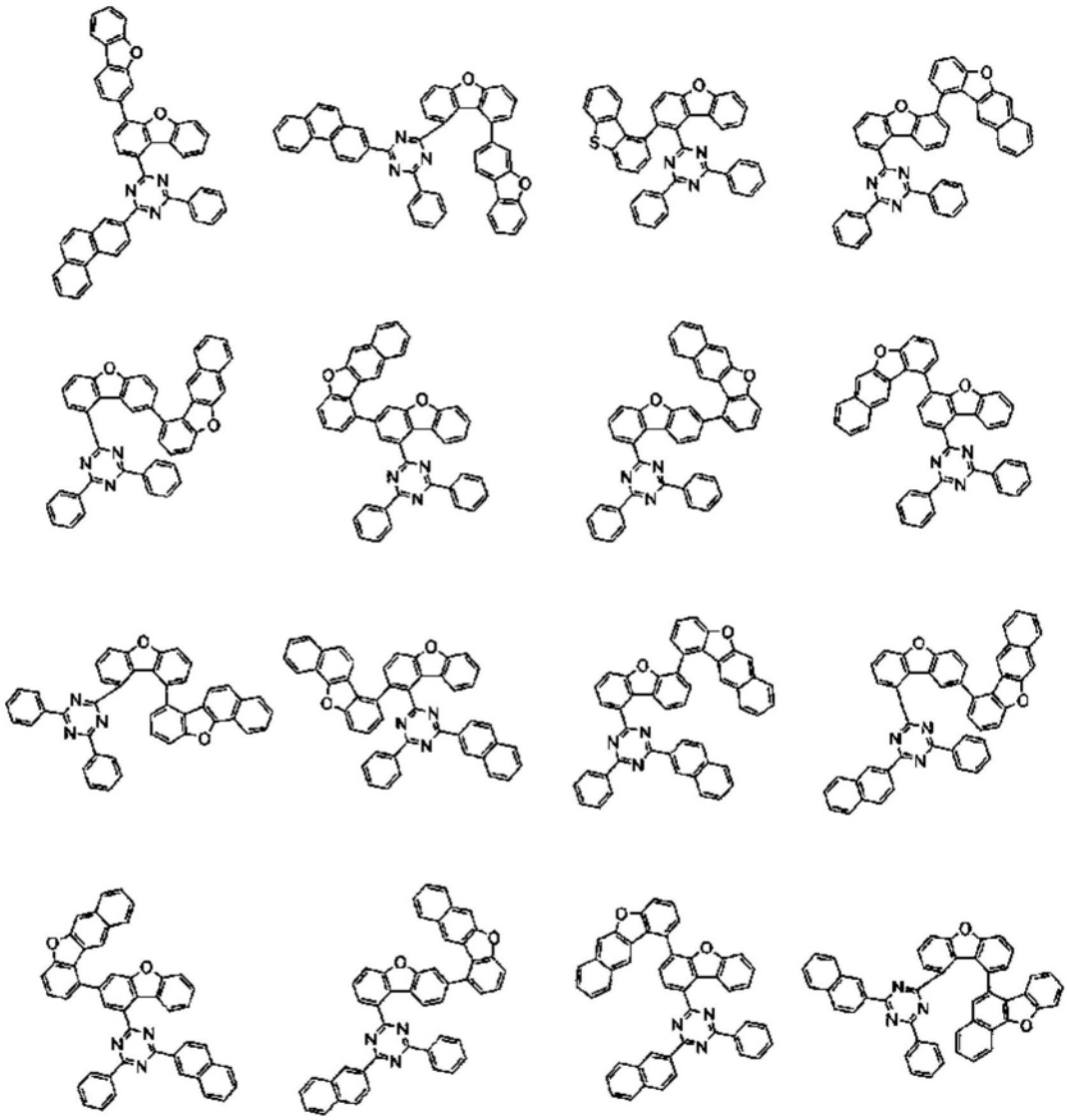
[0144]

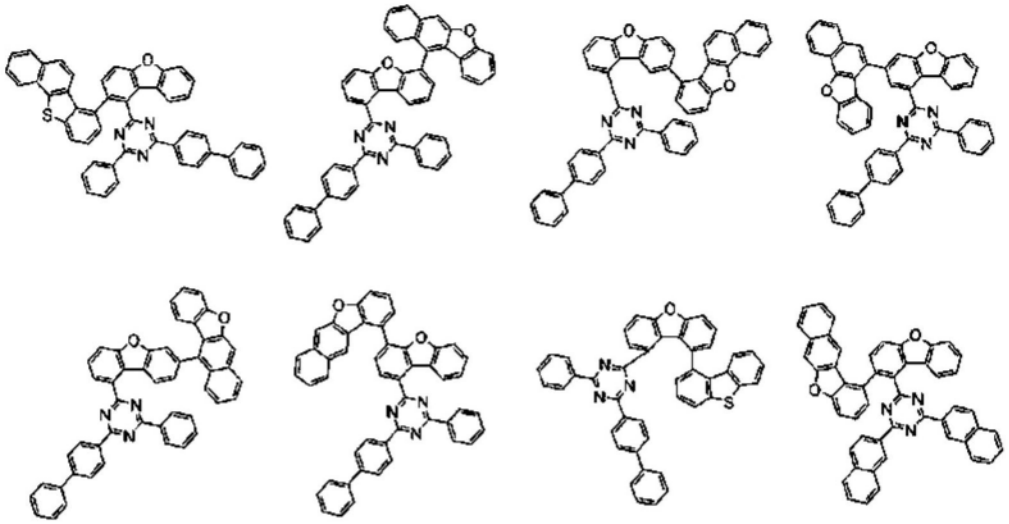


[0145]

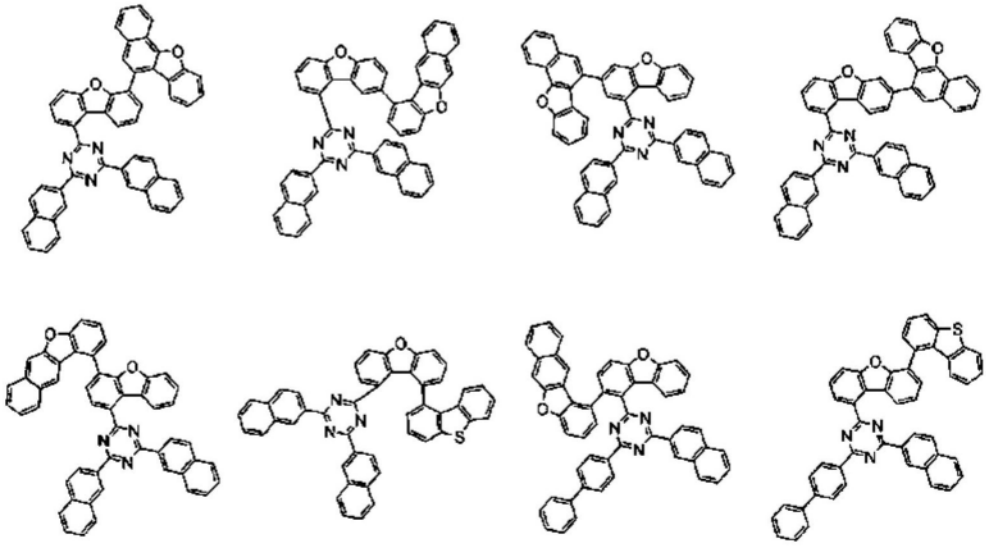


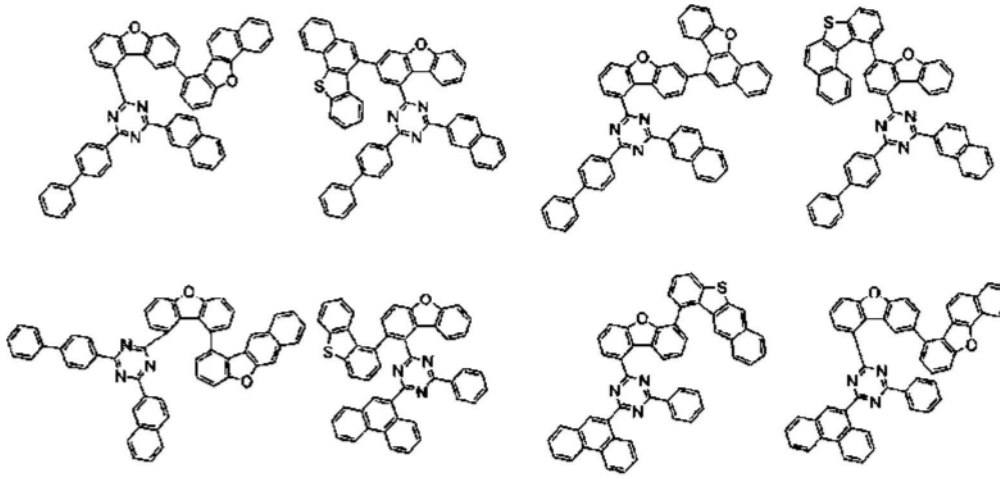
[0146]



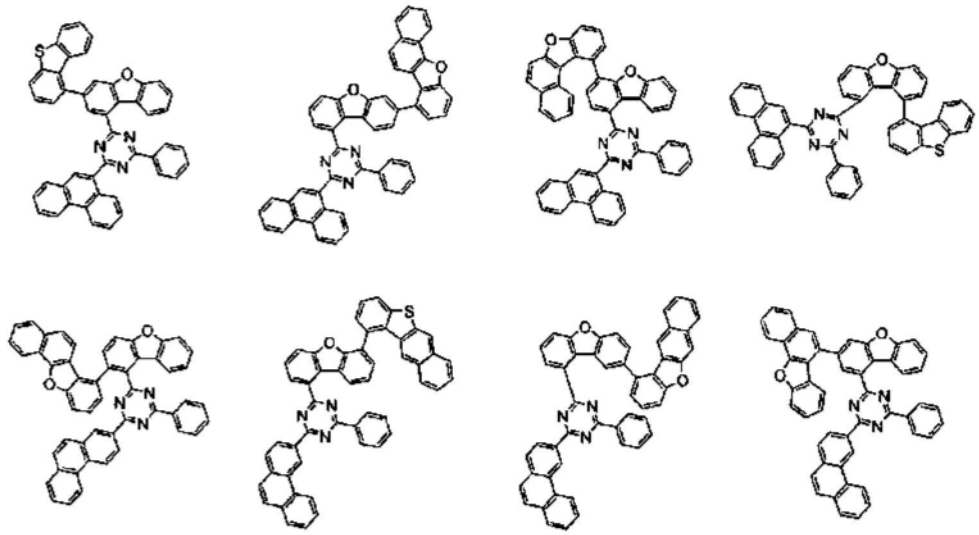


[0147]

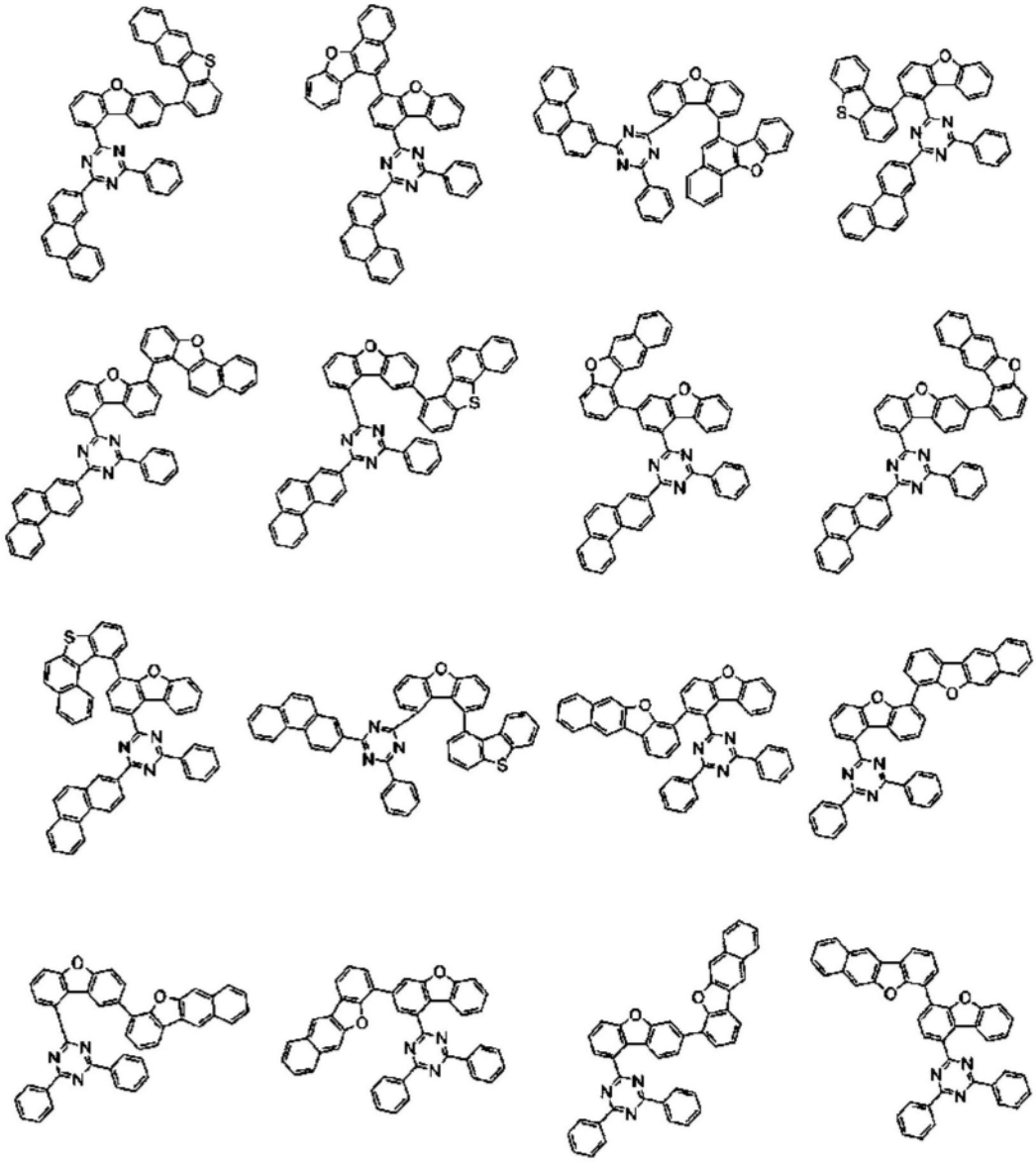


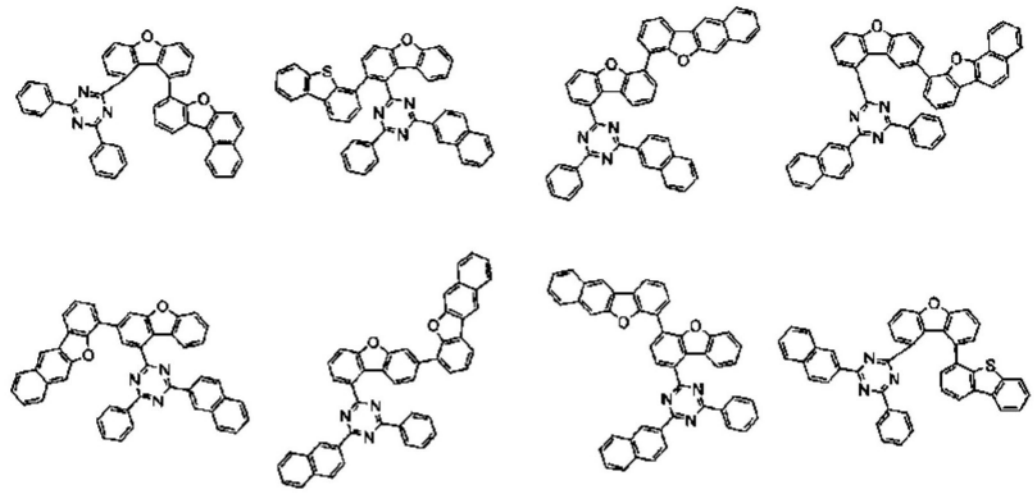


[0148]

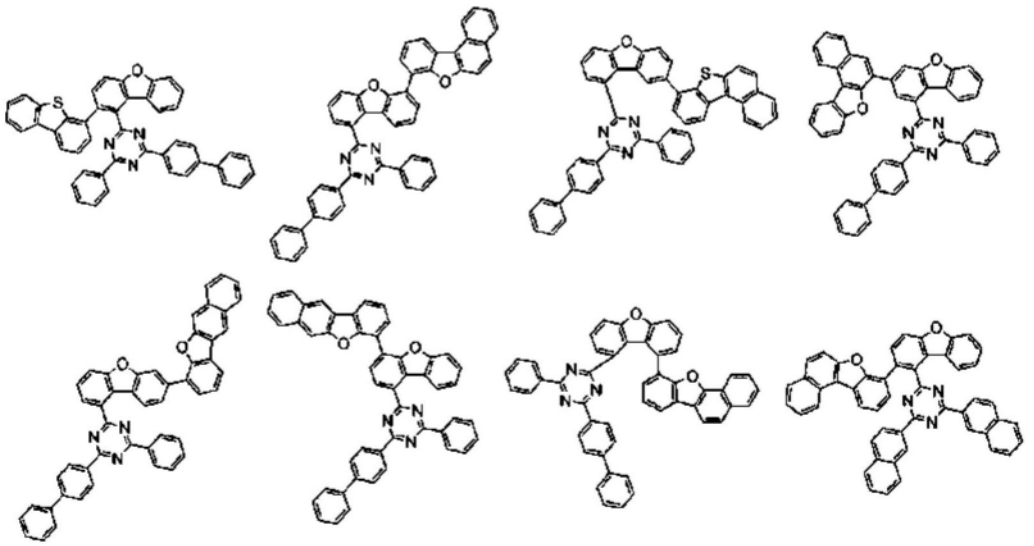


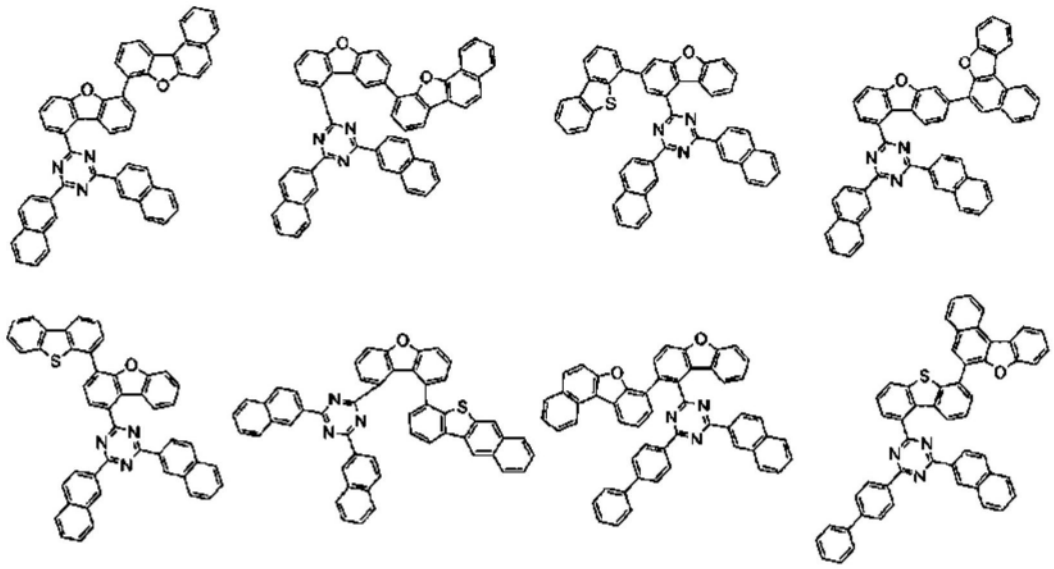
[0149]



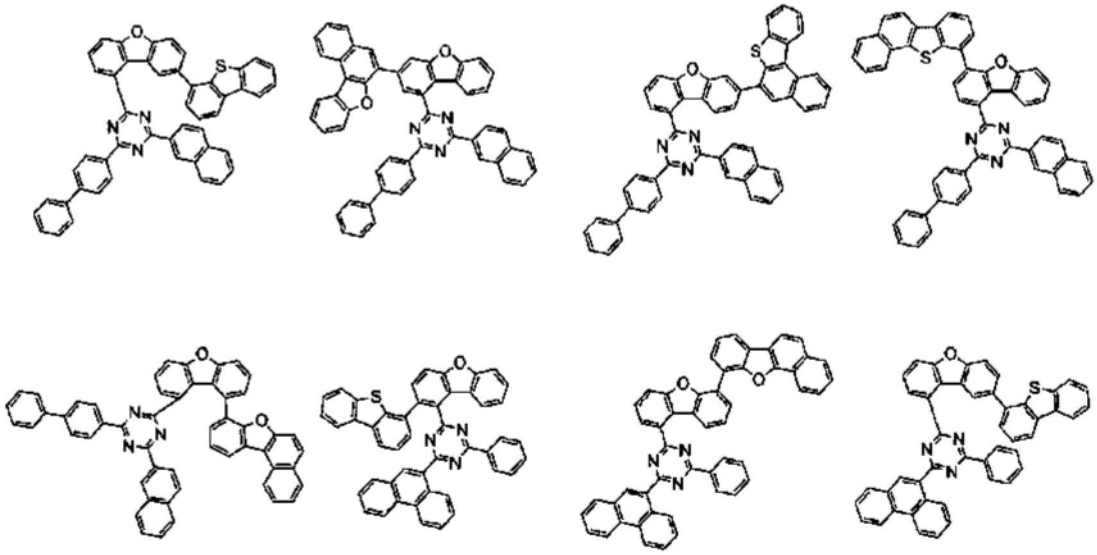


[0150]

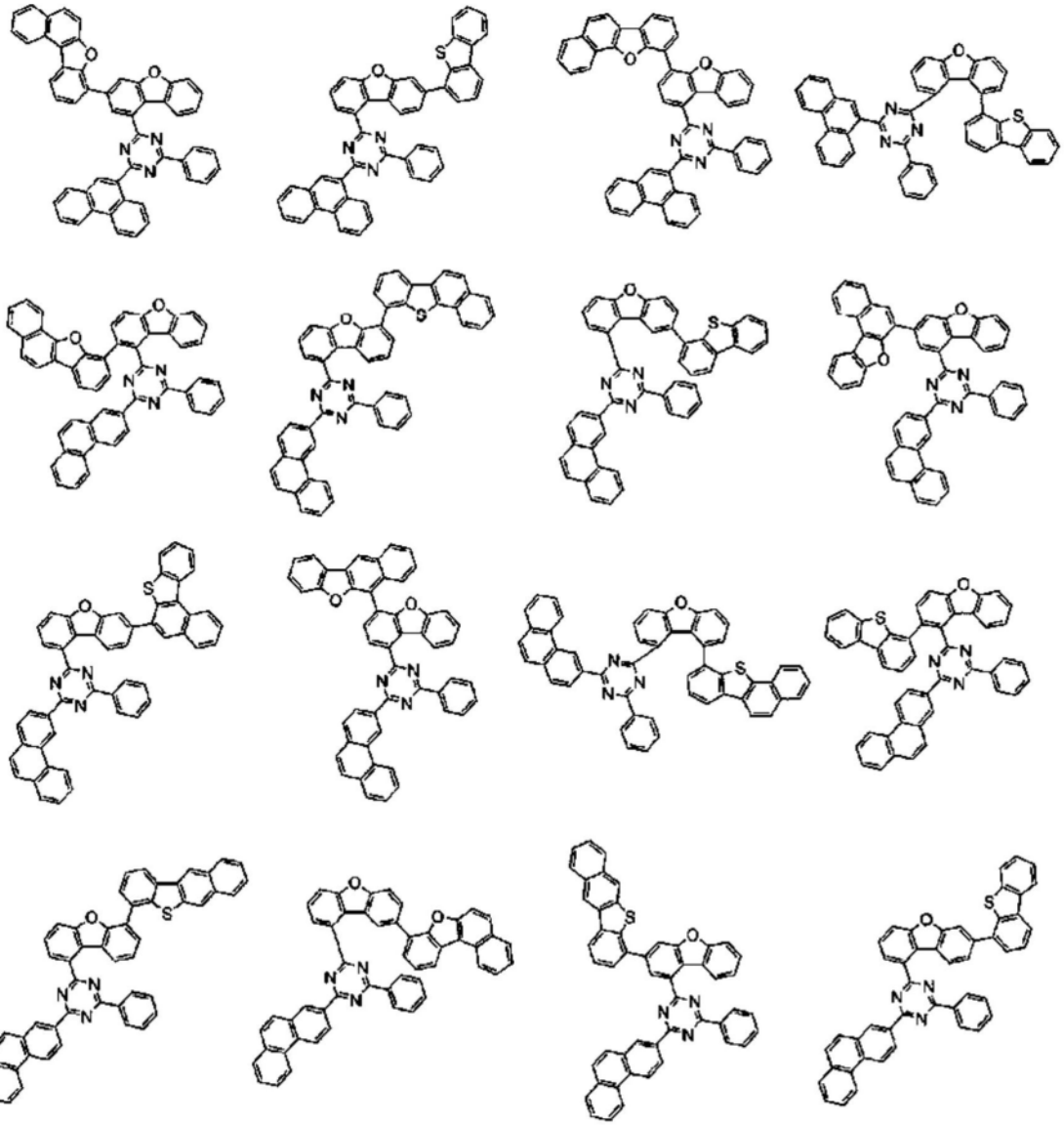


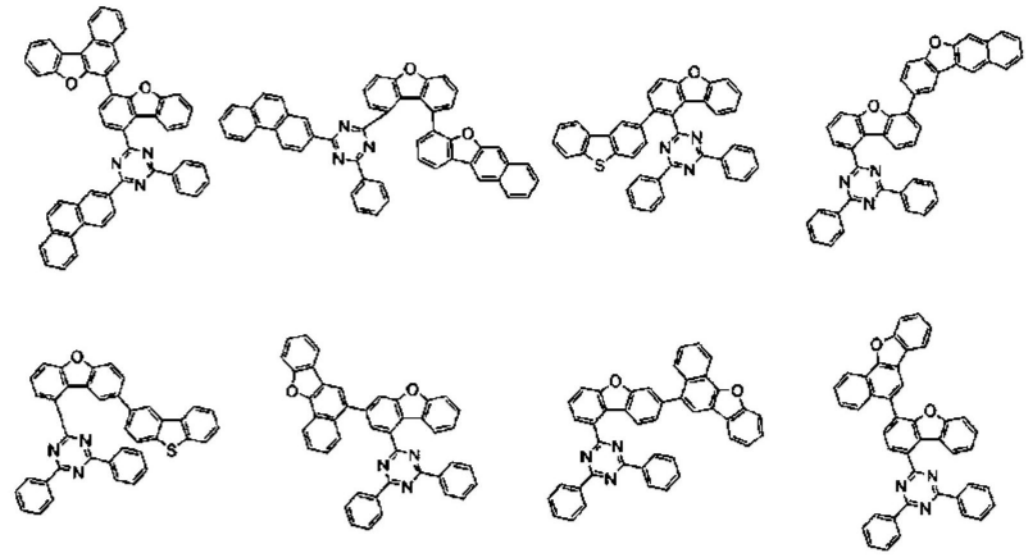


[0151]

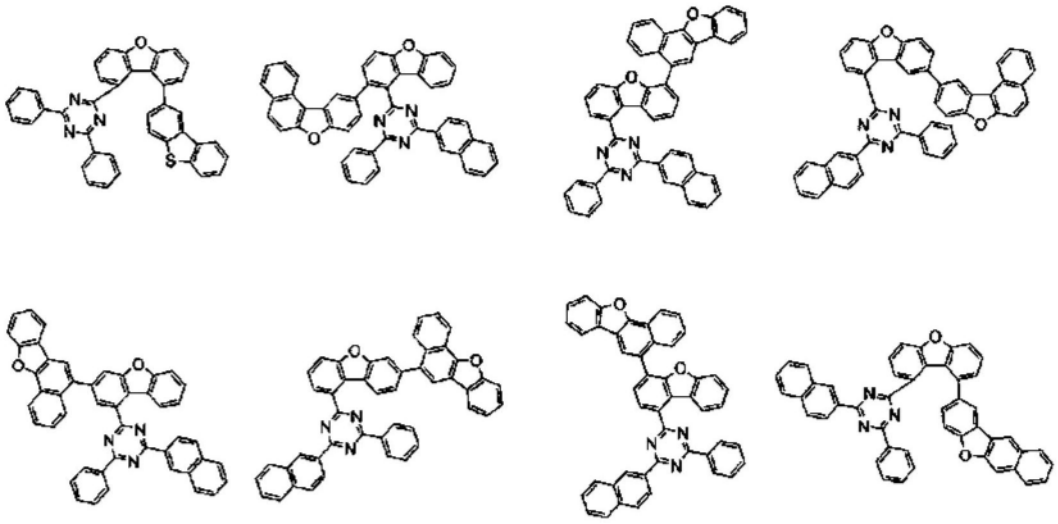


[0152]

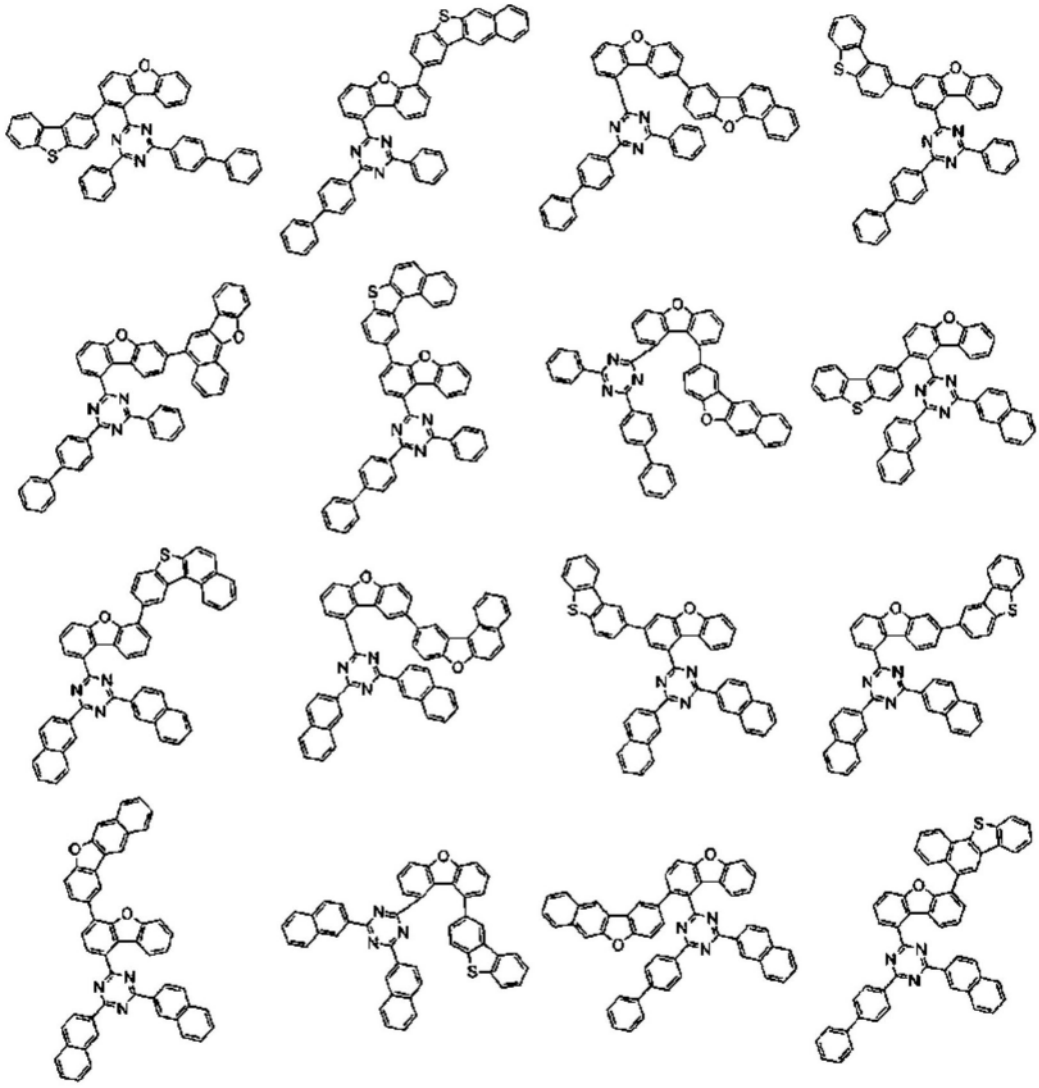


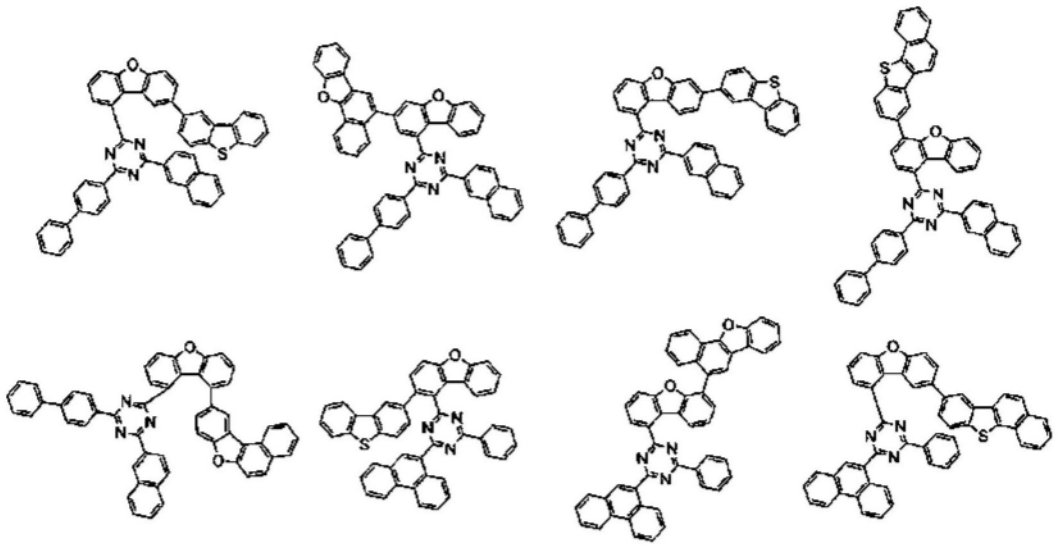


[0153]

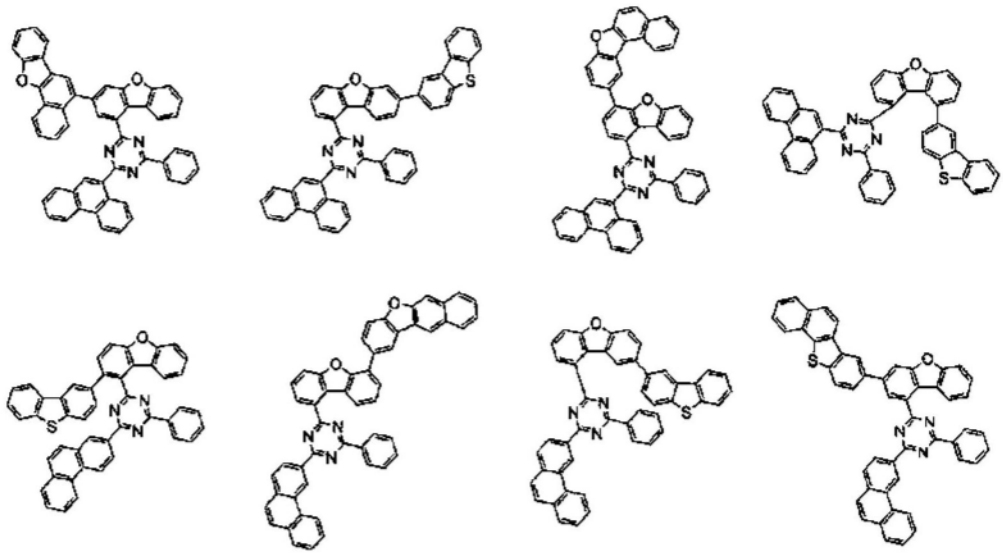


[0154]

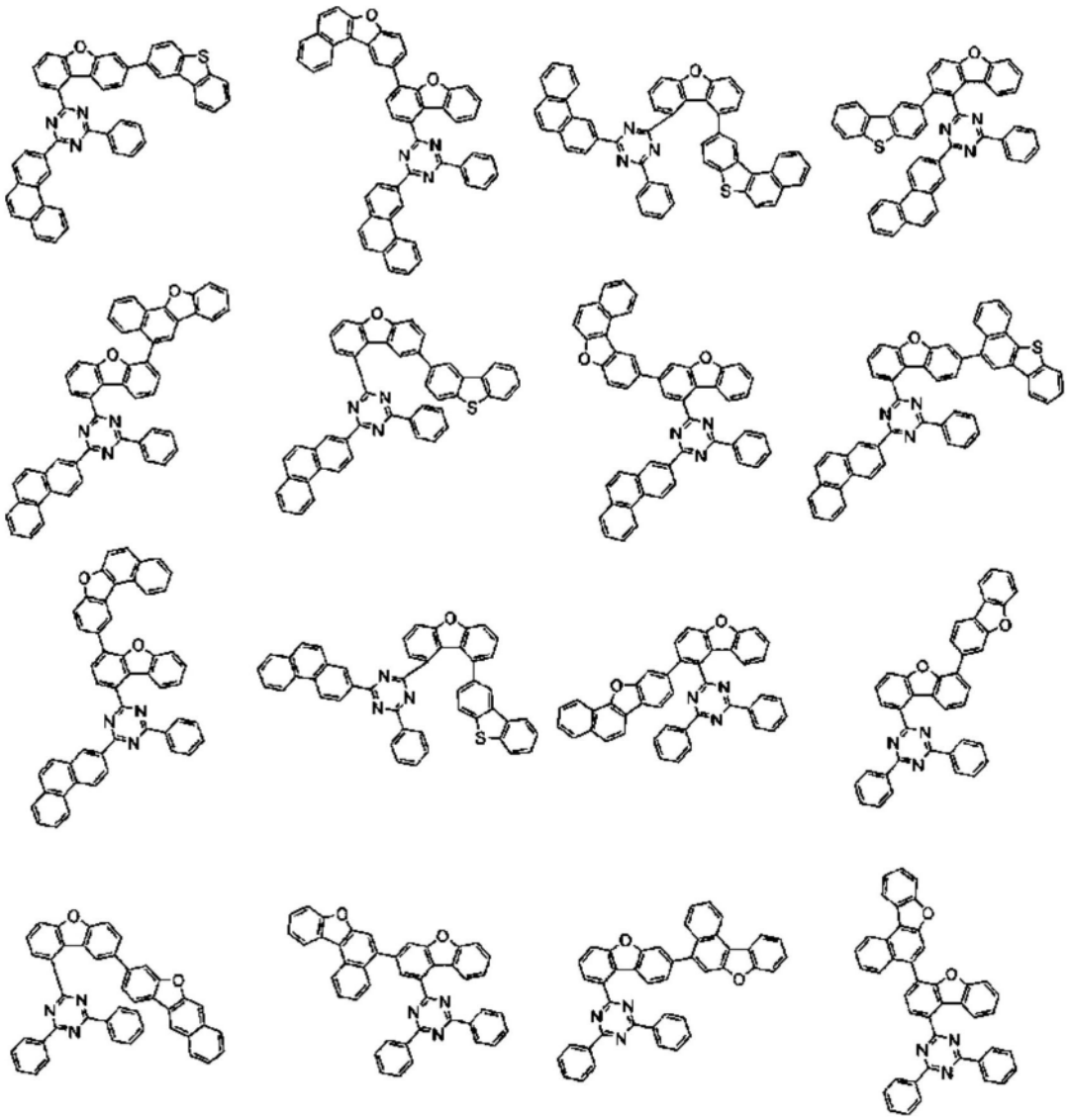


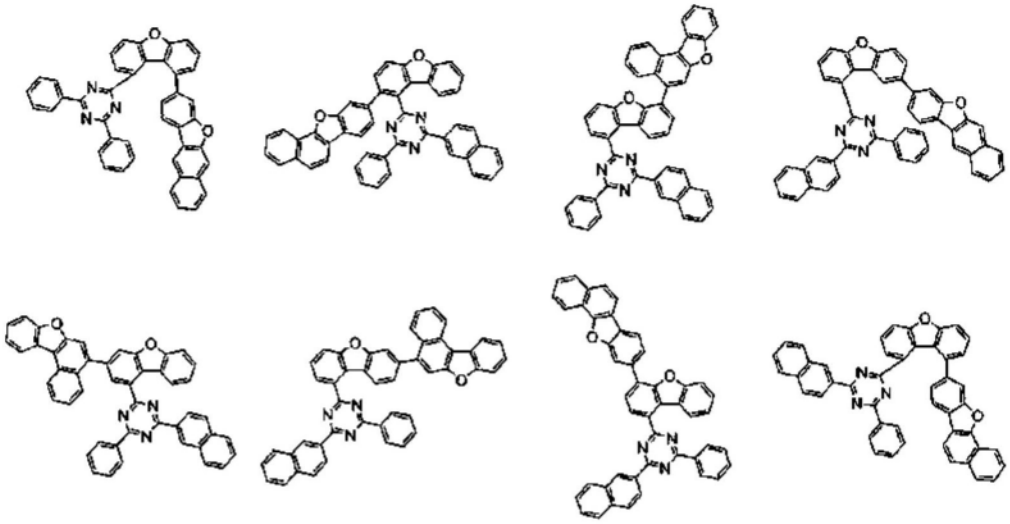


[0155]

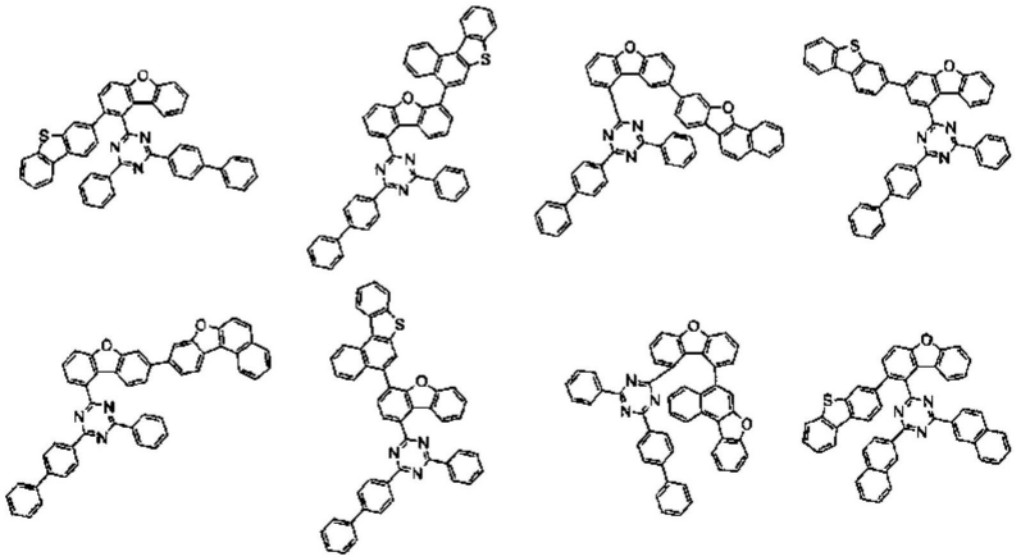


[0156]

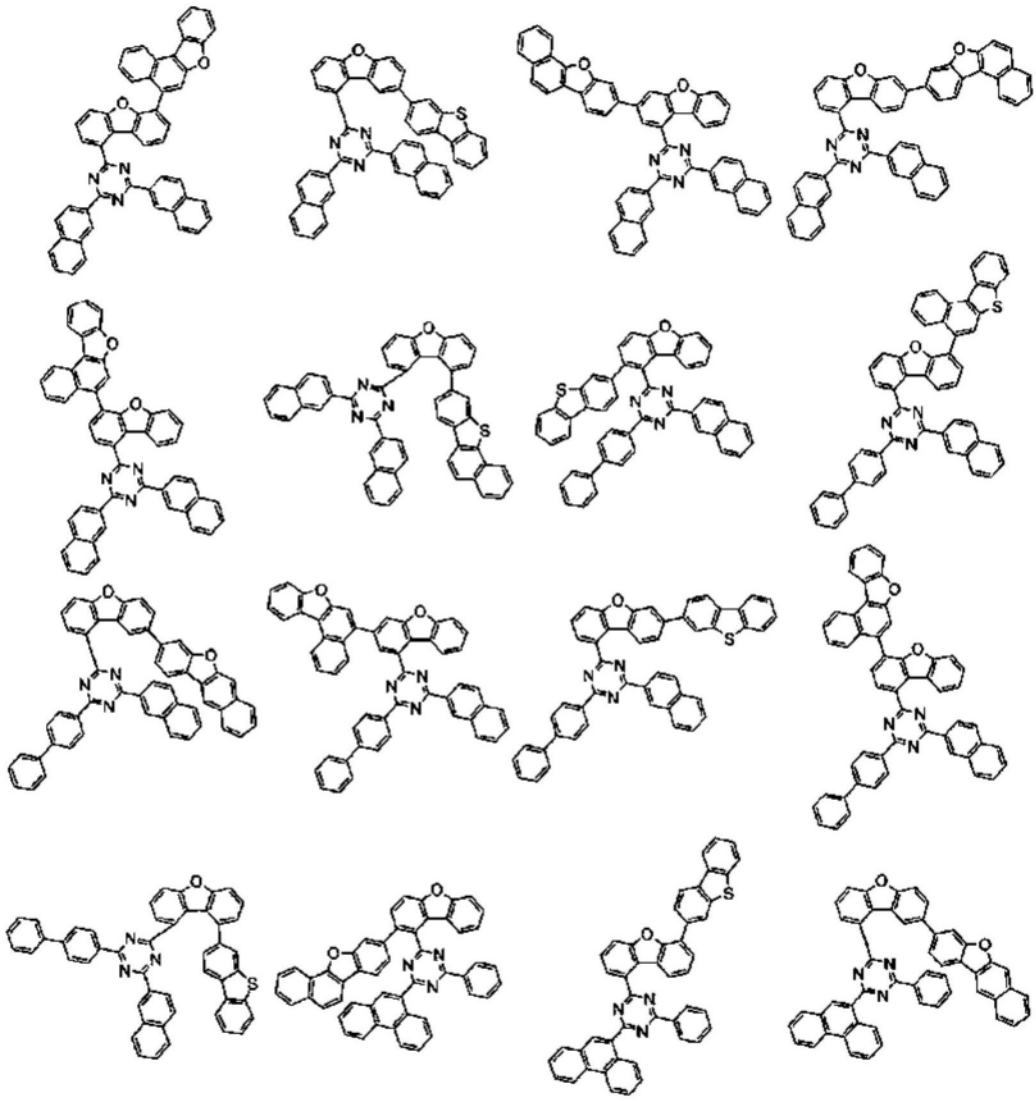


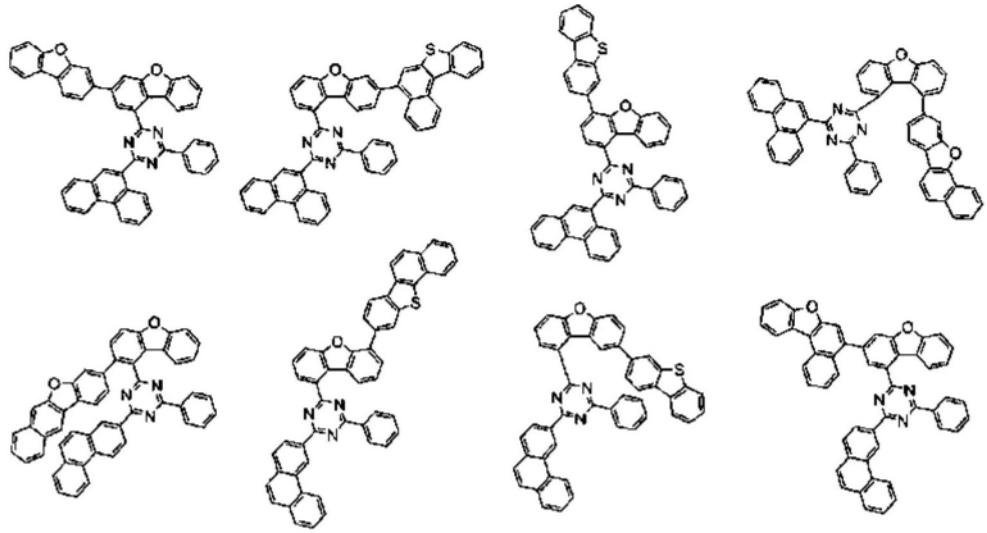


[0157]

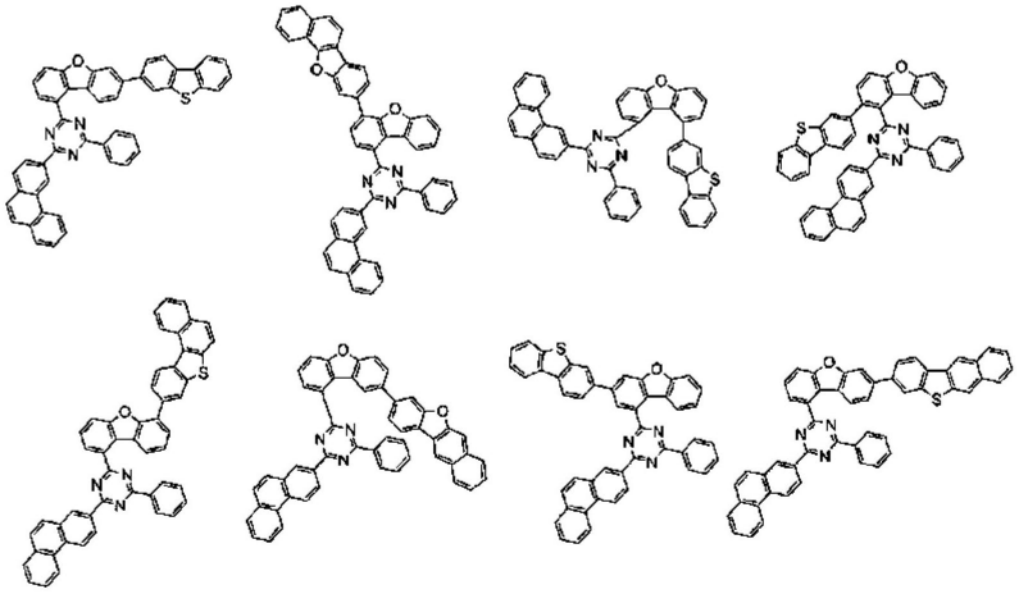


[0158]

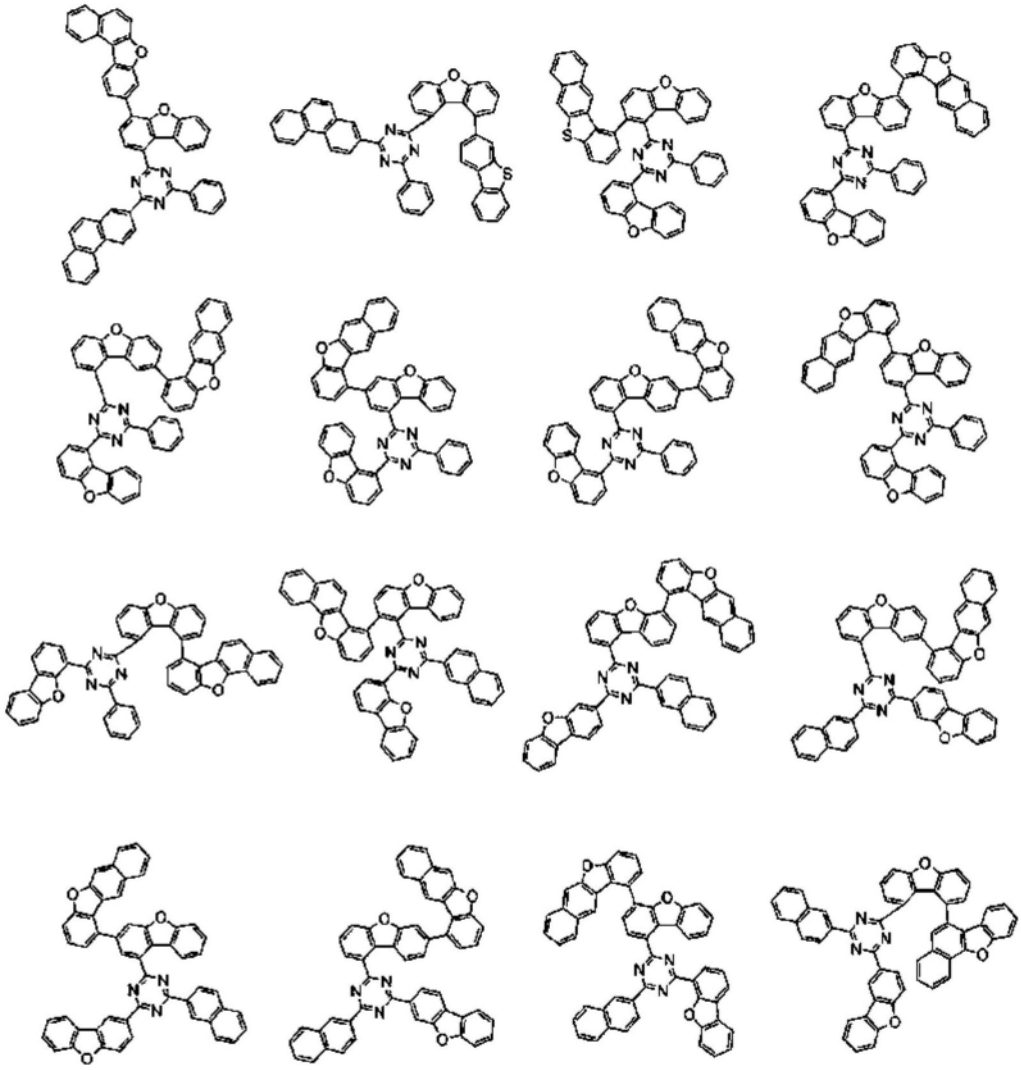


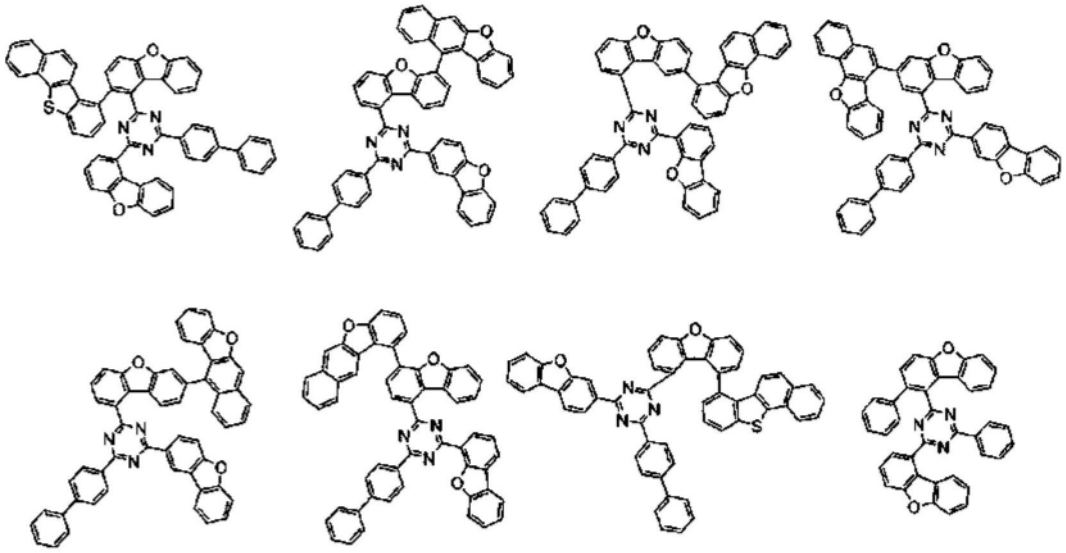


[0159]

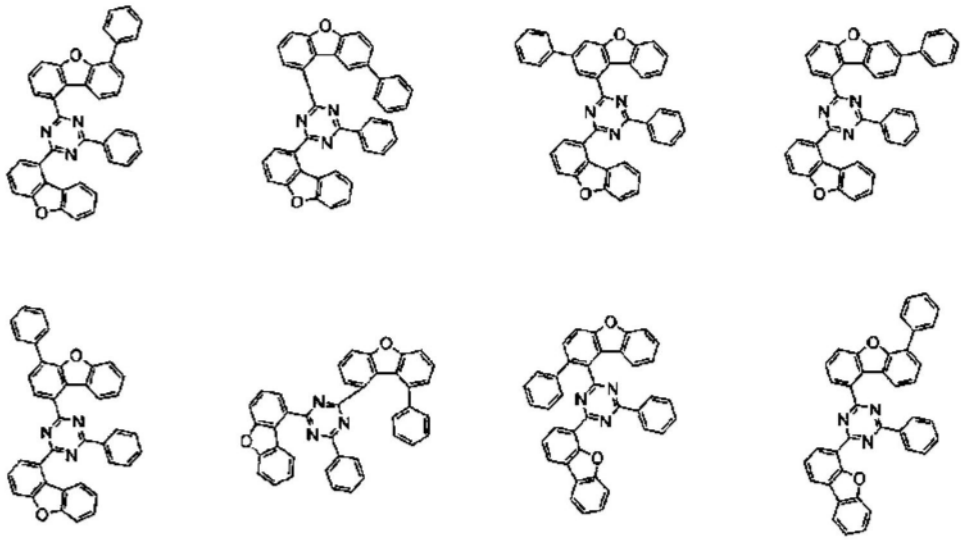


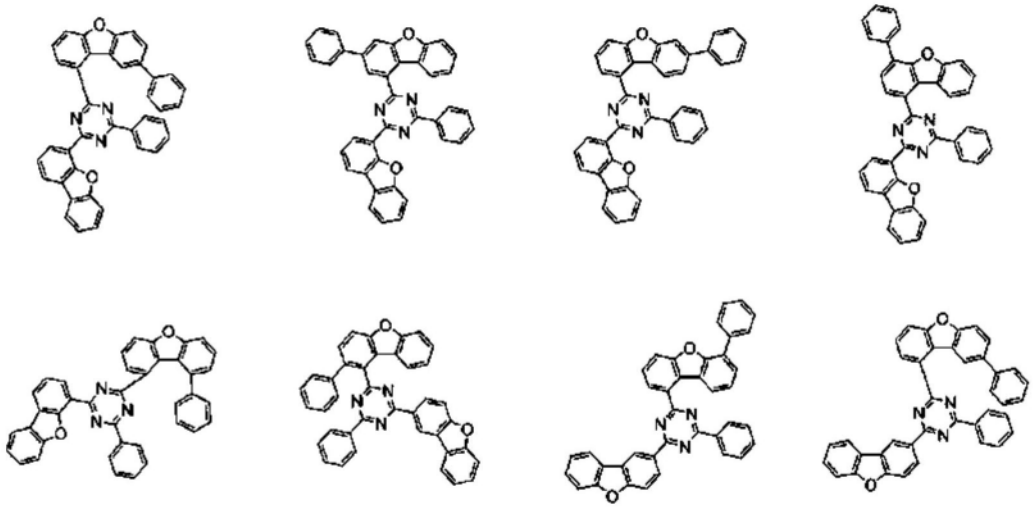
[0160]



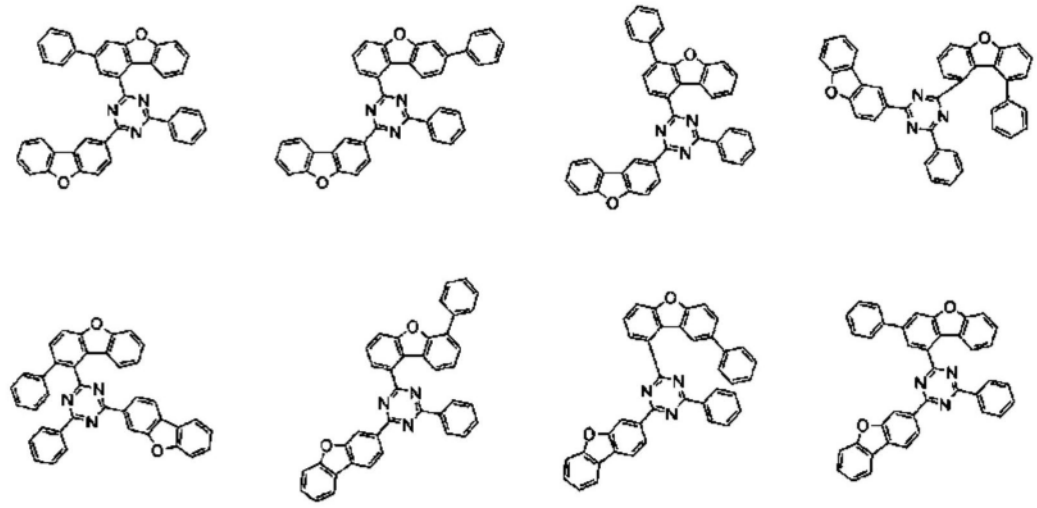


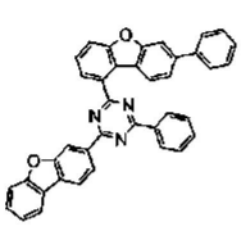
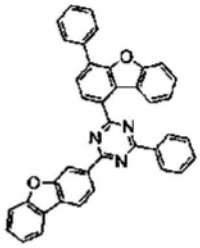
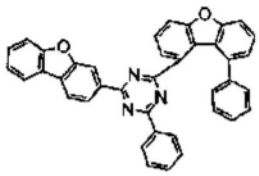
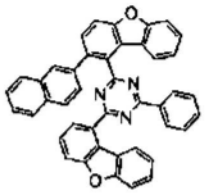
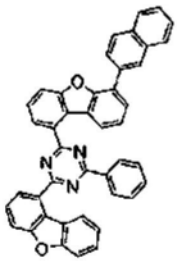
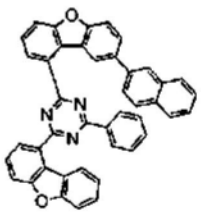
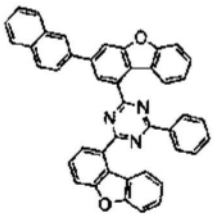
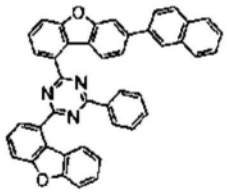
[0161]



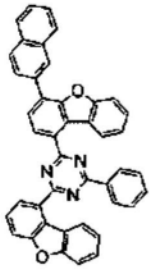
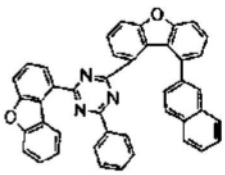
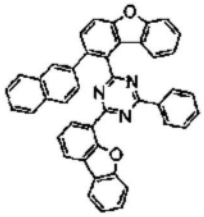
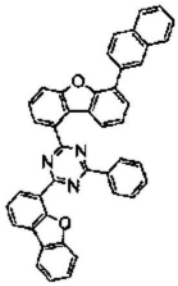
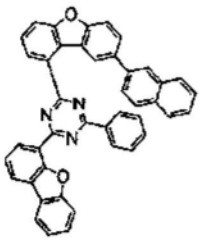
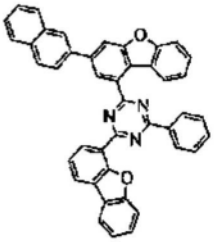
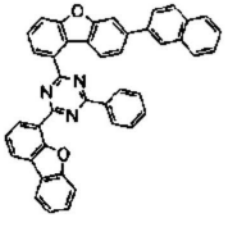
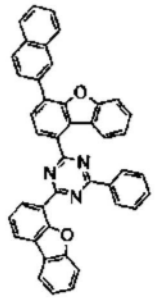


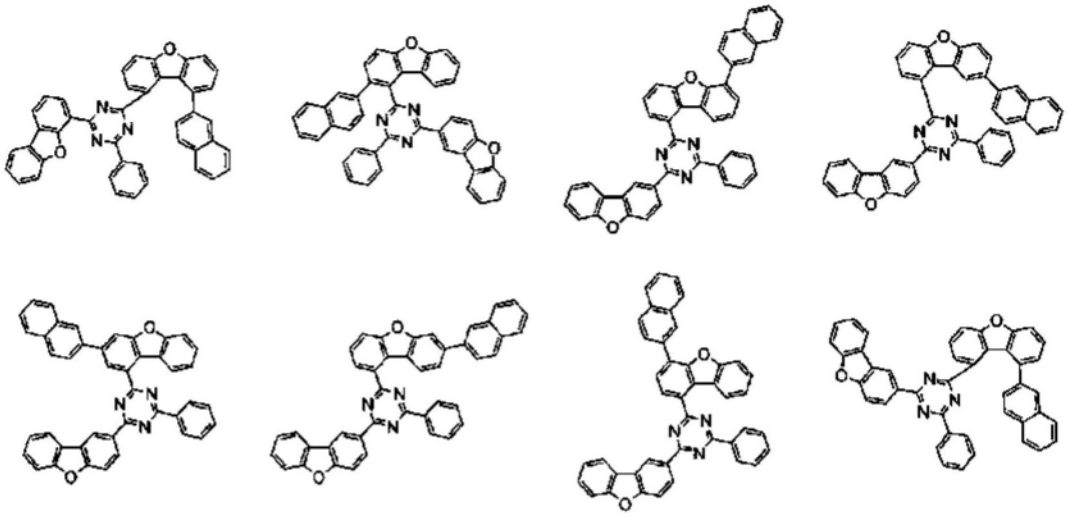
[0162]



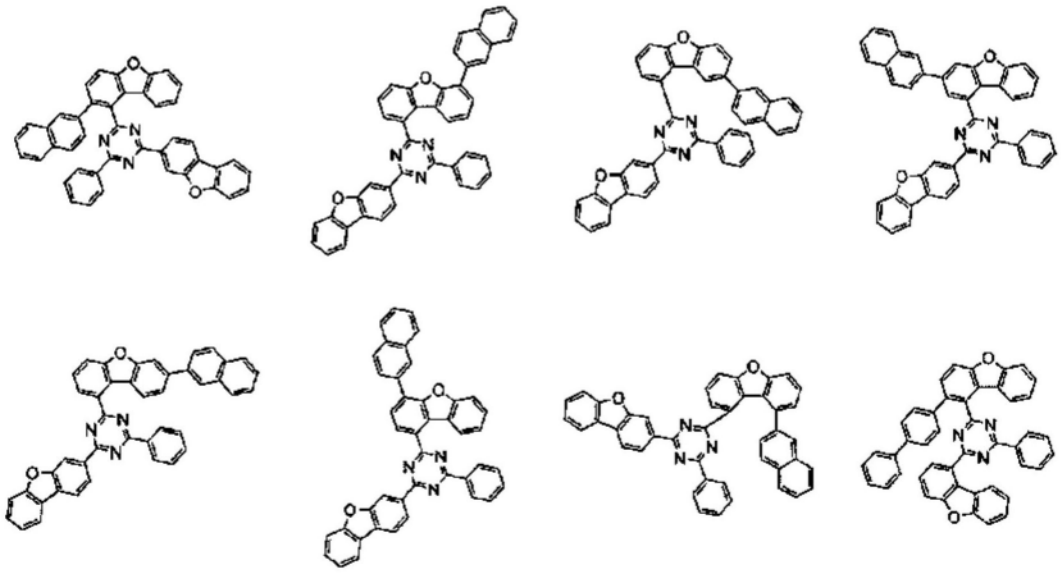









[0163]

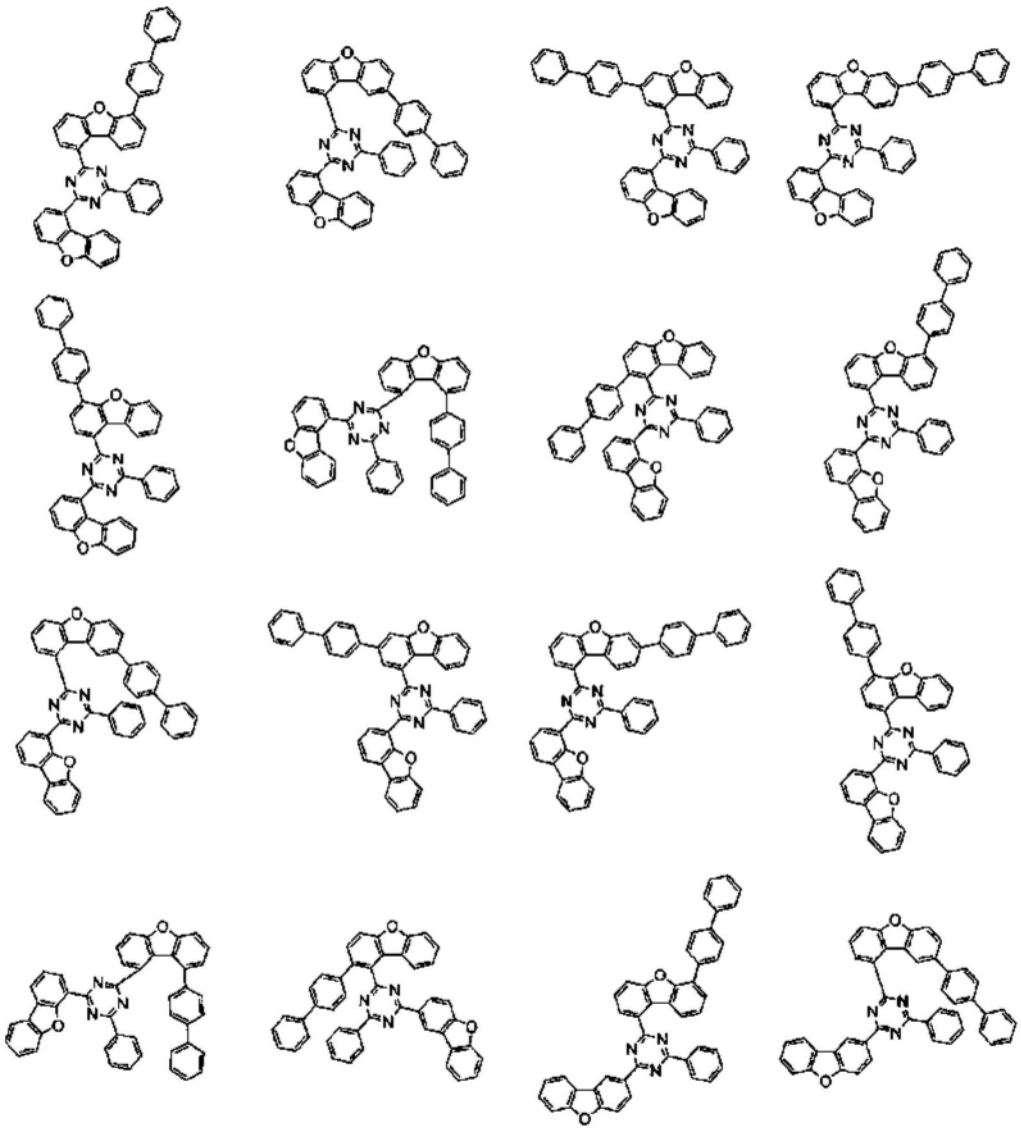











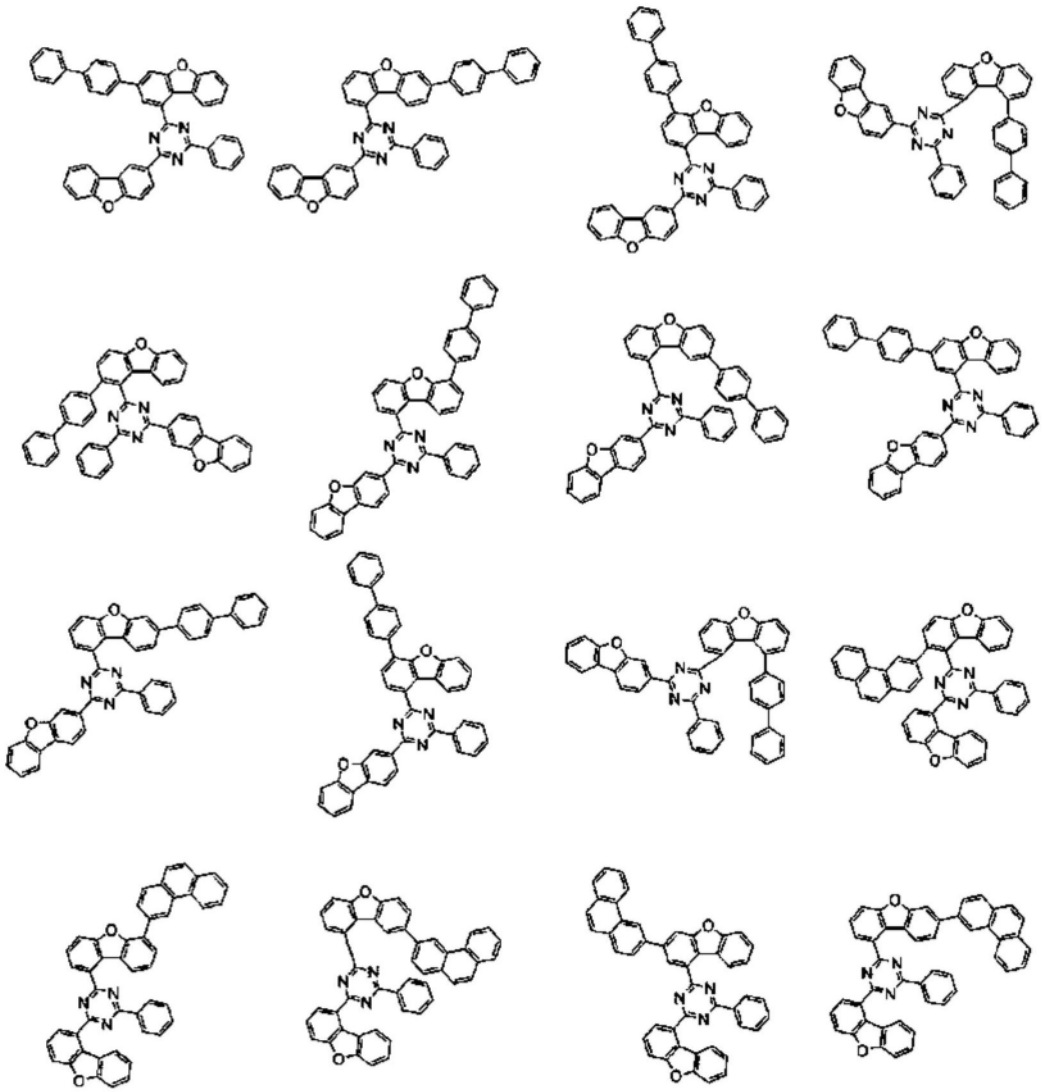
[0164]

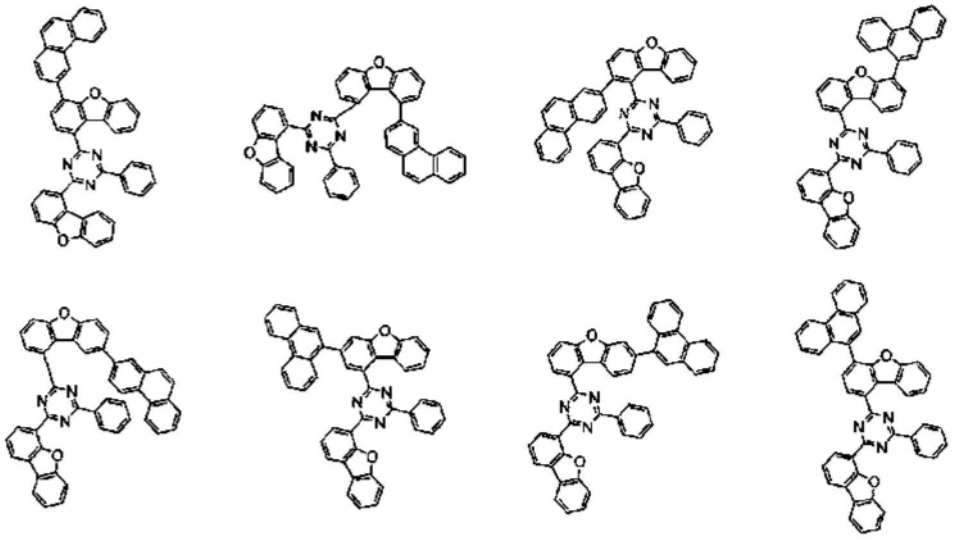


[0165]

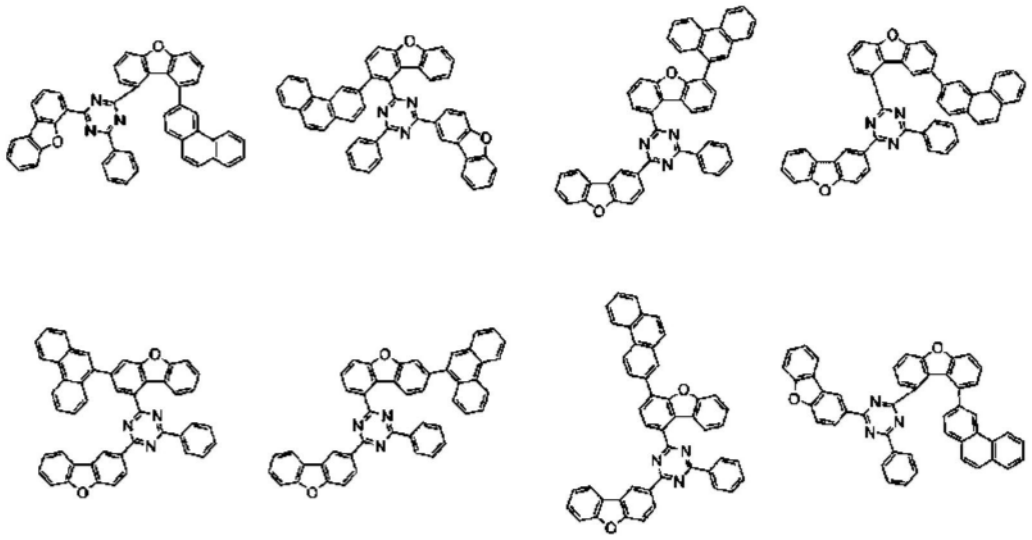


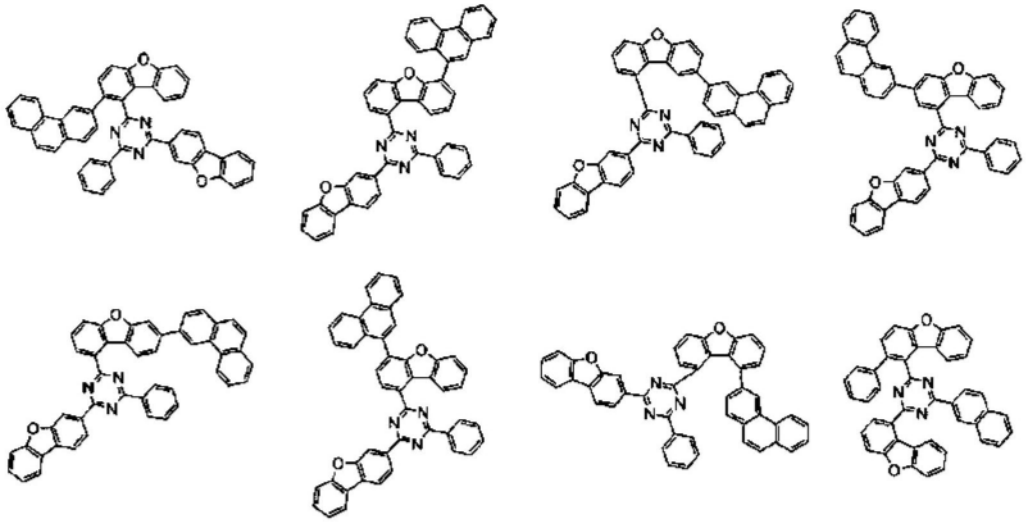
[0166]



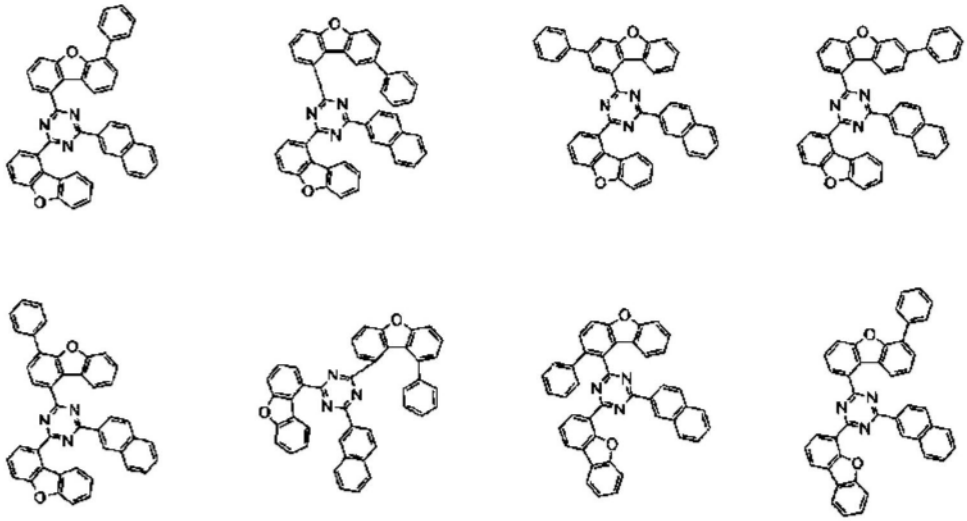


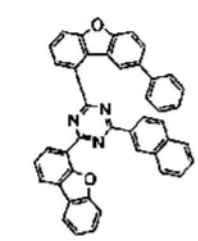
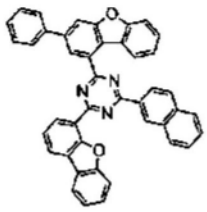
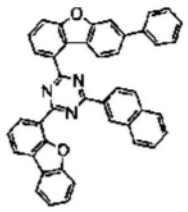
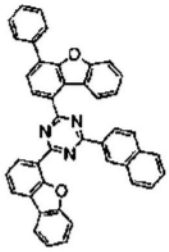
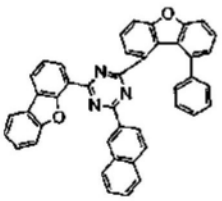
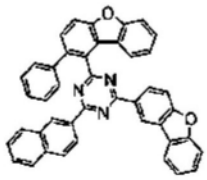
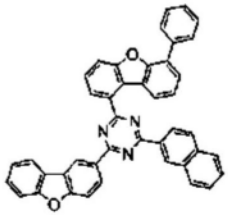
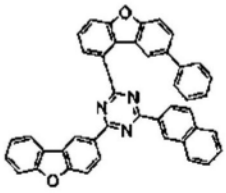
[0167]



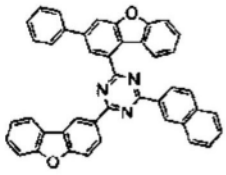
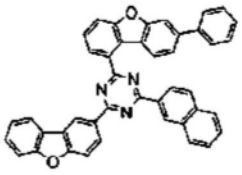
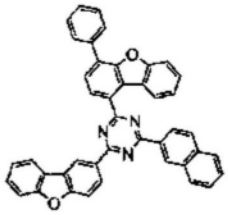
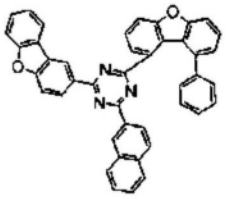
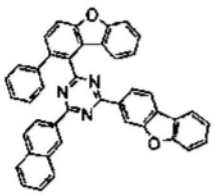
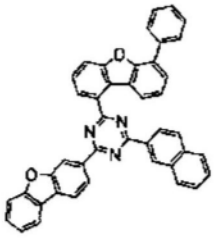
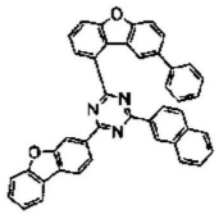
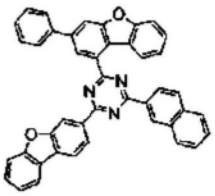


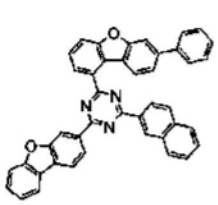
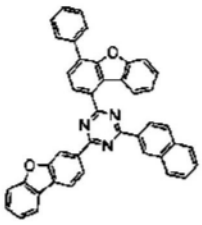
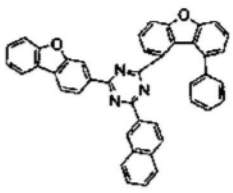
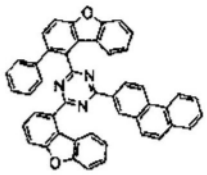
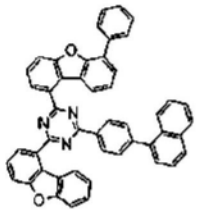
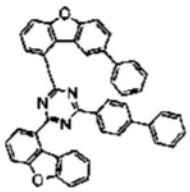
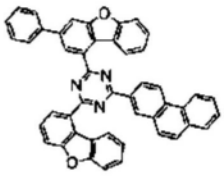
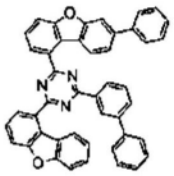
[0168]



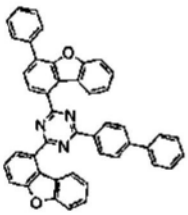
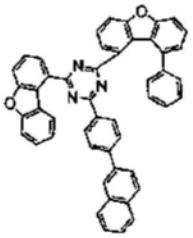
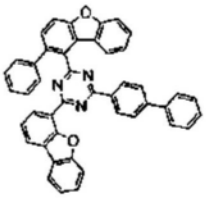
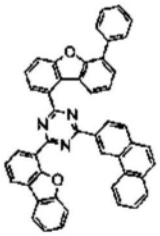
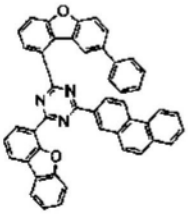
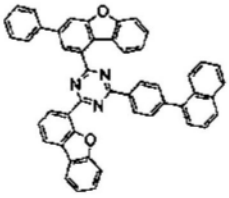
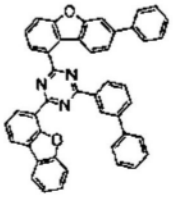
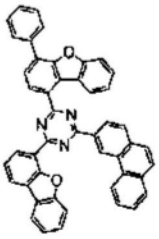









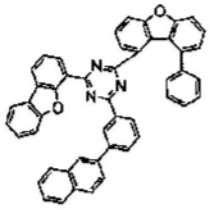
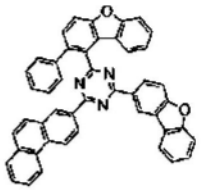
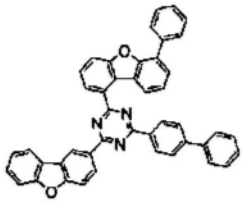
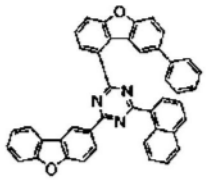
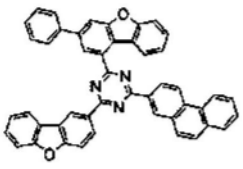
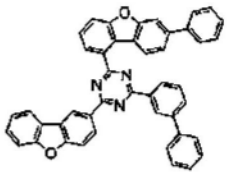
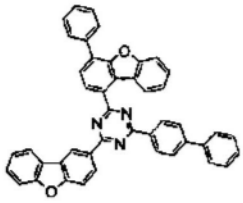
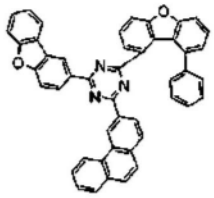
[0169]

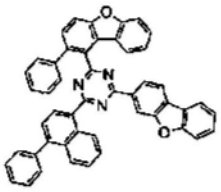
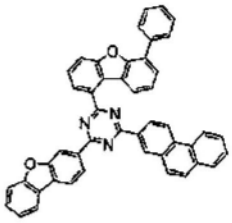
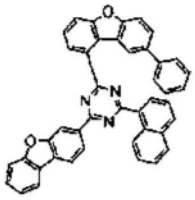
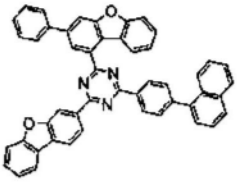
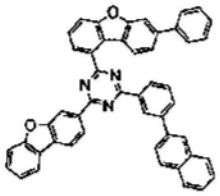
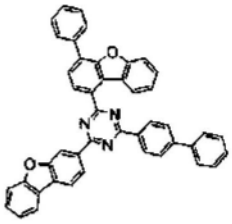
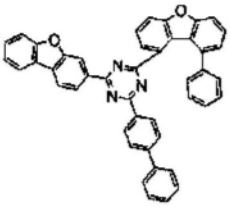
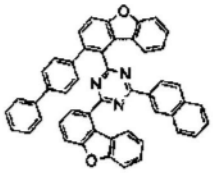









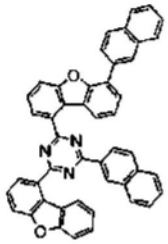
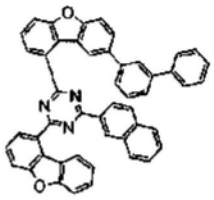
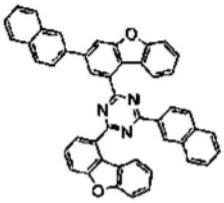
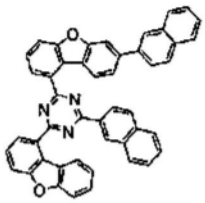
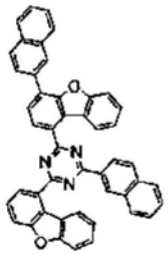
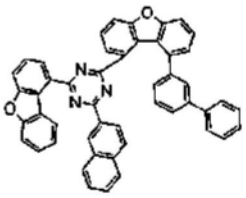
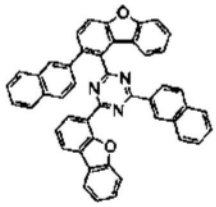
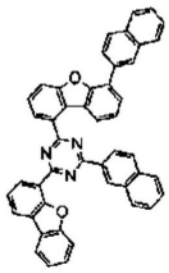
[0170]

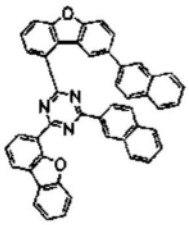
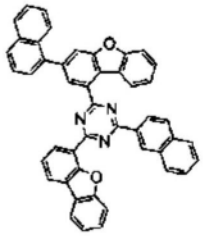
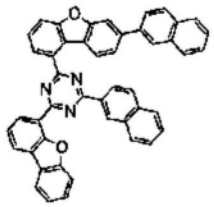
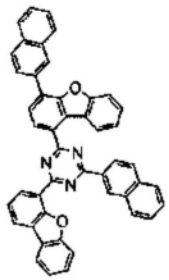
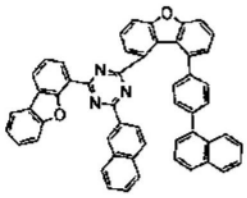
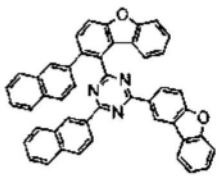
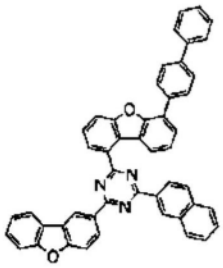
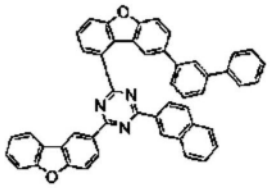









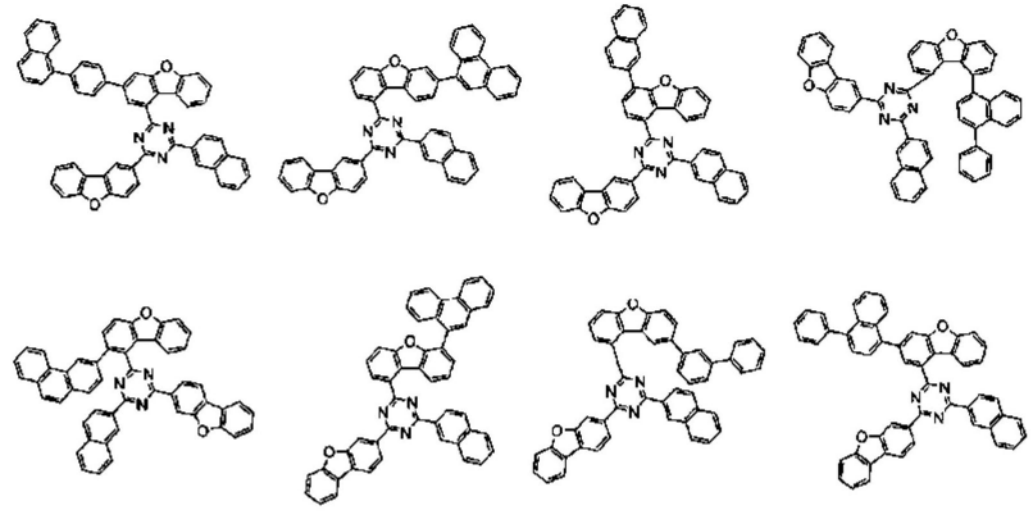
[0171]

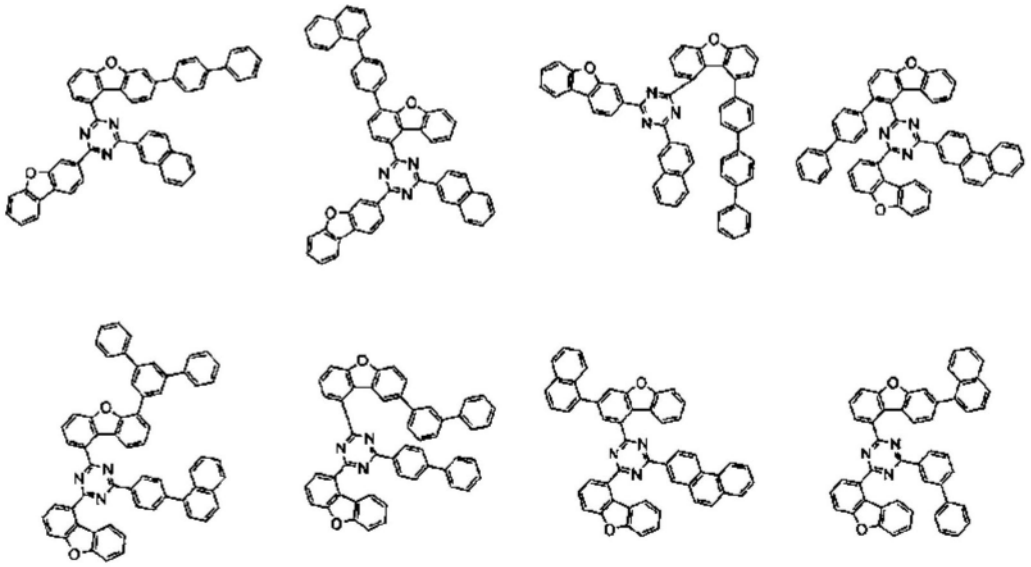









[0172]

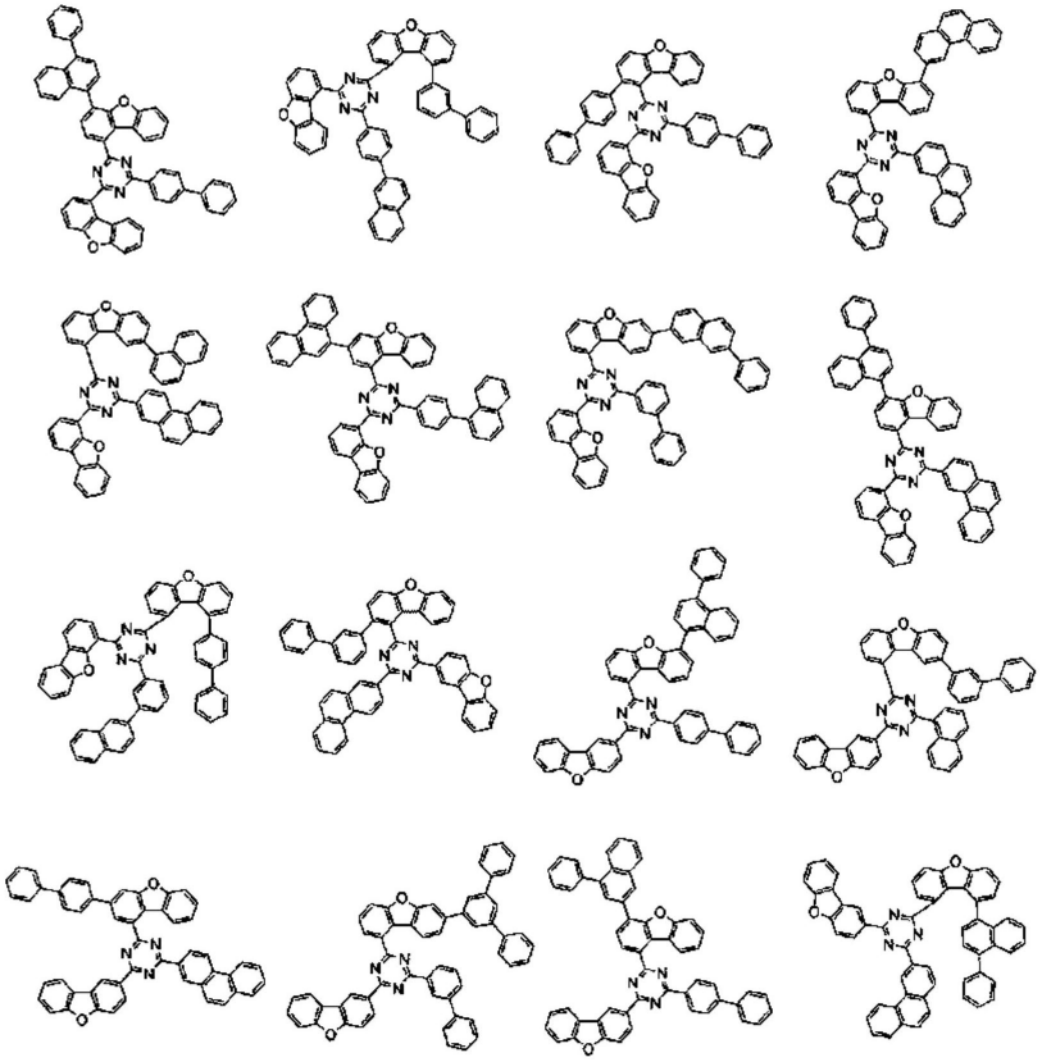









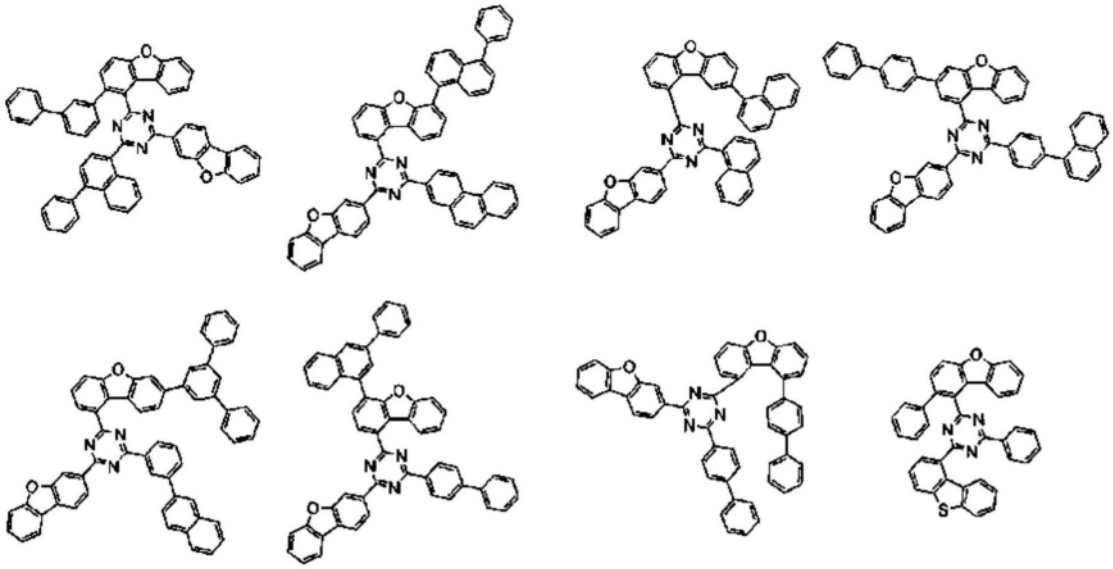


[0173]

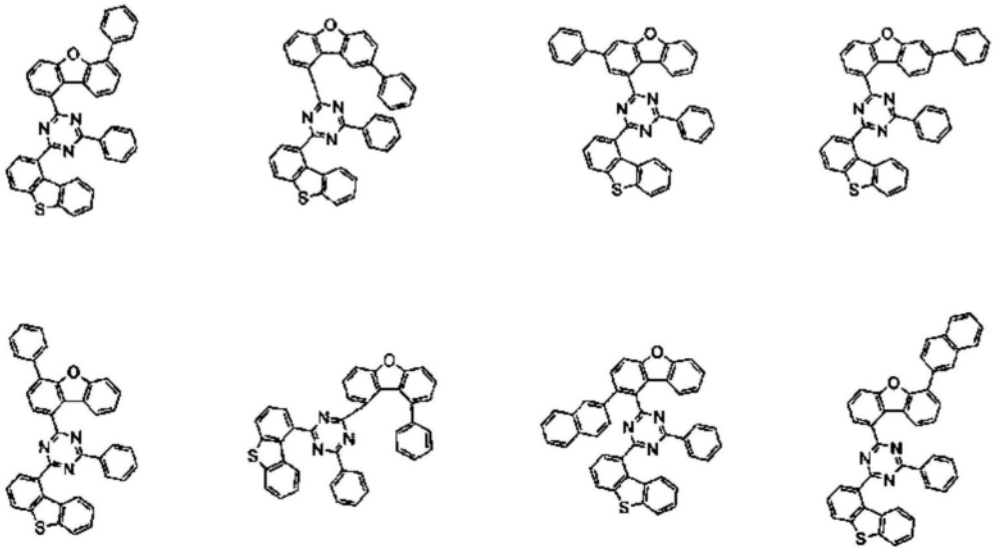


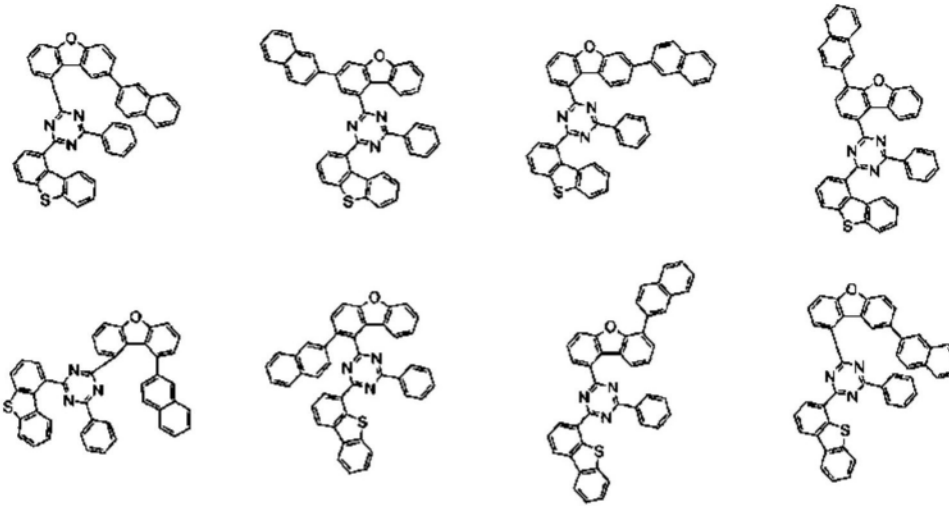
[0174]



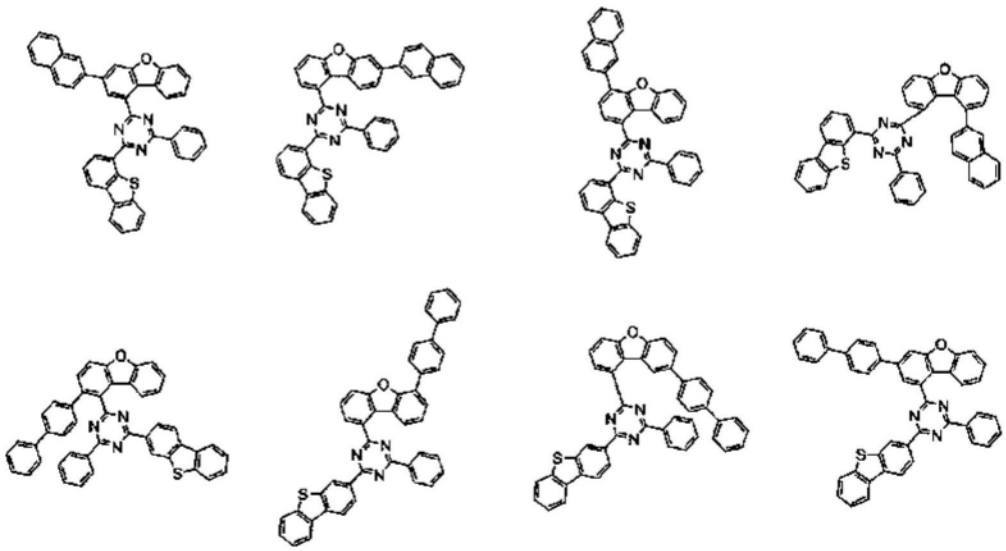


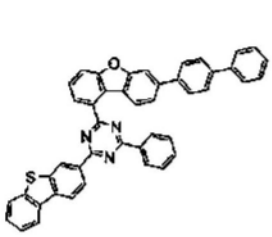
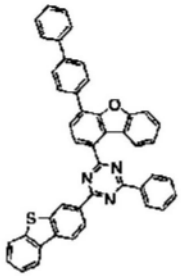
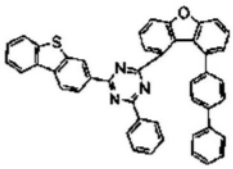
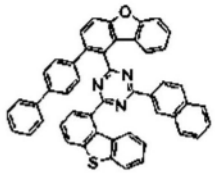
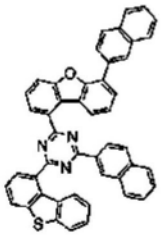
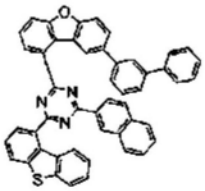
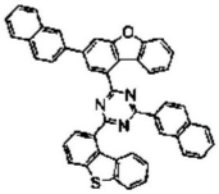
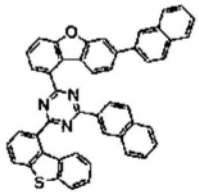
[0175]



