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(54) **ELECTROLUMINESCENT MATERIALS AND DEVICES**

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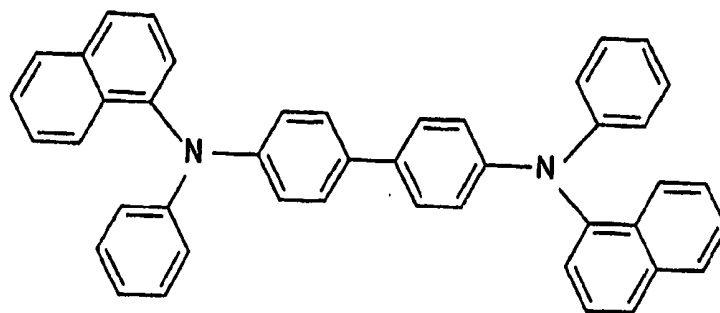
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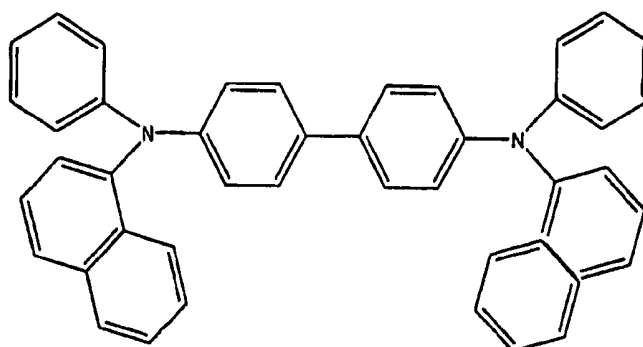
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(57) **ABSTRACT**

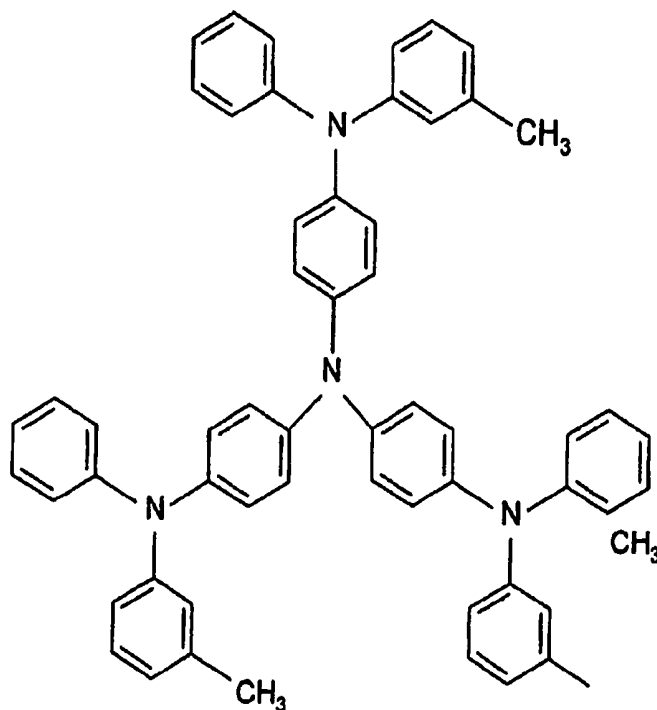
An electroluminescent material is a metal complex of 1-phenyl-3-methyl-4-trimethylacetyl-pyrazol-5-one of formula (I). An electroluminescent device comprising the compound of formula (I) in the luminescent layer is also part of the invention.



α -NPB



β -NPD



mTADATA

Fig. 1

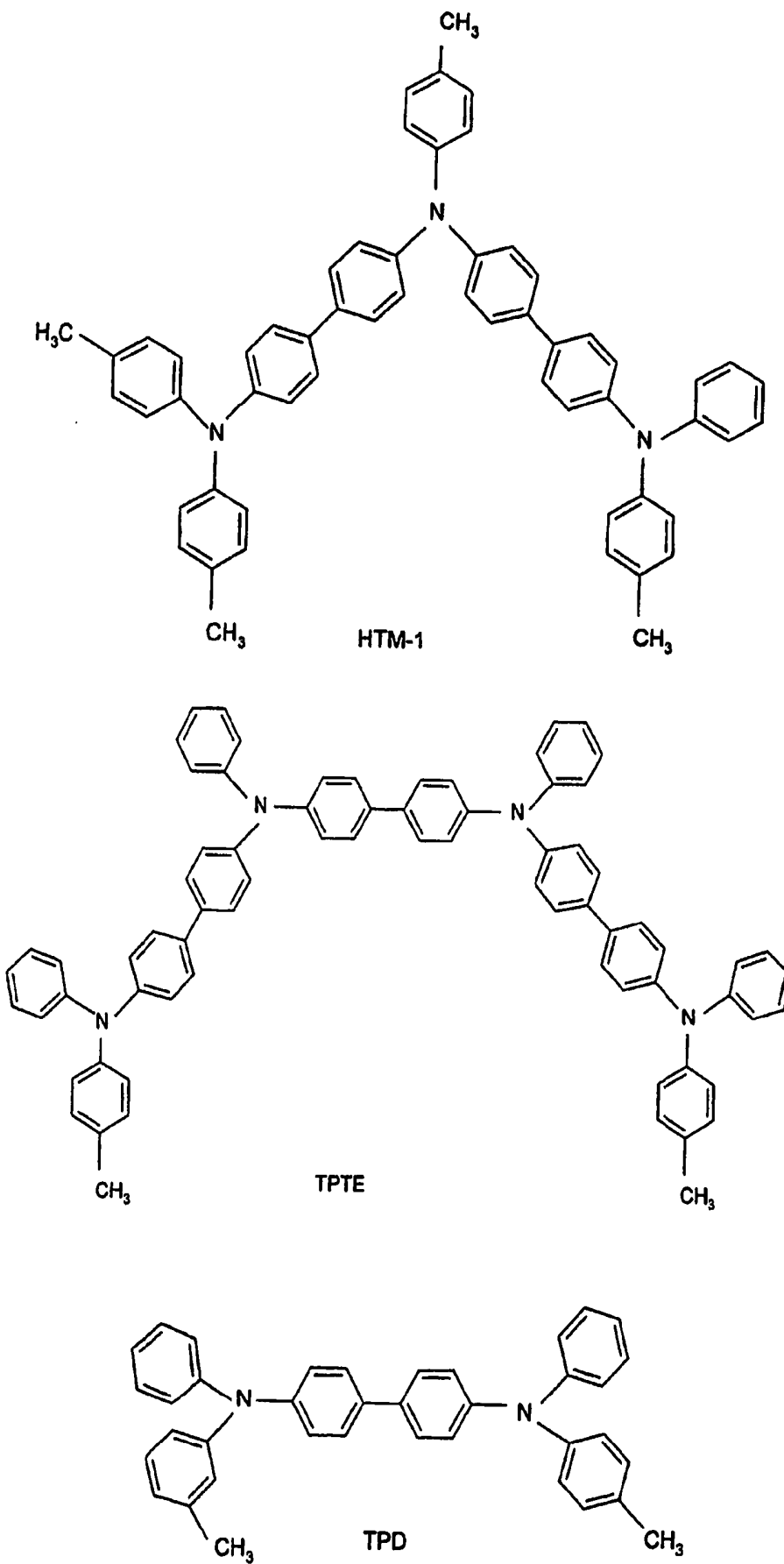


Fig. 2

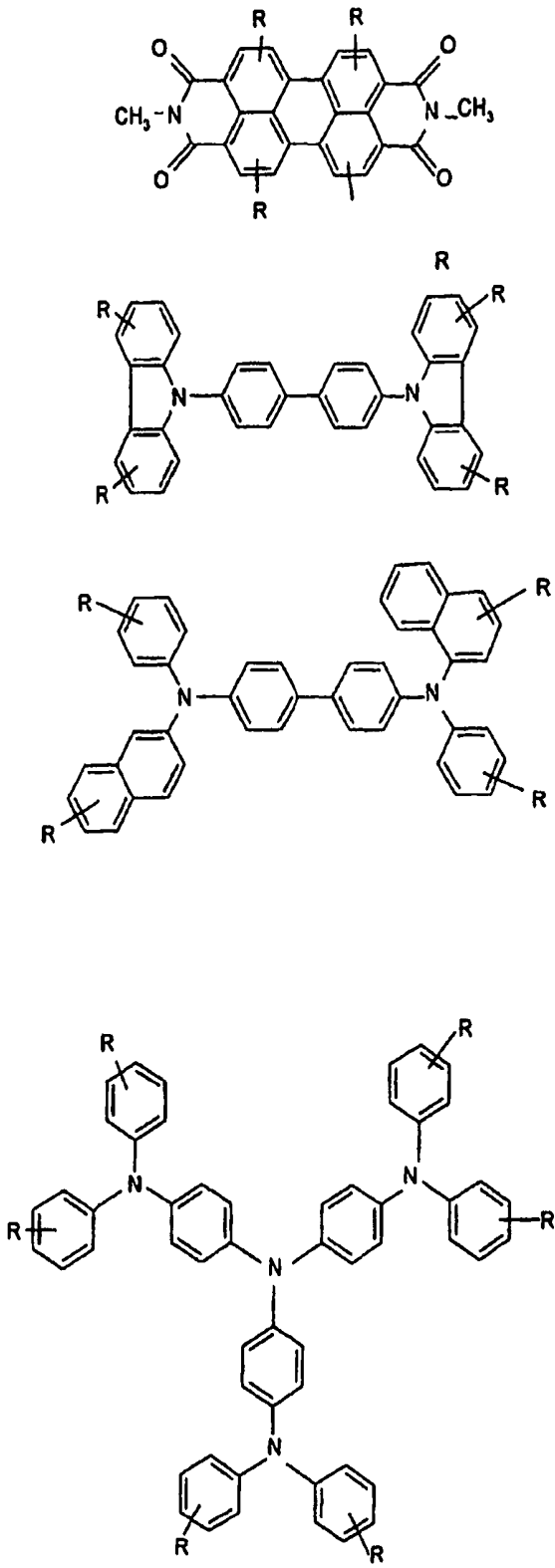


Fig. 3

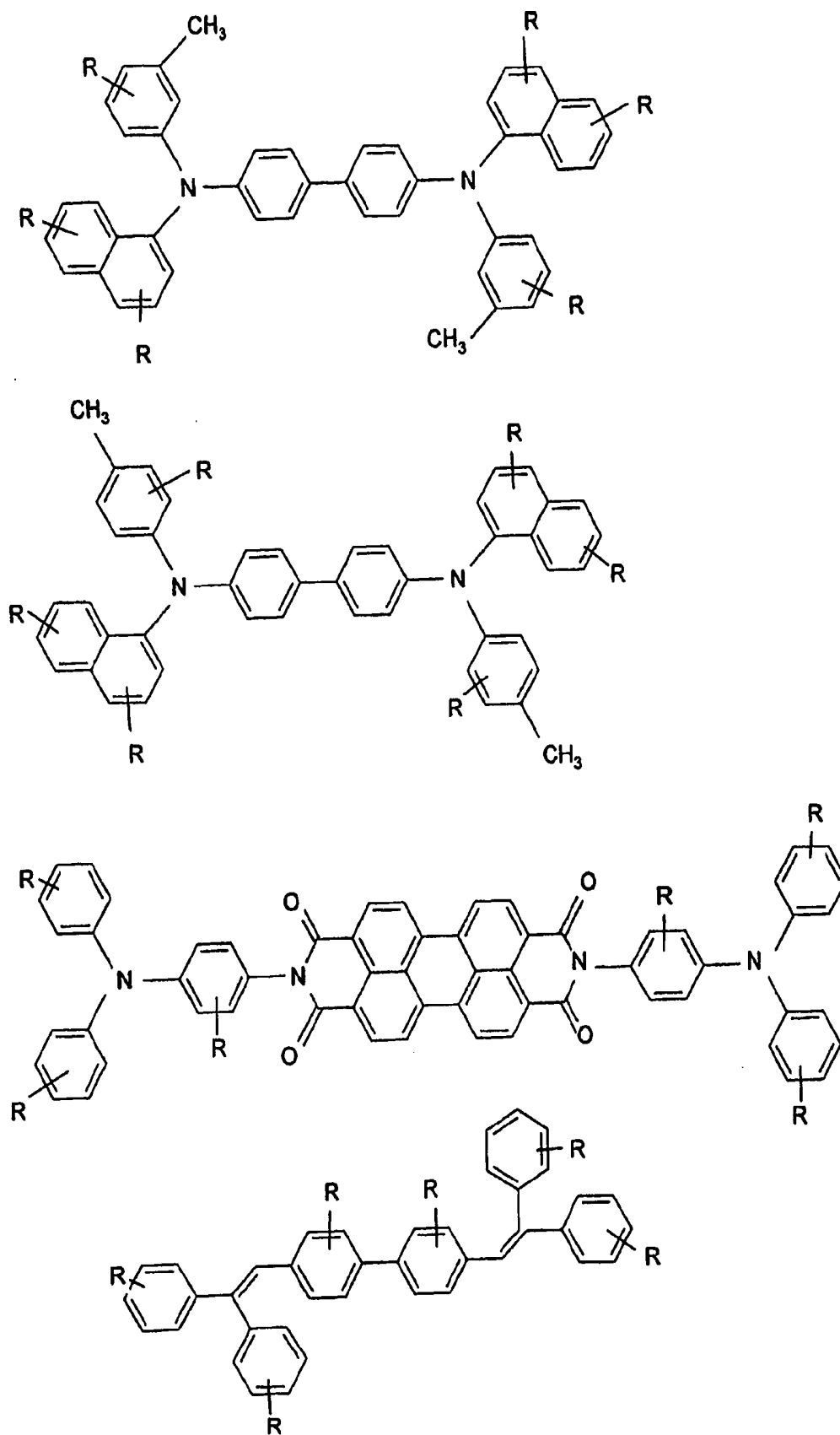
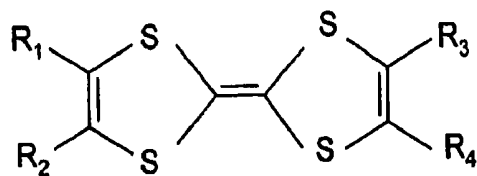
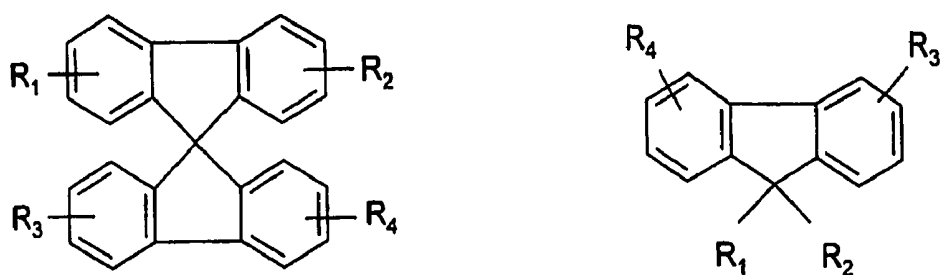


Fig. 4



or

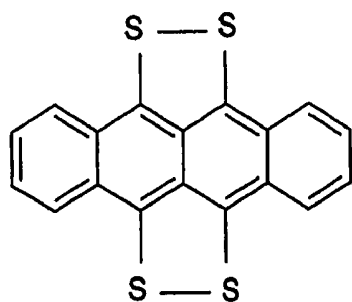
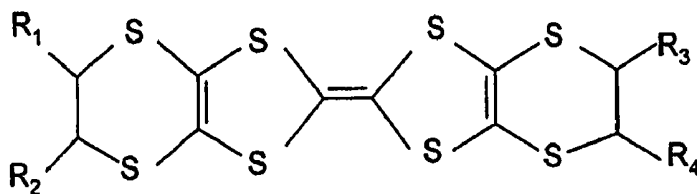
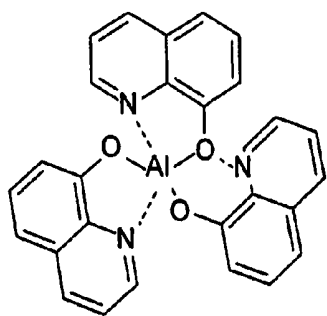
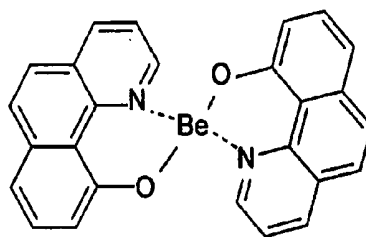


