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(54) Organic electroluminescent element

Organisches elektrolumineszierendes Element

Elément électroluminescent organique

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Remarks:

The file contains technical information submitted after the application was filed and not included in this specification

Description

TECHNICAL FIELD

[0001] The present invention relates to materials for organic electroluminescence devices which are used as a light source such as a planar light emitting member of televisions and a back light of displays, exhibit high efficiency of light emission and have excellent heat resistance and a long life, and organic electroluminescence devices using the materials.

BACKGROUND ART

[0002] Electroluminescence (EL) devices using organic compounds are expected to be used for inexpensive full color display devices of the solid light emission type which can display a large area and development thereof has been actively conducted. In general, an EL device is constituted with a light emitting layer and a pair of electrodes faced to each other at both sides of the light emitting layer. When a voltage is applied between the electrodes, electrons are injected at the side of the cathode and holes are injected at the side of the anode. The electrons are combined with the holes in the light emitting layer and an excited state is formed. When the excited state returns to the normal state, the energy is emitted as light.

[0003] Heretofore, organic EL devices require higher driving voltages and show inferior luminance of emitted light and inferior efficiencies of light emission in comparison with inorganic devices. Moreover, properties of organic EL devices deteriorate very rapidly. Therefore, heretofore, organic EL devices have not been used practically. Although the properties of organic EL devices have been improved, organic EL devices exhibiting a sufficient efficiency of light emission and having sufficient heat resistance and life have not been obtained. For example, a phenylanthracene derivative which can be used for EL devices is disclosed in Japanese Patent Application Laid-Open No. Heisei 8(1996)-12600. However, an organic EL device using this compound exhibited an efficiency of light emission as low as about 2 to 4 cd/A and improvement in the efficiency is desired. In Japanese Patent Application Laid-Open No. Heisei 8(1996)-199162, an EL device having a light emitting layer containing a fluorescent dopant of a derivative of an amine or a diamine is disclosed. However, this EL device has a life as short as 700 hours at an initial luminance of emitted light of 300 cd/m^2 although the efficiency of light emission is 4 to 6 dc/A and improvement in the life is desired. In Japanese Patent Application Laid-Open No. Heisei 9(1997)-268284, a material for EL devices having phenylanthracene group is disclosed. This material exhibits a marked decrease in the luminance of emitted light when the material is used at a high temperature for a long time and heat resistance is insufficient. Moreover, these devices do not emit light in the region of orange to red color. Since emission of red color is indispensable for the full color display by an EL device, a device emitting light in the region of orange to red color is desired. When these materials are used as the host material and other compounds are used as the doping material, a long life cannot be obtained. It is necessary for practical use that an initial luminance of emitted light of 10,000 d/m^2 or greater be exhibited. However, this value has not been achieved. In Japanese Patent Application Laid-Open No. Heisei 11(1999)-152253; an example is disclosed in which a material for organic EL devices having a binaphthalene structure is added to a light emitting layer having the property to transfer electrons such as a layer of an aluminum complex or the like. However, in this example, the aluminum complex or the like emits light and the material for organic EL devices does not function as the light emitting center since the energy gap of the light emitting layer of the aluminum complex or the like is smaller than the energy gap of the material for organic EL devices.

[0004] Materials for organic electroluminescence devices comprising an anthracene nucleus substituted with arylamine groups are described in EP-A-0 866 110 and EP-A-0 765 106.

[0005] Further materials comprising two connected anthracene groups as the nucleus are described in US-B1-6,203,933.

[0006] Synthesis of arylamines used as a material for organic EL devices has been conducted by the Ullmann reaction using an amine and an iodobenzene. It is described, for example, in Chem. Lett., pp. 1145 to 1148, 1989; the specification of United States Patent No. 4,764,625; and Japanese Patent Application Laid-Open No. Heisei 8(1996)-48974 that a triarylamine is produced by the reaction of a corresponding iodobenzene and a diarylamine in an inert hydrocarbon solvent such as decaline at 150°C or higher in the presence of one equivalent or more of copper powder and a base such as potassium hydroxide as the typical example.

[0007] However, the process using the Ullmann reaction has drawbacks in that an expensive iodide must be used as the reacting agent, that the reaction cannot be applied to many types of compounds, that the yield of the reaction is not sufficient, that the reaction requires a temperature as high as 150°C and a long time and that waste liquid containing a great amount of copper is formed since copper powder is used in a great amount and environmental problems arise.

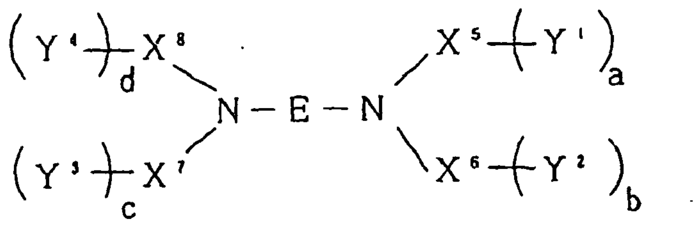
DISCLOSURE OF THE INVENTION

[0008] The present invention has been made to overcome the above problems and has an object to provide a material for organic electroluminescence devices, and an organic electroluminescence device which exhibit high efficiency of light emission and have a long life and excellent heat resistance.

[0009] As the result of extensive studies by the present inventors to develop the material for organic EL devices having the advantageous properties described above and an organic EL device using the material, it was found that the object can be achieved by using the compounds represented by general formula (9) which are shown below. The present invention has been completed based on this knowledge.

[0010] The material for organic electroluminescence devices (referred to as the material for organic EL devices) of the present invention is a compound represented by following general. formula [9]:

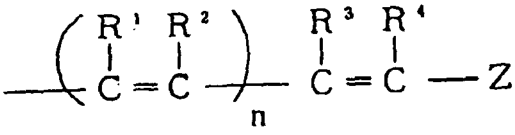
General formula [9]



[9]

wherein E represents a divalent group comprising an anthracene nucleus which is substituted with at least two aryl groups, X^5 to X^8 each independently represent a substituted or unsubstituted arylene group having 6 to 20 carbon atoms, X^5 and X^6 may be bonded to each other, X^7 and X^8 may be bonded to each other, Y^1 to Y^4 each independently represent an organic group represented by general formula [2], a to d each represent an integer of 0 to 2; general formula [2] being:

General formula [2]



$$-\left(\overset{R^1}{\underset{}{C}}=\overset{R^2}{C}\right)_n-\overset{R^3}{C}=\overset{R^4}{C}-Z \qquad [2]$$

wherein R^1 to R^4 each independently represent hydrogen atom, a substituted or unsubstituted alkyl group having 1 to 20 carbon atoms, a substituted or unsubstituted aryl group having 6 to 20 carbon atoms or cyano group or form a triple bond by a linkage of R^1 and R^2 or R^3 and R^4, Z represents a substituted or unsubstituted aryl group having 6 to 20 carbon atoms and n represents 0 or 1.

[0011] The material for organic EL devices of the present invention which is represented by the general formula (9) can be used also as the light emitting material for organic electroluminescence devices.

[0012] The organic electroluminescence (EL) device of the present invention comprises a light emitting layer or a plurality of thin films of organic compounds comprising a light emitting layer disposed between a pair of electrodes, wherein at least one of the thin films of organic compounds is a layer comprising a materials for organic EL devices represented by the general formula (9).

[0013] It is preferable that, in the above organic EL device, a layer comprising the material for organic EL devices represented by the general formula (9) as at least one material selected from a group consisting of a hole injecting material, a hole transporting material and a doping material is disposed between the pair of electrodes

[0014] It is preferable that, in the above organic EL device, the light emitting layer comprises 0.1 to 20% by weight of a material for organic EL devices represented by the general formula (9).

[0015] It is preferable that, in the above organic electroluminescence device, one or more materials selected from a group consisting of a hole injecting material, a hole transporting material and a doping material each independently comprise 0.1 to 20% by weight of the material for organic EL devices represented by any of general formula (9).

[0016] It is preferable that, in the above organic EL device, the light emitting layer is a layer comprising a stilbene derivative and a material for organic EL devices represented by the general formula (9).

[0017] In the above organic EL device, a layer comprising an aromatic tertiary amine derivative and/or a phthalocyanine derivative is disposed between a light emitting layer and an anode.

BRIEF DESCRIPTION OF THE DRAWINGS

[0018]

Figure 1 shows a $^1H_{NMR}$ chart of compound a synthesized in accordance with the process of the present invention.
Figure 2 shows a $^1H_{NMR}$ chart of compound b synthesized in accordance with the process of the present invention.
Figure 3 shows a $^1H_{NMR}$ chart of compound e synthesized in accordance with the process of the present invention.

THE MOST PREFERRED EMBODIMENT TO CARRY OUT THE INVENTION

[0019] In the compound represented by general formula [9] in the present invention, E represents a divalent group comprising an anthracene nucleus which is substituted with at least two aryl groups. X^5 to X^8 each independently represent a substituted or unsubstituted arylene group having 6 to 20 carbon atoms. Examples of the group represented by X^5 to X^8 include monovalent and divalent groups containing the skeleton structure of phenylene, biphenyl, terphenyl, naphthalene, anthracene, phenanthrene, fluorene and thiophene. X^5 and X^6 may be bonded to each other and X^7 and X^8 may be bonded to each other.

[0020] Y^1 to Y^4 each independently represent an organic group represented by general formula (2) and a to d each represent an integer of 0 to 2.

[0021] In general formula [2] in the present invention, R^1 to R^4 each independently represent hydrogen atom, a substituted or unsubstituted alkyl group having 1 to 20 carbon atoms, a substituted or unsubstituted aryl group having 6 to 20 carbon atoms or cyano group. Examples of the group represented by R^1 to R^4 include substituted and unsubstituted alkyl groups such as methyl group, ethyl group, propyl group, butyl group, sec-butyl group, tert-butyl group, pentyl group, hexyl group, heptyl group, octyl group, stearyl group, 2-phenylisopropyl group, trichloromethyl group, trifluoromethyl group, benzyl group, α-phenoxybenzyl group, α,α-dimethylbenzyl group, α,α-methylphenylbenzyl group, α,α-ditrifluoromethylbenzyl group, triphenylmethyl group and α-benzyloxybenzyl group; and substituted and unsubstituted aryl groups such as phenyl group, 2-methylphenyl group, 3-methylphenyl group, 4-methylphenyl group, 4-ethylphenyl group, biphenyl group, 4-methylbiphenyl group, 4-ethylbiphenyl group, 4-cyclohexylbiphenyl group, terphenyl group, 3,5-dichlorophenyl group, naphthyl group, 5-methylnaphthyl group, anthryl group and pyrenyl group.

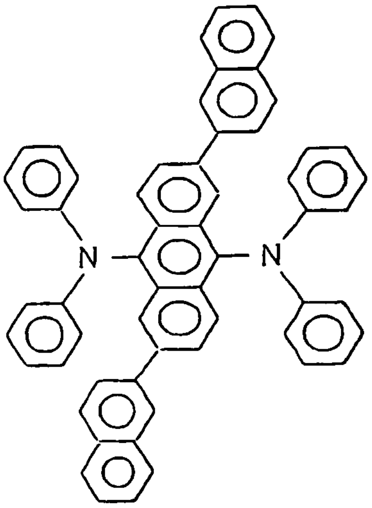
[0022] In general formula [2] in the present invention, Z represents a substituted or unsubstituted aryl group having 6 to 20 carbon atoms. Examples of the group represented by Z include aryl groups such as phenyl group, biphenyl group, terphenyl group, naphthyl group, anthryl group, phenanthryl group, fluorenyl group, pyrenyl group and thiophene group. The above aryl groups may have substituents. Examples of the substituent include alkyl groups and aryl groups described above as the examples of the group represented by R^1 to R^4, alkoxy groups, amino group, cyano group, hydroxyl group, carboxylic acid group, ether group and ester groups. In general formula [2], n represents 0 or 1.

[0023] As described above, since the compound represented by general formula [9] in the present invention has a diamine structure, the ionization energy is 5.6 eV or smaller and holes can be easily injected. The mobility of holes is $10^{-4}m^2/V\cdot s$ or greater. Therefore, the compound has the excellent properties as the hole injecting material and the hole transporting material. Due to the substituted anthracene nucleus at the center, electrons are easily injected.

[0024] When the anthracene nucleus represented by E is unsubstituted, the glass transition temperature is as low as 100°C or lower. The glass transition temperature is elevated by bonding at least two substituents and preferably 2 to 4 substituents to the nucleus as described above. The specific biphenyl structure described above enhances solubility of the compound represented by general formula [9] and purification can be facilitated. When phenyl group is bonded at a position other than the above position, i.e., at the para-position, the content of impurities increases since purification becomes difficult and the properties of the obtained organic EL device deteriorate. By the substitution of aryl groups as described above, formation of pairs of the molecules by association is suppressed and the quantum efficiency of fluorescence emission increases. Thus, the efficiency of light emission of the organic EL device is improved.

[0025] In the following, Compounds (87) to (92), (100) and (101) as the typical examples of the compound represented by general formula [9] are shown. However, the present invention is not limited to these typical examples.

