



(22) Date de dépôt/Filing Date: 1997/07/04

(41) Mise à la disp. pub./Open to Public Insp.: 1998/03/27

(45) Date de délivrance/Issue Date: 2003/09/23

(30) Priorité/Priority: 1996/09/27 (08/721,817) US

(51) Cl.Int.⁶/Int.Cl.⁶ C09D 177/00, G03G 7/00

(72) Inventeurs/Inventors:
SCHANK, RICHARD L., US;
RENFER, DALE S., US;
LIMBURG, WILLIAM W., US;
KUNZMANN, BRENDAN W., US;
PAI, DAMODAR M., US

(73) Propriétaire/Owner:
XEROX CORPORATION, US

(74) Agent: SIM & MCBURNEY

(54) Titre : COMPOSITIONS ET REVETEMENTS DE PHOTORECEPTEUR CONTENANT UN DIHYDROXY ARYLAMIDE
ET UN POLYAMIDE RETICULE

(54) Title: COMPOSITIONS AND PHOTORECEPTOR OVERCOATINGS CONTAINING A DIHYDROXY ARYLAMINE
AND A CROSSLINKED POLYAMIDE

(57) **Abrégé/Abstract:**

An electrophotographic imaging member including a supporting substrate coated with at least a charge generating layer, a charge transport layer and an overcoating layer, said overcoating layer comprising a dihydroxy arylamine dissolved or molecularly dispersed in a crosslinked polyamide matrix. The overcoating layer is formed by crosslinking a crosslinkable coating composition including a polyamide containing methoxy methyl groups attached to amide nitrogen atoms, a crosslinking catalyst and a dihydroxy amine, and heating the coating to crosslink the polyamide. The electrophotographic imaging member may be imaged in a process involving uniformly charging the imaging member, exposing the imaging member with activating radiation in image configuration to form an electrostatic latent image, developing the latent image with toner particles to form a toner image, and transferring the toner image to a receiving member.



ABSTRACT

An electrophotographic imaging member including a supporting substrate coated with at least a charge generating layer, a charge transport layer and an overcoating layer, said overcoating layer comprising a dihydroxy arylamine dissolved or molecularly dispersed in a crosslinked polyamide matrix. The overcoating layer is formed by crosslinking a crosslinkable coating composition including a polyamide containing methoxy methyl groups attached to amide nitrogen atoms, a crosslinking catalyst and a dihydroxy amine, and heating the coating to crosslink the polyamide. The electrophotographic imaging member may be imaged in a process involving uniformly charging the imaging member, exposing the imaging member with activating radiation in image configuration to form an electrostatic latent image, developing the latent image with toner particles to form a toner image, and transferring the toner image to a receiving member.

PATENT APPLICATION

Attorney's Docket No. D/95643

**COMPOSITIONS AND PHOTORECEPTOR OVERCOATINGS CONTAINING A
DIHYDROXY ARYLAMINE AND A CROSSLINKED POLYAMIDE**

BACKGROUND OF THE INVENTION

This invention relates in general to coating compositions and more specifically, to compositions and coated articles containing a dihydroxy arylamine and a crosslinked polyamide.

Electrophotographic imaging members, i.e. photoreceptors, typically include a photoconductive layer formed on an electrically conductive substrate. The photoconductive layer is a good insulator in the dark so that electric charges are retained on its surface. Upon exposure to light, the charge is dissipated.

An electrostatic latent image is formed on the photoreceptor by first uniformly depositing an electric charge over the surface of the photoconductive layer by one of any suitable means well known in the art. The photoconductive layer functions as a charge storage capacitor with charge on its free surface and an equal charge of opposite polarity (the counter charge) on the conductive substrate. A light image is then projected onto the photoconductive layer. On those portions of the photoconductive layer that are exposed to light, the electric charge is conducted through the layer reducing the surface charge. The portions of the surface of the photoconductive not exposed to light retain their surface charge. The quantity of electric charge at any particular area of the photoconductive surface is inversely related to the illumination incident thereon, thus forming an electrostatic latent image. After development of the latent image with toner particles to form a toner image, the toner image is usually transferred to a receiving member such as paper. Transfer is effected by various means such as by electrostatic transfer during which an electrostatic charge is applied to the back side of the receiving member while the front side of the member is in contact with the toner image.

The photodischarge of the photoconductive layer requires that the layer photogenerate free charge carriers and transport this charge through the layer thereby neutralizing the charge on the surface. Two types of photoreceptor structures have been employed: multilayer structures wherein separate layers perform the functions of charge generation and charge transport, respectively, and single layer photoconductors which perform both functions. These layers are formed on an electrically conductive substrate and may include an optional charge blocking and an adhesive layer between the conductive layer and the photoconducting layer or layers. Additionally, the substrate may comprise a non-conducting mechanical support with a conductive surface. Other layers for providing special functions such as incoherent reflection of laser light, dot patterns for pictorial imaging or subbing layers to provide chemical sealing and/or a smooth coating surface may optionally be employed.

One common type of photoreceptor is a multilayered device that comprises a conductive layer, a blocking layer, an adhesive layer, a charge generating layer, and a charge transport layer. The charge transport layer can contain an active aromatic diamine molecule, which enables charge transport, dissolved or molecularly dispersed in a film forming binder. This type of charge transport layer is described, for example in US-A 4,265,990. Other charge transport molecules disclosed in the prior art include a variety of electron donor, aromatic amines, oxadiazoles, oxazoles, hydrazones and stilbenes for hole transport and electron acceptor molecules for electron transport. Another type of charge transport layer has been developed which utilizes a charge transporting polymer wherein the charge transporting moiety is incorporated in the polymer as a group pendant from the backbone of the polymer backbone or as a moiety in the backbone of the polymer. These types of charge transport polymers include materials such as poly(N-vinylcarbazole), polysilylenes, and others including those described, for example, in US-A 4,618,551, 4,806,443, 4,806,444, 4,818,650, 4,935,487, and 4,956,440.

Charge generator layers comprise amorphous films of selenium and alloys of selenium and arsenic, tellurium, germanium and the like, hydrogenated amorphous silicon and compounds of silicon and germanium, carbon, oxygen, nitrogen and the like fabricated by vacuum evaporation or deposition. The charge generator layers may also comprise inorganic pigments of crystalline selenium and its alloys; Group II-VI compounds; and organic pigments such as quinacridones, polycyclic pigments such as dibromo anthanthrone pigments, perylene and perinone diamines, polynuclear aromatic quinones, azo pigments including bis-, tris- and tetrakis-azos; and the like dispersed in a film forming polymeric binder and fabricated by solvent coating techniques.

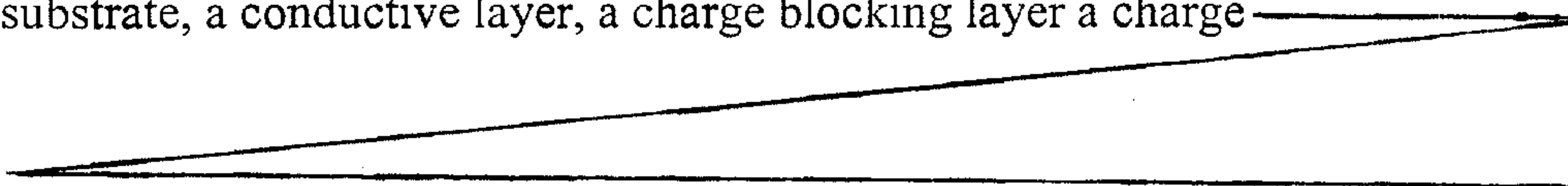
Phthalocyanines have been employed as photogenerating materials for use in laser printers utilizing infrared exposure systems. Infrared sensitivity is required for photoreceptors exposed to low cost semiconductor laser diode light exposure devices. The absorption spectrum and photosensitivity of the phthalocyanines depend on the central metal atom of the compound. Many metal phthalocyanines have been reported and include, oxyvanadium phthalocyanine, chloroaluminum phthalocyanine, copper phthalocyanine, oxytitanium phthalocyanine, chlorogallium phthalocyanine, magnesium phthalocyanine and metal-free phthalocyanine. The phthalocyanines exist in many crystal forms which have a strong influence on photogeneration.

One of the design criteria for the selection of the photosensitive pigment for a charge generator layer and the charge transporting molecule for a transport layer is that, when light photons photogenerate holes in the pigment, the holes be efficiently injected into the charge transporting molecule in the transport layer. More specifically, the injection efficiency from the pigment to the transport layer should be high. A second design criterion is that the injected holes be transported across the charge transport layer in a short time; shorter than the time duration between the exposure and development stations in an imaging device. The transit time across the transport layer is determined by the charge carrier mobility in the transport layer. The charge carrier mobility is the velocity per unit field and

has dimensions of $\text{cm}^2/\text{volt sec}$. The charge carrier mobility is a function of the structure of the charge transporting molecule, the concentration of the charge transporting molecule in the transport layer and the electrically "inactive" binder polymer in which the charge transport molecule is dispersed.

5 Reprographic machines often utilize multilayered organic photoconductors and can also employ corotrons, scorotrons or bias charging rolls to charge the photoconductors prior to imagewise exposure. Further, corotrons, scorotrons or bias transfer rolls may be utilized to transfer toner images from a photoreceptor to a receiving member. Bias transfer rolls for charging purposes
10 have the advantage that they generally emit less ozone than corotrons and scorotrons. It has been found that as the speed and number of imaging of copiers, duplicators and printers are increased, bias transfer rolls and bias charge rolls can cause serious wear problems to the photoreceptors. Bias transfer rolls and bias charge rolls are known in the art. Bias transfer rolls, which are similar to bias
15 charge rolls, are described, for example in US-A 5,420,677, US-A 5,321, 476 and US-A 5,303,014. As a consequence of the abrasive action of the bias transfer rolls and bias charge rolls charge rollers, the operating lifetime of conventional photoreceptors is severely reduced. In a test conducted on a normally abrasion resistant non crosslinked overcoated photoreceptor composition, introduction of
20 bias transfer roll and bias charge roll subsystems causes a greater than eight fold increase in wear of of the overcoated photoreceptor. The precise nature of the electrical/abrasive wearing away of the charge transport layer thickness is unknown, but it is theorized that some degradative process involving charge scission of the binder occurs, or in the case of arylamine hole transporting
25 polymers, the reduction in chain lengths causes the polymers to lose their inherent strength.

As described above, one type of multilayered photoreceptor that has been employed as a belt in electrophotographic imaging systems comprises a substrate, a conductive layer, a charge blocking layer a charge



generating layer, and a charge transport layer. The charge transport layer often comprises an activating small molecule dispersed or dissolved in an polymeric film forming binder. Generally, the polymeric film forming binder in the transport layer is electrically inactive by itself and becomes electrically active when it contains the activating molecule. The expression "electrically active" means that the material is capable of supporting the injection of photogenerated charge carriers from the material in the charge generating layer and is capable of allowing the transport of these charge carriers through the electrically active layer in order to discharge a surface charge on the active layer. The multilayered type of photoreceptor may also comprise additional layers such as an anticurl backing layer, an adhesive layer, and an overcoating layer. Although excellent toner images may be obtained with multilayered belt photoreceptors that are developed with dry developer powder (toner), it has been found that these same photoreceptors become unstable when employed with liquid development systems. These photoreceptors suffer from cracking, crazing, crystallization of active compounds, phase separation of activating compounds and extraction of activating compounds caused by contact with the organic carrier fluid, isoparaffinic hydrocarbons e.g. Isopar, commonly employed in liquid developer inks which, in turn, markedly degrade the mechanical integrity and electrical properties of the photoreceptor. More specifically, the organic carrier fluid of a liquid developer tends to leach out activating small molecules, such as the arylamine containing compounds typically used in the charge transport layers. Representative of this class of materials are: N,N'-diphenyl-N,N'-bis(3-methylphenyl)-[1,1'-biphenyl]-4,4'-diamine; bis-(4-diethylamino-2-methylphenyl)-phenylmethane; 2,5-bis-(4'-dimethylaminophenyl)-1,3,4-oxadiazole; 1-phenyl-3-(4'-diethylaminostyryl)-5-(4"-diethylaminophenyl)-pyrazoline; 1,1-bis-(4-(di-N,N'-p-methylphenyl)-aminophenyl)-cyclohexane; 4-diethylaminobenzaldehyde-1,1-diphenylhydrazone; 1,1-diphenyl-2(p-N,N-diphenyl amino phenyl)-ethylene; N-ethylcarbazole-3-carboxaldehyde-1-methyl-1-phenylhydrazone. The leaching process results in crystallization of the activating small molecules, such as the aforementioned arylamine

compounds, onto the photoreceptor surface and subsequent migration of arylamines into the liquid developer ink. In addition, the ink vehicle, typically a C₁₀-C₁₄ branched hydrocarbon, induces the formation of cracks and crazes in the photoreceptor surface. These effects lead to copy defects and shortened photoreceptor life. The degradation of the photoreceptor manifests itself as increased background and other printing defects prior to complete physical photoreceptor failure. The leaching out of the activating small molecule also increases the susceptibility of the transport layer to solvent/stress cracking when the belt is parked over a belt support roller during periods of non-use. Some carrier fluids may also promote phase separation of the activating small molecules, such as arylamine compounds, in the transport layers, particularly when high concentrations of the arylamine compounds are present in the transport layer binder. Phase separation of activating small molecules also adversely alters the electrical and mechanical properties of a photoreceptor. Similarly, single layer photoreceptors having a single active layer comprising photoconductive particles dispersed in a charge transport film forming binder are also vulnerable to the same degradation problems encountered by the previously described multilayered type of photoreceptor when exposed to liquid developers. Although flexing is normally not encountered with rigid, cylindrical, multilayered photoreceptors which utilize charge transport layers containing activating small molecules dispersed or dissolved in a polymeric film forming binder, electrical degradation are similarly encountered during development with liquid developers. Sufficient degradation of these photoreceptors by liquid developers can occur in less than two hours as indicated by leaching of the small molecule and cracking of the matrix polymer film. Continued exposure for several days severely damages the photoreceptor. Thus, in advanced imaging systems utilizing multilayered belt photoreceptors exposed to liquid development systems, cracking and crazing have been encountered in critical charge transport layers during belt cycling. Cracks developing in charge transport layers during cycling can be manifested as print-out defects adversely affecting copy quality. Furthermore, cracks in the photoreceptor pick up toner

particles which cannot be removed in the cleaning step and may be transferred to the background in subsequent prints. In addition, crack areas are subject to delamination when contacted with blade cleaning devices thus limiting the options in electrophotographic product design.

Photoreceptors have been developed which comprise charge transfer complexes prepared with polymeric molecules. For example, charge transfer complexes formed with polyvinyl carbazole are disclosed in US-A 4,047,948, US-A 4,346,158 and US-A 4,388,392. Photoreceptors utilizing polyvinyl carbazole layers, as compared with current photoreceptor requirements, exhibit relatively poor xerographic performance in both electrical and mechanical properties. Polymeric arylamine molecules prepared from the condensation of di-secondary amine with a di-iodo aryl compound are disclosed in European patent publication 34,425, published August 26, 1981, issued May 16, 1984. Since these polymers are extremely brittle and form films which are very susceptible to physical damage, their use in a flexible belt configuration is precluded. Thus, in advanced imaging systems utilizing multilayered belt photoreceptors exposed to liquid development systems, cracking and crazing have been encountered in critical charge transport layers during belt cycling. Still other arylamine charge transporting polymers such as those disclosed in US-A 4,806,444, US-A 4,806,443, US-A 4,935,487, and US-A 5,030,532 are vulnerable to reduced life because of the highly abrasive conditions presented by imaging systems utilizing bias transfer rolls and/or bias charge rollers.

Protective overcoatings can be somewhat helpful against abrasion. However, most protective overcoatings also fail early when subjected to the highly abrasive conditions presented by imaging systems utilizing bias transfer rolls and/or bias charge rollers. Moreover, many overcoatings tend to accumulate residual charge during cycling. This can cause a condition known as cycle-up in which the residual potential continues to increase with multi-cycle operation. This can give rise to increased densities in the background areas of the final images.

