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PHOSPHORESZIERENDE MATERIALIEN
MATÉRIAUX PHOSPHORESCENTS

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(56) References cited:
KR-B1- 100 662 430 US-A1- 2007 004 918

Remarks:

The file contains technical information submitted after
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specification

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Description

[0001] This application claims priority to U.S. Provisional Application No.: 61/097,488, filed September 16, 2008.

[0002] The claimed invention was made by, on behalf of, and/or in connection with one or more of the following parties to a joint university corporation research agreement: Regents of the University of Michigan, Princeton University, The University of Southern California, and the Universal Display Corporation. The agreement was in effect on and before the date the claimed invention was made, and the claimed invention was made as a result of activities undertaken within the scope of the agreement.

FIELD OF THE INVENTION

[0003] The present invention relates to organic light emitting devices (OLEDs), and specifically to phosphorescent organic materials used in such devices. More specifically, the present invention relates to iridium compounds having a narrow spectrum incorporated into OLEDs.

BACKGROUND

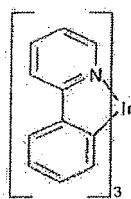
[0004] Opto-electronic devices that make use of organic materials are becoming increasingly desirable for a number of reasons. Many of the materials used to make such devices are relatively inexpensive, so organic opto-electronic devices have the potential for cost advantages over inorganic devices. In addition, the inherent properties of organic materials, such as their flexibility, may make them well suited for particular applications such as fabrication on a flexible substrate. Examples of organic opto-electronic devices include organic light emitting devices (OLEDs), organic phototransistors, organic photovoltaic cells, and organic photodetectors. For OLEDs, the organic materials may have performance advantages over conventional materials. For example, the wavelength at which an organic emissive layer emits light may generally be readily tuned with appropriate dopants.

[0005] OLEDs make use of thin organic films that emit light when voltage is applied across the device. OLEDs are becoming an increasingly interesting technology for use in applications such as flat panel displays, illumination, and backlighting. Several OLED materials and configurations are described in U.S. Pat. Nos. 5,844,363, 6,303,238, and 5,707,745.

[0006] US 2007/004918 A1 discloses red phosphorescence compounds and organic electro-luminescence devices using the same. KR 100662430 B1 discloses similar compounds and devices.

[0007] One application for phosphorescent emissive molecules is a full color display. Industry standards for such a display call for pixels adapted to emit particular colors, referred to as "saturated" colors. In particular, these standards call for saturated red, green, and blue pixels. Color may be measured using CIE coordinates, which are well known to the art.

[0008] One example of a green emissive molecule is tris(2-phenylpyridine) iridium, denoted Ir(ppy)₃, which has the structure of Formula I:



[0009] In this, and later figures herein, we depict the dative bond from nitrogen to metal (here, Ir) as a straight line.

[0010] As used herein, the term "organic" includes polymeric materials as well as small molecule organic materials that may be used to fabricate organic opto-electronic devices. "Small molecule" refers to any organic material that is not a polymer, and "small molecules" may actually be quite large. Small molecules may include repeat units in some circumstances. For example, using a long chain alkyl group as a substituent does not remove a molecule from the "small molecule" class. Small molecules may also be incorporated into polymers, for example as a pendent group on a polymer backbone or as a part of the backbone. Small molecules may also serve as the core moiety of a dendrimer, which consists of a series of chemical shells built on the core moiety. The core moiety of a dendrimer may be a fluorescent or phosphorescent small molecule emitter. A dendrimer may be a "small molecule," and it is believed that all dendrimers currently used in the field of OLEDs are small molecules.

[0011] As used herein, "top" means furthest away from the substrate, while "bottom" means closest to the substrate. Where a first layer is described as "disposed over" a second layer, the first layer is disposed further away from substrate.

There may be other layers between the first and second layer, unless it is specified that the first layer is "in contact with" the second layer. For example, a cathode may be described as "disposed over" an anode, even though there are various organic layers in between.

[0012] As used herein, "solution processible" means capable of being dissolved, dispersed, or transported in and/or deposited from a liquid medium, either in solution or suspension form.

[0013] A ligand may be referred to as "photoactive" when it is believed that the ligand directly contributes to the photoactive properties of an emissive material. A ligand may be referred to as "ancillary" when it is believed that the ligand does not contribute to the photoactive properties of an emissive material, although an ancillary ligand may alter the properties of a photoactive ligand.

[0014] As used herein, and as would be generally understood by one skilled in the art, a first "Highest Occupied Molecular Orbital" (HOMO) or "Lowest Unoccupied Molecular Orbital" (LUMO) energy level is "greater than" or "higher than" a second HOMO or LUMO energy level if the first energy level is closer to the vacuum energy level. Since ionization potentials (IP) are measured as a negative energy relative to a vacuum level, a higher HOMO energy level corresponds to an IP having a smaller absolute value (an IP that is less negative). Similarly, a higher LUMO energy level corresponds to an electron affinity (EA) having a smaller absolute value (an EA that is less negative). On a conventional energy level diagram, with the vacuum level at the top, the LUMO energy level of a material is higher than the HOMO energy level of the same material. A "higher" HOMO or LUMO energy level appears closer to the top of such a diagram than a "lower" HOMO or LUMO energy level.

[0015] As used herein, and as would be generally understood by one skilled in the art, a first work function is "greater than" or "higher than" a second work function if the first work function has a higher absolute value. Because work functions are generally measured as negative numbers relative to vacuum level, this means that a "higher" work function is more negative. On a conventional energy level diagram, with the vacuum level at the top, a "higher" work function is illustrated as further away from the vacuum level in the downward direction. Thus, the definitions of HOMO and LUMO energy levels follow a different convention than work functions.

[0016] More details on OLEDs, and the definitions described above, can be found in US Pat. No. 7,279,704.

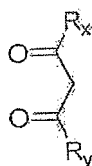
SUMMARY OF THE INVENTION

[0017] Compounds are provided as claimed in claim 1.

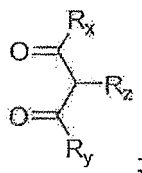
[0018] An organic light emitting device is provided. The device comprises an anode, a cathode, and an organic layer disposed between the anode and the cathode. The organic layer comprises one or more of the inventive compounds. The organic layer can be an emissive layer that contains an emissive dopant and a host, wherein the inventive compound is the emissive dopant and BA1q is the host.

[0019] A consumer product is also provided. The consumer product comprises a device which itself comprises an anode, a cathode, and an organic layer disposed between the anode and the cathode. The organic layer comprises one or more of the inventive compounds.

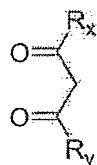
[0020] A method not according to the invention is described, the method comprising reacting



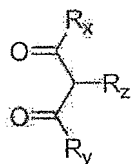
with R_z-X to form the free base



separating unreacted

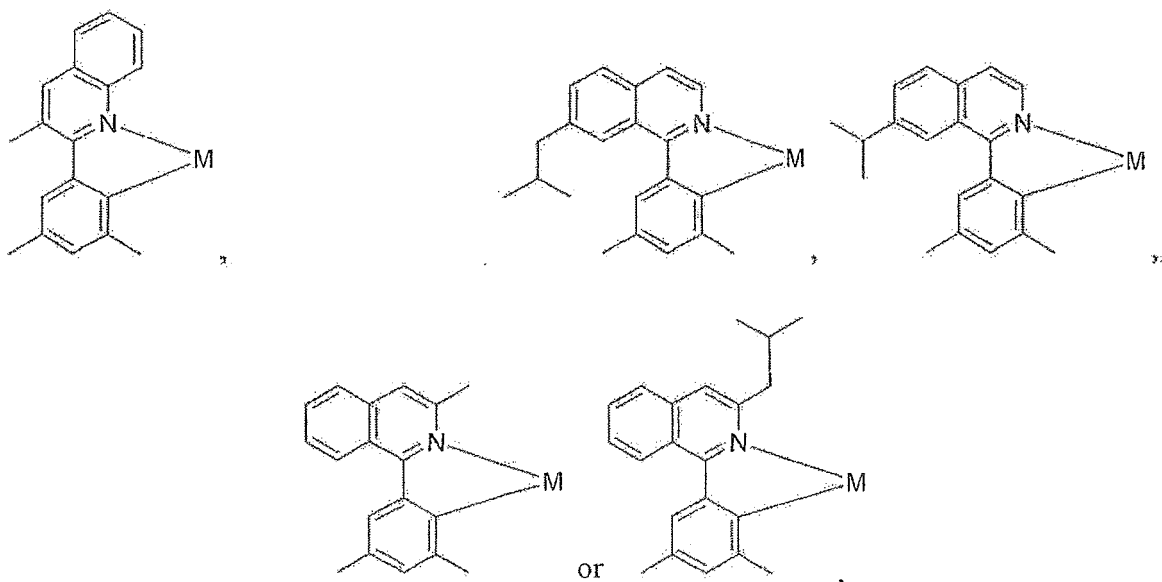


and the product



by column chromatography using a stationary phase consisting of alumina; wherein R_x and R_y are each independently selected from the group consisting of hydrogen, alkyl, heteroalkyl, aryl, or heteroaryl groups; wherein R_z is selected from the group consisting of alkyl, heteroalkyl, aryl, or heteroaryl groups; and wherein $X = Cl, Br, I, OTf, OTs$ or OH .

[0021] An organometallic compound is also provided, the organometallic compound containing a structure selected from the group consisting of:



wherein M is a metal with an atom weight greater than 40.

BRIEF DESCRIPTION OF THE DRAWINGS

[0022]

FIG. 1 shows an organic light emitting device.

FIG. 2 shows an inverted organic light emitting device that does not have a separate electron transport layer.

DETAILED DESCRIPTION

[0023] Generally, an OLED comprises at least one organic layer disposed between and electrically connected to an anode and a cathode. When a current is applied, the anode injects holes and the cathode injects electrons into the organic layer(s). The injected holes and electrons each migrate toward the oppositely charged electrode. When an electron and hole localize on the same molecule, an "exciton," which is a localized electron-hole pair having an excited energy state, is formed. Light is emitted when the exciton relaxes via a photoemissive mechanism. In some cases, the

exciton may be localized on an excimer or an exciplex. Non-radiative mechanisms, such as thermal relaxation, may also occur, but are generally considered undesirable.

[0024] The initial OLEDs used emissive molecules that emitted light from their singlet states ("fluorescence") as disclosed, for example, in U.S. Pat. No. 4,769,292. Fluorescent emission generally occurs in a time frame of less than 10 nanoseconds.

[0025] More recently, OLEDs having emissive materials that emit light from triplet states ("phosphorescence") have been demonstrated. Baldo et al., "Highly Efficient Phosphorescent Emission from Organic Electroluminescent Devices," *Nature*, vol. 395, 151-154, 1998; ("Baldo-I") and Baldo et al., "Very high-efficiency green organic light-emitting devices based on electrophosphorescence," *Appl. Phys. Lett.*, vol. 75, No. 3, 4-6 (1999) ("Baldo-II"). Phosphorescence is described in more detail in US Pat. No. 7,279,704 at cols. 5-6.

[0026] FIG. 1 shows an organic light emitting device 100. The figures are not necessarily drawn to scale. Device 100 may include a substrate 110, an anode 115, a hole injection layer 120, a hole transport layer 125, an electron blocking layer 130, an emissive layer 135, a hole blocking layer 140, an electron transport layer 145, an electron injection layer 150, a protective layer 155, and a cathode 160. Cathode 160 is a compound cathode having a first conductive layer 162 and a second conductive layer 164. Device 100 may be fabricated by depositing the layers described, in order. The properties and functions of these various layers, as well as example materials, are described in more detail in US 7,279,704 at cols. 6-10.

[0027] More examples for each of these layers are available. For example, a flexible and transparent substrate-anode combination is disclosed in U.S. Pat. No. 5,844,363. An example of a p-doped hole transport layer is m-MTDATA doped with F.sub.4-TCNQ at a molar ratio of 50:1, as disclosed in U.S. Patent Application Publication No. 2003/0230980. Examples of emissive and host materials are disclosed in U.S. Pat. No. 6,303,238 to Thompson et al. An example of an n-doped electron transport layer is BPhen doped with Li at a molar ratio of 1:1, as disclosed in U.S. Patent Application Publication No. 2003/0230980. U.S. Pat. Nos. 5,703,436 and 5,707,745, disclose examples of cathodes including compound cathodes having a thin layer of metal such as Mg:Ag with an overlying transparent, electrically-conductive, sputter-deposited ITO layer. The theory and use of blocking layers is described in more detail in U.S. Pat. No. 6,097,147 and U.S. Patent Application Publication No. 2003/0230980. Examples of injection layers are provided in U.S. Patent Application Publication No. 2004/0174116. A description of protective layers may be found in U.S. Patent Application Publication No. 2004/0174116.

[0028] FIG. 2 shows an inverted OLED 200. The device includes a substrate 210, a cathode 215, an emissive layer 220, a hole transport layer 225, and an anode 230. Device 200 may be fabricated by depositing the layers described, in order. Because the most common OLED configuration has a cathode disposed over the anode, and device 200 has cathode 215 disposed under anode 230, device 200 may be referred to as an "inverted" OLED. Materials similar to those described with respect to device 100 may be used in the corresponding layers of device 200. FIG. 2 provides one example of how some layers may be omitted from the structure of device 100.

[0029] The simple layered structure illustrated in FIGS. 1 and 2 is provided by way of non-limiting example, and it is understood that embodiments of the invention may be used in connection with a wide variety of other structures. The specific materials and structures described are exemplary in nature, and other materials and structures may be used. Functional OLEDs may be achieved by combining the various layers described in different ways, or layers may be omitted entirely, based on design, performance, and cost factors. Other layers not specifically described may also be included. Materials other than those specifically described may be used. Although many of the examples provided herein describe various layers as comprising a single material, it is understood that combinations of materials, such as a mixture of host and dopant, or more generally a mixture, may be used. Also, the layers may have various sublayers. The names given to the various layers herein are not intended to be strictly limiting. For example, in device 200, hole transport layer 225 transports holes and injects holes into emissive layer 220, and may be described as a hole transport layer or a hole injection layer. In one embodiment, an OLED may be described as having an "organic layer" disposed between a cathode and an anode. This organic layer may comprise a single layer, or may further comprise multiple layers of different organic materials as described, for example, with respect to FIGS. 1 and 2.

[0030] Structures and materials not specifically described may also be used, such as OLEDs comprised of polymeric materials (PLEDs) such as disclosed in U.S. Pat. No. 5,247,190 to Friend et al. By way of further example, OLEDs having a single organic layer may be used. OLEDs may be stacked, for example as described in U.S. Pat. No. 5,707,745 to Forrest et al. The OLED structure may deviate from the simple layered structure illustrated in FIGS. 1 and 2. For example, the substrate may include an angled reflective surface to improve out-coupling, such as a mesa structure as described in U.S. Pat. No. 6,091,195 to Forrest et al., and/or a pit structure as described in U.S. Pat. No. 5,834,893 to Bulovic et al.

[0031] Unless otherwise specified, any of the layers of the various embodiments may be deposited by any suitable method. For the organic layers, preferred methods include thermal evaporation, ink-jet, such as described in U.S. Pat. Nos. 6,013,982 and 6,087,196, organic vapor phase deposition (OVPD), such as described in U.S. Pat. No. 6,337,102 to Forrest et al., and deposition by organic vapor jet printing (OVJP), such as described in U.S. patent application Ser.

No. 10/233,470. Other suitable deposition methods include spin coating and other solution based processes. Solution based processes are preferably carried out in nitrogen or an inert atmosphere. For the other layers, preferred methods include thermal evaporation. Preferred patterning methods include deposition through a mask, cold welding such as described in U.S. Pat. Nos. 6,294,398 and 6,468,819 and patterning associated with some of the deposition methods such as ink-jet and OVJD. Other methods may also be used. The materials to be deposited may be modified to make them compatible with a particular deposition method. For example, substituents such as alkyl and aryl groups, branched or unbranched, and preferably containing at least 3 carbons, may be used in small molecules to enhance their ability to undergo solution processing. Substituents having 20 carbons or more may be used, and 3-20 carbons is a preferred range. Materials with asymmetric structures may have better solution processibility than those having symmetric structures, because asymmetric materials may have a lower tendency to recrystallize. Dendrimer substituents may be used to enhance the ability of small molecules to undergo solution processing.

[0032] Devices fabricated in accordance with embodiments of the invention may be incorporated into a wide variety of consumer products, including flat panel displays, computer monitors, televisions, billboards, lights for interior or exterior illumination and/or signaling, heads up displays, fully transparent displays, flexible displays, laser printers, telephones, cell phones, personal digital assistants (PDAs), laptop computers, digital cameras, camcorders, viewfinders, micro-displays, vehicles, a large area wall, theater or stadium screen, or a sign. Various control mechanisms may be used to control devices fabricated in accordance with the present invention, including passive matrix and active matrix. Many of the devices are intended for use in a temperature range comfortable to humans, such as 18 degrees C. to 30 degrees C., and more preferably at room temperature (20-25 degrees C.).

[0033] The materials and structures described herein may have applications in devices other than OLEDs. For example, other optoelectronic devices such as organic solar cells and organic photodetectors may employ the materials and structures. More generally, organic devices, such as organic transistors, may employ the materials and structures.

[0034] The terms halo, halogen, alkyl, cycloalkyl, alkenyl, alkynyl, arylkyl, heterocyclic group, aryl, aromatic group, and heteroaryl are known to the art, and are defined in US 7,279,704 at cols. 31-32.

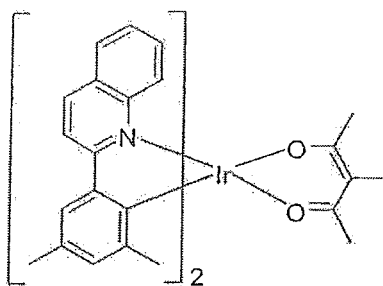
[0035] Numerous Ir(2-phenylquinoline) and Ir(1-phenylisoquinoline) type phosphorescent materials have been synthesized, and OLEDs incorporating them as the dopant emitters have been fabricated. The devices may exhibit advantageously exhibit high current efficiency, high stability, narrow emission, improved processibility (e.g., high solubility and low sublimation temperature), and/or high luminous efficiency: quantum efficiency ratio (LE:EQE).

[0036] Using Ir(3-Meppy)3 as a base structure, different alkyl substitution patterns on both the emitting ligand and the ancillary ligand were studied to establish a structure-property relationship with respect to material processibility (evaporation temperature, evaporation stability, solubility, etc) and device characteristics of Ir(2-phenylquinoline) and Ir(1-phenylisoquinoline) type phosphorescent materials and their PHOLEDs. Alkyl substitutions are particularly important because they offer a wide range of tunability in terms of evaporation temperature, solubility, energy levels, device efficiency and narrowness of the emission spectrum. Moreover, they are stable functional groups chemically and in device operation when applied appropriately.

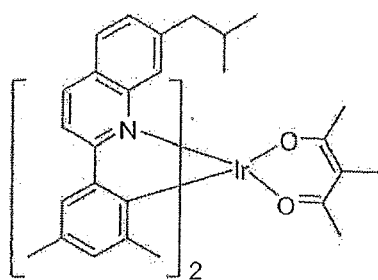
[0037] Compounds are provided, the compounds having the formula as required by claim 1.

[0038] The compounds described herein provide high device efficiency and stability, and a very narrow spectrum among other desirable properties. It is thought that a branched substituents at least at one of R_x and R_y, in combination with the methyl substituents on the phenyl ring (ring B) of the compound may provide for the very narrow emission spectrum and other remarkably good properties of the compound.

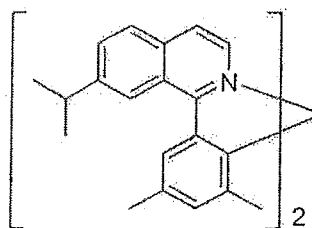
[0039] Specific compounds are provided wherein the compound is selected from the group consisting of:



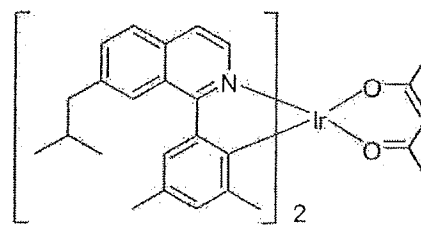
Compound 1



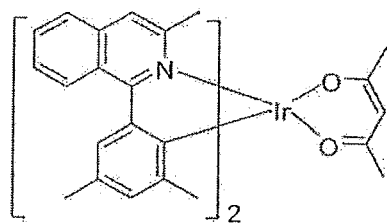
Compound 8



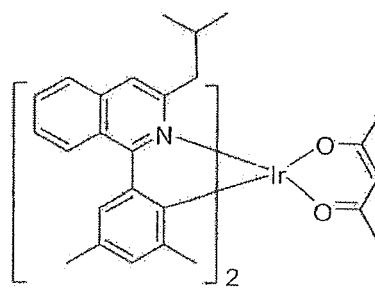
Compound 9



Compound 10



Compound 11

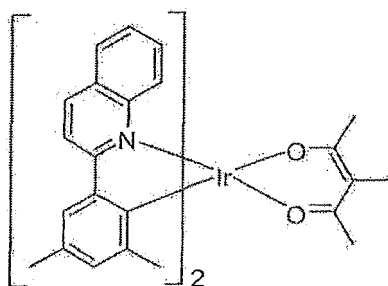


Compound 12

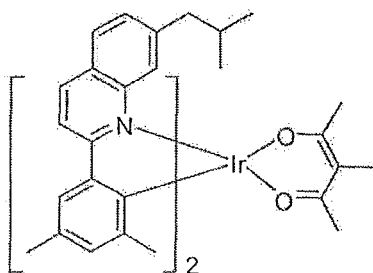
[0040] An organic light emitting device is also provided. The device comprises an anode, a cathode, and an organic layer that is disposed between the anode and the cathode. The organic layer further comprising a compound as defined by claim 5.