(87)



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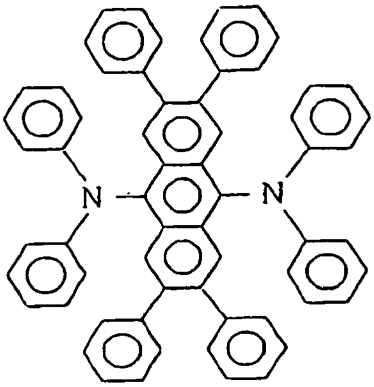

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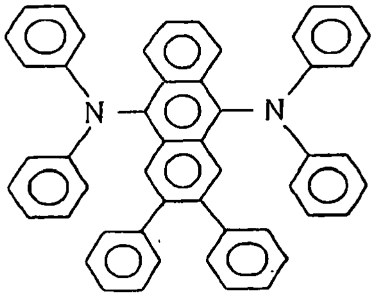

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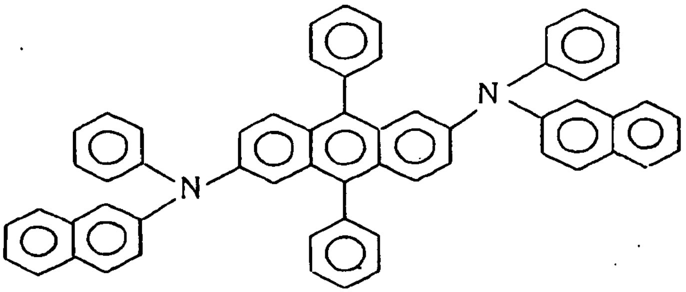

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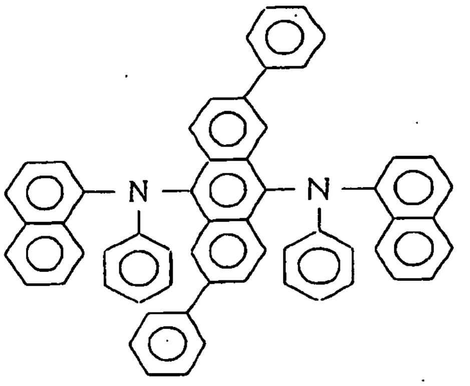

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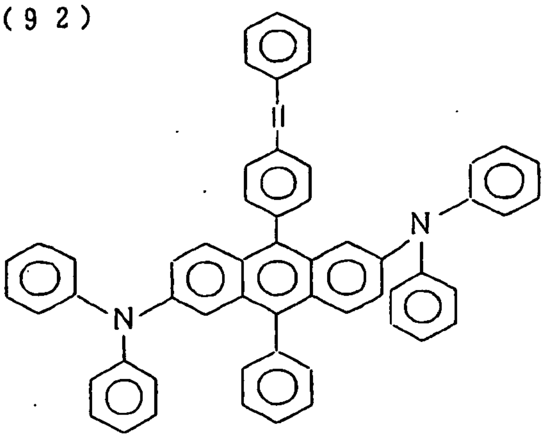

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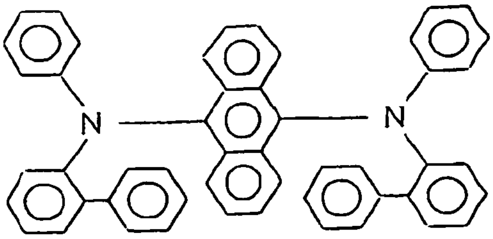

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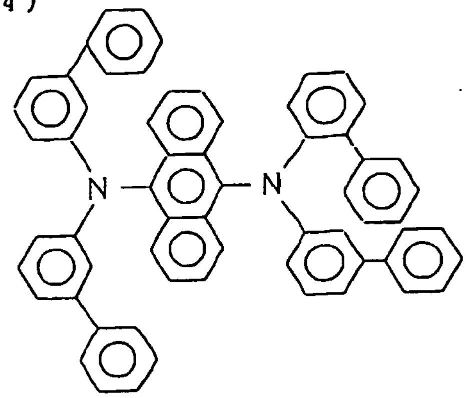

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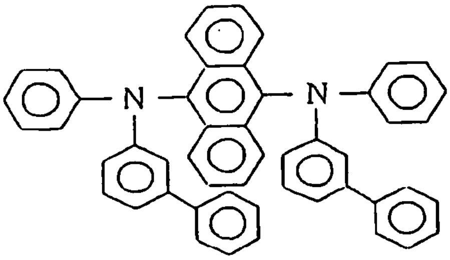

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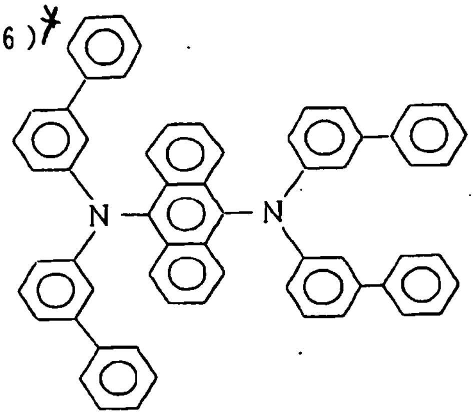

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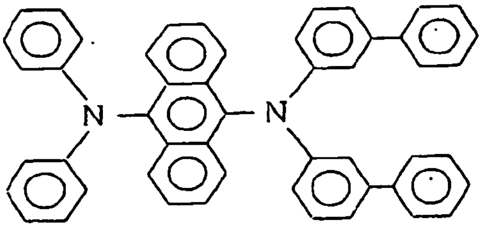

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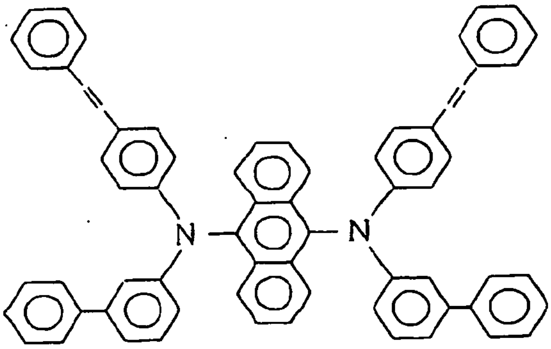

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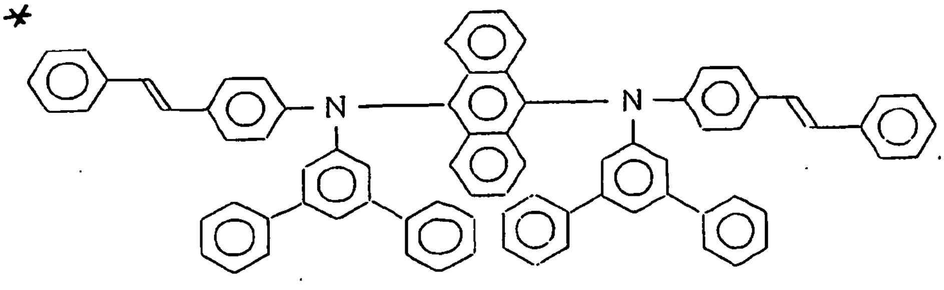

*: not part of the invention

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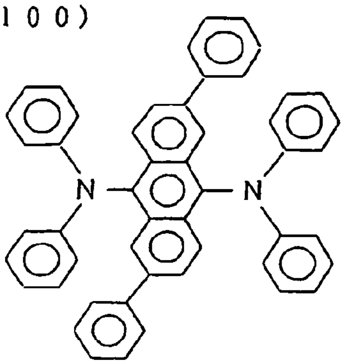

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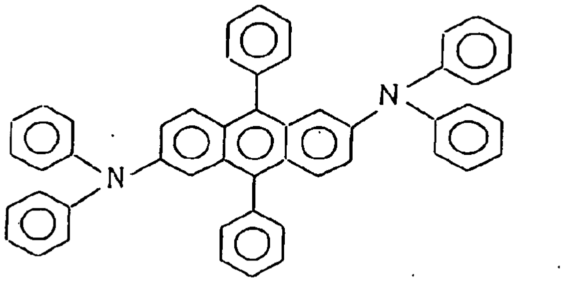

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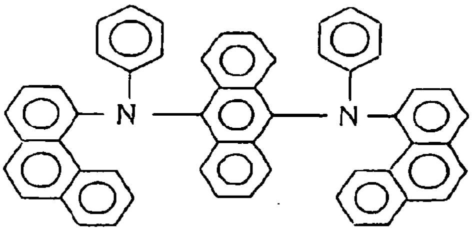

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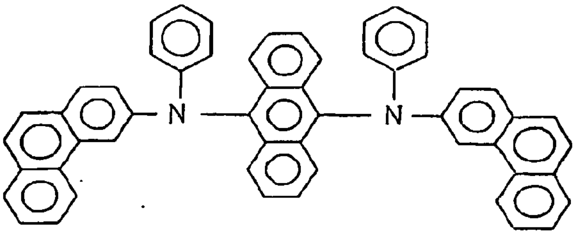

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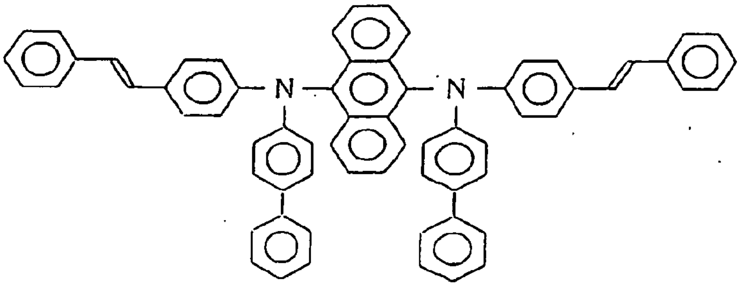


*: not part of the invention

[0026] The compounds represented by general formula (9) of the present invention exhibit strong fluorescence in the solid state, have the excellent light emitting property in the electric field and show a quantum efficiency of fluorescence emission of 0.3 or greater since the polyphenyl structure represented by A or B and the amine structure are connected to each other at the center of the compounds.

[0027] The compounds represented by general formula (9) of the present invention can be used effectively as the light emitting material and may be used also as the hole transporting material, the electron transporting material and the doping material since the compounds have all of the hole injecting property from metal electrodes or organic thin film layers, the hole transporting property, the electron injecting property from metal electrodes or organic thin film layers and the electron transporting property.

[0028] The organic EL device of the present invention is a device in which one or a plurality of organic thin films are disposed between an anode and a cathode. When the device has a single layer, a light emitting layer is disposed between an anode and a cathode. The light emitting layer contains a light emitting material and may also contain a hole injecting material or a electron injecting material to transport holes injected at the anode or electrons injected at the cathode to the light emitting material. However, it is possible that the light emitting layer is formed with the light emitting material of the present invention alone because the light emitting material of the present invention has a very high quantum efficiency of fluorescence emission, excellent ability to transfer holes and excellent ability to transfer electrons and a uniform thin film can be formed. The organic EL device of the present invention having a multi-layer structure has a laminate structure such as: (an anode / a hole injecting layer / a light emitting layer / a cathode), (an anode / a light emitting layer / an electron injecting layer / a cathode) and (an anode / a hole injecting layer / a light emitting layer / an electron injecting layer / a cathode). Since the compounds represented by general formula (9) have the excellent light emitting property and, moreover, the excellent hole injecting property, hole transporting property, electron injecting property and electron transporting property, the compounds can be used for the light emitting layer as the light emitting material.

[0029] In the light emitting layer, where necessary, conventional light emitting materials, doping materials, hole injecting materials and electron injecting materials may be used in addition to the compounds represented by general formula (9) of the present invention. Deterioration in luminance and Life caused by quenching can be prevented by the multi-layer structure of the organic EL. Where necessary, a light emitting materials, a doping materials, a hole injecting materials and an electron injecting materials may be used in combination. By using a doping material, luminance and the efficiency of light emission can be improved and blue light and red light can be emitted. The hole injecting layer, the light emitting layer and the electron injecting layer may each have a multi-layer structure having two or more layers. When the hole injecting layer has a multi-layer structure, the layer into which holes are injected from the electrode is referred to as the hole injecting layer and the layer which receives holes from the hole injecting layer and transports holes from the hole injecting layer to the light emitting layer is referred to as the hole transporting layer. When the electron injecting layer has a multi-layer structure, the layer into which electrons are injected from the electrode is referred to as the electron injecting layer and the layer which receives electrons from the electron injecting layer and transports electrons from the electron injecting layer to the light emitting layer is referred to as the electron transporting layer. These layers are each selected and used in accordance with factors such as the energy level and heat resistance of the material and adhesion with the organic layers or the metal electrodes.

[0030] Examples of the material which can be used in the light emitting layer as the light emitting material or the doping material in combination with the compounds represented by general formula (9) include anthracene, naphthalene, phenanthrene, pyrene, tetracene, coronene, chrysene, fluoresceine, perylene, phthaloperylene, naphthaloperylene, perynone, phthaloperynone, naphthaloperynone, diphenylbutadiene, tetraphenylbutadiene, coumarine, oxadiazole, aldazine, bisbenzoxazoline, bisstyryl, pyrazine, cyclopentadiene, metal complexes of quinoline, metal complexes of aminoquinoline, metal complexes of benzoquinoline, imines, diphenylethylene, vinylanthracene, diamino-carbazole, pyrane, thiopyrane, polymethine, merocyanine, oxinoid compounds chelated with imidazoles, quinacridone, rubrene, stilbene derivatives and fluorescent dyes. However, the examples of the above material are not limited to the above compounds.

[0031] As the hole injecting material, a compound which has the ability to transfer holes, exhibits excellent effect of hole injection from the anode and excellent effect of hole injection to the light emitting layer or the light emitting material, prevents transfer of excited components formed in the light emitting layer into the electron injecting layer or the electron injecting material and has an excellent ability to form a thin film is preferable. Examples of such a compound include phthalocyanine derivatives, naphthalocyanine derivatives, porphyrin derivatives, oxaozole, oxadiazole, triazole, imidazole, imdazolone, imdazolethione, pyrazoline, pyrazolone, tetrahydroimidazole, oxazole, oxadiazole, hydrazone, acylhydrazone, polyarylalkanes, stilbene, butadiene, benzidine-type triphenylamine, styrylamine type triphenylamine, diamine type triphenylamine, derivatives of these compounds and macromolecular compounds such as polyvinylcarbazole, polysilane and conductive macromolecules. However, examples of such a compound are not limited to the compounds described above.

[0032] Among the hole injection materials which can be used in the organic EL device of the present invention, more effective hole injecting materials are aromatic tertiary amine derivatives and phthalocyanine derivatives.

[0033] Examples of the aromatic tertiary amine derivative include triphenylamine, tritolylamine, tolyldiphenylamine, N,N'-diphenyl-N,N'-(3-methylphenyl)-1,1'-biphenyl-4,4'-diamine, N,N,N',N'-(4-methylphenyl)-1,1'-phenyl-4,4'-diamine, N,N,N',N'-(4-methylphenyl)-1,1'-biphenyl-4,4'-diamine, N,N'-diphenyl-N,N'-dinaphthyl-1,1'-biphenyl-4,4'-diamine, N, N'-(methylphenyl)-N,N'-(4-n-butylphenyl)phenanthrene-9,10-diamine, N,N-bis(4-di-4-tolylaminophenyl)-4-phenylcyclohexane and oligomers and polymers having a skeleton structure of these aromatic tertiary amines. However, examples of the aromatic tertiary amine derivative are not limited to the above compounds.

[0034] Examples of the phthalocyanine (Pc) derivative include H_2Pc, CuPc, CoPc, NiPc, ZnPc, PdPc, FePc, MnPc, ClAlPc, ClGaPc, ClInPc, ClSnPc, Cl_2SiPc, (HO)AlPc, (HO)GaPc, VOPc, TiOPc, MoOPc, GaPc-O-GaPc and corresponding derivatives of naphthalocyanine. However, examples of the derivatives of phthalocyanine and naphthalocyanine are not limited to the above compounds.

