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H01L 51/50(2006.01)

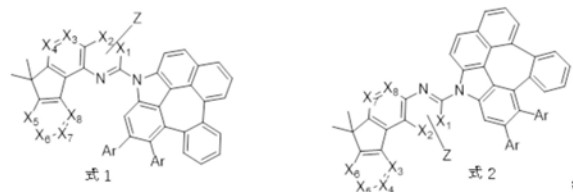
权利要求书9页 说明书25页 附图2页

(54)发明名称

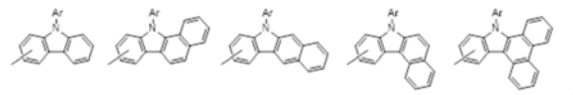
一种磷光化合物以及使用所述化合物的有机电致发光器件

(57)摘要

本发明涉及有机电致发光技术领域,具体地说,涉及一种磷光化合物以及使用所述化合物的有机电致发光器件。其结构式如式1和式2所示:

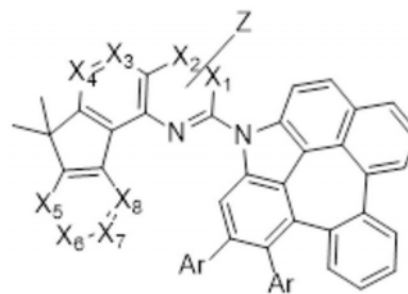


上述的结构式1和结构式2中,Z独立地选自以下结构:

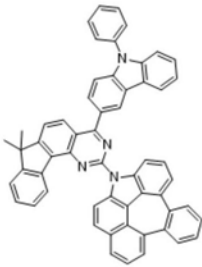


其中X1至X2独立的选自N原子或C原子;其中X1至X2中的至少一个为N;其中Ar独立地选自、C6-C30芳基、C2-C30杂芳基中。该发明的有机电致发光器件随着色纯度增加,可见度降低,甚至当色纯

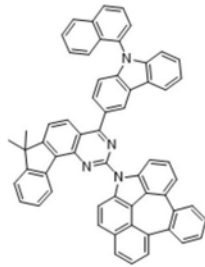
度高时,在低电压下也高效率地运行,并且电流效率增加,在高内量子效率下,发光效率高,同时采用磷光化合物作为有机电致发光器件的发光材料,制造过程简单,工艺效率高,生产成本低。



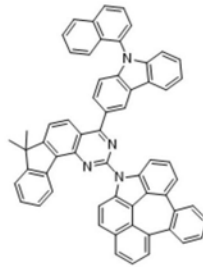




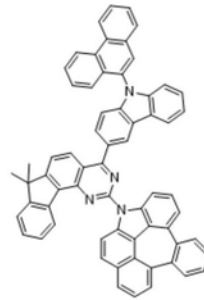
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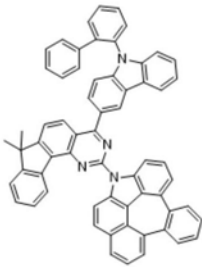
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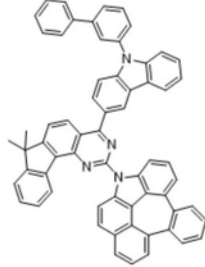
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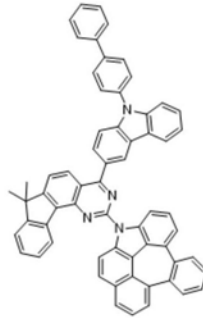
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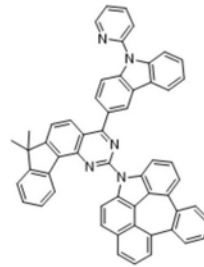
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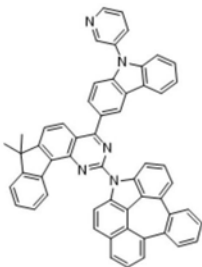
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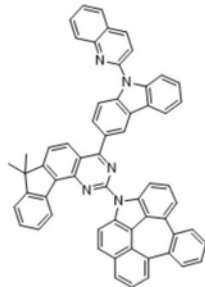
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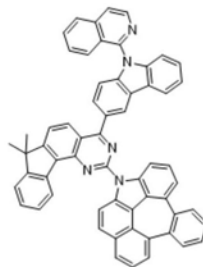
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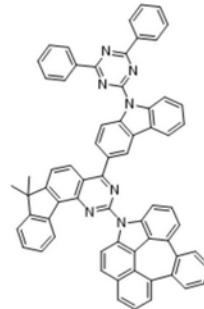
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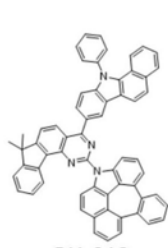
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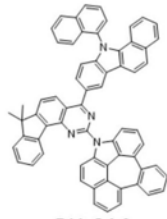
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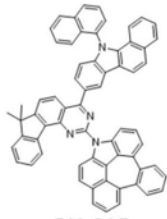
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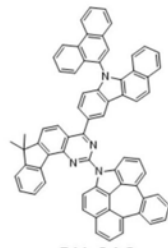
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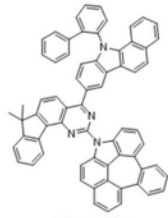
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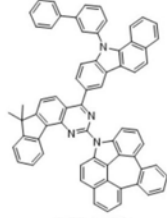
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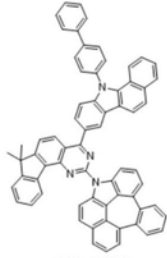
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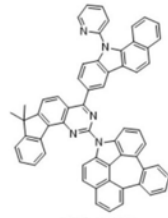
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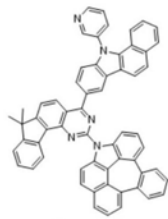
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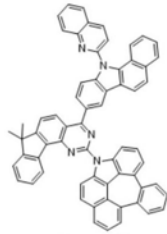
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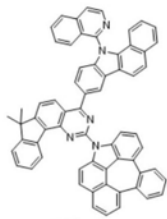
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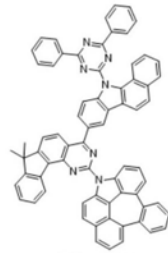
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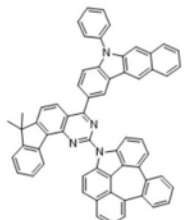
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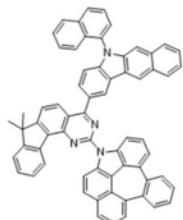
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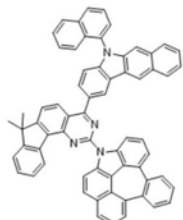
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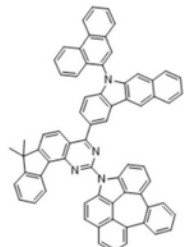
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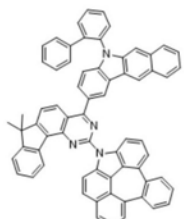
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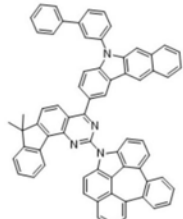
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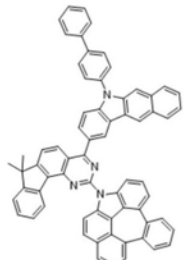
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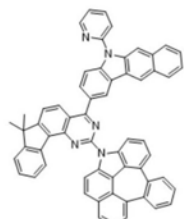
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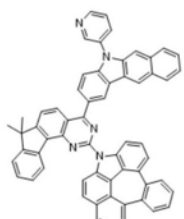
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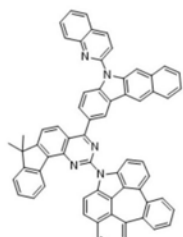
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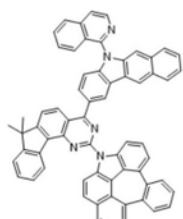
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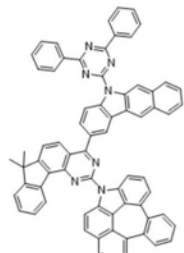
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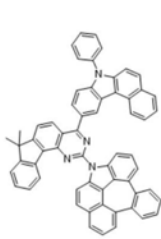
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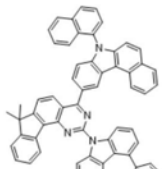
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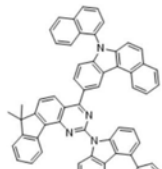
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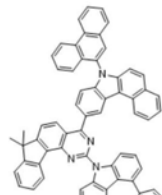
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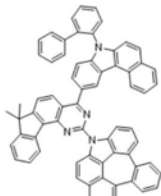
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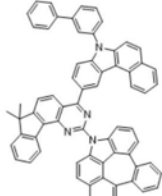
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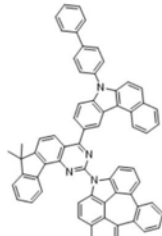
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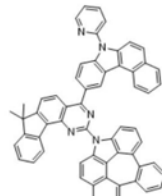
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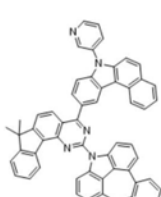
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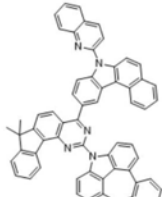
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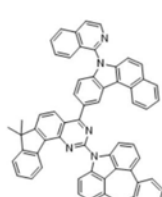
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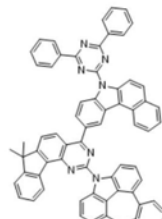
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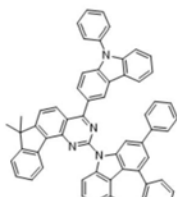
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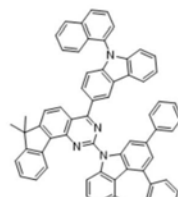
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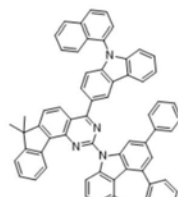
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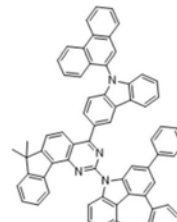
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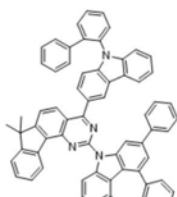
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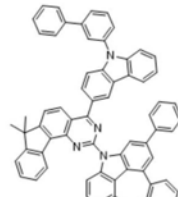
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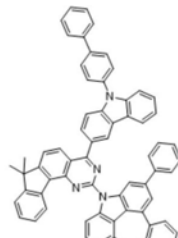
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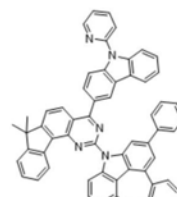
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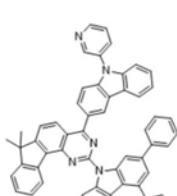
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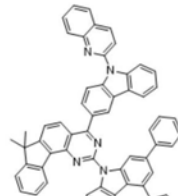
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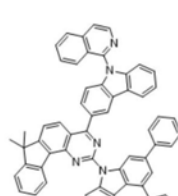
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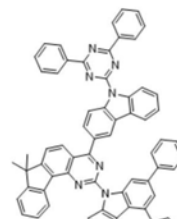
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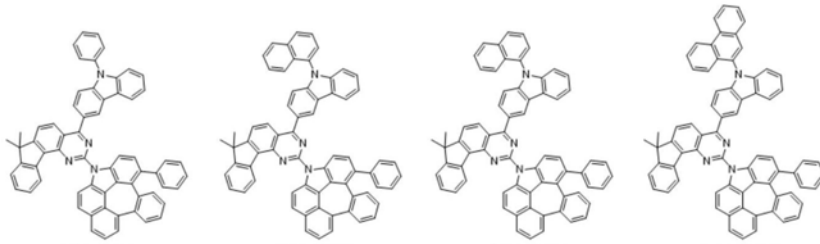
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RH-059



RH-060

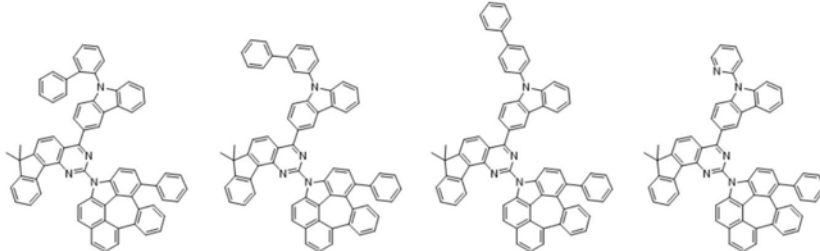


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RH-062

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RH-064

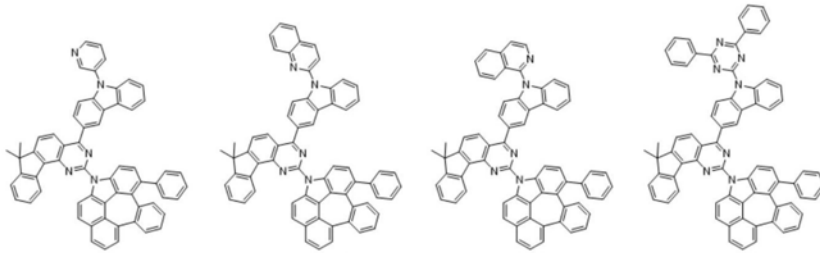


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RH-066

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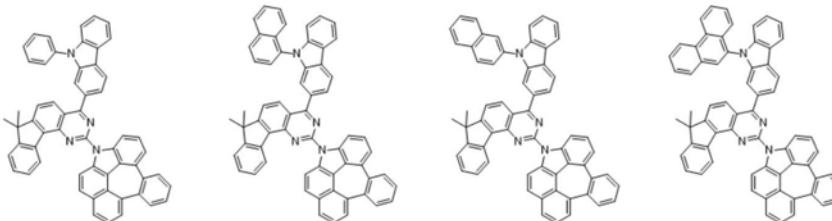


RH-069

RH-070

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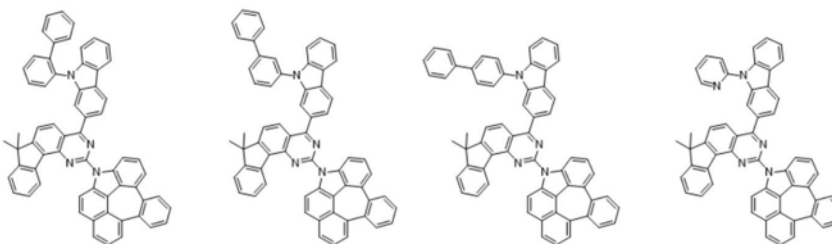


RH-073

RH-074

RH-075

RH-076

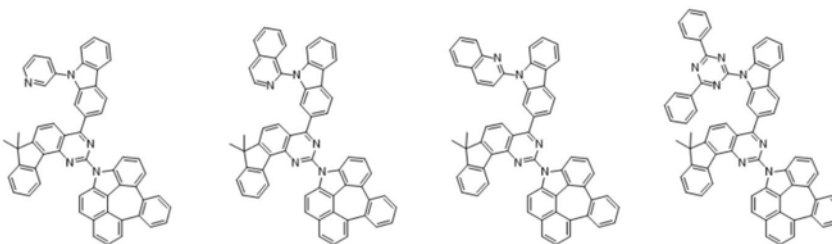


RH-077

RH-078

RH-079

RH-080

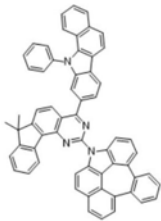


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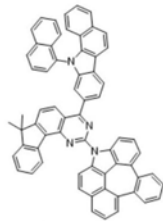
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RH-083

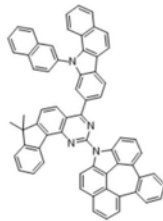
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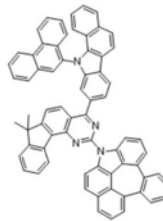
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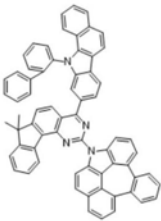
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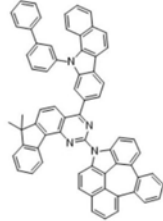
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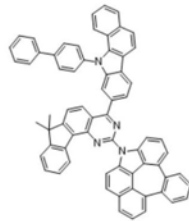
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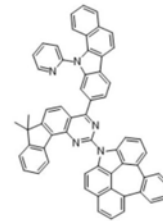
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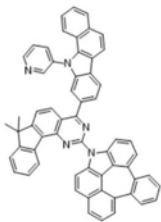
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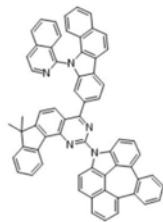
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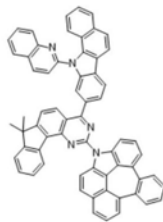
RH-092