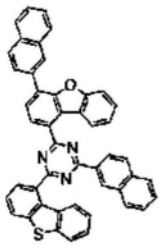
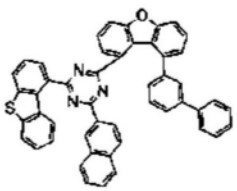
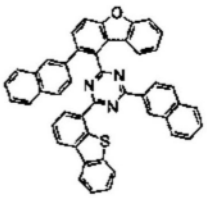
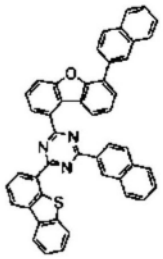
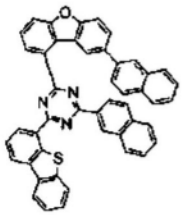
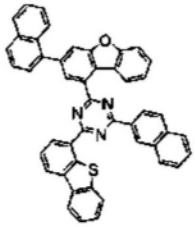
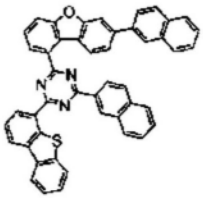
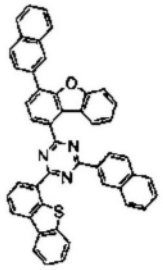


[0176]

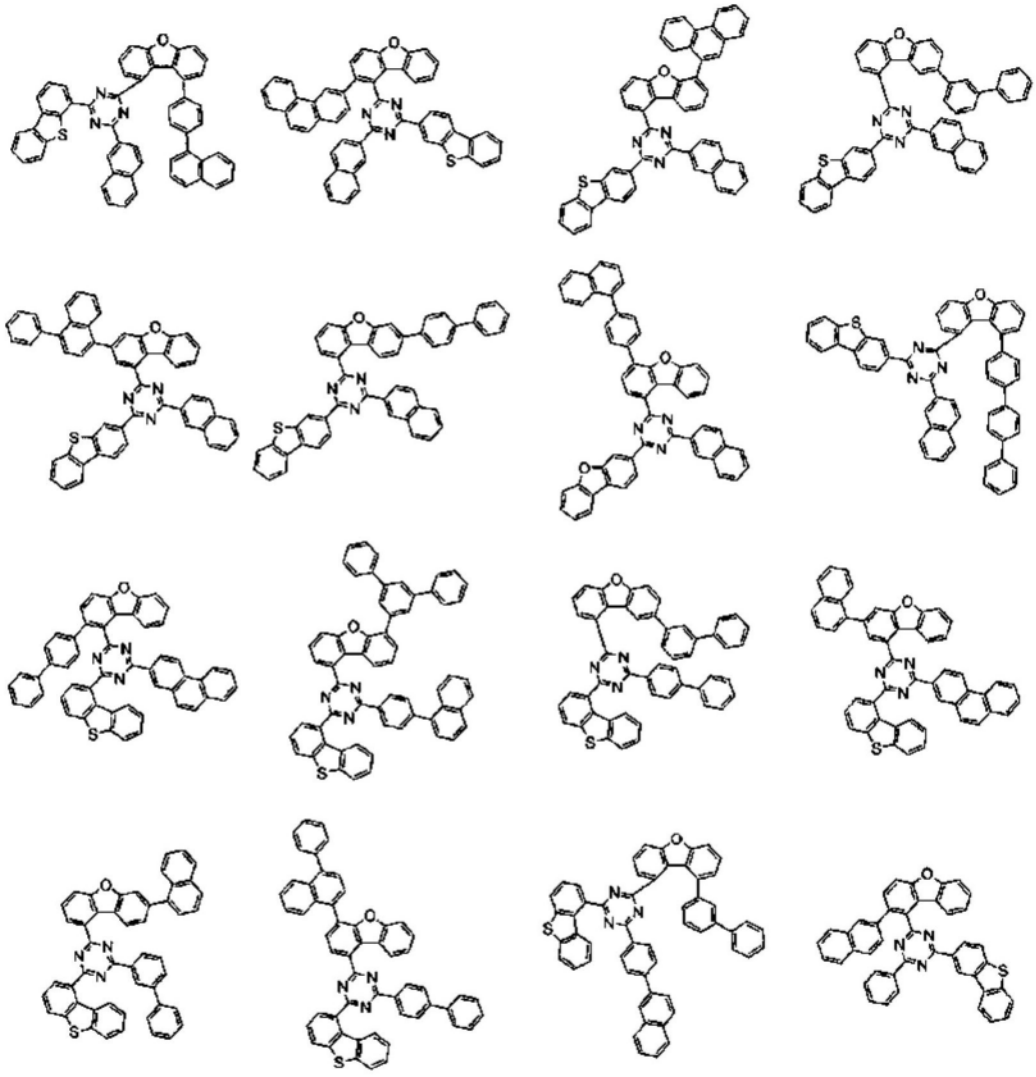


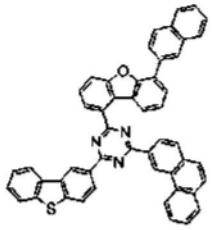
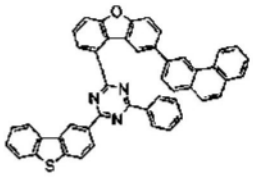
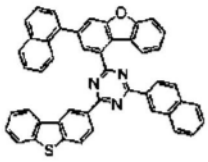
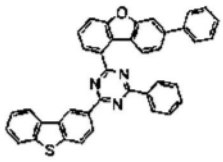
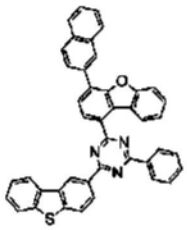
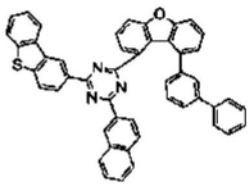
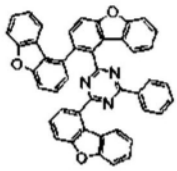
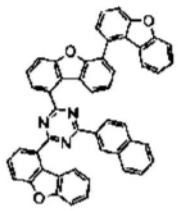









[0177]

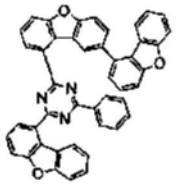
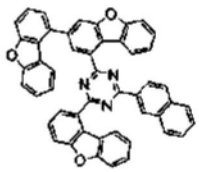
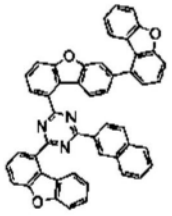
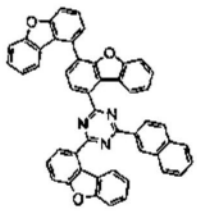
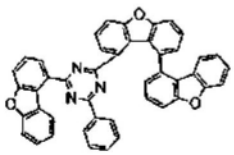
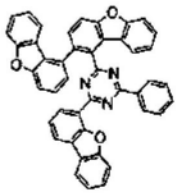
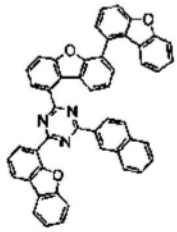
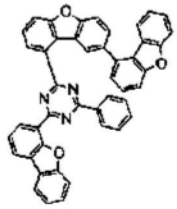









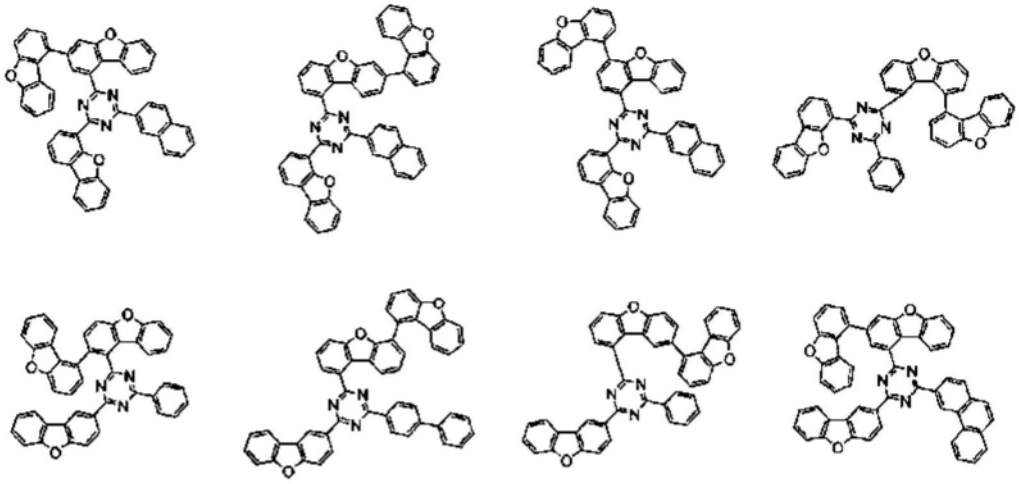
[0178]



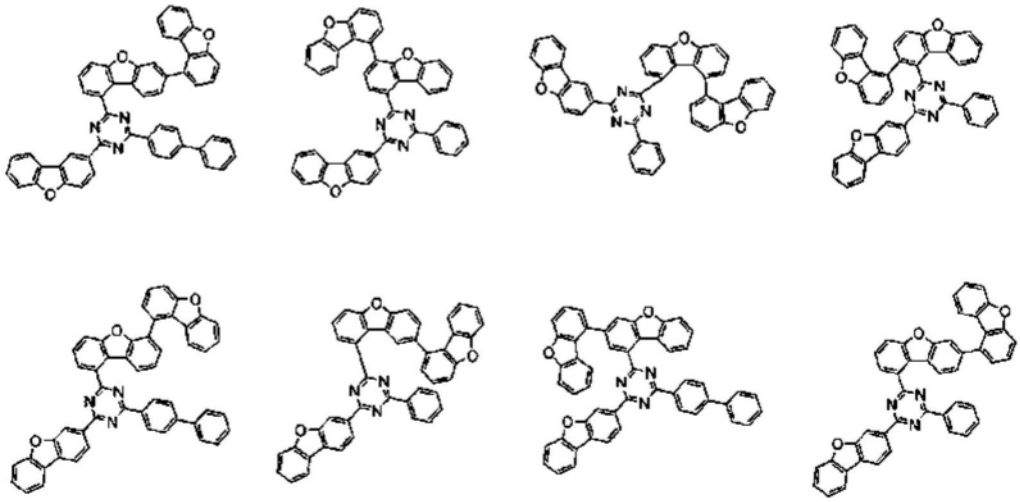









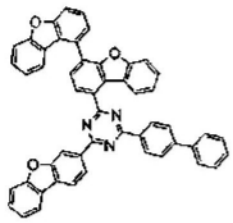
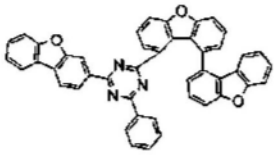
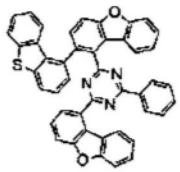
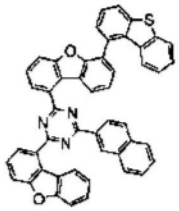
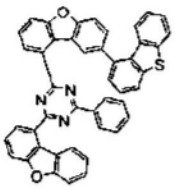
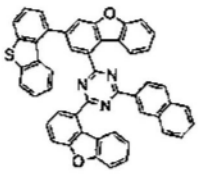
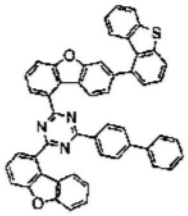
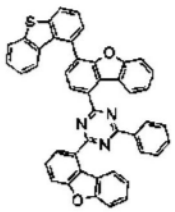
[0179]

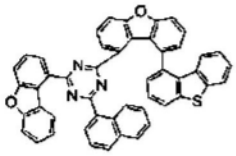
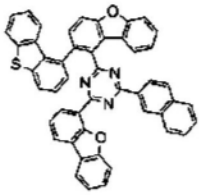
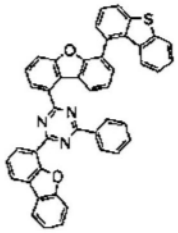
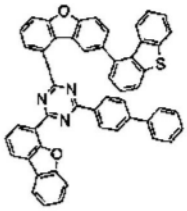
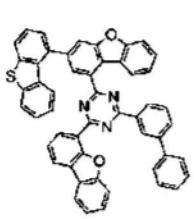
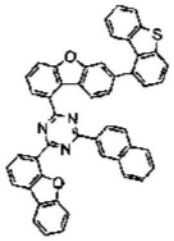
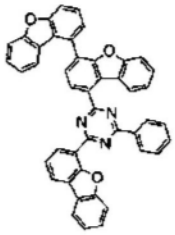
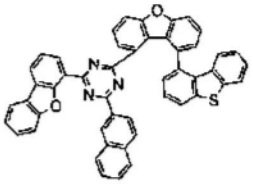


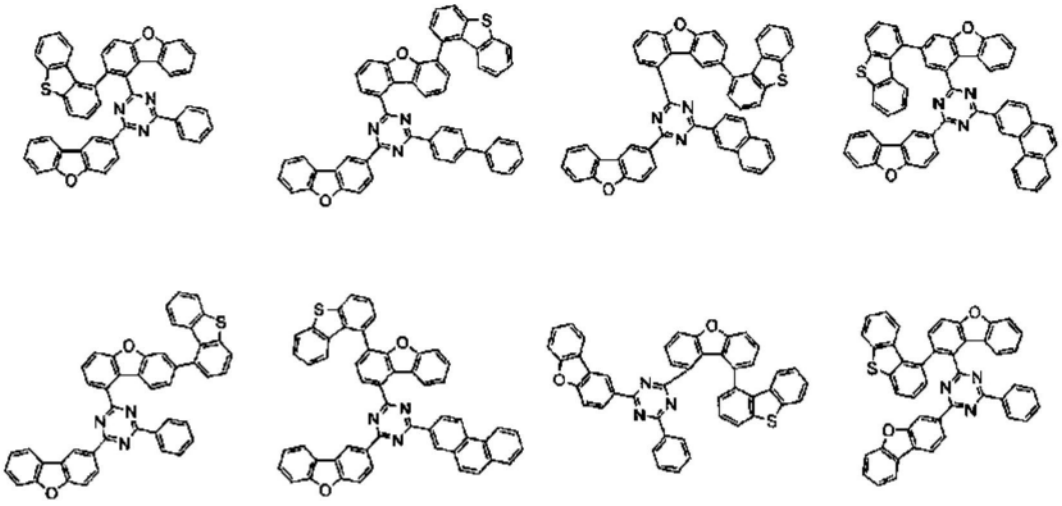
[0180]



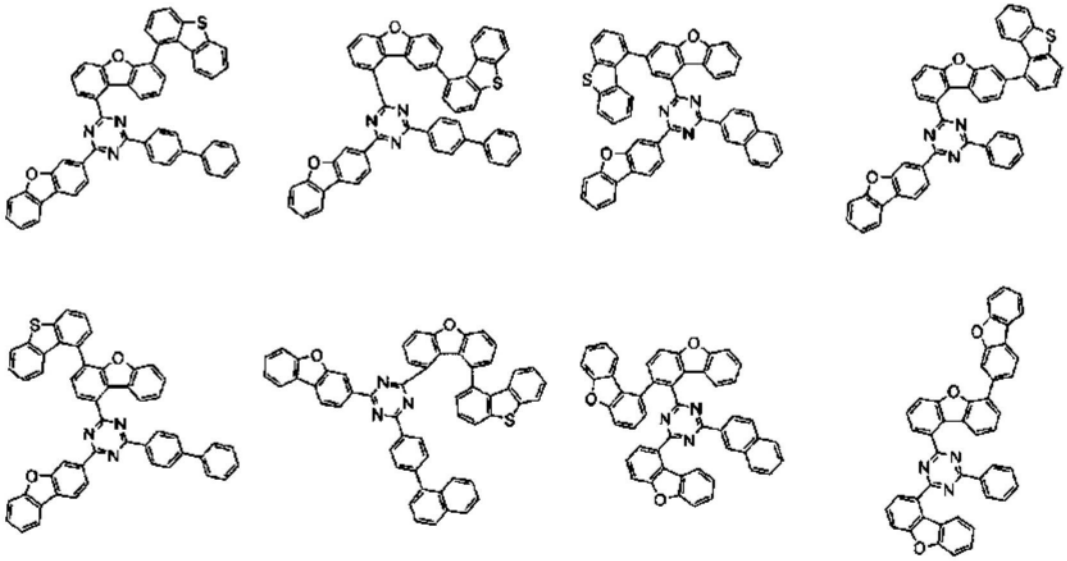









[0181]

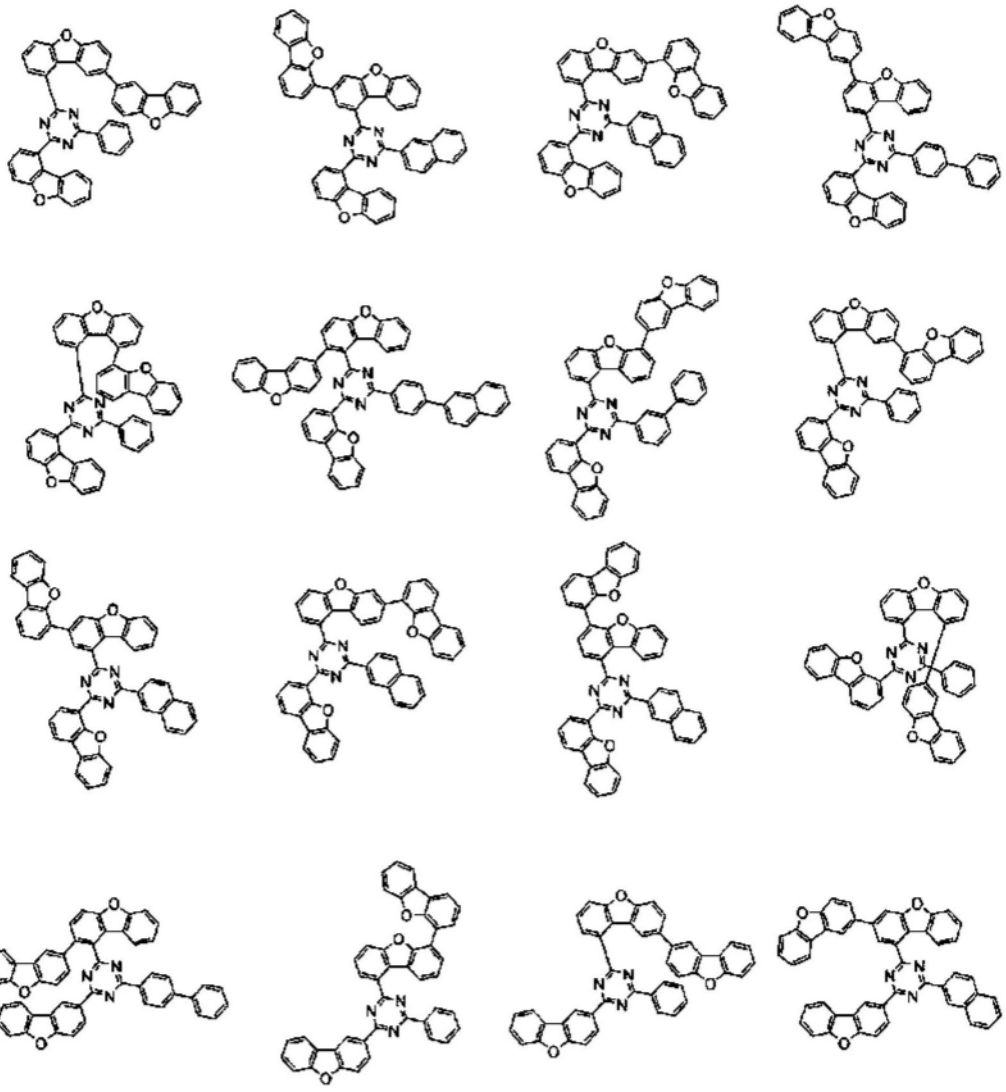









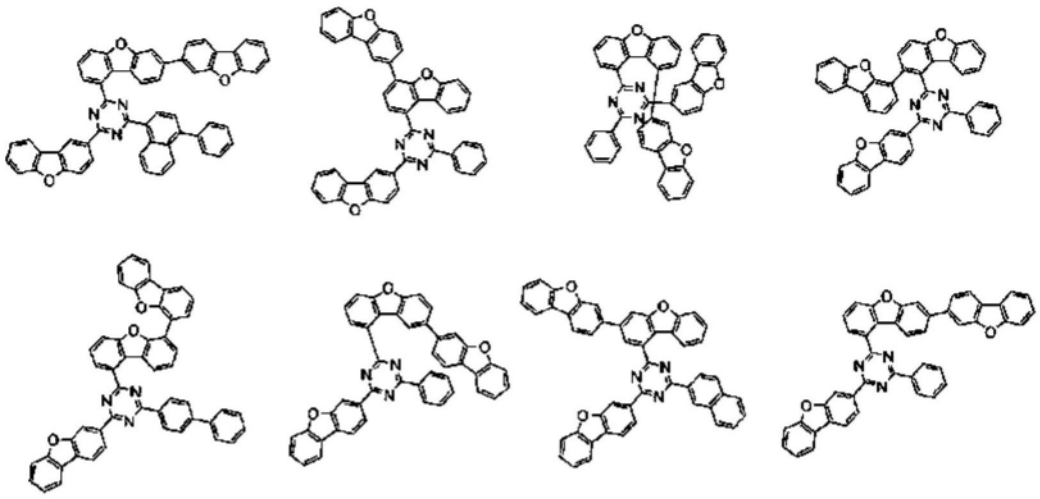


[0182]

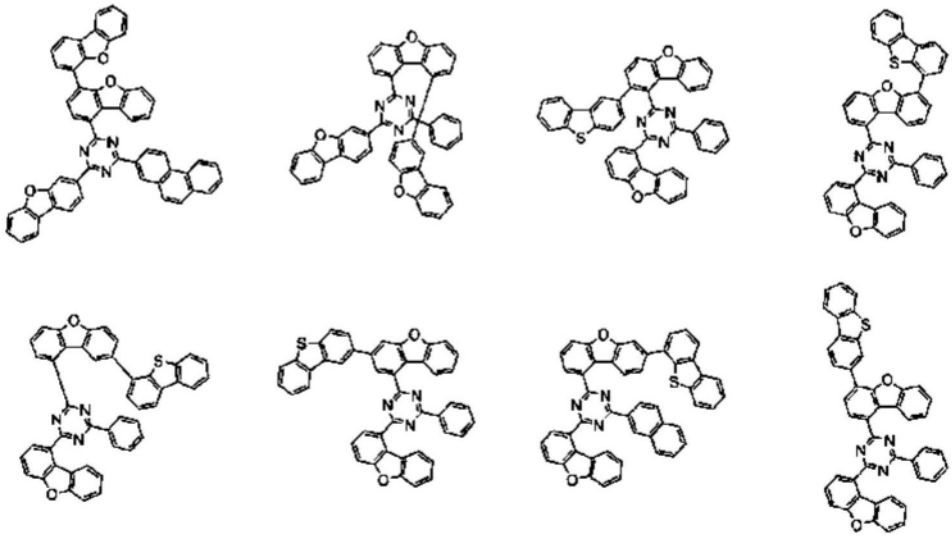


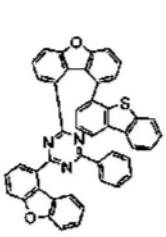
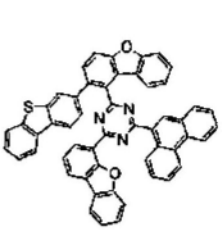
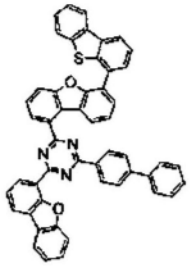
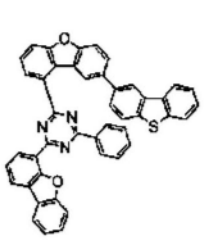
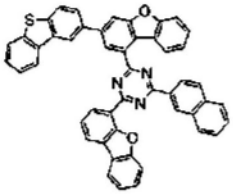
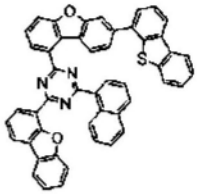
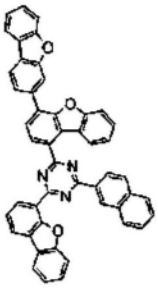
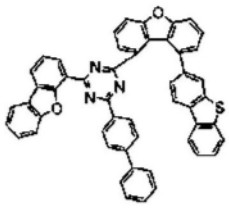
[0183]



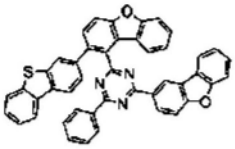
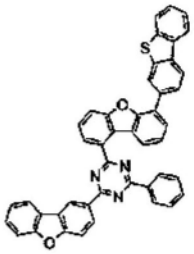
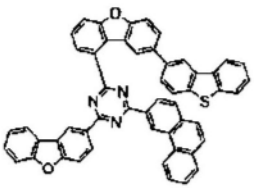
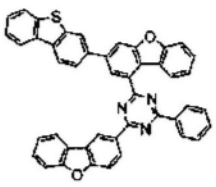
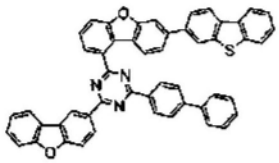
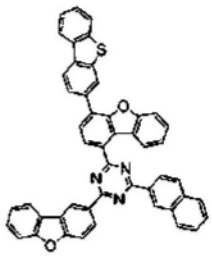
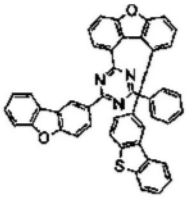
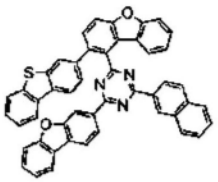


[0184]

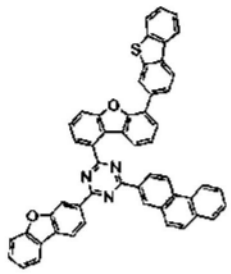
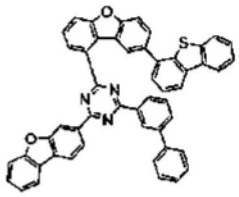
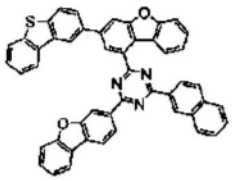
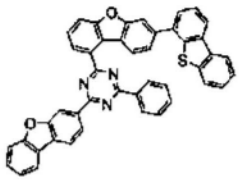
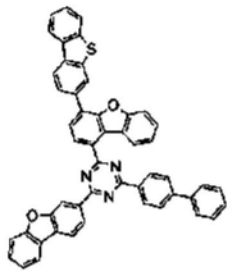
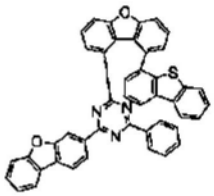
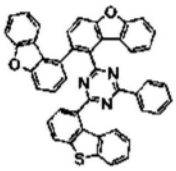
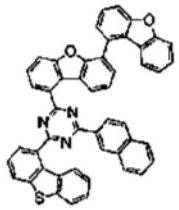
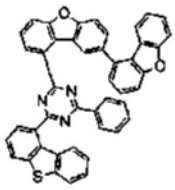
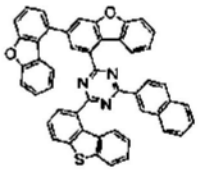
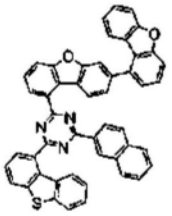
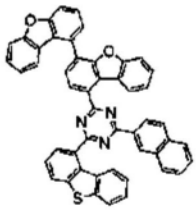
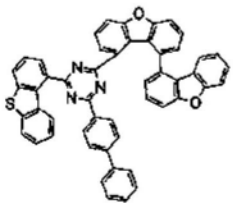
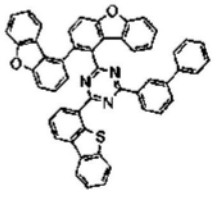
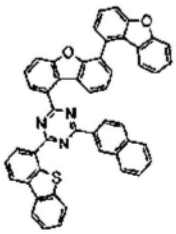
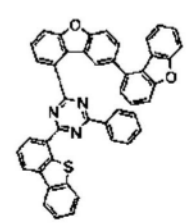


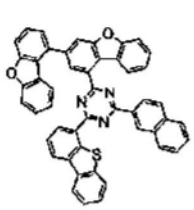
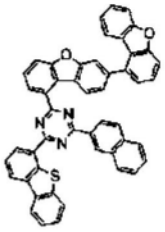
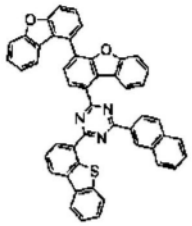
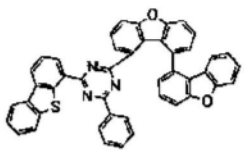
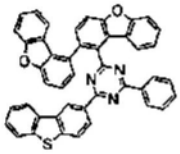
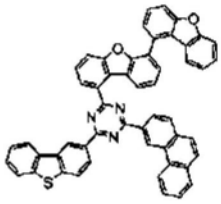
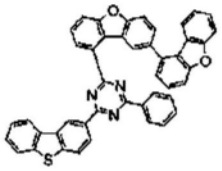
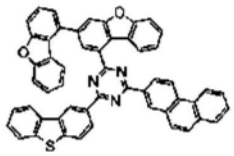









[0185]

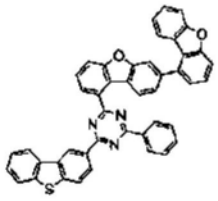
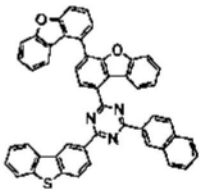
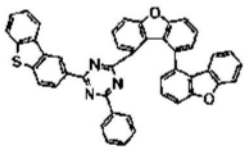
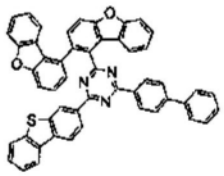
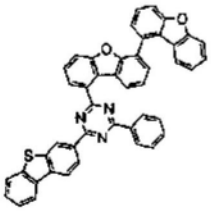
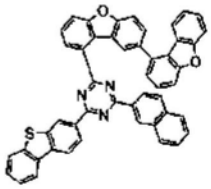
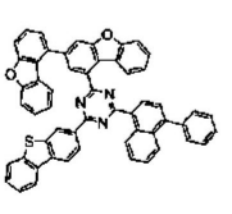
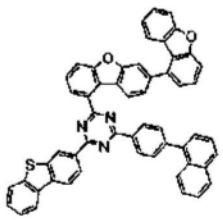









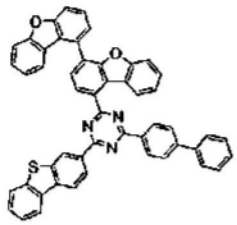
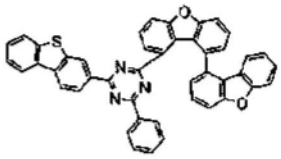
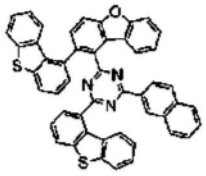
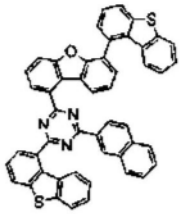
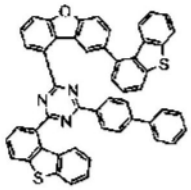
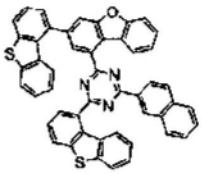
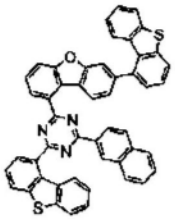
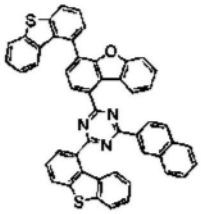
[0186]

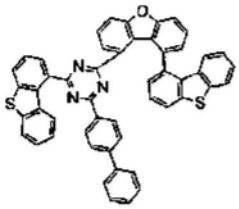
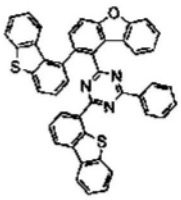
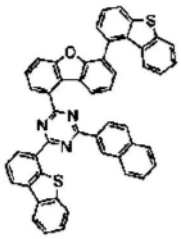
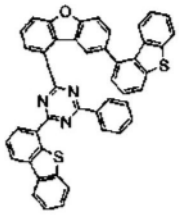
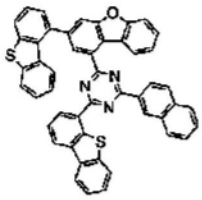
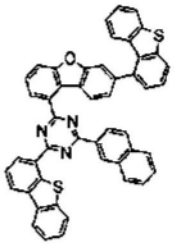
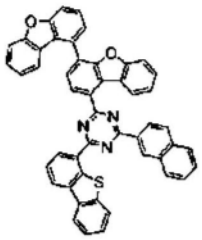
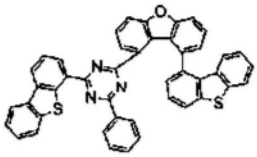









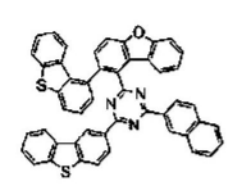
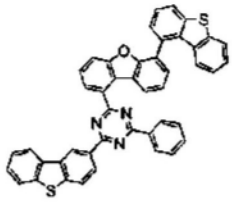
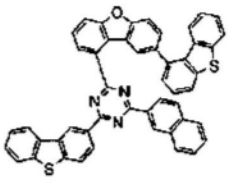
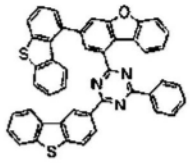
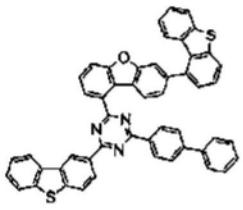
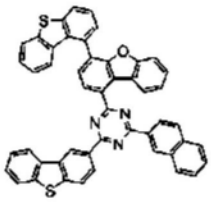
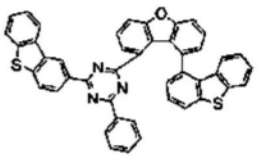
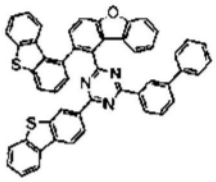
[0187]

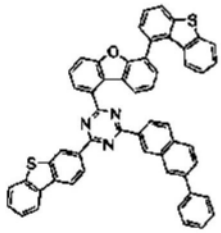
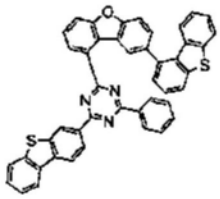
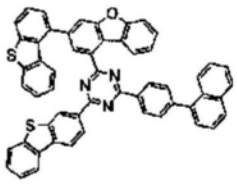
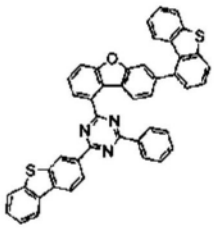
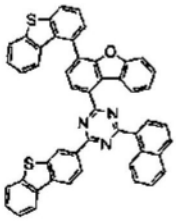
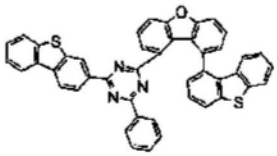
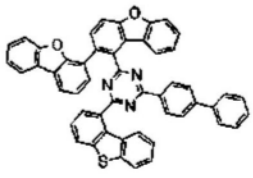
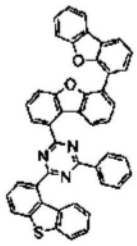









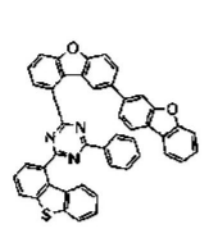
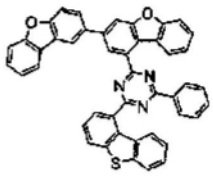
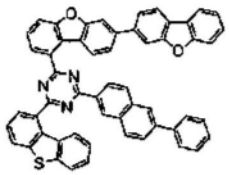
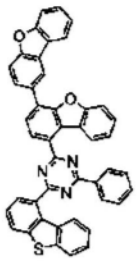
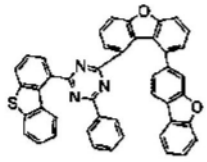
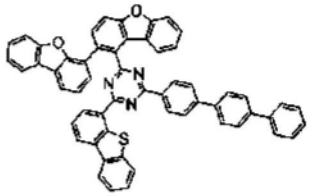
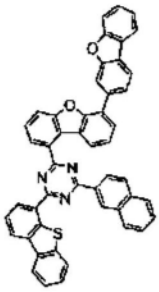
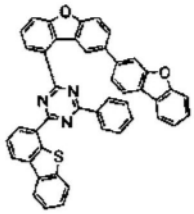
[0188]

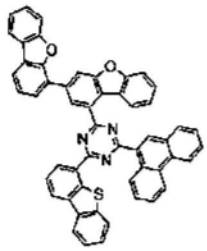
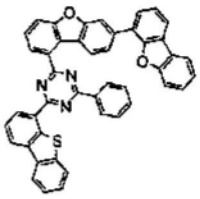
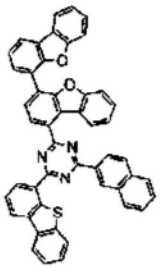
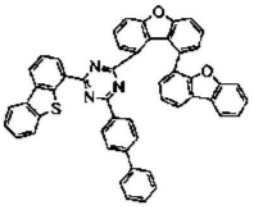
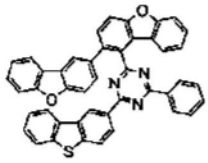
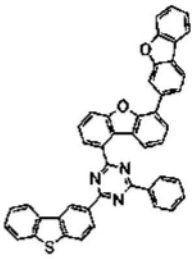
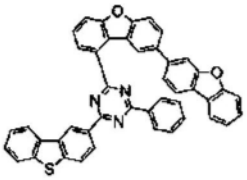
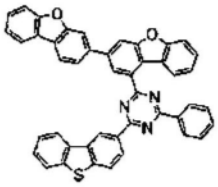









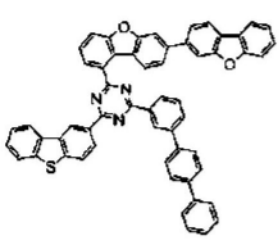
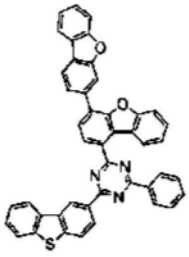
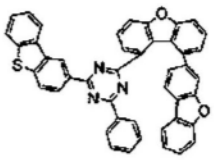
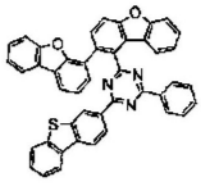
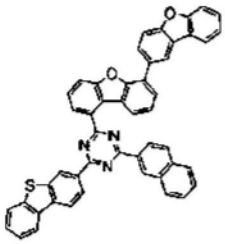
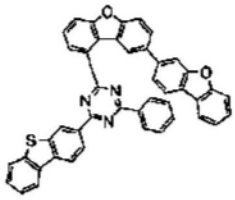
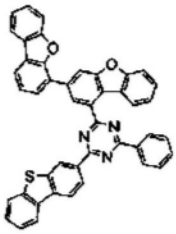
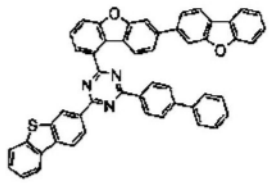
[0189]

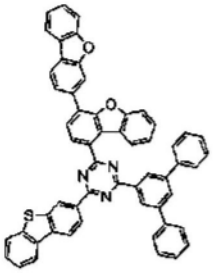
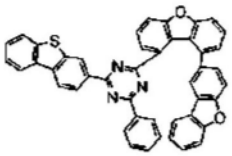
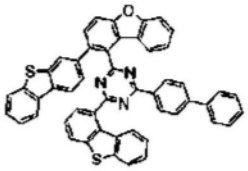
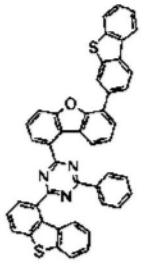
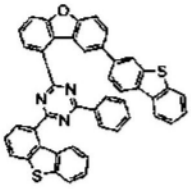
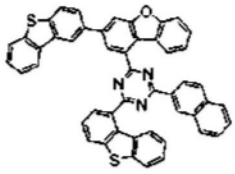
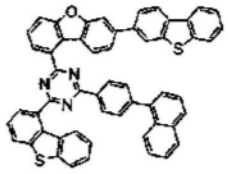
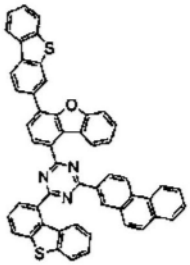









[0190]

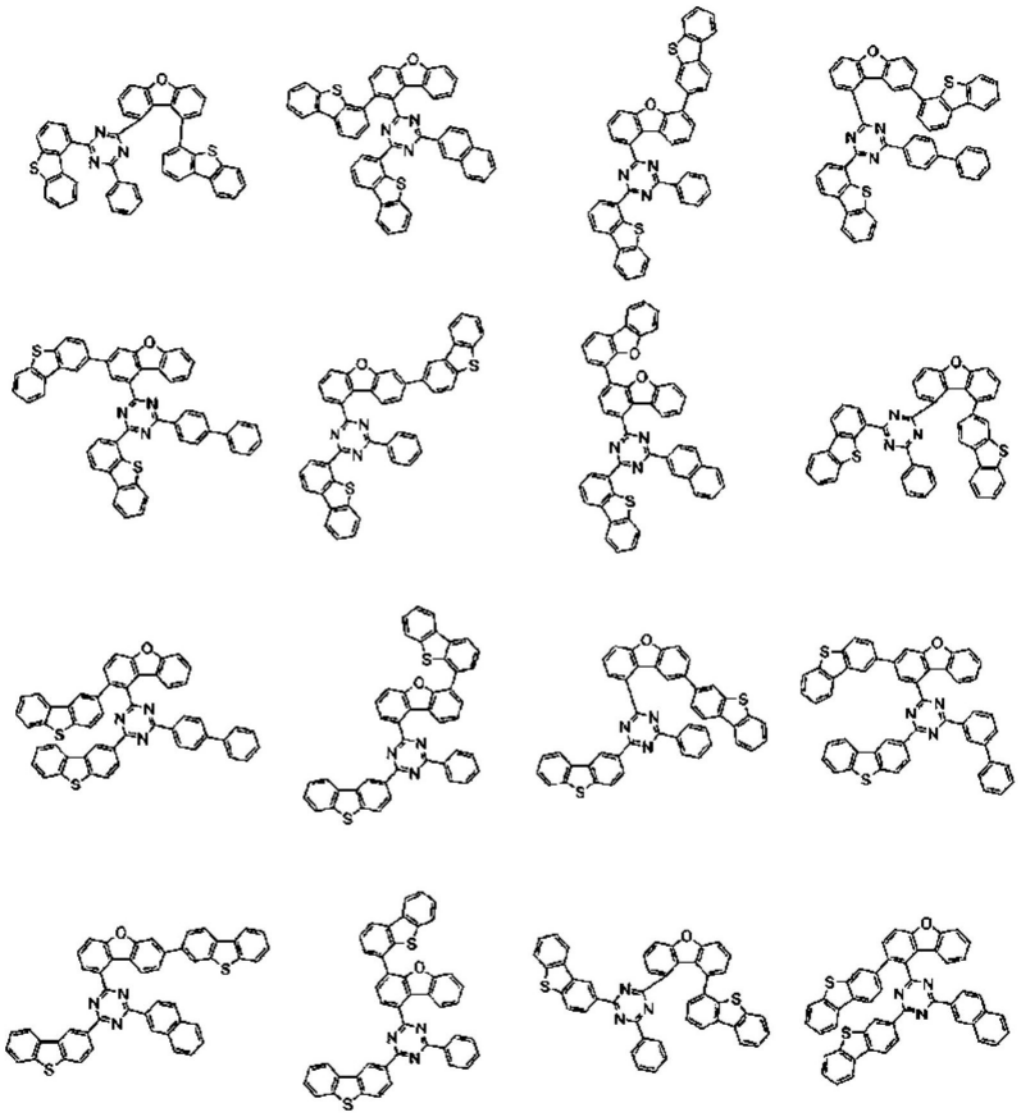









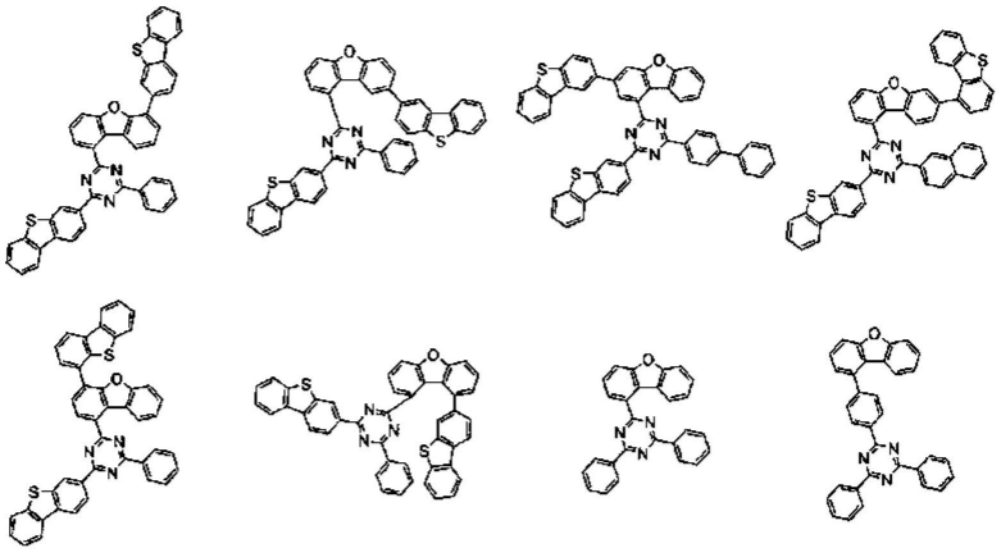









[0191]

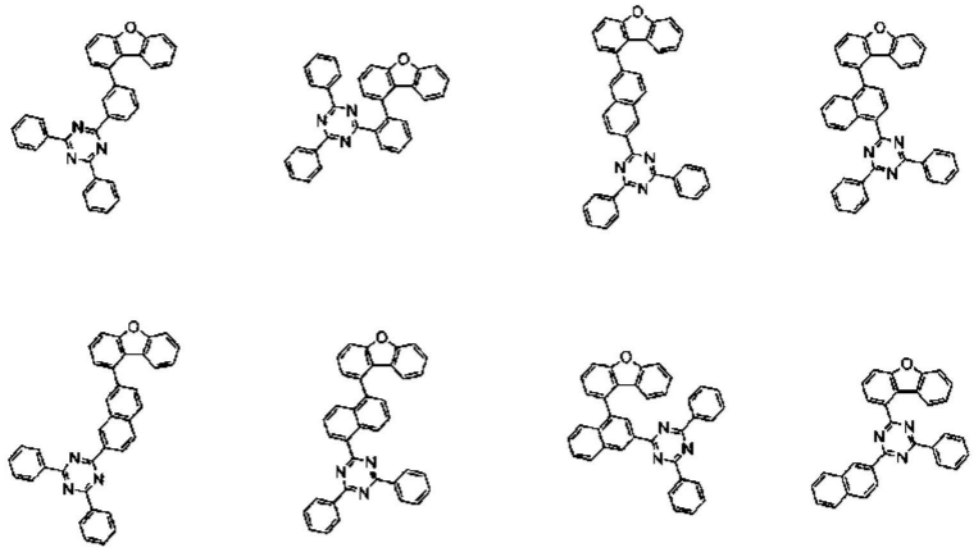









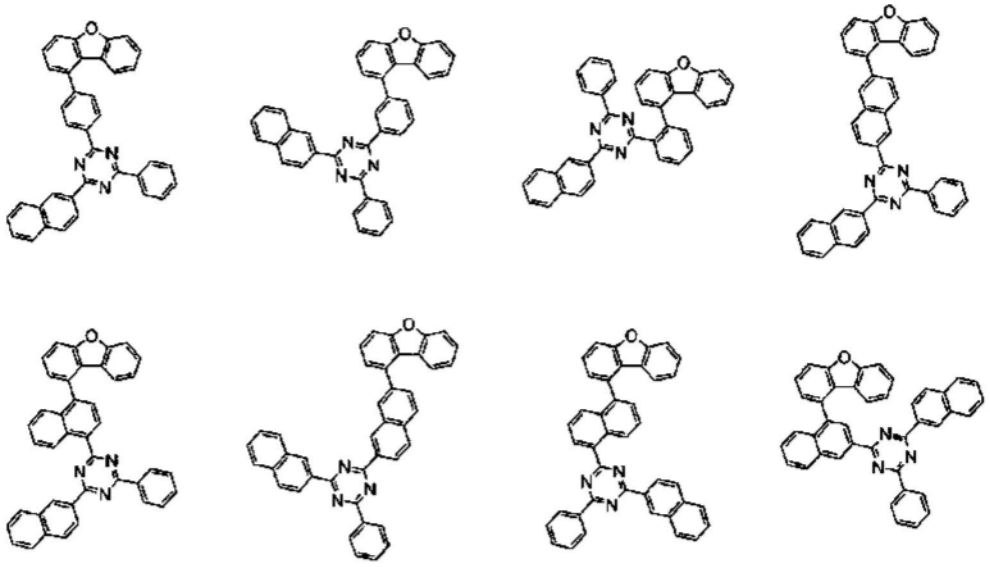
[0192]



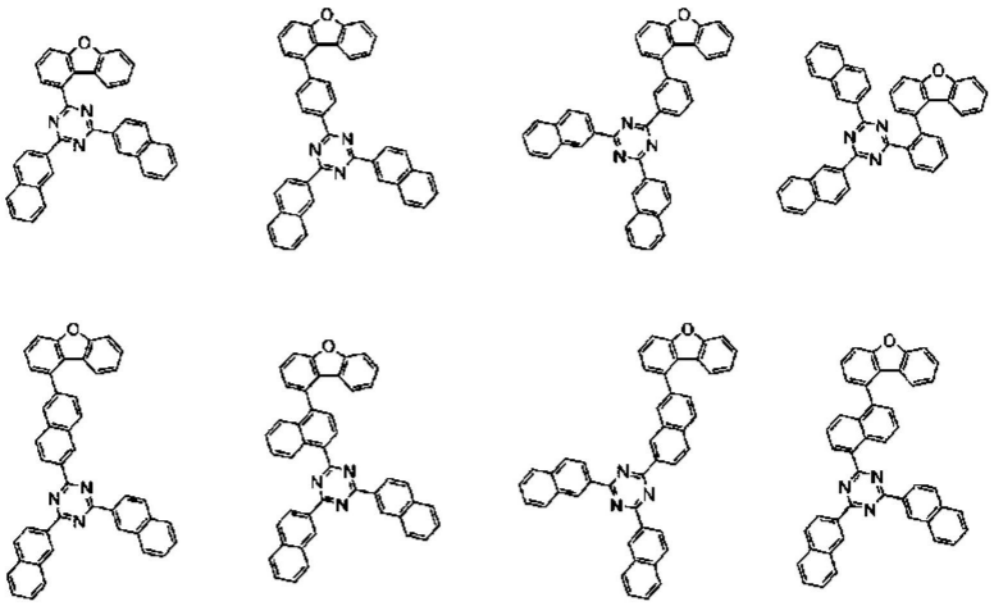


[0193]

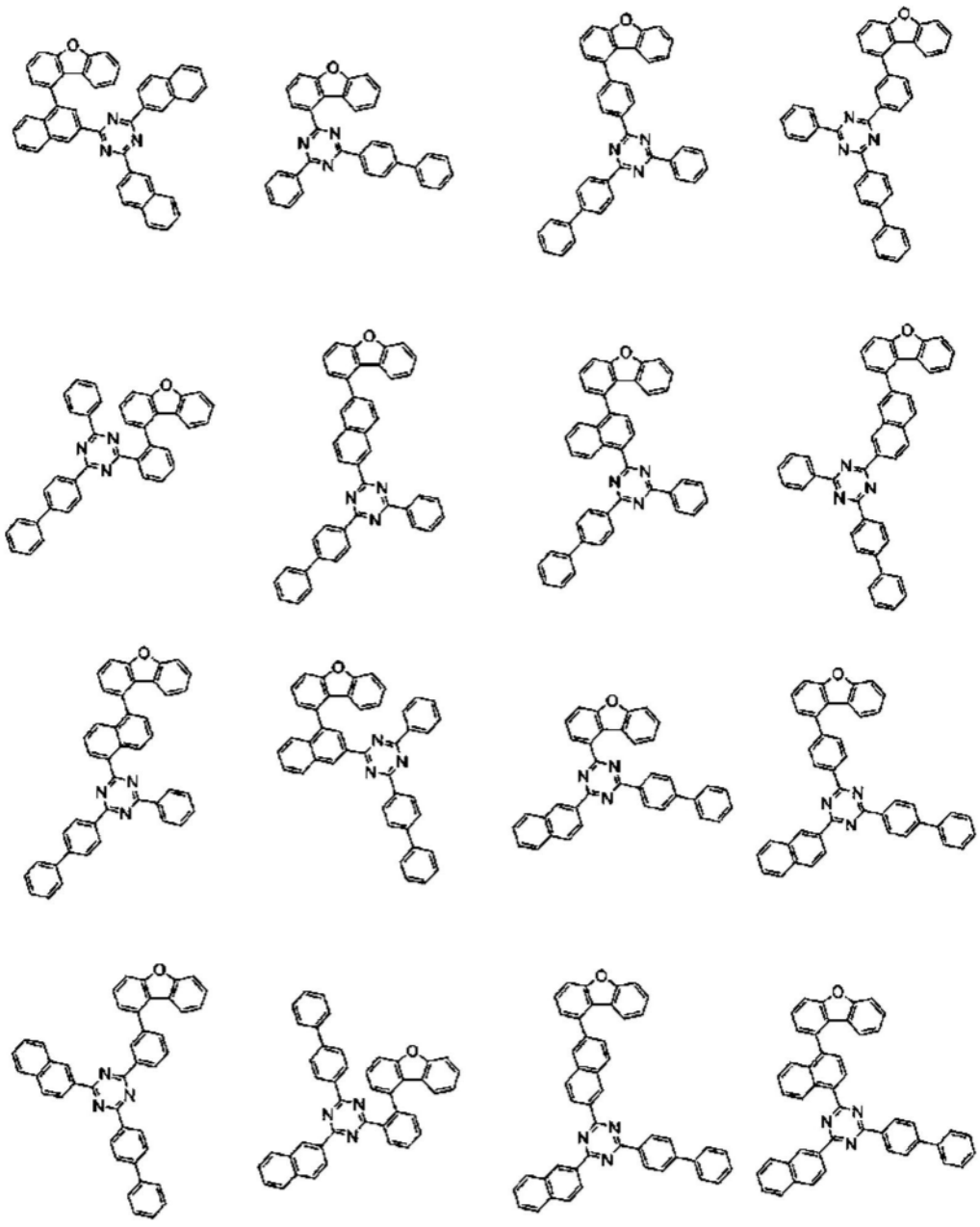


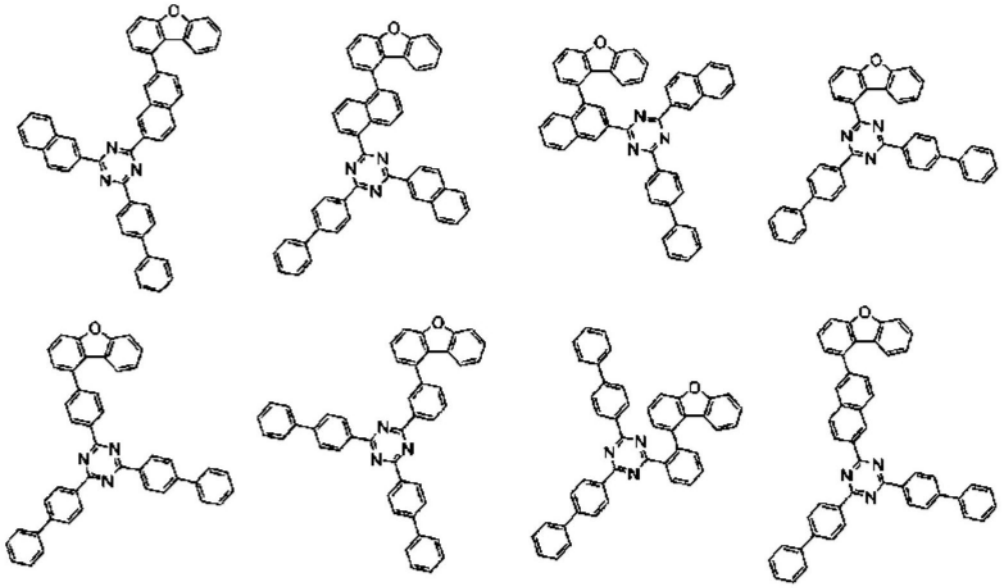


[0194]

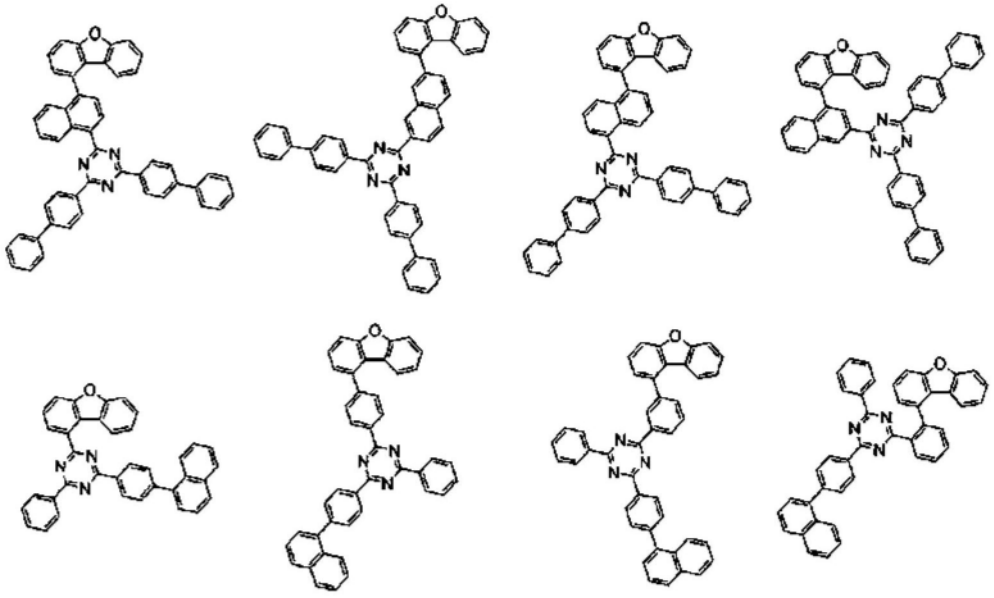


[0195]

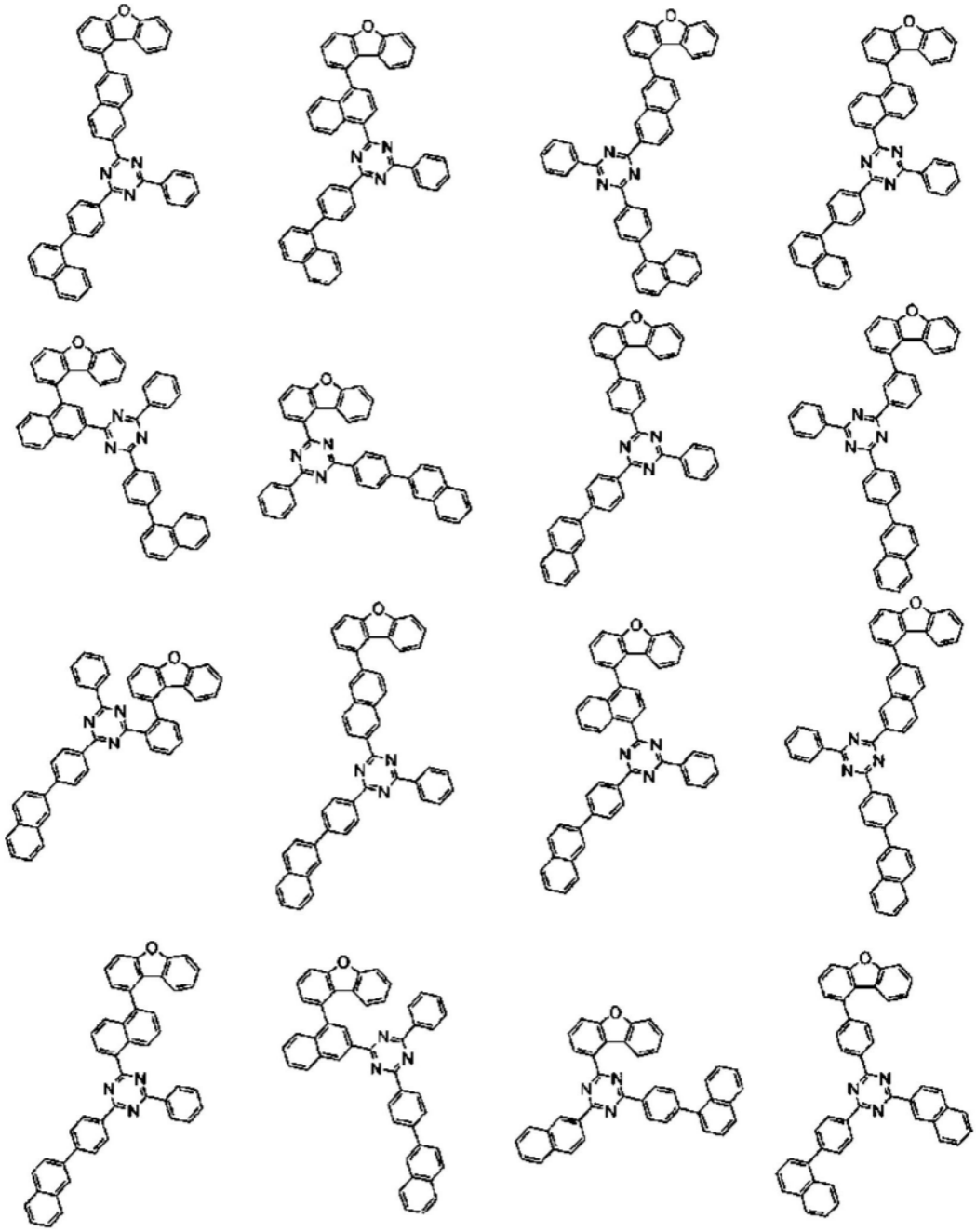




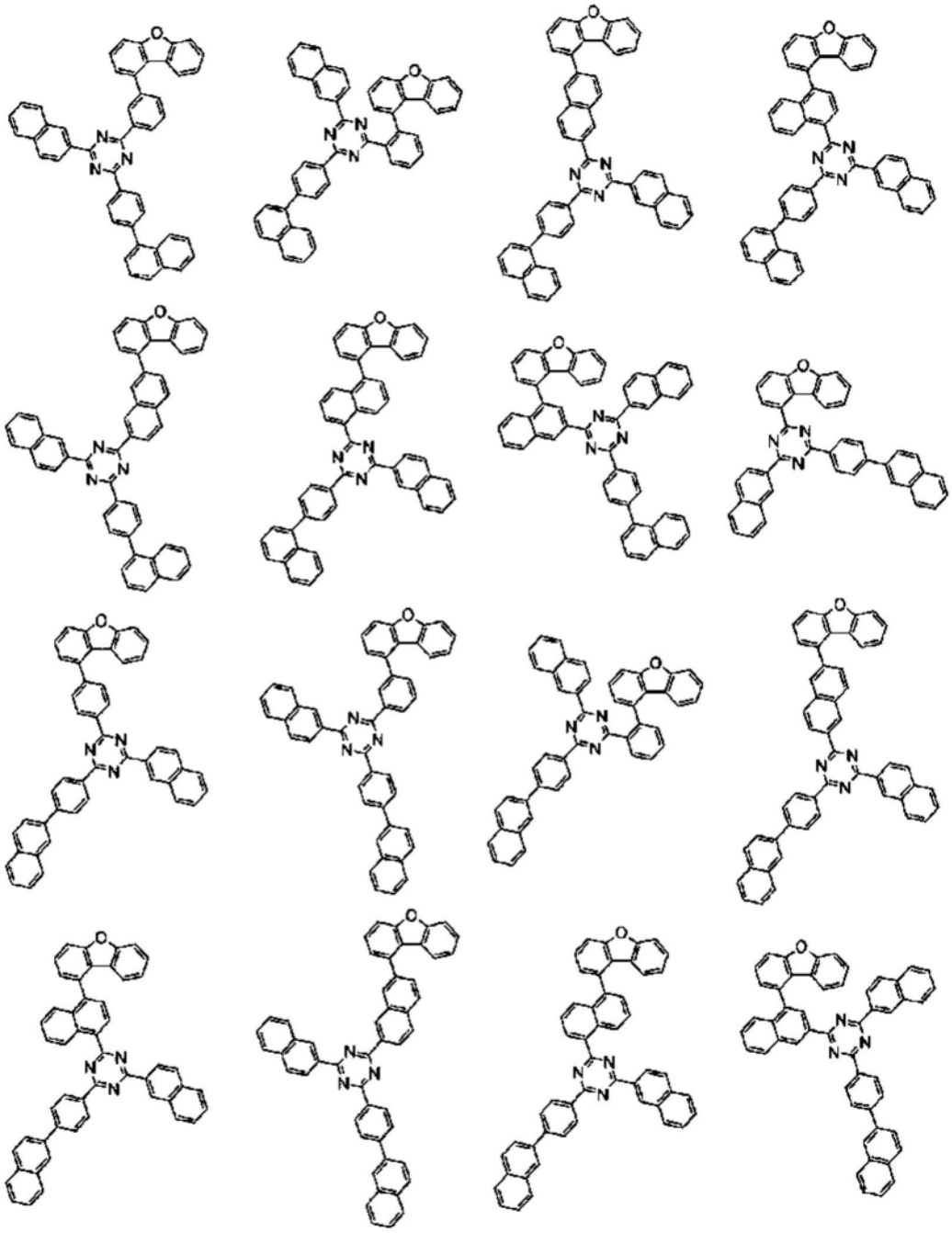
[0196]

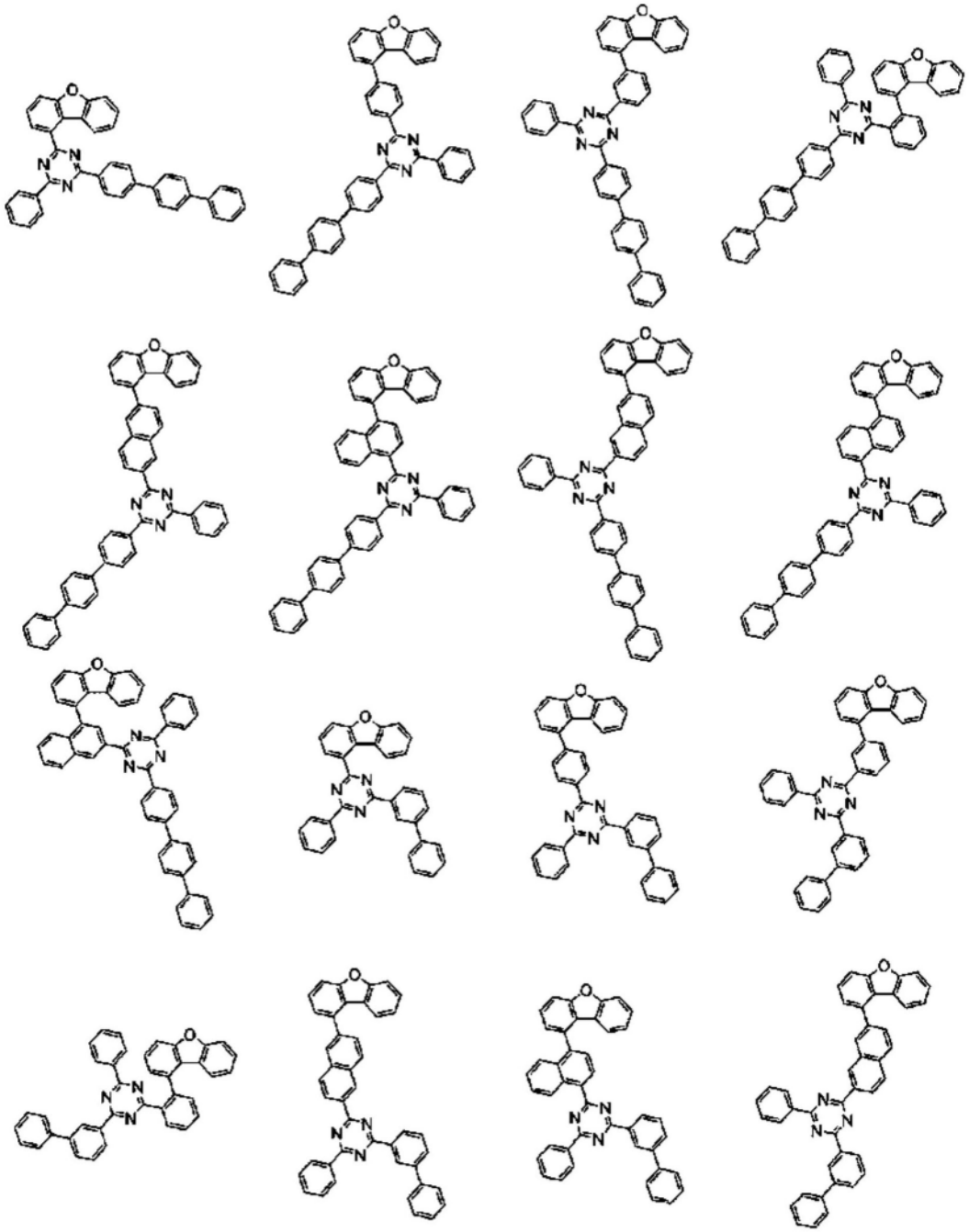


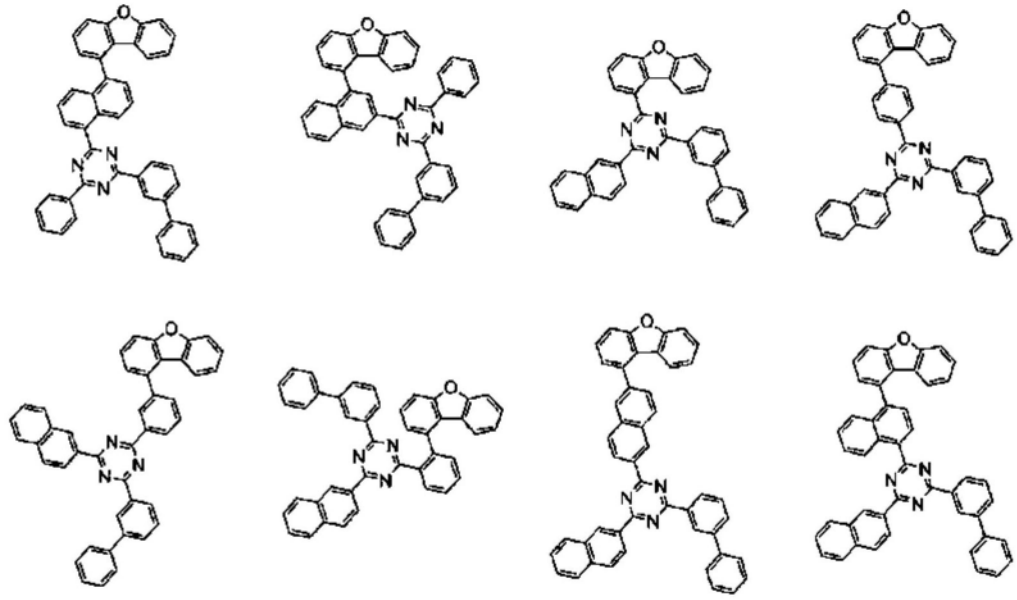
[0197]



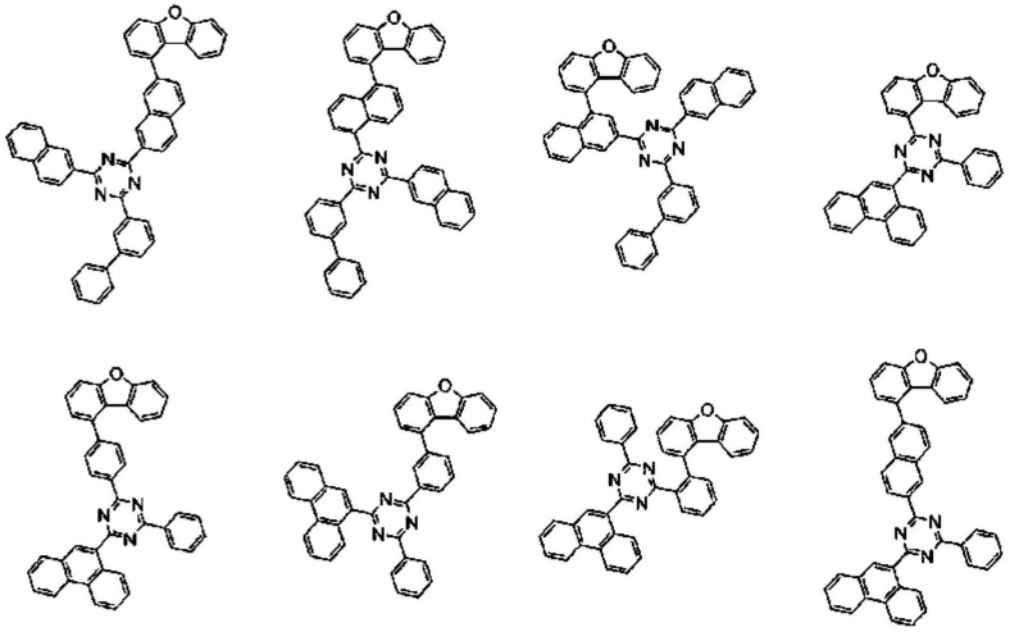
[0198]

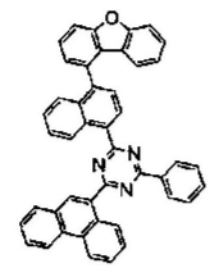
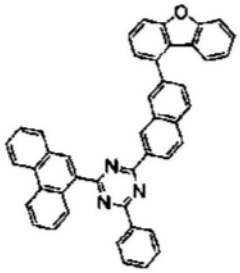
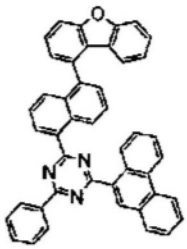
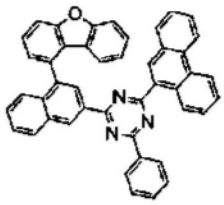
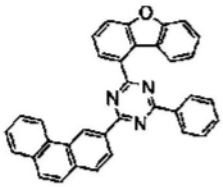
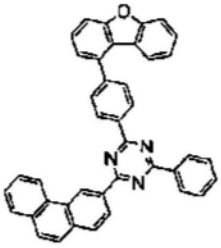
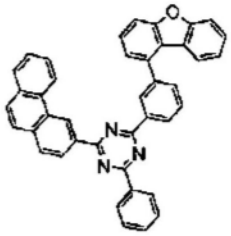
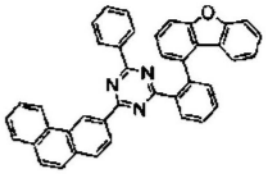


[0199] 

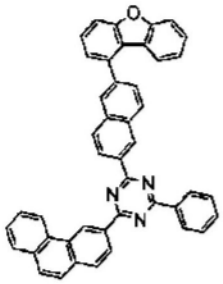
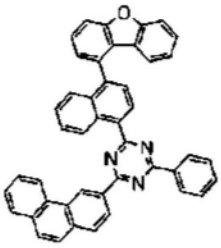
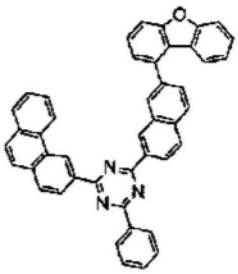
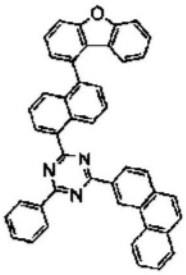
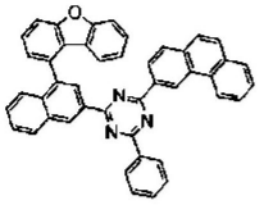
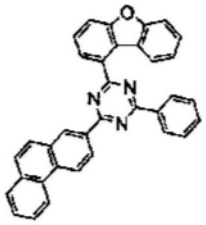
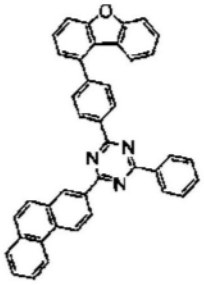
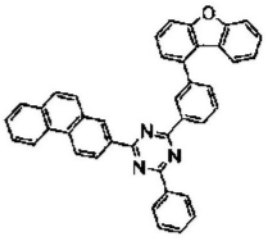


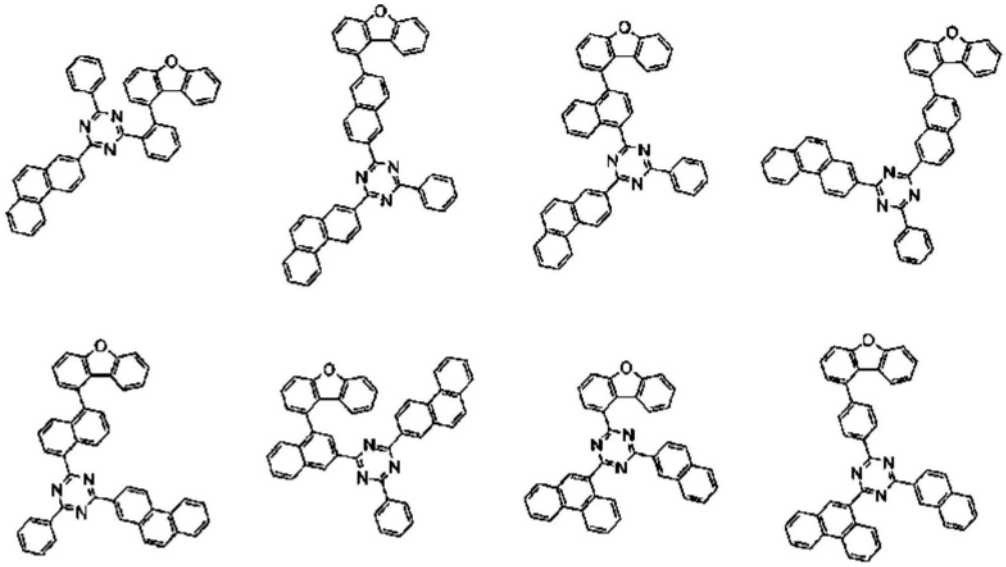
[0200]



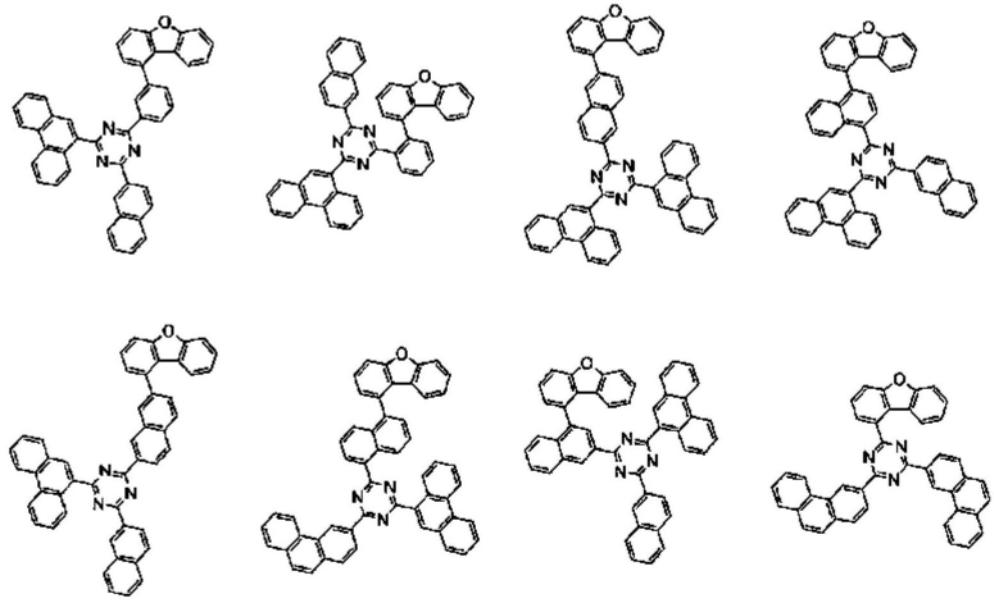









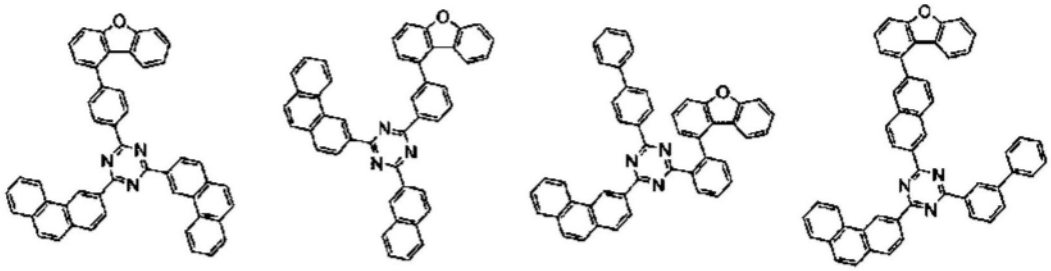
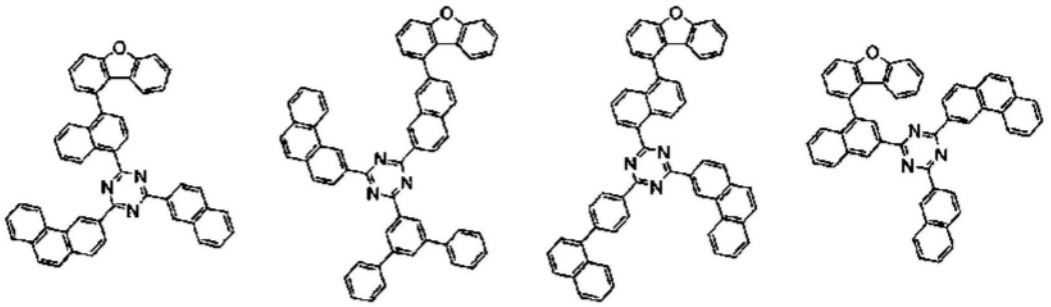
[0201]

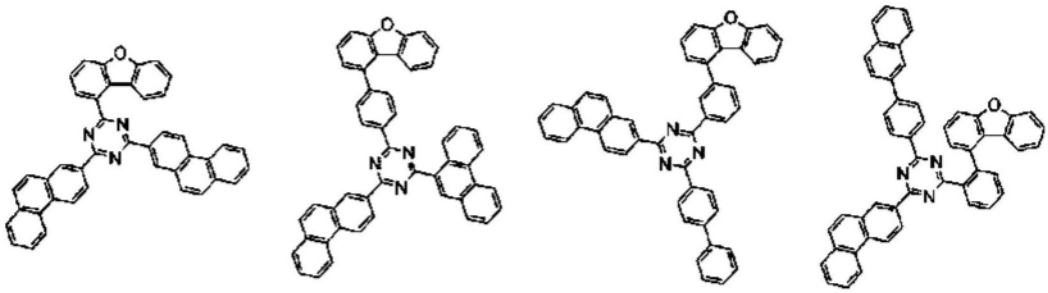
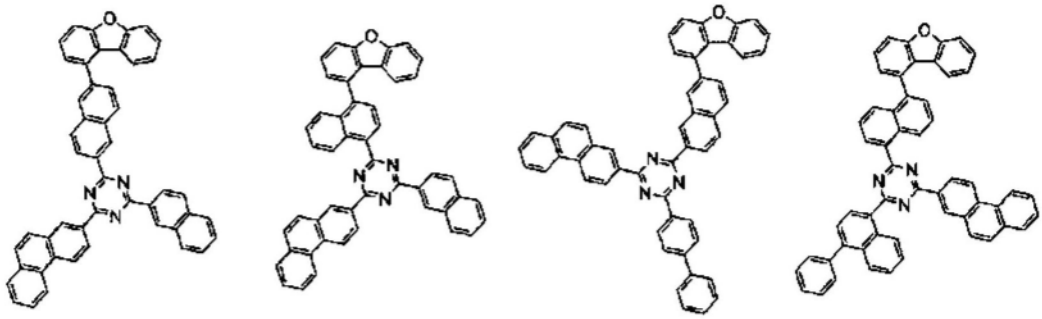


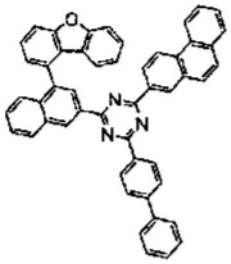
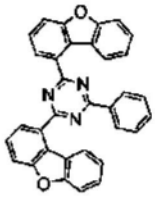
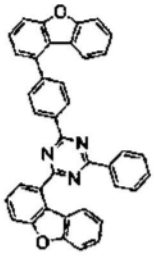
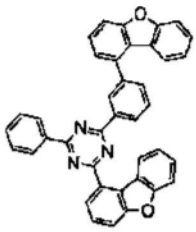
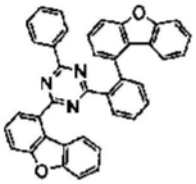
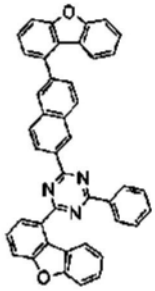
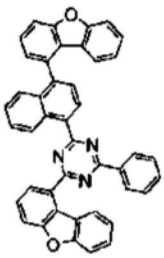
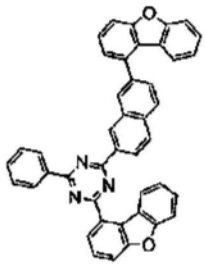
[0202]



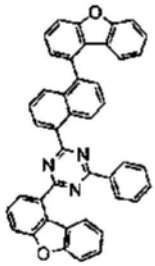
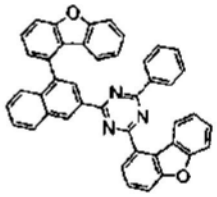
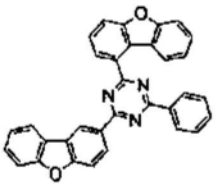
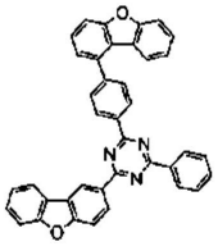
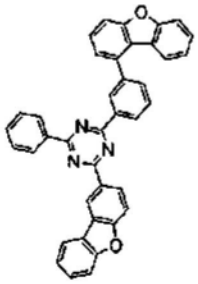
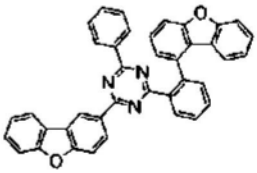
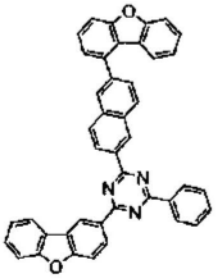
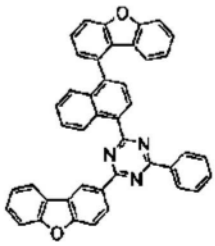



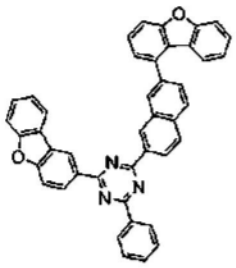
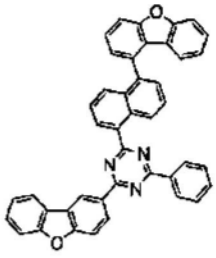
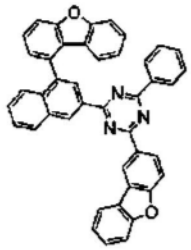
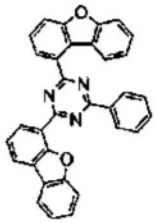
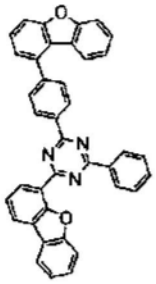
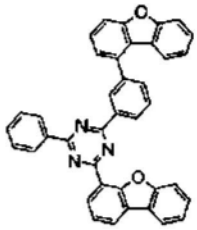
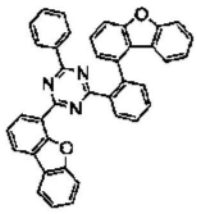
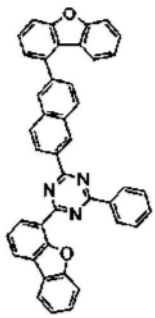
[0203]

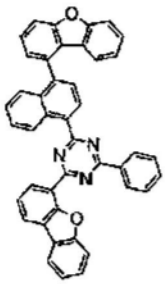
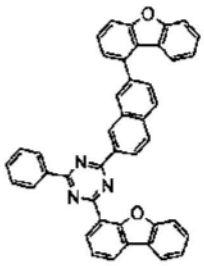
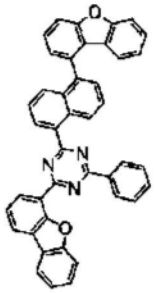
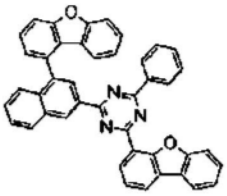
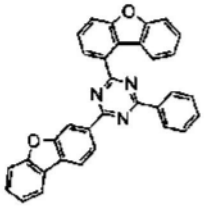
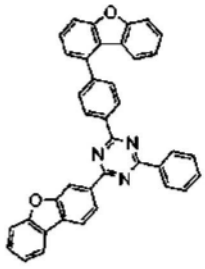
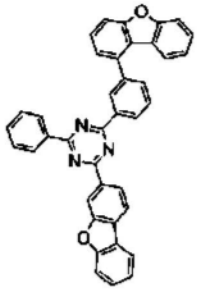
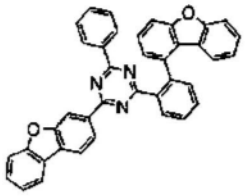









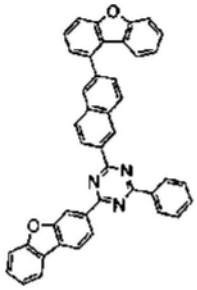
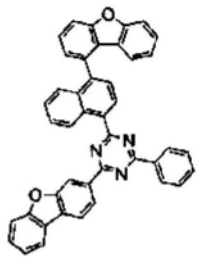
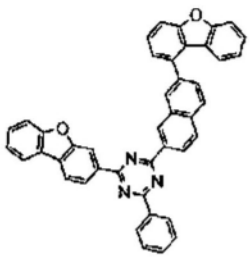
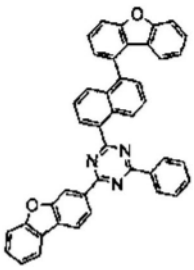
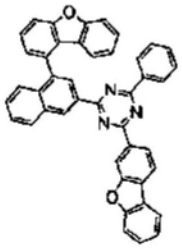
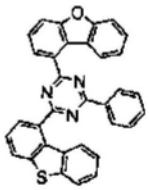
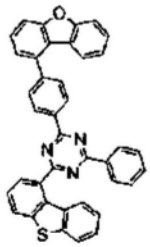
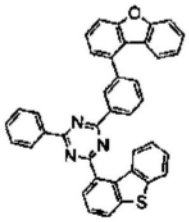
[0204]

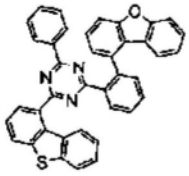
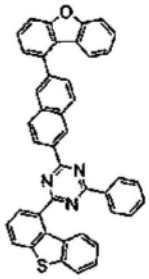
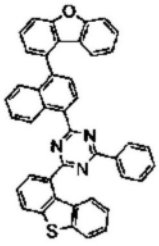
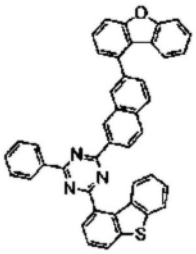
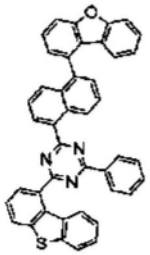
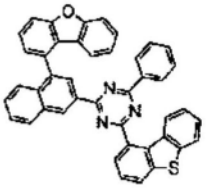
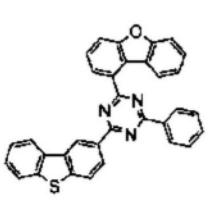
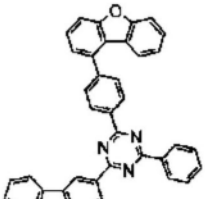









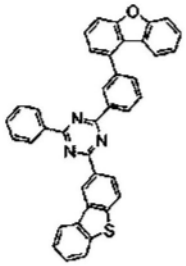
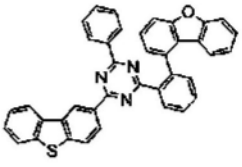
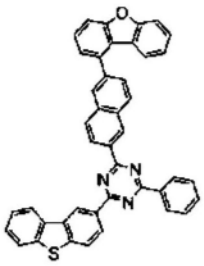
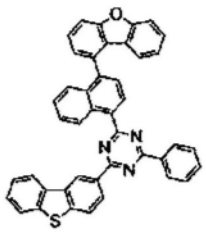
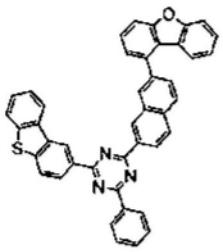
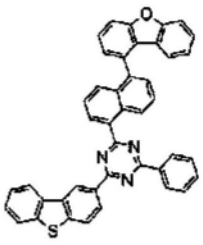
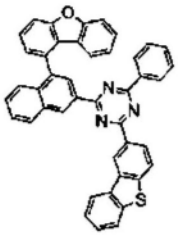
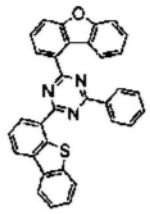
[0205]

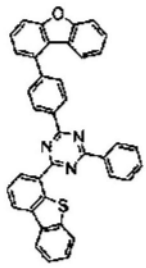
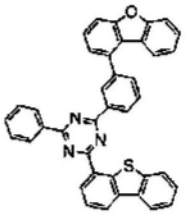
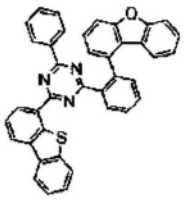
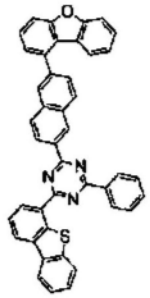
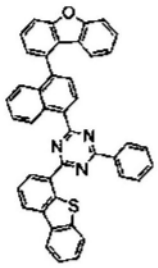
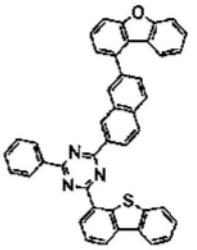
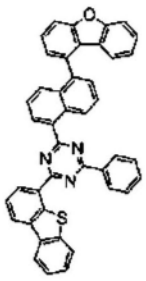
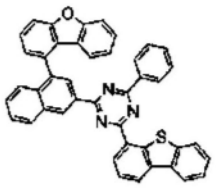









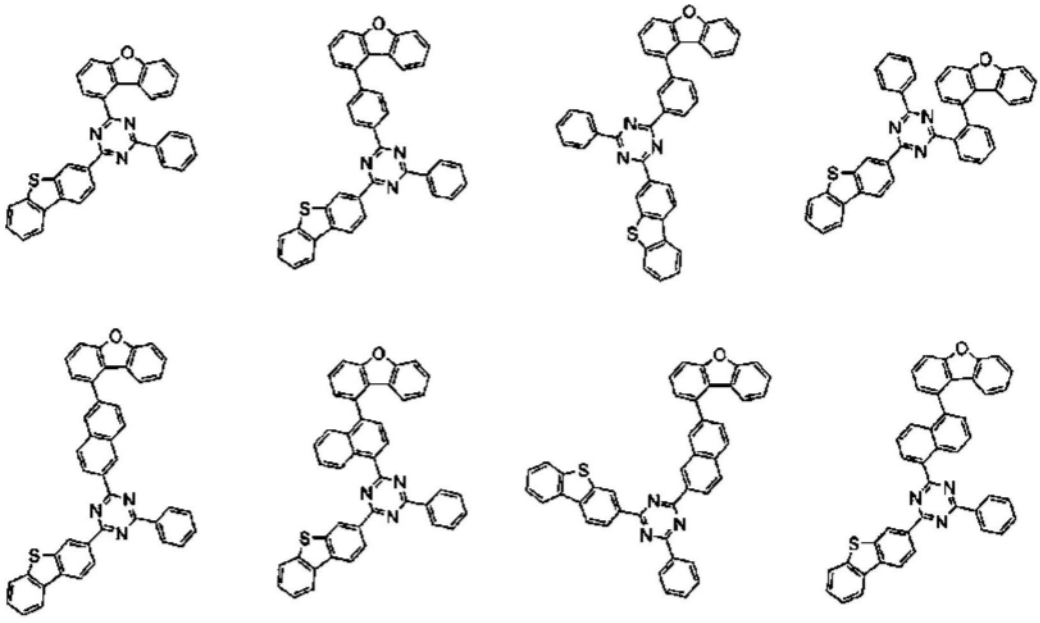
[0206]

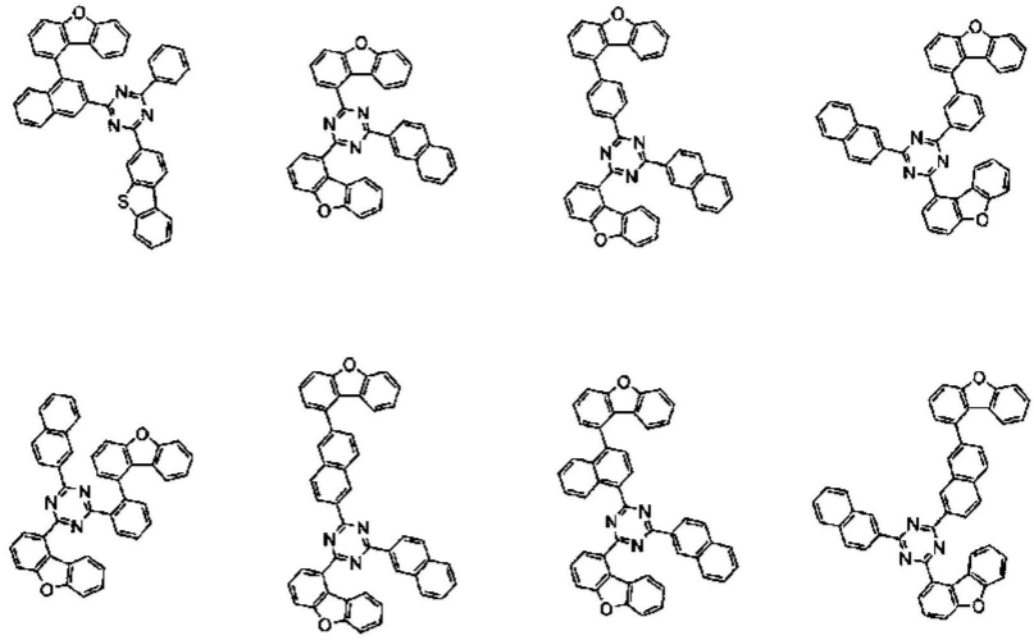









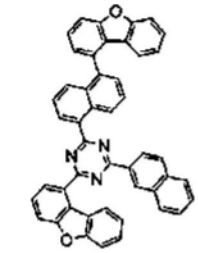
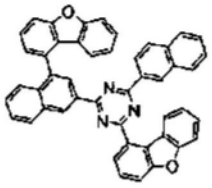
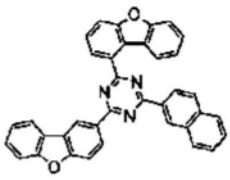
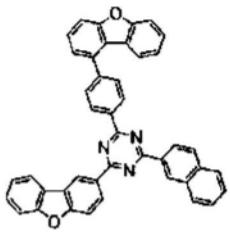
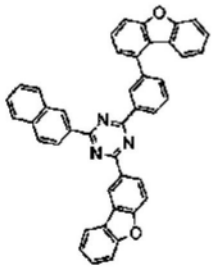
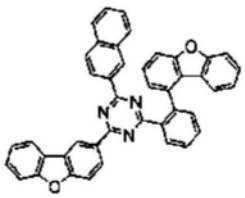
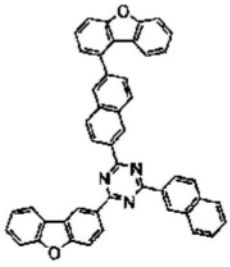
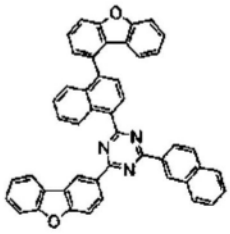
[0207]

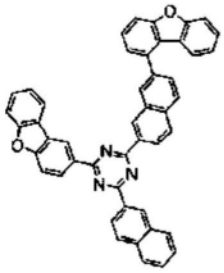
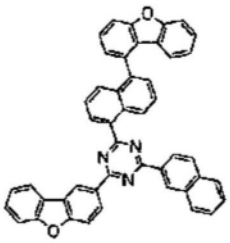
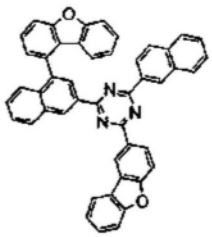
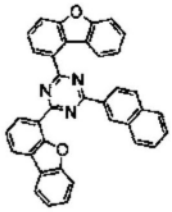
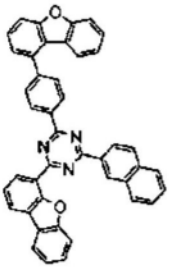
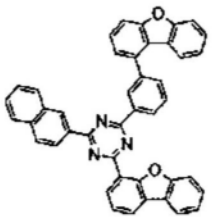
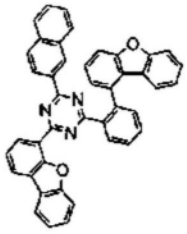
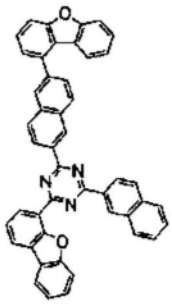


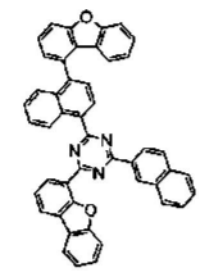
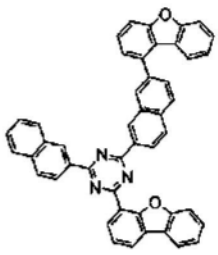
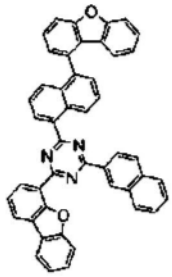
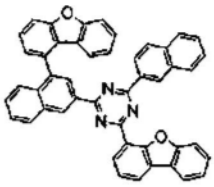
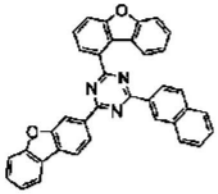
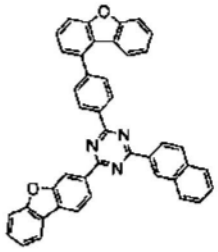
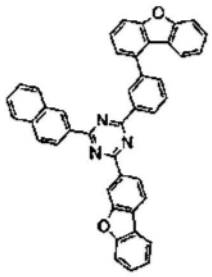
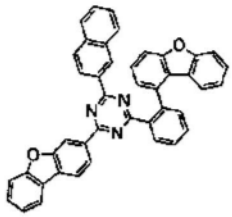
[0208]