INFORMATION DISCLOSURE STATEMENT

US-A 4,871,634 to W. Limburg et al., issued Oct. 3, 1989 - A hydroxy arylamine compound, represented by a specific formula, is disclosed as employable in photoreceptors. The hydroxy arylamine compound can be used as an overcoating with hydroxy arylamine compound bonded to a resin capable of hydrogen bonding such as a polyamide possessing alcohol solubility.

US-A 5,368,967 to R. Shank et al., issued November 29, 1994 - An overcoat layer is disclosed comprising a small molecule hole transporting arylamine having at least two hydroxy functional groups, a hydroxy or multihydroxy triphenyl methane and a polyamide film forming binder capable of forming hydrogen bonds with the hydroxy functional groups of the hydroxy arylamine and hydroxy or multihydroxy triphenyl methane.

CROSS REFERENCE TO RELATED APPLICATIONS

This application is related to the following US-A Patent Applications:

In US Patent 5,612,157 entitled "Charge Blocking Layer For Electrophotographic Imaging Member" - An electrophotographic imaging member is disclosed comprising a substrate, a hole blocking layer comprising a hydrogen bonding or reaction product of a hydrolyzed metal alkoxide molecule or hydrolyzed metal aryloxide molecule and a film forming alcohol soluble nylon polymer containing carboxylic acid amide groups in the polymer backbone, a charge generating layer, and a charge transport layer.

US-A Patent 5,681,679 entitled "OVERCOATED ELECTROPHOTOGRAPHIC IMAGING MEMBER WITH RESILIENT CHARGE TRANSPORT LAYER" - A flexible electrophotographic imaging member is disclosed free of an anticurl backing layer, the imaging member including a supporting substrate uncoated on one side and coated on the opposite side with at least a charge generating layer, a charge transport layer and an overcoating layer, the transport layer including a resilient hole transporting arylamine siloxane polymer and the overcoating

including a polyamide crosslinked with a dihydroxy amine, forming an electrostatic latent image on the imaging member, depositing toner particles on the imaging member in conformance with the latent image to form a toner image, and transferring the toner image to a receiving member. This imaging member may be utilized in an imaging process including forming an electrostatic latent image on the imaging member, depositing toner particles on the imaging member in conformance with the latent image to form a toner image, and transferring the toner image to a receiving member.

US-A Patent 5,670,291 entitled "PROCESS FOR FABRICATING AN ELECTROPHOTOGRAPHIC IMAGING MEMBER" - A process is disclosed for fabricating an electrophotographic imaging member including providing a substrate coated with at least one photoconductive layer, applying a coating composition to the photoconductive layer by dip coating to form a wet layer, the coating composition including finely divided silica particles, a dihydroxy amine charge transport material, an aryl amine charge transport material that is different from the dihydroxy amine charge transport material, a crosslinkable polyamide containing methoxy groups attached to amide nitrogen atoms, a crosslinking catalyst, and at least one solvent for the hydroxy amine charge transport material, aryl amine charge transport material and the crosslinkable polyamide, and heating the wet layer to crosslink the polyamide and remove the solvent to form a dry layer in which the dihydroxy amine charge transport material and the aryl amine charge transport material that is different from the dihydroxy amine charge transport material are molecularly dispersed in a crosslinked polyamide matrix.

US-A Patent 5,709,974 entitled "HIGH SPEED ELECTROPHOTOGRAPHIC IMAGING MEMBER" - An electrophotographic imaging member is disclosed comprising a supporting substrate, a charge generating layer, a charge transport layer and an overcoating layer, the transport layer comprising a charge transporting molecule in a polystyrene matrix and said overcoating layer comprising a film forming

polyamide and a hydroxyaryl amine.

Thus, there is a continuing need for photoreceptors having improved resistance to abrasive cycling conditions and increased densities in the background areas of the final images, and cyclic instabilities. There is also
5 continuing need for improved photoconductors usable in a liquid ink environment.

SUMMARY OF THE INVENTION

It is, therefore, an object of the present invention to provide an
10 improved electrophotographic imaging member which overcomes the above-noted deficiencies.

It is yet another object of the present invention to provide an improved electrophotographic imaging member capable of longer cycling life under abrasive imaging conditions.

15 It is yet another object of the present invention to provide an improved electrophotographic imaging member capable of longer cycling life under abrasive toner/cleaning blade interactions.

It is still another object of the present invention to provide an improved electrophotographic imaging member that is stable against cycle up.

20 It is another object of the present invention to provide an improved electrophotographic imaging member that resists cracking in a liquid development environment.

It is yet another object of the present invention to provide an improved electrophotographic imaging member exhibiting resistance against
25 rough handling in a copier environment.

It is yet another object of the present invention to provide an improved electrophotographic imaging member exhibiting resistance against rough handling during installation and service.

The foregoing objects and others are accomplished in
30 accordance with this invention by providing an electrophotographic imaging member comprising a supporting substrate coated with at least a charge

generating layer, a charge transport layer and an overcoating layer, the overcoating layer comprising a dihydroxy arylamine dissolved or molecularly dispersed in a crosslinked polyamide matrix. The overcoating layer is formed by crosslinking a crosslinkable coating composition comprising an alcohol
5 soluble polyamide containing methoxy methyl groups attached to amide nitrogen atoms, a crosslinking catalyst and a dihydroxy arylamine. The electrophotographic imaging member may be imaged in a process involving uniformly charging the imaging member, exposing the imaging member with activating radiation in image configuration to form an electrostatic latent
10 image, developing the latent image with toner particles to form a toner image, and transferring the toner image to a receiving member.

According to an aspect of the present invention there is provided an electrophotographic imaging member which comprises of a supporting substrate coated with at least a charge generating layer, a charge
15 transport layer and an overcoating layer, the overcoating layer which comprises of a dihydroxy arylamine dissolved or molecularly dispersed in a crosslinked polyamide matrix.

According to another aspect of the present invention there is provided a crosslinkable coating composition which comprises of an alcohol
20 soluble polyamide containing methoxy methyl groups attached to amide nitrogen atoms, a crosslinking catalyst and a dihydroxy arylamine.

According to yet another aspect of the present invention there is a method of forming a coating which comprises of providing a substrate, forming a coating of a crosslinkable composition on the substrate, the
25 crosslinkable coating composition which comprises of a polyamide containing methoxy methyl groups attached to amide nitrogen atoms, a crosslinking catalyst and a dihydroxy amine, and heating the coating to crosslink the polyamide.

According to an aspect of the present invention there is
30 provided an electrophotographic imaging process which comprises of

providing an electrophotographic imaging member which comprises of a supporting substrate coated with at least a charge generating layer, a charge transport layer and an overcoating layer, the overcoating layer which comprises of a dihydroxy arylamine dissolved or molecularly dispersed in a crosslinked polyamide matrix, uniformly charging the imaging member, exposing the imaging member with activating radiation in image configuration to form an electrostatic latent image, developing the latent image with toner particles to form a toner image, and transferring the toner image to a receiving member.

Electrophotographic imaging members are well known in the art. Electrophotographic imaging members may be prepared by any suitable technique. Typically, a flexible or rigid substrate is provided with an electrically conductive surface. A charge generating layer is then applied to the electrically conductive surface. A charge blocking layer may optionally be applied to the electrically conductive surface prior to the application of a charge generating layer. If desired, an adhesive layer may be utilized between the charge blocking layer and the charge generating layer. Usually the charge generation layer is applied onto the blocking layer and a charge transport layer is formed on the charge generation layer. This structure may have the charge generation layer on top of or below the charge transport layer.

The substrate may be opaque or substantially transparent and may comprise any suitable material having the required mechanical properties. Accordingly, the substrate may comprise a layer of an electrically non-conductive or conductive material such as an inorganic or an organic composition. As electrically non-conducting materials there may be employed various resins known for this purpose including polyesters,

polycarbonates, polyamides, polyurethanes, and the like which are flexible as thin webs. An electrically conducting substrate may be any metal, for example, aluminum, nickel, steel, copper, and the like or a polymeric material, as described above, filled with an electrically conducting substance, such as carbon, metallic powder, and the like or an organic electrically conducting material. The electrically insulating or conductive substrate may be in the form of an endless flexible belt, a web, a rigid cylinder, a sheet and the like.

The thickness of the substrate layer depends on numerous factors, including strength desired and economical considerations. Thus, for a drum, this layer may be of substantial thickness of, for example, up to many centimeters or of a minimum thickness of less than a millimeter. Similarly, a flexible belt may be of substantial thickness, for example, about 250 micrometers, or of minimum thickness less than 50 micrometers, provided there are no adverse effects on the final electrophotographic device.

In embodiments where the substrate layer is not conductive, the surface thereof may be rendered electrically conductive by an electrically conductive coating. The conductive coating may vary in thickness over substantially wide ranges depending upon the optical transparency, degree of flexibility desired, and economic factors. Accordingly, for a flexible photoresponsive imaging device, the thickness of the conductive coating may be between about 20 angstroms to about 750 angstroms, and more preferably from about 100 angstroms to about 200 angstroms for an optimum combination of electrical conductivity, flexibility and light transmission. The flexible conductive coating may be an electrically conductive metal layer formed, for example, on the substrate by any suitable coating technique, such as a vacuum depositing technique or electrodeposition. Typical metals include aluminum, zirconium, niobium, tantalum, vanadium and hafnium, titanium, nickel, stainless steel, chromium, tungsten, molybdenum, and the like.

An optional hole blocking layer may be applied to the substrate. Any suitable and conventional blocking layer capable of forming an

electronic barrier to holes between the adjacent photoconductive layer and the underlying conductive surface of a substrate may be utilized.

An optional adhesive layer may applied to the hole blocking layer. Any suitable adhesive layer well known in the art may be utilized. Typical
 5 adhesive layer materials include, for example, polyesters, polyurethanes, and the like. Satisfactory results may be achieved with adhesive layer thickness between about 0.05 micrometer (500 angstroms) and about 0.3 micrometer (3,000 angstroms). Conventional techniques for applying an adhesive layer coating mixture to the charge blocking layer include spraying, dip coating, roll coating,
 10 wire wound rod coating, gravure coating, Bird applicator coating, and the like. Drying of the deposited coating may be effected by any suitable conventional technique such as oven drying, infra red radiation drying, air drying and the like.

Any suitable polymeric film forming binder material may be employed as the matrix in the charge generating (photogenerating) binder
 15 layer. Typical polymeric film forming materials include those described, for example, in U.S. Patent 3,121,006. Thus, typical organic polymeric film forming binders include thermoplastic and thermosetting resins such as polycarbonates, polyesters, polyamides, polyurethanes, polystyrenes, polyarylethers, polyarylsulfones, polybutadienes, polysulfones,
 20 polyethersulfones, polyethylenes, polypropylenes, polyimides, polymethylpentenes, polyphenylene sulfides, polyvinyl acetate, polysiloxanes, polyacrylates, polyvinyl acetals, polyamides, polyimides, amino resins, phenylene oxide resins, terephthalic acid resins, phenoxy resins, epoxy resins, phenolic resins, polystyrene and acrylonitrile copolymers, polyvinylchloride,
 25 vinylchloride and vinyl acetate copolymers, acrylate copolymers, alkyd resins, cellulosic film formers, poly(amideimide), styrene-butadiene copolymers, vinylidenechloride-vinylchloride copolymers, vinylacetate-vinylidenechloride copolymers, styrene-alkyd resins, polyvinylcarbazole, and the like. These polymers may be block, random or alternating copolymers.

The photogenerating composition or pigment is present in the resinous binder composition in various amounts. Generally, however, from about 5 percent by volume to about 90 percent by volume of the photogenerating pigment is dispersed in about 10 percent by volume to about 95 percent by volume of the resinous binder, and preferably from about 20 percent by volume to about 30 percent by volume of the photogenerating pigment is dispersed in about 70 percent by volume to about 80 percent by volume of the resinous binder composition. In one embodiment about 8 percent by volume of the photogenerating pigment is dispersed in about 92 percent by volume of the resinous binder composition. The photogenerator layers can also be fabricated by vacuum sublimation in which case there is no binder.

Any suitable and conventional technique may be utilized to mix and thereafter apply the photogenerating layer coating mixture. Typical application techniques include spraying, dip coating, roll coating, wire wound rod coating, vacuum sublimation and the like. For some applications, the generator layer may be fabricated in a dot or line pattern. Removing of the solvent of a solvent coated layer may be effected by any suitable conventional technique such as oven drying, infrared radiation drying, air drying and the like.

The charge transport layer may comprise a charge transporting small molecule dissolved or molecularly dispersed in a film forming electrically inert polymer such as a polycarbonate. The term "dissolved" as employed herein is defined herein as forming a solution in which the small molecule is dissolved in the polymer to form a homogeneous phase. The expression "molecularly dispersed" is used herein is defined as a charge transporting small molecule dispersed in the polymer, the small molecules being dispersed in the polymer on a molecular scale. Any suitable charge transporting or electrically active small molecule may be employed in the charge transport layer of this invention. The expression charge transporting "small molecule" is defined herein as a monomer that allows the free charge photogenerated in the transport layer to be transported across the transport layer. Typical charge transporting small molecules

include, for example, pyrazolines such as 1-phenyl-3-(4'-diethylamino styryl)-5-(4''-diethylamino phenyl)pyrazoline, diamines such as N,N'-diphenyl-N,N'-bis(3-methylphenyl)-(1, 1'-biphenyl)-4, 4'-diamine, hydrazones such as N-phenyl-N-methyl-3-(9-ethyl)carbazyl hydrazone and 4-diethyl amino benzaldehyde-1, 2-diphenyl hydrazone, and oxadiazoles such as 2,5-bis (4-N,N'-diethylaminophenyl)-1,2,4-oxadiazole, stilbenes and the like. However, to avoid cycle-up, the charge transport layer should be substantially free of triphenyl methane. As indicated above, suitable electrically active small molecule charge transporting compounds are dissolved or molecularly dispersed in electrically inactive polymeric film forming materials. A small molecule charge transporting compound that permits injection of holes from the pigment into the charge generating layer with high efficiency and transports them across the charge transport layer with very short transit times is N,N'-diphenyl-N,N'-bis(3-methylphenyl)-(1, 1'-biphenyl)-4,4'-di-amine.

Any suitable electrically inert polymeric binder may used to disperse the electrically active molecule in the charge transport layer is a poly(4,4'-isopropylidene-diphenylene)carbonate (also referred to as bisphenol-A-polycarbonate), poly(4,4'-isoprpylidene-diphenylene)carbonate, poly(4,4'-diphenyl-1/1'-cyclohexane carbonate), and the like. Other typical inactive resin binders include polyester, polyarylate, polyacrylate, polyether, polysulfone, and the like. Molecular weights can vary, for example, from about 20,000 to about 150,000.

Instead of a small molecule charge transporting compound dissolved or molecularly dispersed in an electrically inert polymeric binder, the charge transport layer may comprise any suitable charge transporting polymer. A typical charge transporting polymers is one obtained from the condensation of N,N'-diphenyl -N,N'-bis (3-hydroxy phenyl)-[1,1'-biphenyl]-4,4'-diamine and diethylene glycol bischloroformate such as disclosed in US-A 4,806,443 and US-A 5,028,687. Another typical charge transporting polymer is poly(N,N'-bis-(3-oxyphenyl)-N,N'-diphenyl [1,1'-biphenyl]-4,4'-diaminesebacoyl) polyethercarbonate obtained from the

condensation of N,N'-diphenyl -N,N'-bis (3-hydroxy phenyl)-[1,1'-biphenyl]-4,4'-diamine and sebacoyl chloride.

