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3,690,971

PYROTECHNIC COMPOSITION FOR COLORED SMOKE PRODUCTION

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No Drawing. Filed Aug. 11, 1970, Ser. No. 63,033

Int. Cl. C06d 3/00

U.S. Cl. 149—19

20 Claims

ABSTRACT OF THE DISCLOSURE

A pyrotechnic composition for colored smoke production including a dye precursor which is capable of reacting in situ to yield a sublimable dye which will recondense as a colored smoke and a heat producing composition which is capable of generating sufficient heat to react the dye precursor to yield the sublimable dye and to sublime the dye without substantial destruction thereof.

BACKGROUND OF THE INVENTION

(1) Field of the invention

This invention is in the field of pyrotechnic compositions for colored smoke production.

(2) Description of the prior art

Colored smokes are conventionally produced either by the atomization of a dye by the use of a charge of high explosive or by volatilization of a dye by the heat produced during the burning of a pyrotechnic composition. A conventional pyrotechnic composition for colored smoke production includes an organic dye mixed with a heat producing composition. The heat producing composition is typically a fuel-oxidizer combination such as sulfur-potassium chlorate or sucrose-potassium chlorate. The fuel-oxidizer combination normally contains a small amount of coolant such as sodium bicarbonate.

Pyrotechnic compositions for colored smoke production are used for target identification, rescue and other forms of visual communication. A typical round of a colored smoke producing composition is a canister which is partially filled with the composition to provide a dead space above the burning charge where the smoke can cool slightly to avoid excessive air oxidation of the heated pigment after it is discharged into the atmosphere. The charge is blended as a dry mix and pressed into the canister to give it a coherent shape. A small amount of glue or lacquer is sometimes added to help hold the pressed charge together. In some formulations a small amount of kerosene may be used to minimize dusting during processing.

To avoid the difficulties inherent in dry pressing these pyrotechnic compositions, polymeric binders have been used to yield a castable system. However, the amount of liquid binder required is excessive when large amounts of finely divided organic pigment are used. Accordingly, to achieve a castable composition, the organic pigment-pyrotechnic mixture is so diluted with binder that the smoke generating capability of the charge is unacceptably reduced.

SUMMARY OF THE INVENTION

A pyrotechnic composition for colored smoke production is provided wherein the composition contains a dye

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precursor which is capable of reacting in situ to yield a sublimable dye which will recondense as a colored smoke and a heat producing composition which is capable of generating sufficient heat to react the dye precursor to yield the sublimable dye and to sublime the dye without substantial destruction thereof.

DESCRIPTION OF THE INVENTION

An object of the present invention is the provision of a pyrotechnic composition for colored smoke production based on a dye precursor which is capable of reacting in situ to yield a sublimable dye which will recondense as a colored smoke.

A further object of the present invention is the provision of a castable pyrotechnic composition for colored smoke production.

Further objects and advantages of the present invention will become apparent upon reading the undergoing specification and claims.

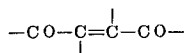
A pyrotechnic composition for colored smoke production is provided which employs dye precursors which will react in situ to yield a sublimable dye. The term "dye" as used herein is used in its broadest sense to mean a material which imparts color by dyeing and includes the term pigment. The term "dye precursor" as used herein means one or more materials which precede the formation of a dye. In its broadest aspects the pyrotechnic composition of the present invention differs from pyrotechnic compositions used in the prior art in that a dye precursor rather than a dye per se is used in the colored smoke producing composition.

The dye precursor is used in combination with a heat producing composition. The heat producing composition generates sufficient heat to react the dye precursor to yield a sublimable dye and to sublime the dye. In preferred form, the heat producing composition generates gas to aid in dispersing the dye in the atmosphere.