[0041] The organic layer of the device is an emissive layer comprising the compound and a host. In one example, the compound is the emissive material. In another example, the host is a metal coordination complex. The host material can be BAiq. In another example, the compound of the device is the emissive material and the host is a metal coordination complex. The host material can be BAiq.

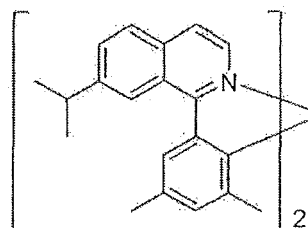
[0042] Additionally, an organic light emitting device is provided. The device comprises an anode, a cathode, and an organic layer disposed between the anode and the cathode, the organic layer comprising a compound selected from the group consisting of:



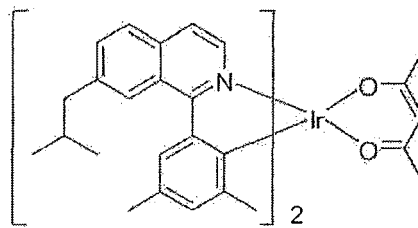
Compound 1



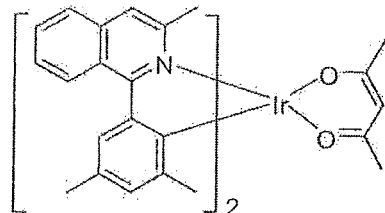
Compound 8



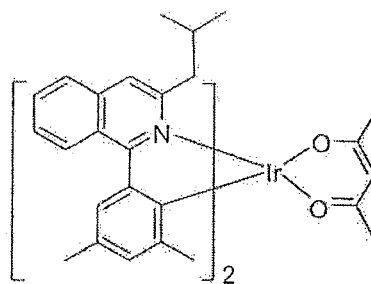
Compound 9



Compound 10



Compound 11

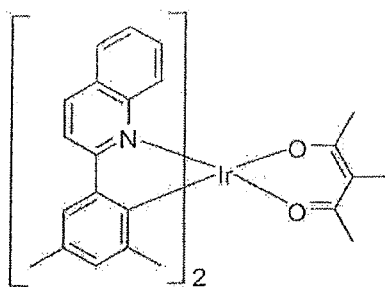


Compound 12

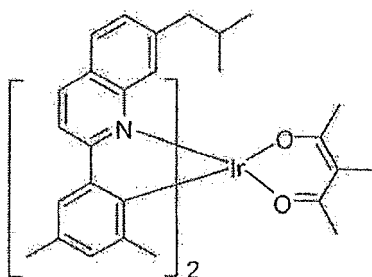
[0043] Additionally, the organic later of the device can be an emissive layer comprising the compound and a host. The inventive compound can be the emissive material and the host can be a metal coordination complex. For example, the host can be BAlq.

[0044] A consumer product is also provided. The consumer product comprising a device, the device further comprising an anode, a cathode, and an organic layer disposed between the anode and the cathode. The organic layer further comprising a compound as defined in claim 1.

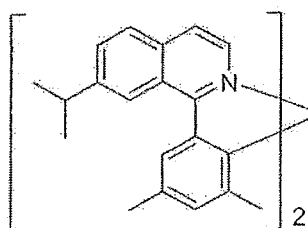
[0045] The consumer product comprises a device, the device further comprising an anode, a cathode, and an organic layer disposed between the anode and the cathode. The organic layer further comprising a compound selected from the group consisting of:



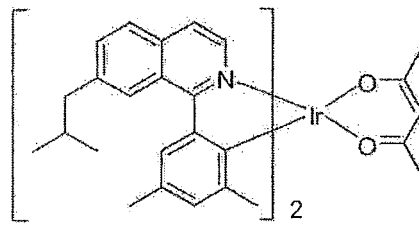
Compound 1



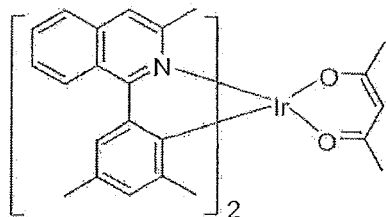
Compound 8



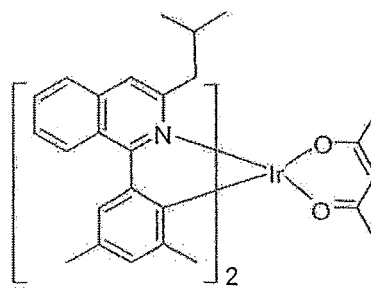
Compound 9



Compound 10

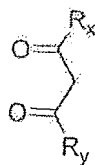


Compound 11

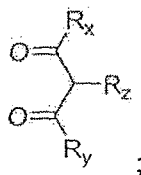


Compound 12

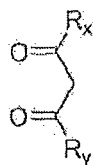
[0046] Additionally, a method not according to the invention is described comprising reacting



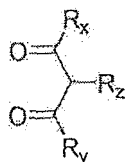
with R_z-X to form the free base



separating unreacted

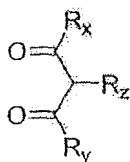


and the product

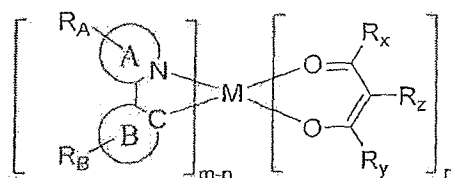


by column chromatography using a stationary phase consisting of alumina; wherein R_x and R_y are each independently selected from the group consisting of hydrogen, alkyl, heteroalkyl, aryl, or heteroaryl groups; wherein R_z is selected from the group consisting of alkyl, heteroalkyl, aryl, or heteroaryl groups; and wherein $X = Cl, Br, I, OTf, OTs$ or OH .

[0047] The method can further comprise reacting

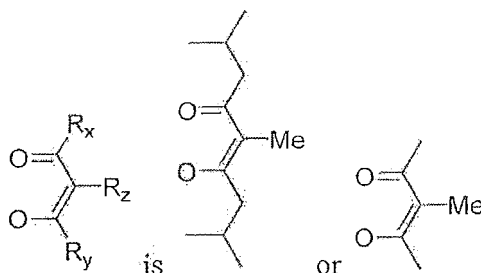


with a metal M and one or more ligands to form a compound having the formula:



wherein M is a metal of atomic weight higher than 40; wherein A and B are each independently a 5 or 6-membered aromatic or heteroaromatic ring, and $A-B$ represents a bonded pair of aromatic or heteroaromatic rings coordinated to the metal via a nitrogen atom on ring A and an sp^2 hybridized carbon atom on ring B ; wherein R_A and R_B each represent no substitution or one or more substituents; wherein each substituent of R_A and R_B is independently selected from the group consisting of alkyl, heteroalkyl, aryl, or heteroaryl groups; wherein m is the oxidation state of the metal; and wherein n is an integer less than m and at least 1.

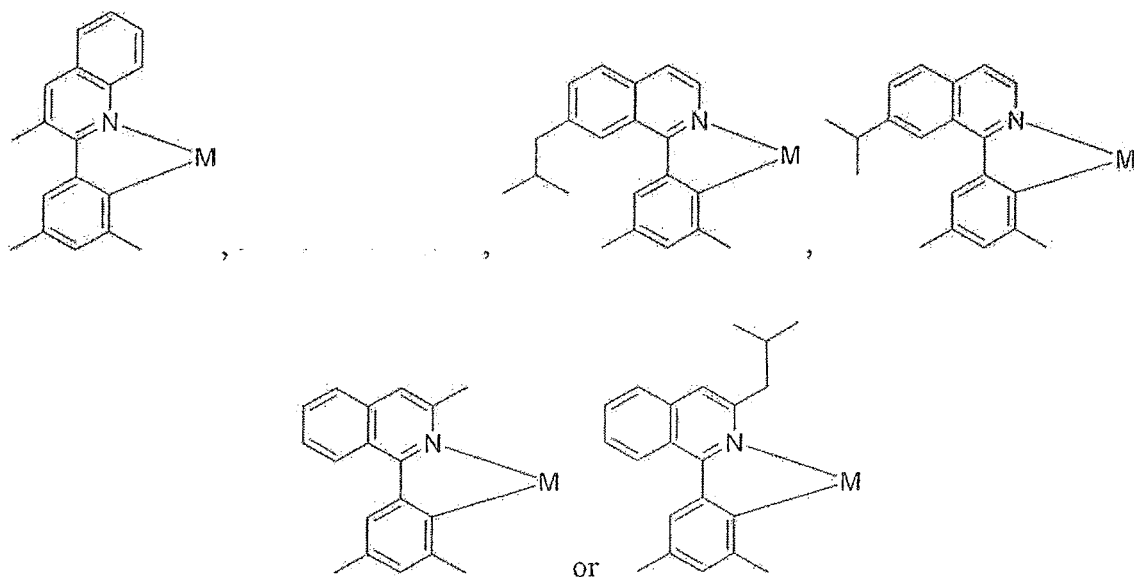
[0048] Additionally, the method can further comprise wherein R_z is a methyl group; and wherein



[0049] Isotopic analogues of the compounds provided herein where hydrogen has been replaced by deuterium are also included.

[0050] Additionally, an organometallic compound is provided. The organometallic compound contains a structure

selected from the group consisting of



wherein M is a metal with an atomic weight greater than 40.

[0051] The organometallic compound provided can have M as Ir.

[0052] The organometallic compound provided can be a phosphorescent material.

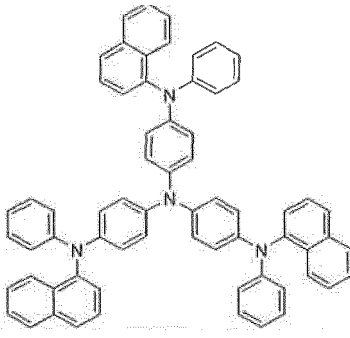
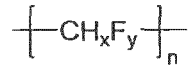
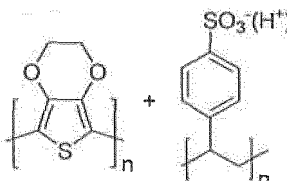
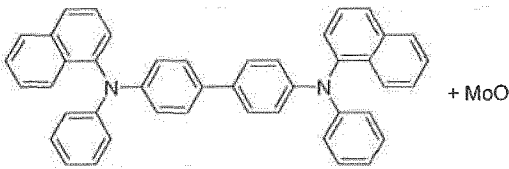
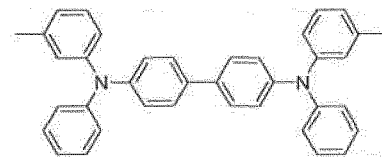
[0053] The materials described herein as useful for a particular layer in an organic light emitting device may be used in combination with a wide variety of other materials present in the device. For example, emissive dopants disclosed herein may be used in conjunction with a wide variety of hosts, transport layers, blocking layers, injection layers, electrodes and other layers that may be present. The materials described or referred to below are non-limiting examples of materials that may be useful in combination with the compounds disclosed herein, and one of skill in the art can readily consult the literature to identify other materials that may be useful in combination.

[0054] In addition to and / or in combination with the materials disclosed herein, many hole injection materials, hole transporting materials, host materials, dopant materials, exciton/hole blocking layer materials, electron transporting and electron injecting materials may be used in an OLED. Non-limiting examples of the materials that may be used in an OLED in combination with materials disclosed herein are listed in Table 1 below. Table 1 lists non-limiting classes of materials, non-limiting examples of compounds for each class, and references that disclose the materials.

TABLE 1


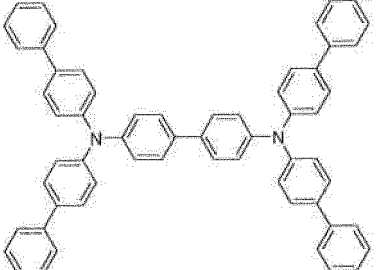
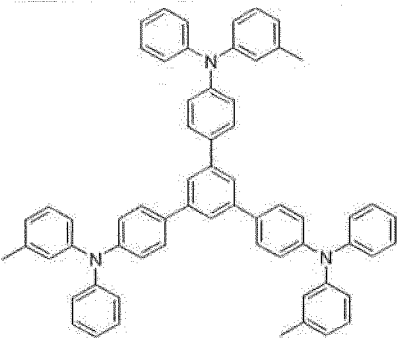
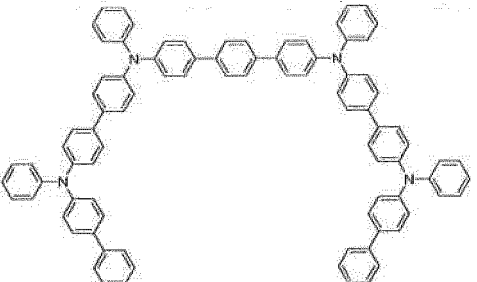
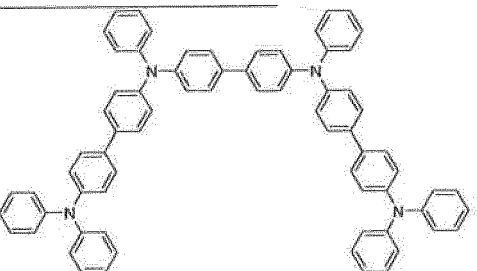
MATERIAL	EXAMPLES OF MATERIAL	PUBLICATIONS
Hole injection materials		
Phthalocyanine and porphyrin compounds		Appl. Phys. Lett. 69, 2160 (1996)

(continued)

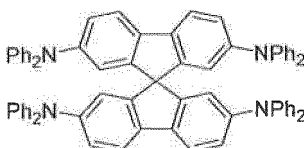
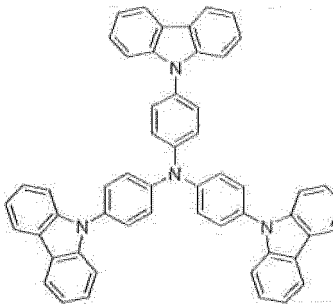
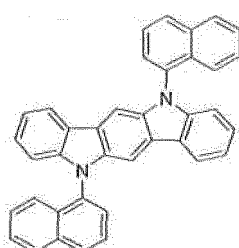
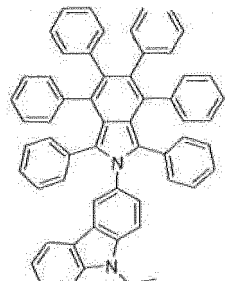
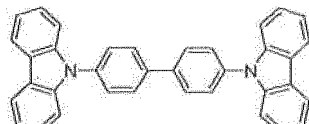
MATERIAL	EXAMPLES OF MATERIAL	PUBLICATIONS
Hole injection materials		
Starburst triarylamines		J. Lumin. 72-74, 985 (1997)
CF_x Fluorohydrocarbon polymer		Appl. Phys. Lett. 78, 673 (2001)
Conducting polymers (e.g., PEDOT: PSS, polyaniline, polythiophene)		Synth. Met. 87, 171 (1997)
Arylamines complexed with metal oxides such as molybdenum and tungsten oxides		SID Symposium Digest, 37, 923 (2006)
Hole transporting materials		
Triarylamines (e.g., TPD, α -NPD)		Appl. Phys. Lett. 51, 913 (1987)

(continued)

Hole transporting materials

5		US5061569
10		EP650955
15		J. Mater. Chem. 3, 319 (1993)
20		Appl. Phys. Lett. 90, 183503 (2007)
25		Appl. Phys. Lett. 90, 183503 (2007)

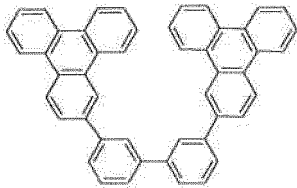
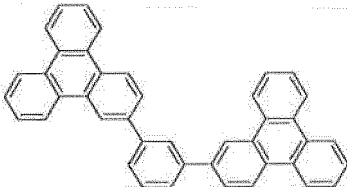
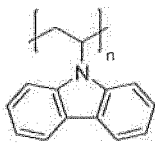
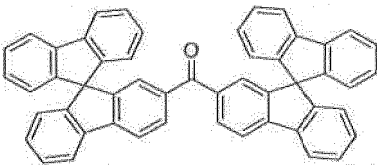
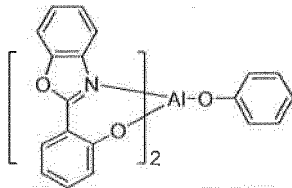
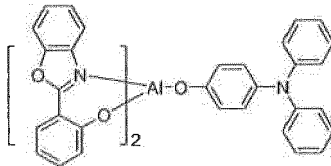
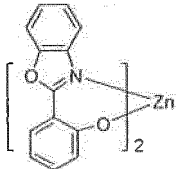
(continued)

Hole transporting materials		
Triaylamine on spirofluorene core		Synth. Met. 91, 209 (1997)
Arylamine carbazole compounds		Adv. Mater. 6, 677 (1994)
Indolocarbazoles		Synth. Met. 111, 421 (2000)
Isoindole compounds		Chem. Mater. 15, 3148 (2003)
Phosphorescent OLED host materials		
Red hosts		
Arylcarbazoles		Appl. Phys. Lett. 78, 1622 (2001)

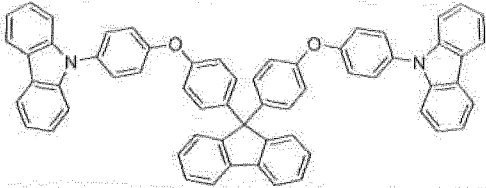
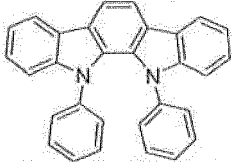
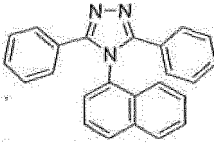
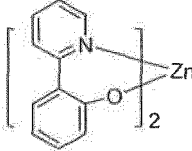
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Phosphorescent OLED host materials		
Red hosts		
Metal 8-hydroxyquinolates (e.g., Alq ₃ , BAlq)		Nature 395, 151 (1998)
		US20060202194
		WO2005014551
Metal phenoxybenzothiazole compounds		Appl. Phys. Lett. 90, 123509 (2007)
Conjugated oligomers and polymers (e.g., polyfluorene)		Org. Electron. 1, 15 (2000)
Green hosts		
Arylcarbazoles		Appl. Phys. Lett. 78, 1622 (2001)
		US2003175553
		WO2001039234

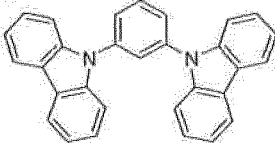
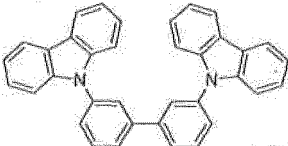
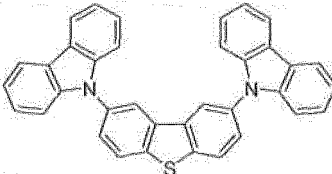
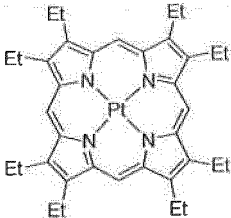
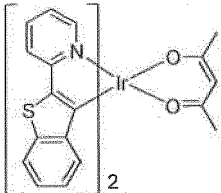
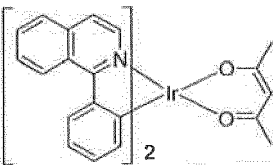
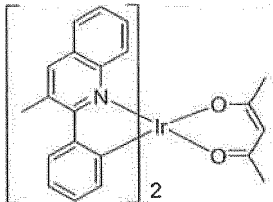
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Green hosts		
Aryltriphenylene compounds		US20060280965
		US20060280965
Polymers (e.g., PVK)		Appl. Phys. Lett. 77, 2280 (2000)
Spirofluorene compounds		WO2004093207
Metal phenoxybenzoxazole compounds		WO05089025
		WO06132173
		JP200511610

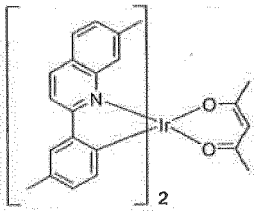
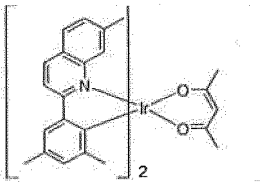
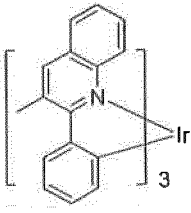
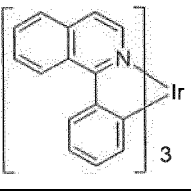
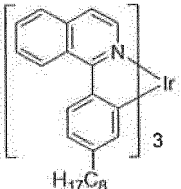
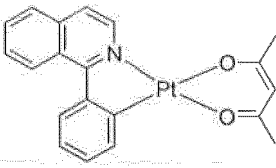
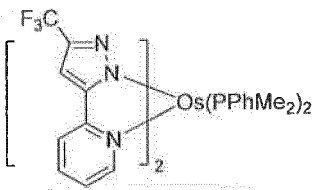
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Green hosts		
Spirofluorene-carbazole compounds		JP2007254297
Indolocabazoles		WO07063796
5-member ring electron deficient heterocycles (e.g., triazole, oxadiazole)		J. Appl. Phys. 90, 5048 (2001)
Metal phenoxy pyridine compounds		WO04107822