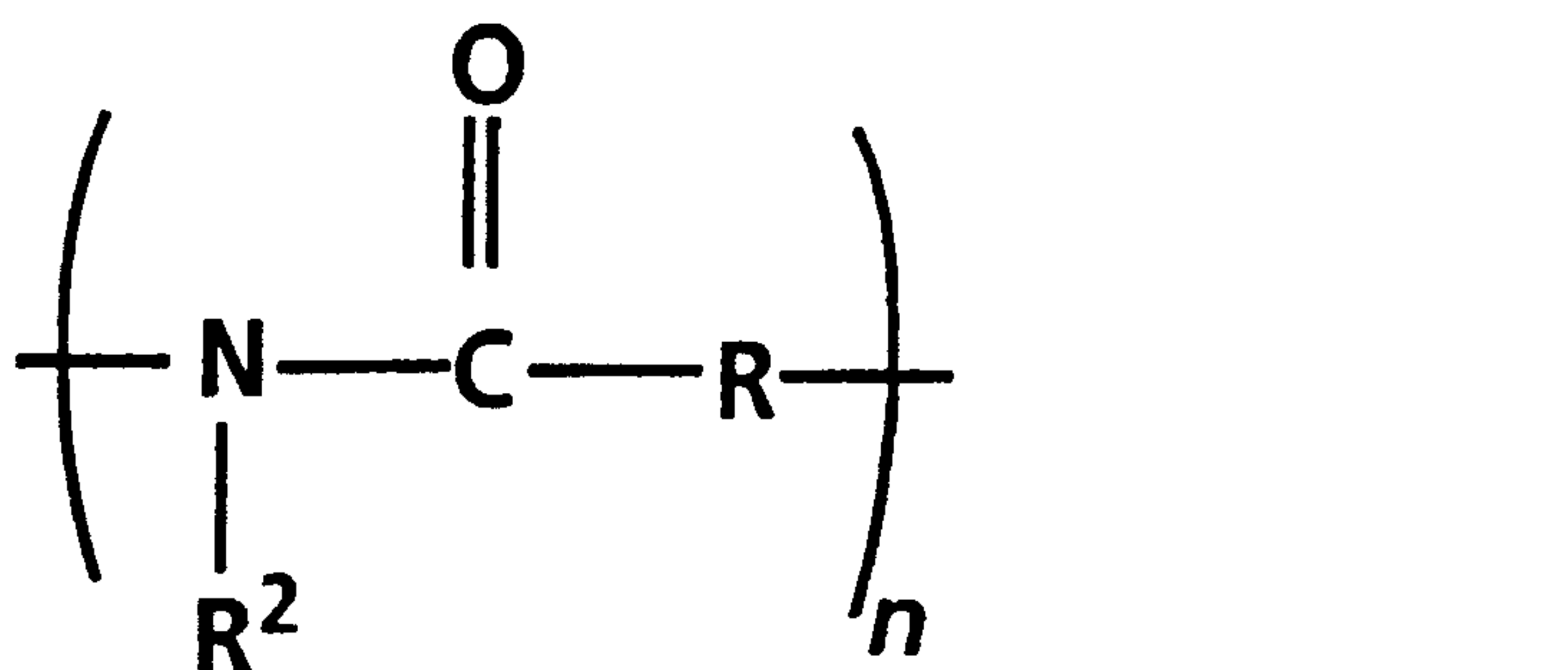
Any suitable and conventional technique may be utilized to mix and thereafter apply the charge transport layer coating mixture to the charge generating layer. Typical application techniques include spraying, dip coating, roll coating, wire wound rod coating, and the like. Drying of the deposited coating may be effected by any suitable conventional technique such as oven drying, infra red radiation drying, air drying and the like.

Generally, the thickness of the charge transport layer is between about 10 and about 50 micrometers, but thicknesses outside this range can also be used. The hole transport layer should be an insulator to the extent that the electrostatic charge placed on the hole transport layer is not conducted in the absence of illumination at a rate sufficient to prevent formation and retention of an electrostatic latent image thereon. In general, the ratio of the thickness of the hole transport layer to the charge generator layers is preferably maintained from about 2:1 to 200:1 and in some instances as great as 400:1. In other words, the charge transport layer, is substantially non-absorbing to visible light or radiation in the region of intended use but is electrically "active" in that it allows the injection of photogenerated holes from the photoconductive layer, i.e., charge generation layer, and allows these holes to be transported through itself to selectively discharge a surface charge on the surface of the active layer.

The overcoat layer of this invention comprises a dihydroxy arylamine dissolved or molecularly dispersed in a crosslinked polyamide matrix. The overcoat layer is formed from a crosslinkable coating composition comprising an alcohol soluble polyamide containing methoxy methyl groups attached to amide nitrogen atoms, a crosslinking catalyst and a dihydroxy arylamine.

Any suitable hole insulating film forming alcohol soluble polyamide polymer having methoxy methyl groups attached to the nitrogen atoms of amide groups in the polymer backbone prior to

crosslinking may be employed in the overcoating of this invention. A preferred alcohol soluble polyamide polymer having methoxy methyl groups attached to the nitrogen atoms of amide groups in the polymer backbone prior to crosslinking is selected from the group consisting of materials represented by the following formulae I and II:



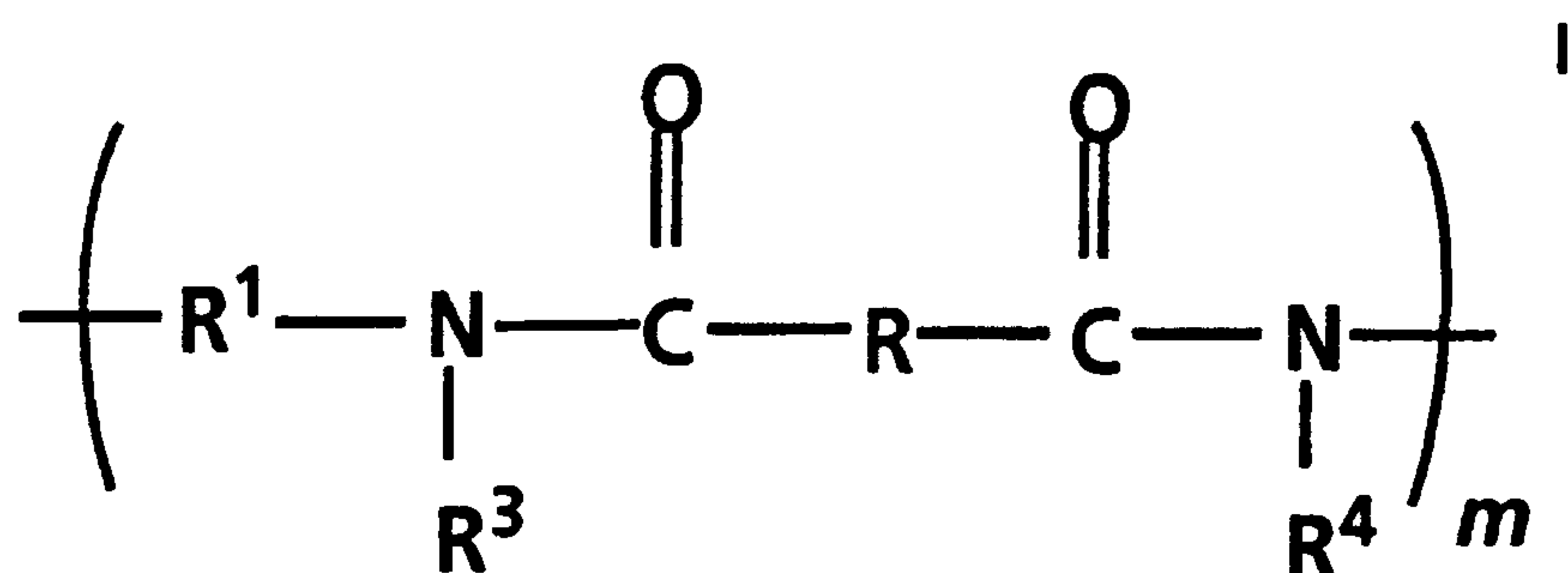
wherein:

n is a positive integer,

R is independently selected from the group consisting of alkylene, arylene or alkarylene units,

between 1 and 99 percent of the R^2 sites are $-H$, and

the remainder of the R^2 sites are $-\text{CH}_2\text{-O-CH}_3$ and



wherein:

m is a positive integer,

R_1 and R are independently selected from the group consisting of
alkylene, arylene or alkarylene units,

5

between 1 and 99 percent of the R^3 and R^4 sites are -H, and

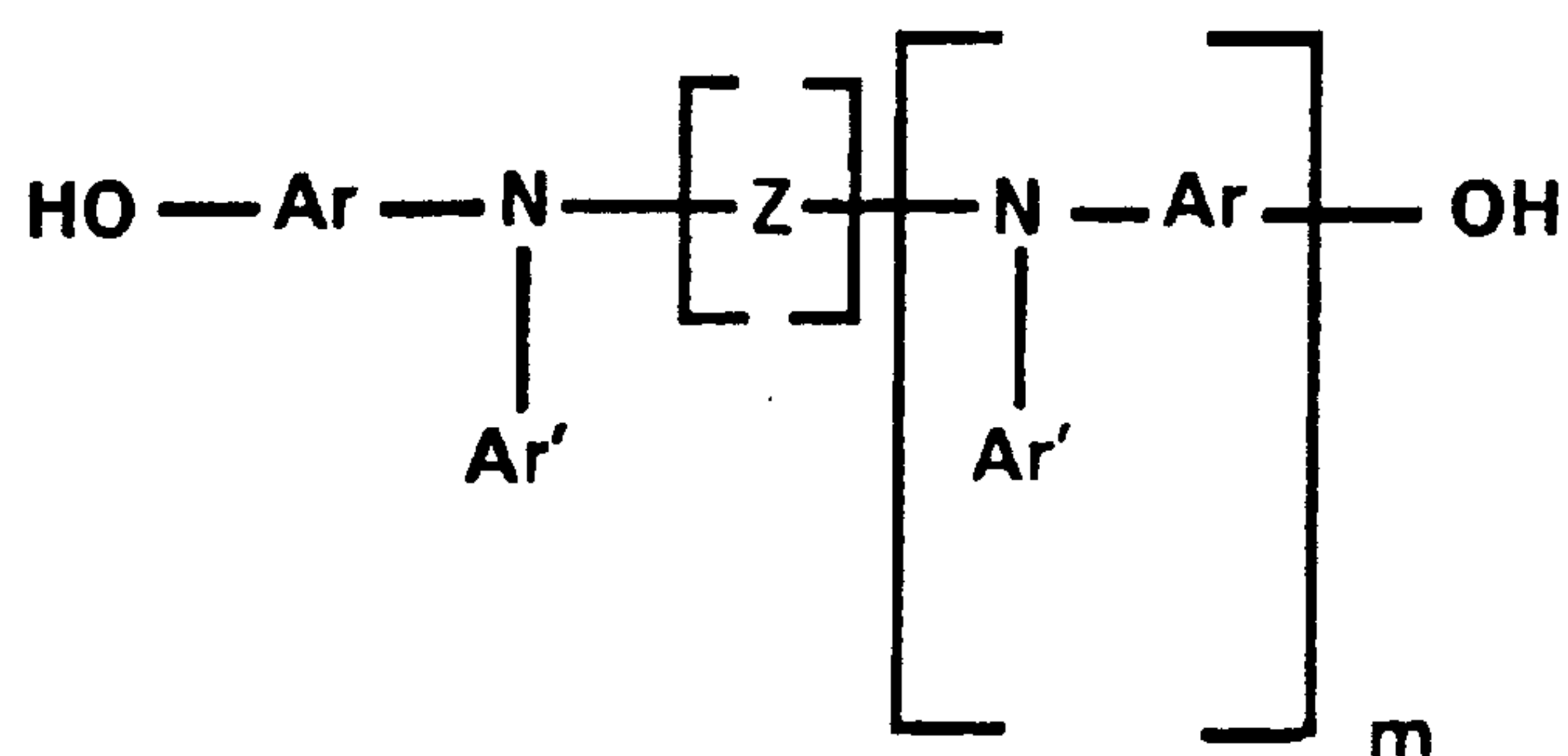
the remainder of the R^3 and R^4 sites are $-CH_2-O-CH_3$.

Between about 1 percent and about 50 mole percent of the total number of repeat
10 units of the nylon polymer should contain methoxy methyl groups attached to the
nitrogen atoms of amide groups. These polyamides should form solid films if
dried prior to crosslinking. The polyamide should also be soluble, prior to
crosslinking, in the alcohol solvents employed. Typical alcohols in which the
polyamide is soluble include, for example, butanol, ethanol, methanol, and the
15 like. Typical alcohol soluble polyamide polymers having methoxy methyl groups
attached to the nitrogen atoms of amide groups in the polymer backbone prior to
crosslinking include, for example, hole insulating alcohol soluble polyamide film
forming polymers include, for example, TMLuckamide 5003 from Dai Nippon Ink,
TMNylon 8 with methoxymethoxy pendant groups, TMCM4000 from Toray
20 Industries, Ltd. and TMCM8000 from Toray Industries, Ltd. and other N-
methoxymethylated polyamides, such as those prepared according to the method
described in Sorenson and Campbell "Preparative Methods of Polymer
Chemistry" second edition, pg 76, John Wiley & Sons Inc. 1968, and the like and
mixtures thereof. These polyamides can be alcohol soluble, for example, with
25 polar functional groups, such as methoxy, ethoxy and hydroxy groups, pendant
from the polymer backbone. It should be noted that polyamides, such as
TMElvamides from DuPont de Nemours & Co., do not contain methoxy methyl
groups attached to the nitrogen atoms of amide groups in the polymer backbone.
The overcoating layer of this invention

preferably comprises between about 50 percent by weight and about 98 percent by weight of the crosslinked film forming crosslinkable alcohol soluble polyamide polymer having methoxy methyl groups attached to the nitrogen atoms of amide groups in the polymer backbone, based on the total weight of the overcoating layer after crosslinking and drying. These film forming polyamides are also soluble in a solvent to facilitate application by conventional coating techniques. Typical solvents include, for example, butanol, methanol, butyl acetate, ethanol, cyclohexanone, tetrahydrofuran, methyl ethyl ketone, and the like and mixtures thereof. Crosslinking is accomplished by heating in the presence of a catalyst. Any suitable catalyst may be employed. Typical catalysts include, for example, oxalic acid, p-toluenesulfonic acid, methanesulfonic acid, and the like and mixtures thereof. Catalysts that transform into a gaseous product during the crosslinking reaction are preferred because they escape the coating mixture and leave no residue that might adversely affect the electrical properties of the final overcoating. A typical gas forming catalyst is, for example, oxalic acid. The temperature used for crosslinking varies with the specific catalyst and heating time utilized and the degree of crosslinking desired. Generally, the degree of crosslinking selected depends upon the desired flexibility of the final photoreceptor. For example, complete crosslinking may be used for rigid drum or plate photoreceptors. However, partial crosslinking is preferred for flexible photoreceptors having, for example, web or belt configurations. The degree of crosslinking can be controlled by the relative amount of catalyst employed. The amount of catalyst to achieve a desired degree of crosslinking will vary depending upon the specific polyamide, catalyst, temperature and time used for the reaction. A typical crosslinking temperature used for Luckamide with oxalic acid as a catalyst is about 125°C for 30 minutes. After crosslinking, the overcoating should be substantially insoluble in the solvent in which it was soluble prior to crosslinking. Thus, no overcoating material will be removed when rubbed with a cloth soaked in the solvent. Crosslinking results in the development of a three dimensional network which restrains the dihydroxy arylamine molecule as a fish is caught in a gill net.

Prolonged attempts to extract the highly fluorescent dihydroxy arylamine hole transport molecule from the crosslinked overcoat, using long exposure to branched hydrocarbon solvents, revealed that the transport molecule is completely immobilized. Thus, when UV light is used to examine the extractant or the applicator pad no fluorescence is observed. The molecule is also locked into the overcoat by hydrogen bonding to amide sites on the polyamide.

