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(71) 申請人：新日鐵住金化學股份有限公司 (日本) NIPPON STEEL &amp; SUMIKIN CHEMICAL CO., LTD. (JP)

日本

(72) 發明人：三宮瑠美 SANNOMIYA, RUMI (JP)；甲斐孝弘 KAI, TAKAHIRO (JP)；古森正樹 KOMORI, MASAKI (JP)；山本敏浩 YAMAMOTO, TOSHIHIRO (JP)

(74) 代理人：詹銘文；葉璟宗

(56) 參考文獻：

TW 201107447A1

審查人員：謝欣秀

申請專利範圍項數：5 項 圖式數：3 共 148 頁

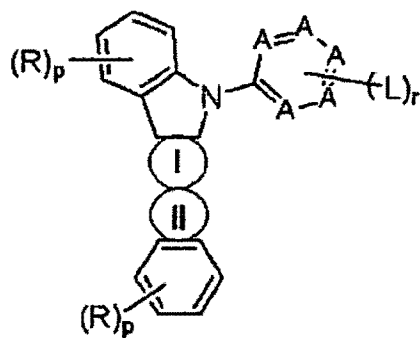
(54) 名稱

有機電場發光元件

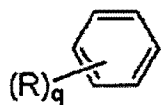
ORGANIC ELECTROLUMINESCENT ELEMENT

(57) 摘要

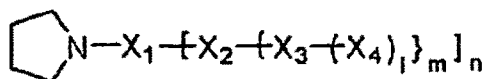
本發明提供一種使用吲哚并咔唑化合物的有機電場發光元件(EL 元件)。該有機 EL 元件是於基板上積層有陽極、包含磷光發光層的多層有機層及陰極而成，且於磷光發光層、電洞傳輸層、電子傳輸層、電洞阻擋層或電子阻擋層中，含有通式(1)所表示的吲哚并咔唑化合物。



(1)



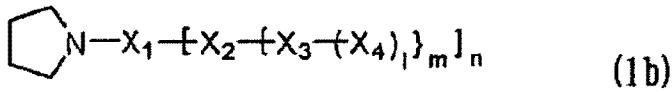
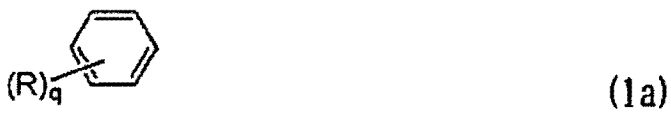
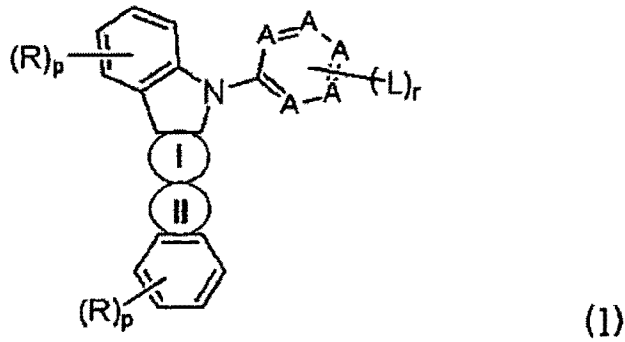
(1a)



(1b)

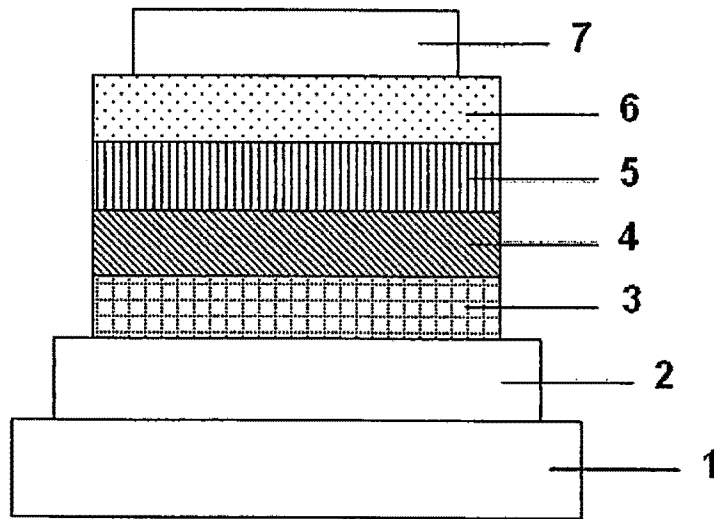
An organic electroluminescent element (EL element) using an indolocarbazole compound is provided. The organic EL element is made by laminating an anode, a plurality of organic layers containing a

phosphorescent light-emitting layer, and a cathode on a substrate. An indolocarbazole compound represented by the general formula (1) is included in the phosphorescent light-emitting layer, a hole transport layer, an electron transport layer, a hole blocking layer or an electron blocking layer.



指定代表圖：

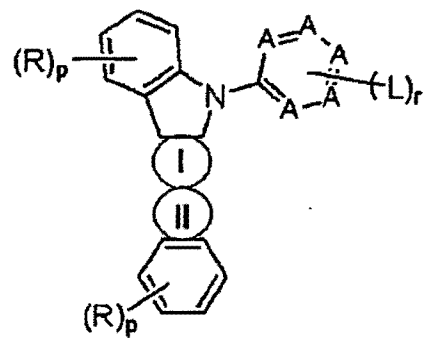
符號簡單說明：



- 1 . . . 基板
- 2 . . . 陽極
- 3 . . . 電洞注入層
- 4 . . . 電洞傳輸層
- 5 . . . 發光層
- 6 . . . 電子傳輸層
- 7 . . . 陰極

圖 1

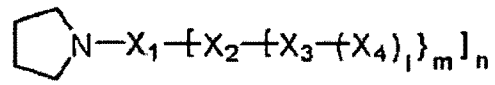
特徵化學式：



(1)



(1a)



(1b)

公告本
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## 發明摘要

※ 申請案號：102108503

※ 申請日：102.3.11

※ IPC 分類：

C09K 11/06 (2006.01)

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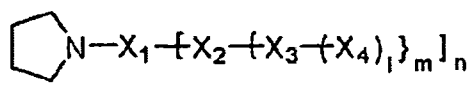
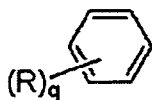
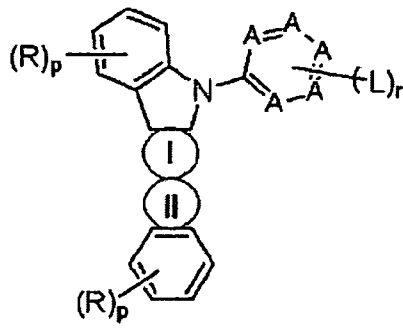
## 【發明名稱】

有機電場發光元件

ORGANIC ELECTROLUMINESCENT ELEMENT

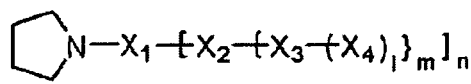
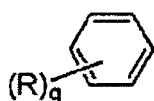
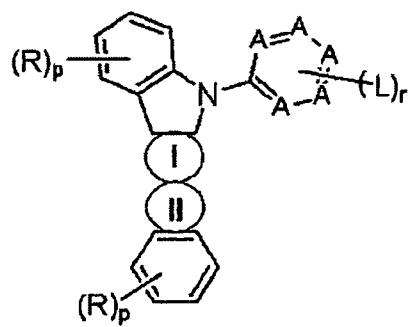
## ● 【中文】

本發明提供一種使用吲哚并咔唑化合物的有機電場發光元件 (EL 元件)。該有機 EL 元件是於基板上積層有陽極、包含磷光發光層的多層有機層及陰極而成，且於磷光發光層、電洞傳輸層、電子傳輸層、電洞阻擋層或電子阻擋層中，含有通式 (1) 所表示的吲哚并咔唑化合物。



## 【英文】

An organic electroluminescent element (EL element) using an indolocarbazole compound is provided. The organic EL element is made by laminating an anode, a plurality of organic layers containing a phosphorescent light-emitting layer, and a cathode on a substrate. An indolocarbazole compound represented by the general formula (1) is included in the phosphorescent light-emitting layer, a hole transport layer, an electron transport layer, a hole blocking layer or an electron blocking layer.



## 【代表圖】

【本案指定代表圖】：圖 1。

【本代表圖之符號簡單說明】：

1：基板

2：陽極

3：電洞注入層

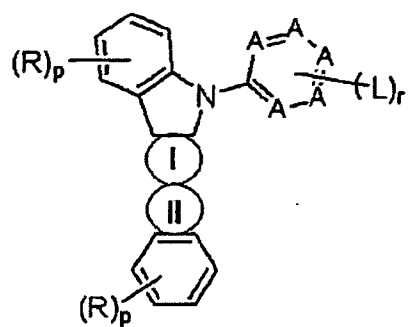
4：電洞傳輸層

5：發光層

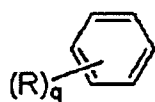
6：電子傳輸層

7：陰極

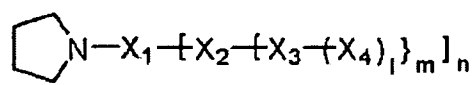
【本案若有化學式時，請揭示最能顯示發明特徵的化學式】：



(1)



(1a)



(1b)

# 發明專利說明書

(本說明書格式、順序，請勿任意更動)

## 【發明名稱】

有機電場發光元件

ORGANIC ELECTROLUMINESCENT ELEMENT

## 【技術領域】

【0001】 本發明是有關於一種含有吡啶并咪唑化合物的有機電場發光元件，詳細而言，本發明是有關於一種對包含有機化合物的發光層施加電場而放出光的薄膜型元件 (device)。

## 【先前技術】

【0002】 通常，對於有機電場發光元件 (以下稱為有機 EL (Electroluminescence) 元件) 而言，其最簡單的結構包含發光層及夾著該層的一對相向電極。即，有機 EL 元件中利用以下現象：若於兩電極間施加電場，則自陰極注入電子，自陽極注入電洞，該等在發光層中再結合而放出光。

【0003】 近年來，正在進行使用有機薄膜的有機 EL 元件的開發。尤其爲了提高發光效率，藉由開發出以自電極注入載子的效率的提高爲目的而進行電極種類的最適化、且於電極間以薄膜的形式設有包含芳香族二胺的電洞傳輸層及包含 8-羥基喹啉鋁錯合物 (以下稱為 Alq3) 的發光層的元件，與先前的使用蒽 (anthracene) 等單晶的元件相比較，大幅度地改善了發光效率，故以使具有自

發光、高速響應性等特徵的高性能平板（flat panel）實用化為目的而推進上述元件。

【0004】另外，作為提高元件的發光效率的嘗試，亦正在研究使用磷光而非螢光。以上述設有包含芳香族二胺的電洞傳輸層及包含 Alq3 的發光層的元件為代表的大多數元件利用螢光發光，而藉由使用磷光發光（即利用自三重態激發狀態的發光），與先前的使用螢光（單重態）的元件相比較，可期待 3 倍～4 倍左右的效率提高。出於該目的，正在研究將香豆素衍生物或二苯甲酮衍生物製成發光層，但僅可獲得極低的亮度。另外，作為利用三重態狀態的嘗試，正在研究使用銻錯合物，但其亦未達成高效率的發光。近年來，如專利文獻 1 所列舉般，以發光的高效率化或長壽命化為目的，而以銻錯合物等有機金屬錯合物為中心進行了大量的磷光發光摻雜材料的研究。

【0005】 [先前技術文獻]

[專利文獻 1]WO01/041512 號公報

[專利文獻 2]日本專利特開 2001-313178 號公報

[專利文獻 3]日本專利特開平 11-162650 號公報

[專利文獻 4]日本專利特開平 11-176578 號公報

[專利文獻 5]WO2008/056746 號公報

[專利文獻 6]WO2008/146839 號公報

[專利文獻 7]WO2010/113726 號公報

[專利文獻 8]WO2010/113755 號公報

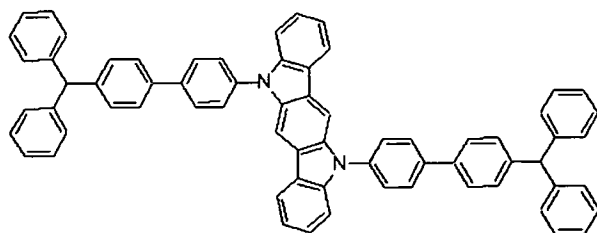


【0006】 爲了獲得高發光效率，與上述摻雜材料同時使用的主體材料變重要。作爲主體材料而提出的具代表性者可列舉專利文獻 2 中介紹的咪唑化合物的 4,4'-雙(9-咪唑基)聯苯（以下稱爲 CBP）。CBP 於用作三(2-苯基吡啶)銦錯合物（以下稱爲 Ir(ppy)<sub>3</sub>）所代表的綠色磷光發光材料的主體材料的情形時，有容易流通電洞而不易流通電子的特性，因而電荷注入平衡被破壞，過剩的電洞於電子傳輸層側流出，結果自 Ir(ppy)<sub>3</sub> 的發光效率降低。

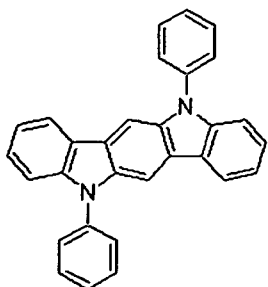
【0007】 如上所述，爲了於有機 EL 元件中獲得高發光效率，需要具有高的三重態激發能量、且兩電荷（電洞、電子）注入傳輸特性取得了平衡的主體材料。進而，期望電化學穩定、且具有高耐熱性並且具有優異的非晶穩定性的化合物，需求進一步的改良。

【0008】 於專利文獻 3 中，揭示有如下所示的吡啶并咪唑化合物作爲電洞傳輸材料。

【0009】

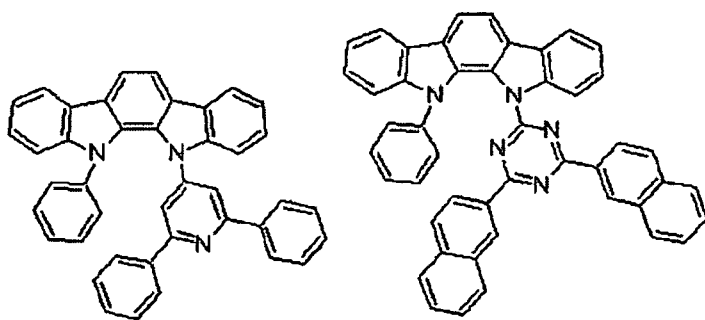


【0010】 於專利文獻 4 中，揭示有如下所示的吡啶并咪唑化合物作爲電洞傳輸材料。



【0011】 然而，該些文獻雖然推薦將具有吲哚并咔唑骨架的化合物用作電洞傳輸材料，但並未揭示於吲哚并咔唑骨架的 N 上取代基上取代有含氮 6 員環的化合物或其有用性。

【0012】 專利文獻 5 及專利文獻 6 中揭示有如下所示的吲哚并咔唑化合物作為磷光主體材料，且揭示使用該吲哚并咔唑化合物的有機 EL 元件改善了發光效率，變得具有高的驅動穩定性。



【0013】 另外，專利文獻 7 及專利文獻 8 中揭示有於吲哚并咔唑的 N 位上具有含氮 6 員環、且於該含氮 6 員環上具有縮合雜環作為取代基的化合物。

### 【發明內容】

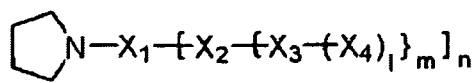
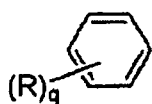
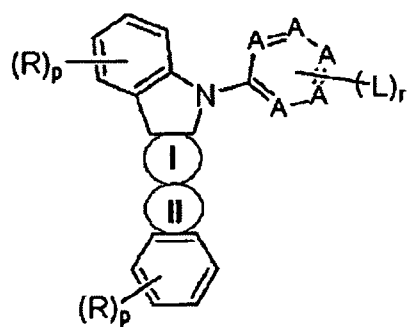
【0014】 為了將有機 EL 元件應用於平板顯示器等顯示元件，必須在改善元件的發光效率的同時充分確保驅動時的穩定性。鑒於上

述現狀，本發明的目的在於提供一種高效率且具有高的驅動穩定性的於實用上有用的有機 EL 元件及適於該有機 EL 元件的化合物。

【0015】本發明者等人進行了潛心研究，結果發現，藉由將具有特定結構的吡啶并咪唑骨架的化合物用作有機 EL 元件，而顯示優異的特性，從而完成了本發明。

【0016】本發明是有關於一種有機電場發光元件，其是於基板上積層有陽極、包含磷光發光層的多層有機層及陰極而成，該有機電場發光元件的特徵在於：於選自由磷光發光層、電洞傳輸層、電子傳輸層、電洞阻擋層及電子阻擋層所組成的群組中的至少一層有機層中，含有通式（1）所表示的吡啶并咪唑化合物。

【0017】

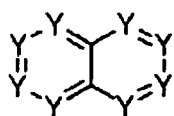


【0018】通式（1）中，環 I 表示於任意的位置與鄰接環縮合的式

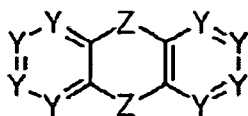
(1a) 所表示的芳香族烴環，環 II 表示於任意的位置與鄰接環縮合的式 (1b) 所表示的雜環。A 表示 C-R 或 N，但至少一個為 N。L 獨立地表示經取代或未經取代的碳數 6~18 的芳香族烴基或碳數 3~17 的芳香族雜環基，r 表示 1~4 的整數。其中，L 的至少一個為由 2 環~4 環的環所形成的縮合雜環基。通式 (1) 及式 (1a) 中，R 分別獨立地表示碳數 1~10 的脂肪族烴基、碳數 6~18 的芳香族烴基或碳數 3~17 的芳香族雜環基，p 分別獨立地表示 0~4 的整數，q 表示 0~2 的整數。式 (1b) 中，X<sub>1</sub>~X<sub>4</sub> 分別獨立地表示碳數 6~18 的芳香族烴基或碳數 3~17 的芳香族雜環基，l、m 及 n 分別獨立地表示 0~5 的整數。此處，於 l、m 或 n 為 2 以上的情形時，X<sub>2</sub>、X<sub>3</sub> 及 X<sub>4</sub> 分別可相同亦可不同。

【0019】於通式 (1) 中，較佳為至少一個 L 為下述式 (2) 或式 (3) 的任一個所表示的一價芳香族雜環基，更佳為下述式 (4) 所表示的化合物所表示的一價芳香族雜環基。

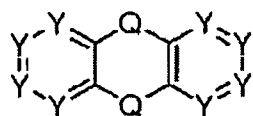
【0020】



(2)



(3)



(4)

(式(2)~式(4)中，Y分別獨立地表示次甲基、取代次甲基或氮，Y的任一個為形成一價基的碳原子。Z獨立地表示單鍵、-S-、-O-或-N(Ar)-的任一個，Q表示單鍵、-S-、或-O-的任一個，於式(2)中，Y的至少一個為雜原子，於式(3)中，Y或Z的至少一個為雜原子，於式(4)中，Y或Q的至少一個為雜原子。此處，Ar表示碳數6~18的芳香族烴基或碳數6~17的芳香族雜環基)

【0021】而且，較佳為式(3)~式(4)中的Z及Q的一個為單鍵，另一個為-S-或-O-。

【0022】於通式(1)的L中，更佳為至少一個L為取代或未取代的二苯并呋喃基或二苯并噻吩基。

【0023】式(1b)中，X<sub>1</sub>~X<sub>4</sub>的總數的較佳範圍為2~10，更佳為4~7。

【0024】另外，本發明的其他實施方式為上述有機電場發光元件，其中含有吡啶并咪唑化合物的有機層為含有磷光發光摻雜劑的發光層。

### 【圖式簡單說明】

### 【0025】

圖1為表示有機EL元件的一結構例的剖面圖。

圖 2 表示吡啶并咪唑化合物 (A-81) 的  $^1\text{H}$ -核磁共振 (Nuclear Magnetic Resonance, NMR) 譜圖。

圖 3 表示吡啶并咪唑化合物 (C-90) 的  $^1\text{H}$ -NMR 譜圖。

### 【實施方式】

【0026】 本發明的有機電場發光元件於至少一層有機層中含有上述通式 (1) 所表示的吡啶并咪唑化合物 (以下亦稱為通式 (1) 所表示的化合物或吡啶并咪唑化合物)。

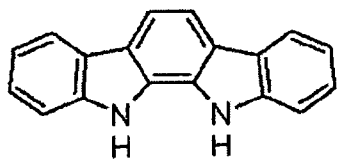
【0027】 於通式 (1) 中，環 I 表示於任意的位置與鄰接環縮合的式 (1a) 所表示的芳香族烴環，環 II 表示於任意的位置與鄰接環縮合的式 (1b) 所表示的雜環。

【0028】 於通式 (1) 所表示的吡啶并咪唑骨架中，式 (1a) 所表示的芳香族烴環可於任意的位置與 2 個鄰接環縮合，但有結構上無法縮合的位置。式 (1a) 所表示的芳香族烴環具有 6 條邊，不可於鄰接的 2 條邊上與 2 個鄰接環縮合。另外，式 (1b) 所表示的雜環可於任意的位置與 2 個鄰接環縮合，但有結構上無法縮合的位置。即，式 (1b) 所表示的雜環具有 5 條邊，不可於鄰接的 2 條邊上與 2 個鄰接環縮合，另外，不可於含有氮原子的邊上與鄰接環縮合。因此，吡啶并咪唑骨架的種類受限。具體而言，吡啶并咪唑骨架可於咪唑骨架的 2,3-位、3,4-位、4,5-位上縮合，存在 5 種骨架的異構物。

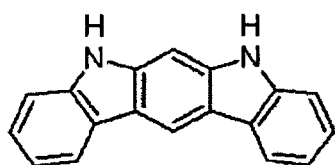
【0029】 於通式 (1) 中，吡啶并咪唑骨架較佳為以下形態所表示者。根據該例可理解吡啶并咪唑骨架中的芳香族烴環及雜環的較

佳縮合位置。

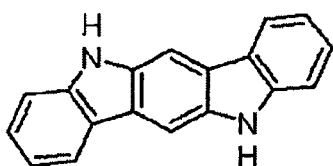
【0030】



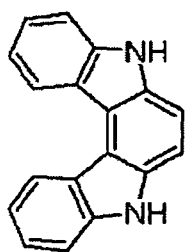
(IC-1)



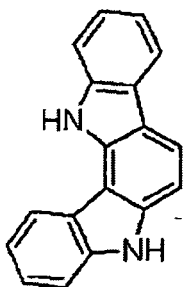
(IC-2)



(IC-3)



(IC-4)



(IC-5)

【0031】 通式 (1) 中，A 表示 C-R 或 N。此處，A 的至少一個為 N。較佳為 1 個～3 個為 N。

【0032】 通式 (1) 中，L 獨立地表示經取代或未經取代的碳數 6

~18 的芳香族烴基或碳數 3~17 的芳香族雜環基，但至少一個為 2 環~4 環的縮合雜環基。

【0033】 L 為未經取代的碳數 6~18 的芳香族烴基或碳數 3~17 的芳香族雜環基的情形的具體例可列舉自以下芳香環中去掉氫而生成的基團：苯、并環戊二烯 (pentalene)、茛、萘、蔥、菲、吡咯、咪唑、吡唑、噻唑、噻吩、吡啶、吡嗪、嘧啶、噻嗪、三嗪、異吲哚 (isoindole)、吲唑 (indazole)、嘌呤、苯并咪唑、吲哚嗪、苯并吡喃 (chromene)、苯并噁唑、異苯并呋喃、喹嗪、異喹啉、咪唑、萘啶、酞嗪、喹啉、喹噁啉、肉啉 (cinnoline)、喹啉、喋啶、萘嵌間二氮雜苯 (perimidine)、啡啉、啡啶 (phenanthridine)、吡啶、吩嗪、吩噻嗪 (phenothiazine)、吩噁嗪 (phenoxazine)、環戊基硼酸 (phenazasiline)、二苯并二噁英 (dibenzodioxin)、卡啉 (carboline)、吲哚、吲哚并吲哚、卡唑、呋喃、苯并呋喃、異苯并呋喃、苯并噻唑、噁蔥、二苯并呋喃、噻吩、噻嘧、噻蔥 (thianthrene)、吩噁噻 (phenoxathiin)、噻茛、異硫茛 (isothianaphthene)、并噻吩 (thiophthene)、萘并噻吩 (thiophanthrene) 或二苯并噻吩等。

【0034】 於 L 為由 2 環~4 環的環所形成的縮合雜環基的情形時，其具體例可列舉自上述 L 的說明中所示的芳香環中的縮合雜環中 去掉氫而生成的基團。更佳例為上述式 (2) ~ 式 (4) 的任一個所表示的一價芳香族雜環基。式 (2) ~ 式 (4) 中，Y 分別獨立地表示次甲基、取代次甲基或氮，Y 的任一個為碳原子 (C)，該



碳原子形成可成爲一價基的自由基。Z 表示單鍵、-S-、-O-或-N(Ar)-的任一個，Q 表示單鍵、-S-、-O-的任一個，式(2)~式(4)分別爲含有至少一個雜原子的縮合雜環基。此處，Ar 表示碳數 6~18 的芳香族烴基或碳數 6~17 的芳香族雜環基。再者，L 爲經取代的碳數 6~18 的芳香族烴基或碳數 3~17 的芳香族雜環基的情形、Y 爲取代次甲基的情形時的取代基較佳爲碳數 1~10 的烷基、碳數 3~11 的環烷基、碳數 6~18 的芳香族烴基或碳數 3~17 的芳香族雜環基。更佳爲碳數 1~4 的烷基、碳數 3~7 的環烷基、碳數 6~12 的芳香族烴基或碳數 3~12 的芳香族雜環基。較佳具體例可列舉：甲基、乙基、丙基等碳數 1~6 的烷基，環己基、甲基環己基等碳數 6~8 的環烷基，苯基、萘基等碳數 6~12 的芳香族烴基，吡啶基、嘧啶基、三嗪基、呋唑基、二苯并呋喃基、二苯并噻吩基等碳數 3~12 的芳香族雜環基，更佳爲苯基、吡啶基、嘧啶基、三嗪基、呋唑基、二苯并呋喃基、二苯并噻吩基。通式(1)中，r 表示 1~4 的整數，較佳爲 1~3 的整數，更佳爲 1~2 的整數。

**【0035】** 於通式(2)~通式(4)所表示的一價芳香族雜環基中，特佳爲經取代或未經取代的二苯并噻吩基或二苯并呋喃基的任一個。此處，二苯并噻吩基或二苯并呋喃基具有取代基的情形時的取代基與上述 Y 爲取代次甲基的情形時說明的取代基相同。

**【0036】** 通式(1)及式(1a)中，R 分別獨立地表示碳數 1~10 的烷基、碳數 3~11 的環烷基、碳數 6~18 的芳香族烴基或碳數 3

~17 的芳香族雜環基。較佳為碳數 1~4 的烷基、碳數 3~7 的環烷基、碳數 6~12 的芳香族烴基或碳數 3~12 的芳香族雜環基，較佳具體例可列舉：甲基、乙基、丙基、環己基、甲基環己基、苯基、萘基、吡啶基、嘧啶基、三嗪基、呋啞基，更佳為甲基、乙基、苯基、吡啶基。於 R 為芳香族烴基或芳香族雜環基的情形時，亦可具有取代基，取代基可列舉：碳數 1~10 的烷基、碳數 3~11 的環烷基、碳數 1~2 的烷氧基、乙醯基、碳數 6~18 的二級胺基、碳數 6~18 的二級磷基 (phosphanyl)、碳數 3~18 的矽烷基、碳數 6~18 的芳香族烴基、碳數 3~17 的芳香族雜環基。較佳為碳數 1~4 的烷基、碳數 3~7 的環烷基、碳數 6~12 的芳香族烴基、碳數 3~12 的芳香族雜環基，較佳具體例可列舉：甲基、乙基、丙基、環己基、甲基環己基、苯基、萘基、吡啶基、嘧啶基、三嗪基、呋啞基，更佳為甲基、乙基、苯基、吡啶基。

【0037】於通式 (1) 及式 (1a) 中，p 獨立地表示 0~4 的整數，q 表示 0~2 的整數。較佳為 p 及 q 為 0 或 1，R 的合計個數較佳為 0~3 的範圍。

【0038】式 (1b) 中， $X_1 \sim X_4$  表示碳數 6~18 的芳香族烴基或碳數 3~17 的芳香族雜環基。該些芳香族烴基或芳香族雜環基可為單環，亦可為縮合環，但不為芳香族基連結成鏈狀的基團。

【0039】此處， $X_2$  表示連結於  $X_1$  的基團， $X_3$  表示連結於  $X_2$  的基團， $X_4$  表示連結於  $X_3$  的基團， $X_4$  為一價基，但  $X_1 \sim X_3$  為根據 n、m、l 的個數所決定的一價以上的基團。 $X_1 \sim X_4$  為碳數 6~18 的芳