[0035] As the electron injecting material, a compound which has the ability to transport electrons, exhibits excellent effect of electron injection from the cathode and excellent effect of electron injecting to the light emitting layer or the light emitting material, prevents transfer of excited components formed in the light emitting layer into the hole injecting layer or the hole injecting material and has an excellent ability to form a thin film is preferable. Examples of such a compound include fluorenone, anthraquinodimethane, diphenoquinone, thiopyrane dioxide, oxazole, oxadiazole, triazole, imidazole, perylenetetetracarboxylic acid, fluorenylidenemethane, anthraquinodimethane, anthrone and derivatives of these compounds. However, examples of such a compound is not limited to the compounds described above. The electron injecting property can be improved by adding an electron accepting material to the hole injecting material or an electron donating material to the electron injecting material.

[0036] In the organic EL device of the present invention, more effective electron injecting materials are metal complex compounds and five-membered derivatives containing nitrogen.

[0037] Examples of the metal complex compound include 8-hydroxyquinolinatolithium, bis(8-hydroxyquinolinato)zinc, bis(8-hydroxyquinolinato)copper, bis(8-hydroxyquinolinato)manganese, tris(8-hydroxyquinolinato)aluminum, tris(2-methyl-8-hydroxyquinolinato)-aluminum, tris(8-hydroxyquinilinato)gallium, bis(10-hydroxybenzo-[h]quinolinato)beryllium, bis(10-hydroxybenzo[h]quinolinato)zinc, bis(2-methyl-8-quinolinato)chlorogallium, bis(2-methyl-8-quinolinato)(o-cresolato)gallium, bis(2-methyl-8-quinolinato)(1-naphtholato)aluminum and bis(2-methyl-8-quinolinato)(2-naphtholato)gallium. However, examples of the metal complex compound are not limited to the above compounds.

[0038] Preferable examples of the five-membered derivative containing nitrogen include derivatives of oxazoles, thiazoles, thiadiazoles and triazoles. Specific examples include 2,5-bis(1-phenyl)-1,3,4-oxazole, dimethylPOPOP, 2,5-bis (1-phenyl)-1,3,4-thiazole, 2,5-bis(1-phenyl)-1,3,4-oxadiazole, 2-(4'-tert-butylphenyl)-5-(4"-biphenyl)-1,3,4-oxadiazole, 2,5-bis(1-naphthyl)-1,3,4-oxadiazole, 1,4-bis[2-(5-phenyloxadiazolyl)]benzene, 1,4-bis[2-(5-phenyloxadiazolyl)-4-tert-butylbenzene], 2-(4'-tert-butylphenyl)-5-(4"-biphenyl)-1,3,4-thiadiazole, 2,5-bis(1-naphthyl)-1,3,4-thiadiazole, 1,4-bis [2-(5-phenylthiadiazolyl)]benzene, 2-(4'tert-butylphenyl)-5-(4"-biphenyl)-1,3,4-triazole, 2,5-bis(1-naphthyl)-1,3,4-triazole and 1,4-bis[2-(5-phenyltriazolyl)]benzene. However, examples of the five-membered derivative containing nitrogen are not limited to the above compounds.

[0039] As the conductive material used as the anode of the organic EL device, a material having a work function of 4 eV or greater is suitable. Examples of such a material include carbon, aluminum, vanadium, iron, cobalt, nickel, tungsten, silver, gold, platinum, palladium, alloys of these metals, metal oxides used for ITO substrates and NESA substrates such as tin oxide and indium oxide and organic conductive resins such as polythiophene and polypyrrol. As the conductive material used for the cathode, a material having a work function smaller than 4 eV is suitable. Examples of such a material include magnesium, calcium, tin, lead, titanium, yttrium, lithium, ruthenium, manganese, aluminum and alloys of these metals. However, examples of the materials used for the anode and the cathode are not limited to the above examples. Typical examples of the alloy include alloys of magnesium and silver, alloys of magnesium and indium and alloys of lithium and aluminum. However, examples of the alloy are not limited to these alloys. The composition of the alloy is determined by the temperature of the source of vapor deposition, the atmosphere and the degree of vacuum and a suitable composition is selected. The anode and the cathode may have a multi-layer structure having two or more layers, where necessary.

[0040] In the organic EL device, it is preferable that at least one face of the device is sufficiently transparent in the wave length region of emitted light to achieve efficient light emission. It is preferable that the substrate is also transparent. In the preparation of the transparent electrode, the above conductive material is used and vapor deposition or sputtering is conducted so that the prescribed transparency is surly obtained. It is preferable that the electrode disposed on the light emitting face has a light transmittance of 10% or greater. The substrate is not particularly limited as long as the substrate has mechanical strength and strength at high temperatures and is transparent. Glass substrates or transparent films of resins may be used. Example of the transparent films of resins include films of polyethylene, ethylene-vinyl acetate copolymers, ethylene-vinyl alcohol copolymers, polypropylene, polystyrene, polymethyl methacrylate, polyvinyl chloride, polyvinyl alcohol, polyvinyl butyral, nylon, polyether ether ketones, polsulfones, polyether sulfones, tetrafluoroethylene-perfluoroalkyl vinyl ether copolymers, polyvinyl fluoride, tetrafluoroethylene-ethylene copolymers, tetrafluoroethylene-hexafluoropropylene copolymers, polychlorotrifluoroethylene, polyvinylidene fluoride, polyesters, polycarbonates, polyurethanes, polyimides, polyether imides, polyimides and polypropylene.

[0041] Each layer of the organic EL device of the present invention can be produced suitably in accordance with a dry process of film formation such as vacuum vapor deposition, sputtering and plasma and ion plating or a wet process of film formation such as spin coating, dipping and flow coating. The thickness of the film is not particularly limited. However, it is necessary that the thickness be set at a suitable value. When the thickness is greater than the suitable value, a great voltage must be applied to obtain a prescribed output of light and the efficiency deteriorates. When the thickness is smaller than the suitable value, pin holes are formed and a sufficient luminance cannot be obtained even when the electric field is applied. In general, the suitable range of the thickness is 5 nm to 10 μm. A thickness in the range of 10 nm to 0.2 μm is preferable.

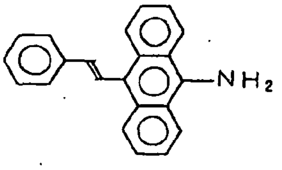
[0042] When the device is produced in accordance with a wet process, materials forming each layer are dissolved or dispersed in a suitable solvent such as ethanol, chloroform, tetrahydrofuran and dioxane and a film is formed from the solution or the suspension. The solvent is not particularly limited. In any organic thin layer, suitable resins and additives may be used to improve the property to form a film and to prevent formation of pin holes. Examples of the resin which can be used include insulating resins such as polystyrene, polycarbonates, polyarylates, polyesters, polyamides, polyurethanes, polysulfones, polymethyl methacrylate, polymethyl acrylate and cellulose, copolymers derived from these resins, photoconductive resins such as poly-N-vinylcarbazole and polysilane and conductive resins such as polythiophene and polypyrrol. Examples of the additive include antioxidants, ultraviolet light absorbents and plasticizers.

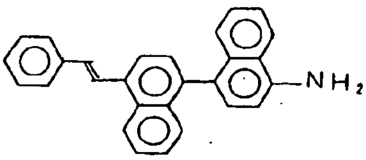
[0043] As described above, by using the materials of the present invention for the light emitting layer of the organic EL device, practically sufficient luminance can be obtained under application of a low voltage. Therefore, the organic EL device exhibiting a high efficiency of light emission and having a long life due to suppressed degradation and excellent heat resistance can be obtained.

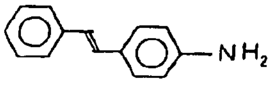
[0044] The organic EL device of the present invention can be used for a planar light emitting member such as a flat panel display of wall televisions, a back light for copiers, printers and liquid crystal displays, a light source of instruments, display panels and a marker light.

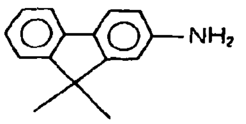
[0045] The materials of the present invention can be used not only for the organic EL devices but also in the field of electronic photosensitive materials, opto-electric conversion devices, solar batteries and image sensors.

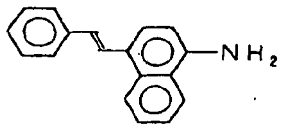
[0046] Examples of the primary amine represented by general formula [15] which is used in the process for producing a material for organic EL devices of the present invention include primary alkylamines such as methylamine, ethylamine, n-propylamine, isopropylamine, n-butylamine, isobutylamine, sec-butylamine, tert-butylamine, n-amylamine, isoamylamine, tert-amylamine, cyclohexylamine, n-hexylamine, heptylamine, 2-aminoheptane, 3-aminoheptane, octylamine, nonylamine, decylamine, undecylamine, dodecylamine, tridecylamine, 1-tetradecylamine, pentadecylamine, 1-hexadecylamine and octadecylamine; primary alkyldiamines such as ethylenediamine, 1,2-diaminopropane, 1,3-diaminopropane and 1,4-diaminobutane; arylamines such as aniline, o-fluoroaniline, m-fluoroaniline, p-fluoroaniline, o-toluidine, m-toluidine, p-toluidine, o-anisidine, m-anisidine, p-anisidine, 1-naphthylamine, 2-naphthylamine, 1-aminoanthracene, 2-aminoanthracene, 2-aminobiphenyl, 4-aminobiphenyl, 9-aminophenanthrene, 2-trifluoromethyltoluidine, 3-trifluoromethyltoluidine and 4-trifluoromethyltoluidine; aryldiamines such as o-phenylenediamine, m-phenylenediamine, p-phenylenediamine, fluorenediamine and 1,8-naphthalenediamine; and the following compounds:

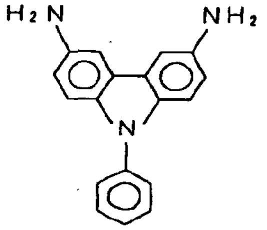


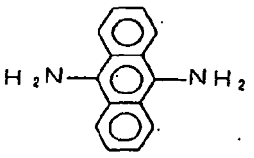


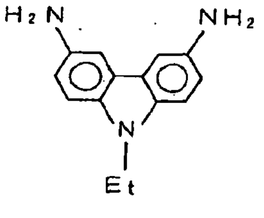




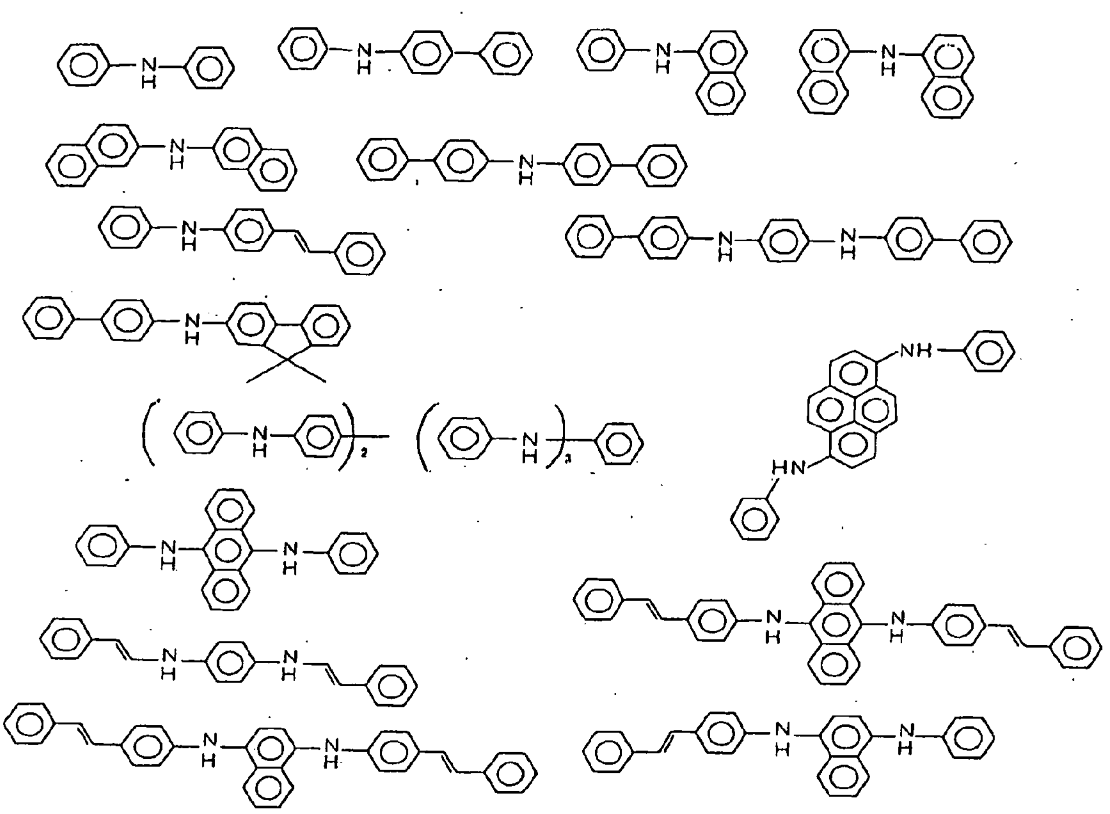






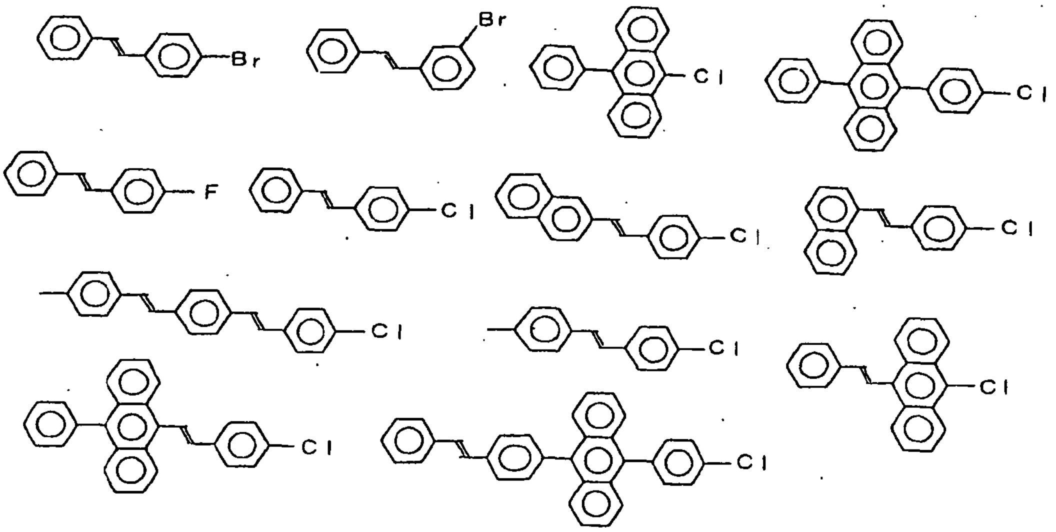


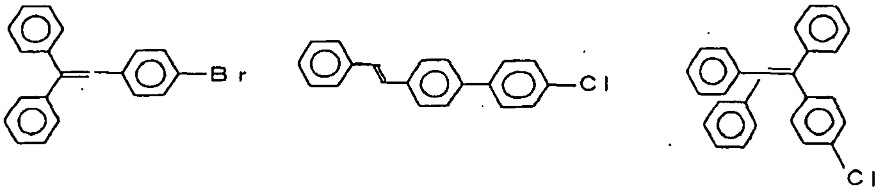
[0047] Examples of the secondary amine represented by general formula [15] include the following compounds:



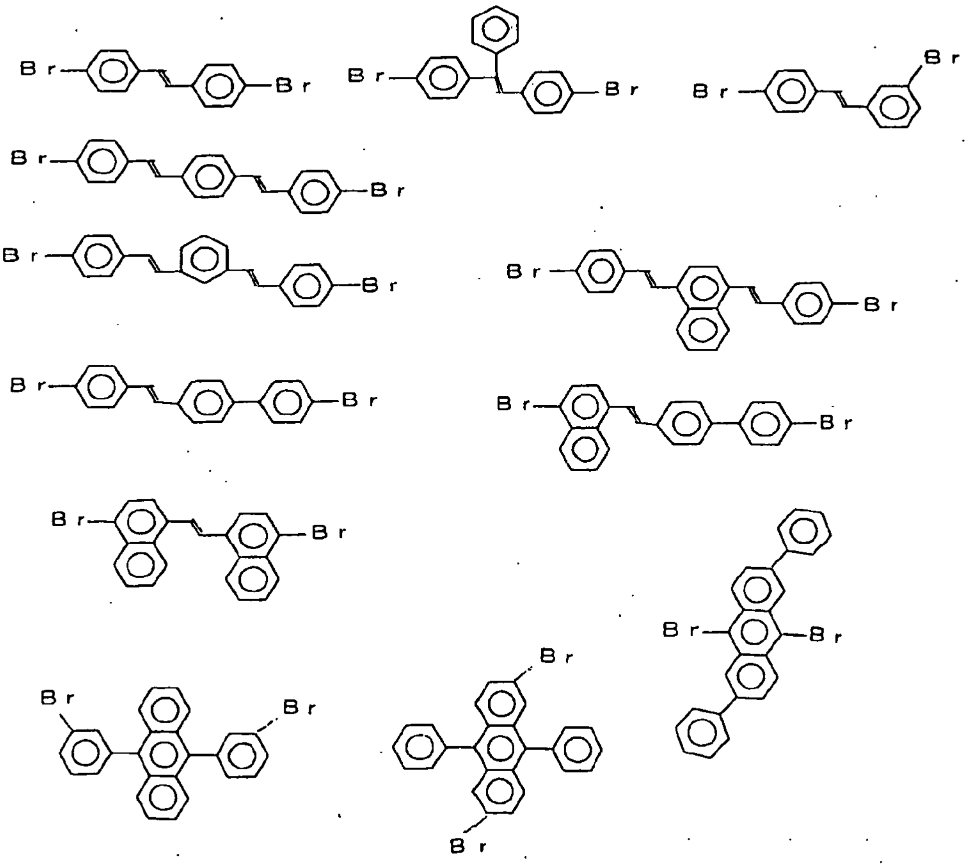
[0048] The aryl halide represented by general formula [16] is not particularly limited. The group represented by Ar is, in general, an alkyl group having 1 to 18 carbon atoms or a substituted or unsubstituted aryl group having 6 to 22 carbon atoms. The aromatic ring may have substituents. In the present invention, the aryl group include hydrocarbon groups having condensed rings.

[0049] Examples of the aryl halide include aryl bromides such as bromobenzene, o-bromoanisole, m-bromoanisole, p-bromoanisole, o-bromotoluene, m-bromotoluene, p-bromotoluene, o-bromophenol, m-bromophenol, p-bromophenol, 2-bromobenzotrifluoride, 3-bromobenzotrifluoride, 4-bromobenzenetrifluoride, 1-bromo-2,4-dimethoxybenzene, 1-bromo-2,5-dimethoxybenzene, 2-bromophenetyl alcohol, 3-bromophenetyl alcohol, 4-bromophenetyl alcohol, 5-bromo-1,2,4-trimethylbenzene, 2-bromo-m-xylene, 2-bromo-p-xylene, 3-bromo-o-xylene, 4-bromo-o-xylene, 4-bromo-m-xylene, 5-bromo-m-xylene, 1-bromo-3-(trifluoromethoxy)benzene, 1-bromo-4-(trifluoromethoxy)benzene, 2-bromobiphenyl, 3-bromobiphenyl, 4-bromobiphenyl, 4-bromo-1,2-(methylenedioxy)benzene, 1-bromonaphthalene, 2-bromonaphthalene, 1-bromo-2-methylnaphthalene and 1-bromo-4-methylnaphthalene; aryl chlorides such as chlorobenzene, o-chloroanisole, m-chloroanisole, p-chloroanisole, o-chlorotoluene, m-chlorotoluene, p-chlorotoluene, o-chlorophenol, m-chlorophenol, p-chlorophenol, 2-chlorobenzotrifluoride, 3-chlorobenzotrifluoride, 4-chlorobenzenetrifluoride, 1-chloro-2,4-dimethoxybenzene, 1-chloro-2,5-dimethoxybenzene, 2-chlorophenetyl alcohol, 3-chlorophenetyl alcohol, 4-chlorophenetyl alcohol, 5-chloro-1,2,4-trimethylbenzene, 2-chloro-m-xylene, 2-chloro-p-xylene, 3-chloro-o-xylene, 4-chloro-o-xylene, 4-chloro-m-xylene, 5-chloro-m-xylene, 1-chloro-3-(trifluoromethoxy)benzene, 1-chloro-4-(trifluoromethoxy) benzene, 2-chlorobiphenyl, 3-chlorobiphenyl, 4-chlorobiphenyl, 1-chloronaphthalene, 2-chloronaphthalene, 1-chloro-2-methylnaphthalene and 1-chloro-4-methylnaphthalene; aryl iodides such as iodobenzene, o-iodoanisole, m-iodoanisole, p-iodoanisole, o-iodotoluene, m-iodotoluene, p-iodotoluene, o-iodophenol, m-iodophenol, p-iodophenol, 2-iodobenzotrifluoride, 3-iodobenzotrifluoride, 4-iodobenzenetrifluoride, 1-iodo-2,4-dimethoxybenzene, 1-iodo-2,5-dimethoxybenzene, 2-iodophenetyl alcohol, 3-iodophenetyl alcohol, 4-iodophenetyl alcohol, 5-iodo-1,2,4-trimethylbenzene, 2-iodo-m-xylene, 2-iodo-p-xylene, 3-iodo-o-xylene, 4-iodo-o-xylene, 4-iodo-m-xylene, 5-iodo-m-xylene, 1-iodo-3-(trifluoromethoxy)benzene, 1-iodo-4-(trifluoromethoxy)benzene, 2-iodobiphenyl, 3-iodobiphenyl, 4-iodobiphenyl, 1-iodonaphthalene, 2-iodonaphthalene, 1-iodo-2-methylnaphthalene and 1-iodo-4-methylnaphthalene; aryl fluorides such as fluorobenzene, o-fluoroanisole, m-fluoroanisole, p-fluoroanisole, o-fluorotoluene, m-fluorotoluene, p-fluorotoluene, o-fluorophenol, m-fluorophenol, p-fluorophenol, 2-fluorobenzotrifluoride, 3-fluorobenzotrifluoride, 4-fluorobenzenetrifluoride, 1-fluoro-2,4-dimethoxybenzene, 1-fluoro-2,5-dimethoxybenzene, 2-fluorophenetyl alcohol, 3-fluorophenetyl alcohol, 4-fluorophenetyl alcohol, 5-fluoro-1,2,4-trimethylbenzene, 2-fluoro-m-xylene, 2-fluoro-p-xylene, 3-fluoro-o-xylene, 4-fluoro-o-xylene, 4-fluoro-m-xylene, 5-fluoro-m-xylene, 1-fluoro-3-(trifluoromethoxy)benzene, 1-fluoro-4-(trifluoromethoxy)benzene, 2-fluorobiphenyl, 3-fluorobiphenyl, 4-fluorobiphenyl, 4-fluoro-1,2-(methylenedioxy)benzene, 1-fluoronaphthalene, 2-fluoronaphthalene, 1-fluoro-2-methylnaphthalene and 1-fluoro-4-methylnaphthalene; and the following compounds:





[0050] Aryl halides having 2 or more halogen atoms and preferably 2 or 3 halogen atoms can also be used as long as the object of the present invention is not adversely affected. Examples of the aryl halide having 2 or more halogen atoms include 1,2-dibromobenzene, 1,3-dibromobenzene, 1,4-dibromobenzene, 9,10-dibromoanthracene, 9,10-dichloroanthracene, 4,4'-dibromobiphenyl, 4,4'-dichlorobiphenyl, 4,4'-diiodobiphenyl, 1-bromo-2-fluorobenzene, 1-bromo-3-fluorobenzene, 1-bromo-4-fluorobenzene, 2-bromochlorobenzene, 3-bromochlorobenzene, 4-bromochlorobenzene, 2-bromo-5-chlorotoluene, 3-bromo-4-chlorobenzotrifluoride, 5-bromo-2-chlorobenzotrifluoride, 1-bromo-2,3-dichlorobenzene, 1-bromo-2,6-dichlorobenzene, 1-bromo-3,5-dichlorobenzene, 2-bromo-4-fluorotoluene, 2-bromo-5-fluorotoluene, 3-bromo-4-fluorotoluene, 4-bromo-2-fluorotoluene, 4-bromo-3-fluorotoluene, tris(4-bromophenyl)amine, 1,3,5-tribromobenzene and the following compounds:



[0051] In the process for producing materials for organic EL devices of the present invention, the method of addition of the aryl halide is not particularly limited. For example, two different types of aryl halides may be mixed with a primary amine before starting the reaction and the reaction may be conducted using the obtained mixture. Alternatively, a primary amine may be reacted with one of two types of aryl halides. Then, the obtained secondary amine may be added to the other aryl halide and the reaction is conducted. The latter method in which different aryl halides are added successively is preferable because a tertiary amine can be produced more selectively.

[0052] The amount of the added aryl halide is not particularly limited. When the two types of aryl halides are added to the primary amine simultaneously, it is suitable that the amount of the aryl halide is in the range of 0.5 to 10 moles per 1 mole of the primary amine. From the standpoint of economy and easier treatments after the reaction such as separation of the unreacted aryl halide, it is preferable that the amount of the aryl halide is in the range of 0.7 to 5 moles per 1 mole of the primary amine. When the two types of aryl halides are added successively to the primary amine, the aryl halide which is added first is added to the reaction system in an amount in the range of 0.5 to 1.5 moles per 1 mole of the amino group in the primary amine. From the standpoint of improving the selectivity of the tertiary amine of the object compound, it is preferable that the above aryl halide is added to the reaction system in an amount of 0. 9 to 1.1 mole per 1 mole of the amino group in the primary amine.

[0053] The aryl halide which is added after preparation of the secondary amine is added in an amount of 0.1 to 10 mole per 1 mole of the amino group in the primary amine used as the starting material. To prevent complicated operations in separation of the unreacted aryl halide and the unreacted secondary amine after the reaction, it is preferable that the aryl halide is added in an amount of 0.9 to 5 mole per 1 mole of the amino group in the primary amine used as the starting material.

[0054] The palladium compound used as the catalyst component is not particularly limited as long as it is a compound of palladium. Examples of the palladium compound include compounds of tetravalent palladium such as sodium hexachloropalladadate(IV) tetrahydrate and potassium hexachloropalladate(IV); compounds of divalent palladium such as palladium(II) chloride, palladium(II) bromide, palladium(II) acetate, palladium acetylacetonate(II), dichlorobis-(benzonitrile)palladium(II), dichlorobis(acetonitrile)palladium(II), dichloro(bis(diphenylphosphino)ethane)palladium(II), dichlorobis-(triphenylphosphine)palladium(II), dichlorotetraamminepalladium(II), dichloro(cycloocta-1,5-diene)palladium(II) and palladium trifluoroacetate(II); and compounds of zero-valent palladium such as tris(dibenzylideneacetone) dipalladium (0) ($Pd_2(dba)_3$), chloroform complex of tris(dibenzylideneacetone) dipalladium(0), tetrakis(triphenylphosphine)palladium (0) and bis(bis(diphenylphosphino)ethane-palladium(0). In the process, the amount of the palladium compound is not particularly limited. The amount of the palladium compound is 0.00001 to 20.0% by mole as the amount of palladium per 1 mol of the primary amine. The tertiary amine can be synthesized with a high selectivity when the amount of the palladium compound is in the above range. Since the palladium compound is expensive, it is preferable that the amount of the palladium compound is 0.001 to 5.0 mole as the amount of palladium per 1 mole of the primary amine.