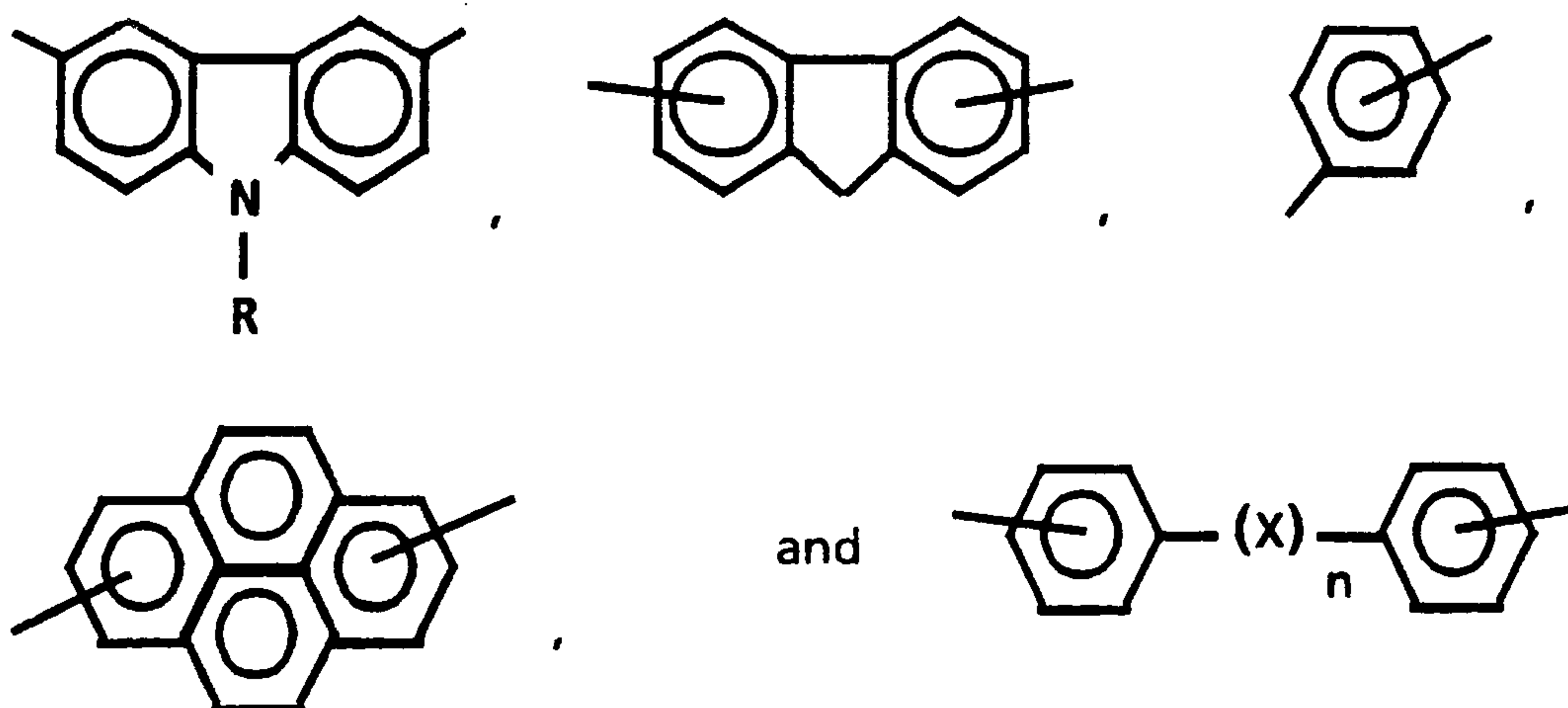
The overcoating of this invention also includes a dihydroxy arylamine. Preferably, the dihydroxy arylamine is represented by the following formula:



wherein:

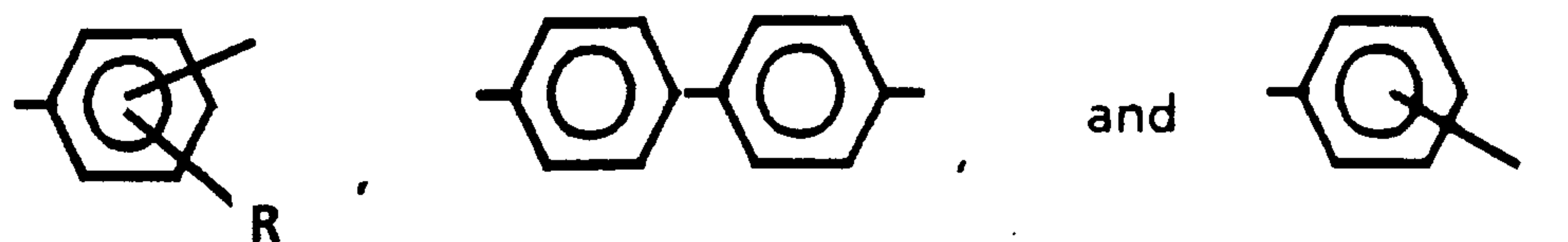
m is 0 or 1,

Z is selected from the group consisting of:



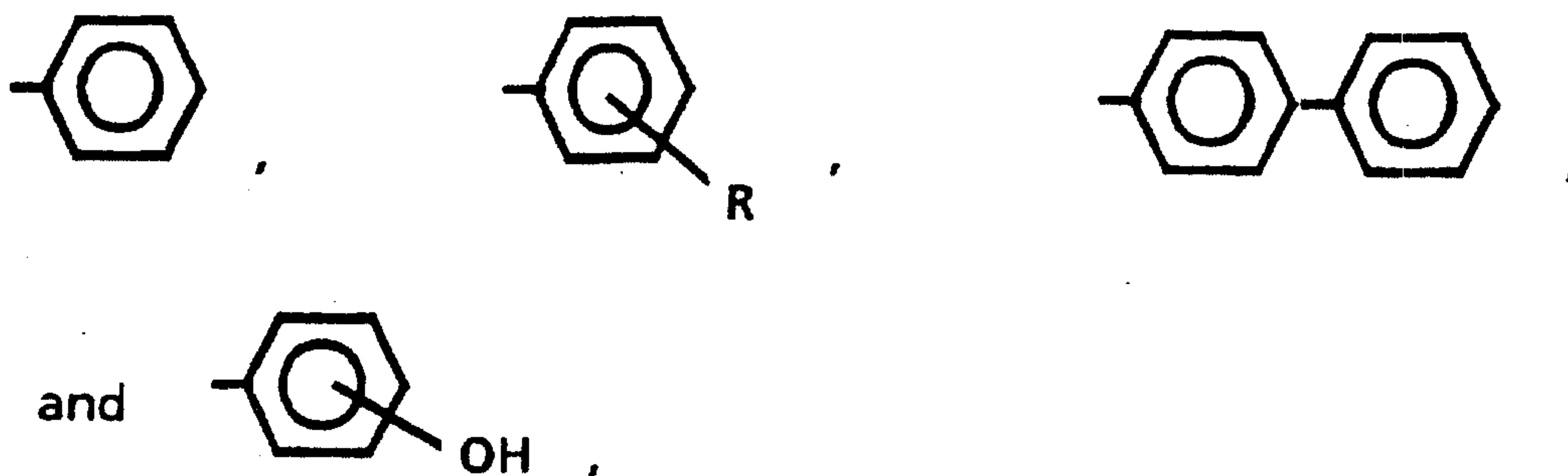
n is 0 or 1,

Ar is selected from the group consisting of:

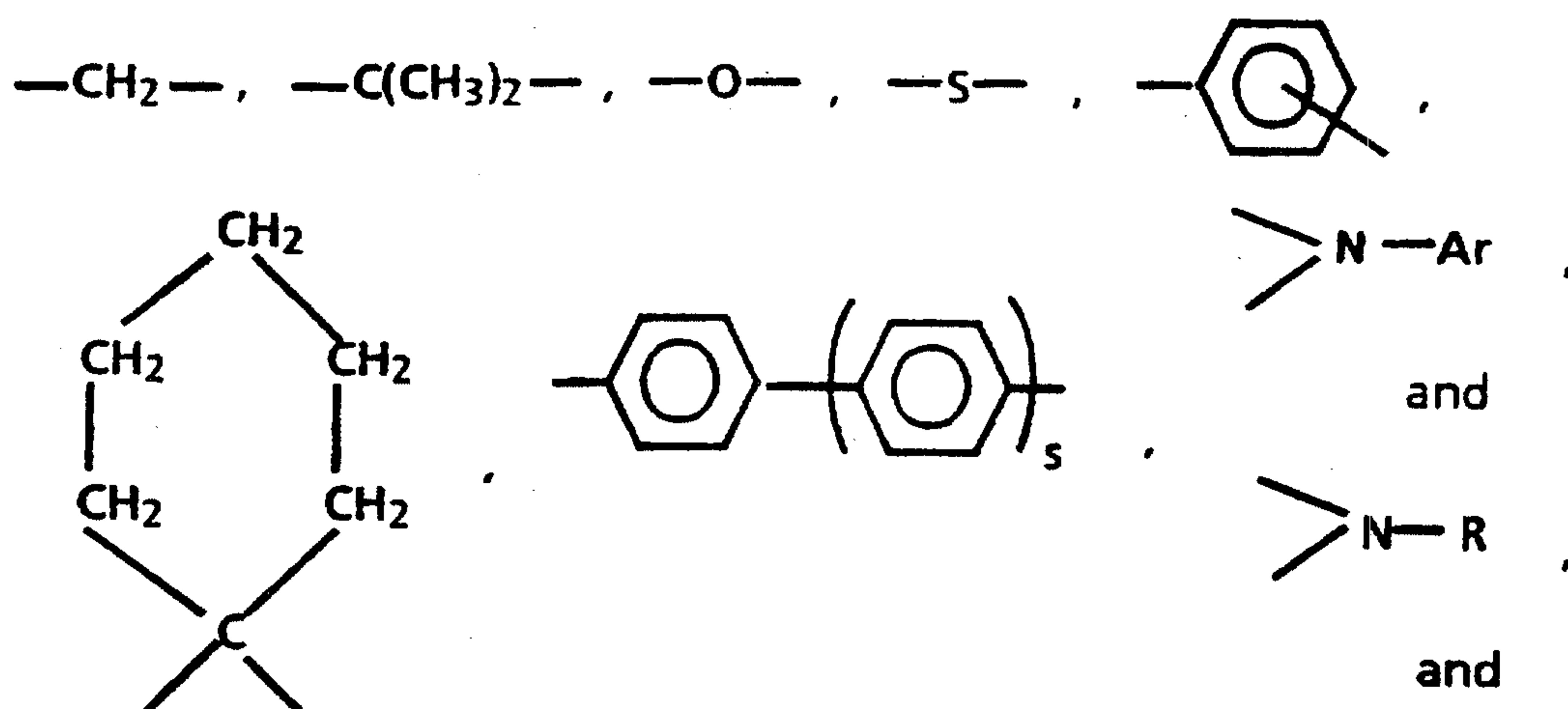


R is selected from the group consisting of $-\text{CH}_3$, $-\text{C}_2\text{H}_5$, $-\text{C}_3\text{H}_7$, and $-\text{C}_4\text{H}_9$,

Ar' is selected from the group consisting of:



X is selected from the group consisting of:



s is 0, 1 or 2.

This hydroxyarylamine compound is described in detail in US-A 4,871,635 and may be free of any direct conjugation between off groups and the nearest nitrogen atom through one or more aromatic rings.

Generally, the hydroxy arylamine compounds are prepared, for example, by hydrolizing an dialkoxy arylamine. A typical process for

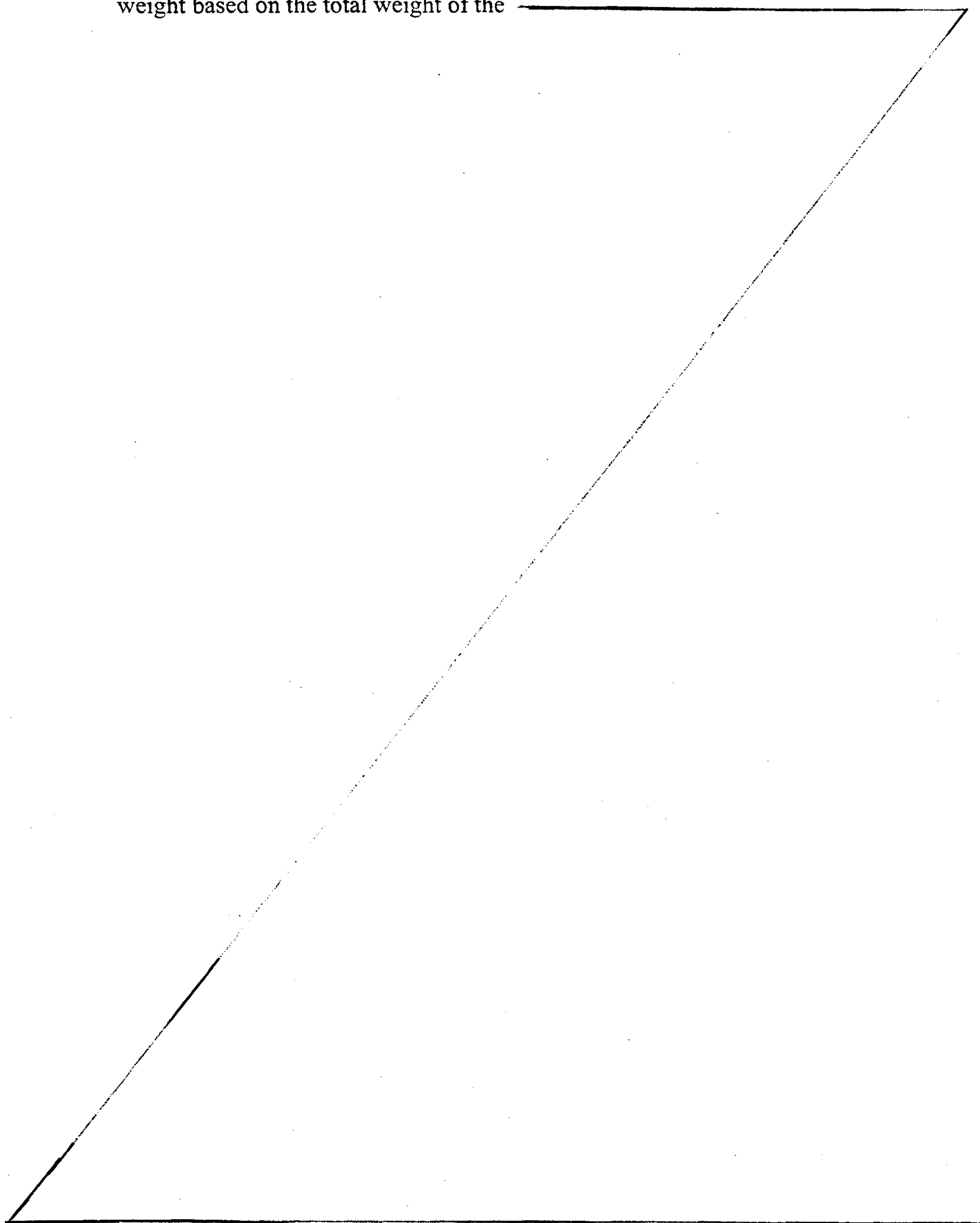
preparing alkoxy arylamines is disclosed in Example I of U.S. Patent 4,588,666 to Stolka et al.