The sublimable dye produced in situ by the reaction of the dye precursor should be heat and oxidation resistant and should be capable of recondensing as a colored smoke. A broad class of dyes can be generated in situ according to the present dye precursor technique. The dyes include nitro, azo, triphenylmethane, xanthene, indigoid, quinone-imine, thiazole, anthraquinone, naphthalic acid derivative, diamionaphthalene derivative and quinoline derivative dyes. Applicable nitro dyes include those dyes with the characteristic nitroarylamine structural unit as, for example, Lithol Fast Yellow GG (CI 14). Usable azo dyes generically include soluble polyazo dyes with the characteristic structural unit $\text{Ar}-\text{N}=\text{N}-\text{Ar}'$. The preferred soluble polyazo dyes are the diazo dyes. Of the diazo dyes derivatives of tetrazotized diamines such as Chrysamine G (CI 419) and primary diazo dyes such as Bismarck Brown G (CI 331) can be mentioned as illustrative. Other preferred azo dyes are the mordant dyes with the characteristic o-hydroxyazo group such as Alizarine Yellow 5G (CI 112). From the triphenylmethane genus, usable sublimable dyes from the diamine, triamine and phenol subgenera can be employed. Malachite Green (CI 657) is illustrative of the triphenylmethane diamines (Malachite Green series) which can be employed. Pararosaniline (CI 42500) is illustrative of the triphenylmethane triamines (Magenta series) which can be employed. Of the triphenylmethane phenols (Rosolic Acid series), Rosolic Acid (CI 724) and Chrome Violet (CI 727) can be mentioned. Usable xanthenes

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include diphenylmethane derivatives such as Pyronine G (CI 739) and Rhodamine S (CI 743) and triphenylmethane derivatives (phthaleines) such as Fluorescein (CI 45350) and Rhodamine 5G (CI 746). 2,2'-derivatives of indole and thionaphthene with the characteristic structural unit



are illustrative of the indigoids which can be employed. Usable species are Indigo (CI 1177) and Thioindigo (CI 1207). Usable quinoneimines include indophenols such as Indophenol Blue (CI 821) and indamines such as phenylene Blue (CI 49400). Primuline (CI 812) is an example drawn from the thiazole genus. Usable anthraquinones include vat dyes such as 1,12-benzoperylene-dicarboxylic anhydride from the anthrone derivative subgenus. Derivatives of naphthalic acid which can be mentioned are Indanthrene Scarlet R (CI 71140) and Indanthrene Scarlet GG (CI 71110). Phthaloperinone is illustrative of the diamionaphthalene derivatives. Quinoline Yellow (CI 800) is illustrative of the quinoline derivatives which can be employed. The color index (CI) numbers used herein refer to Rowe, Colour Index, Society of Dyers and Colourist, Bradford, 1924. The dye precursors from which the above mentioned sublimable dyes are prepared are well known in the art and can be found, for example, in K. Venkataraman, The Chemistry of Synthetic Dyes, Academic Press, Inc., N.Y., 1952. For example, Chrysamine G (CI 410) is prepared by the reaction of diazotized benzidine and salicylic acid and Alizarin Yellow 5G (CI 112) is prepared by the reaction of diazotized o-anisidine and salicylic acid.

The heat producing composition is usually a conventional fuel-oxidizer combination. Suitable fuels include sulfur, sucrose, charcoal, liquid polyhydroxy compounds such as lower alkyl triols (e.g. glycerol) and liquid alkylene oxide polymers (e.g., liquid ethylene oxide polymers). Suitable oxidizers include alkali metal perchlorates such as lithium and potassium perchlorate, alkali metal chlorates such as potassium chlorate, alkali metal nitrates such as sodium nitrate and ammonium compounds such as ammonium nitrate and ammonium perchlorate. The preferred class of oxidizers are the alkali metal chlorates and, in particular, potassium chlorate.

The pyrotechnic composition can additionally contain a coolant which generates gas and acts as a combustion controlling agent and serves primarily to prevent the composition from burning with an open flame. Suitable coolants include alkali metal carbonates such as sodium or potassium carbonate, alkali metal bicarbonates such as sodium or potassium bicarbonate, alkali metal and alkaline earth metal salts of citric acid such as calcium alkaline earth metal salts of oxalic acids, ammonium chloride and alkali metal salts of barbituric acid.

The heat producing composition will normally be one which is capable of heating the pyrotechnic composition to a temperature between 600 and 1000° C. since a temperature in this range is normally required to sublime the dye. The sublimation temperature is normally well above the reaction temperature of the dye precursors employed. Accordingly, the heat producing composition will be satisfactory if it generates sufficient heat to sublime the dye. It is general practice in the smoke production art to try to maintain a burning temperature between 600 and 750° C. This temperature range is therefore preferred and can be maintained by a proper choice of oxidizer and coolant as is well known in the art. Temperatures above 1000° C. should be avoided since temperatures in this range may destroy a substantial portion of the dye which is generated by pyrolysis.