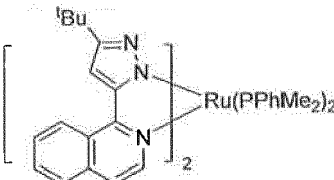
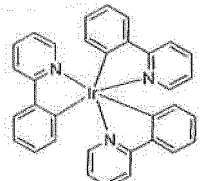
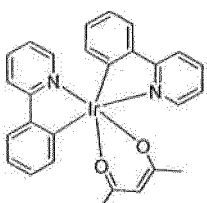
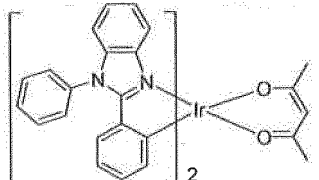
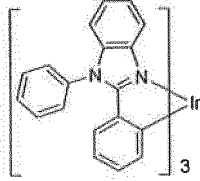
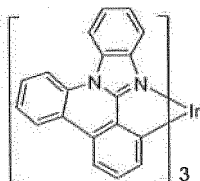
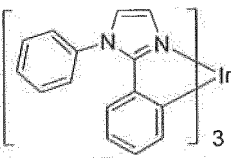
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Blue hosts		
Arylcarbazoles		Appl. Phys. Lett., 82, 2422 (2003)
		US20070190359
Dibenzothiophene-carbazole compounds		WO2006114966
Phosphorescent dopants		
Red dopants		
Heavy metal porphyrins (e.g., PtOEP)		Nature 395, 151 (1998)
Iridium(III) organometallic complexes		Appl. Phys. Lett. 78, 1622 (2001)
		US06835469
		US06835469

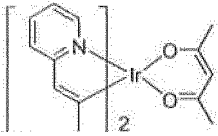
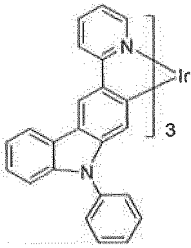
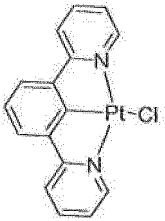
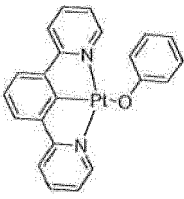
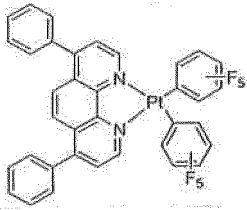
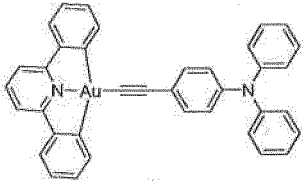
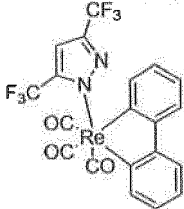
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Phosphorescent dopants		
Red dopants		
		US20060202194
		US20060202194
		US07087321
		US07087321
		Adv. Mater. 19, 739 (2007)
Platinum(II) organometallic complexes		WO2003040257
Osmium(III) complexes		Chem. Mater. 17, 3532 (2005)

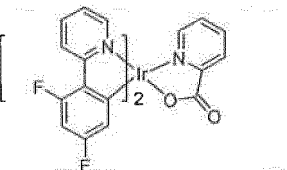
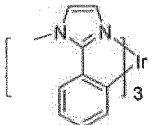
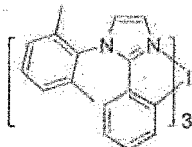
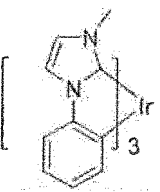
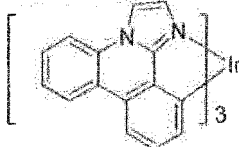
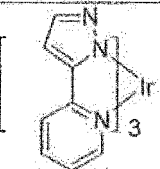
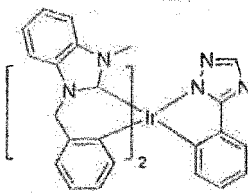
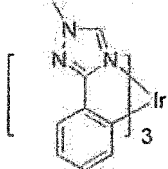
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Phosphorescent dopants		
Red dopants		
Ruthenium(II) complexes		Adv. Mater. 17, 1059 (2005)
Green dopants		
Iridium(III) organometallic complexes	 and its derivatives	Inorg. Chem. 40, 1704 (2001)
		US2002034656
		US06687266
		Chem. Mater. 16,2480 (2004)
		US2007190359
		US 2006008670 JP2007123392

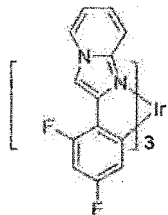
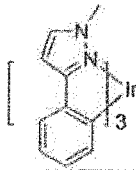
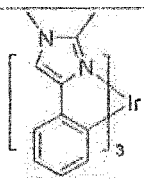
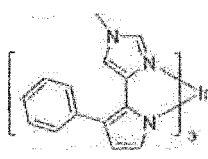
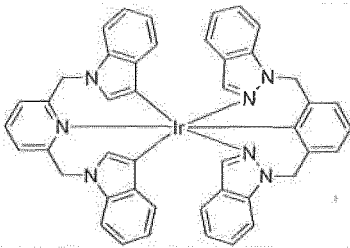
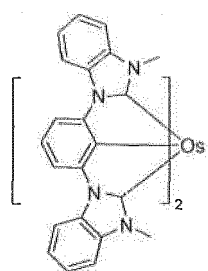
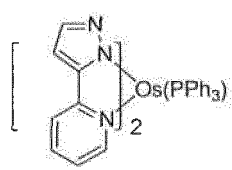
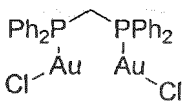
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Green dopants		
		Adv. Mater. 16, 2003 (2004)
		Angew. Chem. Int. Ed. 2006, 45, 7800
Pt(II) organometallic complexes		Appl. Phys. Lett. 86, 153505 (2005)
		Appl. Phys. Lett. 86, 153505 (2005)
		Chem. Lett. 34, 592 (2005)
Gold complexes		Chem. Commun. 2906 (2005)
Rhenium(III) complexes		Inorg. Chem. 42, 1248 (2003)

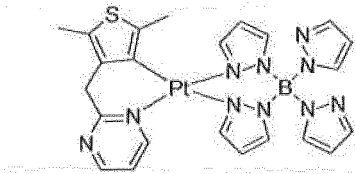
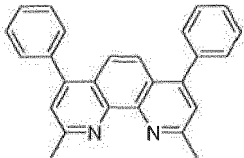
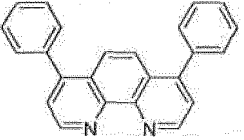
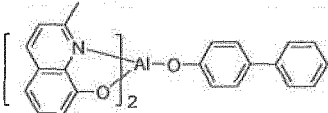
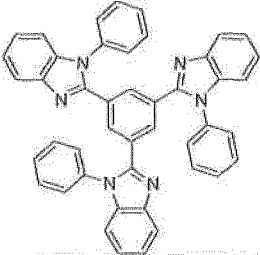
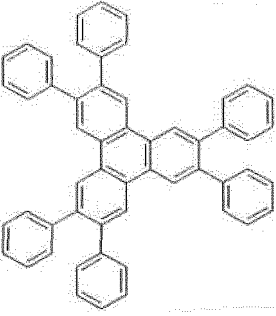
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Blue dopants		
Iridium(III) organometallic complexes		WO2002002714
		WO2006009024
		US2006251923
		WO2006056418, US2005260441
		US2007190359
		US2002134984
		Angew. Chem. Int. Ed. 47, 1 (2008)
		Chem. Mater. 18, 5119 (2006)

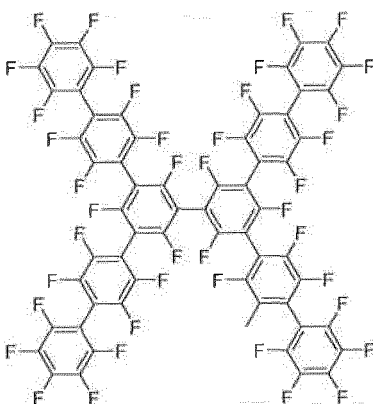
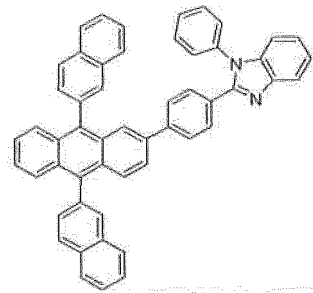
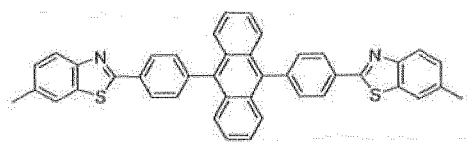
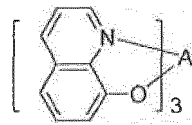
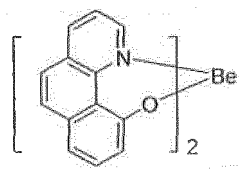
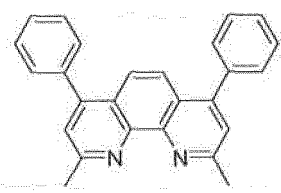
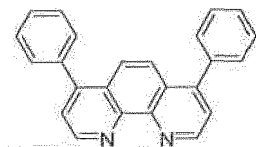
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Blue dopants		
5		Inorg. Chem. 46, 4308 (2007)
10		
15		WO05123873
20		WO05123873
25		WO07004380
30		WO06082742
35		
40	Osmium(II) complexes	US2005260449
45		
50		Organometallics 23, 3745 (2004)
55	Gold complexes	Appl. Phys. Lett. 74, 1361 (1999)
		

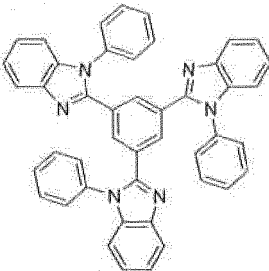
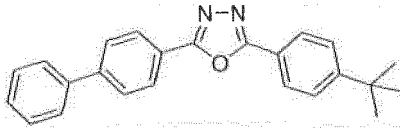
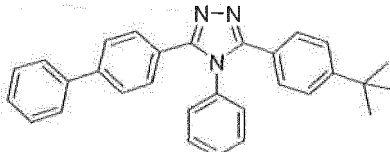
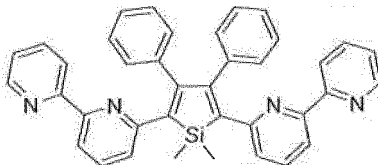
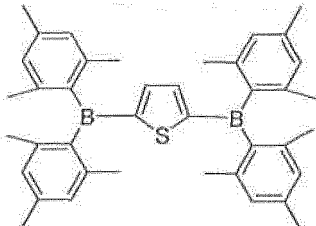
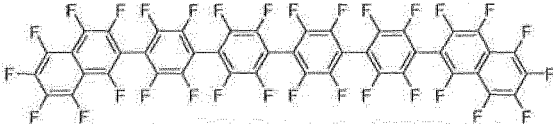
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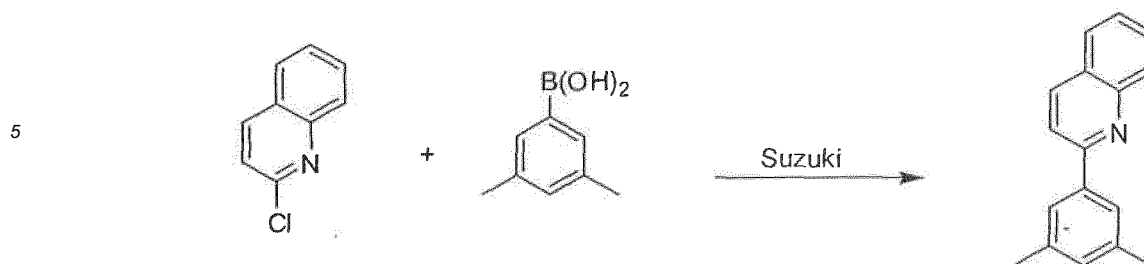
Blue dopants		
Platinum(II) complexes		WO06098120, WO06103874
Exciton/hole blocking layer materials		
Bathocuprine compounds (e.g., BCP, BPhen)		Appl. Phys. Lett. 75, 4 (1999)
		Appl. Phys. Lett. 79, 449 (2001)
Metal 8-hydroxyquinolates (e.g., BAlq)		Appl. Phys. Lett. 81, 162 (2002)
5-member ring electron deficient heterocycles such as triazole, oxadiazole, imidazole, benzoimidazole		Appl. Phys. Lett. 81, 162 (2002)
Triphenylene compounds		US20050025993

(continued)

Exciton/hole blocking layer materials		
Fluorinated aromatic compounds		Appl. Phys. Lett. 79, 156 (2001)
Electron transporting materials		
Anthracene-benzimidazole compounds		WO03060956
Anthracene-benzothiazole compounds		Appl. Phys. Lett. 89, 063504 (2006)
Metal 8-hydroxyquinolates (e.g., Alq ₃)		Appl. Phys. Lett. 51, 913 (1987)
Metal hydroxybenoquinolates		Chem. Lett. 5, 905 (1993)
Bathocuprine compounds such as BCP, BPhen, etc		Appl. Phys. Lett. 91, 263503 (2007)
		Appl. Phys. Lett. 79, 449 (2001)

(continued)

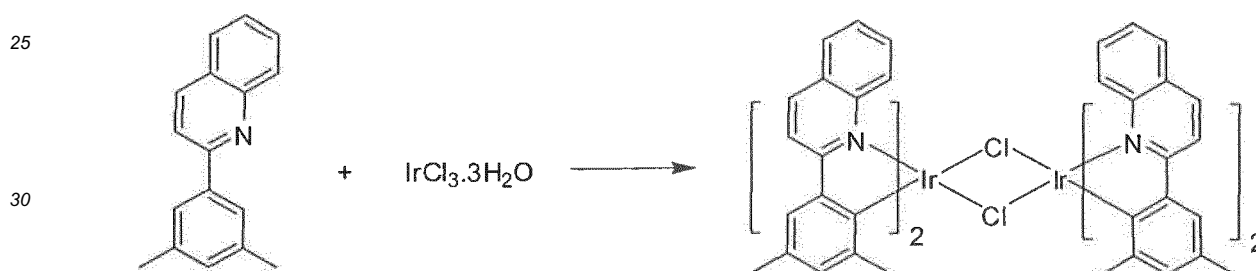
Electron transporting materials		
5-member ring electron deficient heterocycles (e.g., triazole, oxadiazole, imidazole, benzoimidazole)		Appl. Phys. Lett. 74, 865 (1999)
		Appl. Phys. Lett. 55, 1489 (1989)
		Jpn. J. Apply. Phys. 32, L917 (1993)
Silole compounds		Org. Electron. 4, 113 (2003)
Arylborane compounds		J. Am. Chem. Soc. 120, 9714 (1998)
Fluorinated aromatic compounds		J. Am. Chem. Soc. 122, 1832 (2000)



[0056] 2-chloroquinoline (9.0g, 54.4 mmol), 3,5-dimethylphenylboronic acid (9.2g, 59.8 mmol), $\text{Pd}(\text{PPh}_3)_4$ (1.8g, 1.5mmol), K_2CO_3 (22.4g, 163mmol), 1,2-dimethoxyethane (150 mL) and water (150 mL) were charged in a 500mL round bottom flask. The reaction mixture was heated to reflux under nitrogen for 18h. The reaction mixture was then cooled to ambient and the organic phase was separated from the aqueous phase. The aqueous phase was washed with ethyl acetate and all the organic components were combined and dried over anhydrous magnesium sulphate. The solvent was then removed under vacuum and the product was purified using silica gel chromatography (10% ethyl acetate in hexane as eluent). The material obtained was further purified by vacuum distillation to yield 12.2 g (95% yield) of product as a colorless oil.

20 Step 2

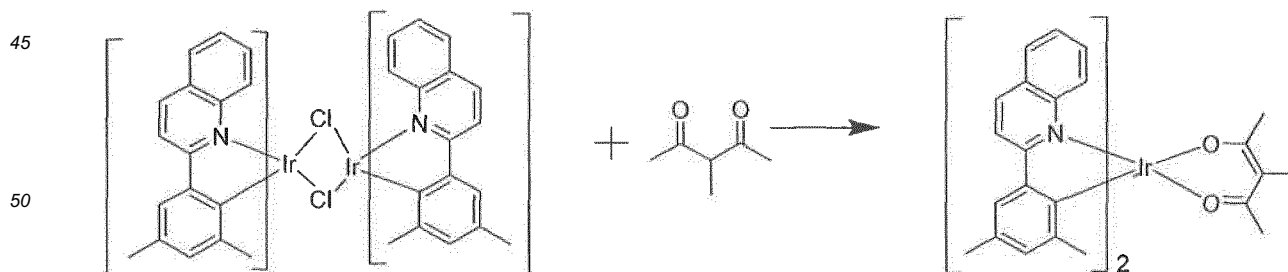
[0057]



[0058] The ligand from step 1 (46g, 197.4 mmol), 2-ethoxyethanol (536 mL) and water (178 mL) were charged in a 1L three-neck round bottom flask. Nitrogen gas was bubbled through the reaction mixture 45 min. $\text{IrCl}_3 \cdot \text{H}_2\text{O}$ (32.0 g 86.2 mmol) was then added and the reaction mixture was heated to reflux under nitrogen for 17 hours. The reaction mixture was cooled to ambient and filtered. The dark gray residue was washed with methanol (4 x 150 mL) followed by hexanes (3 x 300 mL). 36.5 gram of the dichlorobridged Iridium dimer was obtained after drying in vacuum oven.

40 Step 3

[0059]



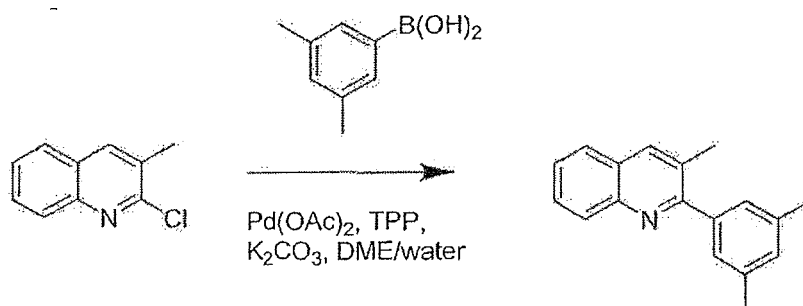
[0060] Dichlorobridged Iridium dimer from step 2 (3.0g, 2.2 mmol), 10 mol eq 3-methyl-2,4-pentanedione (2.5g.), 20 mol eq of Na_2CO_3 (6.3g) and 25 mL of 2-ethoxyethanol were placed in a 250 mL round bottom flask. The reaction mixture was stirred at ambient for 24 hours. 2g of celite and 200mL of dichloromethane was added to the reaction mixture to dissolve the product. The mixture was then filtered through a bed of celite. The filtrate was then passed through a silica/alumina plug and washed with dichloromethane. The clarified solution was then filtered through GF/F filter paper

the filtrate was heated to remove most of the dichloromethane. 20 mL of isopropanol was then added and the slurry was cooled to ambient and the product was filtered and washed with isopropanol and dried to give 3.2g of crude product (97% yield). This product was then recrystallised twice using dichloromethane and isopropanol and then sublimed.

5 Synthesis of Compound 2 (not according to the invention)

Step 1

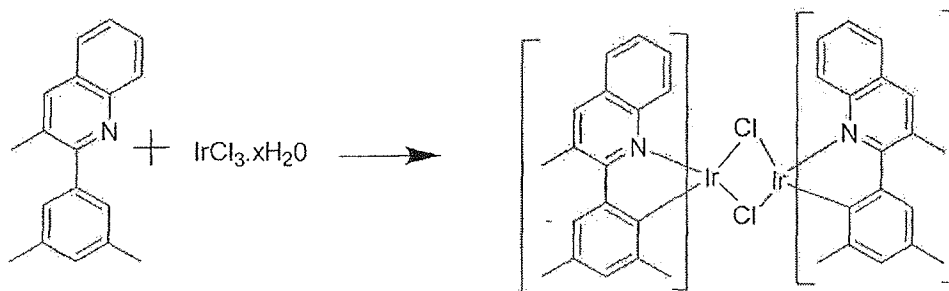
[0061]



[0062] 2-chloro-3-methyl-quinoline (4.5g, 25.0 mmol), dimethylphenylboronic acid (4.6g, 30mmol), triphenylphosphine (1.60g, 6.11mmol), and potassium carbonate (12.67g, 91.69mmol) were charged in a 250mL round bottom flask. 25mL water and 25mL of dimethoxyethane was added to the flask. Nitrogen was bubbled through the reaction mixture for 30 min. Palladium acetate (0.34g, 1.53mmol) was then added to the reaction mixture was then refluxed overnight under an atmosphere of nitrogen. The product was extracted with ethyl acetate, washed with water, and dried over anhydrous magnesium sulfate. The product was purified using silica gel chromatography (5-15% ethyl acetate in hexane as eluent) to give a light yellow oil (85% yield). Further purification was done via vacuum distillation

Step 2

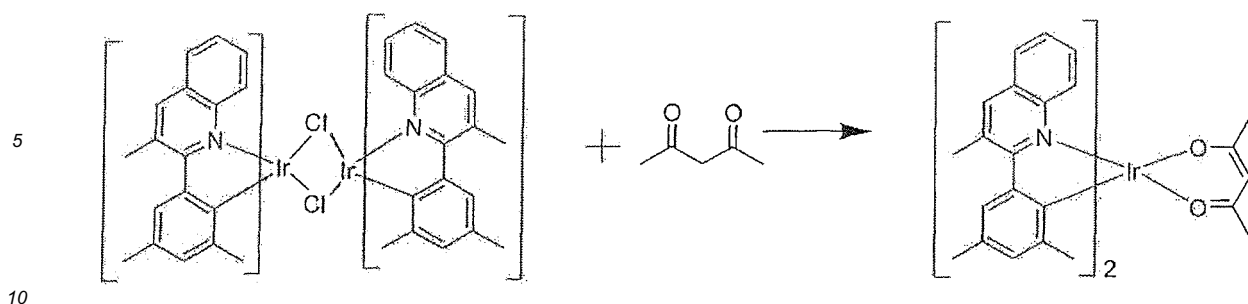
[0063]



[0064] Ligand from step 1 (16g, 65mmol), iridium chloride (5.0g, 14mmol), 2-ethoxyethanol (75mL) and water (12.5mL) was charged in a 250mL round bottom flask.. The reactor contents were heated to 102°C under an atmosphere of nitrogen for 16-19h. The dichloro iridium bridged dimer was not isolated.