Typical hydroxy arylamine compounds of this invention include, for example:

- 5 N,N'-diphenyl-N,N'-bis(3-hydroxyphenyl)-[1,1'-biphenyl]-4,4'-diamine;
N,N,N',N',-tetra(3-hydroxyphenyl)-[1,1'-biphenyl]-4,4'-diamine;
N,N-di(3-hydroxyphenyl)-m-toluidine;
1,1-bis-[4-(di-N,N-m-hydroxypyphenyl)-aminophenyl]-cyclohexane;
- 10 1,1-bis[4-(N-m-hydroxyphenyl)-4-(N-phenyl)-aminophenyl]-cyclohexane;
Bis-(N-(3-hydroxyphenyl)-N-phenyl-4-aminophenyl)-methane;
Bis[(N-(3-hydroxyphenyl)-N-phenyl)-4-aminophenyl]-isopropylidene;
N,N'-diphenyl-N,N'-bis(3-hydroxyphenyl)-[1,1':4',1"-terphenyl]-4,4"-
diamine;
- 15 9-ethyl-3,6-bis[N-phenyl-N-3(3-hydroxyphenyl)-amino]-carbazole;
2,7-bis[N,N-di(3-hydroxyphenyl)-amino]-fluorene;
1,6-bis[N,N-di(3-hydroxyphenyl)-amino]-pyrene;
1,4-bis[N-phenyl-N-(3-hydroxyphenyl)]-phenylenediamine
N,N'-diphenyl-N,N'-bis(4-hydroxy phenyl)[1,1'-biphenyl]-4,4'diamine
- 20 N,N,N',N',-tetra(4-hydroxyphenyl)-[1,1'-biphenyl]-4,4'-diamine;
N,N-di(4-hydroxyphenyl)-m-toluidine;
1,1-bis-[4-(di-N,N-p-hydroxypyphenyl)-aminophenyl]-cyclohexane;
1,1-bis[4-(N-o-hydroxyphenyl)-4-(N-phenyl)-aminophenyl]-cyclohexane;
Bis-(N-(4-hydroxyphenyl)-N-phenyl-4-aminophenyl)-methane;
- 25 Bis[(N-(4-hydroxyphenyl)-N-phenyl)-4-aminophenyl]-isopropylidene;
Bis-N,N-[(4'-hydroxy-4-(1,1'-biphenyl)]-aniline
Bis-N,N-[(2'-hydroxy-4-(1,1'-biphenyl)]-aniline

The concentration of the hydroxy arylamine in the overcoat can be between about 2 percent and about 50 percent by weight based on the total weight
30 of the dried overcoat. Preferably, the concentration of the hydroxy arylamine in

the overcoat layer is between about 10 percent by weight and about 50 percent by weight based on the total weight of the



dried overcoat. When less than about 10 percent by weight of hydroxy arylamine is present in the overcoat, a residual voltage may develop with cycling resulting in background problems. If the amount of hydroxy arylamine in the overcoat exceeds about 50 percent by weight based on the total weight of the overcoating layer, crystallization may occur resulting in residual cycle-up. In addition, mechanical properties, abrasive wear properties are negatively impacted.

The thickness of the continuous overcoat layer selected depends upon the abrasiveness of the charging (e.g., bias charging roll), cleaning (e.g., blade or web), development (e.g., brush), transfer (e.g., bias transfer roll), etc., system employed and can range up to about 10 micrometers. A thickness of between about 1 micrometer and about 5 micrometers in thickness is preferred. Any suitable and conventional technique may be utilized to mix and thereafter apply the overcoat layer coating mixture to the charge generating layer. Typical application techniques include spraying, dip coating, roll coating, wire wound rod coating, and the like. Drying of the deposited coating may be effected by any suitable conventional technique such as oven drying, infrared radiation drying, air drying and the like. The dried overcoating of this invention should transport holes during imaging and should not have too high a free carrier concentration. Free carrier concentration in the overcoat increases the dark decay. Preferably the dark decay of the overcoated layer should be the same as that of the unovercoated device.

Other suitable layers may also be used such as a conventional electrically conductive ground strip along one edge of the belt or drum in contact with the conductive surface of the substrate to facilitate connection of the electrically conductive layer of the photoreceptor to ground or to an electrical bias. Ground strips are well known and usually comprise conductive particles dispersed in a film forming binder.

In some cases an anti-curl back coating may be applied to the side opposite the photoreceptor to provide flatness and/or abrasion resistance for belt or web type photoreceptors. These anti-curl back coating layers are well known in the art and may comprise thermoplastic

organic polymers or inorganic polymers that are electrically insulating or slightly semiconducting.

The photoreceptor of this invention may be used in any conventional electrophotographic imaging system. As described above, electrophotographic imaging usually involves depositing a uniform electrostatic charge on the photoreceptor, exposing the photoreceptor to a light image pattern to form an electrostatic latent image on the photoreceptor, developing the electrostatic latent image with electrostatically attractable marking particles to form a visible toner image, transferring the toner image to a receiving member and repeating the depositing, exposing, developing and transferring steps at least once.

A number of examples are set forth hereinbelow and are illustrative of different compositions and conditions that can be utilized in practicing the invention. All proportions are by weight unless otherwise indicated. It will be apparent, however, that the invention can be practiced with many types of compositions and can have many different uses in accordance with the disclosure above and as pointed out hereinafter.

EXAMPLE I

Three photoreceptors were prepared by forming coatings using conventional techniques on a substrate comprising a vacuum deposited titanium layer on a polyethylene terephthalate film. The first coating was a siloxane barrier layer formed from hydrolyzed gamma aminopropyltriethoxysilane having a thickness of 0.005 micrometer (50 Angstroms). The barrier layer coating composition was prepared by mixing 3-aminopropyltriethoxysilane (available from PCR Research Chemicals of Florida) with ethanol in a 1:50 volume ratio. The coating composition was applied by a multiple clearance film applicator to form a coating having a wet thickness of 0.5 mil. The coating was then allowed to dry for 5 minutes at room temperature, followed by curing for 10 minutes at 110 degree centigrade in a forced air oven. The second coating was an adhesive layer of polyester resin (49,000, available from E. I. duPont de Nemours & Co.) having a thickness of 0.005 micron (50 Angstroms). The second coating

composition was prepared by dissolving 0.5 gram of 49,000 polyester resin in 70 grams of tetrahydrofuran and 29.5 grams of cyclohexanone. The second coating composition was applied using a 0.5 mil bar and the resulting coating was cured in a forced air oven for 10 minutes. This adhesive interface layer was thereafter coated with a photogenerating layer containing 40 percent by volume hydroxygallium phthalocyanine and 60 percent by volume of a block copolymer of styrene (82 percent) / 4-vinyl Pyridine (18 percent) having a Mw of 11,000. This photogenerating coating composition was prepared by dissolving 1.5 grams of the block copolymer of styrene / 4-vinyl pyridine in 42 ml of toluene. To this solution was added 1.33 grams of hydroxygallium phthalocyanine and 300 grams of 1/8 inch diameter stainless steel shot. This mixture was then placed on a ball mill for 20 hours. The resulting slurry was thereafter applied to the adhesive interface with a Bird applicator to form a layer having a wet thickness of 0.25 mil. This layer was dried at 135°C for 5 minutes in a forced air oven to form a photogenerating layer having a dry thickness 0.4 micrometer. The next applied layer was a transport layer which was formed by using a Bird coating applicator to apply a solution containing one gram of N,N'-diphenyl-N,N'-bis(3-methyl-phenyl)-(1,1' biphenyl)-4,4'-diamine and one gram of polycarbonate resin [poly(4,4'-isopropylidene-diphenylene carbonate (available as Makrolon^R from Farbenfabriken Bayer A.G.) dissolved in 11.5 grams of methylene chloride solvent. The N,N'-diphenyl-N,N'-bis(3-methyl-phenyl)-(1,1' biphenyl)-4,4'-diamine is an electrically active aromatic diamine charge transport small molecule whereas the polycarbonate resin is an electrically inactive film forming binder. The coated device was dried at 80°C for half an hour in a forced air oven to form a dry 25 micrometer thick charge transport layer.

EXAMPLE II

A second device was prepared by overcoating a photoreceptor of Example I with an overcoat layer material. This overcoat material is described in

US-A 5,368,967. Prior to application of the overcoat layer, the photoreceptor of Example I was primed by applying 0.1 percent by weight of TMElvacite 2008 in 90:10 weight ratio of isopropyl alcohol and water using a # 3 Meyer rod. This prime coating was air dried in a hood. The overcoat composition was prepared by mixing 10 grams of a 10 percent by weight solution of a polyamide containing methoxymethyl groups (TMLuckamide 5003, available from Dai Nippon Ink) in a 90:10 weight ratio solvent of methanol and n-propanol and 10 grams of N,N'-diphenyl-N,N'-bis (3-hydroxyphenol)-[1,1'-biphenyl]-4,4" - diamine (a dihydroxy arylamine) in a roll mill for 2 hours. This coating solution was applied to the primed photoreceptor using a # 20 Meyer rod. This overcoat layer was air dried in a hood for 30 minutes. The air dried film was then dried in a forced air oven at 125°C for 30 minutes. The overcoat layer thickness was approximately 3 micrometers.

15

EXAMPLE III

A third device was prepared by overcoating a photoreceptor of Example I with an overcoat layer material of this invention. Prior to application of the overcoat layer, the photoreceptor of Example I was primed by applying 0.1 percent by weight of TMElvacite 2008 in 90:10 weight ratio of isopropyl alcohol and water using a # 3 Meyer rod. This prime coating was air dried in a hood. The overcoat layer was prepared by mixing 10 grams of a 10 percent by weight solution of polyamide containing methoxymethyl groups (TMLuckamide 5003, available from Dai Nippon Ink) in a 90:10 weight ratio solvent of methanol and n-propanol and 10 grams of N,N'-diphenyl-N,N'-bis (3-hydroxyphenol)-[1,1'-biphenyl] -4,4"-diamine (a dihydroxy arylamine) in a roll mill for 2 hours. Immediately prior to application of the overcoat layer mixture, 0.1 gram of oxalic acid was added and the resulting mixture was roll milled briefly to assure dissolution. This coating solution was applied to the primed photoreceptor using a # 20 Meyer rod. This overcoat layer was air dried in a hood for 30 minutes. The air dried film was then dried in a forced air oven at 125°C for 30 minutes. The overcoat layer thickness was approximately 3 micrometers. The oxalic

acid caused crosslinking of the methoxymethyl groups of the polyamide to yield a tough, abrasion resistant, hydrocarbon resistant top surface.

EXAMPLE IV

Devices of Example I (device without the overcoat), Example II (device with the overcoat of US-A 5,368,967) and Example III (device with the cross linked overcoat of this invention) were first tested for xerographic sensitivity and cyclic stability. Each photoreceptor device was mounted on a cylindrical aluminum drum substrate which is rotated on a shaft of a scanner. Each photoreceptor was charged by a corotron mounted along the periphery of the drum. The surface potential was measured as a function of time by capacitively coupled voltage probes placed at different locations around the shaft. The probes were calibrated by applying known potentials to the drum substrate. The photoreceptors on the drums were exposed by a light source located at a position near the drum downstream from the corotron. As the drum was rotated, the initial (pre exposure) charging potential was measured by voltage probe 1. Further rotation lead to the exposure station, where the photoreceptor was exposed to monochromatic radiation of known intensity. The photoreceptor was erased by light source located at a position upstream of charging. The measurements made included charging of the photoreceptor in a constant current or voltage mode. The photoreceptor was charged to a negative polarity corona. As the drum was rotated, the initial charging potential was measured by voltage probe 1. Further rotation lead to the exposure station, where the photoreceptor was exposed to monochromatic radiation of known intensity. The surface potential after exposure was measured by voltage probes 2 and 3. The photoreceptor was finally exposed to an erase lamp of appropriate intensity and any residual potential was measured by voltage probe 4. The process was repeated with the magnitude of the exposure automatically changed during the next cycle. The photodischarge characteristics was obtained by plotting the potentials at voltage probes 2 and 3 as a function of light exposure. The charge acceptance and dark decay were also measured in the scanner. A slight increase in sensitivity was

observed in the overcoated photoreceptors. This increase corresponded to the three micrometer increase in thickness due to the presence of the overcoatings. The residual potential was equivalent (15 volts) for all three photoreceptors and no cycle-up was observed when cycled for 10,000 cycles in a continuous mode. The overcoat clearly did not introduce any deficiencies.

EXAMPLE V

Three electrophotographic imaging members were prepared by applying by dip coating a charge blocking layer onto the honed surface of an aluminum drum having a diameter of 4 cm and a length of 31 cm. The blocking layer coating mixture contained a solution of 8 weight percent polyamide (nylon 6) dissolved in a 92 weight percent butanol, methanol and water solvent mixture. The butanol, methanol and water mixture component percentages were 55, 36 and 9 percent by weight, respectively. The blocking layer coating was applied at a coating bath withdrawal rate of 300 mm/minute. After drying in a forced air oven, the blocking layer had a thickness of 1.5 micrometer. The dried blocking layer was coated with a charge generating layer containing 2.5 weight percent hydroxy gallium phthalocyanine pigment particles, 2.5 weight percent polyvinylbutyral film forming polymer and 95 weight percent cyclohexanone solvent. The coating was applied at a coating bath withdrawal rate of 300 millimeters/minute. After drying in a forced air oven, the charge generating layer had a thickness of 0.2 micrometer. The dried generating layer was coated with a charge transport layer containing 8 weight percent N,N'-diphenyl-N,N'-bis(3-methylphenyl)-1,1'-biphenyl-4,4'-diamine, 12 weight percent polycarbonate resin (TMMakrolon 5705, available from Farbensabricken Bayer A.G.) and 80 weight percent monochlorobenzene solvent. The charge transport layer coating was applied at a coating bath withdrawal rate of 100 millimeters/minute. After drying in a forced air oven, the transport layer had a thickness of 20 micrometers. The first imaging member was tested without an overcoat. An overcoating layer was applied to devices on the second and third imaging members by a

lathe-type coating device, a product of Anakenesis Corp., which applies the solution from an open cell polyurethane pad which is replenished from a reservoir and is capable of coating to a thickness having less than 5 percent variation across the drum and no measurable variation around the circumference. The overcoating coating mixture for application to the second imaging member contained a solution of 5.4 weight percent N,N'-diphenyl-N,N'-bis (3-hydroxy phenyl)-[1,1'-biphenyl]-4,4'-diamine and 54 weight percent polyamide solution [prepared by the dissolution of 10 weight percent Luckamide 5003 in 90 weight percent methanol/propanol (90/10)] dissolved in 40.6 weight percent isopropanol and a trace of water solvent mixture. Luckamide 5003 is a polyamide having methoxymethoxy groups pendant from the polymer backbone and is available from Dai Nippon Ink. After application and drying in a forced air oven at a temperature of 125°C for 30 minutes, the overcoat layer had a thickness of 4 to 6 micrometers. The device on the third photoreceptor was overcoated with an overcoat similar to the overcoat for the second photoreceptor except that the coating composition was adjusted to contain 0.5 weight percent oxalic acid dissolved in the coating solution mixture. After application and drying in a forced air oven at a temperature of 125°C, the overcoat layer had a thickness of 4 to 6 micrometers. The three photoreceptors of this Example, i.e., first photoreceptor without the overcoat, second photoreceptor containing an overcoat of the prior art (US-A 5,368,967) and third photoreceptor containing the crosslinked overcoat of this invention were tested for wear and print test capabilities in following Examples.

EXAMPLE VI

The electrical properties of the photoreceptors prepared according to Example V were evaluated with a xerographic testing scanner. The drums were rotated in a scanner at a constant surface speed of 5.66 cm per second. A direct current wire scorotron, narrow wavelength band exposure light, erase light, and four electrometer probes were mounted around the periphery of the mounted photoreceptor samples. Each sample

charging time was 177 milliseconds. The exposure light had an output wavelength of 680 nm and the erase light had an output wavelength of 550 nm. The photodischarge characteristics was obtained by plotting the potentials at voltage probes 2 and 3 as a function of light exposure. The charge acceptance and dark decay were also measured in the scanner. A slight increase in sensitivity was observed in the overcoated devices. This increase corresponded to the 4-6 micron increase in thickness due to the overcoating. The residual potential was equivalent (15 volts) for all four devices and no cycle-up was observed when cycled for 1000 cycles in a continuous mode. The overcoat clearly did not introduce any electrical deficiencies.

EXAMPLE VII

The three photoreceptors of Example V were print tested in a Xerox 4510 machine for 500 consecutive prints. There was no loss of image sharpness, no problem with background or any other defect resulting from the overcoats.