[0055] In the process, the trialkylphosphine compound used as the catalyst component is not particularly limited. Examples of the trialkylphosphine compound include triethylphosphine, tricyclohexylphosphine, triisopropylphosphine, tri-n-butylphosphine, triisobutylphosphine, tri-sec-butylphosphine and tri-tert-butylphosphine. Among these.compounds, tri-tert-butylphosphine is preferable because of the high reaction activity. The triarylphosphine compound is not particularly limited. Examples of the triarylphosphine include triphenylphosphine, benzyldiphenylphosphine, tri-o-toluylphosphine, trim-toluylphosphine and tri-p-toluylphosphine. Among these compounds, triphenylphosphine and tri-o-toluylphosphine are preferable. The diphosphine compound is not particularly limited. Examples of the diphosphine compound include bis(dimethylphosphino)methane, bis(dimethylphosphino)ethane, bis(dicyclohexylphosphino)methane, bis(dicyclohexylphosphino)ethane, bis(diphenylphosphino)ethane, 1,3-bis(diphenylphosphino)propane, 1,4-bis(diphenylphosphino)butane, bis(diphenylphosphino)ferrocene, (R)-2,2'-bis(diphenylphosphino)-1,1'-binaphthyl((R)-BINAP), (S)-2,2'-bis(diphenylphosphino)-1,1'-binaphthyl-((S)-BINAP), 2,2'-bis(diphenylphosphino)-1,1'-bisnaphthyl((±)-BINAP), 2S,3S-bis (diphenylphosphino)butane((S,S)-CHIRAPHOS), 2R,3R-bis(diphenylphosphino)butane((R,R)-CHIRAPHOS), 2,3-bis (diphenylphosphino)butane(±)-CHIRAPHOS), (R)-2,2'-bis(di-p-toluylphosphino)-1,1-binaphthyl((R)-Tol-BINAP), (S)-2,2'-bis(di-p-toluylphosphino)-1,1'-binaphthyl((S)-Tol-BINAP), 2,2'-bis(di-p-toluylphosphino)-1,1'-bisnaphthyl((±)-Tol-BINAP), 4R,5R-bis(diphenylphosphinomethyl)-2,2-dimethyl-1,3-dioxorane((R,R)-DIOP), 4S,5S-bis(diphenylphosphinomethyl)-2,2-dimethyl-1,3-dioxorane(S,S)-DIOP), 4,5-bis(diphenyl-phosphinomethyl)-2,2-dimethyl-1, 3-dixorane((±)-DIOP), N,N'-dimethyl-(S)-1-[(R)-1',2-bis(diphenylphosphino)ferrocenyl]ethylamine((S),(R)-BPPFA), N,N'-dimethyl-(R)-1-[(S)-1',2-bis(diphenylphosphino)ferrocenyl]-ethylamine((R),(S)-BPPFA) and N,N'-dimethyl-1-[1',2-bis(diphenyl-phosphino)ferrocenyl]ethylamine((±)-BPPFA). Among these compounds, bis(diphenylphosphino)ethane, 1,3-bis(diphenylphosphino)propane, bis(diphenylphosphino)ferrocene and BINAPs are preferable. BINAPs may be either optically active compounds or racemic compounds.

[0056] The amounts of the trialkylphosphine compound, the triphenylphosphine compound and the diphosphine compound are 0.01 to 10,000 mole per 1 mole of the palladium compound. As long as the amounts are in this range, the selectivity of the arylamine does not change. However, it is preferable that the amount is 0.1 to 10 mole per 1 mol of the palladium compound since the phosphine compounds are expensive.

[0057] In the process, the palladium compound and the phosphine compound are the essential components of the catalyst. The combination of these components is added to the reaction system as the catalyst. As the method of addition of the components, the two components may be added to the reaction system separately or in the form of a complex which is prepared in advance.

[0058] The base which can be used in the present reaction is not particularly limited and can be selected from inorganic bases such as sodium carbonate and potassium carbonate and alkali metal alkoxides and organic bases such as tertiary amines. Preferable examples of the base include alkali metal alkoxides such as sodium mothoxide, sodium ethoxide, potassium methoxide, potassium ethoxide, lithium tert-butoxide, sodium tert-butoxide, potassium tert-butoxide and cesium carbonate (Cs_2CO_3). The base may be added into the reaction field without any treatment. Alternatively, the base may be prepared from an alkali metal, a hydrogenated alkali metal or a alkali metal hydroxide and an alcohol at the place of reaction and used in the reaction field.

[0059] The amount of the base is not particularly limited. It is preferable that the amount is 0.5 mole or more per 1 mole of the halogen atom in the two different types of aryl halides which are added to the reaction system. When the amount of the base is less than 0.5 mol, the activity of the reaction decreases and the yield of the arylamine decreases. Therefore, such an amount is not preferable. When the base is added in a great excess amount, the yield of the arylamine does not change and, on the other hand, treatments after the reaction become complicated. Therefore, it is more preferable that the amount is 1.0 mole or more and less than 5 mole per 1 mole of the halogen atom.

[0060] The reaction in the process is conducted, in general, in the presence of an inert solvent. The solvent is not particularly limited as long as the solvent does not adversely affect the reaction much. Examples of the solvent include aromatic hydrocarbon solvents such as benzene, toluene and xylene, ether solvents such as diethyl ether, tetrahydrofuran and dioxane, acetonitrile, dimethylformamide, dimethylsulfoxide and hexamethylphosphotriamide. Aromatic hydrocarbon solvents such as benzene, toluene and xylene are preferable.

[0061] It is preferable that the process is conducted under the ordinary pressure in an atmosphere of an inert gas such as nitrogen and argon. The process can be conducted also under an added pressure.

[0062] In the process, the temperature of the reaction can be selected in the range of 20 to 300°C and preferably in the range of 50. to 200°C. The time of the reaction can be selected in the range of several minutes to 72 hours.

[0063] The process in which the arylamine compound is obtained in the presence of the catalyst comprising the phosphine compound and the palladium compound and the base is specifically described in Synthesis Examples.

[0064] The present invention will be described more specifically with reference to examples in the following. However, the present invention is not limited to the examples.

Synthesis Example 1 (Compound (100))

Synthesis of Intermediate Compound H

[0065] In a 1 liter three-necked flask equipped with a condenser, 22.7 g (0.1 mole) of 4-bromophthalic anhydride and 42.4 g (0.4 mole) of sodium carbonate were suspended in 300 ml of water and the components were dissolved by heating at 60°C under an argon stream. After the mixture was dissolved, the resulting mixture was cooled to the room temperature. To the cooled mixture, 18.3 g (0.15 mole) of phenylboric acid and 0.7 g (3% by mole) of palladium acetate were added and the obtained mixture was stirred at the room temperature for one night. After the reaction was completed, separated crystals were dissolved by adding water. After the catalyst was removed by filtration, crystals were precipitated by adding concentrated hydrochloric acid. The crystals were separated by filtration and washed with water. The obtained crystals was dissolved in ethyl acetate and the organic layer was extracted. The extract was dried with magnesium sulfate and concentrated in vacuo using a rotary evaporator and 23.7 g (the yield: 98%) of Intermediate Compound H of the object compound was obtained.

Synthesis of Intermediate Compound I

[0066] In a 500 ml flask having an egg plant shape and equipped with a condenser, 23.7 g (98 mmole) of Intermediate Compound H and 200 ml of acetic anhydride were placed and the resulting mixture was stirred at 80°C for 3 hours. After the reaction was completed, acetic anhydride in an excess amount was removed by distillation and 22 g (the yield: 10%) of Intermediate Compound I of the object compound was obtained.

Synthesis of Intermediate Compound J

[0067] In a 500 ml three-necked flask equipped with a condenser, 7.7 g (50 mmole) of biphenyl, 13.4 g (0.1 mole) of anhydrous aluminum chloride and 200 ml of 1,2-dichloroethane were placed under an argon stream and the resulting mixture was cooled to 0°C. To the cooled mixture, 22 g (98 mmole) of Intermediate Compound I was slowly added and the resulting mixture was stirred at 40°C for 2 hours. After the reaction was completed, ice water was added to the reaction mixture and the resulting mixture was extracted with chloroform. The extract was dried with magnesium sulfate and concentrated in vacuo using a rotary evaporator and 19.0 g (the yield: 100%) of Intermediate Compound J of the object compound was obtained.

Synthesis of Intermediate Compound K

[0068] In a 500 ml flask having an egg plant shape and equipped with a condenser, 200 ml of polyphosphoric acid was placed and heated to 150°C. Then, 19 g (50 mmole) of Intermediate Compound J was added in small portions and the resulting mixture was stirred at the same temperature for 3 hours. After the reaction was completed, ice water was added to the reaction mixture and the resulting mixture was extracted with chloroform. The extract was dried with magnesium sulfate and concentrated in vacuo using a rotary evaporator. The obtained crude crystals were purified in accordance with the column chromatography (silica gel, chloroform/methanol=99/1) and 19 g (the yield: 55%) of Intermediate Compound K of the object compound was obtained.

Synthesis of Intermediate Compound L

[0069] In a 500 ml flask having an egg plant shape and equipped with a condenser, 19.0 g (28 mmole) of Intermediate Compound K, 0.19 g (1 mmole) of tin chloride, 100 ml of acetic acid and 50 ml of concentrated hydrochloric acid were placed under an argon stream and the resulting mixture was heated under refluxing for 2 hours. After the reaction was completed, the reaction mixture was cooled with ice water and precipitated crystals were separated, washed with water to give 19 g (the yield: 100%) of Intermediate Compound L of the object compound.

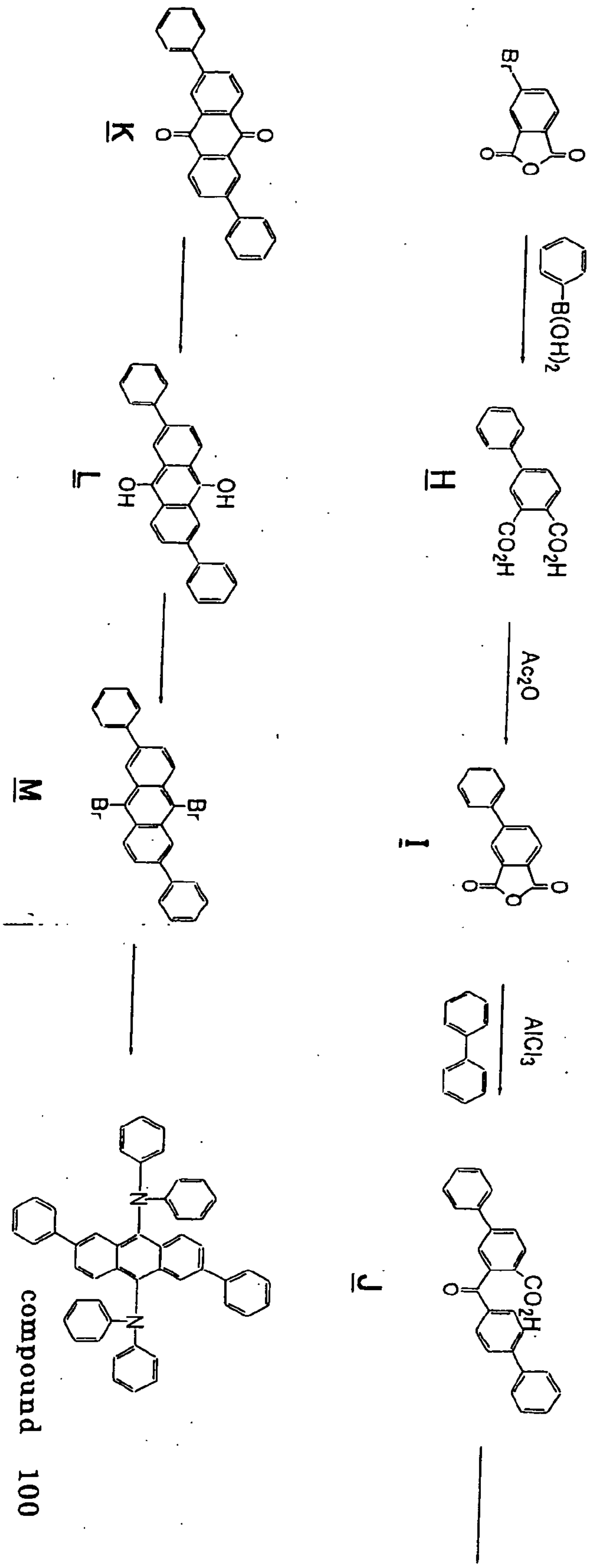
Synthesis of Intermediate Compound M

[0070] In a 500 ml three-necked flask equipped with a condenser, 19.0 g (28 mmole) of Intermediate Compound L, 16 g (60 mmole) of triphenylphosphine and 200 ml of dimethylformamide were placed under an argon stream. To this was added 9.6 g (60 mmole) of iodine dissolved in 50 ml of dimethylformamide slowly dropwise and the resulting mixture was stirred under heating at 200°C for 8 hours. After the reaction was completed, the reaction mixture was cooled with ice water and precipitated crystals were separated. The obtained crystals were washed with water and methanol and 6.7 g (the yield: 50%) of Intermediate Compound M of the object compound was obtained.

Synthesis of Compound (100)

[0071] In a 200 ml three-necked flask equipped with a condenser, 4.9 g (10 mmole) of Intermediate Compound M, 5.1 g (30 mmole) of diphenylamine, 0.14 g (1.5% by mole) of tris(dibenzylideneacetone)-dipalladium, 0.91 g (3% by mole) of tri-o-toluylphosphine, 2.9 g (30 mmole) of sodium t-butoxide and 50 ml of dry toluene were placed under an argon stream. The resulting mixture was stirred overnight under heating at 100°C. After the reaction was completed, precipitated crystals were separated by filtration and washed with 100 ml of methanol and 4.0 g of yellow powder was obtained. The obtained powder was identified to be Compound (100) by the measurements in accordance with NMR, IR and FD-MS (the yield: 60%).

[0072] The chemical structures of Intermediate Compounds and the route of synthesis of Compound (100) are shown in the following.



compound 100

Synthesis Example 2 (Compound (101))

Synthesis of Intermediate Compound N

[0073] In a 500 ml flask having an egg plant shape and equipped with a condenser, 12 g (50 mmole) of 2,6-dihydroxyanthraquinone, 42.5 g (0.3 mole) of methyl iodide, 17 g (0.3 mole) of potassium hydroxide and 200 ml of dimethylsulfoxide were placed under an argon stream and the resulting mixture was stirred at the room temperature for 2 hours. After the reaction was completed, precipitated crystals were separated by filtration. The obtained crystals were washed with 100 ml of methanol and 10.7 g (the yield: 80%) of Intermediate Compound N of the object compound was obtained.

Synthesis of Intermediate Compound O

[0074] In a 500 ml three-necked flask equipped with a condenser, 10.7 g (40 mmole) of Intermediate Compound N and 200 ml of dry tetrahydrofuran were placed under an argon stream and the resulting mixture was cooled to -40°C. To the cooled mixture, 53 ml (80 mmole) of a 1.5 M hexane solution of phenyllithium was added slowly dropwise. After the addition was completed, the reaction mixture was stirred at the room temperature for one night. After the reaction was completed, precipitated crystals were separated by filtration and washed with 100 ml of methanol and 100 ml of acetone. The obtained crude crystals of a diol was used in the following reaction without further purification.

[0075] In a 500 ml flask having an egg plant shape and equipped with a condenser, the crude crystals obtained above, 100 ml of a 57% aqueous solution of hydrogen iodide and 200 ml of acetic acid were placed and the resulting mixture was heated under refluxing for 3 hours. After the reaction was cooled to the room temperature, a small amount of hypophosphorous acid was added to quench hydrogen iodide in an excess amount. Precipitated crystals were separated by filtration and washed with 100 ml of water, 100 ml of methanol and 100 ml of acetone, successively, and 10.1 g (the yield: 70%) of Intermediate Compound O of the object compound was obtained.