Step 3

[0065]



[0066] The reactor contents from step 2 were cooled to ambient. 2,4-pentanedione (14.0g 140 mmol) and sodium carbonate (30.0g, 280mmol) were added to the reactor. The reaction mixture was stirred at ambient for 24h. 5g of celite and 500mL of dichloromethane was added to the reaction mixture to dissolve the product. The mixture was then filtered through a bed of celite. The filtrate was then passed through a silica/alumina plug and washed with dichloromethane. The clarified solution was then filtered through GF/F filter paper the filtrate was heated to remove most of the dichloromethane. 20 mL of isopropanol was then added and the slurry was cooled to ambient and the product was filtered and washed with isopropanol and dried to give 6.3g of crude product (57% yield). This product was then recrystallised twice using dichloromethane and isopropanol and then sublimed.

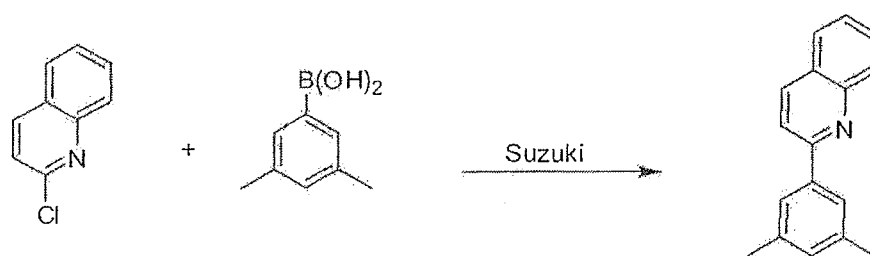
Synthesis of Compound 3 (not according to the invention)

Step 1

[0067]

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[0068] 2-chloroquinoline (9.0g, 54.4 mmol), 3,5-dimethylphenylboronic acid (9.2g, 59.8 mmol), $\text{Pd}(\text{PPh}_3)_4$ (1.8g, 1.5mmol), K_2CO_3 (22.4g, 163mmol), 1,2-dimethoxyethane (150 mL) and water (150 mL) were charged in a 500mL round bottom flask. The reaction mixture was heated to reflux under nitrogen for 18h. The reaction mixture was then cooled to ambient and the organic phase was separated from the aqueous phase. The aqueous phase was washed with ethyl acetate and all the organic components were combined and dried over anhydrous magnesium sulphate. The solvent was then removed under vacuum and the product was purified using silica gel chromatography (10% ethyl acetate in hexane as eluent). The material obtained was further purified by vacuum distillation to yield 12.2 g (95% yield) of product as a colorless oil.

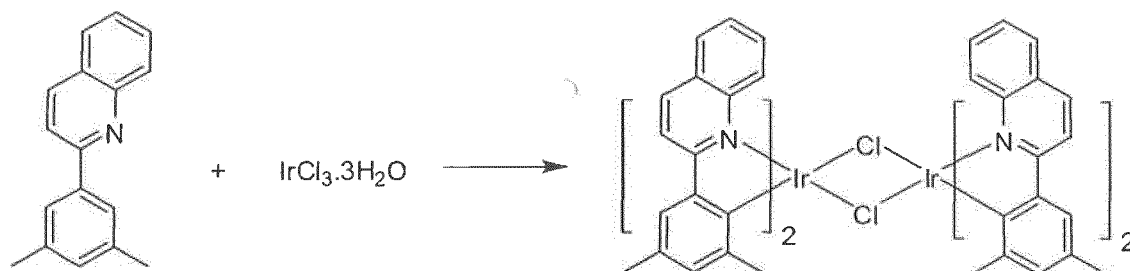
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Step 2

[0069]

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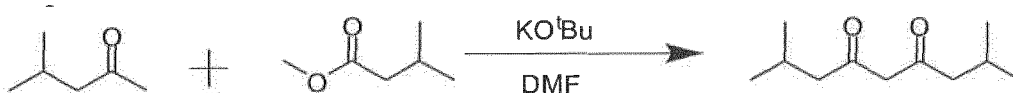
55



[0070] The ligand from step 1 (46g, 197.4 mmol), 2-ethoxyethanol (536 mL) and water (178 mL) were charged in a 1L three-neck round bottom flask. Nitrogen gas was bubbled through the reaction mixture 45 min. $\text{IrCl}_3 \cdot \text{H}_2\text{O}$ (32.0 g 86.2 mmol) was then added and the reaction mixture was heated to reflux under nitrogen for 17 hours. The reaction mixture was cooled to ambient and filtered. The dark gray residue was washed with methanol (4 x 150 mL) followed by hexanes (3 x 300 mL). 36.5 gram of the dichlorobridged Iridium dimer was obtained after drying in vacuum oven.

Step 3

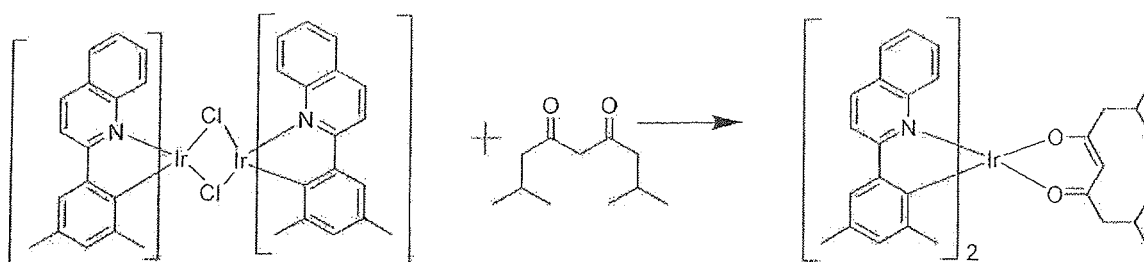
[0071]



[0072] N,N dimethylformamide (DMF) (1L) and potassium tert-butoxide (135.0g 1.2mol) were heated to 50°C under nitrogen. Methyl 3-methylbutanoate (86.0g, 0.75mol) was added dropwise from a dropping funnel followed by a solution of 4-methylpentane-2-one (50g, 1mol) in 100mL DMF. The progress of the reaction was monitored by GC. When the reaction was completed, the mixture was cooled to ambient and slowly neutralized with 20% H_2SO_4 solution. Water (300mL) was added and two layers formed. The layer containing the 2,8-dimethylnonane-4,6-dione was purified using vacuum distillation to give 40g of a pink oil (43% yield).

Step 4

[0073]

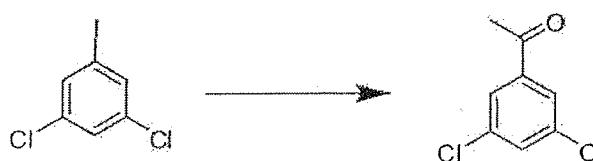


[0074] Dichlorobridged Iridium dimer from step 2 (3.0g, 2.2 mmol), 10 mol eq 2,8-dimethylnonane-4,6-dione (4.1g), 20 mol eq of Na_2CO_3 (6.3g) and 25 mL of 2-ethoxyethanol were placed in a 250 mL round bottom flask. The reaction mixture was stirred at ambient for 24 hours. 2g of celite and 200mL of dichloromethane was added to the reaction mixture to dissolve the product. The mixture was then filtered through a bed of celite. The filtrate was then passed through a silica/alumina plug and washed with dichloromethane. The clarified solution was then filtered through GF/F filter paper the filtrate was heated to remove most of the dichloromethane. 20 mL of isopropanol was then added and the slurry was cooled to ambient and the product was filtered and washed with isopropanol and dried to give 2.9g of crude product (79% yield). This product was then recrystallized twice using dichloromethane and isopropanol and then sublimed.

Synthesis of Compound 4 (not according to the invention)

Step 1

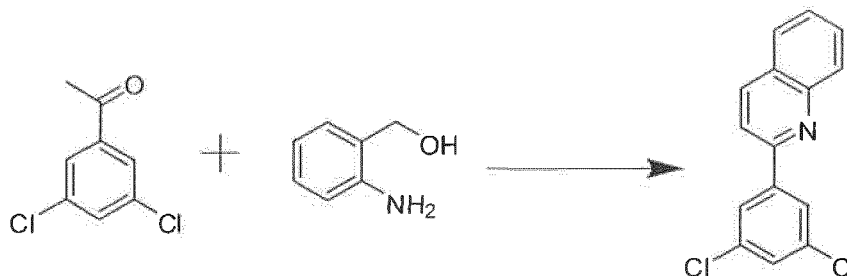
[0075]



[0076] Dichloriodobenzene (37.0g 136mmol), $\text{Pd}_2(\text{dba})_3$ (1.5g, 1.6mmol), Lithium chloride (29.0g, 682mmol) was dissolved in 100mL of DMF in a 500mL round bottom flask. 64.0mL of acetic anhydride and 47.0mL of N-Ethyldiispropylamine was then added to the reaction mixture. The reaction was heated to 100°C for 8 hours. Water was added to the reaction mixture and the product was extracted with ethylacetate and chromatographed using a silica gel column with ethyl acetate and hexanes as the eluent. 8g of product was obtained.

Step 2

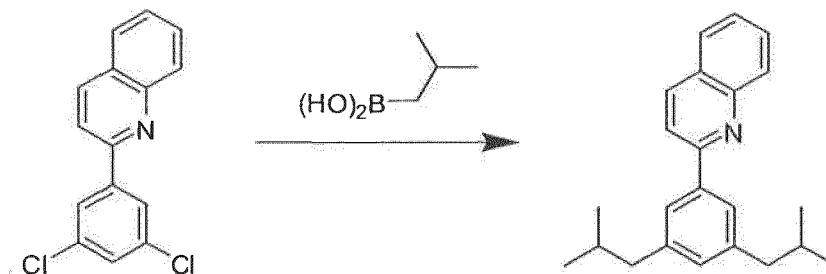
[0077]



[0078] 2-aminobenzyl alcohol (6.0g, 48mmol), 3,5-dichloroacetophenone (12.0g, 63.5mmol), $\text{RuCl}_2(\text{PPh}_3)_3$ (0.5g 10mol%), and KOH (2.4g, 42.0mmol) was refluxed in 100ml of toluene for 10 hours. Water was collected from the reaction using a Dean-Stark trap. The reaction mixture was allowed to cool to room temperature and filtered through a silica gel plug. The product was further purified with a silica gel column using 2% ethyl acetate in hexanes as the eluent. 4.0g product was obtained after column (30% yield). The product was further recrystallized from isopropanol. 3.5g of desired product was obtained.

Step 3

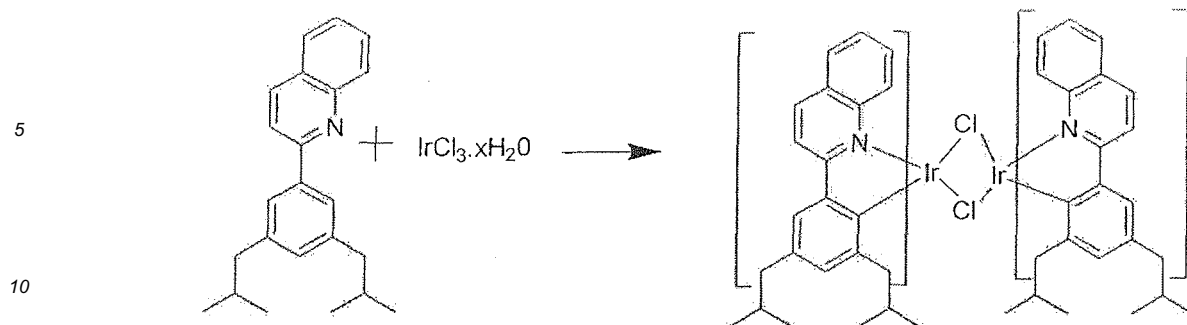
[0079]



[0080] 2-(3,5-dichlorophenyl)quinoline (4.0g, 14.6mmol), isobutylboronic acid (6.0g, 58.4mol), $\text{Pd}_2(\text{dba})_3$ (0.13g, 1mol%), dicyclohexylphosphino-2',6'-dimethoxybiphenyl (0.24, 4mol%), potassium phosphate monohydrate (10g, 13.8mmol) was mixed in 100mL of toluene in a 250mL round bottom flask. Nitrogen was bubbled through the mixture for 20 minutes and the mixture refluxed in a nitrogen atmosphere overnight. The reaction mixture was allowed to cool and the solvent removed under vacuum. The crude product was chromatographed using a silica gel column with 2% ethyl acetate in hexanes as the eluent. The solvent was then removed under vacuo to give 3.5g of product.

Step 4

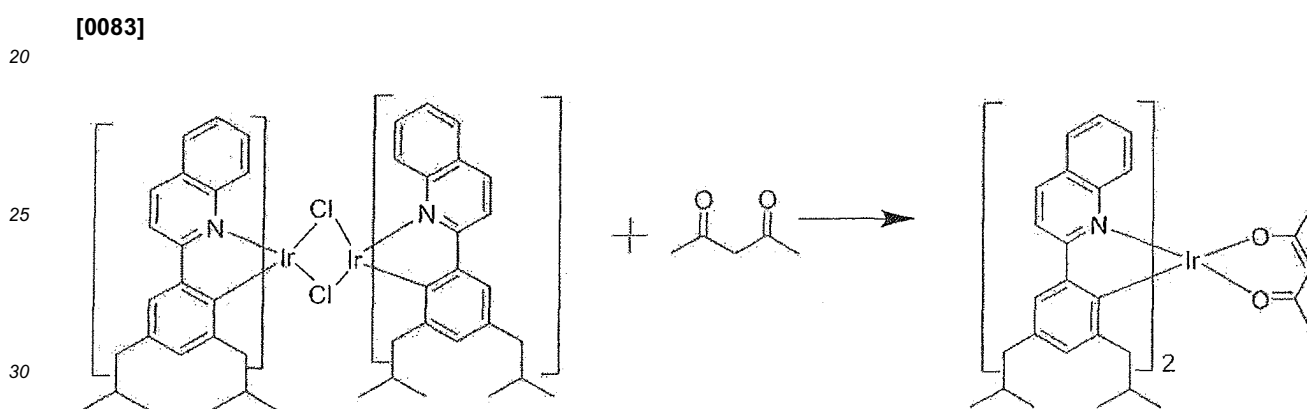
[0081]



[0082] Ligand from step 3 (20g, 65mmol), iridium chloride (5.0g, 14mmol), 2-ethoxyethanol (75mL) and water (12.5mL) was charged in a 250mL round bottom flask. The reactor contents were heated to 102°C under an atmosphere of nitrogen for 16-19h. The dichloro iridium bridged dimer was not isolated.

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Step 5



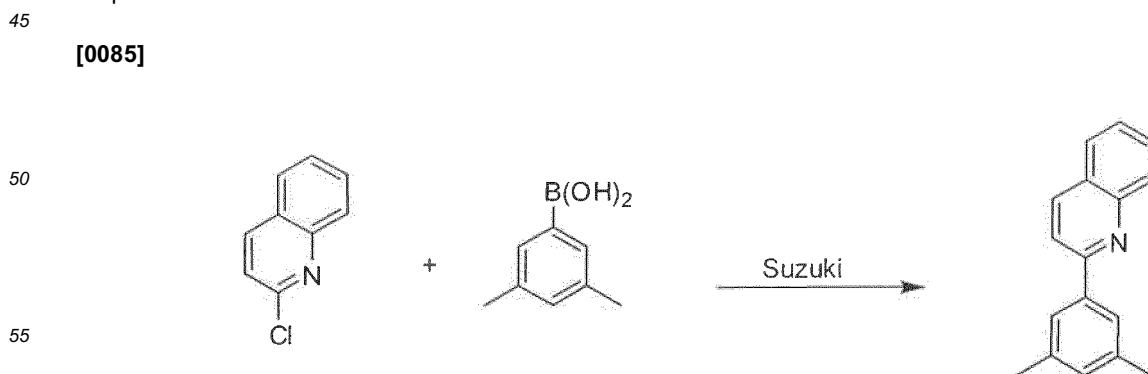
[0084] The reactor contents from step 4 were cooled to ambient. 2,4-pentanedione (14.0g 140 mmol) and sodium carbonate (30.0g, 280mmol) were added to the reactor. The reaction mixture was stirred at ambient for 24h. 5g of celite and 500mL of dichloromethane was added to the reaction mixture to dissolve the product. The mixture was then filtered through a bed of celite. The filtrate was then passed through a silica/alumina plug and washed with dichloromethane. The clarified solution was then filtered through GF/F filter paper the filtrate was heated to remove most of the dichloromethane. 20 mL of isopropanol was then added and the slurry was cooled to ambient and the product was filtered and washed with isopropanol and dried to give 7.1g of crude product(55%yield). This product was then recrystallised twice using dichloromethane and isopropanol and then sublimed.

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Synthesis of Compound 5 (not according to the invention)

Step 1

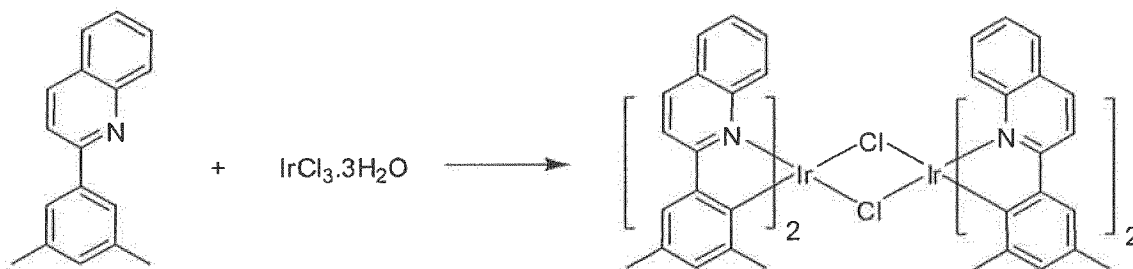


[0086] 2-chloroquinoline (9.0g, 54.4 mmol), 3,5-dimethylphenylboronic acid (9.2g, 59.8 mmol), Pd(PPh₃)₄ (1.8g,

1.5mmol), K_2CO_3 (22.4g, 163mmol), 1,2-dimethoxyethane (150 mL) and water (150 mL) were charged in a 500mL round bottom flask. The reaction mixture was heated to reflux under nitrogen for 18h. The reaction mixture was then cooled to ambient and the organic phase was separated from the aqueous phase. The aqueous phase was washed with ethyl acetate and all the organic components were combined and dried over anhydrous magnesium sulphate. The solvent was then removed under vacuum and the product was purified using silica gel chromatography (10% ethyl acetate in hexane as eluent). The material obtained was further purified by vacuum distillation to yield 12.2 g (95% yield) of product as a colorless oil.

Step 2

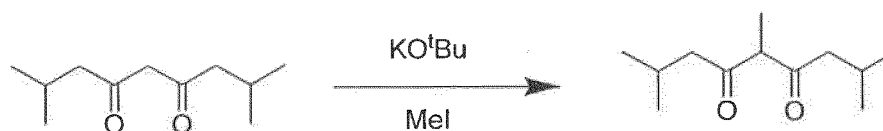
[0087]



[0088] The ligand from step 1 (46g, 197.4 mmol), 2-ethoxyethanol (536 mL) and water (178 mL) were charged in a 1L three-neck round bottom flask. Nitrogen gas was bubbled through the reaction mixture 45 min. $IrCl_3 \cdot H_2O$ (32.0 g 86.2 mmol) was then added and the reaction mixture was heated to reflux under nitrogen for 17 hours. The reaction mixture was cooled to ambient and filtered. The dark gray residue was washed with methanol (4 x 150 mL) followed by hexanes (3 x 300 mL). 36.5 gram of the dichlorobridged Iridium dimer was obtained after drying in vacuum oven.

Step 3

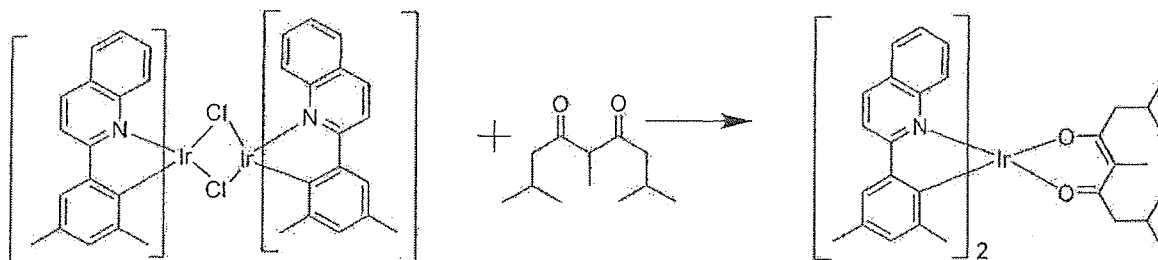
[0089]



[0090] 2,8-dimethylnonane-4,6-dione (10.0g, 5.4mmol), potassium tert butoxide (7.0g, 6.5mmol) and 150mL of anhydrous THF was charged in a 3neck 250mL dry round bottom flask.. The reaction mixture was stirred under an atmosphere of nitrogen at ambient for 1h. Iodomethane (15g, 105mmol) was added to the reaction mixture via a needle and syringe. The reaction mixture was continued to stir at ambient for a further 4h. The reaction was monitored by GC. The reaction was quenched with 100mL of water and acidified using 1M hydrochloric acid. The product was extracted with ethyl acetate and chromatographed using silica gel chromatography (using 1-5%ethyl acetate in hexanes). HPLC revealed that the product (2,5,8-trimethylnonane-4,6-dione) contained a mixture of 2,8-dimethylnonane-4,6-dione (starting material). These two products were separated via chromatography using deactivated basic alumina with 1-5% ethyl acetate in hexanes as the mobile phase to give 3.6g of product (33%yield).