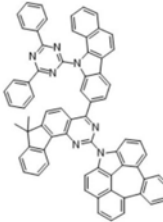
RH-093



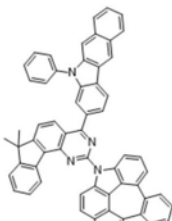
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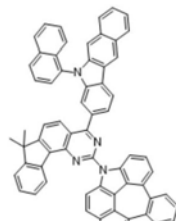
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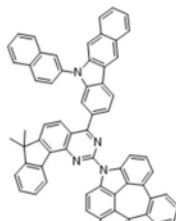
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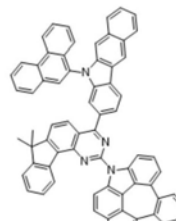
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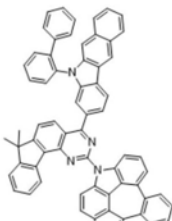
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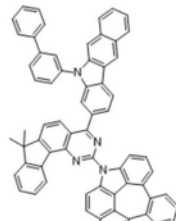
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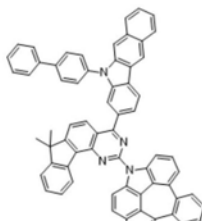
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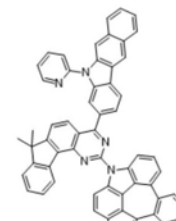
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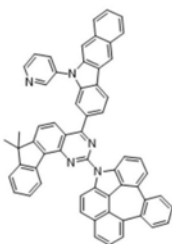
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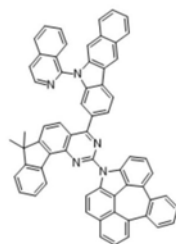
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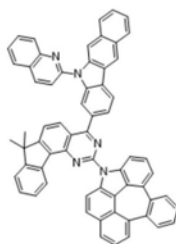
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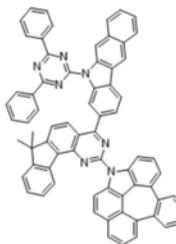
RH-105



RH-106

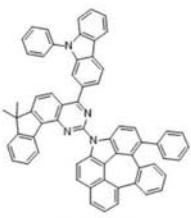


RH-107

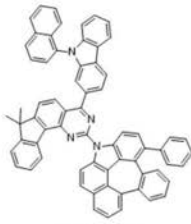


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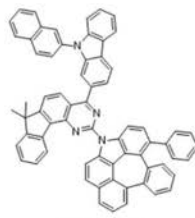




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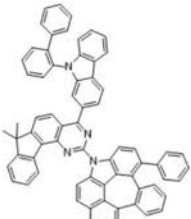
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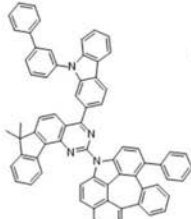
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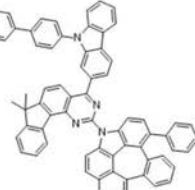
RH-136



RH-137



RH-138



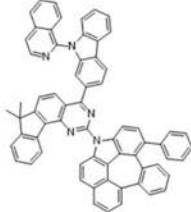
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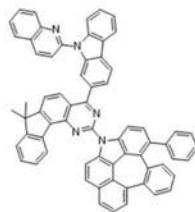
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RH-141



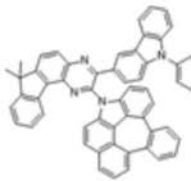
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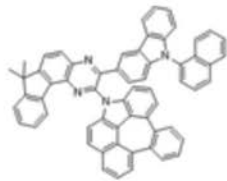
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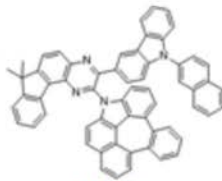
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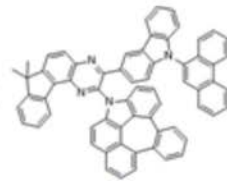
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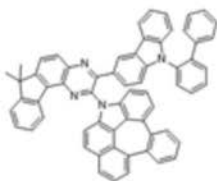
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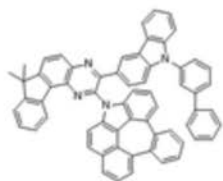
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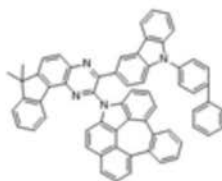
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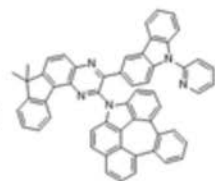
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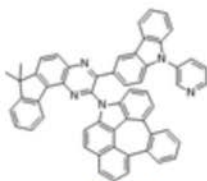
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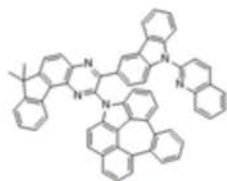
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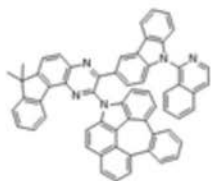
RH-152



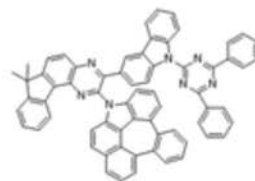
RH-153



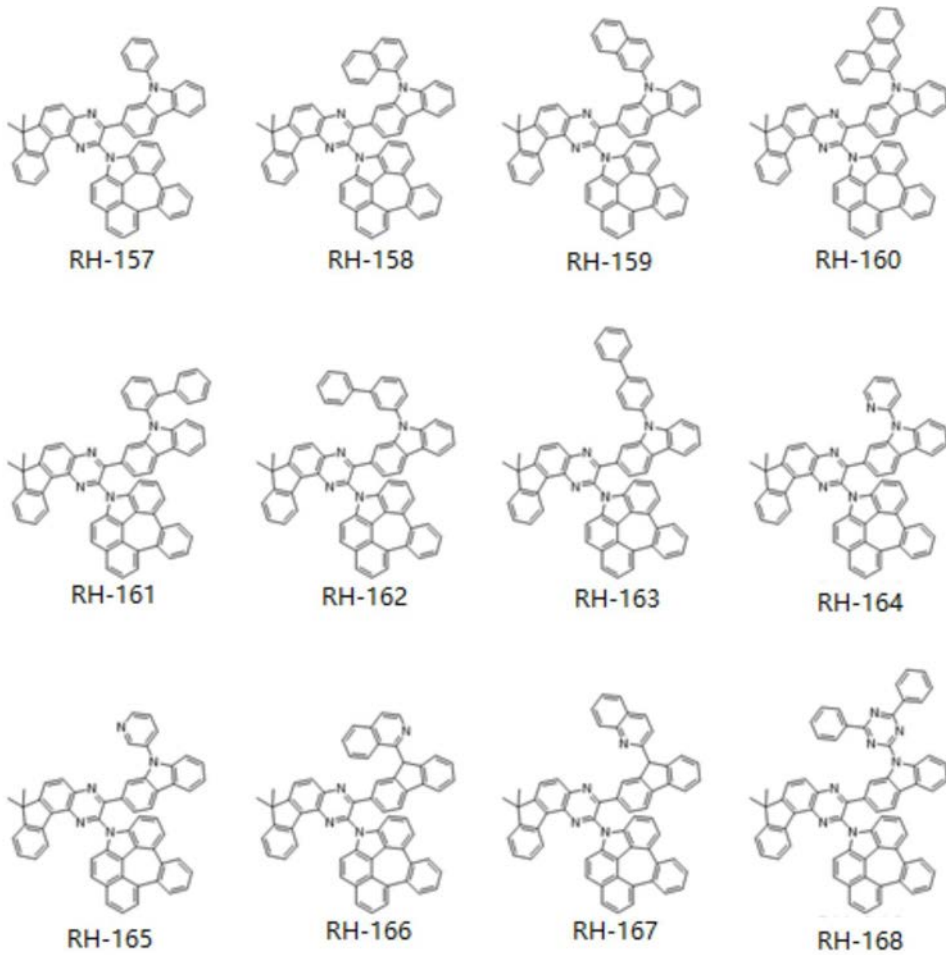
RH-154



RH-155



RH-156



6. 一种使用权利要求5所述的磷光化合物的有机电致发光器件,包括有机电致发光器件,其特征在于:所述有机电致发光器件顺次包括沉积的阳极、空穴注入层、空穴传输层、发光层、电子传输层、电子注入层和阴极,所述磷光化合物作为发光层的主体材料。

## 一种磷光化合物以及使用所述化合物的有机电致发光器件

### 技术领域

[0001] 本发明涉及有机电致发光技术领域,具体地说,涉及一种磷光化合物以及使用所述化合物的有机电致发光器件。

### 背景技术

[0002] 近来,对于平板显示器(例如液晶显示器和等离子显示面板)的需求在增加。但是,这些平板显示器与阴极射线管(CRT)相比具有较低的响应时间和较窄的视角。有机发光二极管(OLED)器件是能够解决以上问题并占地较小的下一代平板显示器之一。

[0003] OLED器件的元件可以在柔性基板(例如塑料基板)上形成。另外,OLED器件在视角、驱动电压、能耗和色纯度方面具有优势。此外,OLED器件足以产生全色图像。通常,OLED器件的发光二极管包括阳极、空穴注入层(HIL)、空穴输送层(HTL)、发光材料层(EML)、电子输送层(ETL)、电子注入层(EIL)和阴极。OLED器件通过以下方式发光:由作为电子注入电极的阴极和由作为空穴注入电极的阳极分别将电子和空穴注入发光化合物层中,从而使电子与空穴复合以产生激子,并使激子由激发态跃迁至基态。

[0004] 发光原理可以分为荧光发光和磷光发光。在荧光发光中,单线态激发状态的有机分子跃迁至基态,由此发出光。另一方面,在磷光发光中,三线态激发状态的有机分子跃迁至基态,由此发出光。当发光材料层发射对应于能带隙的光时,具有0自旋的单线态激子和具有1自旋的三线态激子以1:3的比例产生。有机材料的基态为单线态,这使单线态激子可以跃迁至基态并伴随发光。但是,由于三线态激子不能发生伴随发光的跃迁,因此使用荧光材料的OLED器件的内量子效率被限制在25%以内。

[0005] 另一方面,如果自旋轨道耦合动量很高,则单线态和三线态混合以使得在单线态和三线态之间产生系间跨越,并且三线态激子也可以跃迁至基态并伴随发光。磷光材料可以使用三线态激子和单线态激子,以使得使用磷光材料的OLED器件可以具有100%的内量子效率。

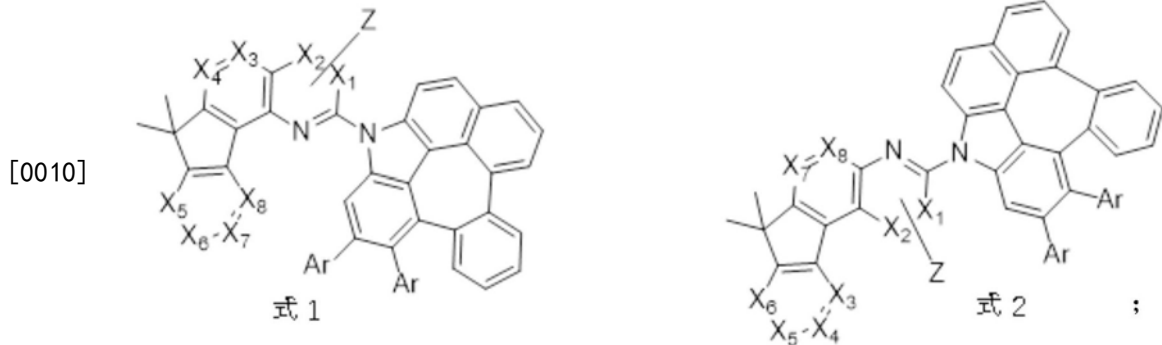
[0006] 近来,已将铱络合物,例如双(2-苯基喹啉)(乙酰丙酮)铱(III)(Ir(2-phq)<sub>2</sub>(acac))、双(2-苯并[b]噻吩-2-基吡啶)(乙酰丙酮)铱(III)(Ir(btp)<sub>2</sub>(acac))和三(2-苯基喹啉)铱(III)Ir(2-phq)<sub>3</sub>掺杂剂引入。为了利用磷光材料获得高电流发光效率(Cd/A),需要优异的内部量子效率、高的色纯度和长寿命。特别是,色纯度越高,即,CIE(X)越高,颜色灵敏度越差。结果,在高内量子效率下,非常难获得发光效率。因此,需要具有优异色纯度(CIE(X) ≥ 0.65)和高发光效率的新型红色磷光化合物。

[0007] 另一方面,除了上述的铱络合物之外,例如,4,4'-N,N'咔唑联苯(CBP)或其他金属络合物用作红色磷光化合物。然而,这些化合物在溶剂中不具有理想的溶解度,因而不能通过溶液工艺来形成发光层。发光层应当通过沉积工艺形成,因此,制造过程极为复杂,工艺效率也极低。另外,沉积工艺中的废料非常多,导致生产成本增大。

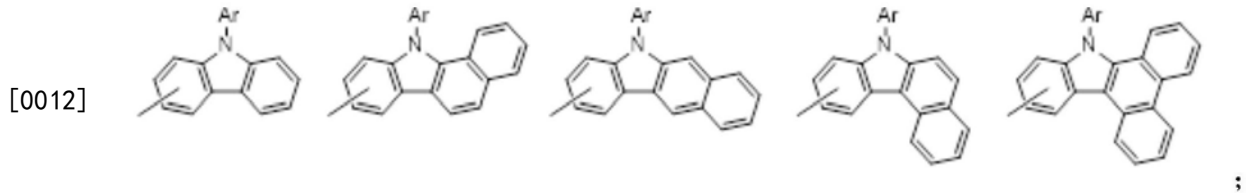
发明内容

[0008] 本发明的目的在于提供一种磷光化合物以及使用所述化合物的有机电致发光器件,以解决上述背景技术中提出的某种或某些缺陷。

[0009] 为实现上述目的,本发明提供一种磷光化合物,其结构式如式1和式2所示:



[0011] 上述的结构式1和结构式2中,Z独立地选自以下结构:



[0013] 其中X1至X2独立的选自N原子或C原子;

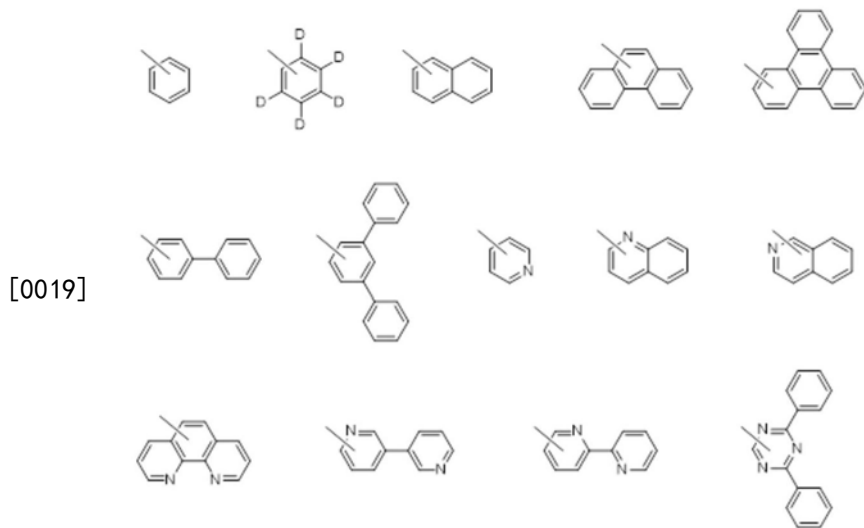
[0014] 其中X1至X2中的至少一个为N;