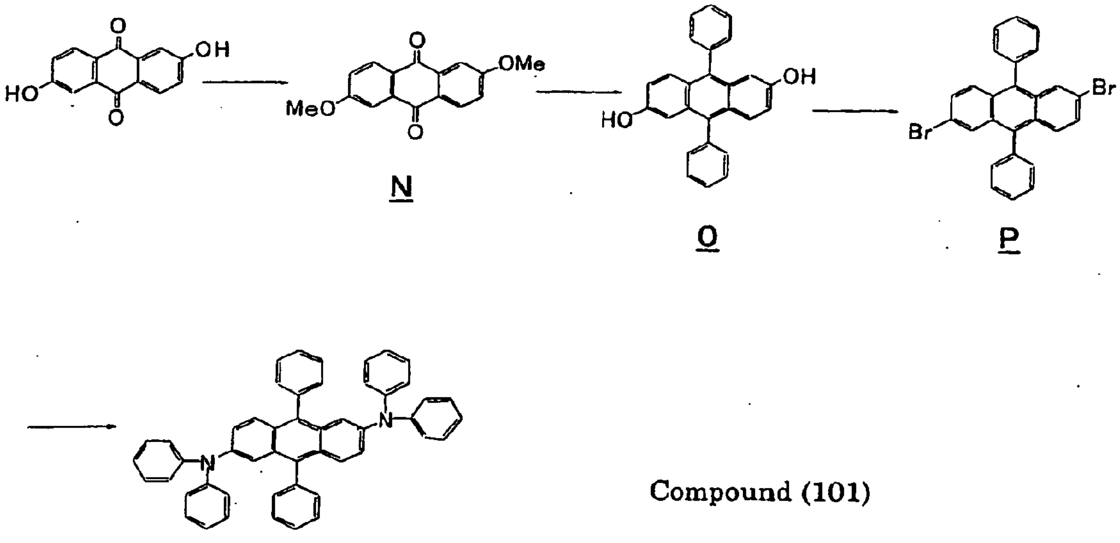
Synthesis of Intermediate Compound P

[0076] In a 500 ml flask having an egg plant shape and equipped with a condenser, 10.1 g (28 mmole) of Intermediate Compound O, 7.9 g (30 mmole) of triphenylphosphine and 200 ml of dimethylformamide were placed under an argon stream. To the resulting mixture, 4.8 g (30 mmole) of bromine dissolved in 50 ml of dimethylformamide was slowly added dropwise and the obtained mixture was stirred under heating at 200°C for 8 hours. After the reaction was completed, the reaction mixture was cooled with ice water and precipitated crystals were separated by filtration. The obtained crystals were washed with water and methanol and 8.2 g (the yield: 60%) of Intermediate Compound P of the object compound was obtained.

Synthesis of Compound (101)

[0077] In a 200 ml three-necked flask equipped with a condenser, 4.9 g (30 mmole) of Intermediate Compound P, 5.1 g (30 mmole) of diphenylamine, 0.14 g (1.5% by mole) of tris(dibenzylideneacetone)-dipalladium, 0.91 g (3% by mole) of tri-o-toluylphosphine, 2.9 g (30 mmole) of sodium t-butoxide and 50 ml of dry toluene were placed under an argon stream. The resulting mixture was stirred overnight under heating at 100°C. After the reaction was completed, precipitated crystals were separated by filtration and washed with 100 ml of methanol and 4.0 g of yellow powder was obtained. The obtained powder was identified to be Compound (101) by the measurements in accordance with NMR, IR and FD-MS (the yield: 60%).

[0078] The chemical structures of Intermediate Compounds and the route of synthesis of Compound (101) are shown in the following.



Compound (101)

Synthesis Example 3 (Compound (93)) (not part of the invention)

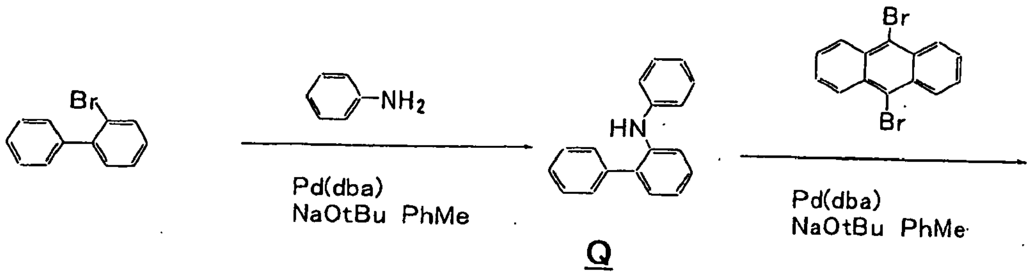
Synthesis of Intermediate Compound Q

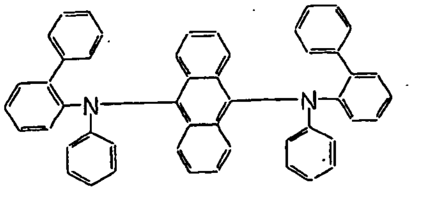
[0079] In a 300 ml three-necked flask equipped with a condenser, 11.7 g (50 mmole) of 2-bromobiphenyl, 19 g (0.2 mole) of aniline, 0.69 g (1.5% by mole) of tris(dibenzylideneacetone)dipalladium, 0.46 g (3% by mole) of tri-o-toluylphosphine, 7.2 g (75 mmole) of sodium t-butoxide and 100 ml of dry toluene were placed under an argon stream. The resulting mixture was stirred overnight under heating at 100°C. After the reaction was completed, precipitated crystals were separated by filtration and washed with 100 ml of methanol. The obtained crude crystals were recrystallized from 50 ml of ethyl acetate and 9.8 g (the yield: 80%) of Intermediate Compound Q of the object compound was obtained.

Synthesis of Compound (93)

[0080] In a 200 ml three-necked flask equipped with a condenser, 2.4 g (10 mmole) of 9,10-dibromoanthracene, 7.4 g (30 mmole) of Intermediate Compound Q, 0.14 g (1.5% by mole) of tris(dibenzylideneacetone)dipalladium, 0.91 g (3% by mole) of tri-o-toluylphosphine, 2.9 g (30 mmole) of sodium t-butoxide and 50 ml of dry toluene were placed under an argon stream. The resulting mixture was stirred overnight under heating at 100°C. After the reaction was completed, precipitated crystals were separated by filtration and washed with 100 ml of methanol and 4.3 g of yellow powder was obtained. The obtained powder was identified to be Compound (93) by the measurements in accordance with NMR, IR and FD-MS (the yield: 65%).

[0081] The chemical structure of-Intermediate Compound and the route of synthesis of Compound (93) are shown in the following.





Compound (93)

Synthesis Example 4 (Compound (95)) (not part of the invention)

Synthesis of Intermediate Compound R

[0082] In a 1 liter three-necked flask equipped with a condenser, 34 g (0.2 mole) of 3-phenylphenol, 58 g (0.22 mmole) of triphenylphosphine and 300 ml of dimethylformamide were placed under an argon stream. To the resulting mixture, 35 g (0.22 mmole) of bromine dissolved in 100 ml of dimethylformamide was slowly added dropwise and the obtained mixture was stirred at 200°C for 8 hours. After the reaction was completed, the reaction mixture was cooled with ice water and precipitated crystals were separated by filtration. The obtained crystals were washed with water and methanol and 37 g (the yield: 80%) of Intermediate Compound R of the object compound was obtained.

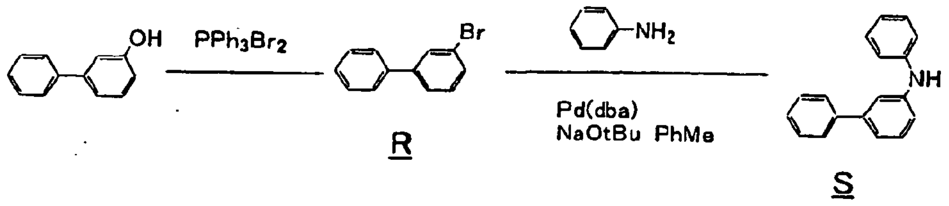
Synthesis of Intermediate Compound S

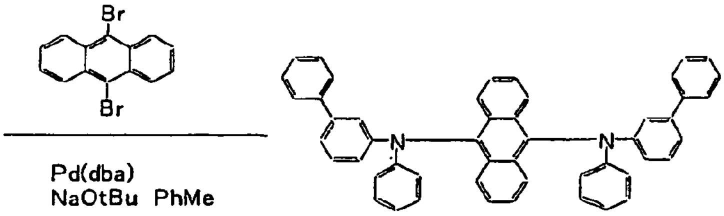
[0083] In a 300 ml three-necked flask equipped with a condenser, 19 g (0.2 mmole) of aniline, 0.69 g (1.5% by mole) of tris(dibenzylideneacetone)dipalladium, 0.46 g (3% by mole) of tri-o-toluylphosphine, 7.2 g (75 mmole) of sodium t-butoxide and 100 ml of dry toluene were placed under an argon stream. The resulting mixture was stirred overnight under heating at 100°C. After the reaction was completed, precipitated crystals were separated by filtration and washed with 100 ml of methanol. The obtained crude crystals were recrystallized from 50 ml of ethyl acetate and 9.8 g (the yield: 80%) of Intermediate Compound S of the object compound was obtained.

Synthesis of Compound (95)

[0084] In a 200 ml three-necked flask equipped with a condenser, 2.4 g (10 mmole) of 9,10-dibromoanthracene, 7.4 g (30 mole) of Intermediate Compound S, 0.14 g (1.5% by mole) of tris(dibenzylideneacetone)dipalladium, 0.91 g (3% by mole) of tri-o-toluylphosphine, 2.9 g (30 mmole) of sodium t-butoxide and 50 ml of dry toluene were placed under an argon stream. The resulting mixture was stirred overnight under heating at 100°C. After the reaction was completed, precipitated crystals were separated by filtration and washed with 100 ml of methanol and 4.2 g of yellow powder was obtained. The obtained powder was identified to be Compound (95) by the measurements in accordance with NMR, IR and FD-MS (the yield: 70%).

[0085] The chemical structures of Intermediate Compounds and the route of synthesis of Compound (95) are shown in the following.





Compound (95)

Synthesis Example 5 (Compound (104)) (not part of the invention)

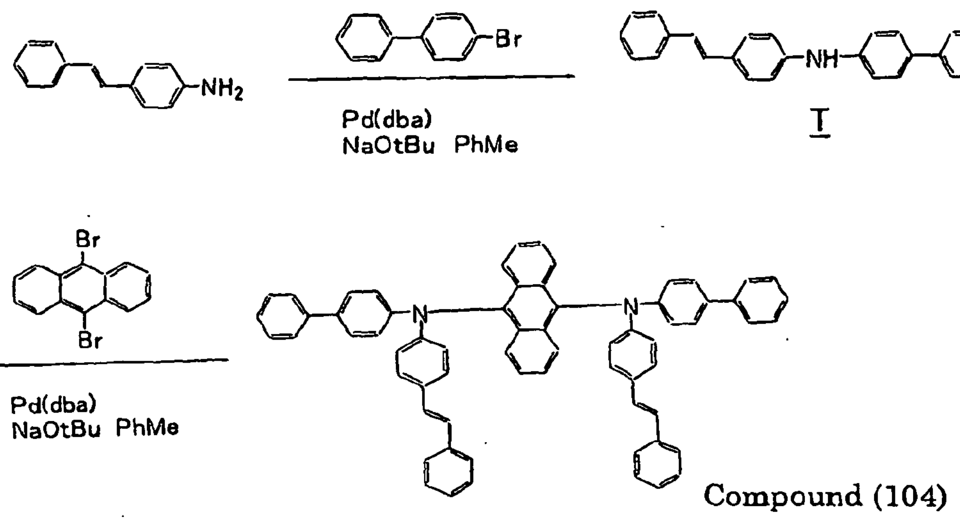
Synthesis of Intermediate Compound T

[0086] In a 300 ml three-necked flask equipped with a condenser, 23 g (0.1 mole) of 4-bromobiphenyl, 9.8 g (50 mmole) of aminostilbene, 0.69 g (1.5% by mole) of tris(dibenzylideneacetone)dipalladium, 0.46 g (3% by mole) of tri-o-toluylphosphine, 7.2 g (75 mmole) of sodium t-butoxide and 100 ml of dry toluene were placed under an argon stream. The resulting mixture was stirred overnight under heating at 100°C. After the reaction was completed, precipitated crystals were separated by filtration and washed with 100 ml of methanol. The obtained crude crystals were recrystallized from 50 ml of ethyl acetate and 13.9 g (the yield: 80%) of Intermediate Compound T of the object compound was obtained.

Synthesis of Compound (104)

[0087] Into a 200 ml three-necked flask equipped with a condenser, 2.4 g (10 mmole) of 9,10-dibromoanthracene, 7.4 g (30 mmole) of Intermediate Compound T, 0.14 g (1.5% by mole) of tris(dibenzylideneacetone)dipalladium, 0.91 g (3% by mole) of tri-o-toluylphosphine, 2.9 g (30 mmole) of sodium t-butoxide and 50 ml of dry toluene were placed under an argon stream. The resulting mixture was stirred overnight under heating at 100°C. After the reaction was completed, precipitated crystals were separated by filtration and washed with 100 ml of methanol and 4.5 g of yellow powder was obtained. The obtained powder was identified to be Compound (104) by the measurements in accordance with NMR, IR and FD-MS (the yield: 70%).

[0088] The chemical structure of Intermediate Compound and the route of synthesis of Compound (104) are shown in the following.