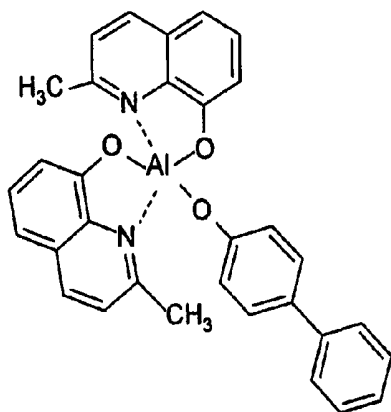
Fig. 5



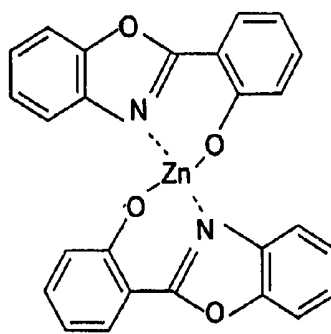
Alq



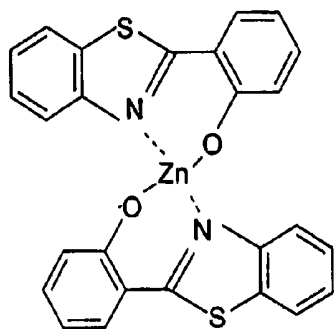
Bebq



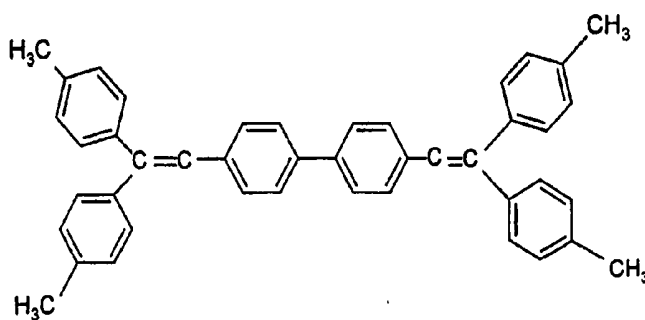
BAlq1



ZnPBO



ZnPBT



DTVb1

Fig. 6

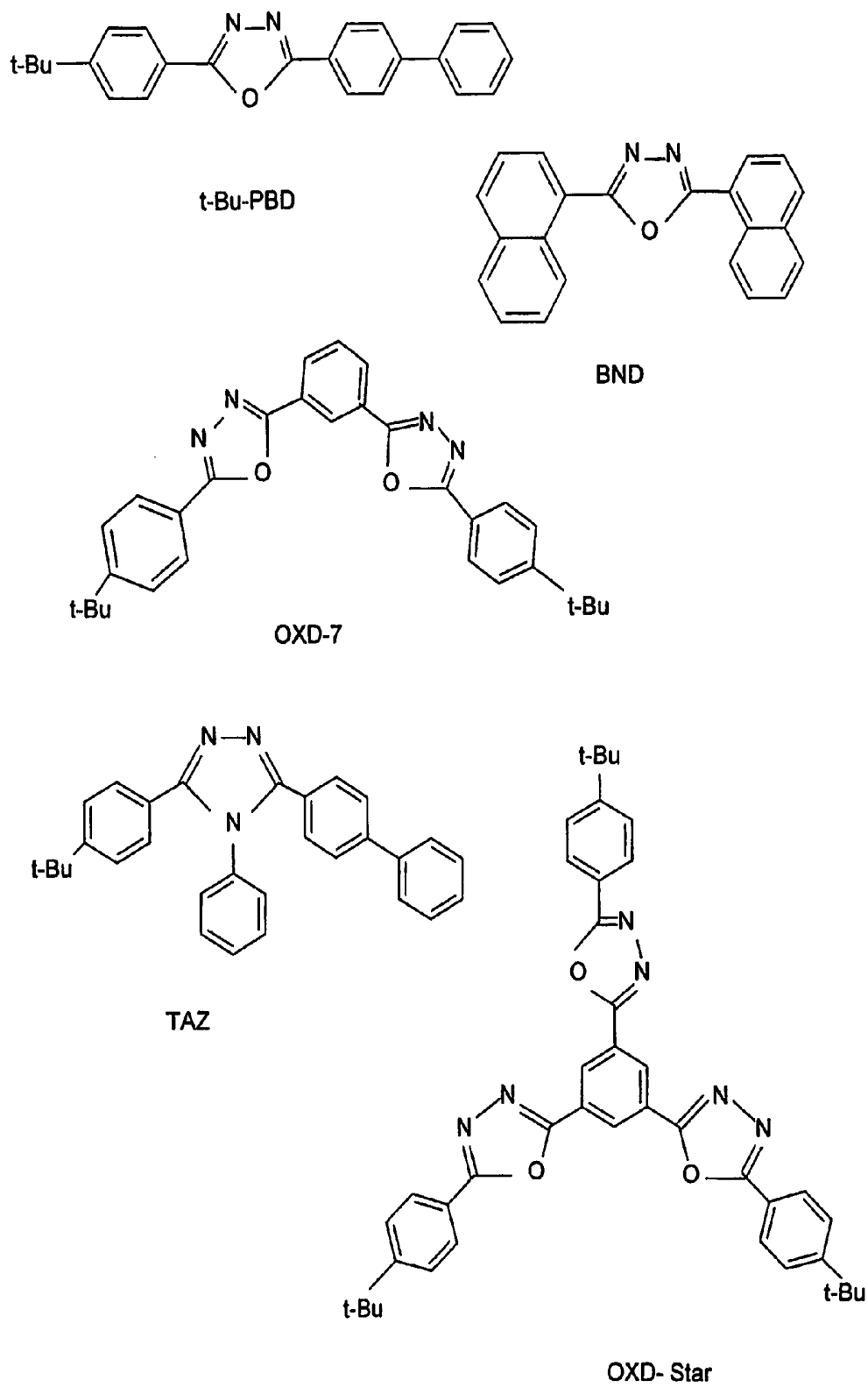


Fig. 7

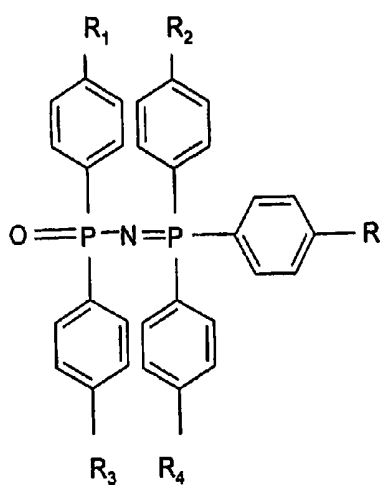


Fig. 8

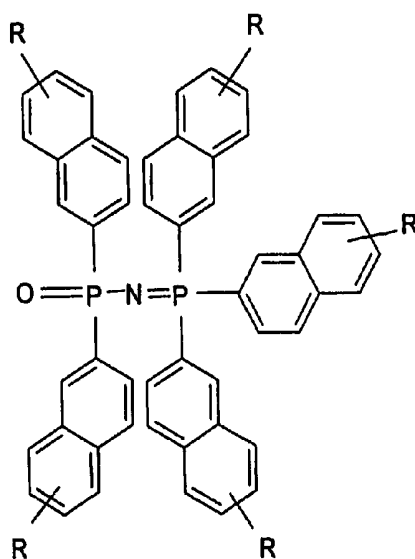


Fig. 9b

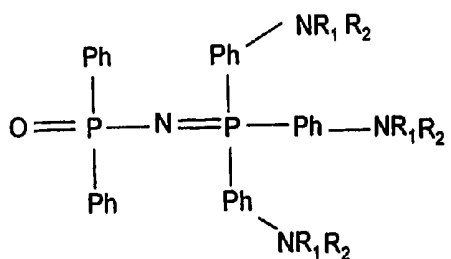


Fig. 9a

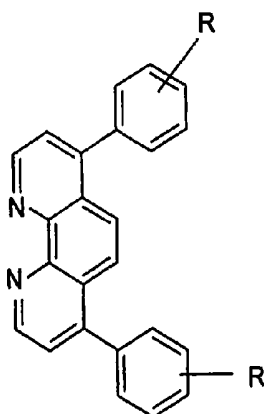


Fig. 10

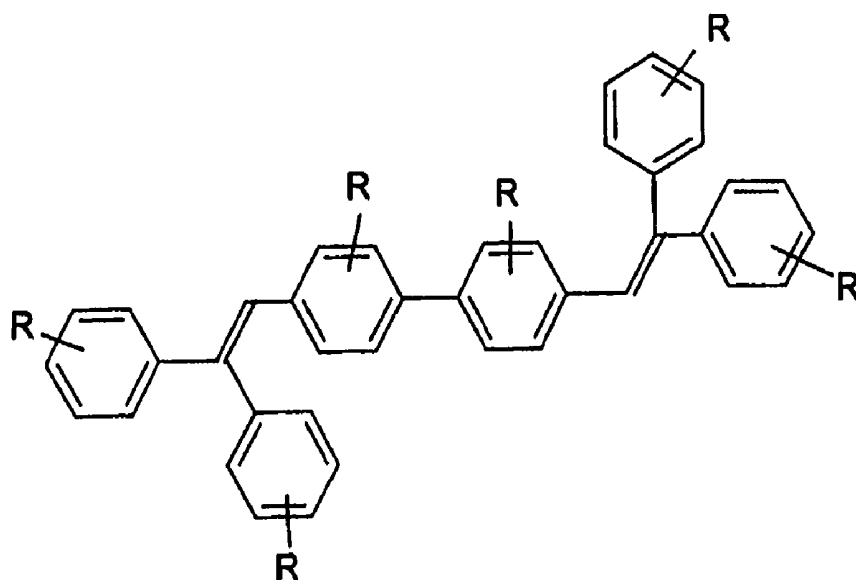
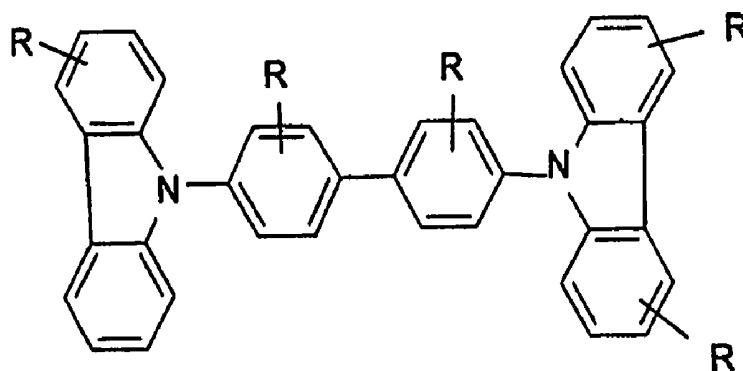
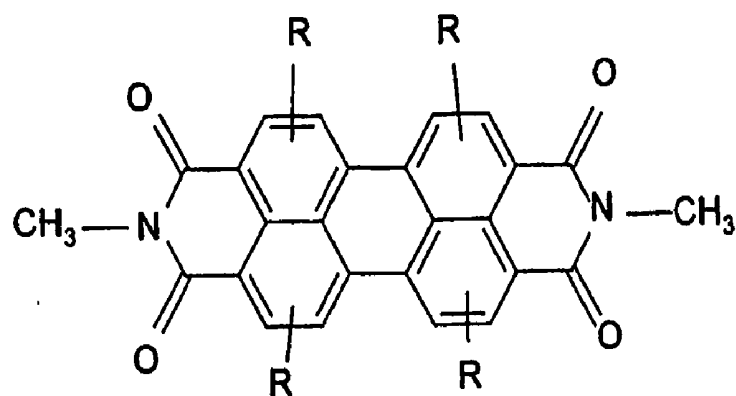