Step 4

[0091]

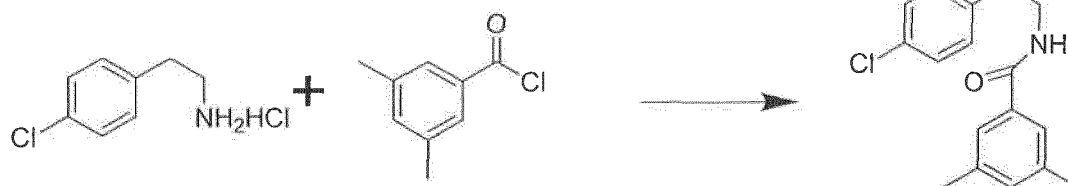


[0092] Dichlorobridged Iridium dimer from step 2 (1.0g, 0.7 mmol), 10 mol eq 2,5,8-trimethylnonane-4,6-dione (1.4g), 20 mol eq of Na_2CO_3 (2.0g) and 25 mL of 2-ethoxyethanol were placed in a 250 mL round bottom flask. The reaction mixture was stirred at ambient for 24 hours. 1 g of celite and 100mL of dichloromethane was added to the reaction mixture to dissolve the product. The mixture was then filtered through a bed of celite. The filtrate was then passed through a silica/alumina plug and washed with dichloromethane. The clarified solution was then filtered through GF/F filter paper the filtrate was heated to remove most of the dichloromethane. 10 mL of isopropanol was then added and the slurry was cooled to ambient and the product was filtered and washed with isopropanol and dried to give 0.7g of crude product (57% yield). This product was then recrystallised twice using dichloromethane and isopropanol and then sublimed.

Synthesis of Compound 6 (not according to the invention)

Step 1

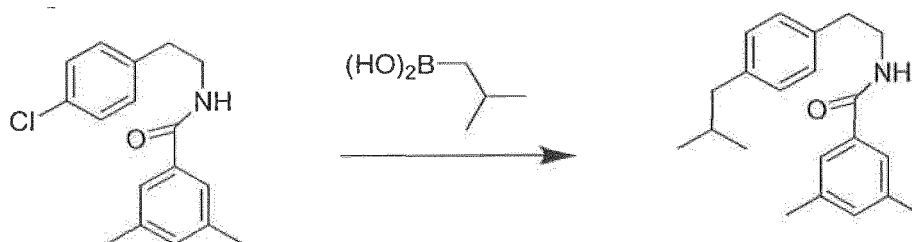
[0093]



[0094] 4-chlorophenylethylamine hydrochloride (10.0g, 64mmol), pyridine (15.3g, 193mmol) and dichloromethane (50mL) were added to a 3-neck round bottom flask. The solution was cooled in an ice bath and 3,5-dimethylbenzoyl chloride (10.8g, 64mmol) was added slowly. The solution was allowed to warm to room temperature and stirred for 12 hours. Dichloromethane was added and the organic phase was washed with water, followed by 5% HCl solution, then 5% NaOH solution and then dried over anhydrous MgSO_4 . The solvent was evaporated under vacuum resulting in 15g of crude product (82% yield) which was used without further purification.

Step 2

[0095]

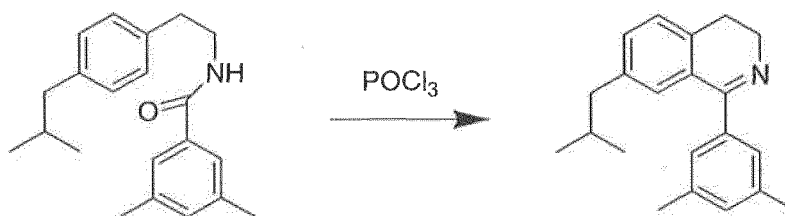


[0096] N-(4-chlorophenylethyl)benzamide (15g, 52mmol), isobutylboronic acid (10.6g, 104mmol), $\text{Pd}_2(\text{dba})_3$ (1mol%), 2-dicyclohexylphosphino-2',6'-dimethoxybiphenyl (4 mol%), potassium phosphate monohydrate (22.0g 212mmol) 200ml of toluene was charged in a 250mL round bottom flask. Nitrogen was bubbled through the reaction mixture for 20 minutes

and heated to reflux for 18h overnight. The reaction mixture was allowed to cool to ambient temperature and the crude product was purified by column chromatography using 2% ethyl acetate in hexanes as solvent. 15g of desired product was obtained (93%yield).

Step 3

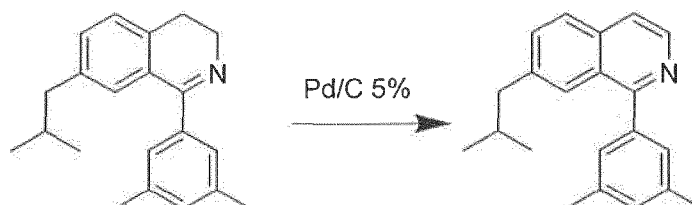
[0097]



[0098] N-(4-p-isobutylphenylethyl)benzamide (15.0g), phosphorous pentoxide (50g) phosphorous oxychloride 50mL and xylenes (160mL) was refluxed for 3 h in a 1L round bottom flask. After the reaction mixture was allowed to cool to room temperature, the solvent was decanted and ice was slowly added to the solid in the bottom of the flask. The water-residue mixture was made weakly alkaline with 50% NaOH and the product was extracted with toluene. The organic layer was washed with water and dried over anhydrous MgSO_4 . The solvent was evaporated to give 12.4g of crude product (88% yield) which was used without further purification.

Step 4

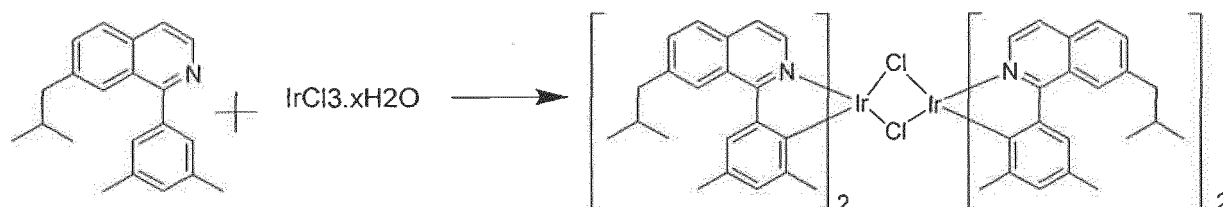
[0099]



[0100] 1-(3,5-dimethylphenyl)-7-isobutyl-3,4-dihydroisoquinoline (12.4g, 42.5mmol) and 2g of 5% Pd/C (~10% by weight) were added to a 500mL round bottom flask with 100 mL of xylenes. The solution was refluxed for 24 hrs and the formation of the product was monitored by TLC. The solvent was removed under vacuum and the product was purified by silica gel column chromatography with 5% ethyl acetate in hexanes as the eluent. The product was then vacuum distilled to give (6.0g, 21mmol) of pure product.

Step 5

[0101]

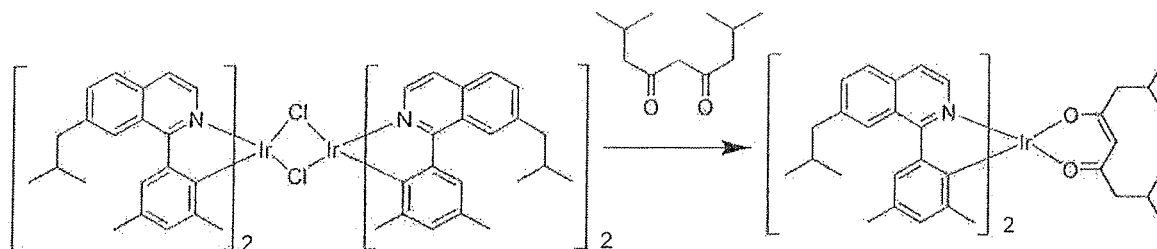


[0102] The ligand from step 4 (4.7g, 14 mmol), 2-ethoxyethanol (25 mL) and water (5 mL) were charged in a 1L three-neck round bottom flask. Nitrogen gas was bubbled through the reaction mixture for 45 min. $\text{IrCl}_3 \cdot \text{H}_2\text{O}$ (1.2 g 3.6 mmol) was then added and the reaction mixture was heated to reflux under nitrogen for 17 hours. The reaction mixture was cooled to ambient and filtered. The dark red residue was washed with methanol (2 x 25 mL) followed by hexanes (2 x

25 mL). 2.5g of the dichlorobridged Iridium dimer was obtained after drying in vacuum oven.

Step 6

[0103]

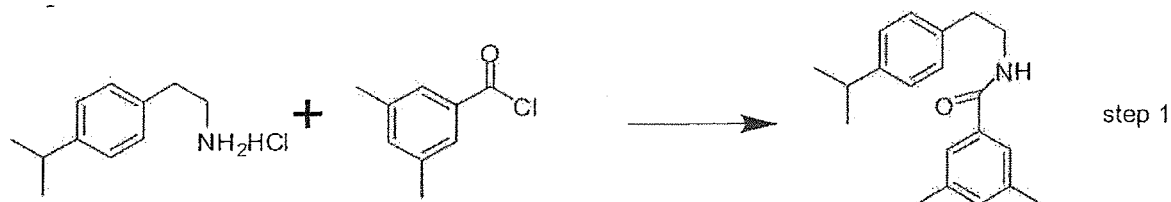


[0104] Dichlorobridged Iridium dimer from step 5 (2.5g, 1.5 mmol), 10 mol eq 2,8-dimethylnonane-4,6-dione (2.8g.), 20 mol eq of Na_2CO_3 (4.3g) and 25 mL of 2-ethoxyethanol were placed in a 250 mL round bottom flask. The reaction mixture was stirred at ambient for 24 hours. 2g of celite and 200mL of dichloromethane was added to the reaction mixture to dissolve the product. The mixture was then filtered through a bed of celite. The filtrate was then passed through a silica/alumina plug and washed with dichloromethane. The clarified solution was then filtered through GF/F filter paper the filtrate was heated to remove most of the dichloromethane. 20 mL of isopropanol was then added and the slurry was cooled to ambient and the product was filtered and washed with isopropanol and dried to give 2.5g of crude product (86% yield). This product was then recrystallised twice using dichloromethane and isopropanol and then sublimed.

Synthesis of Compound 7 (not according to the invention)

Step 1

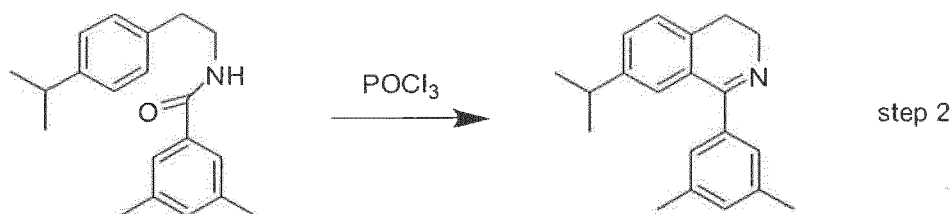
[0105]



[0106] 4-isopropylphenylethylamine hydrochloride (5.0g, 25mmol), pyridine (5.9g, 75mmol) and dichloromethane (25mL) were added to a 3-neck round bottom flask. The solution was cooled in an ice bath and 3,5-dimethylbenzoyl chloride (4.2g, 25mmol) was added slowly. The solution was then allowed to warm to room temperature and stirred for 12 hours. Dichloromethane was added and the organic phase was washed with water, followed by 5% HCl solution, then 5% NaOH solution and then dried over anhydrous MgSO_4 . The solvent was evaporated under vacuum resulting in 7.5g of crude product (82% yield) which was used without further purification.

Step 2

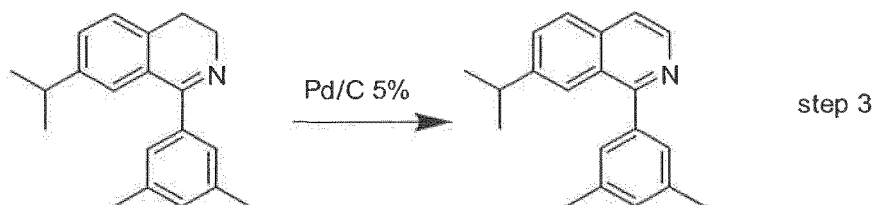
[0107]



[0108] N-(4-p-isopropylphenylethyl)benzamide (7.5g) in 80mL xylenes was refluxed for 3 hrs together with 25g phosphorous pentoxide and 25mL phosphorous oxychloride. After cooling, the solvent was decanted and ice was slowly added to the solid in the bottom of the flask. The water-residue mixture was made weakly alkaline with 50% NaOH and the product was extracted with toluene. The organic layer was washed with water and dried over anhydrous MgSO_4 . The solvent was removed under vacuum to give 6.2g of crude product which was used without further purification.

Step 3

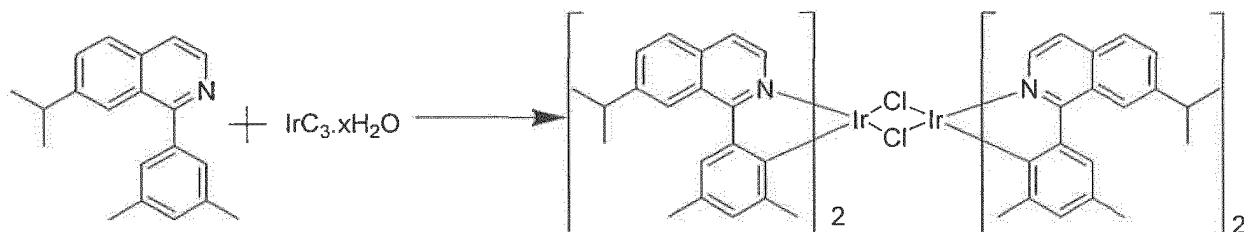
[0109]



[0110] 6.2g of 7-isopropyl-1-phenyl-3,4-dihydroisoquinoline and 1g of 5% Pd/C (~10% by weight) were added to a round bottom flask with 100 mL of xylenes. The solution was refluxed for 24 hrs and the formation of the product was monitored by TLC. The xylenes solvent was removed and the product was purified by column chromatography with ethyl acetate/hexanes. The pure fractions were collected and the solvent was removed. The product was then distilled in a kugelrohr apparatus at 185°C affording 1.8g (0.0073mol) of pure product. The overall yield of ligand formation was ~15%.

Step 4

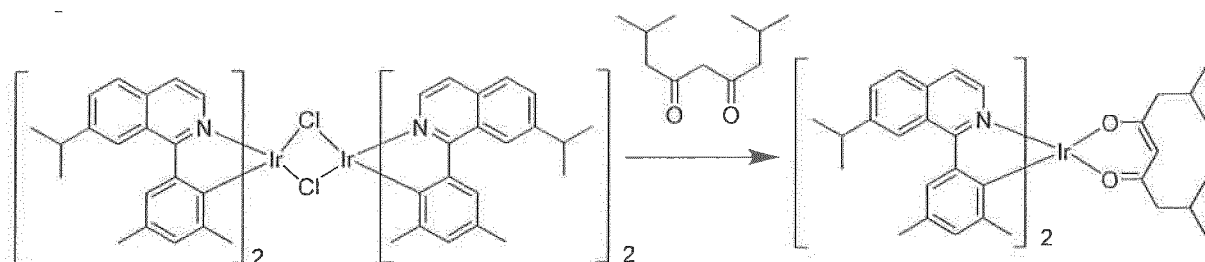
[0111]



[0112] The ligand from step 3 (1.8g, 7.3 mmol), 2-ethoxyethanol (25 mL) and water (5 mL) were charged in a 1L three-neck round bottom flask. Nitrogen gas was bubbled through the reaction mixture 45 min. $\text{IrCl}_3 \cdot \text{H}_2\text{O}$ (1.2 g 3.6 mmol) was then added and the reaction mixture was heated to reflux under nitrogen for 17 hours. The reaction mixture was cooled to ambient and filtered. The dark red residue was washed with methanol (2 x 25 mL) followed by hexanes (2 x 25 mL). 1.3 gram of the dichlorobridged Iridium dimer was obtained after drying in vacuum oven

Step 5

[0113]



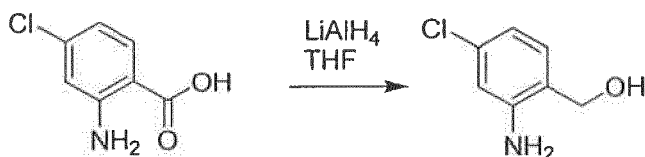
[0114] Dichlorobridged Iridium dimer from step 2 (1.3g, 0.9 mmol), 10 mol eq 2,8-dimethylnonane-4,6-dione (1.6g),

20 mol eq of Na_2CO_3 (2.5g) and 25 mL of 2-ethoxyethanol were placed in a 250 mL round bottom flask. The reaction mixture was stirred at ambient for 24 hours. 2g of celite and 200mL of dichloromethane was added to the reaction mixture to dissolve the product. The mixture was then filtered through a bed of celite. The filtrate was then passed through a silica/alumina plug and washed with dichloromethane. The clarified solution was then filtered through GF/F filter paper the filtrate was heated to remove most of the dichloromethane. 20 mL of isopropanol was then added and the slurry was cooled to ambient and the product was filtered and washed with isopropanol and dried to give 1.4g of crude product(92%yield). This product was then recrystallised twice using dichloromethane and isopropanol and then sublimed.

Synthesis of Compound 8

Step 1

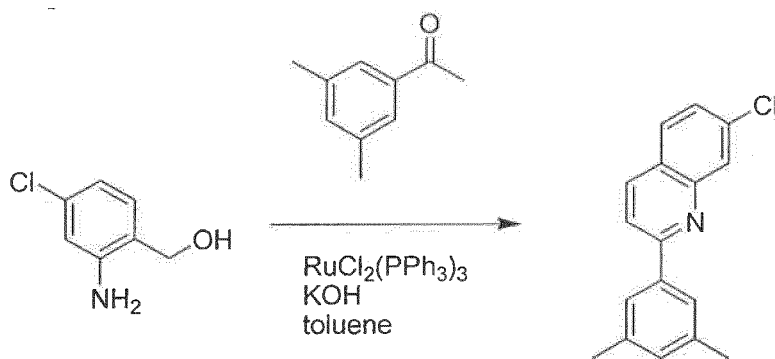
[0115]



[0116] 2-amino-4-chlorobenzoic acid (42.8g, 0.25mol) was dissolved in 200mL of anhydrous THF and cooled in an ice-water bath. To the solution was added lithium aluminum hydride chips(11.76g, 0.31mol). The resulting mixture was stirred at room temperature for 8 hours. 12g of water was added, and then 12g 15% NaOH. 36g of water was then added. The slurry was stirred at room temperature for 30min. The slurry was filtered. The solid was washed with ethyl acetate. The liquid was combined and the solvent was evaporated. The crude material was used for next step without purification.

Step 2

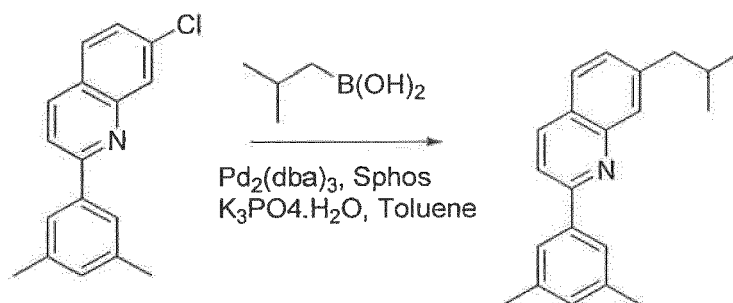
[0117]



[0118] 2-amino-4-chlorophenylmethanol (6.6g, 0.04mol) 1-(3,5-dimethylphenyl)ethanone(10.0g 0.068mol), $\text{RuCl}_2(\text{PPh}_3)_3$ 0.1g) and 2.4g of KOH was refluxed in 100ml of toluene for 10 hours. Water was collected from the reaction using a Dean-Stark trap. After the reaction was cooled to room temperature, the mixture was filtered through a silica gel plug. The product was further purified with column chromatography using 2% ethyl acetate in hexanes as eluent. 9g product was obtained after column. The product was further recrystallized from isopropanol. 5g of desired product was obtained.