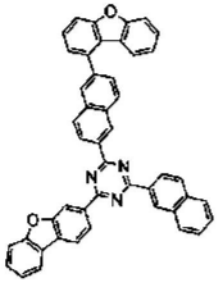
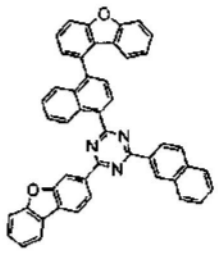
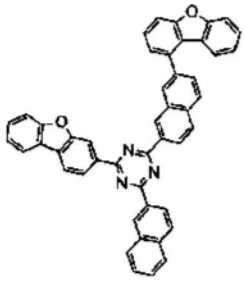
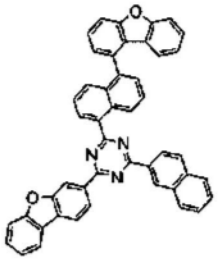
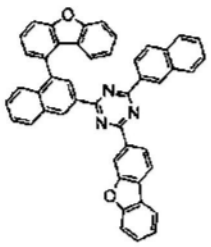
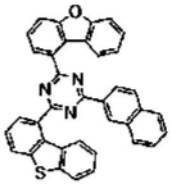
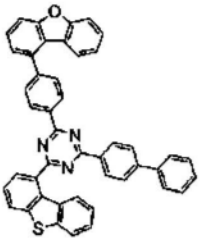
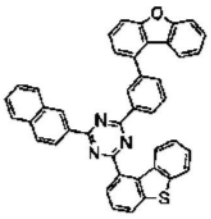









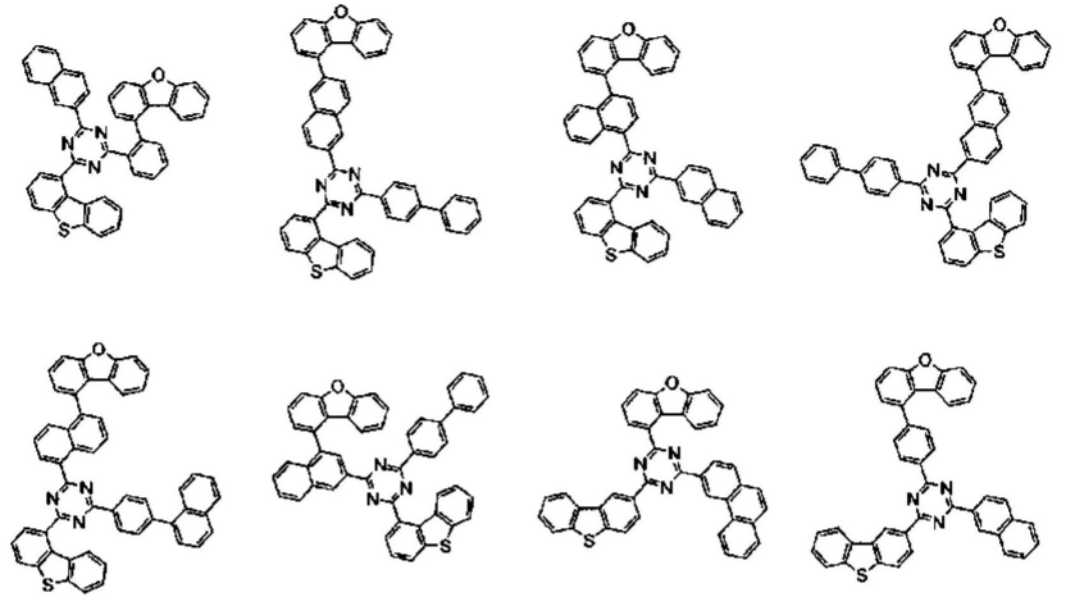
[0209]

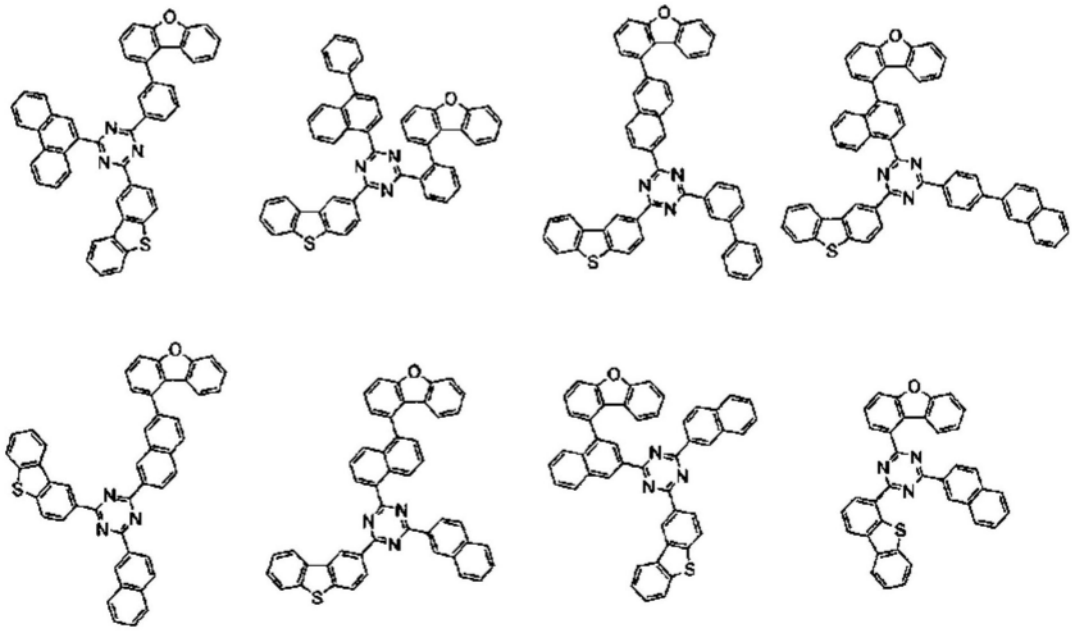









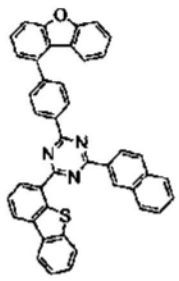
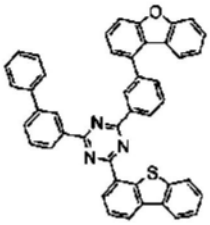
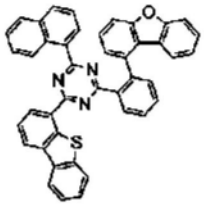
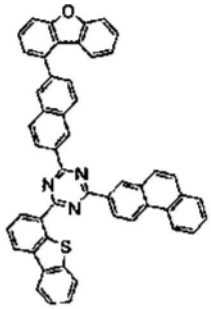
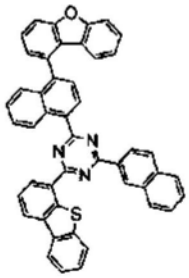
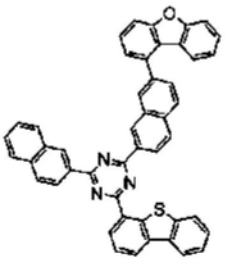
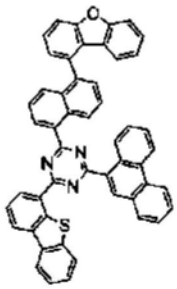
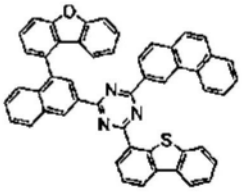
[0210]

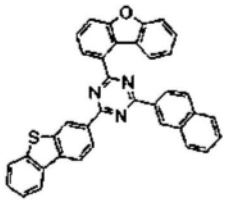
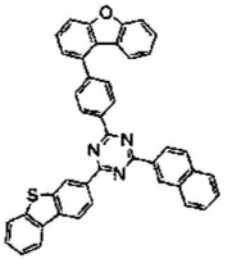
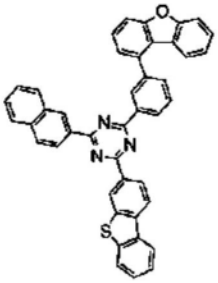
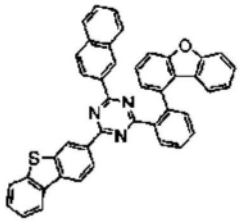
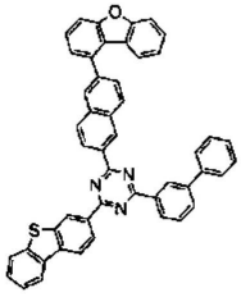
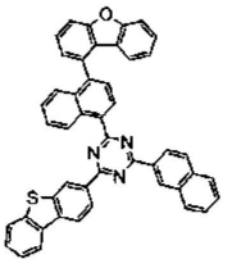
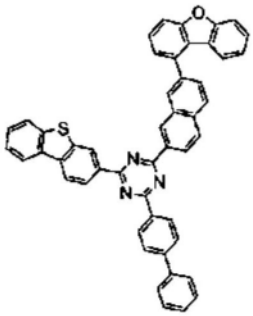
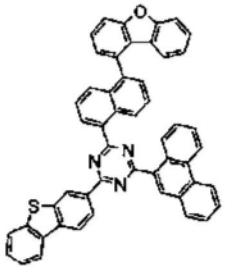


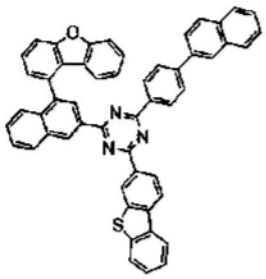
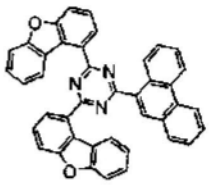
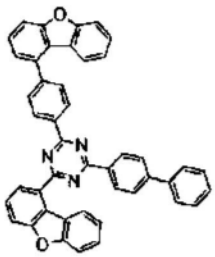
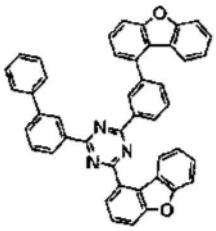
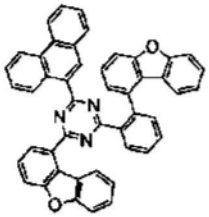
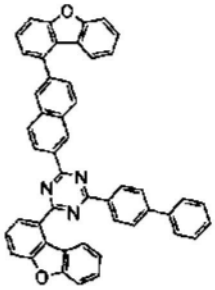
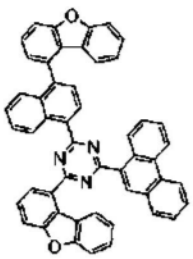
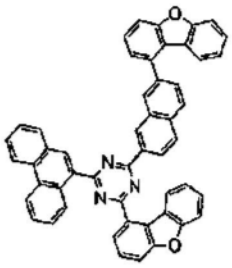
[0211]



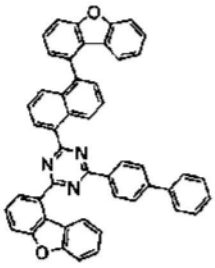
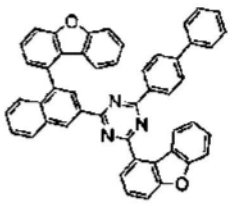
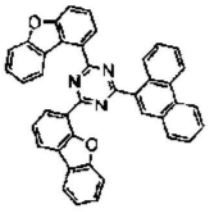
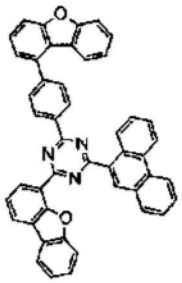
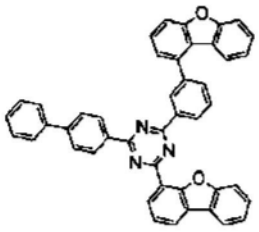
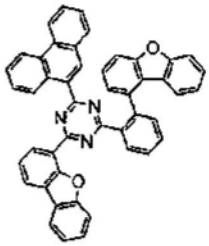
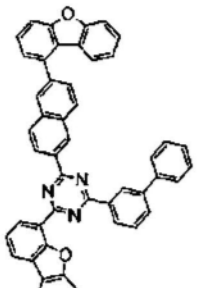
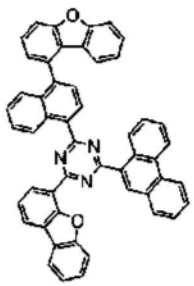









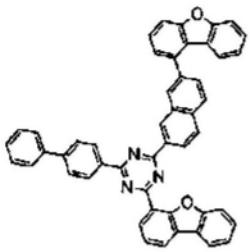
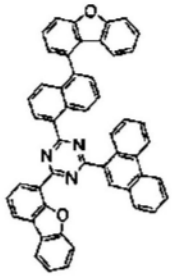
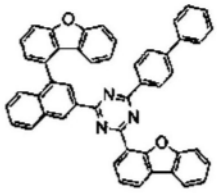
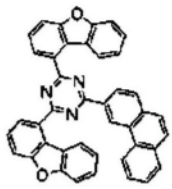
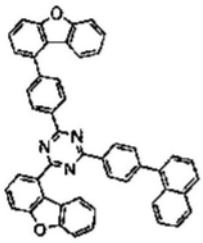
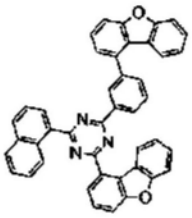
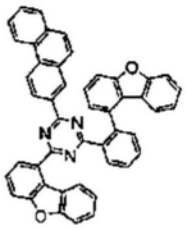
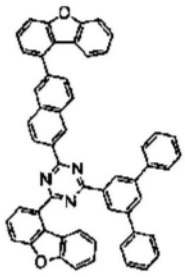
[0212]

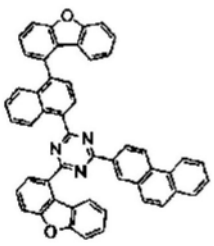
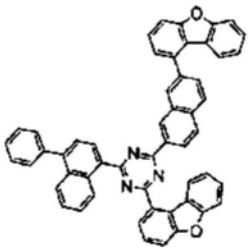
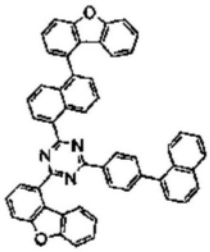
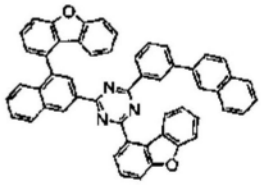
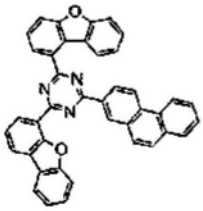
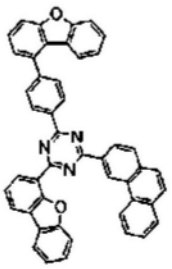
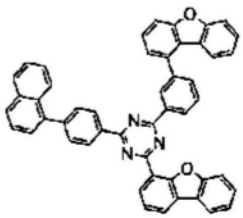
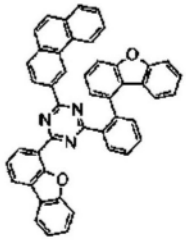









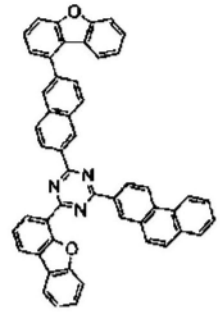
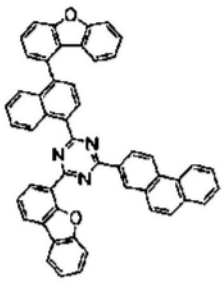
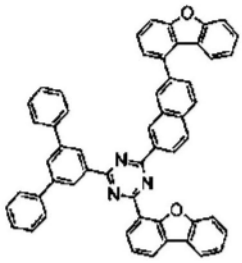
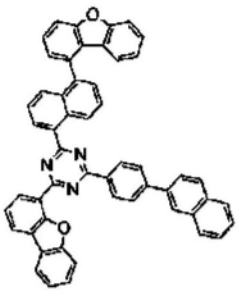
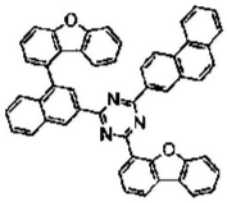
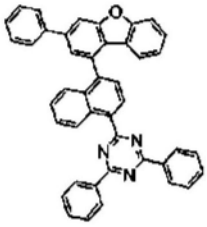
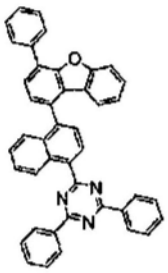
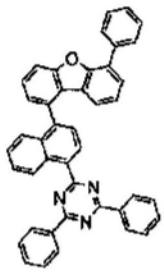
[0213]

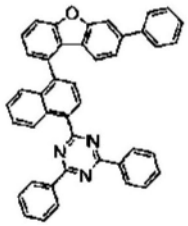
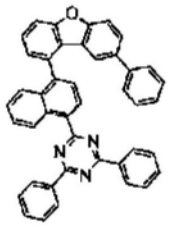
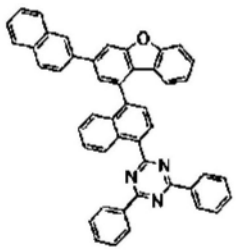
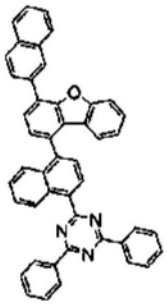
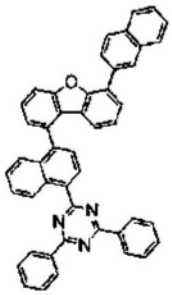
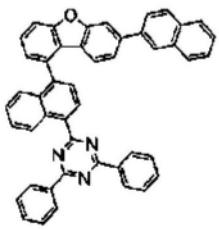
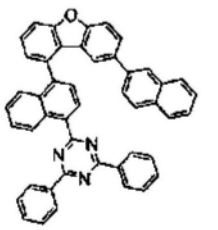
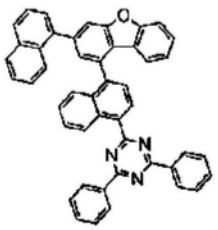









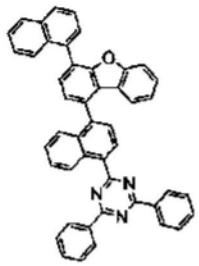
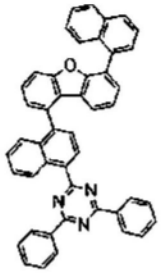
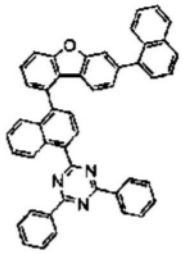
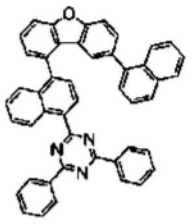
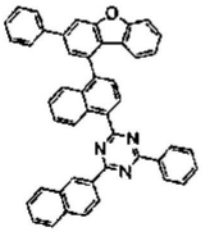
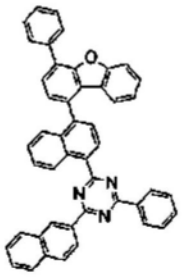
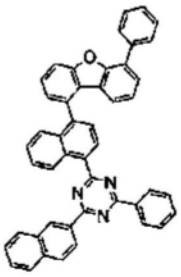
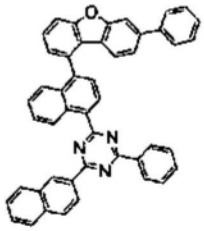
[0214]

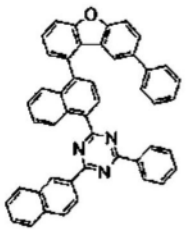
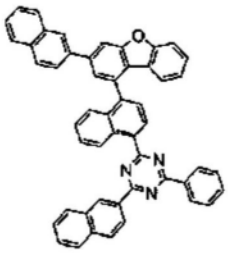
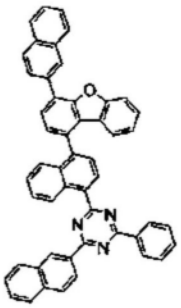
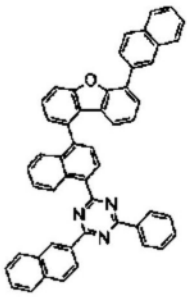
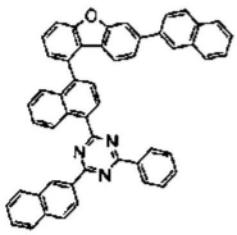
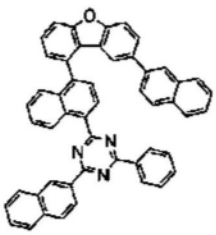
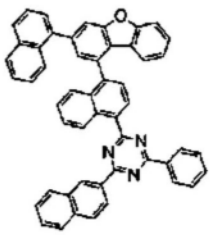
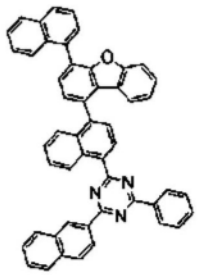









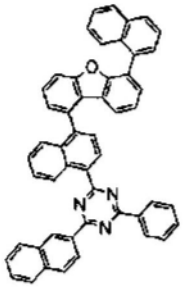
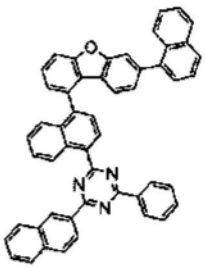
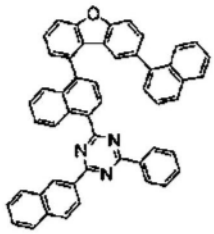
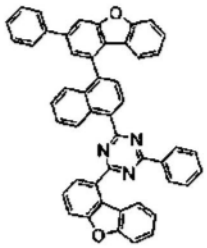
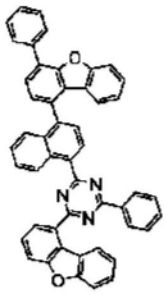
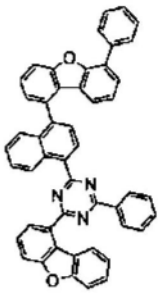
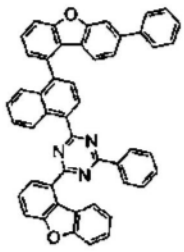
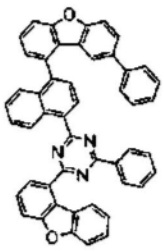
[0215]

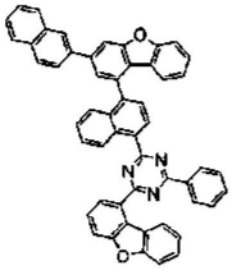
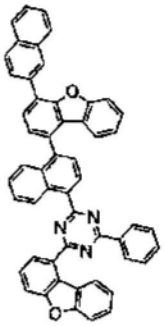
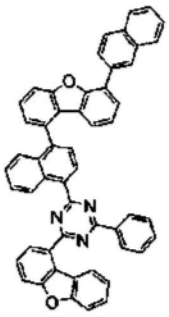
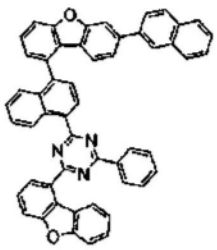
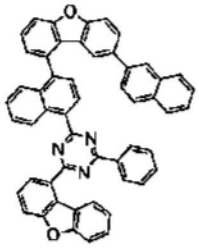
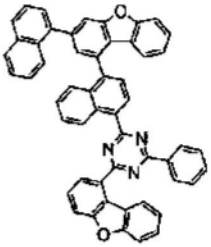
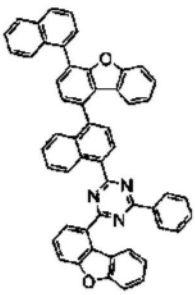
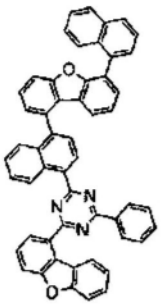









[0216]

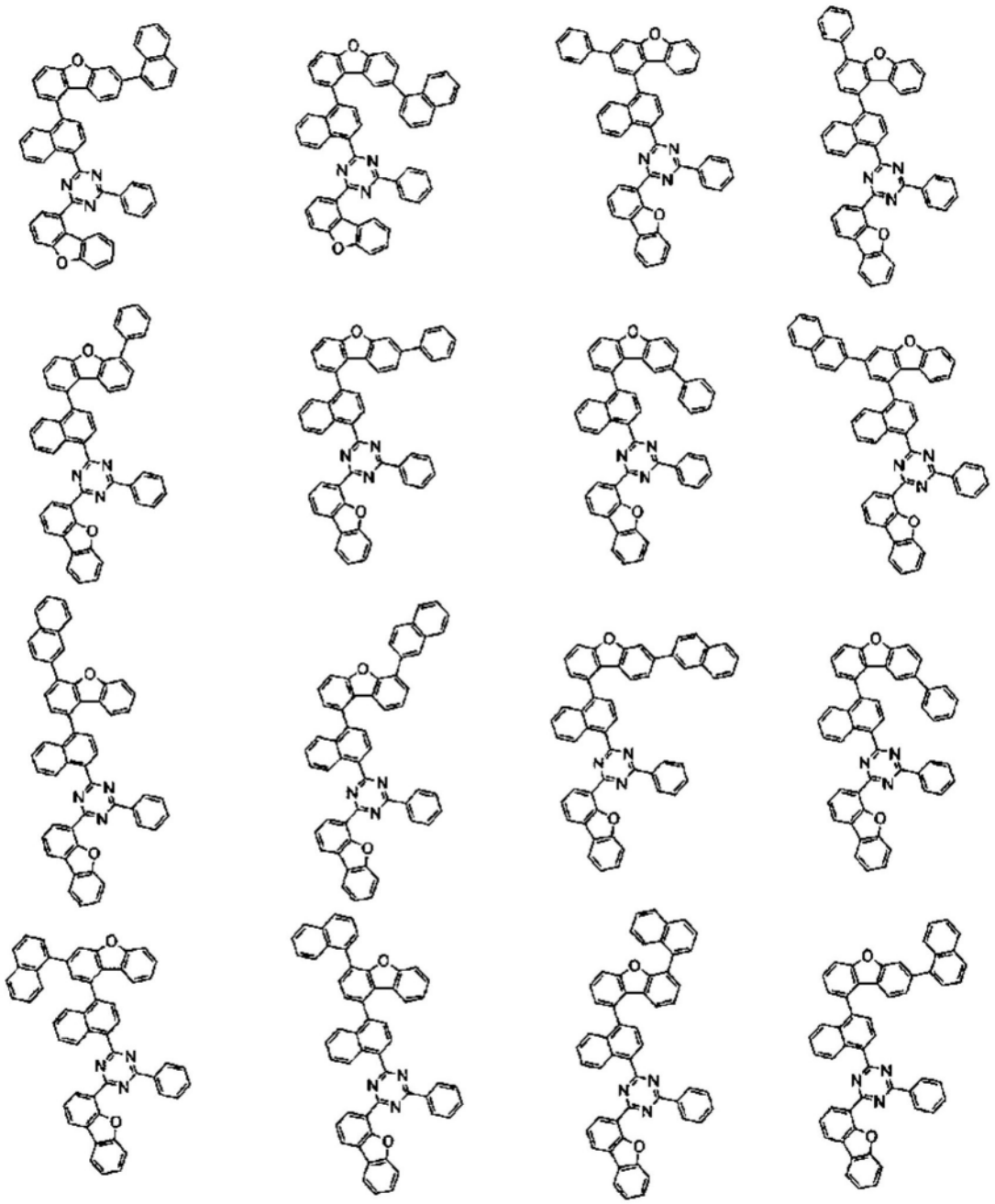









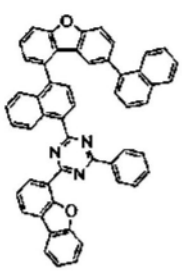
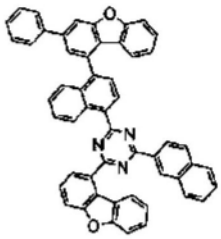
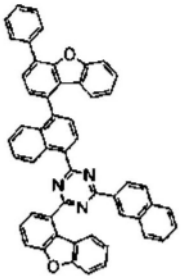
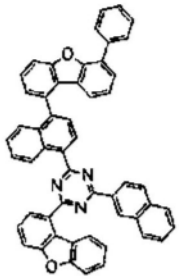
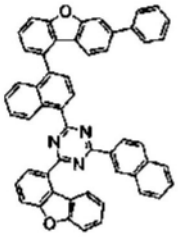
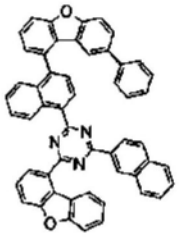
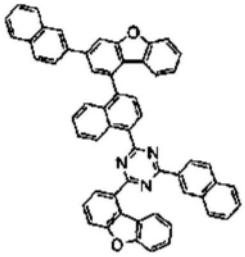
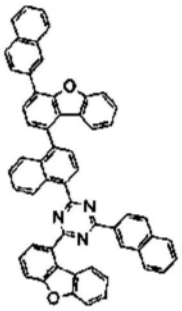









[0217]

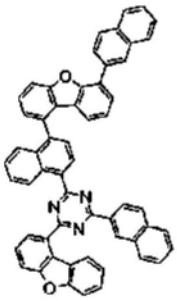
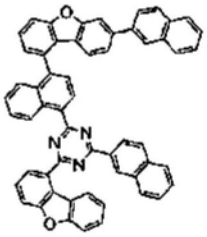
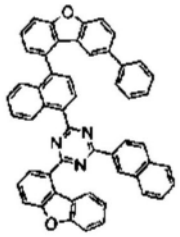
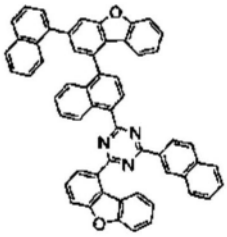
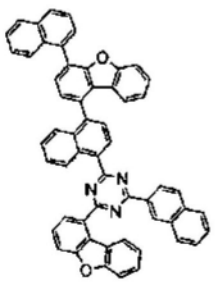
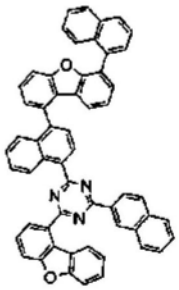
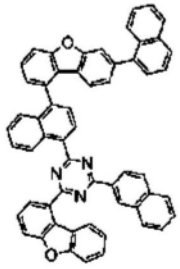
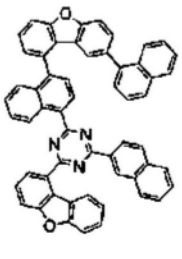









[0218]

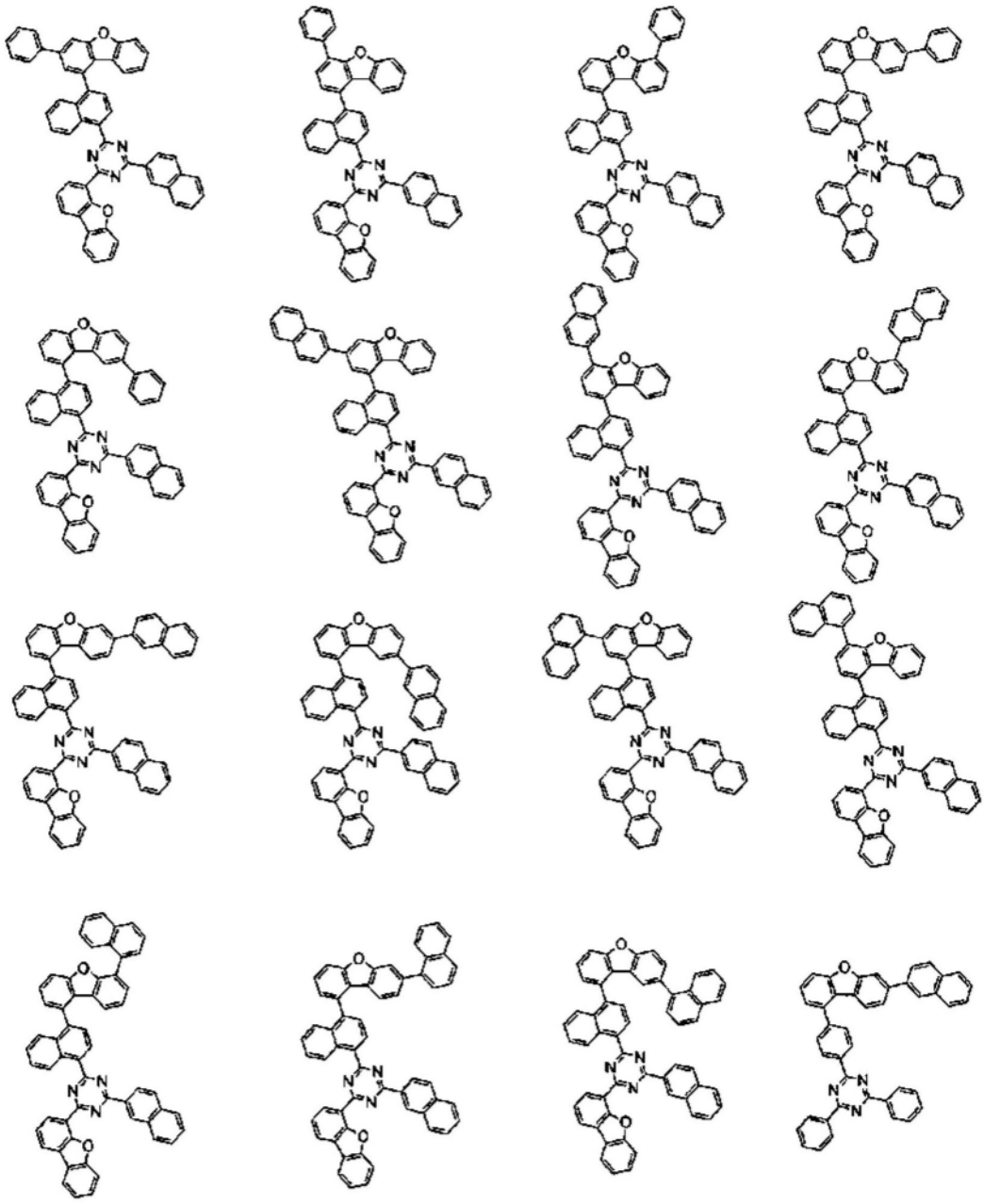


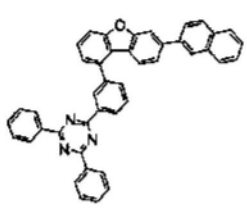
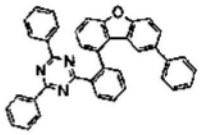
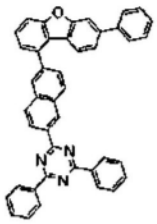
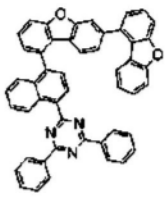
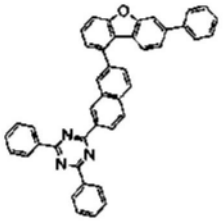
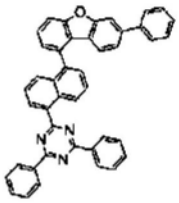
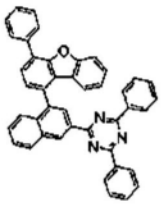
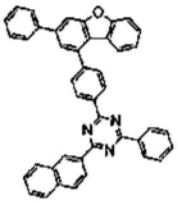









[0219]

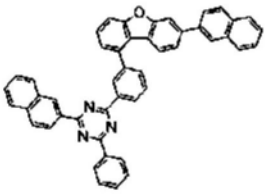
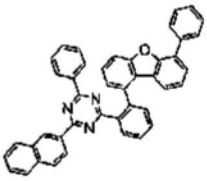
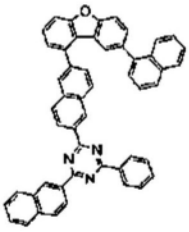
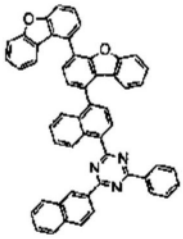
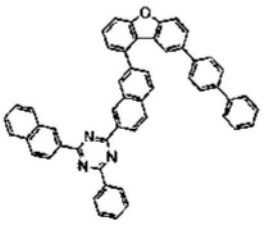
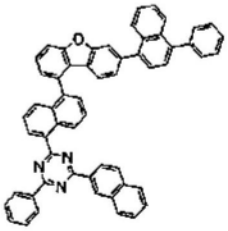
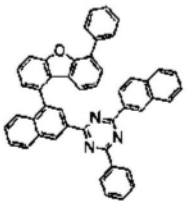
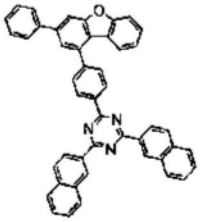









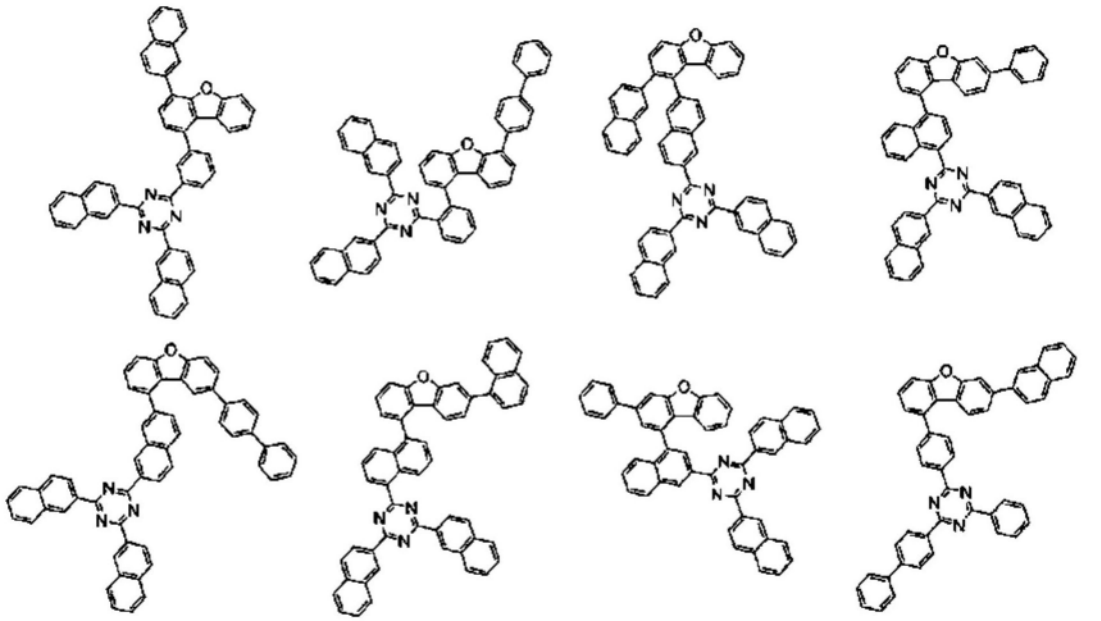
[0220]



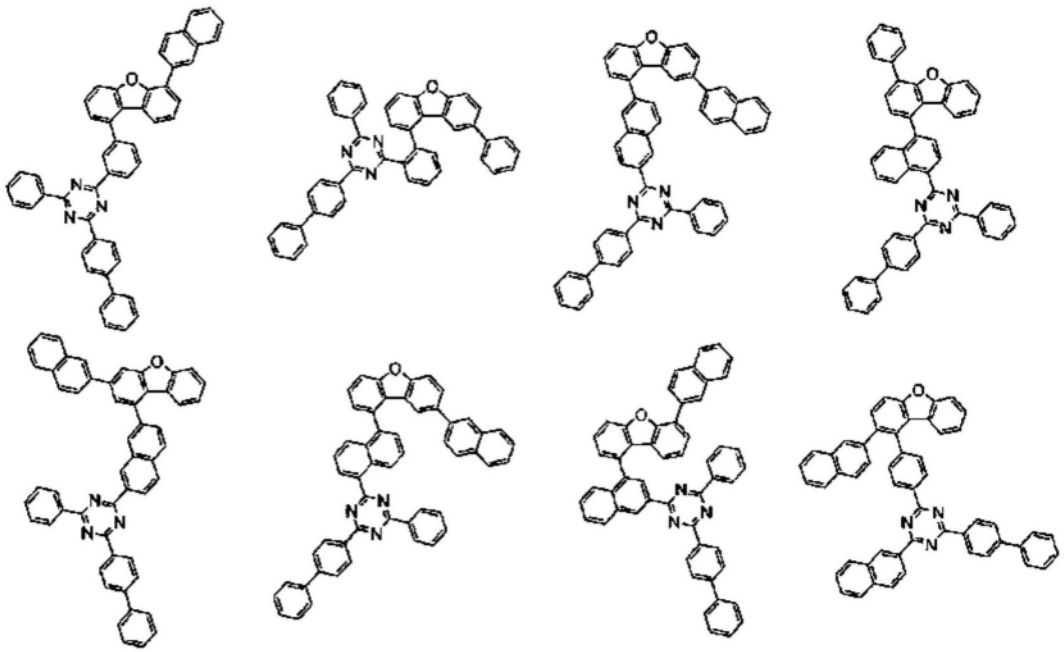









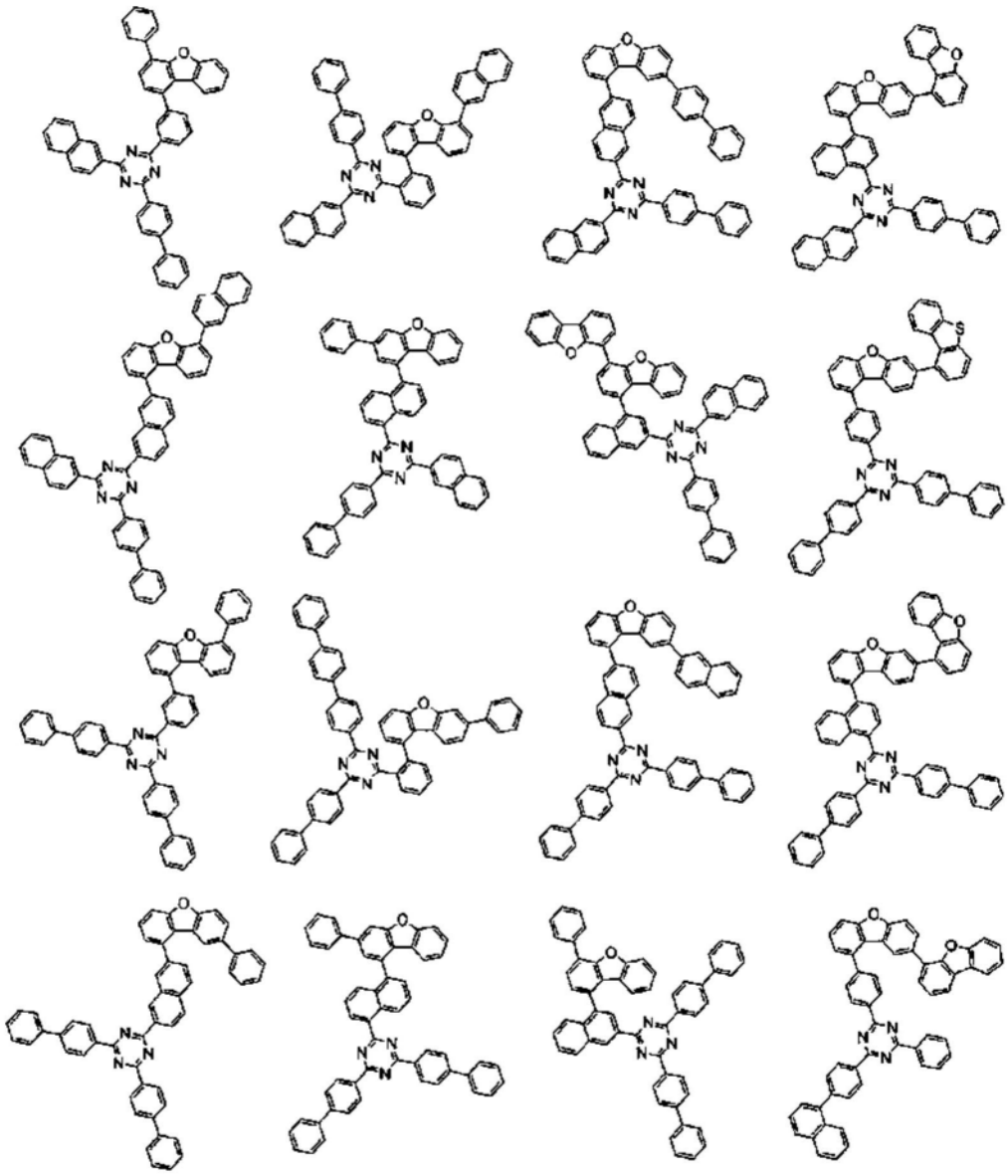
[0221]

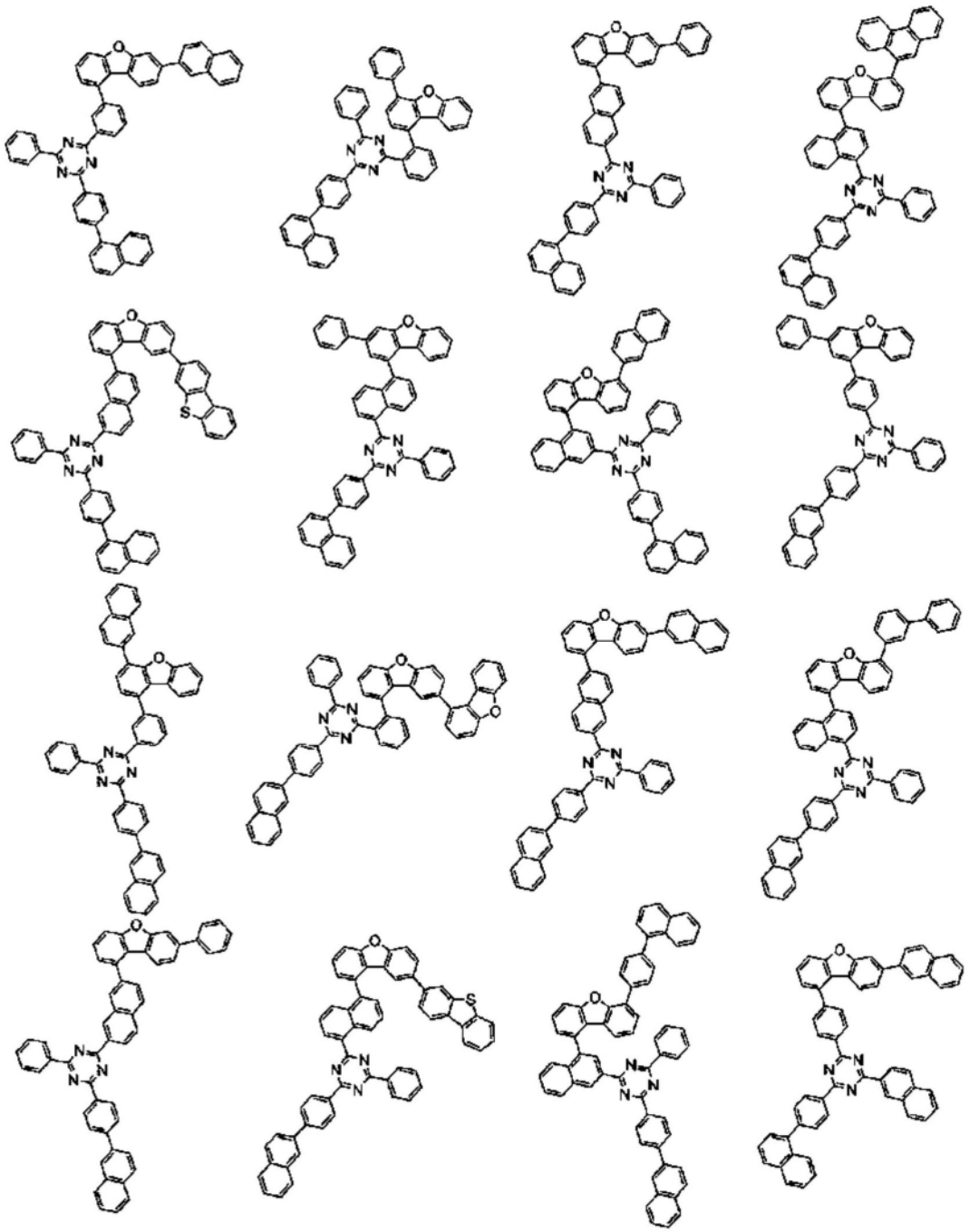


[0222]

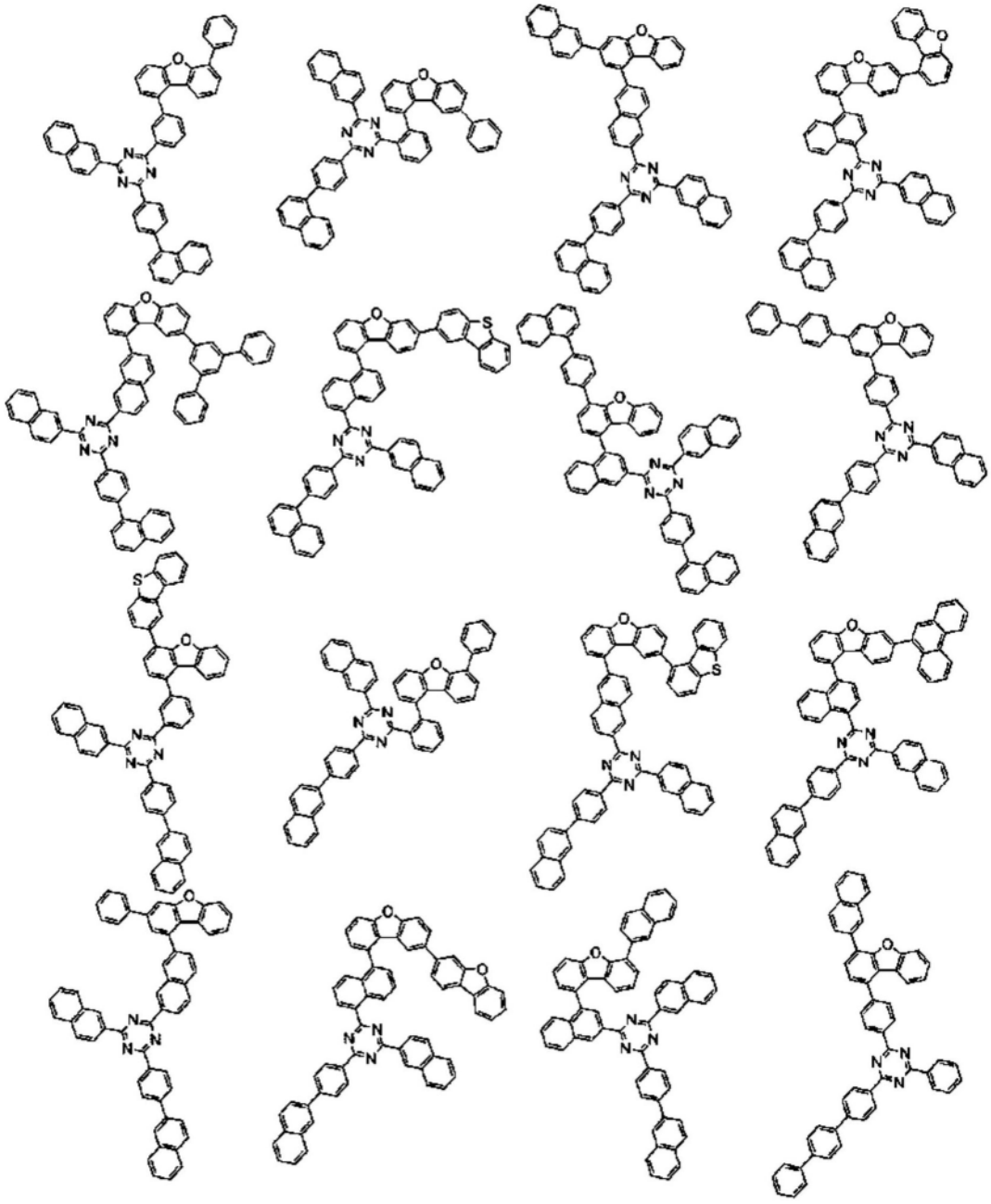


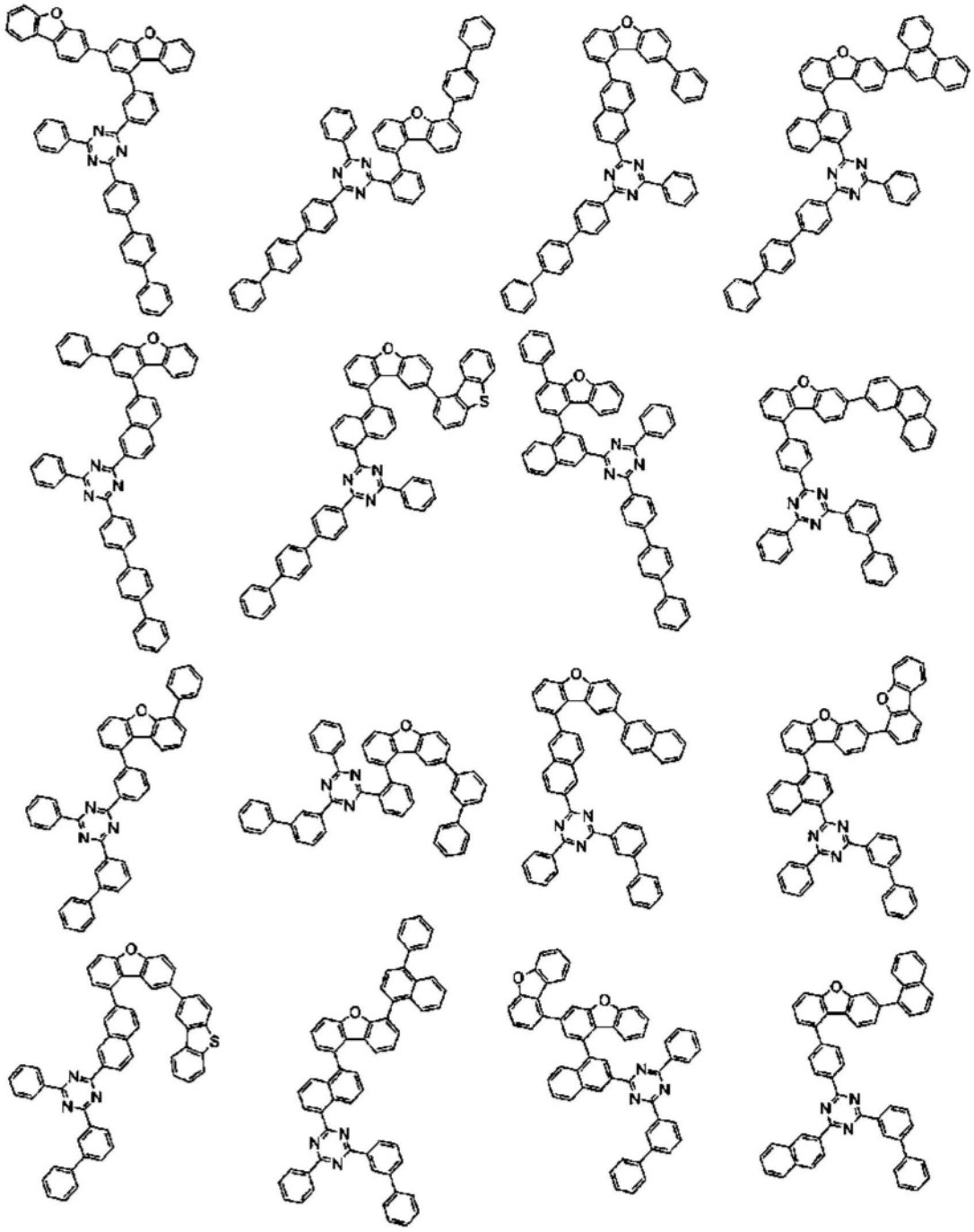
[0223] 

[0224]

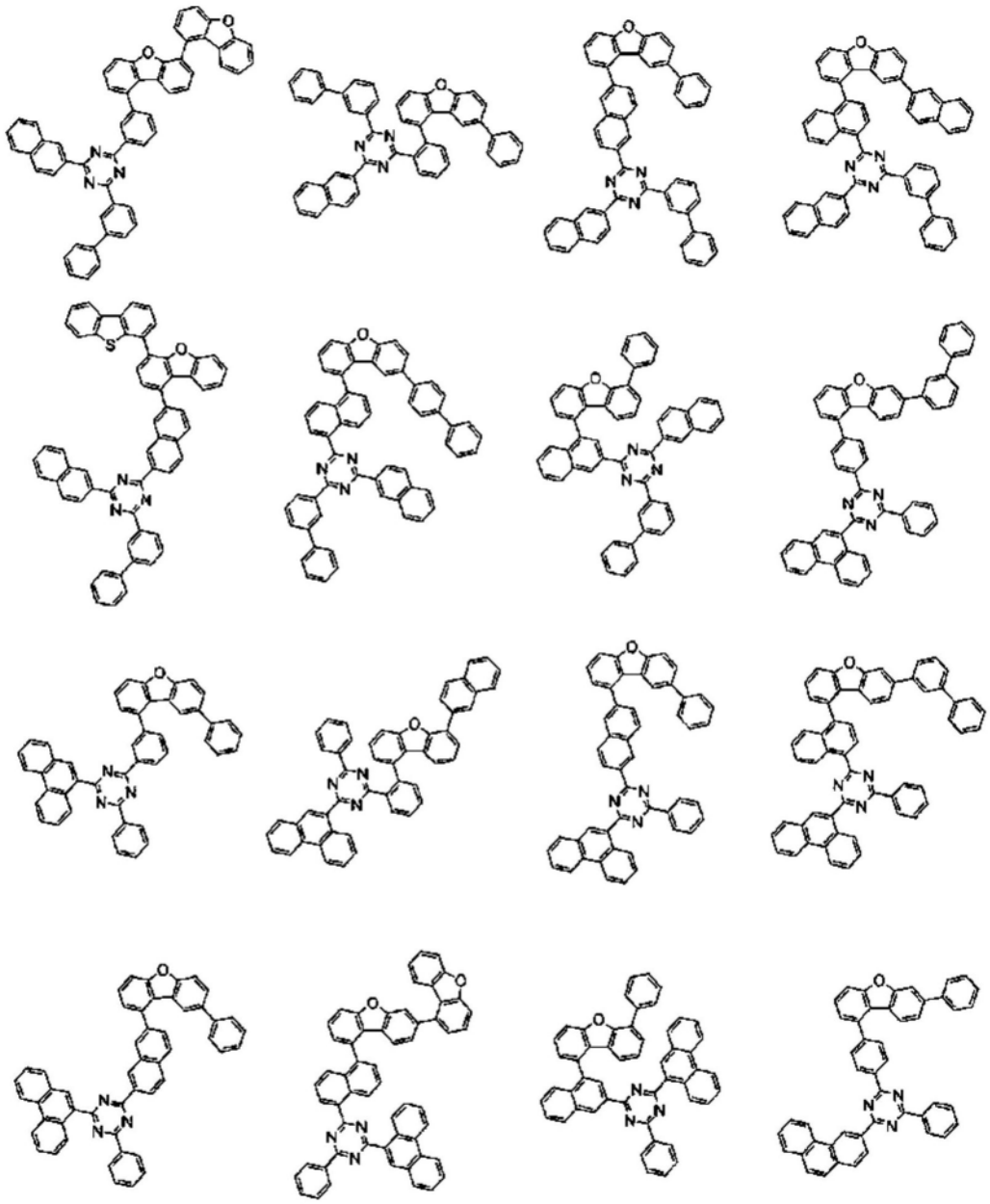


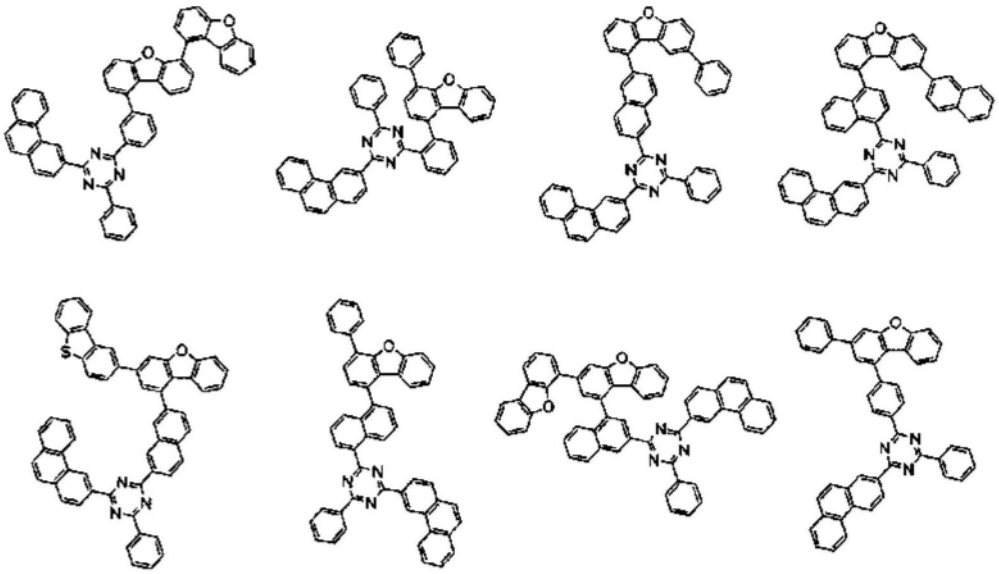
[0225]



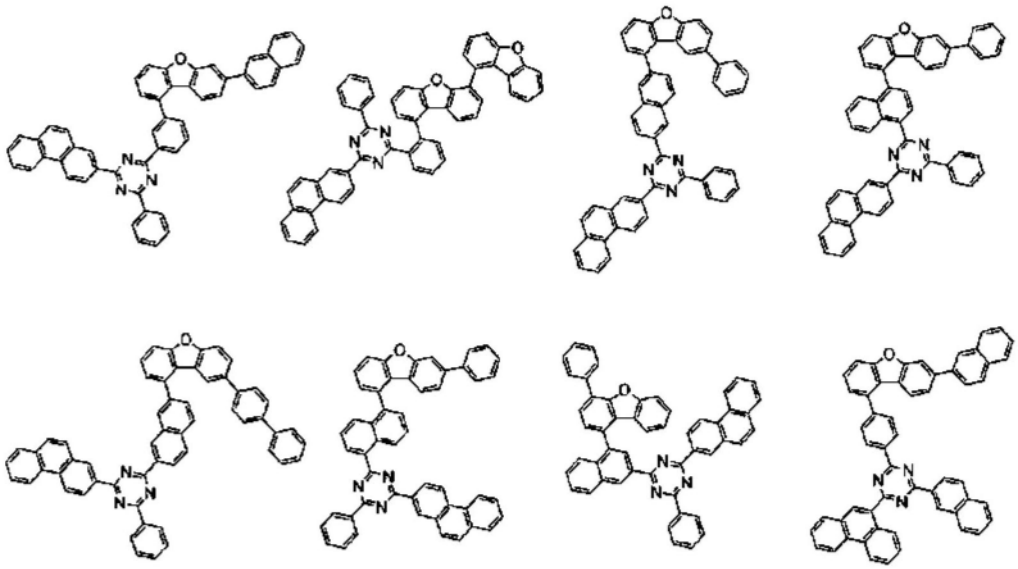
[0226] 

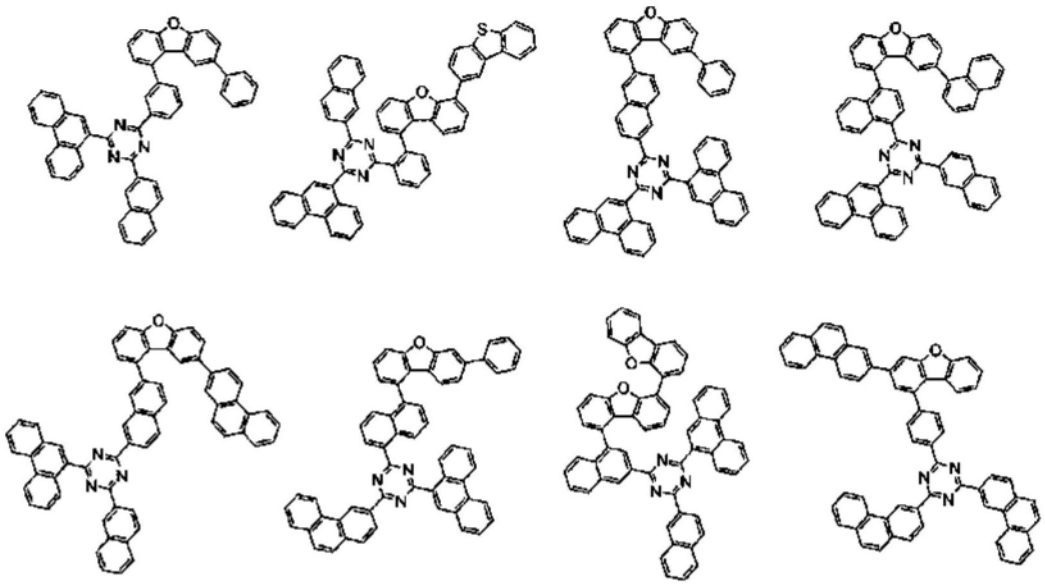
[0227]



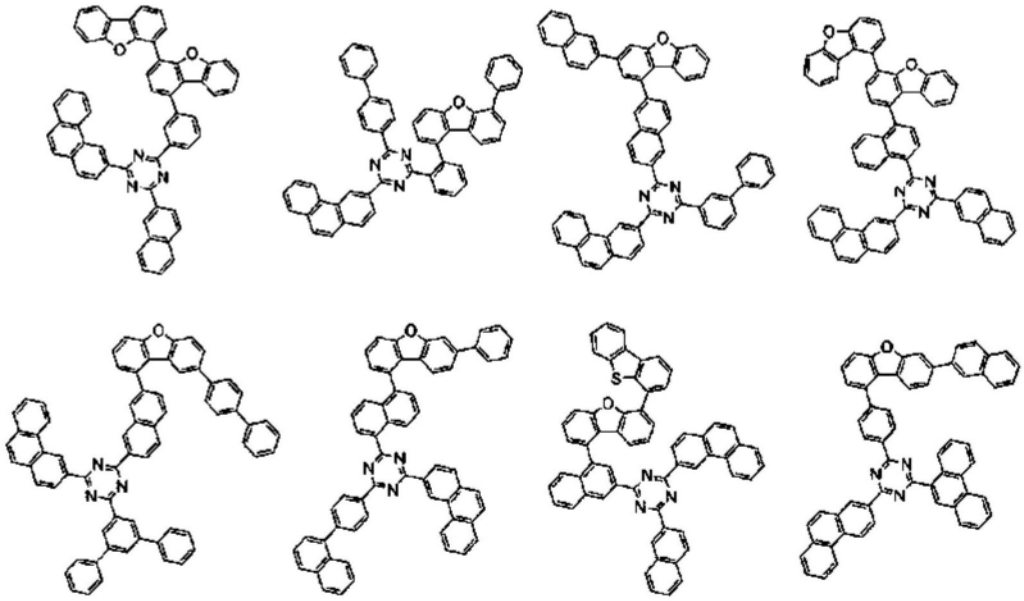


[0228]

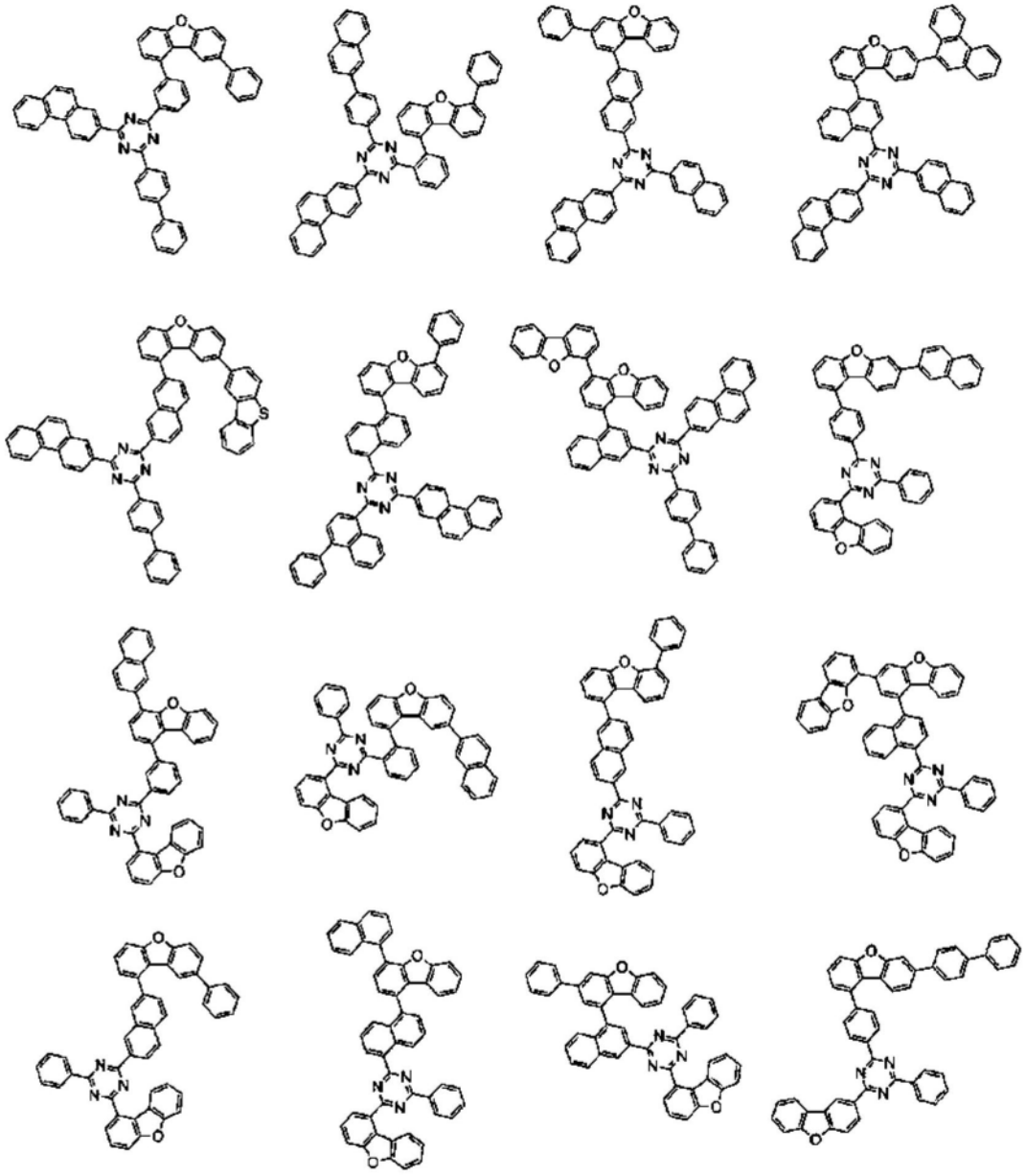


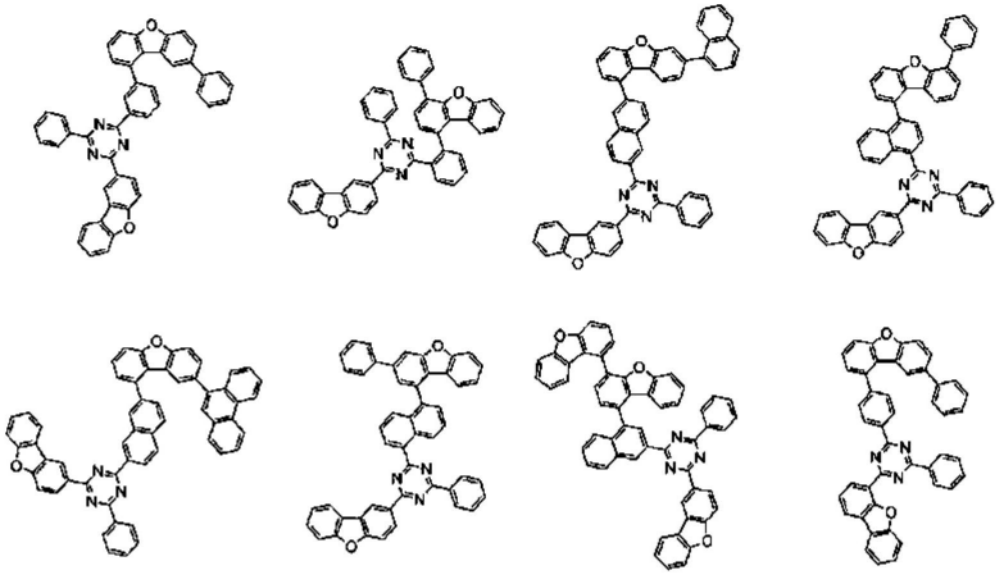


[0229]

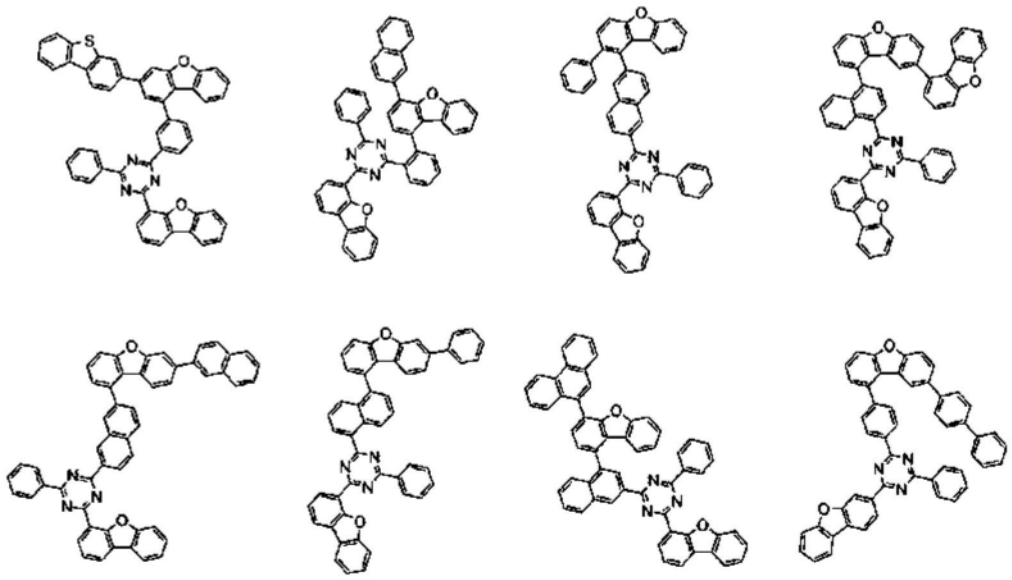


[0230]

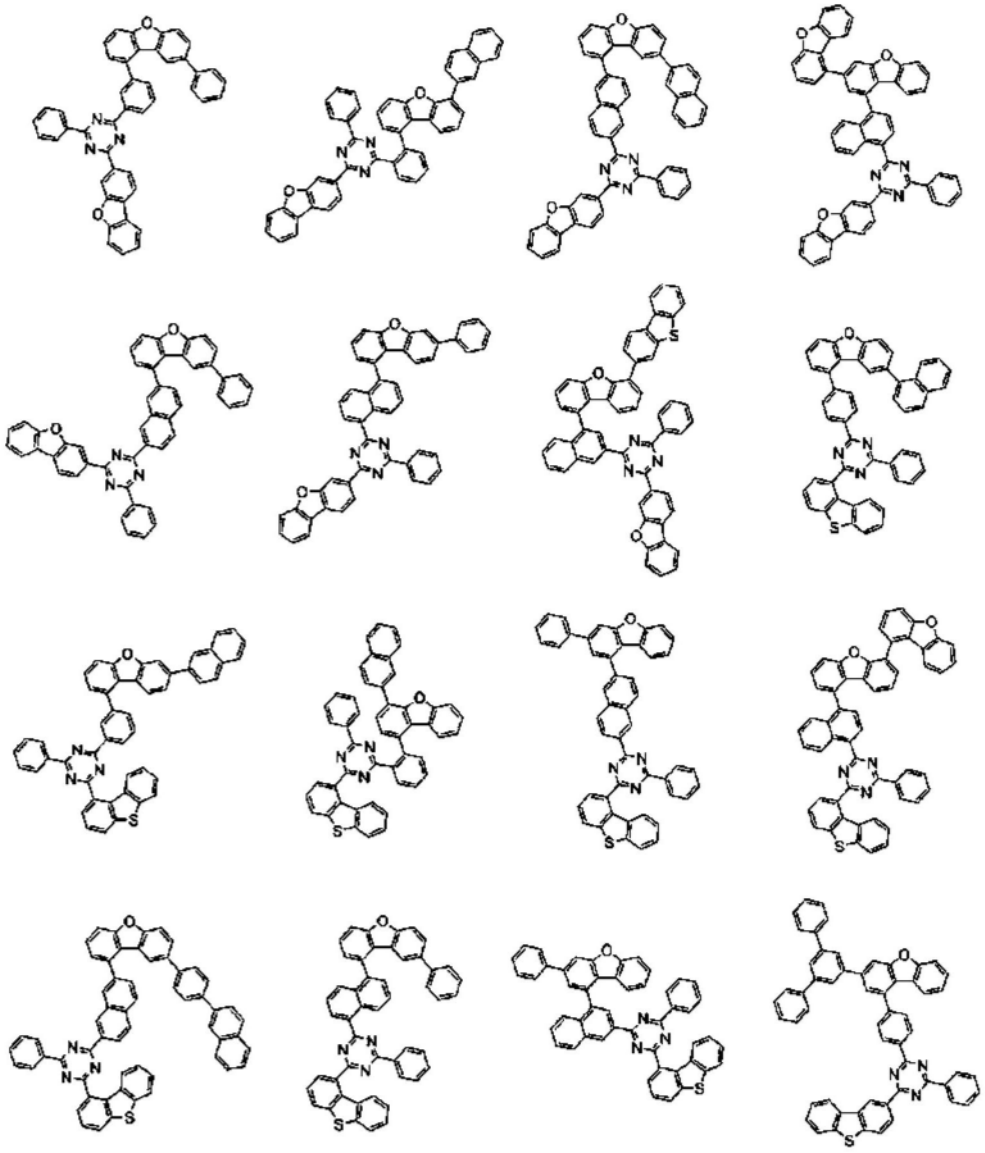


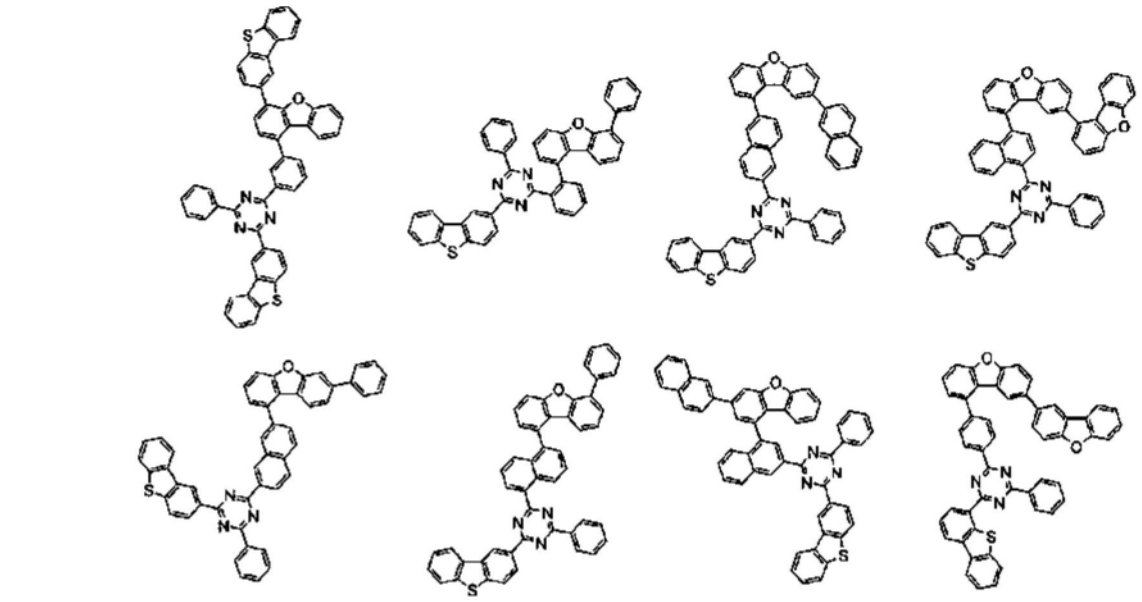


[0231]

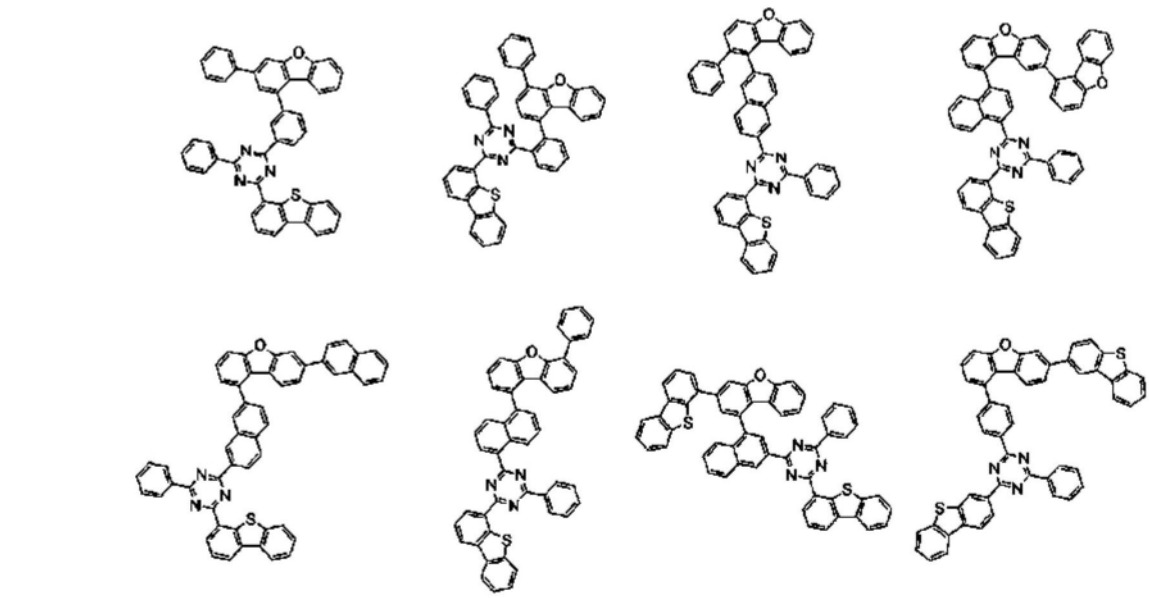


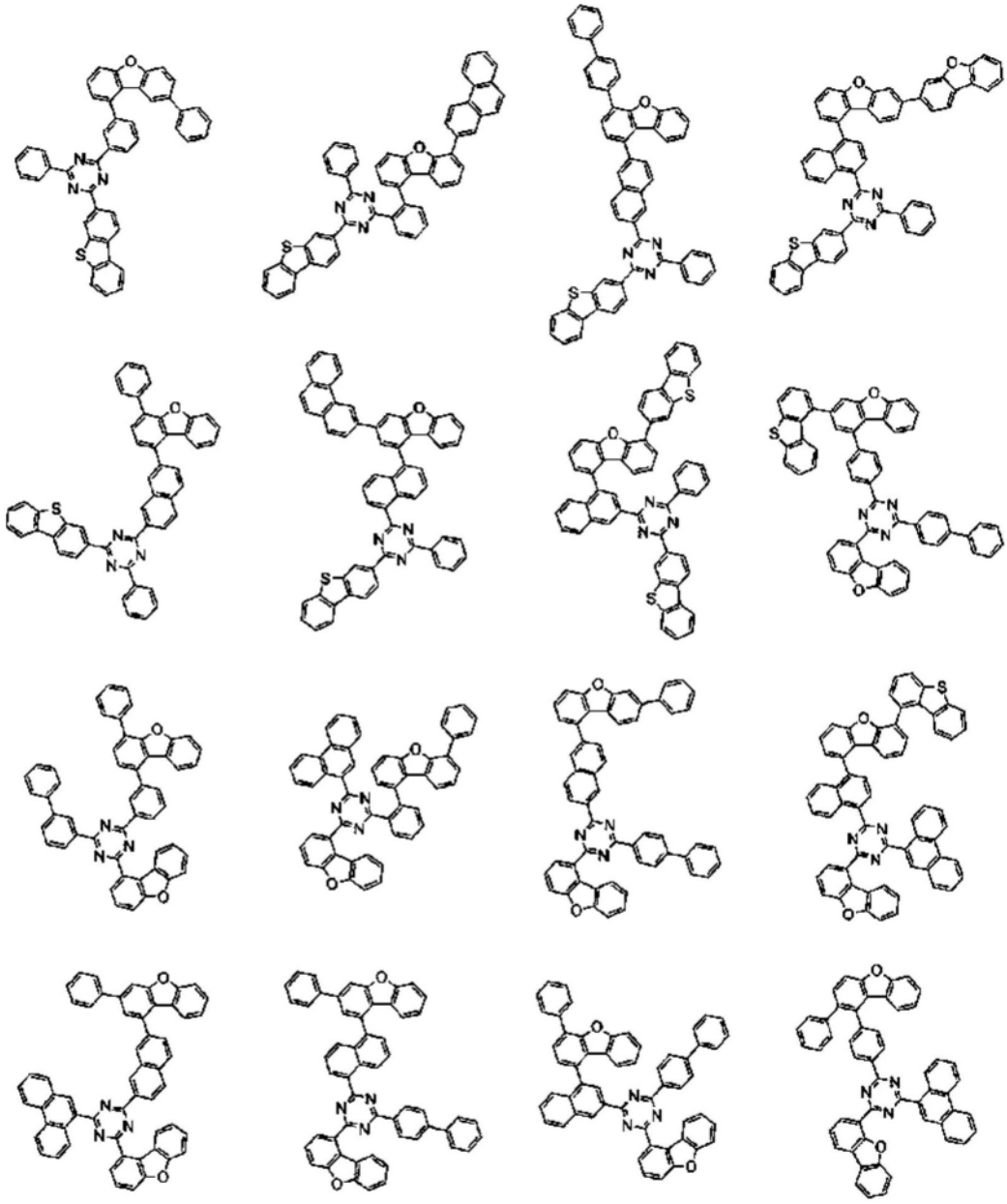
[0232]



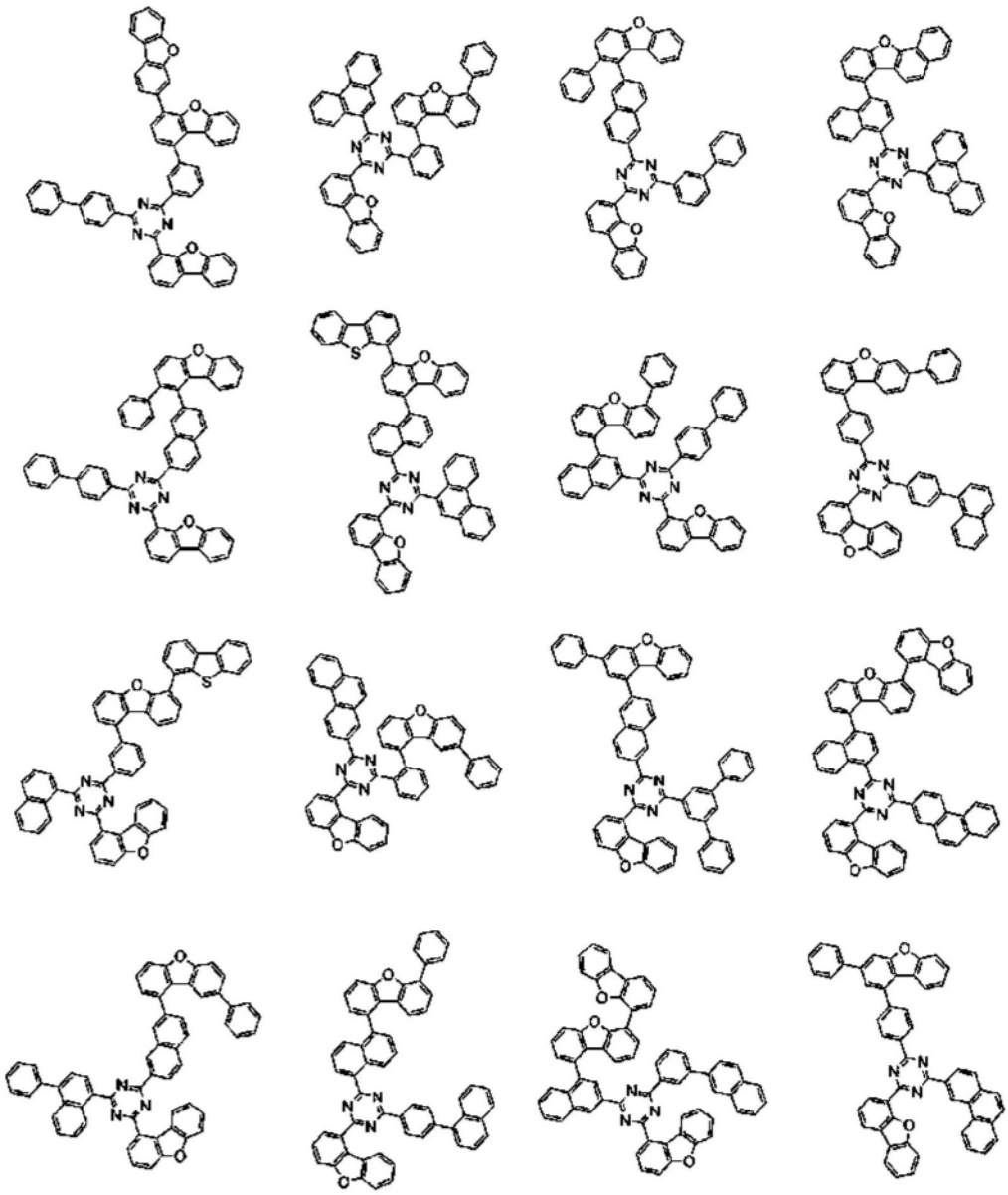


[0233]

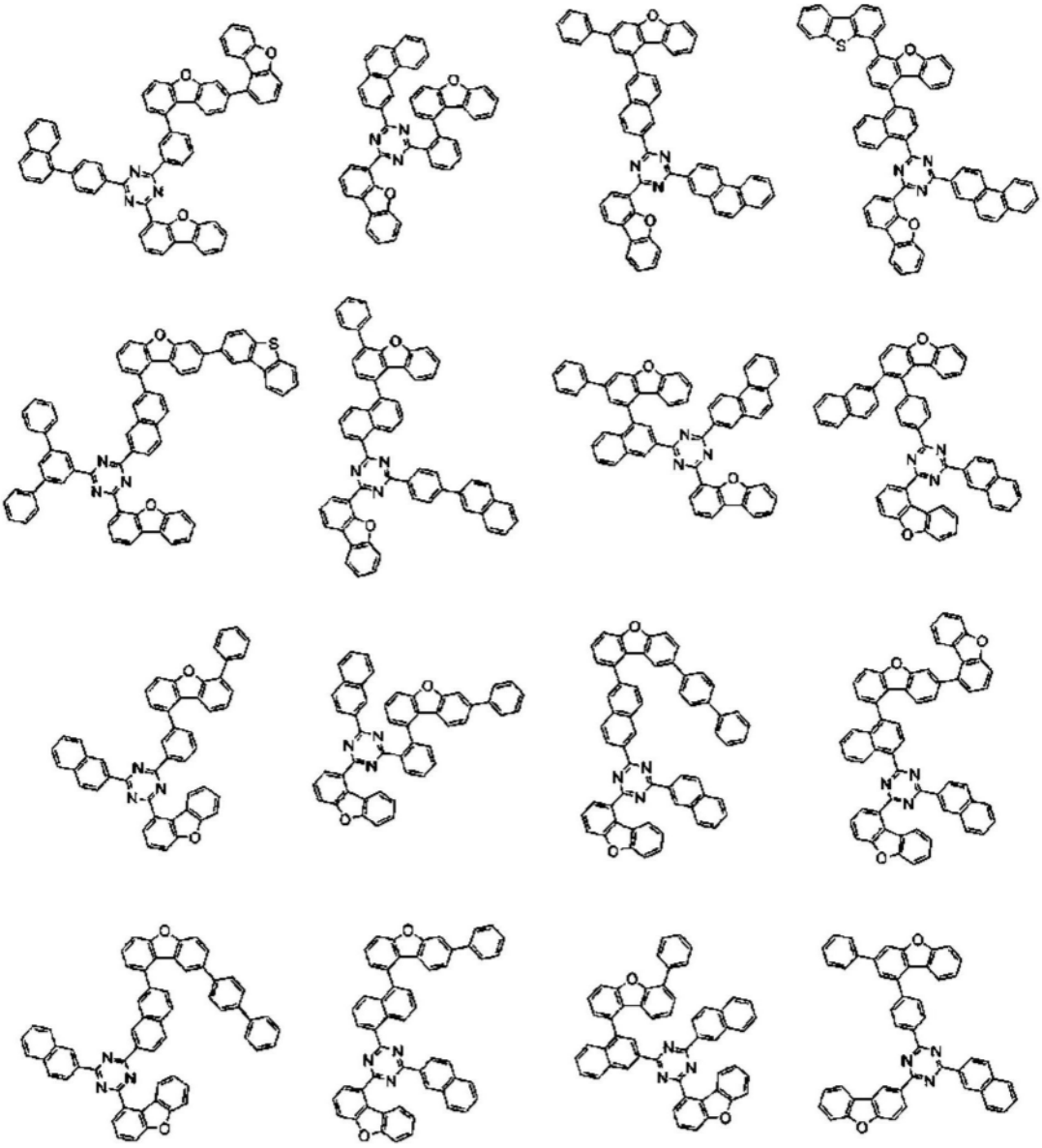


[0234] 

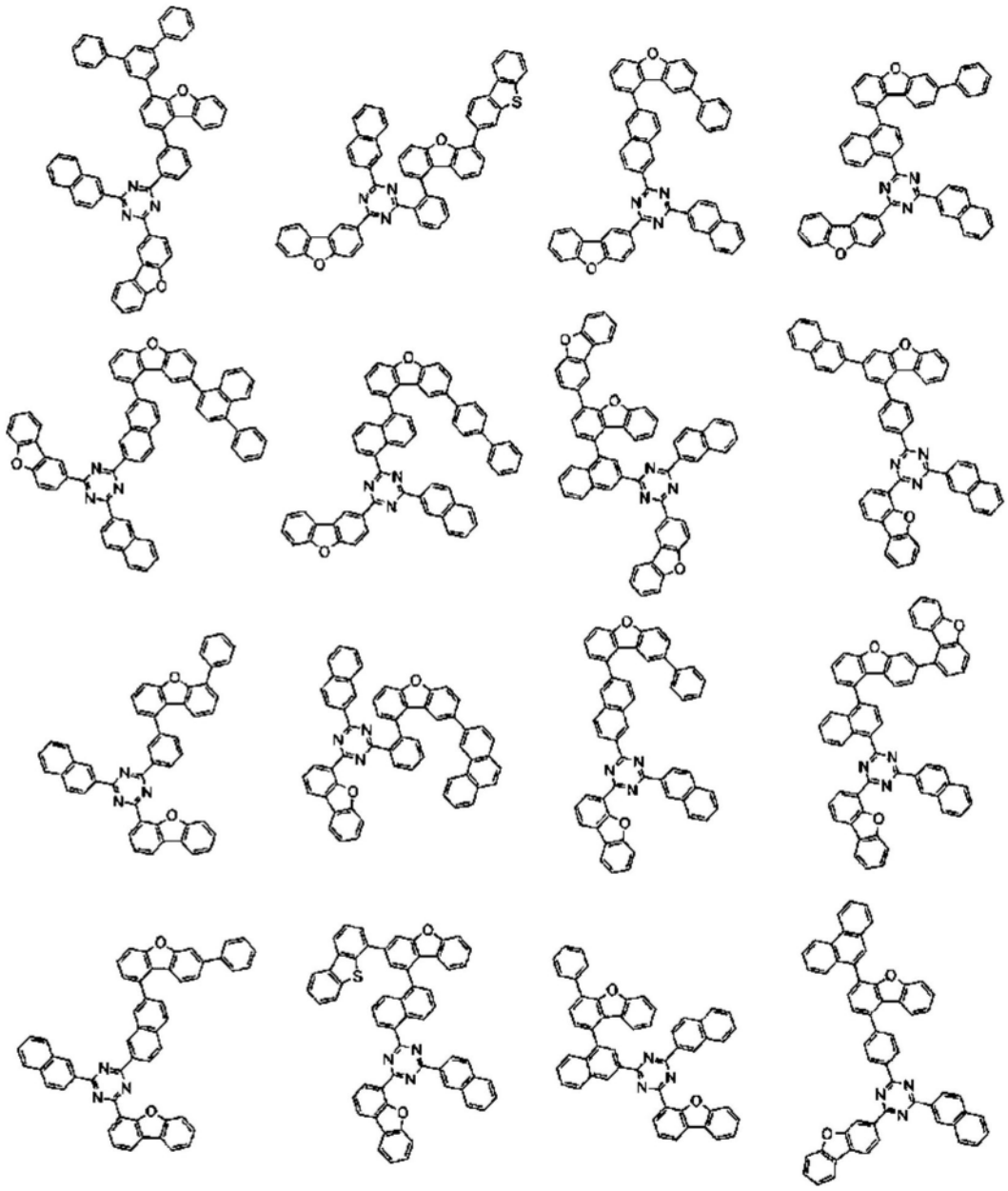
[0235]



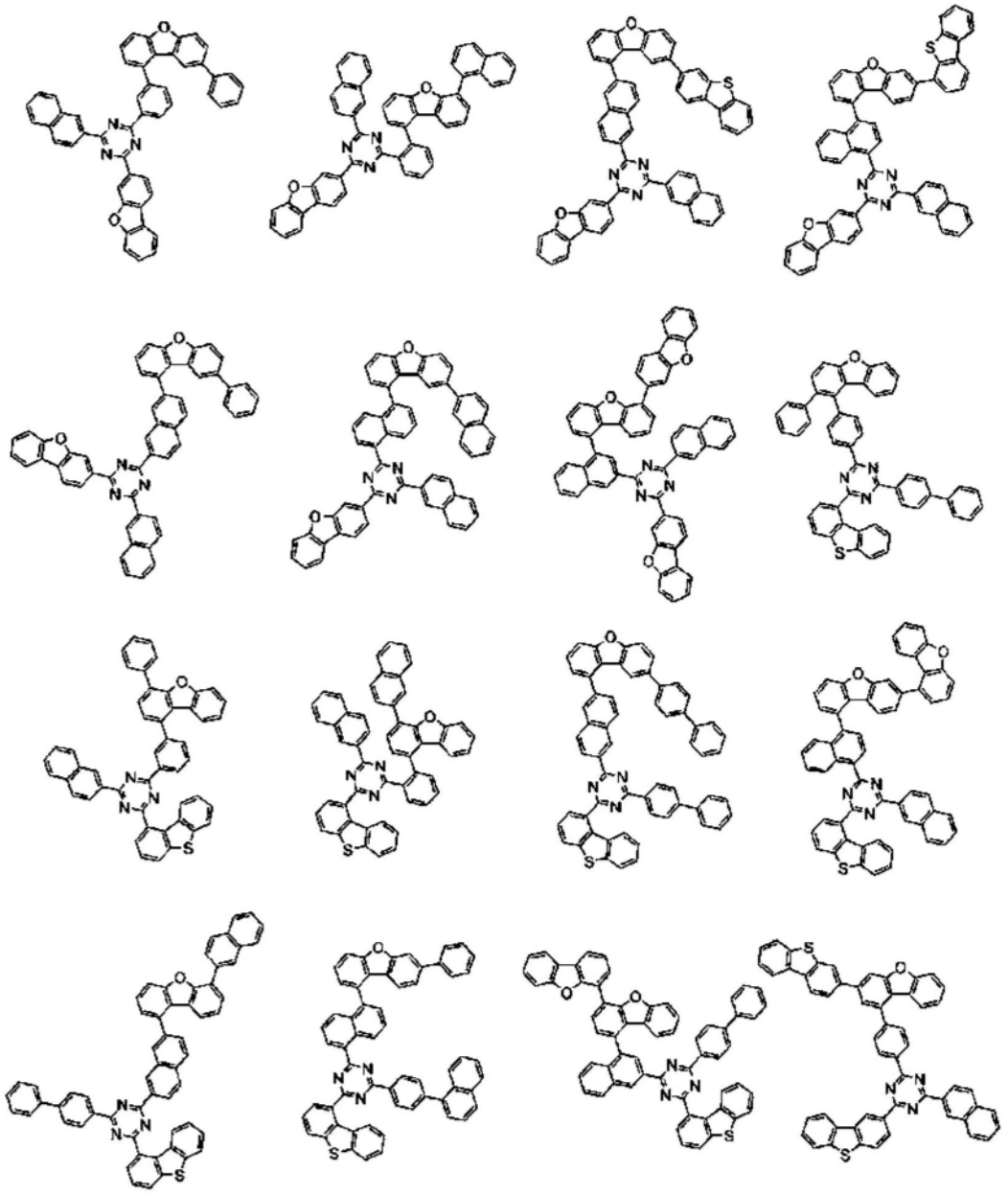
[0236]



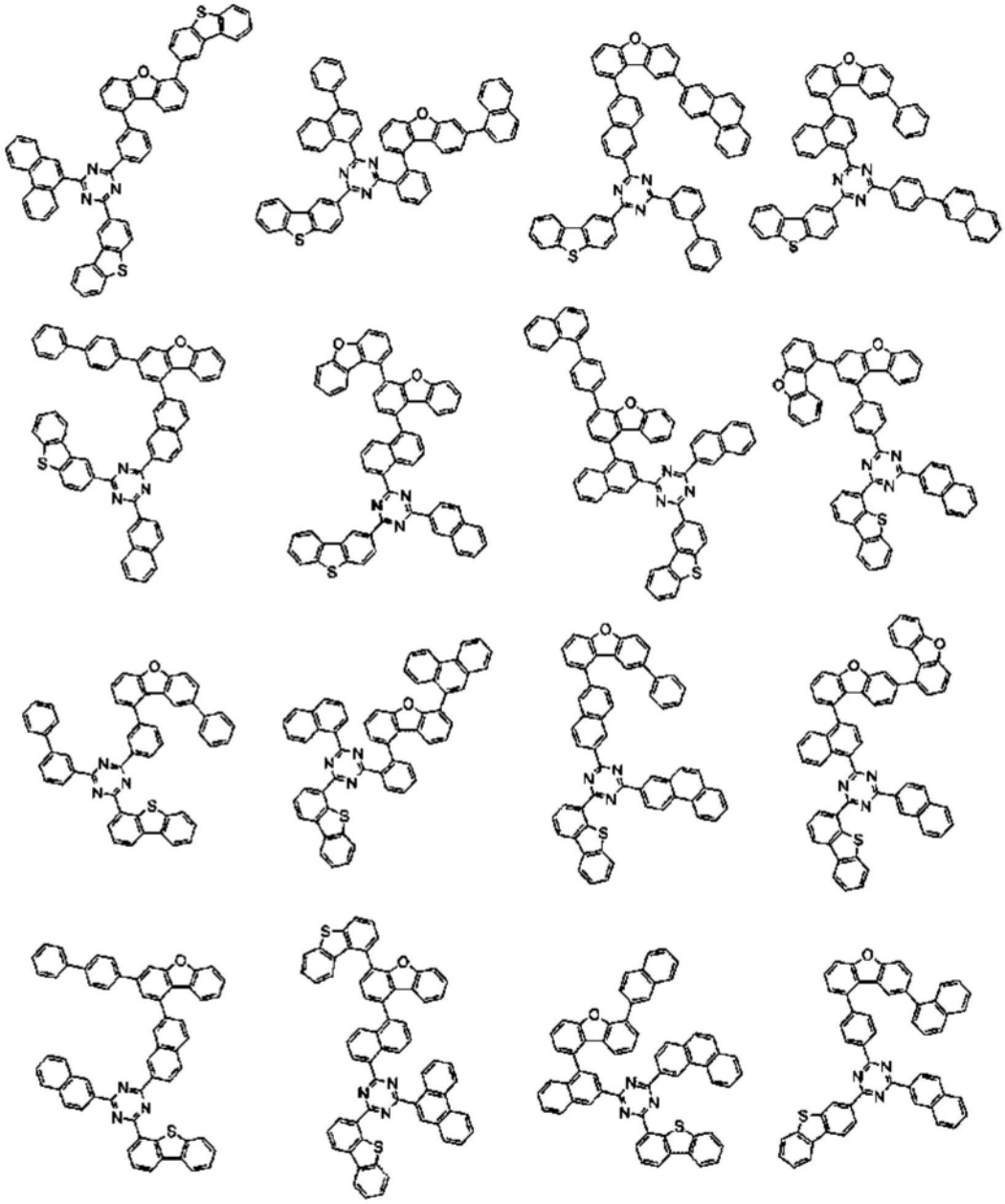
[0237]