[0015] 其中Ar独立地选自、C6-C30芳基、C2-C30杂芳基中。

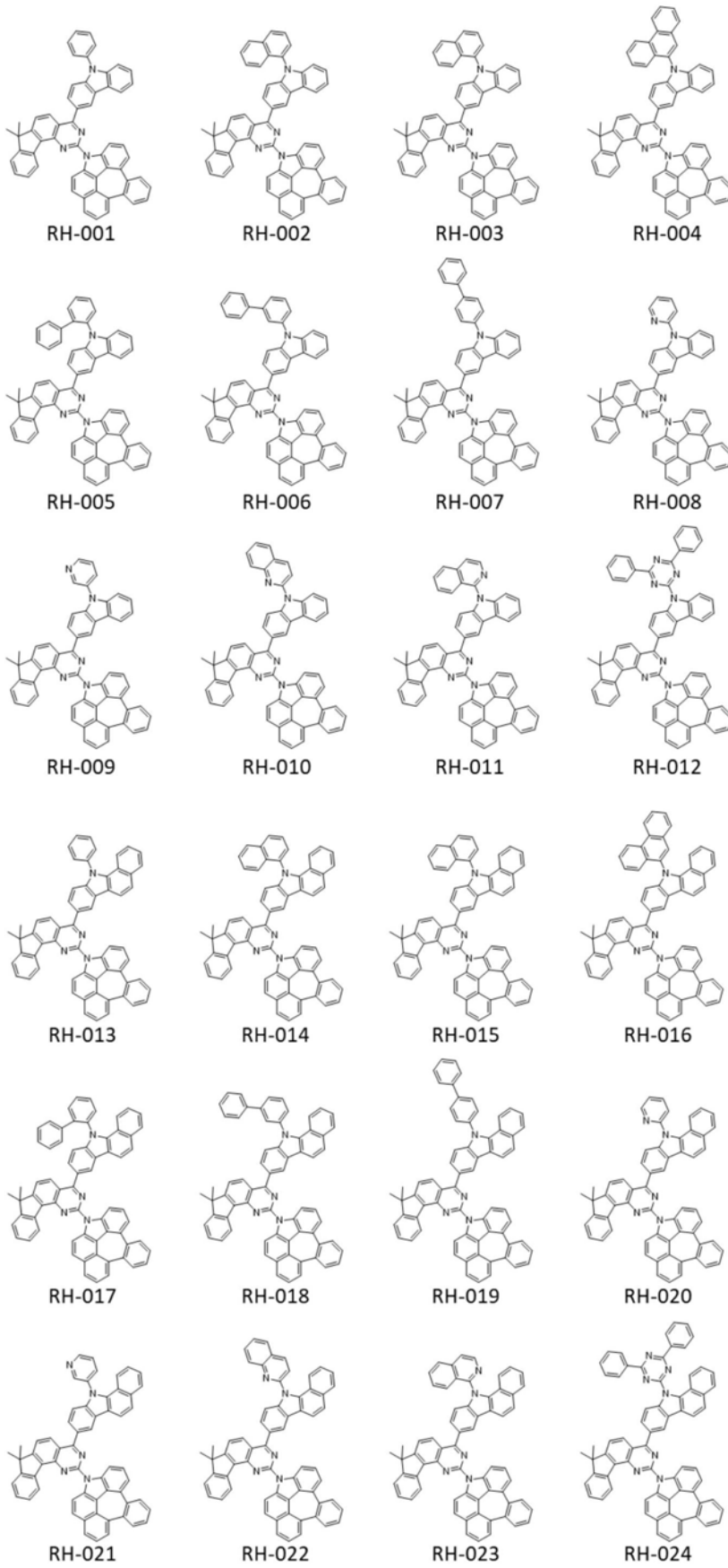
[0016] 作为优选,所述C6-C30芳基选自苯基、萘基、联苯基、三联苯基和菲基中的一种。

[0017] 作为优选,所述C2-C30杂芳基选自吡啶基、联吡啶基、喹啉基、异喹啉基、菲咯啉基和三嗪基中的一种。

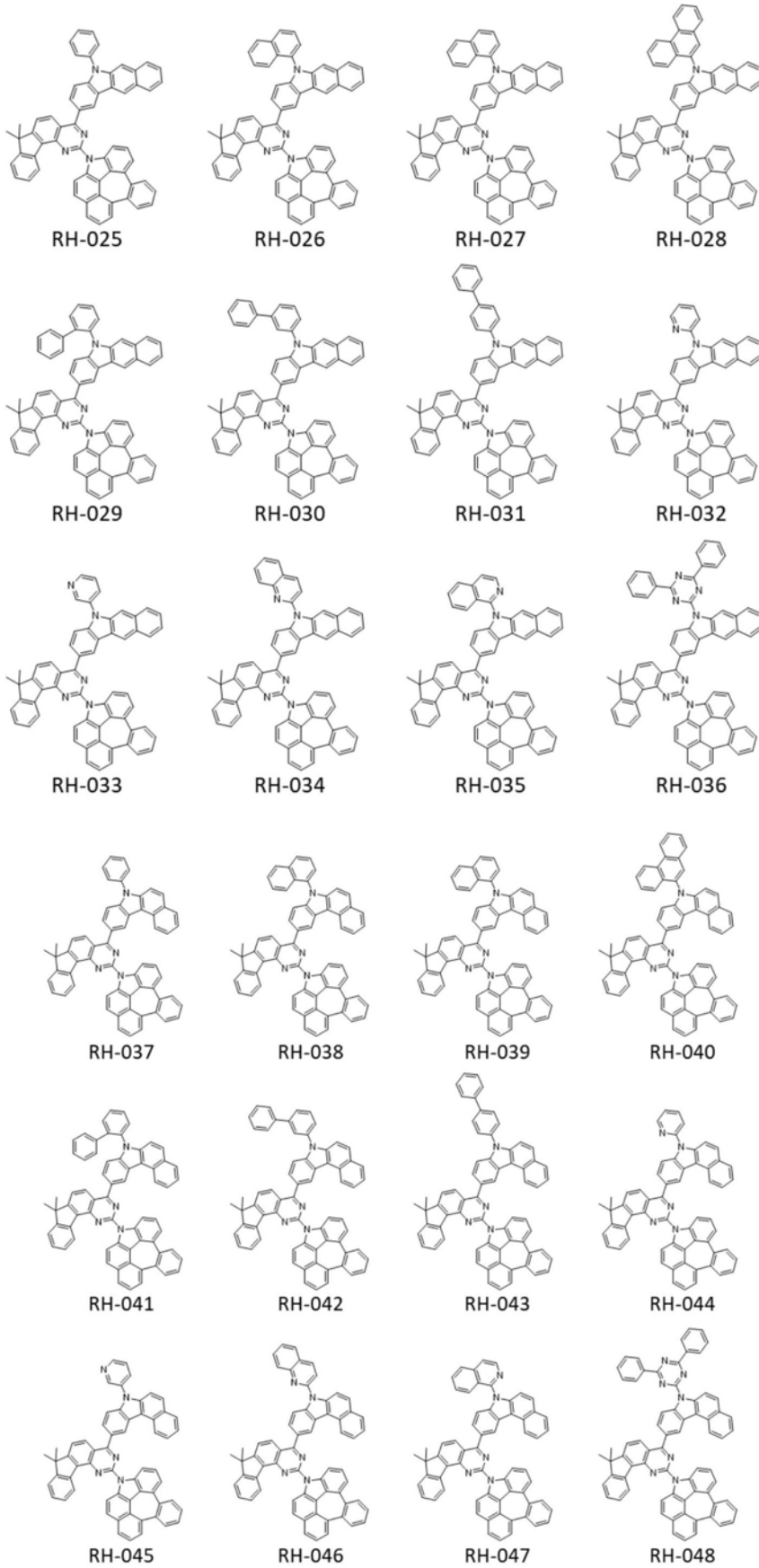
[0018] 作为优选,所述Ar独立地选自以下:



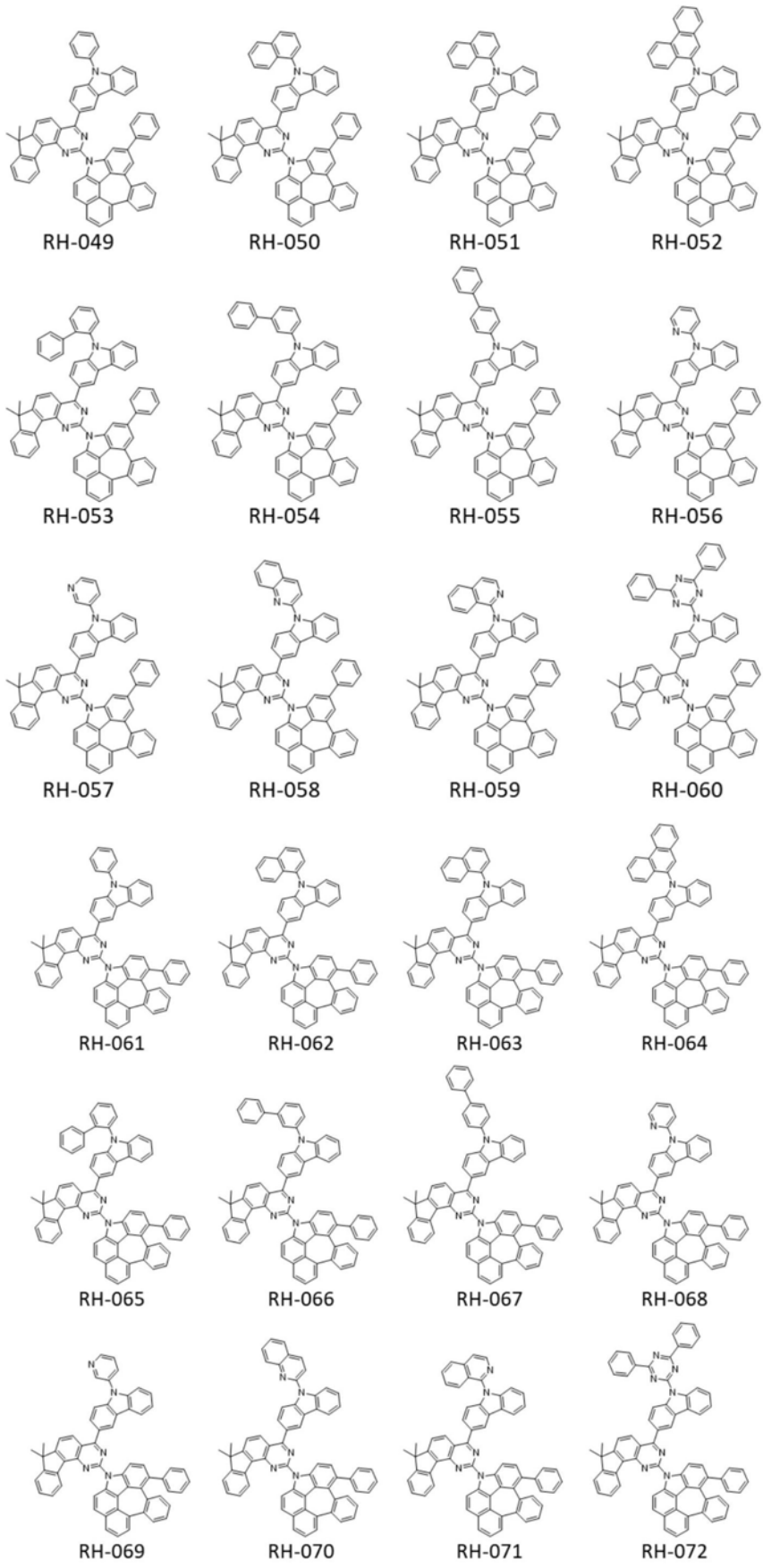
[0020] 作为优选,所述磷光化合物独立地选自下列化合物:



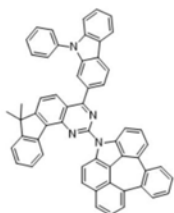
[0021]



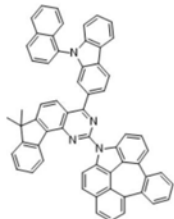
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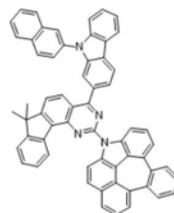
[0023]



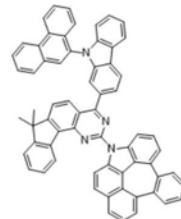
RH-073



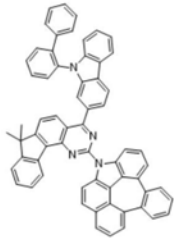
RH-074



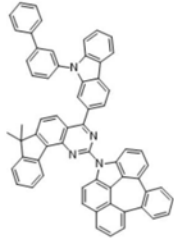
RH-075



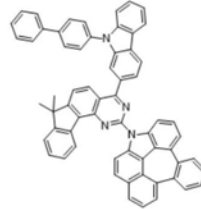
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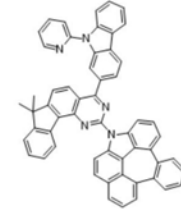
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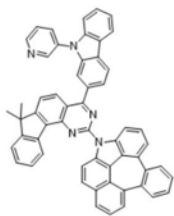
RH-078



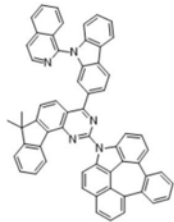
RH-079



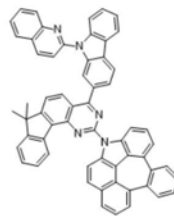
RH-080



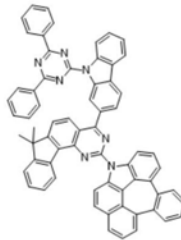
RH-081



RH-082

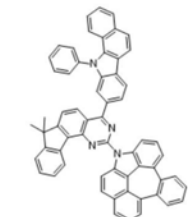


RH-083

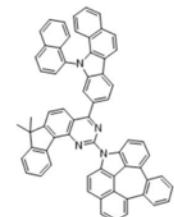


RH-084

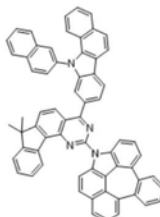
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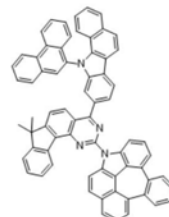
RH-085



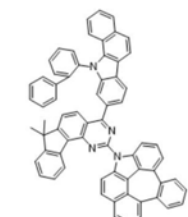
RH-086



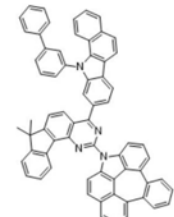
RH-087



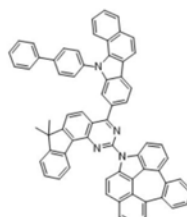
RH-088



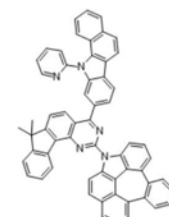
RH-089



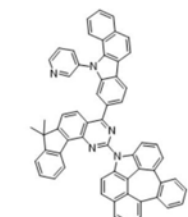
RH-090



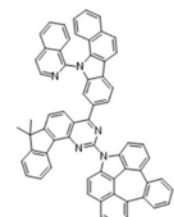
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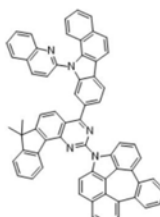
RH-092



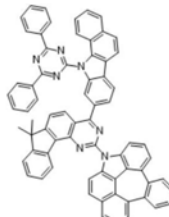
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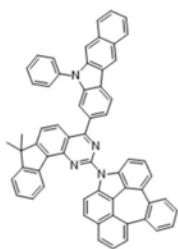
RH-094