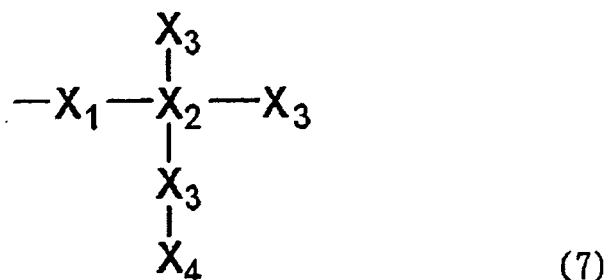
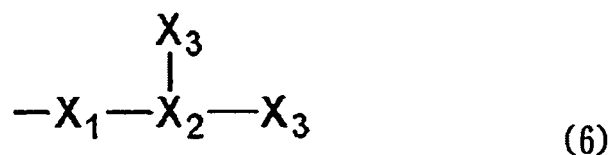
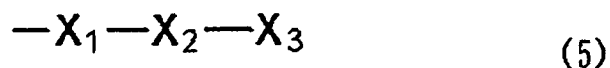
香族烴基或碳數 3~17 的芳香族雜環基的情形時的具體例可列舉：上文中作為 L 為碳數 6~18 的芳香族烴基或碳數 3~17 的芳香族雜環基的情形時的具體例而例示的自芳香環中去掉既定個數的氫而成的一價以上的基團。

【0040】式 (1b) 中， $l$ 、 $m$ 、 $n$  分別表示 0~5 的整數，較佳為 0~3 的整數。

【0041】另外，式 (1b) 中， $X_1 \sim X_4$  的總數以 2~10 為佳，較佳為 4~7，更佳為 5~7。於  $l$ 、 $m$  或  $n$  為 2 以上的情形時， $X_2$ 、 $X_3$  及  $X_4$  分別可相同亦可不同。 $X_1 \sim X_4$  的總數是以  $1+n+mn+mnl=1+n(1+m+ml)$  來計算。因此，為了使  $X_1 \sim X_4$  的總數為 2~10， $n$  成為 1 以上的整數；為了使總數為 5~7，當  $n$  為 1 時 ( $m+ml$ ) 成為 3~5，當  $n$  為 2 時 ( $m+ml$ ) 成為 1~2。

【0042】於  $-X_1-[X_2-\{X_3-(X_4)_l\}_m]_n$  中， $X_1 \sim X_4$  的總數為多數的情形時的例子可列舉下述式。

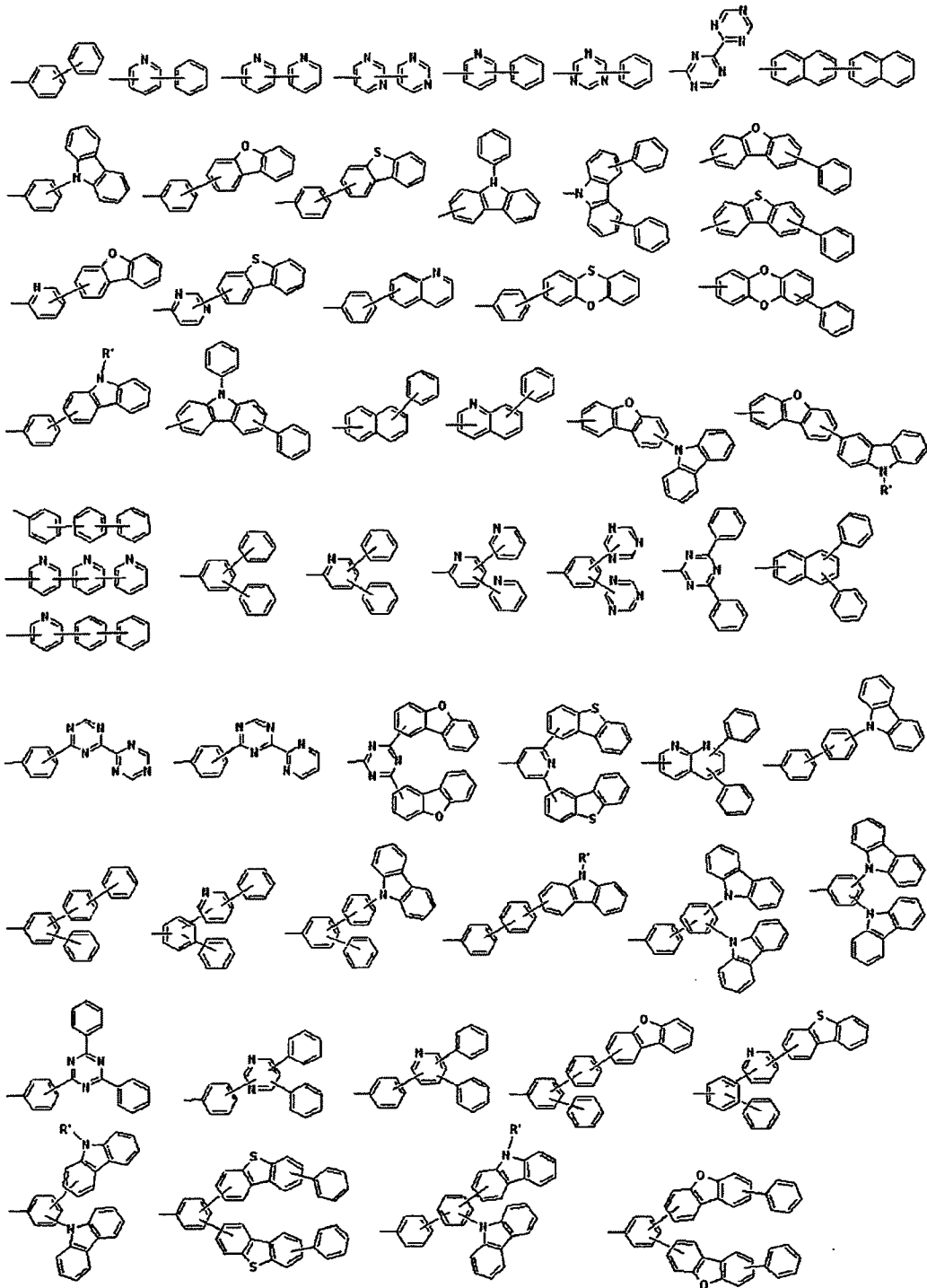
【0043】

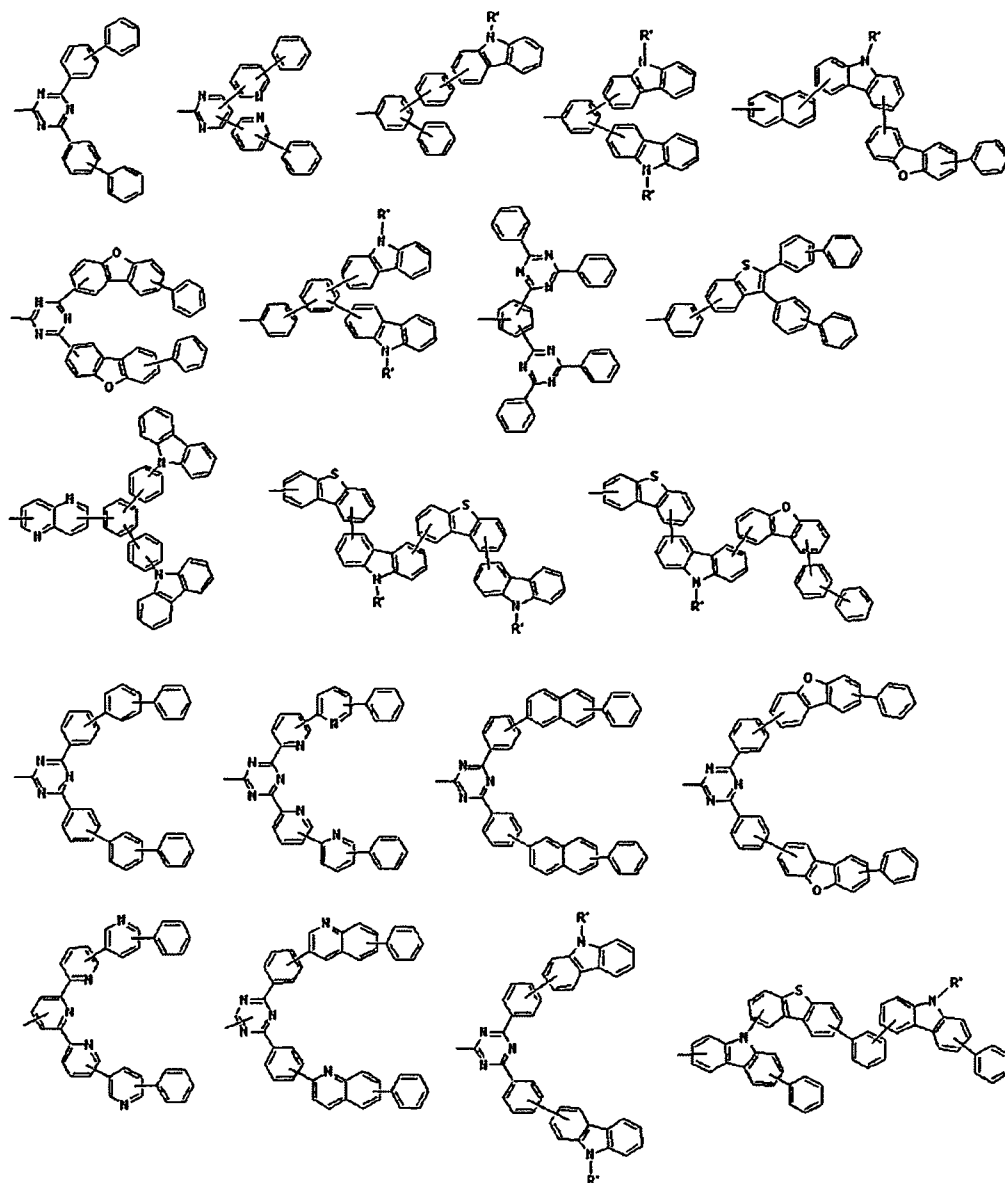


【0044】式(5)~式(7)中， $X_1 \sim X_4$ 表示芳香族烴基或芳香族雜環基，該等的一部分或全部亦可為縮合環。

【0045】 $-X_1-[X_2-\{X_3-(X_4)_1\}_m]_n$ 所表示的芳香環多個連結而生成的基團的具體例例如可列舉如下的一價基。於以下所示的一價基中， $R'$ 表示碳數6~18的芳香族烴基或碳數3~17的芳香族雜環基。碳數6~18的芳香族烴基或碳數3~17的芳香族雜環基的具體例與上述A中說明者相同。

【0046】



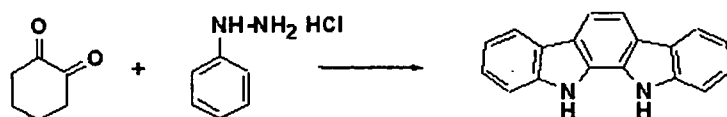


**【0047】** 通式 (1) 所表示的咪唑并咪唑化合物的較佳骨架有式 (IC-1) ~ 式 (IC-4) 的任一個所表示的骨架。通式 (1) 為包含式 (IC-1) ~ (IC-4) 所表示的骨架的概念，可利用通式 (1) 所表示的化合物來代表說明該些骨架。

**【0048】** 可認為通式 (1) 所表示的咪唑并咪唑化合物為式 (IC-1) ~ 式 (IC-4) 的形態所表示般的骨架，該等可根據目標化合物的結構來選擇原料，並使用公知的方法來合成。

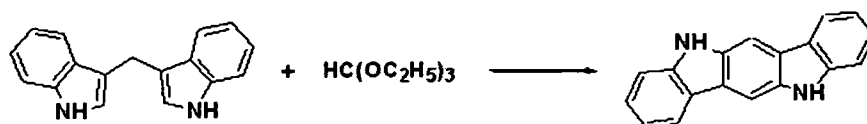
【0049】 例如，式 (IC-1) 所表示的吲哚并咪唑骨架可參考《合成有機化學快報 (Synlett)》，2005, No.1, p42-p48 所示的合成例藉由以下的反應式來合成。

【0050】



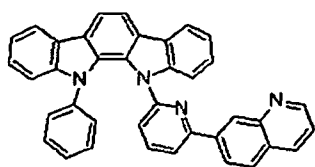
【0051】 另外，式 (IC-3) 所表示的吲哚并咪唑骨架可參考《製藥文獻 (魏茵海姆、德國) (Archiv der Pharmazie(Weinheim, Germany))》1987, 320(3), p280-2 中所示的合成例藉由以下的反應式來合成。

【0052】

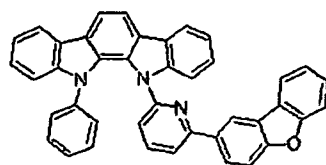


【0053】 以下示出通式 (1) 所表示的吲哚并咪唑化合物的具體例，但本發明的有機電場發光元件用材料不限定於該等。

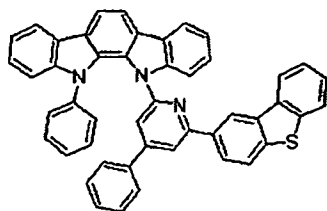
## 【0054】



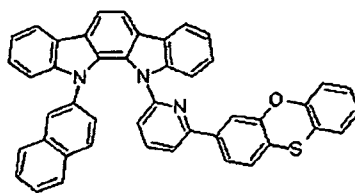
(A-1)



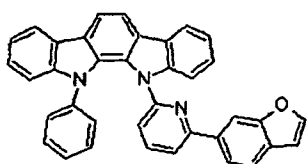
(A-2)



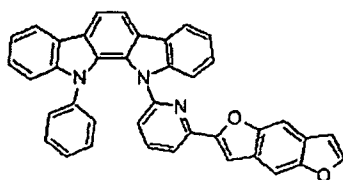
(A-3)



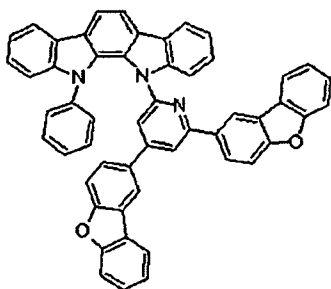
(A-4)



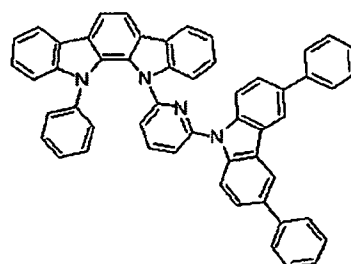
(A-5)



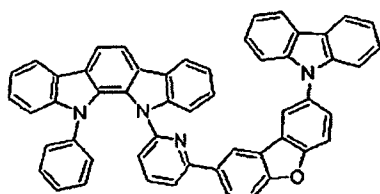
(A-6)



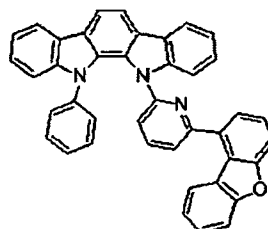
(A-7)



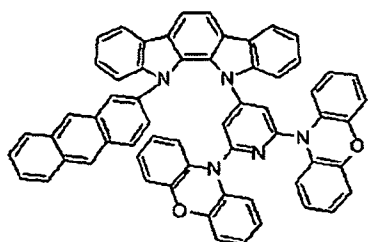
(A-8)



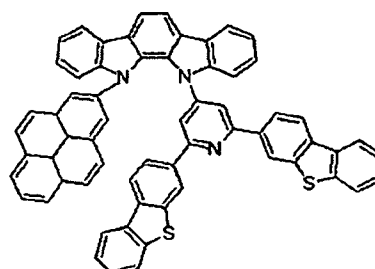
(A-9)



(A-10)

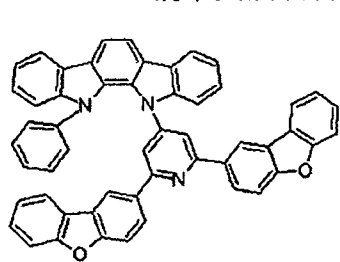


(A-11)

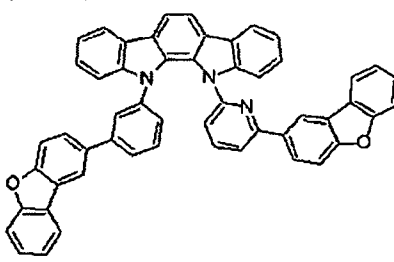


(A-12)

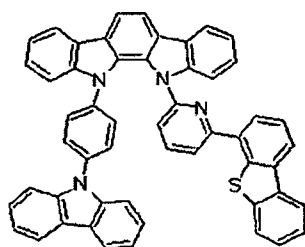




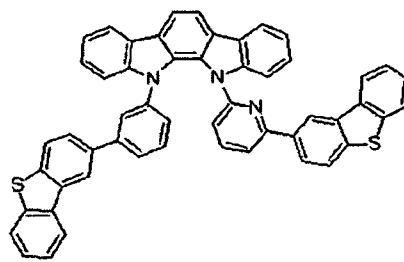
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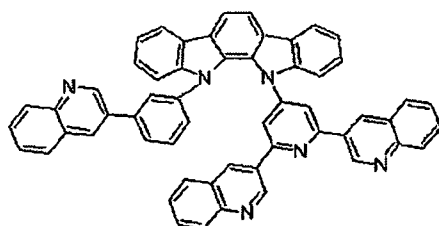
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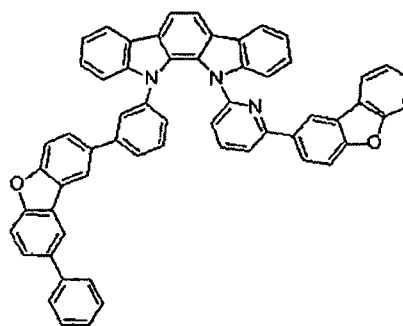
(A-15)



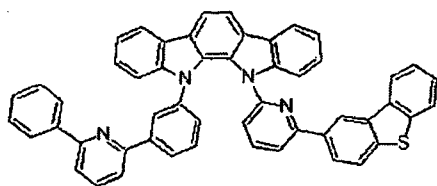
(A-16)



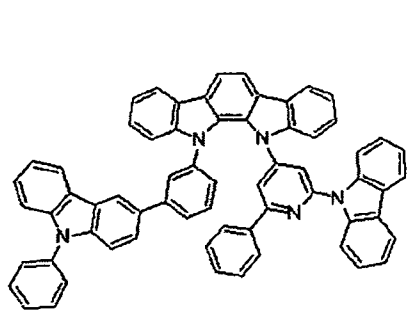
(A-17)



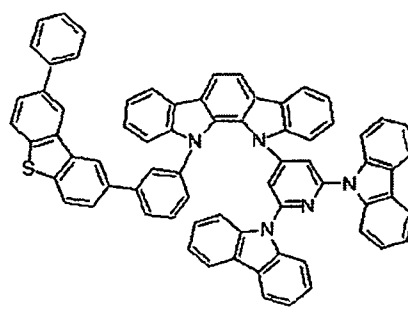
(A-18)



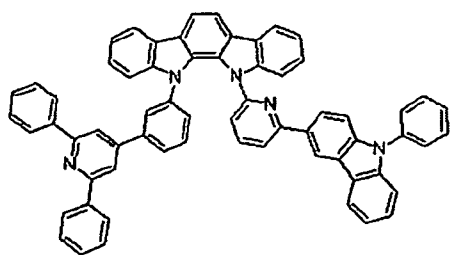
(A-19)



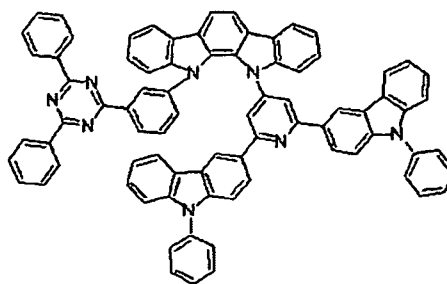
(A-20)



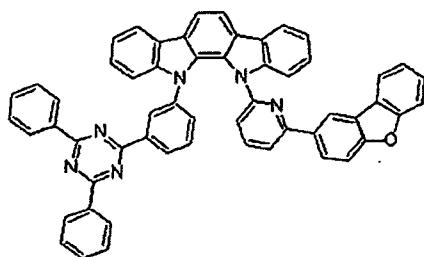
(A-21)



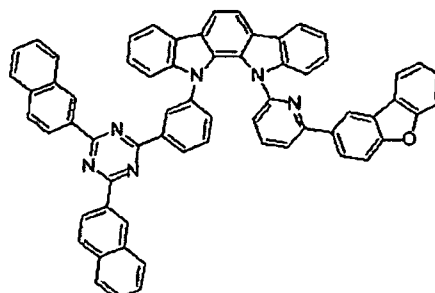
(A-22)



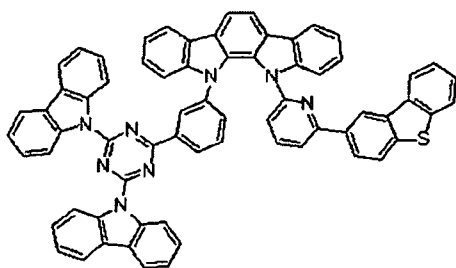
(A-23)



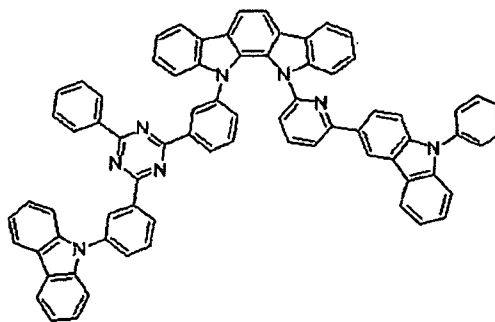
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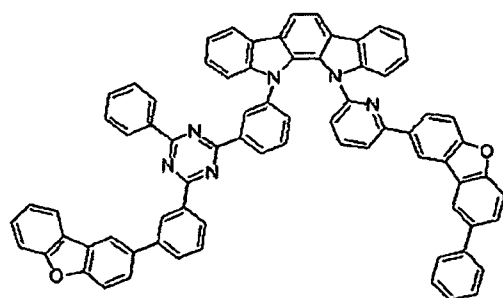
(A-25)



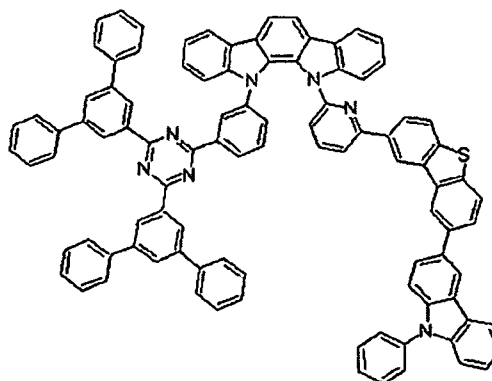
(A-26)



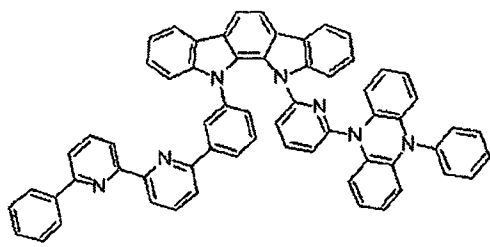
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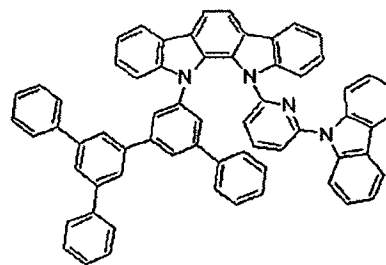
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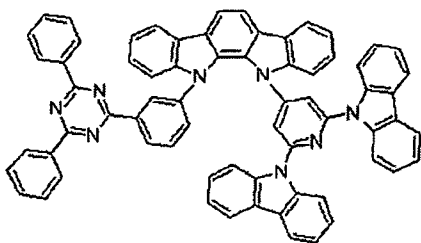
(A-29)



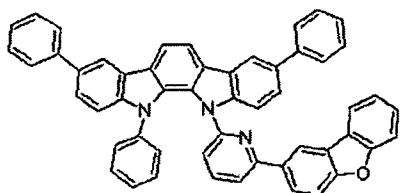
(A-30)



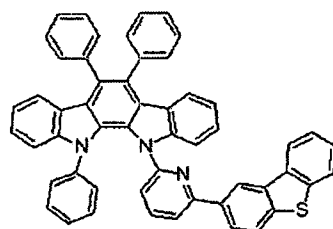
(A-31)



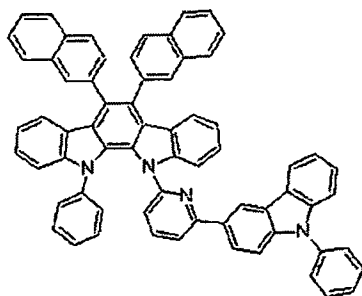
(A-32)



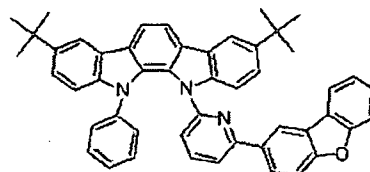
(A-33)



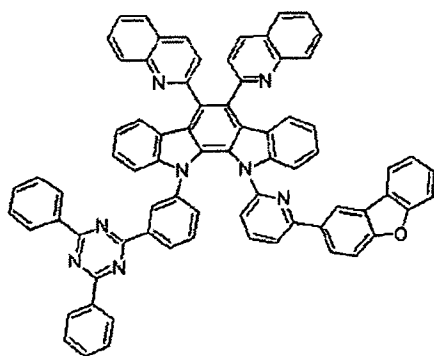
(A-34)



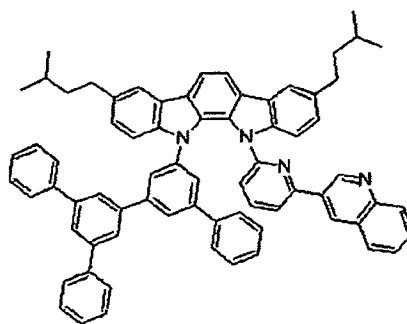
(A-35)



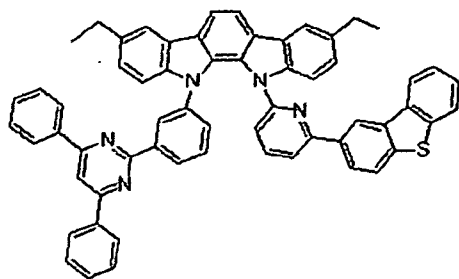
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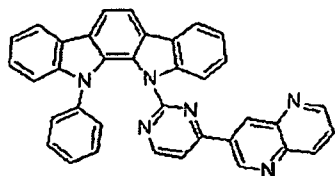
(A-37)



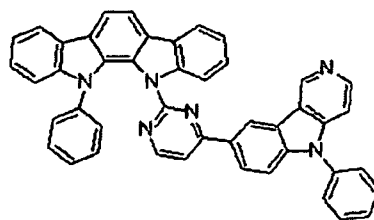
(A-38)



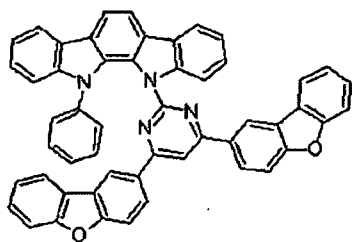
(A-39)



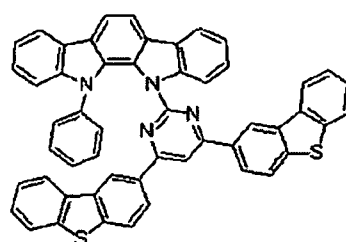
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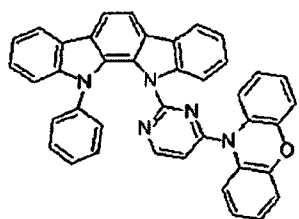
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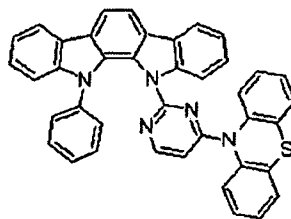
(A-42)



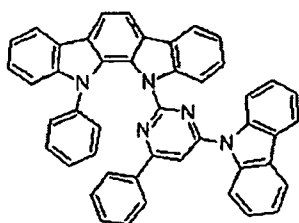
(A-43)



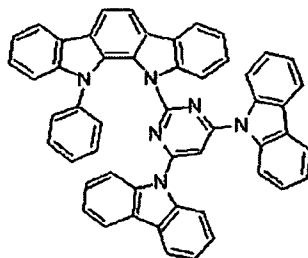
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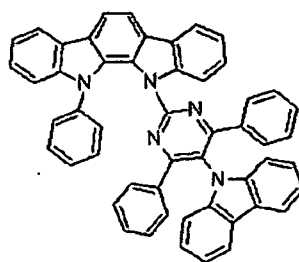
(A-45)