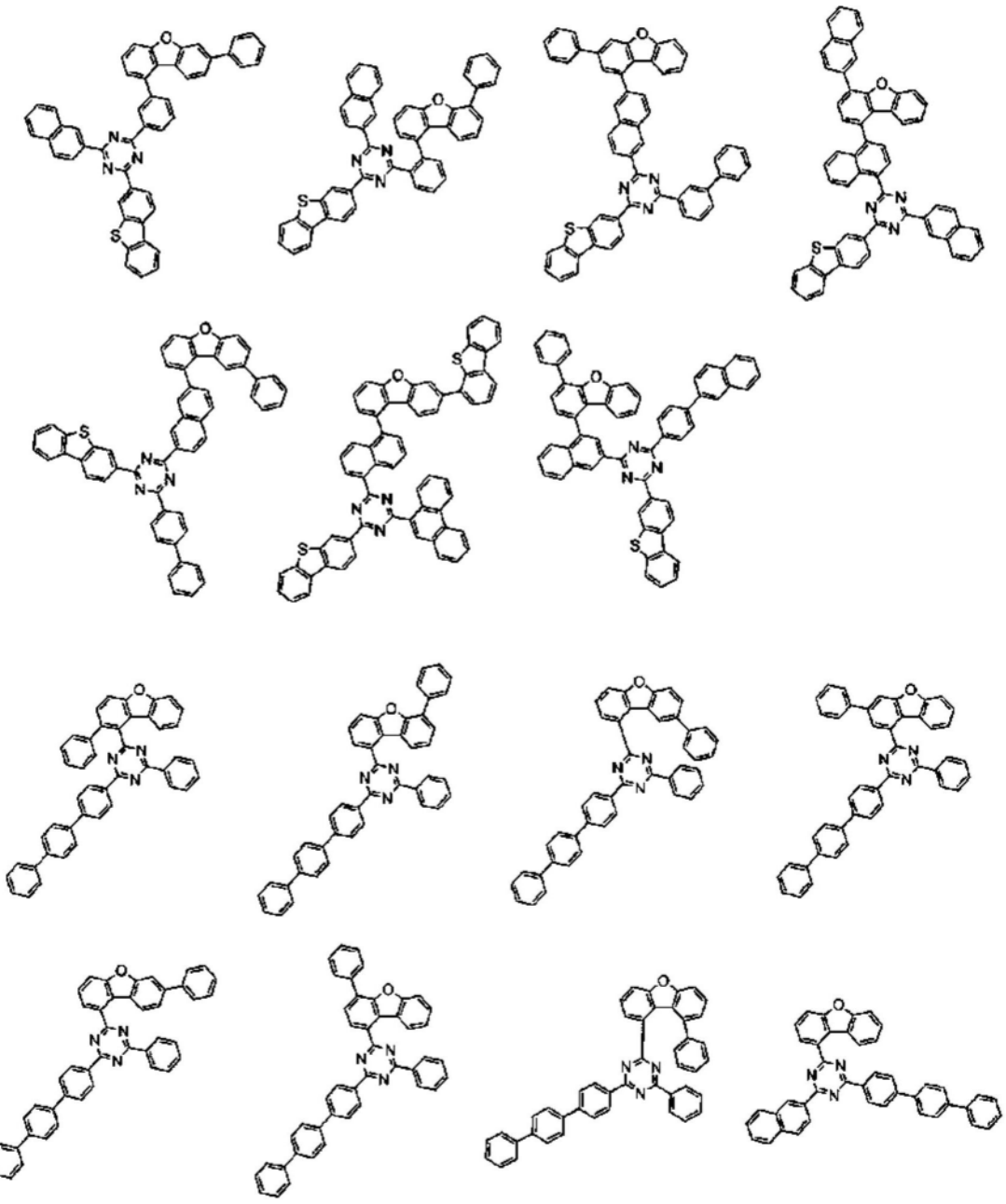
[0238]



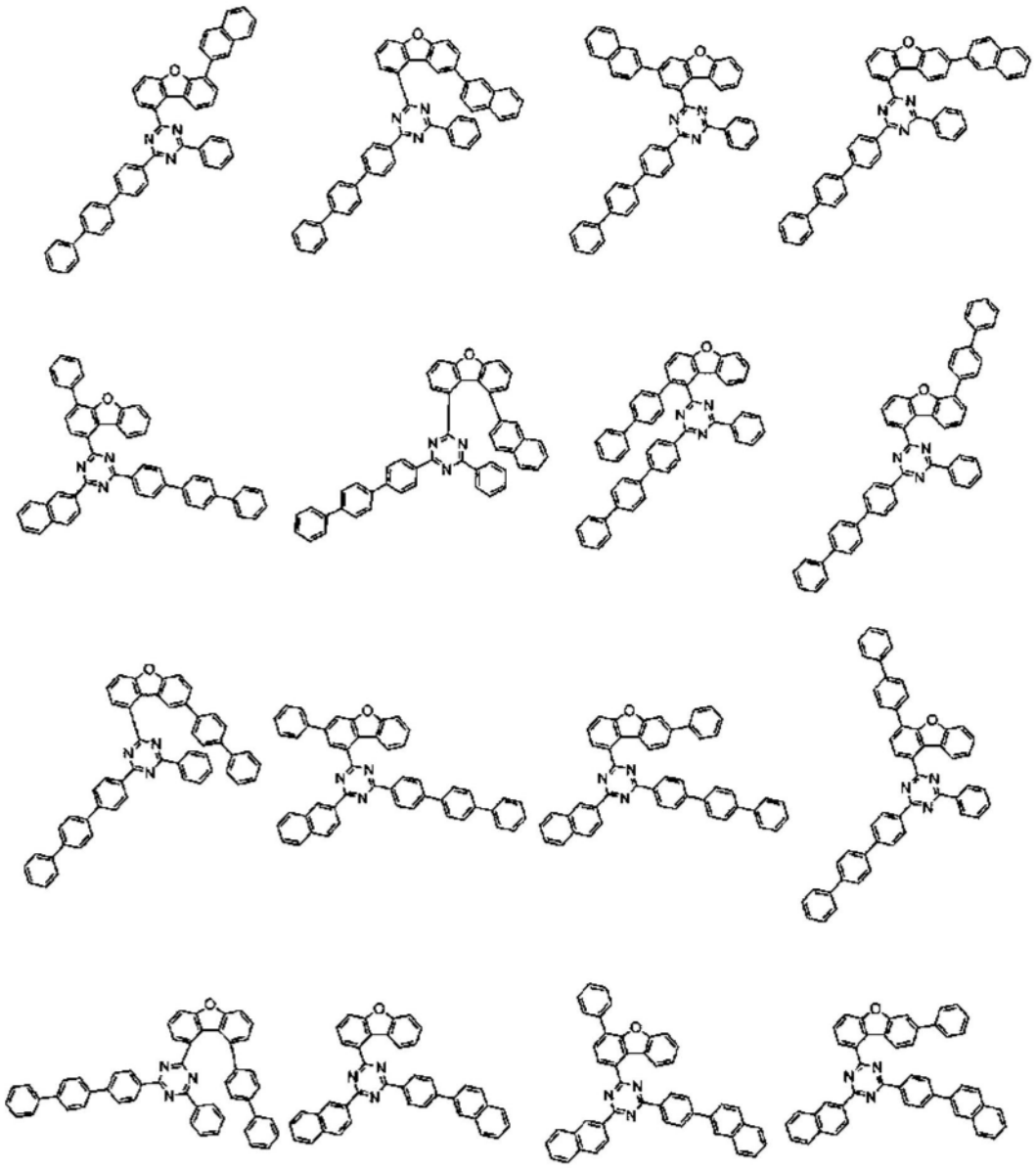
[0239]

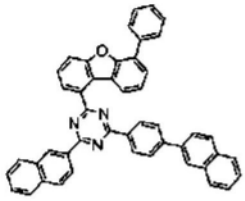
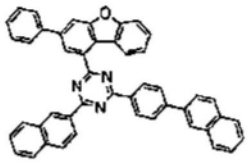
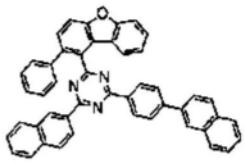
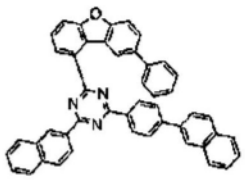
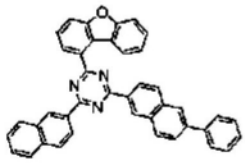
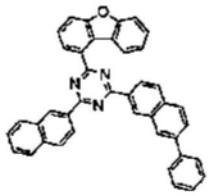
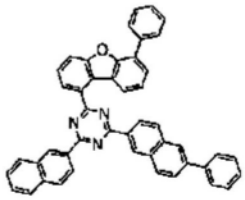
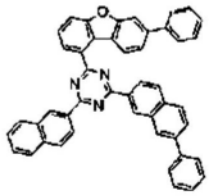


[0240]

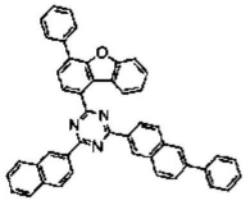
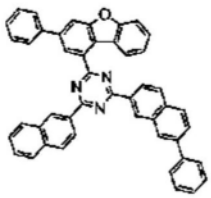
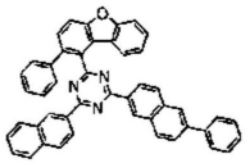
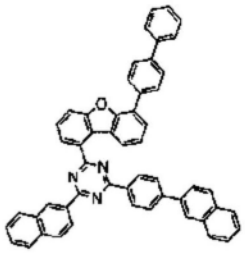
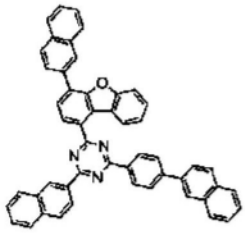
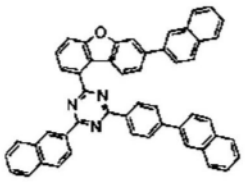
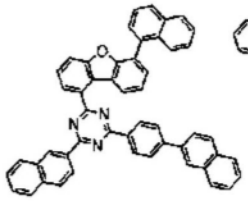
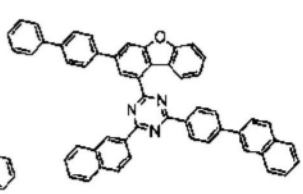


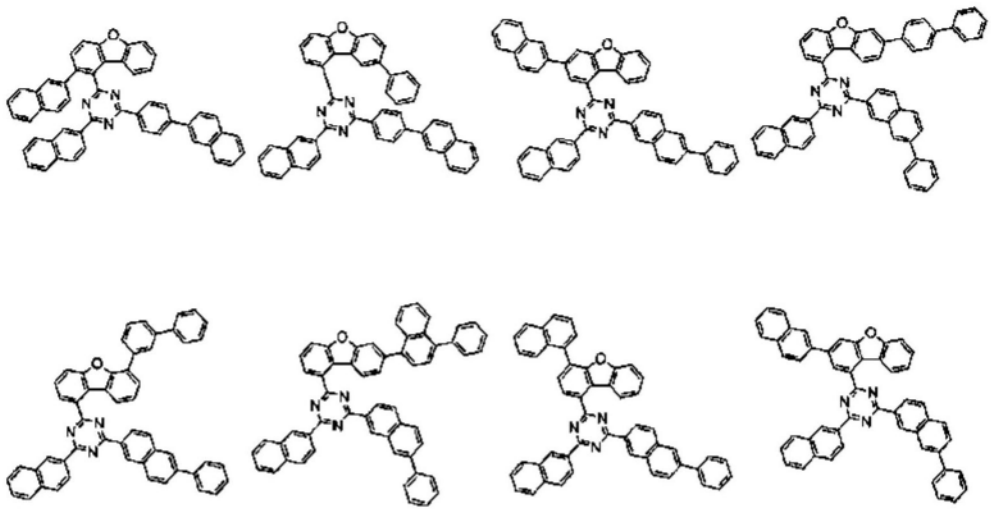
[0241]



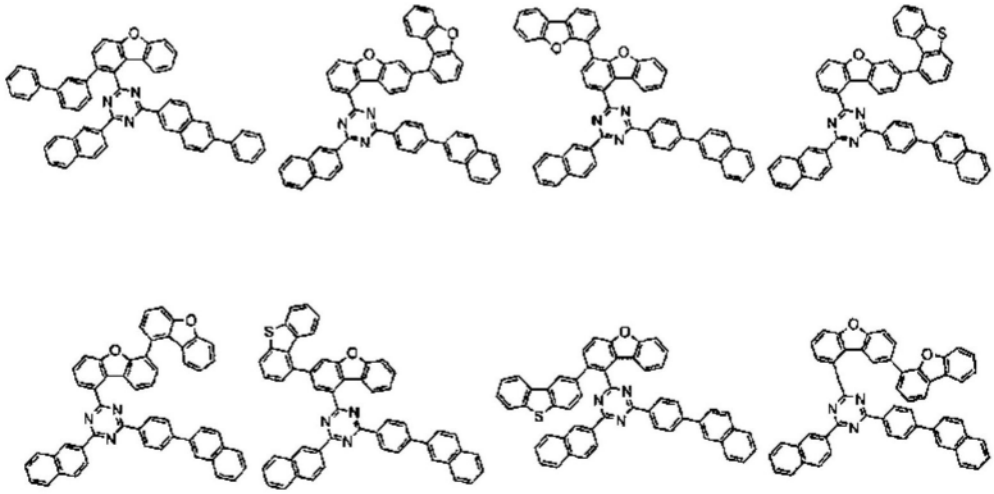









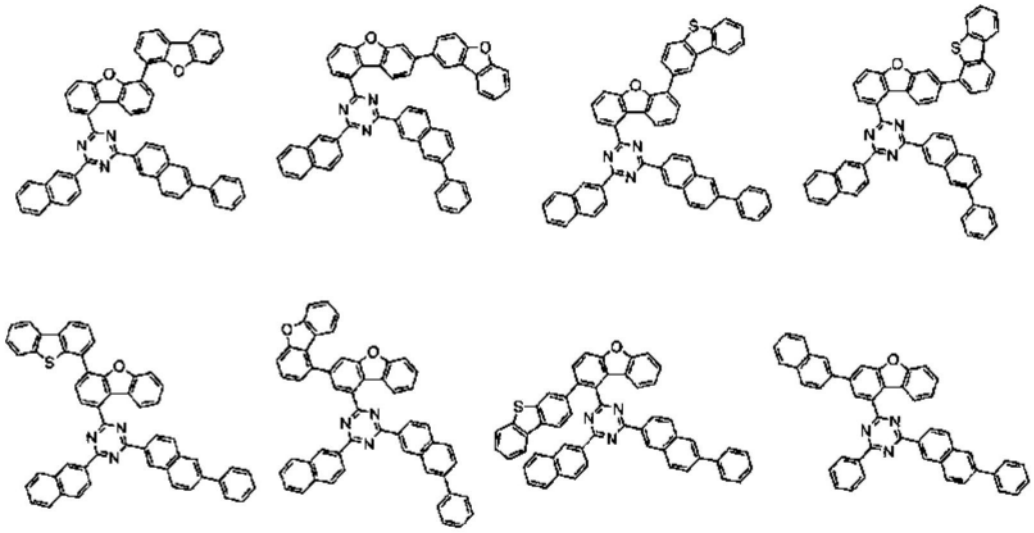
[0242]

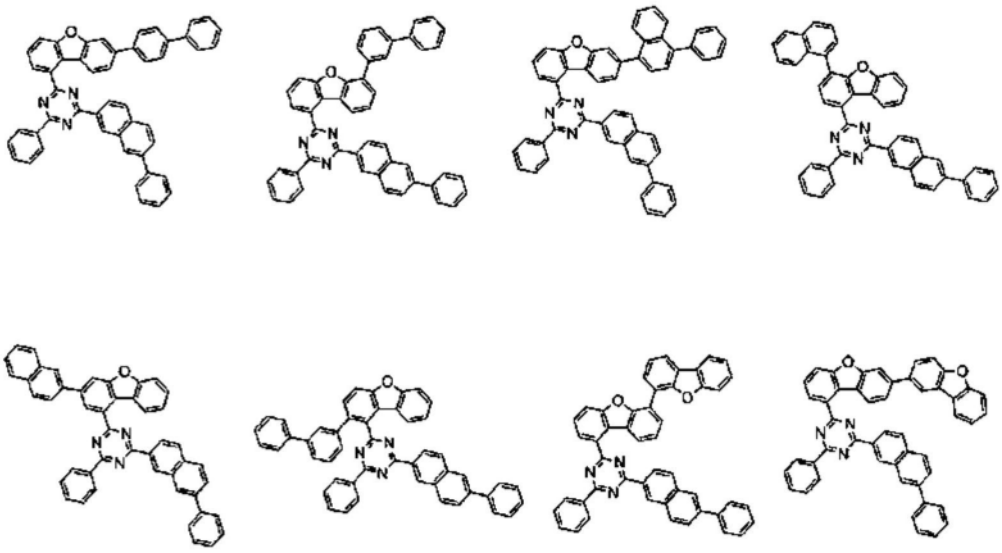


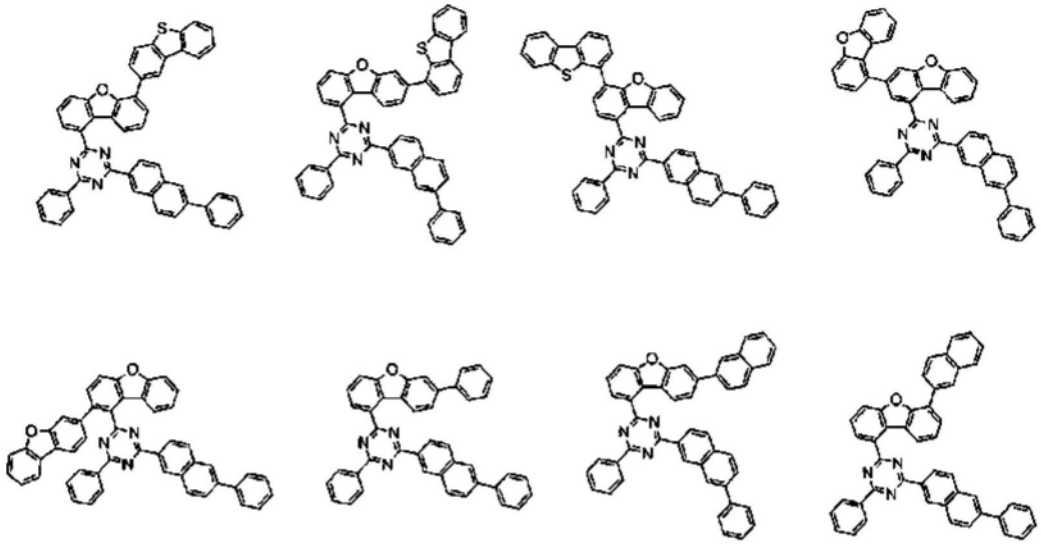
[0243]



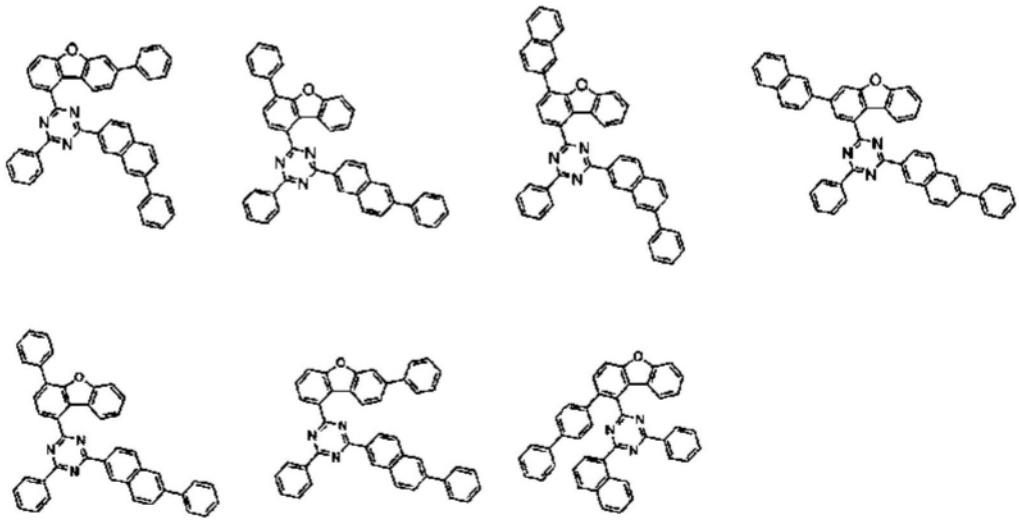


[0244]



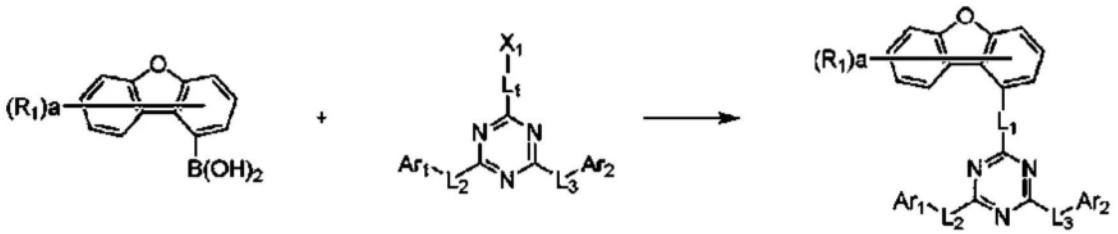


[0245]



[0246] 例如,可以根据如以下反应方案1中所示的制备方法来制备由化学式1表示的化合物,并且可以以类似的方式制备其他剩余的化合物。

[0247] [反应方案1]:

[0248] 

[0249] 其中在反应方案1中,Ar_1、Ar_2、L_1至L_3、R_1和a如化学式1中所限定,并且X_1为卤素,优选地X_1为氯或溴。

[0250] 反应方案1为Suzuki偶联反应,其优选地在钯催化剂和碱的存在下进行,并且用于Suzuki偶联反应的反应性基团可以如本领域中已知的进行修改。以上制备方法可以在下文描述的制备例中进一步呈现。

[0251] 优选地,Ar_3可以为氢;经取代或未经取代的C_{6-20}芳基;或者经取代或未经取代的包

含选自N、O和S中的一者或更多者的C_{2-20}杂芳基,以及

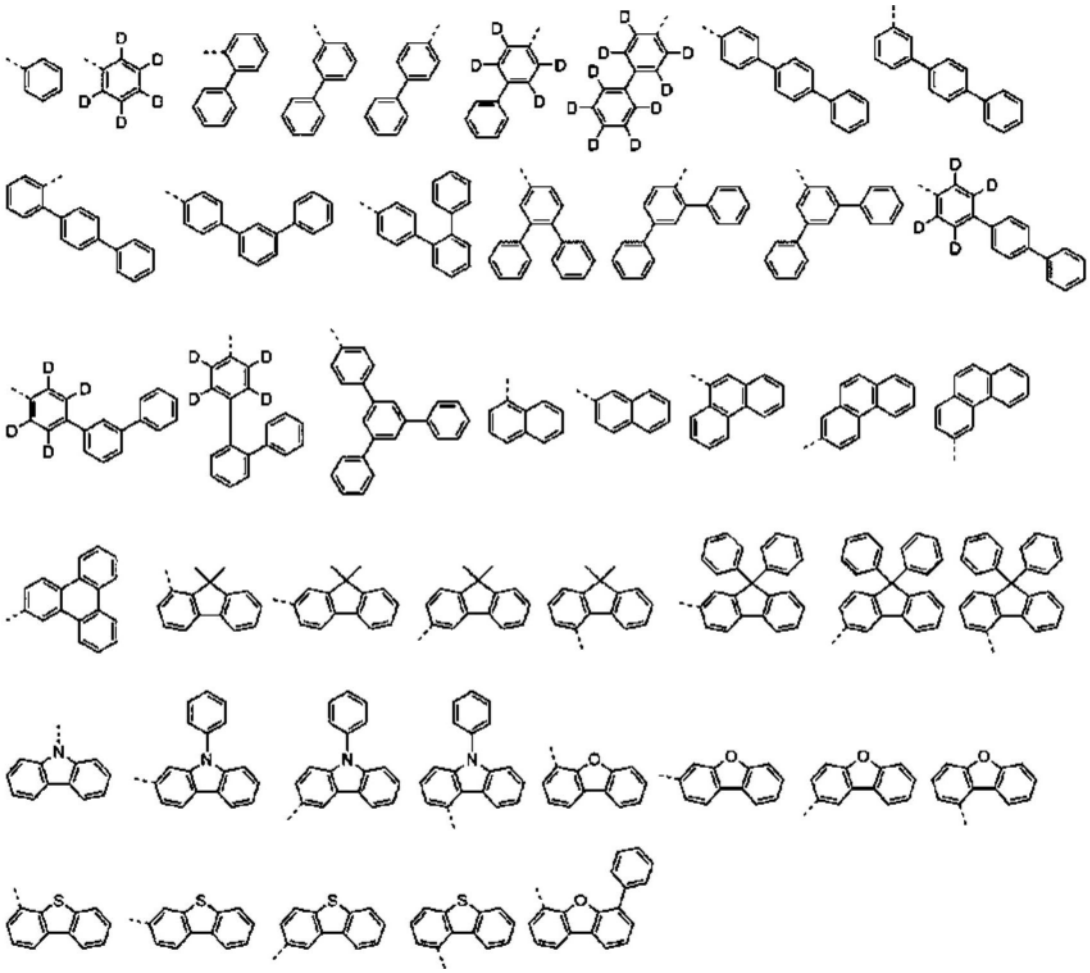
[0252] 更优选地,Ar_3可以为氢或苯基。

[0253] 优选地,Ar_4和Ar_5可以各自独立地为经取代或未经取代的C_{6-20}芳基;或者经取代或未经取代的包含选自N、O和S中的一者或更多者的C_{2-20}杂芳基,

[0254] 更具体地,Ar_4和Ar_5可以各自独立地为苯基、经5个氘取代的苯基、联苯基、经4个氘取代的联苯基、经9个氘取代的联苯基、三联苯基、经4个氘取代的三联苯基、四联苯基、萘基、菲基、三亚苯基、二甲基芴基、二苯基芴基、咔唑基、苯基咔唑基、二苯并呋喃基、二苯并噻吩基或苯基二苯并呋喃基,以及

[0255] 最优选地,Ar_4和Ar_5可以各自独立地为选自以下中的任一者:

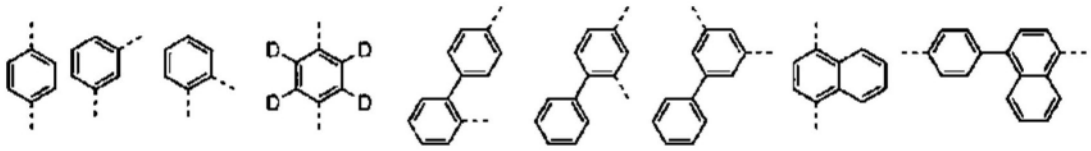
[0256]



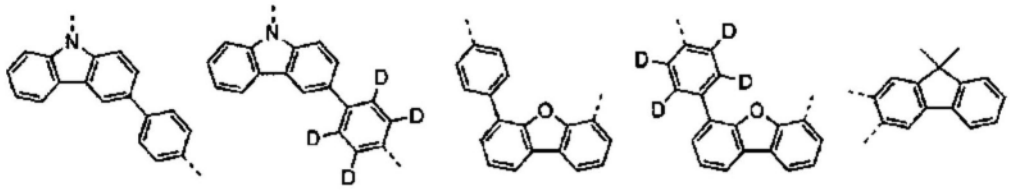
[0257] 优选地,L_4至L_6可以各自独立地为单键;经取代或未经取代的C_{6-20}亚芳基;或者经取代或未经取代的包含选自N、O和S中的一者或更多者的C_{2-20}亚杂芳基,

[0258] 更优选地,L_4至L_6可以各自独立地为单键、亚苯基、经4个氘取代的亚苯基、亚联苯基、亚萘基、苯基亚萘基、亚咔唑基、苯基亚咔唑基、经4个氘取代的苯基亚咔唑基、亚二苯并呋喃基、苯基亚二苯并呋喃基、经4个氘取代的苯基亚二苯并呋喃基、或二甲基亚芴基,以及

[0259] 最优选地,L_4至L_6可以各自独立地为单键或选自以下中的任一者:



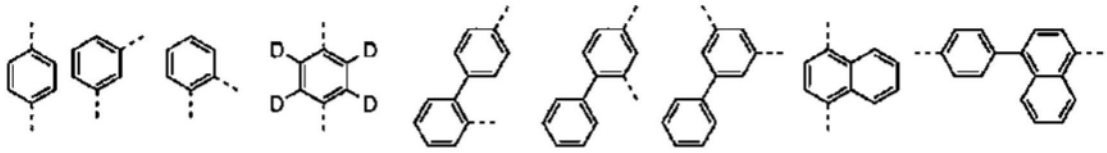
[0260]



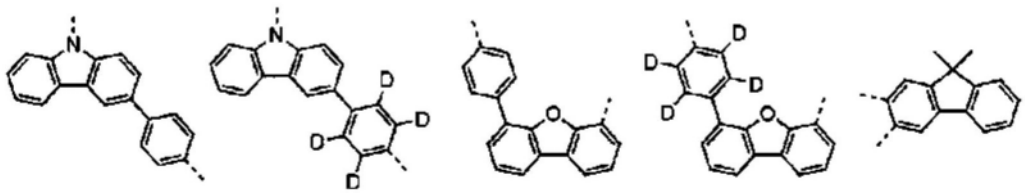
[0261] 优选地，L_4为单键，并且L_5和L_6可以各自独立地为单键；经取代或未经取代的C_{6-20}亚芳基；或者经取代或未经取代的包含选自N、O和S中的至少一者的C_{2-20}亚杂芳基，

[0262] 更优选地，L_4为单键，并且L_5和L_6可以各自独立地为单键、亚苯基、经4个氘取代的亚苯基、亚联苯基、亚萘基、苯基亚萘基、亚咔唑基、苯基亚咔唑基、经4个氘取代的苯基亚咔唑基、亚二苯并呋喃基、苯基亚二苯并呋喃基、经4个氘取代的苯基亚二苯并呋喃基、或二甲基亚芴基，以及

[0263] 最优选地，L_4为单键，并且L_5和L_6可以各自独立地为单键或选自以下中的任一者：



[0264]



[0265] 优选地，L_7可以为经取代或未经取代的C_{6-20}亚芳基，

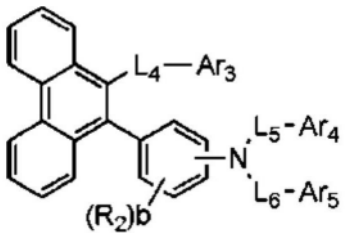
[0266] 更优选地，L_7可以为经取代或未经取代的亚苯基、经取代或未经取代的亚联苯基、或者经取代或未经取代的亚萘基，以及

[0267] 最优选地，L_7可以为亚苯基、经4个氘取代的亚苯基、亚联苯基或亚萘基。

[0268] 优选地，由化学式2表示的化合物可以由以下化学式2-1表示：

[0269] [化学式2-1]

[0270]



[0271] 其中在化学式2-1中，

[0272] Ar_3至Ar_5和L_4至L_6如化学式2中所限定，

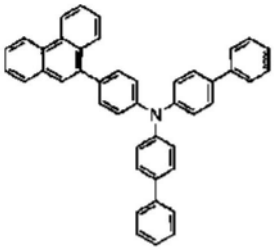
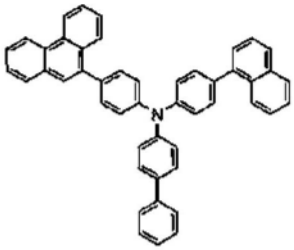
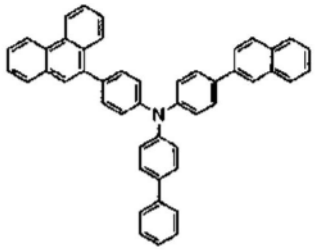
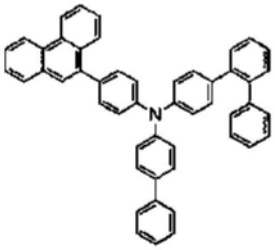
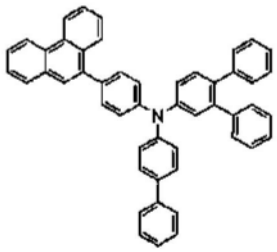
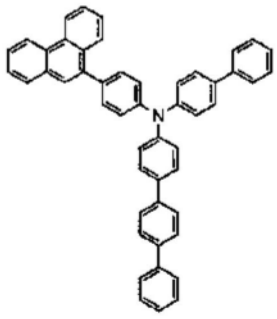
[0273] R_2为氢、氘、或者经取代或未经取代的C_{6-60}芳基，以及

[0274] b为0至4的整数。

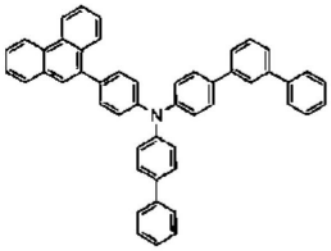
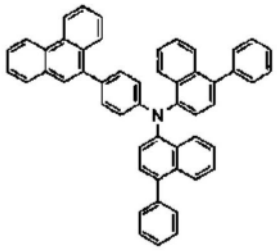
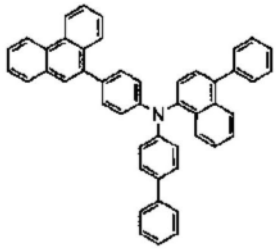
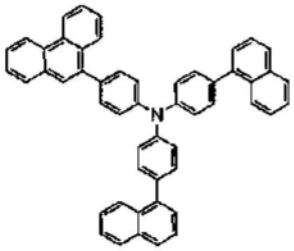
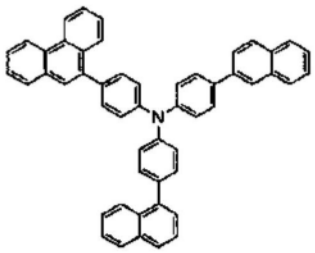
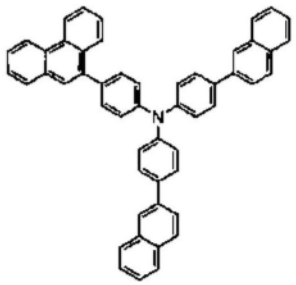
[0275] 优选地，R_2可以为氢、氘、或者经取代或未经取代的C_{6-20}芳基，以及

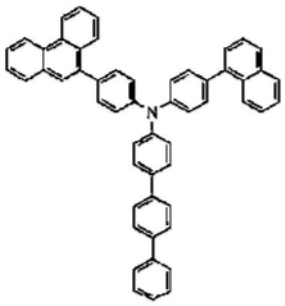
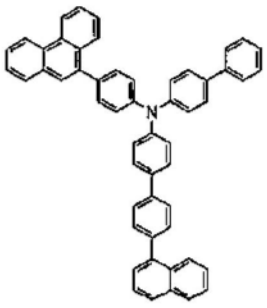
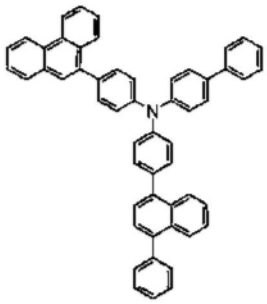
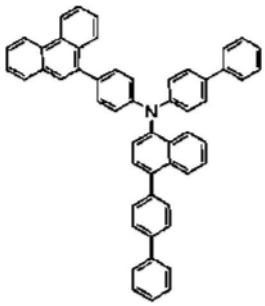
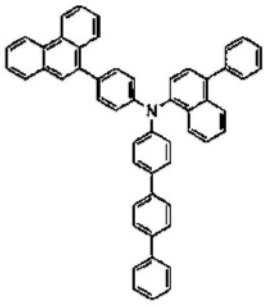
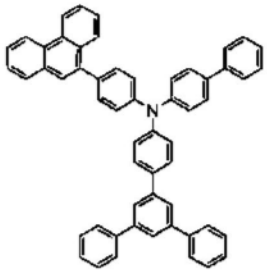
[0276] 更优选地，R_2可以为氢或氘。

[0277] 由化学式2表示的化合物的代表性实例如下：

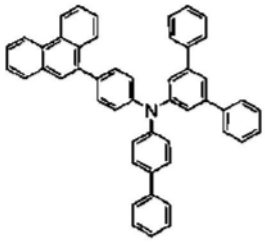
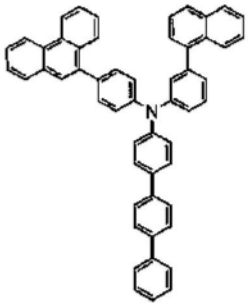
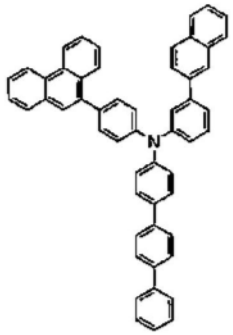
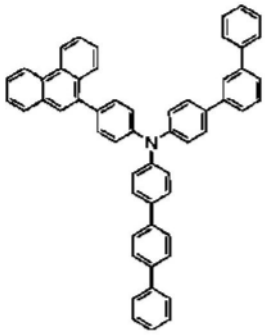
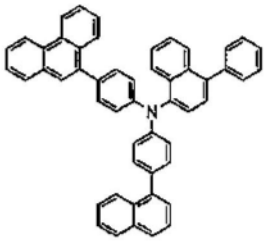
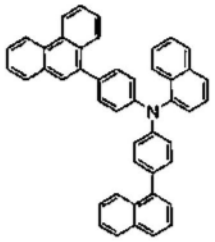







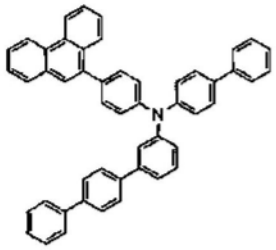
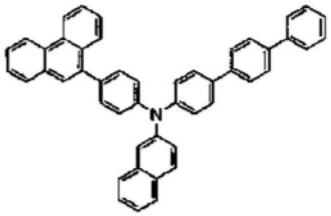
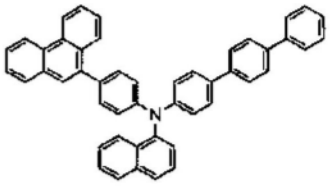
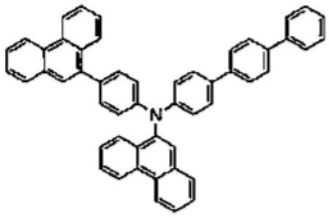
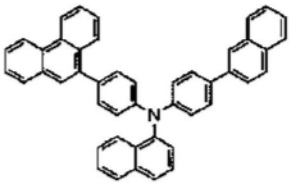
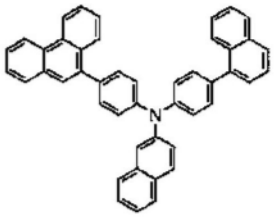
[0278]

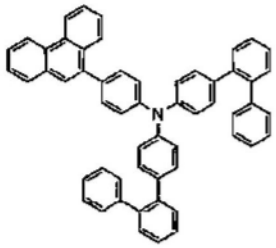
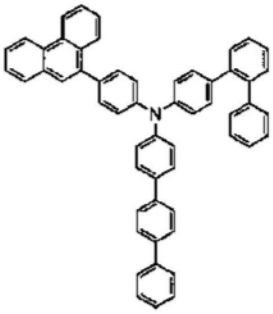
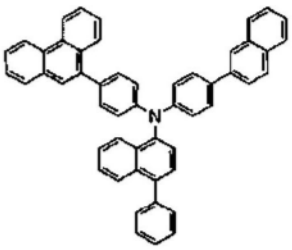
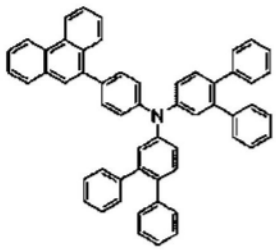
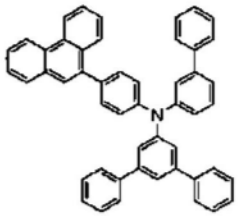
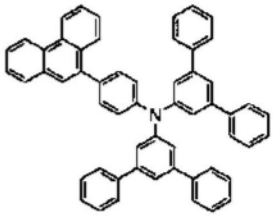







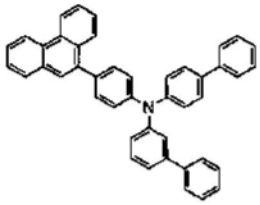
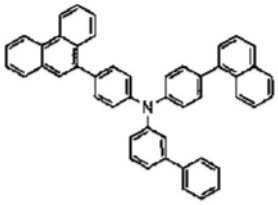
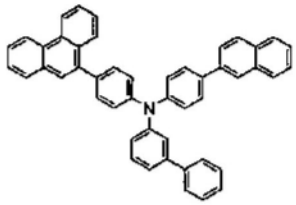
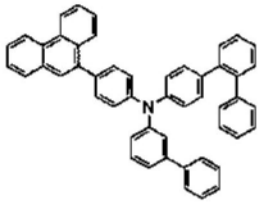
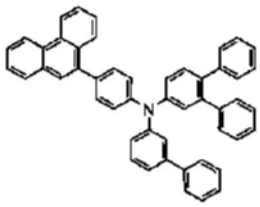
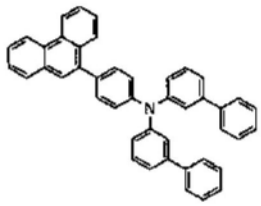
[0279]

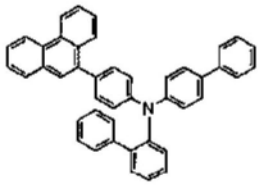
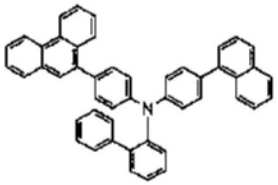
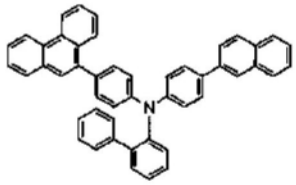
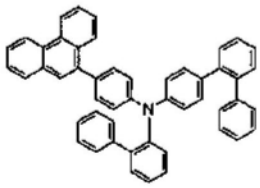
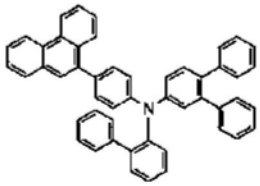
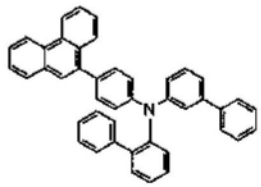







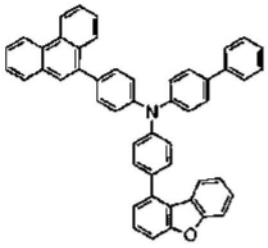
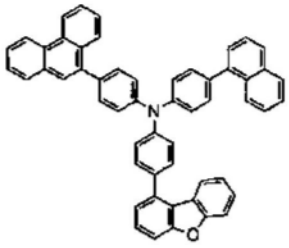
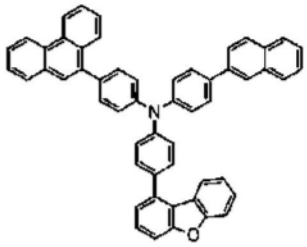
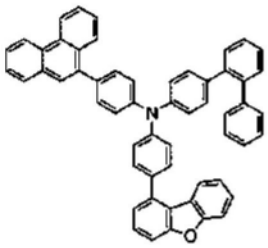
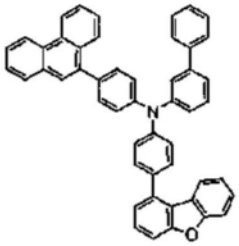
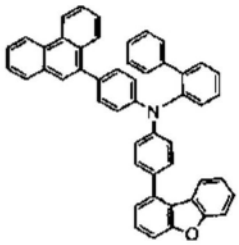
[0280]

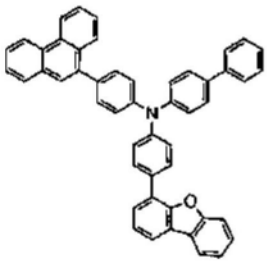
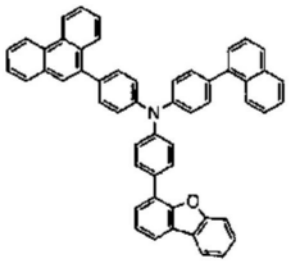
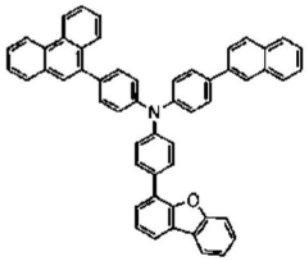
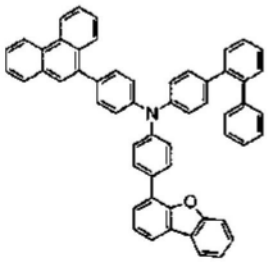
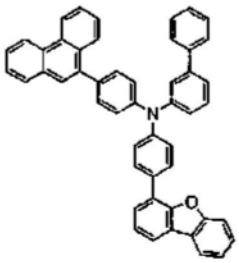
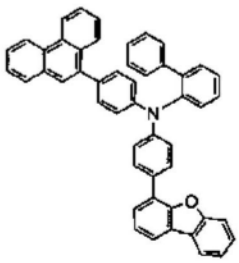







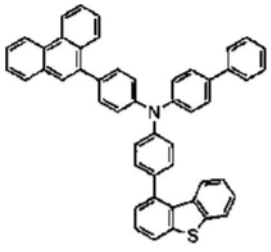
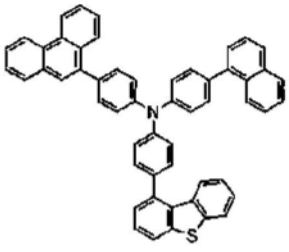
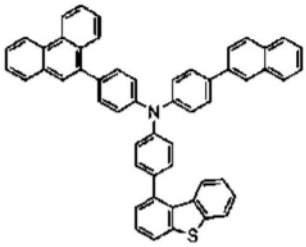
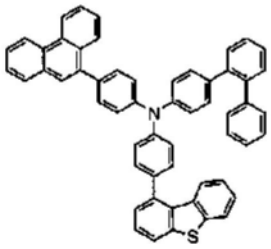
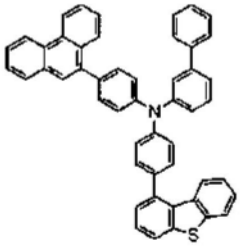
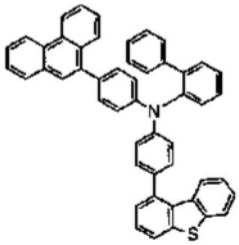
[0281]

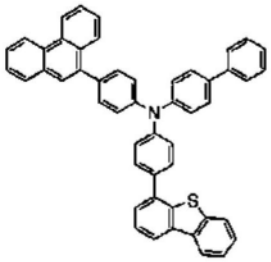
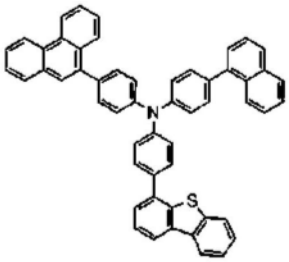
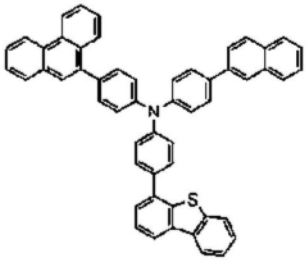
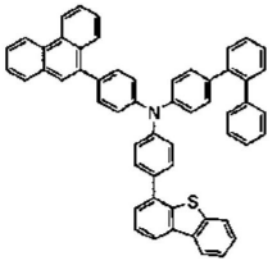
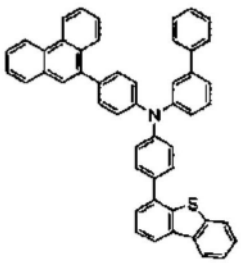
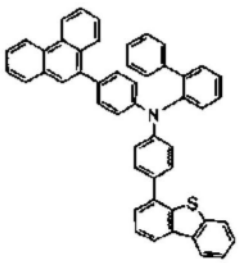







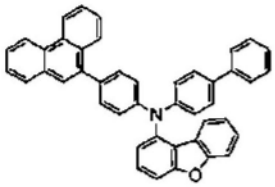
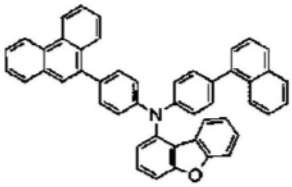
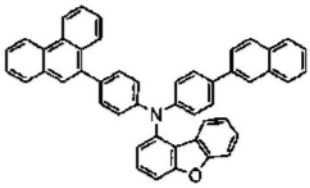
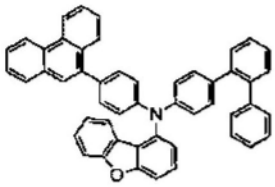
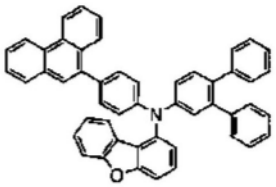
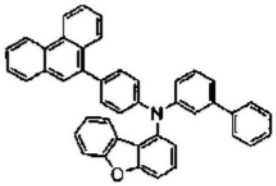
[0282]

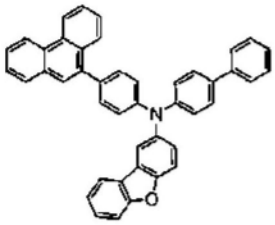
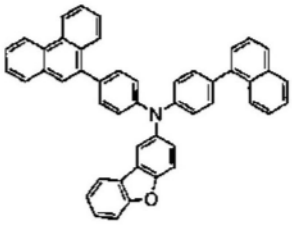
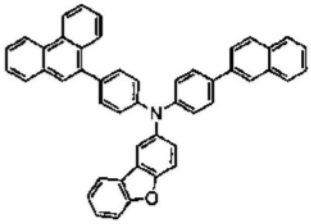
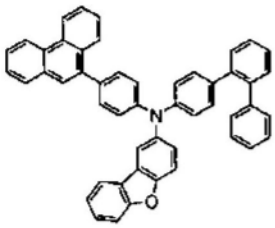
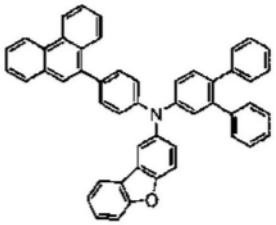
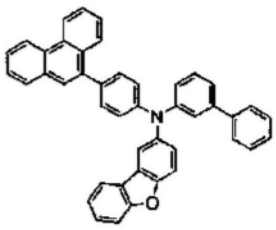







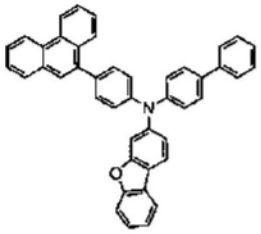
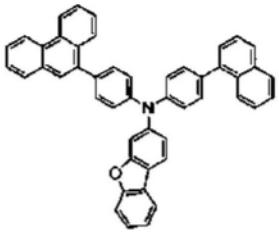
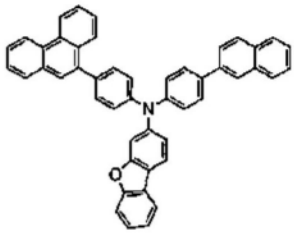
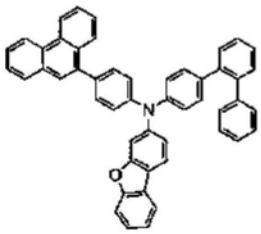
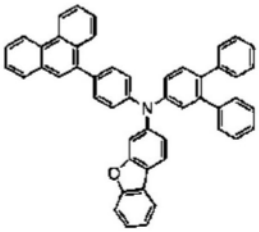
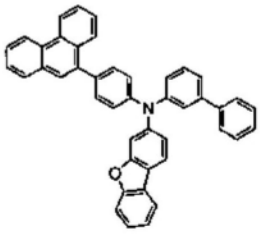
[0283]

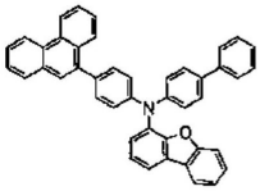
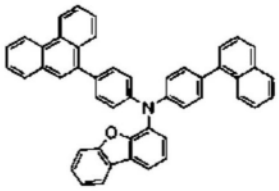
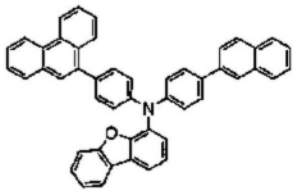
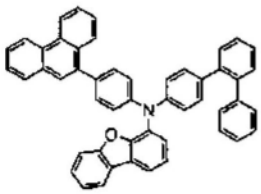
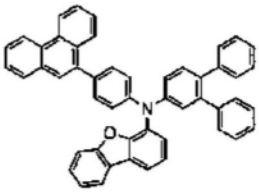
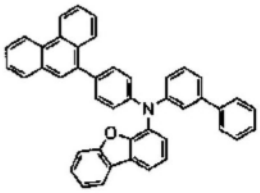







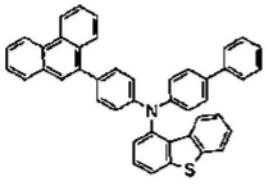
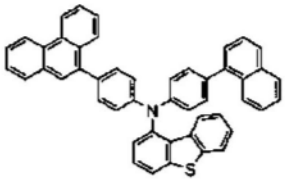
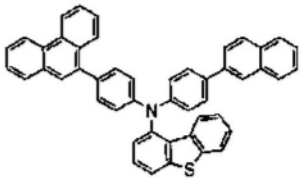
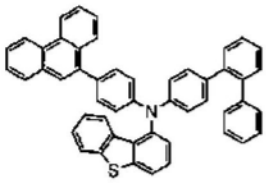
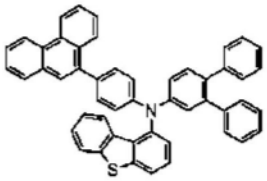
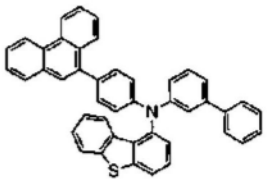
[0284]

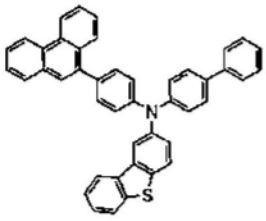
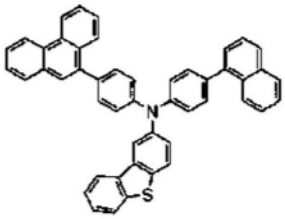
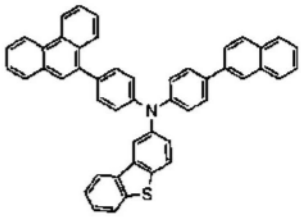
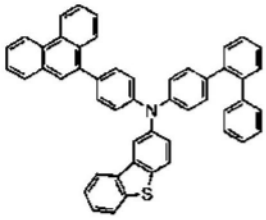
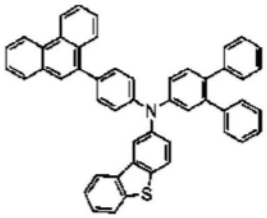
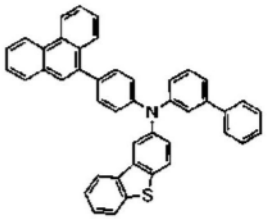







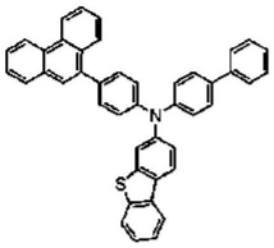
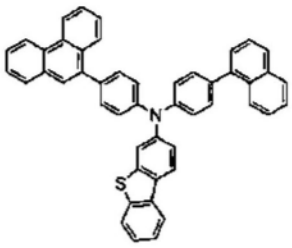
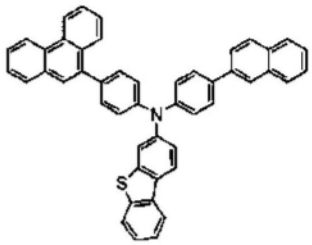
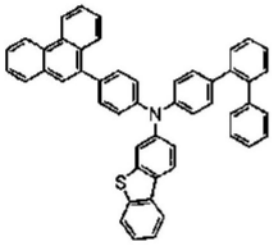
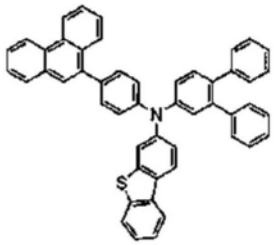
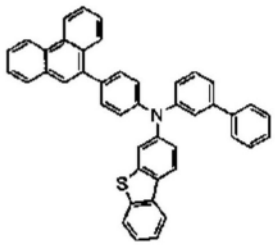
[0285]

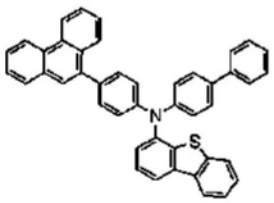
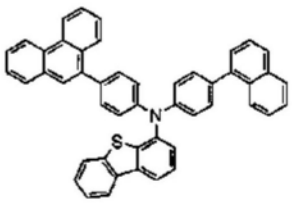
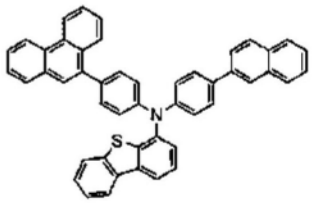
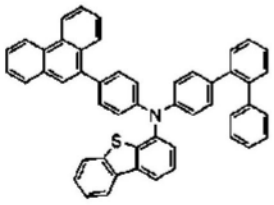
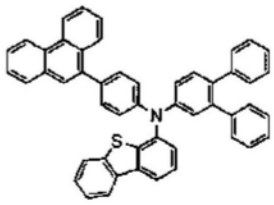
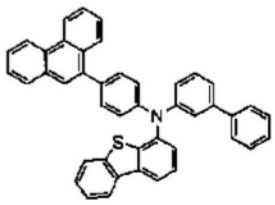







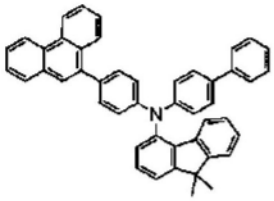
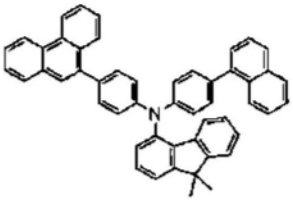
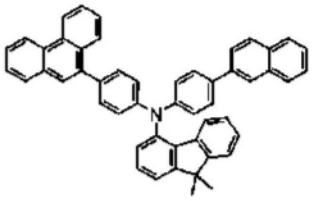
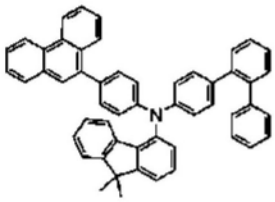
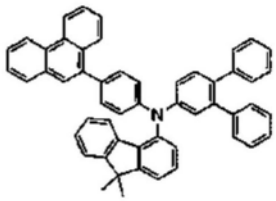
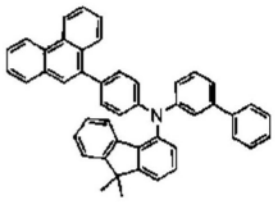
[0286]

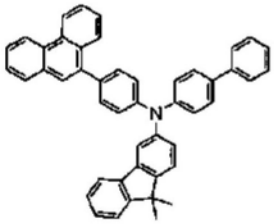
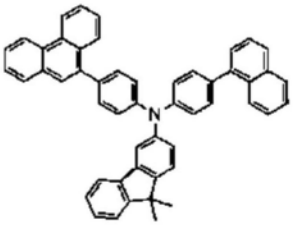
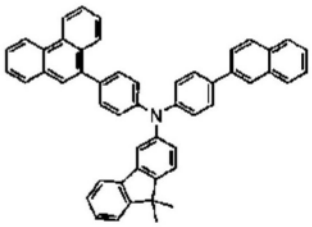
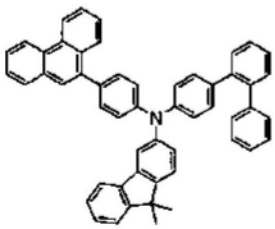
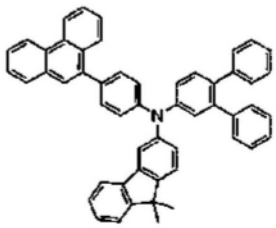
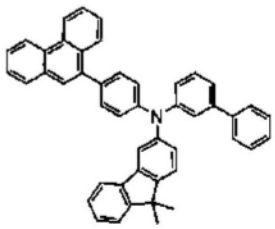







[0287]

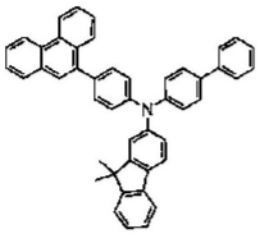
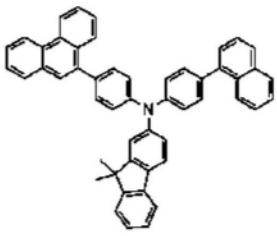
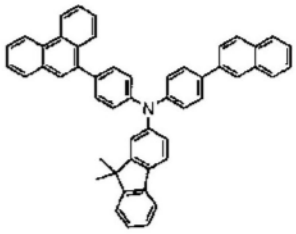
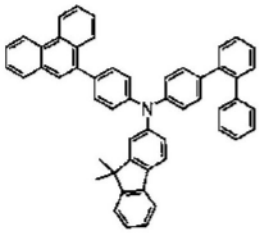
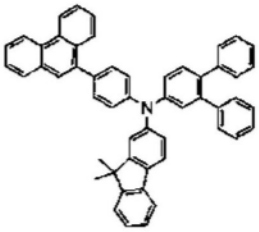
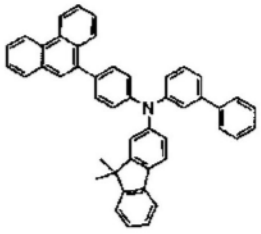
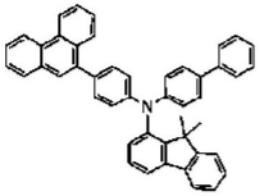
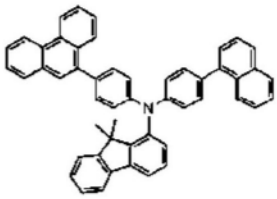
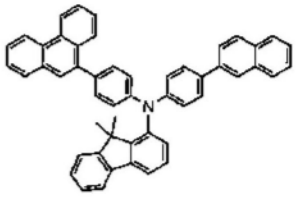
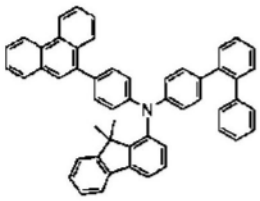
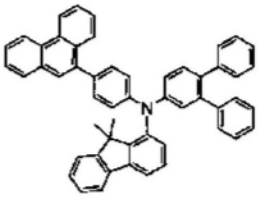
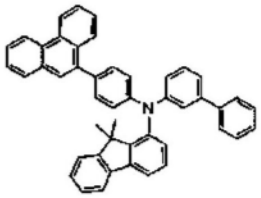







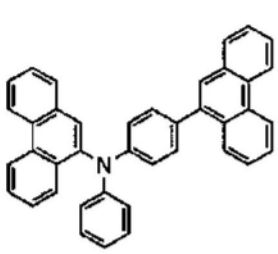
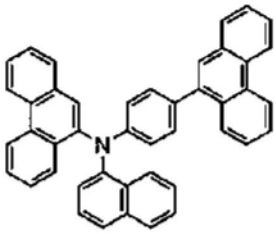
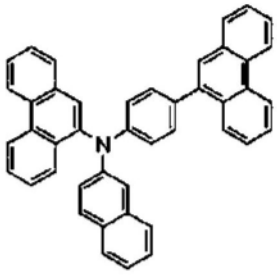
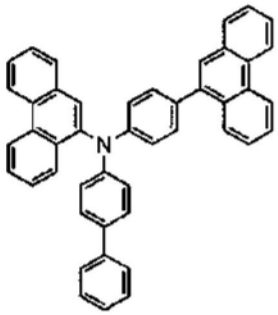
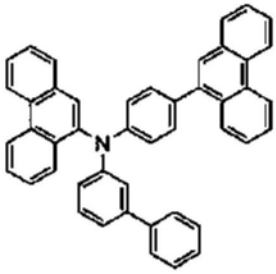
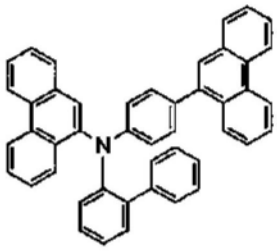







[0288]

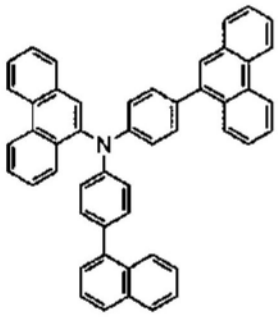
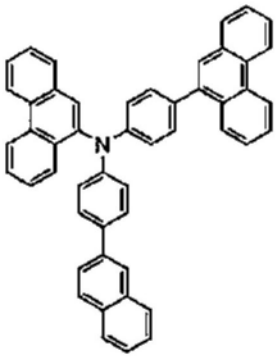
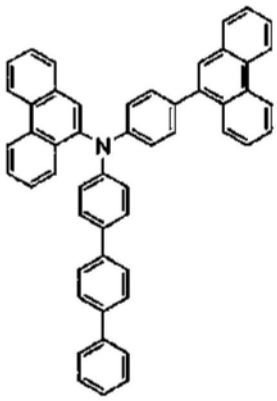
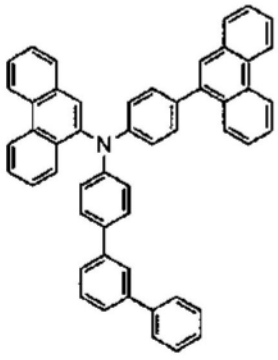
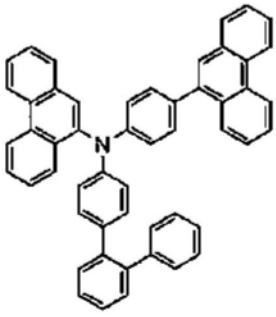
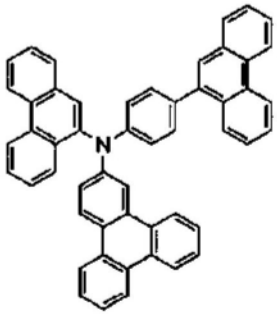







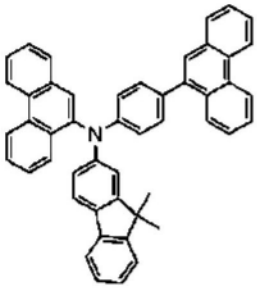
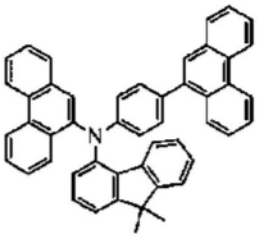
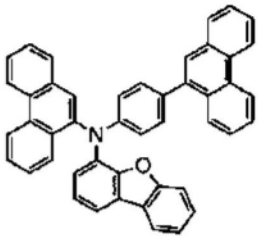
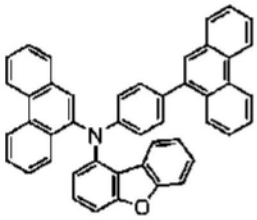
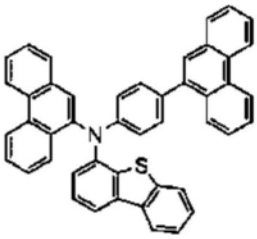
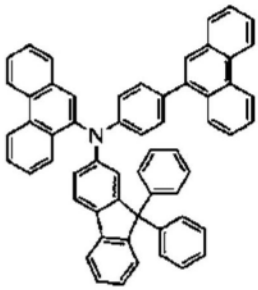
[0289]

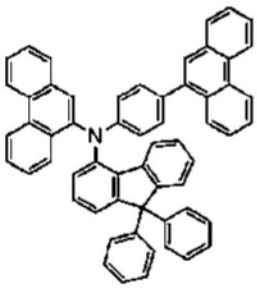
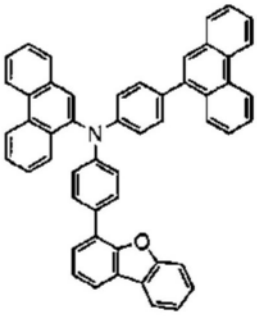
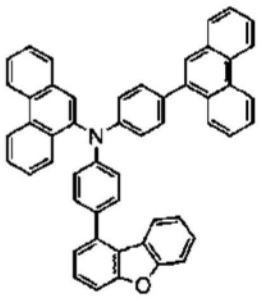
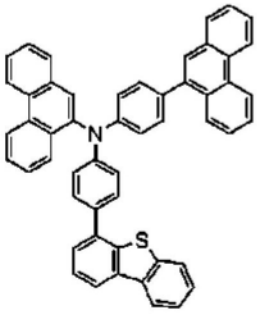
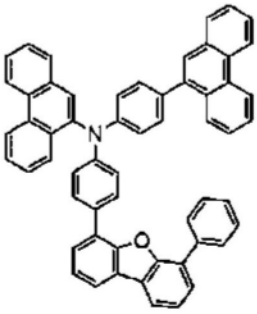
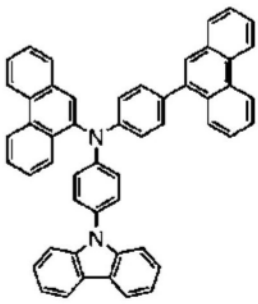







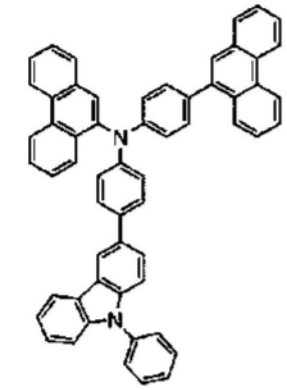
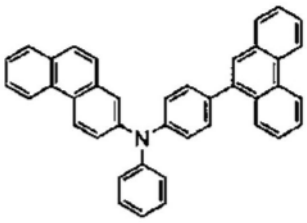
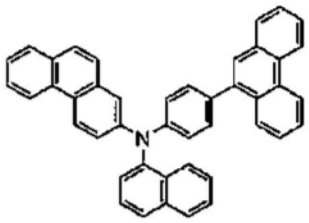
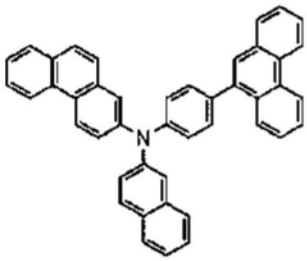
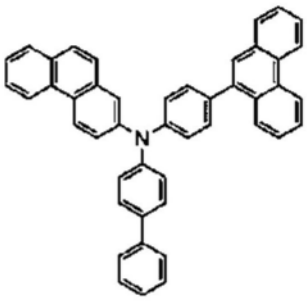
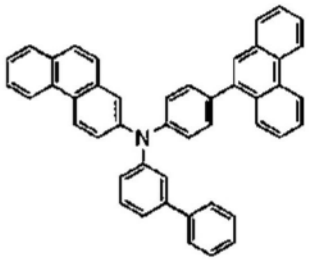
[0290]

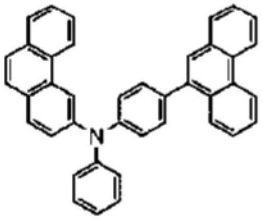
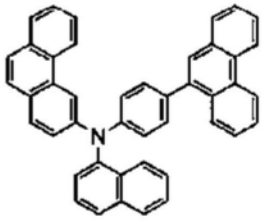
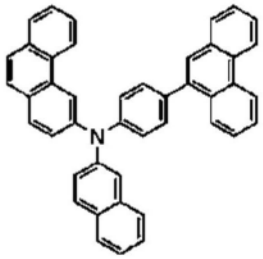
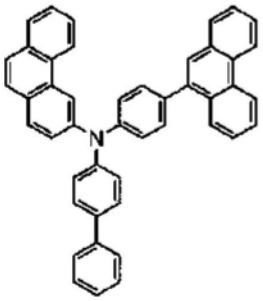
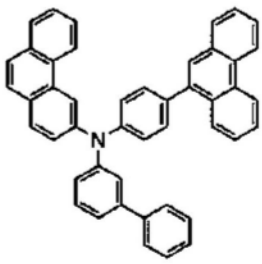
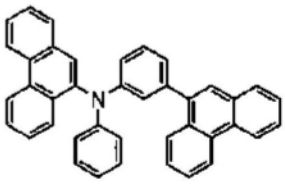







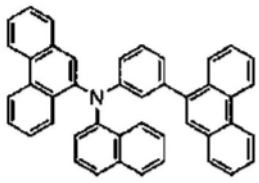
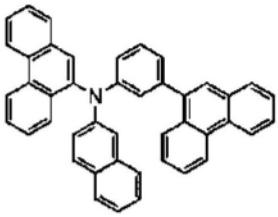
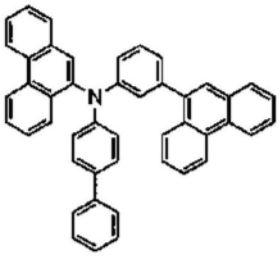
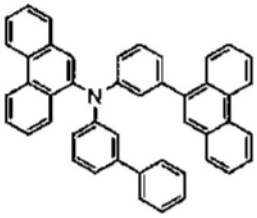
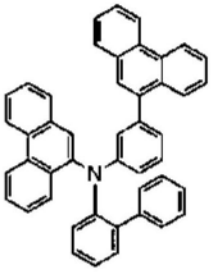
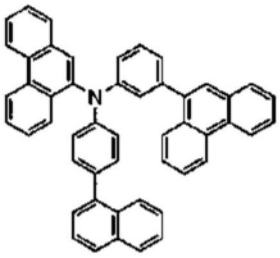
[0291]

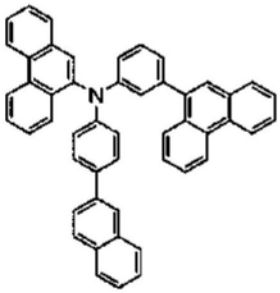
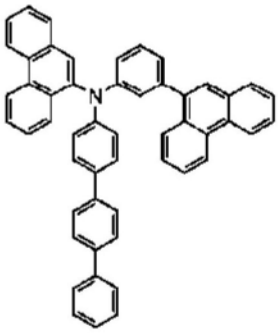
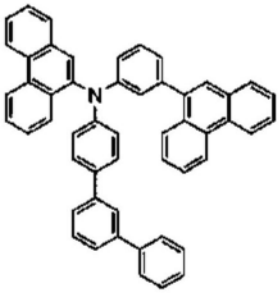
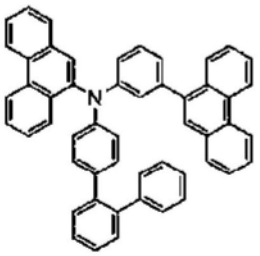
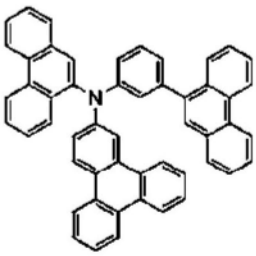
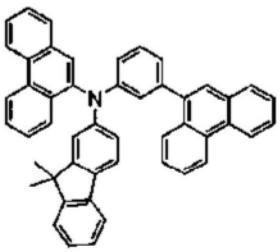







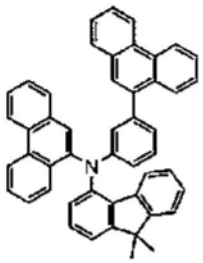
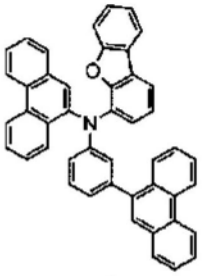
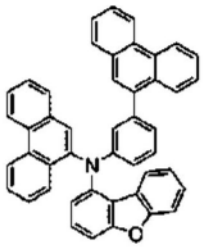
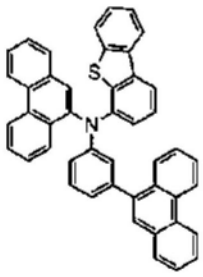
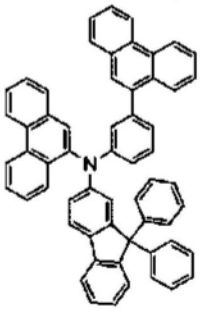
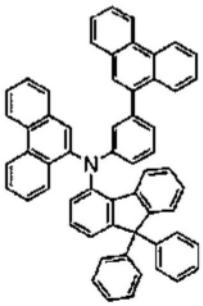
[0292]

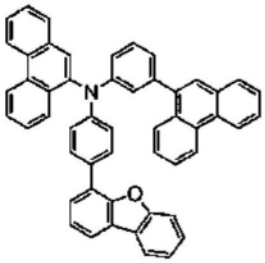
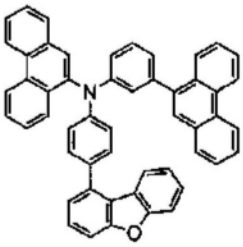
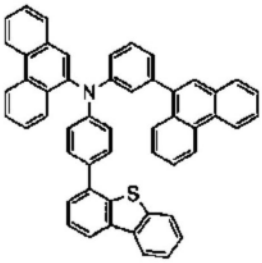
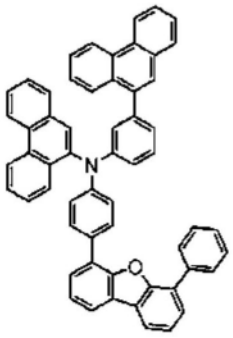
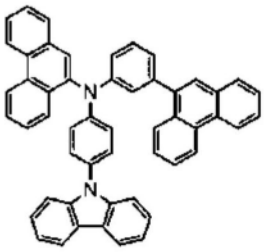
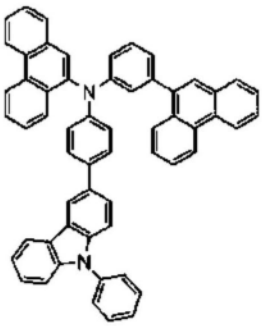







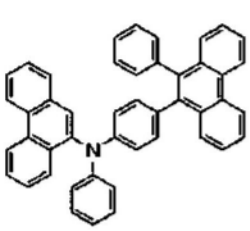
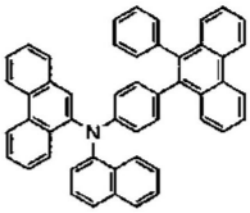
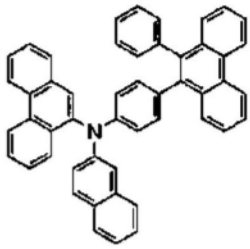
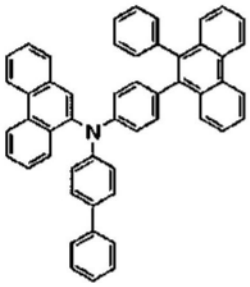
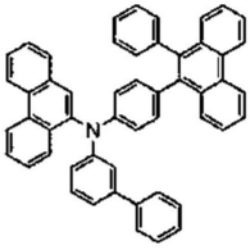
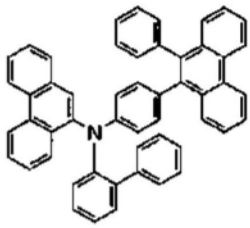
[0293]

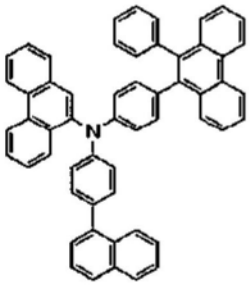
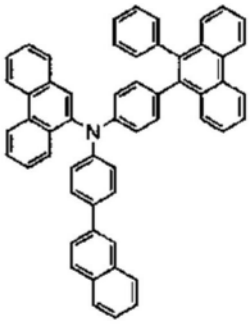
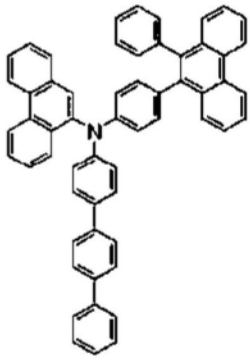
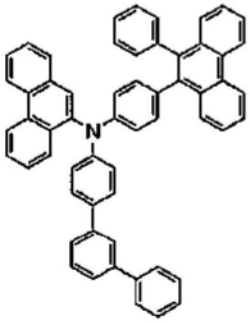
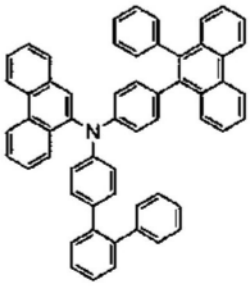
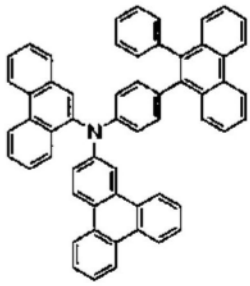







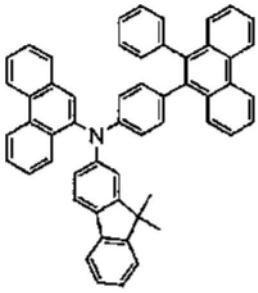
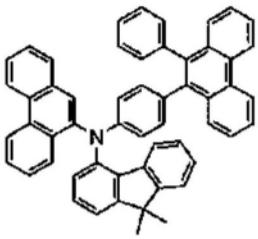
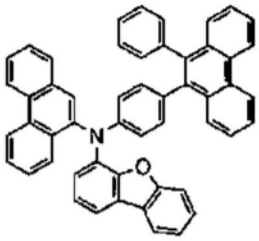
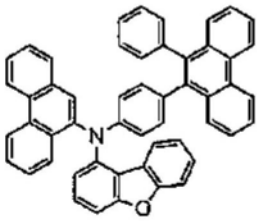
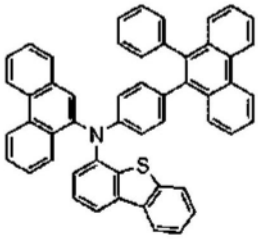
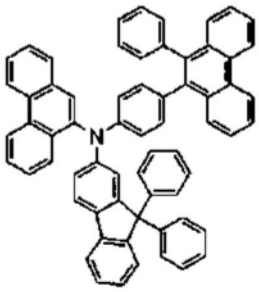
[0294]

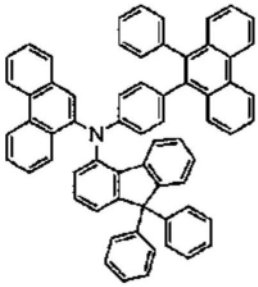
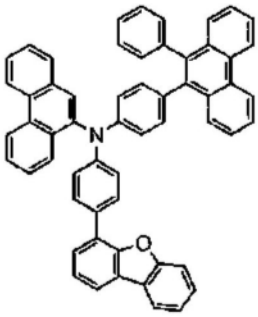
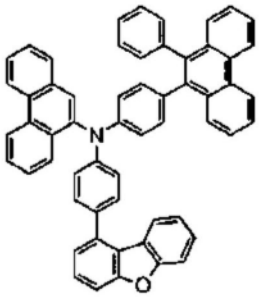
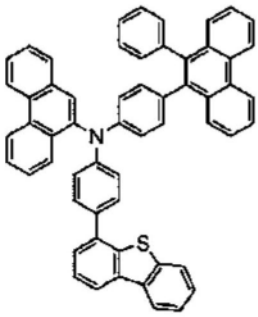
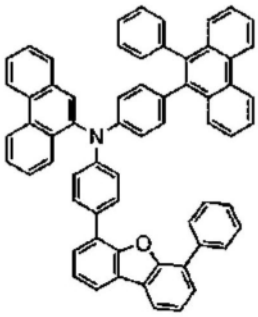
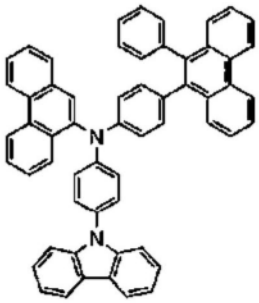







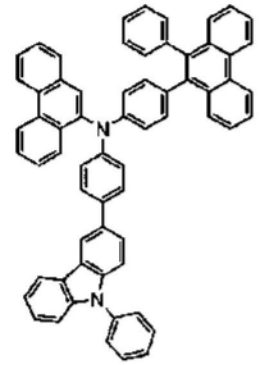
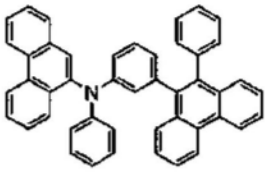
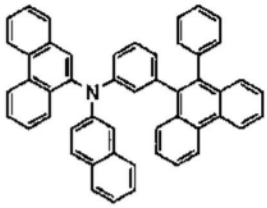
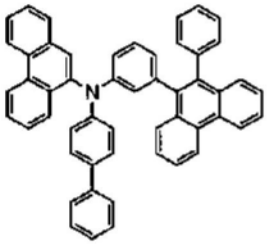
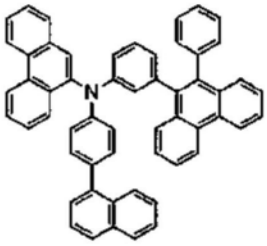
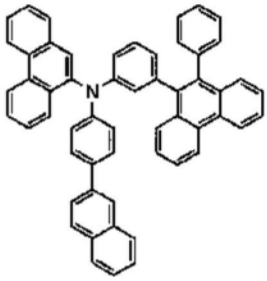
[0295]

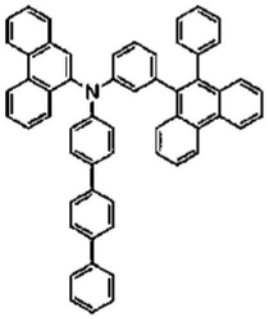
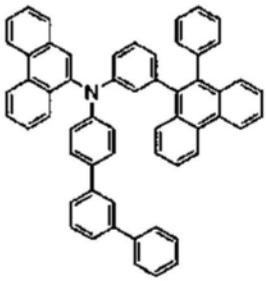
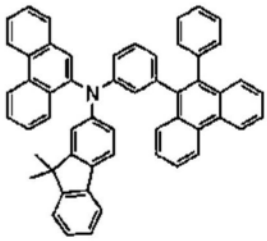
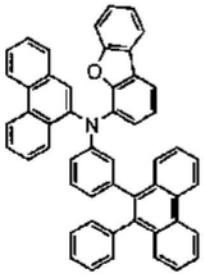
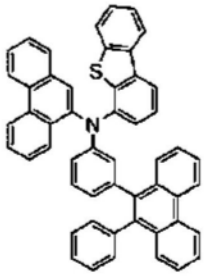
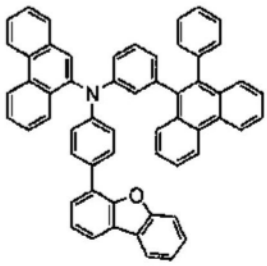







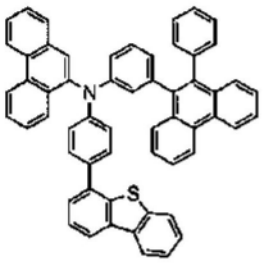
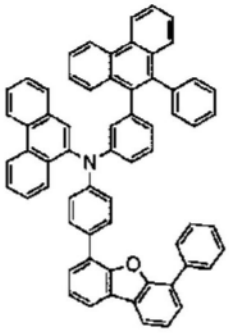
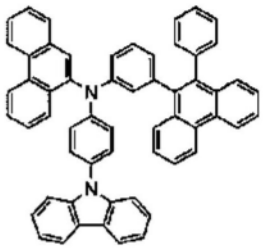
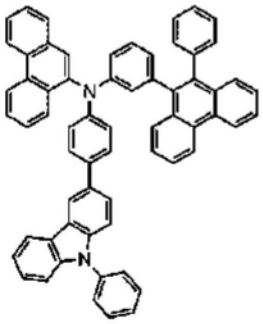
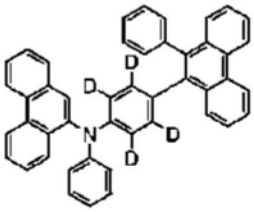
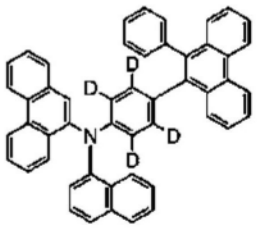
[0296]

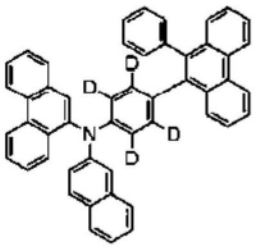
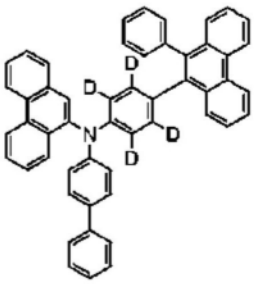
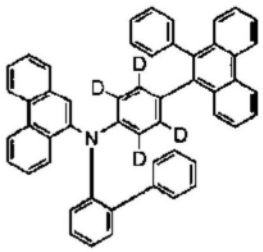
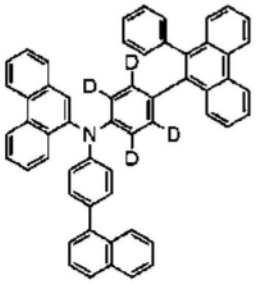
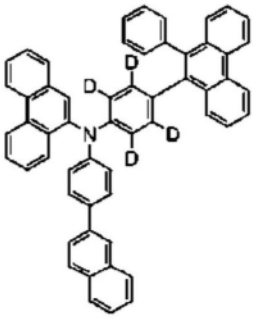
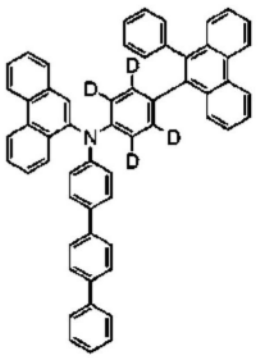







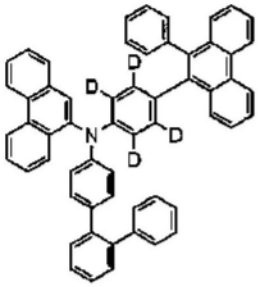
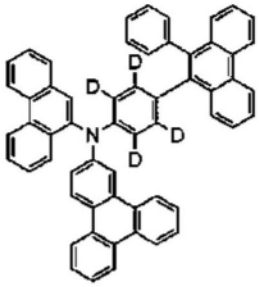
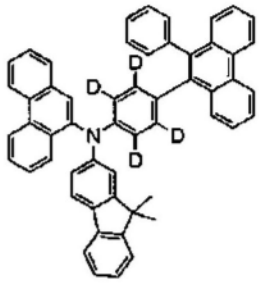
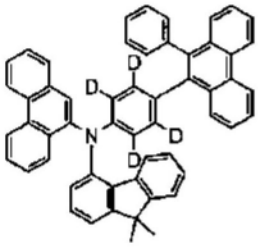
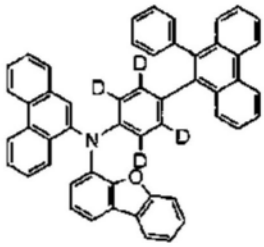
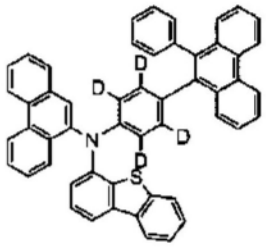
[0297]

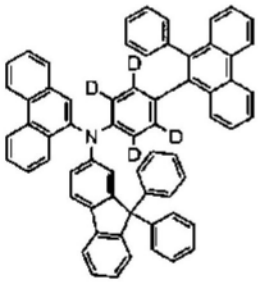
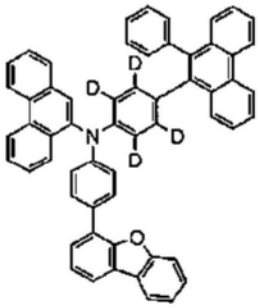
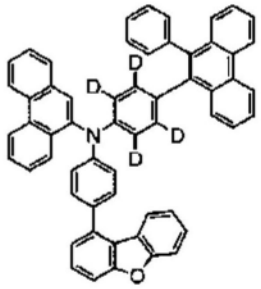
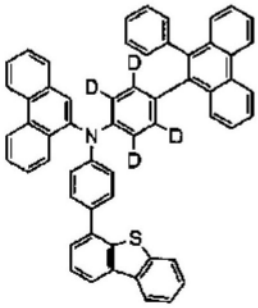
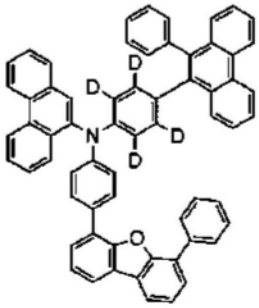
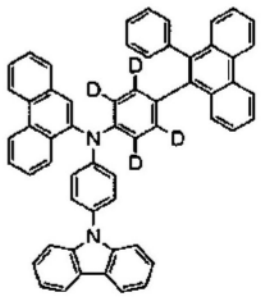







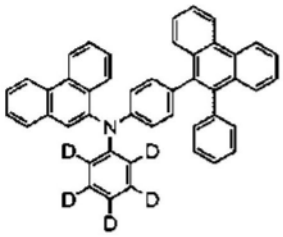
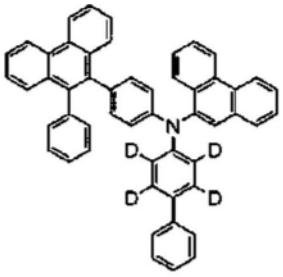
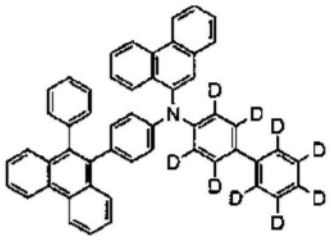
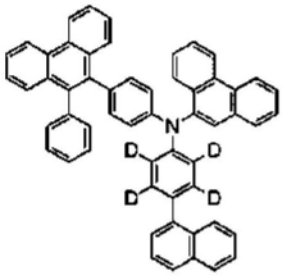
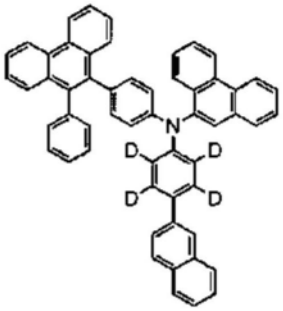
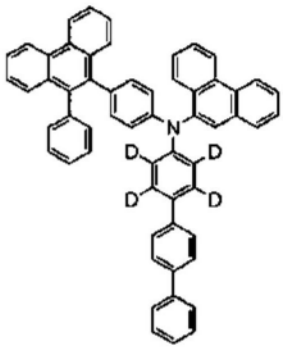
[0298]

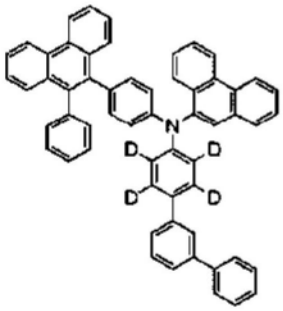
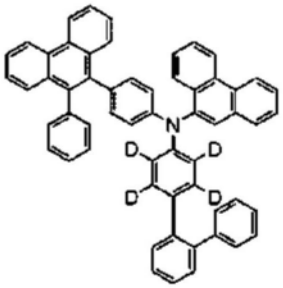
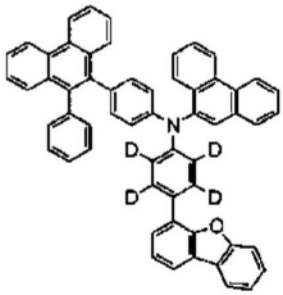
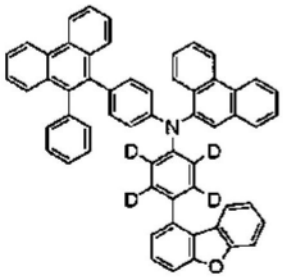
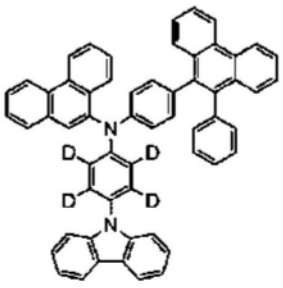
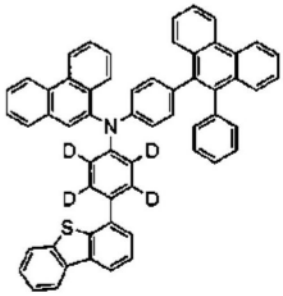







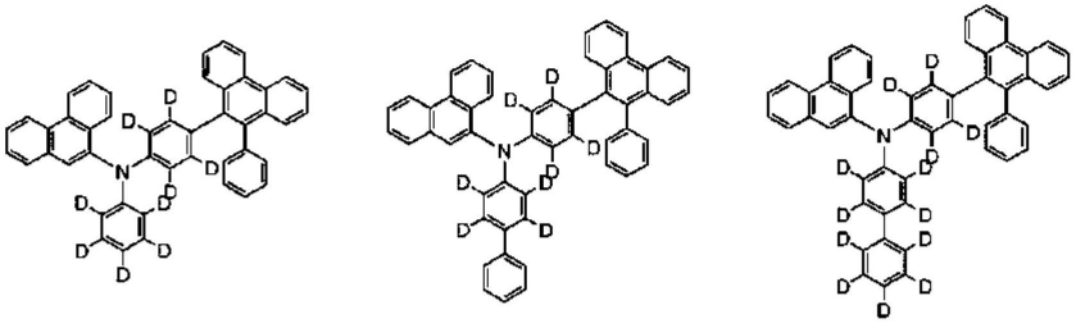
[0299]

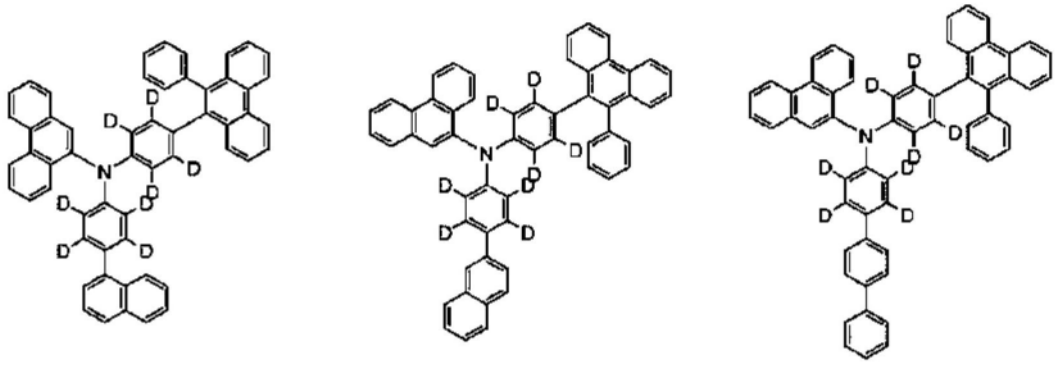







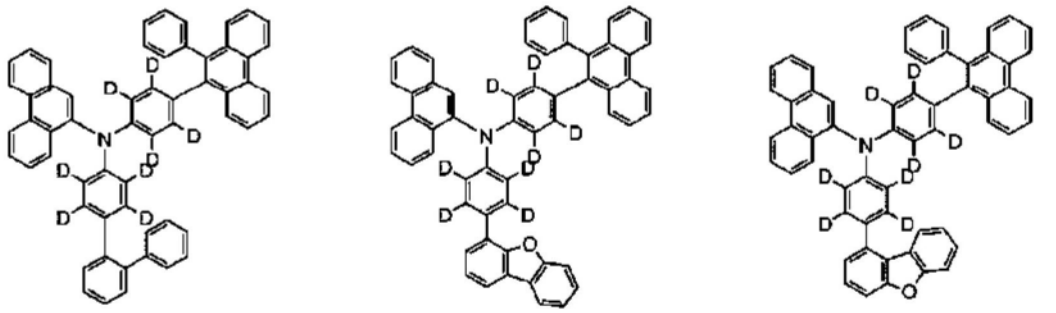
[0300]

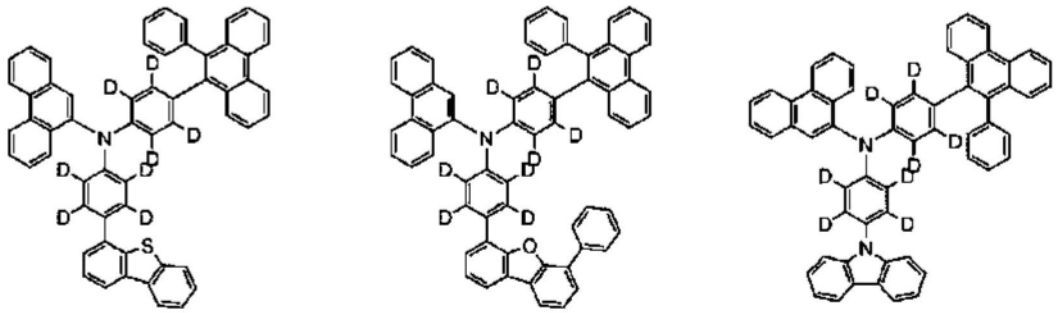







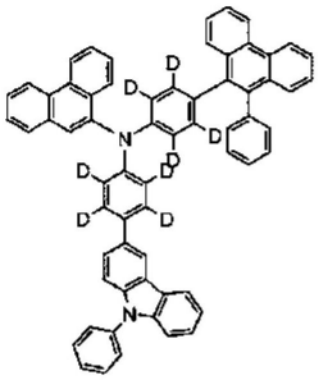
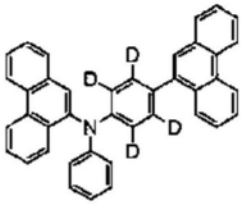
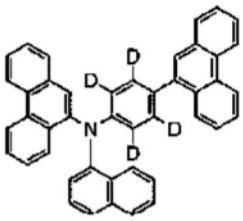
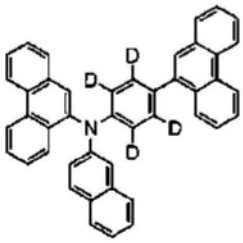
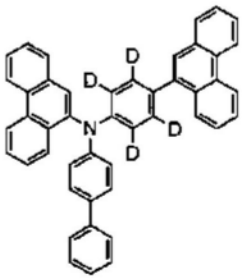
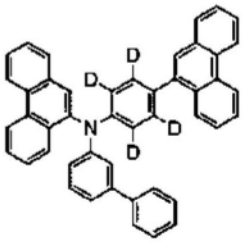




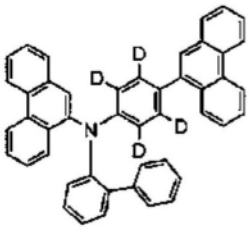
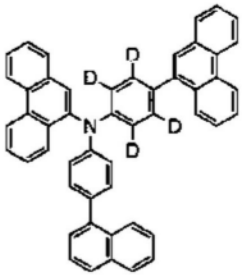
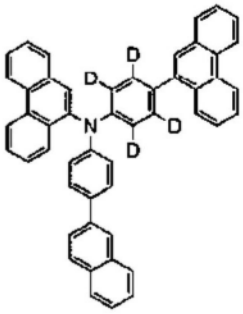
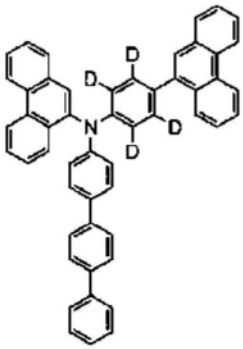
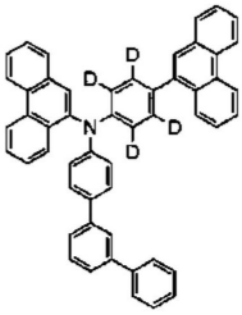
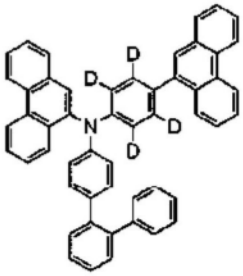
[0301]

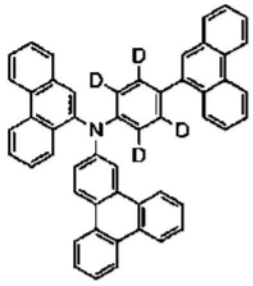
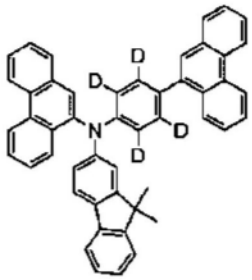
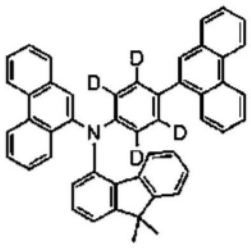
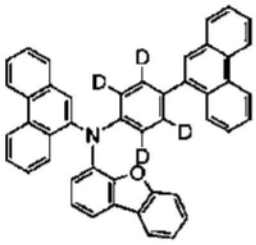
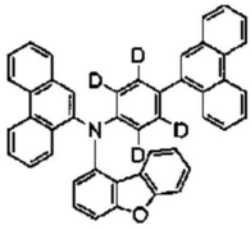
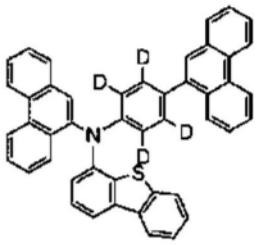




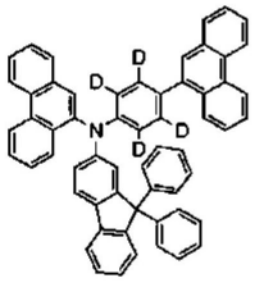
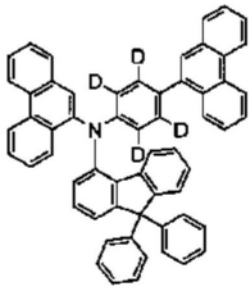
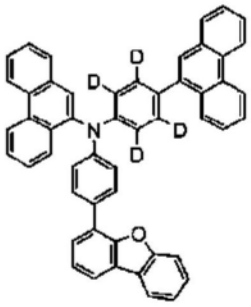
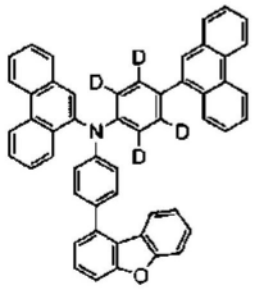
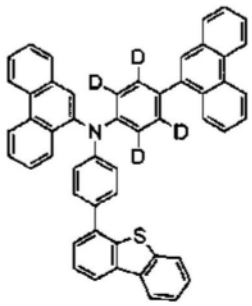
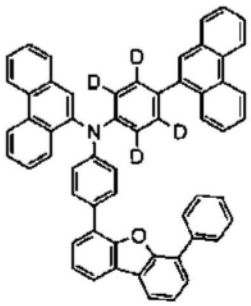