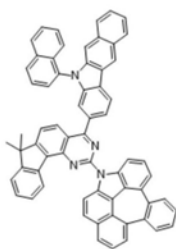
RH-095



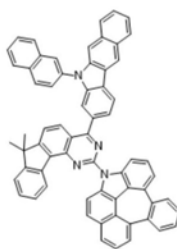
RH-096



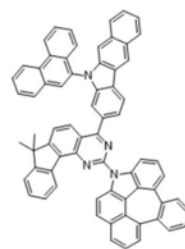
RH-097



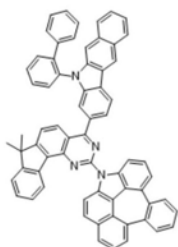
RH-098



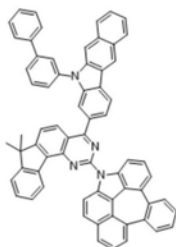
RH-099



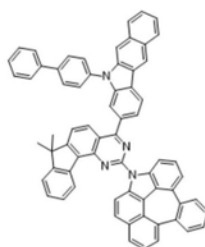
RH-100



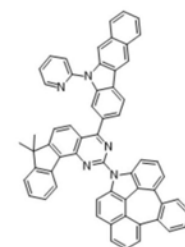
RH-101



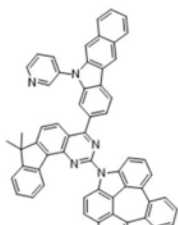
RH-102



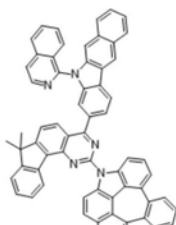
RH-103



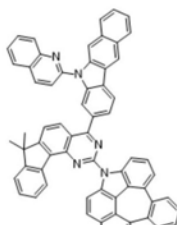
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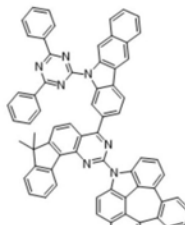
RH-105



RH-106

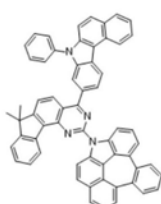


RH-107

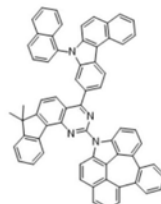


RH-108

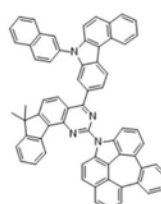
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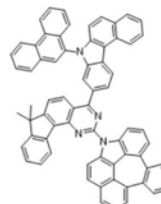
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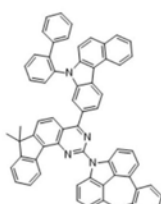
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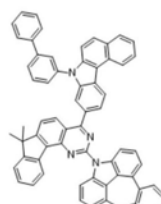
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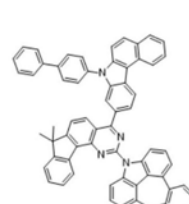
RH-112



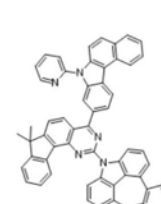
RH-113



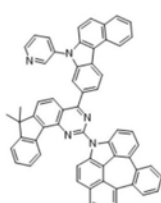
RH-114



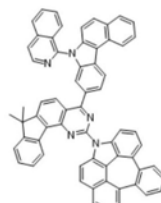
RH-115



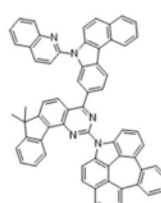
RH-116



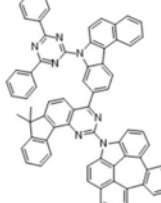
RH-117



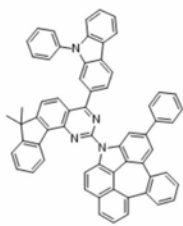
RH-118



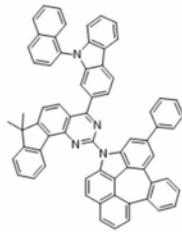
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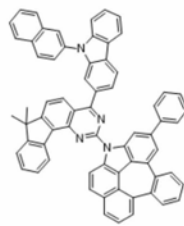
RH-120



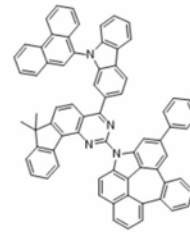
RH-121



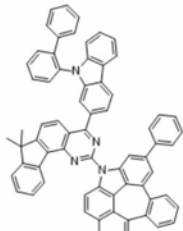
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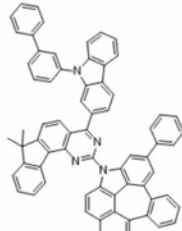
RH-123



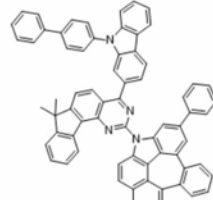
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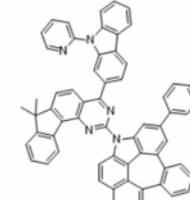
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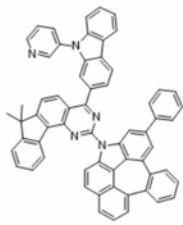
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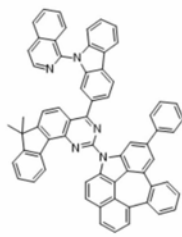
RH-127



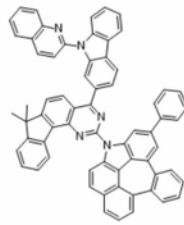
RH-128



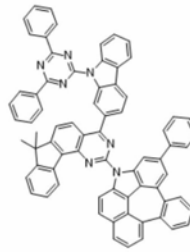
RH-129



RH-130

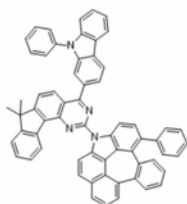


RH-131

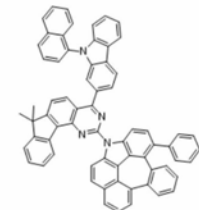


RH-132

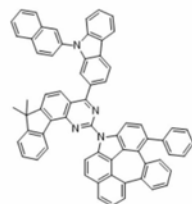
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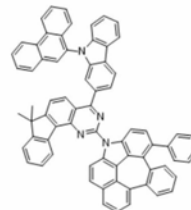
RH-133



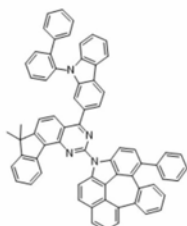
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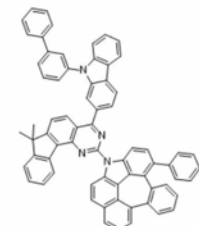
RH-135



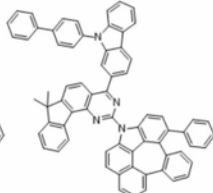
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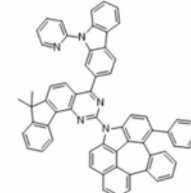
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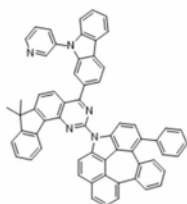
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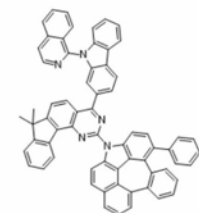
RH-139



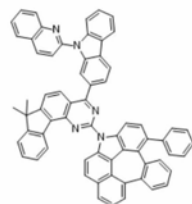
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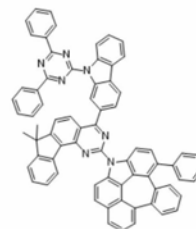
RH-141



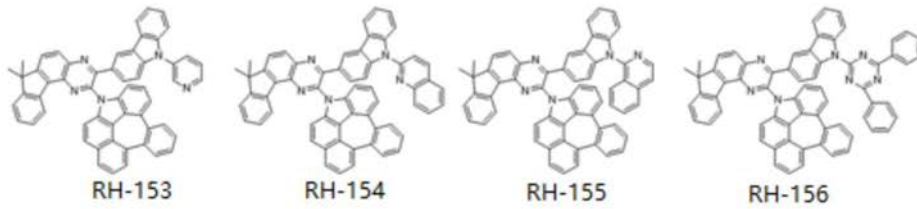
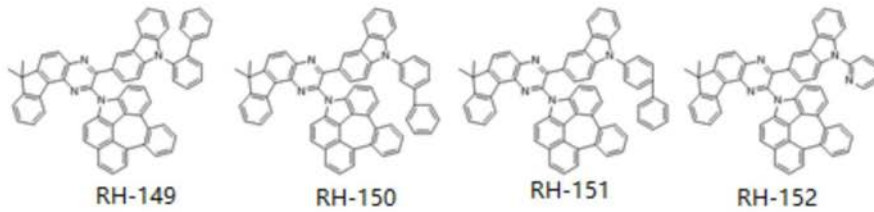
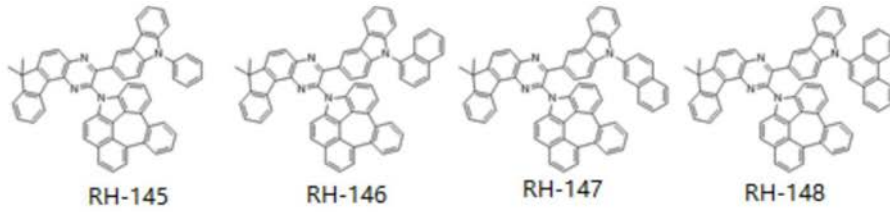
RH-142



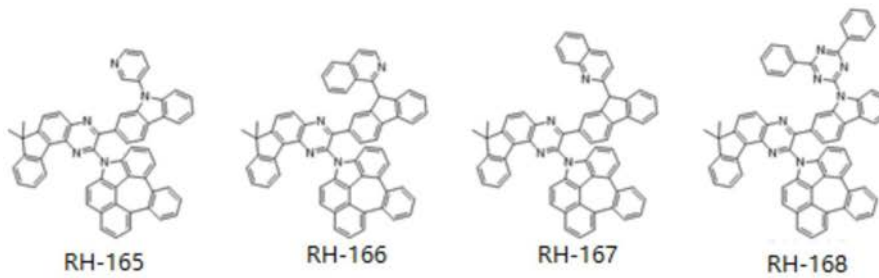
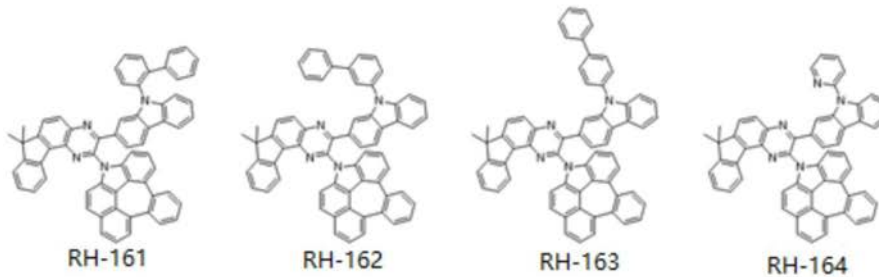
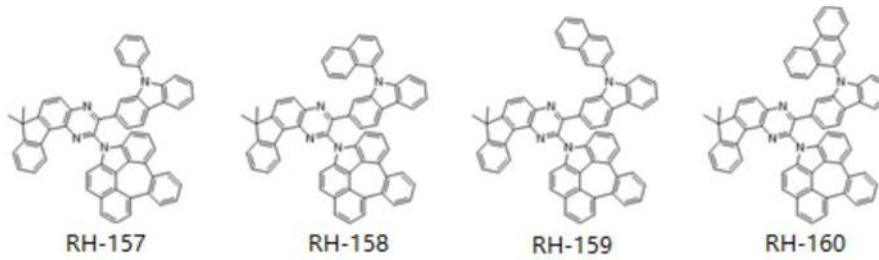
RH-143



RH-144



[0027]



[0028] 一种使用上述磷光化合物的有机电致发光器件,包括有机电致发光器件,所述有机电致发光器件顺次包括沉积的阳极、空穴注入层、空穴传输层、发光层、电子传输层、电

子注入层和阴极,所述磷光化合物作为发光层的主体材料。

[0029] 与现有技术相比,本发明的有益效果:

[0030] 1、该磷光化合物以及使用所述化合物的有机电致发光器件中,在电流为0.9mA情况下,亮度可达到1101cd/m<sup>2</sup>,而普通发光材料在同等电流情况下,亮度仅仅为780cd/m<sup>2</sup>,有效提高光源亮度。

[0031] 2、该磷光化合物以及使用所述化合物的有机电致发光器件中,在电流为0.9mA情况下,电流效率能达到为11.0cd/A,而普通发光材料在同等电流情况下,电流效率仅有7.8cd/A,电流效率增加40%以上。

[0032] 3、该磷光化合物以及使用所述化合物的有机电致发光器件中,在电流为0.9mA情况下,功率效率最高能达到为5.7lm/W,而普通发光材料在同等电流情况下,功率效率仅有3.3lm/W,有效提高功率效率。

[0033] 4、该磷光化合物以及使用所述化合物的有机电致发光器件中,在电流为0.9mA情况下,带动有机电致发光器件工作的电压最低只需要6.0V,而普通发光材料在同等电流情况下,电压需要7.5V,实现节能效果。

[0034] 5、该磷光化合物以及使用所述化合物的有机电致发光器件中,当色纯度高时,该发明提供的有机发光管件电压为6.2V,同时功率效率高达5.1lm/W,而普通发光材料在同等色纯度时,电压为7.5V,功率效率仅为3.3lm/W,同时电流效率增加45%以上。

[0035] 6、该磷光化合物以及使用所述化合物的有机电致发光器件中,采用磷光化合物作为发光元件,制造过程简单,工艺效率高,生产成本低。

[0036] 7、该磷光化合物以及使用所述化合物的有机电致发光器件中,N在X2位上,性能更好,相同条件下,寿命延长一倍。

## 附图说明

[0037] 图1为本发明的红色磷光化合物结构式一示意图;

[0038] 图2为本发明的红色磷光化合物结构式二示意图;

[0039] 图3为本发明的有机发光器件结构层图;

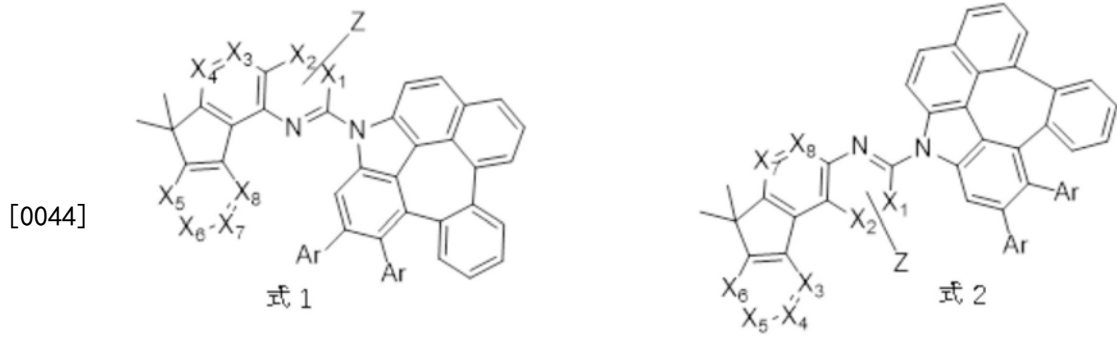
[0040] 图4为本发明的有机发光器件发光色度和可见度关系图。

## 具体实施方式

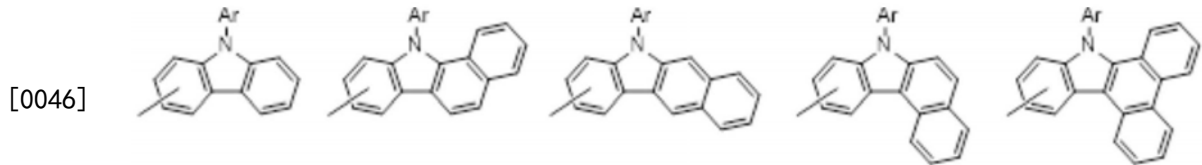
[0041] 下面将结合本发明实施例中的附图,对本发明实施例中的技术方案进行清楚、完整地描述,显然,所描述的实施例仅仅是本发明一部分实施例,而不是全部的实施例。基于本发明中的实施例,本领域普通技术人员在没有做出创造性劳动前提下所获得的所有其他实施例,都属于本发明保护的范围。

[0042] 请参阅图1-图4,本发明提供一种方案:

[0043] 本发明提供一种磷光化合物,其结构式如式1和式2所示:



[0045] 上述的结构式1和结构式2中,Z独立地选自以下结构:



[0047] 其中X1至X2独立的选自N原子或C原子;

