

(19)



(11)

EP 1 791 928 B1

(12)

EUROPEAN PATENT SPECIFICATION

(45) Date of publication and mention
of the grant of the patent:
11.01.2012 Bulletin 2012/02

(21) Application number: **05856355.2**

(22) Date of filing: **23.09.2005**

(51) Int Cl.:
C09K 11/06 (2006.01)

(86) International application number:
PCT/KR2005/003170

(87) International publication number:
WO 2006/080638 (03.08.2006 Gazette 2006/31)

(54) **NEW COMPOUND AND ORGANIC LIGHT EMITTING DEVICE USING THE SAME (7)**

NEUE VERBINDUNG UND DIESE VERWENDENDE ORGANISCHE LICHEMITTIERENDE
VORRICHTUNG (7)

NOUVEAU COMPOSE ET DISPOSITIF ELECTROLUMINESCENT ORGANIQUE UTILISANT CE
COMPOSE (7)

(84) Designated Contracting States:
DE GB NL

(30) Priority: **24.09.2004 KR 20040077214**

(43) Date of publication of application:
06.06.2007 Bulletin 2007/23

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• **TRITSCHLER W ET AL: "SYNTHESE UND KONFORMATION VON SPIROACRIDANEN// SYNTHESIS AND CONFORMATION OF SPIROACRIDANS" CHEMISCHE BERICHTE, VERLAG CHEMIE GMBH. WEINHEIM, DE, vol. 117, 1 January 1984 (1984-01-01), pages 2703-2713, XP008051454 ISSN: 0009-2940**

• **TRITSCHLER ET AL. CHEMISCHE BERICHTE vol. 117, no. 8, 1984, pages 2703 - 2713, XP008095206**

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Description**Technical Field**

5 **[0001]** The present invention relates to an organic light emitting device in which a novel compound capable of significantly improving a lifespan, efficiency, and electrochemical and thermal stabilities of the organic light emitting device is contained in an organic compound layer.

[0002] Background Art

10 **[0003]** An organic light emission phenomenon is an example of a conversion of current into visible rays through an internal process of a specific organic molecule. The organic light emission phenomenon is based on the following mechanism. When organic material layers are interposed between an anode and a cathode, if voltage is applied between the two electrodes, electrons and holes are injected from the cathode and the anode into the organic material layer. The electrons and the holes which are injected into the organic material layer are recombined to form an exciton, and the exciton is reduced to a bottom state to emit light. An organic light emitting device which is based on the above mechanism typically comprises a cathode, an anode, and organic material layer(s), for example, organic material layers including a hole injection layer, a hole transport layer, a light emitting layer, and an electron transport layer, interposed therebetween.

15 **[0004]** The materials used in the organic light emitting device are mostly pure organic materials or complexes of organic material and metal. The material used in the organic light emitting device may be classified as a hole injection material, a hole transport material, a light emitting material, an electron transport material, or an electron injection material, according to its use. In connection with this, an organic material having a p-type property, which is easily oxidized and is electrochemically stable when it is oxidized, is mostly used as the hole injection material or the hole transport material. Meanwhile, an organic material having an n-type property, which is easily reduced and is electrochemically stable when it is reduced, is used as the electron injection material or the electron transport material. As the light emitting layer material, an organic material having both p-type and n-type properties is preferable, which is stable when it is oxidized and when it is reduced. Also a material having high light emission efficiency for conversion of the exciton into light when the exciton is formed is preferable.

20 **[0005]** In addition, it is preferable that the material used in the organic light emitting device further have the following properties.

30 **[0006]** First, it is preferable that the material used in the organic light emitting device have excellent thermal stability. The reason is that joule heat is generated by movement of electric charges in the organic light emitting device. NPB, which has recently been used as the hole transport layer material, has a glass transition temperature of 100°C or lower, thus it is difficult to apply to an organic light emitting device requiring a high current.

35 **[0007]** Second, in order to produce an organic light emitting device that is capable of being actuated at low voltage and has high efficiency, holes and electrons which are injected into the organic light emitting device must be smoothly transported to a light emitting layer, and must not be released out of the light emitting layer. To achieve this, a material used in the organic light emitting device must have a proper band gap and a proper HOMO or LUMO energy levels. A LUMO energy level of PEDOT:PSS, which is currently used as a hole transport material of an organic light emitting device produced using a solution coating method, is lower than that of an organic material used as a light emitting layer material, thus it is difficult to produce an organic light emitting device having high efficiency and a long lifespan.

40 **[0008]** Moreover, the material used in the organic light emitting device must have excellent chemical stability, electric charge mobility, and interfacial characteristic with an electrode or an adjacent layer. That is to say, the material used in the organic light emitting device must be little deformed by moisture or oxygen. Furthermore, proper hole or electron mobility must be assured so as to balance densities of the holes and of the electrons in the light emitting layer of the organic light emitting device to maximize the formation of excitons. Additionally, it has to be able to have a good interface with an electrode including metal or metal oxides so as to assure stability of the device.

45 The document WO 2004/020371 discloses fluorene compounds wherein the two benzene rings are substituted with arylamine moieties. These compounds are applied in organic light-emitting devices in the light emitting layer or as hole transporting material. The document US 2004/0219386 discloses triarylamine derivatives suitable as hole-transporting materials in organic light-emitting devices.

50 **[0009]** Accordingly, there is a need to develop an organic light emitting device including an organic material having the above-mentioned requirements in the art

[0010] Disclosure of Invention**Technical Problem**

55 **[0011]** Therefore, the object of the present inventions is to provide an organic light emitting device which is capable of satisfying conditions required of a material usable for an organic light emitting device, for example, a proper energy level, electrochemical stability, and thermal stability, and which includes a fluorene derivative having a chemical structure

capable of playing various roles required in the organic light emitting device, depending on a substituent group.

[0012] Technical Solution

[0013] The present invention provides an organic light emitting device which comprises a first electrode,; organic material layers comprising a light emitting layer and one of the hole-injection layer, a hole-transport layer and a layer which both injects and transports holes and a second electrode, wherein the first electrode, the organic material layers , and the second electrode form a layered structure, and the hole-injection layer, the hole-transport layer or the layer which both injects and transports holes includes a compound as defined in claim 1.