Fig. 11a

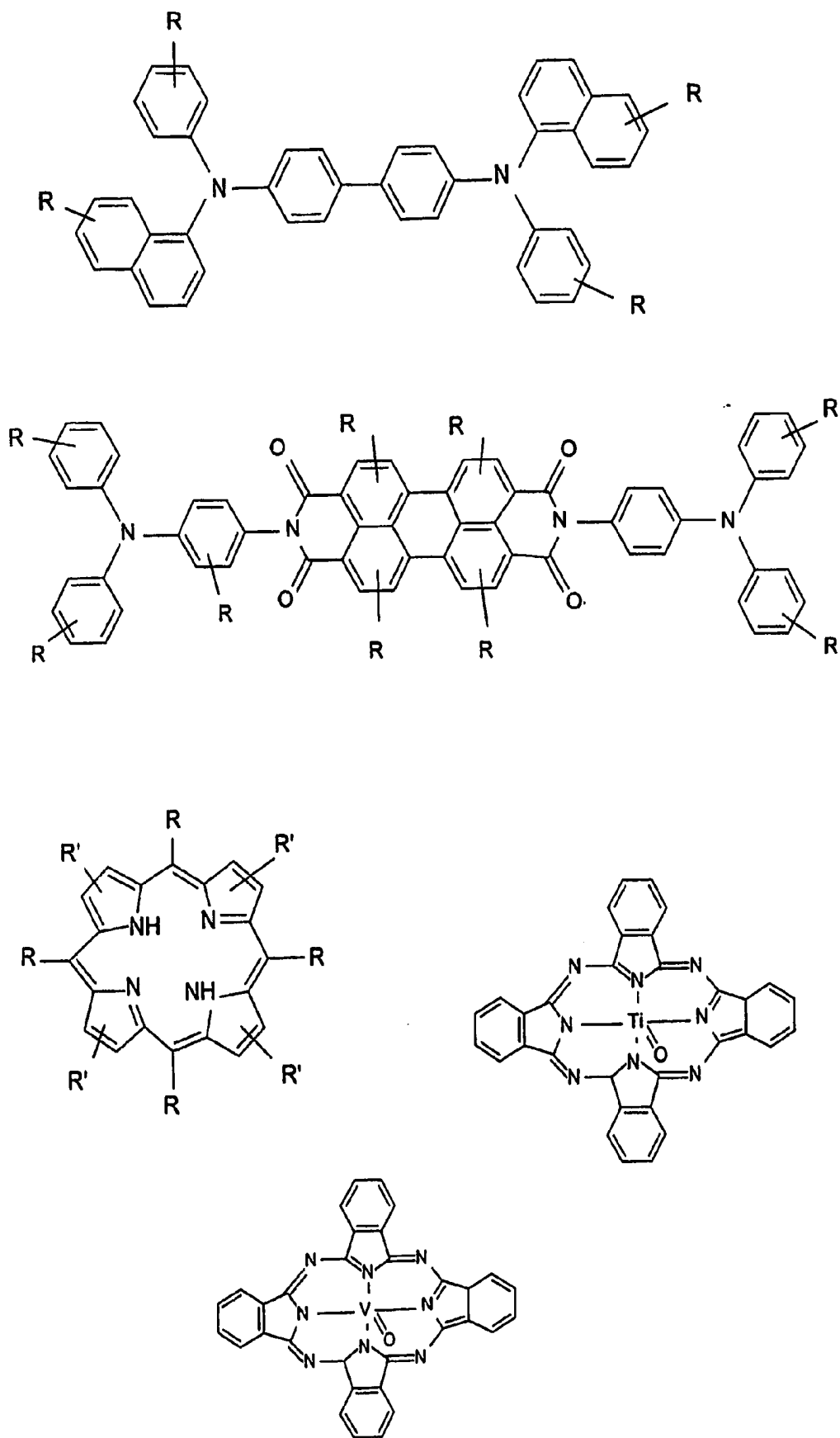


Fig. 11b

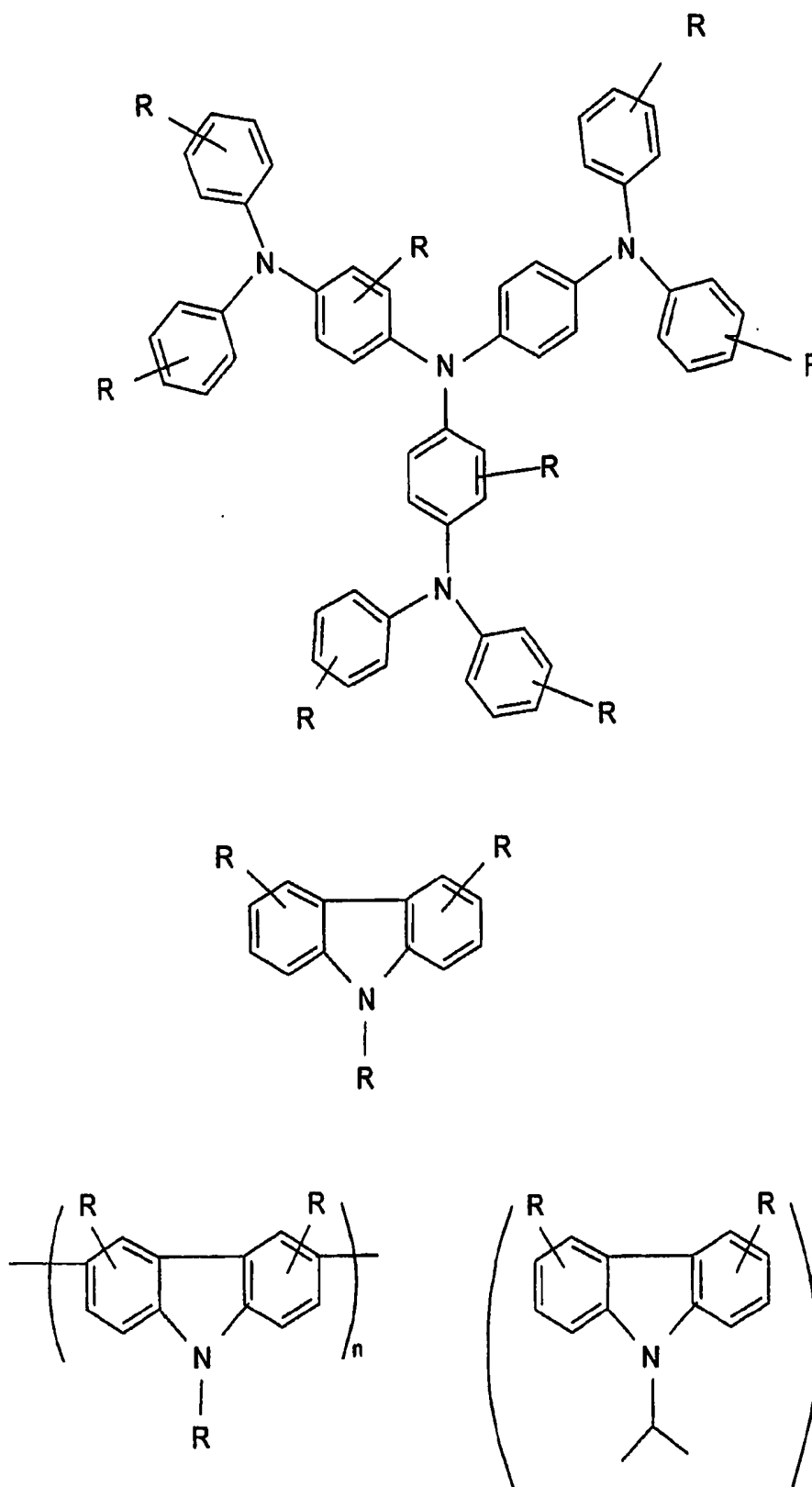


Fig. 11c

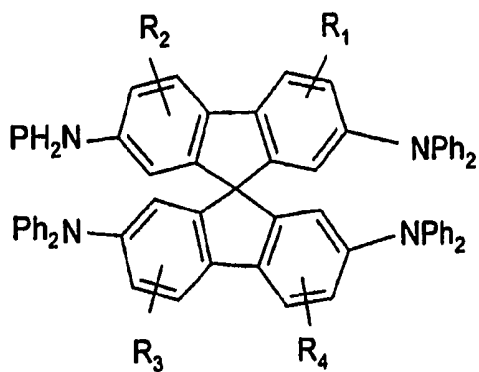
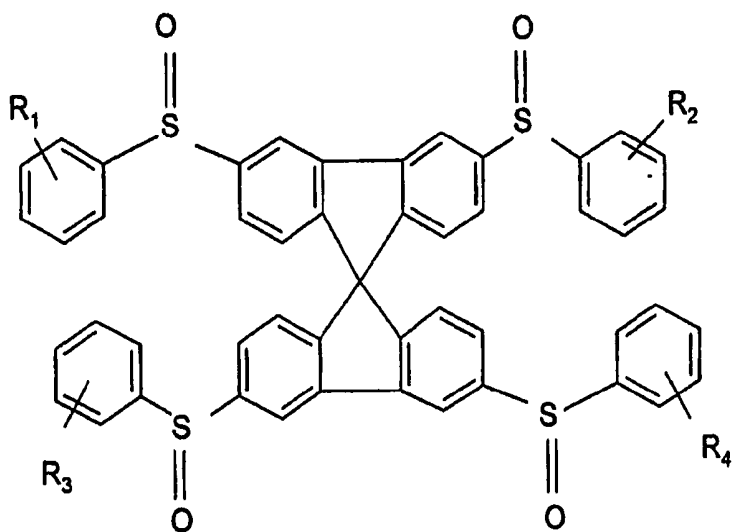
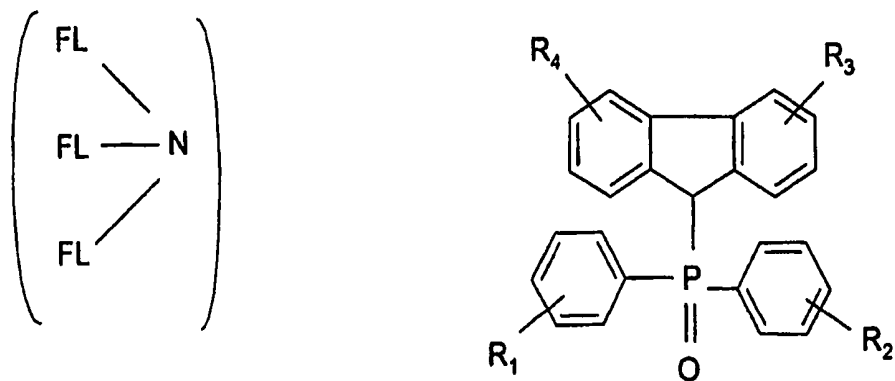


Fig. 12a

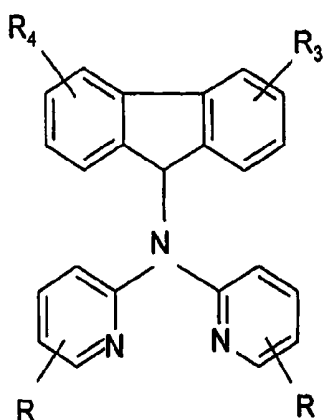


Fig. 12b

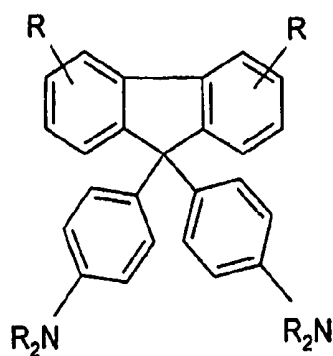


Fig. 12c

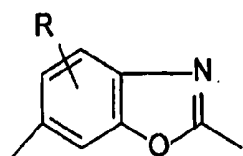


Fig. 13a

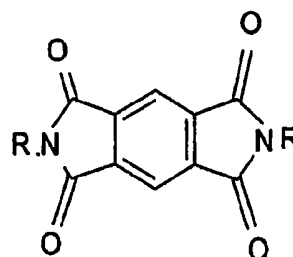


Fig. 13b

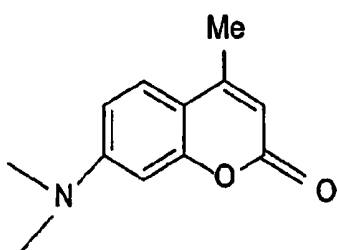


Fig. 13c

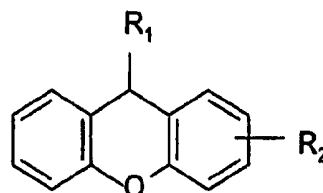


Fig. 13d

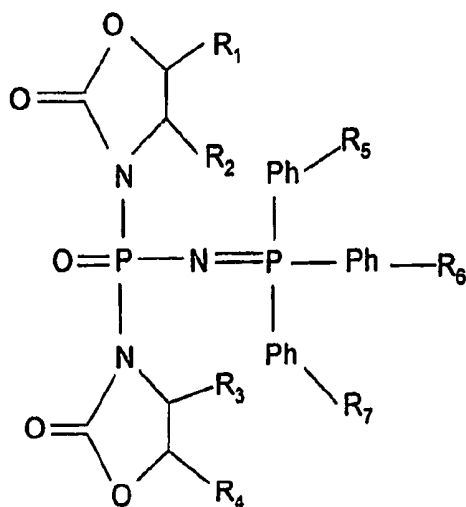


Fig. 13e

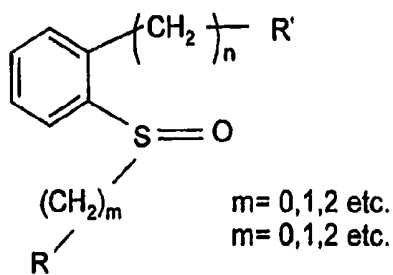
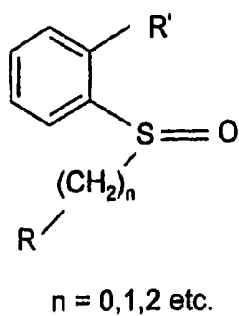
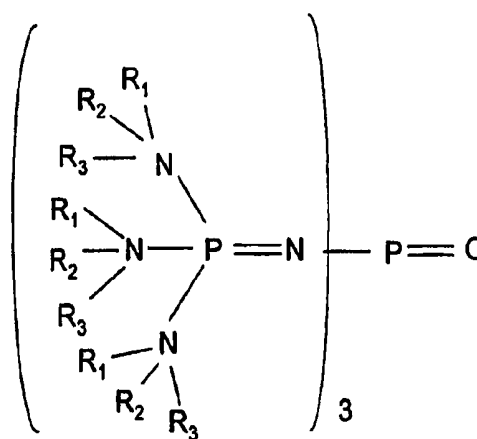
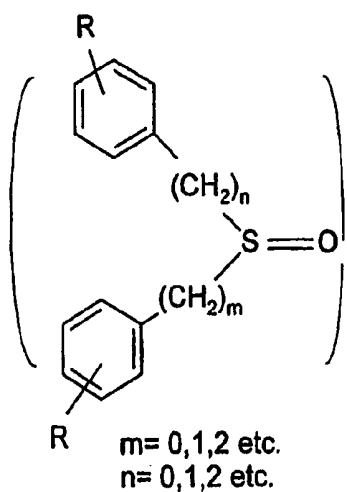
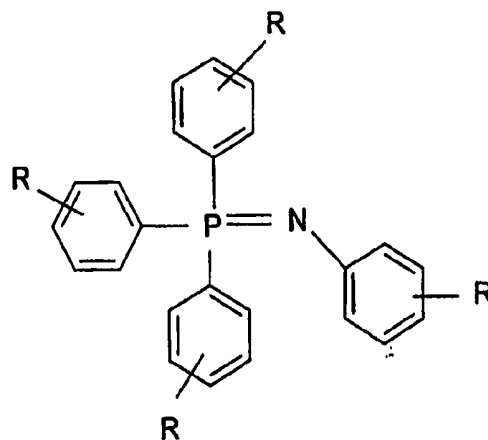
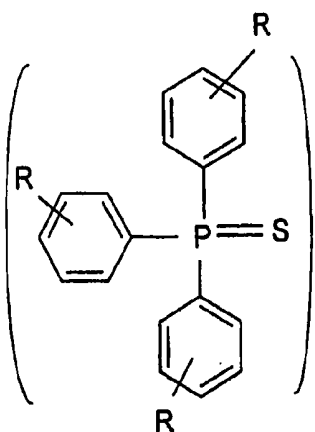


Fig. 14

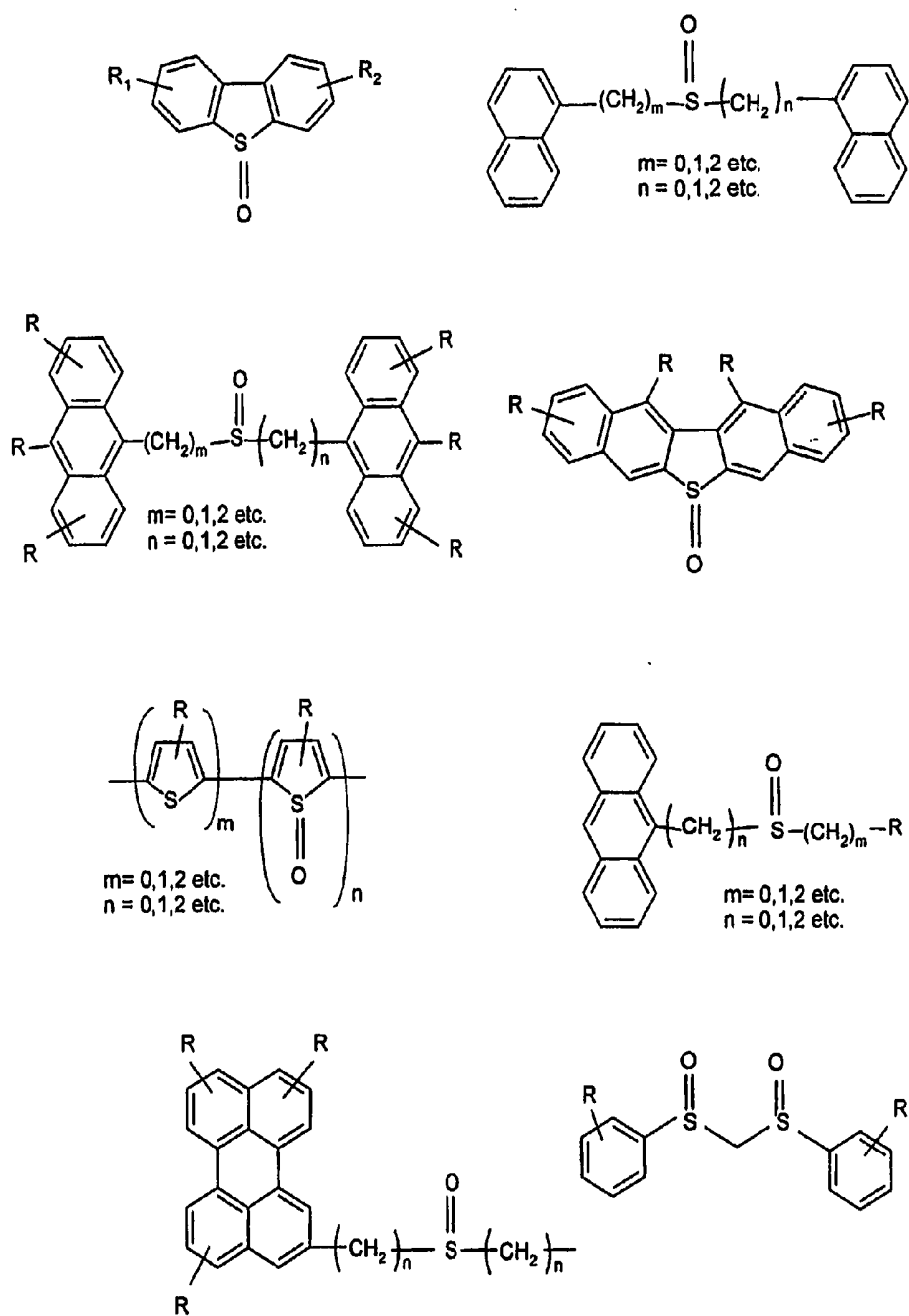


Fig. 15

ITO (H) α -NPB (75 nm)/Tb(pyr)₃OPNP (50 nm)/BCP (20 nm)/Alq₃ (40 nm)/LiF (0.5 nm)/Al