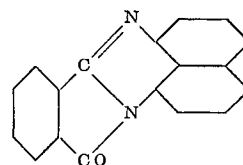
The pyrotechnic composition of the present invention can be made castable by using several techniques. Liquid fuels such as liquid alkylene oxide polymers can be em-

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ployed. Additionally, liquid dye precursors which will act as plasticizers in a castable composition can be employed in appropriate dye precursor systems. If desired, polymeric binders can be added to the system per se or generated in situ. The in situ generation of the polymeric binder can be accomplished by polymerizing a liquid monomer in situ or by crosslinking part of the fuel and/or dye precursor. Suitable crosslinking agents include compounds which contain a plurality of oxirane or isocyanate groups such as vinylcyclohexene dioxide, dicyclopentadiene dioxide, condensation products of epichlorohydrin and bisphenol-A and 2,4-toluene diisocyanate. Crosslinking agents containing other functional end groups are also usable as will be apparent to one skilled in the art. The polymeric binder will serve a dual function as binder and fuel in those systems where the heat generated by the pyrotechnic composition is above the combustion temperature of the binder. When a binder is employed, a small amount of thickener can be added to prevent segregation of the ingredients of the pyrotechnic composition during the curing cycle.

The smoke producing compositions will usually contain, by weight, 25 to 75% dye precursor and 25 to 75% heat producing composition. When a fuel-oxidizer combination is used as the heat producing composition, the smoke producing composition will usually contain 1 to 25% fuel (exclusive of binder) and 20 to 50% oxidizer. The oxidizer will normally be present in the pyrotechnic composition in a weight ratio of 4 or 5 to 1 based on the weight of fuel present. When coolants, binders and thickeners are employed, they will normally be present in from 0 to 10 weight percent, 5 to 25 weight percent and 0 to 5 weight percent, respectively, based on the total smoke producing composition weight. The weight percent of binder set forth herein is based on the amount of polymer, monomer or crosslinking agent added to the pyrotechnic composition and is exclusive of fuel and/or dye precursor which may react with the crosslinking agent.

A referred pyrotechnic composition for orange smoke production is a smoke precursor system based on phthalic anhydride and 1,8-diamionaphthalene. The dye precursors phthalic anhydride and 1,8-diamionaphthalene react in situ to yield phthaloperinone which has the following structure:



This smoke precursor system per se is not castable because the dye precursors phthalic anhydride and 1,8-diamionaphthalene are not liquids. However, a castable system can be formulated by using a liquid fuel, by employing various binders and/or by replacing part or all of the phthalic anhydride with a half ester of phthalic acid which is a liquid or has a low melting point. Suitable liquid fuels are glycerol and liquid polyethylene glycols. Suitable binders include epoxy resins such as the condensation products of epichlorohydrin and bisphenol-A of which Epon® 828 (Shell Chemical Co., New York, N.Y.) is illustrative. Suitable half esters of phthalic acid include monoalkyl acid phthalates such as n-hexyl acid phthalate or n-octyl acid phthalate. When a coolant is employed with the orange smoke composition for gas-generation and cooling, it is preferred to employ sodium barbiturate or calcium citrate. When a castable orange smoke composition is employed, thickeners such as an-

hydrous and particulate silica can be employed to thicken the initial mix and prevent segregation of the ingredients during curing. An example of a suitable silica thickener is Cab-O-Sil (Cabot Corp., Boston, Mass.).

A preferred non-castable pyrotechnic composition for orange smoke production contains 40 to 50 weight percent oxidizer, 10 to 25 weight percent fuel and 25 to 50 weight percent dye precursor. The dye precursor system preferably contains about 50 mole percent phthalic anhydride and about 50 mole percent 1,8 - diamino-naphthalene. The preferred fuel is sucrose or glycerol.

A preferred castable pyrotechnic composition for orange smoke production contains, by weight, 20 to 40% oxidizer, 1 to 15% fuel, 5 to 15% binder and 20 to 50% dye precursor. The preferred dye precursor system contains about 12.5 to 50 mole percent phthalic anhydride, 0 to 37.5 mole percent n-hexyl acid phthalate and 50 mole percent 1,8-diaminonaphthalene. The preferred fuel is glycerol.