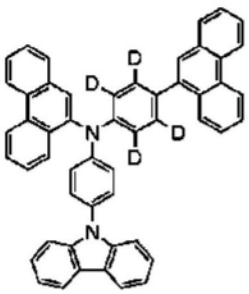
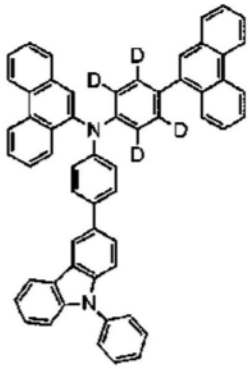
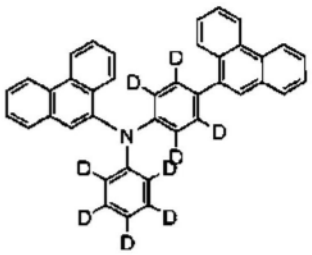
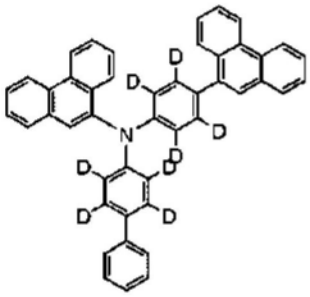
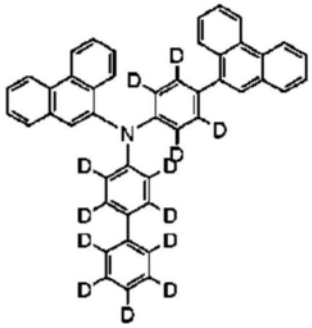
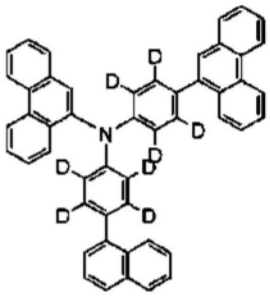
[0302]

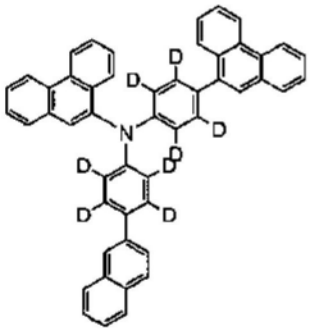
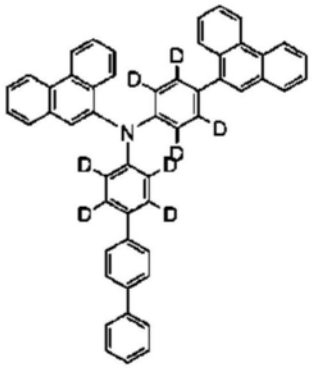
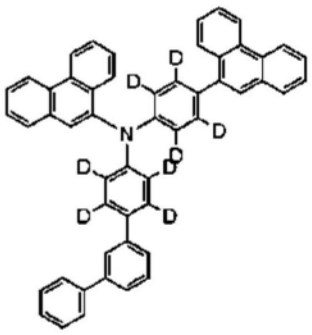
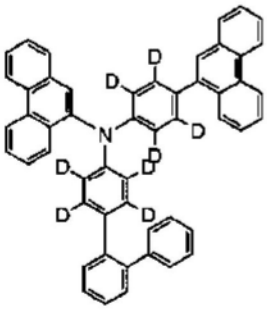
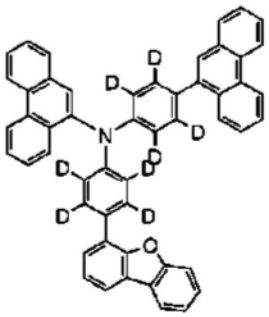
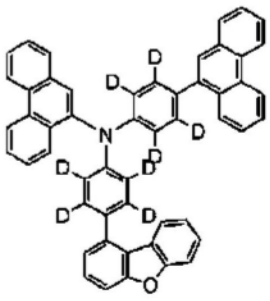







[0303]

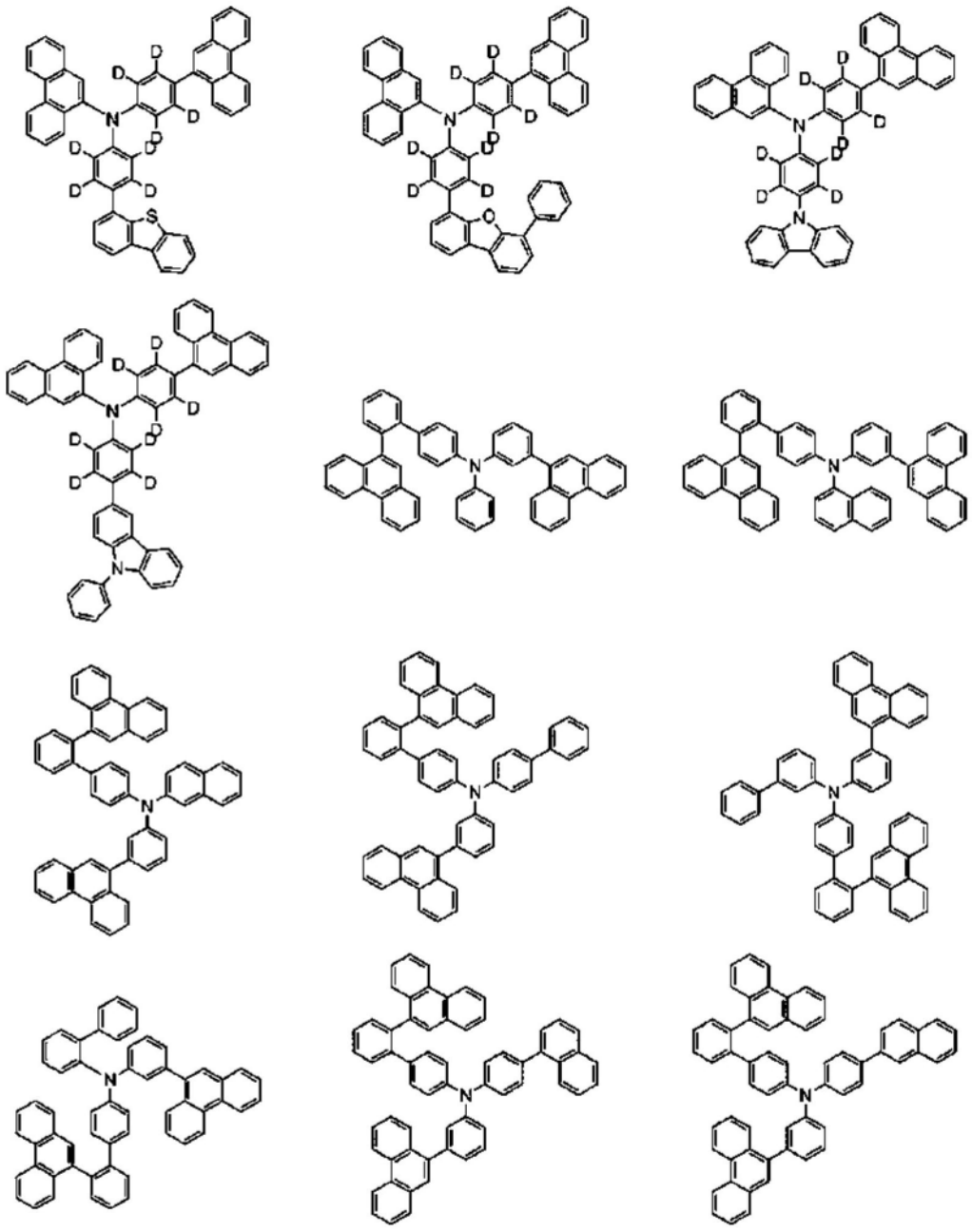







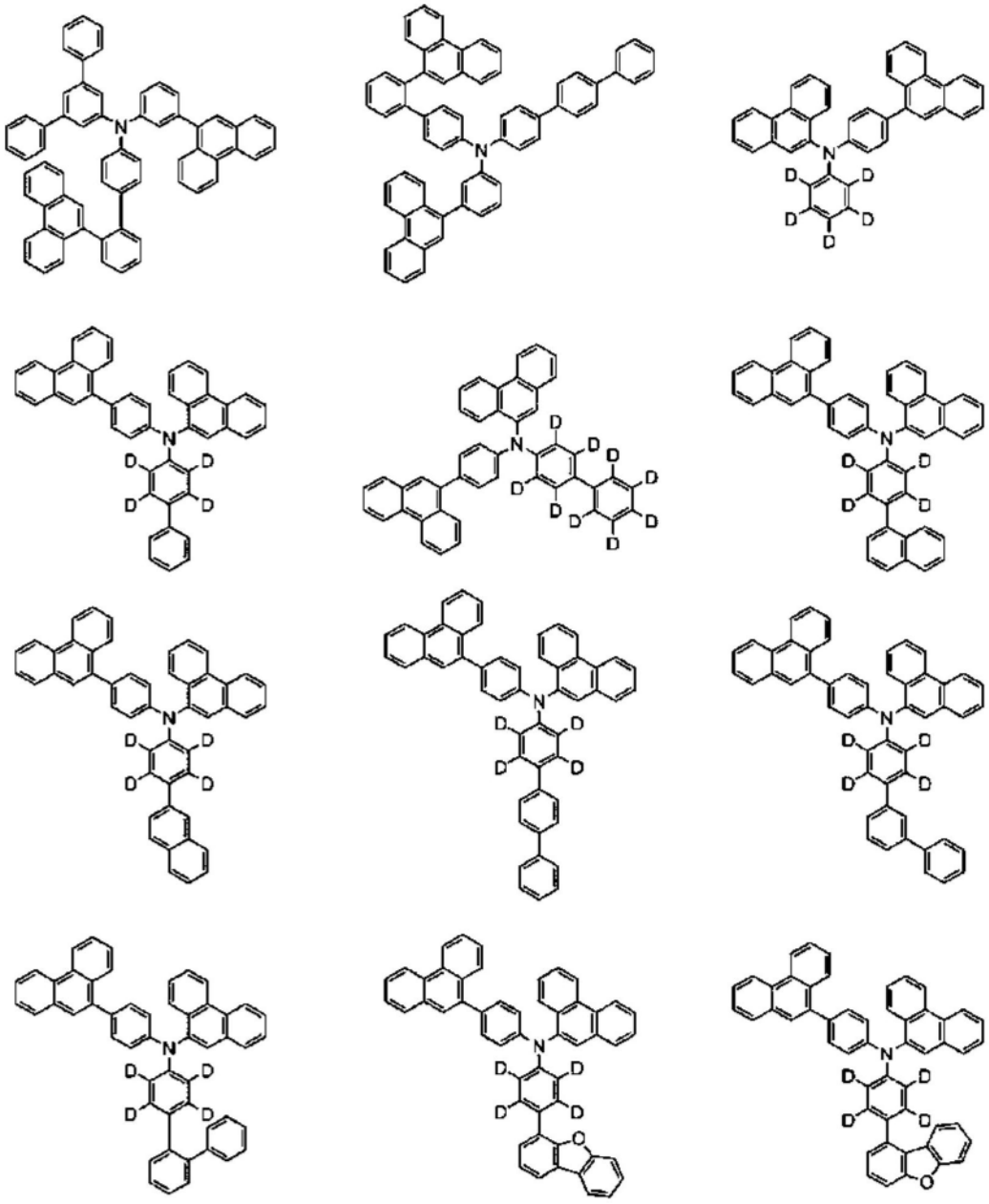
[0304]

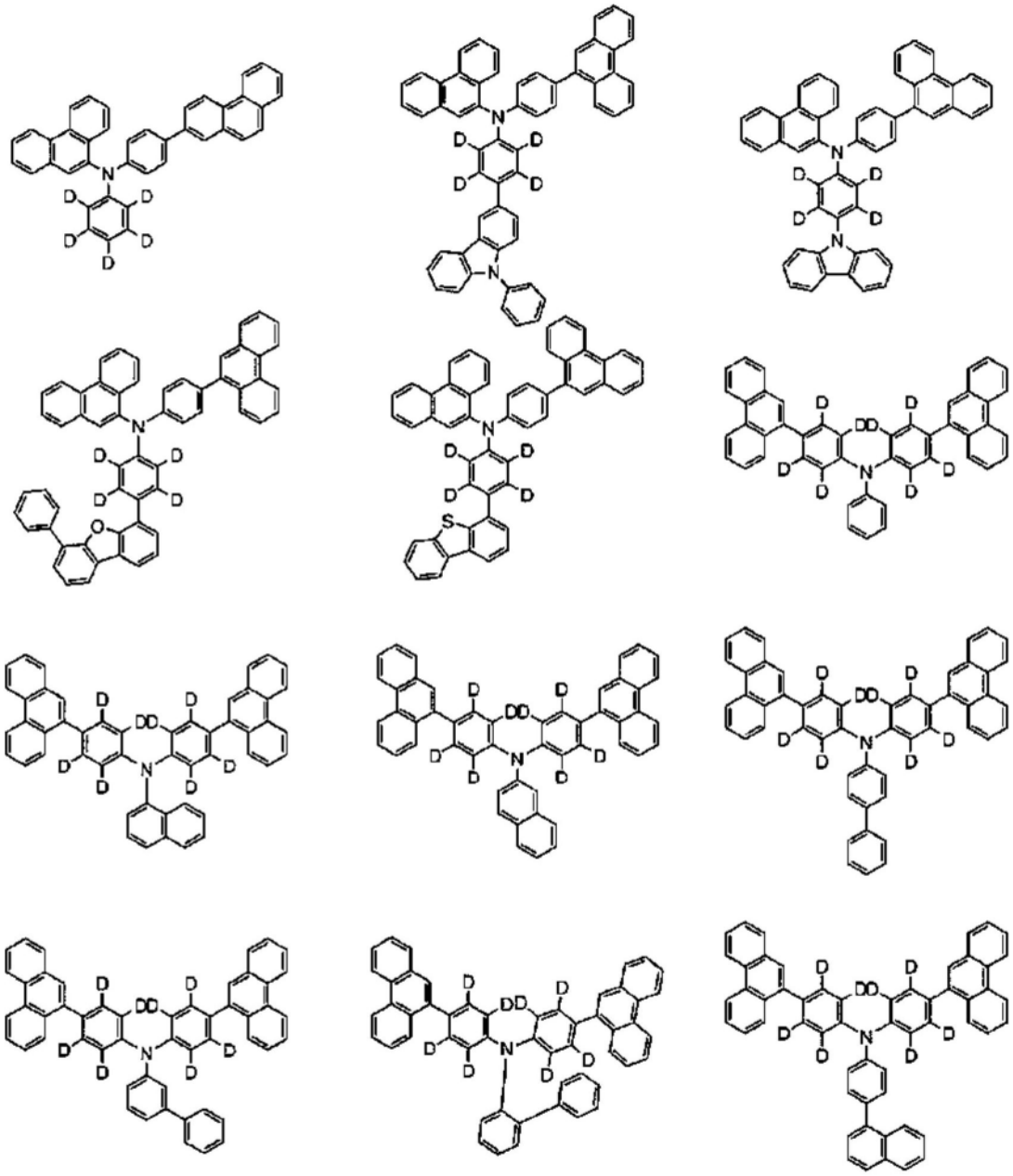
[0305]



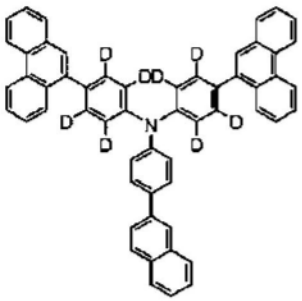
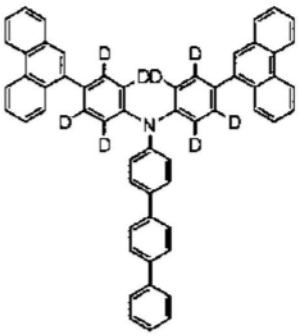
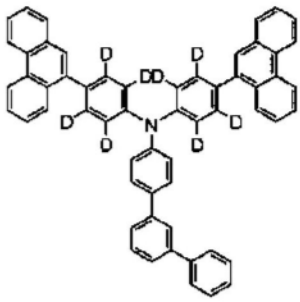
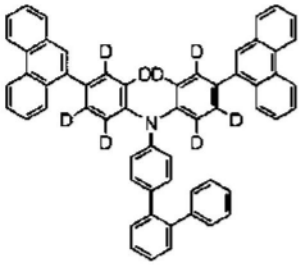
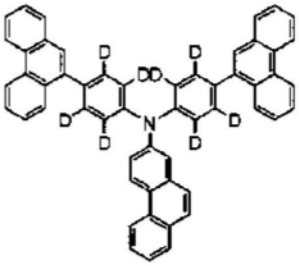
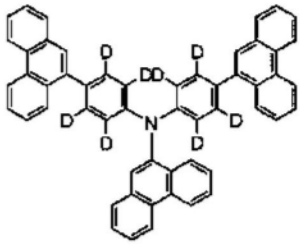
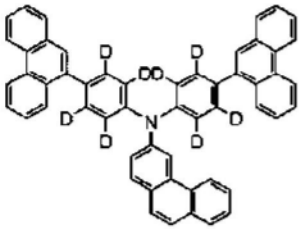
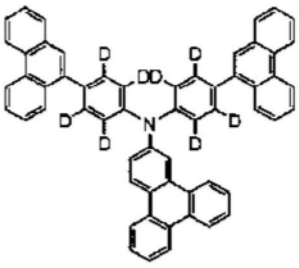
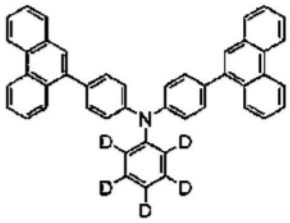
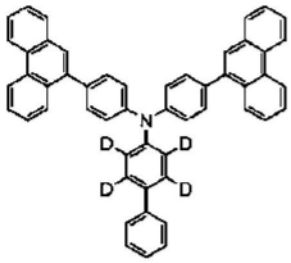
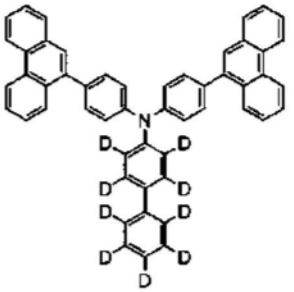
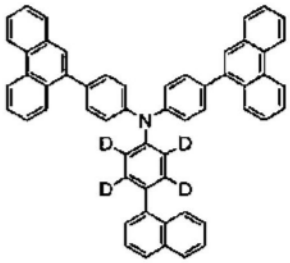
[0306]

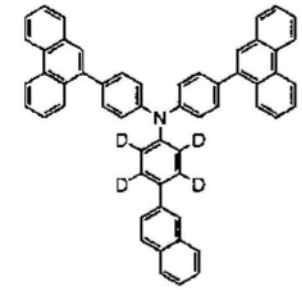
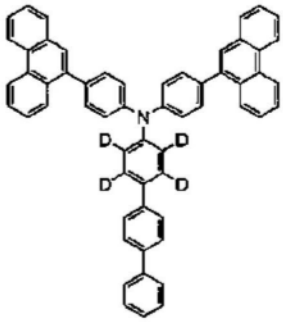
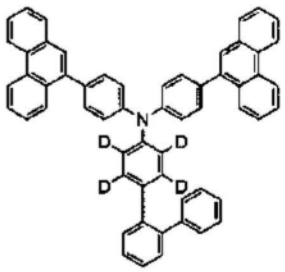
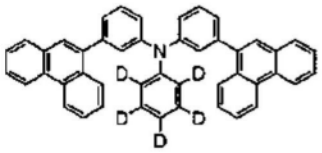
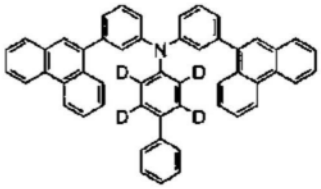
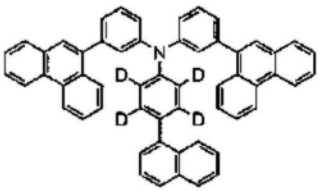


[0307]

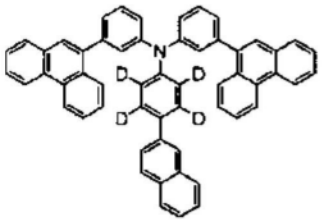
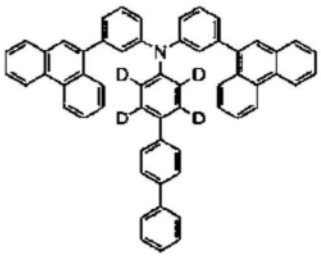
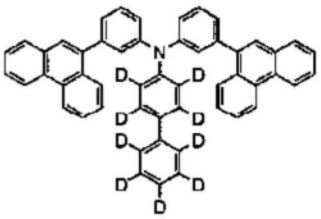
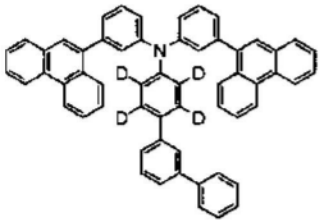
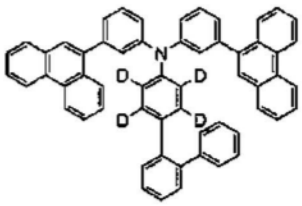
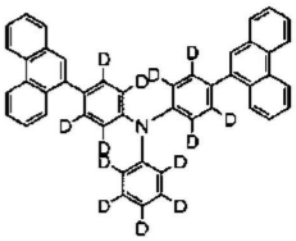


[0308]

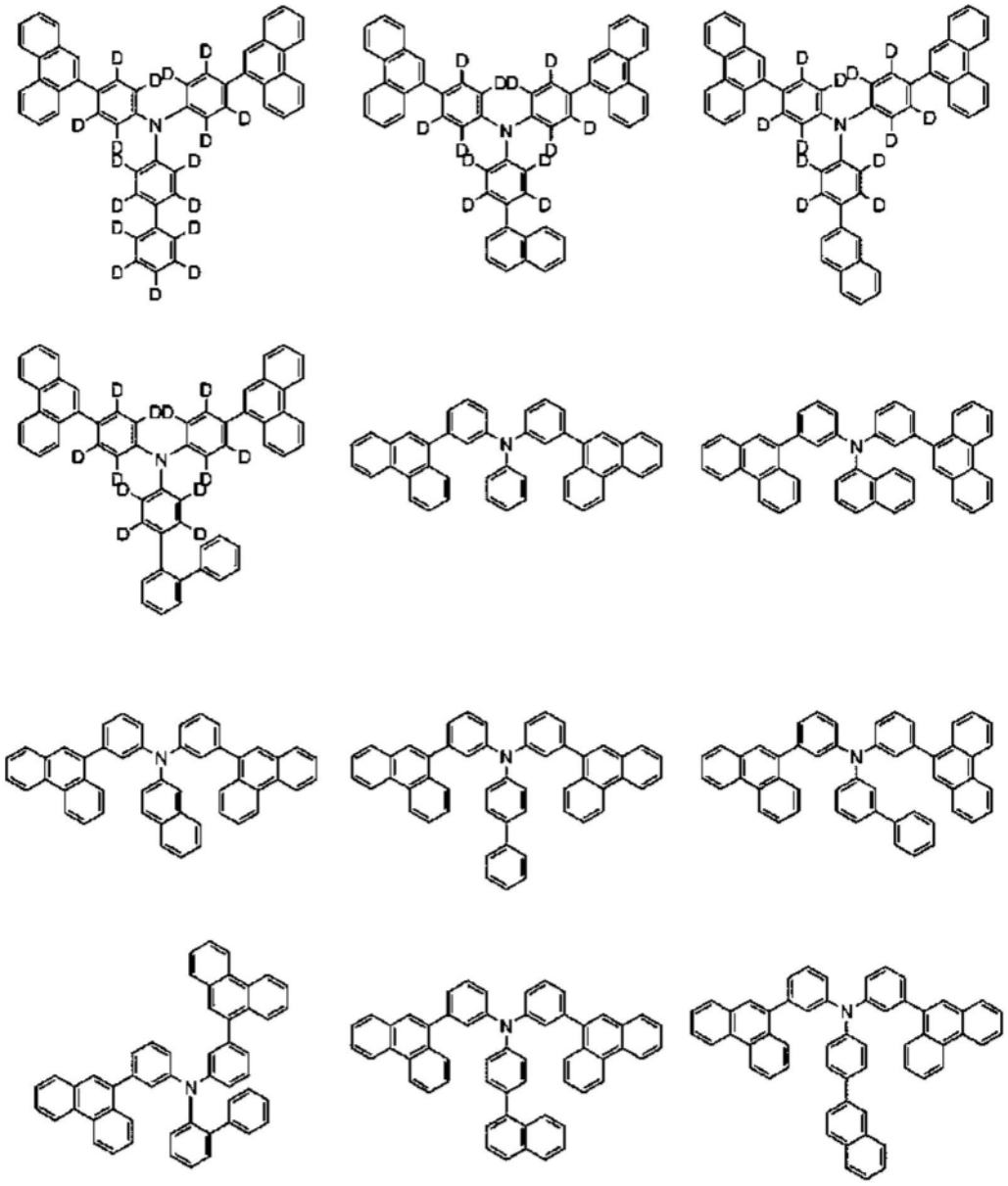













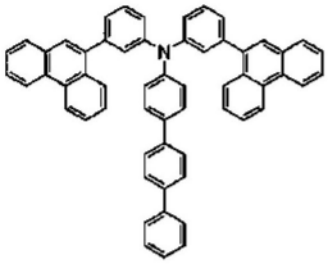
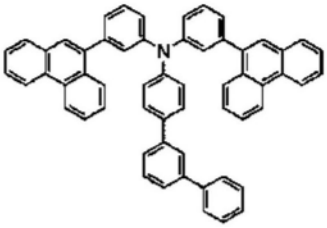
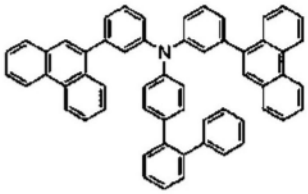
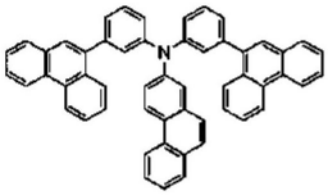
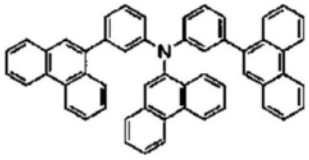
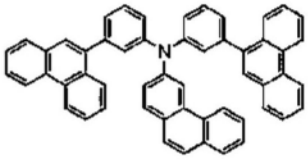







[0309]

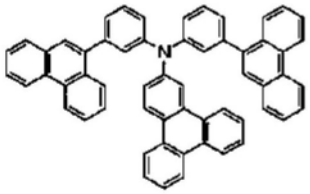
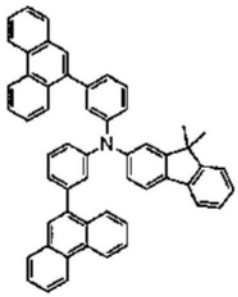
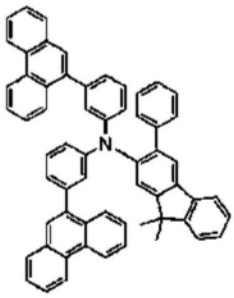
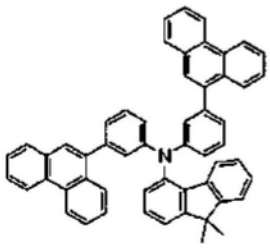
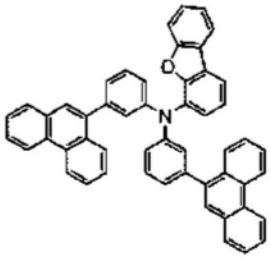
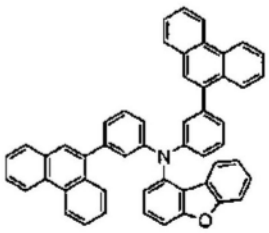







[0310]

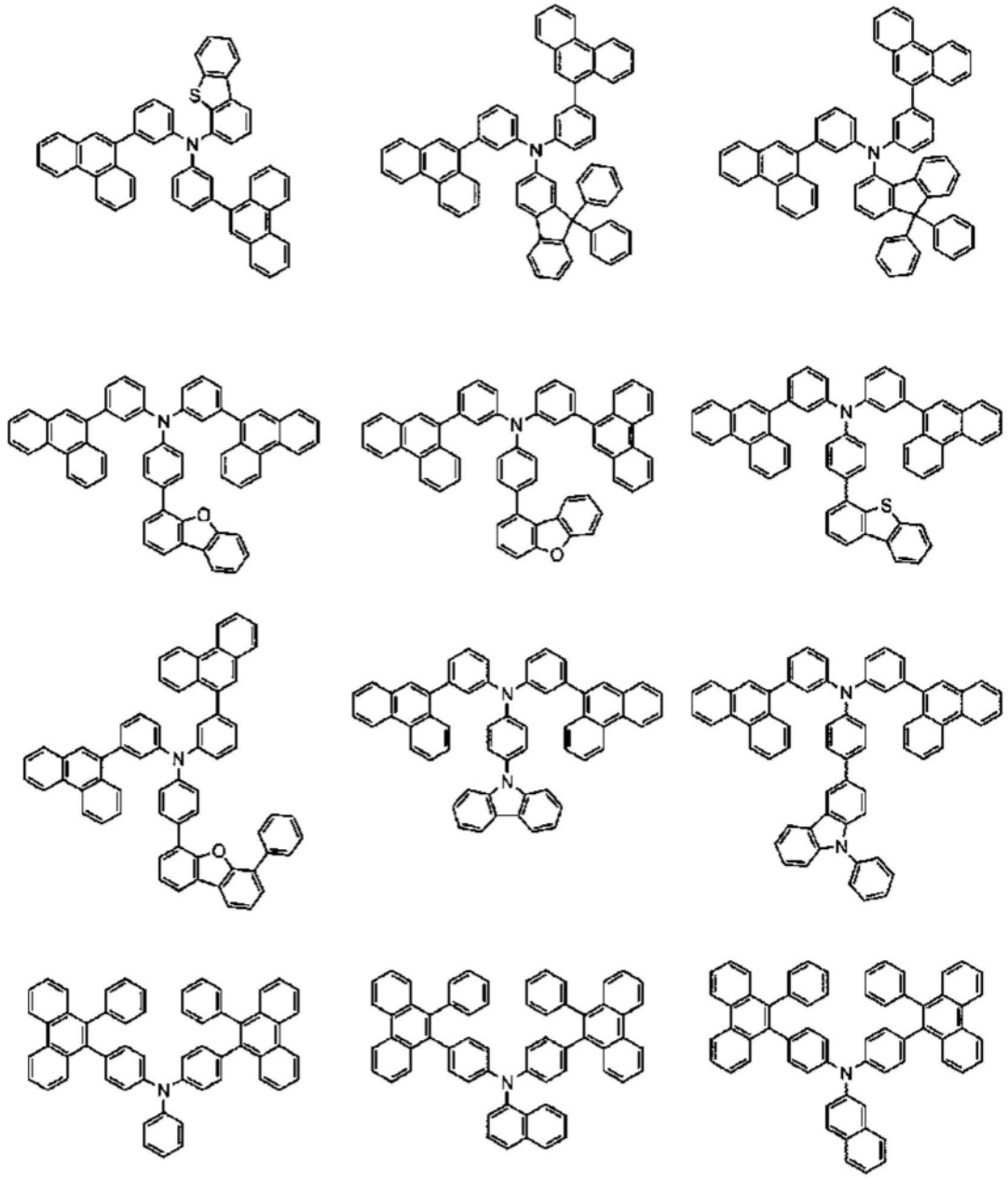


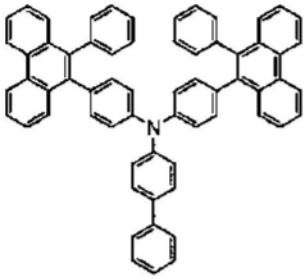
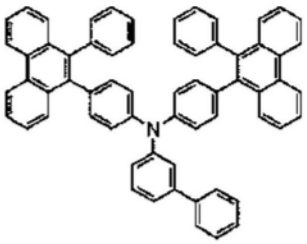
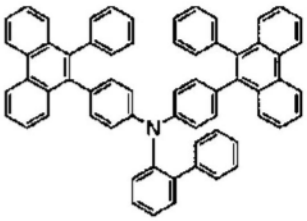
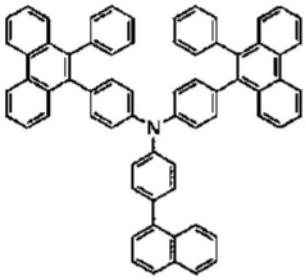
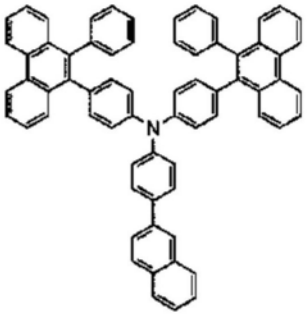
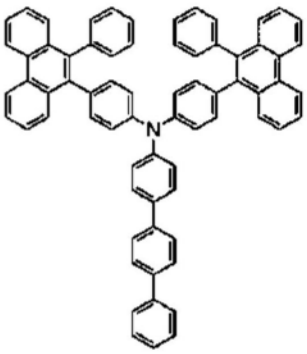







[0311]

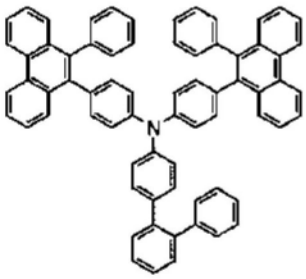
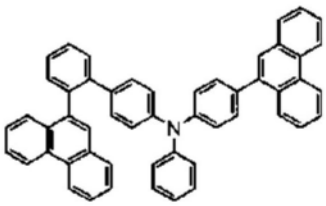
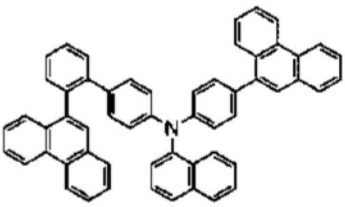
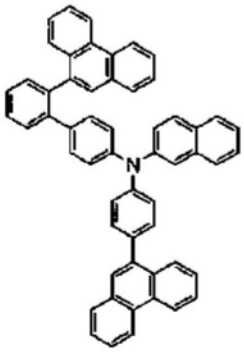
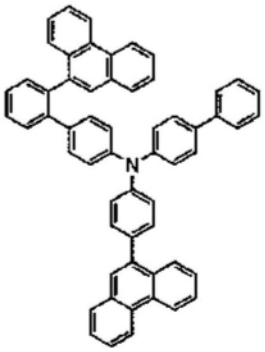
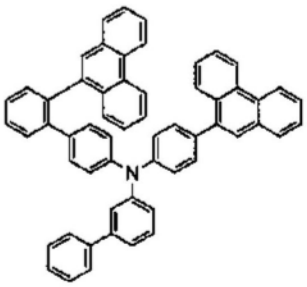







[0312]

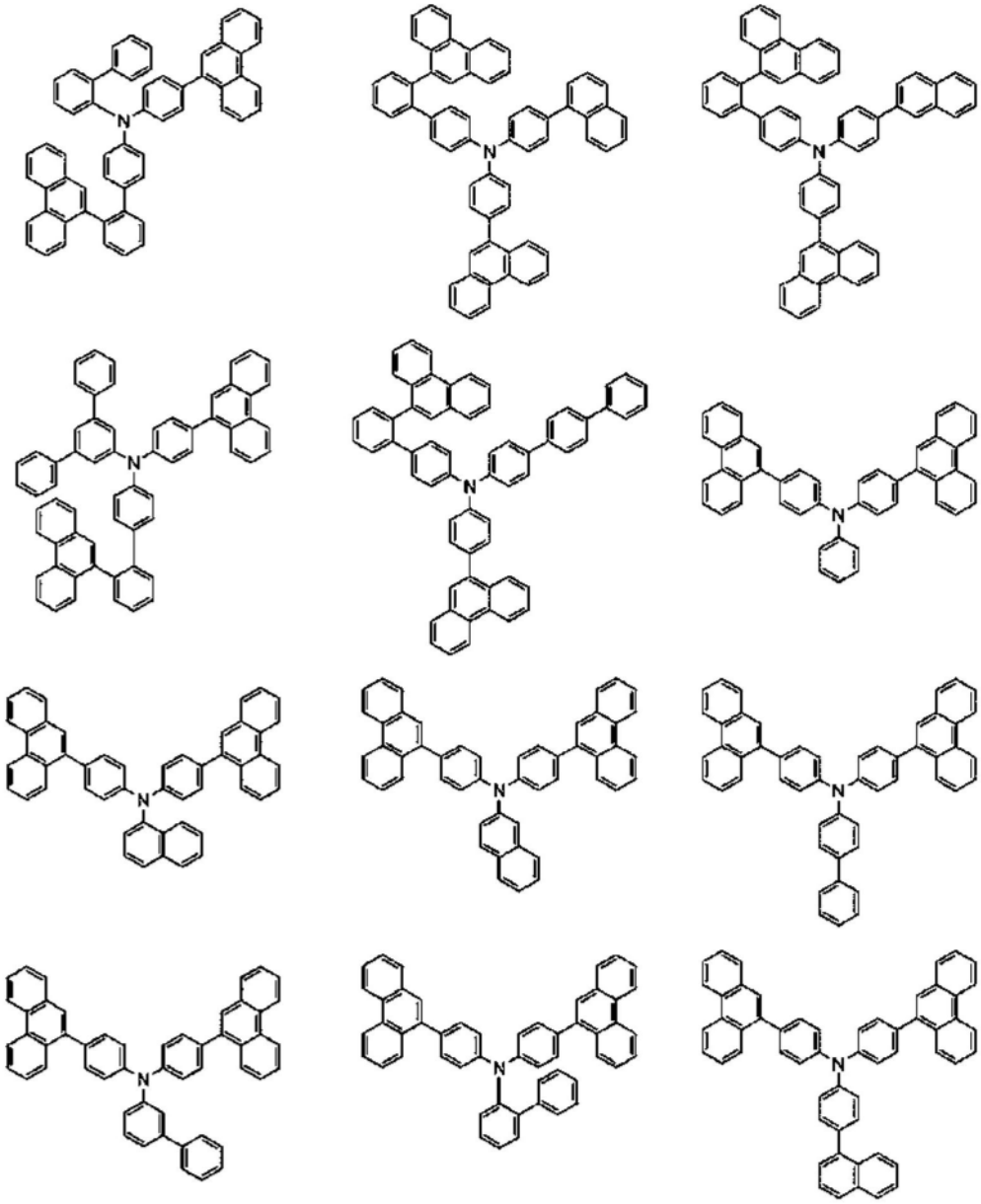


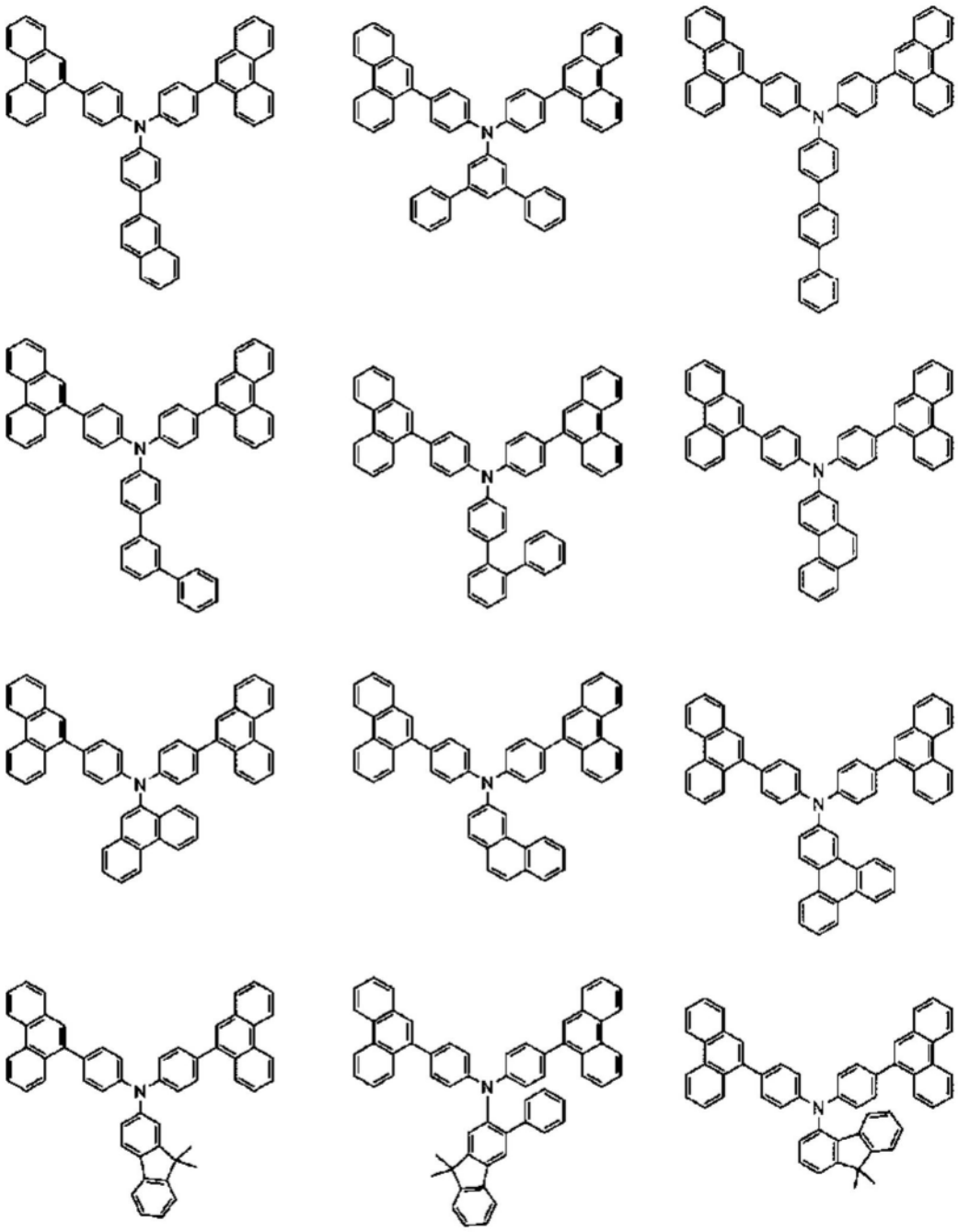
[0313]

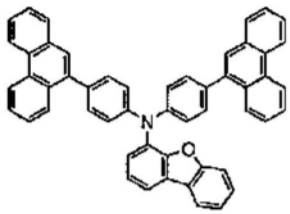
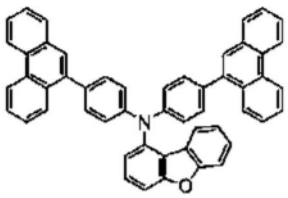
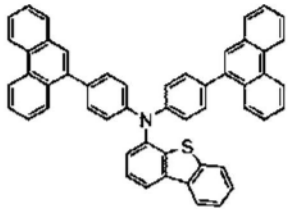
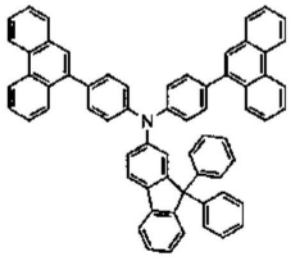
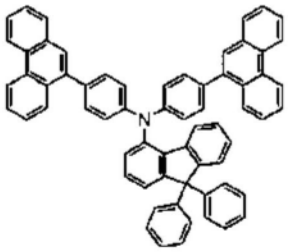
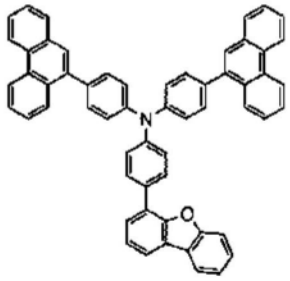







[0314]

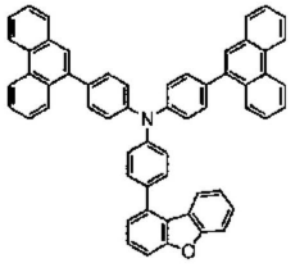
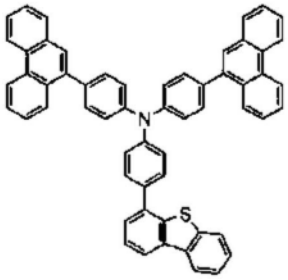
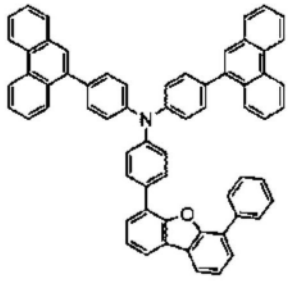
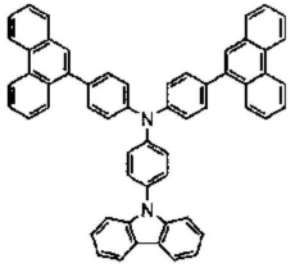
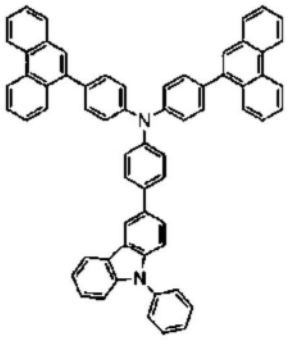
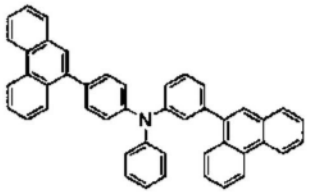


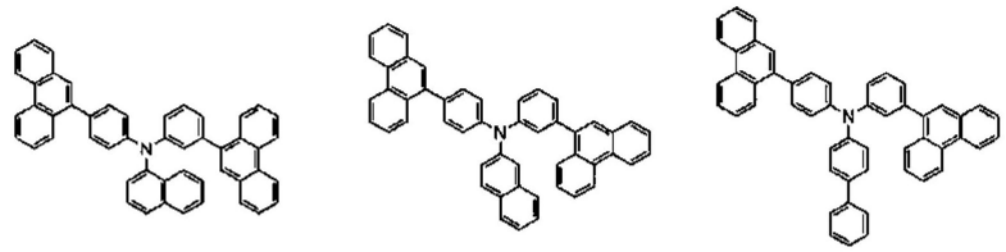
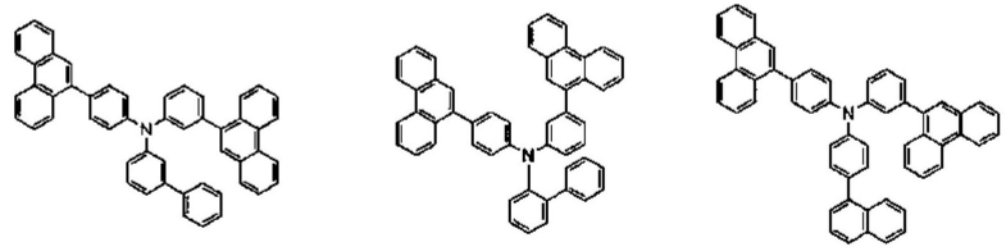
[0315]



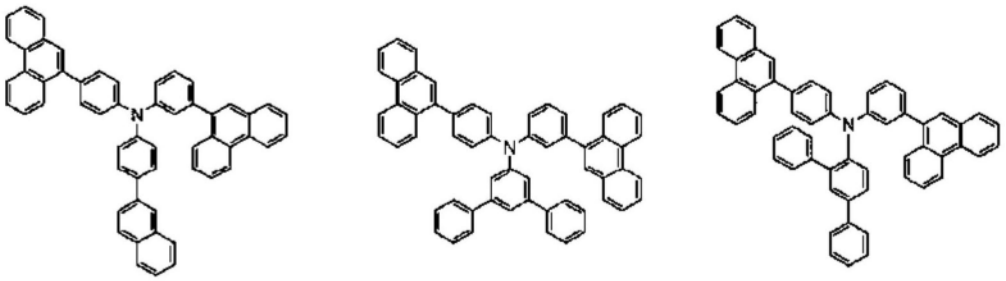
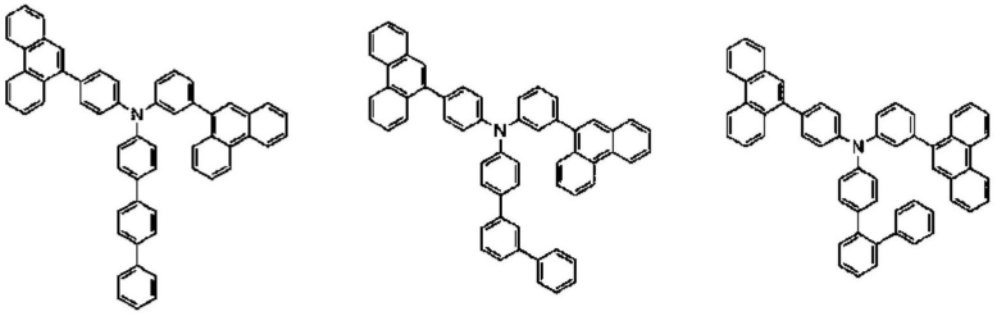







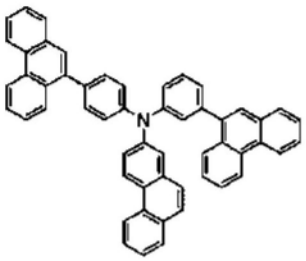
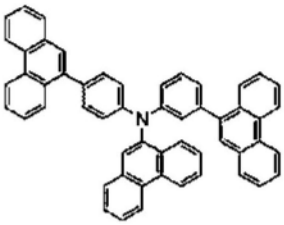
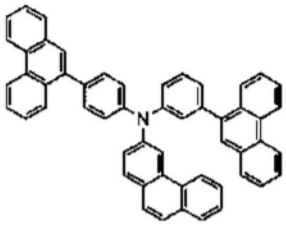
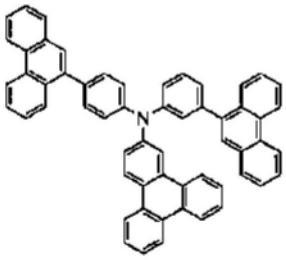
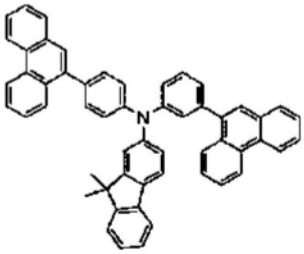
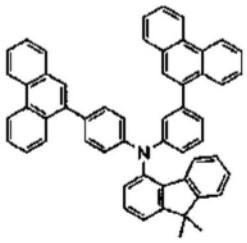
[0316]

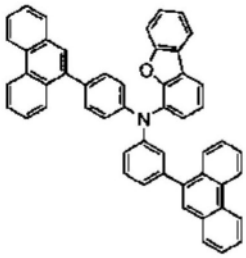
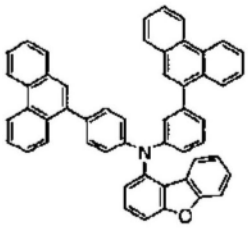
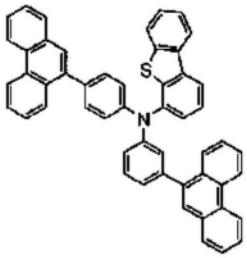
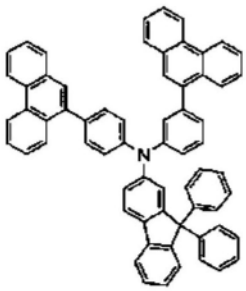
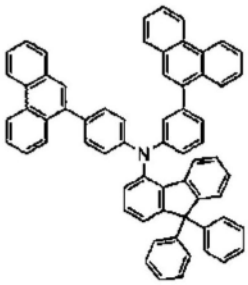
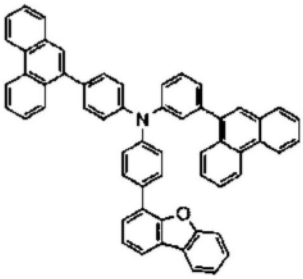



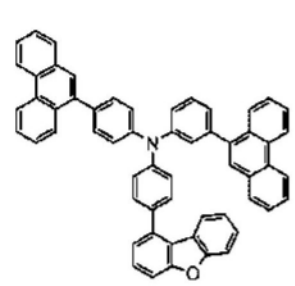
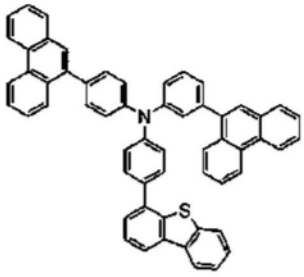
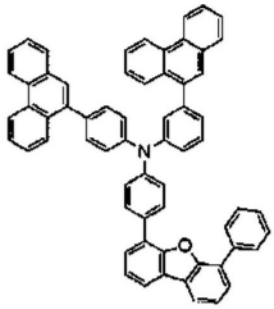
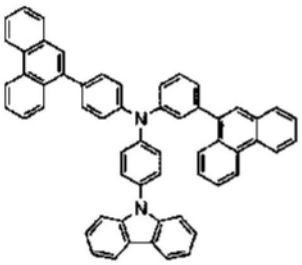
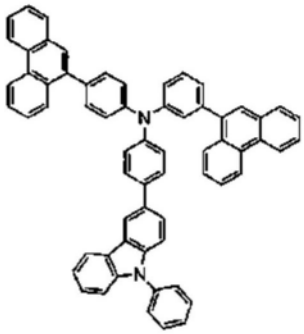
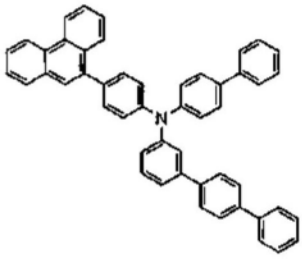
[0317]

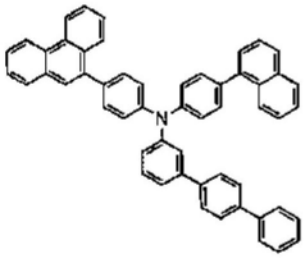
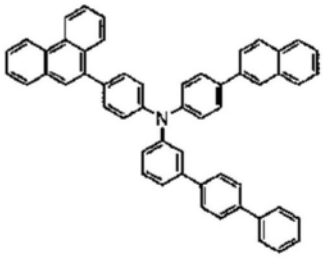
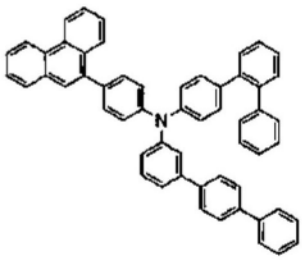
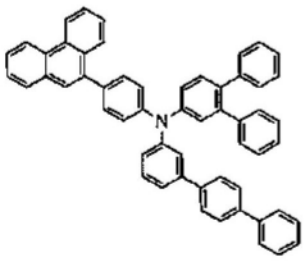
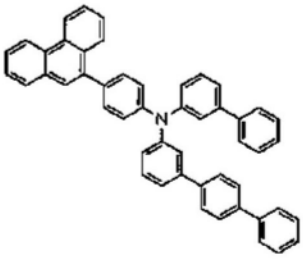
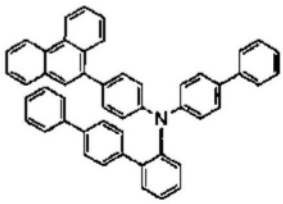







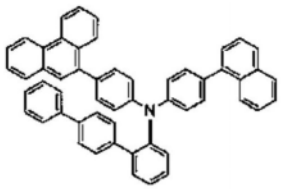
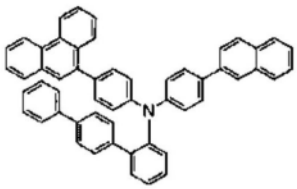
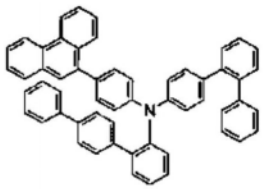
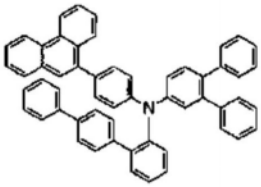
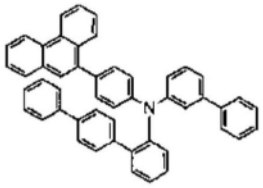
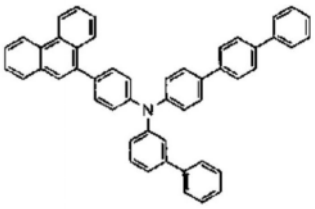
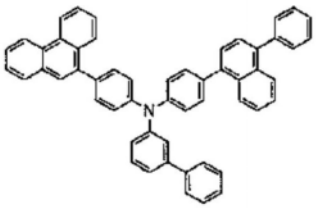
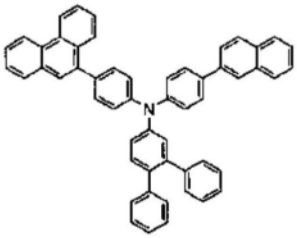
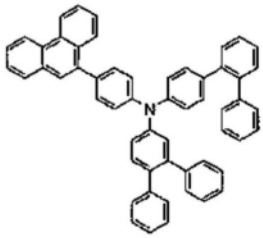
[0318]

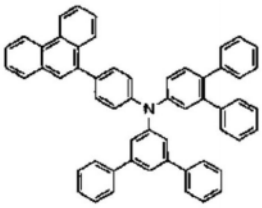
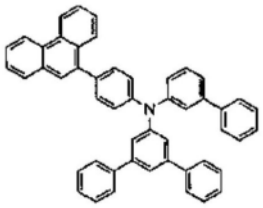
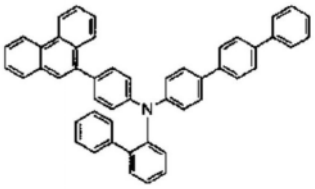
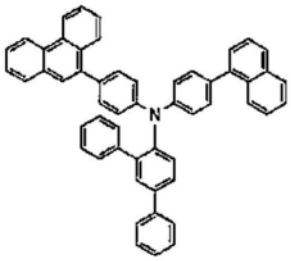
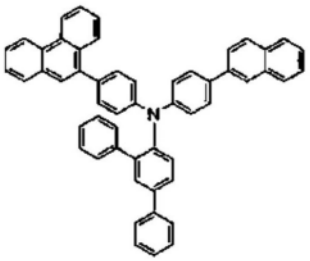
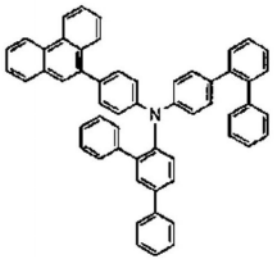
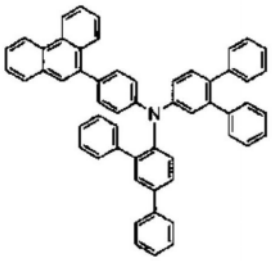
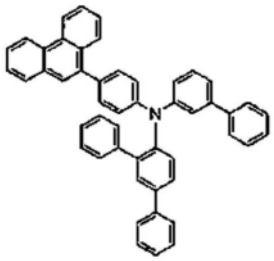







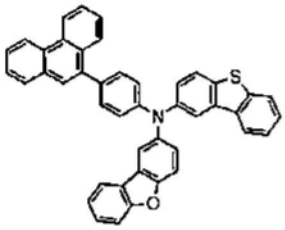
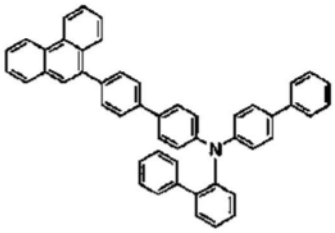
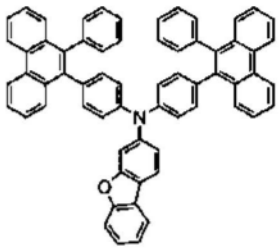
[0319]

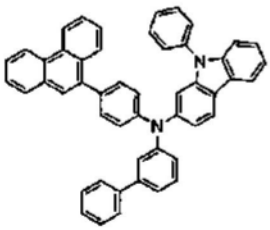
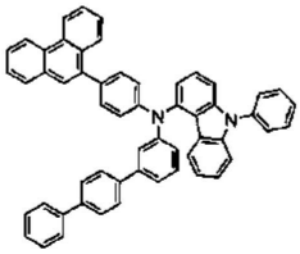
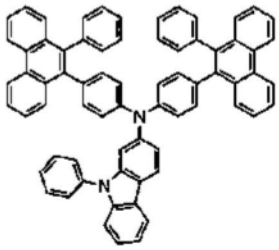










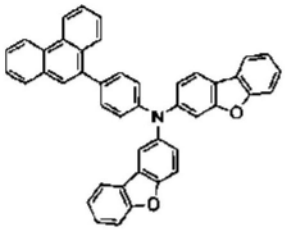
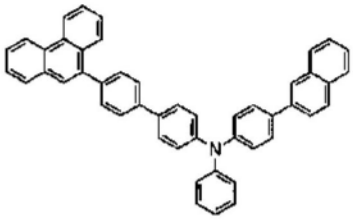
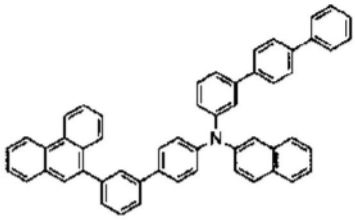
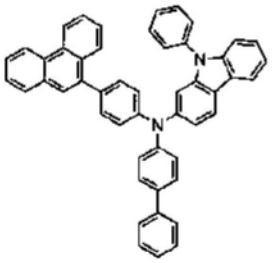
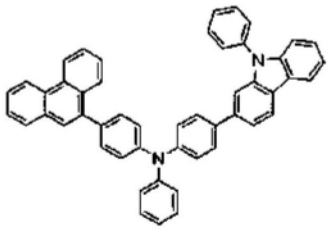
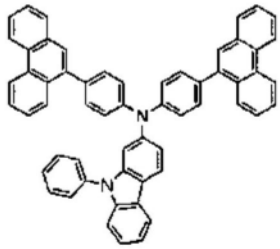
[0320]

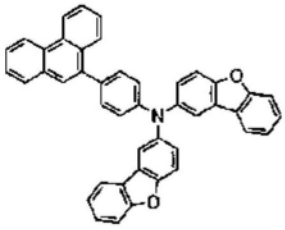
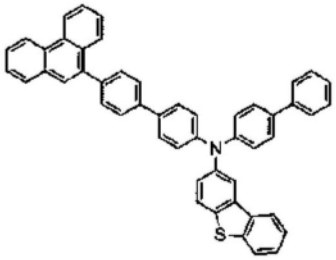
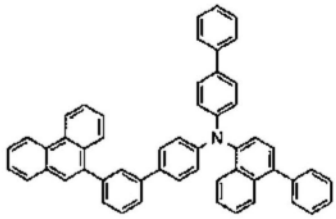
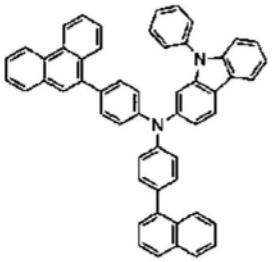
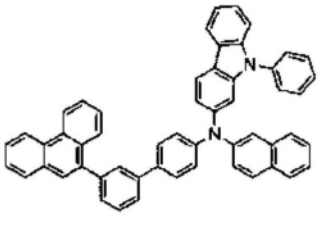
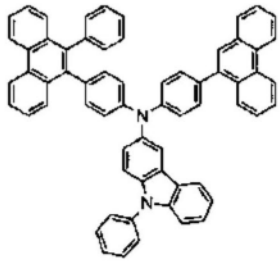




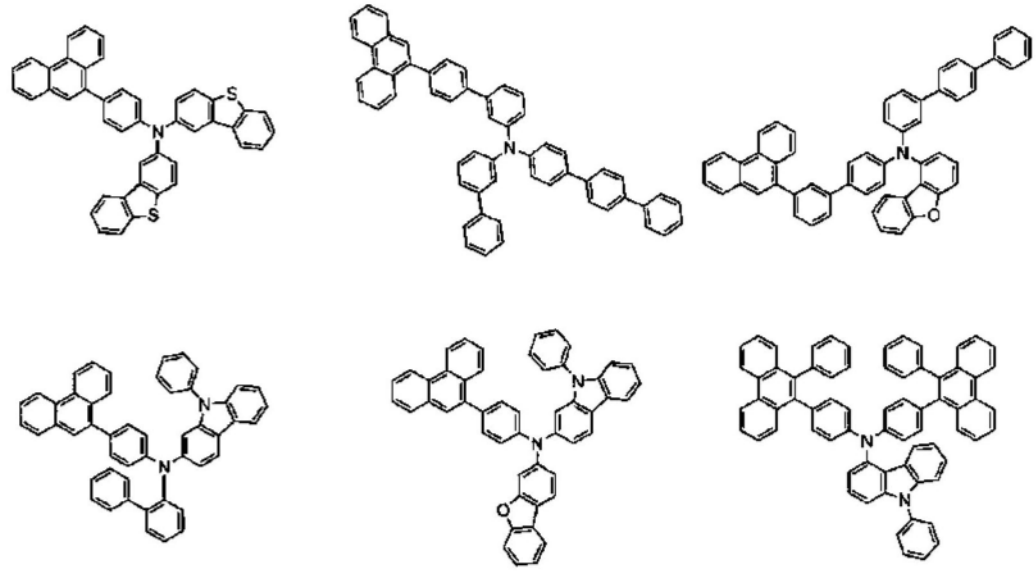
[0321]

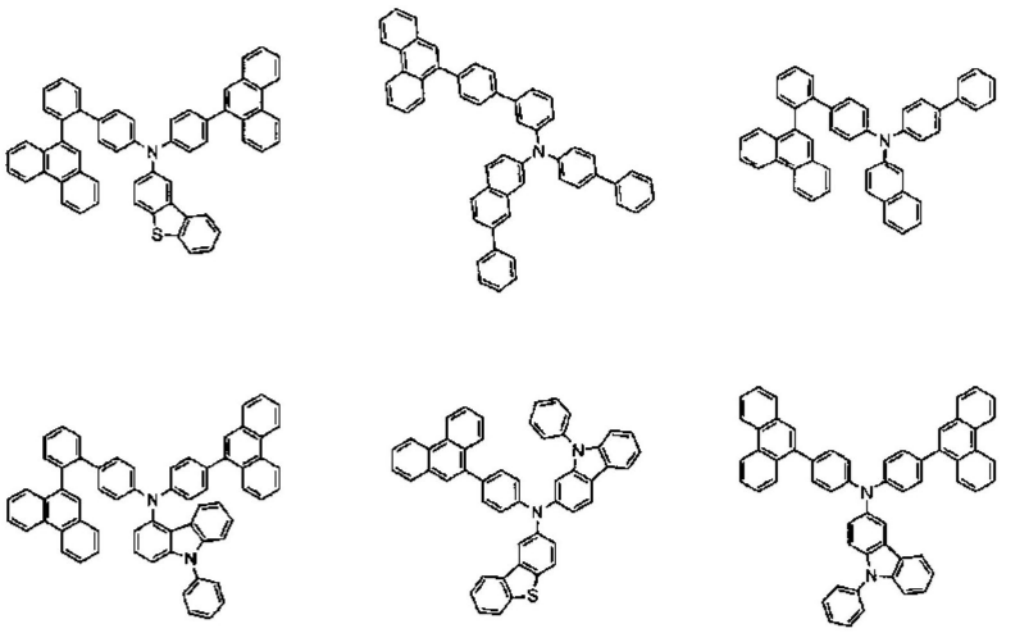







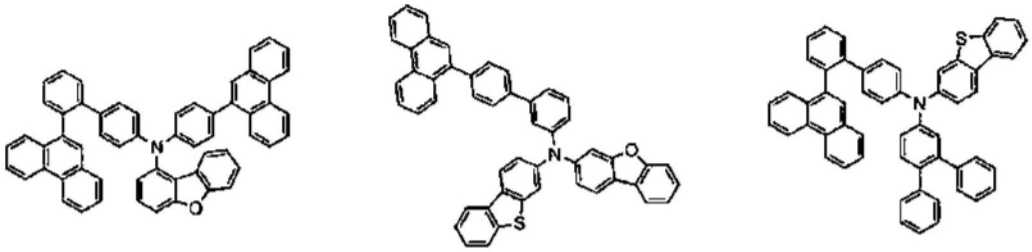
[0322]

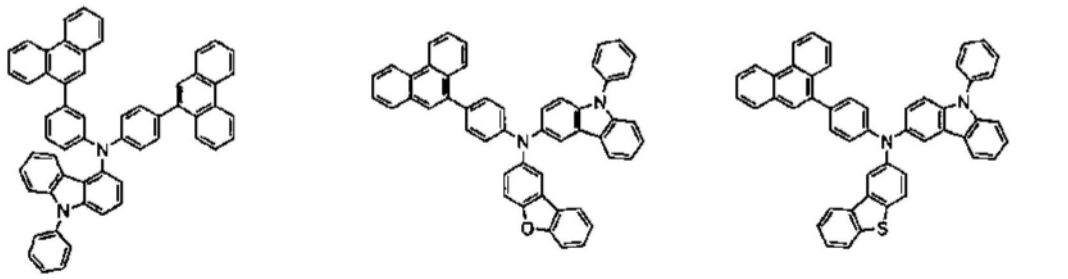


[0323]





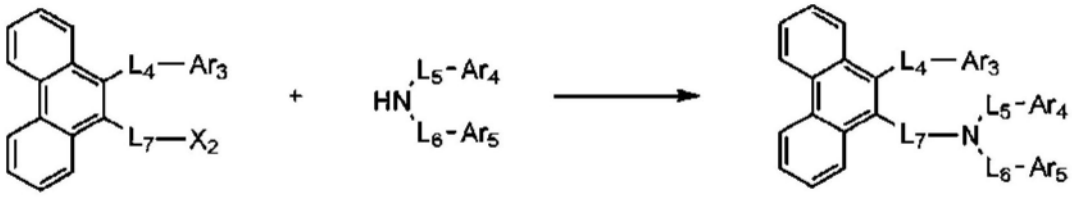
[0324]



。

[0325] 例如,可以根据如以下反应方案2中所示的制备方法来制备由化学式2表示的化合物,并且可以以类似的方式制备其他剩余的化合物。

[0326] [反应方案2]:

[0327] 

[0328] 其中在反应方案2中,Ar_3至Ar_5和L_4至L_7如化学式2中所限定,并且X_2为卤素,优选地X_2为氯或溴。

[0329] 反应方案2为胺取代反应,其优选地在钯催化剂和碱的存在下进行,并且用于胺取代反应的反应性基团可以如本领域已知的进行修改。以上制备方法可以在下文描述的制备例中进一步呈现。

[0330] 优选地,发光层中的由化学式1表示的化合物与由化学式2表示的化合物的重量比为10:90至90:10,更优选地为20:80至80:20、30:70至70:30或40:60至60:40。

[0331] 同时,除了主体之外,发光层还可以包含掺杂剂。掺杂剂材料没有特别限制,只要其是用于有机发光器件的材料即可。作为实例,可以提及芳族胺衍生物、苯乙烯胺化合物、硼配合物、荧蒽化合物、金属配合物等。芳族胺衍生物的具体实例包括经取代或未经取代的具有芳基氨基的稠合芳族环衍生物,其实例包括具有芳基氨基的芘、蒽、䓛和二茚并芘等。苯乙烯基胺化合物为其中经取代或未经取代的芳基胺中取代有至少一个芳基乙烯基的化合物,其中选自芳基、甲硅烷基、烷基、环烷基和芳基氨基中的一个或两个或更多个取代基为经取代或未经取代的。其具体实例包括苯乙烯基胺、苯乙烯基二胺、苯乙烯基三胺、苯乙烯基四胺等,但不限于此。此外,金属配合物的实例包括铱配合物、铂配合物等,但不限于此。

[0332] 空穴阻挡层

[0333] 空穴阻挡层是设置在电子传输层与发光层之间以防止注入阳极的空穴被转移至电子传输层而不在发光层中复合的层,其也可以被称为空穴抑制层或空穴阻止层。空穴阻挡层优选为具有大的电离能的材料。

[0334] 电子传输层

[0335] 如有必要,根据本公开的有机发光器件可以包括在发光层上的电子传输层。

[0336] 电子传输层是接收来自形成在阴极上的电子注入层和阴极的电子并将电子传输至发光层,并且抑制空穴从发光层转移的层,并且电子传输材料适当地为这样的材料:其可以很好地接收来自阴极的电子并将电子转移至发光层,并且具有大的电子迁移率。

[0337] 电子传输材料的具体实例包括:8-羟基喹啉的Al配合物、包含Alq_3的配合物、有机自由基化合物、羟基黄酮-金属配合物等,但不限于此。电子传输层可以与如根据常规技术使用的任何期望的阴极材料一起使用。特别地,阴极材料的合适的实例为具有小功函数,后接铝层或银层的典型材料。其具体实例包括铯、钡、钙、镱和钐,在每种情况下都后接铝层或银层。

[0338] 电子注入层

[0339] 如果必要,根据本公开内容的有机发光器件还可以包括在发光层上(或者当存在电子传输层时,在电子传输层上)的电子注入层。

[0340] 电子注入层是注入来自电极的电子的层,并且优选为这样的化合物:其具有传输电子的能力,具有注入来自阴极的电子的效应和优异的将电子注入到发光层或发光材料中的效应,防止从发光层产生的激子移动至空穴注入层,并且在形成薄膜的能力方面也优异。

[0341] 可以用作电子注入层的材料的具体实例包括芴酮、蒽醌二甲烷、联苯醌、噻喃二氧化物、噁唑、噁二唑、三唑、咪唑、苝四羧酸、亚芴基甲烷、蒽酮等及其衍生物、金属配合物化合物、含氮5元环衍生物,等等,但不限于此。

[0342] 金属配合物化合物的实例包括8-羟基喹啉锂、双(8-羟基喹啉)锌、双(8-羟基喹啉)铜、双(8-羟基喹啉)锰、三(8-羟基喹啉)铝、三(2-甲基-8-羟基喹啉)铝、三(8-羟基喹啉)镓、双(10-羟基苯并[h]喹啉)铍、双(10-羟基苯并[h]喹啉)锌、双(2-甲基-8-喹啉)氯镓、双(2-甲基-8-喹啉)(邻甲酚)镓、双(2-甲基-8-喹啉)(1-萘酚)铝、双(2-甲基-8-喹啉)(2-萘酚)镓等,但不限于此。

[0343] 同时,在本公开内容中,“电子注入和传输层”是同时起到电子注入层和电子传输层的作用的层,并且用作各层的材料可以单独使用或组合使用,但不限于此。

[0344] 有机发光器件

[0345] 图1示出了根据本公开内容的有机发光器件的结构。图1示出了包括基底1、阳极2、发光层3和阴极4的有机发光器件的一个实例。图2示出了包括基底1、阳极2、空穴注入层5、空穴传输层6、电子阻挡层7、发光层3、空穴阻挡层8、电子注入和传输层9以及阴极4的有机发光器件的一个实例。

[0346] 根据本公开内容的有机发光器件可以通过顺序地堆叠上述结构来制造。在这种情况下,有机发光器件可以通过如下来制造:通过使用PVD(物理气相沉积)法例如溅射法或电子束蒸镀法在基底上沉积金属、具有导电性的金属氧化物、或其合金以形成阳极,在阳极上形成上述各层,然后在其上沉积可以用作阴极的材料。除了这样的方法之外,有机发光器件还可以通过以上述配置的相反顺序在基底上从阴极材料至阳极材料顺序地沉积来制造(WO

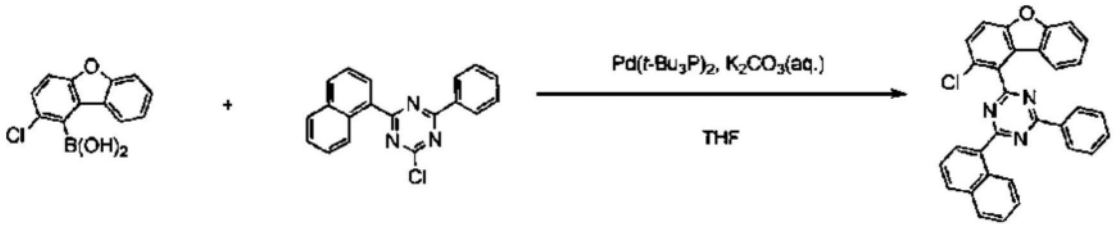
2003/012890)。此外,发光层可以通过使主体和掺杂剂经受真空沉积法和溶液涂覆法来形成。在本文中,溶液涂覆法意指旋涂、浸涂、刮涂、喷墨印刷、丝网印刷、喷洒法、辊涂等,但不限于此。

[0347] 另一方面,根据使用的材料,根据本公开内容的有机发光器件可以为前侧发射型、后侧发射型、或双侧发射型。

[0348] 在下文中,提出本公开内容的优选实施例以帮助理解本发明。然而,提出这些实施例仅出于举例说明目的,并且本公开内容的范围不限于此。

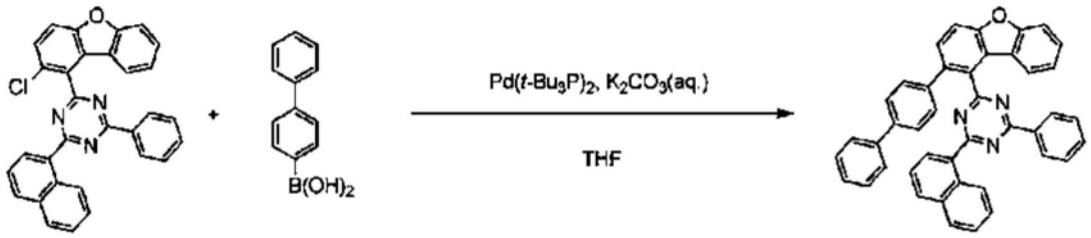
[0349] [制备例]

[0350] 制备例1-1:化合物1-1的制备

[0351] 

1-A Trz27 sub1-A-1

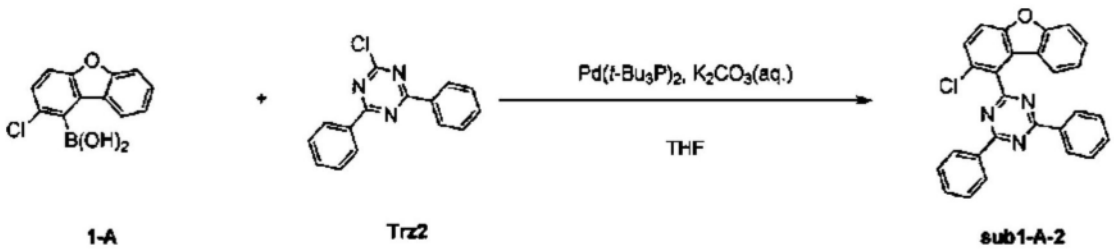
[0352] 在氮气气氛下将化合物1-A(15g,60.9mmol)和化合物Trz27(19.3g,60.9mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(16.8g,121.7mmol)溶解在50ml水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)(0.3g,0.6mmol)。在12小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到20.9g化合物sub1-A-1。(产率:71%,MS:$[M+H]^+$=484)

[0353] 

sub1-A-1 sub1 1-1

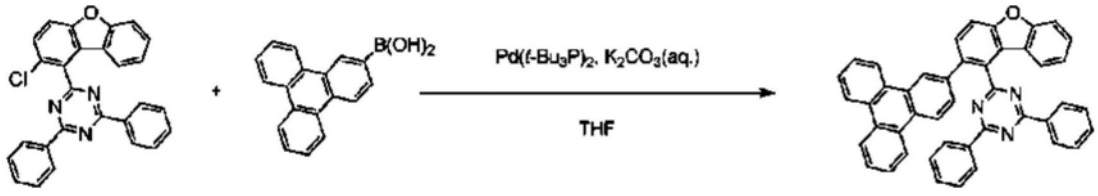
[0354] 在氮气气氛下将化合物sub1-A-1(15g,31mmol)和化合物sub1(6.1g,31mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(8.6g,62mmol)溶解在26ml水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)(0.2g,0.3mmol)。在10小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到12.3g化合物1-1。(产率:66%,MS:$[M+H]^+$=602)

[0355] 制备例1-2:化合物1-2的制备

[0356] 

1-A Trz2 sub1-A-2

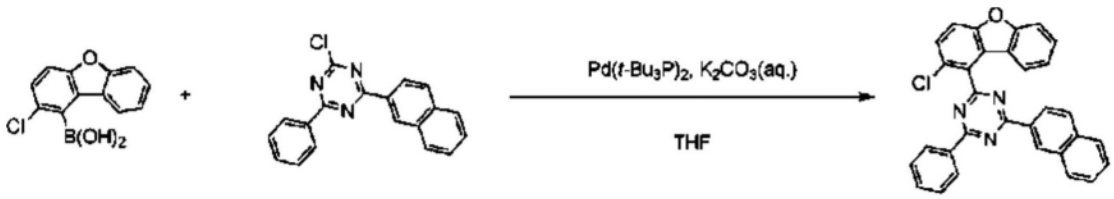
[0357] 在氮气气氛下将化合物1-A(15g,60.9mmol)和化合物Trz2(16.3g,60.9mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(25.2g,182.6mmol)溶解在76ml水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)(0.3g,0.6mmol)。在10小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到19.5g化合物sub1-A-2。(产率:74%,MS:$[M+H]^+$=434)

[0358] 

sub1-A-2 sub2 1-2

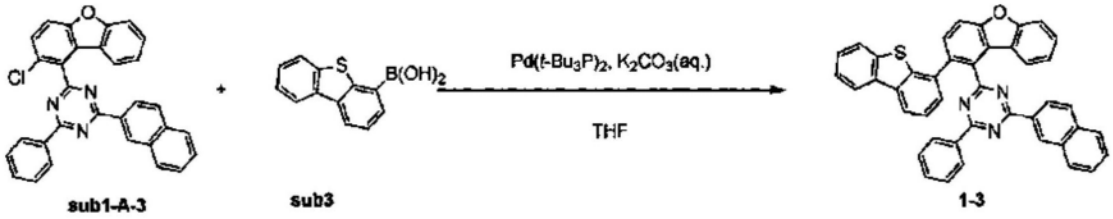
[0359] 在氮气气氛下将化合物sub1-A-2(15g,34.6mmol)和化合物sub2(9.4g,34.6mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(9.6g,69.1mmol)溶解在29ml水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)(0.2g,0.3mmol)。在8小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到14.3g化合物1-2。(产率:66%,MS:$[M+H]^+$=626)

[0360] 制备例1-3:化合物1-3的制备

[0361] 

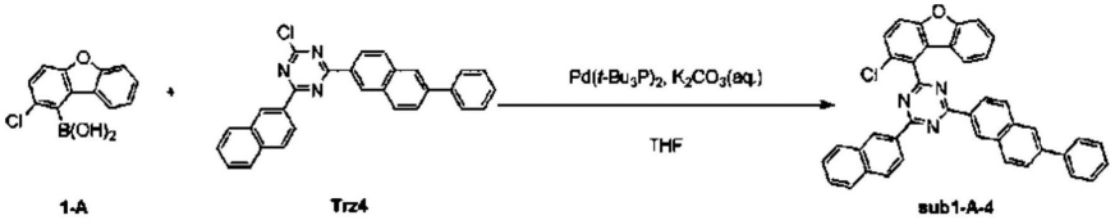
1-A Trz3 sub1-A-3

[0362] 在氮气气氛下将化合物1-A(15g,60.9mmol)和化合物Trz3(19.3g,60.9mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(25.2g,182.6mmol)溶解在76ml水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)(0.3g,0.6mmol)。在9小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到23.2g化合物sub1-A-3。(产率:79%,MS:$[M+H]^+$=484)

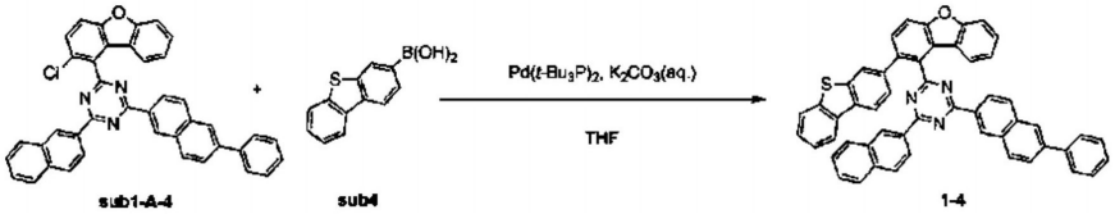
[0363] 

[0364] 在氮气气氛下将化合物sub1-A-3(15g,31mmol)和化合物sub3(7.1g,31mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(8.6g,62mmol)溶解在26ml水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)(0.2g,0.3mmol)。在12小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到12.9g化合物1-3。(产率:66%,MS:[M+H]$^+$=632)

[0365] 制备例1-4:化合物1-4的制备

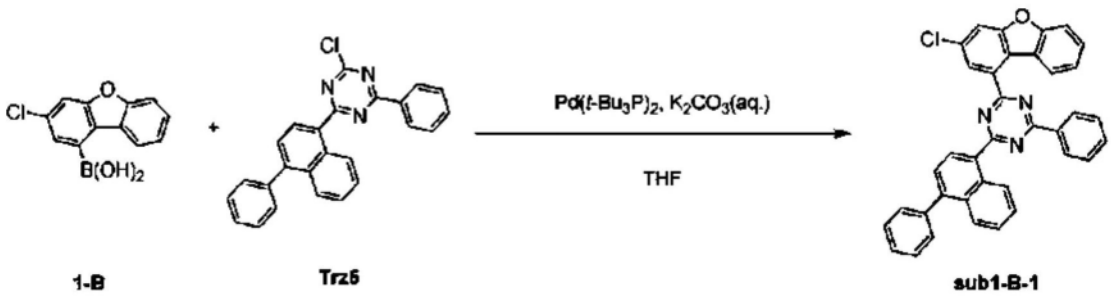
[0366] 

[0367] 在氮气气氛下将化合物1-A(15g,60.9mmol)和化合物Trz4(27g,60.9mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(25.2g,182.6mmol)溶解在76ml水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)(0.3g,0.6mmol)。在9小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到26g化合物sub1-A-4。(产率:70%,MS:[M+H]$^+$=610)

[0368] 

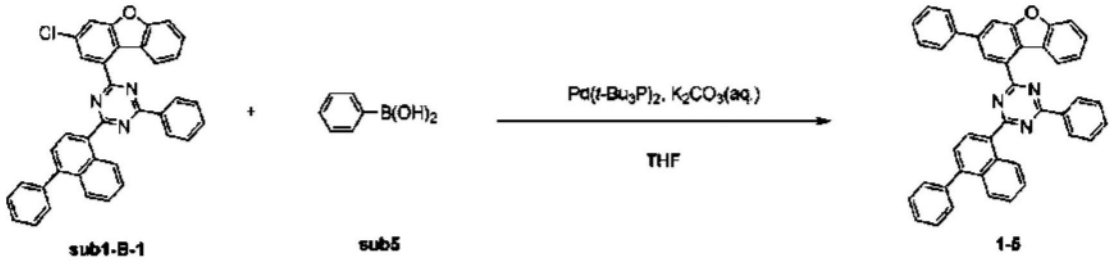
[0369] 在氮气气氛下将化合物sub1-A-4(15g,24.6mmol)和化合物sub4(5.6g,24.6mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(6.8g,49.2mmol)溶解在20ml水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)(0.1g,0.2mmol)。在11小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到11.2g化合物1-4。(产率:60%,MS:[M+H]$^+$=758)

[0370] 制备例1-5:化合物1-5的制备

[0371] 

1-B Trz5 sub1-B-1

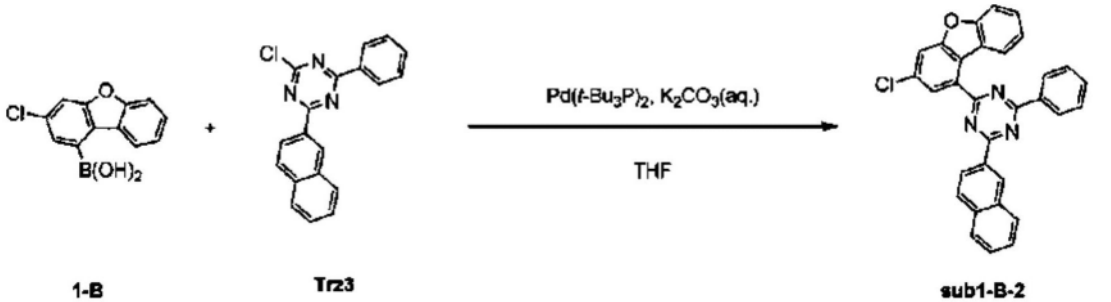
[0372] 在氮气气氛下将化合物1-B(15g,60.9mmol)和化合物Trz5(24g,60.9mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(25.2g,182.6mmol)溶解在76ml水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)(0.3g,0.6mmol)。在10小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到26.2g化合物sub1-B-1。(产率:77%,MS:$[M+H]^+$=560)

[0373] 

sub1-B-1 sub5 1-5

[0374] 在氮气气氛下将化合物sub1-B-1(15g,26.8mmol)和化合物sub5(3.3g,26.8mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(7.4g,53.6mmol)溶解在22ml水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)(0.1g,0.3mmol)。在10小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到12.9g化合物1-5。(产率:80%,MS:$[M+H]^+$=602)

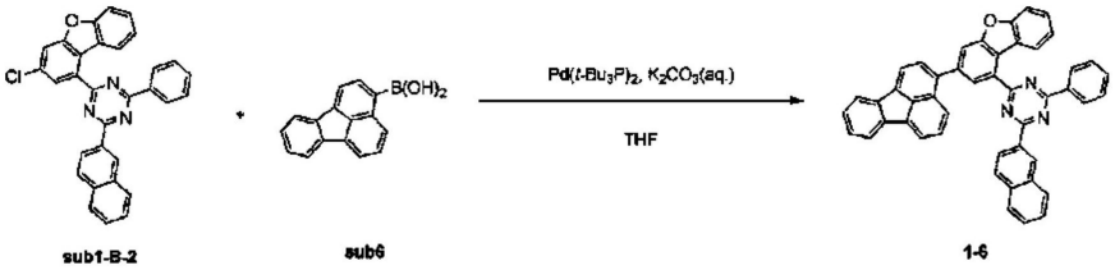
[0375] 制备例1-6:化合物1-6的制备

[0376] 

1-B Trz3 sub1-B-2

[0377] 在氮气气氛下将化合物1-B(15g,60.9mmol)和化合物Trz3(19.3g,60.9mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(25.2g,182.6mmol)溶解在76ml水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)(0.3g,0.6mmol)。在11小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水

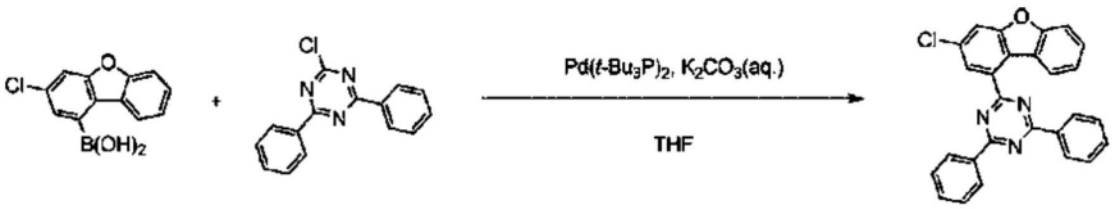
硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到18.2g化合物sub1-B-2。(产率:62%,MS:[M+H]$^+$=484)

[0378] 

sub1-B-2 sub6 1-6

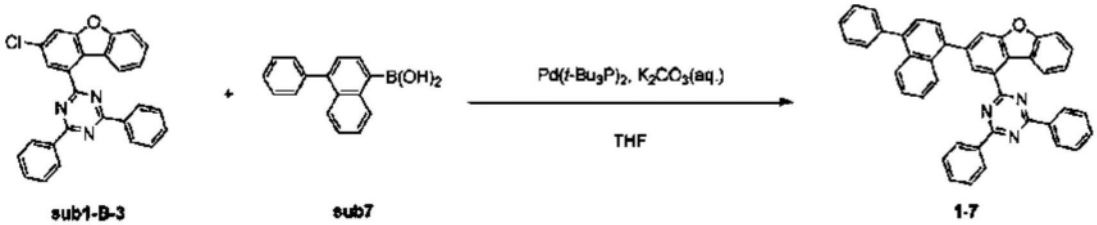
[0379] 在氮气气氛下将化合物sub1-B-2(15g,31mmol)和化合物sub6(7.6g,31mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(8.6g,62mmol)溶解在26ml水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)(0.2g,0.3mmol)。在11小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到15.3g化合物1-6。(产率:76%,MS:[M+H]$^+$=650)

[0380] 制备例1-7:化合物1-7的制备

[0381] 

1-B Trz2 sub1-B-3

[0382] 在氮气气氛下将化合物1-B(15g,60.9mmol)和化合物Trz2(16.3g,60.9mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(25.2g,182.6mmol)溶解在76ml水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)(0.3g,0.6mmol)。在12小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到20.8g化合物sub1-B-3。(产率:79%,MS:[M+H]$^+$=434)

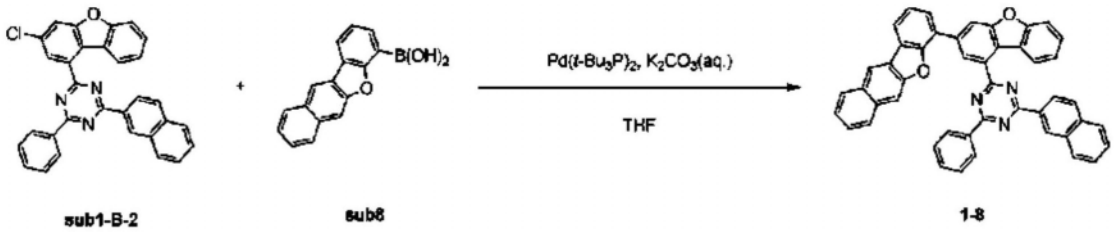
[0383] 

sub1-B-3 sub7 1-7

[0384] 在氮气气氛下将化合物sub1-B-3(15g,34.6mmol)和化合物sub7(8.6g,34.6mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(9.6g,69.1mmol)溶解在29ml水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)(0.2g,0.3mmol)。在8小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫

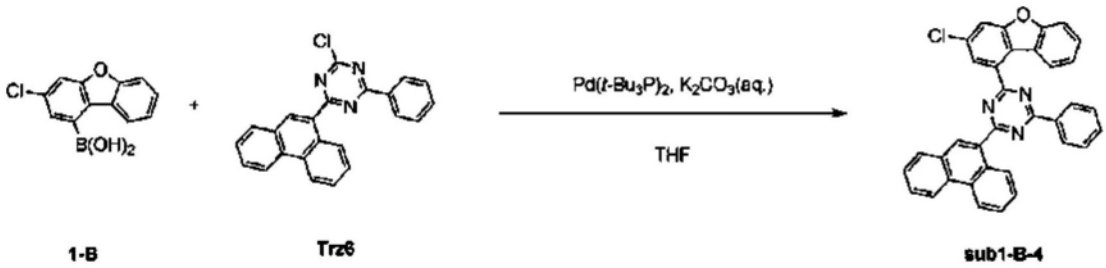
酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到15.4g化合物1-7。(产率:74%,MS:[M+H]$^+$=602)

[0385] 制备例1-8:化合物1-8的制备

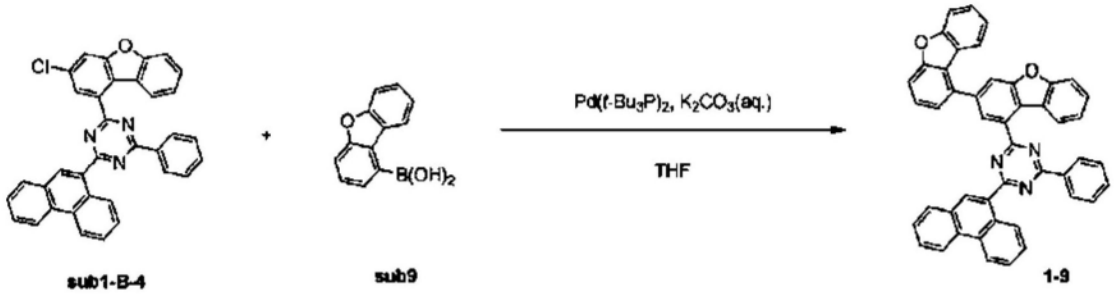
[0386] 

[0387] 在氮气气氛下将化合物sub1-B-2(15g,31mmol)和化合物sub8(8.1g,31mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(8.6g,62mmol)溶解在26ml水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)(0.2g,0.3mmol)。在10小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到15.5g化合物1-8。(产率:75%,MS:[M+H]$^+$=666)

[0388] 制备例1-9:化合物1-9的制备

[0389] 

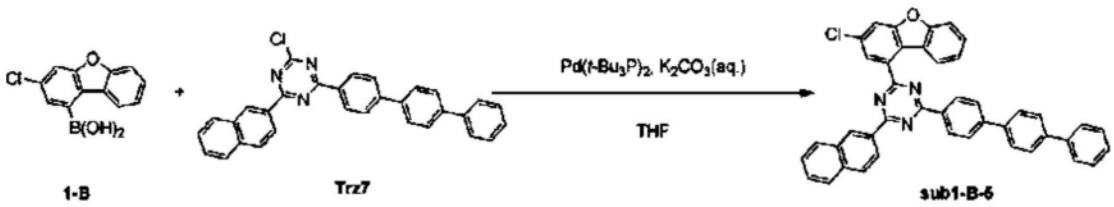
[0390] 在氮气气氛下将化合物1-B(15g,60.9mmol)和化合物Trz6(22.4g,60.9mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(25.2g,182.6mmol)溶解在76ml水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)(0.3g,0.6mmol)。在9小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到23.7g化合物sub1-B-4。(产率:73%,MS:[M+H]$^+$=534)

[0391] 

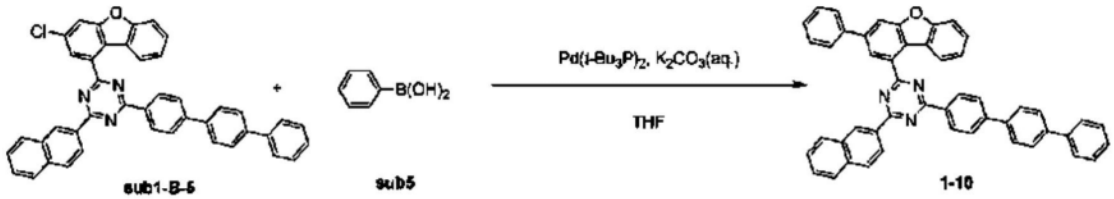
[0392] 在氮气气氛下将化合物sub1-B-4(15g,28.1mmol)和化合物sub9(6g,28.1mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(7.8g,56.2mmol)溶解在23ml水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)(0.1g,

0.3mmol)。在8小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到11.6g化合物1-9。(产率:62%,MS:$[M+H]^+$=666)

[0393] 制备例1-10:化合物1-10的制备

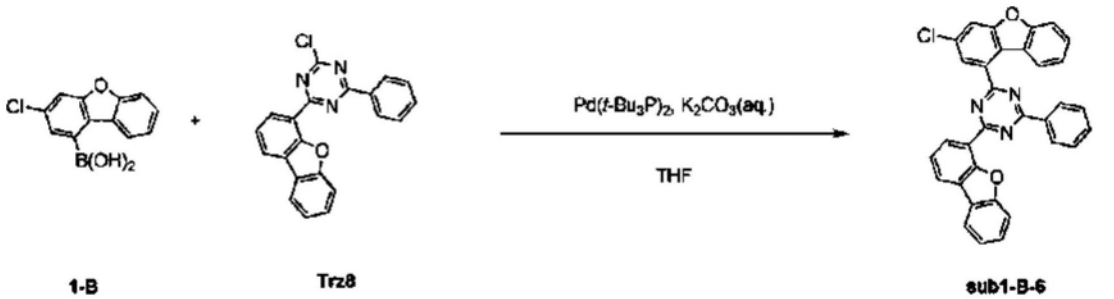
[0394] 

[0395] 在氮气气氛下将化合物1-B(15g,60.9mmol)和化合物Trz7(28.6g,60.9mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(25.2g,182.6mmol)溶解在76ml水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)(0.3g,0.6mmol)。在10小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到28.6g化合物sub1-B-5。(产率:74%,MS:$[M+H]^+$=636)

[0396] 

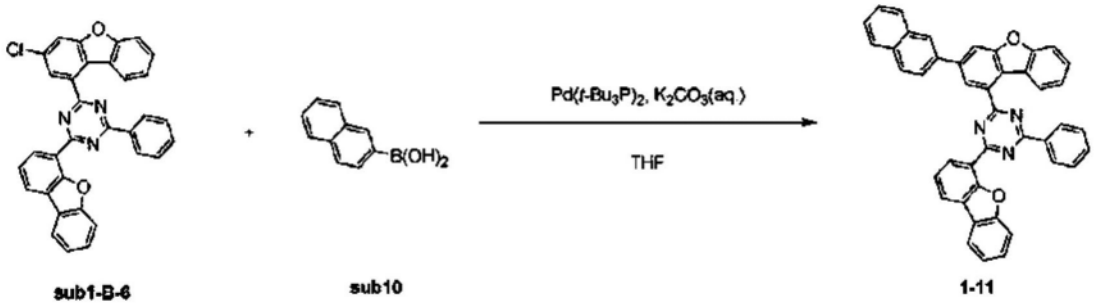
[0397] 在氮气气氛下将化合物sub1-B-5(15g,23.6mmol)和化合物sub5(2.9g,23.6mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(6.5g,47.2mmol)溶解在20ml水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)(0.1g,0.2mmol)。在11小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到10.4g化合物1-10。(产率:65%,MS:$[M+H]^+$=678)

[0398] 制备例1-11:化合物1-11的制备

[0399] 

[0400] 在氮气气氛下将化合物1-B(15g,60.9mmol)和化合物Trz8(21.8g,60.9mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(25.2g,182.6mmol)溶解在76ml水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)(0.3g,

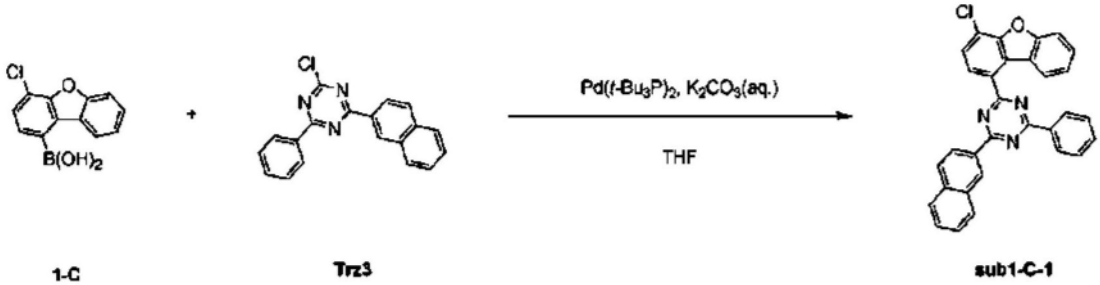
0.6mmol)。在8小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到20.1g化合物sub1-B-6。(产率:63%,MS:$[M+H]^+$=524)

[0401] 

sub1-B-6 sub10 1-11

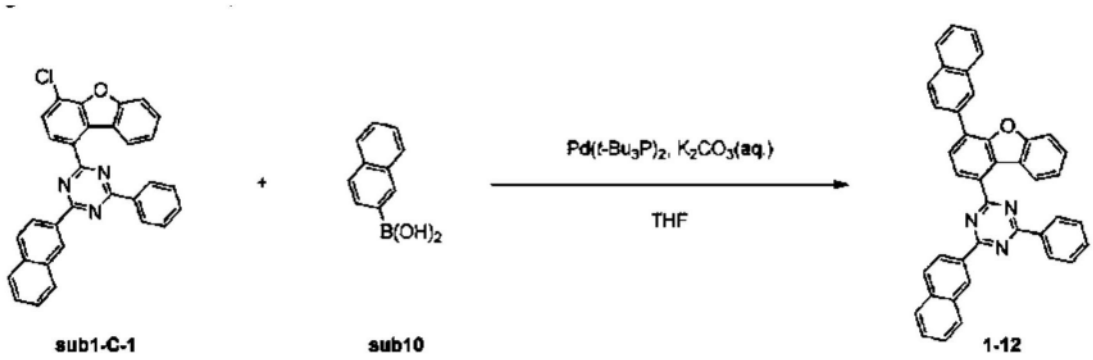
[0402] 在氮气气氛下将化合物sub1-B-6(15g,28.6mmol)和化合物sub10(4.9g,28.6mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(7.9g,57.3mmol)溶解在24ml水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)(0.1g,0.3mmol)。在8小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到11.4g化合物1-11。(产率:65%,MS:$[M+H]^+$=616)

[0403] 制备例1-12:化合物1-12的制备

[0404] 

1-C Trz3 sub1-C-1

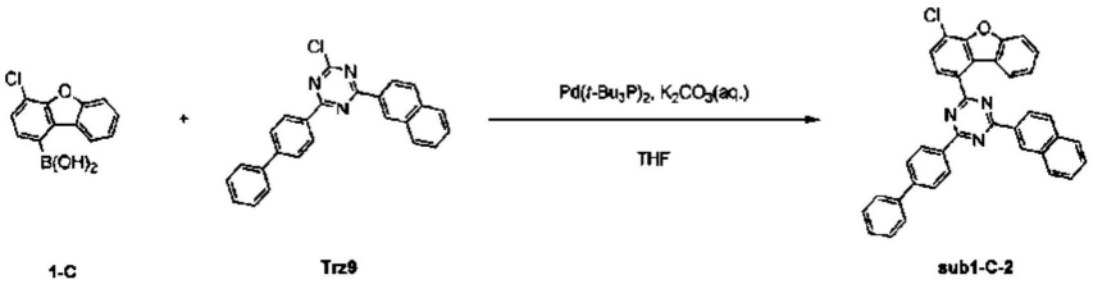
[0405] 在氮气气氛下将化合物1-C(15g,60.9mmol)和化合物Trz3(19.3g,60.9mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(25.2g,182.6mmol)溶解在76ml水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)(0.3g,0.6mmol)。在11小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到17.6g化合物sub1-C-1。(产率:60%,MS:$[M+H]^+$=484)