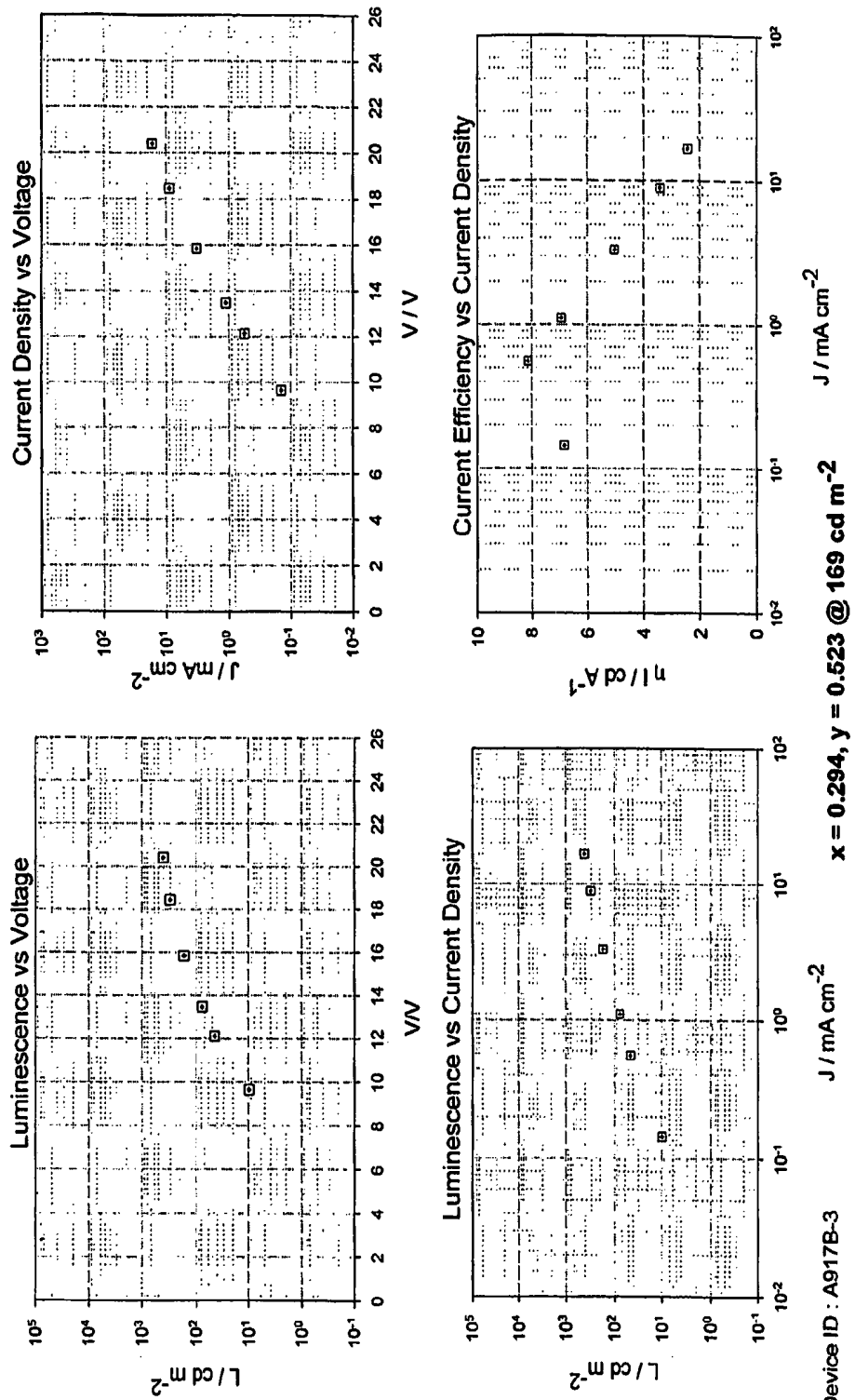
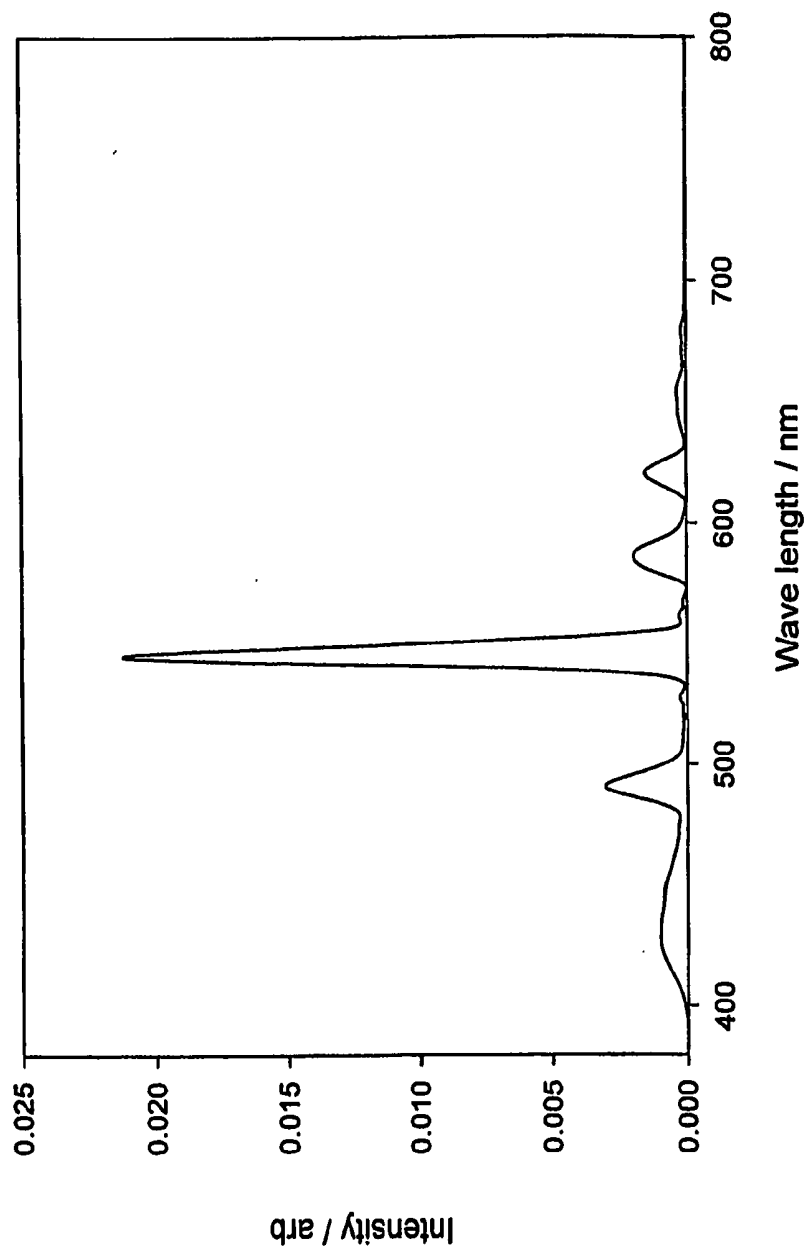


Fig. 16

ITO (H)/ α -NPB (75 nm)/Tb(pyr)₃OPNP (50 nm)/BCP (20 nm)/Alq₃ (40 nm)/LIF (0.5 nm)/Al



x = 0.294, y = 0.523 @ 169 cd m⁻²

Fig. 17

ITO (H) α -NPB (10 nm)/Tb(pyr)₃OPNP (50 nm)/BCP (20 nm)/Alq₃ (40 nm)/LiF (0.7 nm)/Al

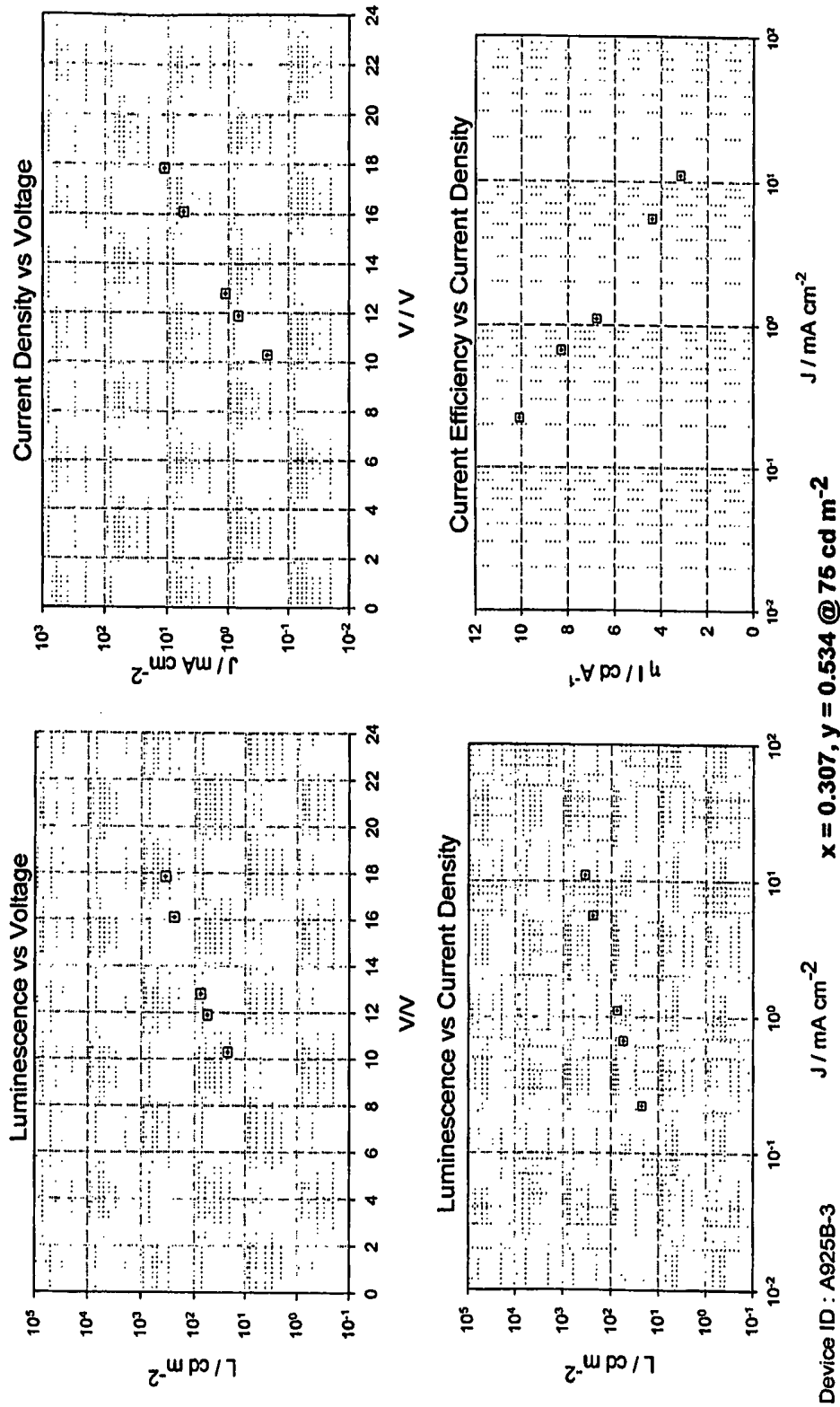
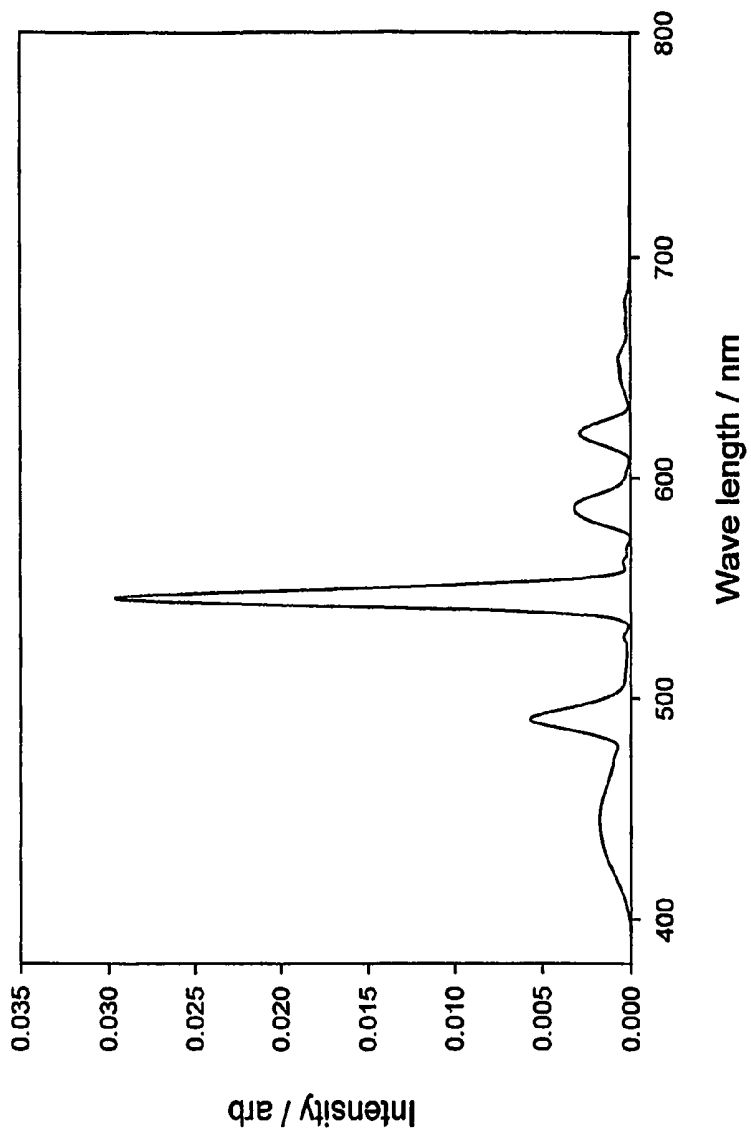


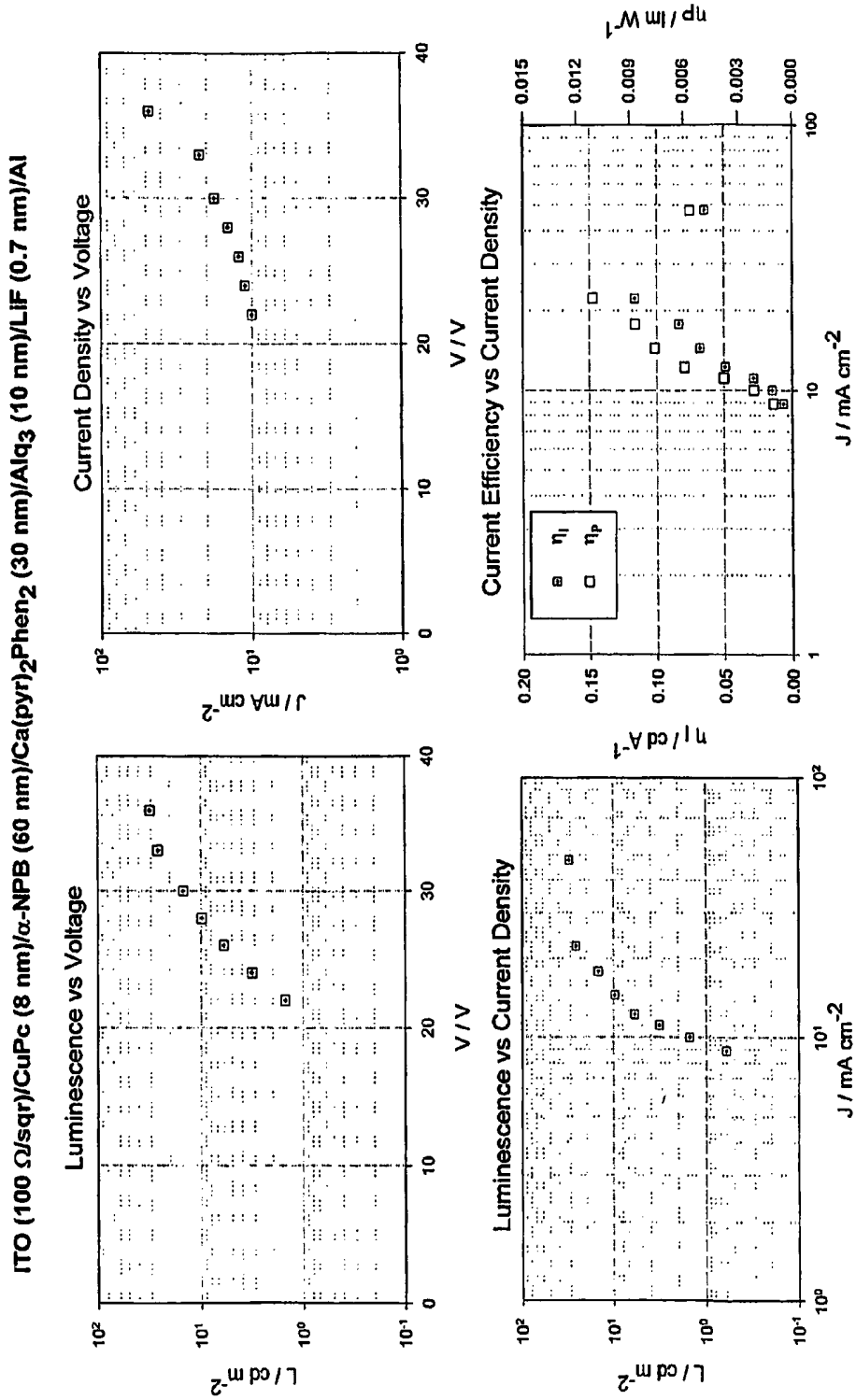
Fig. 18

ITO (H)/ α -NPB (10 nm)/Tb(pyr)₃OPNP (50 nm)/BCP (20 nm)/Alq₃ (40 nm)/LiF (0.7 nm)/Al