[0014] Brief Description of the Drawings

[0015] FIG. 1 illustrates an organic light emitting device comprising a substrate 1, an anode 2, a light emitting layer 3, and a cathode 4; and

[0016] FIG. 2 illustrates an organic light emitting device comprising a substrate 1, an anode 2, a hole injection layer 5, a hole transport layer 6, a light emitting layer 7, an electron transport layer 8, and a cathode 4.

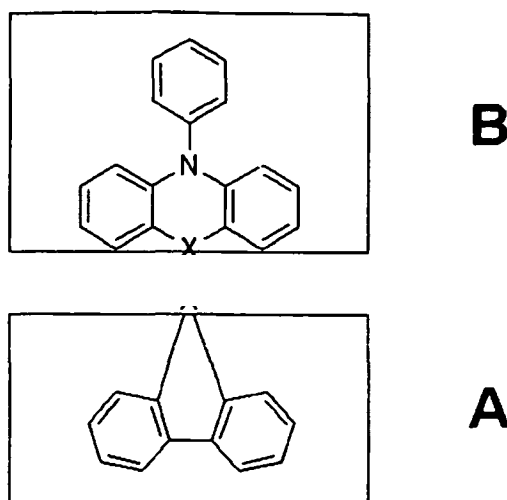
[0017] Best Mode for Carrying Out the Invention

[0018] Hereinafter, a detailed description will be given of the present invention.

[0019] Various substituent groups can be introduced into the core structure of the compound contained in one of the organic material layers. The core structure contains a fluorene group bonded to a combination of an acridine group and a carbazolyl group to form a spiro structure; thereby the compound has characteristics suitable for use in an organic material layer organic light emitting device. This will be described in detail, below.

[0020] The steric core structure of the compound can be divided into two portions, A and B, for explanation as shown in the following figure.

[0021]



[0022] The compound has the steric core structure in which a plane A meets with a plane B at right angles around X (a carbon atom), and conjugation does not occur between the A and B portions around X. Furthermore, since one nitrogen atom is positioned among three aryl groups in the plane B, conjugation is limited in the plane B.

[0023] The conjugation length of the compound has a close relationship with an energy band gap. In detail, the energy band gap is reduced as the conjugation length of the compound increases. As described above, since a conjugation structure is limited in the core structure of the compound, the core structure has a large energy band gap.

[0024] Various substituent groups are introduced into the core structure to produce compounds having intrinsic characteristics of the substituent groups. For example, substituent groups, which are frequently applied to hole injection layer, hole transport layer, light emitting layer, and electron transport layer materials during the production of the organic light emitting device, are introduced into the core structure so as to produce substances capable of satisfying the requirements of each organic material layer. Particularly, since the core structure of the compound includes the arylamine structure, it has an energy level suitable for the hole injection and/or hole transport materials in the organic light emitting device. In the present invention, the compound having the proper energy level is selected depending on the substituent group, thereby it is possible to realize a device having a low actuating voltage and a high light efficiency.

[0025] Furthermore, various substituent groups are asymmetrically introduced into the core structure (the A portion is located at one side of the core structure) so as to precisely control the energy band gap, improve interfacial characteristics with organic materials, and apply the compound to various fields.

[0026] In addition, since the number of nitrogen contained in the substituent group A is 1, it is possible to precisely

control the HOMO and LUMO energy levels and the energy band gap, and on the other hand interfacial characteristics with the organic materials is improved and thereby make it possible to apply the compound to various fields.

[0027] Meanwhile, since the compound has a high glass transition temperature (T_g), it has excellent thermal stability. For example, the glass transition temperature of the compound of Formula 3-2 is 143°C , which is still higher than that of conventionally used NPB (T_g : 96°C). Such increase in thermal stability is an important factor providing actuating stability to the device.

[0028] Furthermore, the compound may be used to form the organic material layer using a vacuum deposition process or a solution coating process during the production of the organic light emitting device. In connection with this, illustrative, but non-limiting, examples of the solution coating process include a spin coating process, a dip coating process, an inkjet printing process, a screen printing process, a spray process, and a roll coating process.

[0029] For example, the compound has excellent solubility in a polar solvent, such as xylene, dichloroethane, or NMP, which is used during the production of the device, and forms a thin film very well through the process using a solution, thus the solution coating process may be applied to produce the device.

[0030] Tertiary alcohol, which is produced by a reaction of a lithiated aryl and keto group, is heated in the presence of an acid catalyst to form a hexagonal cyclic structure while water is removed, thereby producing the compound having a spiro structure according to the present invention. The above-mentioned procedure for producing the compound is well known in the art, and those skilled in the art can change the production conditions during the production of the compound. The production will be described in detail in the preparation examples later.

[0031] The organic light emitting device of the present invention can be produced using known materials through a known process, modified only in that the hole-injection layer, hole-transport layer or layer which both injects and transports holes includes the compound of the present invention.

[0032] The organic light emitting device of the present invention may comprise a hole injection layer, a hole transport layer, a light emitting layer, an electron transport layer, and an electron injection layer as the organic material layers. However, the structure of the organic light emitting device is not limited to this, but may comprise a smaller number of organic material layers.

[0033] Furthermore, the organic light emitting device of the present invention may be produced, for example, by sequentially layering a first electrode, organic material layer(s), and a second electrode on a substrate. In connection with this, a physical vapor deposition (PVD) method, such as a sputtering method or an e-beam evaporation method, may be used, but the method is not limited to these.

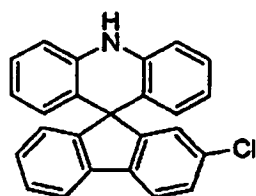
[0034] A method of producing the compound of the invention and the production of the organic light emitting device using the same will be described in detail in the following preparation examples and examples. However, the following preparation examples and examples are set forth to illustrate, but are not to be construed to limit the present invention.

Mode for the Invention

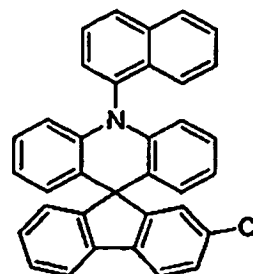
[0035] A better understanding of a method of producing an organic compound according to the invention and the production of an organic light emitting device using the same may be obtained in light of the following preparation examples and examples which are set forth to illustrate, but are not to be construed to limit the present invention.