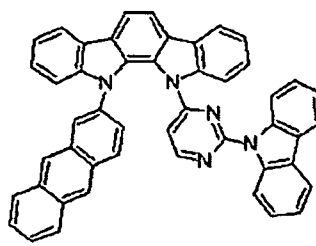
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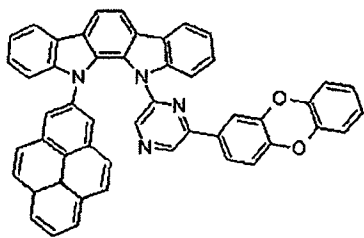
(A-47)



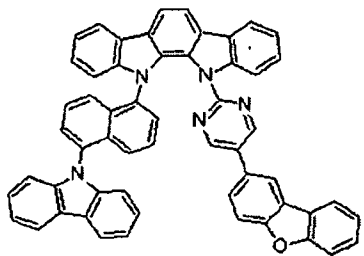
(A-48)



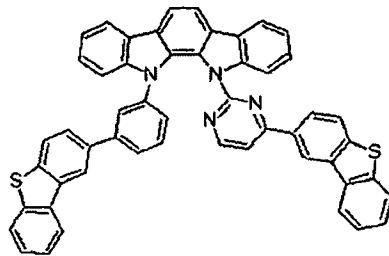
(A-49)



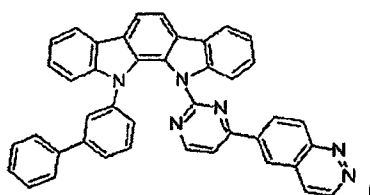
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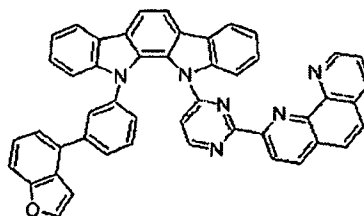
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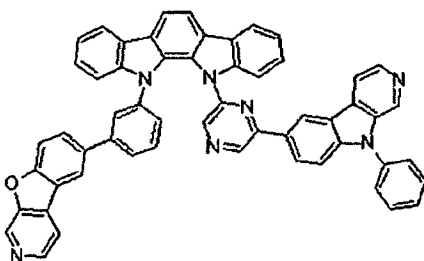
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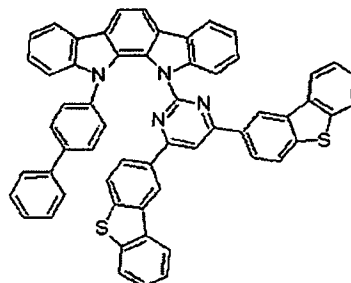
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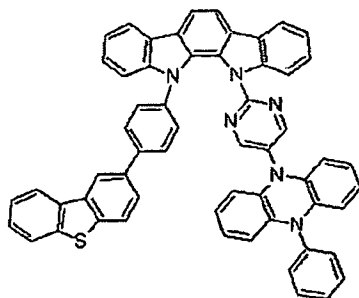
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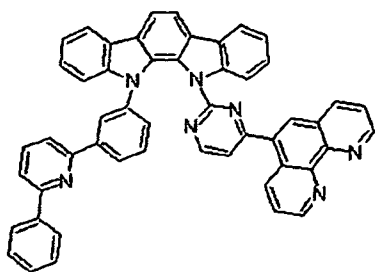
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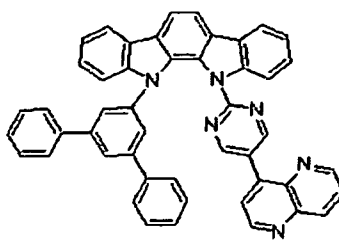
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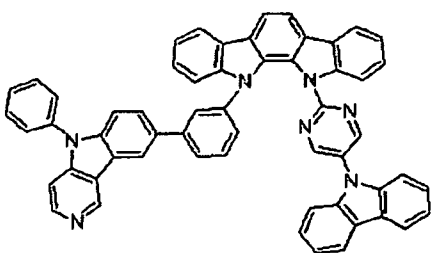
(A-57)



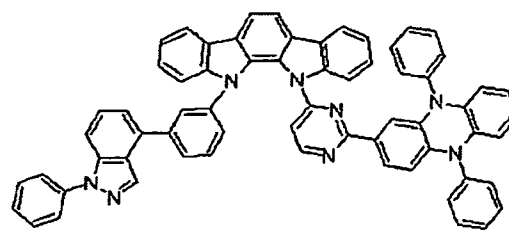
(A-58)



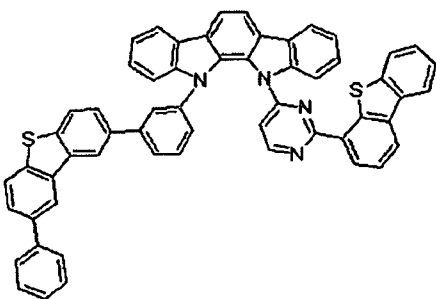
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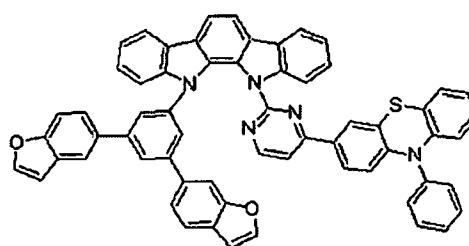
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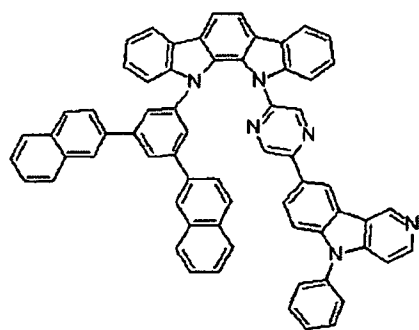
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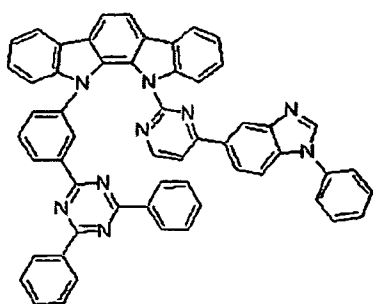
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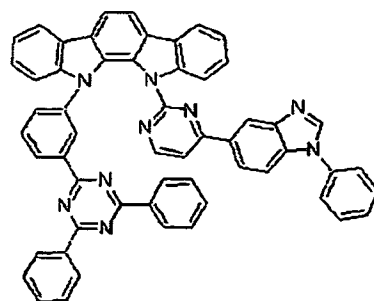
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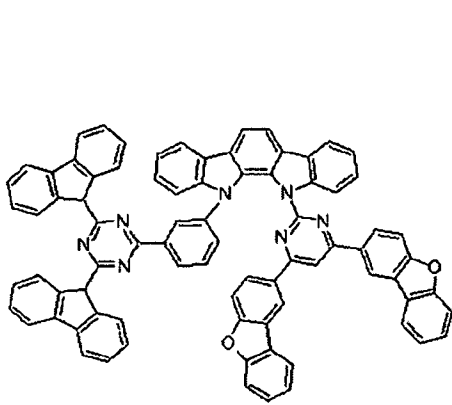
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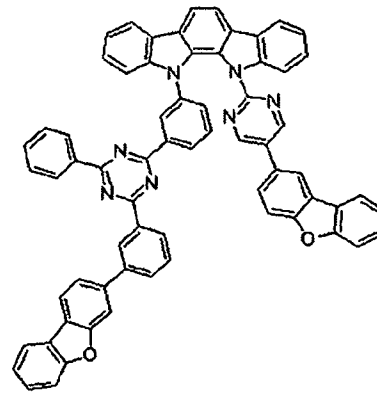
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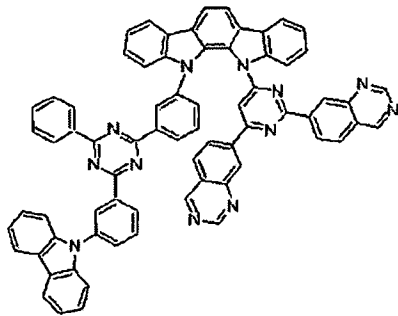
(A-66)



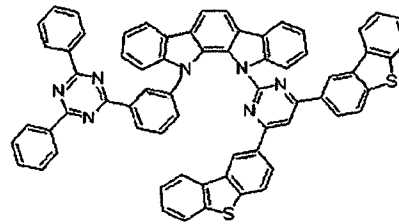
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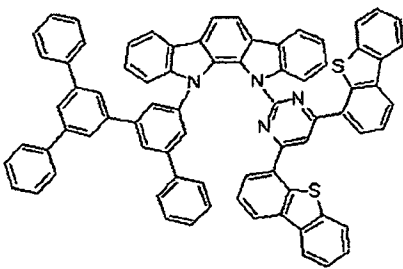
(A-68)



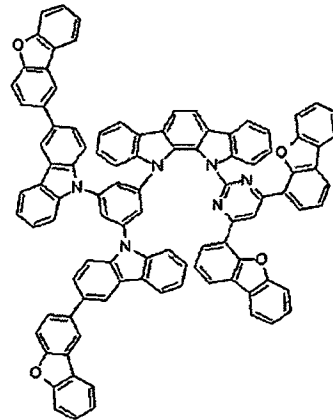
(A-69)



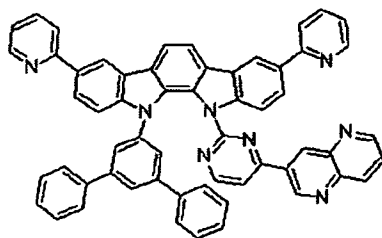
(A-70)



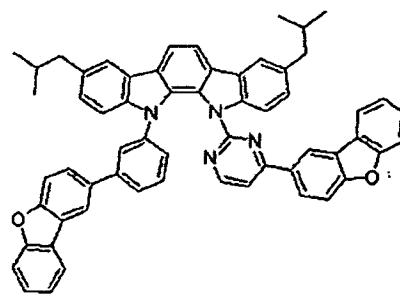
(A-71)



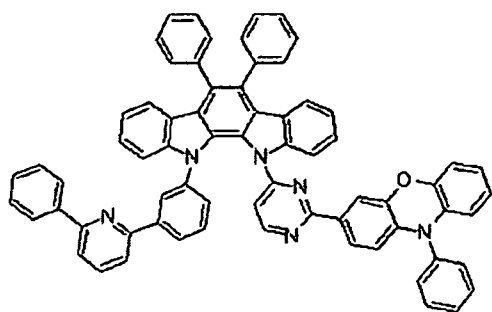
(A-72)



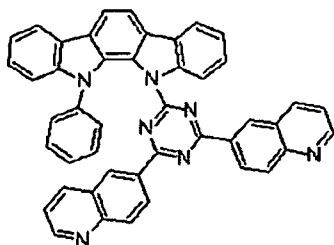
(A-73)



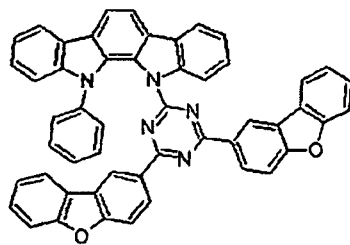
(A-74)



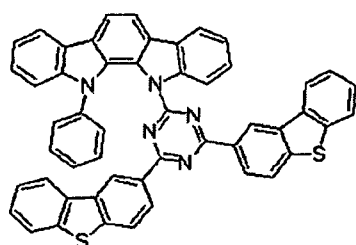
(A-75)



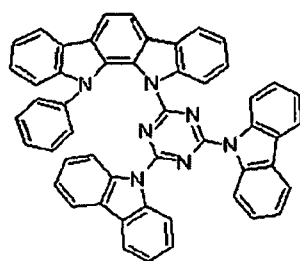
(A-76)



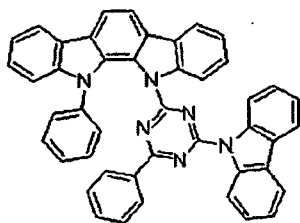
(A-77)



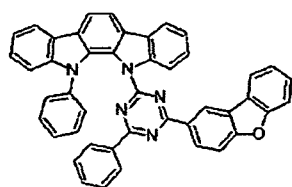
(A-78)



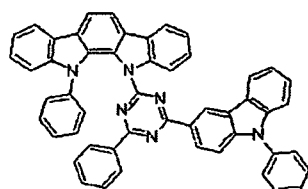
(A-79)



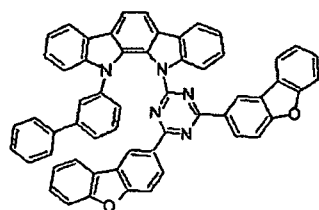
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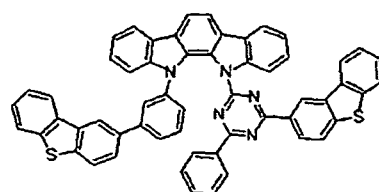
(A-81)



(A-82)

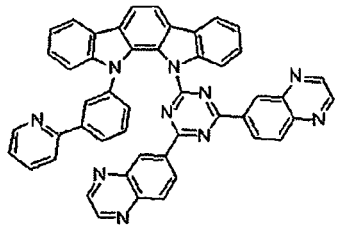


(A-83)

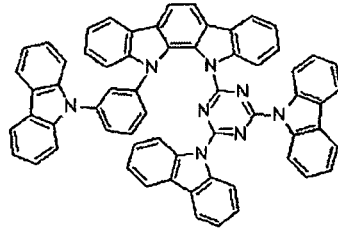


(A-84)

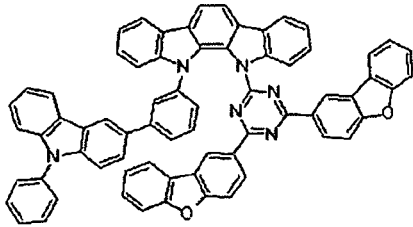




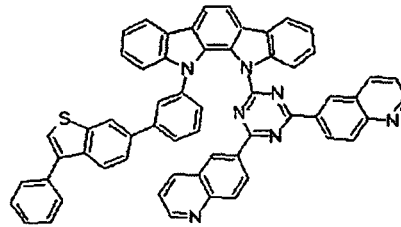
(A-85)



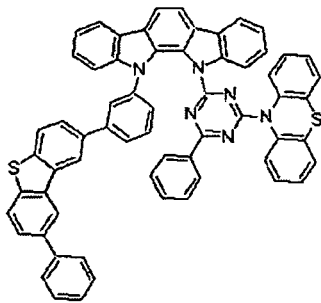
(A-86)



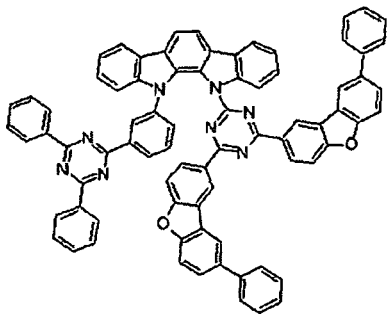
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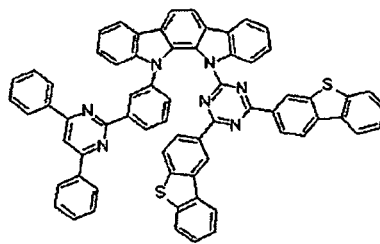
(A-88)



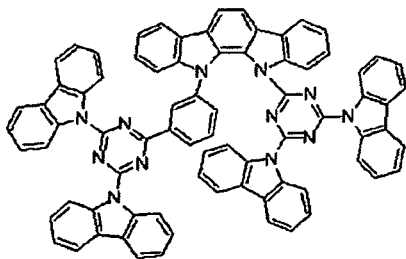
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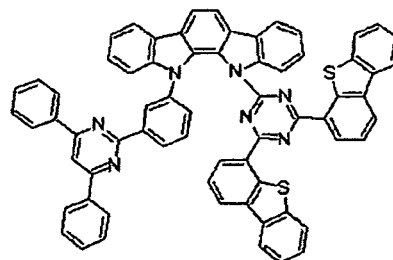
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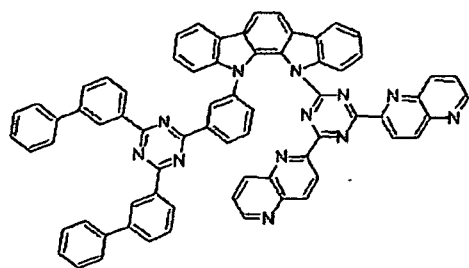
(A-91)



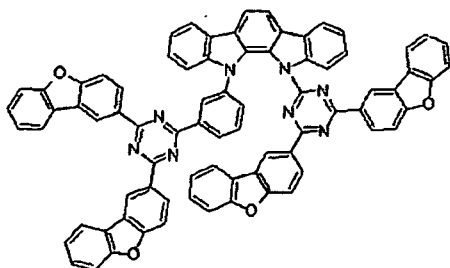
(A-92)



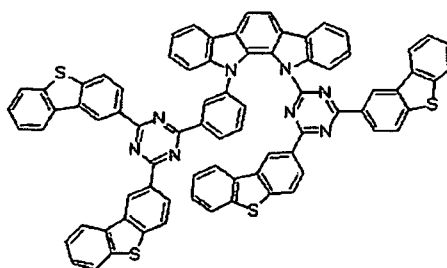
(A-93)



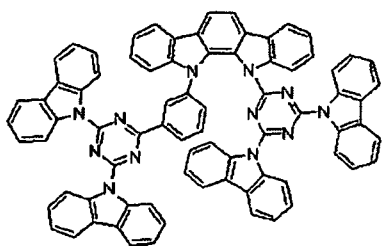
(A-94)



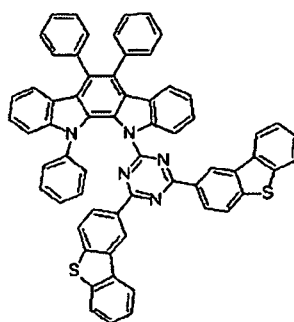
(A-95)



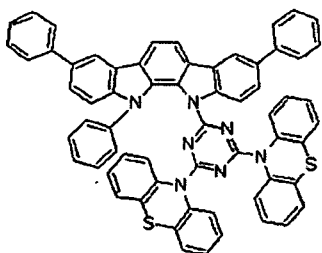
(A-96)



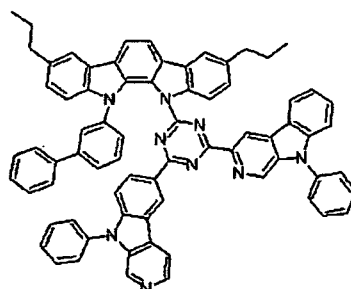
(A-97)



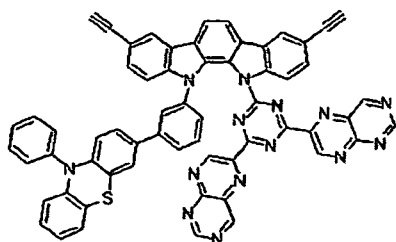
(A-98)



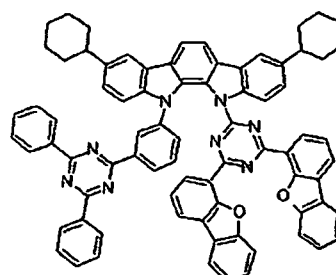
(A-99)



(A-100)

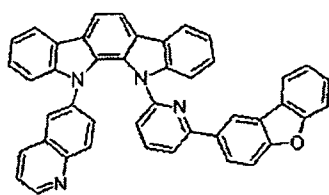


(A-101)

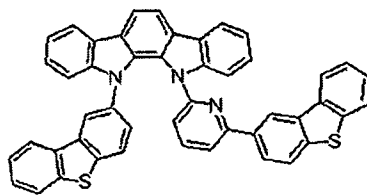


(A-102)

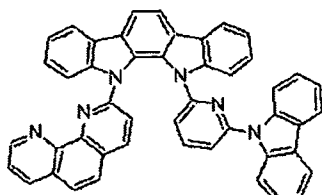
【0055】



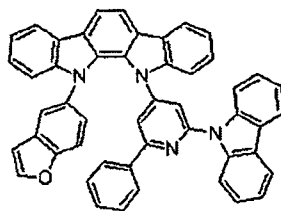
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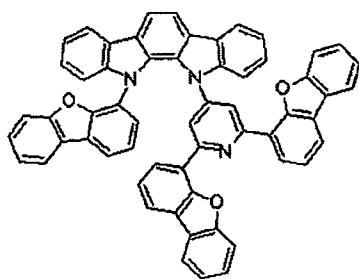
(B-2)



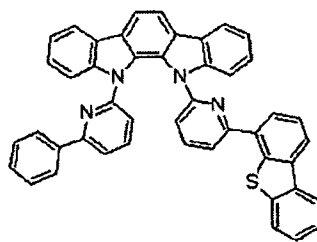
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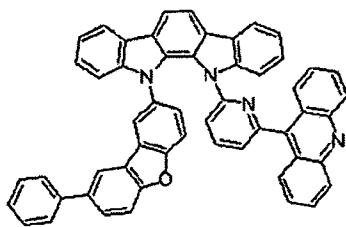
(B-4)



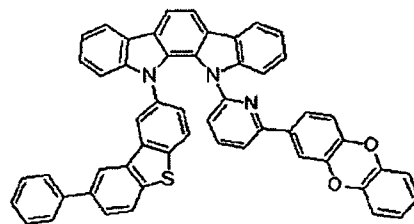
(B-5)



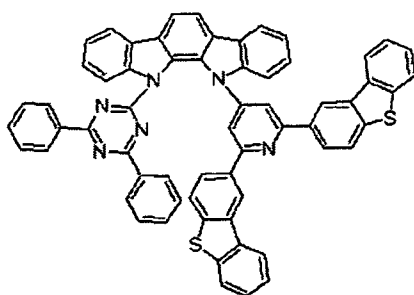
(B-6)



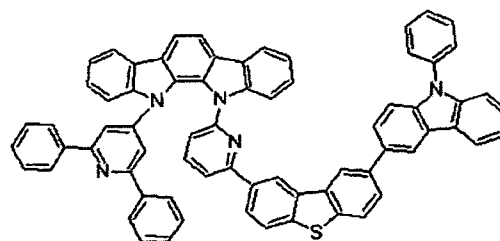
(B-7)



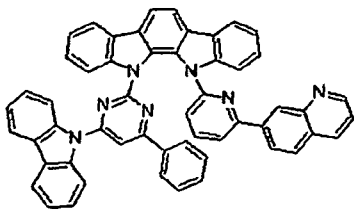
(B-8)



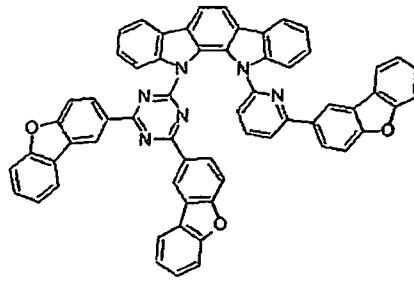
(B-9)



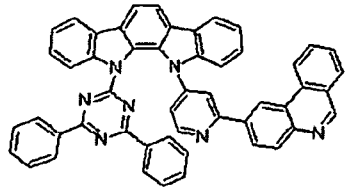
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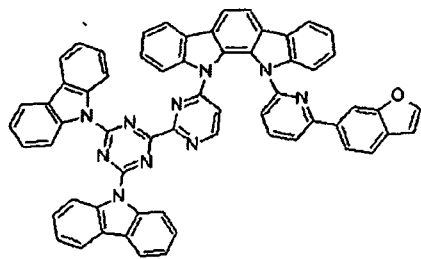
(B-11)



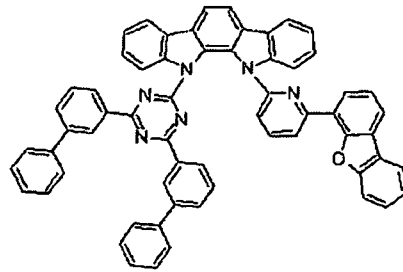
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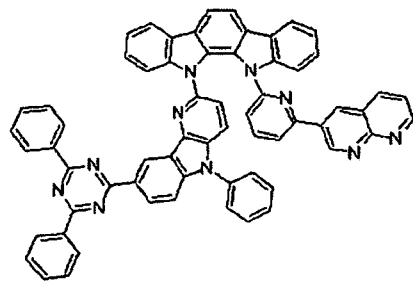
(B-13)



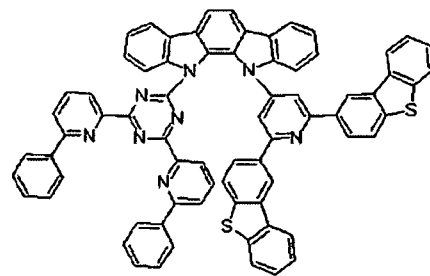
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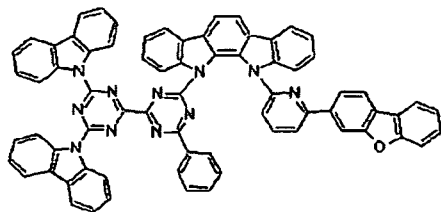
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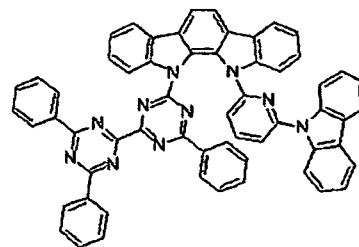
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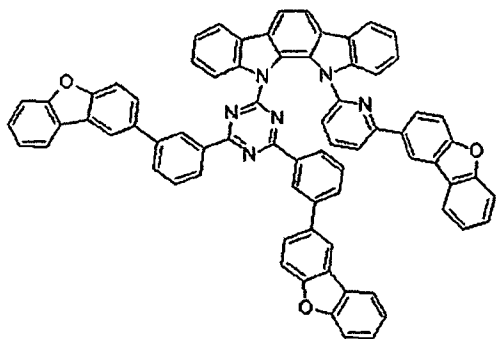
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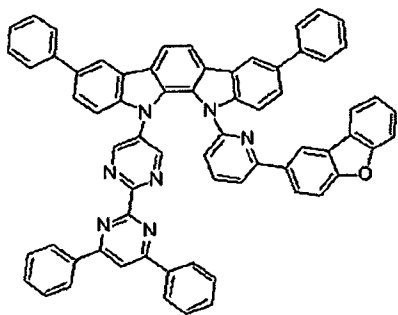
(B-18)



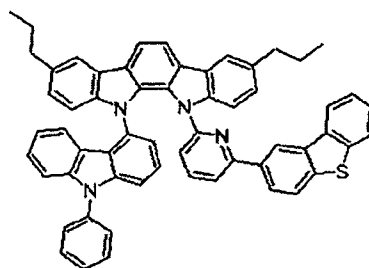
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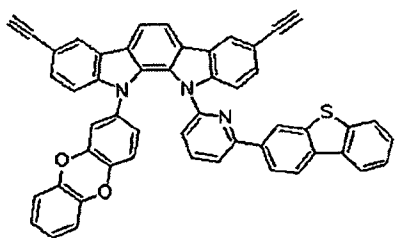
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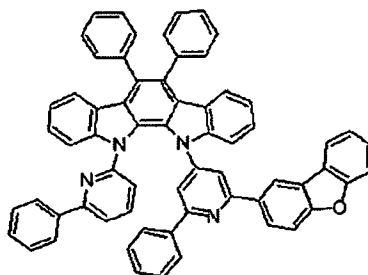
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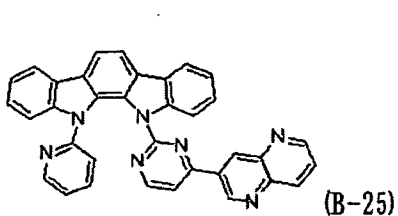
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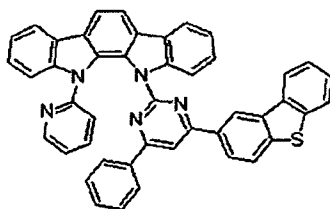
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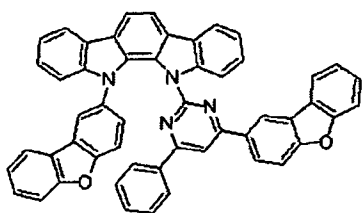
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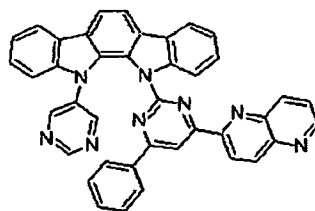
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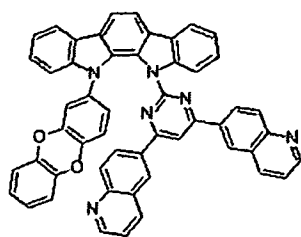
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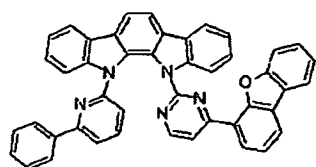
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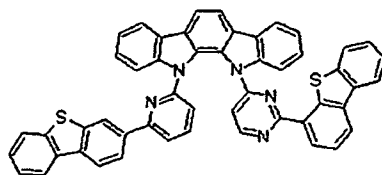
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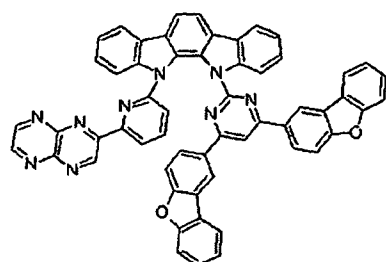
(B-29)