[0406] 

sub1-C-1 sub10 1-12

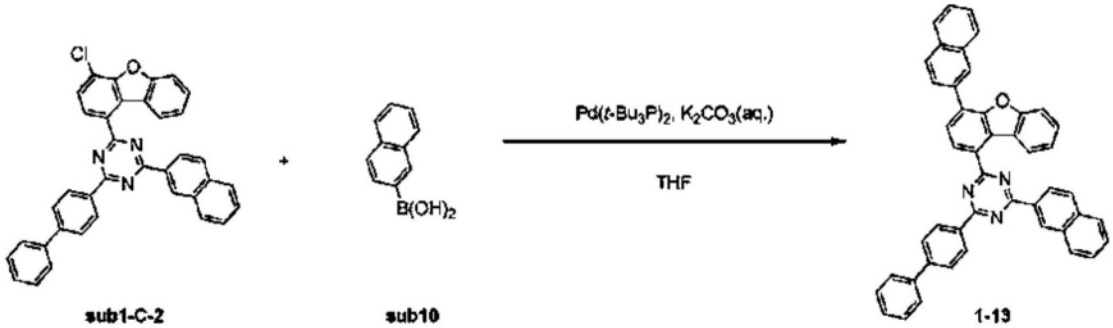
[0407] 在氮气气氛下将化合物sub1-C-1(15g,31mmol)和化合物sub10(5.3g,31mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(8.6g,62mmol)溶解在26ml水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)(0.2g,0.3mmol)。在11小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到12.8g化合物1-12。(产率:72%,MS:$[M+H]^+$=576)

[0408] 制备例1-13:化合物1-13的制备

[0409] 

1-C Trz9 sub1-C-2

[0410] 在氮气气氛下将化合物1-C(15g,60.9mmol)和化合物Trz9(24g,60.9mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(25.2g,182.6mmol)溶解在76ml水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)(0.3g,0.6mmol)。在12小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到23.5g化合物sub1-C-2。(产率:69%,MS:$[M+H]^+$=560)

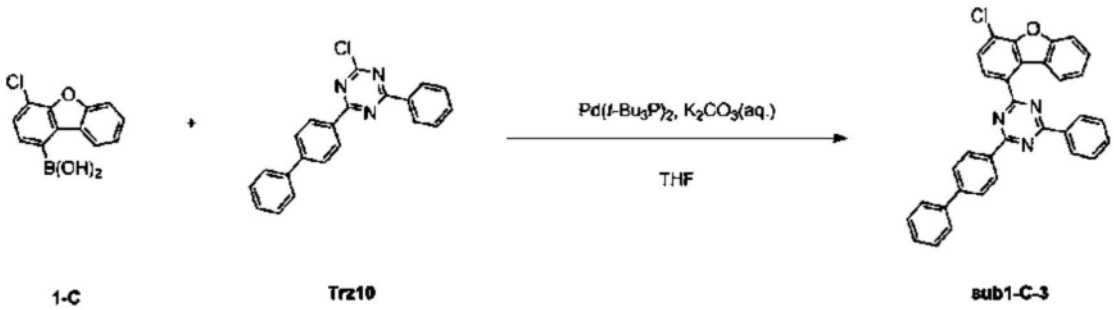
[0411] 

sub1-C-2 sub10 1-13

[0412] 在氮气气氛下将化合物sub1-C-2(15g,26.8mmol)和化合物sub10(4.6g,26.8mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(7.4g,53.6mmol)溶解在22ml水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)

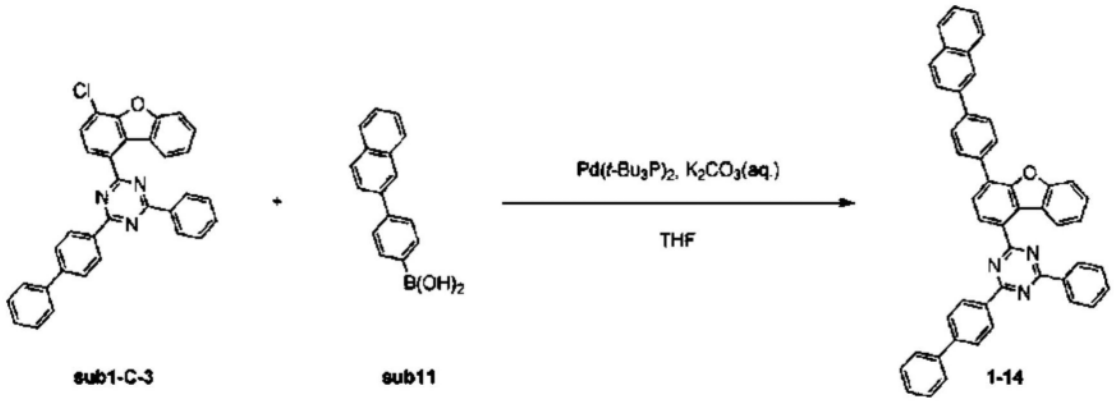
(0.1g,0.3mmol)。在11小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到14g化合物1-13。(产率:80%,MS:$[M+H]^+$=652)

[0413] 制备例1-14:化合物1-14的制备

[0414] 

1-C Trz10 sub1-C-3

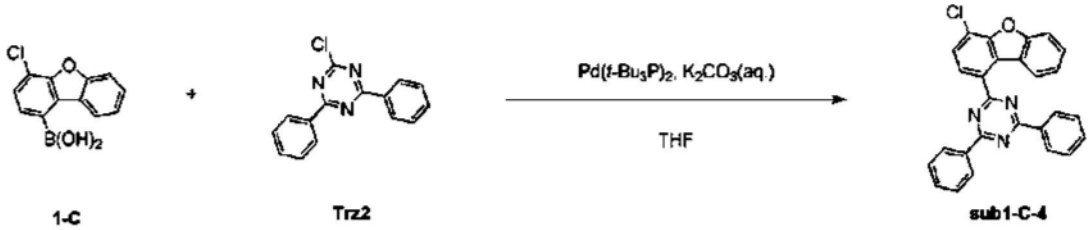
[0415] 在氮气气氛下将化合物1-C(15g,60.9mmol)和化合物Trz10(20.9g,60.9mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(25.2g,182.6mmol)溶解在76ml水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)(0.3g,0.6mmol)。在11小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到20.5g化合物sub1-C-3。(产率:66%,MS:$[M+H]^+$=510)

[0416] 

sub1-C-3 sub11 1-14

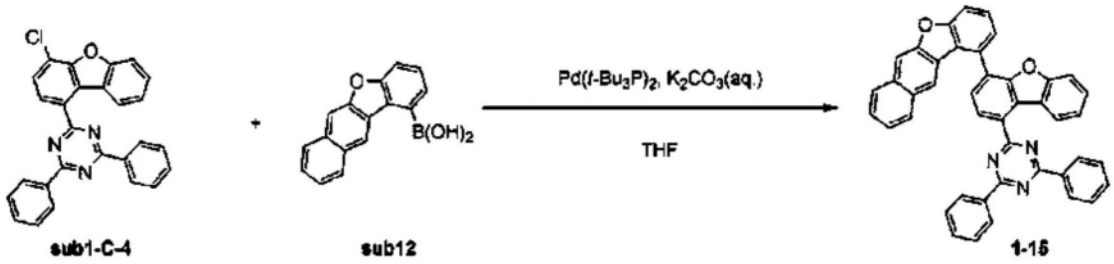
[0417] 在氮气气氛下将化合物sub1-C-3(15g,29.4mmol)和化合物sub11(7.3g,29.4mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(8.1g,58.8mmol)溶解在24ml水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)(0.2g,0.3mmol)。在10小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到15.3g化合物1-14。(产率:77%,MS:$[M+H]^+$=678)

[0418] 制备例1-15:化合物1-15的制备

[0419] 

1-C Trz2 sub1-C-4

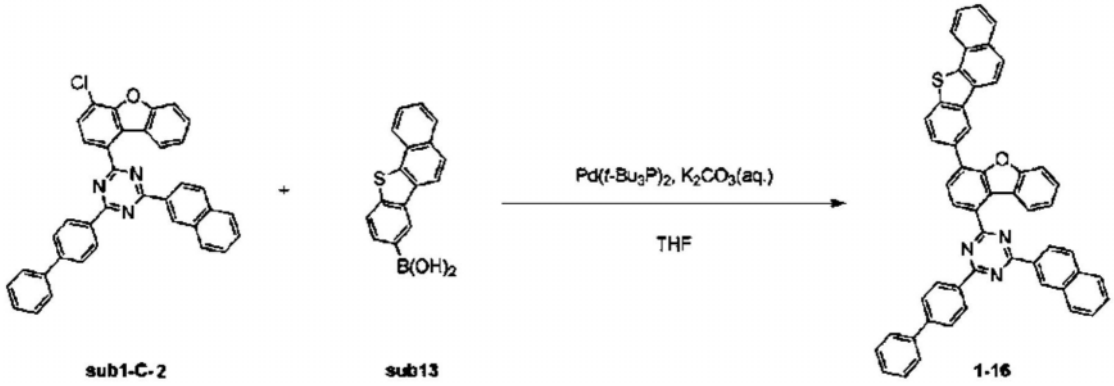
[0420] 在氮气气氛下将化合物1-C(15g,60.9mmol)和化合物Trz2(16.3g,60.9mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(25.2g,182.6mmol)溶解在76ml水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)(0.3g,0.6mmol)。在9小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到18.7g化合物sub1-C-4。(产率:71%,MS:$[M+H]^+$=434)

[0421] 

sub1-C-4 sub12 1-15

[0422] 在氮气气氛下将化合物sub1-C-4(15g,37.1mmol)和化合物sub12(9.7g,37.1mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(10.3g,74.3mmol)溶解在31ml水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)(0.2g,0.4mmol)。在10小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到14.6g化合物1-15。(产率:64%,MS:$[M+H]^+$=616)

[0423] 制备例1-16:化合物1-16的制备

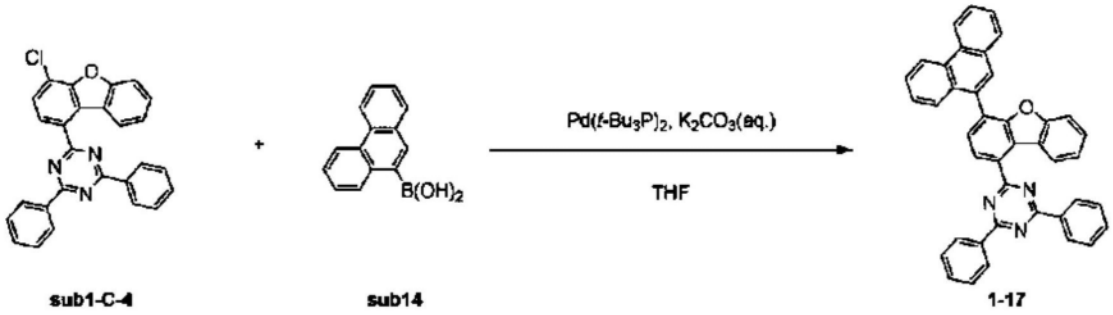
[0424] 

sub1-C-2 sub13 1-16

[0425] 在氮气气氛下将化合物sub1-C-2(15g,26.8mmol)和化合物sub13(7.4g,26.8mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(7.4g,53.6mmol)溶解在22ml水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)(0.1g,0.3mmol)。在9小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加

无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到16.2g化合物1-16。(产率:80%,MS:[M+H]$^+$=758)

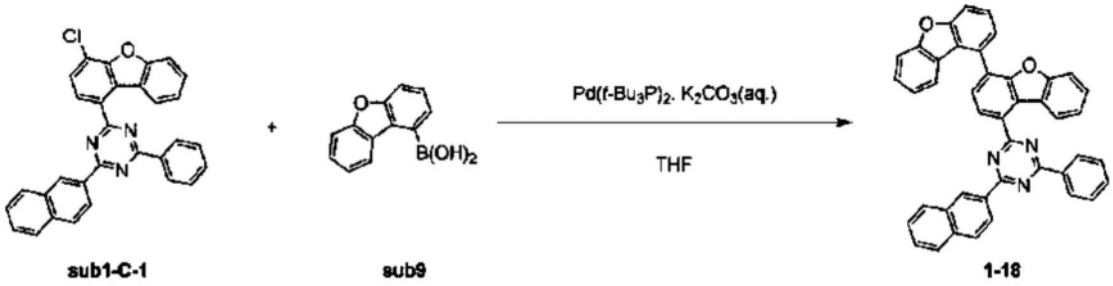
[0426] 制备例1-17:化合物1-17的制备

[0427] 

sub1-C-4 sub14 1-17

[0428] 在氮气气氛下将化合物sub1-C-4(15g,34.6mmol)和化合物sub14(7.7g,34.6mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(9.6g,69.1mmol)溶解在29ml水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)(0.2g,0.3mmol)。在8小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到12.3g化合物1-17。(产率:62%,MS:[M+H]$^+$=576)

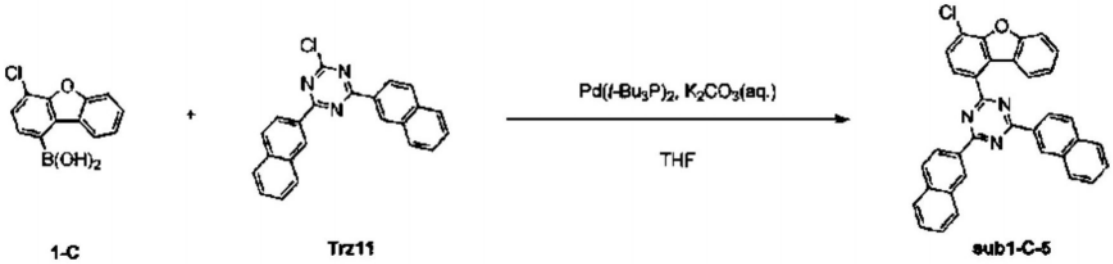
[0429] 制备例1-18:化合物1-18的制备

[0430] 

sub1-C-1 sub9 1-18

[0431] 在氮气气氛下将化合物sub1-C-1(15g,31mmol)和化合物sub9(6.6g,31mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(8.6g,62mmol)溶解在26ml水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)(0.2g,0.3mmol)。在9小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到12g化合物1-18。(产率:63%,MS:[M+H]$^+$=616)

[0432] 制备例1-19:化合物1-19的制备

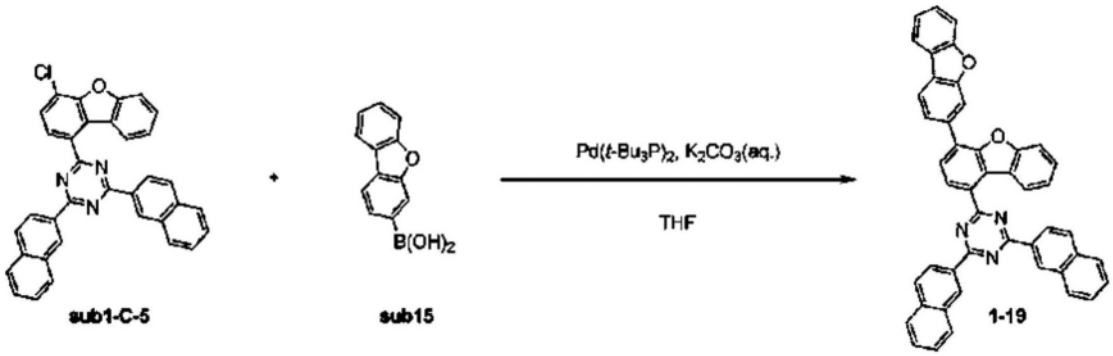
[0433] 

1-C Trz11 sub1-C-5

[0434] 在氮气气氛下将化合物1-C(15g,60.9mmol)和化合物Trz11(22.4g,60.9mmol)添

加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(25.2g,182.6mmol)溶解在76ml水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)(0.3g,0.6mmol)。在11小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到22.4g化合物sub1-C-5。(产率:69%,MS:[M+H]$^+$=534)

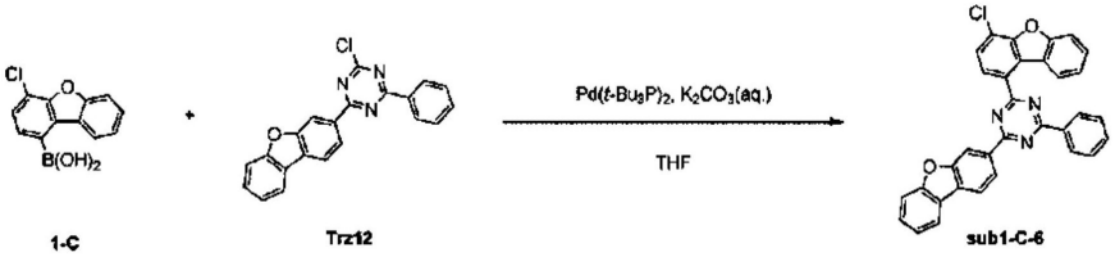
[0435]



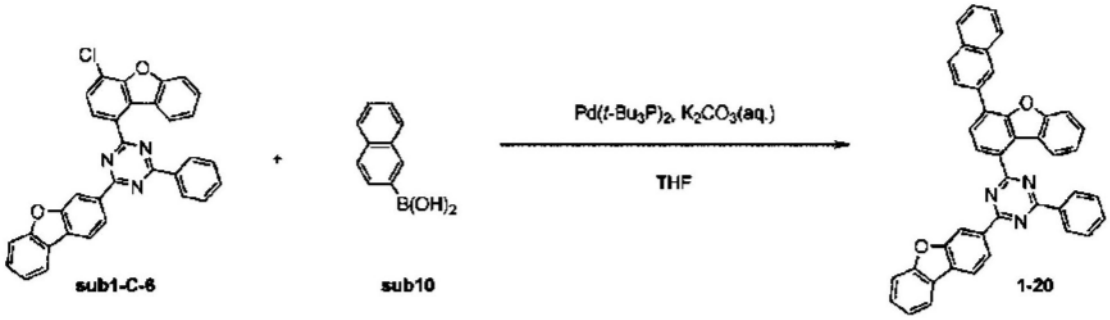
[0436] 在氮气气氛下将化合物sub1-C-5(15g,28.1mmol)和化合物sub15(6g,28.1mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(7.8g,56.2mmol)溶解在23ml水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)(0.1g,0.3mmol)。在10小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到13.3g化合物1-19。(产率:71%,MS:[M+H]$^+$=666)

[0437] 制备例1-20:化合物1-20的制备

[0438]

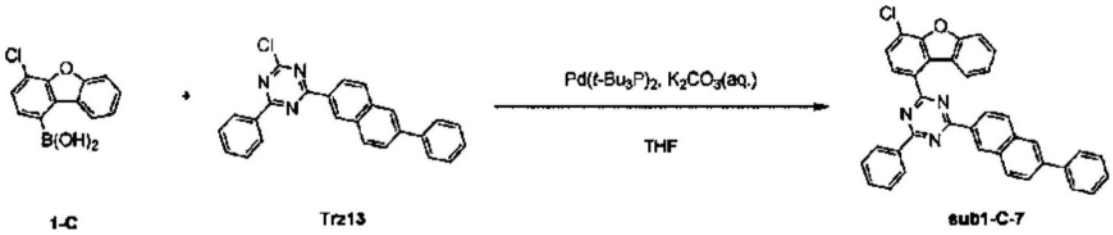


[0439] 在氮气气氛下将化合物1-C(15g,60.9mmol)和化合物Trz12(21.8g,60.9mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(25.2g,182.6mmol)溶解在76ml水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)(0.3g,0.6mmol)。在10小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到21g化合物sub1-C-6。(产率:66%,MS:[M+H]$^+$=524)

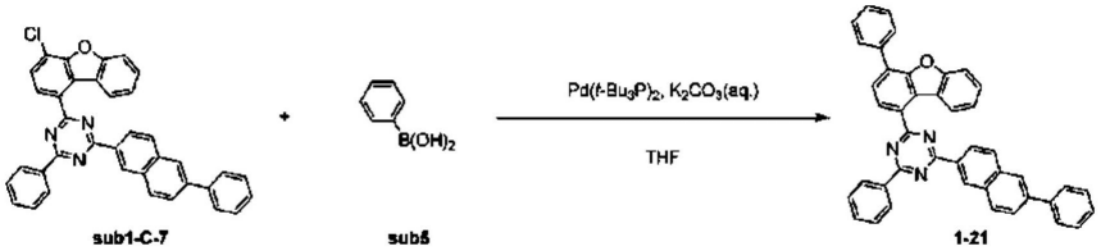
[0440] 

[0441] 在氮气气氛下将化合物sub1-C-6(15g,28.6mmol)和化合物sub10(4.9g,28.6mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(11.9g,85.9mmol)溶解在36ml水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)(0.1g,0.3mmol)。在10小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到12.3g化合物1-20。(产率:70%,MS:$[M+H]^+$=616)

[0442] 制备例1-21:化合物1-21的制备

[0443] 

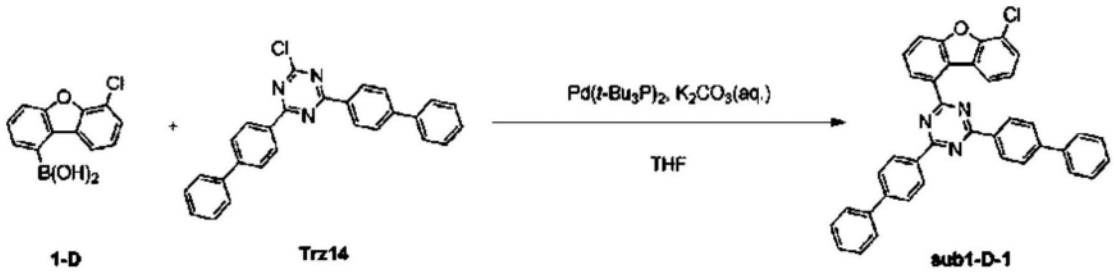
[0444] 在氮气气氛下将化合物1-C(15g,60.9mmol)和化合物Trz13(24g,60.9mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(25.2g,182.6mmol)溶解在76ml水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)(0.3g,0.6mmol)。在12小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到26.2g化合物sub1-C-7。(产率:77%,MS:$[M+H]^+$=560)

[0445] 

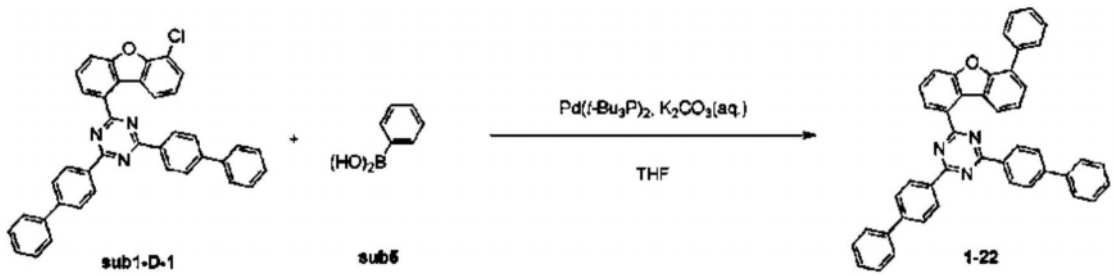
[0446] 在氮气气氛下将化合物sub1-C-7(15g,26.8mmol)和化合物sub5(3.3g,26.8mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(11.1g,80.3mmol)溶解在33ml水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)(0.1g,0.3mmol)。在8小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯

化以得到10.5g化合物1-21。(产率:65%,MS:[M+H]$^+$=602)

[0447] 制备例1-22:化合物1-22的制备

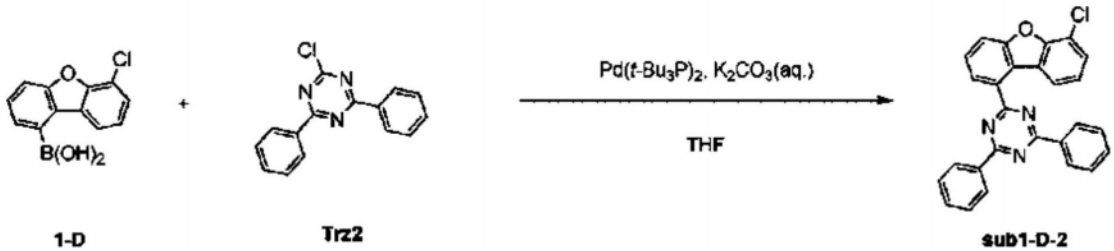
[0448] 

[0449] 在氮气气氛下将化合物1-D(15g,60.9mmol)和化合物Trz14(19.3g,60.9mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(25.2g,182.6mmol)溶解在76ml水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)(0.3g,0.6mmol)。在11小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到23.9g化合物sub1-D-1。(产率:67%,MS:[M+H]$^+$=586)

[0450] 

[0451] 在氮气气氛下将化合物sub1-D-1(15g,25.6mmol)和化合物sub5(3.1g,25.6mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(10.6g,76.8mmol)溶解在32ml水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)(0.1g,0.3mmol)。在8小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到10.3g化合物1-22。(产率:64%,MS:[M+H]$^+$=628)

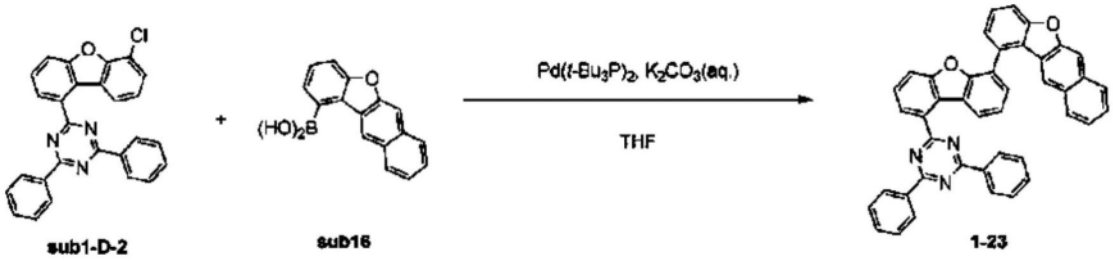
[0452] 制备例1-23:化合物1-23的制备

[0453] 

[0454] 在氮气气氛下将化合物1-D(15g,60.9mmol)和化合物Trz2(16.3g,60.9mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(25.2g,182.6mmol)溶解在76ml水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)(0.3g,0.6mmol)。在10小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将

有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到20g化合物sub1-D-2。(产率:76%,MS:[M+H]$^+$=434)

[0455]

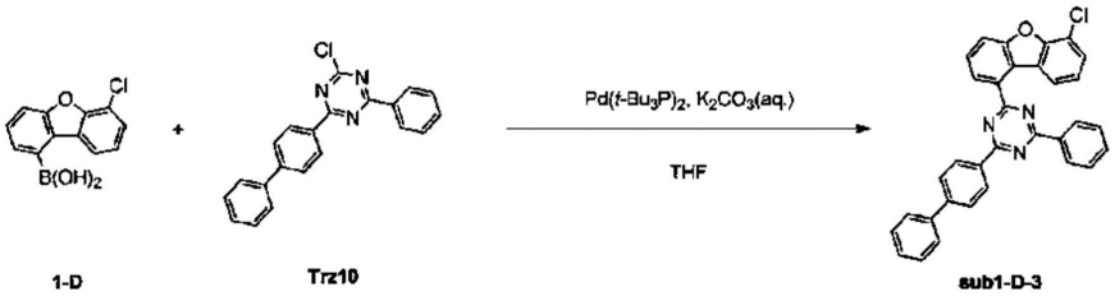


sub1-D-2 sub16 1-23

[0456] 在氮气气氛下将化合物sub1-D-2(15g,34.6mmol)和化合物sub16(9.1g,34.6mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(14.3g,103.7mmol)溶解在43ml水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)(0.2g,0.3mmol)。在9小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到14g化合物1-23。(产率:66%,MS:[M+H]$^+$=616)

[0457] 制备例1-24:化合物1-24的制备

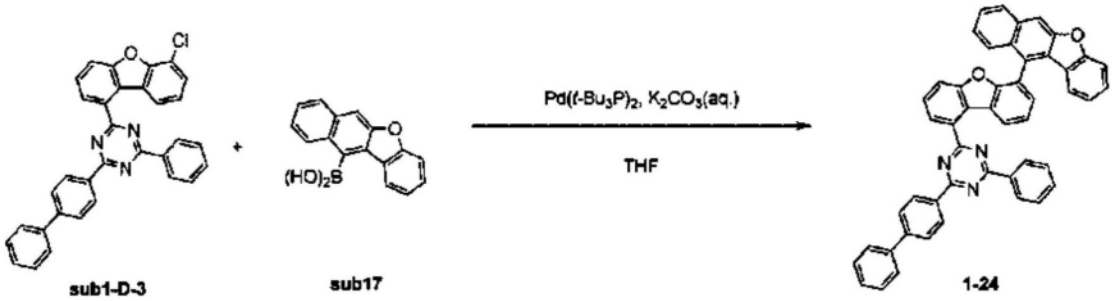
[0458]



1-D Trz10 sub1-D-3

[0459] 在氮气气氛下将化合物1-D(15g,60.9mmol)和化合物Trz10(20.9g,60.9mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(25.2g,182.6mmol)溶解在76ml水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)(0.3g,0.6mmol)。在12小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到20.8g化合物sub1-D-3。(产率:67%,MS:[M+H]$^+$=510)

[0460]

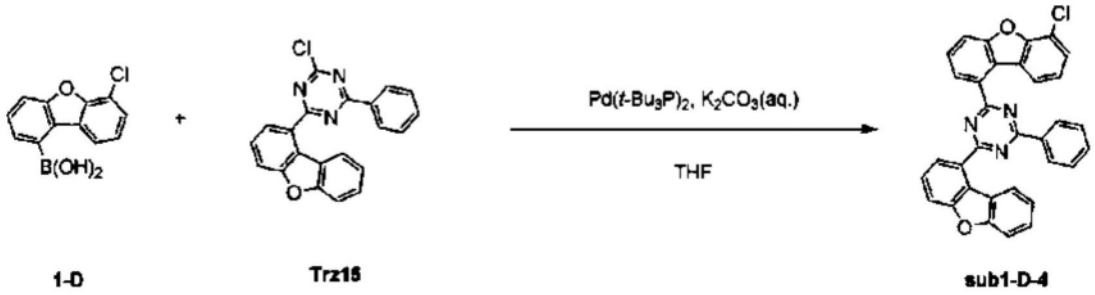


sub1-D-3 sub17 1-24

[0461] 在氮气气氛下将化合物sub1-D-3(15g,29.4mmol)和化合物sub17(7.7g,29.4mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(12.2g,88.2mmol)

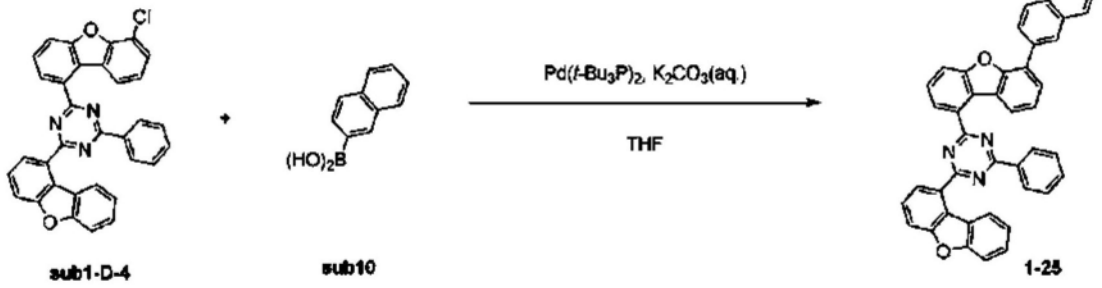
溶解在37ml水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)(0.2g,0.3mmol)。在10小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到12.4g化合物1-24。(产率:61%,MS:$[M+H]^+$=692)

[0462] 制备例1-25:化合物1-25的制备

[0463] 

1-D Trz15 sub1-D-4

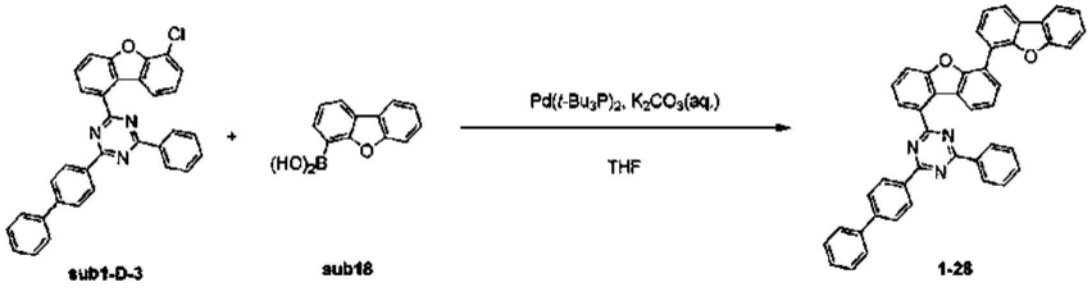
[0464] 在氮气气氛下将化合物1-D(15g,60.9mmol)和化合物Trz15(21.8g,60.9mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(25.2g,182.6mmol)溶解在76ml水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)(0.3g,0.6mmol)。在11小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到21.3g化合物sub1-D-4。(产率:67%,MS:$[M+H]^+$=524)

[0465] 

sub1-D-4 sub10 1-25

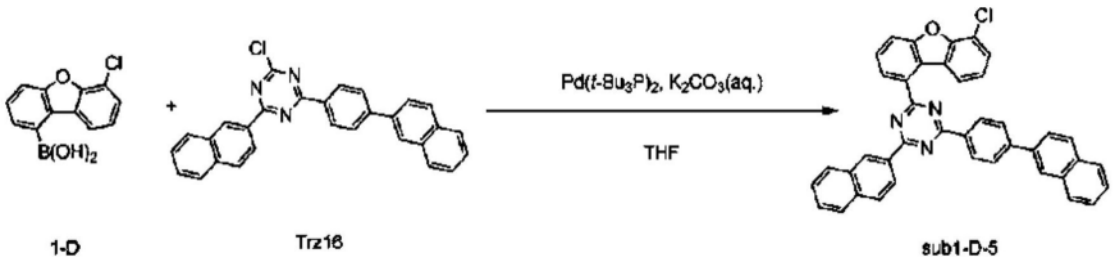
[0466] 在氮气气氛下将化合物sub1-D-4(15g,28.6mmol)和化合物sub10(4.9g,28.6mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(11.9g,85.9mmol)溶解在36ml水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)(0.1g,0.3mmol)。在10小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到10.7g化合物1-25。(产率:61%,MS:$[M+H]^+$=616)

[0467] 制备例1-26:化合物1-26的制备

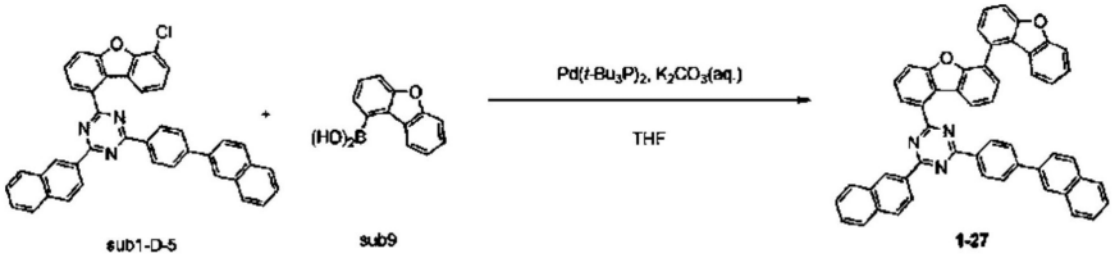
[0468] 

[0469] 在氮气气氛下将化合物sub1-D-3(15g,29.4mmol)和化合物sub18(6.2g,29.4mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(12.2g,88.2mmol)溶解在37ml水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)(0.2g,0.3mmol)。在9小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到14.3g化合物1-26。(产率:76%,MS:$[M+H]^+$=642)

[0470] 制备例1-27:化合物1-27的制备

[0471] 

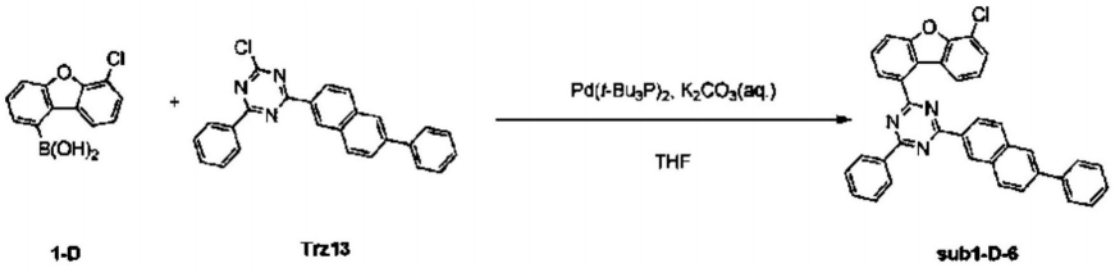
[0472] 在氮气气氛下将化合物1-D(15g,60.9mmol)和化合物Trz16(27g,60.9mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(25.2g,182.6mmol)溶解在76ml水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)(0.3g,0.6mmol)。在11小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到27.1g化合物sub1-D-5。(产率:73%,MS:$[M+H]^+$=610)

[0473] 

[0474] 在氮气气氛下将化合物sub1-D-5(15g,24.6mmol)和化合物sub9(5.2g,24.6mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(10.2g,73.8mmol)溶解在31ml水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)(0.1g,0.2mmol)。在9小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯

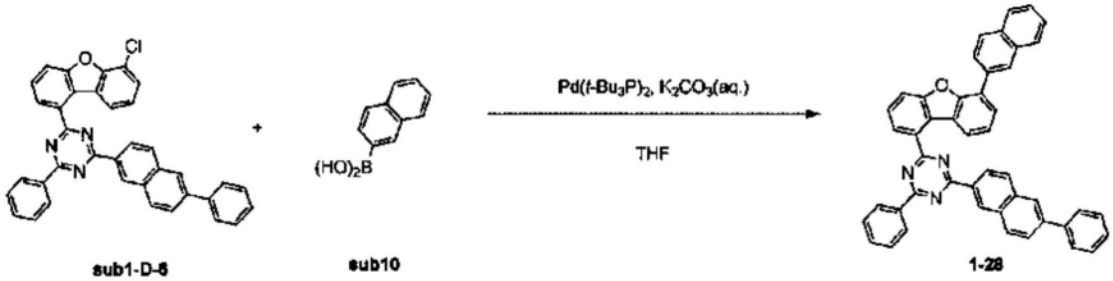
化以得到12.8g化合物1-27。(产率:70%,MS:[M+H]$^+$=742)

[0475] 制备例1-28:化合物1-28的制备

[0476] 

1-D Trz13 sub1-D-6

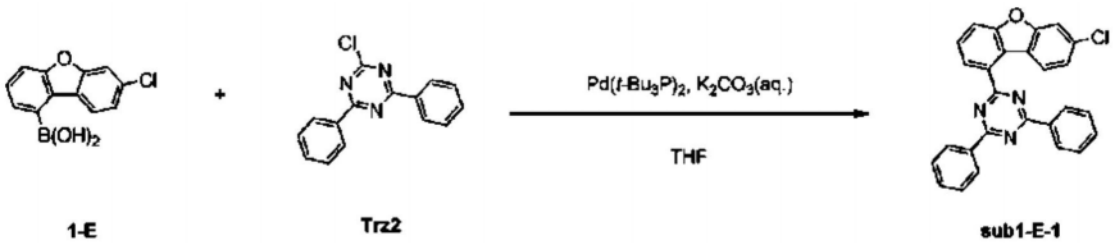
[0477] 在氮气气氛下将化合物1-D(15g,60.9mmol)和化合物Trz13(24g,60.9mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(25.2g,182.6mmol)溶解在76ml水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)(0.3g,0.6mmol)。在9小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到20.8g化合物sub1-D-6。(产率:61%,MS:[M+H]$^+$=560)

[0478] 

sub1-D-6 sub10 1-28

[0479] 在氮气气氛下将化合物sub1-D-6(15g,26.8mmol)和化合物sub10(4.6g,26.8mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(11.1g,80.3mmol)溶解在33ml水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)(0.1g,0.3mmol)。在9小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到12.2g化合物1-28。(产率:70%,MS:[M+H]$^+$=652)

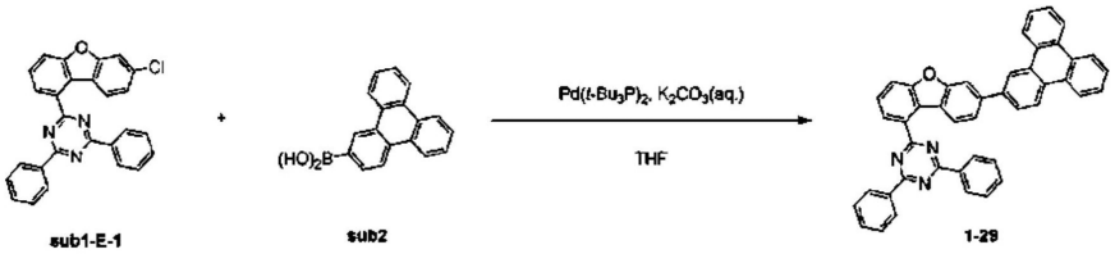
[0480] 制备例1-29:化合物1-29的制备

[0481] 

1-E Trz2 sub1-E-1

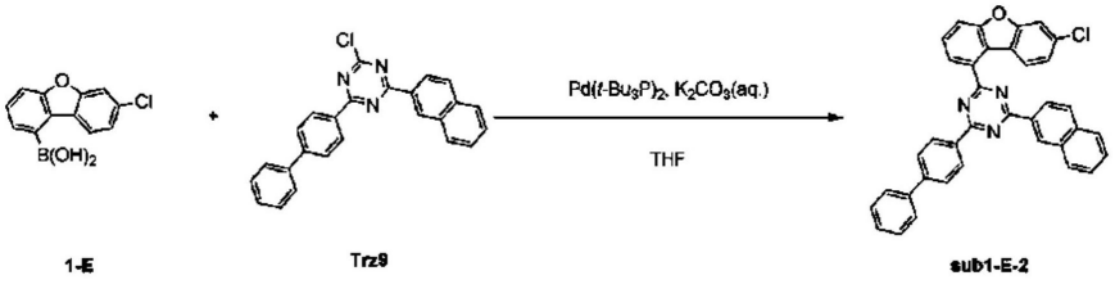
[0482] 在氮气气氛下将化合物1-E(15g,60.9mmol)和化合物Trz12(16.3g,60.9mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(25.2g,182.6mmol)溶解在76ml水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)(0.3g,0.6mmol)。在10小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将

有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到17.1g化合物sub1-E-1。(产率:65%,MS:[M+H]$^+$=434)

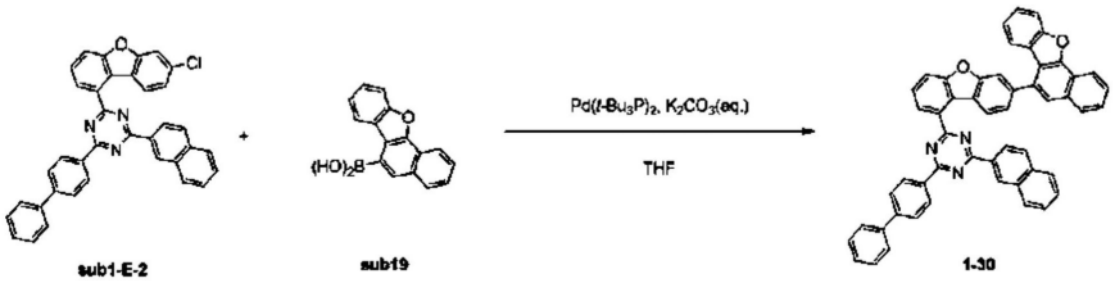
[0483] 

[0484] 在氮气气氛下将化合物sub1-E-1(15g,34.6mmol)和化合物sub2(9.4g,34.6mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(14.3g,103.7mmol)溶解在43ml水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)(0.2g,0.3mmol)。在8小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到14.5g化合物1-29。(产率:67%,MS:[M+H]$^+$=626)

[0485] 制备例1-30:化合物1-30的制备

[0486] 

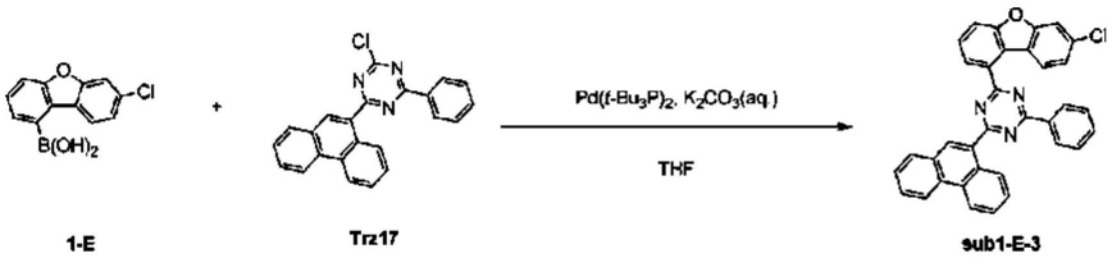
[0487] 在氮气气氛下将化合物1-E(15g,60.9mmol)和化合物Trz9(24g,60.9mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(25.2g,182.6mmol)溶解在76ml水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)(0.3g,0.6mmol)。在8小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到26.9g化合物sub1-E-2。(产率:79%,MS:[M+H]$^+$=560)

[0488] 

[0489] 在氮气气氛下将化合物sub1-E-2(15g,26.8mmol)和化合物sub19(7g,26.8mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(11.1g,80.3mmol)溶解在33ml水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)(0.1g,

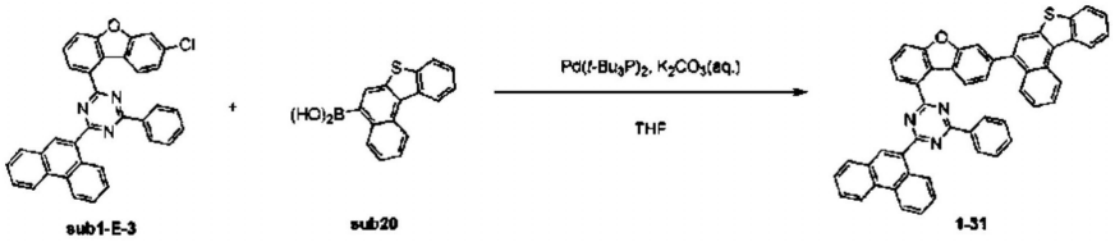
0.3mmol)。在11小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到15.9g化合物1-30。(产率:80%,MS:$[M+H]^+$=742)

[0490] 制备例1-31:化合物1-31的制备

[0491] 

1-E Trz17 sub1-E-3

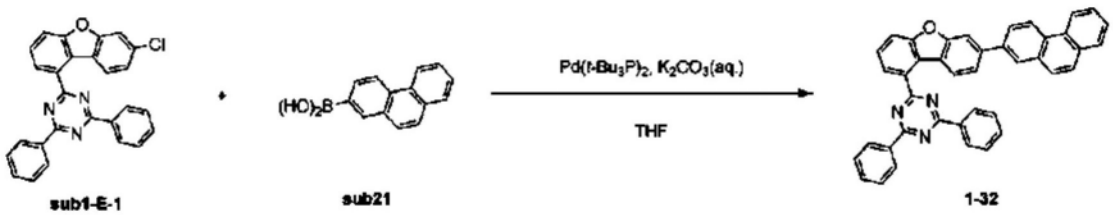
[0492] 在氮气气氛下将化合物1-E(15g,60.9mmol)和化合物Trz17(22.4g,60.9mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(25.2g,182.6mmol)溶解在76ml水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)(0.3g,0.6mmol)。在11小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到25.3g化合物sub1-E-3。(产率:78%,MS:$[M+H]^+$=534)

[0493] 

sub1-E-3 sub20 1-31

[0494] 在氮气气氛下将化合物sub1-E-3(15g,28.1mmol)和化合物sub20(7.8g,28.1mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(11.6g,84.3mmol)溶解在35ml水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)(0.1g,0.3mmol)。在9小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到14.8g化合物1-31。(产率:72%,MS:$[M+H]^+$=732)

[0495] 制备例1-32:化合物1-32的制备

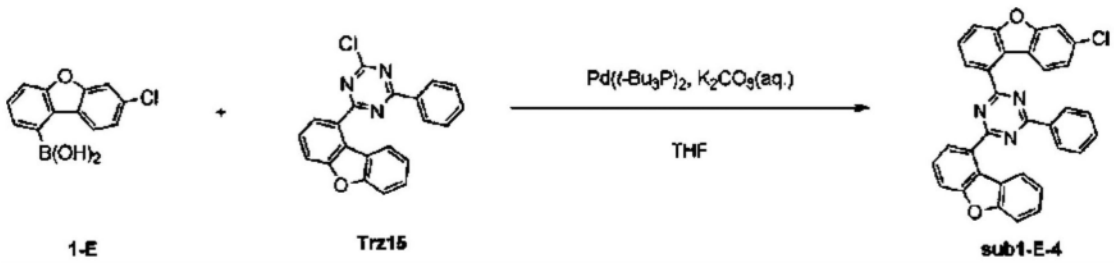
[0496] 

sub1-E-1 sub21 1-32

[0497] 在氮气气氛下将化合物sub1-E-1(15g,34.6mmol)和化合物sub21(7.7g,34.6mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(14.3g,103.7mmol)溶解在43ml水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁

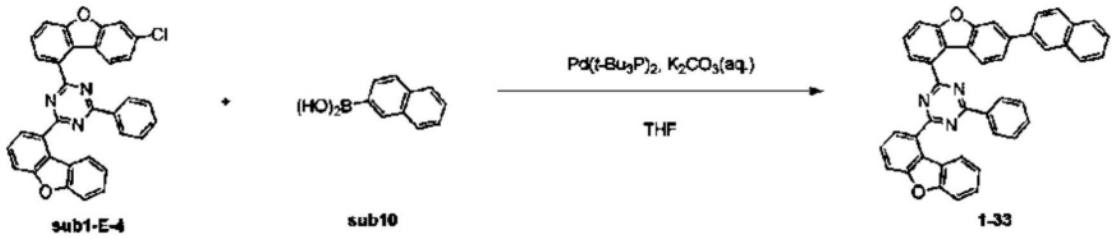
基膦)钯(0)(0.2g,0.3mmol)。在9小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到12.9g化合物1-32。(产率:65%,MS:$[M+H]^+$=576)

[0498] 制备例1-33:化合物1-33的制备

[0499] 

1-E Trz15 sub1-E-4

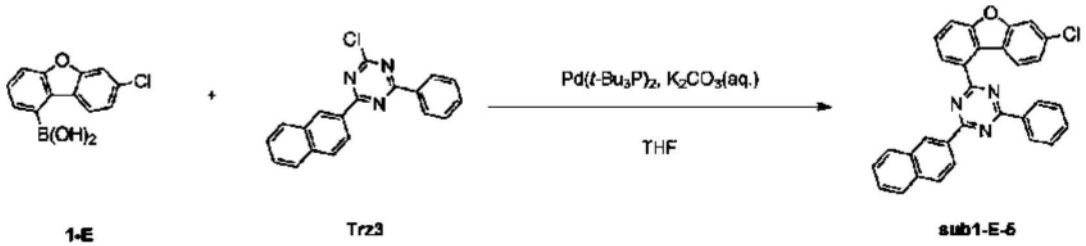
[0500] 在氮气气氛下将化合物1-E(15g,60.9mmol)和化合物Trz15(21.8g,60.9mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(25.2g,182.6mmol)溶解在76ml水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)(0.3g,0.6mmol)。在10小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到25.5g化合物sub1-E-4。(产率:80%,MS:$[M+H]^+$=524)

[0501] 

sub1-E-4 sub10 1-33

[0502] 在氮气气氛下将化合物sub1-E-4(15g,28.6mmol)和化合物sub10(4.9g,28.6mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(11.9g,85.9mmol)溶解在36ml水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)(0.1g,0.3mmol)。在11小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到10.6g化合物1-33。(产率:60%,MS:$[M+H]^+$=616)

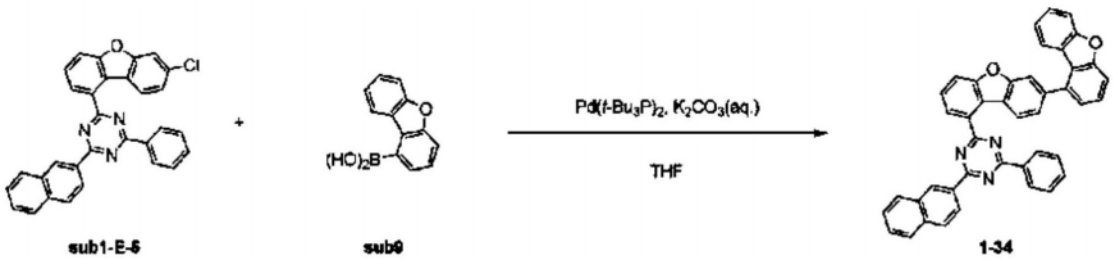
[0503] 制备例1-34:化合物1-34的制备

[0504] 

1-E Trz3 sub1-E-5

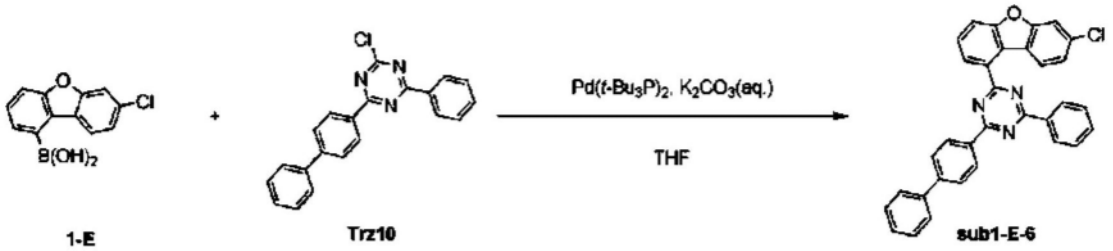
[0505] 在氮气气氛下将化合物1-E(15g,60.9mmol)和化合物Trz3(19.3g,60.9mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(25.2g,182.6mmol)溶解在76ml水

中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)(0.3g,0.6mmol)。在11小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到17.6g化合物sub1-E-5。(产率:60%,MS:$[M+H]^+$=484)

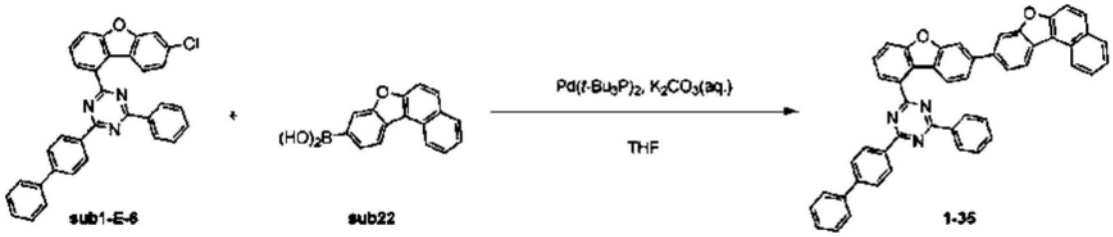
[0506] 

[0507] 在氮气气氛下将化合物sub1-E-5(15g,31mmol)和化合物sub9(6.6g,31mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(12.9g,93mmol)溶解在39ml水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)(0.2g,0.3mmol)。在11小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到11.4g化合物1-34。(产率:60%,MS:$[M+H]^+$=616)

[0508] 制备例1-35:化合物1-35的制备

[0509] 

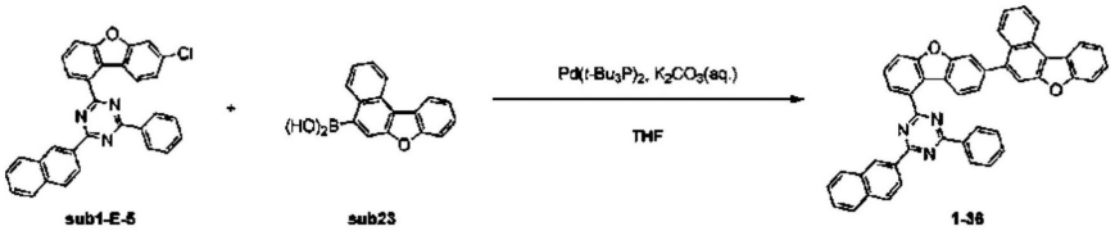
[0510] 在氮气气氛下将化合物1-E(15g,60.9mmol)和化合物Trz10(20.9g,60.9mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(25.2g,182.6mmol)溶解在76ml水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)(0.3g,0.6mmol)。在8小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到21.7g化合物sub1-E-6。(产率:70%,MS:$[M+H]^+$=510)

[0511] 

[0512] 在氮气气氛下将化合物sub1-E-6(15g,29.4mmol)和化合物sub22(7.7g,29.4mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(12.2g,88.2mmol)

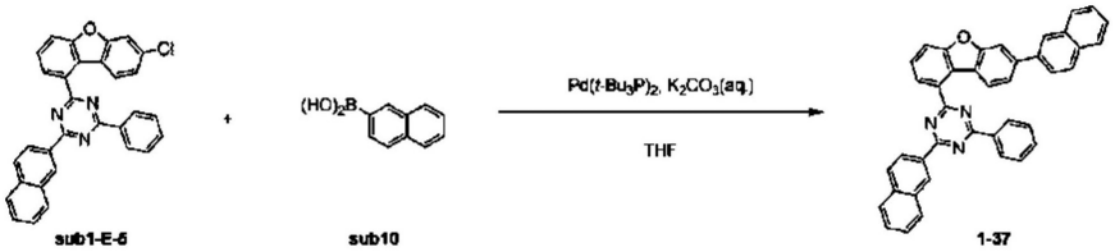
溶解在37ml水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)(0.2g,0.3mmol)。在9小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到14.6g化合物1-35。(产率:72%,MS:$[M+H]^+$=692)

[0513] 制备例1-36:化合物1-36的制备

[0514] 

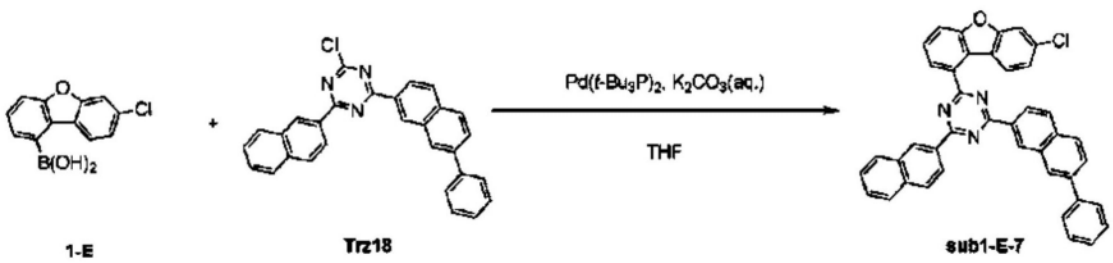
[0515] 在氮气气氛下将化合物sub1-E-5(15g,31mmol)和化合物sub23(8.1g,31mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(12.9g,93mmol)溶解在39ml水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)(0.2g,0.3mmol)。在12小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到12.4g化合物1-36。(产率:60%,MS:$[M+H]^+$=666)

[0516] 制备例1-37:化合物1-37的制备

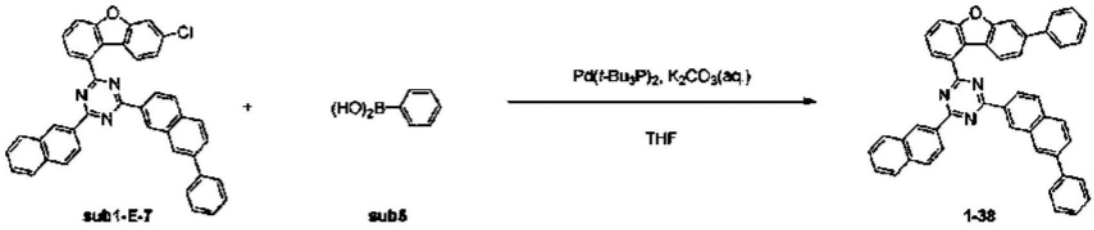
[0517] 

[0518] 在氮气气氛下将化合物sub1-E-5(15g,31mmol)和化合物sub10(5.3g,31mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(12.9g,93mmol)溶解在39ml水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)(0.2g,0.3mmol)。在12小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到14.1g化合物1-37。(产率:79%,MS:$[M+H]^+$=576)

[0519] 制备例1-38:化合物1-38的制备

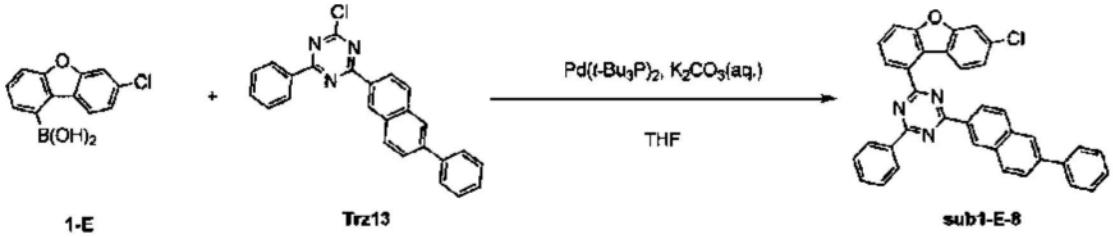
[0520] 

[0521] 在氮气气氛下将化合物1-E(15g,60.9mmol)和化合物Trz18(27g,60.9mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(25.2g,182.6mmol)溶解在76ml水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)(0.3g,0.6mmol)。在11小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到24.1g化合物sub1-E-7。(产率:65%,MS:[M+H]$^+$=610)

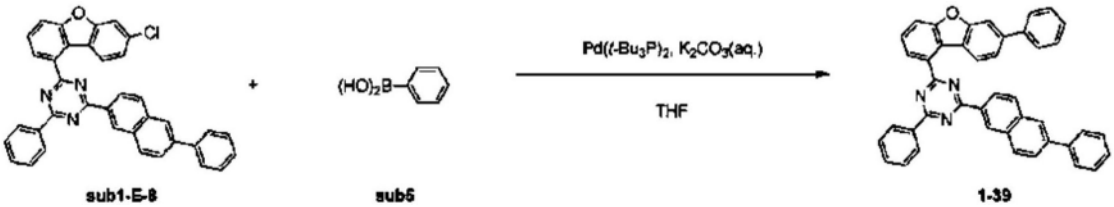
[0522] 

[0523] 在氮气气氛下将化合物sub1-E-7(15g,24.6mmol)和化合物sub5(3g,24.6mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(10.2g,73.8mmol)溶解在31ml水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)(0.1g,0.2mmol)。在9小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到10.1g化合物1-38。(产率:63%,MS:[M+H]$^+$=652)

[0524] 制备例1-39:化合物1-39的制备

[0525] 

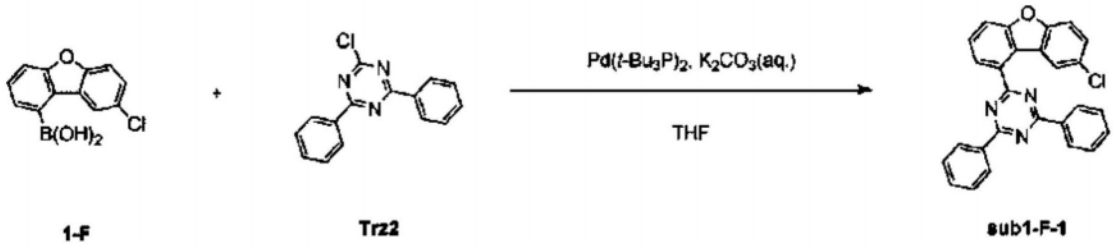
[0526] 在氮气气氛下将化合物1-E(15g,60.9mmol)和化合物Trz13(24g,60.9mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(25.2g,182.6mmol)溶解在76ml水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)(0.3g,0.6mmol)。在12小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到26.2g化合物sub1-E-8。(产率:77%,MS:[M+H]$^+$=560)

[0527] 

[0528] 在氮气气氛下将化合物sub1-E-8(15g,26.8mmol)和化合物sub5(3.3g,26.8mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(11.1g,80.3mmol)溶解在

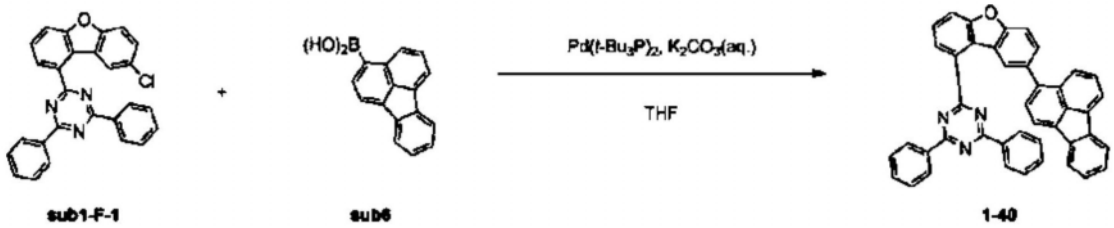
33ml水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)(0.1g,0.3mmol)。在11小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到10.9g化合物1-39。(产率:68%,MS:$[M+H]^+$=602)

[0529] 制备例1-40:化合物1-40的制备

[0530] 

1-F Trz2 sub1-F-1

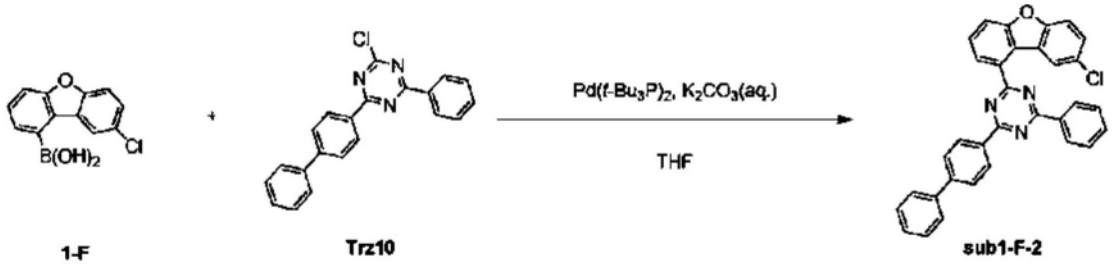
[0531] 在氮气气氛下将化合物1-F(15g,60.9mmol)和化合物Trz2(16.3g,60.9mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(25.2g,182.6mmol)溶解在76ml水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)(0.3g,0.6mmol)。在12小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到19.2g化合物sub1-F-1。(产率:73%,MS:$[M+H]^+$=434)

[0532] 

sub1-F-1 sub6 1-40

[0533] 在氮气气氛下将化合物sub1-F-1(15g,34.6mmol)和化合物sub6(8.5g,34.6mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(14.3g,103.7mmol)溶解在43ml水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)(0.2g,0.3mmol)。在10小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到14.7g化合物1-40。(产率:71%,MS:$[M+H]^+$=600)

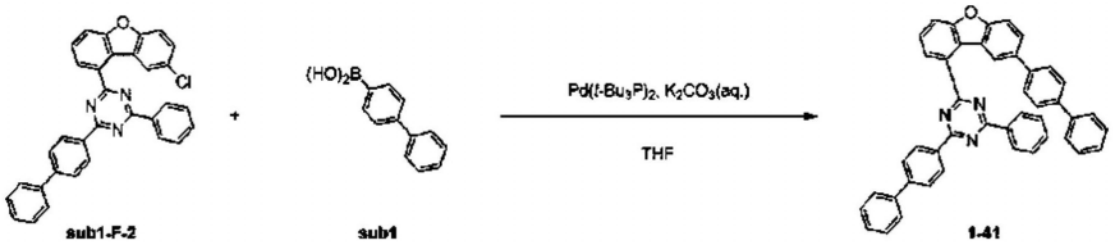
[0534] 制备例1-41:化合物1-41的制备

[0535] 

1-F Trz10 sub1-F-2

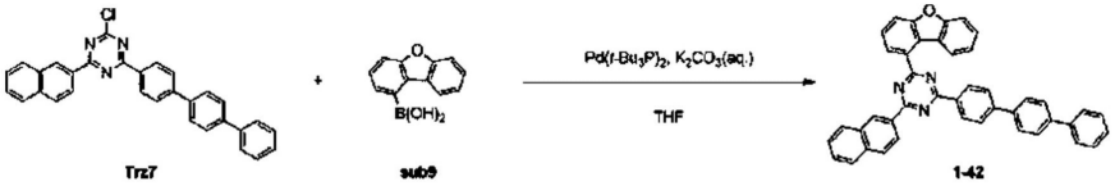
[0536] 在氮气气氛下将化合物1-F(15g,60.9mmol)和化合物Trz10(20.9g,60.9mmol)添

加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(25.2g,182.6mmol)溶解在76ml水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)(0.3g,0.6mmol)。在11小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到21.1g化合物sub1-F-2。(产率:68%,MS:[M+H]$^+$=510)

[0537] 

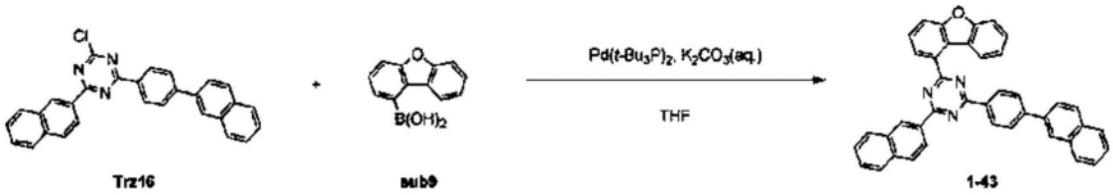
[0538] 在氮气气氛下将化合物sub1-F-2(15g,29.4mmol)和化合物sub1(5.8g,29.4mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(12.2g,88.2mmol)溶解在37ml水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)(0.2g,0.3mmol)。在12小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到14.2g化合物1-41。(产率:77%,MS:[M+H]$^+$=628)

[0539] 制备例1-42:化合物1-42的制备

[0540] 

[0541] 在氮气气氛下将化合物Trz7(15g,31.9mmol)和化合物sub9(6.8g,31.9mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(13.2g,95.8mmol)溶解在40ml水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)(0.2g,0.3mmol)。在9小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到15.2g化合物1-42。(产率:79%,MS:[M+H]$^+$=602)

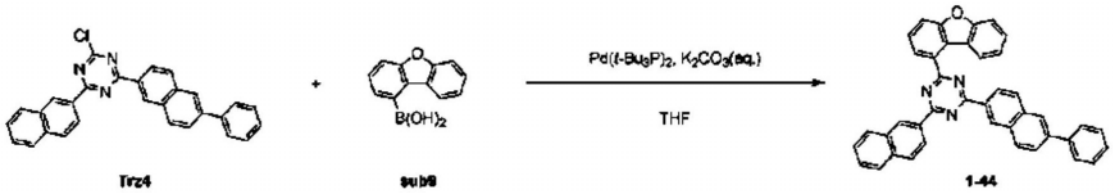
[0542] 制备例1-43:化合物1-43的制备

[0543] 

[0544] 在氮气气氛下将化合物Trz16(15g,33.8mmol)和化合物sub9(7.2g,33.8mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(14g,101.4mmol)溶解在42ml水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)(0.2g,

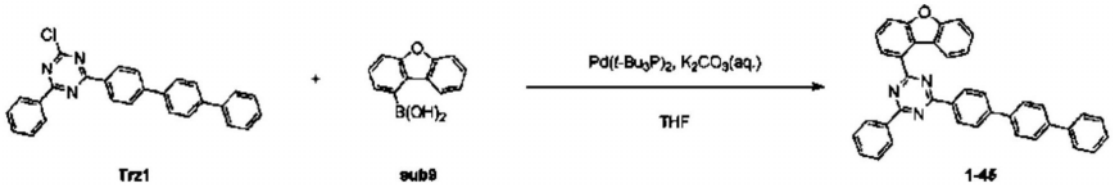
0.3mmol)。在12小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到15g化合物1-43。(产率:77%,MS:$[M+H]^+$=576)

[0545] 制备例1-44:化合物1-44的制备

[0546] 

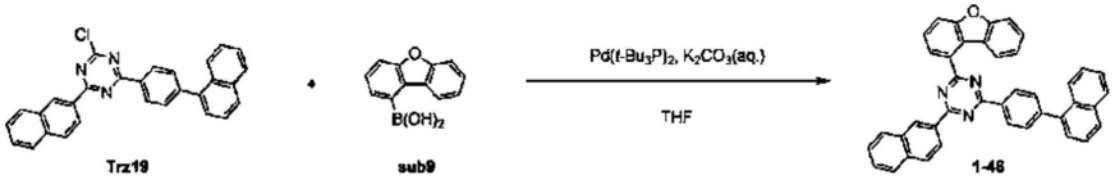
[0547] 在氮气气氛下将化合物Trz4(15g,33.8mmol)和化合物sub9(7.2g,33.8mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(14g,101.4mmol)溶解在42ml水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)(0.2g,0.3mmol)。在11小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到14.2g化合物1-44。(产率:73%,MS:$[M+H]^+$=576)

[0548] 制备例1-45:化合物1-45的制备

[0549] 

[0550] 在氮气气氛下将化合物Trz1(15g,35.7mmol)和化合物sub9(7.6g,35.7mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(14.8g,107.2mmol)溶解在44ml水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)(0.2g,0.4mmol)。在9小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到12.2g化合物1-45。(产率:62%,MS:$[M+H]^+$=552)

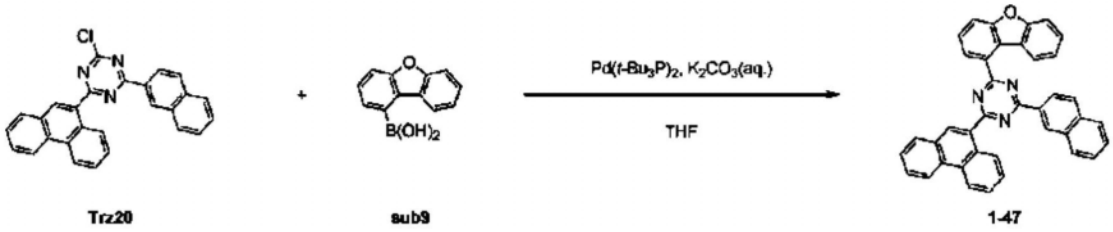
[0551] 制备例1-46:化合物1-46的制备

[0552] 

[0553] 在氮气气氛下将化合物Trz19(15g,33.8mmol)和化合物sub9(7.2g,33.8mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(14g,101.4mmol)溶解在42ml水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)(0.2g,0.3mmol)。在8小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫

酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到13.6g化合物1-46。(产率:70%,MS:[M+H]$^+$=576)

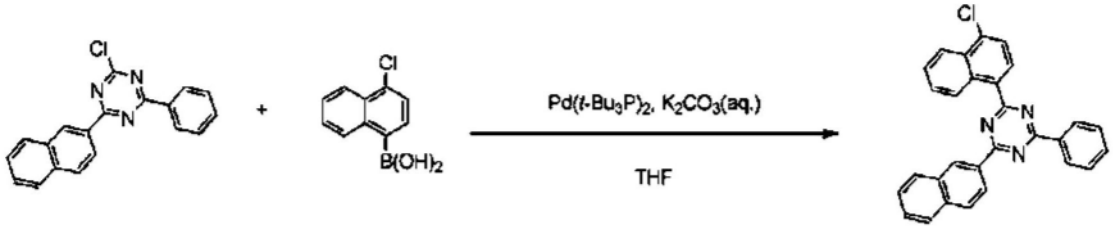
[0554] 制备例1-47:化合物1-47的制备

[0555] 

Trz20 sub9 1-47

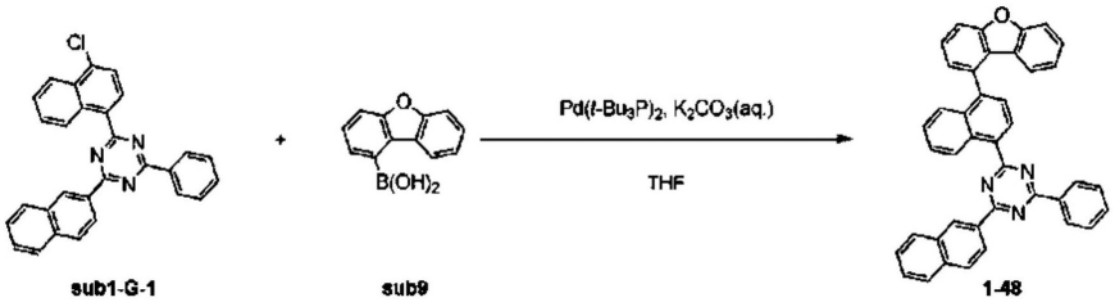
[0556] 在氮气气氛下将化合物Trz20(15g,35.9mmol)和化合物sub9(7.6g,35.9mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(14.9g,107.7mmol)溶解在45ml水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)(0.2g,0.4mmol)。在8小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到15g化合物1-47。(产率:76%,MS:[M+H]$^+$=550)

[0557] 制备例1-48:化合物1-48的制备

[0558] 

Trz3 sub24 sub1-G-1

[0559] 在氮气气氛下将化合物Trz3(15g,47.2mmol)和化合物sub24(9.7g,47.2mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(19.6g,141.6mmol)溶解在59ml水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)(0.2g,0.5mmol)。在11小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到13g化合物sub1-G-1。(产率:62%,MS:[M+H]$^+$=444)

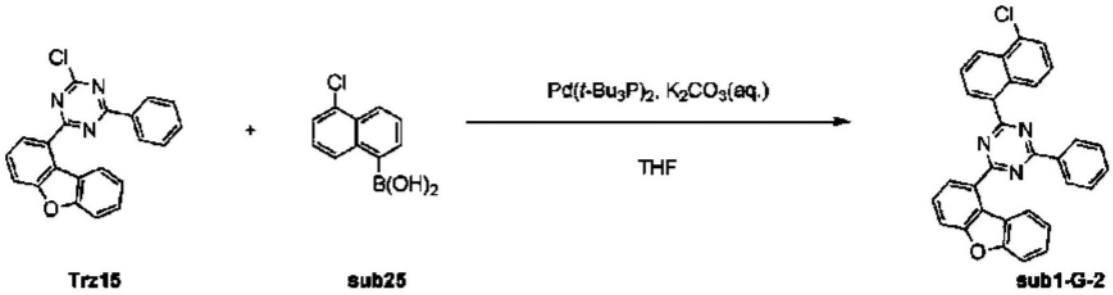
[0560] 

sub1-G-1 sub9 1-48

[0561] 在氮气气氛下将化合物sub1-G-1(15g,33.8mmol)和化合物sub9(7.2g,33.8mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(14g,101.4mmol)溶解在42ml水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)(0.2g,

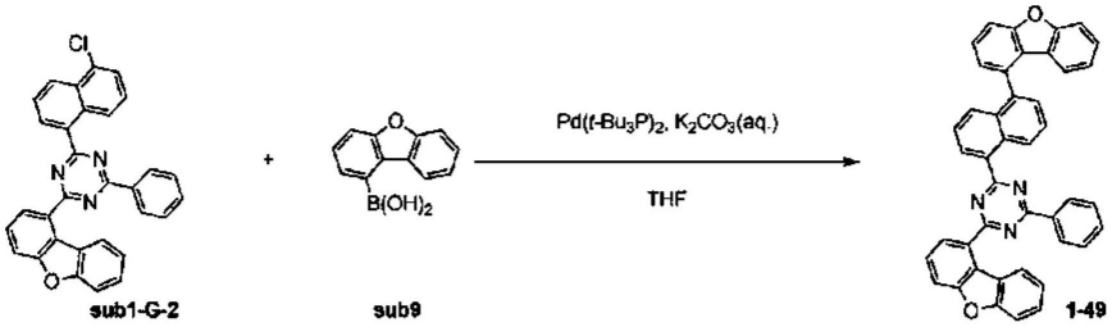
0.3mmol)。在10小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到15.2g化合物1-48。(产率:78%,MS:$[M+H]^+$=576)

[0562] 制备例1-49:化合物1-49的制备

[0563] 

Trz15 sub25 sub1-G-2

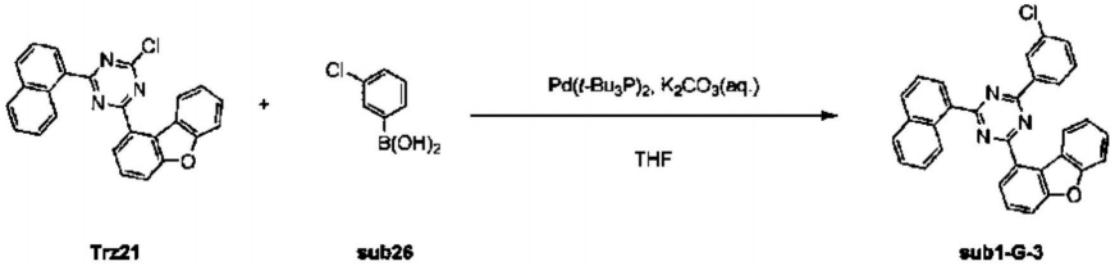
[0564] 在氮气气氛下将化合物Trz15(15g,41.9mmol)和化合物sub25(8.7g,41.9mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(17.4g,125.8mmol)溶解在52ml水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)(0.2g,0.4mmol)。在8小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到12.6g化合物sub1-G-2。(产率:62%,MS:$[M+H]^+$=484)

[0565] 

sub1-G-2 sub9 1-49

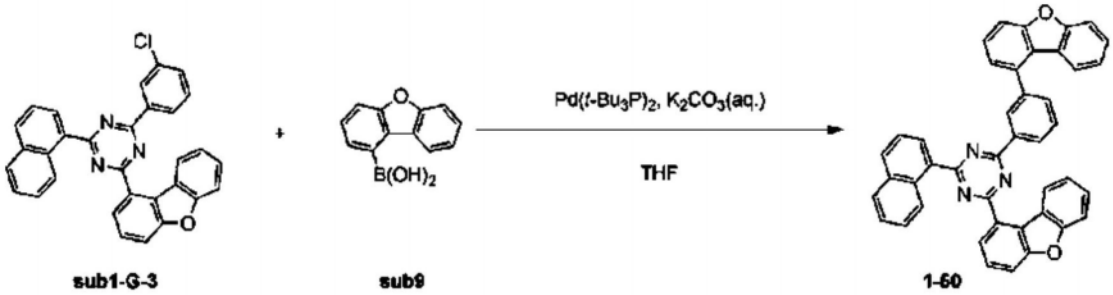
[0566] 在氮气气氛下将化合物sub1-G-2(15g,31mmol)和化合物sub9(6.6g,31mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(12.9g,93mmol)溶解在39ml水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)(0.2g,0.3mmol)。在8小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到13.7g化合物1-49。(产率:72%,MS:$[M+H]^+$=616)

[0567] 制备例1-50:化合物1-50的制备

[0568] 

Trz21 sub26 sub1-G-3

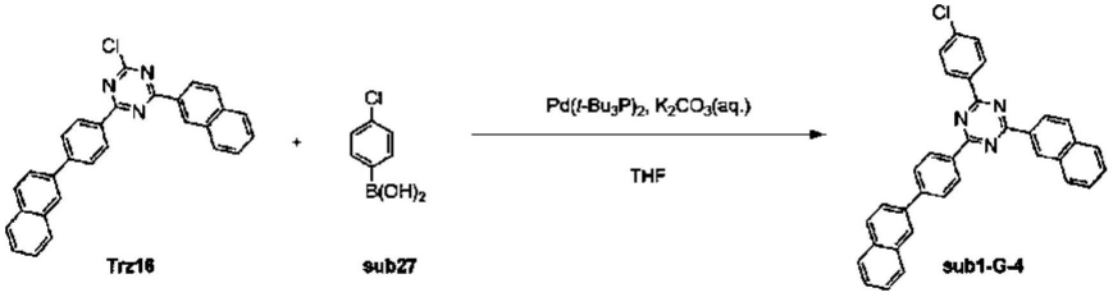
[0569] 在氮气气氛下将化合物Trz21(15g,36.8mmol)和化合物sub26(5.8g,36.8mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(15.2g,110.3mmol)溶解在46ml水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)(0.2g,0.4mmol)。在9小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到12.8g化合物sub1-G-3。(产率:72%,MS:$[M+H]^+$=484)

[0570] 

sub1-G-3 sub9 1-50

[0571] 在氮气气氛下将化合物sub1-G-3(15g,31mmol)和化合物sub9(6.6g,31mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(12.9g,93mmol)溶解在39ml水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)(0.2g,0.3mmol)。在10小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到13.2g化合物1-50。(产率:69%,MS:$[M+H]^+$=616)

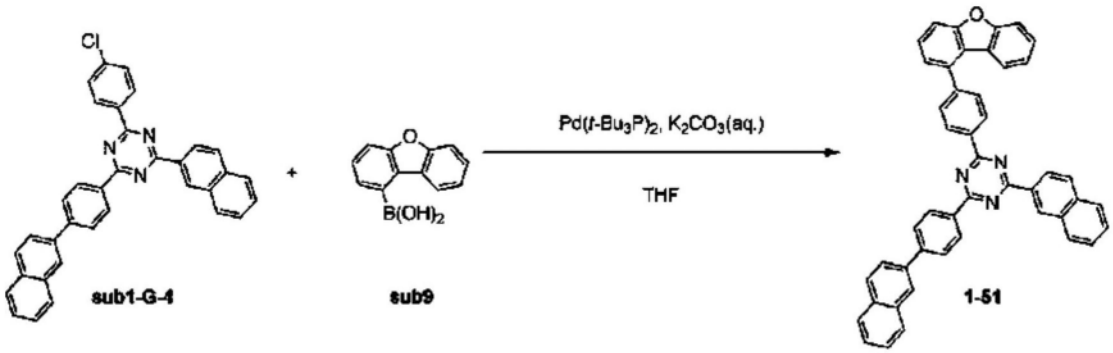
[0572] 制备例1-51:化合物1-51的制备

[0573] 

Trz16 sub27 sub1-G-4

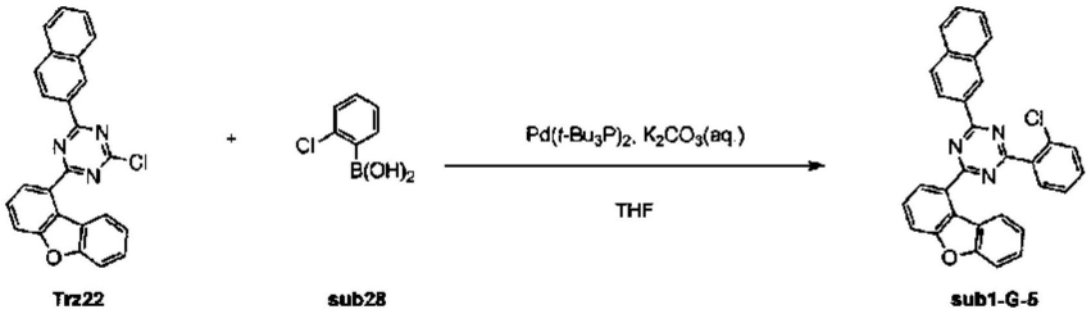
[0574] 在氮气气氛下将化合物Trz16(15g,36.8mmol)和化合物sub27(5.3g,33.8mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(14g,101.4mmol)溶解在42ml水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)(0.2g,0.3mmol)。在11小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水

硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到13.3g化合物sub1-G-4。(产率:76%,MS:[M+H]$^+$=520)

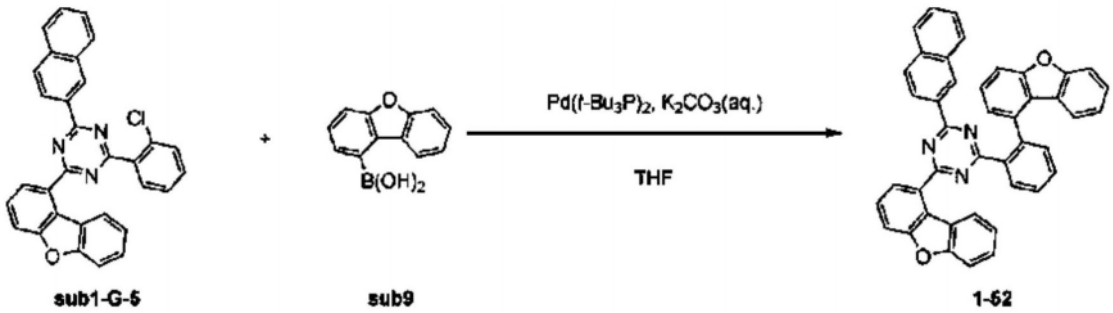
[0575] 

[0576] 在氮气气氛下将化合物sub1-G-4(15g,28.8mmol)和化合物sub9(6.1g,28.8mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(12g,86.5mmol)溶解在36ml水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)(0.1g,0.3mmol)。在9小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到13.3g化合物1-51。(产率:71%,MS:[M+H]$^+$=652)

[0577] 制备例1-52:化合物1-52的制备

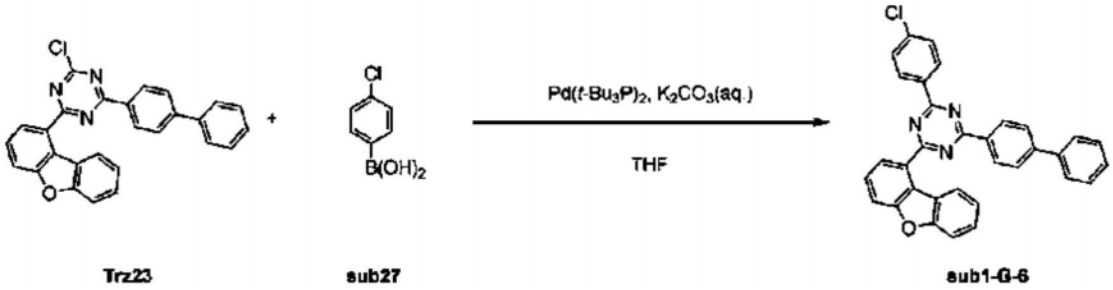
[0578] 

[0579] 在氮气气氛下将化合物Trz22(15g,36.8mmol)和化合物sub28(5.8g,36.8mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(15.2g,110.3mmol)溶解在46ml水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)(0.2g,0.4mmol)。在12小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到12.8g化合物sub1-G-5。(产率:72%,MS:[M+H]$^+$=484)

[0580] 

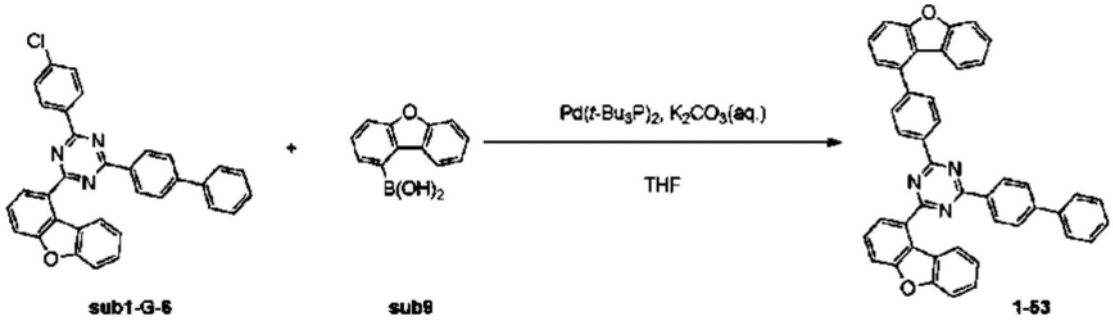
[0581] 在氮气气氛下将化合物sub1-G-5(15g,31mmol)和化合物sub9(6.6g,31mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(12.9g,93mmol)溶解在39ml水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)(0.2g,0.3mmol)。在10小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到13g化合物1-52。(产率:68%,MS:[M+H]$^+$=616)

[0582] 制备例1-53:化合物1-53的制备

[0583] 

Trz23 sub27 sub1-G-6

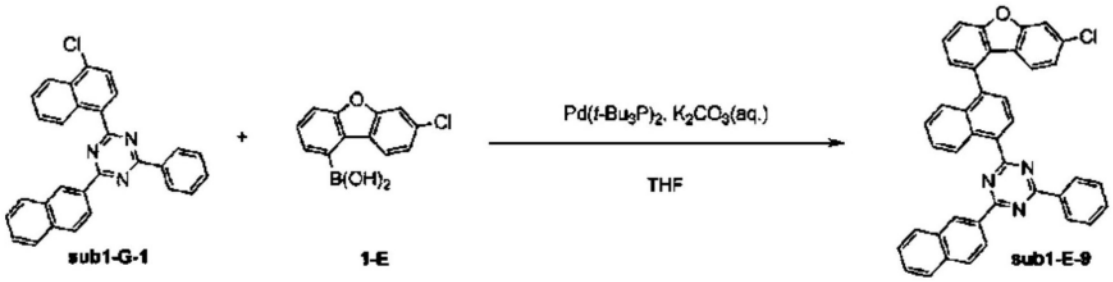
[0584] 在氮气气氛下将化合物Trz23(15g,34.6mmol)和化合物sub27(5.4g,34.6mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(14.3g,103.7mmol)溶解在43ml水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)(0.2g,0.3mmol)。在10小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到11.3g化合物sub1-G-6。(产率:64%,MS:[M+H]$^+$=510)

[0585] 

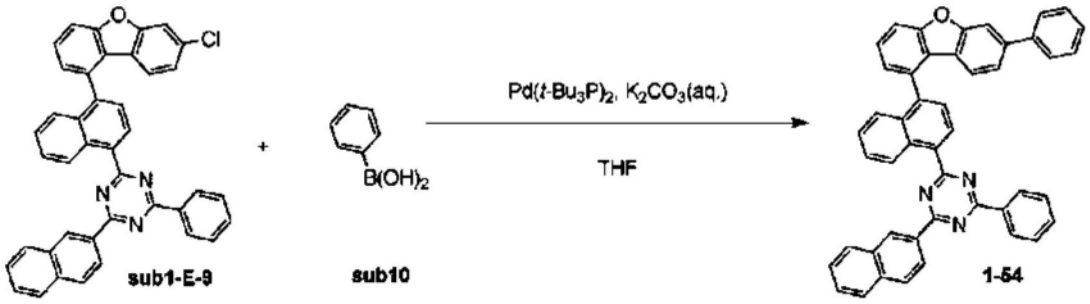
sub1-G-6 sub9 1-53

[0586] 在氮气气氛下将化合物sub1-G-6(15g,31mmol)和化合物sub9(6.6g,31mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(12.9g,93mmol)溶解在39ml水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)(0.2g,0.3mmol)。在10小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到13g化合物1-53。(产率:68%,MS:[M+H]$^+$=616)

[0587] 制备例1-54:化合物1-54的制备

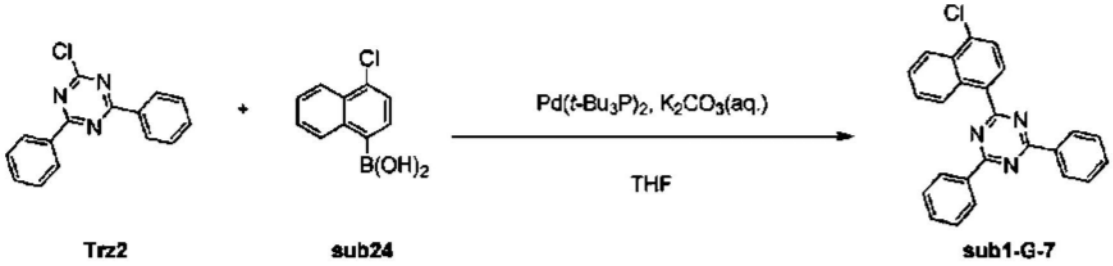
[0588] 

[0589] 在氮气气氛下将化合物sub-G-1(15g,33.8mmol)和化合物1-E(8.3g,33.8mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(14g,101.4mmol)溶解在42ml水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)(0.2g,0.3mmol)。在8小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到14.4g化合物sub1-E-9。(产率:70%,MS:[M+H]$^+$=610)

[0590] 

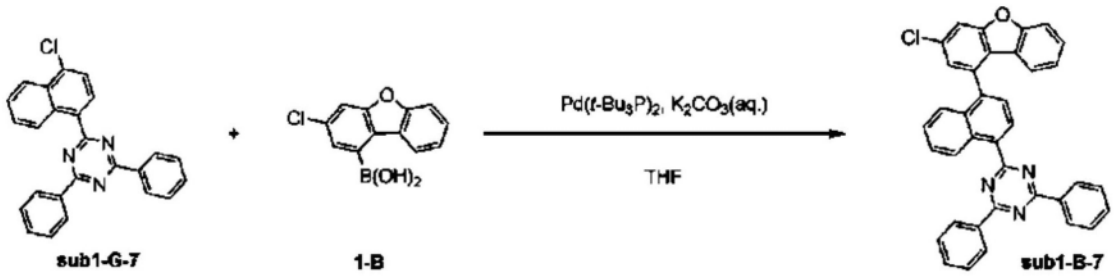
[0591] 在氮气气氛下将化合物sub1-E-9(15g,24.6mmol)和化合物sub10(3g,24.6mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(10.2g,73.8mmol)溶解在31ml水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)(0.1g,0.2mmol)。在11小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到12.2g化合物1-54。(产率:76%,MS:[M+H]$^+$=652)

[0592] 制备例1-55:化合物1-55的制备

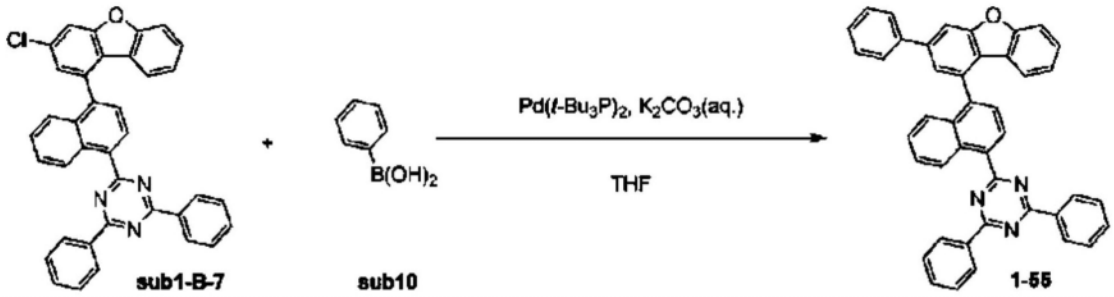
[0593] 

[0594] 在氮气气氛下将化合物Trz2(15g,56mmol)和化合物sub24(11.6g,56mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(23.2g,168.1mmol)溶解在70ml水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)(0.3g,0.6mmol)。在11小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水

硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到15.6g化合物sub1-G-7。(产率:71%,MS:[M+H]$^+$=394)

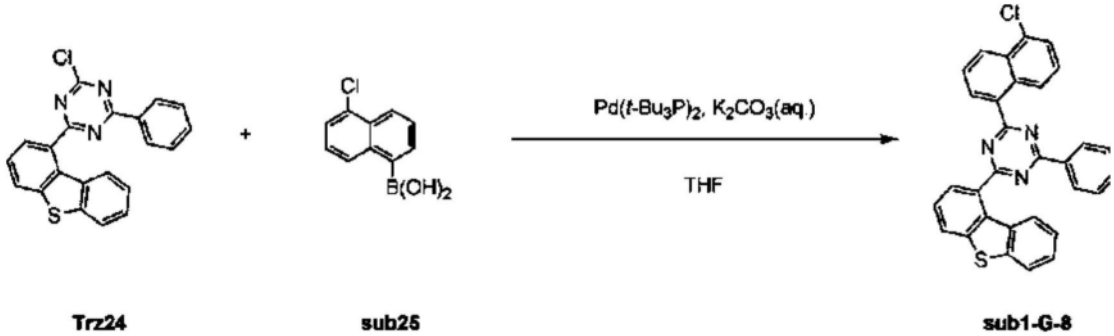
[0595] 

[0596] 在氮气气氛下将化合物sub1-G-7(15g,38.1mmol)和化合物1-B(9.4g,38.1mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(15.8g,114.3mmol)溶解在47ml水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)(0.2g,0.4mmol)。在10小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到13.8g化合物sub1-B-7。(产率:65%,MS:[M+H]$^+$=560)

[0597] 

[0598] 在氮气气氛下将化合物sub1-B-7(15g,26.8mmol)和化合物sub10(3.3g,26.8mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(11.1g,80.3mmol)溶解在33ml水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)(0.1g,0.3mmol)。在9小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到12.9g化合物1-55。(产率:80%,MS:[M+H]$^+$=602)

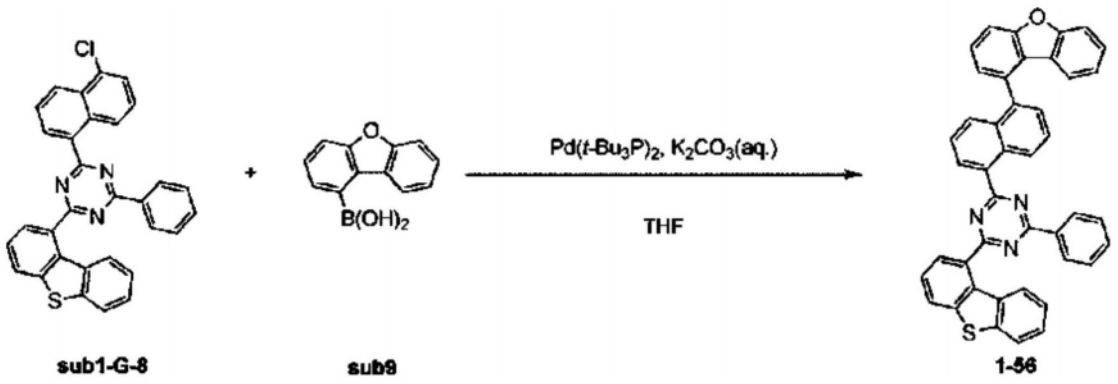
[0599] 制备例1-56:化合物1-56的制备

[0600] 

[0601] 在氮气气氛下将化合物Trz24(15g,38.1mmol)和化合物sub25(9.4g,38.1mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(15.8g,114.3mmol)溶解在47ml

水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)(0.2g,0.4mmol)。在10小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到13.8g化合物sub1-G-8。(产率:65%,MS:[M+H]$^+$=560)

[0602]

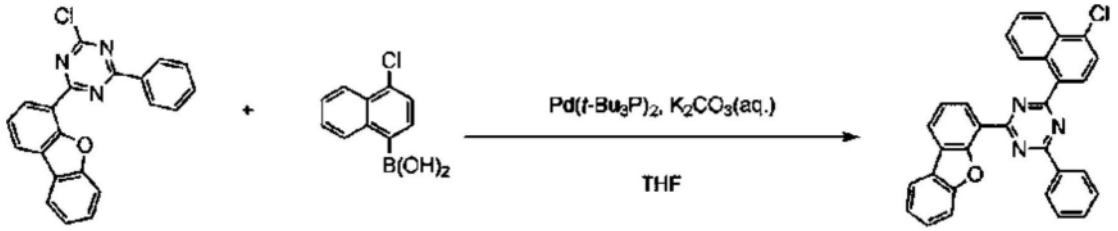


sub1-G-8 sub9 1-56

[0603] 在氮气气氛下将化合物sub1-G-8(15g,30mmol)和化合物sub9(6.4g,30mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(12.4g,90mmol)溶解在37ml水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)(0.2g,0.3mmol)。在10小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到13.4g化合物1-56。(产率:71%,MS:[M+H]$^+$=632)

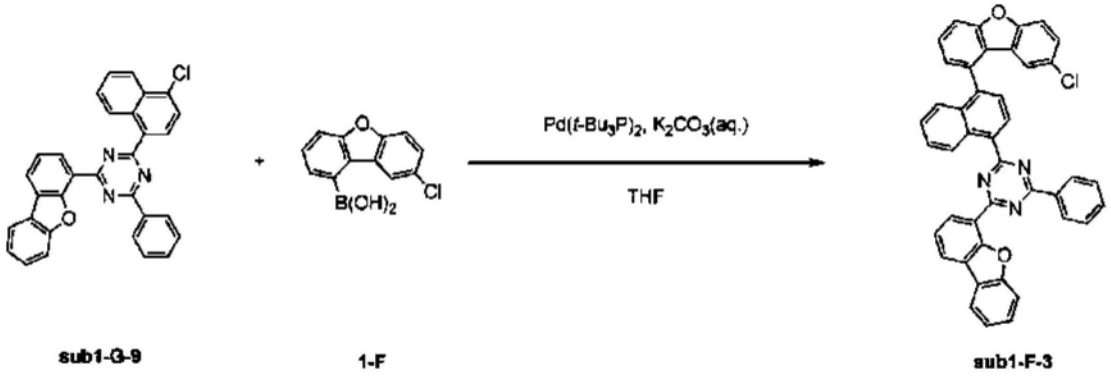
[0604] 制备例1-57:化合物1-57的制备

[0605]