[0036] In order to produce the compound of the invention compounds of the [85] following Formula a or b may be used as a starting material.

[0037]



[Formula a]



[Formula b]

[0038] PREPARATION EXAMPLE 1: Preparation of a starting material represented by Formula a

[0039] 1) After 10 g of diphenylamine (59 mmol) and 8.04 ml of bromomethyl methyl ether (88.6 mmol) were dissolved

in 100 ml of tetrahydrofuran, 12.4 ml of triethylamine (88.6 mmol) were added thereto. Stirring was conducted in a nitrogen atmosphere for 5 hours, and an organic layer was then extracted using distilled water. The extracted organic layer was subjected to a column separation process at a ratio of n-hexane/tetrahydrofuran of 15:1, and vacuum dried to produce 12 g of tertiary amine (yield 90 %).

[0040] 2) The amine compound produced in 1) (12.0 g, 56.3 mmol) was dissolved in 100 ml of purified THF and cooled to -78°C, and n-BuLi (2.5 M hexane solution, 22.5 ml, 56.3 mmol) was slowly dropped thereon. Stirring was conducted at the same temperature for 30 min, and a 2-chloro-9-fluorenone compound (12.1 g, 56.3 mmol) was added thereto. After stirring at the same temperature for 40 min, the temperature was raised to normal temperature and stirring was carried out for an additional 3 hours. The reaction was completed in an ammonium chloride aqueous solution, and extraction was conducted with ethyl ether. Water was removed from an organic material layer using anhydrous magnesium sulfate, and an organic solvent was then removed therefrom. The produced solid was dispersed in ethanol, stirred for one day, filtered, and vacuum dried. After an intermediate material was dispersed in 100 ml of acetic acid, ten drops of concentrated sulfuric acid were added thereto and reflux was conducted for 4 hours. The resulting solid was filtered, washed with ethanol, and vacuum dried to produce 20 g of amine (97 % yield). MS: [M+H]⁺ = 366.

[0041] PREPARATION EXAMPLE 2: Preparation of a starting material represented by Formula b

[0042] A compound of Formula a (8.23 g, 22.5 mmol), iodobenzene (9.18 g, 45 mmol), potassium carbonate (6.22 g, 45 mmol), copper iodide (214 mg, 1.13 mmol), and xylene (250 ml) were heated in a nitrogen atmosphere overnight. After cooling to normal temperature, a product was extracted with ethyl acetate, water was removed with anhydrous magnesium sulfate, and the solvent was removed at a reduced pressure. The resulting product was passed through a silica gel column using a hexane solvent to produce a compound, the solvent was removed at a reduced pressure, and vacuum drying was conducted to produce the compound of Formula b (5.2 g, 47 % yield). M: [M+H]⁺ = 493.

[0043] EXAMPLE 1: Preparation of the compound represented by Formula 3-2

[0044] After 4.37 g of the compound of Formula b (8.88 mmol) and 2.34 g of N-phenyl-1-naphthylamine (10.7 mmol) were dissolved in 120 ml of toluene, 2.90 g of sodium-tert-butoxide (30.2 mmol), 0.12 g of bis(dibenzylidene acetone) palladium(O) (0.21 mmol), and 0.16 ml of 50 wt% tri-tert-butylphosphine toluene solution (0.32 mmol) were added thereto, and reflux was conducted in a nitrogen atmosphere for 2 hours. Distilled water was added to the reaction solution to complete the reaction, and the organic layer was extracted. A column separation process was conducted using a solvent of n-hexane and tetrahydrofuran at a ratio of 9:1, stirring was conducted using petroleum ether, and vacuum drying was conducted to produce the compound of Formula 3-2 (5.2 g, yield 86.8 %). MS: [M+H]⁺ = 675.

[0045] EXAMPLE 2: Preparation of the compound represented by Formula 3-8

[0046] 1) Synthesis of arylamine (1,4-naphthylbiphenylamine) to produce the compound represented by Formula 3-8: 1-aminonaphthalene (7.4 g, 51.48 mmol) and 4-bromobiphenyl (12 g, 51.48 mmol) were dissolved in 200 ml of toluene, and bis(dibenzylidene acetone)palladium(O) (Pd(dba)₂, 0.89 g, 1.54 mmol), 50 wt% tri-tert-butylphosphine (0.60 ml, 1.54 mmol), and sodium-tert-butoxide (9.90 g, 103.0 mmol) were then added thereto. After reflux was conducted in a nitrogen atmosphere for 2 hours, distilled water was added to the reaction solution to complete the reaction. The organic layer was extracted, and a column separation process was conducted using a developing solvent of n-hexane and tetrahydrofuran (n-hexane/THF = 15/1), stirring was conducted using petroleum ether, and vacuum drying was conducted to produce arylamine (6.3 g, yield 42 %). MS: [M+H]⁺ = 295.

[0047] 2) After 4.37 g of the compound of Formula b (8.88 mmol) and 3.16 g of naphthyl-biphenylamine (10.7 mmol) were dissolved in 120 ml of toluene, 2.90 g of sodium-tert-butoxide (30.2 mmol), 0.12 g of bis(dibenzylidene acetone) palladium(O) (0.21 mmol), and 0.16 ml of 50 wt% tri-tert-butylphosphine toluene solution (0.32 mmol) were added thereto, and reflux was conducted in a nitrogen atmosphere for 2 hours. Distilled water was added to the reaction solution to complete the reaction, and the organic layer was extracted. A column separation process was conducted using a solvent of n-hexane and tetrahydrofuran at a ratio of 9:1, stirring was conducted using petroleum ether, and vacuum drying was conducted to produce the compound of Formula 3-8 (4.5 g, yield 67.5 %). MS: [M+H]⁺ = 751.