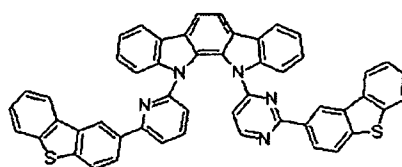
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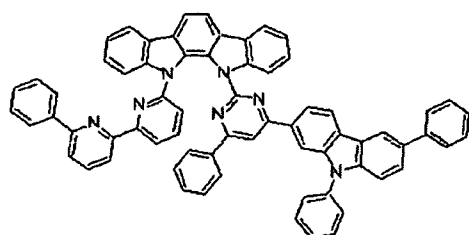
(B-31)



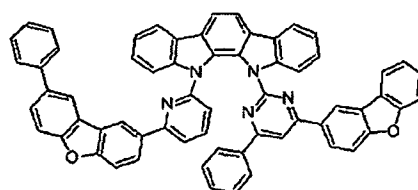
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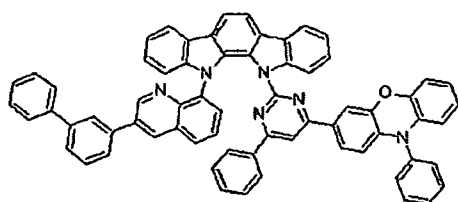
(B-33)



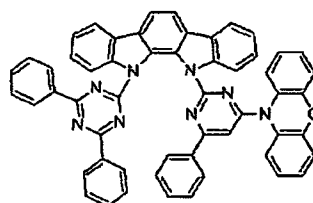
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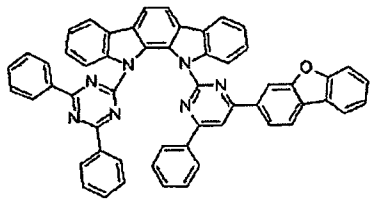
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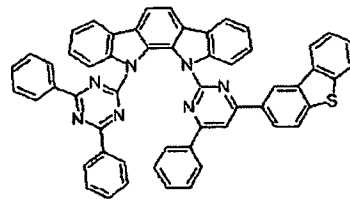
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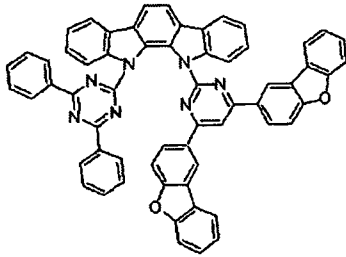
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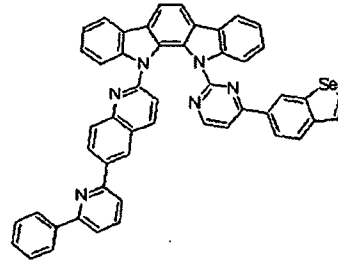
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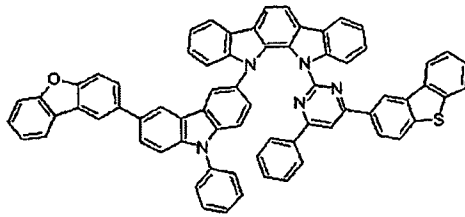
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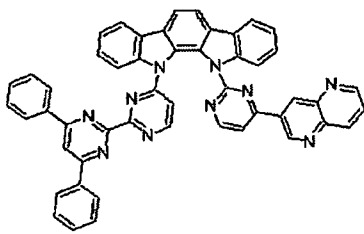
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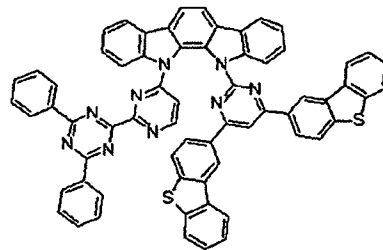
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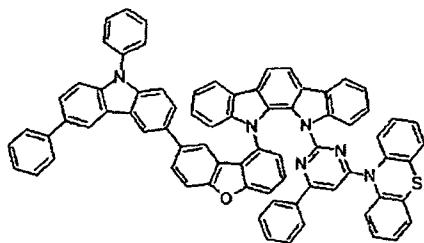
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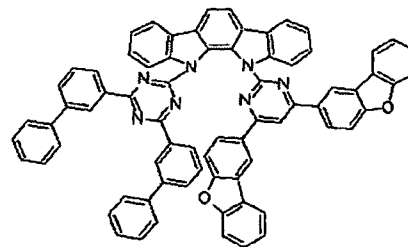
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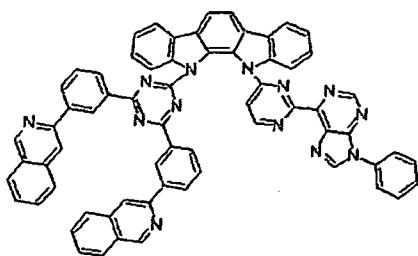
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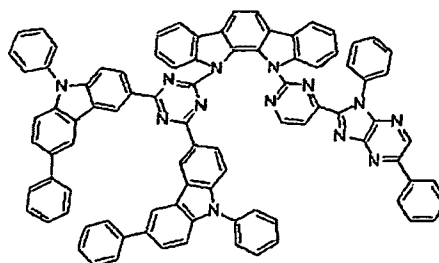
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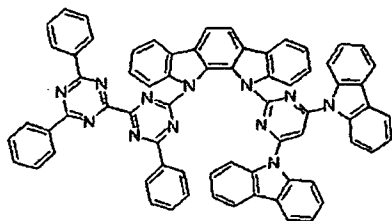
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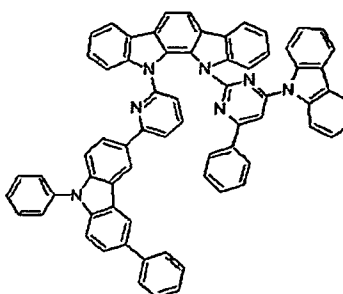
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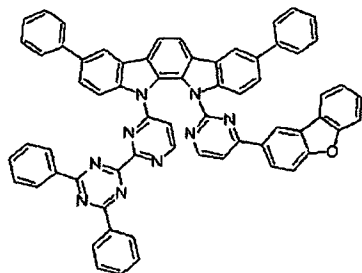
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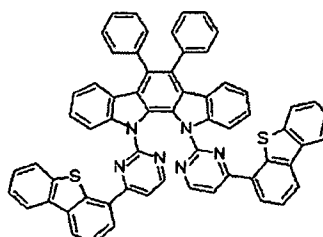
(B-49)



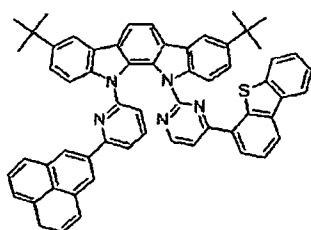
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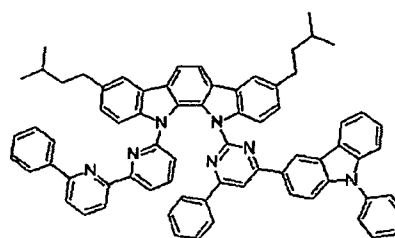
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(B-52)

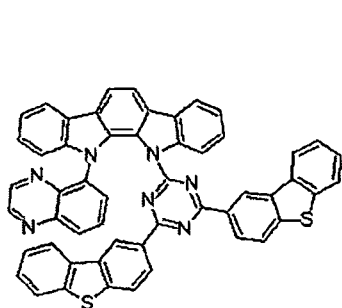


(B-53)

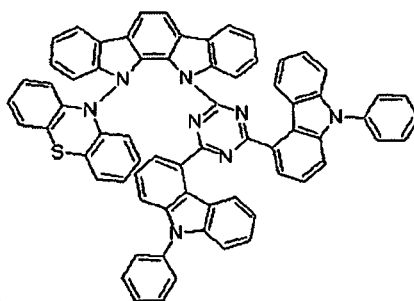


(B-54)

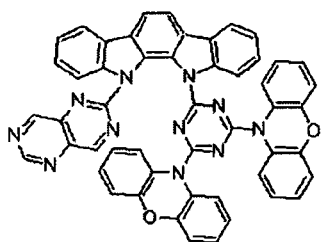




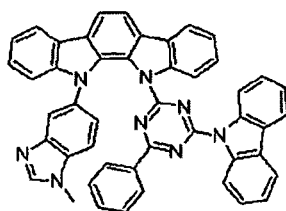
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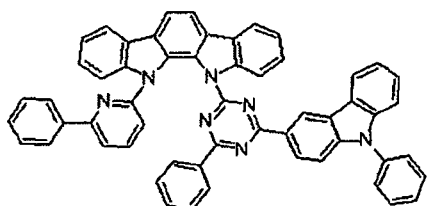
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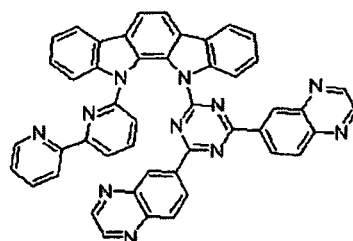
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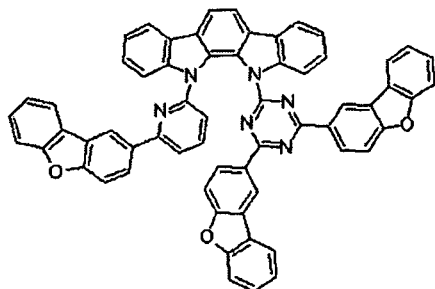
(B-58)



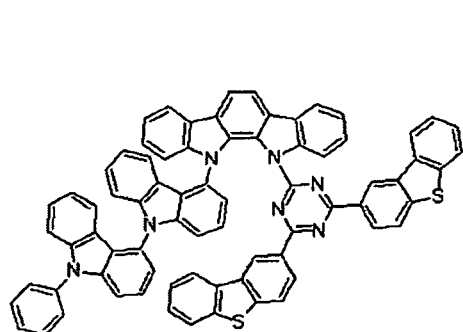
(B-59)



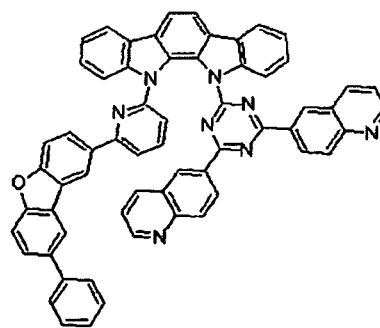
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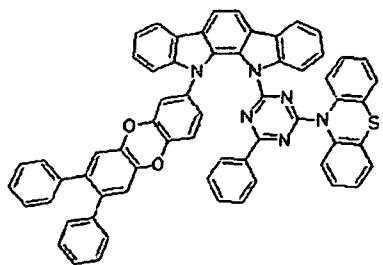
(B-61)



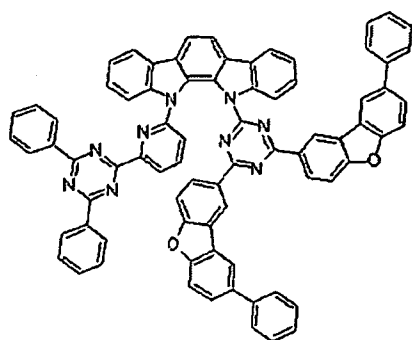
(B-62)



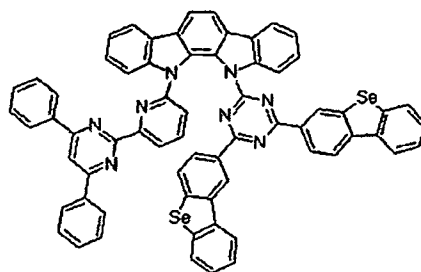
(B-63)



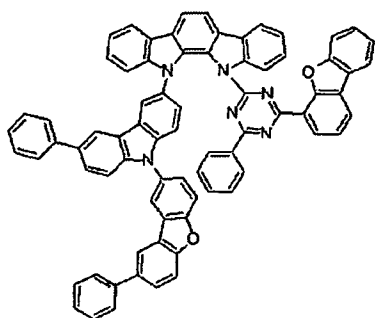
(B-64)



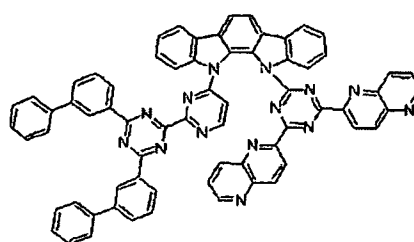
(B-65)



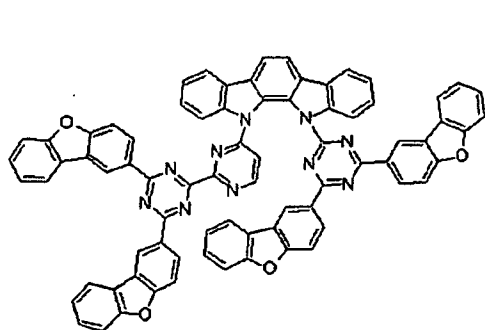
(B-66)



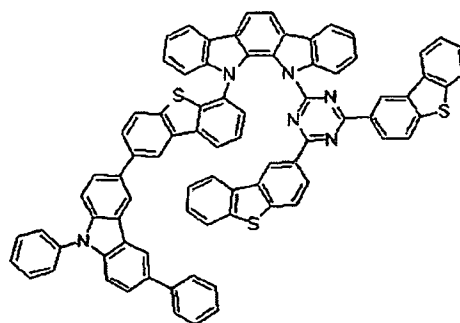
(B-67)



(B-68)

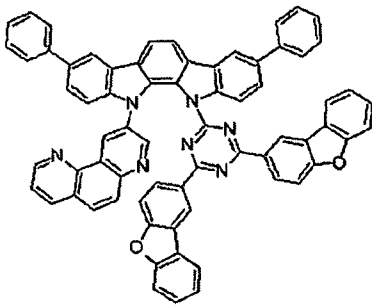


(B-69)

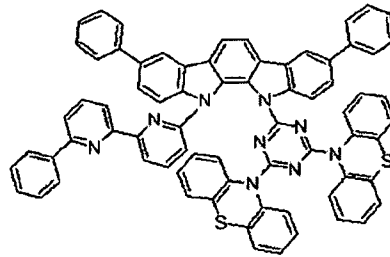


(B-70)

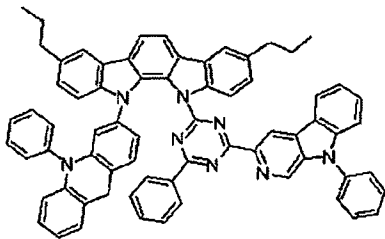
【0056】



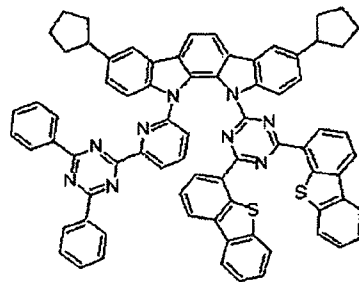
(B-71)



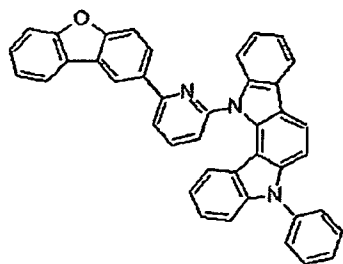
(B-72)



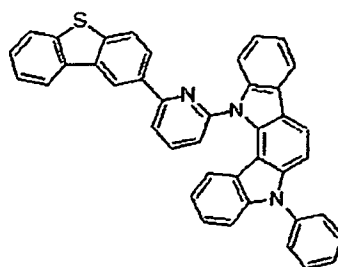
(B-73)



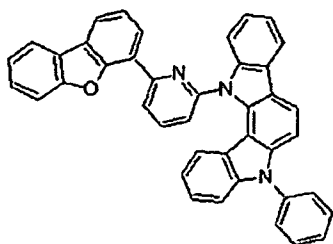
(B-74)



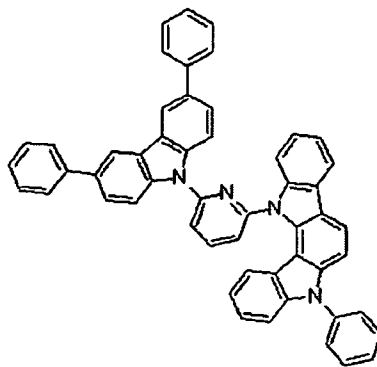
(C-1)



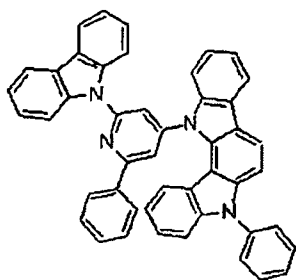
(C-2)



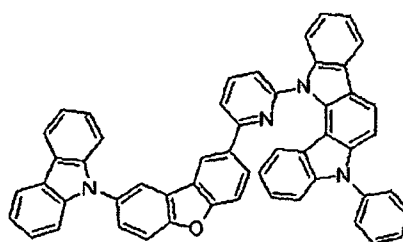
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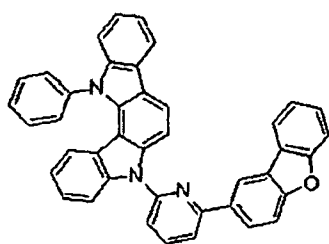
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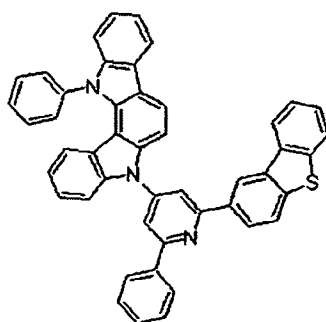
(C-5)



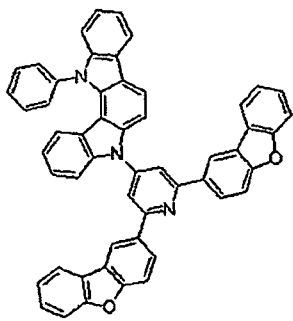
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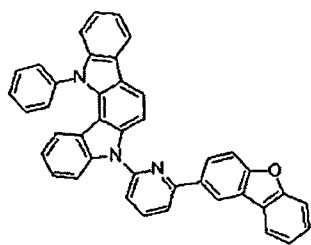
(C-7)



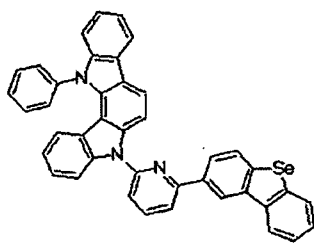
(C-8)



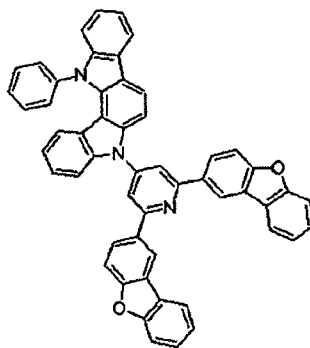
(C-9)



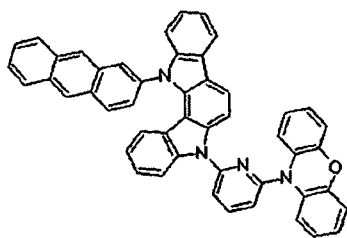
(C-10)



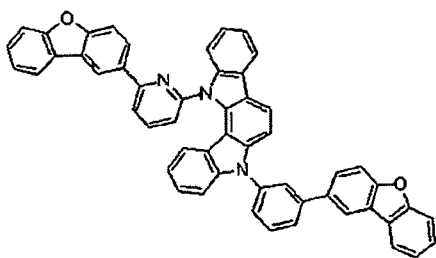
(C-11)



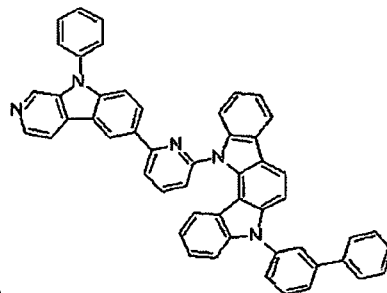
(C-12)



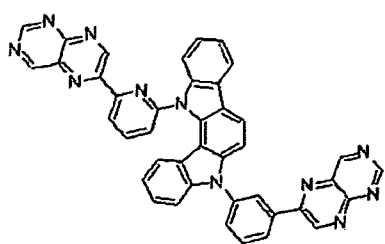
(C-13)



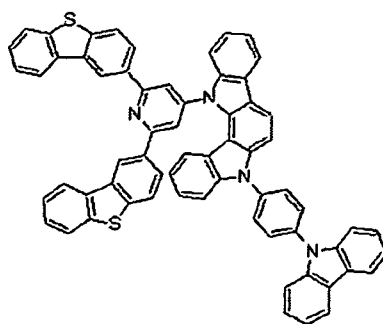
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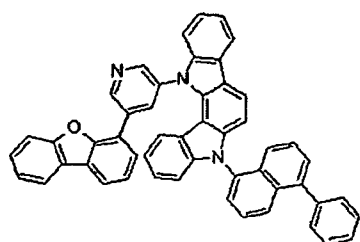
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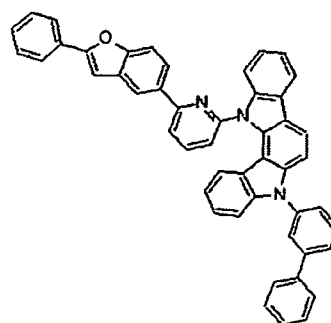
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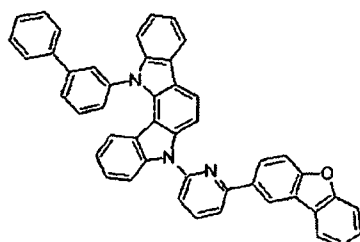
(C-17)



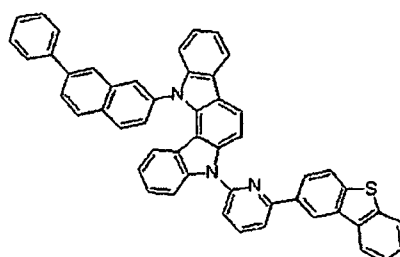
(C-18)



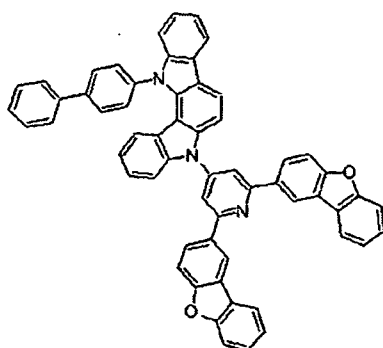
(C-19)



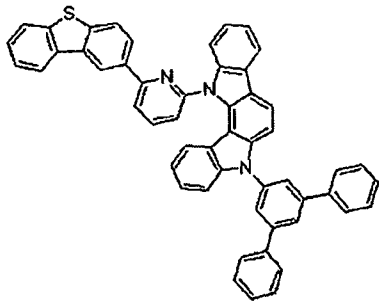
(C-20)



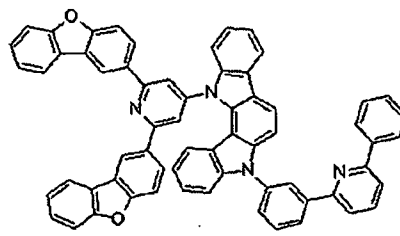
(C-21)



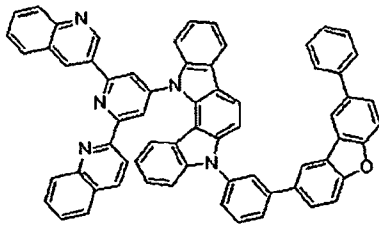
(C-22)



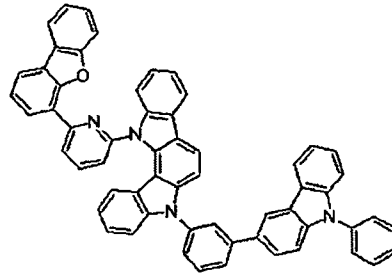
(C-23)



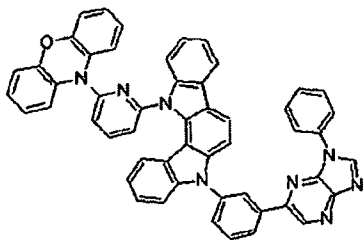
(C-24)



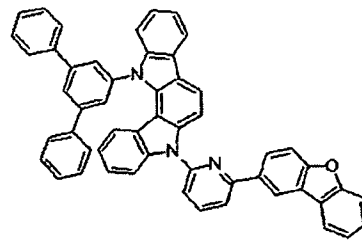
(C-25)



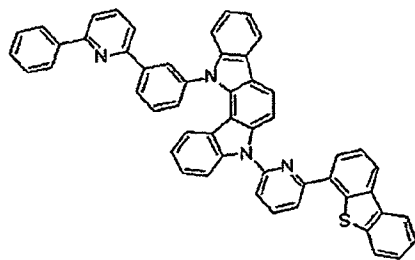
(C-26)



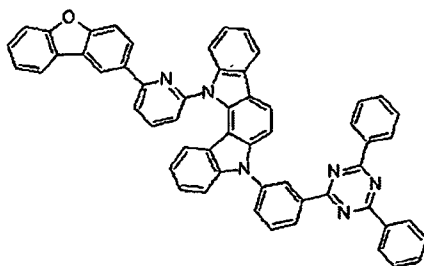
(C-27)



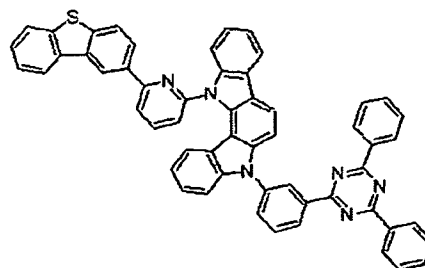
(C-28)



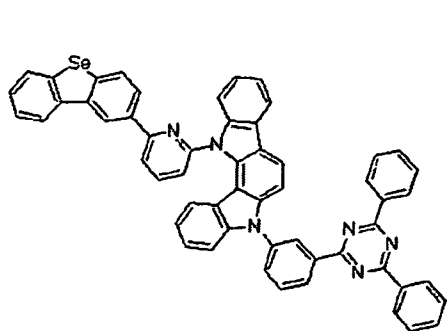
(C-29)



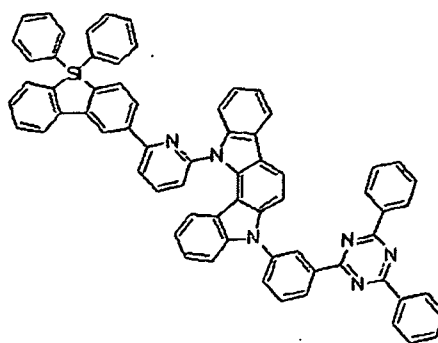
(C-30)



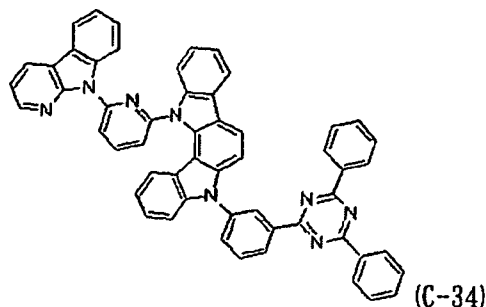
(C-31)



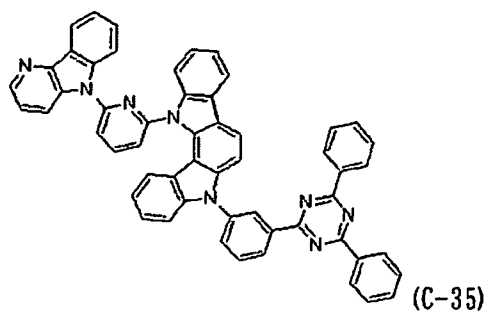
(C-32)