Yellow smoke production is preferably based on the oxidation of primary aromatic amines. The dye precursors of choice for yellow smoke production are primary phenylene amines. The preferred primary phenylene amines are aminophenols such as p-aminophenol, phenylene diamines such as p-phenylene diamine and diphenylene diamines such as p-diaminodiphenyl. These dye precursors are believed to yield low molecular weight sublimable azo compounds or polymers by oxidative coupling. The preferred non-castable yellow smoke generating composition contains, by weight, 30 to 50% oxidizer, 10 to 25% fuel and 25 to 60% primary phenylene amine dye precursor. The preferred castable yellow smoke generating composition contains, by weight, 20 to 40% oxidizer, 1 to 25% fuel, 10 to 25% binder and 20 to 50% primary phenylene amine dye precursor. The preferred dye precursor is p-phenylene diamine.

A violet smoke producing composition can be prepared based on the dye precursors 1,8-diaminonaphthalene and pyromellitic dianhydride which yield on combustion a dye of the phthaloperinone type. The pressable violet smoke producing composition preferably contains, by weight, 20 to 50% oxidizer, 1 to 20% fuel and 50 to 75% dye precursor. The castable violet smoke generating composition preferably contains, by weight, 20 to 40% oxidizer, 1 to 15% fuel, 10 to 25% binder and 35 to 65% dye precursor. The dye precursor system preferably contains about 66.6 mole percent 1,8 - diamino-naphthalene and about 33.3 mole percent pyromellitic dianhydride.

Red smoke can be produced by using the dye precursors 1,8 - diamino-naphthalene and tetrabromophthalic anhydride to yield a dye of the phthaloperinone type. A preferred red smoke producing composition contains about 20 to 50 weight percent oxidizer, 1 to 20 weight percent fuel and 30 to 70 weight percent dye precursor wherein the dye precursor system contains about 50 mole percent 1,8 - diamino-naphthalene and about 50 mole percent tetrabromophthalic anhydride.

A pyrotechnic composition for green smoke production is based on the dye precursor p-dimethylaminobenzaldehyde. A preferred green smoke producing composition contains, by weight, about 30 to 50% oxidizer, 5 to 25% fuel and 25 to 50% p-dimethylaminobenzaldehyde.

The pyrotechnic compositions of the present invention can be ignited by any conventional technique such as by the use of an electric squib or a small amount of first fire pyrotechnic mixture of fuel and oxidizer ignited by a match. Solids are used in particle sizes which are conventional in the pyrotechnic art. Percentages are based on weight herein unless specified to the contrary.

The following non-limitive examples illustrate the invention:

EXAMPLE 1

A batch of an orange smoke producing composition was prepared by handmixing the following ingredients:

Ingredients:	Wt. percent
Potassium chlorate	42.8
Sucrose	23.9
Phthalic anhydride	15.9
1,8-diaminonaphthalene	17.1

The handmixed composition was pressed by hand into a size 9 nickel-plated sewing thimble and ignited by placing a small amount of a stoichiometric mixture of potassium chlorate and sulfur on top of the handmixed composition and applying a match to the mixture. The composition burned to produce an orange smoke.

EXAMPLE 2

A batch of an orange smoke producing composition was prepared by handmixing the following ingredients:

Ingredients:	Wt. percent
Potassium chlorate	49.3
Glycerol	19.5
Phthalic anhydride	19.3
1,8-diaminonaphthalene	20.9

The handmixed composition was transferred from the mixing vessel to a size 9 nickel-plated sewing thimble. The composition was ignited by placing a match to a stoichiometric mixture of potassium chlorate and sulfur and burned with an orange smoke.

EXAMPLE 3

A batch of an orange smoke producing composition was prepared by handmixing the following ingredients:

Ingredients:	Wt. percent
Potassium chlorate	39.9
Polyethylene glycol (molecular wt. 400)	5.9
Glycerol	13.9
Phthalic anhydride	19.4
1,8-diaminonaphthalene	20.9

The handmixed composition was transferred from the mixing vessel to a size 9 nickel-plated sewing thimble. The composition was ignited by placing a match to a stoichiometric mixture of potassium chlorate and sulfur and burned with an orange smoke.