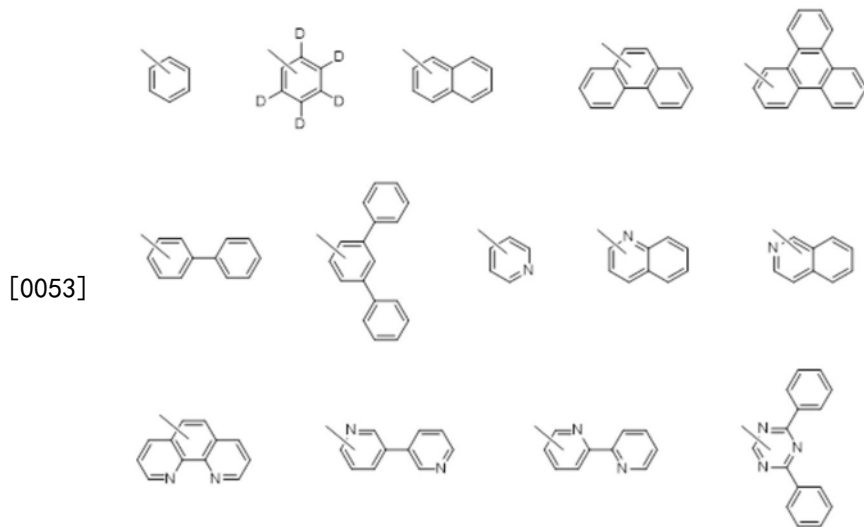
[0048] 其中X1至X2中的至少一个为N;

[0049] 其中Ar独立地选自、C6-C30芳基、C2-C30杂芳基中。

[0050] C6-C30芳基选自苯基、萘基、联苯基、三联苯基和菲基中的一种。

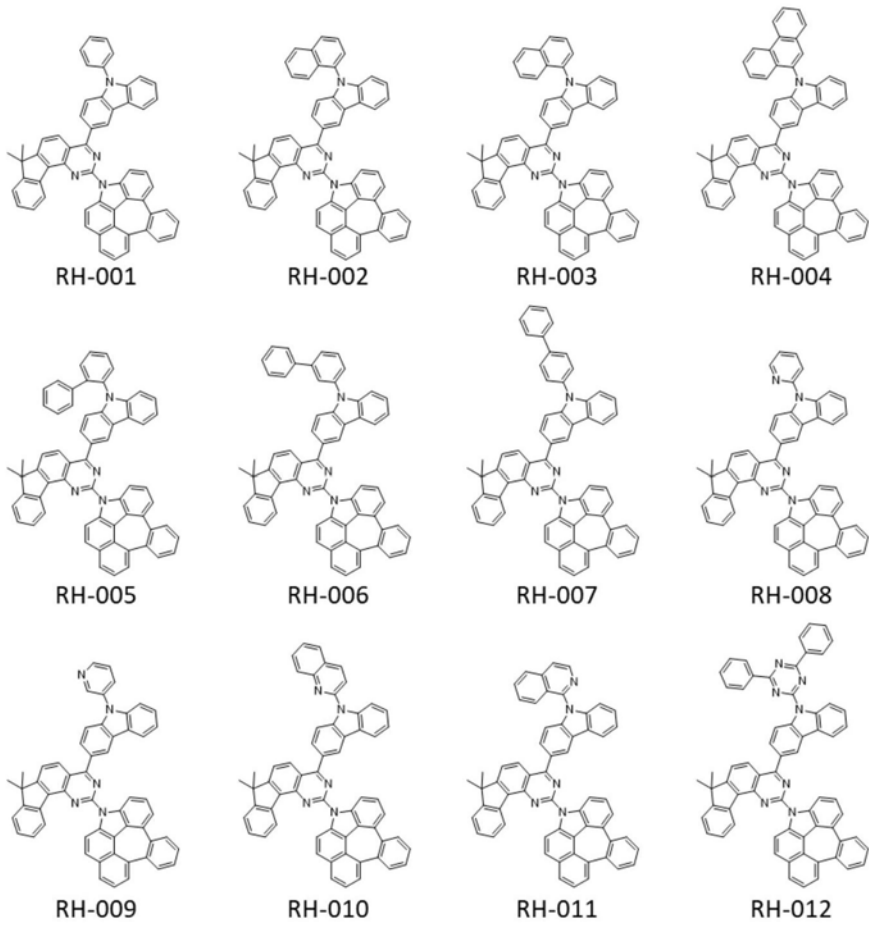
[0051] C2-C30杂芳基选自吡啶基、联吡啶基、喹啉基、异喹啉基、菲咯啉基和三嗪基中的一种。

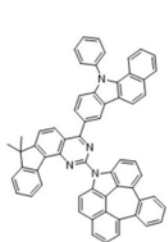
[0052] Ar独立地选自以下:



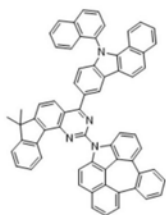
[0054] 磷光化合物独立地选自下列化合物:

[0055]

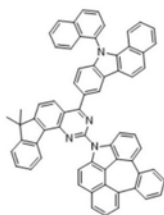




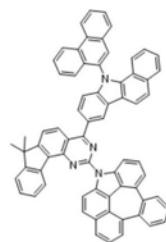
RH-013



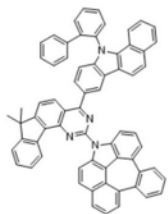
RH-014



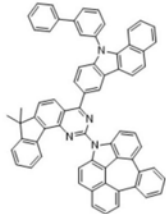
RH-015



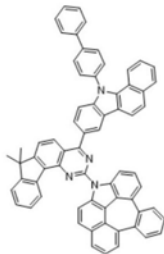
RH-016



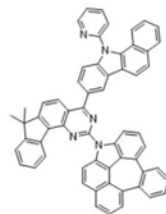
RH-017



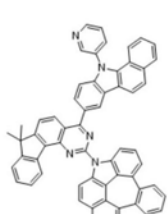
RH-018



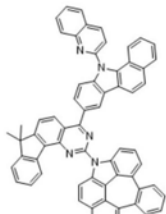
RH-019



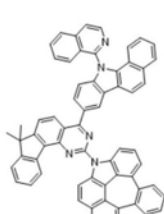
RH-020



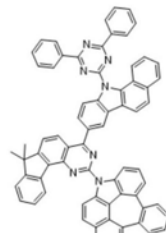
RH-021



RH-022

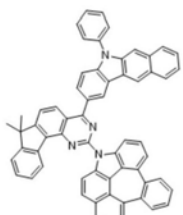


RH-023

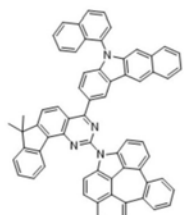


RH-024

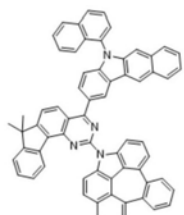
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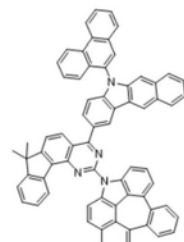
RH-025



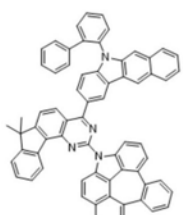
RH-026



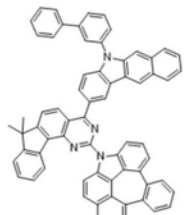
RH-027



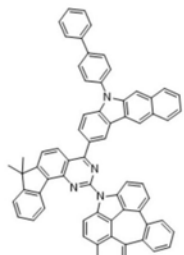
RH-028



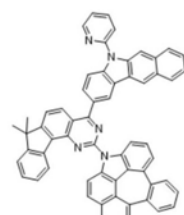
RH-029



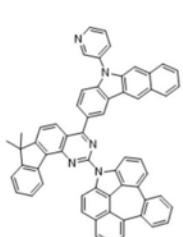
RH-030



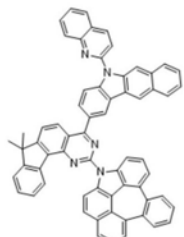
RH-031



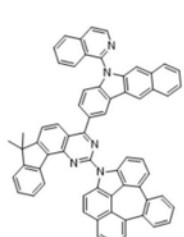
RH-032



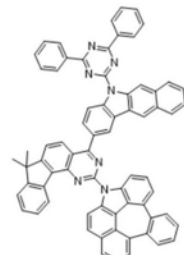
RH-033



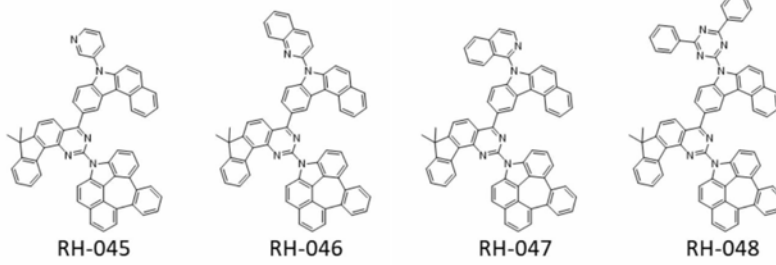
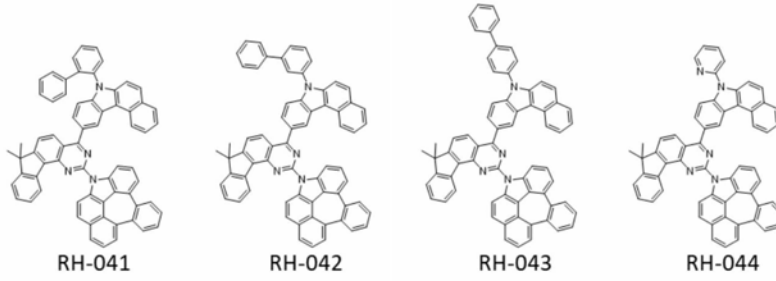
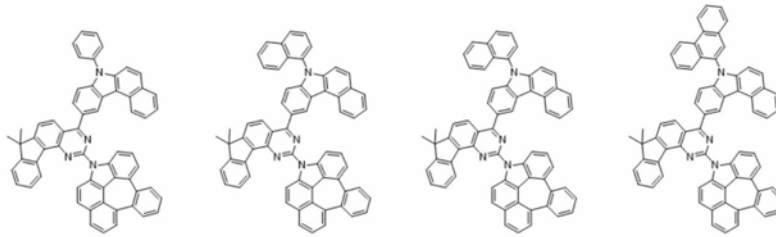
RH-034



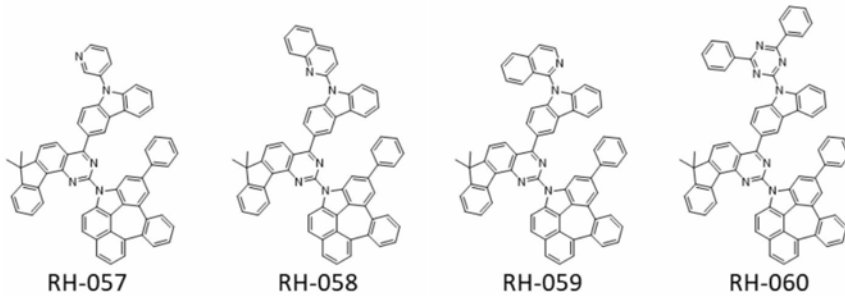
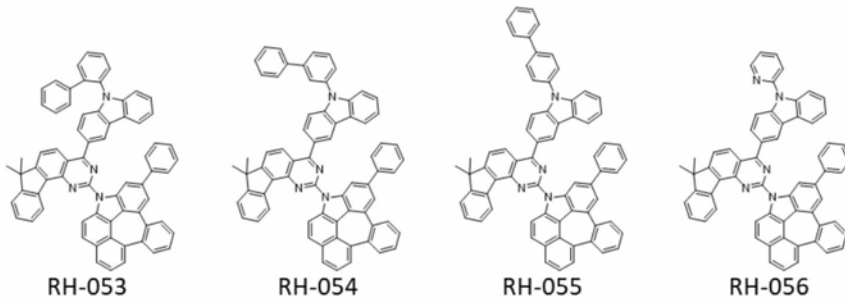
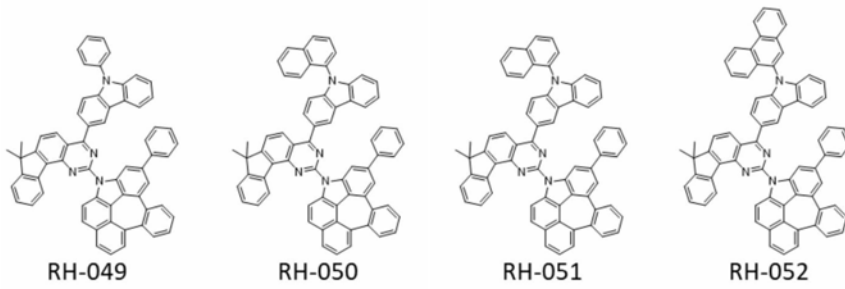
RH-035

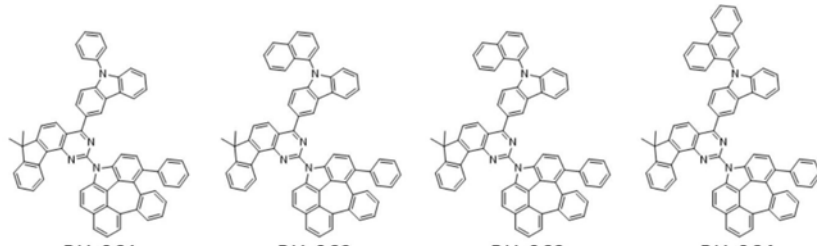


RH-036



[0057]



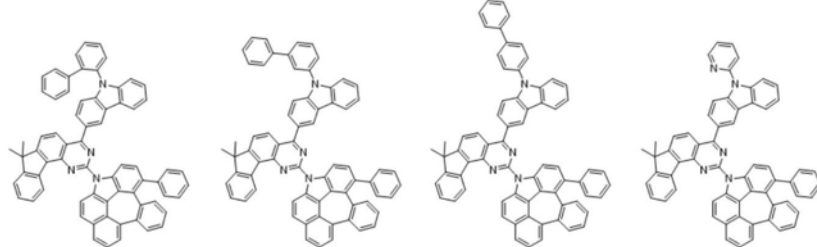


RH-061

RH-062

RH-063

RH-064

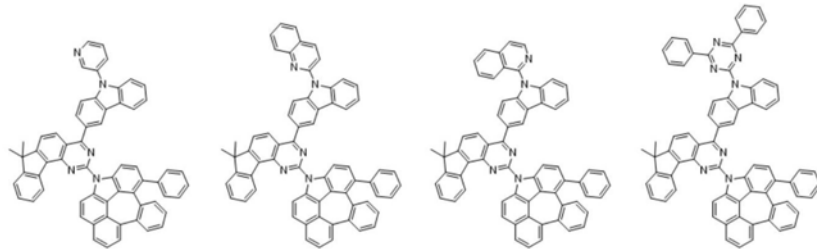


RH-065

RH-066

RH-067

RH-068



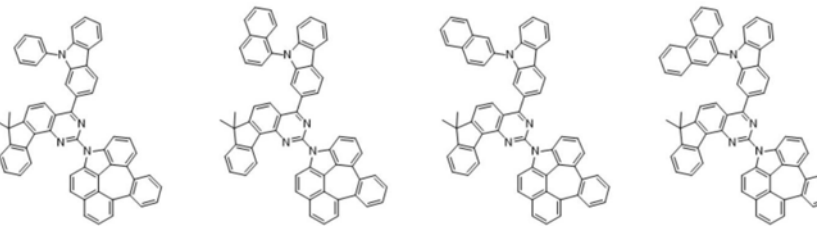
RH-069

RH-070

RH-071

RH-072

[0058]