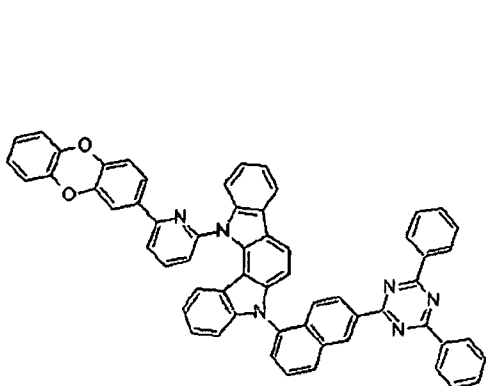
(C-33)



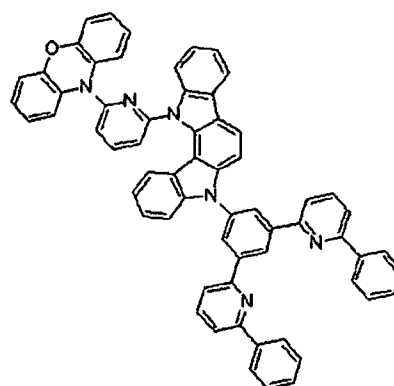
(C-34)



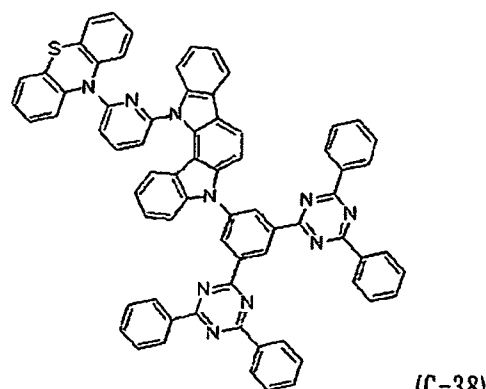
(C-35)



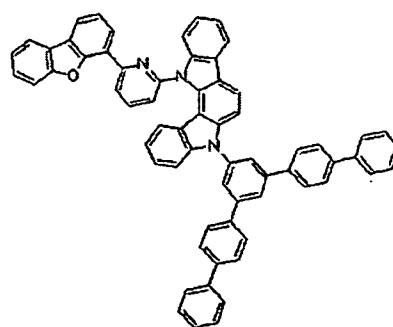
(C-36)



(C-37)

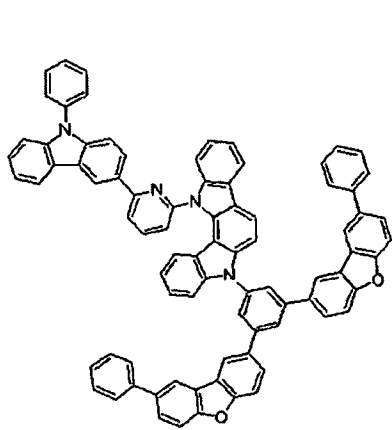


(C-38)

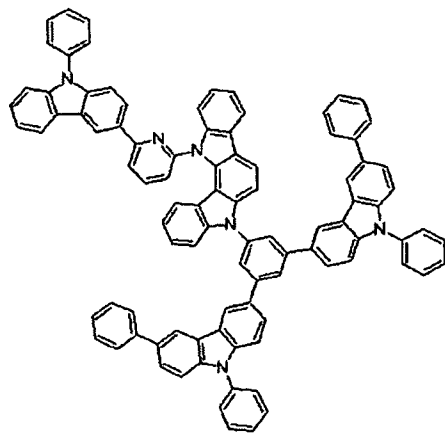


(C-39)

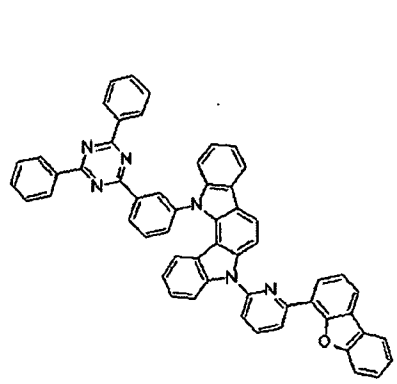




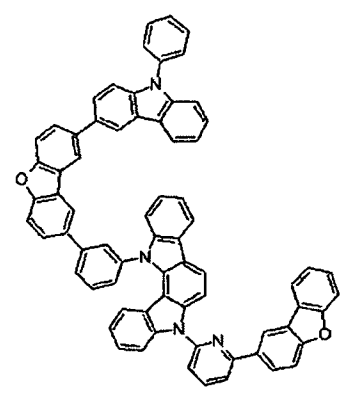
(C-40)



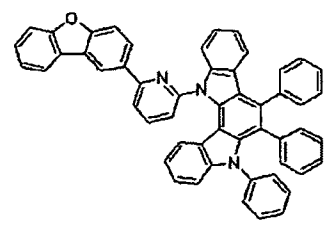
(C-41)



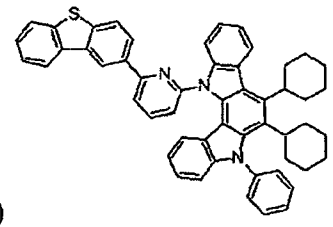
(C-42)



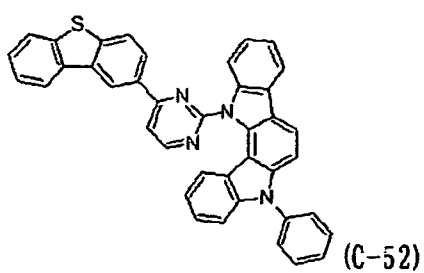
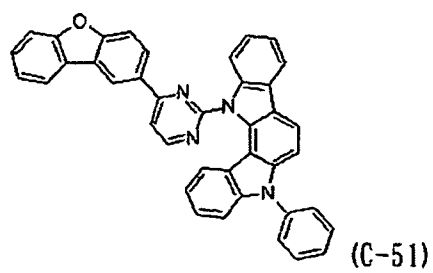
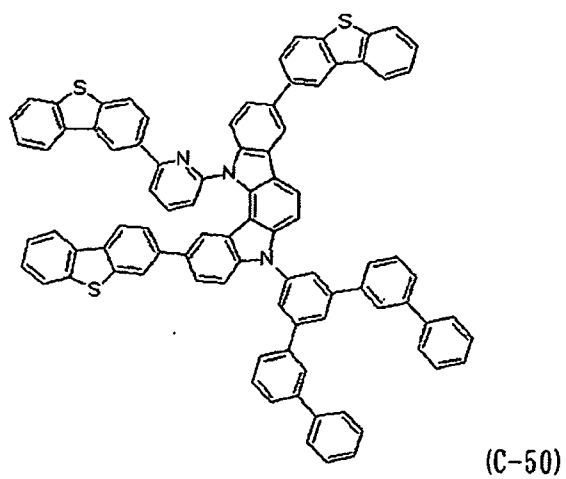
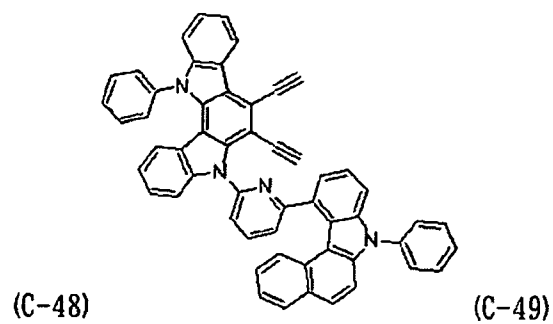
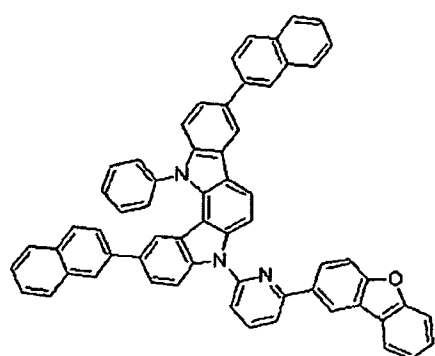
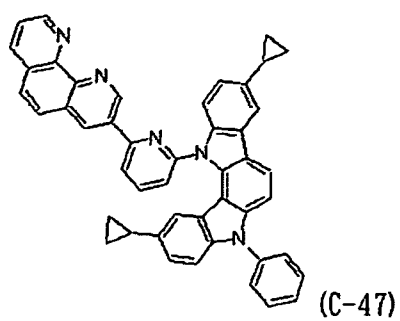
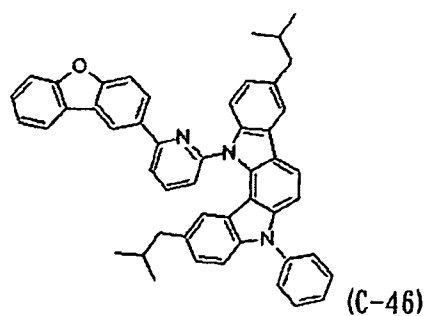
(C-43)

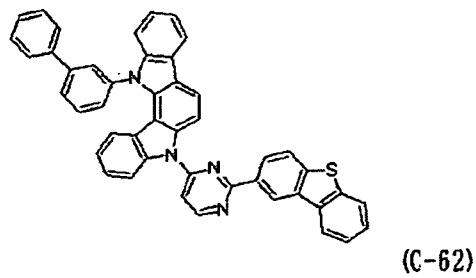
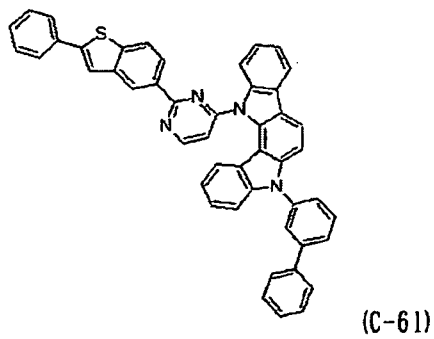
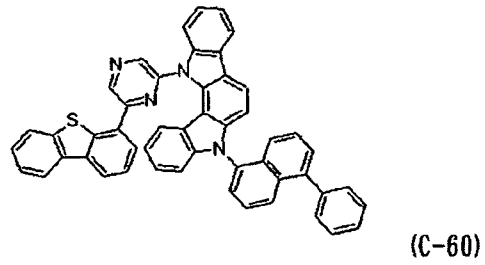
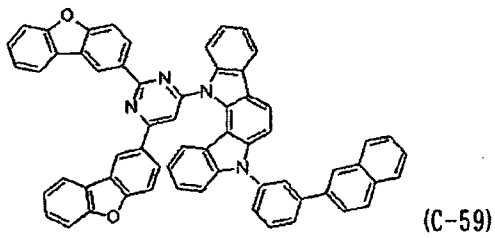
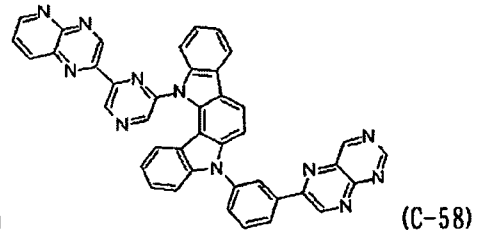
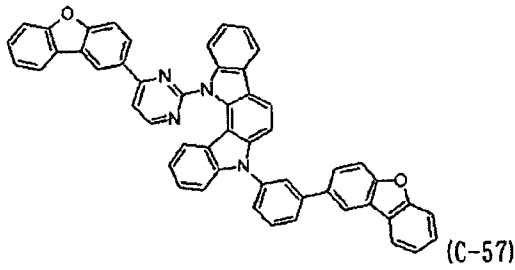
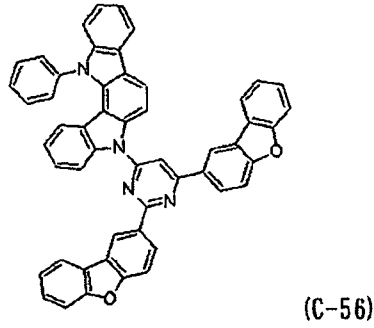
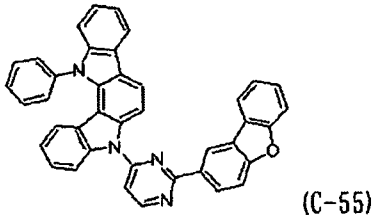
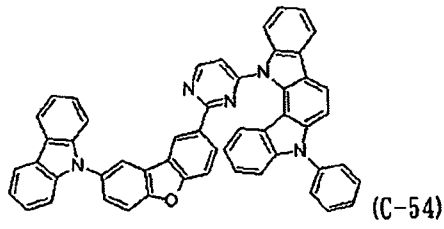
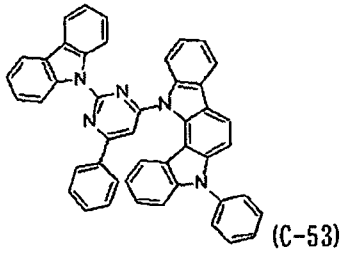


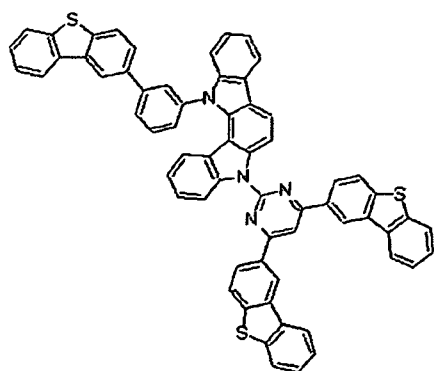
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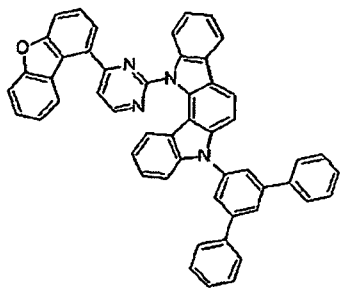
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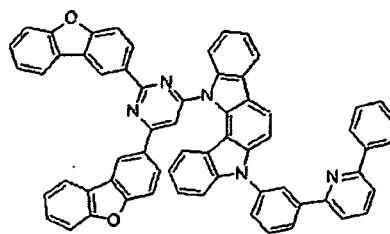




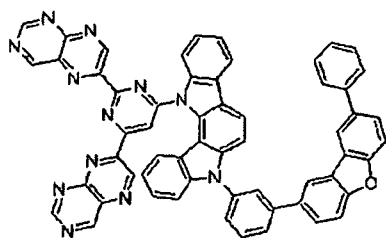
(C-63)



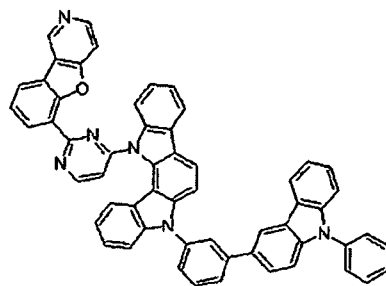
(C-64)



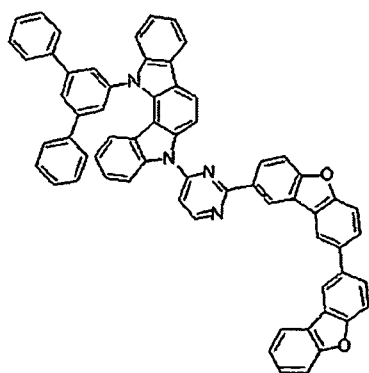
(C-65)



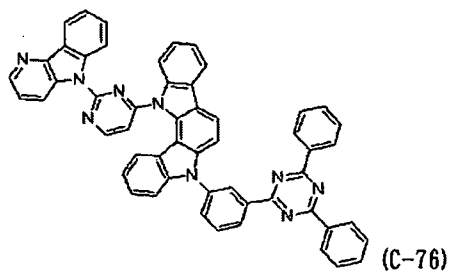
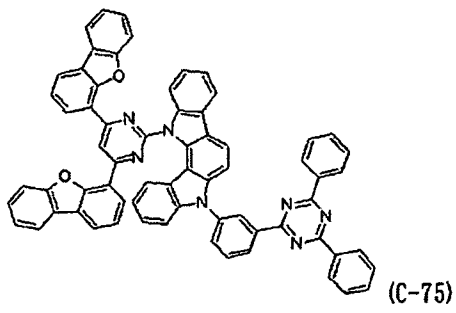
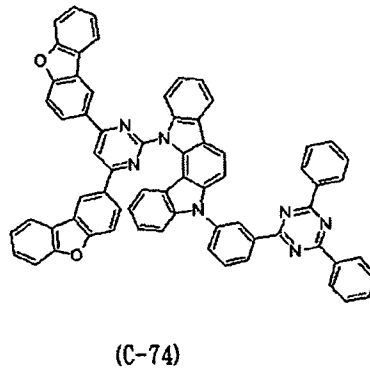
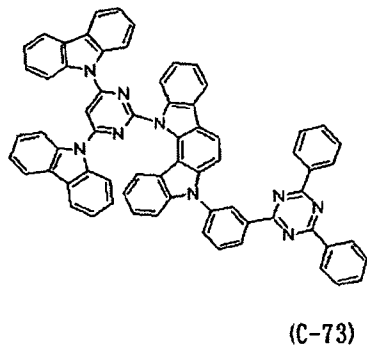
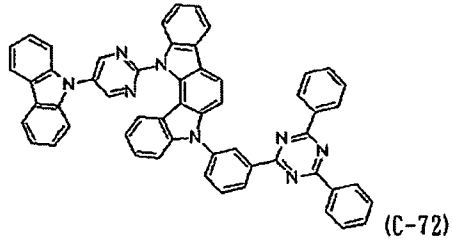
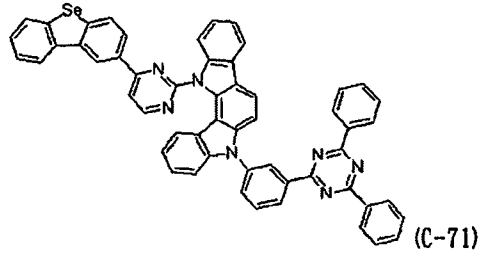
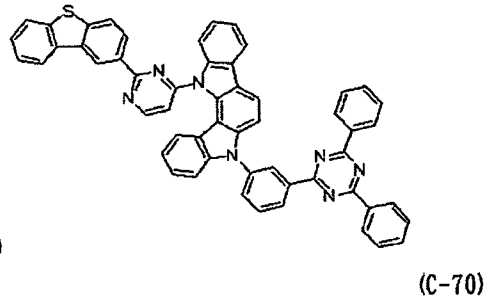
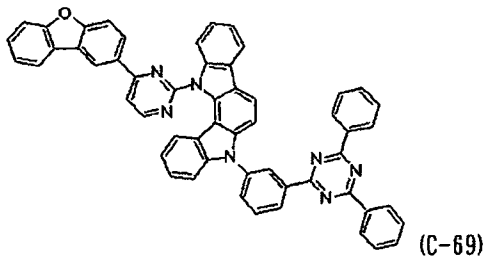
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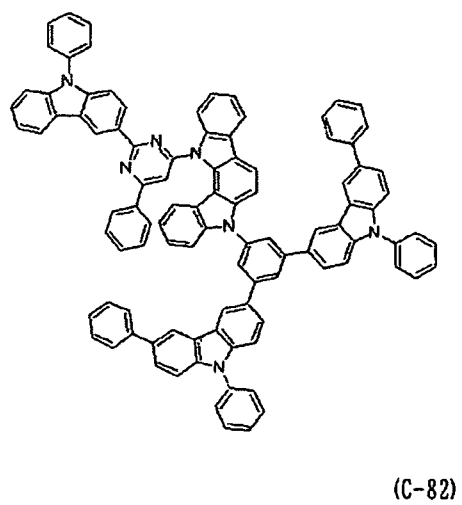
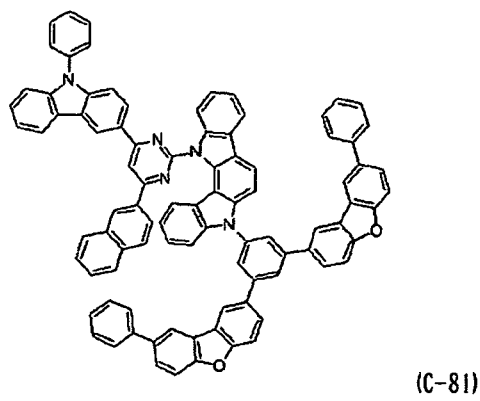
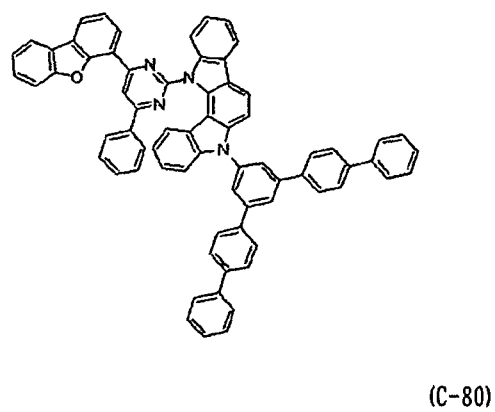
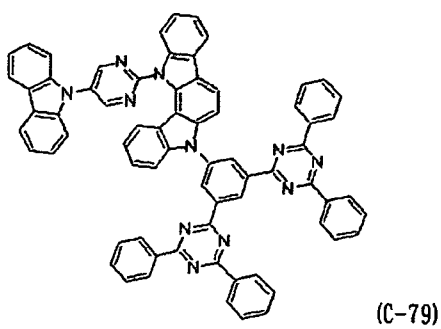
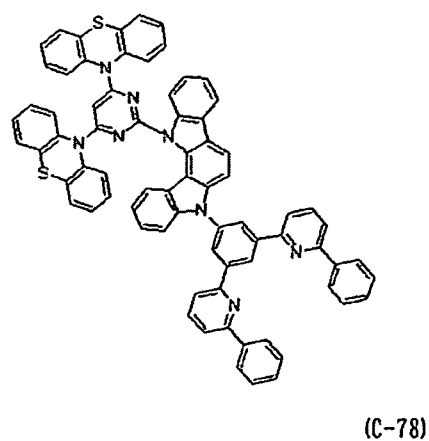
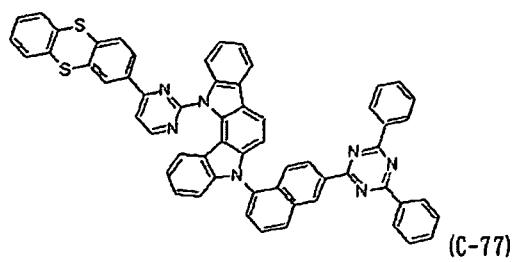


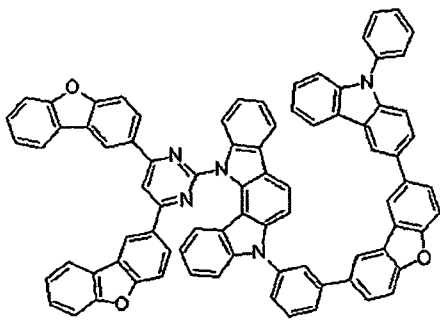
(C-67)



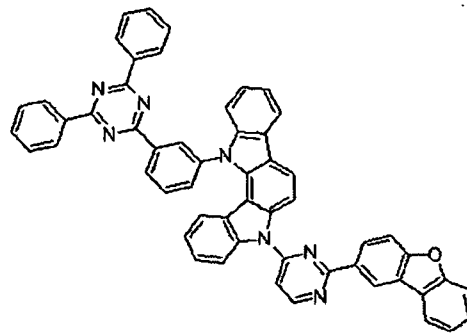
(C-68)



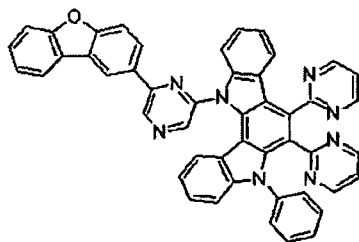




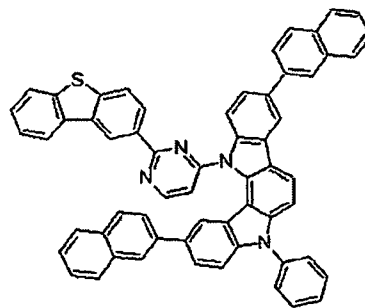
(C-83)



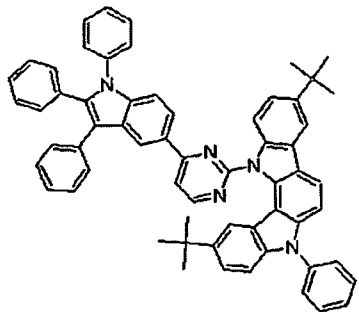
(C-84)



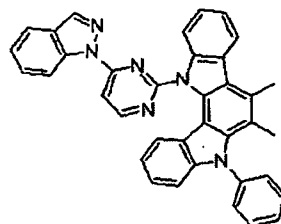
(C-85)



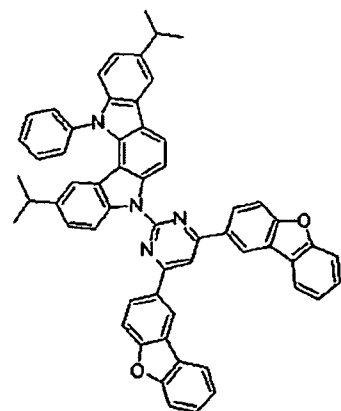
(C-86)



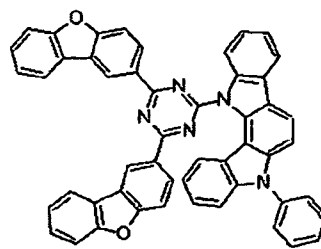
(C-87)



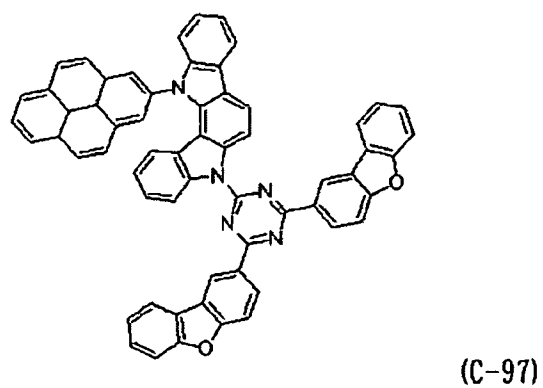
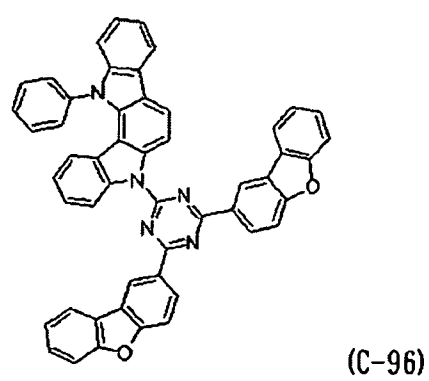
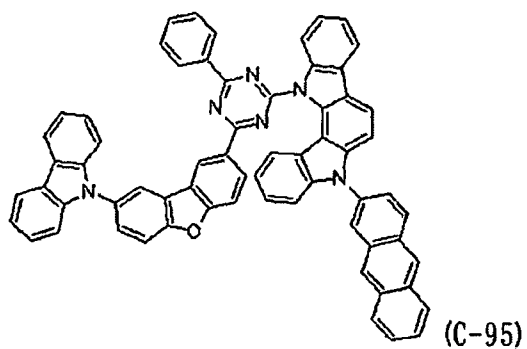
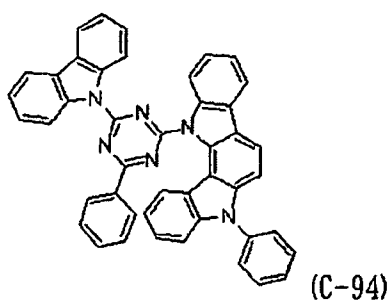
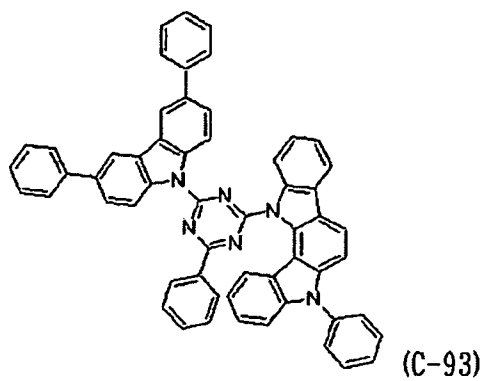
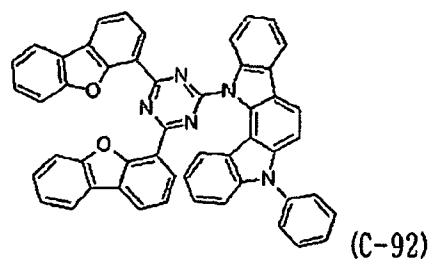
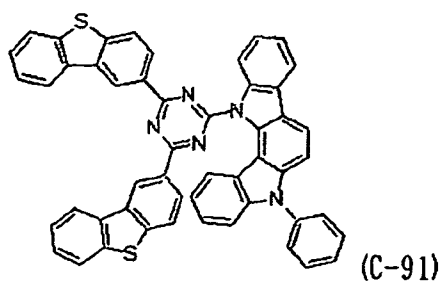
(C-88)