Step 3

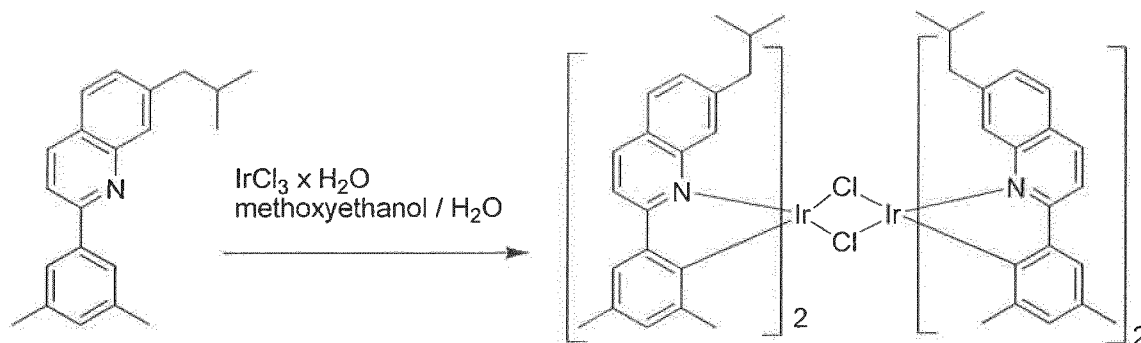
[0119]



[0120] 7-chloro-2-(3,5-dimethylphenyl)quinoline (3.75g, 0.014mol) isobutylboronic acid (2.8g, 0.028mol), $\text{Pd}_2(\text{dba})_3$ (1mol%), 2-dicyclohexylphosphino-2',6'-dimethoxybiphenyl (4 mol%), potassium phosphate monohydrate (16.0g) 100mL of toluene was charged in a 250mL round bottom flask. Nitrogen was bubbled through the reaction mixture for 20 minutes and heated to reflux for 18h overnight. The reaction mixture was allowed to cool to ambient temperature and the crude product was purified by column chromatography using 2% ethyl acetate in hexanes as solvent. 3.6g of desired product was obtained.

Step 4

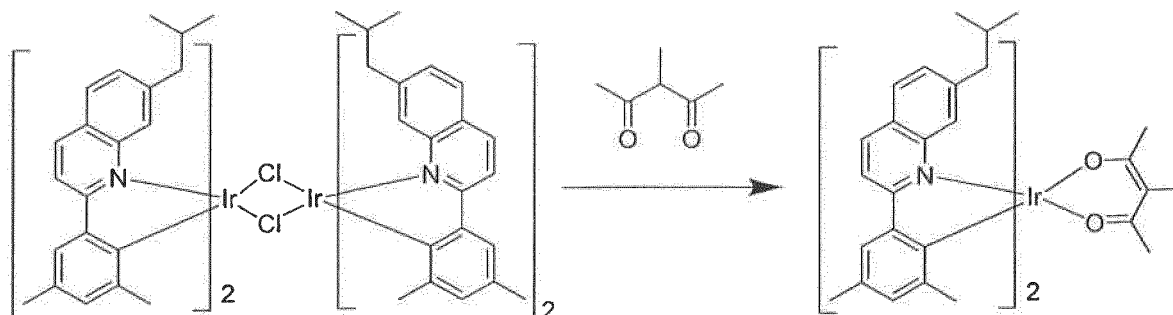
[0121]



[0122] The ligand from step 3 (4.6g, 16 mmol), 2-ethoxyethanol (25 mL) and water (5 mL) were charged in a 1L three-neck round bottom flask. Nitrogen gas was bubbled through the reaction mixture 45 min. $\text{IrCl}_3 \cdot \text{H}_2\text{O}$ (1.2 g 3.6 mmol) was then added and the reaction mixture was heated to reflux under nitrogen for 17 hours. The reaction mixture was cooled to ambient and filtered. The dark red residue was washed with methanol (2 x 25 mL) followed by hexanes (2 x 25 mL). 1.3 gram of the dichlorobridged Iridium dimer was obtained after drying in vacuum oven

Step 5

[0123]

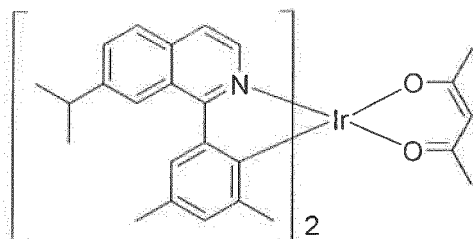


[0124] Dichlorobridged Iridium dimer from step 2 (1.0g, 0.6 mmol), 10 mol eq 3-methyl-2,4-pentanedione (0.8g.), 20

mol eq of Na_2CO_3 (3g) and 25 mL of 2-ethoxyethanol were placed in a 250 mL round bottom flask. The reaction mixture was stirred at ambient for 24 hours. 2g of celite and 200mL of dichloromethane was added to the reaction mixture to dissolve the product. The mixture was then filtered through a bed of celite. The filtrate was then passed through a silica/alumina plug and washed with dichloromethane. The clarified solution was then filtered through GF/F filter paper the filtrate was heated to remove most of the dichloromethane. 20 mL of isopropanol was then added and the slurry was cooled to ambient and the product was filtered and washed with isopropanol and dried to give 1.1g of crude product (97% yield). This product was then recrystallised twice using dichloromethane and isopropanol and then sublimed.

Synthesis of Compound 9

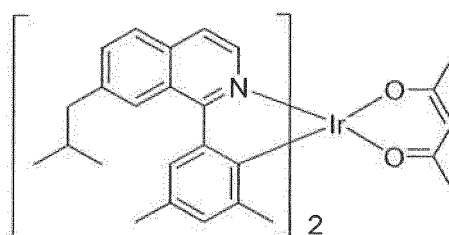
[0125]



[0126] Compound 9 can be synthesized using the same procedure as outlined for invention compound 7. In this case the dichlorobridged iridium dimer that is formed should be cleaved with 2,4-pentane dione to afford the product.

Synthesis of Compound 10

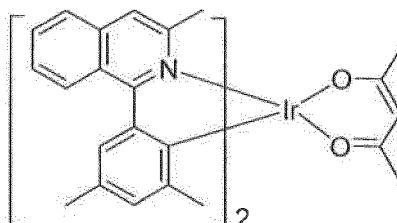
[0127]



[0128] Compound 10 can be synthesized using the same procedure as outlined for invention compound 6. In this case the dichlorobridged iridium dimer that is formed should be cleaved with 2,4-pentane dione to afford the product.

Synthesis of Compound 11

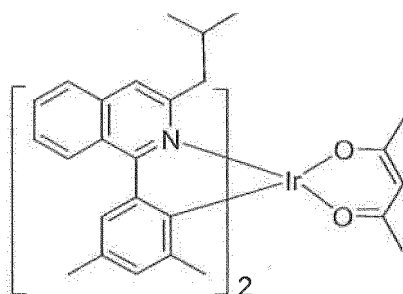
[0129]



[0130] Synthesis of compound 11 can be easily synthesized using common synthetic approaches already disclosed.

Synthesis of Compound 12

[0131]



[0132] Synthesis of compound 11 can be easily synthesized using common synthetic approaches already disclosed.

Device Examples

[0133] All device examples were fabricated by high vacuum ($<10^{-7}$ Torr) thermal evaporation. The anode electrode is 1200Å of indium tin oxide (ITO). The cathode consisted of 10Å of LiF followed by 1000Å of Al. All devices were encapsulated with a glass lid sealed with an epoxy resin in a nitrogen glove box (<1 ppm of H_2O and O_2) immediately after fabrication, and a moisture getter was incorporated inside the package.

[0134] The organic stack of the Device Examples 1-8 in Table 2, consisted of sequentially, from the ITO surface, 100Å of $Ir(3-Mepq)_3$ as the hole injection layer (HIL), 400Å of 4,4'-bis[N-(1-naphthyl)-N-phenylamino]biphenyl (a-NPD) as the hole transporting later (HTL), 300Å of BAiq doped with 8-12% of the inventive compound as the emissive layer (EML), and 550Å of Alq_3 (tris-8-hydroxyquinoline aluminum) as the ETL.

[0135] Comparative Examples 1 and 2 were fabricated similarly to the Device Examples, except that $Ir(3-Mepq)_2(acac)$ or $Ir(piq)_2(acac)$ was used as the emissive dopant.

[0136] The device structures and data are summarized in Table 2. As used herein, the following compounds have the following structures:

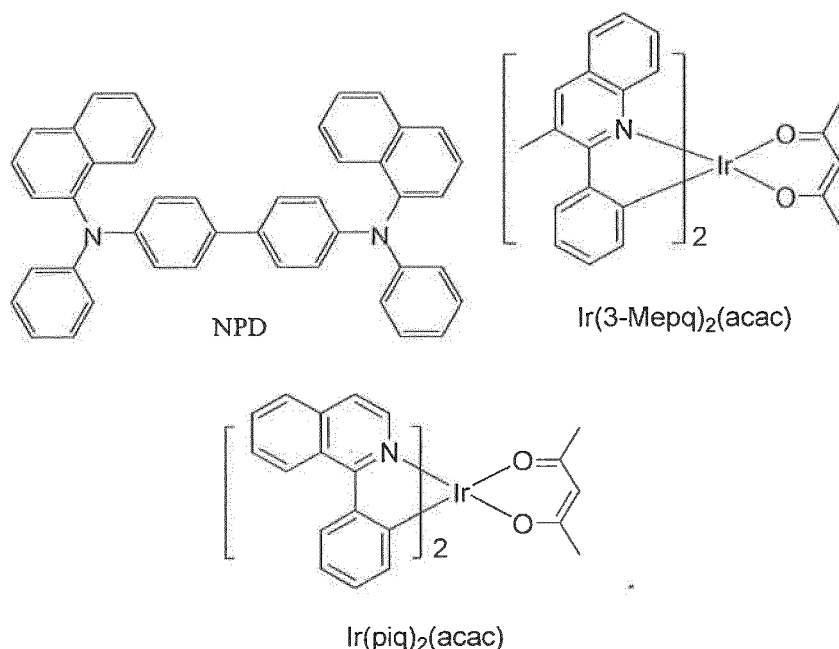


TABLE 2.

Device Example		Emitter (doping %)	T _{sub} at 0.24 Å/s	At 10 mA/cm ²						At 40 mA/cm ²		
				Em _{max} (nm)	FWHM (nm)	CIE	V (V)	LE (cd/A)	EQE (%)	LE:EQE	L ₀ (cd/m ²)	T _{0.8} at 70°C
comparative 1	comparative 2	Ir(3-Mepq) ₂ (acac) (9)	206	622	94	0.65 0.35	8.1	14.3	14.1	1.01	4,817	60
		Ir(piq) ₂ (acac) (9)	229	632	84	0.68 0.32	8.8	10.6	15.2	0.70	3,757	240
1	1(8) 2(8) * 3(8) * 4(12) * 5(12) * 6(12) * 7(12) *		177	634	68	0.682 0.316	8.6	12.6	16.2	0.78	4,200	70
2			198	636	66	0.688 0.308	8.5	8.5	13	0.66	2,895	80
3			180	618	59	0.662 0.335	8.4	21.1	18.2	1.16	7,042	55
4			186	621	61	0.666 0.331	9.2	18.7	17.4	1.07	6,443	31
5			190	633	67	0.681 0.316	8.5	11.9	15.3	0.78	4,029	25
6			205	631	55	0.691 0.307	8.5	14.0	20.4	0.68	4,694	69
7			210	632	55	0.690 0.307	8.1	13.3	19.4	0.68	4,493	190
* reference example, not according to the invention												

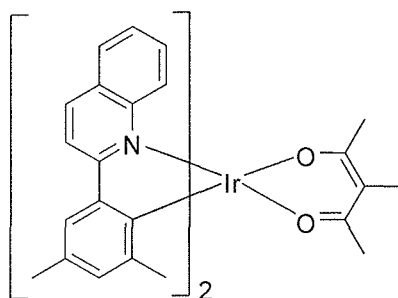
[0137] It can be seen from Table 2 that the Device Examples containing inventive compounds show similar or higher device efficiency and lifetime and also extremely narrow emission spectra versus the Comparative Examples containing Ir(3-Mepq)₂(acac) or Ir(piq)₂(acac). Several of the Device Examples show particularly good properties. For example, the LE and EQE of Example 3 are 21.1 cd/A and 18.2% respectively, at CIE of (0.662, 0.335). Also, the LE and EQE of Example 4 are 18.7 cd/A and 11.4% respectively, at CIE of (0.666, 0.331). These efficiencies are significantly higher than that for Comparative Example 1, which has LE and EQE of 14.3cd/A and 14.1% and has slightly bluer CIE (0.65, 0.35). Additionally, the LE and EQE of Example 6 are 14.0 cd/A and 20.4% respectively, at CIE of (0.691, 0.307) and the LE and EQE of Example 8 are 13.3cd/A and 19.4% respectively, at CIE of (0.690, 0.307), compared to Comparative Example 2 which has LE and EQE of 11.1cd/A and 15.4% at CIE (0.68, 0.32). Of note is that even though these examples are much deeper red than our Comparative Example 2, the efficiencies for these examples are still significantly higher. The Full Width Half Max (FWHM) of the EL for examples 3, 4, 6 and 7 are 59, 61, 55 and 55nm respectively. These are by far narrower than the EL measured for Comparative Examples 1 and 2 with FWHM 94 and 84nm, respectively. Device Examples 6 and 7 have the narrowest FWHM of any red iridium complex reported to date. Therefore, the inventive compounds may be advantageously used in devices to improve efficiency, stability and luminescence. The sublimation temperatures using the branched diketone ligand, for example, Compounds 3, 6 and 7, are also quite low which are well suited for long term thermal evaporation required in manufacturing.

[0138] Additionally, the 70°C lifetime comparison shows that Device Example 8 is more stable than both Comparative Example 1 and 2. Therefore, the inventive compounds may be advantageously used in devices to improve device lifetime.

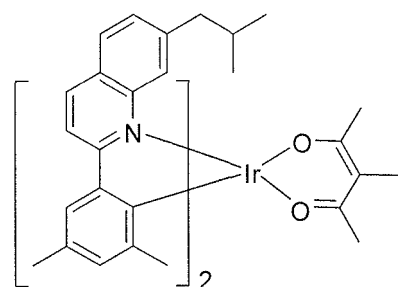
[0139] It is understood that the various embodiments described herein are by way of example only, and are not intended to limit the scope of the invention. The present invention as claimed may therefore include variations from the particular examples and preferred embodiments described herein, as will be apparent to one of skill in the art. It is understood that various theories as to why the invention works are not intended to be limiting.

Claims

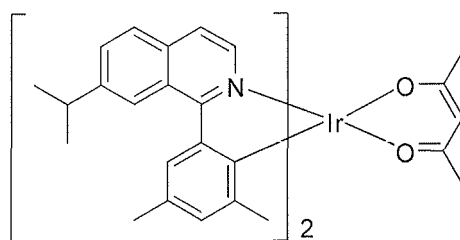
1. A compound selected from the group consisting of:



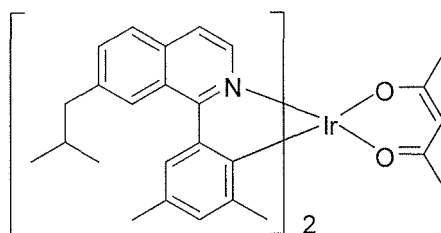
Compound 1



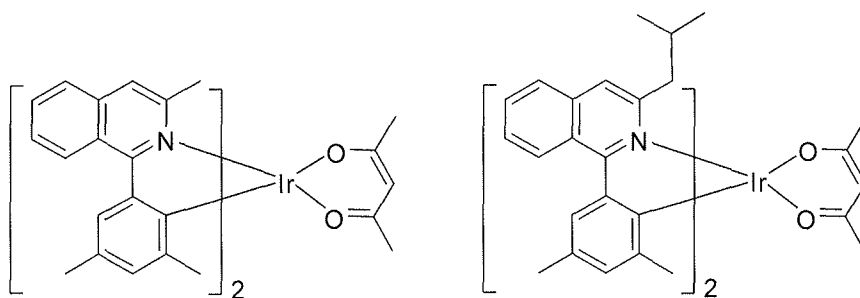
Compound 8



Compound 9



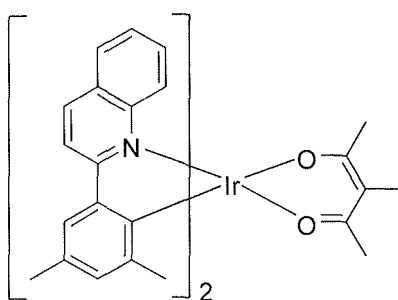
Compound 10



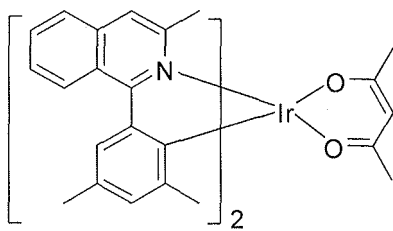
Compound 11

Compound 12

2. The compound of claim 1, wherein the compound is selected from the group consisting of:

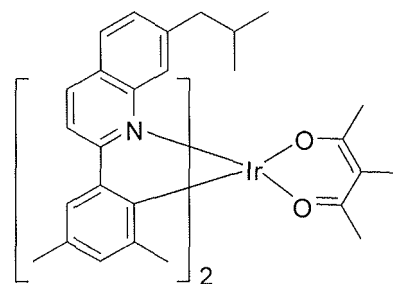


Compound 1

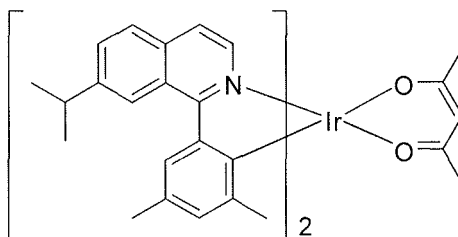


Compound 11

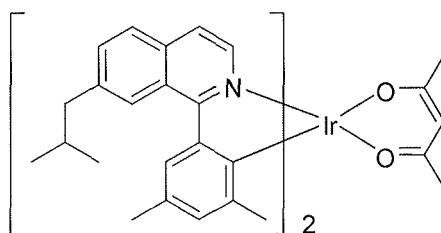
3. The compound of claim 1, wherein the compound is selected from the group consisting of:



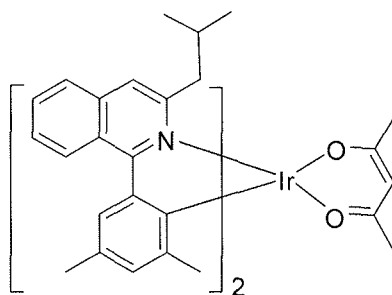
Compound 8



Compound 9

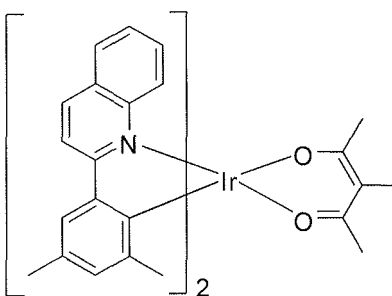


Compound 10

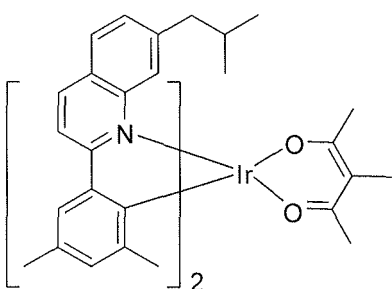


Compound 12

4. The compound of claim 1, wherein the compound is selected from the group consisting of:



Compound 1



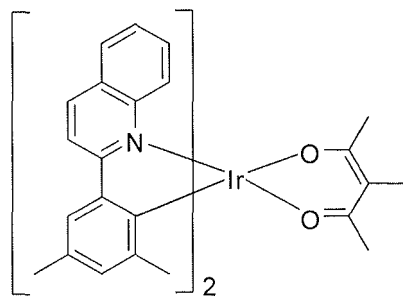
Compound 8

5. An organic light emitting device (100; 200) comprising:

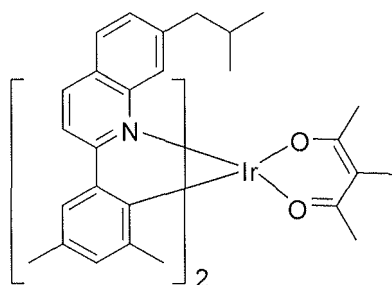
an anode (115; 260);

a cathode (160; 215); and

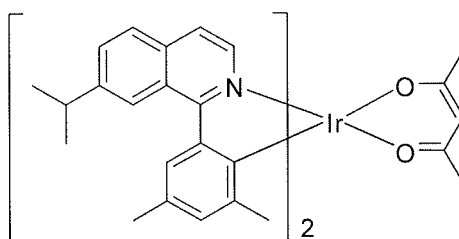
an organic layer, disposed between the anode (115; 260) and the cathode (160; 215), the organic layer further comprising a compound selected from the group consisting of:



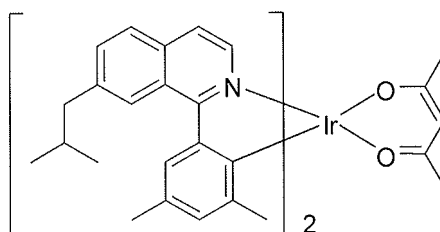
Compound 1



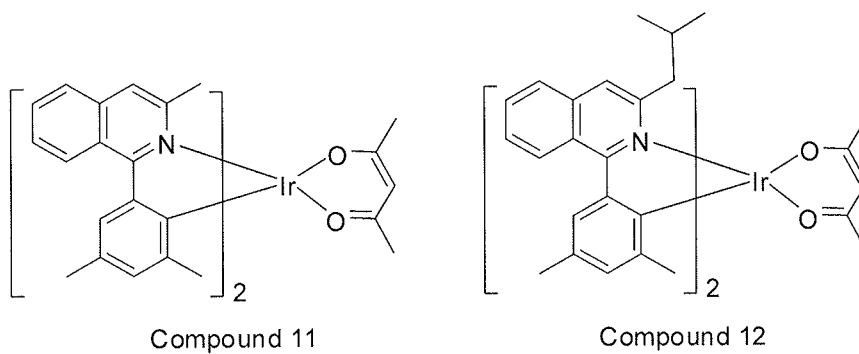
Compound 8



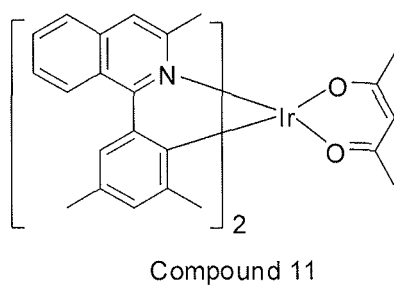
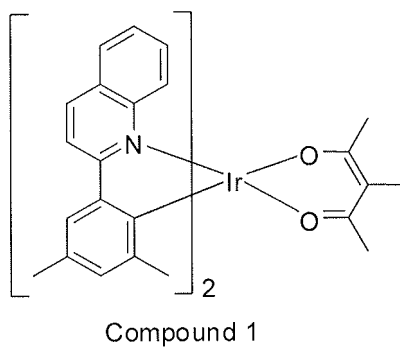
Compound 9