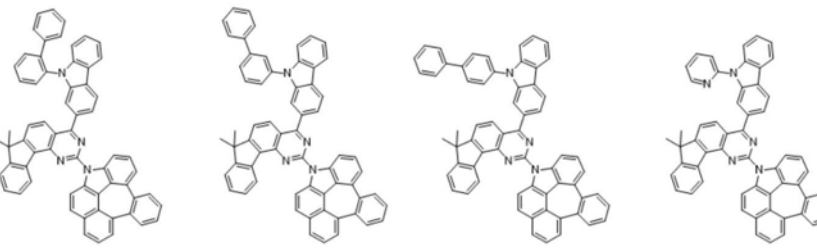


RH-073

RH-074

RH-075

RH-076

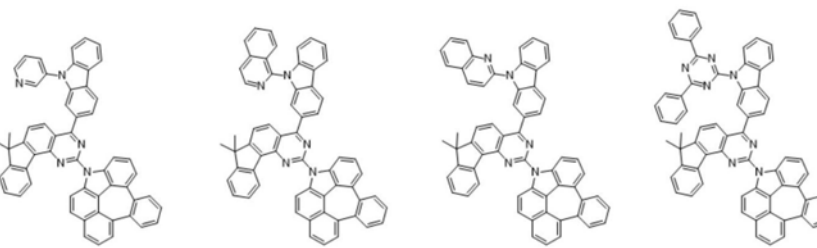


RH-077

RH-078

RH-079

RH-080

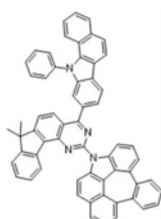


RH-081

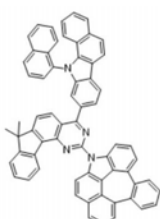
RH-082

RH-083

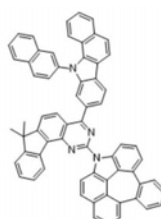
RH-084



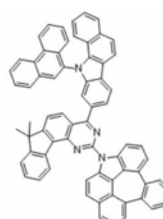
RH-085



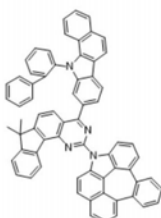
RH-086



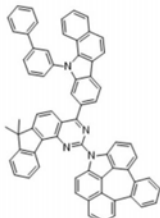
RH-087



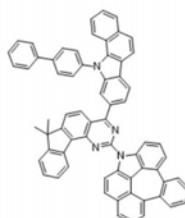
RH-088



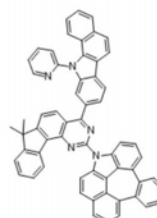
RH-089



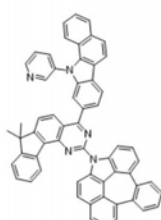
RH-090



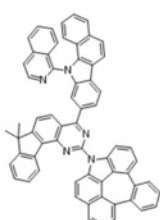
RH-091



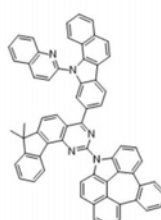
RH-092



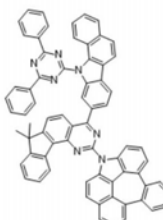
RH-093



RH-094

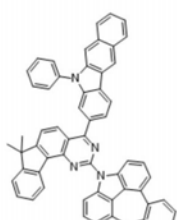


RH-095

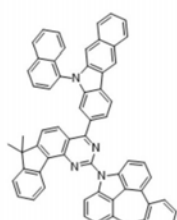


RH-096

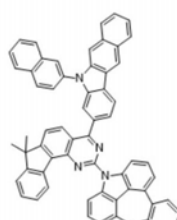
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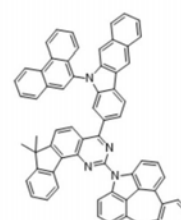
RH-097



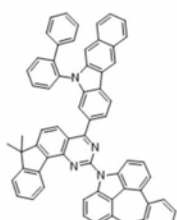
RH-098



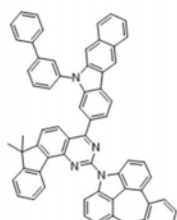
RH-099



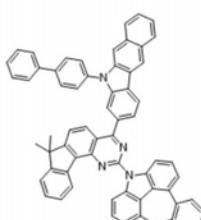
RH-100



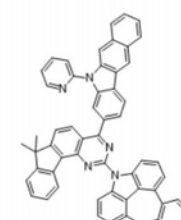
RH-101



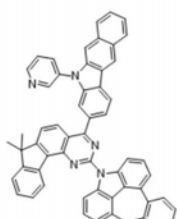
RH-102



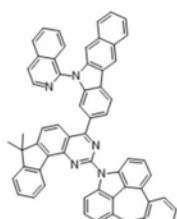
RH-103



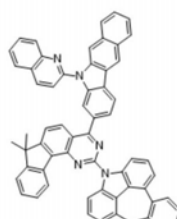
RH-104



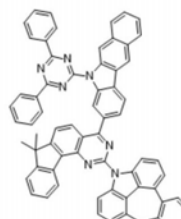
RH-105



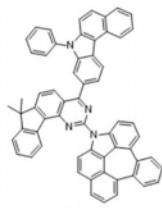
RH-106



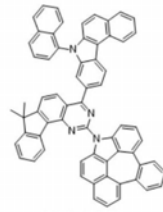
RH-107



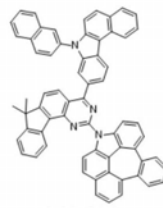
RH-108



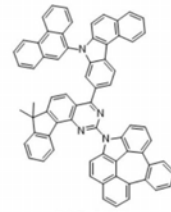
RH-109



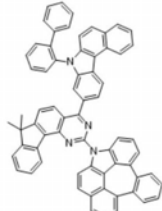
RH-110



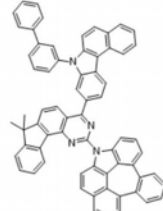
RH-111



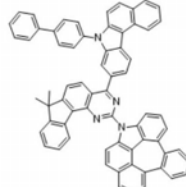
RH-112



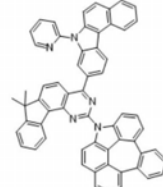
RH-113



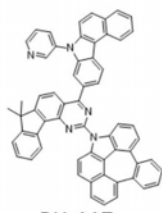
RH-114



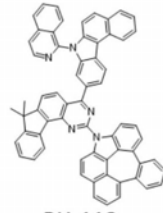
RH-115



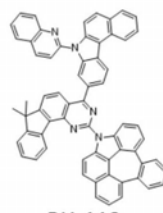
RH-116



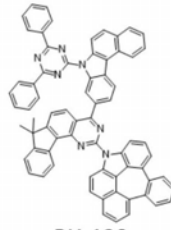
RH-117



RH-118

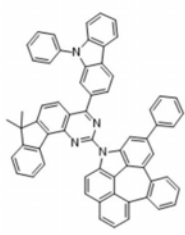


RH-119

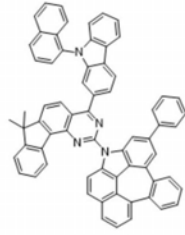


RH-120

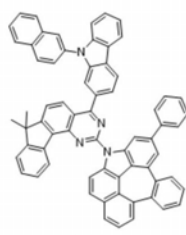
[0060]



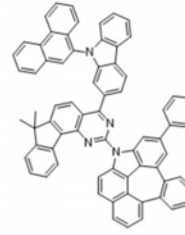
RH-121



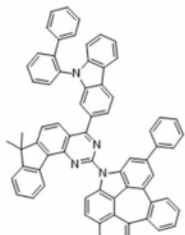
RH-122



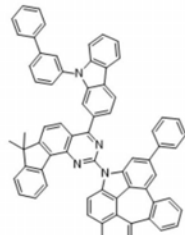
RH-123



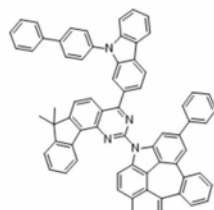
RH-124



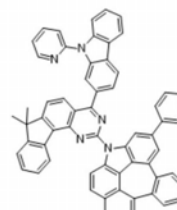
RH-125



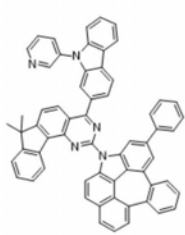
RH-126



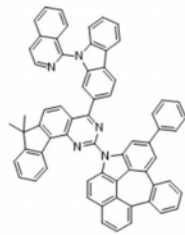
RH-127



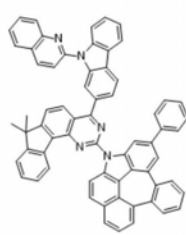
RH-128



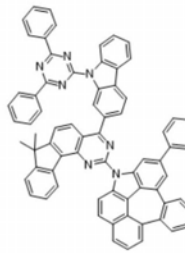
RH-129



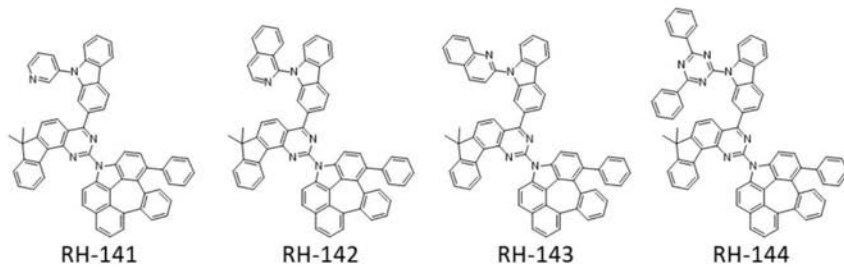
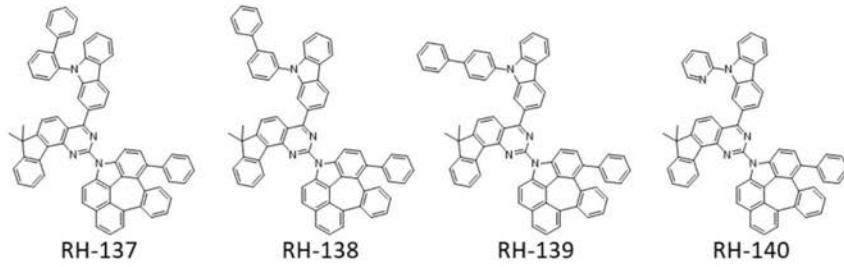
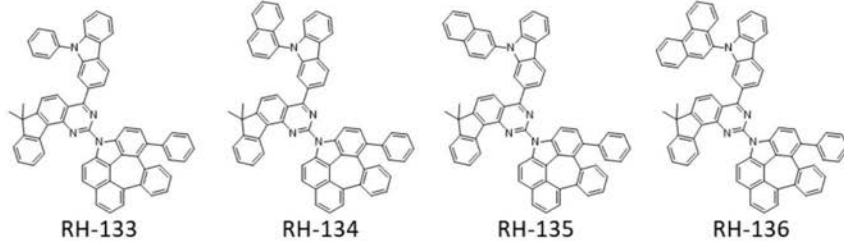
RH-130



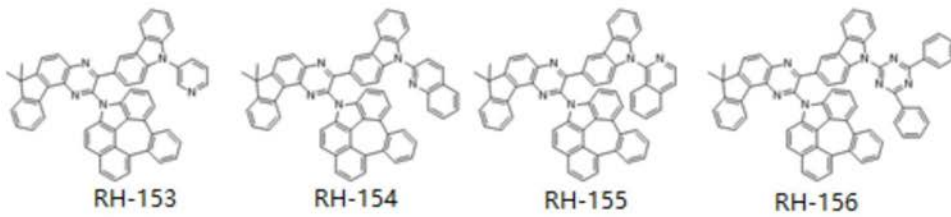
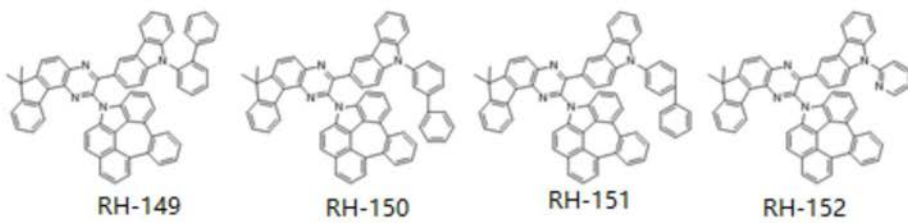
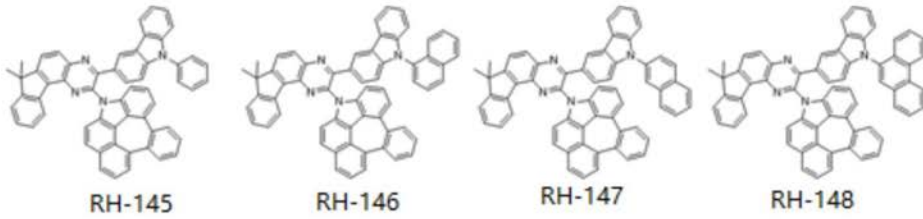
RH-131

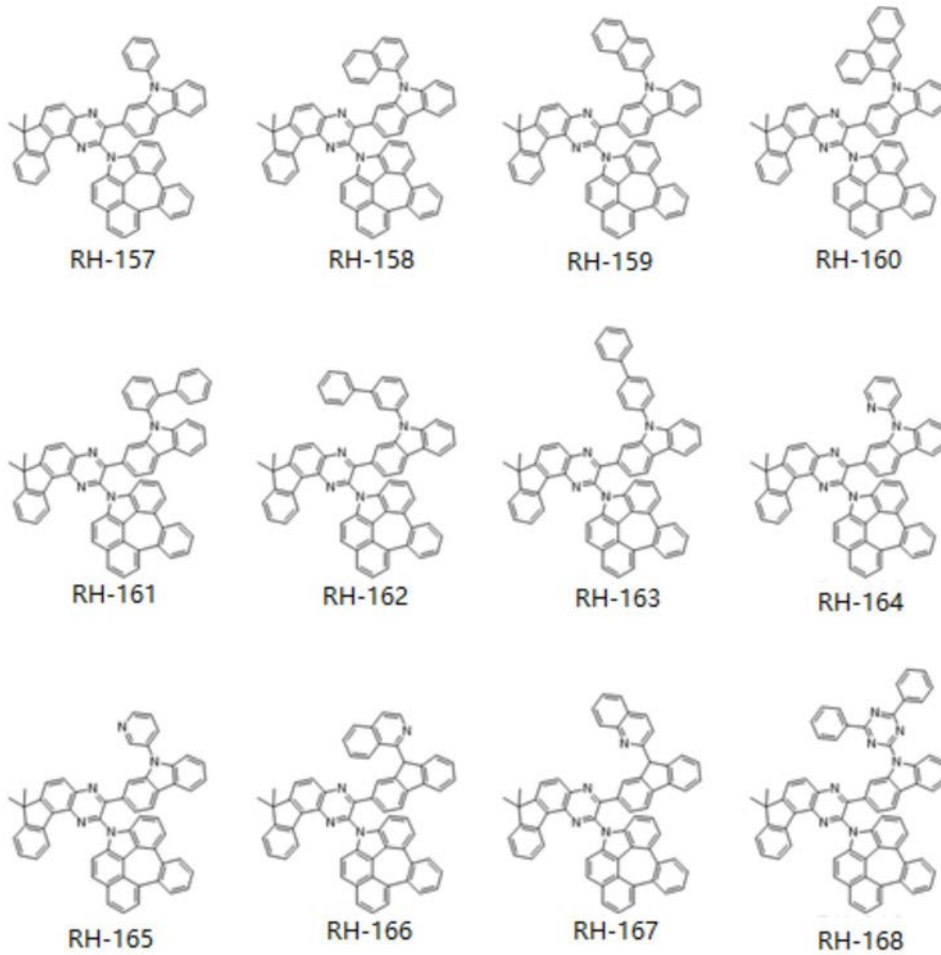


RH-132



[0061]





[0062]

[0063] 一种使用上述磷光化合物的有机电致发光器件,包括有机电致发光器件,有机电致发光器件顺次包括沉积的阳极、空穴注入层、空穴传输层、发光层、电子传输层、电子注入层和阴极,磷光化合物作为发光层的主体材料。

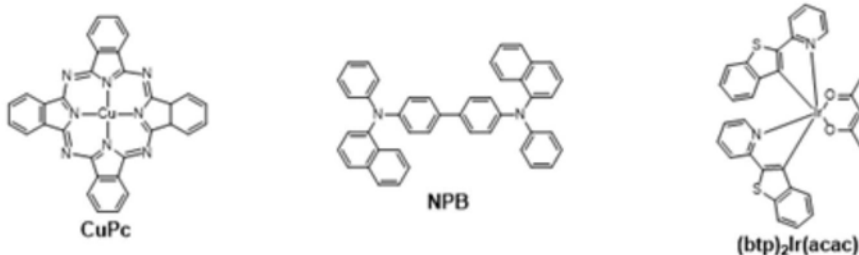
[0064] 为了使本发明实现的技术手段、创作特征、达成目的与功效易于明白了解,下面结合图示与具体实施例,进一步阐述本发明。