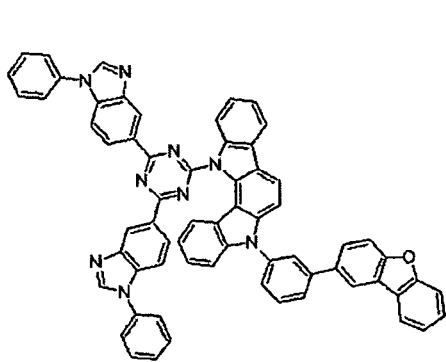
(C-89)



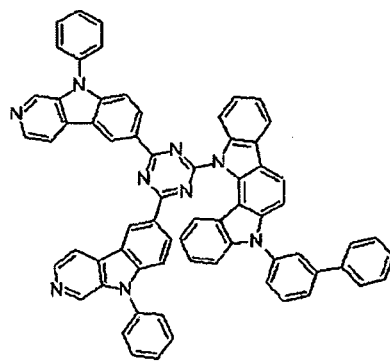
(C-90)



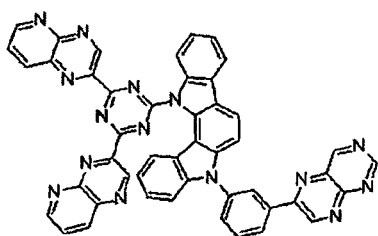




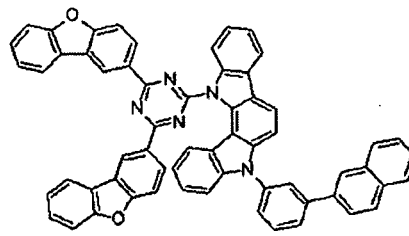
(C-98)



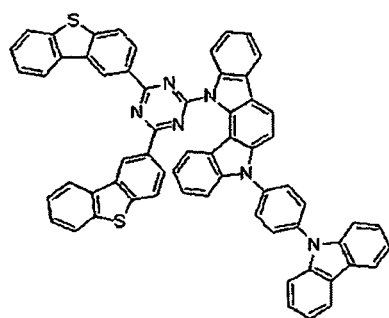
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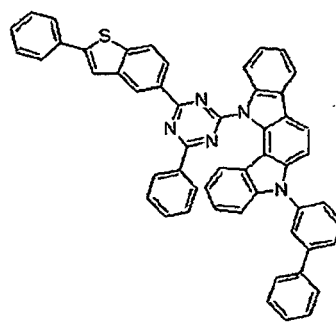
(C-100)



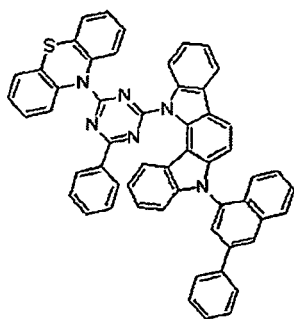
(C-101)



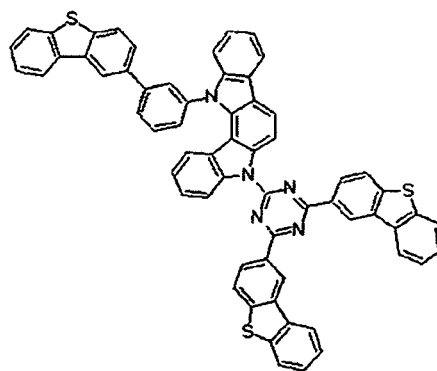
(C-102)



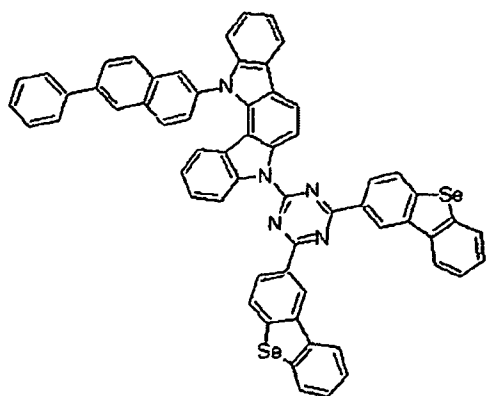
(C-103)



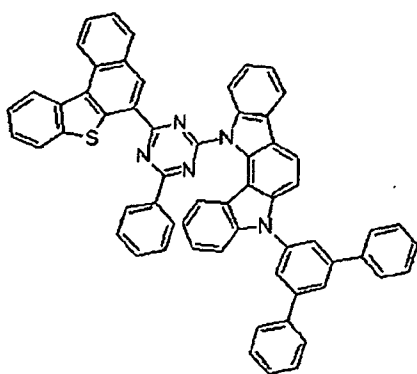
(C-104)



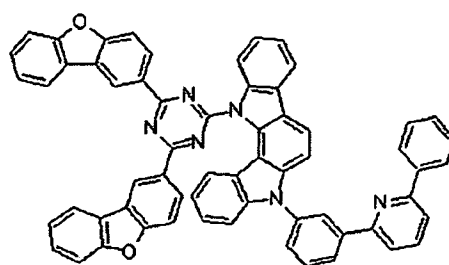
(C-105)



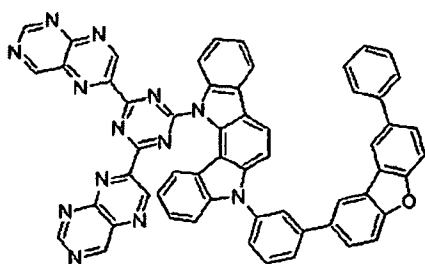
(C-106)



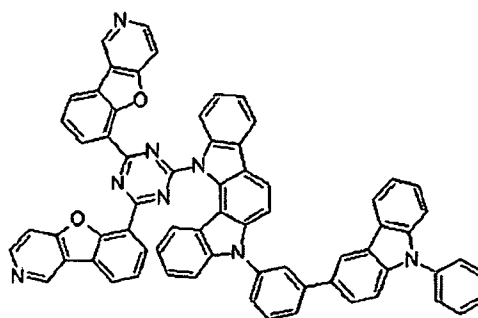
(C-107)



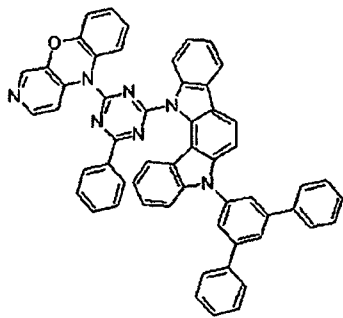
(C-108)



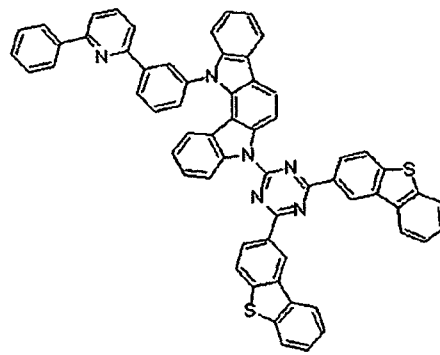
(C-109)



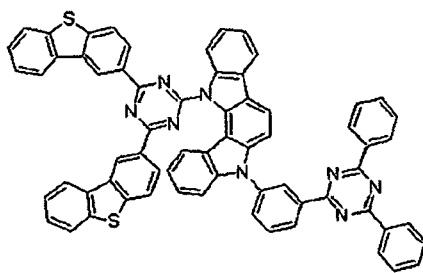
(C-110)



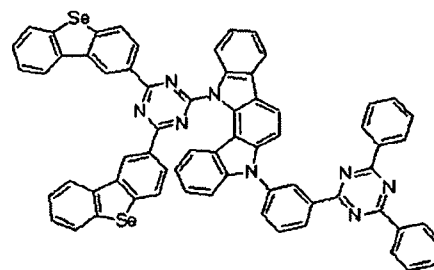
(C-111)



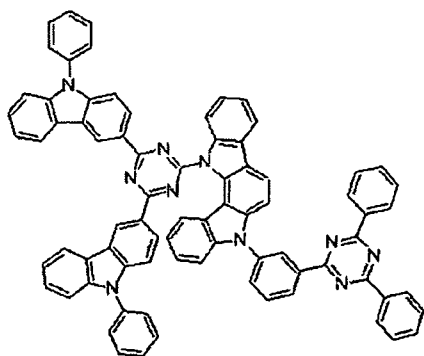
(C-112)



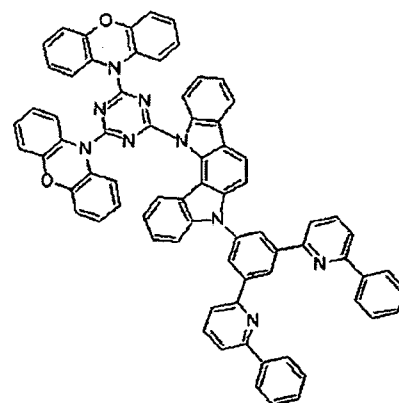
(C-113)



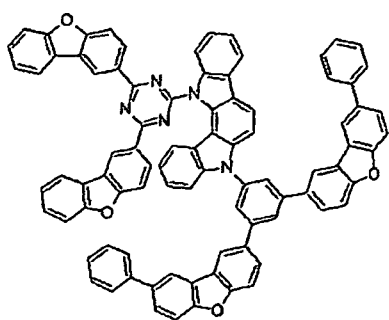
(C-114)



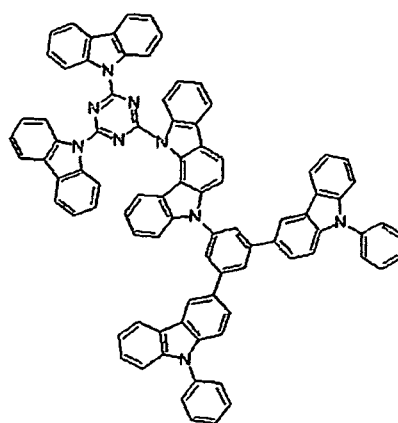
(C-115)



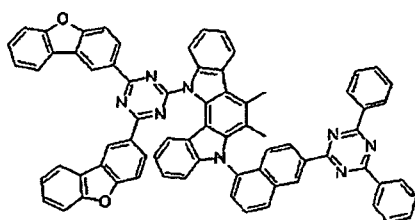
(C-116)



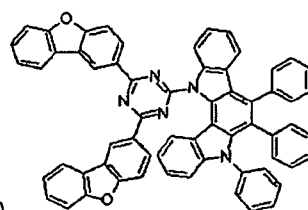
(C-117)



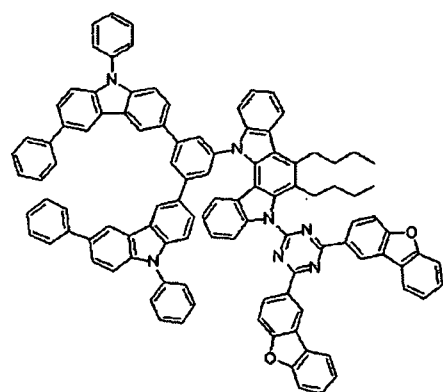
(C-118)



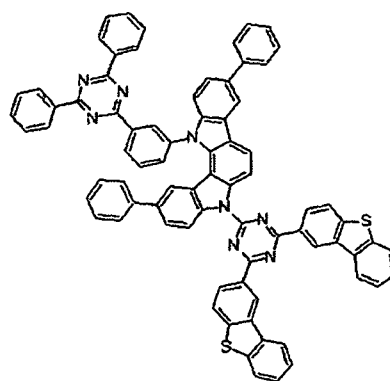
(C-119)



(C-120)

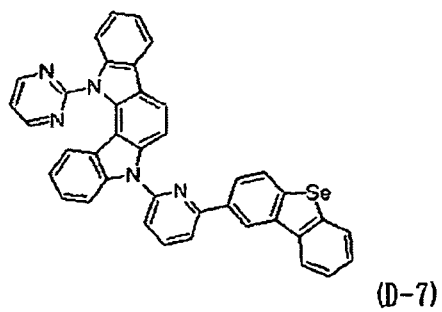
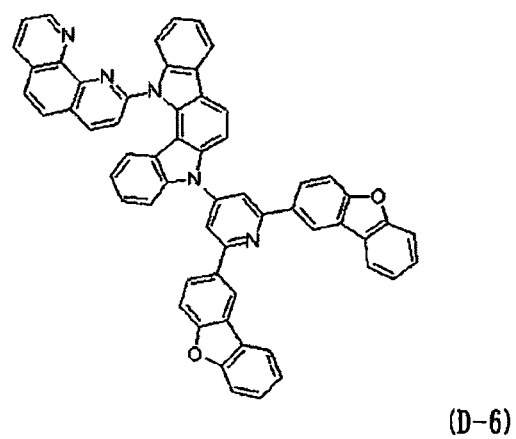
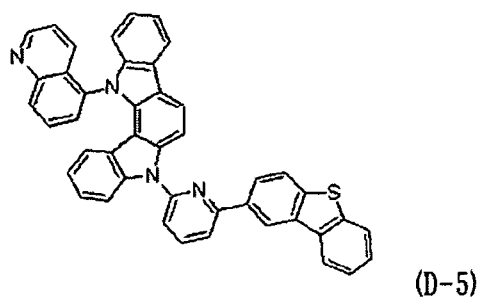
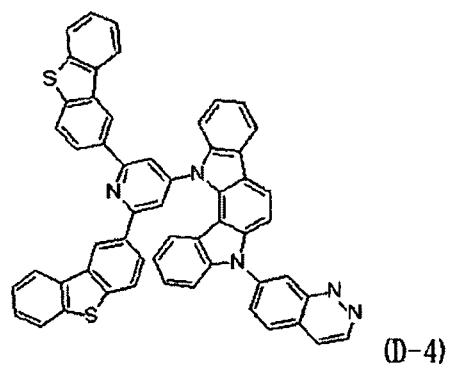
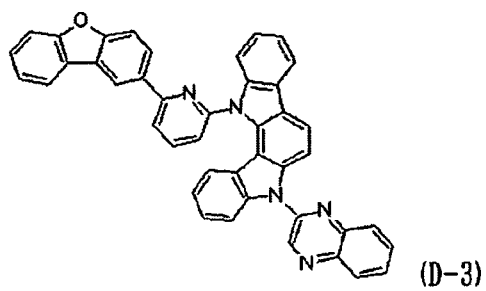
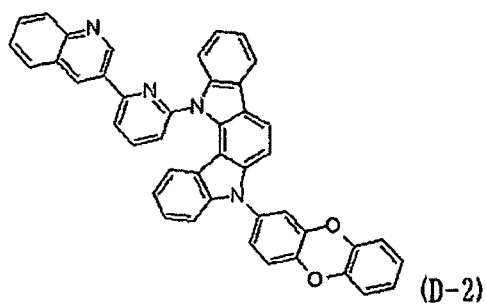
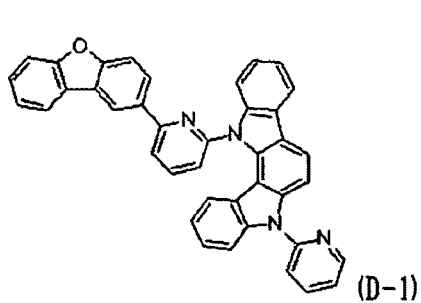


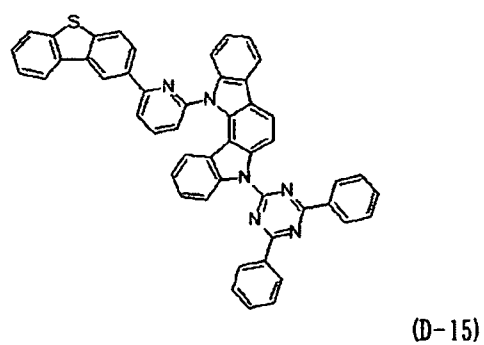
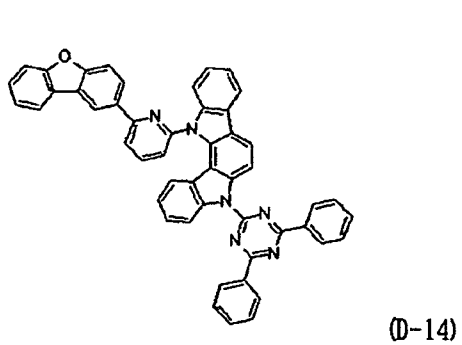
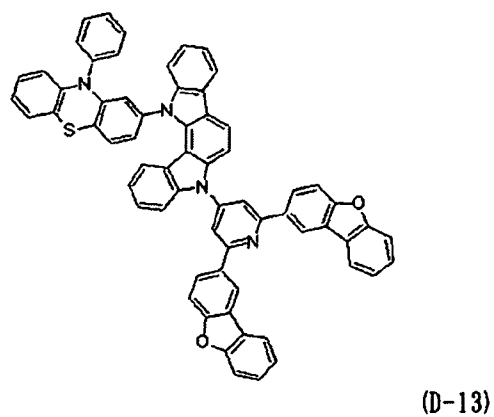
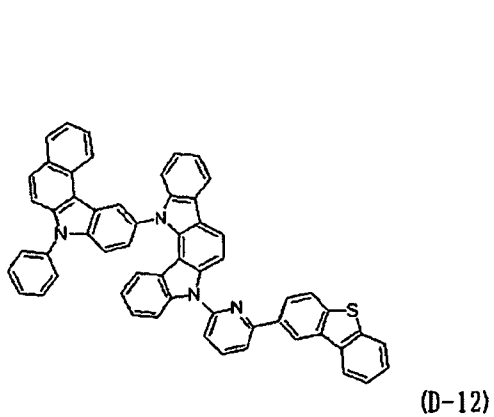
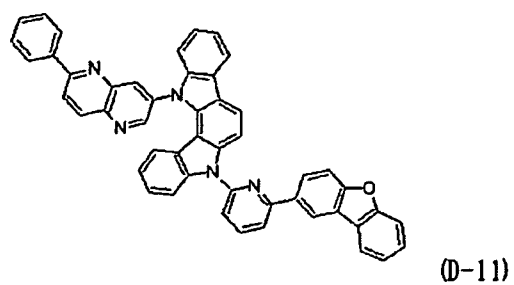
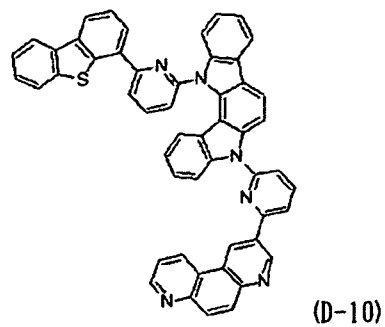
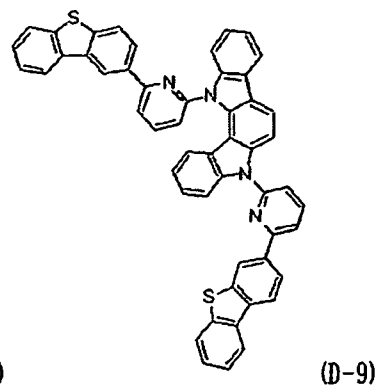
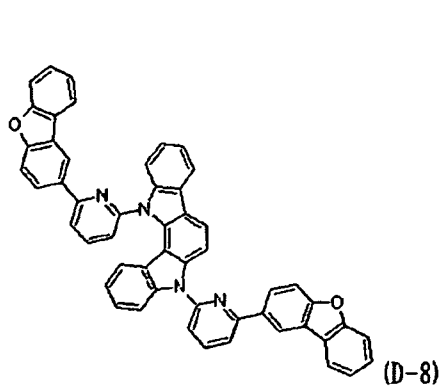
(C-121)

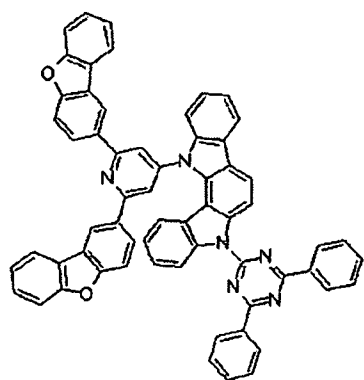


(C-122)

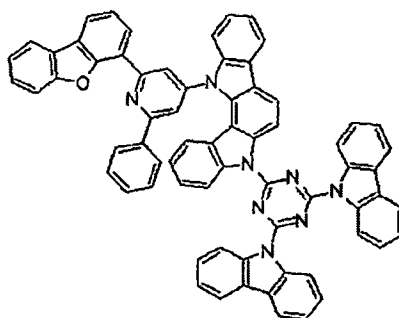
【0057】



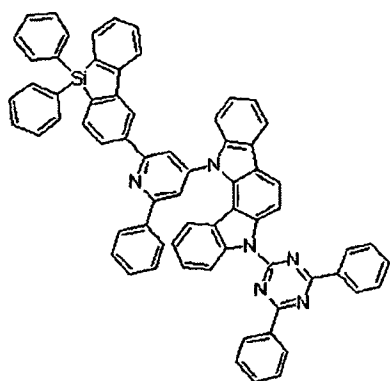




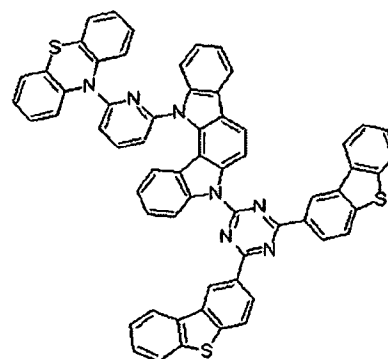
(D-16)



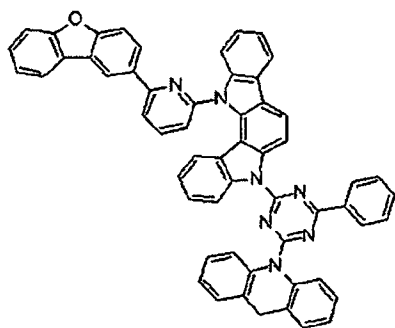
(D-17)



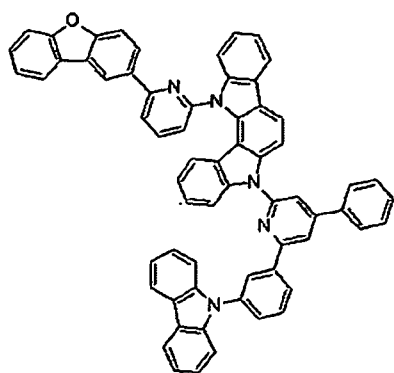
(D-18)



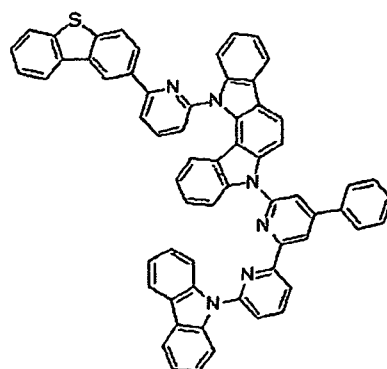
(D-19)



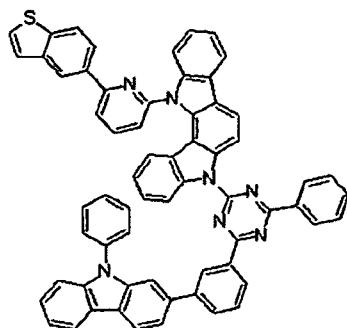
(D-20)



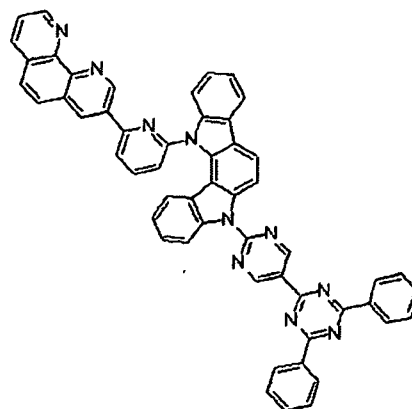
(D-21)



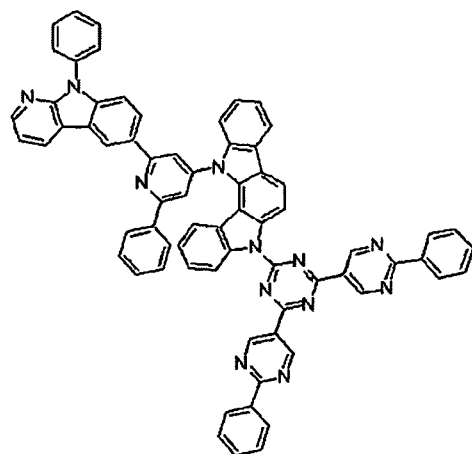
(D-22)



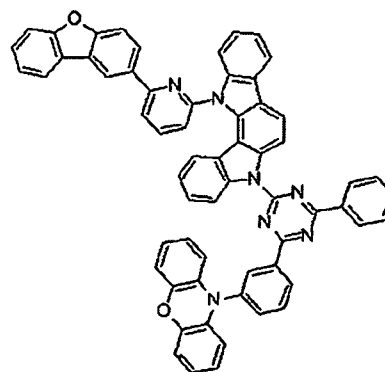
(D-23)



(D-24)

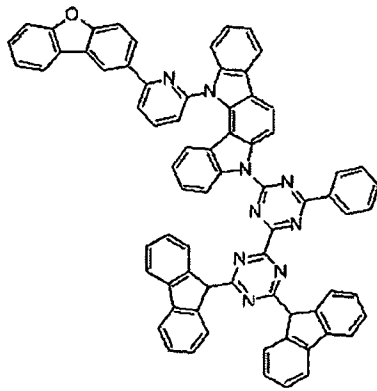


(D-25)

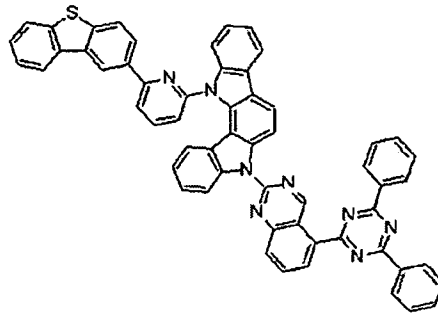


(D-26)

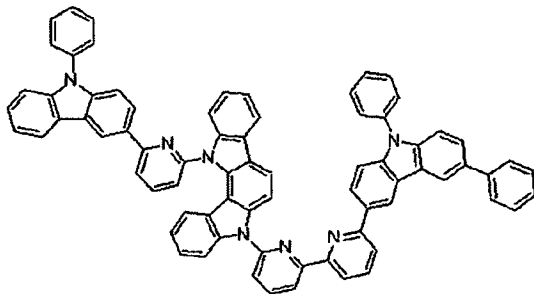




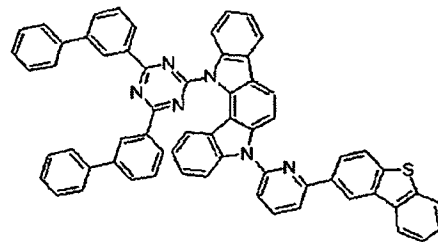
(D-27)



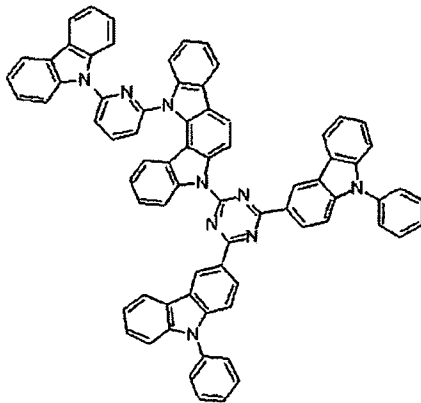
(D-28)



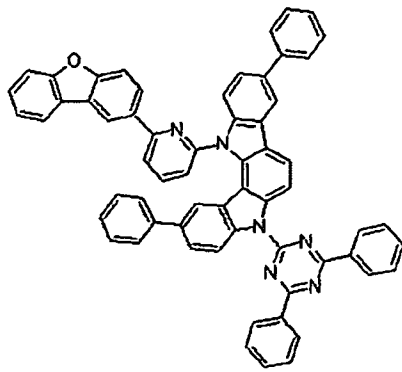
(D-29)



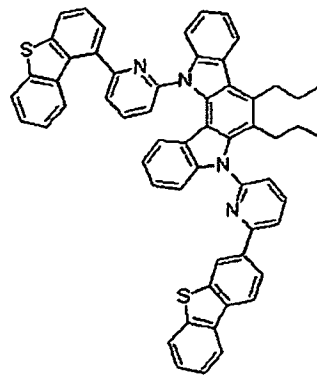
(D-30)