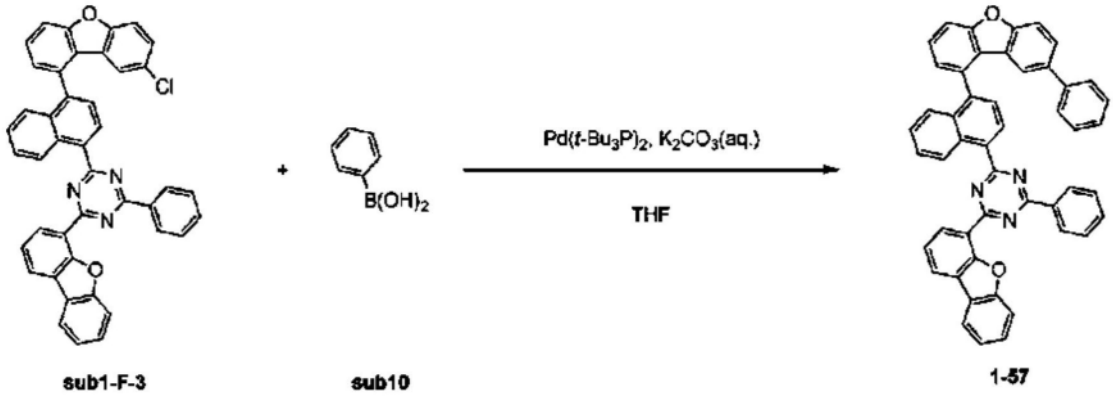
Trz25 sub24 sub1-G-9

[0606] 在氮气气氛下将化合物Trz25(15g,41.9mmol)和化合物sub24(8.7g,41.9mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(17.4g,125.8mmol)溶解在52ml水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)(0.2g,0.4mmol)。在11小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到12.4g化合物sub1-G-9。(产率:61%,MS:[M+H]$^+$=484)

[0607] 

sub1-G-9 1-F sub1-F-3

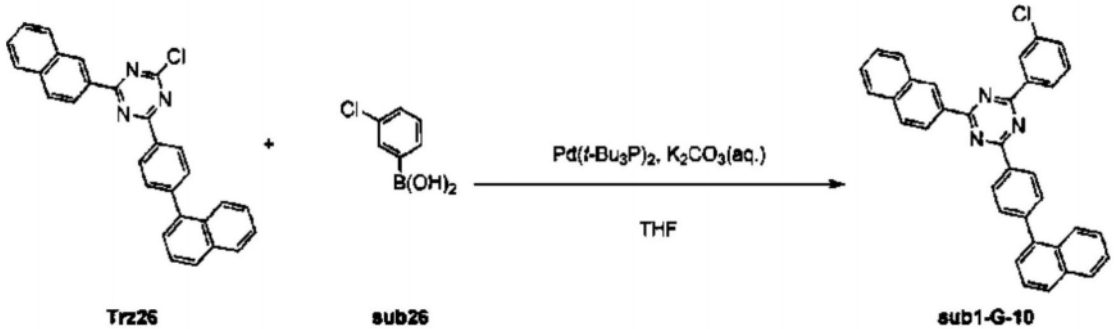
[0608] 在氮气气氛下将化合物sub1-G-9(15g,31mmol)和化合物1-F(7.6g,31mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(12.9g,93mmol)溶解在39ml水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)(0.2g,0.3mmol)。在9小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到12.5g化合物sub1-F-3。(产率:62%,MS:[M+H]$^+$=650)

[0609] 

sub1-F-3 sub10 1-57

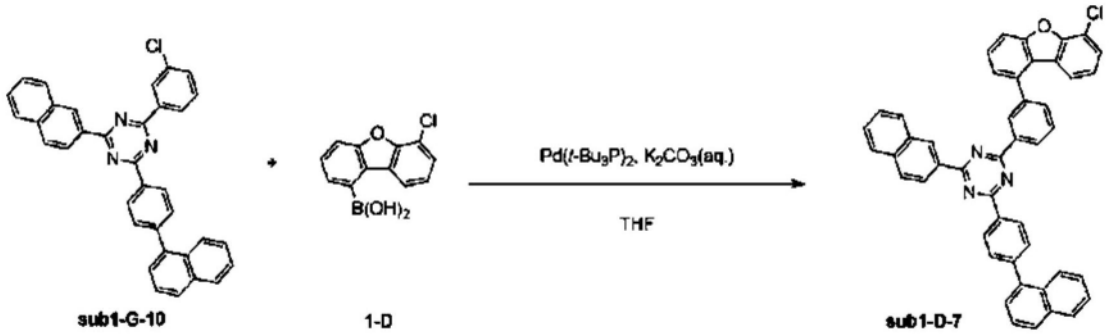
[0610] 在氮气气氛下将化合物sub1-F-3(15g,23.1mmol)和化合物sub10(2.8g,23.1mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(9.6g,69.2mmol)溶解在29ml水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)(0.1g,0.2mmol)。在11小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到12.8g化合物1-57。(产率:80%,MS:[M+H]$^+$=692)

[0611] 制备例1-58:化合物1-58的制备

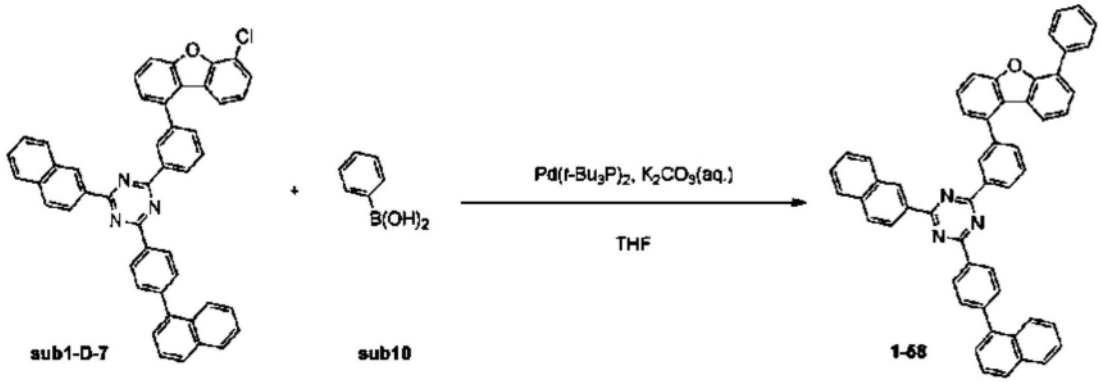
[0612] 

Trz26 sub26 sub1-G-10

[0613] 在氮气气氛下将化合物Trz26(15g,33.8mmol)和化合物sub26(5.3g,33.8mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(14g,101.4mmol)溶解在42ml水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)(0.2g,0.3mmol)。在10小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到10.5g化合物sub1-G-10。(产率:60%,MS:[M+H]$^+$=520)

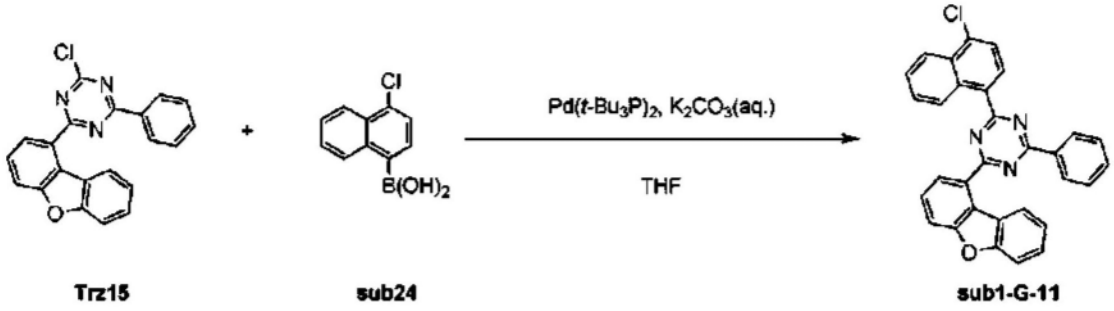
[0614] 

[0615] 在氮气气氛下将化合物sub1-G-10(15g,28.8mmol)和化合物1-D(7.1g,28.8mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(12g,86.5mmol)溶解在36ml水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)(0.1g,0.3mmol)。在10小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到15g化合物sub1-D-7。(产率:76%,MS:[M+H]$^+$=686)

[0616] 

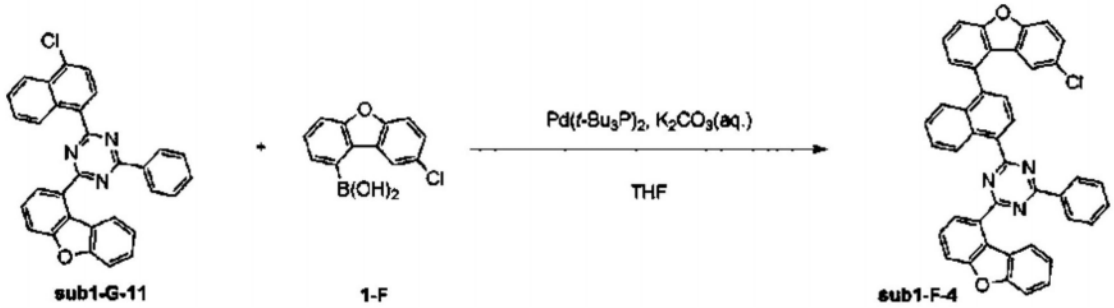
[0617] 在氮气气氛下将化合物sub1-D-7(15g,21.9mmol)和化合物10(2.7g,21.9mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(9.1g,65.6mmol)溶解在27ml水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)(0.1g,0.2mmol)。在12小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到9.9g化合物1-58。(产率:62%,MS:[M+H]$^+$=728)

[0618] 制备例1-59:化合物1-59的制备

[0619] 

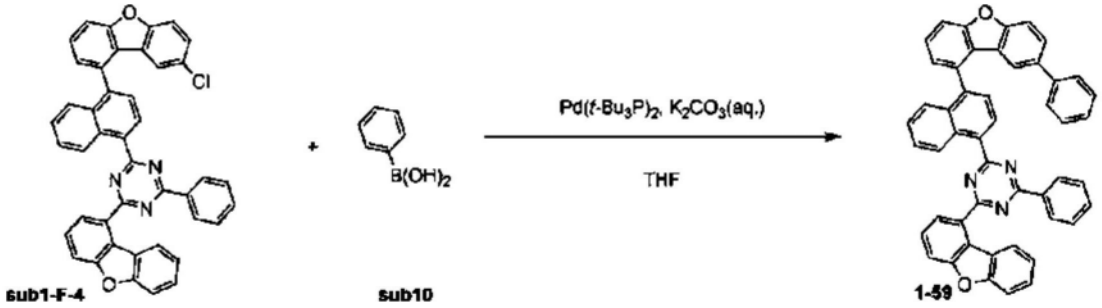
Trz15 sub24 sub1-G-11

[0620] 在氮气气氛下将化合物Trz15(15g,41.9mmol)和化合物sub24(8.7g,41.9mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(17.4g,125.8mmol)溶解在52ml水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)(0.2g,0.4mmol)。在11小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到12.4g化合物sub1-G-11。(产率:61%,MS:$[M+H]^+$=484)

[0621] 

sub1-G-11 1-F sub1-F-4

[0622] 在氮气气氛下将化合物sub1-G-11(15g,28.8mmol)和化合物1-F(7.1g,28.8mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(12g,86.5mmol)溶解在36ml水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)(0.1g,0.3mmol)。在10小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到15g化合物sub1-F-4。(产率:76%,MS:$[M+H]^+$=686)

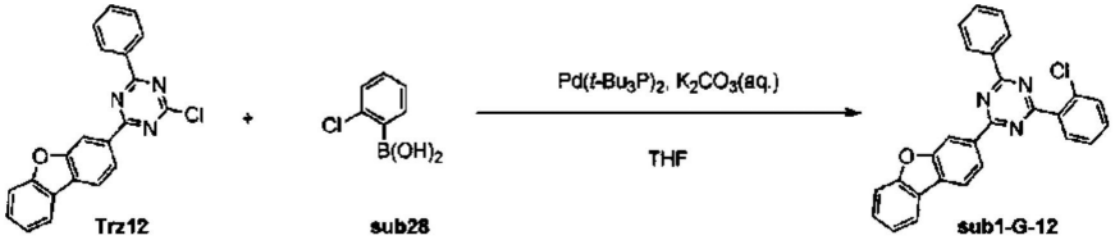
[0623] 

sub1-F-4 sub10 1-59

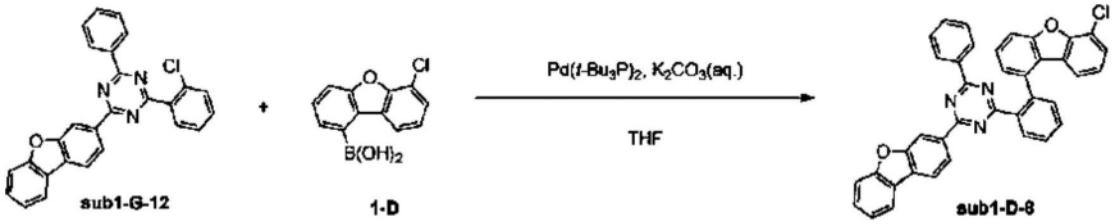
[0624] 在氮气气氛下将化合物sub1-F-4(15g,23.1mmol)和化合物sub10(2.8g,23.1mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(9.6g,69.2mmol)溶解在29ml水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)(0.1g,0.2mmol)。在10小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添

加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到12.1g化合物1-59。(产率:76%,MS:[M+H]$^+$=692)

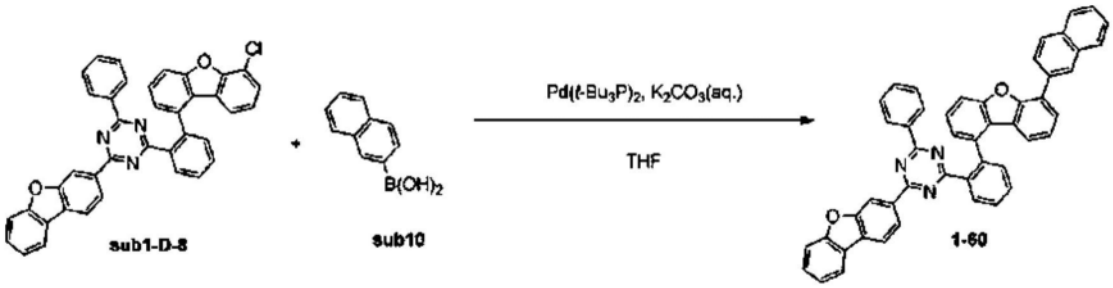
[0625] 制备例1-60:化合物1-60的制备

[0626] 

[0627] 在氮气气氛下将化合物Trz12(15g,41.9mmol)和化合物sub28(6.6g,41.9mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(17.4g,125.8mmol)溶解在52ml水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)(0.2g,0.4mmol)。在10小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到11.1g化合物sub1-G-12。(产率:61%,MS:[M+H]$^+$=434)

[0628] 

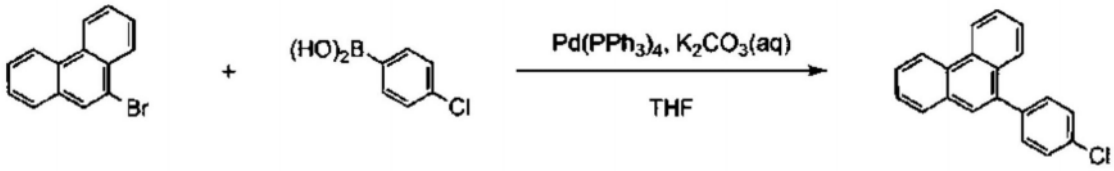
[0629] 在氮气气氛下将化合物sub1-G-12(15g,34.6mmol)和化合物1-D(8.5g,34.6mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(14.3g,103.7mmol)溶解在43ml水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)(0.2g,0.3mmol)。在9小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到13.6g化合物sub1-D-8。(产率:79%,MS:[M+H]$^+$=500)

[0630] 

[0631] 在氮气气氛下将化合物sub1-D-8(15g,25mmol)和化合物sub10(4.3g,25mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(10.4g,75mmol)溶解在31ml水中,添加至混合物中,并将混合物充分搅拌,然后添加双(三叔丁基膦)钯(0)(0.1g,0.2mmol)。在10小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水

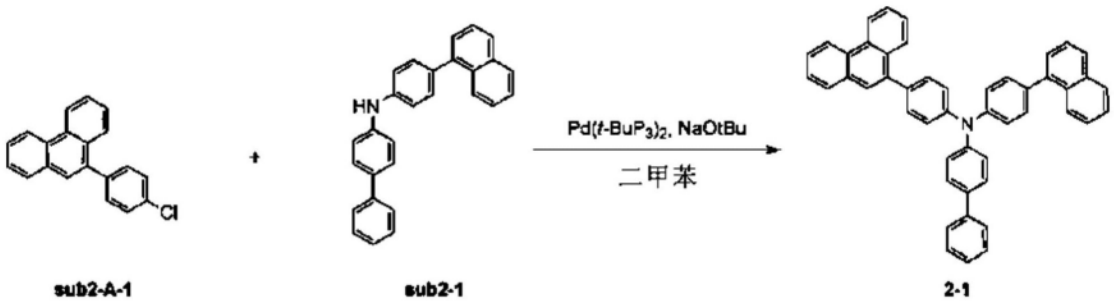
硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到13.3g化合物1-60。(产率:77%,MS:$[M+H]^+$=692)

[0632] 制备例2-1:化合物2-1的制备

[0633] 

2-A 2-B sub2-A-1

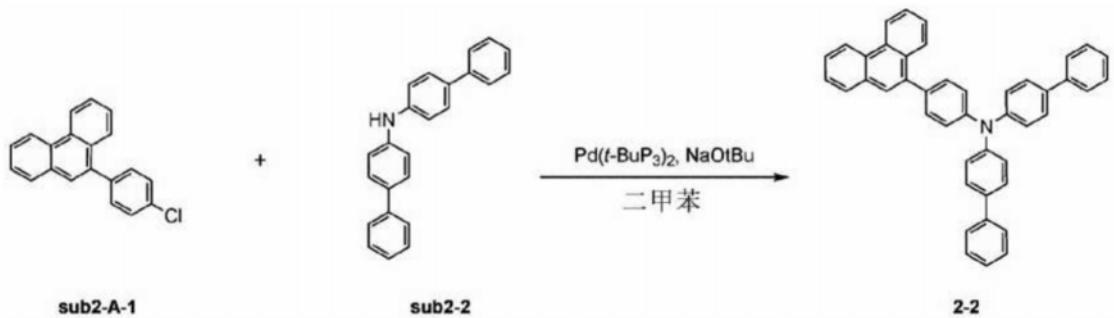
[0634] 在氮气气氛下将化合物2-A(15g,58.3mmol)和化合物2-B(10g,64.2mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(16.1g,116.7mmol)溶解在48ml水中,添加至混合物中,并将混合物充分搅拌,然后添加四(三苯基膦)钯(0)(1.3g,1.2mmol)。在11小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到12.6g化合物sub2-A-1。(产率:75%,MS:$[M+H]^+$=289)

[0635] 

sub2-A-1 sub2-1 2-1

[0636] 在氮气气氛下将化合物sub2-A-1(10g,34.6mmol)、化合物sub2-1(12.9g,34.6mmol)和叔丁醇钠(4.3g,45mmol)添加至200ml二甲苯中,并将混合物搅拌并回流。然后,向其中添加双(三叔丁基膦)钯(0)(0.2g,0.3mmol)。5小时之后,当反应完成时,将反应混合物冷却至室温并在减压下除去溶剂。然后,将化合物再次完全溶解在氯仿中,用水洗涤两次,然后分离有机层,用无水硫酸镁处理,然后过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到12.7g化合物2-1。(产率:59%,MS:$[M+H]^+$=624)

[0637] 制备例2-2:化合物2-2的制备

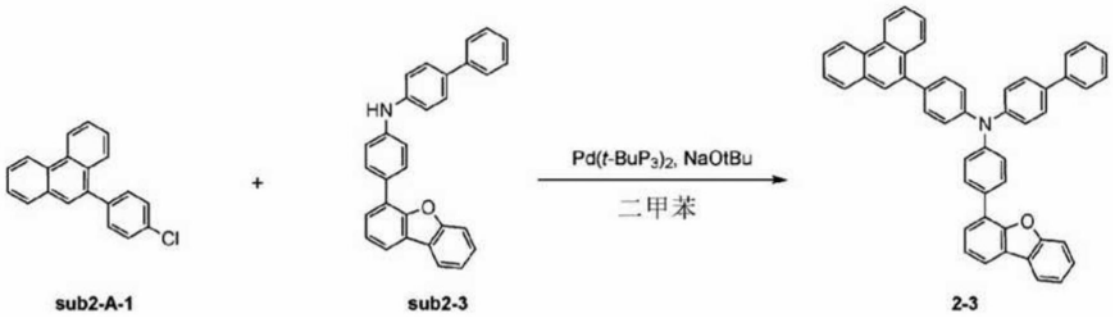
[0638] 

sub2-A-1 sub2-2 2-2

[0639] 在氮气气氛下将化合物sub2-A-1(10g,34.6mmol)、化合物sub2-2(11.1g,34.6mmol)和叔丁醇钠(4.3g,45mmol)添加至200ml二甲苯中,并将混合物搅拌并回流。然

后,向其中添加双(三叔丁基膦)钯(0)(0.2g,0.3mmol)。5小时之后,当反应完成时,将反应混合物冷却至室温并在减压下除去溶剂。然后,将化合物再次完全溶解在氯仿中,用水洗涤两次,然后分离有机层,用无水硫酸镁处理,然后过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到10.1g化合物2-2。(产率:51%,MS:$[M+H]^+$=574)

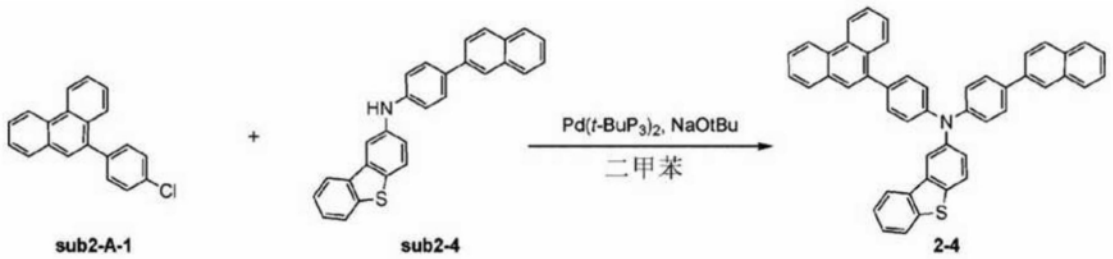
[0640] 制备例2-3:化合物2-3的制备

[0641] 

sub2-A-1 sub2-3 2-3

[0642] 在氮气气氛下将化合物sub2-A-1(10g,34.6mmol)、化合物sub2-3(14.3g,34.6mmol)和叔丁醇钠(4.3g,45mmol)添加至200ml二甲苯中,并将混合物搅拌并回流。然后,向其中添加双(三叔丁基膦)钯(0)(0.2g,0.3mmol)。5小时之后,当反应完成时,将反应混合物冷却至室温并在减压下除去溶剂。然后,将化合物再次完全溶解在氯仿中,用水洗涤两次,然后分离有机层,用无水硫酸镁处理,然后过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到12.2g化合物2-3。(产率:53%,MS:$[M+H]^+$=664)

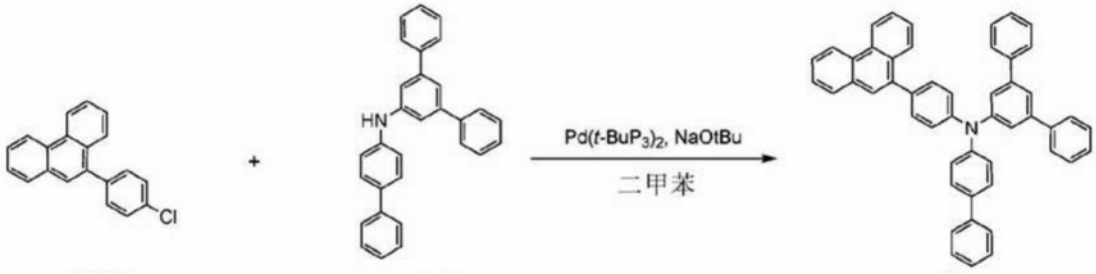
[0643] 制备例2-4:化合物2-4的制备

[0644] 

sub2-A-1 sub2-4 2-4

[0645] 在氮气气氛下将化合物sub2-A-1(10g,34.6mmol)、化合物sub2-4(13.9g,34.6mmol)和叔丁醇钠(4.3g,45mmol)添加至200ml二甲苯中,并将混合物搅拌并回流。然后,向其中添加双(三叔丁基膦)钯(0)(0.2g,0.3mmol)。5小时之后,当反应完成时,将反应混合物冷却至室温并在减压下除去溶剂。然后,将化合物再次完全溶解在氯仿中,用水洗涤两次,然后分离有机层,用无水硫酸镁处理,然后过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到14g化合物2-4。(产率:62%,MS:$[M+H]^+$=654)

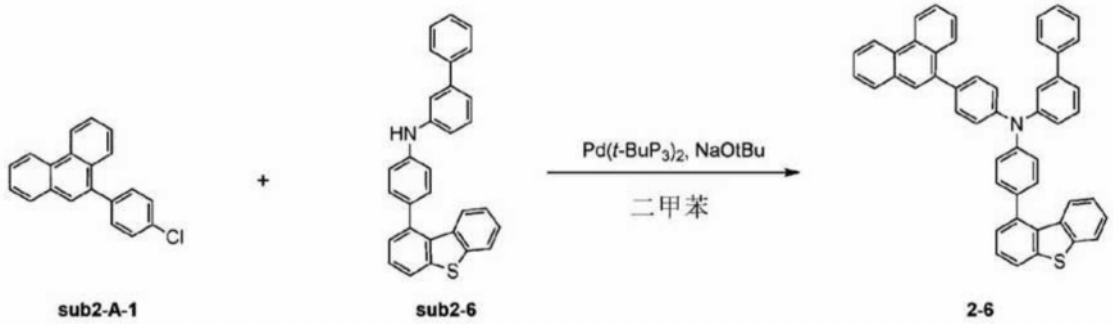
[0646] 制备例2-5:化合物2-5的制备

[0647] 

sub2-A-1 sub2-5 2-5

[0648] 在氮气气氛下将化合物sub2-A-1(10g,34.6mmol)、化合物sub2-5(13.8g,34.6mmol)和叔丁醇钠(4.3g,45mmol)添加至200ml二甲苯中,并将混合物搅拌并回流。然后,向其中添加双(三叔丁基膦)钯(0)(0.2g,0.3mmol)。5小时之后,当反应完成时,将反应混合物冷却至室温并在减压下除去溶剂。然后,将化合物再次完全溶解在氯仿中,用水洗涤两次,然后分离有机层,用无水硫酸镁处理,然后过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到11.2g化合物2-5。(产率:50%,MS:$[M+H]^+$=650)

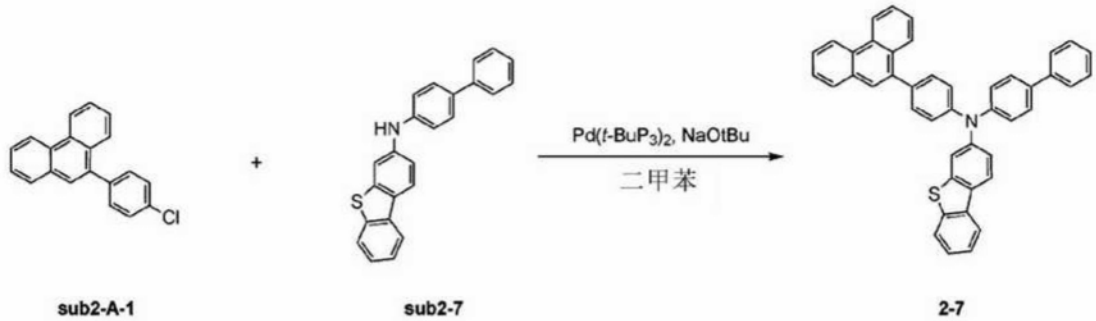
[0649] 制备例2-6:化合物2-6的制备

[0650] 

sub2-A-1 sub2-6 2-6

[0651] 在氮气气氛下将化合物sub2-A-1(10g,34.6mmol)、化合物sub2-6(14.8g,34.6mmol)和叔丁醇钠(4.3g,45mmol)添加至200ml二甲苯中,并将混合物搅拌并回流。然后,向其中添加双(三叔丁基膦)钯(0)(0.2g,0.3mmol)。5小时之后,当反应完成时,将反应混合物冷却至室温并在减压下除去溶剂。然后,将化合物再次完全溶解在氯仿中,用水洗涤两次,然后分离有机层,用无水硫酸镁处理,然后过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到12.2g化合物2-6。(产率:52%,MS:$[M+H]^+$=680)

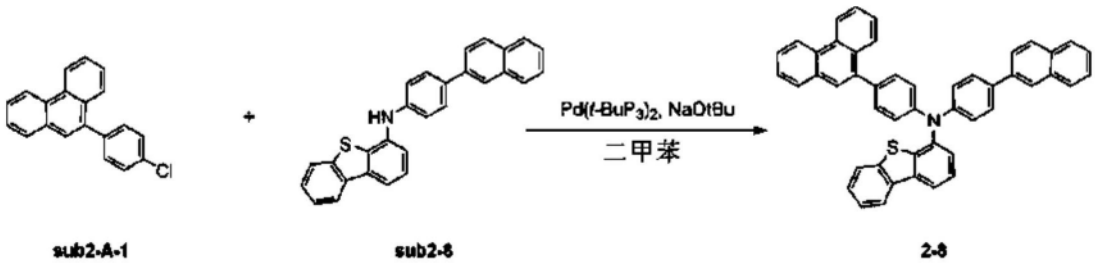
[0652] 制备例2-7:化合物2-7的制备

[0653] 

sub2-A-1 sub2-7 2-7

[0654] 在氮气气氛下将化合物sub2-A-1(10g,34.6mmol)、化合物sub2-7(12.2g,34.6mmol)和叔丁醇钠(4.3g,45mmol)添加至200ml二甲苯中,并将混合物搅拌并回流。然后,向其中添加双(三叔丁基膦)钯(0)(0.2g,0.3mmol)。5小时之后,当反应完成时,将反应混合物冷却至室温并在减压下除去溶剂。然后,将化合物再次完全溶解在氯仿中,用水洗涤两次,然后分离有机层,用无水硫酸镁处理,然后过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到1g化合物2-7。(产率:50%,MS:$[M+H]^+$=61)

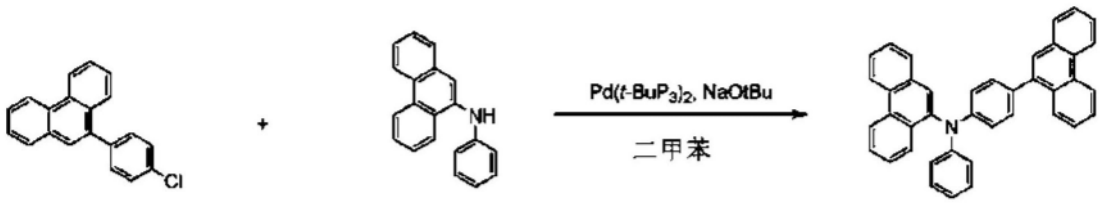
[0655] 制备例2-8:化合物2-8的制备

[0656] 

sub2-A-1 sub2-8 2-8

[0657] 在氮气气氛下将化合物sub2-A-1(10g,34.6mmol)、化合物sub2-8(13.9g,34.6mmol)和叔丁醇钠(4.3g,45mmol)添加至200ml二甲苯中,并将混合物搅拌并回流。然后,向其中添加双(三叔丁基膦)钯(0)(0.2g,0.3mmol)。5小时之后,当反应完成时,将反应混合物冷却至室温并在减压下除去溶剂。然后,将化合物再次完全溶解在氯仿中,用水洗涤两次,然后分离有机层,用无水硫酸镁处理,然后过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到13.3g化合物2-8。(产率:59%,MS:$[M+H]^+$=654)

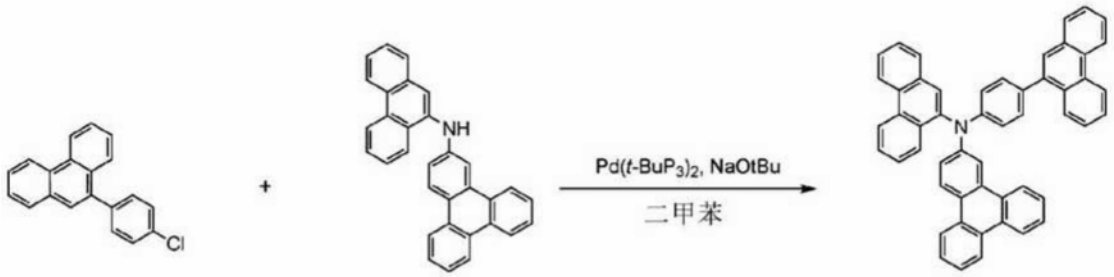
[0658] 制备例2-9:化合物2-9的制备

[0659] 

sub2-A-1 sub2-9 2-9

[0660] 在氮气气氛下将化合物sub2-A-1(10g,34.6mmol)、化合物sub2-9(9.3g,34.6mmol)和叔丁醇钠(4.3g,45mmol)添加至200ml二甲苯中,并将混合物搅拌并回流。然后,向其中添加双(三叔丁基膦)钯(0)(0.2g,0.3mmol)。5小时之后,当反应完成时,将反应混合物冷却至室温并在减压下除去溶剂。然后,将化合物再次完全溶解在氯仿中,用水洗涤两次,然后分离有机层,用无水硫酸镁处理,然后过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到11.2g化合物2-9。(产率:62%,MS:$[M+H]^+$=522)

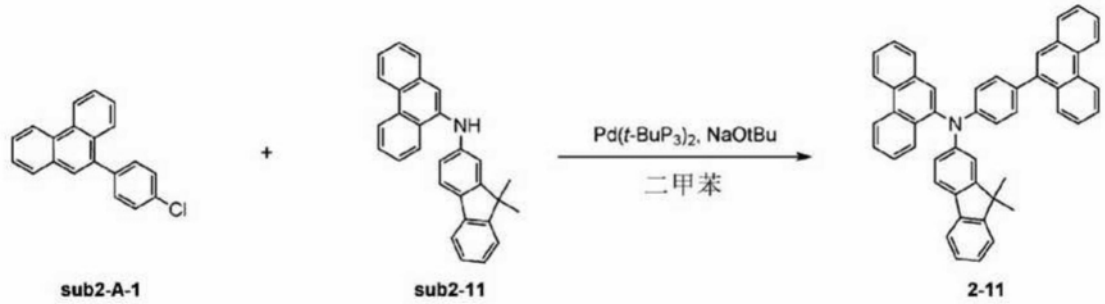
[0661] 制备例2-10:化合物2-10的制备

[0662] 

sub2-A-1 sub2-10 2-10

[0663] 在氮气气氛下将化合物sub2-A-1(10g,34.6mmol)、化合物sub2-10(14.5g,34.6mmol)和叔丁醇钠(4.3g,45mmol)添加至200ml二甲苯中,并将混合物搅拌并回流。然后,向其中添加双(三叔丁基膦)钯(0)(0.2g,0.3mmol)。5小时之后,当反应完成时,将反应混合物冷却至室温并在减压下除去溶剂。然后,将化合物再次完全溶解在氯仿中,用水洗涤两次,然后分离有机层,用无水硫酸镁处理,然后过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到14.4g化合物2-10。(产率:62%,MS:$[M+H]^+$=572)

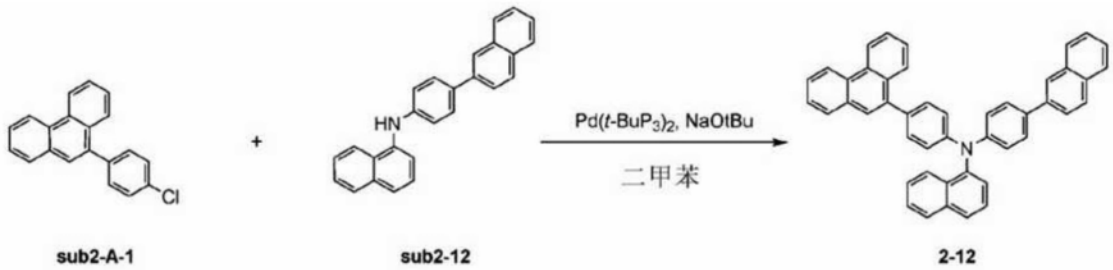
[0664] 制备例2-11:化合物2-11的制备

[0665] 

sub2-A-1 sub2-11 2-11

[0666] 在氮气气氛下将化合物sub2-A-1(10g,34.6mmol)、化合物sub2-11(13.4g,34.6mmol)和叔丁醇钠(4.3g,45mmol)添加至200ml二甲苯中,并将混合物搅拌并回流。然后,向其中添加双(三叔丁基膦)钯(0)(0.2g,0.3mmol)。5小时之后,当反应完成时,将反应混合物冷却至室温并在减压下除去溶剂。然后,将化合物再次完全溶解在氯仿中,用水洗涤两次,然后分离有机层,用无水硫酸镁处理,然后过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到12.4g化合物2-11。(产率:56%,MS:$[M+H]^+$=638)

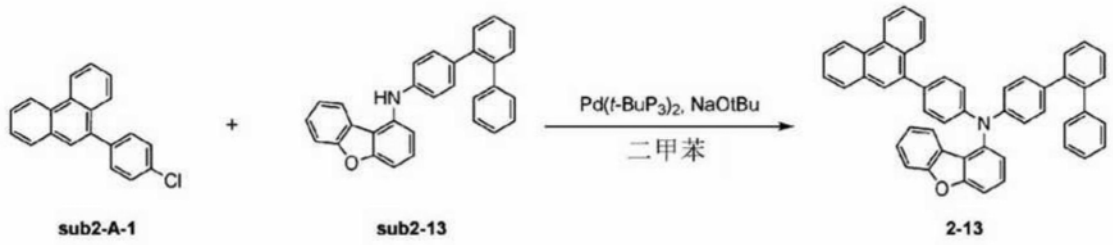
[0667] 制备例2-12:化合物2-12的制备

[0668] 

sub2-A-1 sub2-12 2-12

[0669] 在氮气气氛下将化合物sub2-A-1(10g,34.6mmol)、化合物sub2-12(12g,34.6mmol)和叔丁醇钠(4.3g,45mmol)添加至200ml二甲苯中,并将混合物搅拌并回流。然后,向其中添加双(三叔丁基膦)钯(0)(0.2g,0.3mmol)。5小时之后,当反应完成时,将反应混合物冷却至室温并在减压下除去溶剂。然后,将化合物再次完全溶解在氯仿中,用水洗涤两次,然后分离有机层,用无水硫酸镁处理,然后过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到11g化合物2-12。(产率:53%,MS:$[M+H]^+$=598)

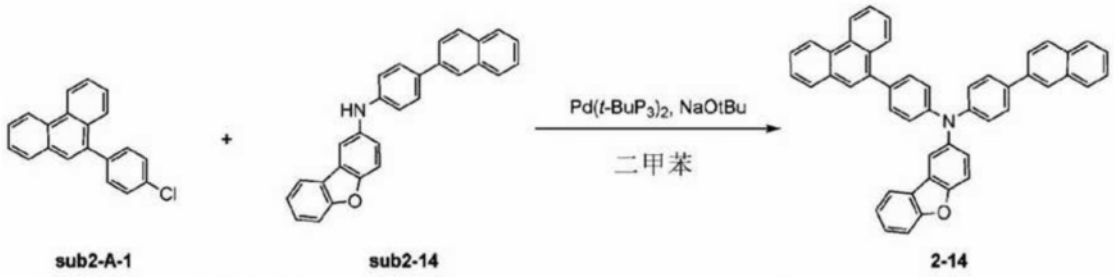
[0670] 制备例2-13:化合物2-13的制备

[0671] 

sub2-A-1 sub2-13 2-13

[0672] 在氮气气氛下将化合物sub2-A-1(10g,34.6mmol)、化合物sub2-13(14.3g,34.6mmol)和叔丁醇钠(4.3g,45mmol)添加至200ml二甲苯中,并将混合物搅拌并回流。然后,向其中添加双(三叔丁基膦)钯(0)(0.2g,0.3mmol)。5小时之后,当反应完成时,将反应混合物冷却至室温并在减压下除去溶剂。然后,将化合物再次完全溶解在氯仿中,用水洗涤两次,然后分离有机层,用无水硫酸镁处理,然后过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到15.6g化合物2-13。(产率:68%,MS:$[M+H]^+$=664)

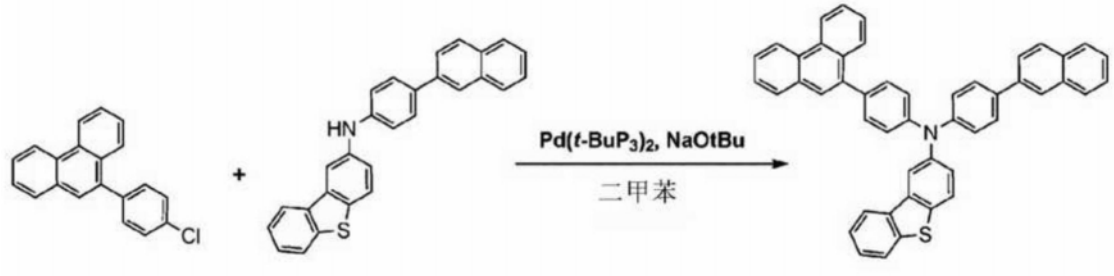
[0673] 制备例2-14:化合物2-14的制备

[0674] 

sub2-A-1 sub2-14 2-14

[0675] 在氮气气氛下将化合物sub2-A-1(10g,34.6mmol)、化合物sub2-14(13.3g,34.6mmol)和叔丁醇钠(4.3g,45mmol)添加至200ml二甲苯中,并将混合物搅拌并回流。然后,向其中添加双(三叔丁基膦)钯(0)(0.2g,0.3mmol)。5小时之后,当反应完成时,将反应混合物冷却至室温并在减压下除去溶剂。然后,将化合物再次完全溶解在氯仿中,用水洗涤两次,然后分离有机层,用无水硫酸镁处理,然后过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到13.2g化合物2-14。(产率:60%,MS:$[M+H]^+$=638)

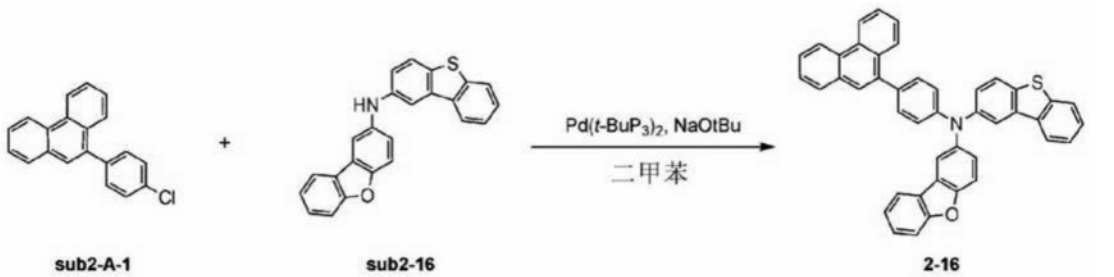
[0676] 制备例2-15:化合物2-15的制备

[0677] 

sub2-A-1 sub2-15 2-15

[0678] 在氮气气氛下将化合物sub2-A-1(10g,34.6mmol)、化合物sub2-15(13.9g,34.6mmol)和叔丁醇钠(3.7g,38.1mmol)添加至200ml二甲苯中,并将混合物搅拌并回流。然后,向其中添加双(三叔丁基膦)钯(0)(0.2g,0.3mmol)。5小时之后,当反应完成时,将反应混合物冷却至室温并在减压下除去溶剂。然后,将化合物再次完全溶解在氯仿中,用水洗涤两次,然后分离有机层,用无水硫酸镁处理,然后过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到12g化合物2-15。(产率:53%,MS:$[M+H]^+$=654)

[0679] 制备例2-16:化合物2-16的制备

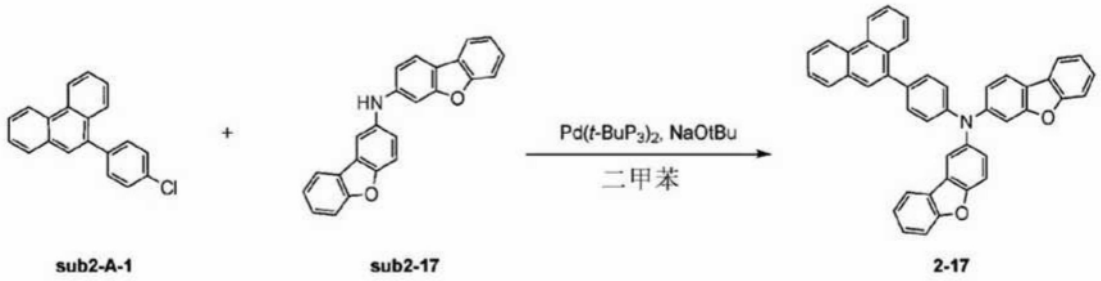
[0680] 

sub2-A-1 sub2-16 2-16

[0681] 在氮气气氛下将化合物sub2-A-1(10g,34.6mmol)、化合物sub2-16(12.7g,34.6mmol)和叔丁醇钠(4.3g,45mmol)添加至200ml二甲苯中,并将混合物搅拌并回流。然后,向其中添加双(三叔丁基膦)钯(0)(0.2g,0.3mmol)。5小时之后,当反应完成时,将反应混合物冷却至室温并在减压下除去溶剂。然后,将化合物再次完全溶解在氯仿中,用水洗涤两次,然后分离有机层,用无水硫酸镁处理,然后过滤,并将滤液在减压下蒸馏。通过硅胶柱

色谱法将浓缩的化合物纯化以得到13.7g化合物2-16。(产率:64%,MS:[M+H]$^+$=618)

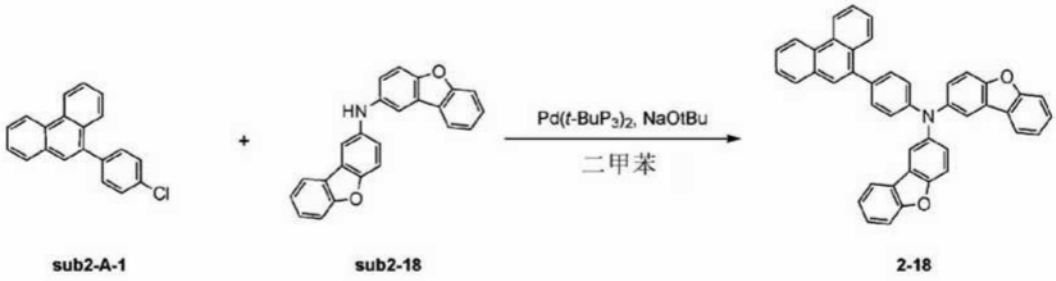
[0682] 制备例2-17:化合物2-17的制备

[0683] 

sub2-A-1 sub2-17 2-17

[0684] 在氮气气氛下将化合物sub2-A-1(10g,34.6mmol)、化合物sub2-17(12.1g,34.6mmol)和叔丁醇钠(4.3g,45mmol)添加至200ml二甲苯中,并将混合物搅拌并回流。然后,向其中添加双(三叔丁基膦)钯(0)(0.2g,0.3mmol)。5小时之后,当反应完成时,将反应混合物冷却至室温并在减压下除去溶剂。然后,将化合物再次完全溶解在氯仿中,用水洗涤两次,然后分离有机层,用无水硫酸镁处理,然后过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到11.5g化合物2-17。(产率:55%,MS:[M+H]$^+$=602)

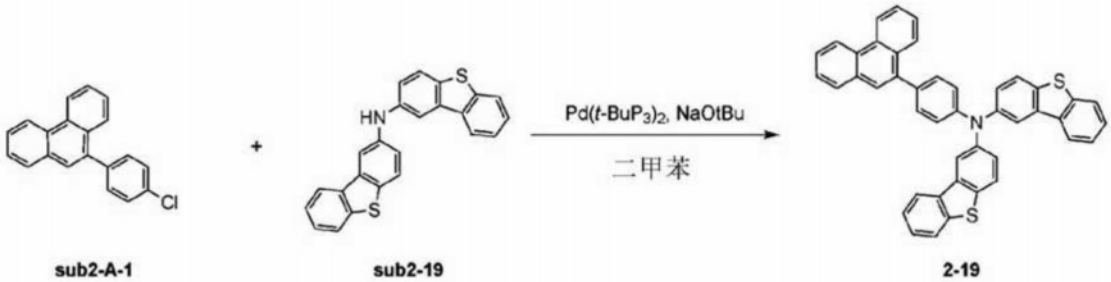
[0685] 制备例2-18:化合物2-18的制备

[0686] 

sub2-A-1 sub2-18 2-18

[0687] 在氮气气氛下将化合物sub2-A-1(10g,34.6mmol)、化合物sub2-18(12.1g,34.6mmol)和叔丁醇钠(4.3g,45mmol)添加至200ml二甲苯中,并将混合物搅拌并回流。然后,向其中添加双(三叔丁基膦)钯(0)(0.2g,0.3mmol)。5小时之后,当反应完成时,将反应混合物冷却至室温并在减压下除去溶剂。然后,将化合物再次完全溶解在氯仿中,用水洗涤两次,然后分离有机层,用无水硫酸镁处理,然后过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到14.4g化合物2-18。(产率:69%,MS:[M+H]$^+$=602)

[0688] 制备例2-19:化合物2-19的制备

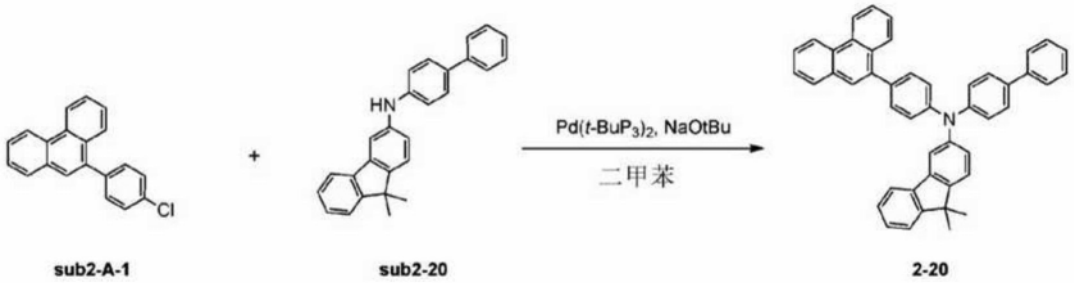
[0689] 

sub2-A-1 sub2-19 2-19

[0690] 在氮气气氛下将化合物sub2-A-1(10g,34.6mmol)、化合物sub2-19(13.2g,34.6mmol)和叔丁醇钠(4.3g,45mmol)添加至200ml二甲苯中,并将混合物搅拌并回流。然后,向其中添加双(三叔丁基膦)钯(0)(0.2g,0.3mmol)。5小时之后,当反应完成时,将反应混合物冷却至室温并在减压下除去溶剂。然后,将化合物再次完全溶解在氯仿中,用水洗涤

两次,然后分离有机层,用无水硫酸镁处理,然后过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到11.4g化合物2-19。(产率:52%,MS:[M+H]$^+$=634)

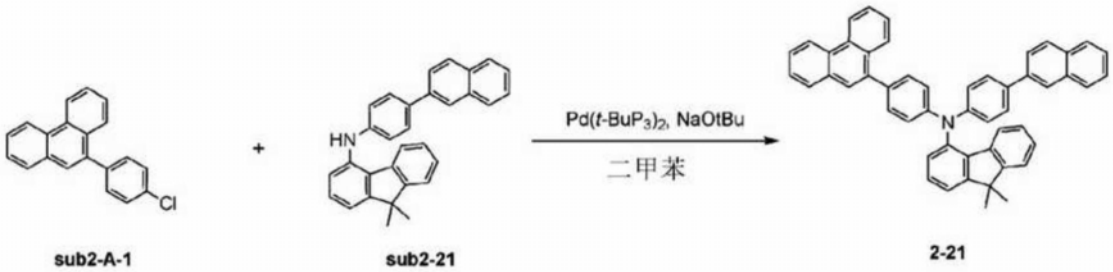
[0691] 制备例2-20:化合物2-20的制备

[0692] 

sub2-A-1 sub2-20 2-20

[0693] 在氮气气氛下将化合物sub2-A-1(10g,34.6mmol)、化合物sub2-20(12.5g,34.6mmol)和叔丁醇钠(4.3g,45mmol)添加至200ml二甲苯中,并将混合物搅拌并回流。然后,向其中添加双(三叔丁基膦)钯(0)(0.2g,0.3mmol)。5小时之后,当反应完成时,将反应混合物冷却至室温并在减压下除去溶剂。然后,将化合物再次完全溶解在氯仿中,用水洗涤两次,然后分离有机层,用无水硫酸镁处理,然后过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到13.2g化合物2-20。(产率:62%,MS:[M+H]$^+$=614)

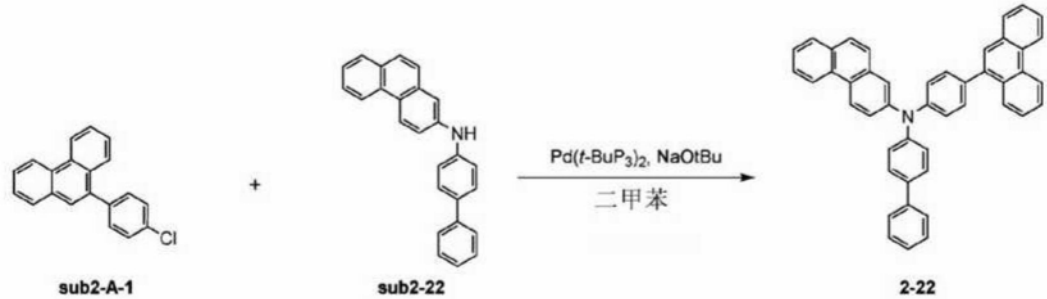
[0694] 制备例2-21:化合物2-21的制备

[0695] 

sub2-A-1 sub2-21 2-21

[0696] 在氮气气氛下将化合物sub2-A-1(10g,34.6mmol)、化合物sub2-21(14.3g,34.6mmol)和叔丁醇钠(4.3g,45mmol)添加至200ml二甲苯中,并将混合物搅拌并回流。然后,向其中添加双(三叔丁基膦)钯(0)(0.2g,0.3mmol)。5小时之后,当反应完成时,将反应混合物冷却至室温并在减压下除去溶剂。然后,将化合物再次完全溶解在氯仿中,用水洗涤两次,然后分离有机层,用无水硫酸镁处理,然后过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到14.2g化合物2-21。(产率:62%,MS:[M+H]$^+$=664)

[0697] 制备例2-22:化合物2-22的制备

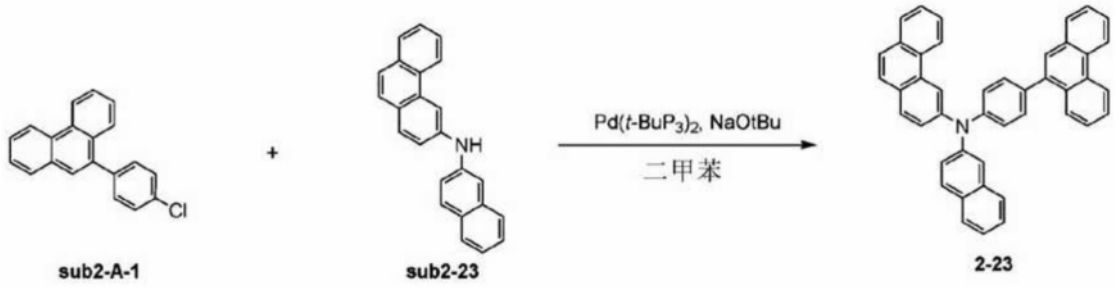
[0698] 

sub2-A-1 sub2-22 2-22

[0699] 在氮气气氛下将化合物sub2-A-1(10g,34.6mmol)、化合物sub2-22(12g,34.6mmol)和叔丁醇钠(4.3g,45mmol)添加至200ml二甲苯中,并将混合物搅拌并回流。然后,向其中添加双(三叔丁基膦)钯(0)(0.2g,0.3mmol)。5小时之后,当反应完成时,将反应

混合物冷却至室温并在减压下除去溶剂。然后,将化合物再次完全溶解在氯仿中,用水洗涤两次,然后分离有机层,用无水硫酸镁处理,然后过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到11.2g化合物2-22。(产率:54%,MS:$[M+H]^+$=598)

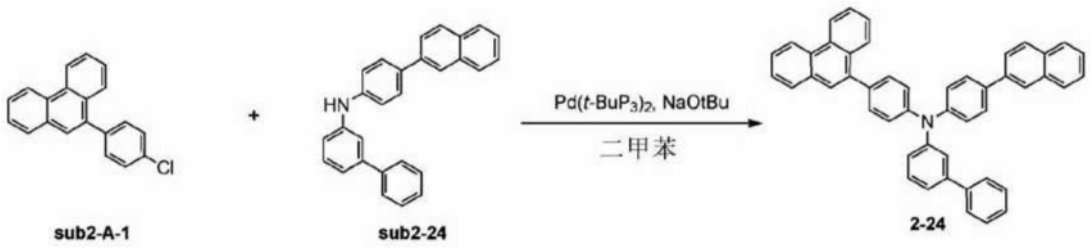
[0700] 制备例2-23:化合物2-23的制备

[0701] 

sub2-A-1 sub2-23 2-23

[0702] 在氮气气氛下将化合物sub2-A-1(10g,34.6mmol)、化合物sub2-23(11.1g,34.6mmol)和叔丁醇钠(4.3g,45mmol)添加至200ml二甲苯中,并将混合物搅拌并回流。然后,向其中添加双(三叔丁基膦)钯(0)(0.2g,0.3mmol)。5小时之后,当反应完成时,将反应混合物冷却至室温并在减压下除去溶剂。然后,将化合物再次完全溶解在氯仿中,用水洗涤两次,然后分离有机层,用无水硫酸镁处理,然后过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到11.9g化合物2-23。(产率:60%,MS:$[M+H]^+$=572)

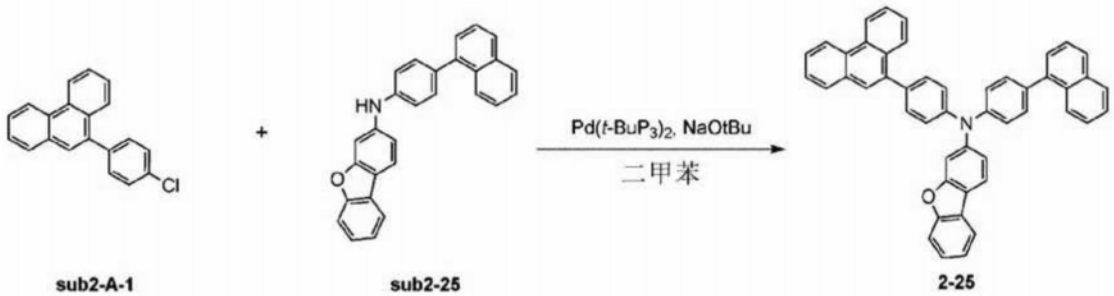
[0703] 制备例2-24:化合物2-24的制备

[0704] 

sub2-A-1 sub2-24 2-24

[0705] 在氮气气氛下将化合物sub2-A-1(10g,34.6mmol)、化合物sub2-24(12.9g,34.6mmol)和叔丁醇钠(4.3g,45mmol)添加至200ml二甲苯中,并将混合物搅拌并回流。然后,向其中添加双(三叔丁基膦)钯(0)(0.2g,0.3mmol)。5小时之后,当反应完成时,将反应混合物冷却至室温并在减压下除去溶剂。然后,将化合物再次完全溶解在氯仿中,用水洗涤两次,然后分离有机层,用无水硫酸镁处理,然后过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到13.6g化合物2-24。(产率:63%,MS:$[M+H]^+$=624)

[0706] 制备例2-25:化合物2-25的制备

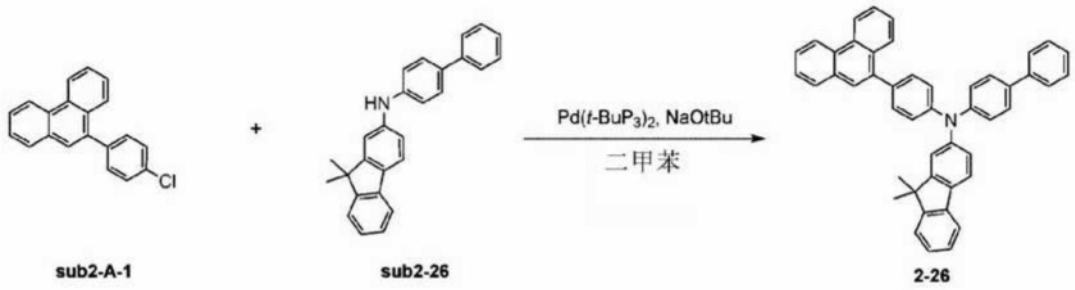
[0707] 

sub2-A-1 sub2-25 2-25

[0708] 在氮气气氛下将化合物sub2-A-1(10g,34.6mmol)、化合物sub2-25(13.3g,34.6mmol)和叔丁醇钠(4.3g,45mmol)添加至200ml二甲苯中,并将混合物搅拌并回流。然

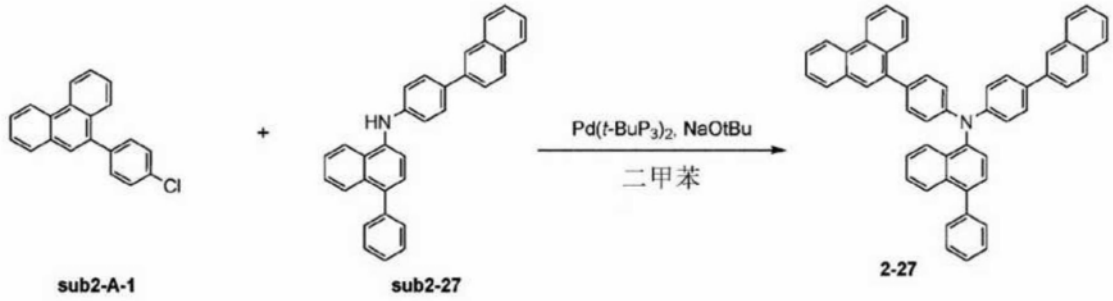
后,向其中添加双(三叔丁基膦)钯(0)(0.2g,0.3mmol)。5小时之后,当反应完成时,将反应混合物冷却至室温并在减压下除去溶剂。然后,将化合物再次完全溶解在氯仿中,用水洗涤两次,然后分离有机层,用无水硫酸镁处理,然后过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到14.3g化合物2-25。(产率:65%,MS:$[M+H]^+$=638)

[0709] 制备例2-26:化合物2-26的制备

[0710] 

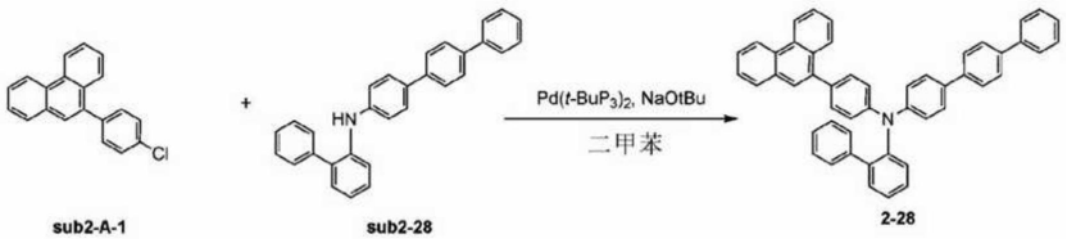
[0711] 在氮气气氛下将化合物sub2-A-1(10g,34.6mmol)、化合物sub2-26(12.5g,34.6mmol)和叔丁醇钠(4.3g,45mmol)添加至200ml二甲苯中,并将混合物搅拌并回流。然后,向其中添加双(三叔丁基膦)钯(0)(0.2g,0.3mmol)。5小时之后,当反应完成时,将反应混合物冷却至室温并在减压下除去溶剂。然后,将化合物再次完全溶解在氯仿中,用水洗涤两次,然后分离有机层,用无水硫酸镁处理,然后过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到10.8g化合物2-26。(产率:51%,MS:$[M+H]^+$=614)

[0712] 制备例2-27:化合物2-27的制备

[0713] 

[0714] 在氮气气氛下将化合物sub2-A-1(10g,34.6mmol)、化合物sub2-27(14.6g,34.6mmol)和叔丁醇钠(4.3g,45mmol)添加至200ml二甲苯中,并将混合物搅拌并回流。然后,向其中添加双(三叔丁基膦)钯(0)(0.2g,0.3mmol)。5小时之后,当反应完成时,将反应混合物冷却至室温并在减压下除去溶剂。然后,将化合物再次完全溶解在氯仿中,用水洗涤两次,然后分离有机层,用无水硫酸镁处理,然后过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到16.1g化合物2-27。(产率:69%,MS:$[M+H]^+$=674)

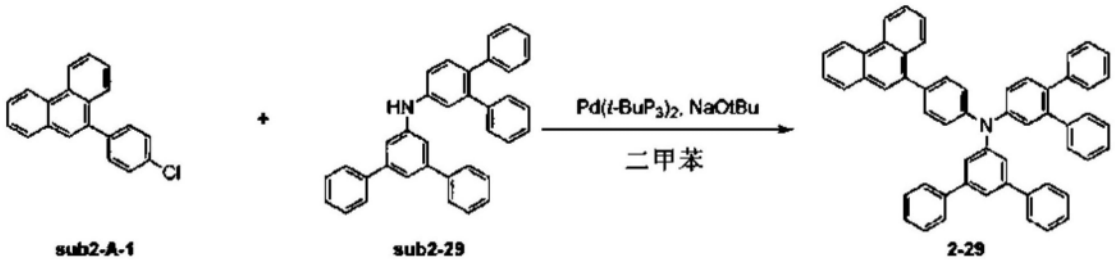
[0715] 制备例2-28:化合物2-28的制备

[0716] 

[0717] 在氮气气氛下将化合物sub2-A-1(10g,34.6mmol)、化合物sub2-28(13.8g,

34.6mmol)和叔丁醇钠(4.3g,45mmol)添加至200ml二甲苯中,并将混合物搅拌并回流。然后,向其中添加双(三叔丁基膦)钯(0)(0.2g,0.3mmol)。5小时之后,当反应完成时,将反应混合物冷却至室温并在减压下除去溶剂。然后,将化合物再次完全溶解在氯仿中,用水洗涤两次,然后分离有机层,用无水硫酸镁处理,然后过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到11.2g化合物2-28。(产率:50%,MS:[M+H]$^+$=650)

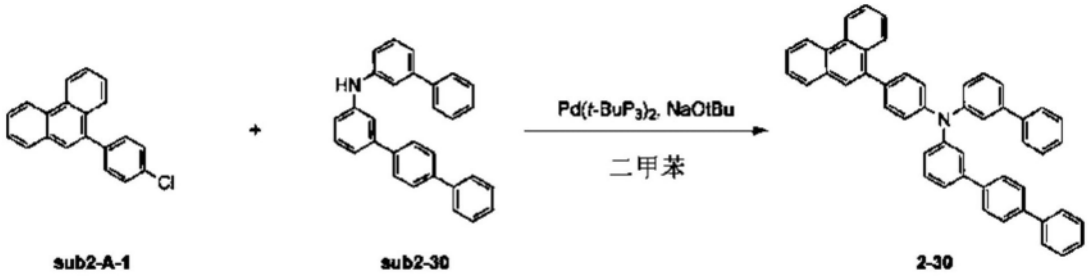
[0718] 制备例2-29:化合物2-29的制备

[0719] 

sub2-A-1 sub2-29 2-29

[0720] 在氮气气氛下将化合物sub2-A-1(10g,34.6mmol)、化合物sub2-29(16.4g,34.6mmol)和叔丁醇钠(4.3g,45mmol)添加至200ml二甲苯中,并将混合物搅拌并回流。然后,向其中添加双(三叔丁基膦)钯(0)(0.2g,0.3mmol)。5小时之后,当反应完成时,将反应混合物冷却至室温并在减压下除去溶剂。然后,将化合物再次完全溶解在氯仿中,用水洗涤两次,然后分离有机层,用无水硫酸镁处理,然后过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到17.1g化合物2-29。(产率:68%,MS:[M+H]$^+$=726)

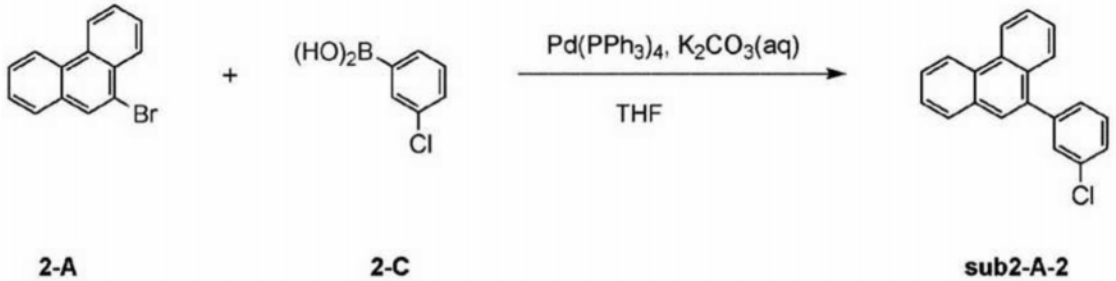
[0721] 制备例2-30:化合物2-30的制备

[0722] 

sub2-A-1 sub2-30 2-30

[0723] 在氮气气氛下将化合物sub2-A-1(10g,34.6mmol)、化合物sub2-30(13.8g,34.6mmol)和叔丁醇钠(4.3g,45mmol)添加至200ml二甲苯中,并将混合物搅拌并回流。然后,向其中添加双(三叔丁基膦)钯(0)(0.2g,0.3mmol)。5小时之后,当反应完成时,将反应混合物冷却至室温并在减压下除去溶剂。然后,将化合物再次完全溶解在氯仿中,用水洗涤两次,然后分离有机层,用无水硫酸镁处理,然后过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到14.4g化合物2-30。(产率:64%,MS:[M+H]$^+$=650)

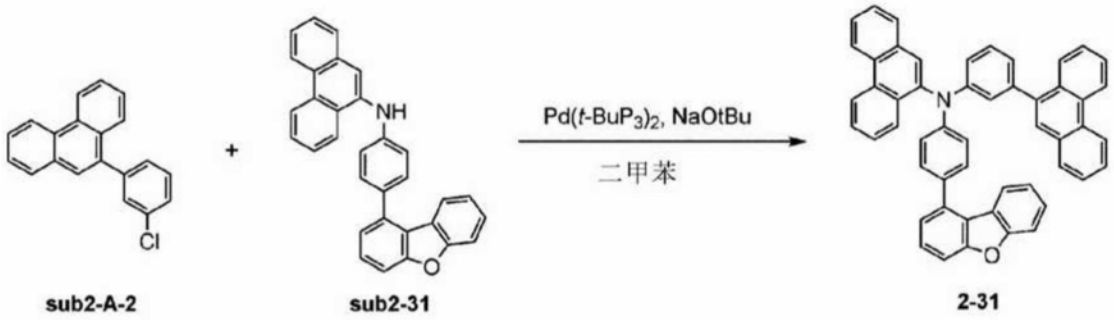
[0724] 制备例2-31:化合物2-31的制备

[0725] 

2-A 2-C sub2-A-2

[0726] 在氮气气氛下将化合物2-A(15g,58.3mmol)和化合物2-C(10g,64.2mmol)添加至

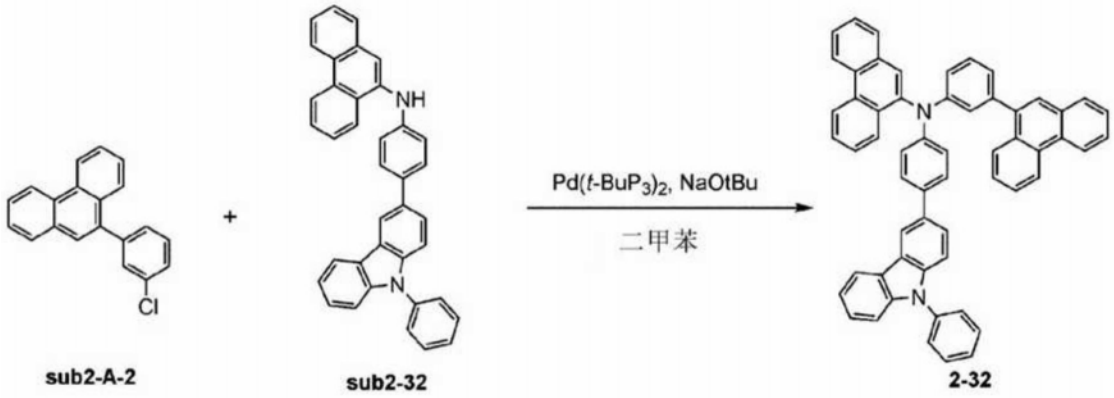
300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(16.1g,116.7mmol)溶解在48ml水中,添加至混合物中,并将混合物充分搅拌,然后添加四(三苯基膦)钯(0)(1.3g,1.2mmol)。在10小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到10.6g化合物sub2-A-2。(产率:63%,MS:$[M+H]^+$=289)

[0727] 

sub2-A-2 sub2-31 2-31

[0728] 在氮气气氛下将化合物sub2-A-2(10g,34.6mmol)、化合物sub2-31(15.1g,34.6mmol)和叔丁醇钠(4.3g,45mmol)添加至200ml二甲苯中,并将混合物搅拌并回流。然后,向其中添加双(三叔丁基膦)钯(0)(0.2g,0.3mmol)。5小时之后,当反应完成时,将反应混合物冷却至室温并在减压下除去溶剂。然后,将化合物再次完全溶解在氯仿中,用水洗涤两次,然后分离有机层,用无水硫酸镁处理,然后过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到16.7g化合物2-31。(产率:70%,MS:$[M+H]^+$=688)

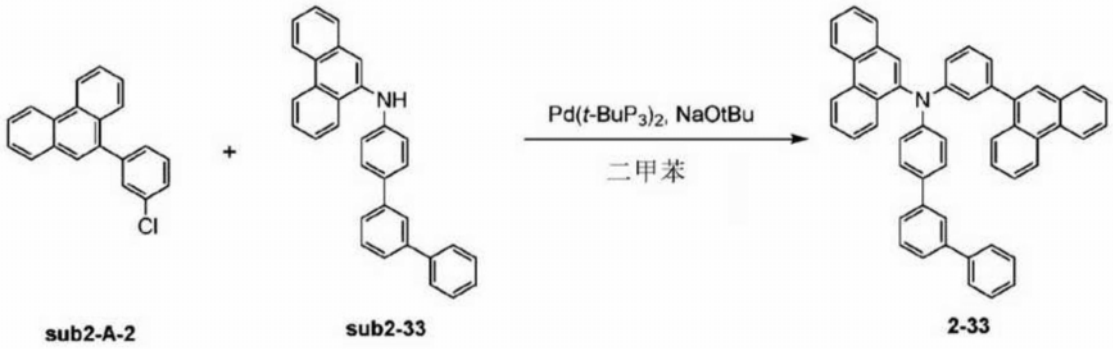
[0729] 制备例2-32:化合物2-32的制备

[0730] 

sub2-A-2 sub2-32 2-32

[0731] 在氮气气氛下将化合物sub2-A-2(10g,34.6mmol)、化合物sub2-32(17.7g,34.6mmol)和叔丁醇钠(4.3g,45mmol)添加至200ml二甲苯中,并将混合物搅拌并回流。然后,向其中添加双(三叔丁基膦)钯(0)(0.2g,0.3mmol)。5小时之后,当反应完成时,将反应混合物冷却至室温并在减压下除去溶剂。然后,将化合物再次完全溶解在氯仿中,用水洗涤两次,然后分离有机层,用无水硫酸镁处理,然后过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到16.6g化合物2-32。(产率:63%,MS:$[M+H]^+$=763)

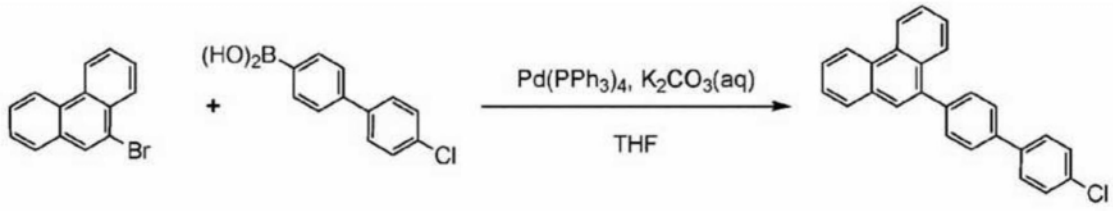
[0732] 制备例2-33:化合物2-33的制备

[0733] 

sub2-A-2 sub2-33 2-33

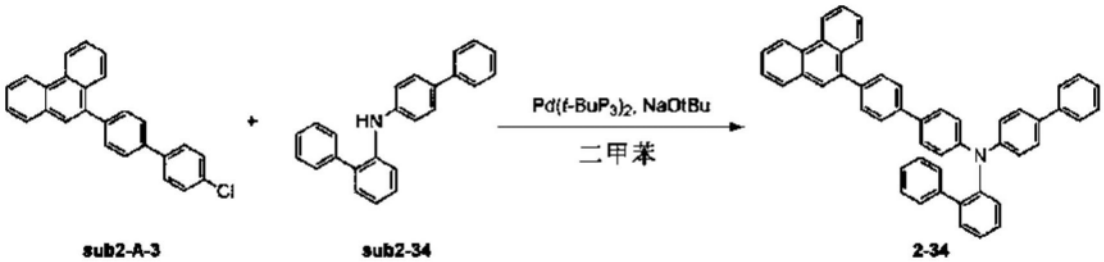
[0734] 在氮气气氛下将化合物sub2-A-2(10g,34.6mmol)、化合物sub2-33(14.6g,34.6mmol)和叔丁醇钠(4.3g,45mmol)添加至200ml二甲苯中,并将混合物搅拌并回流。然后,向其中添加双(三叔丁基膦)钯(0)(0.2g,0.3mmol)。5小时之后,当反应完成时,将反应混合物冷却至室温并在减压下除去溶剂。然后,将化合物再次完全溶解在氯仿中,用水洗涤两次,然后分离有机层,用无水硫酸镁处理,然后过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到12.6g化合物2-33。(产率:54%,MS:$[M+H]^+$=674)

[0735] 制备例2-34:化合物2-34的制备

[0736] 

2-A 2-D sub2-A-3

[0737] 在氮气气氛下将化合物2-A(15g,58.3mmol)和化合物2-D(14.9g,64.2mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(16.1g,116.7mmol)溶解在48ml水中,添加至混合物中,并将混合物充分搅拌,然后添加四(三苯基膦)钯(0)(1.3g,1.2mmol)。在10小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到16.8g化合物sub2-A-3。(产率:79%,MS:$[M+H]^+$=365)

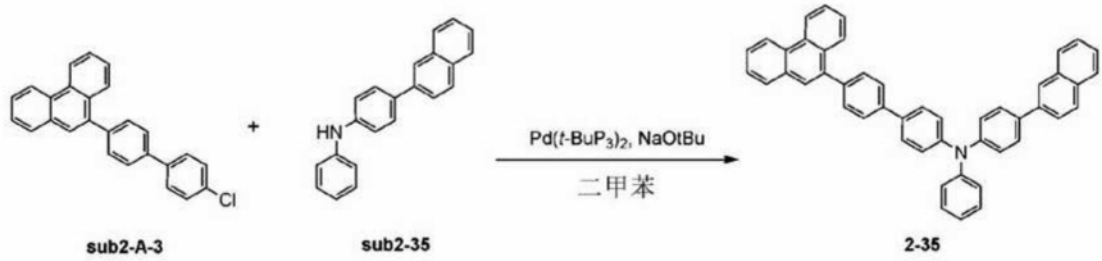
[0738] 

sub2-A-3 sub2-34 2-34

[0739] 在氮气气氛下将化合物sub2-A-3(10g,27.4mmol)、化合物sub2-34(8.8g,27.4mmol)和叔丁醇钠(3.4g,35.7mmol)添加至200ml二甲苯中,并将混合物搅拌并回流。然后,向其中添加双(三叔丁基膦)钯(0)(0.1g,0.3mmol)。5小时之后,当反应完成时,将反应混合物冷却至室温并在减压下除去溶剂。然后,将化合物再次完全溶解在氯仿中,用水洗涤两次,然后分离有机层,用无水硫酸镁处理,然后过滤,并将滤液在减压下蒸馏。通过硅胶柱

色谱法将浓缩的化合物纯化以得到11.2g化合物2-34。(产率:63%,MS:[M+H]$^+$=650)

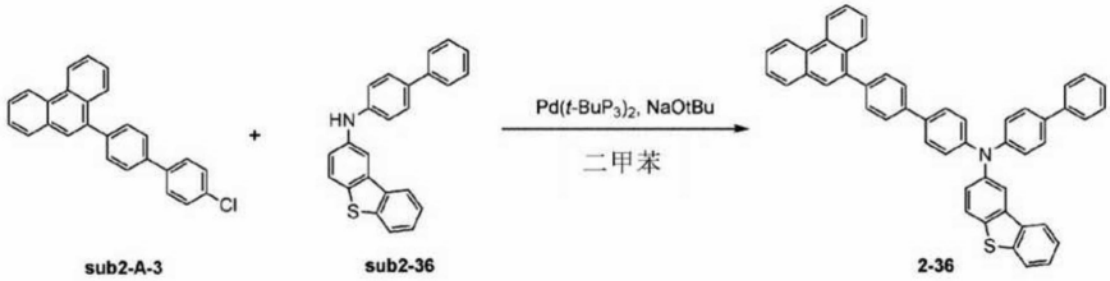
[0740] 制备例2-35:化合物2-35的制备

[0741] 

sub2-A-3 sub2-35 2-35

[0742] 在氮气气氛下将化合物sub2-A-3(10g,27.4mmol)、化合物sub2-35(8.1g,27.4mmol)和叔丁醇钠(3.4g,35.7mmol)添加至200ml二甲苯中,并将混合物搅拌并回流。然后,向其中添加双(三叔丁基膦)钯(0)(0.1g,0.3mmol)。5小时之后,当反应完成时,将反应混合物冷却至室温并在减压下除去溶剂。然后,将化合物再次完全溶解在氯仿中,用水洗涤两次,然后分离有机层,用无水硫酸镁处理,然后过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到8.7g化合物2-35。(产率:51%,MS:[M+H]$^+$=624)

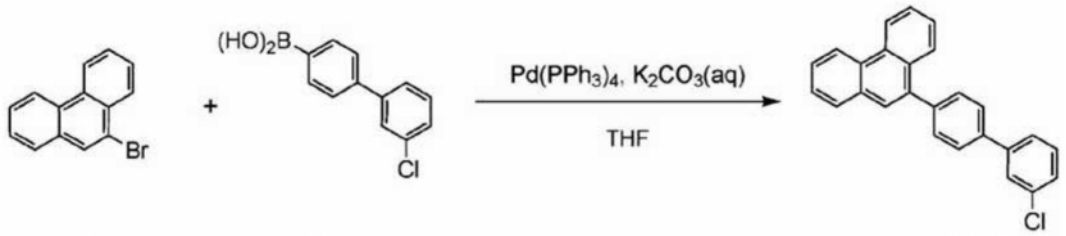
[0743] 制备例2-36:化合物2-36的制备

[0744] 

sub2-A-3 sub2-36 2-36

[0745] 在氮气气氛下将化合物sub2-A-3(10g,27.4mmol)、化合物sub2-36(9.6g,27.4mmol)和叔丁醇钠(3.4g,35.6mmol)添加至200ml二甲苯中,并将混合物搅拌并回流。然后,向其中添加双(三叔丁基膦)钯(0)(0.1g,0.3mmol)。5小时之后,当反应完成时,将反应混合物冷却至室温并在减压下除去溶剂。然后,将化合物再次完全溶解在氯仿中,用水洗涤两次,然后分离有机层,用无水硫酸镁处理,然后过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到12.1g化合物2-36。(产率:65%,MS:[M+H]$^+$=680)

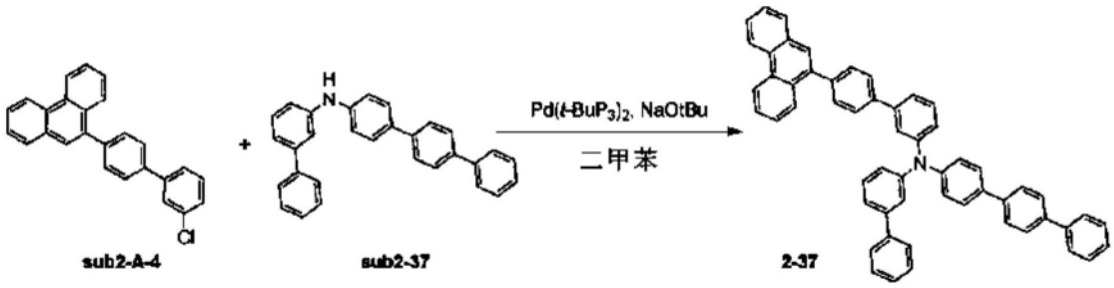
[0746] 制备例2-37:化合物2-37的制备

[0747] 

2-A 2-E sub2-A-4

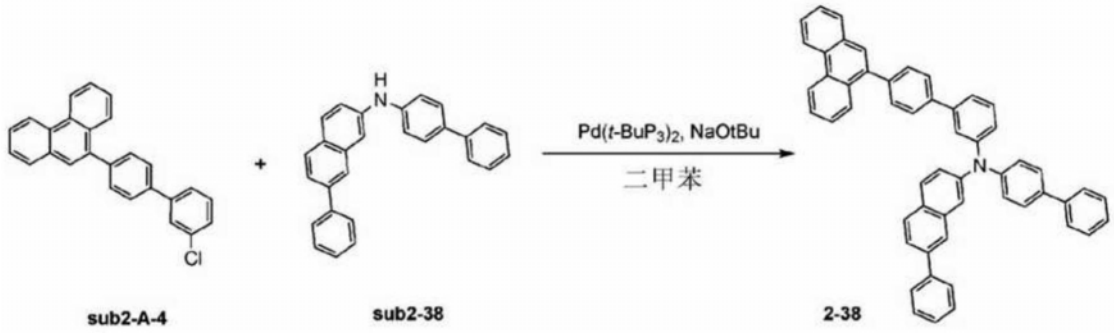
[0748] 在氮气气氛下将化合物2-A(15g,58.3mmol)和化合物2-E(14.9g,64.2mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(16.1g,116.7mmol)溶解在48ml水中,添加至混合物中,并将混合物充分搅拌,然后添加四(三苯基膦)钯(0)(1.3g,1.2mmol)。在11小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将

混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到14.2g化合物sub2-A-4。(产率:67%,MS:[M+H]$^+$=365)

[0749] 

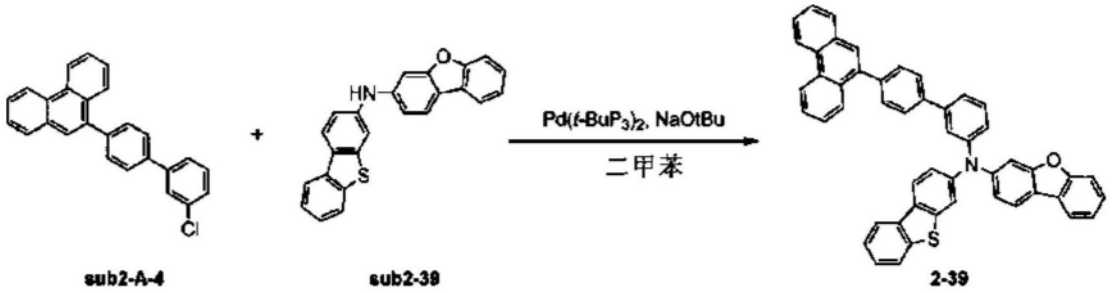
[0750] 在氮气气氛下将化合物sub2-A-4(10g,27.4mmol)、化合物sub2-37(10.9g,27.4mmol)和叔丁醇钠(3.4g,35.6mmol)添加至200ml二甲苯中,并将混合物搅拌并回流。然后,向其中添加双(三叔丁基膦)钯(0)(0.1g,0.3mmol)。5小时之后,当反应完成时,将反应混合物冷却至室温并在减压下除去溶剂。然后,将化合物再次完全溶解在氯仿中,用水洗涤两次,然后分离有机层,用无水硫酸镁处理,然后过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到13.9g化合物2-37。(产率:70%,MS:[M+H]$^+$=726)

[0751] 制备例2-38:化合物2-38的制备

[0752] 

[0753] 在氮气气氛下将化合物sub2-A-4(10g,27.4mmol)、化合物sub2-38(10.2g,27.4mmol)和叔丁醇钠(3.4g,35.6mmol)添加至200ml二甲苯中,并将混合物搅拌并回流。然后,向其中添加双(三叔丁基膦)钯(0)(0.1g,0.3mmol)。5小时之后,当反应完成时,将反应混合物冷却至室温并在减压下除去溶剂。然后,将化合物再次完全溶解在氯仿中,用水洗涤两次,然后将有机层分离,用无水硫酸镁处理,然后过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到10.7g化合物2-38。(产率:56%,MS:[M+H]$^+$=700)

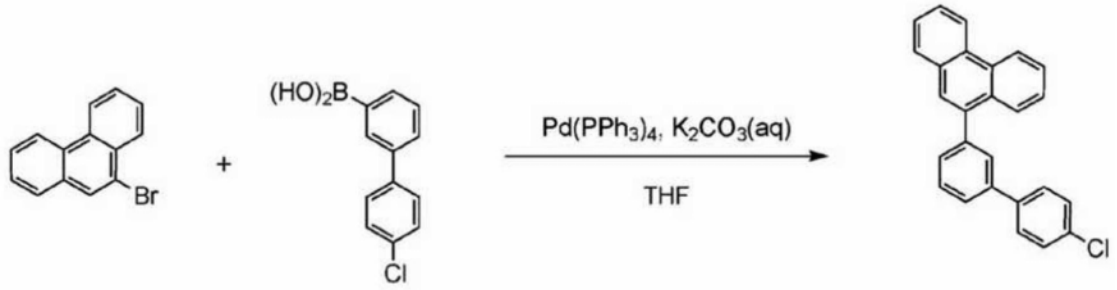
[0754] 制备例2-39:化合物2-39的制备

[0755] 

[0756] 在氮气气氛下将化合物sub2-A-4(10g,27.4mmol)、化合物sub2-39(10g,27.4mmol)和叔丁醇钠(3.4g,35.6mmol)添加至200ml二甲苯中,并将混合物搅拌并回流。然后,向其中添加双(三叔丁基膦)钯(0)(0.1g,0.3mmol)。5小时之后,当反应完成时,将反应

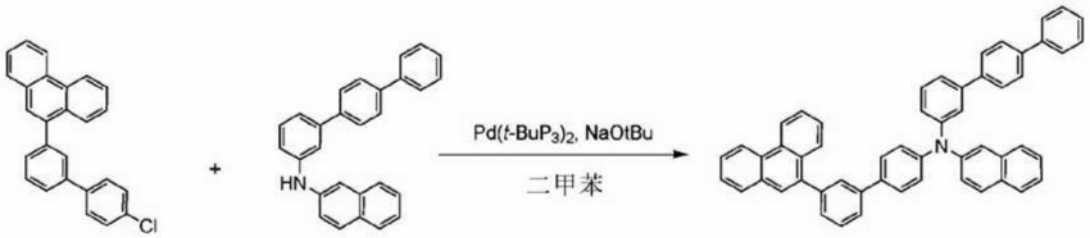
混合物冷却至室温并在减压下除去溶剂。然后,将化合物再次完全溶解在氯仿中,用水洗涤两次,然后分离有机层,用无水硫酸镁处理,然后过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到11.8g化合物2-39。(产率:62%,MS:[M+H]$^+$=694)

[0757] 制备例2-40:化合物2-40的制备

[0758] 

2-A 2-F sub2-A-5

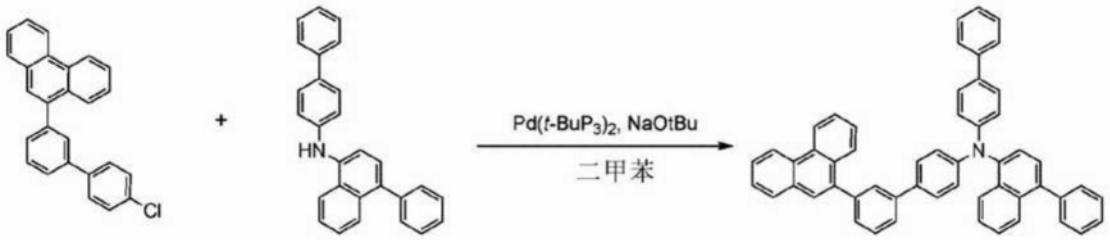
[0759] 在氮气气氛下将化合物2-A(15g,58.3mmol)和化合物2-F(14.9g,64.2mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(16.1g,116.7mmol)溶解在48ml水中,添加至混合物中,并将混合物充分搅拌,然后添加四(三苯基膦)钯(0)(1.3g,1.2mmol)。在8小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到14.4g化合物sub2-A-5。(产率:68%,MS:[M+H]$^+$=365)

[0760] 

sub2-A-5 sub2-40 2-40

[0761] 在氮气气氛下将化合物sub2-A-5(10g,27.4mmol)、化合物sub2-40(10.2g,27.4mmol)和叔丁醇钠(3.4g,35.6mmol)添加至200ml二甲苯中,并将混合物搅拌并回流。然后,向其中添加双(三叔丁基膦)钯(0)(0.1g,0.3mmol)。5小时之后,当反应完成时,将反应混合物冷却至室温并在减压下除去溶剂。然后,将化合物再次完全溶解在氯仿中,用水洗涤两次,然后分离有机层,用无水硫酸镁处理,然后过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到10.7g化合物2-40。(产率:56%,MS:[M+H]$^+$=700)

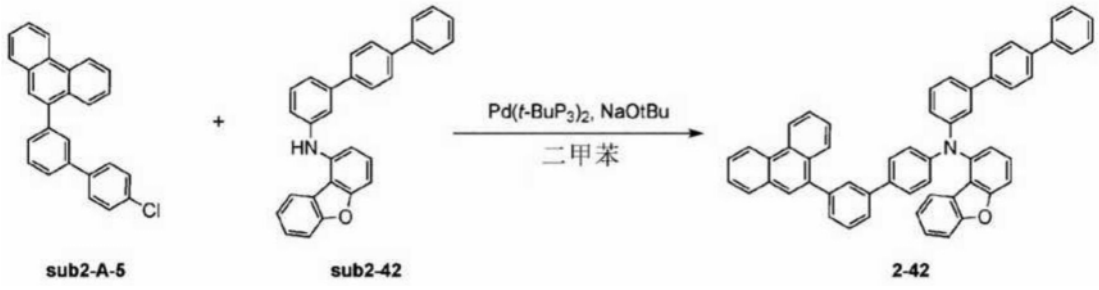
[0762] 制备例2-41:化合物2-41的制备

[0763] 

sub2-A-5 sub2-41 2-41

[0764] 在氮气气氛下将化合物sub2-A-5(10g,27.4mmol)、化合物sub2-41(10.2g,27.4mmol)和叔丁醇钠(3.4g,35.6mmol)添加至200ml二甲苯中,并将混合物搅拌并回流。然后,向其中添加双(三叔丁基膦)钯(0)(0.1g,0.3mmol)。5小时之后,当反应完成时,将反应混合物冷却至室温并在减压下除去溶剂。然后,将化合物再次完全溶解在氯仿中,用水洗涤两次,然后分离有机层,用无水硫酸镁处理,然后过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到9.8g化合物2-41。(产率:51%,MS:$[M+H]^+$=700)

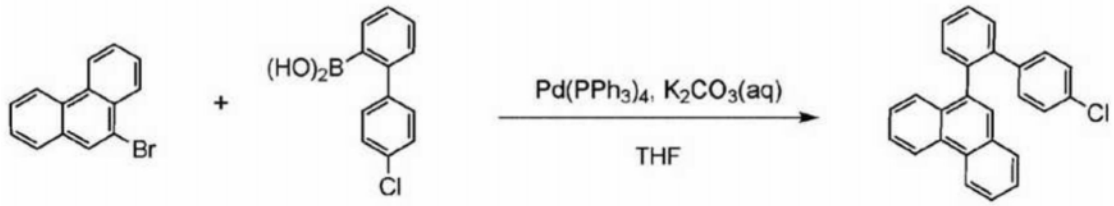
[0765] 制备例2-42:化合物2-42的制备

[0766] 

sub2-A-5 sub2-42 2-42

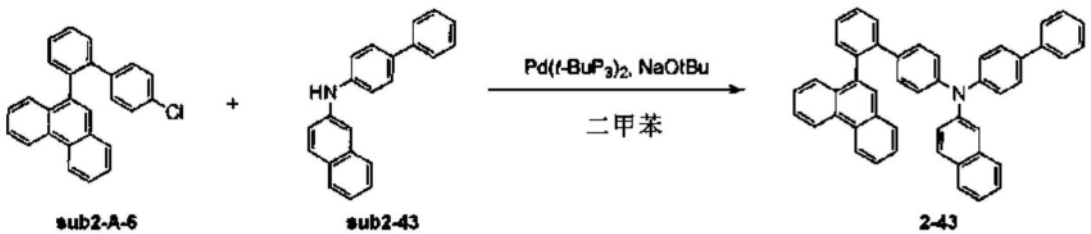
[0767] 在氮气气氛下将化合物sub2-A-5(10g,27.4mmol)、化合物sub2-42(11.3g,27.4mmol)和叔丁醇钠(3.4g,35.6mmol)添加至200ml二甲苯中,并将混合物搅拌并回流。然后,向其中添加双(三叔丁基膦)钯(0)(0.1g,0.3mmol)。5小时之后,当反应完成时,将反应混合物冷却至室温并在减压下除去溶剂。然后,将化合物再次完全溶解在氯仿中,用水洗涤两次,然后分离有机层,用无水硫酸镁处理,然后过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到11.5g化合物2-42。(产率:57%,MS:$[M+H]^+$=740)

[0768] 制备例2-43:化合物2-43的制备

[0769] 

2-A 2-G sub2-A-6

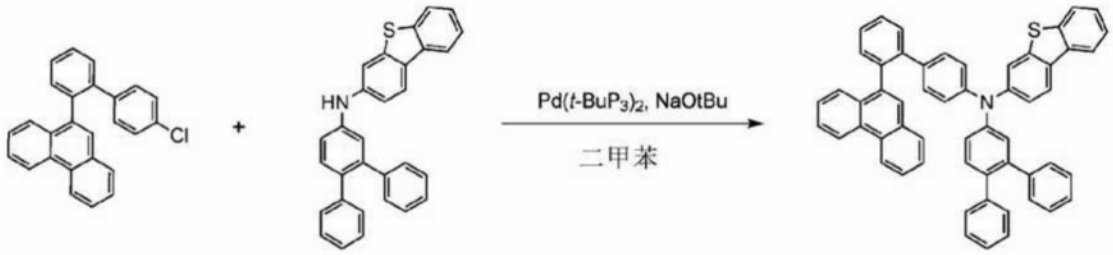
[0770] 在氮气气氛下将化合物2-A(15g,58.3mmol)和化合物2-G(14.9g,64.2mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(16.1g,116.7mmol)溶解在48ml水中,添加至混合物中,并将混合物充分搅拌,然后添加四(三苯基膦)钯(0)(1.3g,1.2mmol)。在9小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到14.7g化合物sub2-A-6。(产率:69%,MS:$[M+H]^+$=365)

[0771] 

sub2-A-6 sub2-43 2-43

[0772] 在氮气气氛下将化合物sub2-A-6(10g,27.4mmol)、化合物sub2-43(8.1g,27.4mmol)和叔丁醇钠(3.4g,35.6mmol)添加至200ml二甲苯中,并将混合物搅拌并回流。然后,向其中添加双(三叔丁基膦)钯(0)(0.1g,0.3mmol)。5小时之后,当反应完成时,将反应混合物冷却至室温并在减压下除去溶剂。然后,将化合物再次完全溶解在氯仿中,用水洗涤两次,然后分离有机层,用无水硫酸镁处理,然后过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到9.7g化合物2-43。(产率:57%,MS:[M+H]$^+$=624)

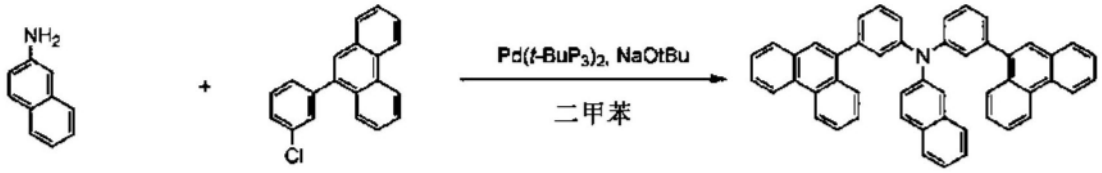
[0773] 制备例2-44:化合物2-44的制备

[0774] 

sub2-A-6 sub2-44 2-44

[0775] 在氮气气氛下将化合物sub2-A-6(10g,27.4mmol)、化合物sub2-44(11.7g,27.4mmol)和叔丁醇钠(3.4g,35.6mmol)添加至200ml二甲苯中,并将混合物搅拌并回流。然后,向其中添加双(三叔丁基膦)钯(0)(0.1g,0.3mmol)。5小时之后,当反应完成时,将反应混合物冷却至室温并在减压下除去溶剂。然后,将化合物再次完全溶解在氯仿中,用水洗涤两次,然后分离有机层,用无水硫酸镁处理,然后过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到12g化合物2-44。(产率:58%,MS:[M+H]$^+$=756)

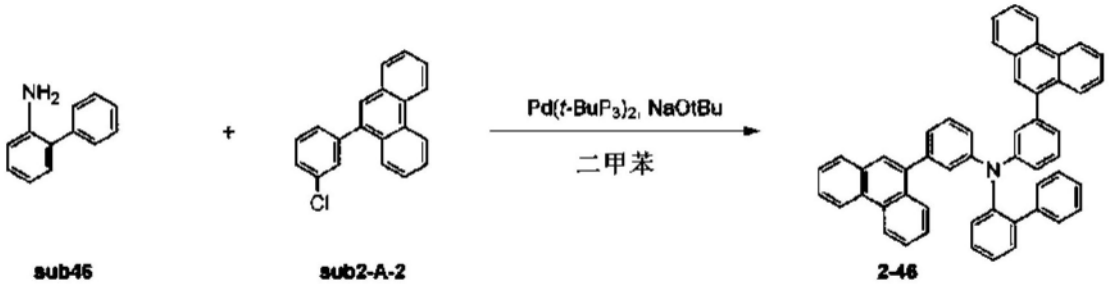
[0776] 制备例2-45:化合物2-45的制备

[0777] 

sub45 sub2-A-2 2-45

[0778] 在氮气气氛下将化合物sub45(10g,70.3mmol)、化合物sub2-A-2(42.6g,147.7mmol)和叔丁醇钠(16.9g,175.8mmol)添加至200ml二甲苯中,并将混合物搅拌并回流。然后,向其中添加双(三叔丁基膦)钯(0)(0.7g,1.4mmol)。5小时之后,当反应完成时,将反应混合物冷却至室温并在减压下除去溶剂。然后,将化合物再次完全溶解在氯仿中,用水洗涤两次,然后分离有机层,用无水硫酸镁处理,然后过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到31g化合物2-45。(产率:68%,MS:[M+H]$^+$=648)

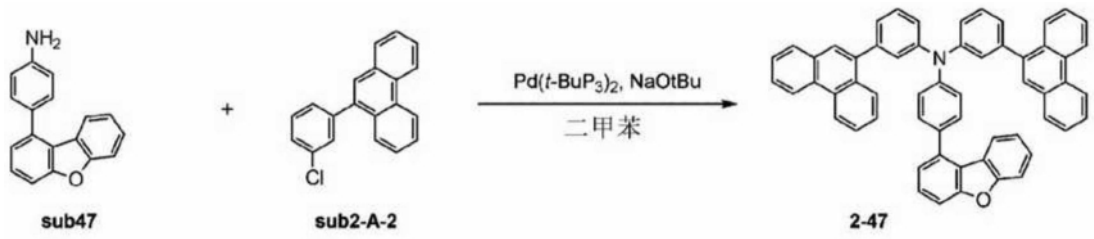
[0779] 制备例2-46:化合物2-46的制备

[0780] 

sub46 sub2-A-2 2-46

[0781] 在氮气气氛下将化合物sub46(10g,59.1mmol)、化合物sub2-A-2(35.8g,124.1mmol)和叔丁醇钠(14.2g,147.7mmol)添加至200ml二甲苯中,并将混合物搅拌并回流。然后,向其中添加双(三叔丁基膦)钯(0)(0.6g,1.2mmol)。5小时之后,当反应完成时,将反应混合物冷却至室温并在减压下除去溶剂。然后,将化合物再次完全溶解在氯仿中,用水洗涤两次,然后分离有机层,用无水硫酸镁处理,然后过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到26.7g化合物2-46。(产率:67%,MS:$[M+H]^+$=674)