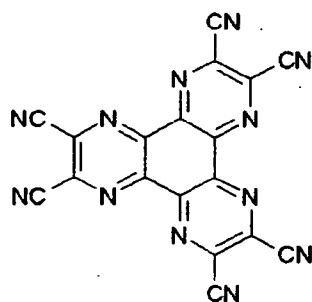
[0048] EXAMPLE 3: Production of an organic light emitting device

[0049] A glass substrate (coming 7059 glass), on which ITO (indium tin oxide) was applied to a thickness of 1000 Å to form a thin film, was put in distilled water, in which a detergent was dissolved, and washed using ultrasonic waves. In connection with this, a product manufactured by Fischer Inc. was used as a detergent, and distilled water was produced by filtering twice using a filter manufactured by Millipore Inc. After ITO was washed for 30 min, ultrasonic washing was conducted twice using distilled water for 10 min. After the washing using distilled water was completed, ultrasonic washing was conducted using isopropyl alcohol, acetone, and methanol solvents, and drying was then conducted. Next, it was transported to a plasma washing machine. Furthermore, the substrate was dry washed using oxygen plasma for 5 min, and then transported to a vacuum evaporator.

[0050] Hexanitrile hexaazatriphenylene (hereinafter, referred to as "HAT") of the following Formula was vacuum deposited to a thickness of 500 Å by heating on a transparent ITO electrode, which was prepared through the above procedure, so as to form an anode including an ITO conductive layer and an N-type organic material.

[0051]

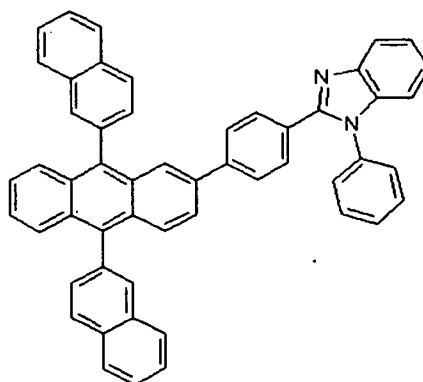
[HAT]



[0052] The compound of Formula 3-2 (400 Å) was vacuum deposited thereon to form a hole transport layer. Alq3 was vacuum deposited to a thickness of 300 Å on the hole transport layer to form a light emitting layer. An electron transport layer material of the following Formula was deposited to a thickness of 200 Å on the light emitting layer to form an electron transport layer.

[0053]

Electron transport layer material



[0054] Lithium fluoride (LiF) having a thickness of 12 Å and aluminum having a thickness of 2000 Å were sequentially deposited on the electron transport layer to form a cathode.

[0055] In the above procedure, the deposition speed of an organic material was maintained at 0.3 - 0.8 Å/sec. Furthermore, lithium fluoride and aluminum were deposited at speeds of 0.3 Å/sec and 1.5 - 2.5 Å/sec, respectively, on the cathode. During the deposition, a vacuum was maintained at $1 \sim 3 \times 10^{-7}$.

[0056] The resulting device had an electric field of 4.53 V at a forward current density of 100 mA/cm², and a spectrum having a light efficiency of 1.91 lm/W. The operation and light emission of the device at the above-mentioned actuating voltage mean that the compound of Formula 3-2, which formed the layer between the hole injection layer and the light emitting layer, functions to transport holes.

[0057] EXAMPLE 4: Production of an organic light emitting device

[0058] The procedure of example 14 was repeated to produce a device except that the compound of Formula 3-8 was applied to the hole transport layer of example 3, instead of the compound of Formula 3-2.

[0059] The resulting device had an electric field of 4.33 V at a forward current density of 100 mA/cm², and a spectrum having a light efficiency of 1.93 lm/W. The operation and light emission of the device at the above-mentioned actuating voltage mean that the compound of Formula 3-8, which formed the layer between the hole injection layer and the light emitting layer, functions to transport holes.

Industrial Applicability

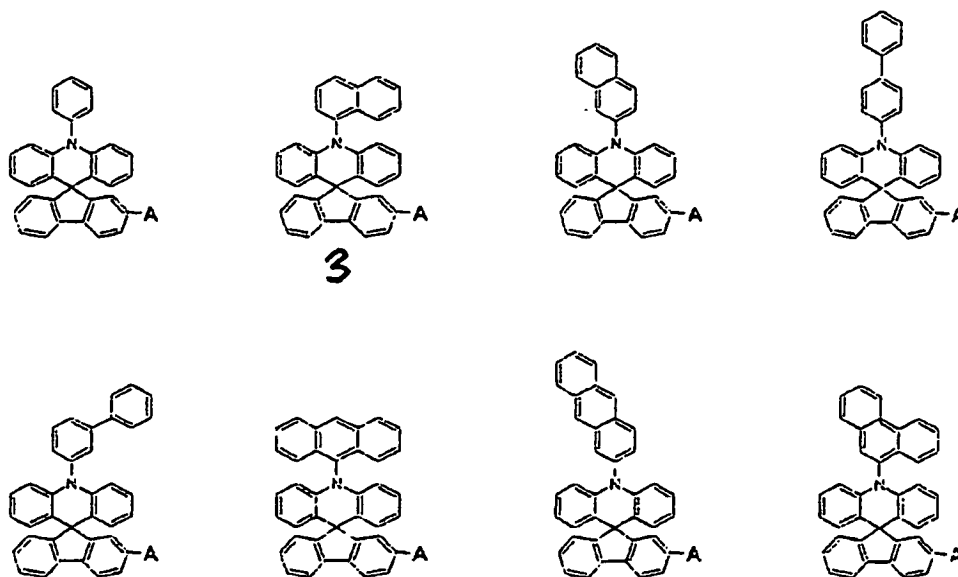
[0060] The compound of the present invention can be used as an organic material layer material, particularly, hole

injection and/or transport materials in an organic light emitting device, and when applied to an organic light emitting device it is possible to reduce the actuating voltage of the device, to improve the light efficiency thereof, and to improve the lifespan of the device through the thermal stability of the compound.