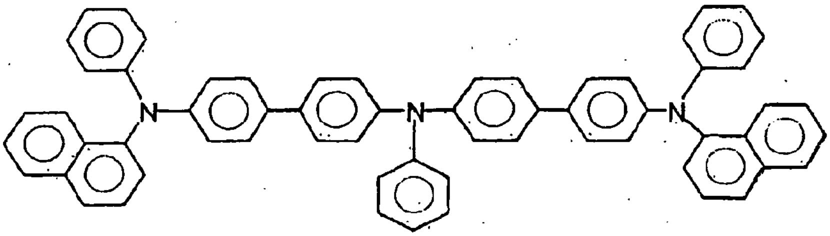
Compound (104)

Example 1

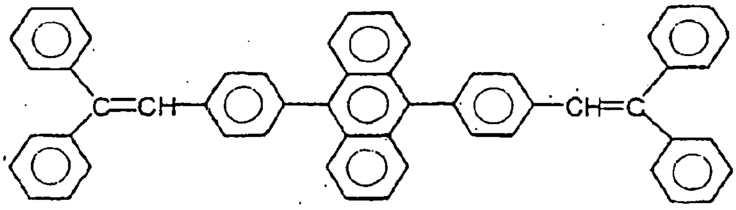
[0089] On a glass substrate having a size of 25 mm X 75 mm X 1.1 mm, a transparent anode of a film of indium tin oxide having a thickness of 100 nm was formed and cleaned for 10 minutes by using ultraviolet light and ozone in combination.

[0090] This glass substrate was placed into an apparatus for vacuum vapor deposition (manufactured by NIPPON SHINKUU GIJUTU Co., Ltd.) and the pressure was reduced to about 10^{-4} Pa. TPD74 described above was vapor deposited at a speed of 0.2 nm/second and a layer having a thickness of 60 nm was formed. Then, TPD78 having the structure shown below was vapor deposited at a speed of 0.2 nm/second and a layer having a thickness of 20 nm was formed.

[0091] On the layer formed above, DPVDPAN having the structure shown below and Compound (100) described above as the light emitting material were simultaneously vapor deposited and a light emitting layer having a thickness of 40 nm was formed. The speed of vapor deposition of DPVDPAN was 0.4 nm/second and the speed of vapor deposition of Compound (100) was 0.01 nm/second. On the layer formed above, Alq described above was vapor deposited at a speed of 0.2 nm/second. Finally, aluminum and lithium were vapor deposited simultaneously and a cathode having a thickness of 150 nm was formed. Thus, an organic EL device was obtained. The speed of vapor deposition of aluminum was 1 nm/second and the speed of vapor deposition of lithium was 0.004 nm/second.



(TPD78)



(DPVDPAN)

[0092] The properties of the obtained organic EL device were evaluated. Luminance of emitted light at the voltage shown in Table 4 was measured and the efficiency of light emission was calculated. The color of emitted light was observed. The organic EL device was driven by a constant electric current under a nitrogen stream at an initial luminance of emitted light of 500 (cd/m^2) and the half life time which was the time before the luminance decreases to 250 (cd/m^2) was measured. The results are shown in Table 1.

Comparative Example 1

[0093] An organic EL devices was prepared in accordance with the same procedures as those conducted in Example 1 except that the diamine compound shown below was used as the light emitting material in place of Compound (100) and the properties were evaluated. The results are shown in Table 1.

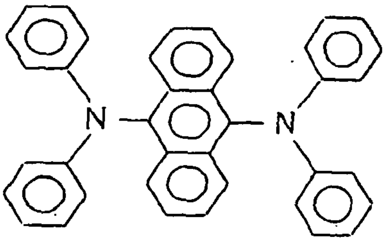


Table 1

	Voltage (V)	Luminance of emitted light (cd/m^2)	Efficiency of light emission (lm/W)	Half life time (hour)	Color of emitted light
Example 1	6.0	120	4.50	1800	green
Comparative Example 1	6.0	150	3.70	1200	green

[0094] As shown in Table 1, the organic EL devices of Example 1 in which the compound represented by general formula [9] of the present invention was used as the light emitting material or the hole transporting material exhibited more excellent luminance of emitted light and efficiencies of light emission and longer lives in comparison with the organic EL device of Comparative Example 1 in which the diamine compound was used.

INDUSTRIAL APPLICABILITY

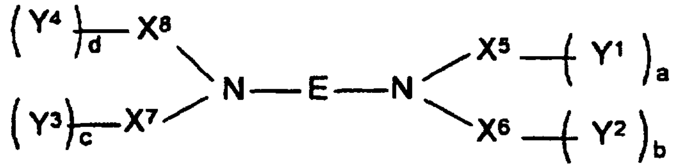
[0095] The organic EL devices of the present invention in which the materials for organic EL devices represented by general formula (9) described above are used as the light emitting material, the hole injecting material, the hole transporting material or the doping material exhibit luminances of light emission sufficient for practical use and high efficiencies of light emission under application of a low voltage, have long lives because the decrease in the properties after use for a long time is suppressed and show no deterioration in the properties in the environment of high temperatures due to excellent heat resistance.

[0096] By producing materials for organic EL devices in accordance with the process of the present invention, materials for organic EL devices exhibiting a high efficiency of light emission, having a long life, showing high activity and containing little impurities can be produced in a high yield.

Claims

1. A material for organic electroluminescence devices represented by following general formula [9]:

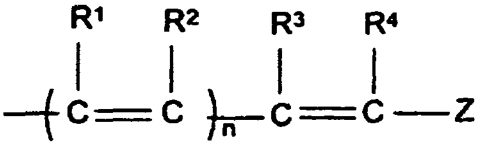
General formula [9]



(9)

wherein E represents a divalent group comprising an anthracene nucleus which is substituted with at least two aryl groups;
X^5 to X^8 each independently represent a substituted or unsubstituted arylene group having 6 to 20 carbon atoms, X^5 and X^6 may be bonded to each other, X^7 and X^8 may be bonded to each other;
Y^1 to Y^4 each independently represent an organic group represented by general formula [2];
a to d each represent an integer of 0 to 2 and,
general formula [2] being:

General formula [2]



$$-\left(\overset{R^1}{\overset{|}{C}}=\overset{R^2}{\overset{|}{C}}\right)_n-\overset{R^3}{\overset{|}{C}}=\overset{R^4}{\overset{|}{C}}-Z \qquad (2)$$

wherein R^1 to R^4 each independently represent hydrogen atom, a substituted or unsubstituted alkyl group having 1 to 20 carbon atoms, a substituted or unsubstituted aryl group having 6 to 20 carbon atoms, cyano group or form a triple bond by a linkage of R^1 and R^2 or R^3 and R^4, Z represents a substituted or unsubstituted aryl group having 6 to 20 carbon atoms and n represents 0 or 1.

2. A material for organic electroluminescence devices according to Claim 1, which is a light emitting material for organic electroluminescence devices.

3. An organic electroluminescence device which comprises a light emitting layer or a plurality of thin films of organic compounds comprising a light emitting layer disposed between a pair of electrodes, wherein at least one of the thin films of organic compounds is a layer comprising a material for organic electroluminescence devices described in Claim 1 or 2.

4. An organic electroluminescence device which comprises a light emitting layer or a plurality of thin films of organic compounds comprising a light emitting layer disposed between a pair of electrodes, wherein a layer comprising a material for organic electroluminescence devices described in Claim 1 or 2 as at least one material selected from the group consisting of a hole injecting material, a hole transporting material and a doping material is disposed between the pair of electrodes.

5. An organic electroluminescence device which comprises a light emitting layer or a plurality of thin films of organic compounds comprising a light emitting layer disposed between a pair of electrodes, wherein the light emitting layer comprises 0.1 to 20% by weight of a material for organic luminescence devices described in Claim 1 or 2.

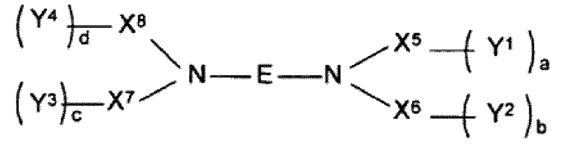
6. An organic electroluminescence device which comprises a light emitting layer or a plurality of thin films of organic compounds comprising a light emitting layer disposed between a pair of electrodes, wherein one or more materials selected from the group consisting of a hole injecting material, a hole transporting material and a doping material each independently comprise 0.1 to 20% by weight of a material for organic electroluminescence devices described in Claim 1 or 2.

7. An organic electroluminescence device which comprises a light emitting layer or a plurality of thin films of organic compounds comprising a light emitting layer disposed between a pair of electrodes, wherein the light emitting layer

is a layer comprising a stilbene derivative and a material for organic electroluminescence devices described in Claim 1 or 2.

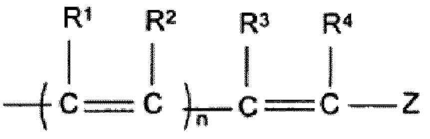
Patentansprüche

1. Material für organische Elektrolumineszenz-Vorrichtungen, dargestellt durch die folgende allgemeine Formel [9]:



(9)

wobei E eine divalente Gruppe darstellt, die einen Anthracenkern umfasst, welcher mit mindestens zwei Arylgruppen substituiert ist;
X^5 bis X^8 jeweils unabhängig eine substituierte oder unsubstituierte Arylengruppe mit 6 bis 20 Kohlenstoffatomen darstellen; X^5 und X^6 aneinander gebunden sein können, X^7 und X^8 aneinander gebunden sein können;
Y^1 bis Y^4 jeweils unabhängig eine durch die allgemeine Formel [2] dargestellte organische Gruppe darstellen;
a bis d jeweils eine ganze Zahl von 0 bis 2 darstellen und,
die allgemeine Formen [2]



$$-\left(\overset{R^1}{\overset{|}{C}}=\overset{R^2}{\overset{|}{C}}\right)_n-\overset{R^3}{\overset{|}{C}}=\overset{R^4}{\overset{|}{C}}-Z$$

(2)

Lautet, wobei R^1 bis R^4 jeweils unabhängig Wasserstoffatom, eine substituierte oder unsubstituierte Alkylgruppe mit 1 bis 20 Kohlenstoffatomen, eine substituierte oder unsubstituierte Arylgruppe mit 6 bis 20 Kohlenstoffatomen, Cyanogruppe darstellen oder eine Dreifachbindung durch eine Bindung von R^1 und R^2 oder R^3 und R^4 bilden, Kohlenstoffatomen darstellt und n 0 oder 1 darstellt.

2. Material für organische Elektrolumineszenz-Vorrichtungen nach Anspruch 1, welches ein lichtemittierendes Material für organische Elektrolumineszenz-Vorrichtungen ist.

3. Organische Elektrolumineszenz-Vorrichtung, welche eine lichtemittierende Schicht oder eine Mehrzahl dünner Filme aus organischen Verbindungen, die eine lichtemittierende Schicht umfassen, zwischen einem Paar von Elektroden angeordnet umfasst, wobei mindestens einer der dünnen Filme aus organischen Verbindungen eine Schicht ist, die ein Material für organische Elektrolumineszenz-Vorrichtungen, wie in Anspruch 1 oder 2 beschrieben, umfasst. Organische Elektrolumineszenz-Vorrichtung, welche eine lichtemittierende Schicht oder eine Mehrzahl dünner Filme aus organischen Verbindungen, die eine lichtemittierende Schicht umfassen, zwischen einem Paar von Elektroden angeordnet umfasst, wobei mindestens der dünnen Filme aus organischen Verbindungen eine Schicht ist, die ein Material für organische Elektrolumineszenz-Vorrichtungen, wie in Anspruch 1 oder 2 beschrieben, umfasst.

4. Organische Elektrolumineszenz-Vorrichtung, welche eine lichtemittierende Schicht oder eine Mehrzahl dünner Filme aus organischen Verbindungen, die eine lichtemittierende Schicht umfassen, zwischen einem Paar von Elektroden angeordnet umfasst, wobei eine Schicht, die ein Material für organische Elektrolumineszenz-Vorrichtungen, wie in Anspruch 1 oder 2 beschrieben, als mindestens ein Material umfasst, das ausgewählt wird der aus einem Lochinjektionsmaterial, einem Lochtransportmaterial und einem Dotierungsmaterial bestehenden Gruppe, zwischen dem Elektrodenpaar angeordnet ist.

5. Organische Elektrolumineszenz-Vorrichtung, welche eine lichtemittierende Schicht oder eine Mehrzahl dünner Filme

aus organischen Verbindungen, die eine lichtemittierende Schicht umfassen, zwischen einem Paar von Elektroden angeordnet umfasst, wobei die lichtemittierende Schicht 0.1 bis 20 Gew.% eines Materials für Elketrolumineszenz-Vorrichtungen, wie in Anspruch 1 oder 2 beschrieben, umfasst.

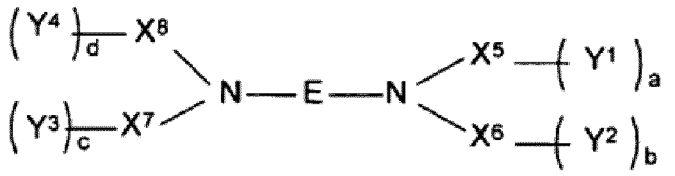
6. Organische Elektrolumineszenz-Vorrichtung, welche eine lichtemittierende Schicht oder eine Mehrzahl dünner Filme aus organischen Verbindungen, die eine lichtemittierende Schicht umfassen, zwischen einem Paar von Elektroden angeordnet umfasst, wobei ein oder mehrere Materiallen, ausgewählt aus der aus einem Lochinjektionsmaterial, einem Lochtransportmaterial und einem Dotierungsmaterial bestehenden Gruppe, jeweils unabhängig 0.1 bis 20 Gew.-% eines Materials für organische Elektrolumineszenz-Vorrichtungen wie in Anspruch 1 oder 2 beschrieben umfasst.

7. Organische Elektrolumineszenz-Vorrichtung, welche eine lichtemittierende Schicht oder eine Mehrzahl dünner Filme aus organischen Verbindungen, die eine lichtemittierende Schicht umfassen, zwischen einem Paar von Elektroden angeordnet umfasst, wobei die lichtemittierende Schicht Schicht ist, die ein Stilben-Derivat und ein Material für organische Elektrolumineszenz-Vorrichtungen wie in Anspruch 1 oder 2 beschrieben umfasst.

Revendications

1. Matériau pour des dispositifs organiques électroluminescents représenté par la formule générale [9] :

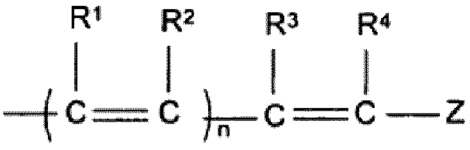
Formule générale [9]



(9)

dans laquelle E représente un groupe bivalent comprenant un noyau anthrancènique qui est substitué avec au moins deux groupes aryles ;
chacun de X^5 à X^8 représente indépendamment un radical arylène substitué ou non substitué avec de 6 à 20 atomes de carbone, X^5 et X^6 peuvent être liés l'un à l'autre, X^7 et X^8 peuvent être liés l'un à l'autre ;
chacun de Y^1 à Y^4 représente indépendamment un groupe organique représenté par la formule général [2] ;
chacun de a à d représente un entier de 0 à 2 et
la formule général [2] étant :

Formule générale [2]



$$-\!\!\left(\overset{R^1}{\underset{}{C}}=\overset{R^2}{C}\right)_{n}\!\!-\overset{R^3}{C}=\overset{R^4}{C}-Z \qquad (2)$$

dans laquelle chacun de R^1 à R^4 représente indépendamment un atome d'hydrogène, un groupe alkyle substitué ou non substitué avec de 1 à 20 atomes de carbone, un groupe aryle substitué ou non substitué avec de 6 à 20 atomes de carbone, un groupe cyano ou ils forment une triple liaison par la liaison entre R^1 et R^2 ou R^3 et R^4, Z représente un groupe aryle substitué ou non substitué avec de 6 à 20 atomes de carbone et n représente 0 ou 1.