$x = 0.307, y = 0.534 @ 75 \text{ cd m}^{-2}$

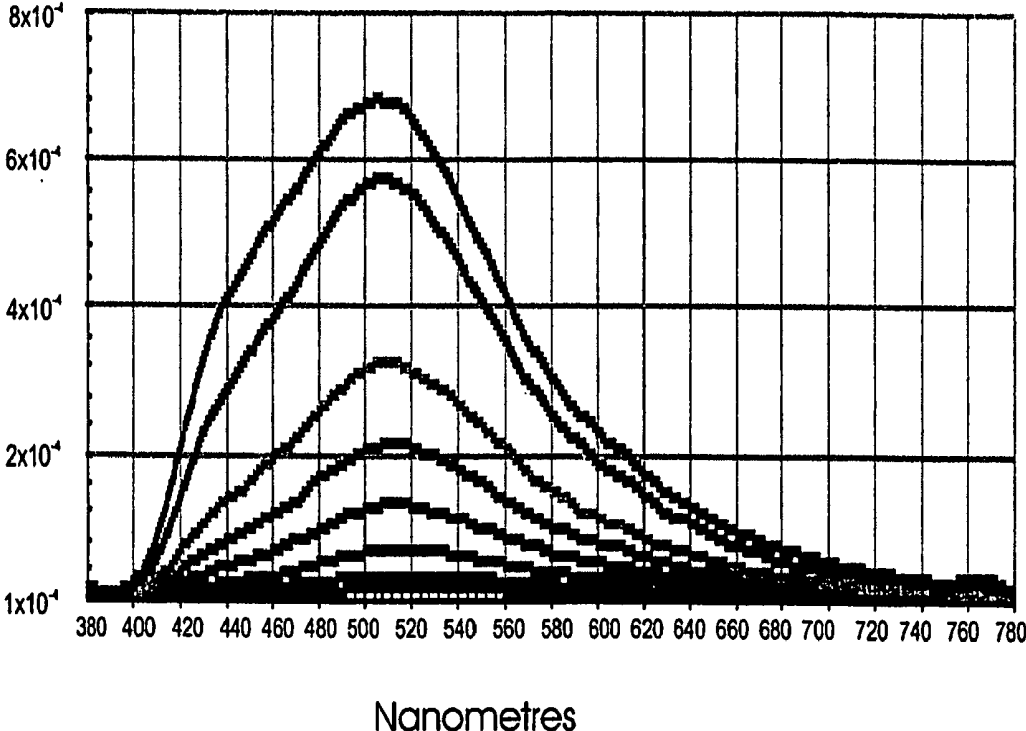
Fig. 19



Device ID: F1H

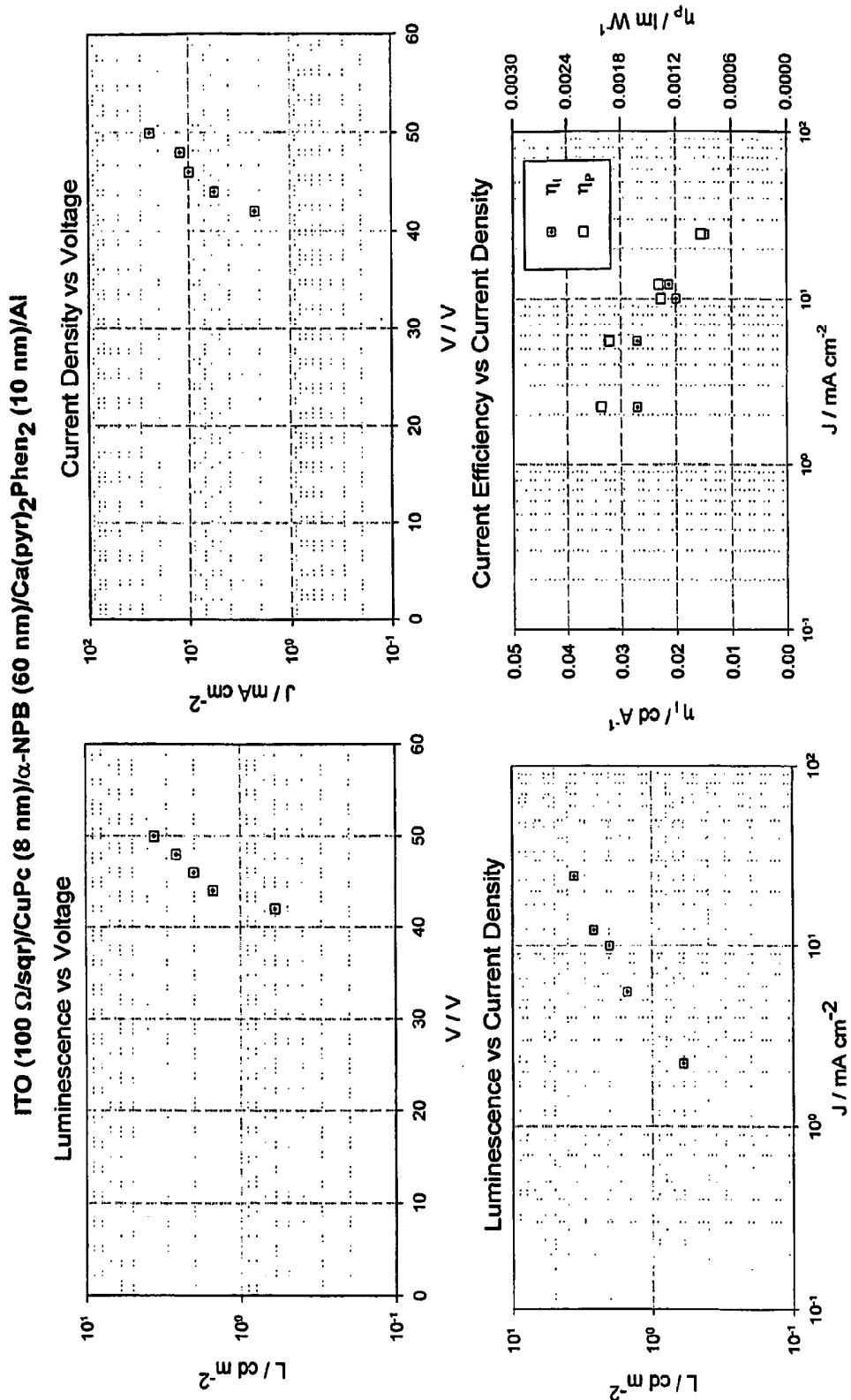
Fig. 20

Spectral radiance peak @508nm



Device 3

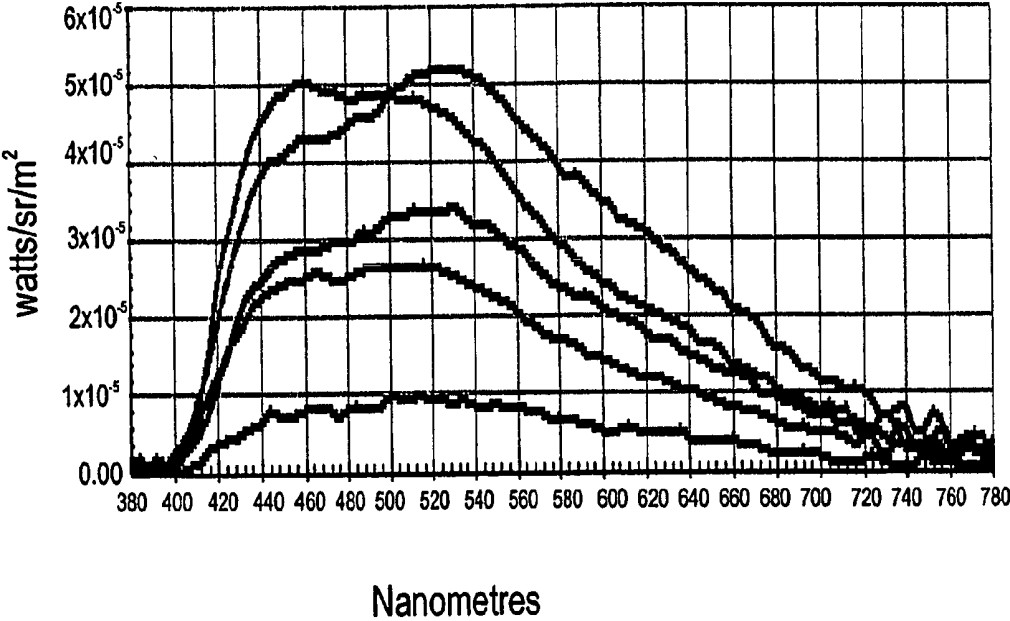
Fig. 21



Device ID: F1V

Fig. 22

Spectral radiance Peak @ 536nm



Device 4

Fig. 23

ELECTROLUMINESCENT MATERIALS AND DEVICES

[0001] The present invention relates to electroluminescent materials and devices incorporating electroluminescent materials.

[0002] Materials which emit light when an electric current is passed through them are well known and used in a wide range of display applications. Liquid crystal devices and devices which are based on inorganic semiconductor systems are widely used; however these suffer from the disadvantages of high energy consumption, high cost of manufacture, low quantum efficiency and the inability to make flat panel displays.

[0003] Organic polymers have been proposed as useful in electroluminescent devices, but it is not possible to obtain pure colours; they are expensive to make and have a relatively low efficiency.

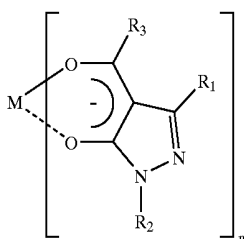
[0004] Another compound which has been proposed is aluminium quinolate, but this requires dopants to be used to obtain a range of colours and has a relatively low efficiency.

[0005] Patent application WO98/58037 describes a range of lanthanide complexes which can be used in electroluminescent devices which have improved properties and give better results. Patent Applications PCT/GB98/01773, PCT/GB99/03619, PCT/GB99/04030, PCT/GB99/04024, PCT/GB99/04028, PCT/GB00/00268 describe electroluminescent complexes, structures and devices using rare earth chelates.

[0006] Hitherto electroluminescent metal complexes have been based on a rare earth, transition metal, lanthanide or an actinide or have been quinolates such as aluminium quinolate.

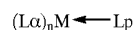
[0007] We have now invented electroluminescent materials which do not include a rare earth, transition metal, lanthanide or an actinide.

[0008] According to the invention there is provided an electroluminescent compound which has the formula

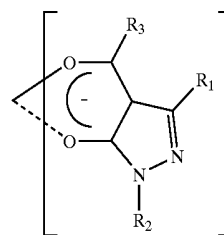


where M is a metal other than aluminium; n is the valency of M; R₁, R₂ and R₃ which may be the same or different are selected from hydrogen, hydrocarbyl groups, substituted and unsubstituted aliphatic groups substituted and unsubstituted aromatic, heterocyclic and polycyclic ring structures, fluorocarbons such as trifluoromethyl groups, halogens such as fluorine or thiophenyl groups or nitrile; R₁ and R₃ can also be form ring structures and R₁, R₂ and R₃ can be copolymerisable with a monomer, e.g. styrene.

[0009] The compounds of formula (I) can be coordinated with a neutral ligand such as L_p To form a complex



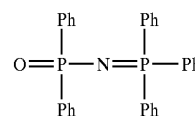
where L α is of formula



(II)

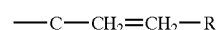
where M is a metal, n is the valency of M and L_p is a neutral ligand.

[0010] The groups L_p can be selected from



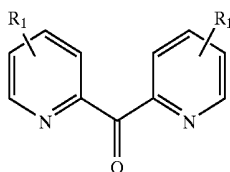
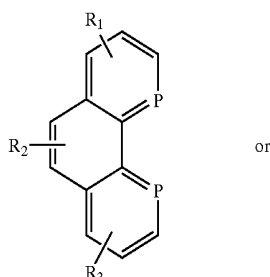
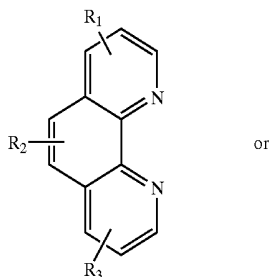
(III)

[0011] Where each Ph which can be the same or different and can be a phenyl (OPNP) or a substituted phenyl group, other substituted or unsubstituted aromatic group, a substituted or unsubstituted heterocyclic or polycyclic group, a substituted or unsubstituted fused aromatic group such as a naphthyl, anthracene, phenanthrene or pyrene group. The substituents can be for example an alkyl, aralkyl, alkoxy, aromatic, heterocyclic, polycyclic group, halogen such as fluorine, cyano, amino. Substituted amino etc. Examples are given in FIGS. 8 and 9 of the drawings where R, R₁, R₂, R₃ and R₄ can be the same or different and are selected from hydrogen, hydrocarbyl groups, substituted and unsubstituted aromatic, heterocyclic and polycyclic ring structures, fluorocarbons such as trifluoromethyl groups, halogens such as fluorine or thiophenyl groups; R, R₁, R₂, R₃ and R₄ can also form substituted and unsubstituted fused aromatic, heterocyclic and polycyclic ring structures and can be copolymerisable with a monomer e.g. styrene. R, R₁, R₂, R₃ and R₄ can also be unsaturated alkylene groups such as vinyl groups or groups

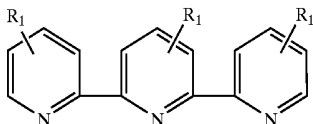
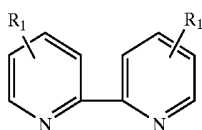


where R is as above.

[0012] L_p can also be compounds of formulae

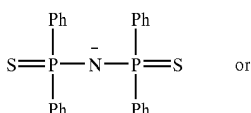


where R_1 , R_2 and R_3 are as referred to above, for example bathophen shown in FIG. 10 of the drawings in which R is as above or

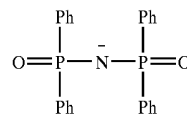


where R_1 , R_2 and R_3 are as referred to above.

[0013] L_p can also be



-continued



where Ph is as above.

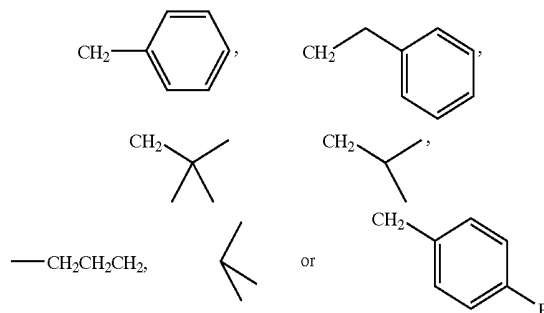
[0014] Other examples of L_p chelates are as shown in FIG. 11 and fluorene and fluorene derivatives e.g. a shown in FIG. 12 and compounds of formulae as shown as shown in FIGS. 13 to 15.

(V) [0015] The invention also provides an electroluminescent device comprising (i) a first electrode, (ii) an electroluminescent layer comprising a layer of a complex of formula (I) and (iii) a second electrode.

[0016] Examples of R_1 and/or R_2 and/or R_3 include aliphatic, aromatic and heterocyclic alkoxy, aryloxy and carboxy groups, substituted and substituted phenyl, fluorophenyl, biphenyl, phenanthrene, anthracene, naphthyl and fluorene groups alkyl groups such as t-butyl, heterocyclic groups such as carbazole.

(VI) [0017] Preferably R_1 and R_2 are Ph_1 and Ph_2 and at least one of Ph_1 and Ph_2 is a substituted or unsubstituted aromatic compound and the other Ph moiety is selected from hydrogen, and substituted and unsubstituted hydrocarbyl groups such as substituted and unsubstituted aliphatic groups, substituted and unsubstituted aromatic, heterocyclic and polycyclic ring structures, fluorocarbons such as trifluoromethyl groups, halogens such as fluorine; substituted and unsubstituted fused aromatic, heterocyclic and polycyclic ring structures and can be copolymerisable with a monomer, e.g. styrene, fluorocarbons such as trifluoromethyl groups, halogens such as fluorine. Examples include aliphatic, aromatic and heterocyclic alkoxy, aryloxy and carboxy groups, substituted and substituted phenyl, fluorophenyl, biphenyl, phenanthrene, anthracene, naphthyl and fluorene groups alkyl groups such as t-butyl, heterocyclic groups such as carbazole.