[0065] 由于结构式如式1和式2的红色磷光化合物均具有优异的纯色度、高亮度和优异的光效率,现以RH-001、RH-007、RH-073和RH-079制备方法和测试结果为例,证明本发明提供的技术方案和达到的技术效果。

[0066] 以下实施方案中,NPB为4,4'-双[N-(1-萘基)-N-苯氨基]联苯,CBP为4,4'-N,N'-二吡哇联苯,CuPc为酞菁铜,LiF氟化锂,ITO为氧化铟锡,Alq3为三(8-羟基喹啉)铝。

[0067] LC-MS:液相色谱-质谱联用仪,M/Z:质子数/电荷数的比值。

[0068] 本发明本实施方案中使用的化合物铜(II)酞菁(CuPc),NPB,(btp)2Ir(acac),Alq3和CBP的结构式如下。

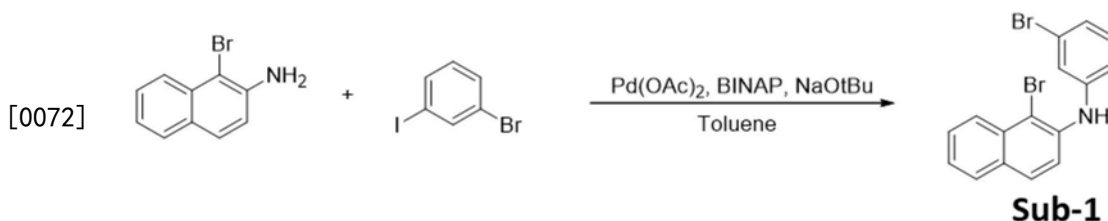


[0069]



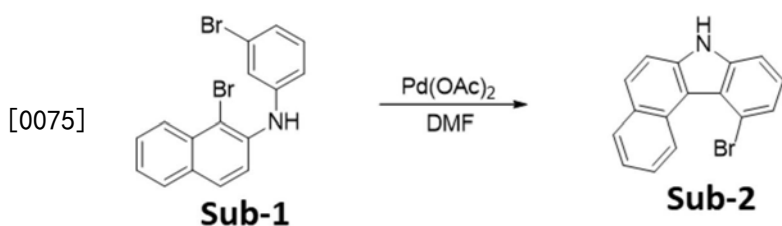
[0070] 形成例

[0071] 1. 中间体Sub-1的合成:



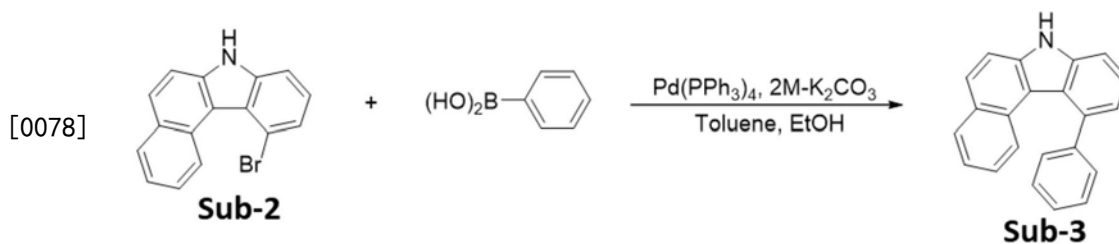
[0073] 在2000mL反应瓶中加入间溴碘苯(50.0g, 176.7mmol), 2-氨基-1-溴萘(43.2g, 194.4mmol)加入干燥并脱气的甲苯(1000mL)作为溶剂。然后加入叔丁醇钠(51.0g, 530.2mmol), 醋酸钯(2mol%)和1,1'-联萘-2,2'-双二苯膦(4mol%)。加料结束后, 将温度升至110℃反应16h。反应终止并冷却至室温。用活性炭吸附后过滤, 除去溶剂。使用甲苯和乙醇重结晶, 得到中间体Sub-1(56.6g, yield85%)。LC-MS:M/Z378.1 (M+H)<sup>+</sup>。

[0074] 2. 中间体Sub-2的合成:



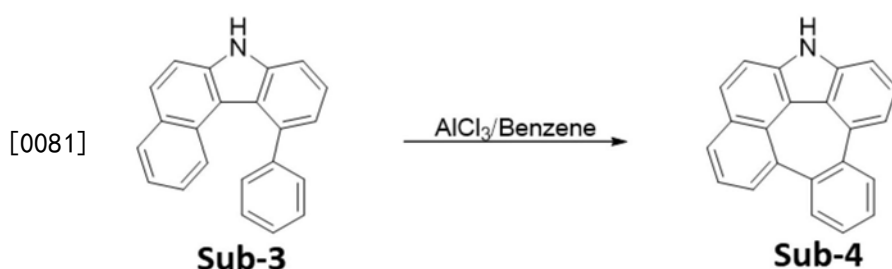
[0076] 在2000mL反应瓶中加入中间体Sub-1(50.0g, 132.6mmol), 然后使用干燥并脱气的DMF(1000mL)作为溶剂。氮气置换15分钟后, 加入催化剂醋酸钯(2mol%)和乙酸钾(89.3g, 397.8mmol)。加料结束后, 将温度升至160℃反应14h。在反应结束后, 冷却至室温, 加水搅拌1小时, 固体生成。泵抽过滤, 然后用少量乙醇洗涤, 干燥。用甲苯和乙醇重结晶, 得到中间体Sub-2(34.6g, yield88%)。LC-MS:M/Z297.2 (M+H)<sup>+</sup>。

[0077] 3. 中间体Sub-3的合成:



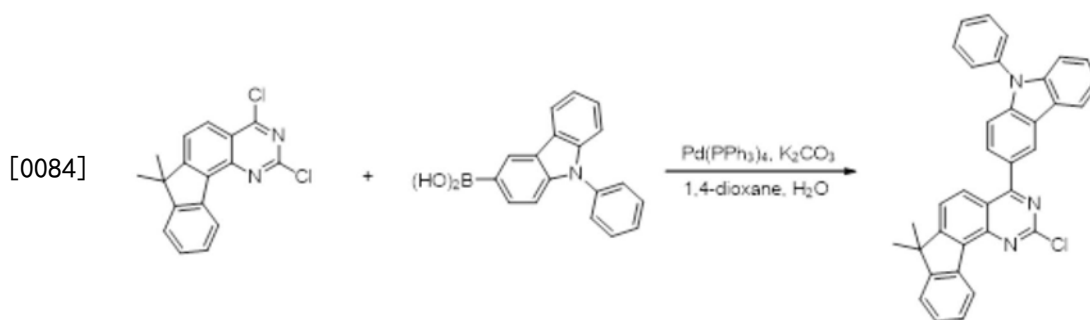
[0079] 在1000ml反应瓶中加入中间体Sub-2 (30.0g, 101.3mmol), 苯硼酸 (14.8g, 121.6mmol), 四(三苯基膦) 钯 (5mol%), 2M-K<sub>2</sub>CO<sub>3</sub> (150mL), 甲苯 (300mL) 和乙醇 (150mL)。反应体系升温至120℃, 氮气保护下反应12小时。反应完成后, 将反应液冷却至室温, 用邻二氯苯和水萃取。有机层用无水硫酸镁干燥, 浓缩, 重结晶所得粗品过硅胶柱得到中间体Sub-3 (23.2g, yield 78%)。LC-MS: M/Z 294.4 (M+H)<sup>+</sup>。

[0080] 4. 中间体Sub-4的合成:



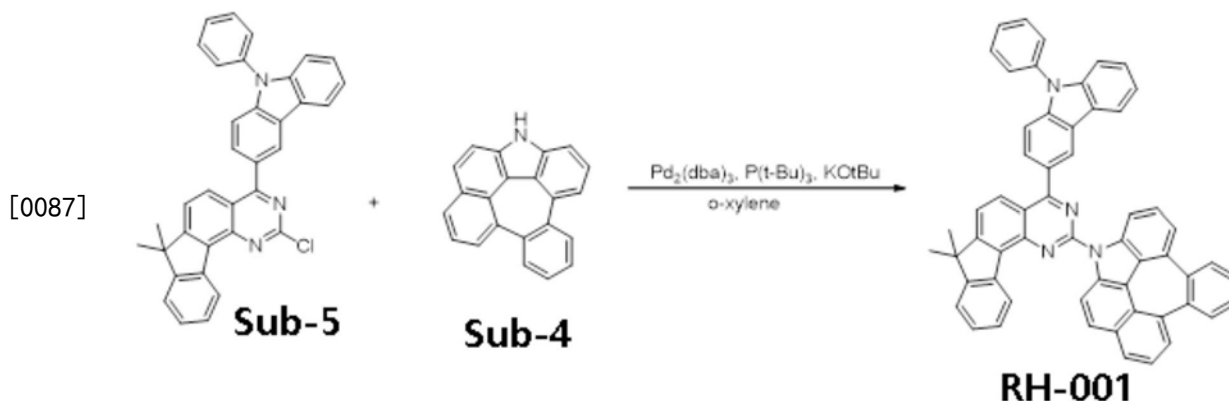
[0082] 在1000ml反应瓶中加入中间体Sub-3 (25.0g, 85.2mmol), NaCl (74.7g, 1278.3mmol) 和AlCl<sub>3</sub> (340.9g, 2556.6mmol) 溶解到苯里, 水解后使用水和碳酸氢钠水溶液洗涤, 使用硫酸镁干燥, 浓缩。然后将浓缩液溶于少量的热对二甲苯中过柱, 残留液使用甲基环己烷重结晶, 得到中间体4。得到中间体Sub-4 (7.4g, yield 30%)。LC-MS: M/Z 292.4 (M+H)<sup>+</sup>。

[0083] 5. 中间体Sub-5的合成:



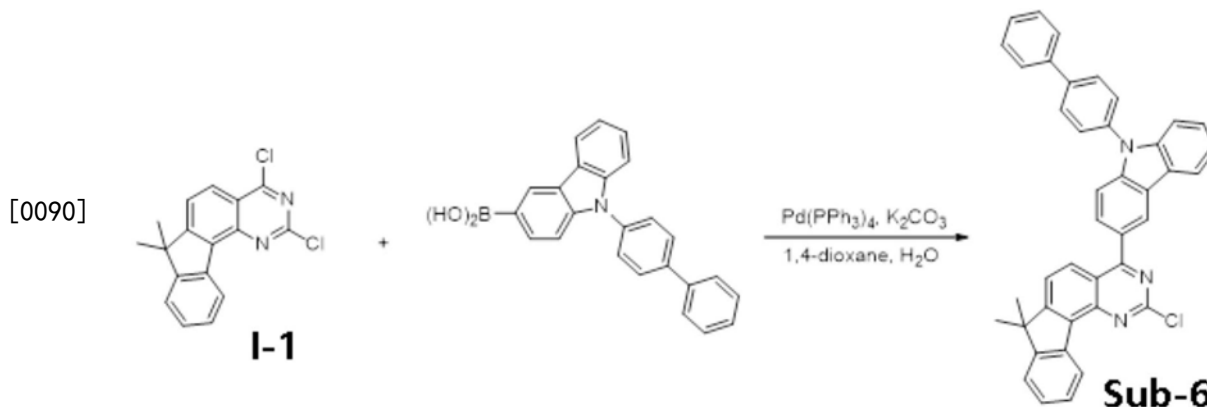
[0085] 在500ml反应瓶中加入中间体I-1 (10.0g, 31.7mmol), (9-苯基-9H-吡啶-3-基) 硼酸 (9.1g, 31.7mmol), 碳酸钾 (11.0g, 79.3mmol), 四三苯基膦钯 (5mol%), 1,4-二氧六环 (140mL) 和水 (70mL)。反应体系升温至60℃, 氮气保护下反应十小时。将反应液倒入450mL甲醇中, 过滤析出的固体。用氯苯溶解析出的固体, 用装有硅藻土和硅胶粉的漏斗过滤。过滤得到的橙色液体浓缩蒸干并用甲醇重结晶得到中间体Sub-5 (10.8g, yield 65%)。LC-MS: M/Z 523.0 (M+H)<sup>+</sup>。

[0086] 6. RH-001的合成:



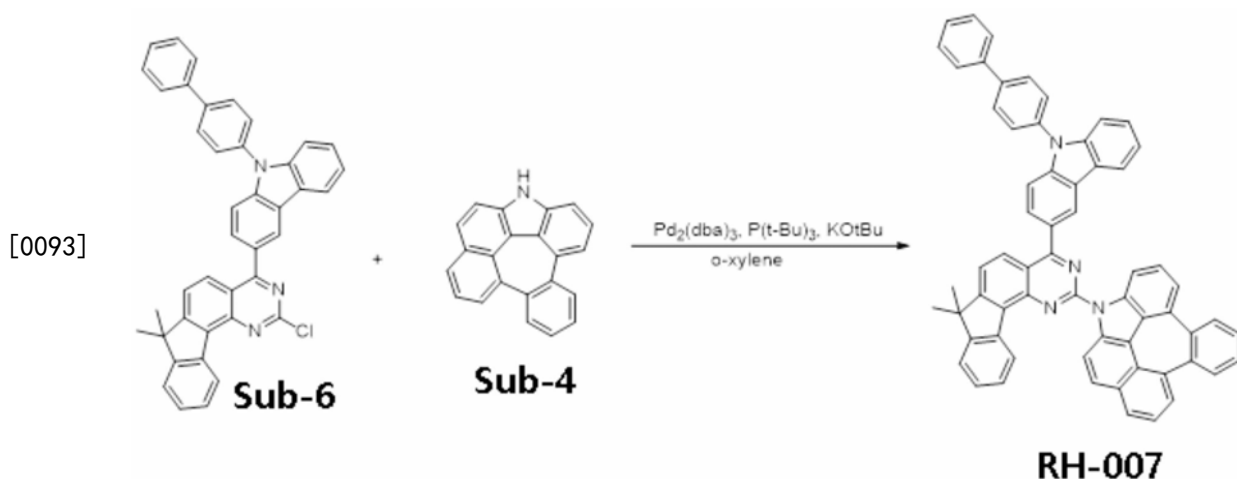
[0088] 在250ml三口烧瓶中加入中间体Sub-5 (5g, 9.6mmol), 中间体Sub-4 (3.1g, 10.5mmol), 三(二亚苄基丙酮)二钯 (4mol%), 三叔丁基膦 (8mol%), 叔丁醇钾 (3.2g, 28.7mmol) 和邻二甲苯 (80mL)。反应体系升温至120℃, 氮气保护下反应12小时。反应完成后, 将反应液冷却至室温, 用邻二氯苯和水萃取。有机层用无水硫酸镁干燥, 浓缩, 重结晶所得粗品过硅胶柱得到RH-001 (5.6, yield 75%)。LC-MS: M/Z 777.9 (M+H)+。

[0089] 7. 中间体Sub-6的合成:



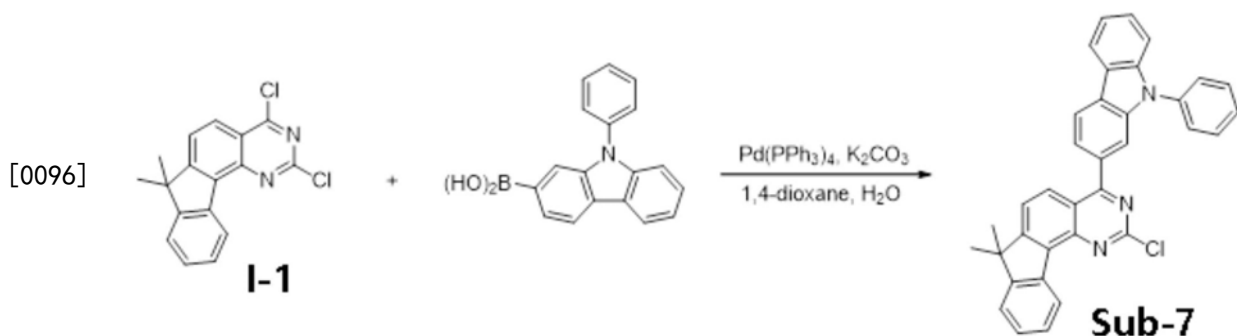
[0091] 在500ml反应瓶中加入中间体I-1 (10.0g, 31.7mmol), 9-(4-联苯基)-3-硼酸喹啉 (11.5g, 31.7mmol), 碳酸钾 (11.0g, 79.3mmol), 四三苯基膦钯 (5mol%), 1,4-二氧六环 (140mL) 和水 (70mL)。反应体系升温至60℃, 氮气保护下反应十小时。将反应液倒入450mL甲醇中, 过滤析出的固体。用氯苯溶解析出的固体, 用装有硅藻土和硅胶粉的漏斗过滤。过滤得到的橙色液体浓缩蒸干并用甲醇重结晶得到中间体Sub-6 (11.4g, yield 60%), LC-MS: M/Z 599.1 (M+H)+。