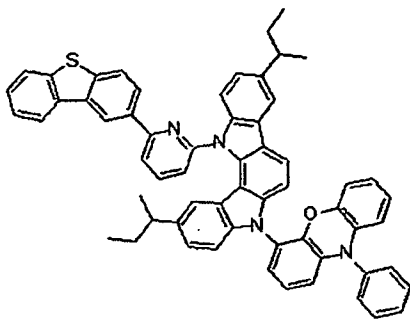
(D-31)



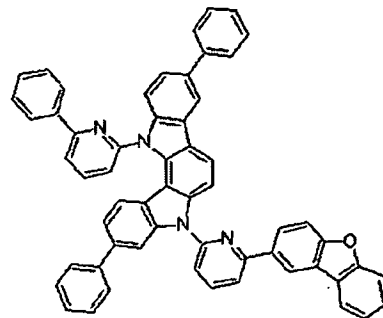
(D-32)



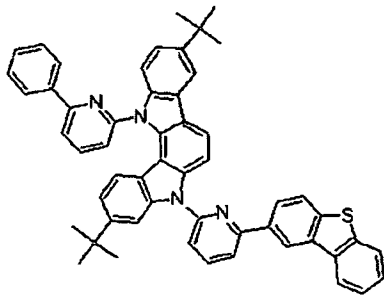
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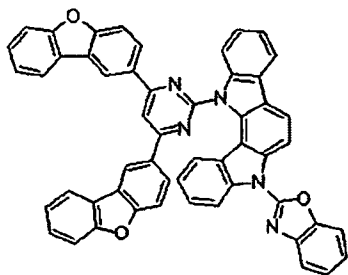
(D-34)



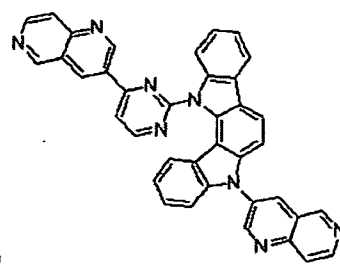
(D-35)



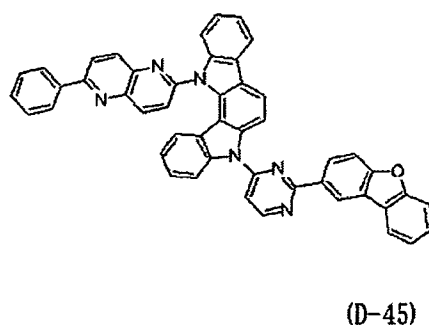
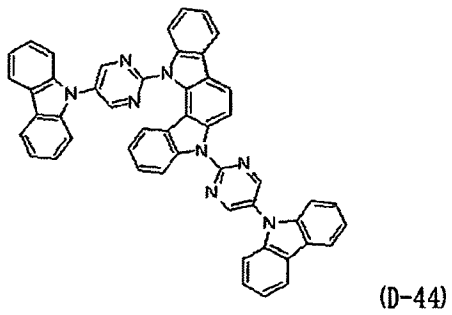
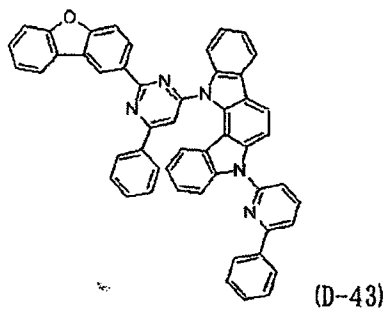
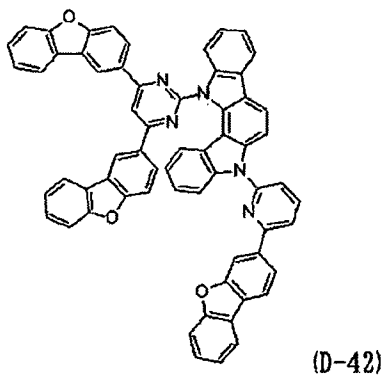
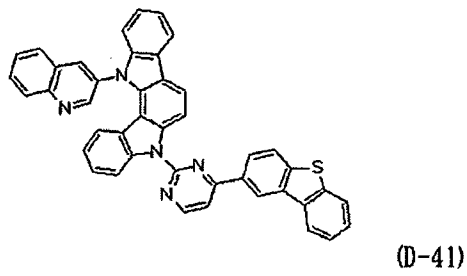
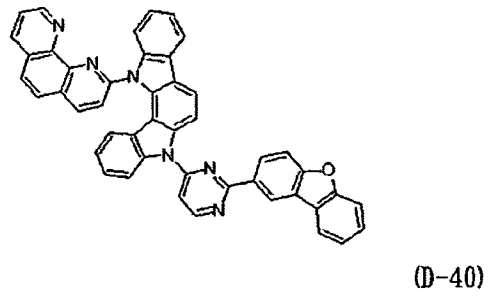
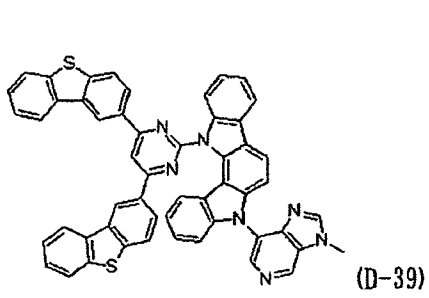
(D-36)

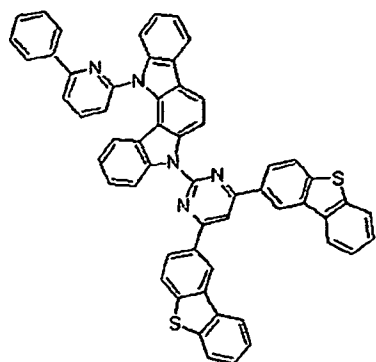


(D-37)

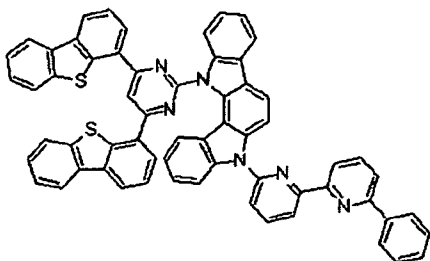


(D-38)

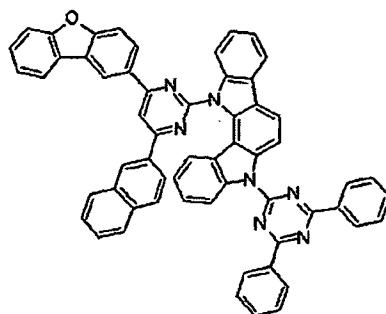




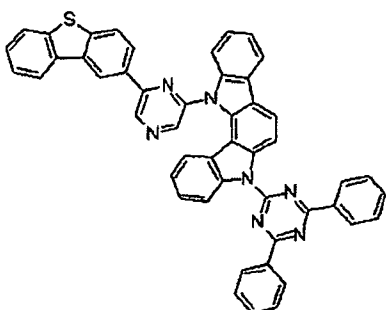
(D-46)



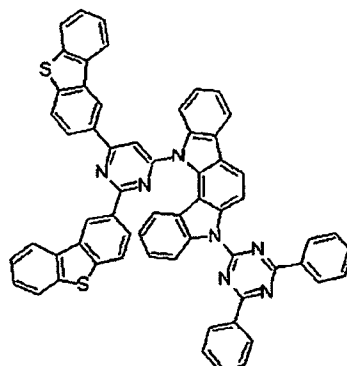
(D-47)



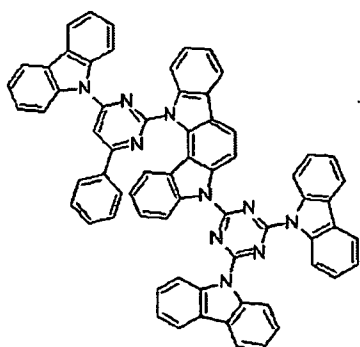
(D-48)



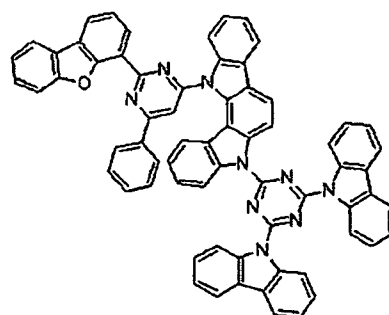
(D-49)



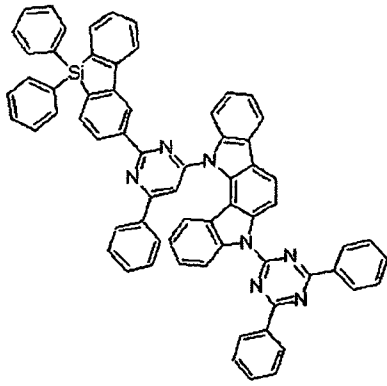
(D-50)



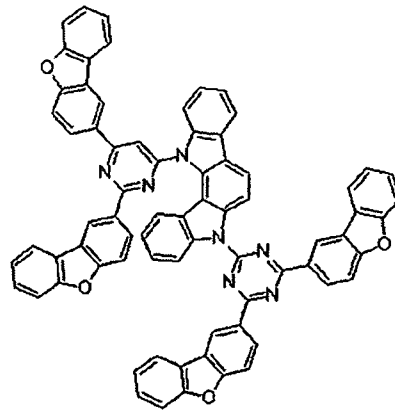
(D-51)



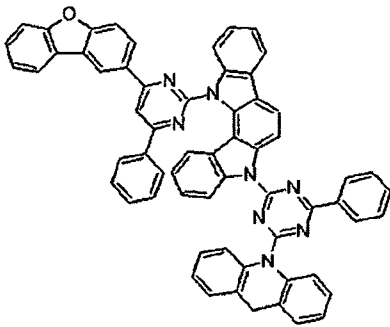
(D-52)



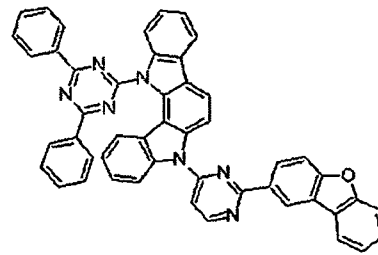
(D-53)



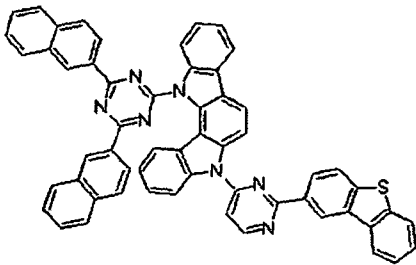
(D-54)



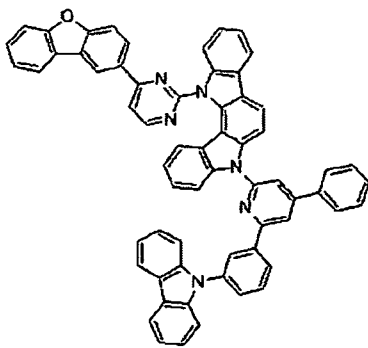
(D-55)



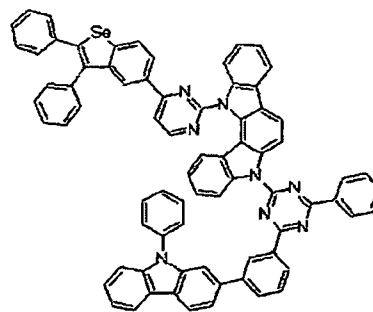
(D-56)



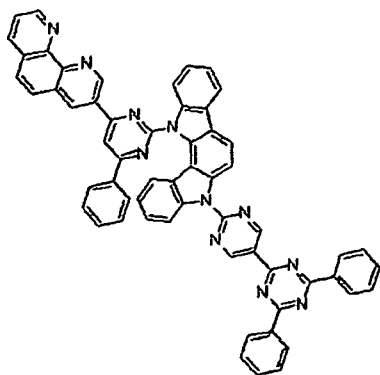
(D-57)



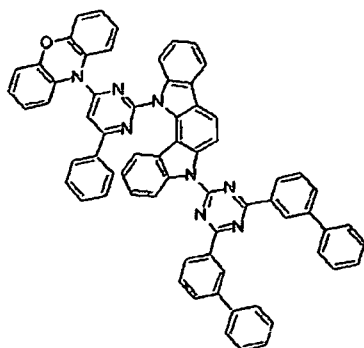
(D-58)



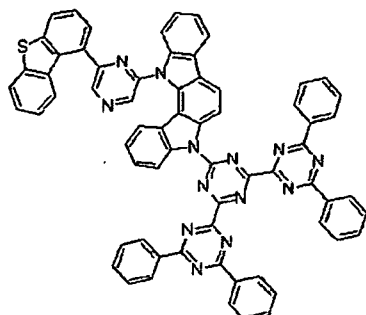
(D-59)



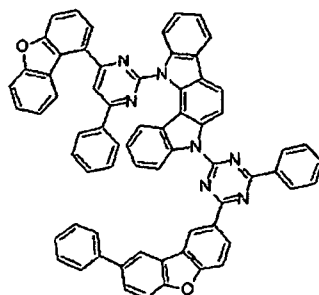
(D-60)



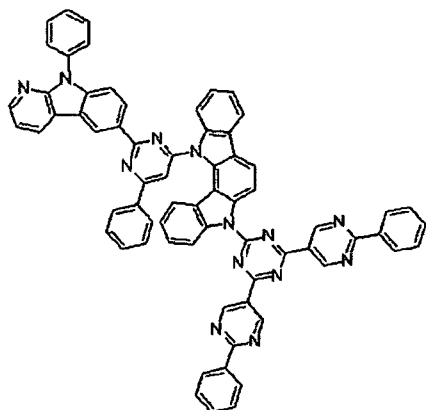
(D-61)



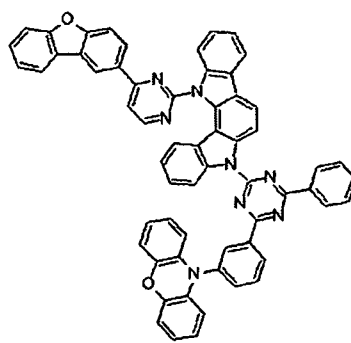
(D-62)



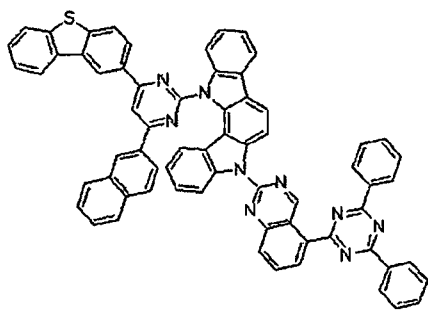
(D-63)



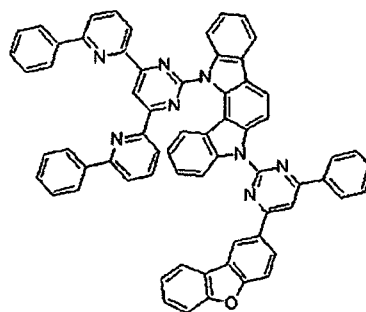
(D-64)



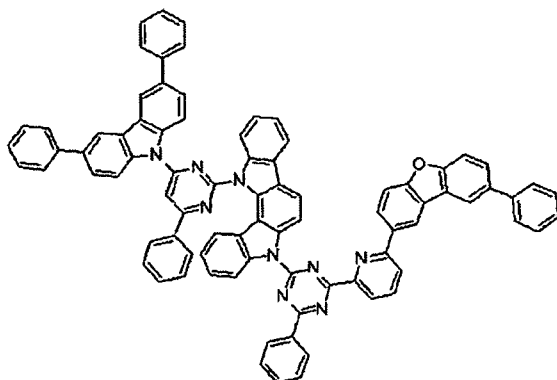
(D-65)



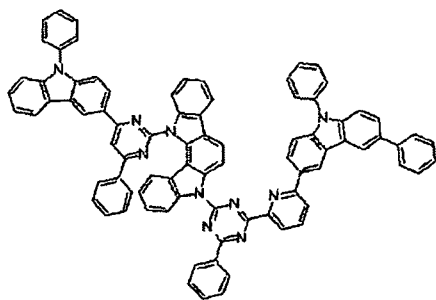
(D-66)



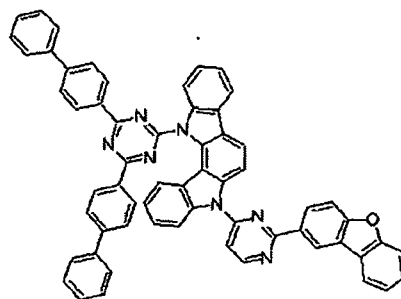
(D-67)



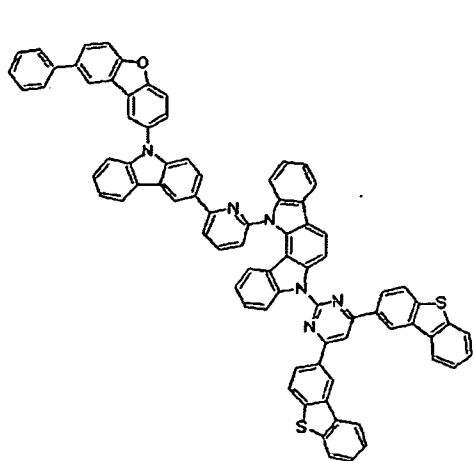
(D-68)



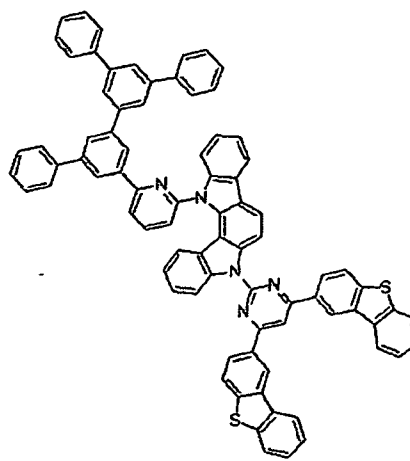
(D-69)



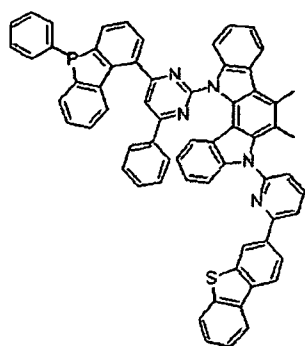
(D-70)



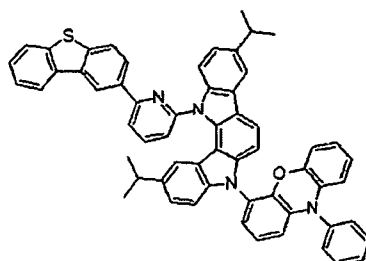
(D-71)



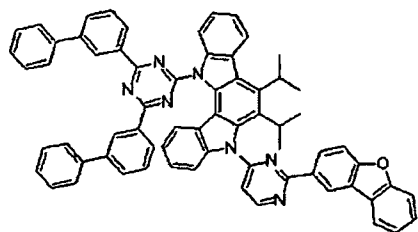
(D-72)



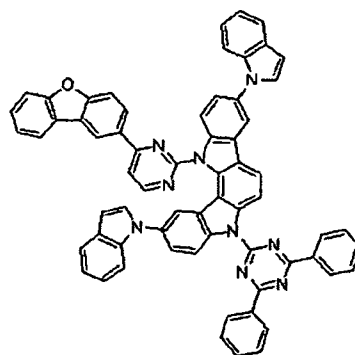
(D-73)



(D-74)

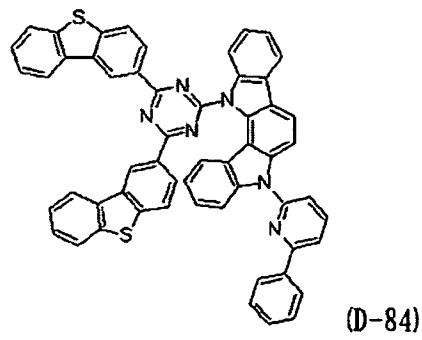
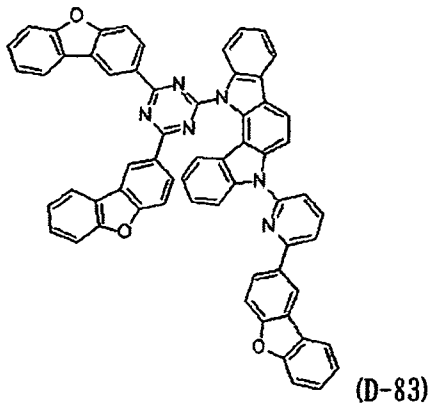
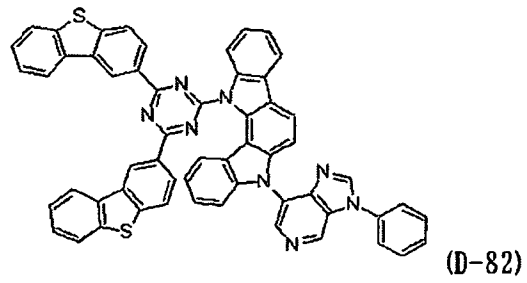
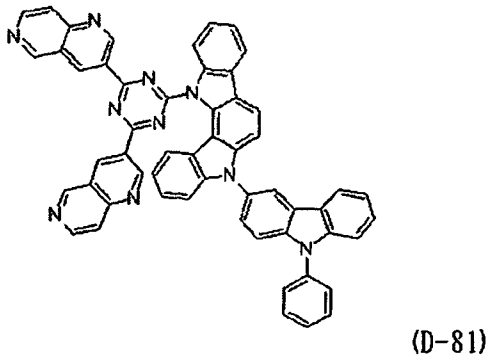
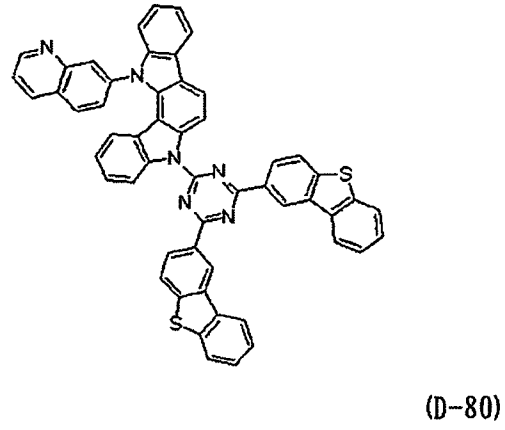
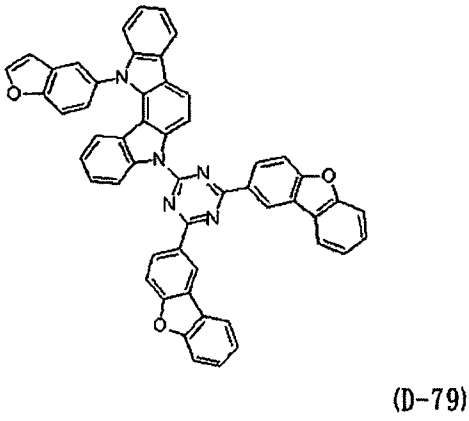
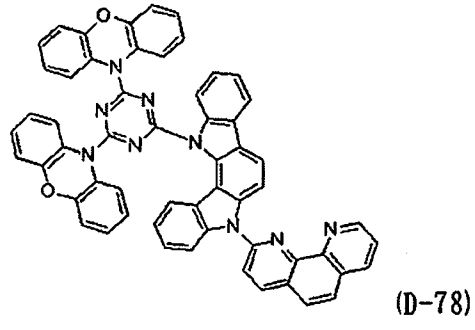
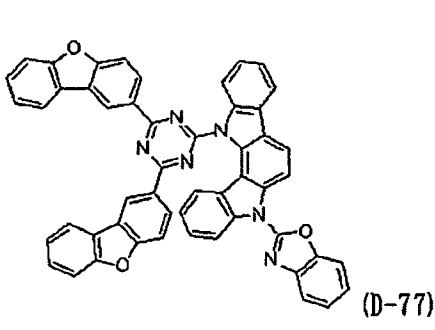


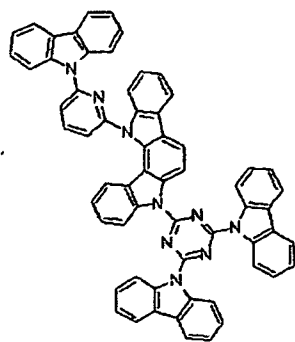
(D-75)



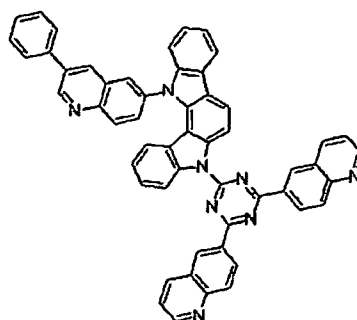
(D-76)



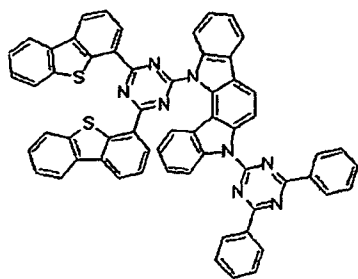




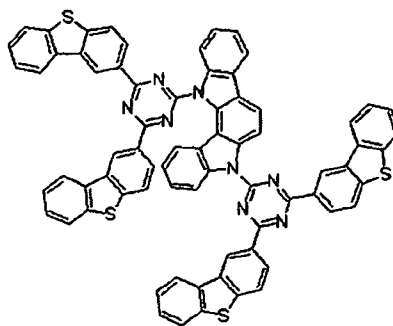
(D-85)



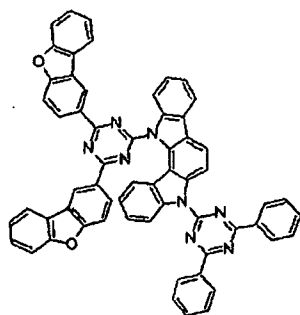
(D-86)



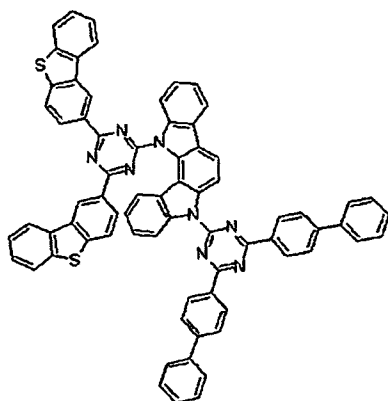
(D-87)



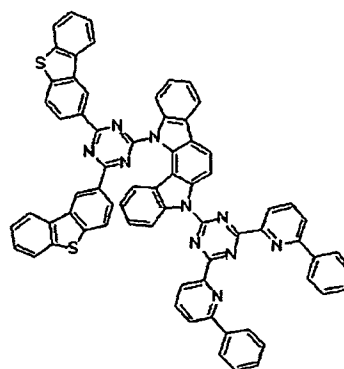
(D-88)



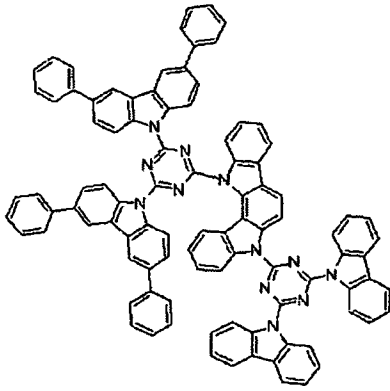
(D-89)



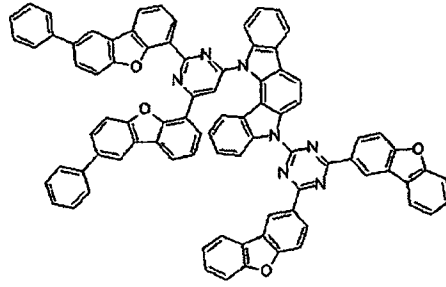
(D-90)



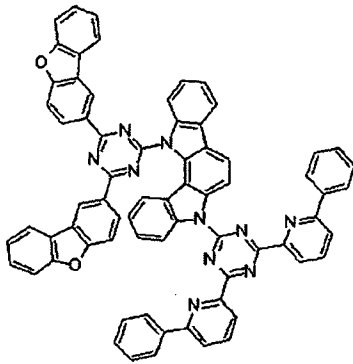
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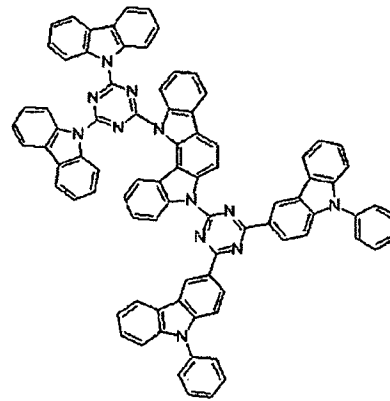
(D-92)