Claims

1. An organic light-emitting device comprising:

a first electrode;
organic material layers comprising a light-emitting layer and one of a hole-injection layer, a hole-transport layer and a layer which both injects and transports holes; and
a second electrode;
wherein the first electrode, the organic material layers and the second electrode form a layered structure; and
the hole-injection layer, the hole-transport layer or the layer which both injects and transports holes comprises a compound represented by one of the following formulae:



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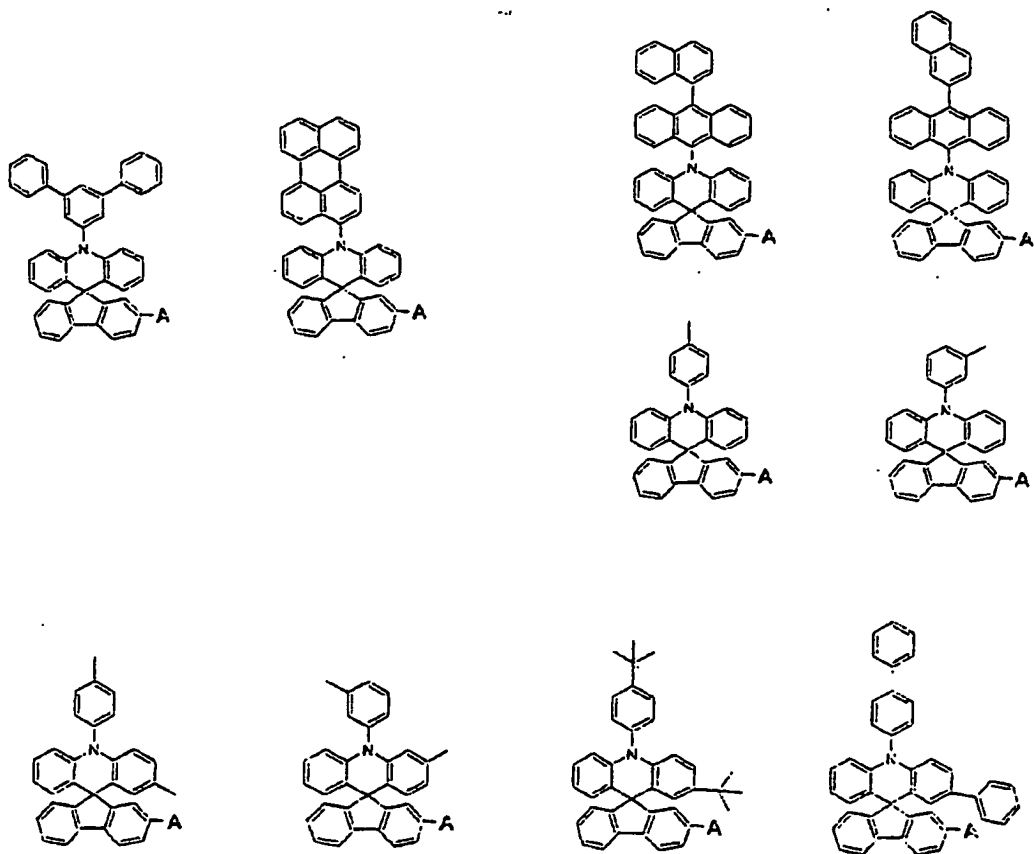
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wherein A represents one of the following groups:

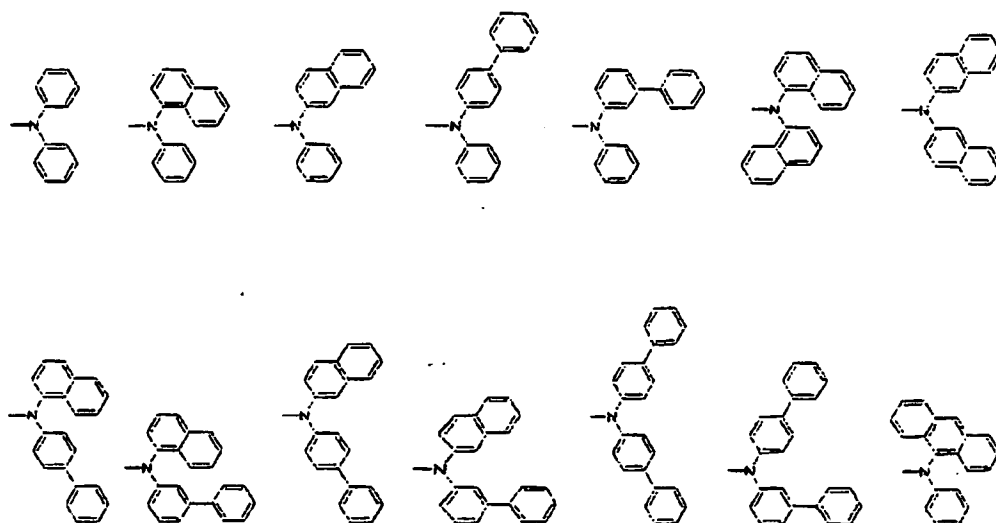
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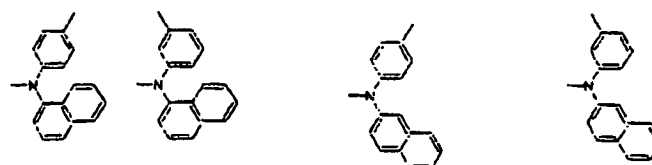
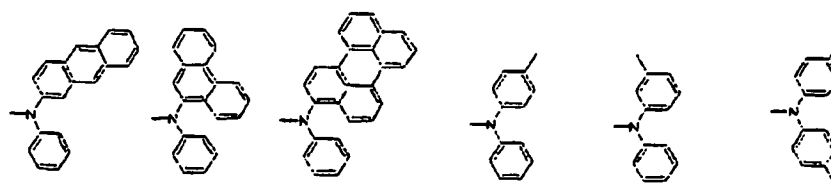
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Patentansprüche