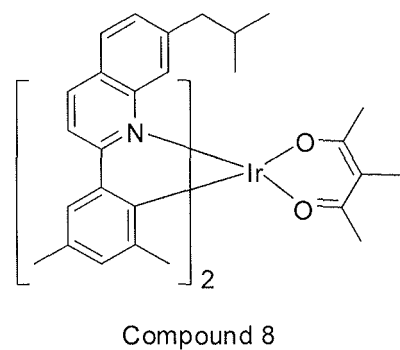
Compound 10

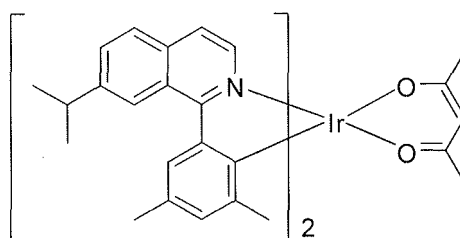


6. The device of claim 5, wherein the compound is selected from the group consisting of:

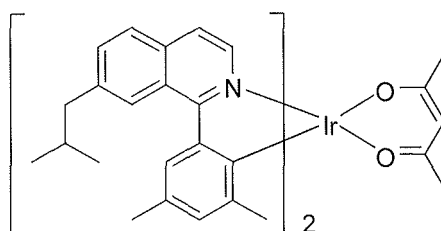


7. The device of claim 5, wherein the compound is selected from the group consisting of:

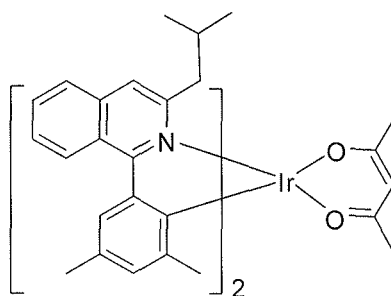




Compound 9

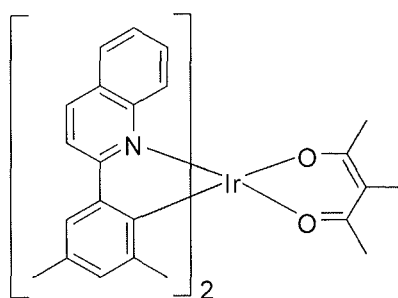


Compound 10

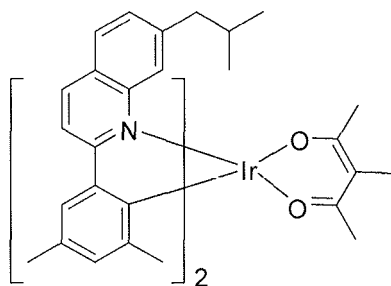


Compound 12

8. The device of claim 5, wherein the compound is selected from the group consisting of:



Compound 1



Compound 8

9. The device of claim 5, wherein the organic layer is an emissive layer (135; 220) comprising the compound and a host.

10. The device of claim 9, wherein the compound is the emissive material.

11. The device of claim 9, wherein the host is a metal coordination complex.

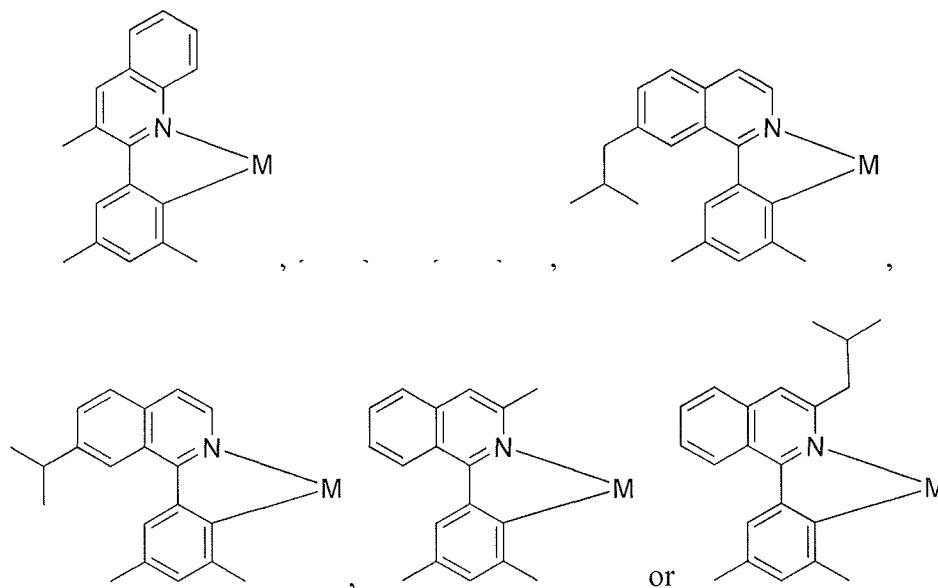
12. A consumer product comprising a device, the device further comprising:

an anode;

a cathode; and

an organic layer, disposed between the anode and the cathode, the organic layer further comprising a compound as defined in claim 1.

13. An organometallic compound containing a structure selected from the group consisting of:



wherein M is a metal with an atomic weight greater than 40.

14. The compound of claim 13, wherein M is Ir.

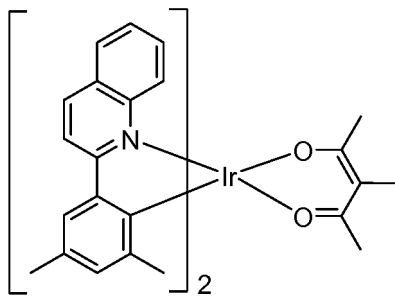
15. The compound of claim 14, wherein the compound is a phosphorescent material.

Patentansprüche

1. Eine Verbindung ausgewählt aus der Gruppe bestehend aus:

5

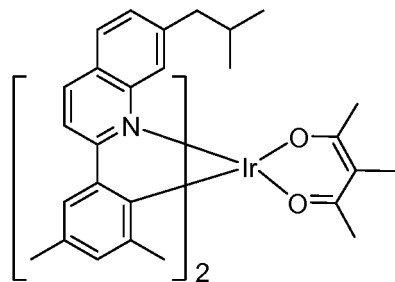
10



Verbindung 1

15

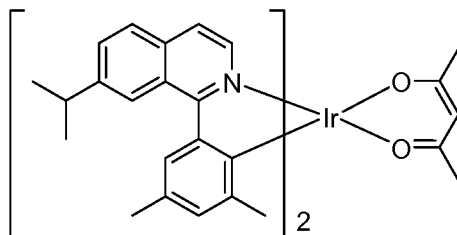
20



Verbindung 8

25

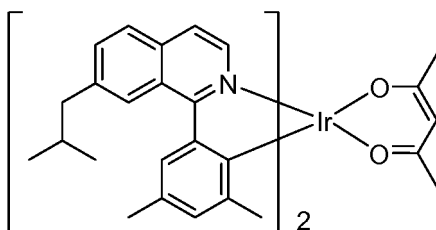
30



Verbindung 9

35

40

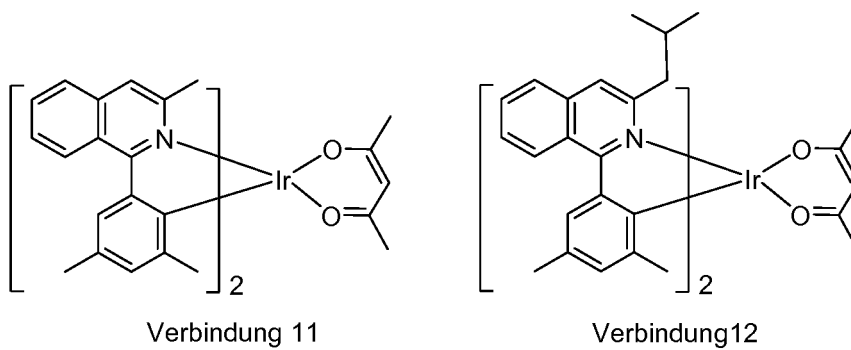


Verbindung 10

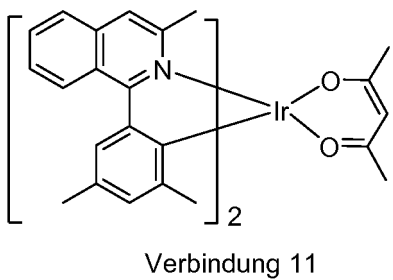
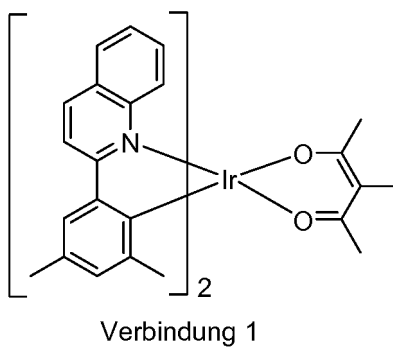
45

50

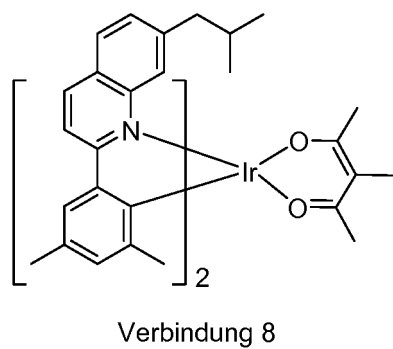
55

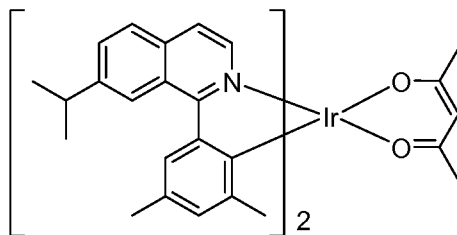


2. Die Verbindung nach Anspruch 1, wobei die Verbindung ausgewählt ist aus der Gruppe bestehend aus:

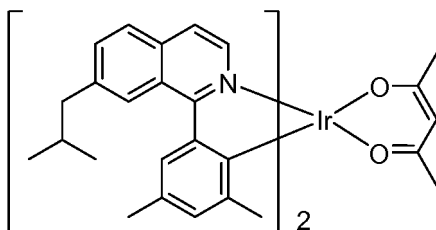


3. Die Verbindung nach Anspruch 1, wobei die Verbindung ausgewählt ist aus der Gruppe bestehend aus:

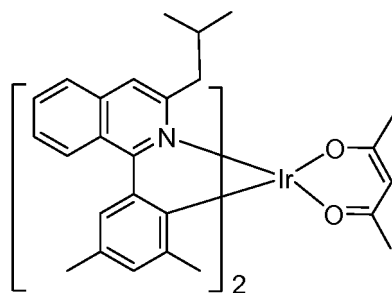




Verbindung 9

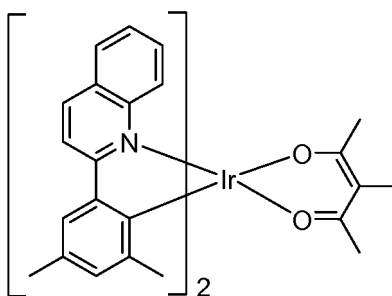


Verbindung 10

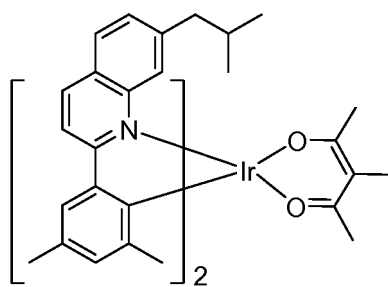


Verbindung 12

4. Die Verbindung nach Anspruch 1, wobei die Verbindung ausgewählt ist aus der Gruppe bestehend aus:



Verbindung 1



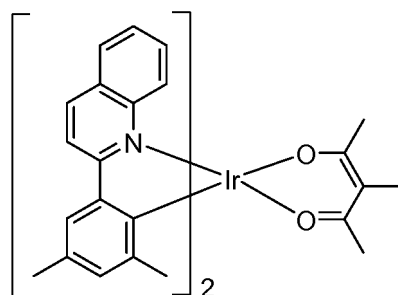
Verbindung 8

5. Eine organische Licht emittierende Vorrichtung (100; 200) umfassend:

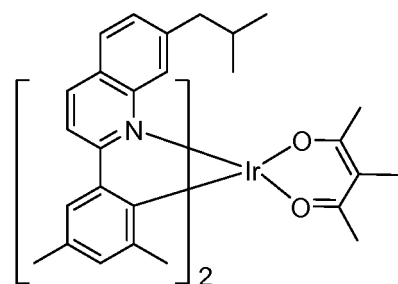
eine Anode (115; 260);

eine Kathode (160; 215); und

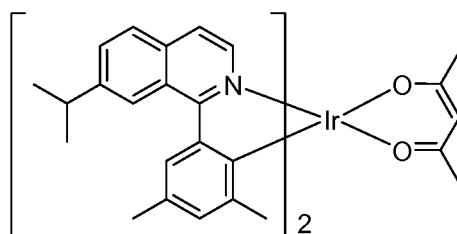
eine organische Schicht angeordnet zwischen der Anode (115; 260) und der Kathode (160; 215), die organische Schicht ferner umfassend eine Verbindung ausgewählt aus der Gruppe bestehend aus:



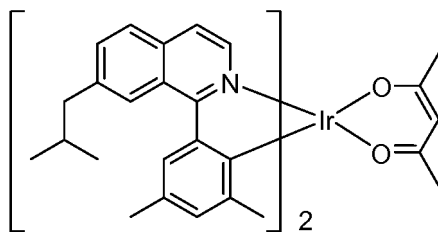
Verbindung 1



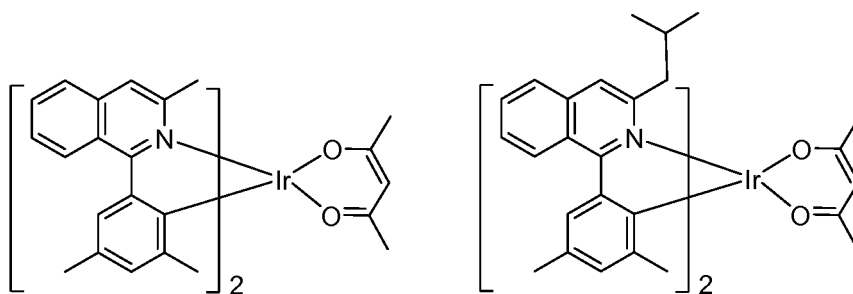
Verbindung 8



Verbindung 9



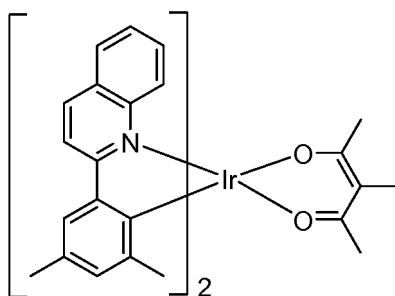
Verbindung 10



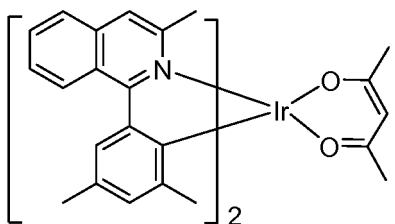
Verbindung 11

Verbindung 12

6. Die Vorrichtung nach Anspruch 5, wobei die Verbindung ausgewählt ist aus der Gruppe bestehend aus:

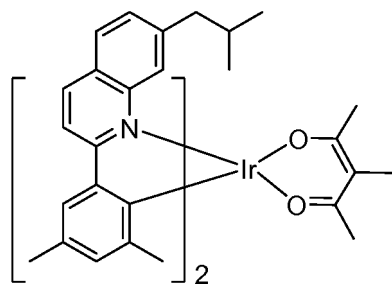


Verbindung 1

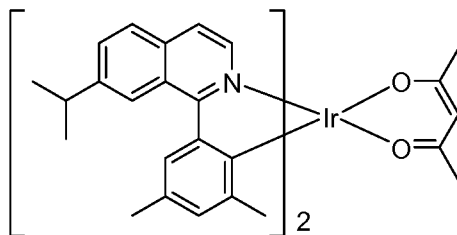


Verbindung 11

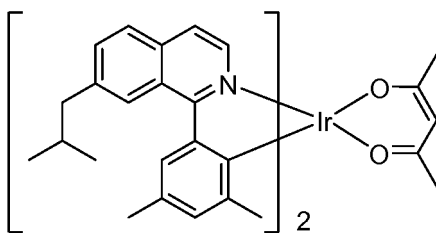
7. Die Vorrichtung nach Anspruch 5, wobei die Verbindung ausgewählt ist aus der Gruppe bestehend aus:



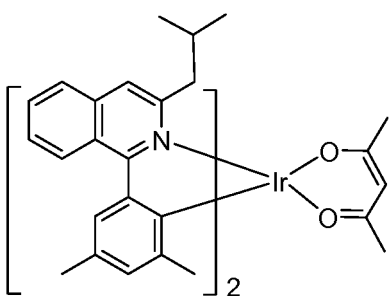
Verbindung 8



Verbindung 9

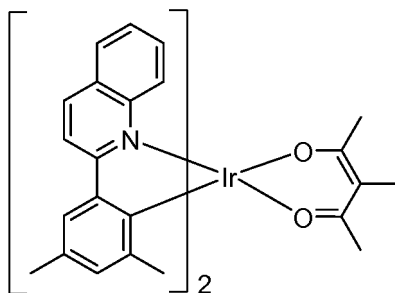


Verbindung 10

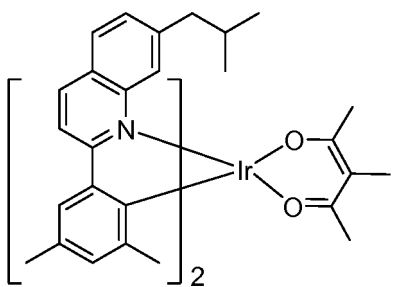


Verbindung 12

8. Die Vorrichtung nach Anspruch 5, wobei die Verbindung ausgewählt ist aus der Gruppe bestehend aus:

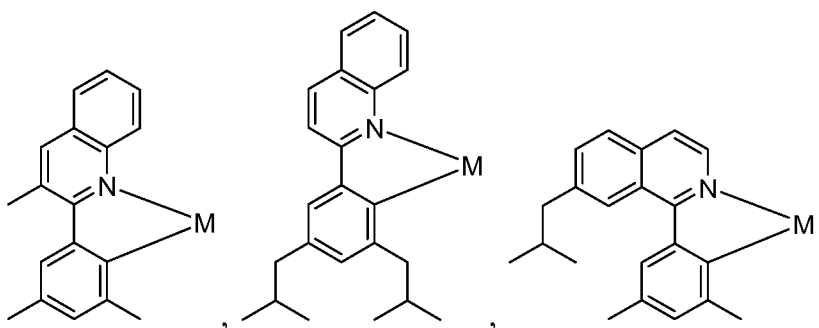


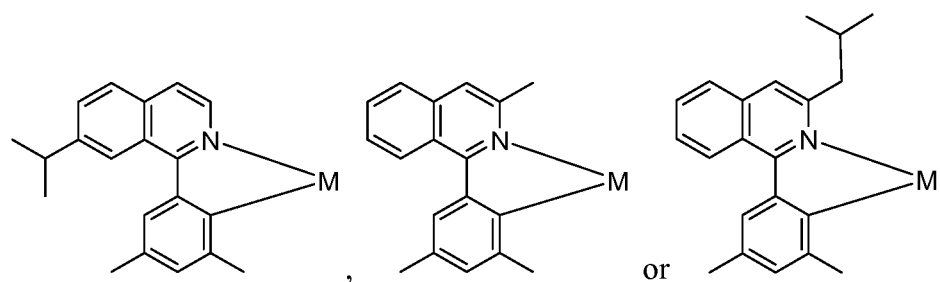
Verbindung 1



Verbindung 8

9. Die Vorrichtung nach Anspruch 5, wobei die organische Schicht eine emittierende Schicht (135; 220) umfassend die Verbindung und einen Wirt ist.
10. Die Vorrichtung nach Anspruch 9, wobei die Verbindung das emittierende Material ist.
11. Die Vorrichtung nach Anspruch 9, wobei der Wirt ein Metallkoordinationskomplex ist.
12. Ein Konsumentenprodukt umfassend eine Vorrichtung, die Vorrichtung ferner umfassend:
 - eine Anode;
 - eine Kathode; und
 - eine organische Schicht, angeordnet zwischen der Anode und der Kathode, wobei die organische Schicht ferner eine Verbindung wie in Anspruch 1 definiert umfasst.
13. Eine metallorganische Verbindung enthaltend eine Struktur ausgewählt aus der Gruppe bestehend aus:





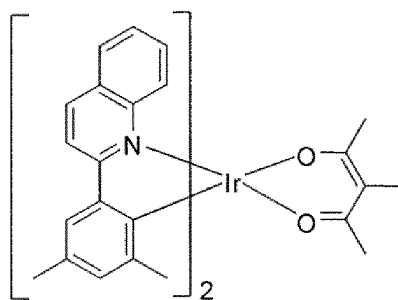
wobei M ein Metall mit einer Atomzahl von mehr als 40 ist.