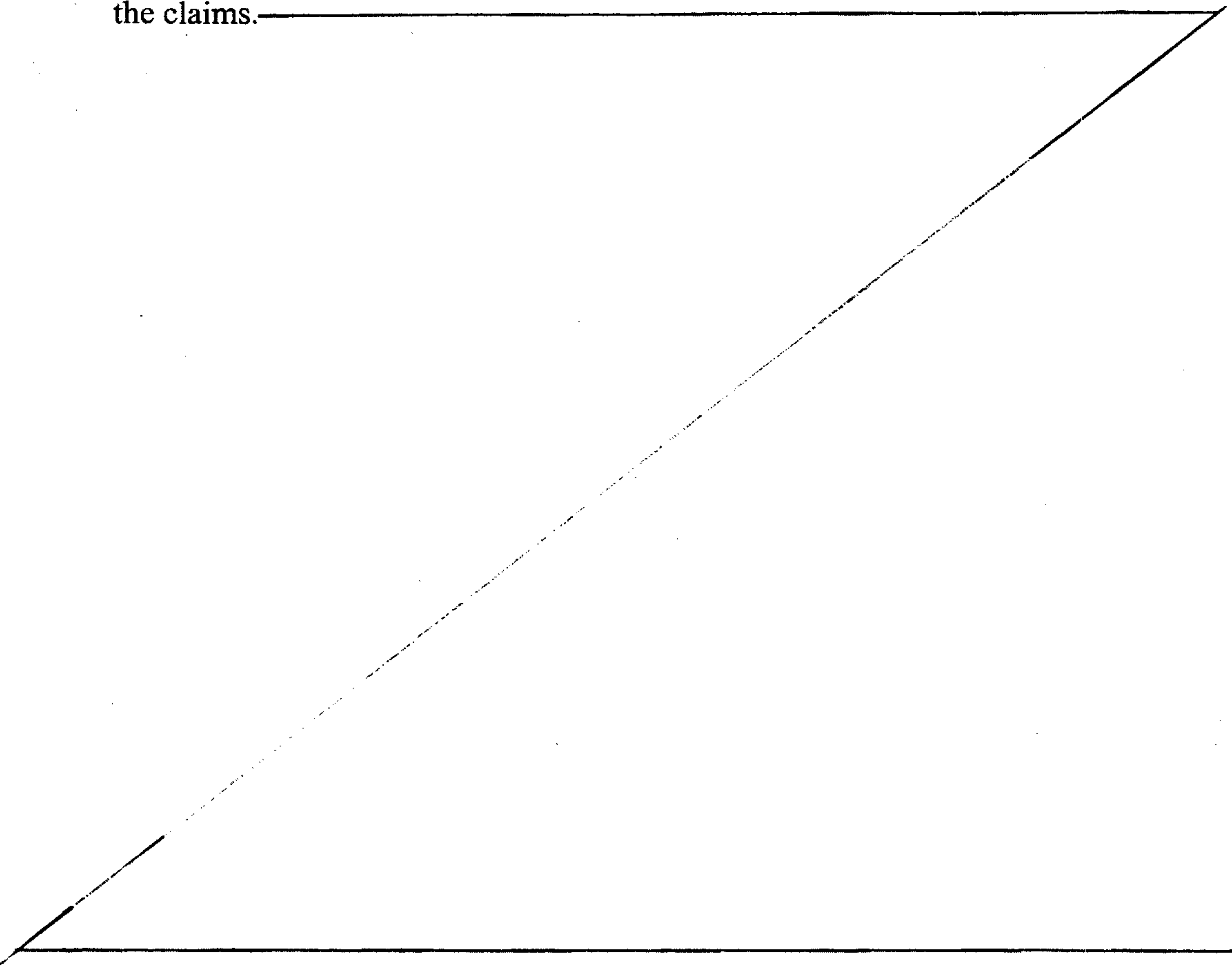
EXAMPLE VIII

The three drum photoreceptors of Example V were tested in a wear fixture that contained a bias charging roll for charging. Wear is calculated in terms of nanometers / kilocycles of rotation (nm/Kc). Reproducibility of calibration standards is about ± 2 nm/Kc. The wear of the drum without the overcoat was > 50 nm/kcycles. Wear of the second photoreceptor was > 50 nm/kcycles. Wear for the third photoreceptor having the crosslinked overcoating of this invention was about 9 nm/kcycle. Thus, the improvement in resistance to wear for the photoreceptor of this invention, when subjected to bias charging roll conditions, was very significant.

EXAMPLE IX

The three drum photoreceptors of Example V were contacted gauze pads soaked with TMIsopar M, a C₁₅ branched hydrocarbon useful in liquid ink development xerography. When the pads which contacted the unovercoated first photoreceptor and the uncrosslinked overcoating of the second photoreceptor were exposed to an ultraviolet lamp, telltale fluorescence (characteristic of the transport molecule) were observed on each pad whereas the pad which contacted the crosslinked overcoating of the third photoreceptor showed no evidence of fluorescence, indicating that the crosslinked sample was resistant to TMIsopar extraction.

Although the invention has been described with reference to specific preferred embodiments, it is not intended to be limited thereto, rather those having ordinary skill in the art will recognize that variations and modifications may be made therein which are within the spirit of the invention and within the scope of the claims.



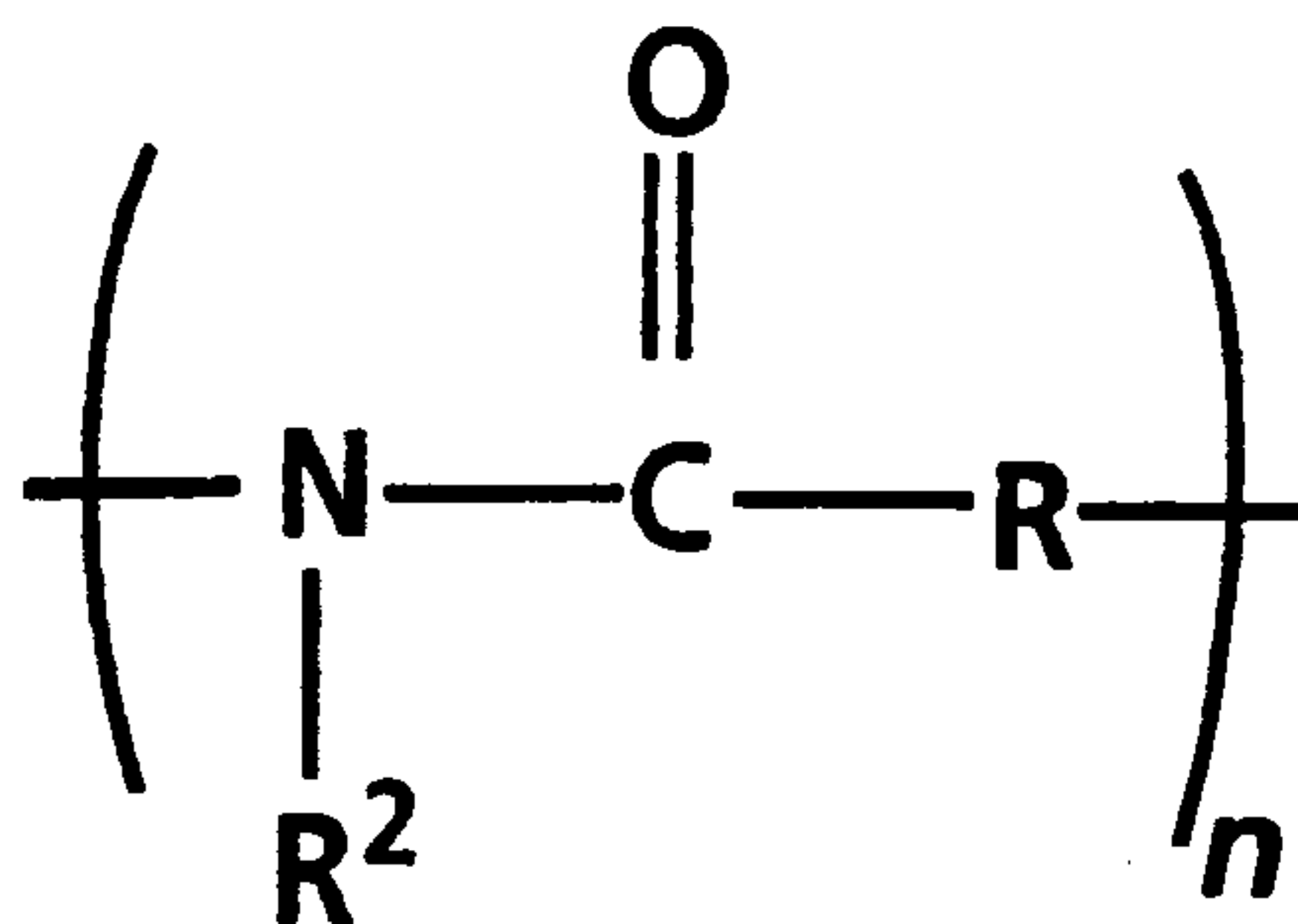
WHAT IS CLAIMED IS:

1. An electrophotographic imaging member comprising a supporting substrate coated with at least a charge generating layer, a charge transport layer and an overcoating layer, said overcoating layer comprising a dihydroxy arylamine dissolved or molecularly dispersed in a crosslinked polyamide matrix.

2. An electrophotographic imaging member according to claim 1 wherein said polyamide is crosslinked in the presence of an oxalic acid catalyst.

3. An electrophotographic imaging member according to claim 1 wherein amide nitrogen atoms on said polyamide contain methoxy methyl groups prior to crosslinking.

4. An electrophotographic imaging member according to claim 1 wherein said polyamide is selected from the group consisting of materials represented by the following formulae I and II:



I

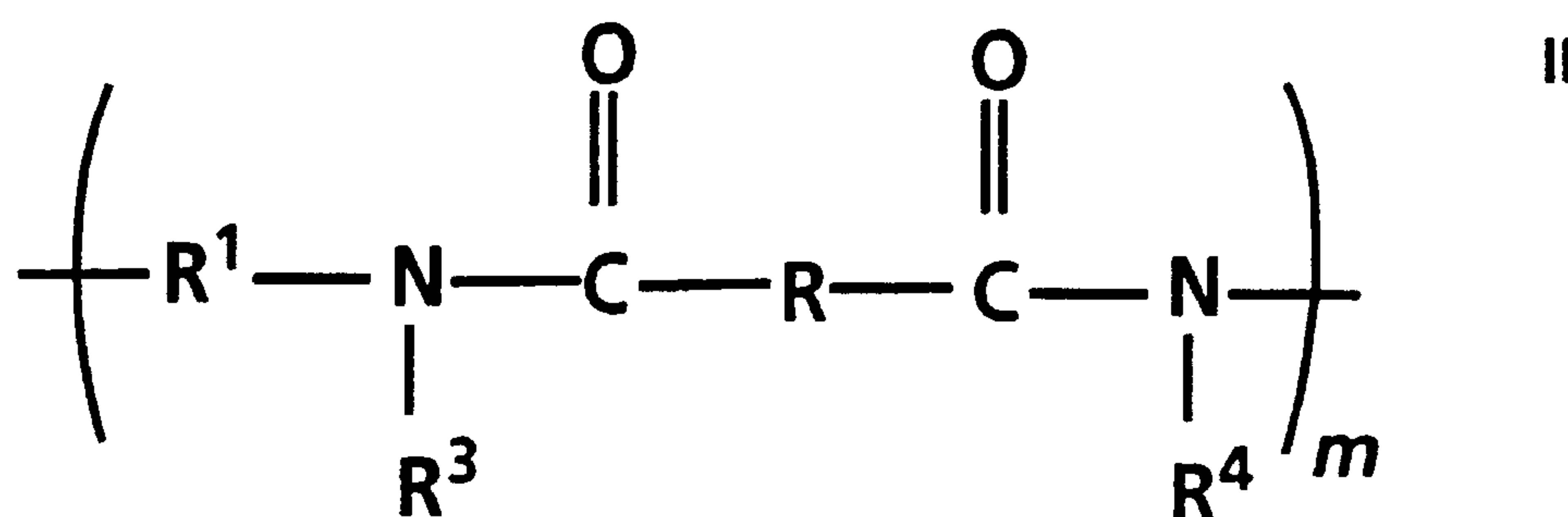
wherein:

n is a positive integer,

R is independently selected from the group consisting of alkylene, arylene or alkarylene units,

between 1 and 99 percent of the R² sites are -H, and

the remainder of the R² sites are -CH₂-O-CH₃ and



wherein:

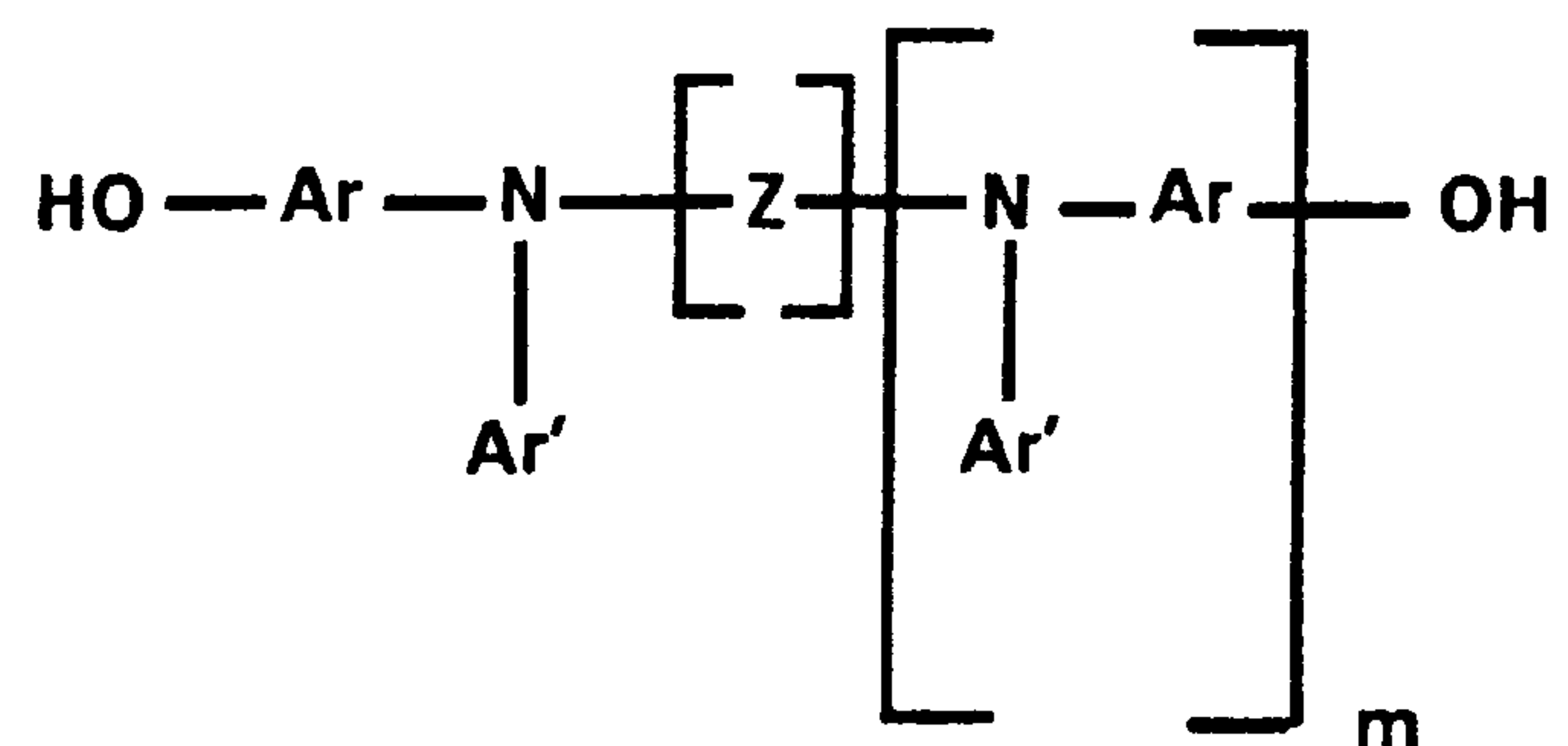
m is a positive integer,

R₁ and R are independently selected from the group consisting of alkylene, arylene or alkarylene units,

between 1 and 99 percent of the R³ and R⁴ sites are -H, and

the remainder of the R³ and R⁴ sites are -CH₂-O-CH₃.