1. Organische Licht-emittierende Vorrichtung, umfassend:

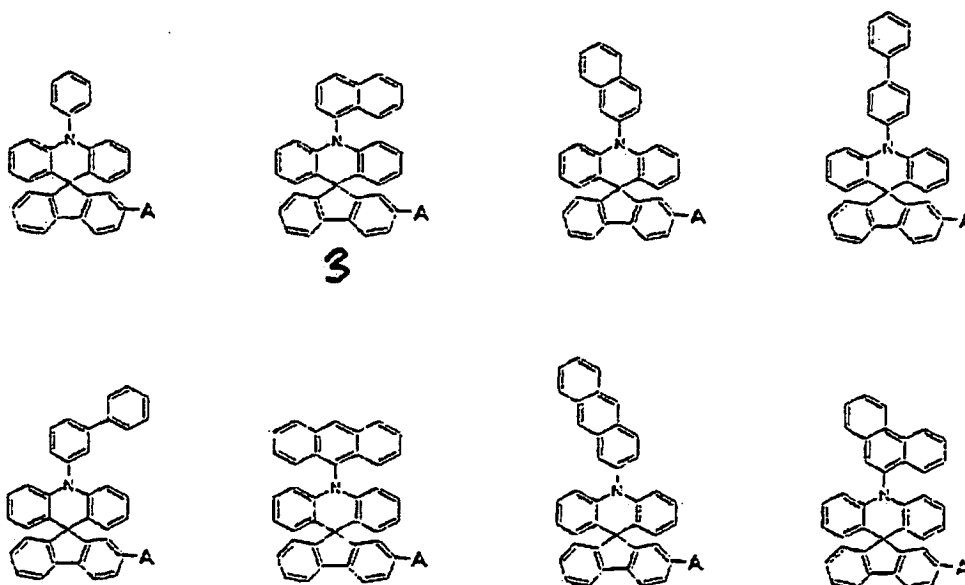
eine erste Elektrode;

Schichten aus organischem Material, umfassend eine Licht-emittierende Schicht und entweder eine Loch-Injektionsschicht, eine Loch-Transportschicht oder eine Schicht, die sowohl Löcher injiziert als auch transportiert und

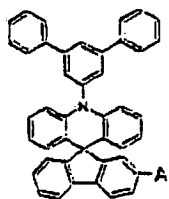
eine zweite Elektrode;

worin die erste Elektrode, die Schichten aus organischem Material und die zweite Elektrode in einer Schichtstruktur gebildet sind und

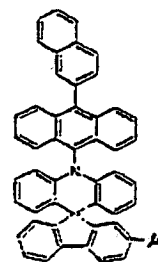
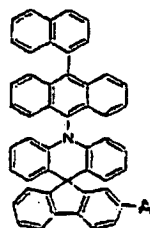
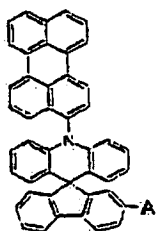
die Loch-Injektionsschicht, die Loch-Transportschicht oder die Schicht, die sowohl Löcher injiziert als auch transportiert, eine durch eine der folgenden Formeln dargestellte Verbindung umfasst:



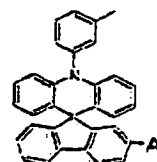
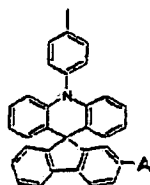
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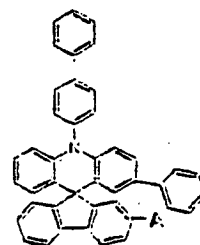
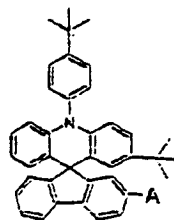
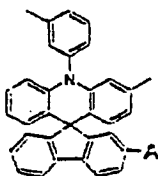
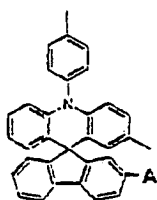


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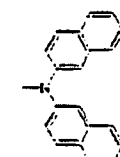
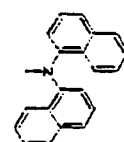
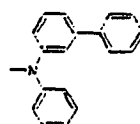
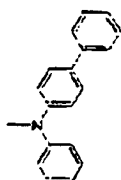
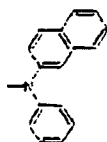
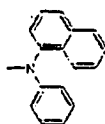
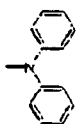
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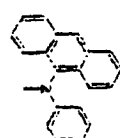
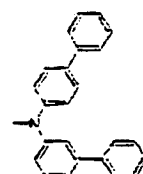
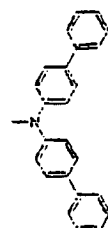
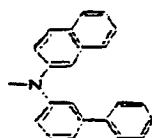
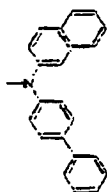
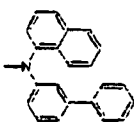
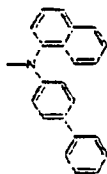
worin A eine der folgenden Gruppen darstellt:

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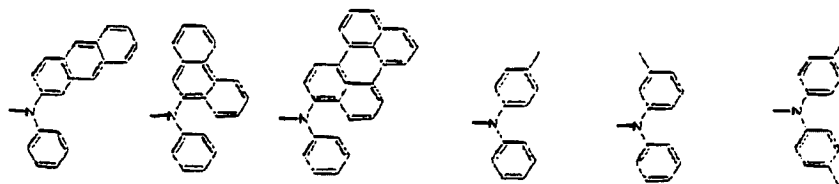
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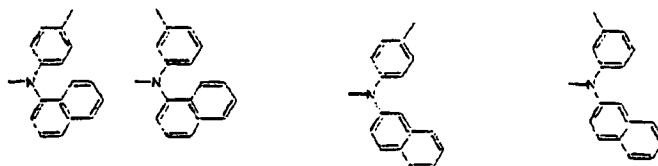
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Revendications

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1. Dispositif électroluminescent organique comprenant :

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une première électrode ;

des couches de matière organique comprenant une couche électroluminescente et l'une d'une couche d'injection de trous, d'une couche de transport de trous et d'une couche qui à la fois injecte et transporte des trous ; et

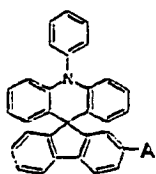
une seconde électrode ;

dans lequel la première électrode, les couches de matière organique et la seconde électrode forment une structure stratifiée ; et