14. Die Verbindung nach Anspruch 13, wobei M für Ir steht.

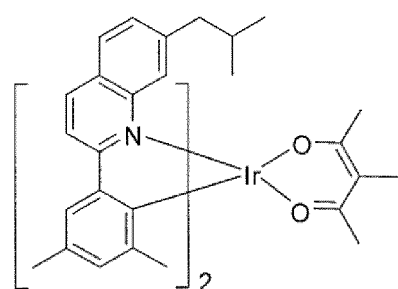
15. Die Verbindung nach Anspruch 14, wobei die Verbindung ein phosphoreszentes Material ist.

Revendications

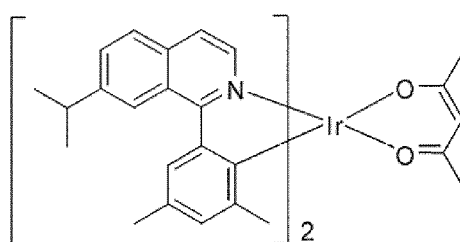
1. Composé choisi dans le groupe constitué par :



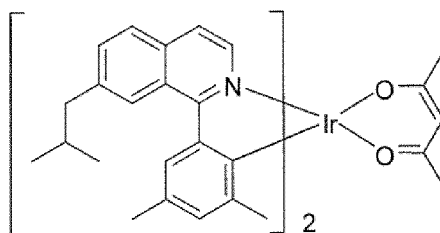
Composé 1



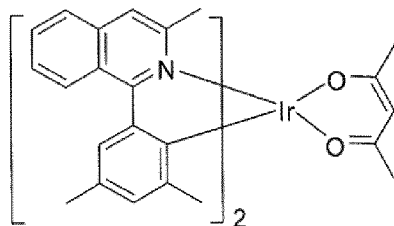
Composé 8



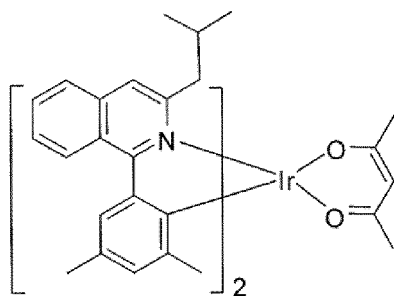
Composé 9



Composé 10

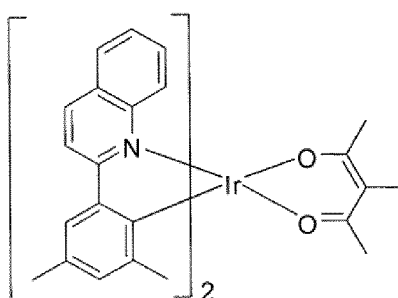


Composé 11

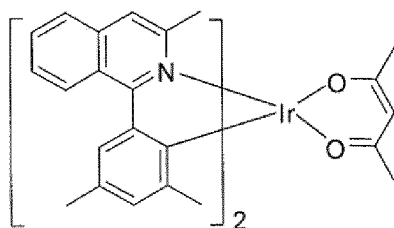


Composé 12.

2. Composé de la revendication 1, le composé étant choisi dans le groupe constitué par :

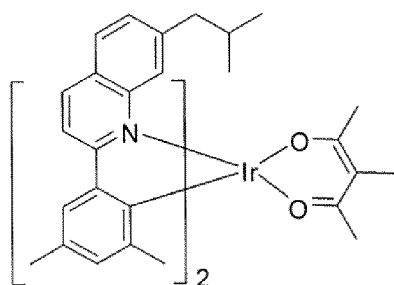


Composé 1

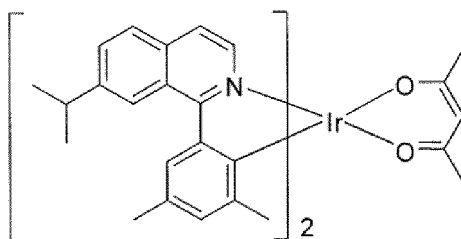


Composé 11.

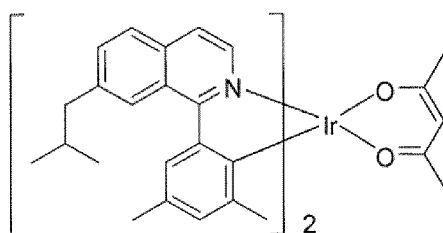
3. Composé de la revendication 1, le composé étant choisi dans le groupe constitué par :



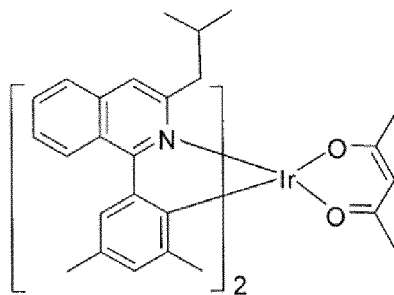
Composé 8



Composé 9

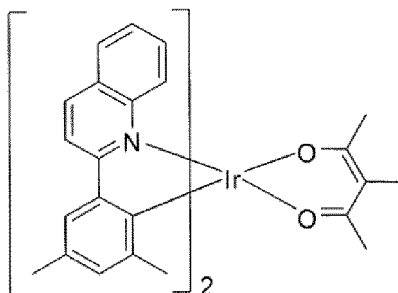


Composé 10

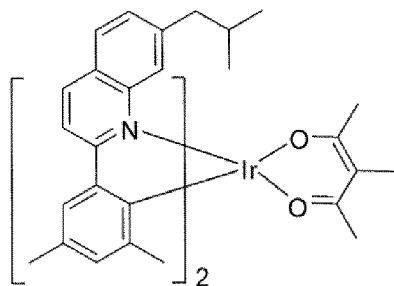


Composé 12.

4. Composé de la revendication 1, le composé étant choisi dans le groupe constitué par :



Composé 1



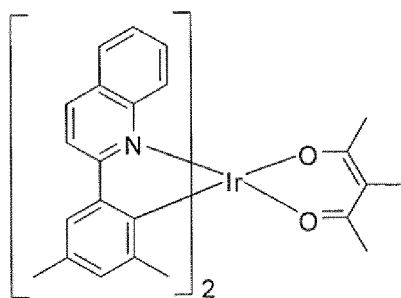
Composé 8.

5. Dispositif électroluminescent organique (100 ; 200) comprenant :

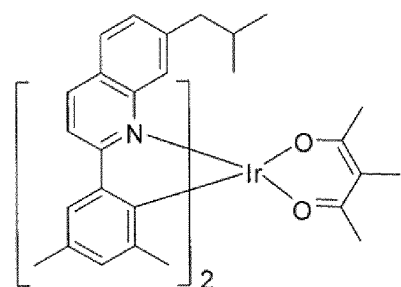
une anode (115 ; 260) ;

une cathode (160 ; 215) ; et

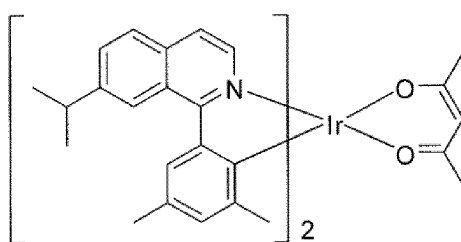
une couche organique, disposée entre l'anode (115 ; 260) et la cathode (160 ; 215), la couche organique comprenant en outre un composé choisi dans le groupe constitué par :



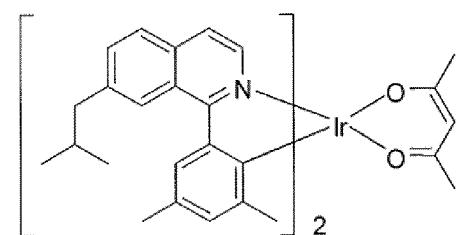
Composé 1



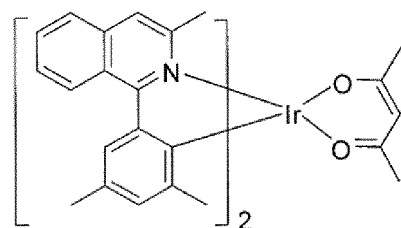
Composé 8



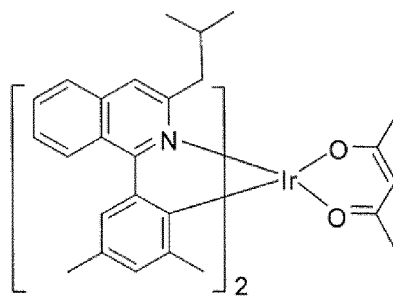
Composé 9



Composé 10

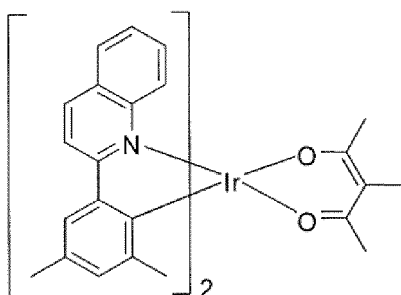


Composé 11

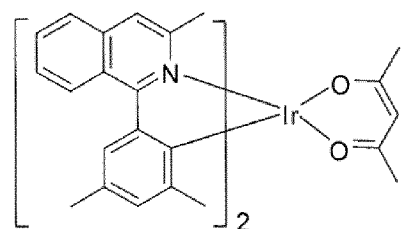


Composé 12.

6. Dispositif de la revendication 5, dans lequel le composé est choisi dans le groupe constitué par :

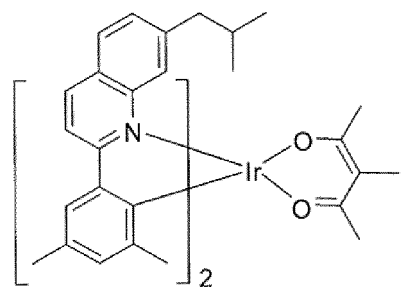


Composé 1

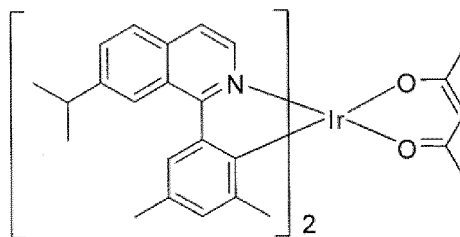


Composé 11.

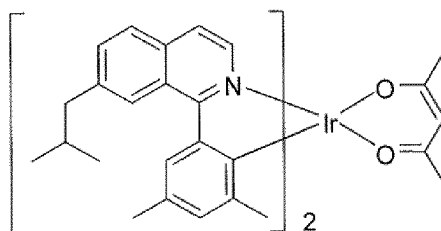
7. Dispositif de la revendication 5, dans lequel le composé est choisi dans le groupe constitué par :



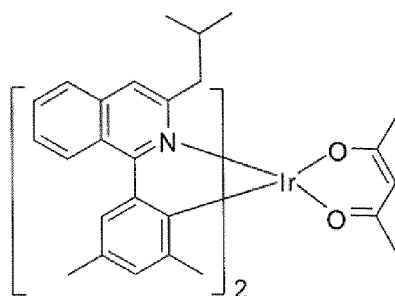
Composé 8



Composé 9

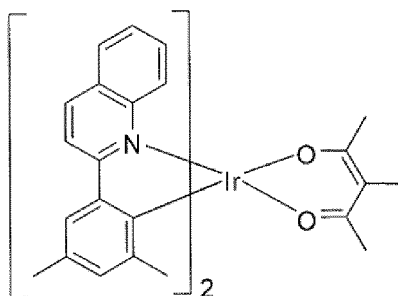


Composé 10

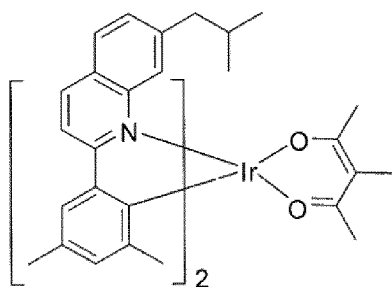


Composé 12.

8. Dispositif de la revendication 5, dans lequel le composé est choisi dans le groupe constitué par :



Composé 1



Composé 8.

9. Dispositif de la revendication 5, dans lequel la couche organique est une couche émissive (135 ; 220) comprenant le composé et un hôte.

10. Dispositif de la revendication 9, dans lequel le composé est la matière émissive.

11. Dispositif de la revendication 9, dans lequel l'hôte est un complexe de coordination de métal.

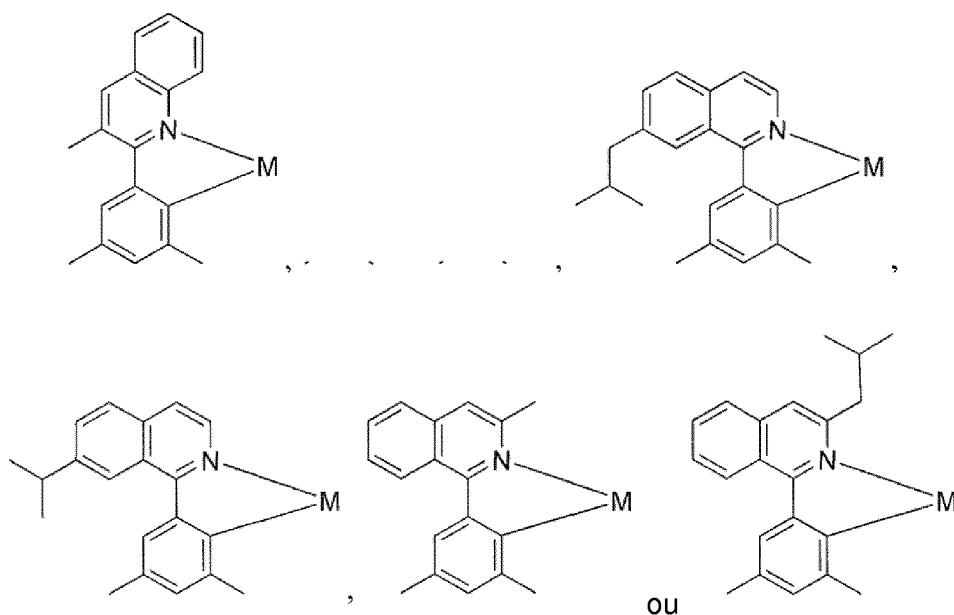
12. Produit de consommation comprenant un dispositif, le dispositif comprenant en outre :

une anode ;

une cathode ; et

une couche organique, disposée entre l'anode et la cathode, la couche organique comprenant en outre un composé tel que défini dans la revendication 1.

13. Composé organométallique contenant une structure choisie dans le groupe constitué par :



dans laquelle M est un métal ayant un poids atomique supérieur à 40.

14. Composé de la revendication 13, dans lequel M est Ir.

15. Composé de la revendication 14, le composé étant une matière phosphorescente.

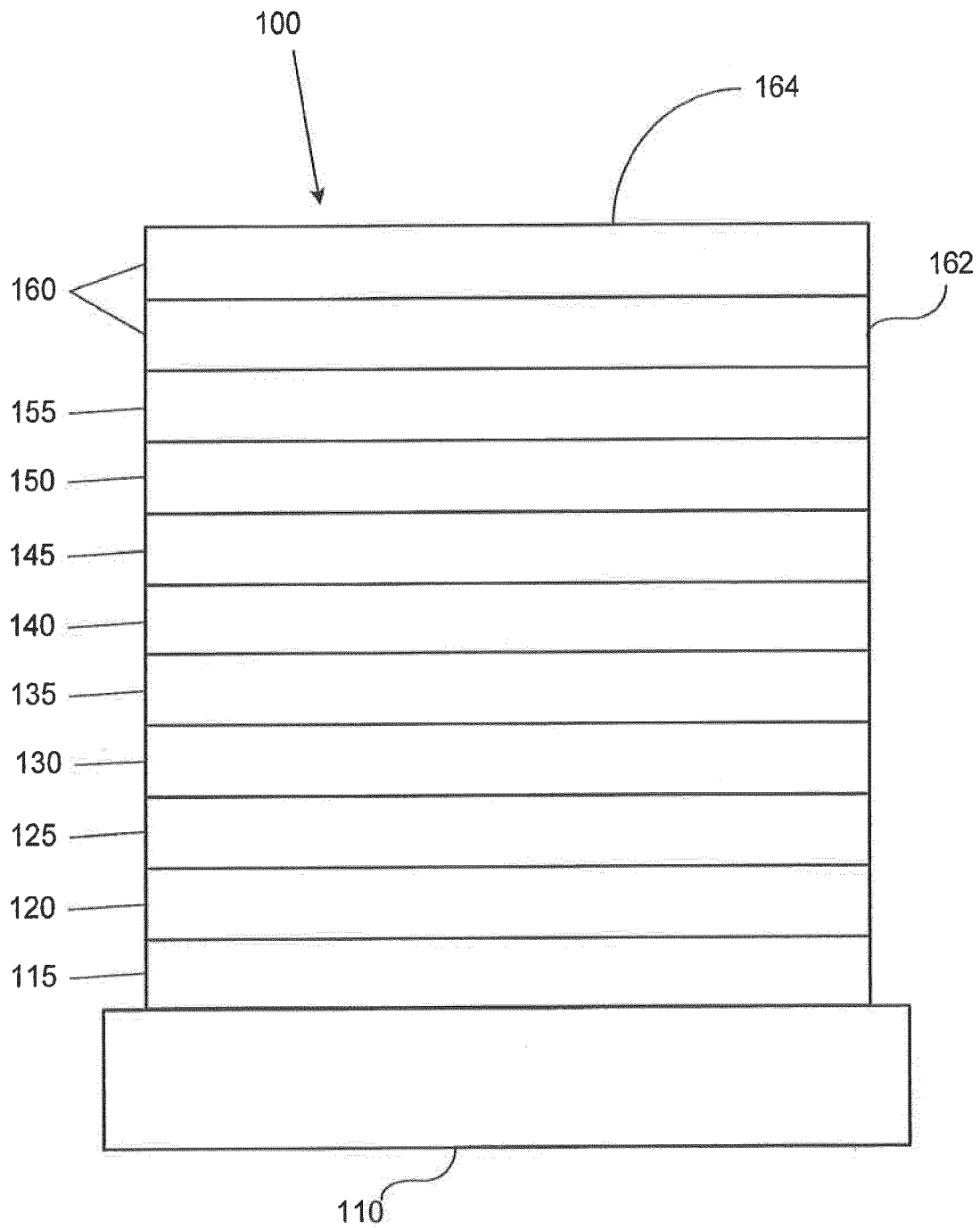


FIGURE 1

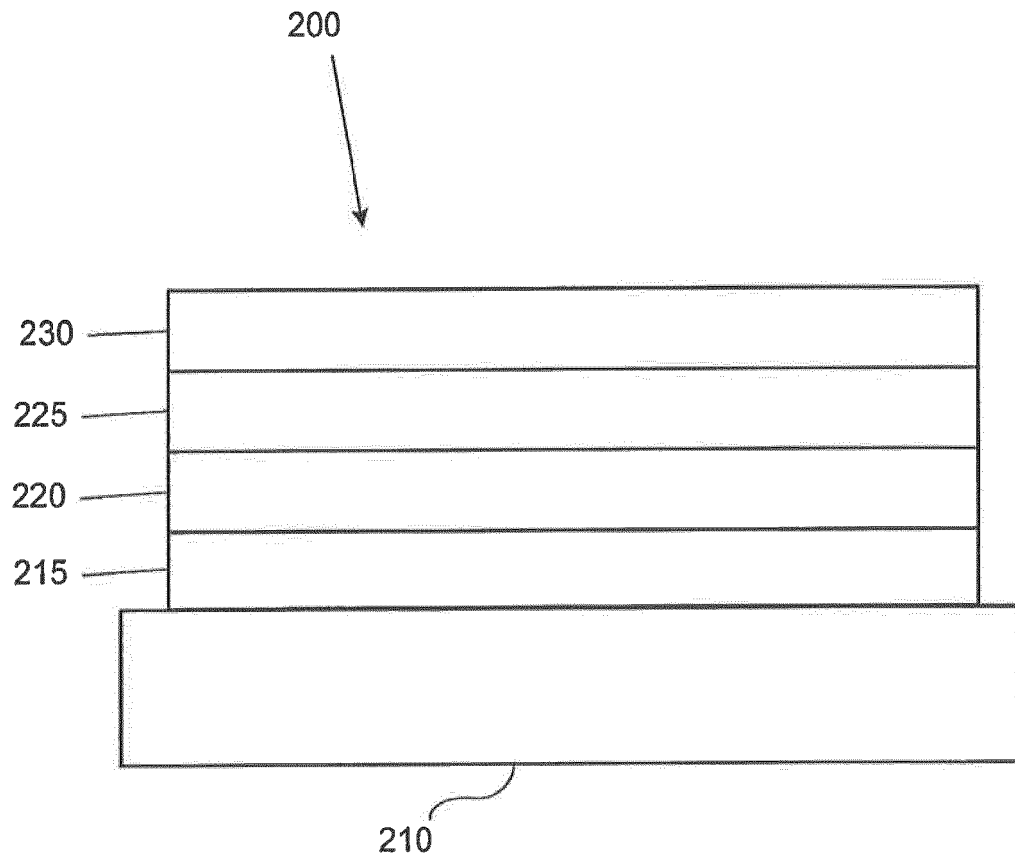


FIGURE 2

REFERENCES CITED IN THE DESCRIPTION

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摘要(译)

本发明涉及有机发光器件 (OLED) , 具体涉及用于这种器件的磷光有机材料。更具体地, 本发明涉及如下定义的铱化合物, 其具有结合到OLED中的窄光谱。