[0018] Preferably R_1 is methyl and R_2 is



(IX)

[0019] Preferred metals are metals other than aluminium, e.g. gallium, indium, germanium, tin (II), tin (IV), antimony (II), antimony (IV), lead (II), lead (IV) and metals of the first, second and third groups of transition metals in different valence states e.g. manganese, iron, ruthenium, osmium,

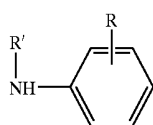
cobalt, nickel, palladium(II), palladium(IV), platinum(II), platinum(IV), cadmium, chromium, titanium, vanadium, zirconium, tantalum, molybdenum, rhodium, iridium, titanium, niobium, scandium, yttrium, and R₃ is preferably a phenyl or substituted phenyl group.

[0020] Preferably there is a hole transmitting layer deposited on the transparent substrate and the electroluminescent material is deposited on the hole transmitting layer. The hole transmitting layer serves to transport holes and to block the electrons, thus preventing electrons from moving into the electrode without recombining with holes. The recombination of carriers therefore mainly takes place in the emitter layer.

[0021] Hole transmitting layers are used in small molecule based polymer electroluminescent devices and in electroluminescent devices based on rare earth metal complexes and any of the known hole transmitting materials in film form can be used.

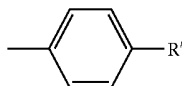
[0022] Hole transmitting layers are used in polymer electroluminescent devices and any of the known hole transmitting materials in film form can be used.

[0023] The hole transmitting layer can be made of a film of an aromatic amine complex such as poly(vinylcarbazole), N,N'-diphenyl-N,N'-bis(3-methylphenyl)-1,1'-biphenyl-4,4'-diamine (TPD), an unsubstituted or substituted polymer of an amino substituted aromatic compound, a polyaniline, substituted polyanilines, polythiophenes, substituted polythiophenes, polysilanes etc. Examples of polyanilines are polymers of



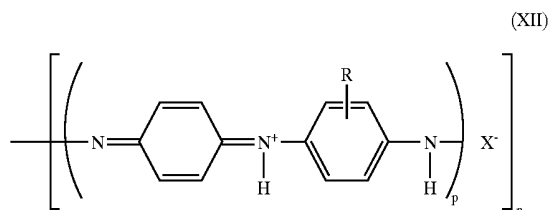
(XI)

where R is in the ortho—or meta-position and is hydrogen, C1-18 alkyl, C1-6 alkoxy, amino, chloro, bromo, hydroxy or the group



where R is alkyl or aryl and R' is hydrogen, C1-6 alkyl or aryl with at least one other monomer of formula I above.

[0024] Polyanilines which can be used in the present invention have the general formula



where p is from 1 to 10 and n is from 1 to 20, R is as defined above and X is an anion, preferably selected from Cl, Br, SO₄, BF₄, PF₆, H₂PO₃, H₂PO₄, arylsulphonate, arenedicarboxylate, polystyrenesulphonate, polyacrylate alkylsulphonate, vinylsulphonate, vinylbenzene sulphonate, cellulose-sulphonate, camphor sulphonates, cellulose sulphate or a perfluorinated polyanion.

[0025] Examples of arylsulphonates are p-toluenesulphonate, benzenesulphonate, 9,10-anthraquinone-sulphonate and antiracenesulphonate, an example of an arenedicarboxylate is phthalate and an example of arenecarboxylate is benzoate.

[0026] We have found that protonated polymers of the unsubstituted or substituted polymer of an amino substituted aromatic compound such as a polyaniline are difficult to evaporate or cannot be evaporated; however we have surprisingly found that if the unsubstituted or substituted polymer of an amino substituted aromatic compound is deprotonated it can be easily evaporated i.e. the polymer is evaporable.

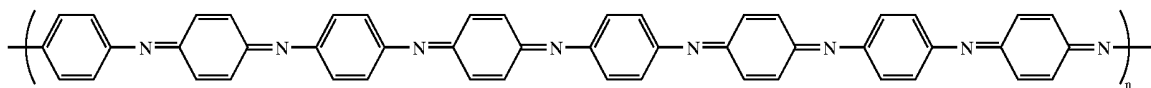
[0027] Preferably evaporable de-protonated polymers of unsubstituted or substituted polymer of an amino substituted aromatic compound are used. The de-protonated unsubstituted or substituted polymer of an amino substituted aromatic compound can be formed by deprotonating the polymer by treatment with an alkali such as ammonium hydroxide or an alkali metal hydroxide such as sodium hydroxide or potassium hydroxide.

[0028] The degree of protonation can be controlled by forming a protonated polyaniline and de-protonating. Methods of preparing polyanilines are described in the article by A. G. MacDiarmid and A. F. Epstein, Faraday Discussions, Chem Soc. 88 P319 1989.

[0029] The conductivity of the polyaniline is dependent on the degree of protonation with the maximum conductivity being when the degree of protonation is between 40 and 60%, e.g. about 50%.

[0030] Preferably the polymer is substantially fully deprotonated.

[0031] A polyaniline can be formed of octamer units i.e. p is four, e.g.



[0032] The polyanilines can have conductivities of the order of 1×10^{-1} Siemen cm^{-1} or higher.

[0033] The aromatic rings can be unsubstituted or substituted, e.g. by a C1 to 20 alkyl group such as ethyl.

[0034] The polyaniline can be a copolymer of aniline and preferred copolymers are the copolymers of aniline with o-anisidine, m-sulphanilic acid or o-aminophenol, or o-toluidine with o-aminophenol, o-ethylaniline, o-phenylene diamine or with amino anthracenes.

[0035] Other polymers of an amino substituted aromatic compound which can be used include substituted or unsubstituted polyaminonaphthalenes, polyaminoanthracenes, polyaminophenanthrenes, etc. and polymers of any other condensed polyaromatic compound. Polyaminoanthracenes and methods of making them are disclosed in U.S. Pat. No. 6,153,726. The aromatic rings can be unsubstituted or substituted, e.g. by a group R as defined above.

[0036] The polyanilines can be deposited on the first electrode by conventional methods, e.g. by vacuum evaporation, spin coating, chemical deposition, direct electrodeposition etc. Preferably the thickness of the polyaniline layer is such that the layer is conductive and transparent and is preferably from 20 nm to 200 nm. The polyanilines can be doped or undoped. When they are doped they can be dissolved in a solvent and deposited as a film, when they are undoped they are solids and can be deposited by vacuum evaporation i.e. by sublimation.

[0037] The structural formulae of some other hole transmitting materials are shown in FIGS. 1, 2, 3, 4 and 5 of the drawings, where R, R₁, R₂ and R₃ can be the same or different and are selected from hydrogen, and substituted and unsubstituted hydrocarbyl groups such as substituted and unsubstituted aliphatic groups, substituted and unsubstituted aromatic, heterocyclic and polycyclic ring structures, fluorocarbons such as trifluoryl methyl groups, halogens such as fluorine or thiophenyl groups; R₁, R₂ and R₃ can also form substituted and unsubstituted fused aromatic, heterocyclic and polycyclic ring structures and can be copolymerisable with a monomer, e.g. styrene. X is Se, S or O, Y can be hydrogen, substituted or unsubstituted hydrocarbyl groups, such as substituted and unsubstituted aromatic, heterocyclic and polycyclic ring structures, fluorine, fluorocarbons such as trifluoryl methyl groups, halogens such as fluorine or thiophenyl groups or nitrile.

[0038] Examples of R₁ and/or R₂ and/or R₃ include aliphatic, aromatic and heterocyclic alkoxy, aryloxy and carboxy groups, substituted and substituted phenyl, fluorophenyl, biphenyl, phenanthrene, anthracene, naphthyl and fluorene groups alkyl groups such as t-butyl, heterocyclic groups such as carbazole.

[0039] The hole transporting material can optionally be mixed with the electroluminescent material in a ratio of 5-95% of the electroluminescent material to 95 to 5% of the hole transporting compound.

[0040] Other hole transporting materials which can be used are conjugated polymers.

[0041] U.S. Pat. No. 5,807,627 discloses an electroluminescence device in which there are conjugated polymers in the electroluminescent layer. The conjugated polymers referred to are defined as polymers for which the main chain is either fully conjugated possessing extended pi molecular orbitals along the length of the chain or else is substantially conjugated, but with interruptions to conjugation, either random or regular along the main chain. They can be homopolymers or copolymers.

[0042] The conjugated polymer used can be any of the conjugated polymers disclosed or referred to in U.S. Pat. No. 5,807,627, PCT/WO90/13148 and PCT/WO92/03490.

[0043] The conjugated polymers disclosed are poly(p-phenylenevinylene)-PPV and copolymers including PPV. Other preferred polymers are poly(2,5 dialkoxyphenylene vinylene) such as poly(2-methoxy-5-(2-methoxypropyloxy)-1,4-phenylene vinylene), poly(2-methoxypropyloxy)-1,4-phenylenevinylene), poly(2-methoxy-5-(2-dodecyloxy-1,4-phenylenevinylene) and other poly(2,5 dialkoxyphenylenevinylene) with at least one of the alkoxy groups being a long chain solubilising alkoxy group, poly fluorenes and oligofluorenes, polyphenylenes and oligophenylenes, polyanthracenes and oligo anthracenes, polythiophenes and oligothiophenes.

[0044] In PPV the phenylene ring may optionally carry one or more substituents, e.g. each independently selected from alkyl, preferably methyl, alkoxy, preferably methoxy or ethoxy.

[0045] Any poly(arylenevinylene) including substituted derivatives thereof can be used and the phenylene ring in poly(p-phenylenevinylene) may be replaced by a fused ring system such as anthracene or naphthylene ring and the number of vinylene groups in each polyphenylenevinylene moiety can be increased, e.g. up to 7 or higher.

[0046] The conjugated polymers can be made by the methods disclosed in U.S. Pat. No. 5,807,627, PCT/WO90/13 148 and PCT/WO92/03490.

[0047] The hole transmitting material and the light emitting metal compound can be mixed to form one layer, e.g. in a proportion of 5 to 95% of the hole transmitting material to 95 to 5% of the light emitting metal compound.

[0048] Optionally there is a layer of an electron transmitting material between the cathode and the electroluminescent material layer. The electron transmitting material is a material which will transport electrons when an electric current is passed through it. Electron transmitting materials include a metal complex such as a metal quinolate, e.g. an aluminium quinolate, lithium quinolate, a cyano anthracene such as 9,10 dicyano anthracene, a polystyrene sulphonate and compounds of formulae shown in FIGS. 6 and 7. Instead of being a separate layer the electron transmitting

material can be mixed with the electroluminescent material to form one layer, e.g. in a proportion of 5 to 95% of the electron transmitting material to 95 to 5% of the light emitting metal compound.

[0049] The electroluminescent layer can comprise a mixture of the light emitting metal compound with the hole transmitting material and electron transmitting material.

[0050] The electroluminescent material can be deposited on the substrate directly by vacuum evaporation or evaporation from a solution in an organic solvent. The solvent which is used will depend on the material but chlorinated hydrocarbons such as dichloromethane and n-methyl pyrrolidone; dimethyl sulphoxide; tetra hydrofuran; dimethylformamide etc. are suitable in many cases.

[0051] Alternatively electroluminescent material can be deposited by spin coating from solution, or by vacuum deposition from the solid state, e.g. by sputtering, or any other conventional method can be used.

[0052] Preferably the first electrode is a transparent substrate such as a conductive glass or plastic material which acts as the anode. Preferred substrates are conductive glasses such as indium tin oxide coated glass, but any glass which is conductive or has a transparent conductive layer such as a metal or conductive polymer can be used.

[0053] Conductive polymers and conductive polymer coated glass or plastics materials can also be used as the substrate.

[0054] The second electrode functions as the cathode and can be any low work function metal, e.g. aluminium, calcium, lithium, silver/magnesium alloys etc; aluminium is a preferred metal.

[0055] The display of the invention may be monochromatic or polychromatic. Electroluminescent rare earth chelate compounds are known which will emit a range of colours, e.g. red, green, and blue light and white light and examples are disclosed in Patent Applications WO98/58037 PCT/GB98/01773, PCT/GB99/03619, PCT/GB99/04030, PCT/GB99/04024, PCT/GB99/04028, PCT/GB00/00268 and can be used to form OLEDs emitting those colours. Thus, a full colour display can be formed by arranging three individual backplanes, each emitting a different primary monochrome colour, on different sides of an optical system, from another side of which a combined colour image can be viewed. Alternatively, rare earth chelate electroluminescent compounds emitting different colours can be fabricated so that adjacent diode pixels in groups of three neighbouring pixels produce red, green and blue light. In a further alternative, field sequential colour filters can be fitted to a white light emitting display.

[0056] Either or both electrodes can be formed of silicon and the electroluminescent material and intervening layers of a hole transporting and electron transporting materials can be formed as pixels on the silicon substrate. Preferably each pixel comprises at least one layer of a rare earth chelate electroluminescent material and an (at least semi-) transparent electrode in contact with the organic layer on a side thereof remote from the substrate.

[0057] Preferably, the substrate is of crystalline silicon and the surface of the substrate may be polished or smoothed to produce a flat surface prior to the deposition of electrode, or

electroluminescent compound. Alternatively a non-planarised silicon substrate can be coated with a layer of conducting polymer to provide a smooth, flat surface prior to deposition of further materials.

[0058] In one embodiment, each pixel comprises a metal electrode in contact with the substrate. Depending on the relative work functions of the metal and transparent electrodes, either may serve as the anode with the other constituting the cathode.

[0059] When the silicon substrate is the cathode an indium tin oxide coated glass can act as the anode and light is emitted through the anode. When the silicon substrate acts as the anode, the cathode can be formed of a transparent electrode which has a suitable work function, for example by an indium zinc oxide coated glass in which the indium zinc oxide has a low work function. The anode can have a transparent coating of a metal formed on it to give a suitable work function. These devices are sometimes referred to as top emitting devices or back emitting devices.

[0060] The metal electrode may consist of a plurality of metal layers, for example a higher work function metal such as aluminium deposited on the substrate and a lower work function metal such as calcium deposited on the higher work function metal. In another example, a further layer of conducting polymer lies on top of a stable metal such as aluminium.

[0061] Preferably, the electrode also acts as a mirror behind each pixel and is either deposited on, or sunk into, the planarised surface of the substrate. However, there may alternatively be a light absorbing black layer adjacent to the substrate.

[0062] In still another embodiment, selective regions of a bottom conducting polymer layer are made non-conducting by exposure to a suitable aqueous solution allowing formation of arrays of conducting pixel pads which serve as the bottom contacts of the pixel electrodes.

[0063] As described in WO00/60669 the brightness of light emitted from each pixel is preferably controllable in an analogue manner by adjusting the voltage or current applied by the matrix circuitry or by inputting a digital signal which is converted to an analogue signal in each pixel circuit. The substrate preferably also provides data drivers, data converters and scan drivers for processing information to address the array of pixels so as to create images. When an electroluminescent material is used which emits light of a different colour, depending on the applied voltage, the colour of each pixel can be controlled by the matrix circuitry.

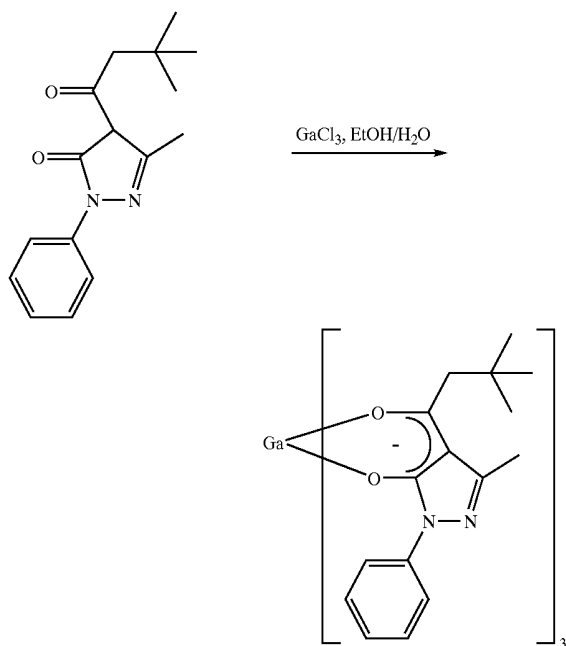
[0064] In one embodiment, each pixel is controlled by a switch comprising a voltage controlled element and a variable resistance element, both of which are conveniently formed by metal-oxide-semiconductor field effect transistors (MOSFETs) or by an active matrix transistor.

[0065] The invention is illustrated in the examples.