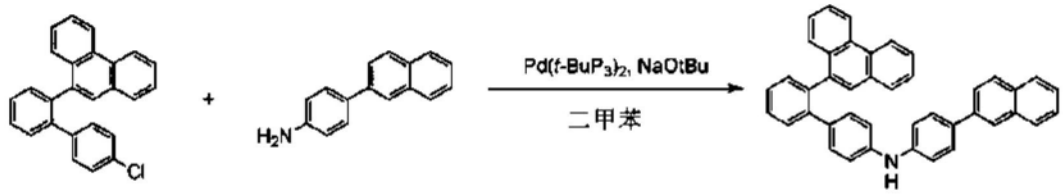
[0782] 制备例2-47:化合物2-47的制备

[0783] 

sub47 sub2-A-2 2-47

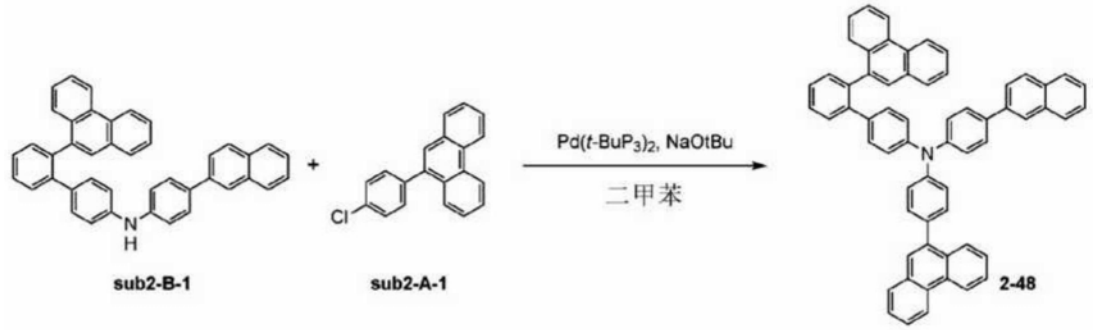
[0784] 在氮气气氛下将化合物sub47(10g,38.6mmol)、化合物sub2-A-2(23.4g,81mmol)和叔丁醇钠(9.3g,96.4mmol)添加至200ml二甲苯中,并将混合物搅拌并回流。然后,向其中添加双(三叔丁基膦)钯(0)(0.4g,0.8mmol)。5小时之后,当反应完成时,将反应混合物冷却至室温并在减压下除去溶剂。然后,将化合物再次完全溶解在氯仿中,用水洗涤两次,然后将有机层分离,用无水硫酸镁处理,然后过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到15g化合物2-47。(产率:51%,MS:$[M+H]^+$=764)

[0785] 制备例2-48:化合物2-48的制备

[0786] 

sub2-A-6 sub48 sub2-B-1

[0787] 在氮气气氛下将化合物sub2-A-6(10g,27.4mmol)、化合物sub48(6g,27.4mmol)和叔丁醇钠(2.9g,30.1mmol)添加至200ml二甲苯中,并将混合物搅拌并回流。然后,向其中添加双(三叔丁基膦)钯(0)(0.1g,0.3mmol)。5小时之后,当反应完成时,将反应混合物冷却至室温并在减压下除去溶剂。然后,将化合物再次完全溶解在氯仿中,用水洗涤两次,然后分离有机层,用无水硫酸镁处理,然后过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到9g化合物sub2-B-1。(产率:60%,MS:$[M+H]^+$=548)

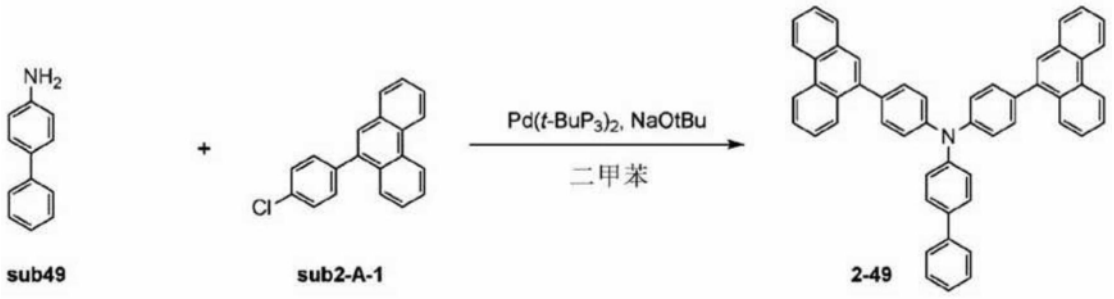
[0788] 

sub2-B-1 sub2-A-1 2-48

[0789] 在氮气气氛下将化合物sub2-B-1(10g,18.3mmol)、化合物sub2-A-1(5.3g,

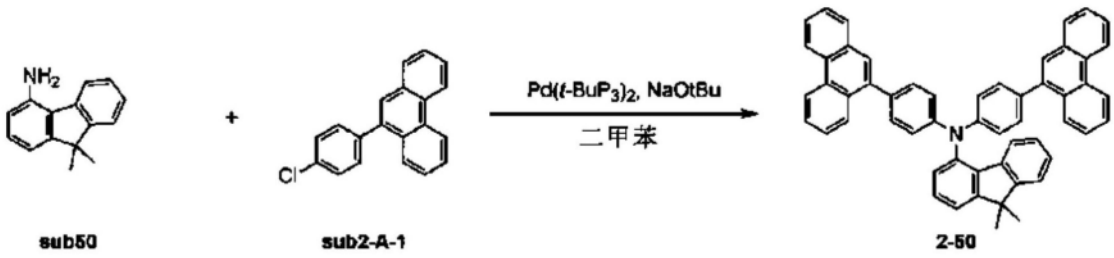
18.3mmol)和叔丁醇钠(2.3g,23.1mmol)添加至200ml二甲苯中,并将混合物搅拌并回流。然后,向其中添加双(三叔丁基膦)钯(0)(0.1g,0.2mmol)。5小时之后,当反应完成时,将反应混合物冷却至室温并在减压下除去溶剂。然后,将化合物再次完全溶解在氯仿中,用水洗涤两次,然后分离有机层,用无水硫酸镁处理,然后过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到7.7g化合物2-48。(产率:53%,MS:$[M+H]^+$=800)

[0790] 制备例2-49:化合物2-49的制备

[0791] 

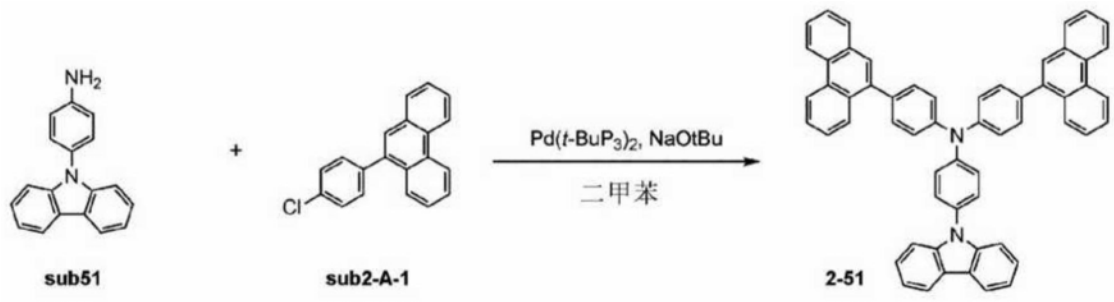
[0792] 在氮气气氛下将化合物sub49(10g,59.1mmol)、化合物sub2-A-1(35.8g,124.1mmol)和叔丁醇钠(14.2g,147.7mmol)添加至200ml二甲苯中,并将混合物搅拌并回流。然后,向其中添加双(三叔丁基膦)钯(0)(0.6g,1.2mmol)。5小时之后,当反应完成时,将反应混合物冷却至室温并在减压下除去溶剂。然后,将化合物再次完全溶解在氯仿中,用水洗涤两次,然后分离有机层,用无水硫酸镁处理,然后过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到22.7g化合物2-49。(产率:57%,MS:$[M+H]^+$=674)

[0793] 制备例2-50:化合物2-50的制备

[0794] 

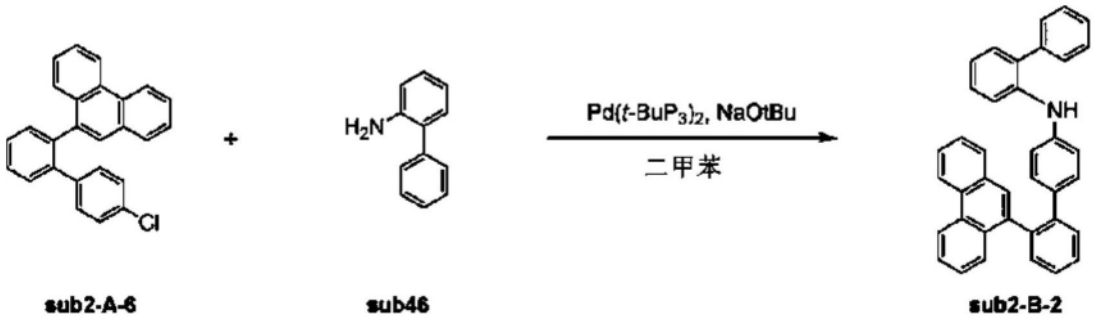
[0795] 在氮气气氛下将化合物sub50(10g,47.8mmol)、化合物sub2-A-1(29g,100.3mmol)和叔丁醇钠(11.5g,119.5mmol)添加至200ml二甲苯中,并将混合物搅拌并回流。然后,向其中添加双(三叔丁基膦)钯(0)(0.5g,1mmol)。5小时之后,当反应完成时,将反应混合物冷却至室温并在减压下除去溶剂。然后,将化合物再次完全溶解在氯仿中,用水洗涤两次,然后分离有机层,用无水硫酸镁处理,然后过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到23.9g化合物2-50。(产率:70%,MS:$[M+H]^+$=714)

[0796] 制备例2-51:化合物2-51的制备

[0797] 

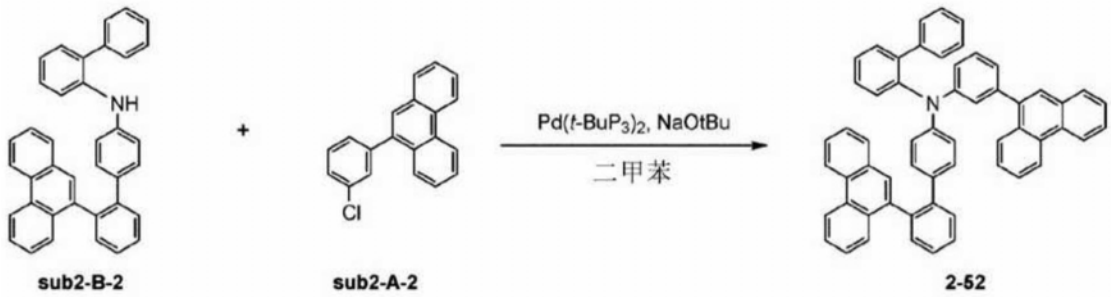
[0798] 在氮气气氛下将化合物sub51(10g,38.7mmol)、化合物sub2-A-1(23.5g,81.3mmol)和叔丁醇钠(9.3g,96.8mmol)添加至200ml二甲苯中,并将混合物搅拌并回流。然后,向其中添加双(三叔丁基膦)钯(0)(0.4g,0.8mmol)。5小时之后,当反应完成时,将反应混合物冷却至室温并在减压下除去溶剂。然后,将化合物再次完全溶解在氯仿中,用水洗涤两次,然后分离有机层,用无水硫酸镁处理,然后过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到16.8g化合物2-51。(产率:57%,MS:$[M+H]^+$=763)

[0799] 制备例2-52:化合物2-52的制备

[0800] 

sub2-A-6 sub46 sub2-B-2

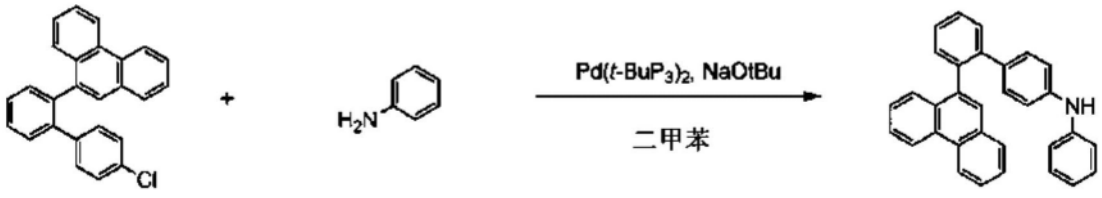
[0801] 在氮气气氛下将化合物sub2-A-6(10g,27.4mmol)、化合物sub46(4.6g,27.4mmol)和叔丁醇钠(2.9g,30.1mmol)添加至200ml二甲苯中,并将混合物搅拌并回流。然后,向其中添加双(三叔丁基膦)钯(0)(0.1g,0.3mmol)。5小时之后,当反应完成时,将反应混合物冷却至室温并在减压下除去溶剂。然后,将化合物再次完全溶解在氯仿中,用水洗涤两次,然后分离有机层,用无水硫酸镁处理,然后过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到9.4g化合物sub2-B-2。(产率:69%,MS:$[M+H]^+$=498)

[0802] 

sub2-B-2 sub2-A-2 2-52

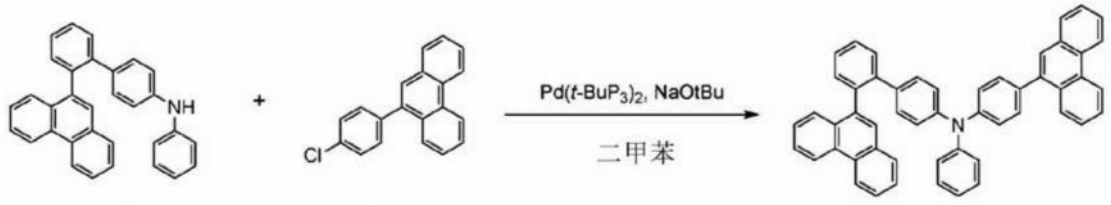
[0803] 在氮气气氛下将化合物sub2-B-2(10g,20.1mmol)、化合物sub2-A-2(5.8g,20.1mmol)和叔丁醇钠(2.5g,26.1mmol)添加至200ml二甲苯中,并将混合物搅拌并回流。然后,向其中添加双(三叔丁基膦)钯(0)(0.1g,0.2mmol)。5小时之后,当反应完成时,将反应混合物冷却至室温并在减压下除去溶剂。然后,将化合物再次完全溶解在氯仿中,用水洗涤两次,然后分离有机层,用无水硫酸镁处理,然后过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到8.3g化合物2-52。(产率:55%,MS:$[M+H]^+$=750)

[0804] 制备例2-53:化合物2-53的制备

[0805] 

sub2-A-6 sub52 sub2-B-3

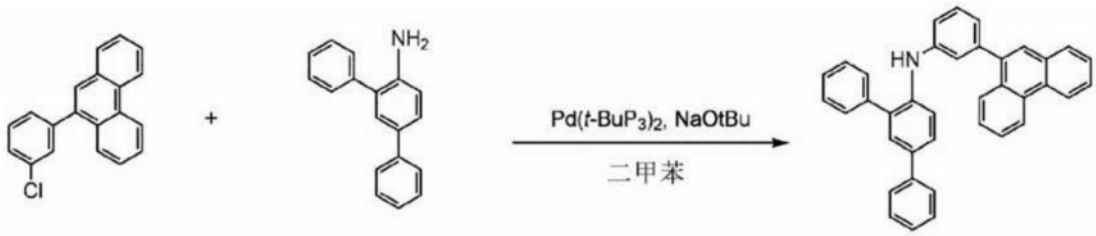
[0806] 在氮气气氛下将化合物sub2-A-6(10g,27.4mmol)、化合物sub52(2.6g,27.4mmol)和叔丁醇钠(2.9g,30.1mmol)添加至200ml二甲苯中,并将混合物搅拌并回流。然后,向其中添加双(三叔丁基膦)钯(0)(0.1g,0.3mmol)。5小时之后,当反应完成时,将反应混合物冷却至室温并在减压下除去溶剂。然后,将化合物再次完全溶解在氯仿中,用水洗涤两次,然后分离有机层,用无水硫酸镁处理,然后过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到5.9g化合物sub2-B-3。(产率:51%,MS:[M+H]$^+$=422)

[0807] 

sub2-B-3 sub2-A-1 2-53

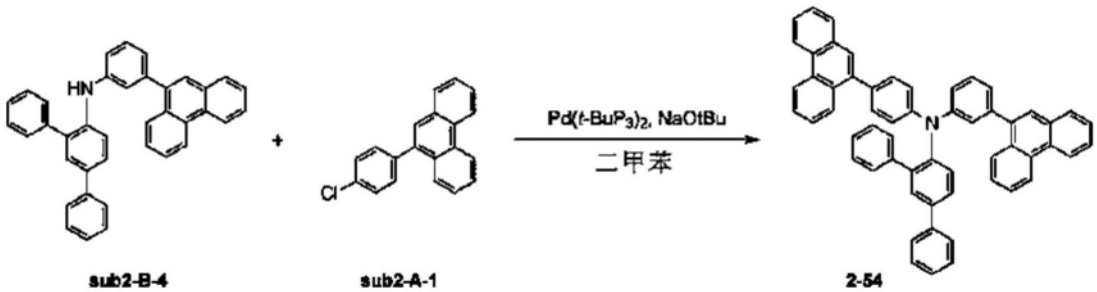
[0808] 在氮气气氛下将化合物sub2-B-3(10g,23.7mmol)、化合物sub2-A-1(6.9g,23.7mmol)和叔丁醇钠(3g,30.8mmol)添加至200ml二甲苯中,并将混合物搅拌并回流。然后,向其中添加双(三叔丁基膦)钯(0)(0.1g,0.2mmol)。5小时之后,当反应完成时,将反应混合物冷却至室温并在减压下除去溶剂。然后,将化合物再次完全溶解在氯仿中,用水洗涤两次,然后分离有机层,用无水硫酸镁处理,然后过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到9.3g化合物2-53。(产率:58%,MS:[M+H]$^+$=674)

[0809] 制备例2-54:化合物2-54的制备

[0810] 

sub2-A-2 sub53 sub2-B-4

[0811] 在氮气气氛下将化合物sub2-A-2(10g,34.6mmol)、化合物sub53(8.5g,34.6mmol)和叔丁醇钠(3.7g,38.1mmol)添加至200ml二甲苯中,并将混合物搅拌并回流。然后,向其中添加双(三叔丁基膦)钯(0)(0.2g,0.3mmol)。5小时之后,当反应完成时,将反应混合物冷却至室温并在减压下除去溶剂。然后,将化合物再次完全溶解在氯仿中,用水洗涤两次,然后分离有机层,用无水硫酸镁处理,然后过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到11.5g化合物sub2-B-4。(产率:67%,MS:[M+H]$^+$=498)

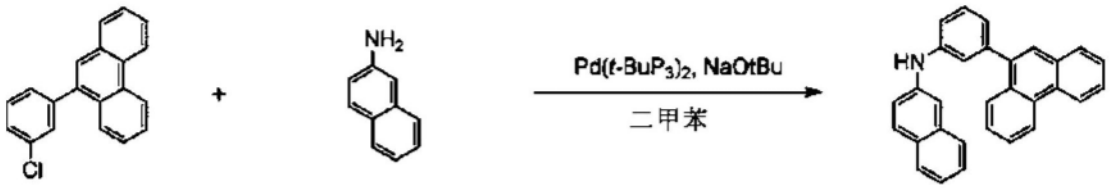
[0812] 

sub2-B-4 sub2-A-1 2-54

[0813] 在氮气气氛下将化合物sub2-B-4(10g,20.1mmol)、化合物sub2-A-1(5.8g,

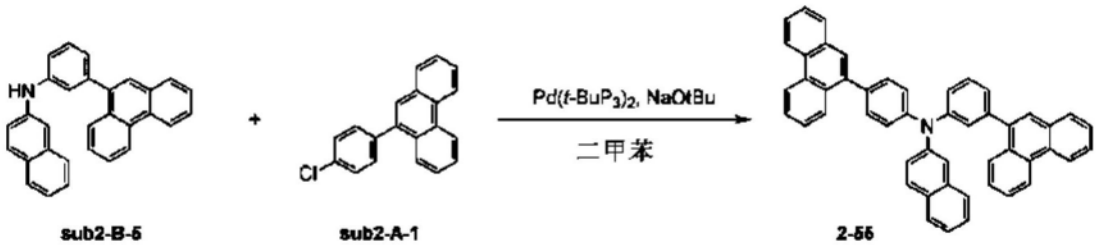
20.1mmol)和叔丁醇钠(2.5g,26.1mmol)添加至200ml二甲苯中,并将混合物搅拌并回流。然后,向其中添加双(三叔丁基膦)钯(0)(0.1g,0.2mmol)。5小时之后,当反应完成时,将反应混合物冷却至室温并在减压下除去溶剂。然后,将化合物再次完全溶解在氯仿中,用水洗涤两次,然后将有机层分离,用无水硫酸镁处理,然后过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到7.5g化合物2-54。(产率:50%,MS:$[M+H]^+$=750)

[0814] 制备例2-55:化合物2-55的制备

[0815] 

sub2-A-2 sub45 sub2-B-5

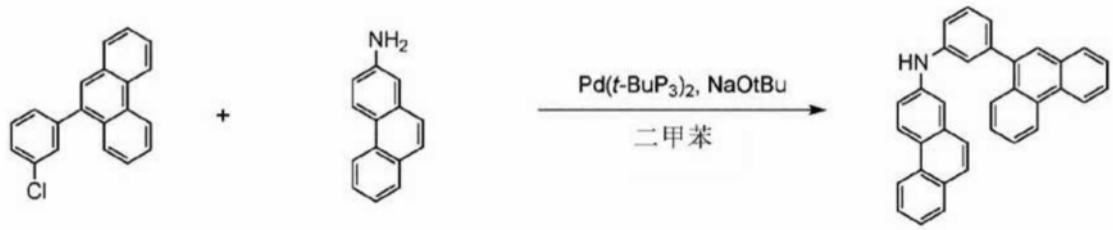
[0816] 在氮气气氛下将化合物sub2-A-2(10g,34.6mmol)、化合物sub45(5g,34.6mmol)和叔丁醇钠(3.7g,38.1mmol)添加至200ml二甲苯中,并将混合物搅拌并回流。然后,向其中添加双(三叔丁基膦)钯(0)(0.2g,0.3mmol)。5小时之后,当反应完成时,将反应混合物冷却至室温并在减压下除去溶剂。然后,将化合物再次完全溶解在氯仿中,用水洗涤两次,然后分离有机层,用无水硫酸镁处理,然后过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到9.3g化合物sub2-B-5。(产率:68%,MS:$[M+H]^+$=396)

[0817] 

sub2-B-5 sub2-A-1 2-56

[0818] 在氮气气氛下将化合物sub2-B-5(10g,25.3mmol)、化合物sub2-A-1(7.3g,25.3mmol)和叔丁醇钠(3.2g,32.9mmol)添加至200ml二甲苯中,并将混合物搅拌并回流。然后,向其中添加双(三叔丁基膦)钯(0)(0.1g,0.3mmol)。5小时之后,当反应完成时,将反应混合物冷却至室温并在减压下除去溶剂。然后,将化合物再次完全溶解在氯仿中,用水洗涤两次,然后分离有机层,用无水硫酸镁处理,然后过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到10g化合物2-55。(产率:61%,MS:$[M+H]^+$=648)

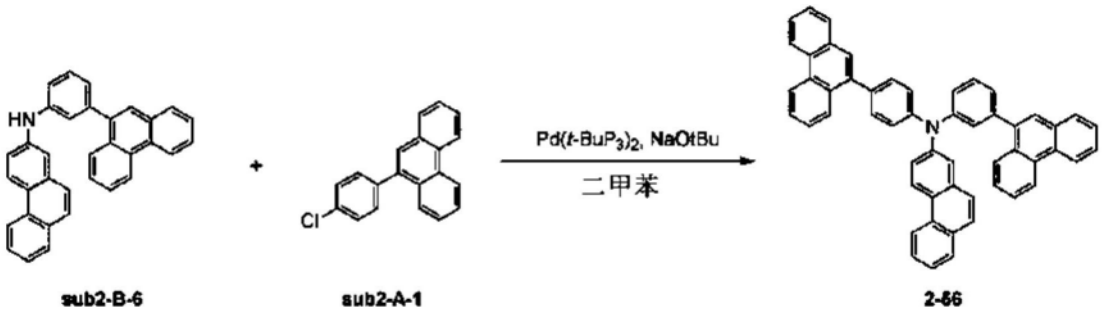
[0819] 制备例2-56:化合物2-56的制备

[0820] 

sub2-A-2 sub54 sub2-B-6

[0821] 在氮气气氛下将化合物sub2-A-2(10g,34.6mmol)、化合物sub54(6.7g,34.6mmol)和叔丁醇钠(3.7g,38.1mmol)添加至200ml二甲苯中,并将混合物搅拌并回流。然后,向其中添加双(三叔丁基膦)钯(0)(0.2g,0.3mmol)。5小时之后,当反应完成时,将反应混合物冷却

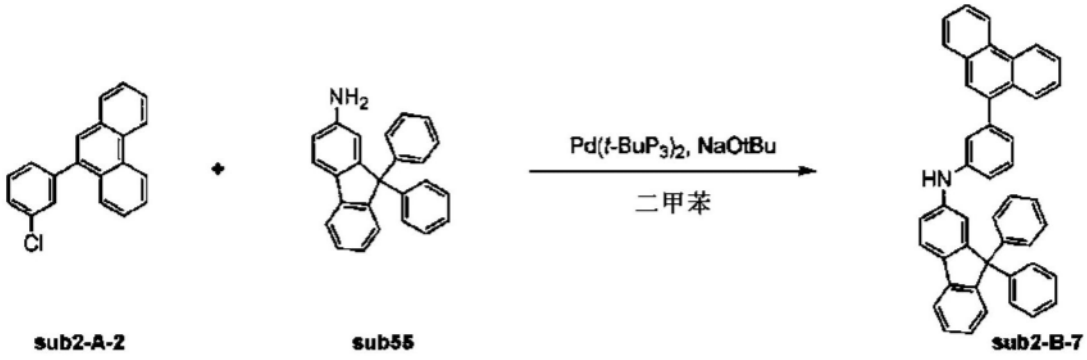
至室温并在减压下除去溶剂。然后,将化合物再次完全溶解在氯仿中,用水洗涤两次,然后分离有机层,用无水硫酸镁处理,然后过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到8.6g化合物sub2-B-6。(产率:56%,MS:[M+H]$^+$=446)

[0822] 

sub2-B-6 sub2-A-1 2-56

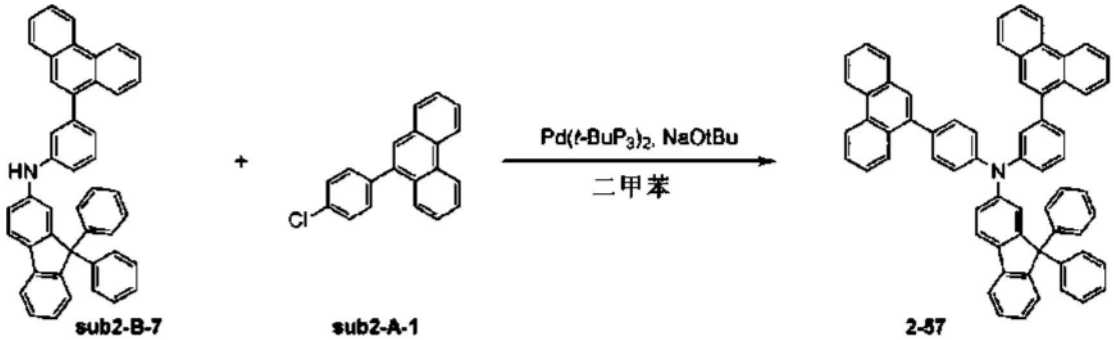
[0823] 在氮气气氛下将化合物sub2-B-6(10g,22.4mmol)、化合物sub2-A-1(6.5g,22.4mmol)和叔丁醇钠(2.8g,29.2mmol)添加至200ml二甲苯中,并将混合物搅拌并回流。然后,向其中添加双(三叔丁基膦)钯(0)(0.1g,0.2mmol)。5小时之后,当反应完成时,将反应混合物冷却至室温并在减压下除去溶剂。然后,将化合物再次完全溶解在氯仿中,用水洗涤两次,然后分离有机层,用无水硫酸镁处理,然后过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到8.8g化合物2-56。(产率:56%,MS:[M+H]$^+$=698)

[0824] 制备例2-57:化合物2-57的制备

[0825] 

sub2-A-2 sub55 sub2-B-7

[0826] 在氮气气氛下将化合物sub2-A-2(10g,34.6mmol)、化合物sub55(11.5g,34.6mmol)和叔丁醇钠(3.7g,38.1mmol)添加至200ml二甲苯中,并将混合物搅拌并回流。然后,向其中添加双(三叔丁基膦)钯(0)(0.2g,0.3mmol)。5小时之后,当反应完成时,将反应混合物冷却至室温并在减压下除去溶剂。然后,将化合物再次完全溶解在氯仿中,用水洗涤两次,然后分离有机层,用无水硫酸镁处理,然后过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到13.2g化合物sub2-B-7。(产率:65%,MS:[M+H]$^+$=586)

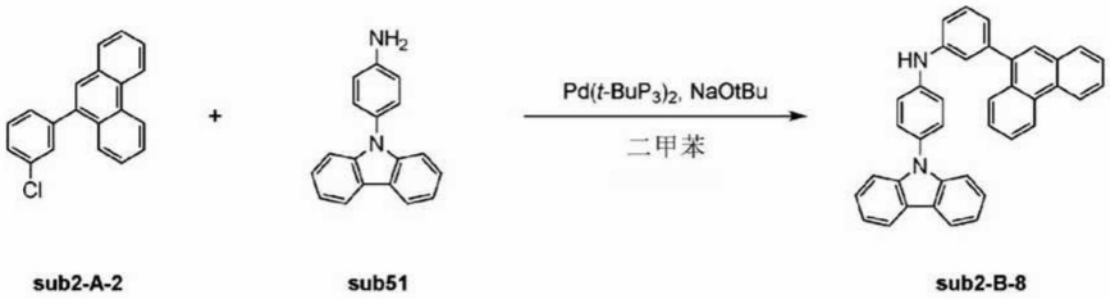
[0827] 

sub2-B-7 sub2-A-1 2-57

[0828] 在氮气气氛下将化合物sub2-B-7(10g,17.1mmol)、化合物sub2-A-1(4.9g,

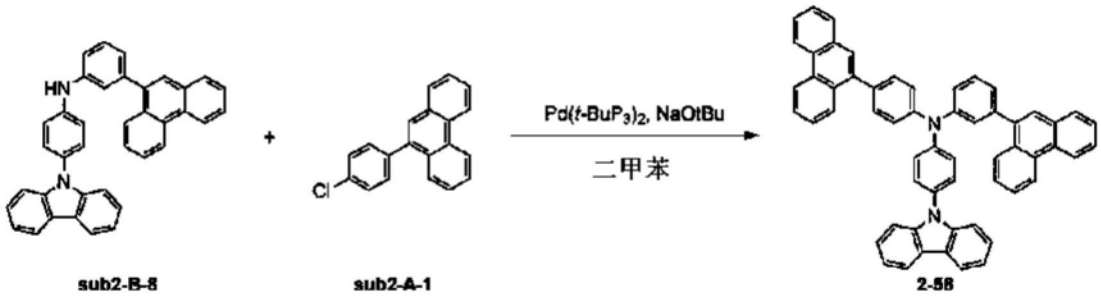
17.1mmol)和叔丁醇钠(2.1g,22.2mmol)添加至200ml二甲苯中,并将混合物搅拌并回流。然后,向其中添加双(三叔丁基膦)钯(0)(0.1g,0.2mmol)。5小时之后,当反应完成时,将反应混合物冷却至室温并在减压下除去溶剂。然后,将化合物再次完全溶解在氯仿中,用水洗涤两次,然后分离有机层,用无水硫酸镁处理,然后过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到7.7g化合物2-57。(产率:54%,MS:$[M+H]^+$=838)

[0829] 制备例2-58:化合物2-58的制备

[0830] 

sub2-A-2 sub51 sub2-B-8

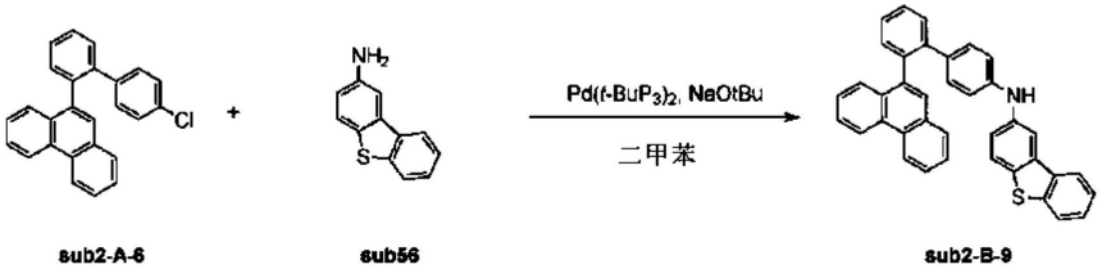
[0831] 在氮气气氛下将化合物sub2-A-2(10g,34.6mmol)、化合物sub51(8.9g,34.6mmol)和叔丁醇钠(3.7g,38.1mmol)添加至200ml二甲苯中,并将混合物搅拌并回流。然后,向其中添加双(三叔丁基膦)钯(0)(0.2g,0.3mmol)。5小时之后,当反应完成时,将反应混合物冷却至室温并在减压下除去溶剂。然后,将化合物再次完全溶解在氯仿中,用水洗涤两次,然后分离有机层,用无水硫酸镁处理,然后过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到10.8g化合物sub2-B-8。(产率:61%,MS:$[M+H]^+$=511)

[0832] 

sub2-B-8 sub2-A-1 2-58

[0833] 在氮气气氛下将化合物sub2-B-8(10g,19.6mmol)、化合物sub2-A-1(5.7g,19.6mmol)和叔丁醇钠(2.4g,25.5mmol)添加至200ml二甲苯中,并将混合物搅拌并回流。然后,向其中添加双(三叔丁基膦)钯(0)(0.1g,0.2mmol)。5小时之后,当反应完成时,将反应混合物冷却至室温并在减压下除去溶剂。然后,将化合物再次完全溶解在氯仿中,用水洗涤两次,然后分离有机层,用无水硫酸镁处理,然后过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到7.6g化合物2-58。(产率:51%,MS:$[M+H]^+$=763)

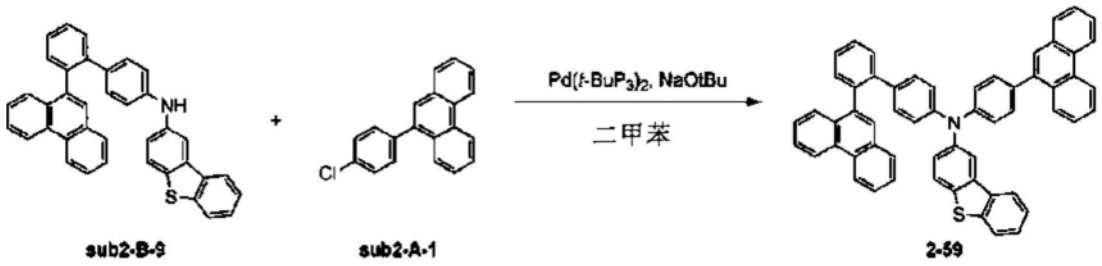
[0834] 制备例2-59:化合物2-59的制备

[0835] 

sub2-A-6 sub56 sub2-B-9

[0836] 在氮气气氛下将化合物sub2-A-6(10g,27.4mmol)、化合物sub56(5.5g,27.4mmol)

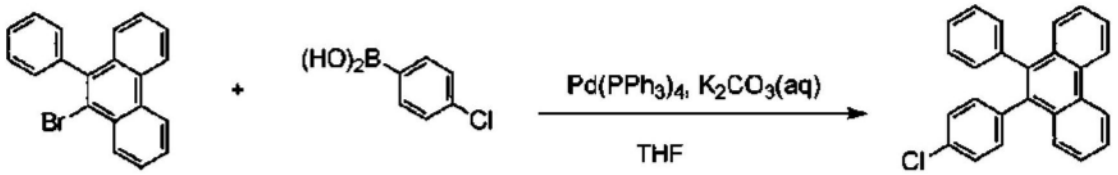
和叔丁醇钠(2.9g,30.2mmol)添加至200ml二甲苯中,并将混合物搅拌并回流。然后,向其中添加双(三叔丁基膦)钯(0)(0.1g,0.3mmol)。5小时之后,当反应完成时,将反应混合物冷却至室温并在减压下除去溶剂。然后,将化合物再次完全溶解在氯仿中,用水洗涤两次,然后分离有机层,用无水硫酸镁处理,然后过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到7.5g化合物sub2-B-9。(产率:52%,MS:$[M+H]^+$=528)

[0837] 

sub2-B-9 sub2-A-1 2-59

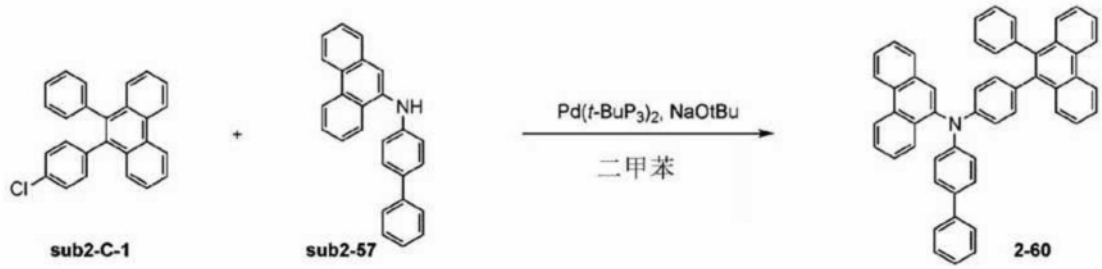
[0838] 在氮气气氛下将化合物sub2-B-9(10g,19mmol)、化合物sub2-A-1(5.5g,19mmol)和叔丁醇钠(2.4g,24.6mmol)添加至200ml二甲苯中,并将混合物搅拌并回流。然后,向其中添加双(三叔丁基膦)钯(0)(0.1g,0.2mmol)。5小时之后,当反应完成时,将反应混合物冷却至室温并在减压下除去溶剂。然后,将化合物再次完全溶解在氯仿中,用水洗涤两次,然后分离有机层,用无水硫酸镁处理,然后过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到8.7g化合物2-59。(产率:59%,MS:$[M+H]^+$=780)

[0839] 制备例2-60:化合物2-60的制备

[0840] 

2-H 2-B sub2-C-1

[0841] 在氮气气氛下将化合物2-H(15g,45mmol)和化合物2-B(7.7g,49.5mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(12.4g,90mmol)溶解在37ml水中,添加至混合物中,并将混合物充分搅拌,然后添加四(三苯基膦)钯(0)(1g,0.9mmol)。在11小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到12.3g化合物sub2-C-1。(产率:75%,MS:$[M+H]^+$=365)

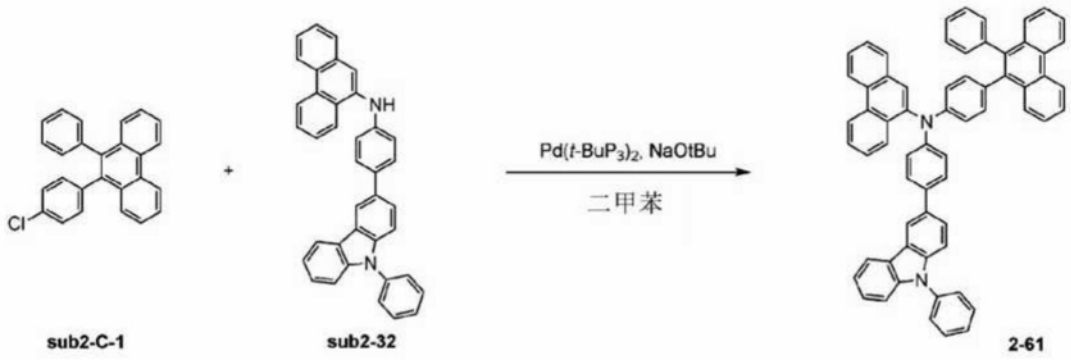
[0842] 

sub2-C-1 sub2-57 2-60

[0843] 在氮气气氛下将化合物sub2-C-1(10g,27.4mmol)、化合物sub2-57(9.5g,27.4mmol)和叔丁醇钠(3.4g,35.6mmol)添加至200ml二甲苯中,并将混合物搅拌并回流。然

后,向其中添加双(三叔丁基膦)钯(0)(0.1g,0.3mmol)。5小时之后,当反应完成时,将反应混合物冷却至室温并在减压下除去溶剂。然后,将化合物再次完全溶解在氯仿中,用水洗涤两次,然后分离有机层,用无水硫酸镁处理,然后过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到12.7g化合物2-60。(产率:69%,MS:$[M+H]^+$=674)

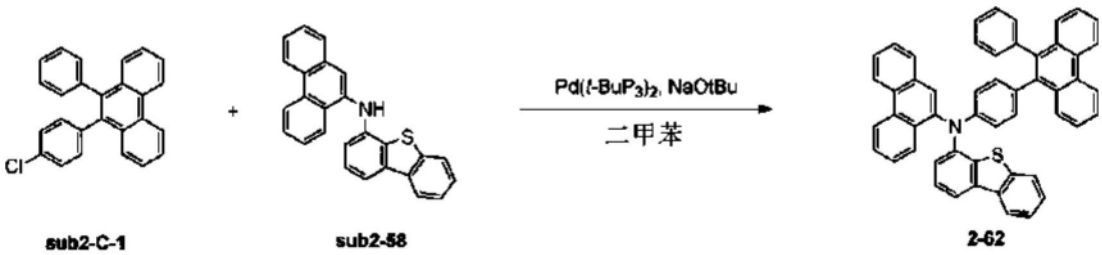
[0844] 制备例2-61:化合物2-61的制备

[0845] 

sub2-C-1 sub2-32 2-61

[0846] 在氮气气氛下将化合物sub2-C-1(10g,27.4mmol)、化合物sub2-32(14g,27.4mmol)和叔丁醇钠(3.4g,35.6mmol)添加至200ml二甲苯中,并将混合物搅拌并回流。然后,向其中添加双(三叔丁基膦)钯(0)(0.1g,0.3mmol)。5小时之后,当反应完成时,将反应混合物冷却至室温并在减压下除去溶剂。然后,将化合物再次完全溶解在氯仿中,用水洗涤两次,然后分离有机层,用无水硫酸镁处理,然后过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到12.6g化合物2-61。(产率:55%,MS:$[M+H]^+$=839)

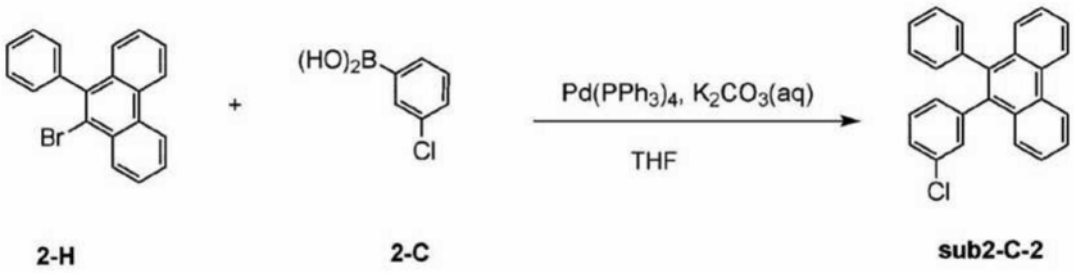
[0847] 制备例2-62:化合物2-62的制备

[0848] 

sub2-C-1 sub2-58 2-62

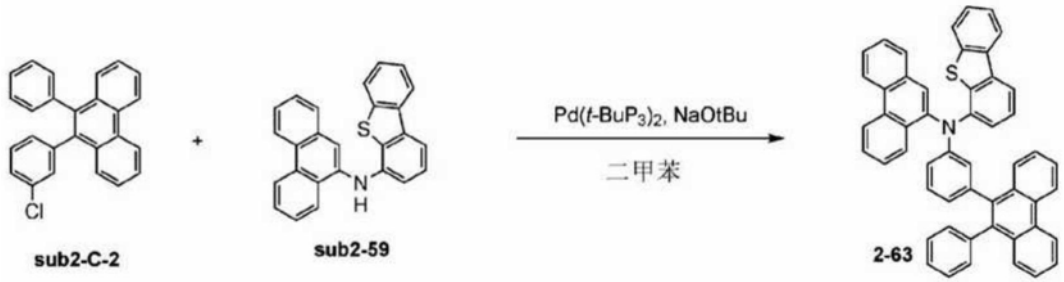
[0849] 在氮气气氛下将化合物sub2-C-1(10g,27.4mmol)、化合物sub2-58(10.3g,27.4mmol)和叔丁醇钠(3.4g,35.6mmol)添加至200ml二甲苯中,并将混合物搅拌并回流。然后,向其中添加双(三叔丁基膦)钯(0)(0.1g,0.3mmol)。5小时之后,当反应完成时,将反应混合物冷却至室温并在减压下除去溶剂。然后,将化合物再次完全溶解在氯仿中,用水洗涤两次,然后分离有机层,用无水硫酸镁处理,然后过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到12.5g化合物2-62。(产率:65%,MS:$[M+H]^+$=704)

[0850] 制备例2-63:化合物2-63的制备

[0851] 

2-H 2-C sub2-C-2

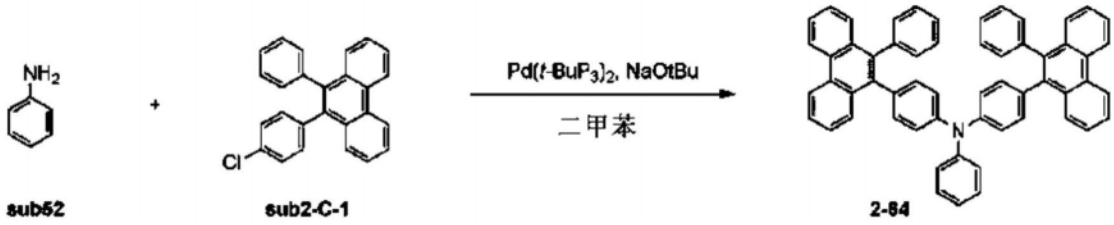
[0852] 在氮气气氛下将化合物2-H(15g,45mmol)和化合物2-C(7.7g,49.5mmol)添加至300ml THF中,并将混合物搅拌并回流。然后,将碳酸钾(12.4g,90mmol)溶解在37ml水中,添加至混合物中,并将混合物充分搅拌,然后添加四(三苯基膦)钯(0)(1g,0.9mmol)。在11小时反应之后,将反应混合物冷却至室温,并将有机层和水层分离,然后将有机层蒸馏。将其再次溶解在氯仿中,用水洗涤两次,然后将有机层分离,向其中添加无水硫酸镁,将混合物搅拌并过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到12.3g化合物sub2-C-2。(产率:75%,MS:$[M+H]^+$=365)

[0853] 

sub2-C-2 sub2-59 2-63

[0854] 在氮气气氛下将化合物sub2-C-2(10g,27.4mmol)、化合物sub2-59(10.3g,27.4mmol)和叔丁醇钠(3.4g,35.6mmol)添加至200ml二甲苯中,并将混合物搅拌并回流。然后,向其中添加双(三叔丁基膦)钯(0)(0.1g,0.3mmol)。5小时之后,当反应完成时,将反应混合物冷却至室温并在减压下除去溶剂。然后,将化合物再次完全溶解在氯仿中,用水洗涤两次,然后分离有机层,用无水硫酸镁处理,然后过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到13.5g化合物2-63。(产率:70%,MS:$[M+H]^+$=704)

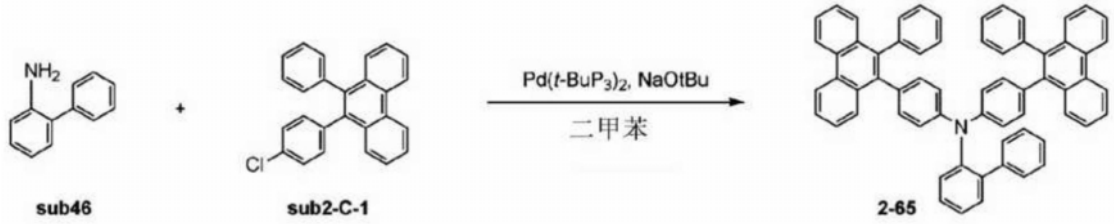
[0855] 制备例2-64:化合物2-64的制备

[0856] 

sub52 sub2-C-1 2-64

[0857] 在氮气气氛下将化合物sub52(10g,107.4mmol)、化合物sub2-C-1(82.3g,225.5mmol)和叔丁醇钠(25.8g,268.4mmol)添加至200ml二甲苯中,并将混合物搅拌并回流。然后,向其中添加双(三叔丁基膦)钯(0)(1.1g,2.1mmol)。5小时之后,当反应完成时,将反应混合物冷却至室温并在减压下除去溶剂。然后,将化合物再次完全溶解在氯仿中,用水洗涤两次,然后分离有机层,用无水硫酸镁处理,然后过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到41g化合物2-64。(产率:51%,MS:$[M+H]^+$=750)

[0858] 制备例2-65:化合物2-65的制备

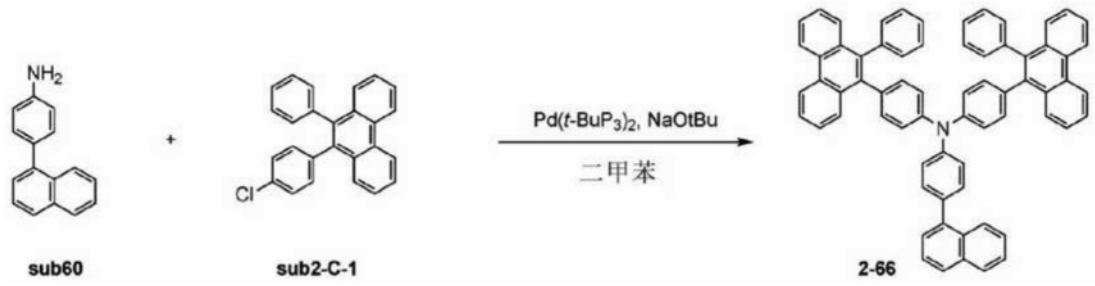
[0859] 

sub46 sub2-C-1 2-65

[0860] 在氮气气氛下将化合物sub46(10g,59.1mmol)、化合物sub2-C-1(45.3g,

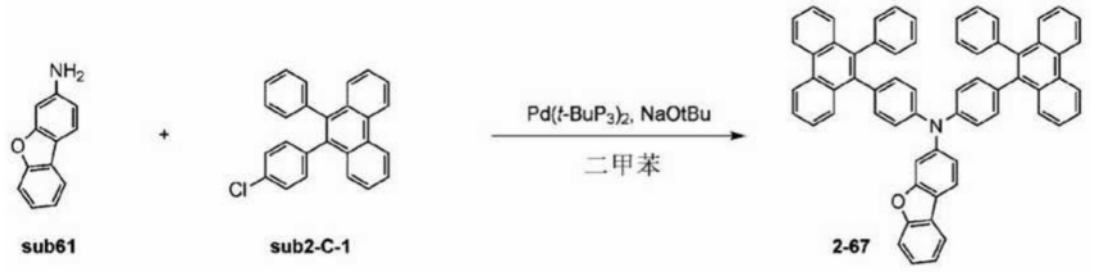
124.1mmol)和叔丁醇钠(14.2g,147.7mmol)添加至200ml二甲苯中,并将混合物搅拌并回流。然后,向其中添加双(三叔丁基膦)钯(0)(0.6g,1.2mmol)。5小时之后,当反应完成时,将反应混合物冷却至室温并在减压下除去溶剂。然后,将化合物再次完全溶解在氯仿中,用水洗涤两次,然后分离有机层,用无水硫酸镁处理,然后过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到31.2g化合物2-65。(产率:64%,MS:$[M+H]^+$=826)

[0861] 制备例2-66:化合物2-66的制备

[0862] 

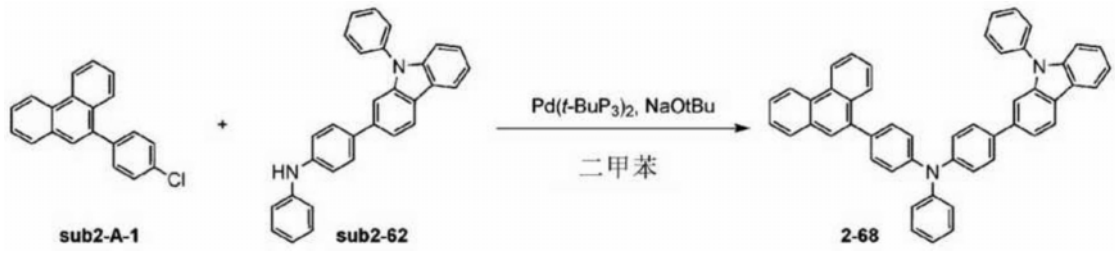
[0863] 在氮气气氛下将化合物sub60(10g,45.6mmol)、化合物sub2-C-1(34.9g,95.8mmol)和叔丁醇钠(11g,114mmol)添加至200ml二甲苯中,并将混合物搅拌并回流。然后,向其中添加双(三叔丁基膦)钯(0)(0.5g,0.9mmol)。5小时之后,当反应完成时,将反应混合物冷却至室温并在减压下除去溶剂。然后,将化合物再次完全溶解在氯仿中,用水洗涤两次,然后分离有机层,用无水硫酸镁处理,然后过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到26.7g化合物2-66。(产率:67%,MS:$[M+H]^+$=876)

[0864] 制备例2-67:化合物2-67的制备

[0865] 

[0866] 在氮气气氛下将化合物sub61(10g,54.6mmol)、化合物sub2-C-1(41.8g,114.6mmol)和叔丁醇钠(13.1g,136.5mmol)添加至200ml二甲苯中,并将混合物搅拌并回流。然后,向其中添加双(三叔丁基膦)钯(0)(0.6g,1.1mmol)。5小时之后,当反应完成时,将反应混合物冷却至室温并在减压下除去溶剂。然后,将化合物再次完全溶解在氯仿中,用水洗涤两次,然后分离有机层,用无水硫酸镁处理,然后过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到32.1g化合物2-67。(产率:70%,MS:$[M+H]^+$=840)

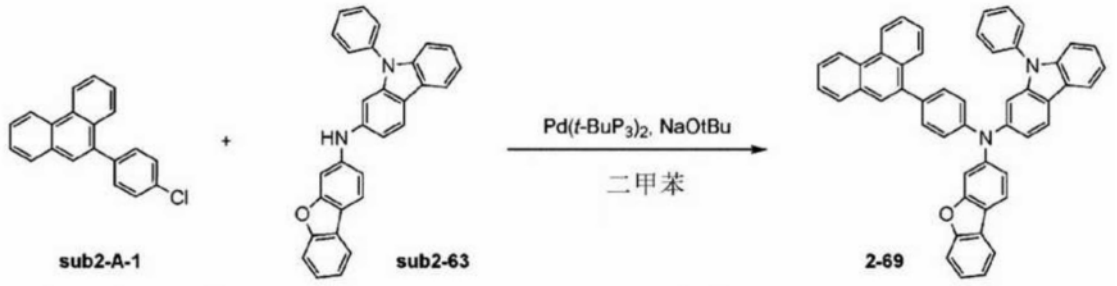
[0867] 制备例2-68:化合物2-68的制备

[0868] 

[0869] 在氮气气氛下将化合物sub2-A-1(10g,34.6mmol)、化合物sub2-62(15.6g,

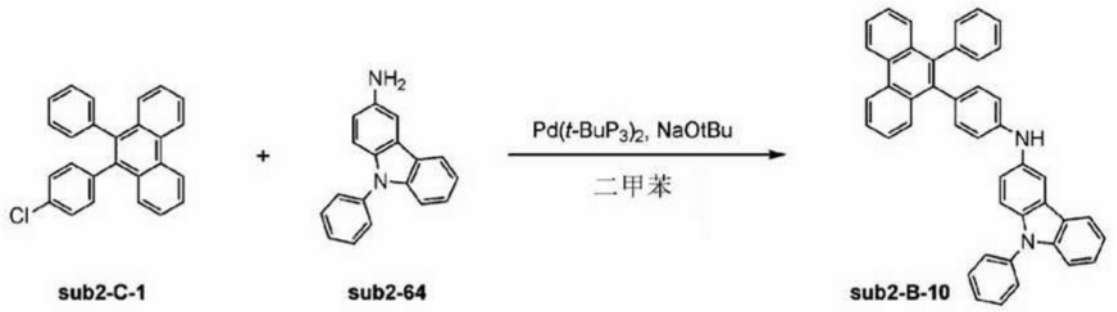
38.1mmol)和磷酸钾(22.1g,103.9mmol)添加至200ml二甲苯中,并将混合物搅拌并回流。然后,向其中添加双(三叔丁基膦)钯(0)(0.4g,0.7mmol)。2小时之后,当反应完成时,将反应混合物冷却至室温并在减压下除去溶剂。然后,将化合物再次完全溶解在氯仿中,用水洗涤两次,然后分离有机层,用无水硫酸镁处理,然后过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到12.6g化合物2-68。(产率:55%,MS:$[M+H]^+$=663)

[0870] 制备例2-69:化合物2-69的制备

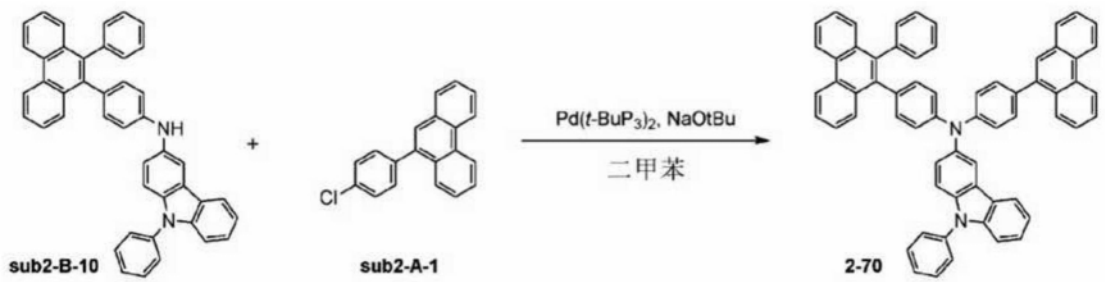
[0871] 

[0872] 在氮气气氛下将化合物sub2-A-1(10g,34.6mmol)、化合物sub2-63(16.2g,38.1mmol)和磷酸钾(22.1g,103.9mmol)添加至200ml二甲苯中,并将混合物搅拌并回流。然后,向其中添加双(三叔丁基膦)钯(0)(0.4g,0.7mmol)。2小时之后,当反应完成时,将反应混合物冷却至室温并在减压下除去溶剂。然后,将化合物再次完全溶解在氯仿中,用水洗涤两次,然后分离有机层,用无水硫酸镁处理,然后过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到12.6g化合物2-69。(产率:54%,MS:$[M+H]^+$=677)

[0873] 制备例2-70:化合物2-70的制备

[0874] 

[0875] 在氮气气氛下将化合物sub2-C-1(10g,27.4mmol)、化合物sub2-64(7.8g,30.1mmol)和磷酸钾(17.5g,82.2mmol)添加至200ml二甲苯中,并将混合物搅拌并回流。然后,向其中添加双(三叔丁基膦)钯(0)(0.3g,0.5mmol)。2小时之后,当反应完成时,将反应混合物冷却至室温并在减压下除去溶剂。然后,将化合物再次完全溶解在氯仿中,用水洗涤两次,然后分离有机层,用无水硫酸镁处理,然后过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到11.2g化合物sub2-B-10。(产率:70%,MS:$[M+H]^+$=587)

[0876] 

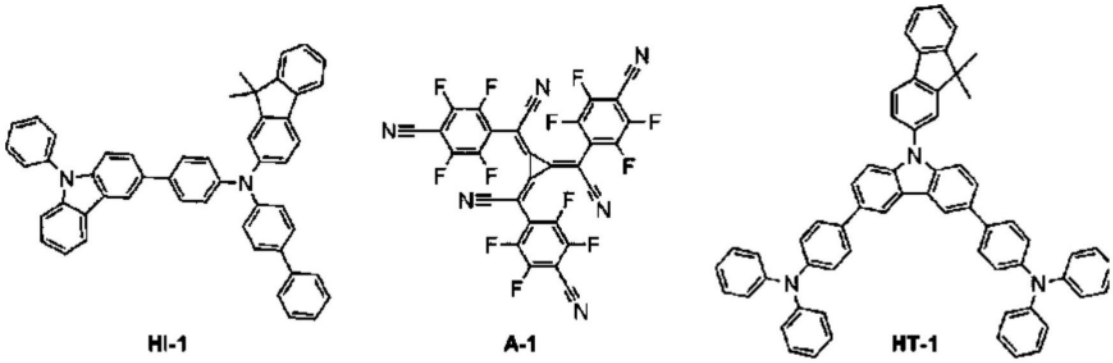
[0877] 在氮气气氛下将化合物sub2-B-10(10g,17mmol)、化合物sub2-A-1(5.4g,18.7mmol)和磷酸钾(10.9g,51.1mmol)添加至200ml二甲苯中,并将混合物搅拌并回流。然后,向其中添加双(三叔丁基膦)钯(0)(0.2g,0.3mmol)。3小时之后,当反应完成时,将反应混合物冷却至室温并在减压下除去溶剂。然后,将化合物再次完全溶解在氯仿中,用水洗涤两次,然后分离有机层,用无水硫酸镁处理,然后过滤,并将滤液在减压下蒸馏。通过硅胶柱色谱法将浓缩的化合物纯化以得到8.7g化合物2-70。(产率:61%,MS:$[M+H]^+$=839)

[0878] [实施例和比较例]

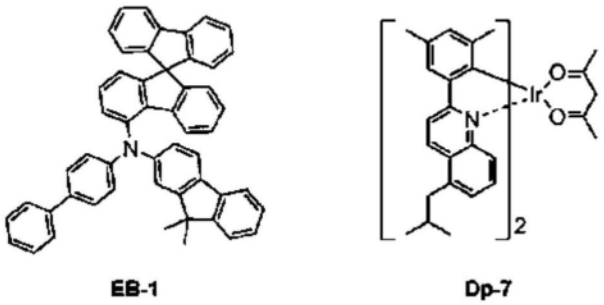
[0879] 比较例1

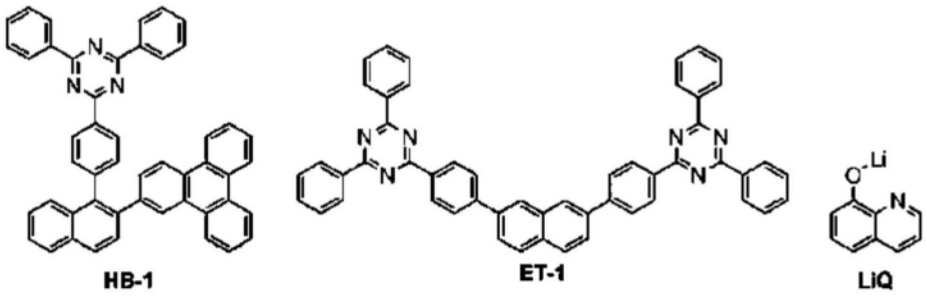
[0880] 将其上涂覆有厚度为1000 Å的ITO(氧化铟锡)薄膜的玻璃基底放入包含溶解在其中的清洁剂的蒸馏水中,并通过超声波洗涤。在这种情况下,使用的清洁剂是可商购自Fischer Co.的产品,并且蒸馏水是通过使用可商购自Millipore Co.的过滤器过滤两次的蒸馏水。将ITO洗涤30分钟,然后通过使用蒸馏水重复两次超声洗涤10分钟。在用蒸馏水洗涤完成之后,将基底用异丙醇、丙酮和甲醇溶剂超声洗涤,并干燥,之后将其转移至等离子体清洗机。然后,将基底用氧等离子体清洗5分钟,然后转移至真空蒸镀器。

[0881] 在由此准备的ITO透明电极上,使以下化合物HI-1形成为空穴注入层,但是使以1.5重量%的量p掺杂以下化合物A-1。在空穴注入层上真空沉积以下化合物HT-1以形成膜厚度为800 Å的空穴传输层。然后,在空穴传输层上真空沉积以下化合物EB-1至膜厚度为150 Å以形成电子阻挡层。然后,在EB-1沉积膜上以98:2的重量比真空沉积以下化合物1-2和以下化合物Dp-7以形成厚度为400 Å的红色发光层。在发光层上真空沉积以下化合物HB-1至厚度为30 Å以形成空穴阻挡层。然后,在空穴阻挡层上以2:1的比率真空沉积以下化合物ET-1和以下化合物LiQ以形成厚度为300 Å的电子注入和传输层。在电子注入和传输层上顺序地沉积氟化锂(LiF)和铝至分别具有12 Å和1,000 Å的厚度以形成阴极。



[0882]





[0883] 在上述过程中,将有机材料的沉积速率保持在0.4 Å/秒至0.7 Å/秒,将阴极的氟化锂和铝的沉积速率分别保持在0.3 Å/秒和2 Å/秒,以及将沉积期间的真空度保持在$2*10^{-7}$托至$5*10^{-6}$托。

[0884] 实施例1至155

[0885] 以与比较例1中相同的方式制造有机发光器件,不同之处在于以1:1的重量比共沉积下表1中列出的第一主体和第二主体并进行使用,以代替作为主体的化合物1-2。

[0886] 比较例2至61

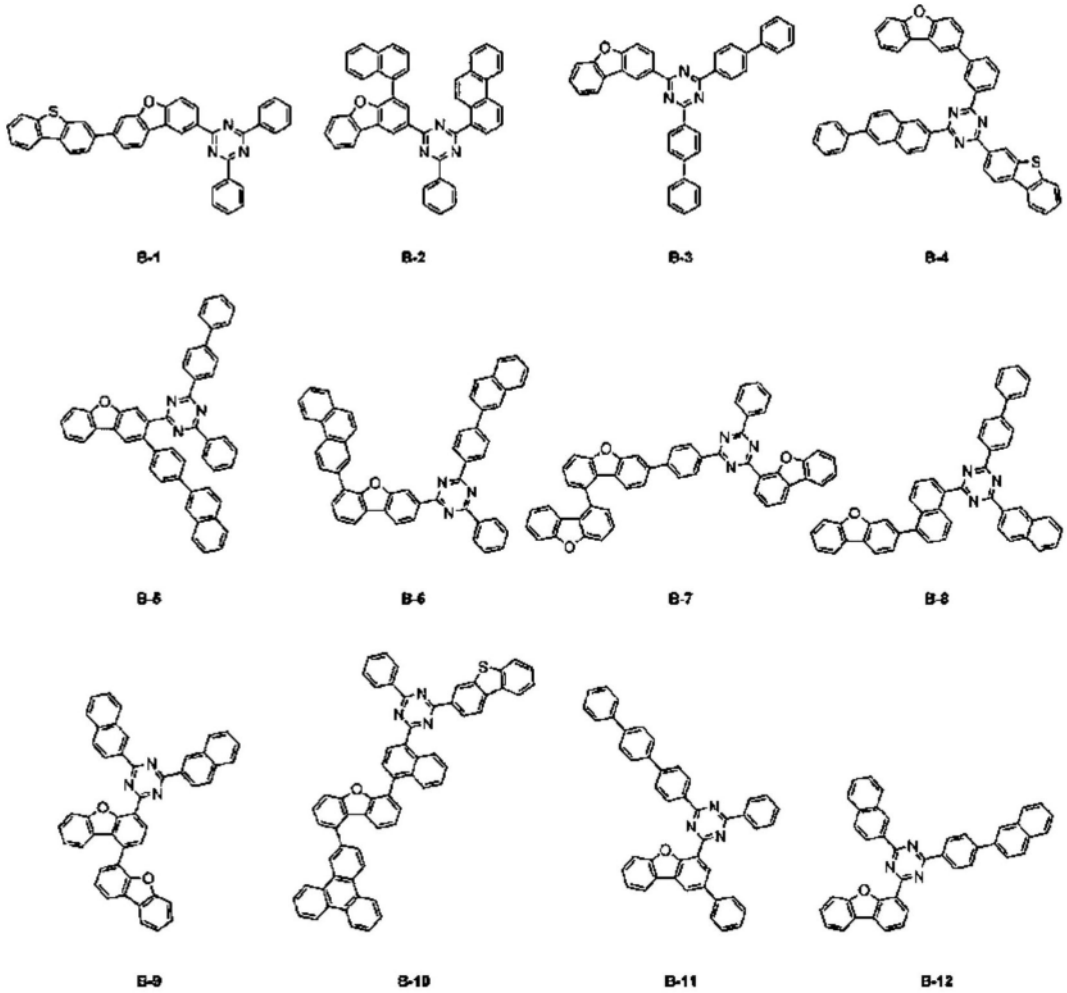
[0887] 以与比较例1中相同的方式制造有机发光器件,不同之处在于使用表2中所示的主体材料代替作为主体的化合物1-2,以及使用下表2中所示的化合物代替作为电子阻挡层材料的化合物EB-1。

[0888] 比较例62至121

[0889] 以与比较例1中相同的方式制造有机发光器件,不同之处在于以1:1的重量比共沉积下表3中列出的第一主体和第二主体并进行使用,以代替作为主体的化合物1-2。

[0890] 表3中的化合物B-1至化合物B-12如下。

[0891]

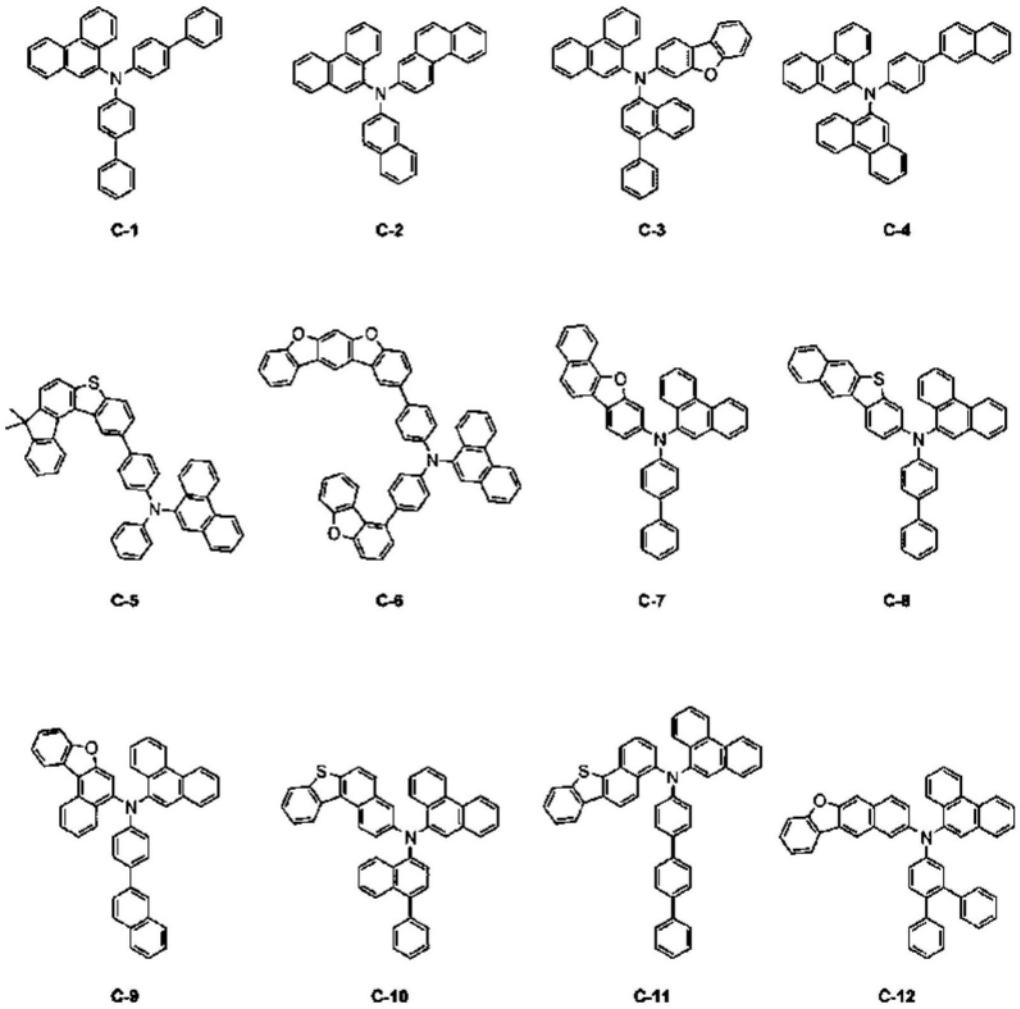


[0892] 比较例122至227

[0893] 以与比较例1中相同的方式制造有机发光器件,不同之处在于以1:1的重量比共沉积下表4中列出的第一主体和第二主体并进行使用,以代替作为主体的化合物1-2。

[0894] 表4中的化合物C-1至化合物C-12如下。

[0895]



[0896] [实验例]

[0897] 通过向实施例1至155和比较例1至227中制造的有机发光器件施加电流来测量电压、效率和寿命(基于15mA/cm^2),并且结果示于下表1至4中。寿命T95意指亮度降低至初始亮度(6000尼特)的95%所需的时间。

[0898] [表1]

[0899]

类别	第一主体	第二主体	驱动电压（V）	效率（cd/A）	寿命T95（小时）	发光颜色
实施例 1	化合物1-2	化合物2-1	3.96	19.6	203	红色
实施例 2	化合物1-2	化合物2-27	3.94	19.3	207	红色
实施例 3	化合物1-2	化合物2-39	3.97	19.8	198	红色
实施例 4	化合物1-2	化合物2-54	3.88	20.4	185	红色
实施例 5	化合物1-2	化合物2-60	3.93	19.1	193	红色
实施例 6	化合物1-3	化合物2-3	3.92	18.9	191	红色
实施例 7	化合物1-3	化合物2-10	3.88	19.2	208	红色
实施例 8	化合物1-3	化合物2-68	3.90	19.6	197	红色
实施例 9	化合物1-3	化合物2-44	3.89	18.7	191	红色
实施例 10	化合物1-3	化合物2-49	3.92	185	205	红色
实施例 11	化合物1-7	化合物2-5	3.86	19.3	213	红色
实施例 12	化合物1-7	化合物2-14	3.82	19.8	212	红色
实施例 13	化合物1-7	化合物2-23	3.86	18.1	207	红色
实施例 14	化合物1-7	化合物2-58	3.83	19.5	218	红色
实施例 15	化合物1-7	化合物2-64	3.81	18.7	195	红色
实施例 16	化合物1-9	化合物2-17	3.88	19.3	203	红色
实施例 17	化合物1-9	化合物2-2	3.80	19.7	210	红色

[0900]

		0				
实施例18	化合物1-9	化合物2-28	3.84	19.0	207	红色
实施例19	化合物1-9	化合物2-35	3.89	18.6	201	红色
实施例20	化合物1-9	化合物2-57	3.86	19.2	195	红色
实施例21	化合物1-11	化合物2-1	3.85	18.4	203	红色
实施例22	化合物1-11	化合物2-27	3.81	18.6	205	红色
实施例23	化合物1-11	化合物2-39	3.80	18.7	197	红色
实施例24	化合物1-11	化合物2-54	3.88	19.0	206	红色
实施例25	化合物1-11	化合物2-60	3.83	18.9	194	红色
实施例26	化合物1-14	化合物2-3	3.89	20.1	213	红色
实施例27	化合物1-14	化合物2-10	3.87	20.3	215	红色
实施例28	化合物1-14	化合物2-68	3.88	20.2	201	红色
实施例29	化合物1-14	化合物2-44	3.86	19.5	217	红色
实施例30	化合物1-14	化合物2-49	3.86	20.4	220	红色
实施例31	化合物1-15	化合物2-5	3.79	19.3	208	红色
实施例32	化合物1-15	化合物2-14	3.72	20.2	211	红色
实施例33	化合物1-15	化合物2-23	3.74	20.7	210	红色
实施例34	化合物1-1	化合物2-5	3.70	19.8	203	红色

[0901]

	5	8				
实施例35	化合物1-15	化合物2-64	3.77	20.6	215	红色
实施例36	化合物1-16	化合物2-17	3.76	19.9	221	红色
实施例37	化合物1-16	化合物2-20	3.78	20.3	216	红色
实施例38	化合物1-16	化合物2-28	3.74	20.1	220	红色
实施例39	化合物1-16	化合物2-35	3.79	20.6	208	红色
实施例40	化合物1-16	化合物2-70	3.82	20.4	215	红色
实施例41	化合物1-17	化合物2-1	3.81	20.1	203	红色
实施例42	化合物1-17	化合物2-27	3.83	20.3	214	红色
实施例43	化合物1-17	化合物2-39	3.80	20.8	209	红色
实施例44	化合物1-17	化合物2-54	3.85	20.5	205	红色
实施例45	化合物1-17	化合物2-60	3.84	21.0	215	红色
实施例46	化合物1-20	化合物2-3	3.93	19.3	201	红色
实施例47	化合物1-20	化合物2-10	3.90	20.0	228	红色
实施例48	化合物1-20	化合物2-68	3.92	20.6	210	红色
实施例49	化合物1-20	化合物2-44	3.88	19.7	224	红色
实施例50	化合物1-20	化合物2-49	3.86	20.0	217	红色
实施例51	化合物1-2	化合物2-5	3.80	20.9	234	红色

[0902]

	2					
实施例52	化合物1-22	化合物2-14	3.81	20.5	221	红色
实施例53	化合物1-22	化合物2-23	3.87	19.9	216	红色
实施例54	化合物1-22	化合物2-58	3.86	20.6	224	红色
实施例55	化合物1-22	化合物2-64	3.93	20.4	225	红色
实施例56	化合物1-24	化合物2-17	3.78	20.3	217	红色
实施例57	化合物1-24	化合物2-20	3.84	21.1	230	红色
实施例58	化合物1-24	化合物2-28	3.80	21.4	228	红色
实施例59	化合物1-24	化合物2-35	3.83	20.7	233	红色
实施例60	化合物1-24	化合物2-57	3.85	20.9	215	红色
实施例61	化合物1-27	化合物2-1	3.76	20.5	238	红色
实施例62	化合物1-27	化合物2-27	3.82	19.3	211	红色
实施例63	化合物1-27	化合物2-39	3.79	19.6	223	红色
实施例64	化合物1-27	化合物2-54	3.78	19.1	226	红色
实施例65	化合物1-27	化合物2-60	3.82	20.3	230	红色
实施例66	化合物1-28	化合物2-3	3.88	19.7	227	红色
实施例67	化合物1-28	化合物2-10	3.84	20.8	218	红色
实施例68	化合物1-2	化合物2-6	3.78	19.3	215	红色

[0903]

	8	8				
实施例69	化合物1-28	化合物2-44	3.82	19.5	220	红色
实施例70	化合物1-28	化合物2-49	3.86	20.2	218	红色
实施例71	化合物1-31	化合物2-5	3.98	20.3	256	红色
实施例72	化合物1-31	化合物2-14	3.92	20.0	248	红色
实施例73	化合物1-31	化合物2-23	3.95	20.5	251	红色
实施例74	化合物1-31	化合物2-58	3.90	20.1	252	红色
实施例75	化合物1-31	化合物2-64	3.93	19.8	265	红色
实施例76	化合物1-33	化合物2-1	3.68	22.3	281	红色
实施例77	化合物1-33	化合物2-27	3.65	22.0	277	红色
实施例78	化合物1-33	化合物2-39	3.70	21.3	288	红色
实施例79	化合物1-33	化合物2-54	3.69	22.7	270	红色
实施例80	化合物1-33	化合物2-60	3.71	21.8	274	红色
实施例81	化合物1-37	化合物2-17	3.76	22.2	263	红色
实施例82	化合物1-37	化合物2-20	3.70	21.9	275	红色
实施例83	化合物1-37	化合物2-28	3.74	22.0	279	红色
实施例84	化合物1-37	化合物2-35	3.79	21.8	256	红色
实施例85	化合物1-3	化合物2-7	3.74	21.5	275	红色

[0904]

	7	0				
实施例86	化合物1-38	化合物2-3	3.72	21.0	275	红色
实施例87	化合物1-38	化合物2-10	3.70	22.1	271	红色
实施例88	化合物1-38	化合物2-68	3.75	21.5	284	红色
实施例89	化合物1-38	化合物2-44	3.74	21.3	268	红色
实施例90	化合物1-38	化合物2-49	3.71	22.3	275	红色
实施例91	化合物1-40	化合物2-5	3.73	20.7	225	红色
实施例92	化合物1-40	化合物2-14	3.70	19.1	238	红色
实施例93	化合物1-40	化合物2-23	3.76	19.7	231	红色
实施例94	化合物1-40	化合物2-58	3.74	19.0	240	红色
实施例95	化合物1-40	化合物2-64	3.80	19.4	234	红色
实施例96	化合物1-41	化合物2-17	3.83	19.0	241	红色
实施例97	化合物1-41	化合物2-20	3.87	19.5	236	红色
实施例98	化合物1-41	化合物2-28	3.83	18.9	228	红色
实施例99	化合物1-41	化合物2-35	3.86	19.8	234	红色
实施例100	化合物1-41	化合物2-57	3.79	19.4	225	红色
实施例101	化合物1-43	化合物2-1	3.81	19.5	197	红色
实施例	化合物1-4	化合物2-2	3.80	20.0	202	红色

[0905]

102	3	7				
实施例103	化合物1-43	化合物2-39	3.84	19.4	213	红色
实施例104	化合物1-43	化合物2-54	3.79	20.8	204	红色
实施例105	化合物1-43	化合物2-60	3.85	19.7	195	红色
实施例106	化合物1-45	化合物2-3	3.87	20.1	194	红色
实施例107	化合物1-45	化合物2-10	3.91	18.8	203	红色
实施例108	化合物1-45	化合物2-68	3.88	19.3	201	红色
实施例109	化合物1-45	化合物2-44	3.93	20.7	198	红色
实施例110	化合物1-45	化合物2-49	3.85	18.9	194	红色
实施例111	化合物1-47	化合物2-5	3.79	20.1	209	红色
实施例112	化合物1-47	化合物2-14	3.82	20.3	201	红色
实施例113	化合物1-47	化合物2-23	3.84	20.0	197	红色
实施例114	化合物1-47	化合物2-58	3.86	19.9	194	红色
实施例115	化合物1-47	化合物2-64	3.80	19.6	205	红色
实施例116	化合物1-48	化合物2-17	3.83	19.6	217	红色
实施例117	化合物1-48	化合物2-20	3.87	18.8	214	红色
实施例118	化合物1-48	化合物2-28	3.80	19.7	203	红色
实施例	化合物1-4	化合物2-3	3.88	19.4	198	红色

[0906]

119	8	5				
实施例120	化合物1-48	化合物2-70	3.92	18.9	201	红色
实施例121	化合物1-52	化合物2-1	3.91	19.6	197	红色
实施例122	化合物1-52	化合物2-27	3.94	19.3	190	红色
实施例123	化合物1-52	化合物2-39	3.92	19.2	203	红色
实施例124	化合物1-52	化合物2-54	3.88	18.9	194	红色
实施例125	化合物1-52	化合物2-60	3.94	19.7	191	红色
实施例126	化合物1-53	化合物2-3	3.87	18.8	203	红色
实施例127	化合物1-53	化合物2-10	3.90	19.1	209	红色
实施例128	化合物1-53	化合物2-68	3.86	18.6	198	红色
实施例129	化合物1-53	化合物2-44	3.93	19.0	204	红色
实施例130	化合物1-53	化合物2-49	3.90	19.3	195	红色
实施例131	化合物1-55	化合物2-5	3.87	20.3	223	红色
实施例132	化合物1-55	化合物2-14	3.82	19.9	224	红色
实施例133	化合物1-55	化合物2-23	3.83	20.6	218	红色
实施例134	化合物1-55	化合物2-58	3.84	20.4	208	红色
实施例135	化合物1-55	化合物2-64	3.90	19.7	225	红色
实施例	化合物1-5	化合物2-1	3.98	20.0	195	红色

[0907]

136	6	7				
实施例137	化合物1-56	化合物2-20	3.94	20.7	192	红色
实施例138	化合物1-56	化合物2-28	3.92	19.4	197	红色
实施例139	化合物1-56	化合物2-35	3.95	20.3	203	红色
实施例140	化合物1-56	化合物2-57	3.97	19.8	195	红色
实施例141	化合物1-57	化合物2-1	3.91	18.1	213	红色
实施例142	化合物1-57	化合物2-27	3.94	19.0	220	红色
实施例143	化合物1-57	化合物2-39	3.88	18.5	229	红色
实施例144	化合物1-57	化合物2-54	3.93	18.9	217	红色
实施例145	化合物1-57	化合物2-60	3.90	19.4	224	红色
实施例146	化合物1-58	化合物2-3	3.87	18.7	199	红色
实施例147	化合物1-58	化合物2-10	3.90	18.5	201	红色
实施例148	化合物1-58	化合物2-68	3.93	19.2	203	红色
实施例149	化合物1-58	化合物2-44	3.87	19.4	195	红色
实施例150	化合物1-58	化合物2-49	3.84	19.0	198	红色
实施例151	化合物1-60	化合物2-5	3.94	18.9	203	红色
实施例152	化合物1-60	化合物2-14	3.85	19.1	197	红色
实施例	化合物1-6	化合物2-2	3.88	18.5	211	红色

[0908]

153	0	3				
实施例154	化合物1-60	化合物2-58	3.95	19.6	207	红色
实施例155	化合物1-60	化合物2-64	3.91	19.4	208	红色

[0909] [表2]

[0910]

类别	主体	电子阻挡层	驱动电压（V）	效率（cd/A）	寿命T95（小时）	发光颜色
比较例 1	化合物1-2	化合物EB-1	4.37	14.8	117	红色
比较例 2	化合物1-2	化合物 2-1	4.39	14.0	101	红色
比较例 3	化合物1-2	化合物2-27	4.48	14.1	103	红色
比较例 4	化合物1-2	化合物2-39	4.49	14.8	98	红色
比较例 5	化合物1-2	化合物2-54	4.40	15.5	92	红色
比较例 6	化合物1-2	化合物2-60	4.43	15.1	111	红色
比较例 7	化合物1-11	化合物 2-3	4.36	15.2	114	红色
比较例 8	化合物1-11	化合物2-10	4.39	15.0	103	红色
比较例 9	化合物1-11	化合物2-68	4.41	15.3	104	红色
比较例10	化合物1-11	化合物2-44	4.42	14.4	107	红色
比较例11	化合物1-11	化合物2-49	4.39	14.9	106	红色
比较例12	化合物1-15	化合物 2-5	4.40	15.2	138	红色
比较例13	化合物1-15	化合物2-14	4.45	15.6	127	红色
比较例14	化合物1-15	化合物2-23	4.43	15.0	121	红色
比较例15	化合物1-15	化合物2-58	4.48	15.3	114	红色
比较例16	化合物1-15	化合物2-64	4.45	15.7	121	红色
比较例17	化合物1-17	化合物2-17	4.43	15.3	105	红色
比较例	化合物1-1	化合物2-20	4.44	15.4	103	红色

[0911]

18	7					
比较例19	化合物1-17	化合物2-28	4.46	15.4	110	红色
比较例20	化合物1-17	化合物2-35	4.41	14.8	107	红色
比较例21	化合物1-17	化合物2-70	4.44	14.9	111	红色
比较例22	化合物1-27	化合物 2-1	4.38	15.0	108	红色
比较例23	化合物1-27	化合物2-27	4.37	14.8	103	红色
比较例24	化合物1-27	化合物2-39	4.36	14.1	100	红色
比较例25	化合物1-27	化合物2-54	4.39	14.4	101	红色
比较例26	化合物1-27	化合物2-60	4.38	14.7	111	红色
比较例27	化合物1-37	化合物 2-3	4.37	15.3	106	红色
比较例28	化合物1-37	化合物2-10	4.39	15.4	108	红色
比较例29	化合物1-37	化合物2-68	4.38	15.3	117	红色
比较例30	化合物1-37	化合物2-44	4.37	15.0	104	红色
比较例31	化合物1-37	化合物2-49	4.36	15.1	111	红色
比较例32	化合物1-41	化合物 2-5	4.42	14.6	109	红色
比较例33	化合物1-41	化合物2-14	4.45	14.0	91	红色
比较例34	化合物1-41	化合物2-23	4.36	14.2	95	红色
比较例	化合物1-4	化合物2-58	4.39	14.3	108	红色

[0912]

35	1					
比较例36	化合物1-41	化合物2-64	4.35	14.0	94	红色
比较例37	化合物1-45	化合物2-17	4.38	14.1	92	红色
比较例38	化合物1-45	化合物2-20	4.39	14.5	98	红色
比较例39	化合物1-45	化合物2-28	4.37	14.3	95	红色
比较例40	化合物1-45	化合物2-35	4.36	14.7	91	红色
比较例41	化合物1-45	化合物2-57	4.38	14.0	89	红色
比较例42	化合物1-53	化合物 2-1	4.38	15.1	97	红色
比较例43	化合物1-53	化合物2-27	4.39	15.3	101	红色
比较例44	化合物1-53	化合物2-39	4.42	15.0	96	红色
比较例45	化合物1-53	化合物2-54	4.40	14.8	98	红色
比较例46	化合物1-53	化合物2-60	4.34	14.9	94	红色
比较例47	化合物1-55	化合物 2-3	4.46	14.7	104	红色
比较例48	化合物1-55	化合物2-10	4.38	15.0	107	红色
比较例49	化合物1-55	化合物2-68	4.39	14.8	106	红色
比较例50	化合物1-55	化合物2-44	4.47	15.3	104	红色
比较例51	化合物1-55	化合物2-49	4.42	15.0	112	红色
比较例	化合物1-5	化合物 2-5	4.41	14.1	101	红色

[0913]

52	7					
比较例53	化合物1-57	化合物2-14	4.40	14.9	107	红色
比较例54	化合物1-57	化合物2-23	4.43	14.6	108	红色
比较例55	化合物1-57	化合物2-58	4.39	14.7	99	红色
比较例56	化合物1-57	化合物2-64	4.45	15.0	101	红色
比较例57	化合物1-60	化合物2-17	4.40	14.5	91	红色
比较例58	化合物1-60	化合物2-20	4.41	15.0	99	红色
比较例59	化合物1-60	化合物2-28	4.42	14.3	104	红色
比较例60	化合物1-60	化合物2-35	4.44	15.1	96	红色
比较例61	化合物1-60	化合物2-70	4.39	14.5	93	红色

[0914] [表3]

[0915]

类别	第一主体	第二主体	驱动电压（V）	效率（cd/A）	寿命T95（小时）	发光颜色
比较例 62	化合物B-1	化合物2-1	4.31	13.1	91	红色
比较例 63	化合物B-1	化合物2-27	4.36	13.8	103	红色
比较例 64	化合物B-1	化合物2-39	4.34	13.6	97	红色
比较例 65	化合物B-1	化合物2-54	4.39	14.0	102	红色
比较例 66	化合物B-1	化合物2-60	4.36	13.9	95	红色
比较例 67	化合物B-2	化合物2-3	4.33	13.3	84	红色
比较例 68	化合物B-2	化合物2-10	4.40	13.8	93	红色
比较例 69	化合物B-2	化合物2-68	4.38	13.6	95	红色
比较例 70	化合物B-2	化合物2-44	4.31	13.9	87	红色
比较例 71	化合物B-2	化合物2-49	4.36	14.1	95	红色
比较例 72	化合物B-3	化合物2-5	4.40	13.2	108	红色
比较例 73	化合物B-3	化合物2-14	4.43	14.0	97	红色
比较例 74	化合物B-3	化合物2-23	4.41	13.4	101	红色
比较例 75	化合物B-3	化合物2-58	4.39	14.3	104	红色
比较例 76	化合物B-3	化合物2-64	4.42	14.2	101	红色
比较例 77	化合物B-4	化合物2-1	4.35	13.8	90	红色

[0916]

		7				
比较例 78	化合物B-4	化合物2-20	4.38	14.2	97	红色
比较例 79	化合物B-4	化合物2-28	4.36	13.6	91	红色
比较例 80	化合物B-4	化合物2-35	4.40	14.1	92	红色
比较例 81	化合物B-4	化合物2-57	4.42	13.7	89	红色
比较例 82	化合物B-5	化合物2-1	4.27	15.0	128	红色
比较例 83	化合物B-5	化合物2-27	4.29	14.8	127	红色
比较例 84	化合物B-5	化合物2-39	4.30	15.3	124	红色
比较例 85	化合物B-5	化合物2-54	4.27	14.9	121	红色
比较例 86	化合物B-5	化合物2-60	4.29	14.7	128	红色
比较例 87	化合物B-6	化合物2-3	4.30	15.3	114	红色
比较例 88	化合物B-6	化合物2-10	4.28	15.0	124	红色
比较例 89	化合物B-6	化合物2-68	4.26	15.5	117	红色
比较例 90	化合物B-6	化合物2-44	4.31	15.8	124	红色
比较例 91	化合物B-6	化合物2-49	4.27	14.6	111	红色
比较例 92	化合物B-7	化合物2-5	4.29	15.6	129	红色
比较例 93	化合物B-7	化合物2-14	4.31	14.7	131	红色
比较例 94	化合物B-7	化合物2-2	4.27	15.0	125	红色

[0917]

		3				
比较例 95	化合物B-7	化合物2-58	4.24	14.9	117	红色
比较例 96	化合物B-7	化合物2-64	4.28	14.6	124	红色
比较例 97	化合物B-8	化合物2-17	4.21	14.0	112	红色
比较例 98	化合物B-8	化合物2-20	4.29	15.3	116	红色
比较例 99	化合物B-8	化合物2-28	4.25	14.1	120	红色
比较例 100	化合物B-8	化合物2-35	4.23	15.2	109	红色
比较例 101	化合物B-8	化合物2-57	4.20	15.0	124	红色
比较例 102	化合物B-9	化合物2-1	4.11	15.5	143	红色
比较例 103	化合物B-9	化合物2-27	4.10	15.7	139	红色
比较例 104	化合物B-9	化合物2-39	4.13	15.8	137	红色
比较例 105	化合物B-9	化合物2-54	4.15	14.9	130	红色
比较例 106	化合物B-9	化合物2-60	4.09	15.1	141	红色
比较例 107	化合物 B-10	化合物2-3	4.13	15.6	137	红色
比较例 108	化合物B-10	化合物2-10	4.15	16.1	126	红色
比较例 109	化合物B-10	化合物2-68	4.17	16.0	134	红色
比较例 110	化合物B-10	化合物2-44	4.10	15.7	126	红色
比较例	化合物B-1	化合物2-4	4.14	15.4	129	红色

[0918]

111	0	9				
比较例112	化合物B-11	化合物2-5	4.10	15.8	142	红色
比较例113	化合物B-11	化合物2-14	4.09	16.3	137	红色
比较例114	化合物B-11	化合物2-23	4.17	15.9	130	红色
比较例115	化合物B-11	化合物2-58	4.16	16.4	124	红色
比较例116	化合物B-11	化合物2-64	4.18	16.1	132	红色
比较例117	化合物B-12	化合物2-17	4.09	16.0	147	红色
比较例118	化合物B-12	化合物2-20	4.12	16.5	129	红色
比较例119	化合物B-12	化合物2-28	4.08	16.3	144	红色
比较例120	化合物B-12	化合物2-35	4.17	15.7	136	红色
比较例121	化合物B-12	化合物2-70	4.13	15.4	141	红色

[0919] [表4]

[0920]

类别	第一主体	第二主体	驱动电压（V）	效率（cd/A）	寿命T95（小时）	发光颜色
比较例122	化合物1-2	化合物C-1	4.21	15.0	131	红色
比较例123	化合物1-11	化合物C-1	4.26	14.8	123	红色
比较例124	化合物1-15	化合物C-1	4.17	15.3	117	红色
比较例125	化合物1-28	化合物C-1	4.25	14.9	128	红色
比较例126	化合物1-33	化合物C-1	4.16	14.7	105	红色
比较例127	化合物1-40	化合物C-1	4.23	15.3	122	红色
比较例128	化合物1-43	化合物C-1	4.30	15.8	111	红色
比较例129	化合物1-55	化合物C-1	4.28	14.6	130	红色
比较例130	化合物1-3	化合物C-2	4.20	14.0	114	红色
比较例131	化合物1-7	化合物C-2	4.16	14.8	105	红色
比较例132	化合物1-17	化合物C-2	4.30	15.3	109	红色
比较例133	化合物 1-24	化合物C-2	4.13	14.9	102	红色
比较例134	化合物1-37	化合物C-2	4.21	15.7	123	红色
比较例135	化合物1-47	化合物C-2	4.19	16.0	127	红色
比较例136	化合物1-48	化合物C-2	4.22	15.4	120	红色
比较例	化合物1-5	化合物C-2	4.31	15.1	114	红色

[0921]

137	8					
比较例138	化合物1-9	化合物C-3	4.18	14.3	135	红色
比较例139	化合物1-16	化合物C-3	4.26	15.1	134	红色
比较例140	化合物1-22	化合物C-3	4.20	14.7	129	红色
比较例141	化合物1-38	化合物C-3	4.12	14.0	135	红色
比较例142	化合物1-41	化合物C-3	4.17	14.9	124	红色
比较例143	化合物1-45	化合物C-3	4.24	15.1	132	红色
比较例144	化合物1-53	化合物C-3	4.22	14.8	127	红色
比较例145	化合物1-57	化合物C-3	4.22	15.6	121	红色
比较例146	化合物1-2	化合物C-4	4.19	17.0	141	红色
比较例147	化合物1-14	化合物C-4	4.20	16.8	135	红色
比较例148	化合物1-20	化合物C-4	4.21	15.7	149	红色
比较例149	化合物1-27	化合物C-4	4.16	17.9	136	红色
比较例150	化合物 1-31	化合物C-4	4.11	16.3	148	红色
比较例151	化合物1-52	化合物C-4	4.17	17.3	152	红色
比较例152	化合物1-56	化合物C-4	4.19	16.8	134	红色
比较例153	化合物1-60	化合物C-4	4.15	17.6	141	红色
比较例	化合物1-2	化合物C-5	4.37	13.0	81	红色

[0922]

154						
比较例155	化合物1-11	化合物C-5	4.34	13.9	92	红色
比较例156	化合物1-15	化合物C-5	4.38	13.1	89	红色
比较例157	化合物1-28	化合物C-5	4.41	12.8	93	红色
比较例158	化合物1-33	化合物C-5	4.39	13.3	95	红色
比较例159	化合物1-40	化合物C-5	4.31	13.8	87	红色
比较例160	化合物1-43	化合物C-5	4.33	13.2	84	红色
比较例161	化合物1-55	化合物C-5	4.34	13.0	80	红色
比较例162	化合物1-3	化合物C-6	4.31	14.1	86	红色
比较例163	化合物1-7	化合物C-6	4.30	14.4	80	红色
比较例164	化合物1-17	化合物C-6	4.35	15.1	93	红色
比较例165	化合物1-24	化合物C-6	4.33	14.7	92	红色
比较例166	化合物1-37	化合物C-6	4.31	14.6	95	红色
比较例167	化合物 1-47	化合物C-6	4.29	15.3	97	红色
比较例168	化合物1-48	化合物C-6	4.32	14.8	92	红色
比较例169	化合物1-58	化合物C-6	4.30	15.3	86	红色
比较例170	化合物1-9	化合物C-7	4.29	16.6	129	红色
比较例	化合物1-1	化合物C-7	4.23	16.1	131	红色

[0923]

171	6					
比较例172	化合物1-22	化合物C-7	4.27	16.5	134	红色
比较例173	化合物1-38	化合物C-7	4.28	15.8	140	红色
比较例174	化合物1-41	化合物C-7	4.20	15.9	137	红色
比较例175	化合物1-45	化合物C-7	4.29	16.3	143	红色
比较例176	化合物1-53	化合物C-7	4.30	16.8	129	红色
比较例177	化合物1-57	化合物C-7	4.27	16.2	143	红色
比较例178	化合物1-2	化合物C-8	4.15	17.2	132	红色
比较例179	化合物1-14	化合物C-8	4.13	17.3	124	红色
比较例180	化合物1-20	化合物C-8	4.21	16.9	126	红色
比较例181	化合物1-27	化合物C-8	4.18	16.2	119	红色
比较例182	化合物1-31	化合物C-8	4.12	16.1	137	红色
比较例183	化合物1-52	化合物C-8	4.17	16.7	124	红色
比较例184	化合物 1-56	化合物C-8	4.20	17.2	130	红色
比较例185	化合物1-60	化合物C-8	4.19	17.5	142	红色
比较例186	化合物1-2	化合物C-9	4.21	15.3	102	红色
比较例187	化合物1-11	化合物C-9	4.16	15.6	107	红色
比较例	化合物1-1	化合物C-9	4.24	15.1	105	红色

[0924]

188	5					
比较例189	化合物1-28	化合物C-9	4.27	16.0	94	红色
比较例190	化合物1-33	化合物C-9	4.19	15.3	103	红色
比较例201	化合物1-40	化合物C-9	4.22	15.8	98	红色
比较例202	化合物1-43	化合物C-9	4.26	14.7	91	红色
比较例203	化合物1-55	化合物C-9	4.27	14.9	97	红色
比较例204	化合物1-3	化合物C-10	4.20	16.9	106	红色
比较例205	化合物1-7	化合物C-10	4.21	15.4	100	红色
比较例206	化合物1-17	化合物C-10	4.19	16.8	109	红色
比较例207	化合物1-24	化合物C-10	4.22	15.0	98	红色
比较例208	化合物1-37	化合物C-10	4.17	16.2	112	红色
比较例209	化合物1-47	化合物C-10	4.26	16.7	101	红色
比较例210	化合物1-48	化合物C-10	4.28	16.0	94	红色
比较例211	化合物 1-58	化合物C-10	4.26	15.4	110	红色
比较例212	化合物1-9	化合物C-11	4.31	17.2	116	红色
比较例213	化合物1-16	化合物C-11	4.30	16.6	121	红色
比较例214	化合物1-22	化合物C-11	4.35	17.1	108	红色
比较例	化合物1-3	化合物C-1	4.35	16.4	129	红色

[0925]

215	8	1				
比较例216	化合物1-41	化合物C-11	4.34	17.8	120	红色
比较例217	化合物1-45	化合物C-11	4.31	17.8	113	红色
比较例218	化合物1-53	化合物C-11	4.32	16.5	114	红色
比较例219	化合物1-57	化合物C-11	4.39	16.9	109	红色
比较例220	化合物1-2	化合物C-12	4.18	16.7	103	红色
比较例221	化合物1-14	化合物C-12	4.21	17.0	92	红色
比较例222	化合物1-20	化合物C-12	4.23	17.4	99	红色
比较例223	化合物1-27	化合物C-12	4.20	16.8	108	红色
比较例224	化合物1-31	化合物C-12	4.27	17.2	96	红色
比较例225	化合物1-52	化合物C-12	4.26	17.3	91	红色
比较例226	化合物1-56	化合物C-12	4.24	17.5	105	红色
比较例227	化合物 1-60	化合物C-12	4.19	16.1	112	红色

[0926] 当向根据实施例1至155和比较例1至227制造的有机发光器件施加电流时,获得以上表1至表4的结果。在比较例1的有机发光器件中,使用了常规广泛使用的材料。

[0927] 在比较例2至61中,通过使用本公开内容的由化学式2表示的化合物作为电子阻挡层并使用单一主体作为发光层以与比较例1中相同的方式来制造有机发光器件。当如表1的实施例中将由化学式1表示的化合物和由化学式2表示的化合物共沉积并用作发光层时,确定了与表2的比较例相比,驱动电压降低并且效率和寿命增加。

[0928] 此外,当如表3中所示将比较例化合物B-1至B-12和本公开内容的由化学式2表示的化合物共沉积并用作发光层时,结果显示与本公开内容的组合相比,驱动电压通常增加并且效率和寿命降低。如表4中所示,即使当将比较例化合物C-1至C-12和本公开内容的由化学式1表示的化合物共沉积并用作发光层时,结果显示驱动电压增加并且效率和寿命降低。

[0929] 从这些结果可以看出,当组合使用作为第一主体的由化学式1表示的化合物和作为第二主体的由化学式2表示的化合物时,向发光层中红色掺杂剂的能量转移很好地进行,从而改善驱动电压并且增加效率和寿命。此外,可以推断,实施例的化合物的组合在发光层内可以形成比比较例的化合物的组合更稳定的平衡,以通过电子和空穴的组合形成激子,从而进一步改善制造的有机发光器件的效率和寿命。

[0930] 即,当组合使用本公开内容的由化学式1表示的化合物和由化学式2表示的化合物作为发光层的主体时,有机发光器件的驱动电压、发光效率和寿命特性可以得到改善。

[0931] <附图标记>

[0932] 1:基底 2:阳极

[0933] 3:发光层 4:阴极

[0934] 5:空穴注入层 6:空穴传输层

[0935] 7:电子阻挡层 8:空穴阻挡层

[0936] 9:电子注入和传输层

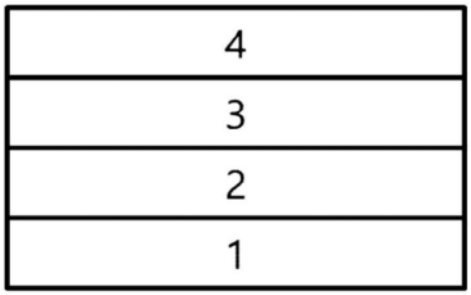


图1

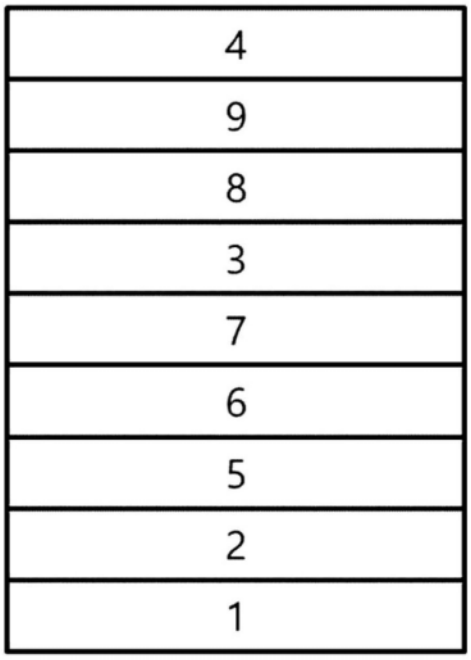


图2