2. Matériau pour des dispositifs organiques électroluminescents selon la revendication 1 qui est un matériau émettant de la lumière pour des dispositifs organiques électroluminescents.

3. Dispositif organique électroluminescent qui comprend une couche émettant de la lumière ou une pluralité de couches minces des composés organique comprenant couche émettant de la lumière disposées entre une paire d'électrodes, dans lequel au moins l'une des couches minces des composés organiques est une couche comprenant un matériau pour des dispositifs organiques électroluminescents selon la revendication 1 ou 2.

4. Dispositif organique électroluminescent qui comprend une couche émettant de la lumière ou une pluralité de couches minces des composés organiques comprenant une couche de la lumière disposées entre une paire d'électrodes, dans lequel une couche comprenant un matériau pour des dispositifs organiques électroluminescents selon la revendication 1 ou 2 est disposé entre la paire d'électrodes comme au moins un matériau sélectionné parmi le groupe consistant en un matériau injectant des trous, un matériau transportant des trous et un matériau de dopage.

5. Dispositif organique électroluminescent qui comprend une couche émettant de la lumière ou une pluralité de couches minces des composés organiques comprenant une couche émettant de la lumière disposées entre une paire d'électrodes, dans lequel la couche émettant de la lumière comprend de 0,1 à 20 % en masse du matériau pour des dispositifs organiques électroluminescents selon la revendication 1 ou 2.

6. Dispositif organique électroluminescent qui comprend une couche émettant de la lumière ou une pluralité de couches minces des composés organiques comprenant une couche émettant de la lumière disposées entre une paire d'électrodes, dans lequel chacun d'un ou plus matériaux sélectionnés parmi le groupe consistant en un matériau injectant des trous, un matériau transportant des trous et un matériau de dopage comprennent indépendamment de 0.1 à 20 % en masse du matériau pour des dispositifs organiques électroluminescents selon la revendication 1 ou 2.

7. Dispositif organique électroluminescent qui comprend une couche émettant de la lumière ou une pluralité de couches minces des composés organiques comprenant une couche émettant de la lumière disposées entre une paire d'électrodes, dans lequel la couche émettant de la lumière est une couche comprenant un dérivé des stilbènes et un matériau pour des dispositifs organiques électroluminescents selon la revendication 1 ou 2.

Fig. 1

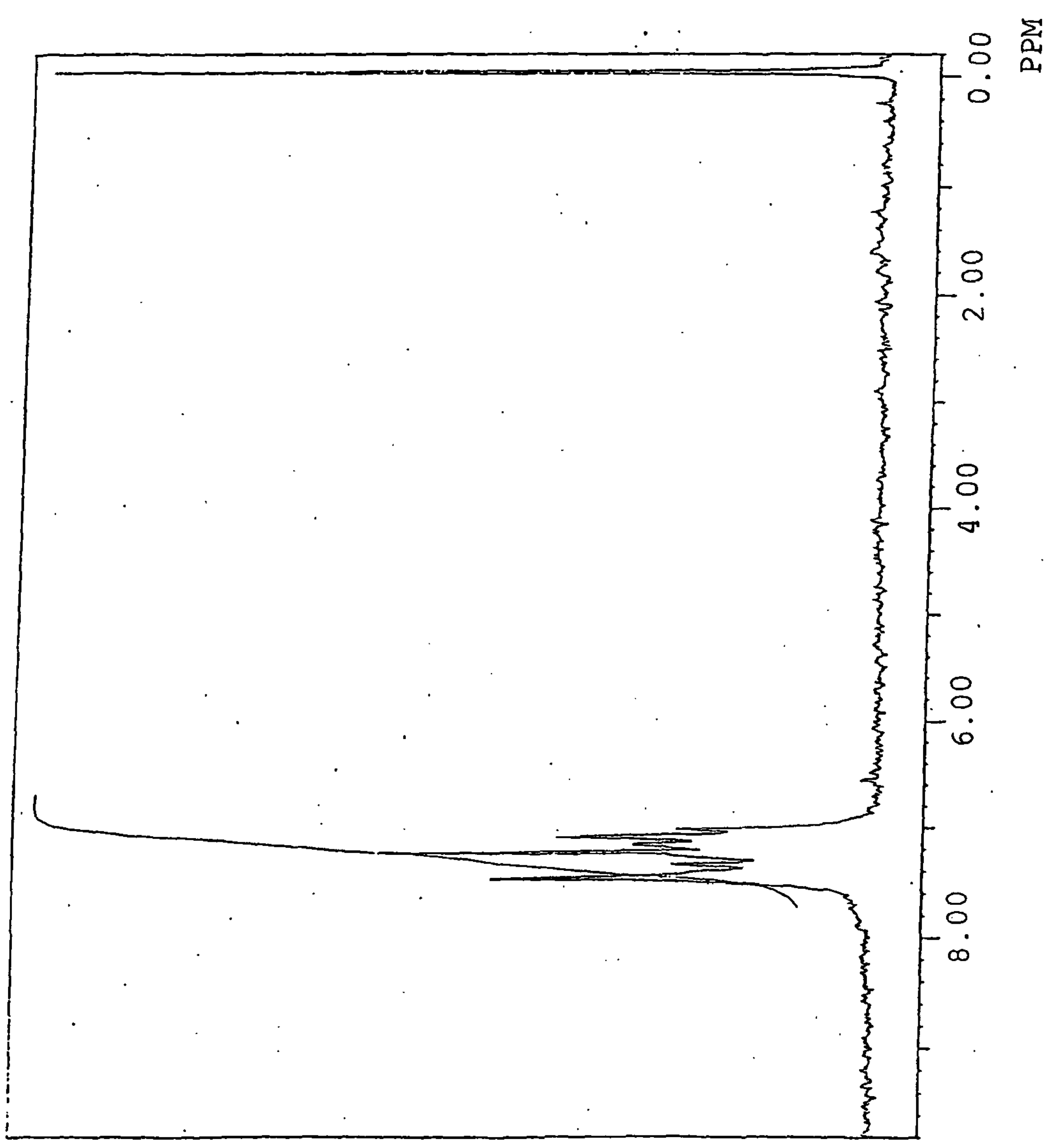


Fig. 2

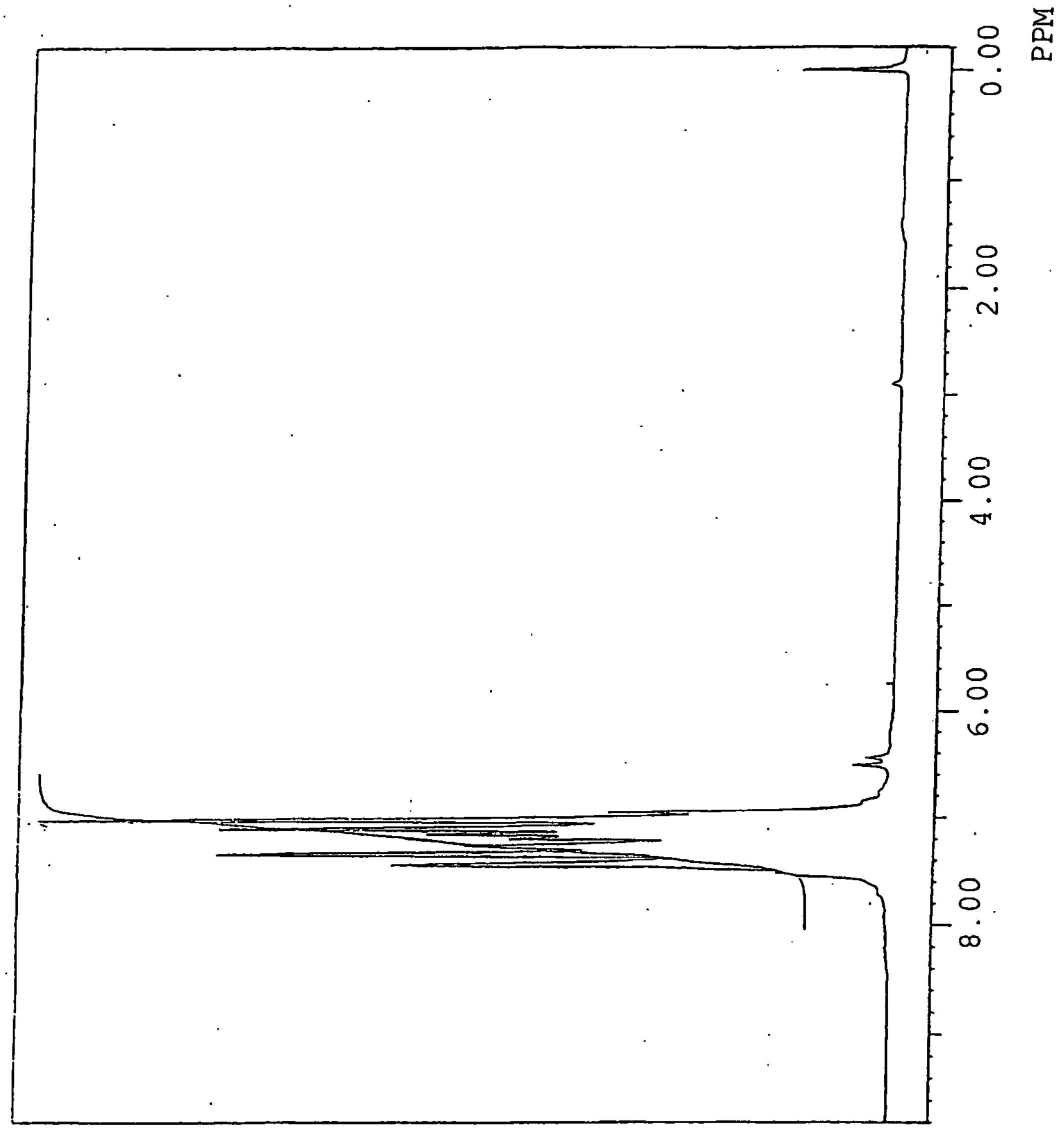
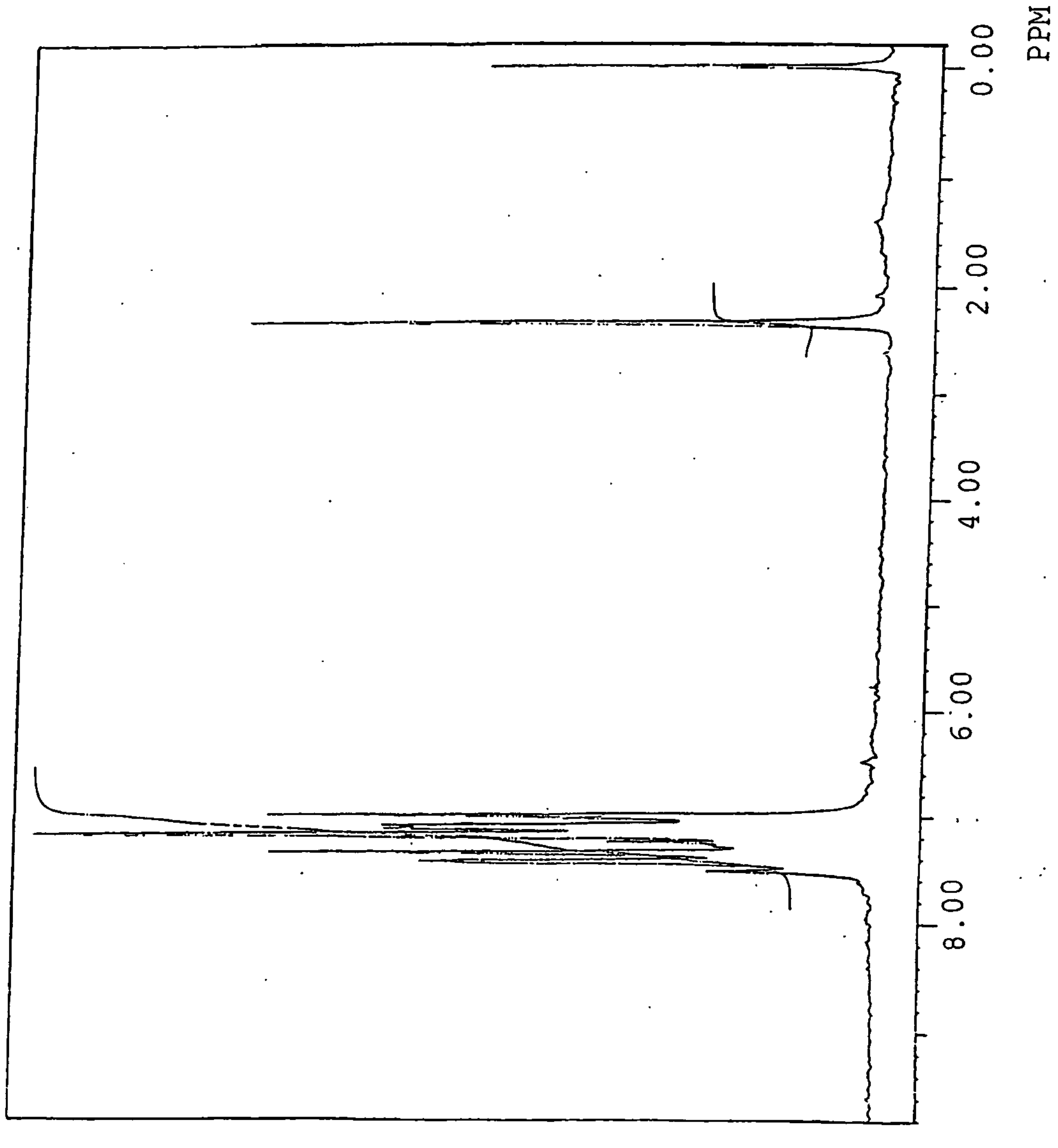


Fig. 3



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