EXAMPLE 4

A 250 gram batch of an orange smoke producing composition was prepared from the following ingredients:

Ingredients:	Wt. percent
Potassium chlorate	31
Glycerol	10
Vinyl cyclohexene dioxide	11
n-Hexyl acid phthalate	26
1,8-diaminonaphthalene	14
Sodium barbiturate	8

The ingredients were mixed in a standard air driven one pint vertical mixer with an input horsepower rating of 0.34. The ingredients were stirred for 20 minutes, the mixer stopped, the blades scraped and the mixing was then continued for an additional 10 minutes. The mixture was cast into an aluminum tube with an inner diameter of 1.5 inches and a length of 3 inches which was provided with a 1/2-inch outer diameter mandrel. The aluminum tube was placed in an oven and the casting cured at 55-60° C. for 72 hours. The mandrel was removed from the casting and the aluminum tube inserted into a cylindrical metal canister with an inner diameter of approximately

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1.5 inches and a length of approximately 3 inches and with a 3/8-inch port in one end. The casting was ignited using a mixture containing silicon, cuprous oxide and lead oxide (PbO₂) in a weight ratio of 1:1:1 ignited by a hot wire and provided copious quantities of orange smoke. The smoke producing composition had a burn time of approximately six minutes.

EXAMPLE 5

A 250 gram batch of an orange smoke producing composition was prepared, cast and fitted in a metal canister as set forth in Example 4. The smoke producing composition was prepared from the following ingredients:

Ingredients:	Wt. percent
Potassium chlorate	31
Glycerol	10
Epon® 828	11
n-Hexyl acid phthalate	12
1,8-diaminonaphthalene	17
Sodium barbiturate	8
Phthalic anhydride	11

The composition burned for approximately 1½ minutes and yielded copious quantities of orange smoke.

EXAMPLE 6

A composition for yellow smoke production was prepared by handmixing 44 parts by weight potassium chlorate, 22 parts by weight sucrose and 34 parts by weight p-phenylene diamine. The mixture was pressed in a standard 25 ton press and ignited to yield copious quantities of yellow smoke.

EXAMPLE 7

A castable composition for yellow smoke production was prepared by combining 23 parts by weight potassium chlorate, 21 parts by weight sucrose, 34 parts by weight p-phenylene diamine and 22 parts by weight vinyl cyclohexene dioxide. The mixture was prepared in a standard air driven one pint vertical mixer with an input horsepower rating of 0.34 by mixing the ingredients for 30 minutes. The mixture was cast into an aluminum tube (1.5 inch inner diameter, 3-inch length) provided with a 1/2-inch outer diameter mandrel. The aluminum tube was placed in an oven and the casting cured overnight at 55–60° C. After the mandrel was removed, the aluminum tube was inserted into a cylindrical metal canister with a 3/8-inch port in one end. The casting was ignited and burned with a yellow smoke.

EXAMPLE 8

A violet smoke generating composition was prepared by handmixing 33 parts by weight potassium chlorate, 7 parts by weight sucrose, 30 parts by weight 1,8-diaminonaphthalene and 30 parts by weight pyromellitic dianhydride. The composition yielded a violet smoke on ignition.

EXAMPLE 9

A violet smoke generating composition was prepared by handmixing 26.9 parts by weight potassium chlorate, 3 parts by weight sucrose, 42 parts by weight 1,8-diaminonaphthalene and 28 parts by weight pyromellitic dianhydride. This composition yielded a violet smoke on ignition.

EXAMPLE 10

A composition for violet smoke production was prepared from the following ingredients:

Ingredients:	Wt. percent
Potassium chlorate	22.4
Sucrose	2.6
Pyromellitic dianhydride	23.2
1,8-diaminonaphthalene	34.7
Vinyl cyclohexene dioxide	17.2

The ingredients were mixed in a standard air driven one pint vertical mixer with an input horsepower rating of

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0.34 for 30 minutes. The mixture was cast into an aluminum tube (1.5-inch inner diameter; 3-inch length) provided with a 1/2-inch outer diameter mandrel. The aluminum tube was placed in an oven and the casting cured at 55–60° C. for 72 hours. After the mandrel was removed, the aluminum tube was inserted into a cylindrical metal canister with a 3/8-inch port in one end. The casting was ignited using a 1:1:1 weight ratio silicon, cuprous oxide and lead oxide (PbO₂) mixture ignited by a hot wire. Copious quantities of violet smoke were produced by the burning casting.