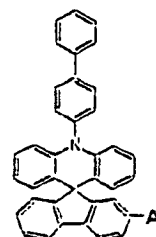
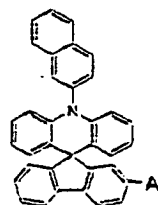
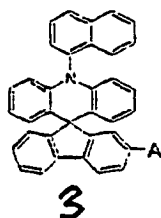
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la couche d'injection de trous, la couche de transport de trous ou la couche qui à la fois injecte et transporte des trous comprend un composé représenté par une des formules suivantes :

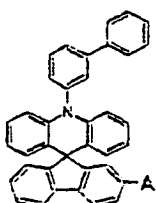
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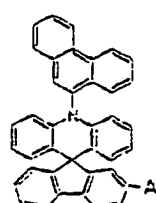
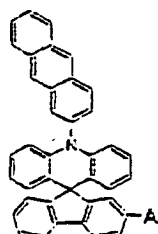
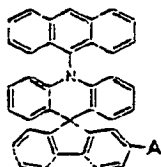
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dans lesquelles A représente l'un des groupes suivants :

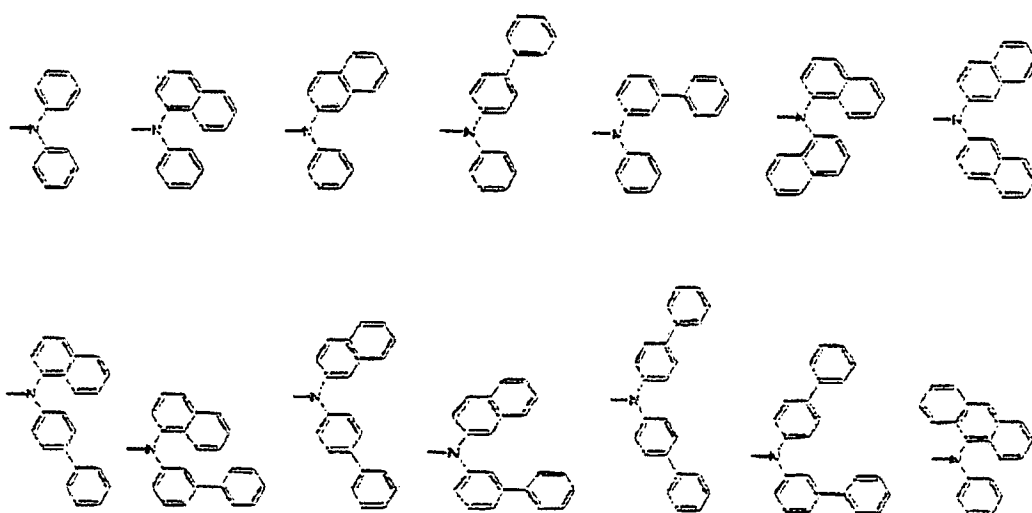
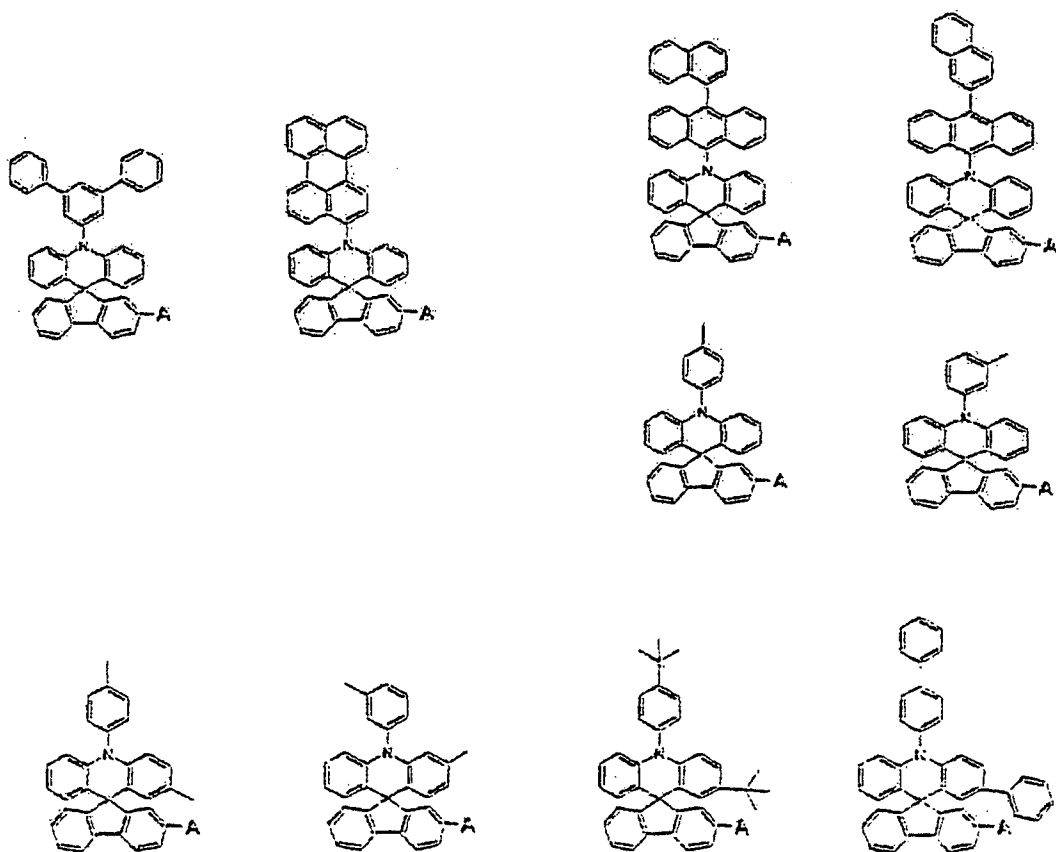
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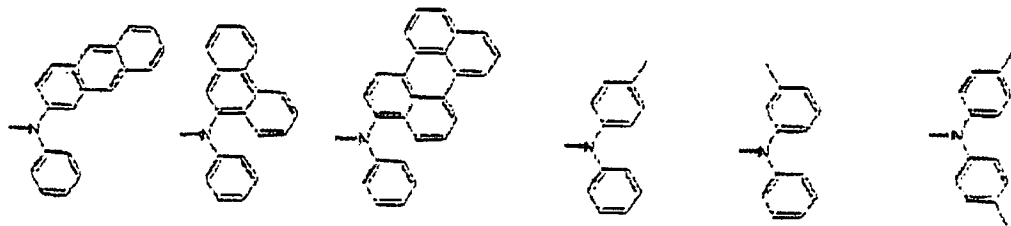
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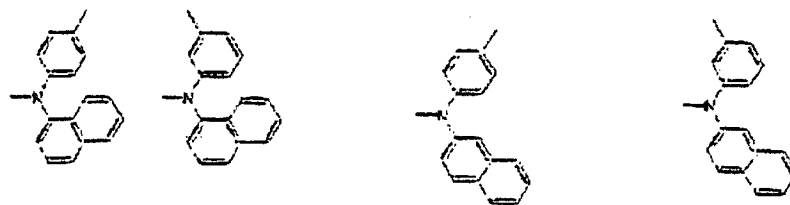