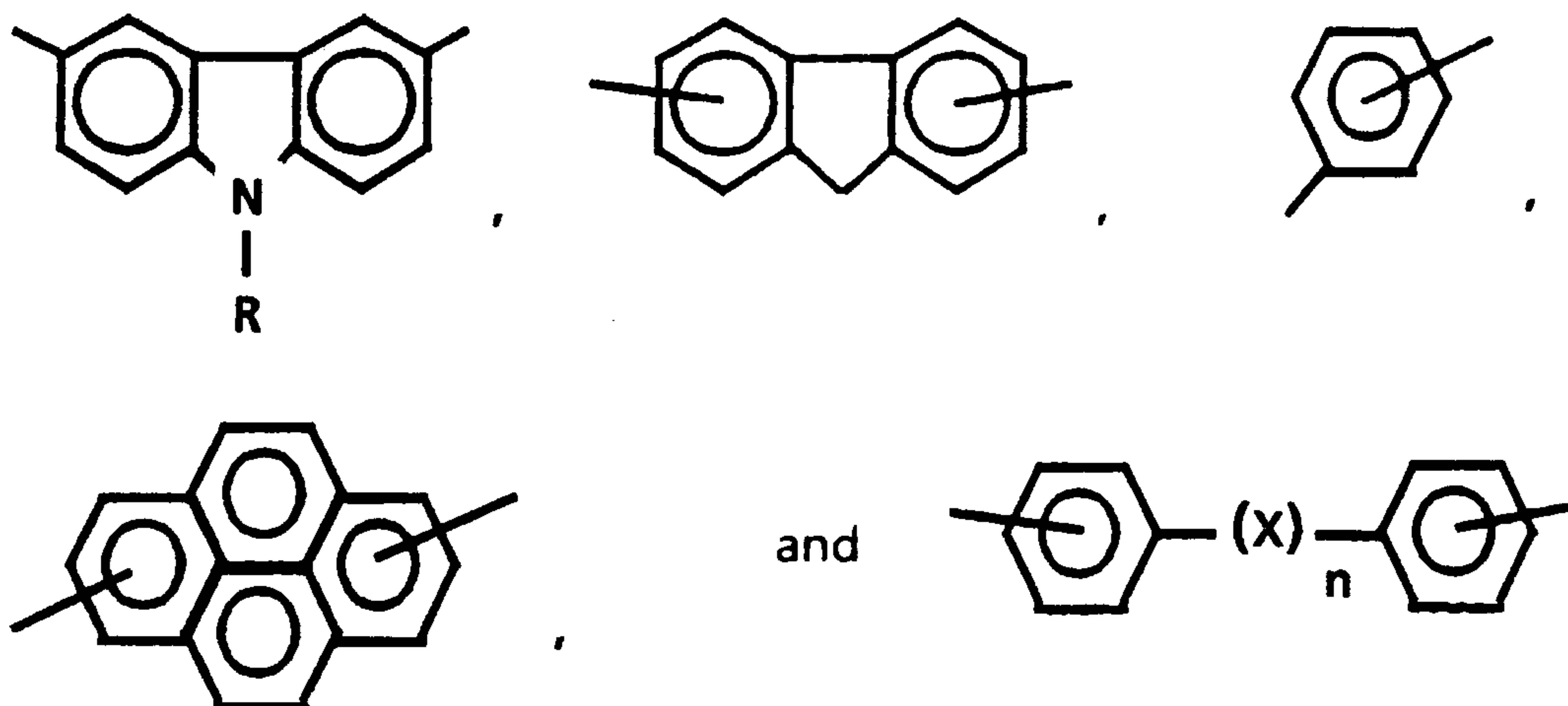
5. An electrophotographic imaging member according to claim 1 wherein said dihydroxy arylamine is represented by the following formula:



wherein:

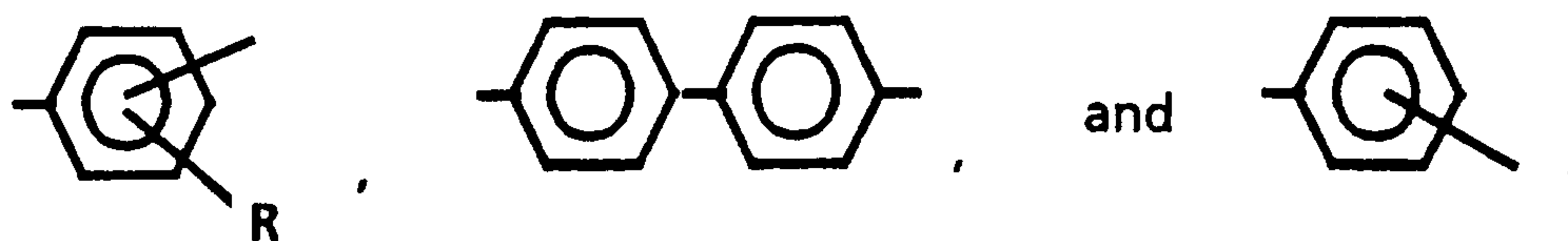
m is 0 or 1,

Z is selected from the group consisting of:



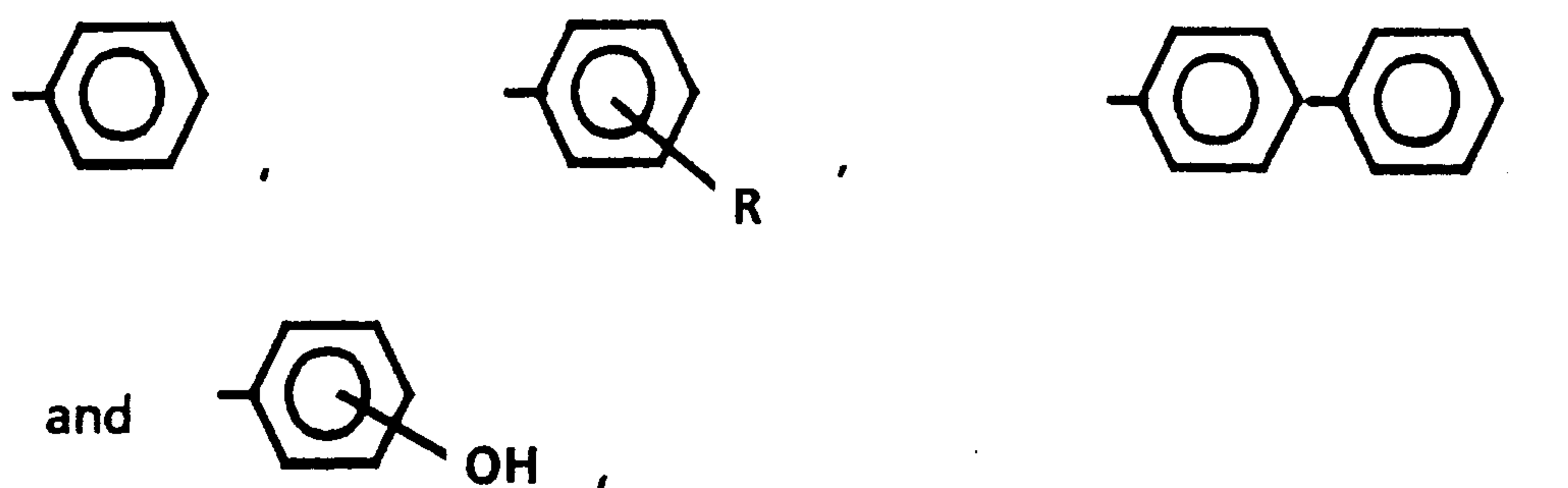
n is 0 or 1,

Ar is selected from the group consisting of:



R is selected from the group consisting of -CH₃, -C₂H₅, -C₃H₇, and -C₄H₉,

Ar' is selected from the group consisting of:



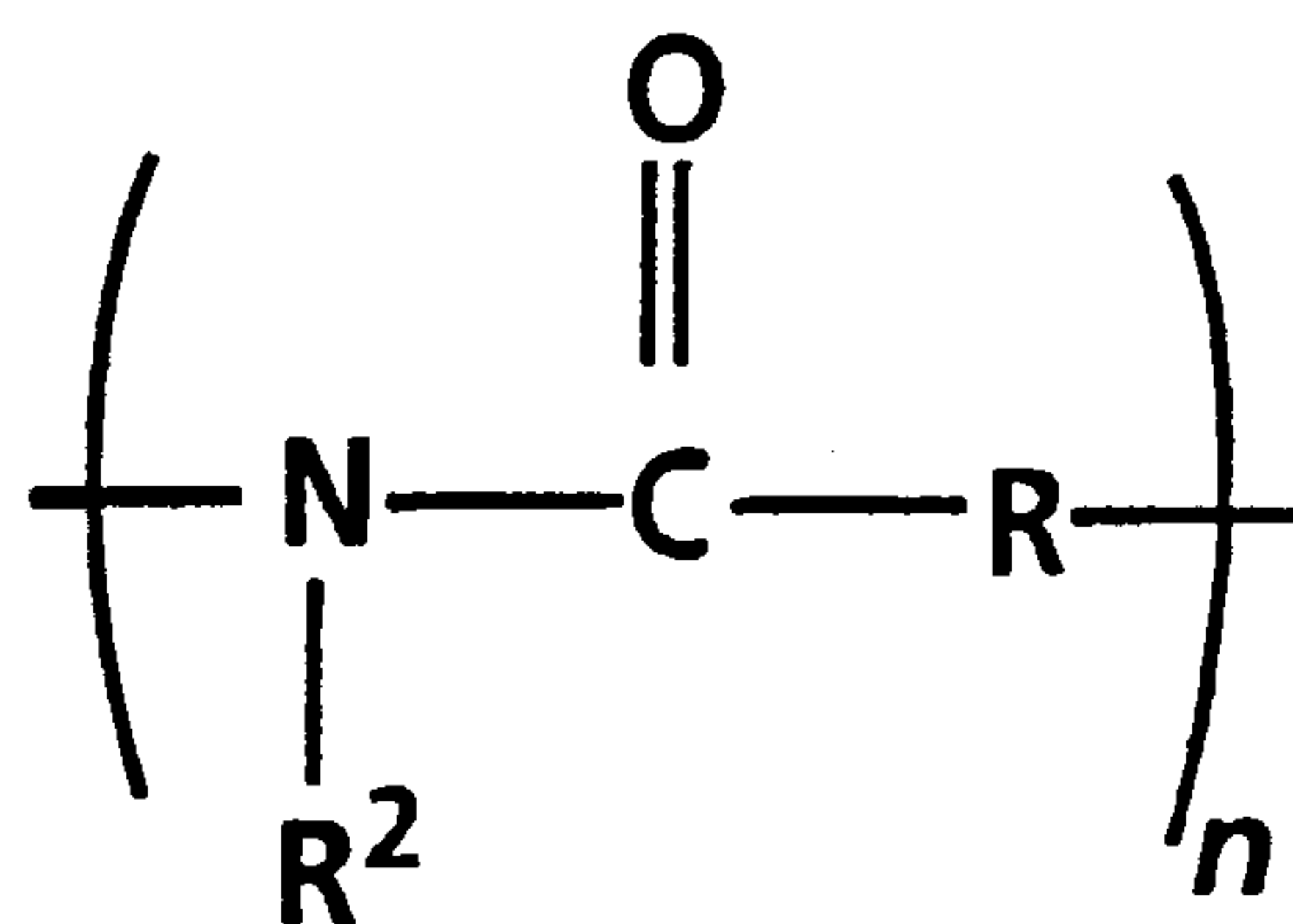
X is selected from the group consisting of:

9. An electrophotographic imaging member according to claim 1 wherein said overcoating has a thickness between about 1 micrometer and about 5 micrometers.

10. An electrophotographic imaging member according to claim 1 wherein said overcoating is hole transporting.

11. A crosslinkable coating composition comprising an alcohol soluble polyamide containing methoxy methyl groups attached to amide nitrogen atoms, a crosslinking catalyst and a dihydroxy arylamine.

12. A crosslinkable coating composition according to claim 11 wherein said polyamide is represented by the formulae I and II:



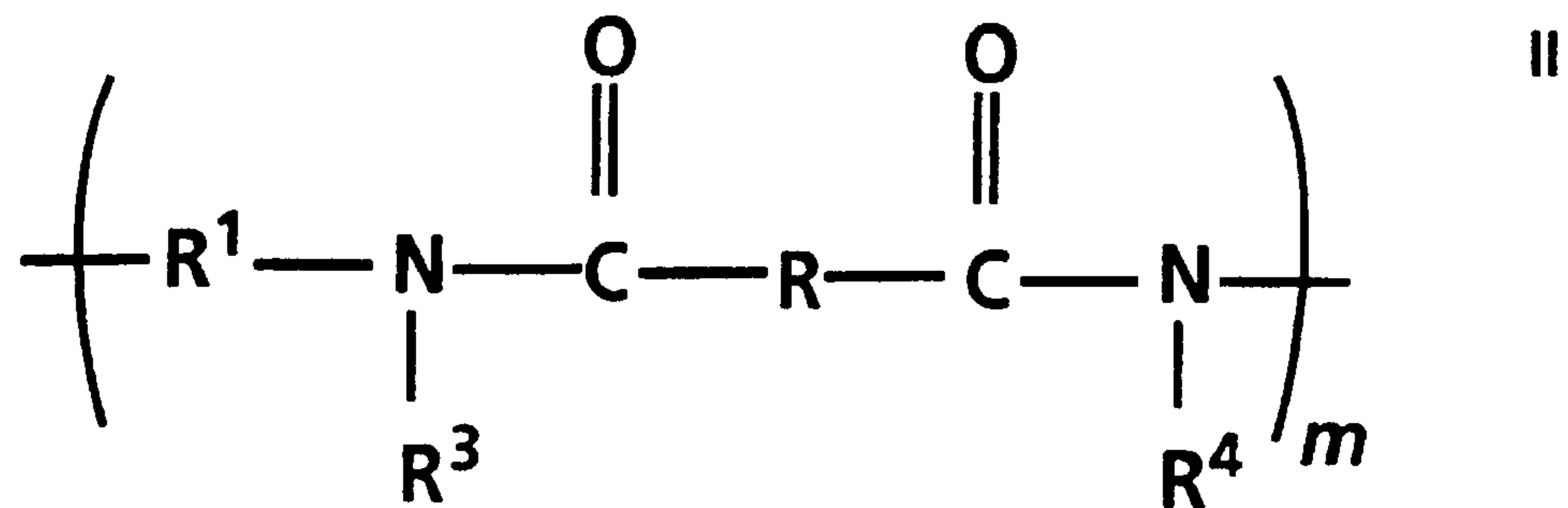
wherein:

n is a positive integer,

R is independently selected from the group consisting of alkylene, arylene or alkarylene units,

between 1 and 99 percent of the R² sites are -H, and

the remainder of the R² sites are -CH₂-O-CH₃.



wherein:

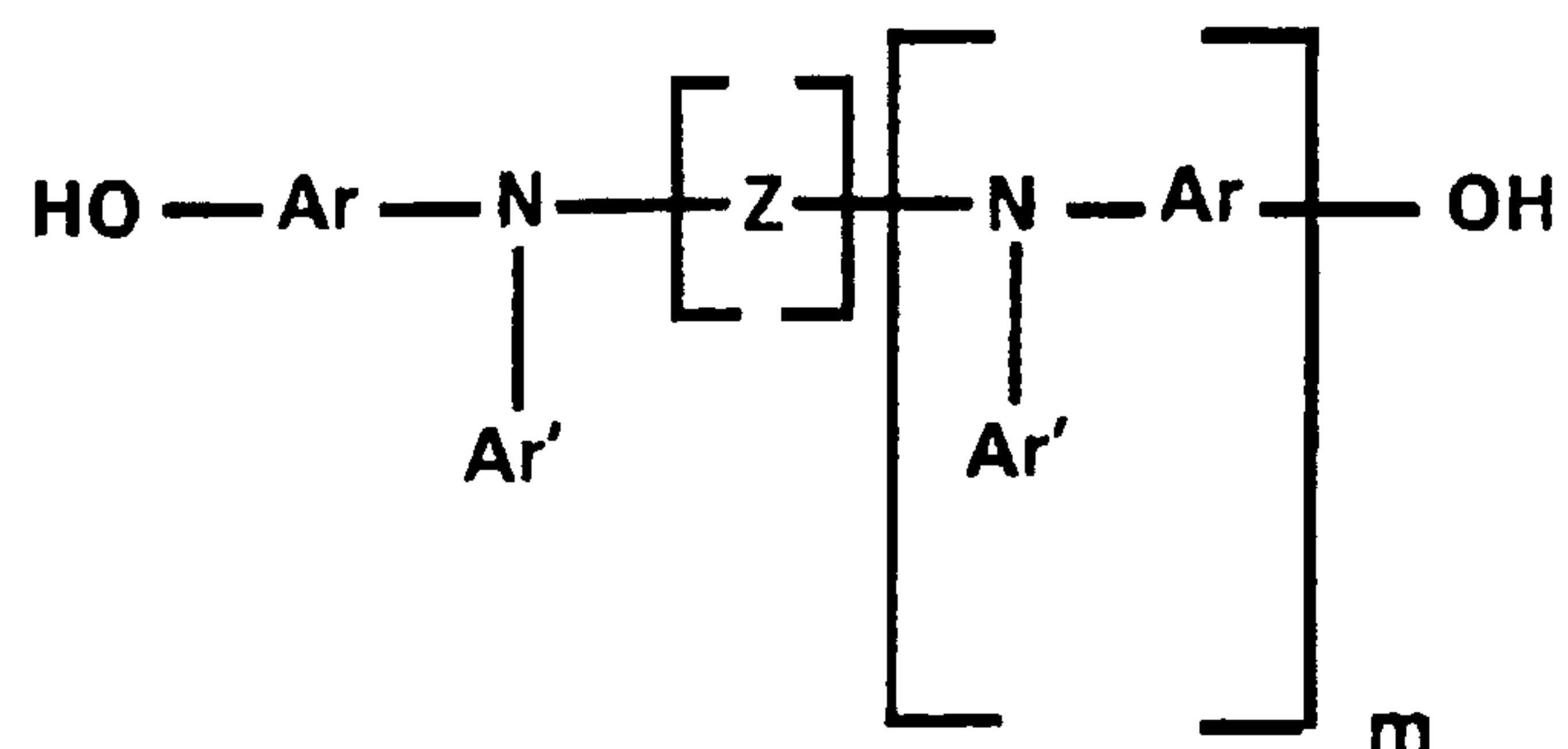
m is a number is a positive integer,

R₁ and R are independently selected from the group consisting of alkylene, arylene or alkarylene units,

between 1 and 99 percent of the R³ and R⁴ sites are -H, and

the remainder of the R³ and R⁴ sites are -CH₂-O-CH₃.

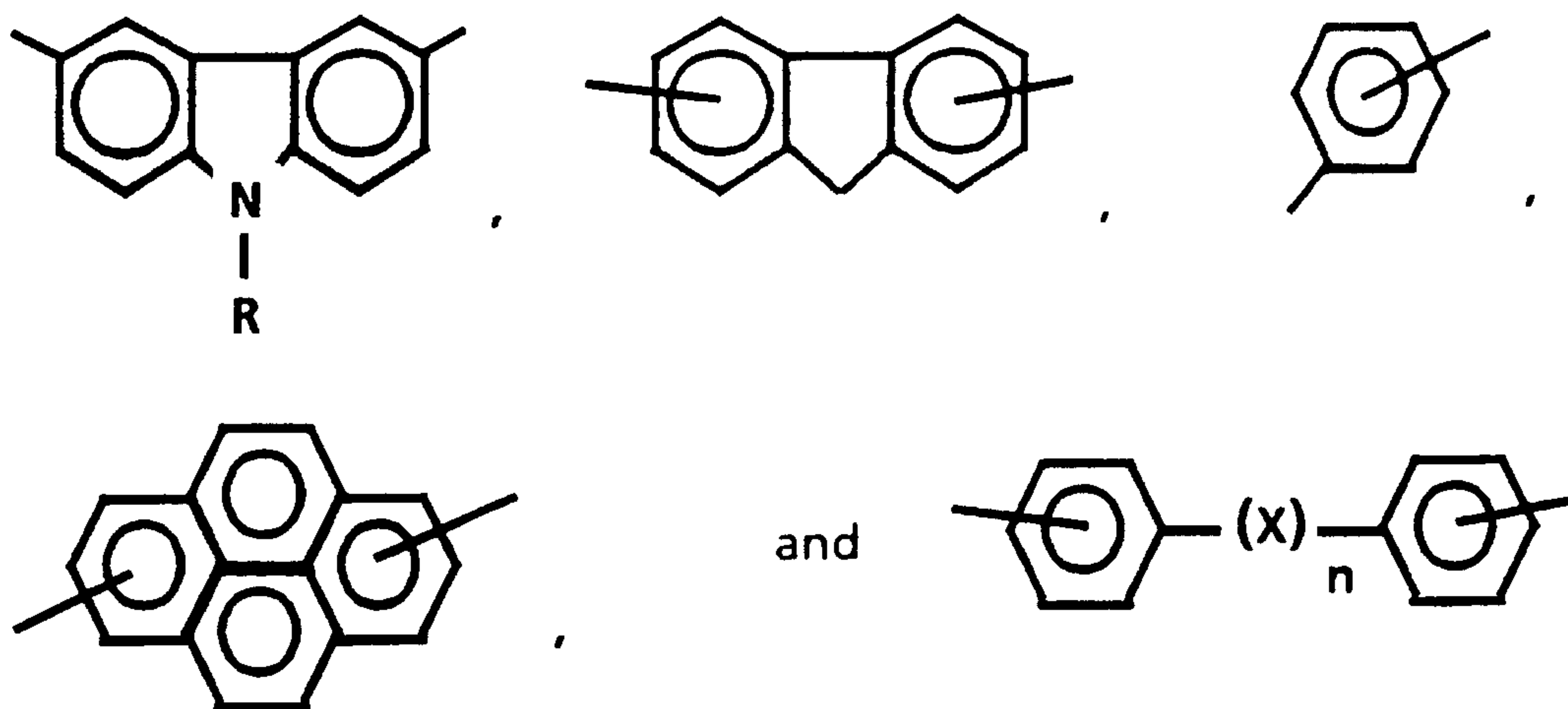
13. A crosslinkable coating composition according to claim 11 wherein said dihydroxy amine is represented by the formula:



wherein:

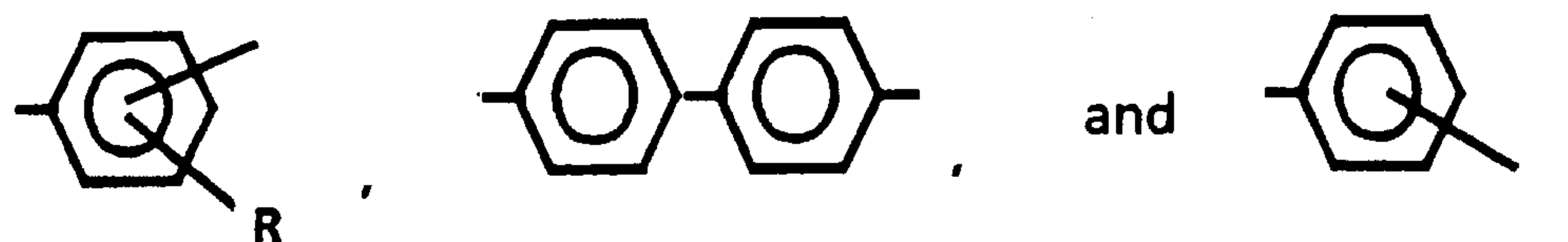
m is 0 or 1,

Z is selected from the group consisting of:



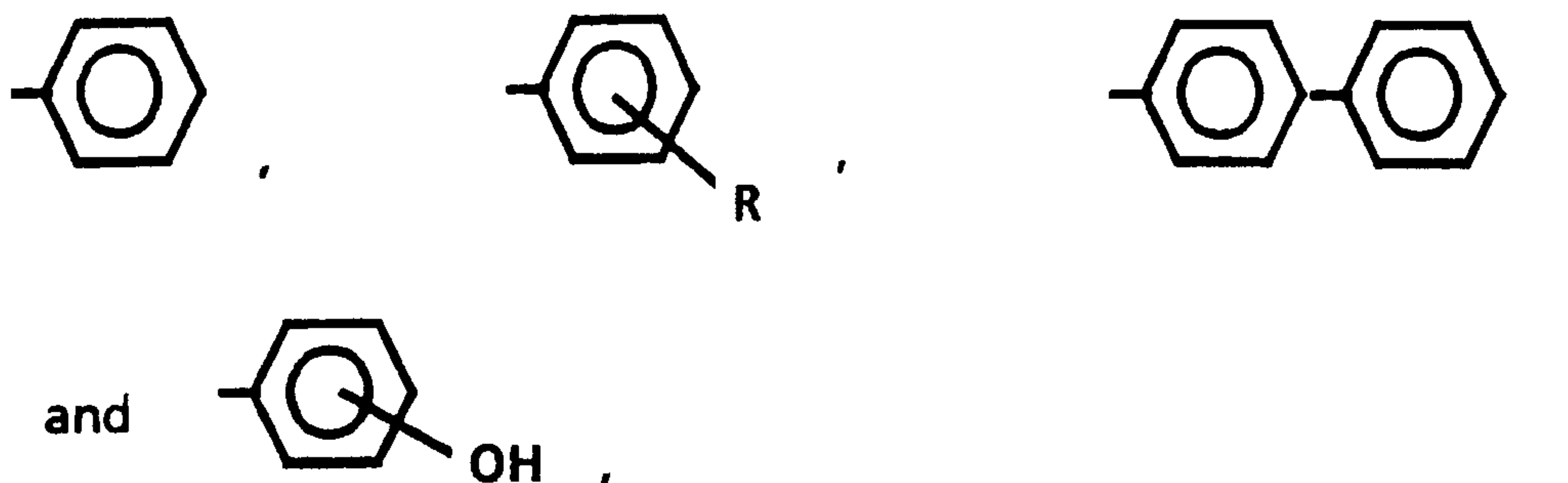
n is 0 or 1,

Ar is selected from the group consisting of:

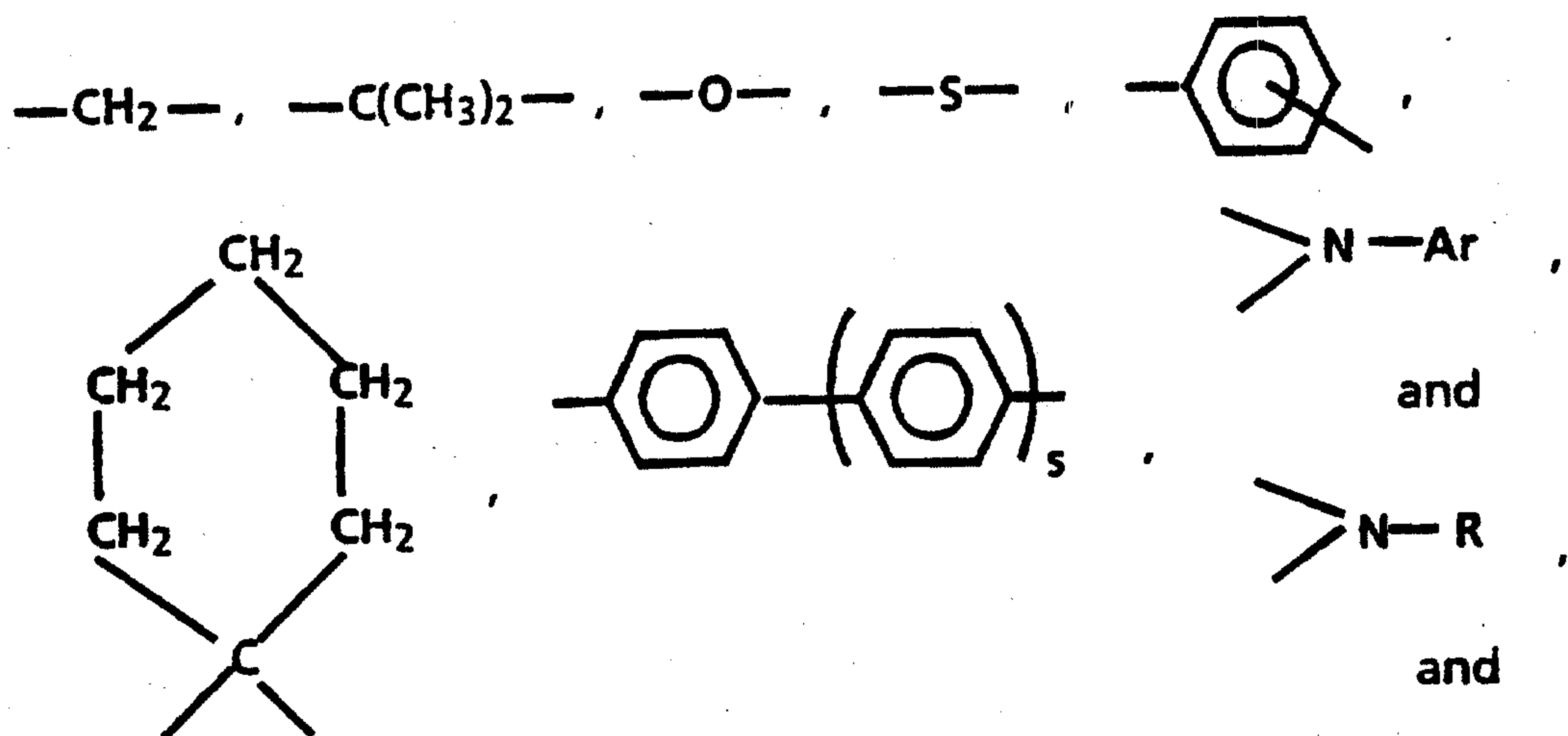


R is selected from the group consisting of -CH₃, -C₂H₅, -C₃H₇, and -C₄H₉,

Ar' is selected from the group consisting of:



X is selected from the group consisting of:



s is 0, 1 or 2,

said hydroxy arylamine compound being free of any direct conjugation between the -OH groups and the nearest nitrogen atom through one or more aromatic rings.

14. A crosslinkable coating composition according to claim 11 wherein said catalyst is oxalic acid.

15. A method of forming a coating comprising providing a substrate, forming a coating of a crosslinkable composition on said substrate, said crosslinkable coating composition comprising a polyamide containing methoxy methyl groups attached to amide nitrogen atoms, a crosslinking catalyst and a dihydroxy amine, and heating said coating to crosslink said polyamide.

16. An electrophotographic imaging process comprising providing an electrophotographic imaging member comprising a supporting substrate coated with at least a charge generating layer, a charge transport layer and an overcoating layer, said overcoating layer comprising a dihydroxy arylamine dissolved or molecularly dispersed in a crosslinked polyamide matrix, uniformly charging said imaging member, exposing said imaging member with activating radiation in image configuration to form an electrostatic latent image, developing said latent image with toner particles to form a toner image, and transferring said toner image to a receiving member.

17. An electrophotographic imaging process according to claim 16 including uniformly charging said imaging member with a contacting bias charging roll.

18. An electrophotographic imaging process according to claim 16 including transferring said toner image to a receiving member with a bias transfer roll.

19. An electrophotographic imaging process according to claim 16 wherein said toner particles are supplied to said latent image in a liquid developer comprising said toner particles dispersed in a liquid carrier.