[0092] 8. RH-007的合成:



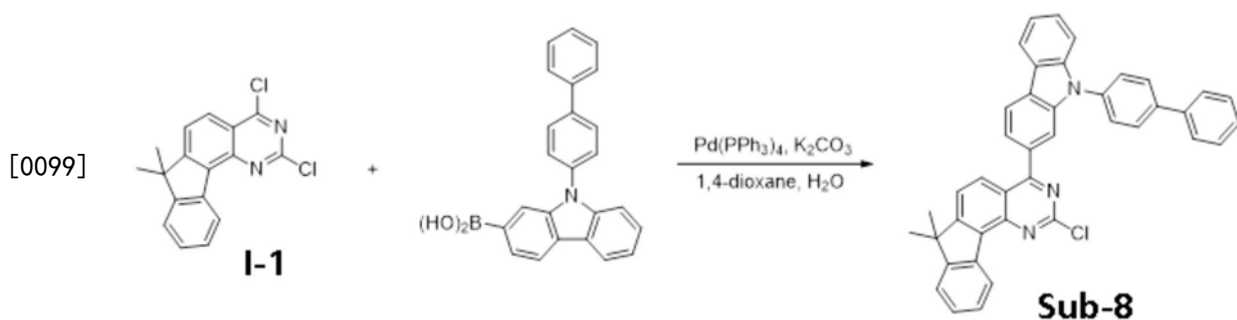
[0094] 在250mL三口烧瓶中加入中间体Sub-6 (5g, 8.4mmol), 中间体Sub-4 (2.7g, 9.2mmol), 三(二亚苄基丙酮)二钯 (4mol%), 三叔丁基膦 (8mol%), 叔丁醇钾 (2.8g, 25.1mmol) 和邻二甲苯 (80mL)。反应体系升温至120℃, 氮气保护下反应12小时。反应完成后, 将反应液冷却至室温, 用邻二氯苯和水萃取。有机层用无水硫酸镁干燥, 浓缩, 重结晶所得粗品过硅胶柱得到RH-007 (4.7g, yield 65%)。LC-MS: M/Z 854.0 (M+H)<sup>+</sup>。

[0095] 9. 中间体Sub-7的合成:



[0097] 在500ml反应瓶中加入中间体I-1 (10.0g, 31.7mmol), (9-苯基-9H-吡唑-2-基)硼酸 (9.1g, 31.7mmol), 碳酸钾 (11.0g, 79.3mmol), 四三苯基膦钯 (5mol%), 1, 4-二氧六环 (140mL) 和水 (70mL)。反应体系升温至60℃, 氮气保护下反应十小时。将反应液倒入450mL甲醇中, 过滤析出的固体。用氯苯溶解析出的固体, 用装有硅藻土和硅胶粉的漏斗过滤。过滤得到的橙色液体浓缩蒸干并用甲醇重结晶得到中间体Sub-7 (10.3g, yield 62%)。

[0098] 10. 中间体Sub-8的合成:



[0100] 在500ml反应瓶中加入中间体I-1 (10.0g, 31.7mmol), 9-(4-联苯基)-2-硼酸吡唑 (11.5g, 31.7mmol), 碳酸钾 (11.0g, 79.3mmol), 四三苯基膦钯 (5mol%), 1,4-二氧六环

(140mL) 和水 (70mL)。反应体系升温至60℃,氮气保护下反应十小时。将反应液倒入450mL 甲醇中,过滤析出的固体。用氯苯溶解析出的固体,用装有硅藻土和硅胶粉的漏斗过滤。过滤得到的橙色液体浓缩蒸干并用甲醇重结晶得到中间体Sub-8(11.6g, yield61%),LC-MS:M/Z599.1 (M+H)<sup>+</sup>。

[0101] 实施方案

[0102] 1. 第一实施方案

[0103] 使ITO玻璃基板图案化,以具有3mm×3mm的发光区域。然后,洗涤图案化的ITO玻璃基板。随后将该基板安放在真空室。标准压力设定为 $1 \times 10^{-6}$ 托。此后,在ITO基板上以CuPc(200Å),NPB(400Å),RH-001+(btp) 2Ir(acac) (5%)(200Å),Alq3(300Å),LiF(5Å),和Al(1000Å)的顺序形成有机物质的层。

[0104] 在0.9mA下,亮度等于1088cd/m<sup>2</sup>(6.0V)。此时,CIE<sub>x</sub>=0.659,y=0.329。

[0105] 2. 第二实施方案

[0106] 使ITO玻璃基板图案化,以具有3mm×3mm的发光区域。然后,洗涤图案化的ITO玻璃基板。随后将该基板安放在真空室。标准压力设定为 $1 \times 10^{-6}$ 托。此后,在ITO基板上以CuPc(200Å),NPB(400Å),RH-007+(btp) 2Ir(acac) (5%)(200Å),Alq3(300Å),LiF(5Å),和Al(1000Å)的顺序形成有机物质的层。

[0107] 在0.9mA下,亮度等于1012cd/m<sup>2</sup>(6.2V)。此时,CIE<sub>x</sub>=0.659,y=0.330。

[0108] 3. 第三实施方案

[0109] 使ITO玻璃基板图案化,以具有3mm×3mm的发光区域。然后,洗涤图案化的ITO玻璃基板。随后将该基板安放在真空室。标准压力设定为 $1 \times 10^{-6}$ 托。此后,在ITO基板上以CuPc(200Å),NPB(400Å),RH-073+(btp) 2Ir(acac) (5%)(200Å),Alq3(300Å),LiF(5Å),和Al(1000Å)的顺序形成有机物质的层。

[0110] 在0.9mA下,亮度等于1101cd/m<sup>2</sup>(6.1V)。此时,CIE<sub>x</sub>=0.659,y=0.329。

[0111] 4. 第四实施方案

[0112] 使ITO玻璃基板图案化,以具有3mm×3mm的发光区域。然后,洗涤图案化的ITO玻璃基板。随后将该基板安放在真空室。标准压力设定为 $1 \times 10^{-6}$ 托。此后,在ITO基板上以CuPc(200Å),NPB(400Å),RH-079+(btp) 2Ir(acac) (5%)(200Å),Alq3(300Å),LiF(5Å),和Al(1000Å)的顺序形成有机物质的层。

[0113] 在0.9mA下,亮度等于1037cd/m<sup>2</sup>(6.3V)。此时,CIE<sub>x</sub>=0.659,y=0.330。

[0114] 5. 对比例

[0115] 使ITO玻璃基板图案化,以具有3mm×3mm的发光区域。然后,洗涤图案化的ITO玻璃基板。随后将该基板安放在真空室。标准压力设定为 $1 \times 10^{-6}$ 托。在ITO基板上以CuPc(200Å),NPB(400Å),CPB+(btp) 2Ir(acac) (5%)(200Å),Alq3(300Å),LiF(5Å),和Al(1000Å)的顺序形成有机物质的层。

[0116] 在0.9mA下,亮度等于780cd/m<sup>2</sup>(7.5V)。此时,CIE<sub>x</sub>=0.659,y=0.329。

[0117] 如图4所示显示有机电致发光器件的色纯度增加(即随色度坐标的X值变得更大)可见度降低。

[0118] 依据上述实施方案和对比例,效率,色度坐标,和亮度的特性显示在下表1中。

[0119] 表1

器件	电压 (V)	电流 (mA)	亮度 (cd/m <sup>2</sup> )	电流效率 (cd/A)	功率效率 (lm/W)	CIE (x)	CIE (y)
第一实施方案	6.0	0.9	1088	10.9	5.7	0.659	0.329
第二实施方案	6.2	0.9	1012	10.1	5.1	0.659	0.330
第三实施方案	6.1	0.9	1101	11.0	5.7	0.659	0.329
第四实施方案	6.3	0.9	1037	10.4	5.2	0.659	0.330
对比例	7.5	0.9	780	7.8	3.3	0.659	0.329

[0121] 如表1所示,甚至当色纯度高时,该器件在低电压下也高效率地运行。并且,与对比例相比,第二实施方案电流效率增加45%以上。

[0122] 同时,N在X2位上,性能更好,相同条件下,寿命延长一倍。

[0123] 以上显示和描述了本发明的基本原理、主要特征和本发明的优点。本行业的技术人员应该了解,本发明不受上述实施例的限制,上述实施例和说明书中描述的仅为本发明的优选例,并不用来限制本发明,在不脱离本发明精神和范围的前提下,本发明还会有各种变化和改进行,这些变化和改进行都落入要求保护的本发明范围内。本发明要求保护范围由所附的权利要求书及其等效物界定。

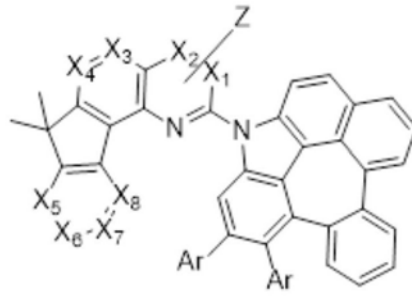


图1

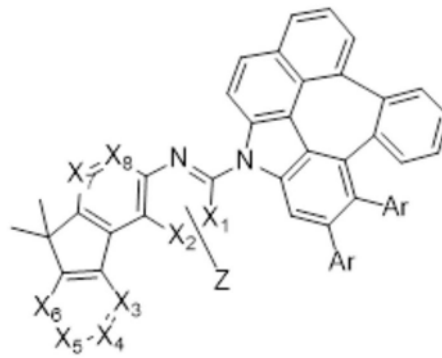


图2

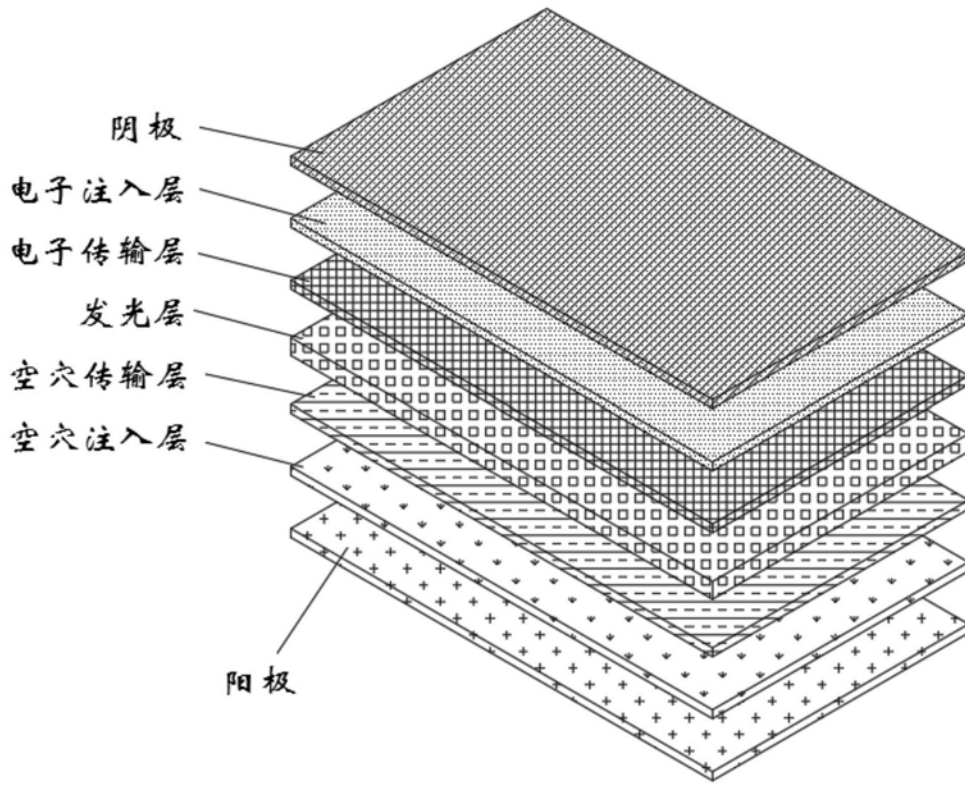


图3

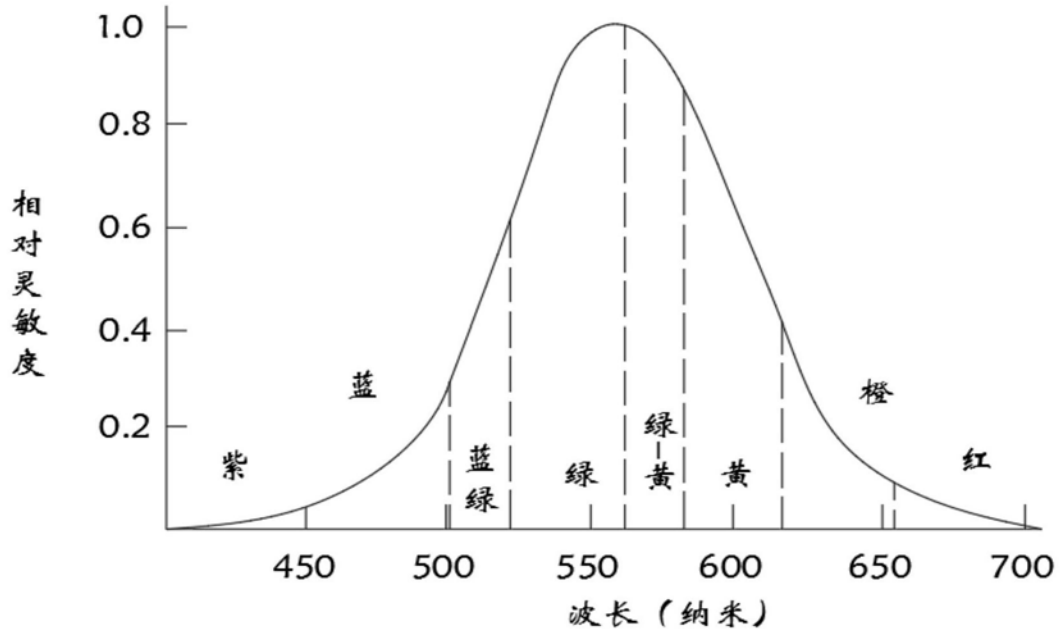


图4

专利名称(译)	一种磷光化合物以及使用所述化合物的有机电致发光器件		
公开(公告)号	<a href="#">CN110951482A</a>	公开(公告)日	2020-04-03
申请号	CN201911010625.5	申请日	2019-10-23
[标]申请(专利权)人(译)	宇瑞(上海)化学有限公司		
申请(专利权)人(译)	宇瑞(上海)化学有限公司		
当前申请(专利权)人(译)	宇瑞(上海)化学有限公司		
[标]发明人	郑贤哲 王世超 华万鸣		
发明人	郑贤哲 王世超 吴信蔚 华万鸣		
IPC分类号	C09K11/06 C07D403/14 C07D401/14 H01L51/50 H01L51/54		
CPC分类号	C07D401/14 C07D403/14 C09K11/06 C09K2211/1007 C09K2211/1011 C09K2211/1029 C09K2211/1044 C09K2211/1059 H01L51/0052 H01L51/0067 H01L51/0072 H01L51/5012		
代理人(译)	钱磊		
外部链接	<a href="#">Espacenet</a> <a href="#">SIPO</a>		

摘要(译)

本发明涉及有机电致发光技术领域，具体地说，涉及一种磷光化合物以及使用所述化合物的有机电致发光器件。其结构式如式1和式2所示：上述的结构式1和结构式2中，Z独立地选自以下结构：其中X1至X2独立的选自N原子或C原子；其中X1至X2中的至少一个为N；其中Ar独立地选自、C6-C30芳基、C2-C30杂芳基中。该发明的有机电致发光器件随着色纯度增加，可见度降低，甚至当色纯度高时，在低电压下也高效率地运行，并且电流效率增加，在高内量子效率下，发光效率高，同时采用磷光化合物作为有机电致发光器件的发光材料，制造过程简单，工艺效率高，生产成本低。