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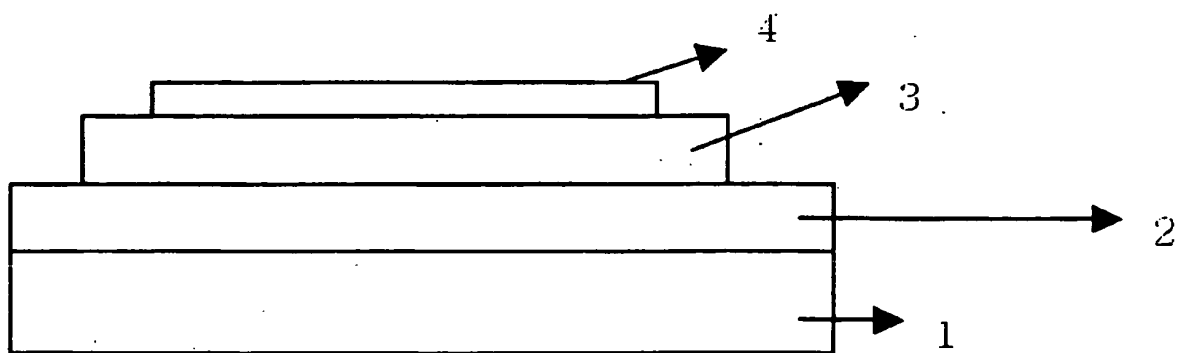
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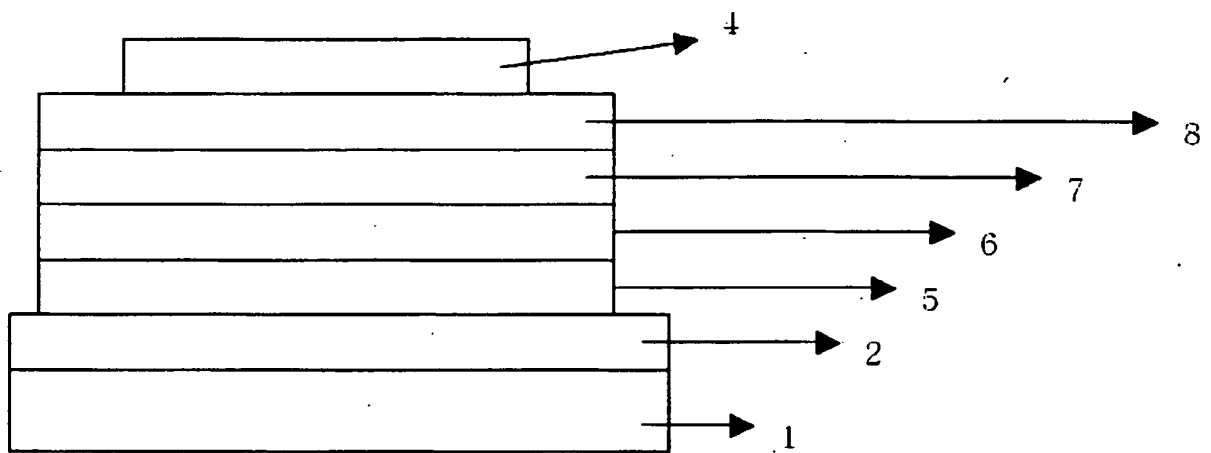
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[Fig. 1]



[Fig. 2]



REFERENCES CITED IN THE DESCRIPTION

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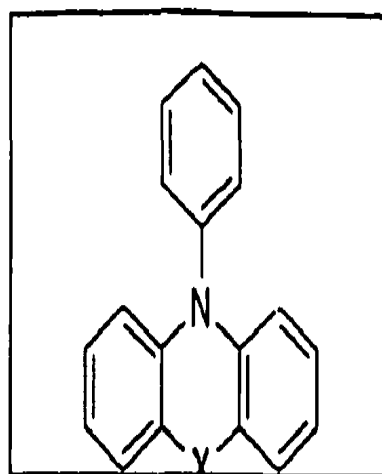
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- WO 2004020371 A [0008]
- US 20040219386 A [0008]

专利名称(译)	新型化合物及使用其的有机发光装置 (7)		
公开(公告)号	EP1791928B1	公开(公告)日	2012-01-11
申请号	EP2005856355	申请日	2005-09-23
[标]申请(专利权)人(译)	乐金化学股份有限公司		
申请(专利权)人(译)	LG化学公司		
当前申请(专利权)人(译)	LG化学公司		
[标]发明人	CHO WOOK DONG KIM JI EUN JEON BYUNG SUN JANG JUN GI YOON SEOK HEE MOON JAE MIN		
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IPC分类号	C09K11/06		
CPC分类号	C09K11/06 C07D221/20 C09K2211/1011 C09K2211/1029 H01L51/0039 H01L51/006 H01L51/5048 H01L51/5088 H05B33/14 Y10S428/917		
优先权	1020040077214 2004-09-24 KR		
其他公开文献	EP1791928A4 EP1791928A1		
外部链接	Espacenet		

摘要(译)

公开了一种有机发光器件。有机发光器件包括第一电极，包括发光层的有机材料层和第二电极。第一电极，有机材料层和第二电极形成层状结构，并且有机材料层的至少一层包括其中热固性或光致的式1的化合物或式1的化合物 -介绍了可交联的官能团。



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