EXAMPLE 1

Preparation of Tris-(4-^tButylacetyl-3-methyl-1-phenyl-pyrazol-5-onato)Gallium (Ga(^tBuPz)₃)

[0066]



[0067] 4-^tButylacetyl-3-methyl-1-phenyl-pyrazol-5-one (2.83 g, 10.39 mmol), in a 100 ml round bottom flask, was dissolved in ethanol (~50 mL) with gentle heating. Gallium(III) chloride (0.61 g, 3.46 mmol) was dissolved in H₂O (~10 mL) and added to the pyrazolone solution. The resulting suspension was heated to reflux for 2 hours and then allowed to cool. The resulting suspension was filtered and washed with H₂O (3×10 ml) and EtOH (3×10 ml) then dried under vacuum at 80° C. to give a pink powder with the following analysis.

Ga(^t BuPz) ₃	C	H	N
Theoretical	65.24	6.50	9.51
Found	65.07	6.57	9.46

[0068] Melting point: 252.1° C. (D.S.C.)

[0069] Emission λ_{max}: ~450 nm

[0070] Photoluminescence Efficiency (x,y): 0.001 cdm²μW⁻¹ (0.21, 0.24)

PL Measurement:

[0071] PL spectra was measured by Lot Oriel Multispec Model 77400 CCD Camera.

[0072] The measurement was carried out from the powder by spreading the powder on a spectroil plate.

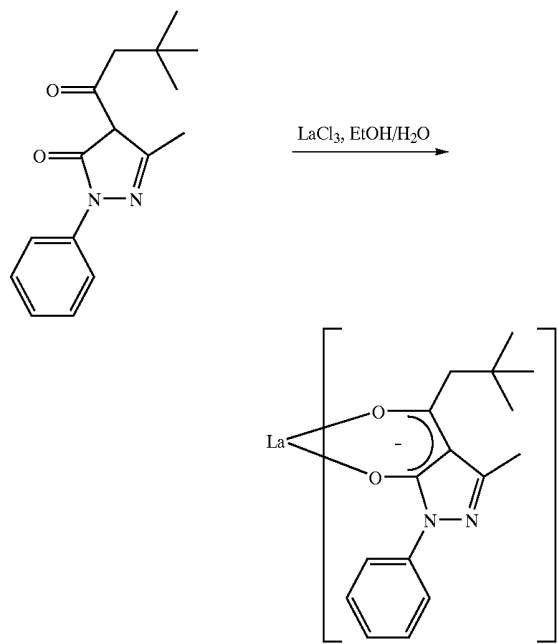
[0073] Reagents Gallium(III) chloride, anhydrous, 99.99%; Aldrich; 45,089-8 4-^tButylacetyl-3-methyl-1-phenyl-pyrazol-5-one as prepared

nyl-pyrazol-5-onato as prepared Ethanol, denatured with 4.8% Methanol; Fluka; 02857

EXAMPLE 2

Preparation of Tris-(4-^tButylacetyl-3-methyl-1-phenyl-pyrazol-5-onato)Lanthanum (La(^tBuPz)₃)

[0074]



[0075] 4-^tButylacetyl-3-methyl-1-phenyl-pyrazol-5-one (2.93 g, 10.76 mmol), in a 100 ml round bottom flask, was dissolved in ethanol (~50 mL) with gentle heating. Lanthanum(III) chloride (1.27 g, 3.59 mmol) was dissolved in H₂O (~10 mL) and added to the pyrazolone solution. The resulting suspension was heated to reflux for 2 hours and then allowed to cool. The resulting suspension was filtered and washed with H₂O (3×10 mL) and EtOH (3×10 mL) then dried under vacuum at 80° C. to give a white powder with the following analysis.

Sc(^t BuPz) ₃	C	H	N
Theoretical	60.48	6.03	8.82
Found	59.92	6.28	8.67

[0076] Melting point: 114.1° C. (T_g)

[0077] Emission λ_{max}: 441.8 nm

[0078] Photoluminescence Efficiency (x,y): 0.003 cdm²μW⁻¹ (0.20, 0.22)

Reagents

[0079] Lanthanum(III) chloride hexahydrate, 99.9%; Strem Chmeicals, 93-5731; Lot no. 251194-S

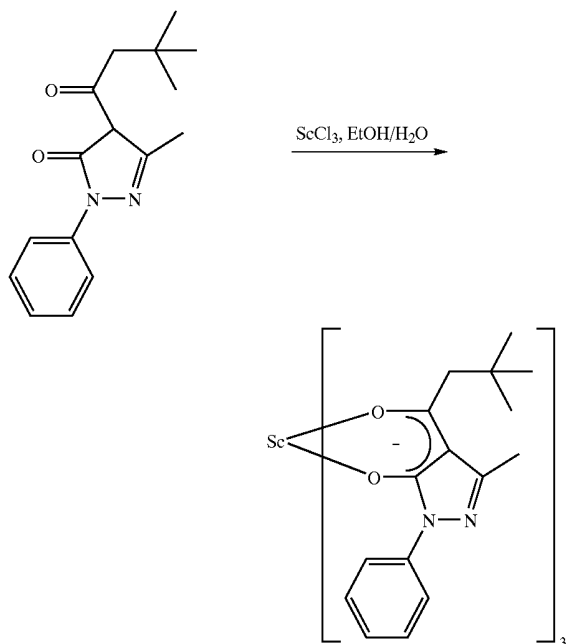
[0080] 4-^tButylacetyl-3-methyl-1-phenyl-pyrazol-5-one as prepared

[0081] Ethanol, denatured with 4.8% Methanol; Fluka; 02857.

EXAMPLE 3

Preparation of Tri-(4-^tButylacetyl-3-methyl-phenyl-pyrazol-5-onato)Scandium (Sc(^tBuPz)₃)

[0082]



[0083] 4-^tButylacetyl-3-methyl-1-phenyl-pyrazol-5-one (1.07 g, 3.93 mmol), in a 100 ml round bottom flask, was dissolved in ethanol (~50 mL) with gentle heating. Scandium(III) chloride hexahydrate (0.34 g, 1.31 mmol) was dissolved in H₂O (~40 mL) and added to the pyrazolone solution. The resulting suspension was heated to reflux for 2 hours and then allowed to cool. The resulting suspension was filtered and washed with H₂O (3×10 mL), and EtOH (3×10 mL) then dried under vacuum at 80° C. to give a white powder with the following analysis.

Sc(^t BuPz) ₃	C	H	N
Theoretical	67.12	6.69	9.78
Found	66.73	6.65	9.62

[0084] Melting point: 275.5° C. (D.S.C.)

[0085] Emission Max.: 448.55 nm

[0086]₂ Photoluminescence Efficiency (x,y): 0.004 cdm μW⁻¹ (0.22, 0.28)

Reagents

[0087] Scandium(III) chloride hexahydrate, 99.9%; Strem Chemicals; 93-2111, Lot no. B4745091

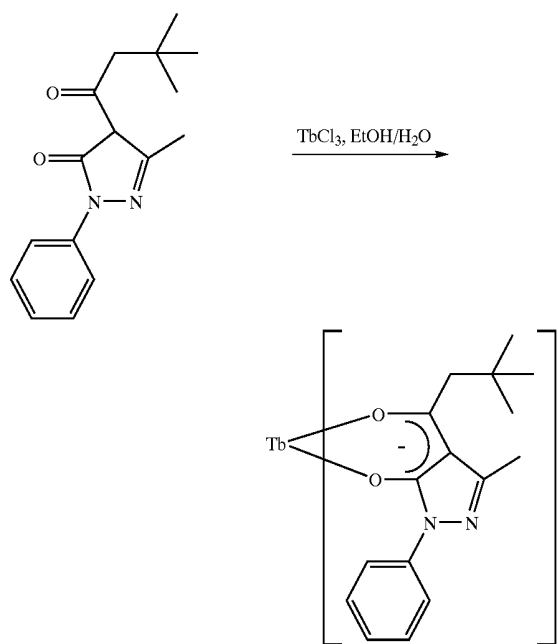
[0088] 4-^tButylacetyl-3-methyl-1-phenyl-pyrazol-5-one as prepared

[0089] Ethanol, denatured with 4.8% Methanol; Fluka; 02857

EXAMPLE 4

Preparation of Tris-(4-^tButylacetyl-3-methyl-1-phenyl-pyrazol-5-onato)Terbium (Tb(^tBuPz)₃)

[0090]



[0091] 4-^tButylacetyl-3-methyl-1-phenyl-pyrazol-5-one (1.45 g, 5.32 mmol), in a 100 ml round bottom flask, was dissolved in ethanol (~50 mL) with gentle heating. Terbium(III) chloride hexahydrate (0.66 g, 1.77 mmol) was dissolved in H₂O (~10 mL) and added to the pyrazolone solution. The resulting suspension was heated to reflux for 2 hours and then allowed to cool. The resulting suspension was filtered and washed with H₂O (3×10 mL) and EtOH (3×10 mL) then dried under vacuum at 80° C. to give a white powder with the following analysis.

Tb(^t BuPz) ₃	C	H	N
Theoretical	59.24	5.91	8.64
Found	59.86	6.23	8.75

[0092] Melting point: 252.6° C. (D.S.C.)

[0093] Emission Max.: 492.4 nm, 547.6 nm

[0094] Photoluminescence Efficiency (x,y): cdm² μW⁻¹

Reagents

[0095] Terbium(III) chloride, 99.9%; Acros

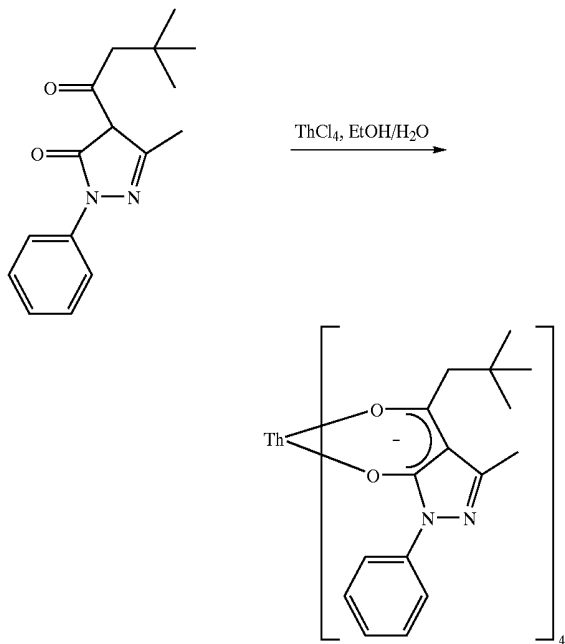
[0096] 4-^tButylacetyl-3-methyl-1-phenyl-pyrazol-5-one as prepared

[0097] Ethanol, denatured with 4.8% Methanol; Fluka; 02857

EXAMPLE 5

Preparation of Tetrakis-(4-^tButylacetyl-3-methyl-1-phenyl-pyrazol-5-onato)Thorium (Th(^tBuPz)₄)

[0098]



[0099] 4-^tButylacetyl-3-methyl-1-phenyl-pyrazol-5-one (1.92 g. 7.06 mmol), in a 100 ml round bottom flask, was dissolved in ethanol (~50 mL) with gentle heating. Thorium(IV) chloride hexahydrate (0.66 g. 1.77 mmol) was dissolved in H₂O (~10 mL) and added to the pyrazolone solution. The resulting suspension was heated to reflux for 2 hours and then allowed to cool. The resulting suspension was filtered and washed with H₂O (3×10 ml) and EtOH (3×10 mL) then dried under vacuum at 80° C. to give a pink powder with the following analysis.

Th(^t BuPz) ₄	C	H	N
Theoretical	58.35	5.81	8.58
Found	58.49	6.06	8.32

[0100] Melting point: 254.7° C. (D.S.C.)

[0101] Emission Max.: 462.8 nm

[0102]₂ Photoluminescence Efficiency (x,y): 0.002 cdm μW⁻¹ (0.27, 0.36)

Reagents

[0103] Thorium(IV) chloride hydrate, 99.9%; Strem Chemicals; 09-3155

[0104] 4-^tButylacetyl-3-methyl-1-phenyl-pyrazol-5-one; as prepared

[0105] Ethanol, denatured with 4.8% Methanol; Fluka; 02857

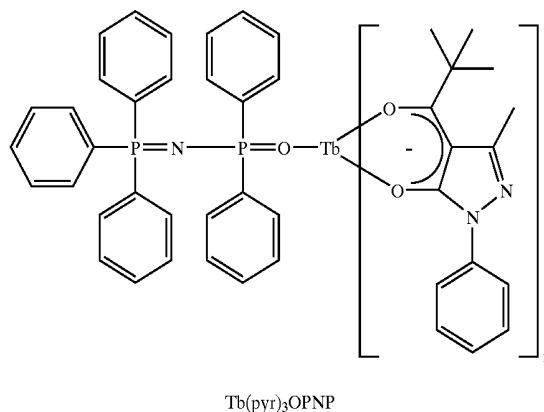
EXAMPLE 6

Tris-(4-^tButylacetyl-3-methyl-1-phenyl-pyrazol-5-onato)Calcium (Ca(^tBuPz)₃)

[0106] (Ca(^tBuPz)₃) was prepared by the method of Example using calcium chloride in place of the Terbium chloride.

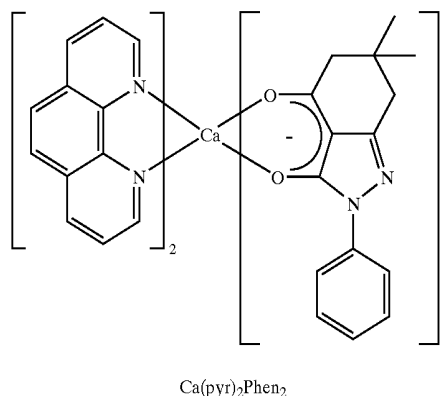
EXAMPLE 7

[0107] The (Tb(^tBuPz)₃) of Example 4 was heated at reflux with diphenylphosphinic-azide in trimethyl pentane and the mixture heated to reflux until a clear solution was obtained (about 1 hour). The solution was allowed to clear yielding (Tb(^tBuPz)₃)di phenylphosponimidetris-phenylphosphorane, (Tb(^tBuPz)₃OPNP[Tb(pyr)₃OPNP] as a crystalline solid.



EXAMPLE 8

[0108] The (Ca₂(^tBuPz)₃) of Example 6 was heated under reflux with phenanthrene in chloroform overnight. The solvent was removed in vacuo to yield a solid which was (Ca₂(^tBuPz)₃Phen₂[Ca(pyr)₂Phen₂]



Device Fabrication

[0109] An indium tin oxide (ITO) coated glass piece (1×1 cm²) had a portion etched out with concentrated hydrochloric acid to remove the ITO and was cleaned and dried. Four devices were fabricated

[0110] Device 1 was fabricated by sequentially forming on the ITO, layers comprising ITO/ α -NPB(75 nm)/Tb(pyr)₃OPNP(50 nm)/BCP(20 nm)/Alq₃(40 nm)/LiF(0.5 nm)/Al where α -NPB is in FIG. 1, BCP is bathocupron, LiF is lithium fluoride and Alq₃ is aluminium quinolate.

[0111] Device 2 was fabricated by sequentially forming on the ITO, layers comprising ITO/ α -NPB(10 nm)/Tb(pyr)₃OPNP(50 nm)/BCP(20 nm)/Alq₃(40 nm)/LiF(0.7 nm)/Al

[0112] Device 3 was fabricated by sequentially forming on the ITO, layers comprising ITO(100 Ω sqr)/CuPc(8 nm)/ α -NPB(60 nm)/Ca(pyr)₂Phen₂(50 nm)/Alq₃(10 nm)/LiF(0.7 nm)/Al where CuPc is copper phthalocyanine,

[0113] Device 4 was fabricated by sequentially forming on the ITO, layers comprising ITO(100 Ω sqr)/CuPc(8 nm)/ α -NPB(60 nm)/Ca(pyr)₂Phen₂(10 nm)/Al

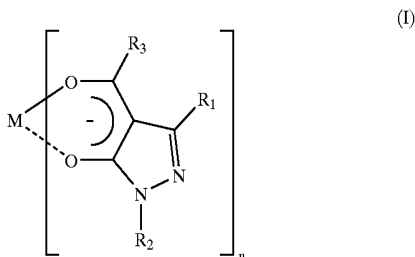
[0114] The organic coating on the portion which had been etched with, the concentrated hydrochloric acid was wiped with a cotton bud. The coated electrodes were stored in a vacuum desiccator over a molecular sieve and phosphorous pentoxide until they were loaded into a vacuum coater (Edwards, 10⁻⁶ torr) and aluminium top contacts made. The active area of the LED's was 0.08 cm² by 0.1 cm² the devices were then kept in a vacuum desiccator until the electroluminescence studies were performed.