EXAMPLE 11

A composition for red smoke production was prepared by handmixing 32.6 parts by weight potassium chloride, 4.2 parts by weight sucrose, 15.9 parts by weight 1,8-diaminonaphthalene and 47.3 parts by weight tetrabromophthalic anhydride. The composition was ignited by conventional technique and burned under a blanket of nitrogen to yield a red smoke.

EXAMPLE 12

A green smoke generating composition was prepared by handmixing 32.6 parts by weight potassium chlorate, 17.9 parts by weight sucrose and 50 parts by weight p-dimethylaminobenzaldehyde. The composition was ignited by conventional technique and burned under a blanket of nitrogen gas with an apple green smoke.

We claim:

1. A pyrotechnic composition for colored smoke production comprising:

(a) an organic dye precursor which is capable of reacting in situ to yield a sublimable dye which will recondense as a colored smoke; and

(b) a heat producing composition which is capable of generating sufficient heat to react said dye precursor to yield said sublimable dye and to sublime said dye without substantial destruction of said dye.

2. The composition of claim 1 in which the heat producing composition is capable of generating gas to disperse said sublimable dye in the atmosphere.

3. The composition of claim 1 in which the pyrotechnic composition contains 25 to 75 weight percent dye precursor and 25 to 75 weight percent heat producing composition.

4. The composition of claim 3 in which the heat producing composition comprises a fuel and an oxidizer capable of oxidizing said fuel.

5. A pyrotechnic composition for colored smoke production comprising:

(a) an organic dye precursor which is capable of reacting in situ to yield a sublimable dye which will recondense as a colored smoke;

(b) a fuel which is capable of generating sufficient heat to react said dye precursor to yield said sublimable dye and to sublime said dye without substantial destruction of said dye; and

(c) an oxidizer which is capable of oxidizing said fuel.

6. The composition of claim 5 in which the pyrotechnic composition contains 25 to 75 weight percent dye precursor, 1 to 25 weight percent fuel and 20 to 50 weight percent oxidizer.

7. The composition of claim 5 in which the dye precursor comprises phthalic anhydride and 1,8-diaminonaphthalene.

8. The composition of claim 7 in which the pyrotechnic composition contains 40 to 50 weight percent oxidizer, 10 to 25 weight percent fuel and 25 to 50 weight percent dye precursor.

9. The composition of claim 7 in which the pyrotechnic composition contains 20 to 40 weight percent oxidizer, 1 to 15 weight percent fuel, 5 to 15 weight percent binder and 20 to 50 weight percent dye precursor.

10. The composition of claim 5 in which the dye precursor is a primary phenylene amine.

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11. The composition of claim 10 in which the primary phenylene amine is p-phenylene diamine.

12. The composition of claim 10 in which the pyrotechnic composition contains 30 to 50 weight percent oxidizer, 10 to 25 weight percent fuel and 25 to 60 weight percent dye precursor.

13. The composition of claim 10 in which the pyrotechnic composition contains 20 to 40 weight percent oxidizer, 1 to 25 weight percent fuel, 10 to 25 weight percent binder and 20 to 50 weight percent dye precursor.

14. The composition of claim 5 in which the dye precursor comprises pyromellitic dianhydride and 1,8-diaminonaphthalene.

15. The composition of claim 14 in which the pyrotechnic composition contains 20 to 50 weight percent oxidizer, 1 to 20 weight percent fuel and 50 to 75 weight percent dye precursor.

16. The composition of claim 14 in which the pyrotechnic composition contains 20 to 40 weight percent oxidizer, 1 to 15 weight percent fuel, 10 to 25 weight percent binder and 35 to 65 weight percent dye precursor.

17. The composition of claim 5 in which the dye precursor comprises tetrabromophthalic anhydride and 1,8-diaminonaphthalene.

18. The composition of claim 17 in which the pyrotechnic composition contains 20 to 50 weight percent oxidizer,

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1 to 20 weight percent fuel and 30 to 70 weight percent dye precursor.

19. The composition of claim 5 in which the dye precursor is p-dimethylaminobenzaldehyde.

20. The composition of claim 19 in which the pyrotechnic composition contains 30 to 50 weight percent oxidizer, 5 to 25 weight percent fuel and 25 to 50 weight percent dye precursor.

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U.S. Cl. X.R.

149—60, 61, 78, 83, 84