[0115] The ITO electrode was always connected to the positive terminal. The current vs. voltage studies were carried out on a computer controlled Keithly 2400 source meter. Electroluminescence spectra were recorded by means of a computer controlled charge coupled device on PR650 system made by Photoresearch Inc.

[0116] The results are shown in FIGS. 16 to 23.

1-23. (canceled)

24. An electroluminescent compound which has the general chemical formula

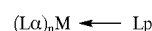


wherein M is a metal other than aluminum; n is the valency of M; R₁, R₂ and R₃, which may be the same or different, are selected from the group consisting of hydrogen, hydrocarbyl groups, substituted and unsubstituted aliphatic groups, substituted and unsubstituted aromatic, heterocyclic and polycyclic ring structures, fluorocarbon groups, halogen groups, thiophenyl groups, and nitrile groups; or alternatively R₁ and

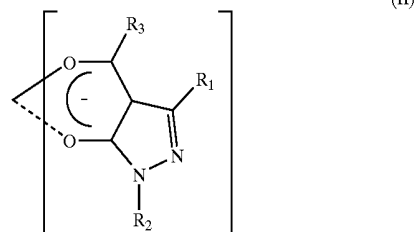
R₃ can form ring structures, or any of R₁, R₂ and R₃ can be copolymerized with a monomer.

25. A compound according to claim 24 wherein M is selected from the group consisting of gallium, indium, germanium, tin (II), tin (IV), antimony (II), antimony (IV), lead (II), lead (IV), metals of the first, second and third groups of transition metals in any valence states, manganese, iron, ruthenium, osmium, cobalt, nickel, palladium(II), palladium(IV), platinum(II), platinum(IV), cadmium, chromium, titanium, vanadium, zirconium, tantalum, molybdenum, rhodium, iridium, niobium, scandium and yttrium.

26. An electroluminescent compound which has the general chemical formula

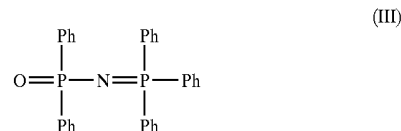


wherein L α has the general chemical formula



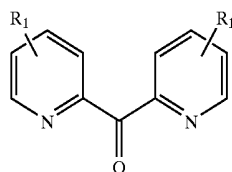
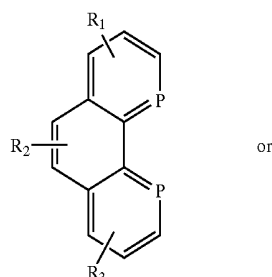
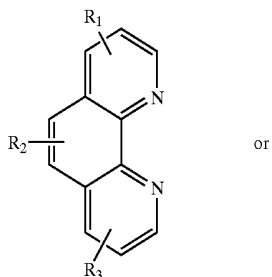
M is a metal, n is the valency of M, L_p is a neutral ligand, and R₁, R₂ and R₃, which may be the same or different, are selected from the group consisting of hydrogen, hydrocarbyl groups, substituted and unsubstituted aliphatic groups, substituted and unsubstituted aromatic, heterocyclic and polycyclic ring structures, fluorocarbon groups, halogen groups, thiophenyl groups, and nitrile groups; or alternatively R₁ and R₃ can form ring structures, or any of R₁, R₂ and R₃ can be copolymerized with a monomer.

27. An electroluminescent compound according to claim 26 wherein the groups L_p are selected from compounds having the general chemical formula



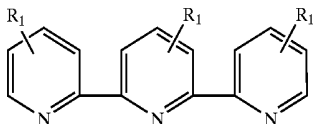
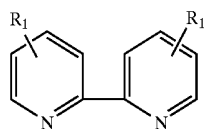
wherein each Ph which can be the same or different and is selected from the group consisting of phenyl (OPNP) and substituted phenyl groups, other substituted or unsubstituted aromatic groups, substituted or unsubstituted heterocyclic or polycyclic groups, substituted or unsubstituted fused aromatic groups, naphthyl groups, anthracene groups, phenanthrene groups, and pyrene groups, and the substituents in substituted phenyl groups are selected from the group consisting of alkyl groups, aralkyl groups, alkoxy groups, aromatic groups, heterocyclic groups, polycyclic groups, halogen groups, cyano groups, amino groups groups having

any of the following general chemical formulas:

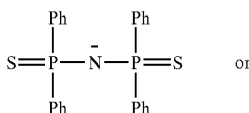


wherein R₁, R₂ and R₃ are as previously defined;

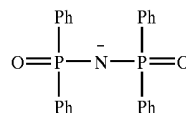
groups having any of the following general chemical formulas:



wherein R₁, R₂ and R₃ are as previously defined; and groups having any of the following general chemical formulas:



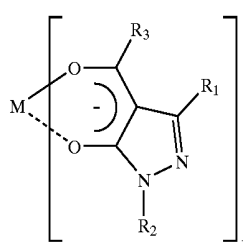
-continued



wherein Ph is as previously defined.

28. An electroluminescent compound according to claim 26 wherein M is a metal selected from the group consisting of gallium, indium, aluminum, germanium, tin (II), tin (IV), antimony (II), antimony (V), lead (II), lead (IV) and metals of the first, second and third groups of transition metals in different valence states e.g. manganese, iron, ruthenium, osmium, cobalt, nickel, palladium(II), palladium(IV), platinum(II), platinum(IV), cadmium, chromium, titanium, vanadium, zirconium, tantalum, molybdenum, rhodium, iridium, niobium, scandium or yttrium.

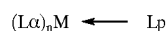
29. An electroluminescent device comprising: (i) a first electrode; (ii) an electroluminescent layer consisting essentially of a layer of an electroluminescent complex according to claim 24 of the general chemical formula



wherein M, n, R₁, R₂ and R₃ are as previously defined; and (iii) a second electrode.

30. An electroluminescent device according to claim 29 wherein M is selected from the group consisting of gallium, indium, germanium, tin (II), tin (IV), antimony (II), antimony (IV), lead (II), lead (IV), metals of the first, second and third groups of transition metals in any valence states, manganese, iron, ruthenium, osmium, cobalt, nickel, palladium(II), palladium(IV), platinum(II), platinum(IV), cadmium, chromium, titanium, vanadium, zirconium, tantalum, molybdenum, rhodium, iridium, niobium, scandium, and yttrium; and further wherein R₃ is a phenyl or substituted phenyl group.

31. An electroluminescent device comprising: (i) a first electrode; (ii) an electroluminescent layer consisting essentially of a layer of an electroluminescent compound according to claim 26 of the general chemical formula

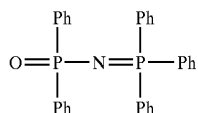


wherein Lα, M, n and Lp are as previously defined; and (iii) a second electrode.

32. An electroluminescent device according to claim 31 wherein M is a metal selected from gallium, indium, aluminum, germanium, tin (II), tin (IV), antimony (II), antimony (IV), lead (II), lead (IV) and metals of the first, second

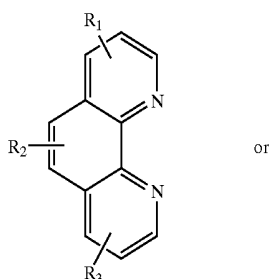
and third groups of transition metals in different valence states e.g. manganese, iron, ruthenium, osmium, cobalt, nickel, palladium(II), palladium(IV), platinum(II), platinum(IV), cadmium, chromium, titanium, vanadium, zirconium, tantalum, molybdenum, rhodium, iridium, niobium, scandium or yttrium.

33. An electroluminescent device according to claim 31 wherein the groups L_p are selected from compounds having the general chemical formula

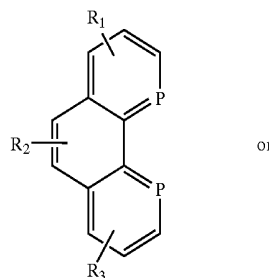


(III)

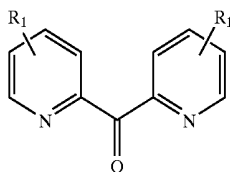
wherein each Ph which can be the same or different and is selected from the group consisting of phenyl (OPNP) and substituted phenyl groups, other substituted or unsubstituted aromatic groups, substituted or unsubstituted heterocyclic or polycyclic groups, substituted or unsubstituted fused aromatic groups, naphthyl groups, anthracene groups, phenanthrene groups, and pyrene groups, and the substituents in substituted phenyl groups are selected from the group consisting of alkyl groups, aralkyl groups, alkoxy groups, aromatic groups, heterocyclic groups, polycyclic groups, halogen groups, cyano groups, amino groups, groups having any of the following general chemical formulas:



(IV)



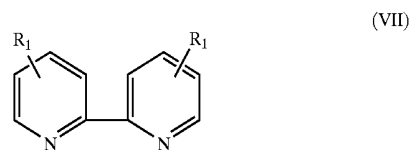
(V)



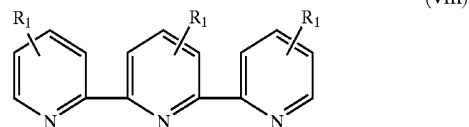
(VI)

wherein R_1 , R_2 and R_3 are as previously defined;

groups having any of the following general chemical formulas:

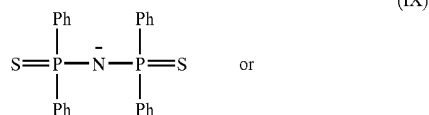


(VII)

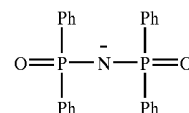


(VIII)

wherein R_1 , R_2 and R_3 are as previously defined; and groups having any of the following general chemical formulas:



(IX)



(X)

wherein Ph is as previously defined.

34. An electroluminescent device according to claim 29 wherein there is a layer of a hole transmitting material between the first electrode and the layer of the electroluminescent complex.

35. An electroluminescent device according to claim 34 wherein the hole transmitting material is selected from the group consisting of aromatic amine complexes and conjugated polymers.

36. An electroluminescent device according to claim 34 wherein the hole transmitting material is a film of a polymer selected from the group consisting of poly(vinylcarbazole); N,N' -diphenyl- N,N' -bis(3-methylphenyl)-1,1'-biphenyl-4,4'-diamine (TPD); polyaniline; substituted polyanilines; polythiophenes; substituted polythiophenes; polysilanes and substituted polysilanes; polymers of cyclic aromatic compounds; poly(p-phenylenevinylene)-PPV and copolymers; PPV; poly(2,5 dialkoxyphenylene vinylene); poly(2-methoxy-5-(2-methoxypropyloxy)-1,4-phenylene vinylene); poly(2-methoxypropyloxy)-1,4-phenylenevinylene); poly(2-methoxy-5-(2-dodecyloxy)-1,4-phenylenevinylene); other poly(2,5 dialkoxyphenylenevinylene)s with at least one of the alkoxy groups being a long chain solubilising alkoxy group; poly fluorenes; oligofluorenes; polyphenylenes; oligophenylenes; polyanthracenes and oligo anthracenes; and polythiophenes and oligothiophenes.

37. An electroluminescent device according to claim 31 wherein there is a layer of a hole transmitting material between the first electrode and the layer of electroluminescent compound, and further wherein the hole transmitting material is selected from the group consisting of aromatic amine complexes and conjugated polymers.

38. An electroluminescent device according to claim 37 wherein the hole transmitting material is a film of a polymer

selected from the group consisting of poly(vinylcarbazole); N,N'-diphenyl-N,N'-bis(3-methylphenyl)-1,1'-biphenyl-4,4'-diamine (TPD); polyaniline; substituted polyanilines; polythiophenes; substituted polythiophenes; polysilanes and substituted polysilanes; polymers of cyclic aromatic compounds, poly(p-phenylenevinylene)-PPV and copolymers; PPV; poly(2,5 dialkoxyphenylene vinylene); poly(2-methoxy-5-(2-methoxypentyloxy-1,4-phenylene vinylene); poly(2-methoxypentyloxy)-1,4-phenylenevinylene); poly(2-methoxy-5-(2-dodecyloxy-1,4-phenylenevinylene); other poly(2,5 dialkoxyphenylenevinylenes) with at least one of the alkoxy groups being a long chain solubilising alkoxy group; poly fluorenes; oligofluorenes; polyphenylenes; oligophenylenes; polyanthracenes and oligo anthracenes; polythiophenes and oligothiophenes.

39. An electroluminescent device according to claim 29 wherein there is a layer of an electron transmitting material between the electroluminescent compound layer and the second electrode.

40. An electroluminescent device according to claim 39 wherein the electron transmitting material is selected from the group consisting of metal quinolates and cyano anthracenes.

41. An electroluminescent device according to claim 39 wherein the electron transmitting material is an aluminum quinolate or lithium quinolate.

42. An electroluminescent device according to claim 31 wherein there is a layer of an electron transmitting material between the electroluminescent compound layer and the second electrode.

43. An electroluminescent device according to claim 42 wherein the electron transmitting material is selected from the group consisting of metal quinolates and cyano anthracenes.

44. An electroluminescent device according to claim 42 wherein the electron transmitting material is an aluminum quinolate or lithium quinolate.

45. An electroluminescent device according to claim 29 wherein the second electrode is a material selected from the group consisting of aluminum, calcium, lithium, and silver/magnesium alloys.

46. An electroluminescent device according to claim 31 wherein the second electrode is a material selected from the group consisting of aluminum, calcium, lithium, and silver/magnesium alloys.

47. An electroluminescent device according to claim 34 wherein the hole transmitting material and the electroluminescent compound are mixed to form one layer in a proportion ranging from about 5% of the electroluminescent compound and 95% of the hole transmitting material to about 95% of the electroluminescent compound and 5% of the hole transmitting material.

48. An electroluminescent device according to claim 39 wherein the electron transmitting material and the light emitting metal compound are mixed to form one layer in a proportion ranging from about 5% of the light emitting metal compound and 95% of the electron transmitting material to about 95% of the light emitting metal compound and 5% of the electroluminescent compound.

49. An electroluminescent device according to claim 48 wherein there is a copper phthalocyanine layer on the first electrode and a lithium fluoride layer on the second electrode.

* * * * *

专利名称(译)	电致发光材料和器件		
公开(公告)号	US20060035110A1	公开(公告)日	2006-02-16
申请号	US10/537315	申请日	2003-12-05
[标]申请(专利权)人(译)	KATHIRGAMANATHAN POOPATHY SURENDRAKUMAR SILVAGNANASUNDRA GEMMELL PATRICK GANESHAMURUGAN SUBRAMANIAM KUMARAVARL MUTTULILINGHAM PARTHEEPAN的Arumugam SURESH SUTHERALINGAM SELVARANJAN SELVADURAL		
申请(专利权)人(译)	KATHIRGAMANATHAN POOPATHY SURENDRAKUMAR SILVAGNANASUNDRA GEMMELL PATRICK GANESHAMURUGAN SUBRAMANIAM KUMARAVARL MUTTULILINGHAM PARTHEEPAN的Arumugam SURESH SUTHERALINGAM SELVARANJAN SELVADURAL		
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[标]发明人	KATHIRGAMANATHAN POOPATHY SURENDRAKUMAR SILVAGNANASUNDRAM GEMMELL PATRICK GANESHAMURUGAN SUBRAMANIAM KUMARAVARL MUTTULILINGHAM PARTHEEPAN ARUMUGAM SURESH SUTHERALINGAM SELVARANJAN SELVADURAL		
发明人	KATHIRGAMANATHAN, POOPATHY SURENDRAKUMAR, SILVAGNANASUNDRAM GEMMELL, PATRICK GANESHAMURUGAN, SUBRAMANIAM KUMARAVARL, MUTTULILINGHAM PARTHEEPAN, ARUMUGAM SURESH, SUTHERALINGAM SELVARANJAN, SELVADURAL		
IPC分类号	H05B33/14 C09K11/06 C07D231/00 C07D231/26 C07F9/535 C07F9/6596 H01L51/00 H01L51/30 H01L51/50		

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H01L51/005 H01L51/0053 H01L51/0059 H01L51/0062 H01L51/007 H01L51/0077 H01L51/0078
H01L51/0081 H01L51/0089 C07F9/5355 Y10S428/917

优先权 2002028335 2002-12-05 GB

其他公开文献 US7718275

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摘要(译)

电致发光材料是式 (1) 的1-苯基-3-甲基-4-三甲基乙酰基 - 吡唑-5-酮的金属络合物。在发光层中包含式 (1) 化合物的电致发光器件也是本发明的一部分。