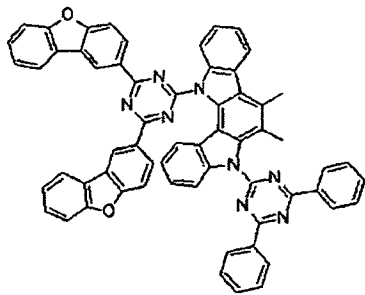
(D-93)



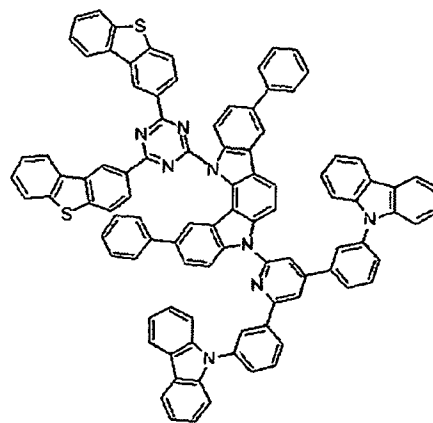
(D-94)



(D-95)

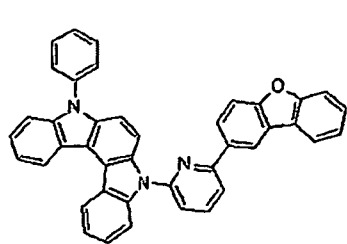


(D-96)

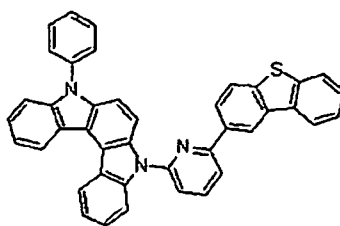


(D-97)

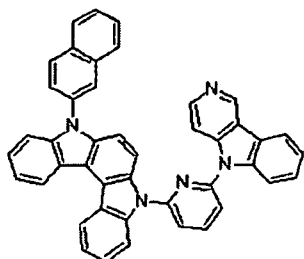
【0058】



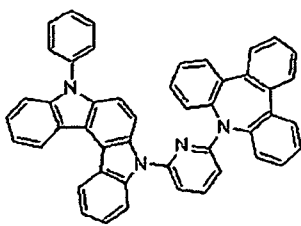
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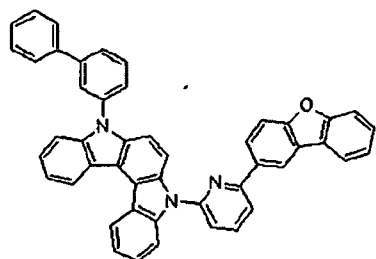
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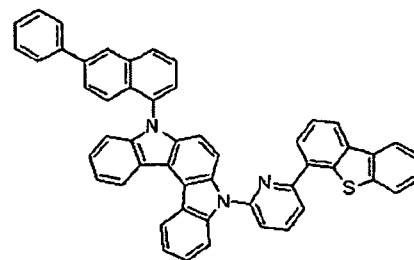
(E-3)



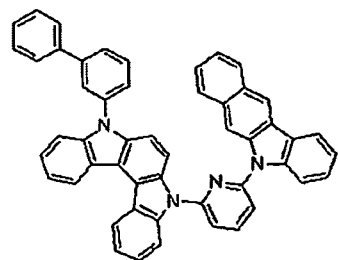
(E-4)



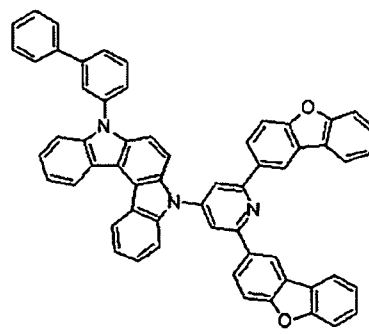
(E-5)



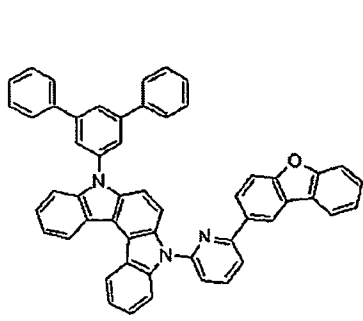
(E-6)



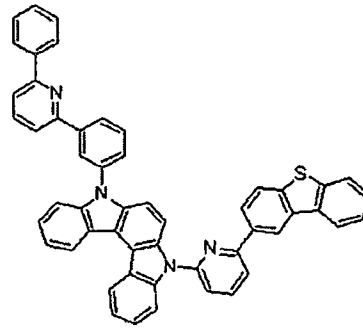
(E-7)



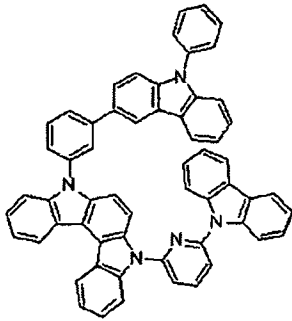
(E-8)



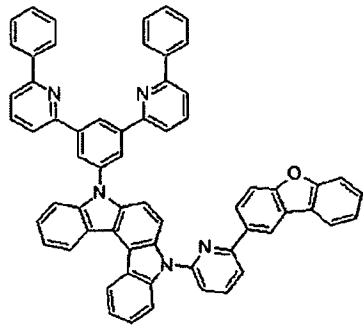
(E-9)



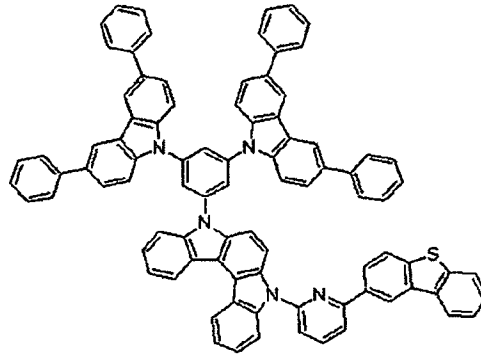
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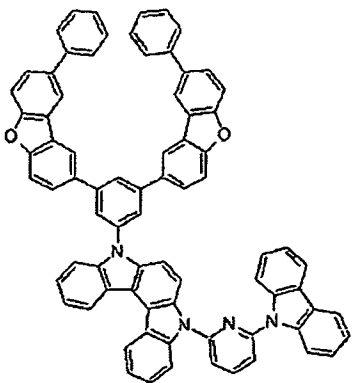
(E-11)



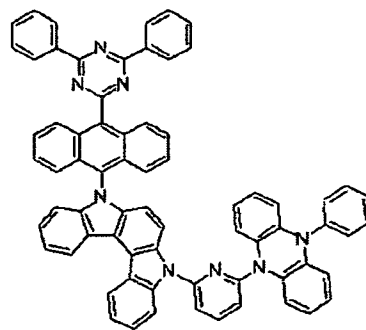
(E-12)



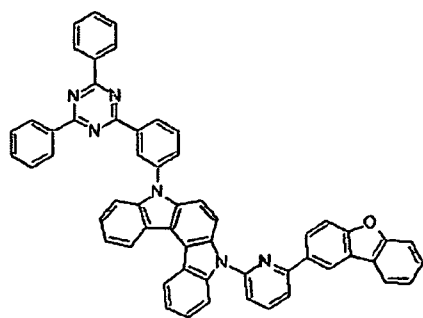
(E-13)



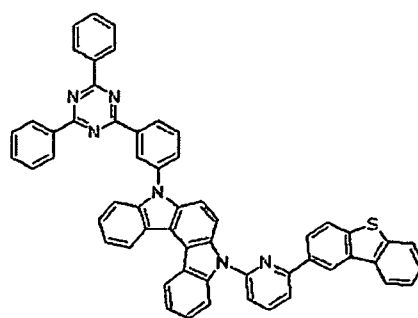
(E-14)



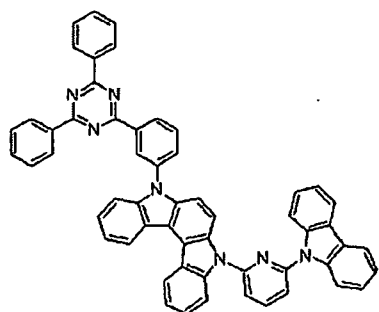
(E-15)



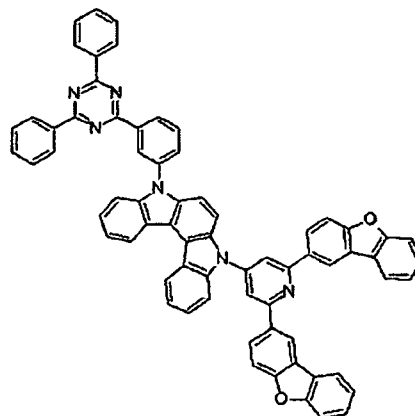
(E-16)



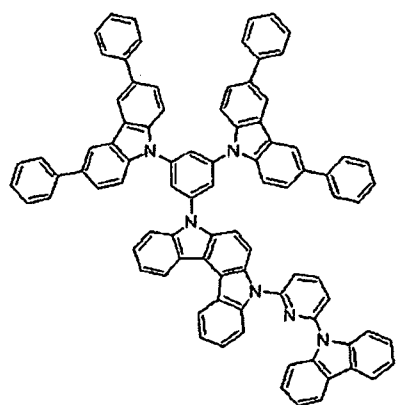
(E-17)



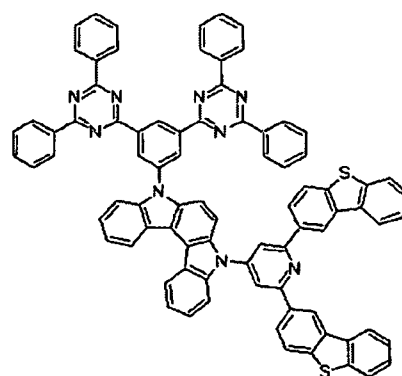
(E-18)



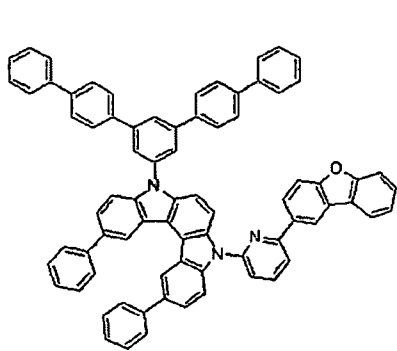
(E-19)



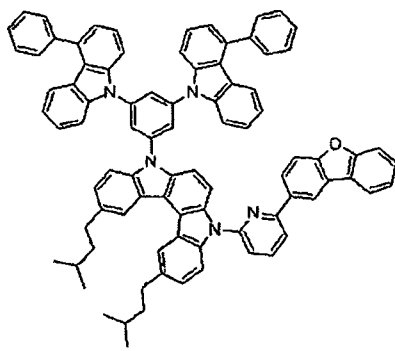
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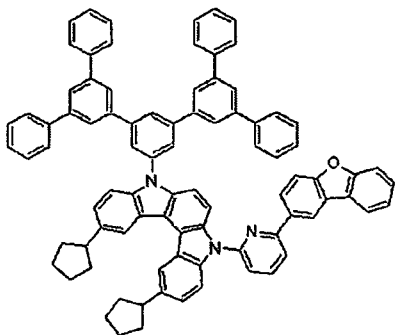
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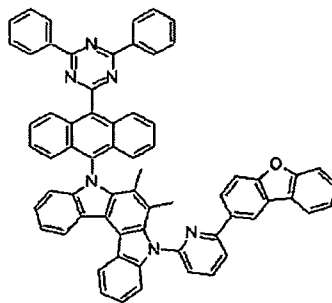
(E-22)



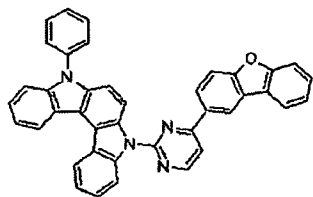
(E-23)



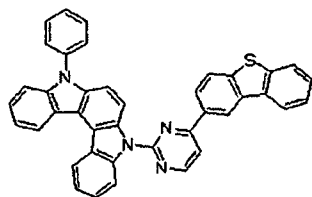
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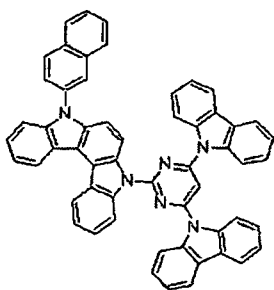
(E-25)



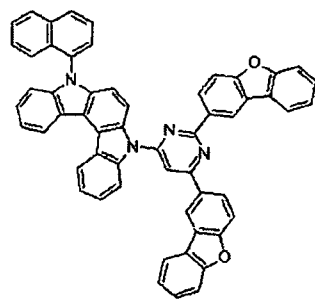
(E-26)



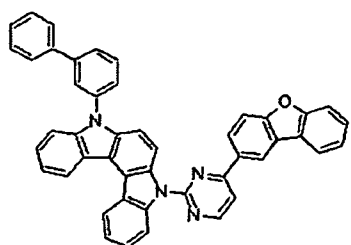
(E-27)



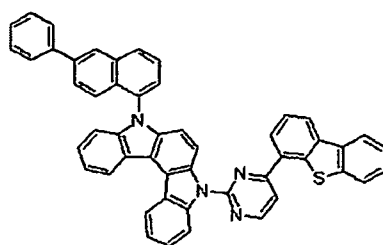
(E-28)



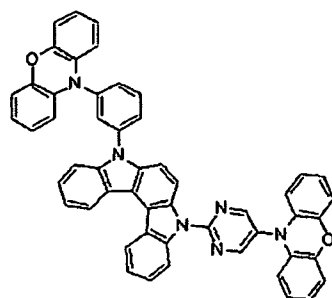
(E-29)



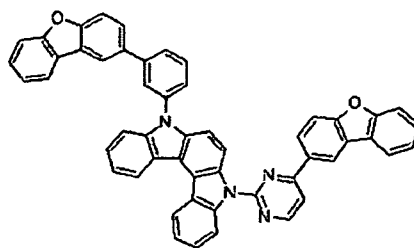
(E-30)



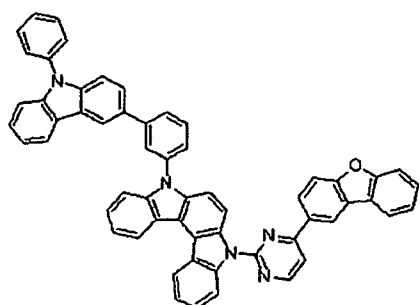
(E-31)



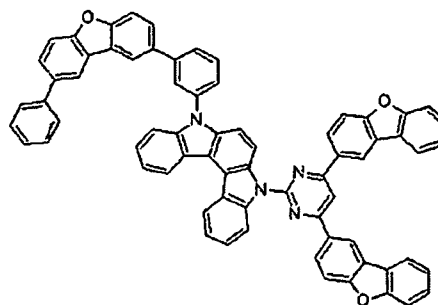
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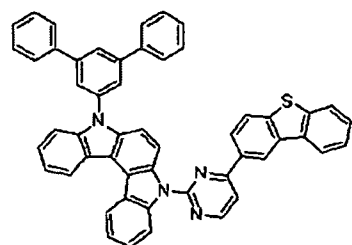
(E-33)



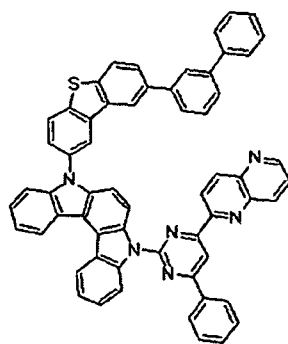
(E-34)



(E-35)

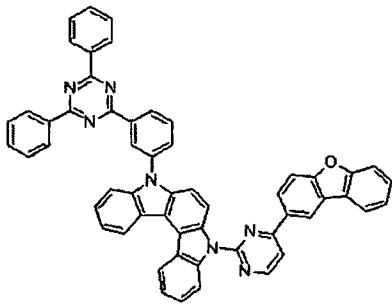


(E-36)

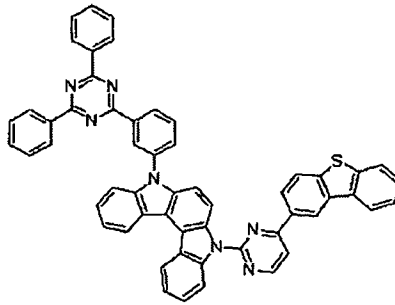


(E-37)

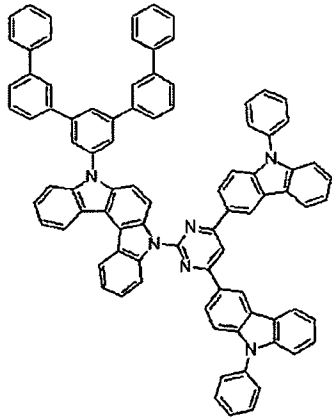




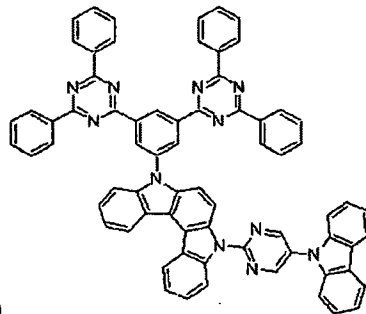
(E-38)



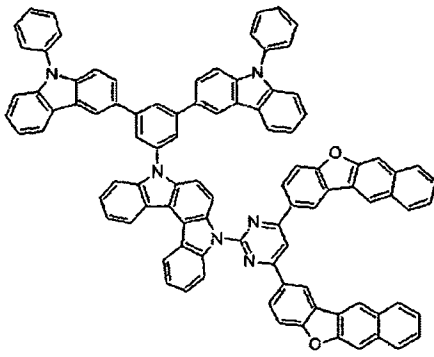
(E-39)



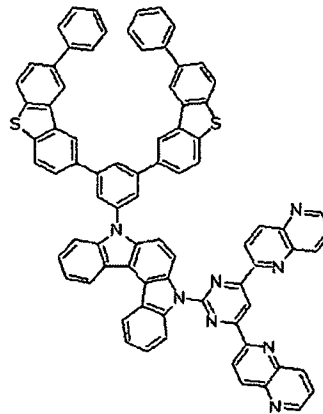
(E-40)



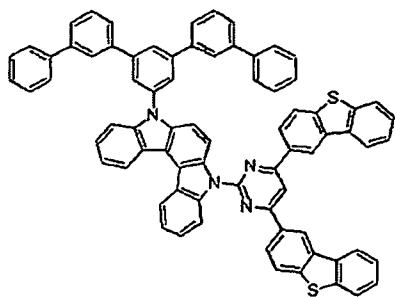
(E-41)



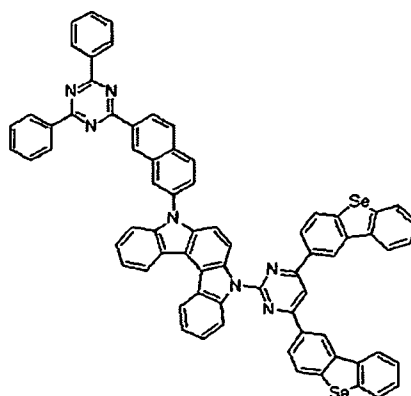
(E-42)



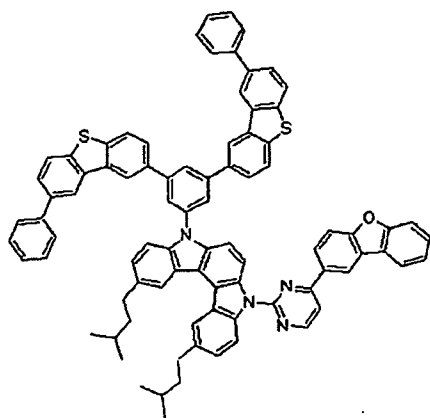
(E-43)



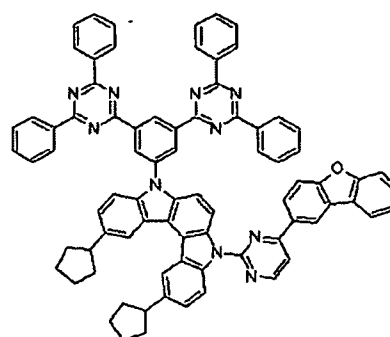
(E-44)



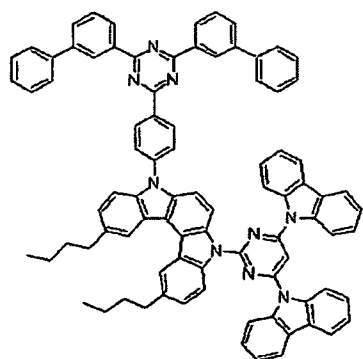
(E-45)



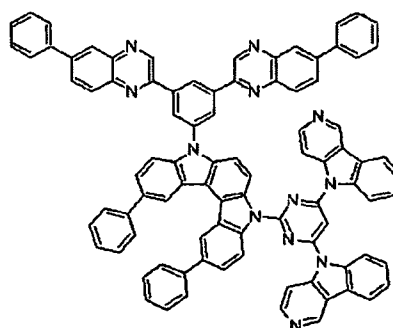
(E-46)



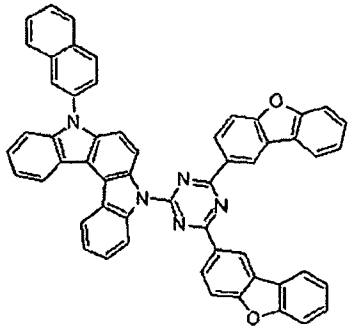
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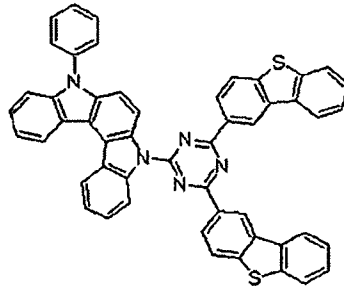
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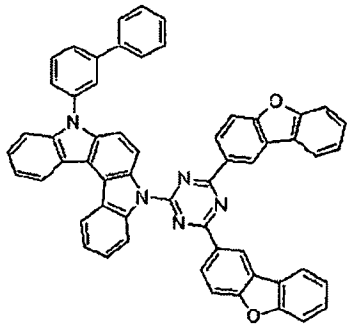
(E-49)



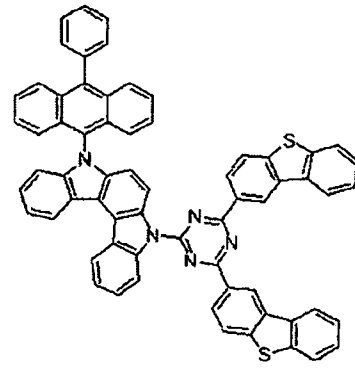
(E-50)



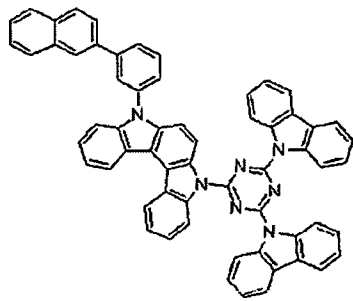
(E-51)



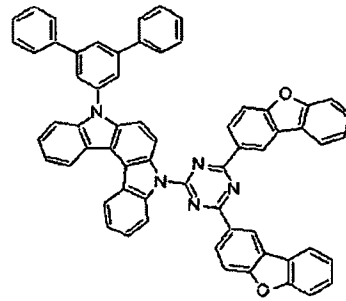
(E-52)



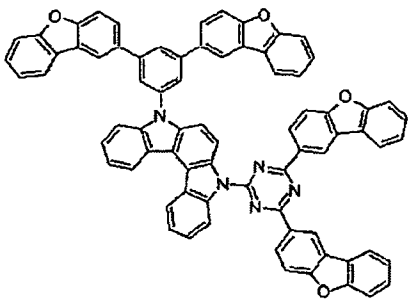
(E-53)



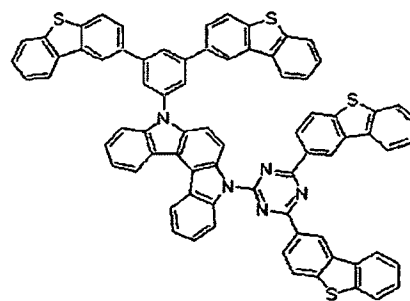
(E-54)



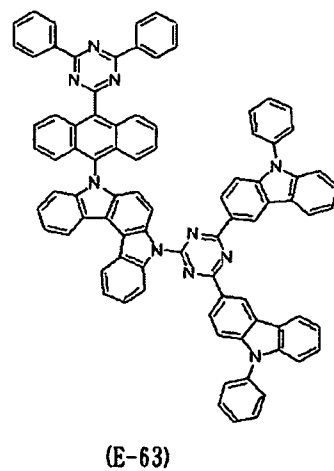
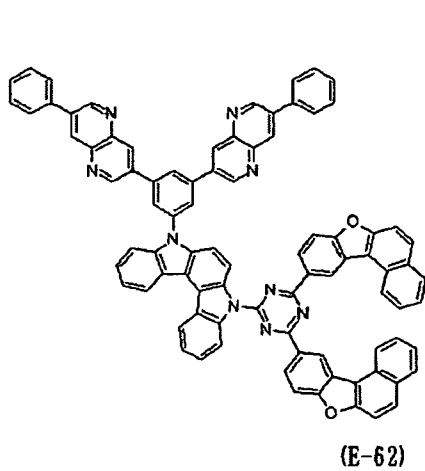
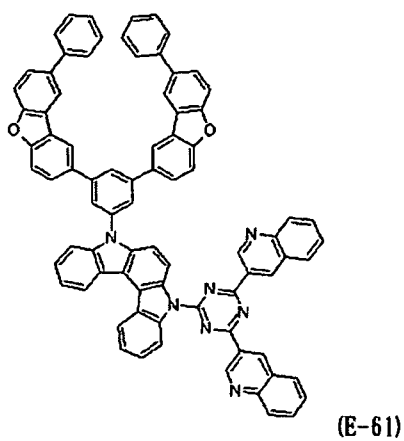
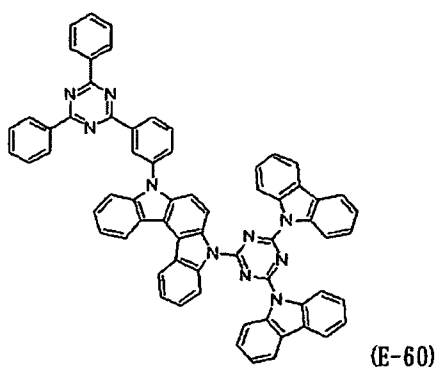
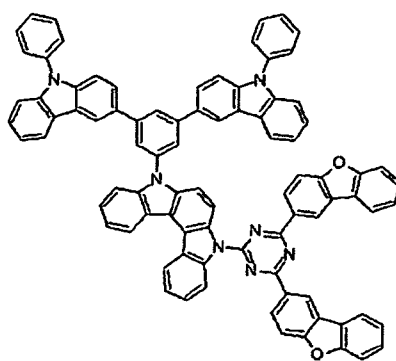
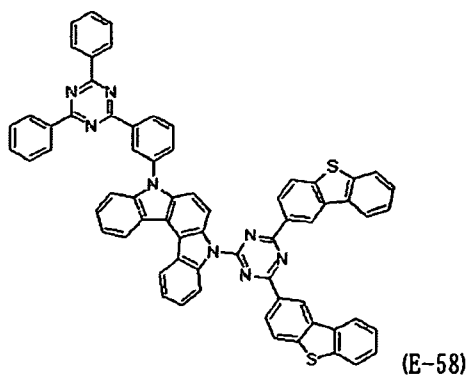
(E-55)

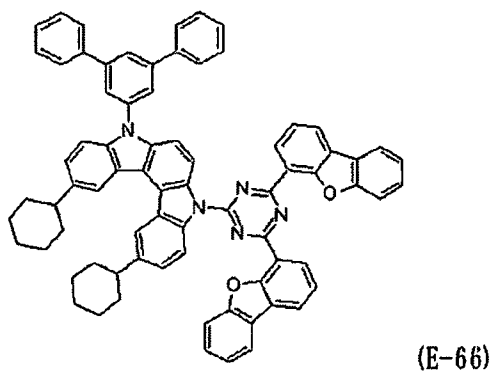
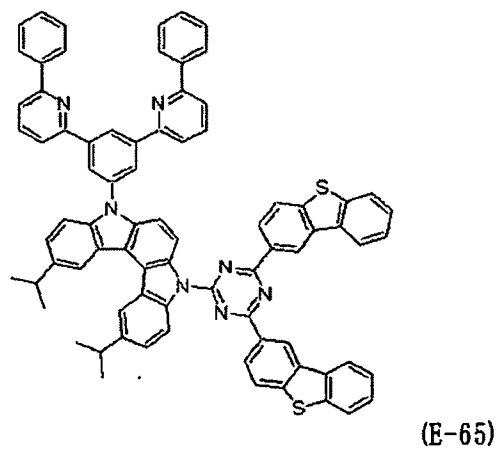
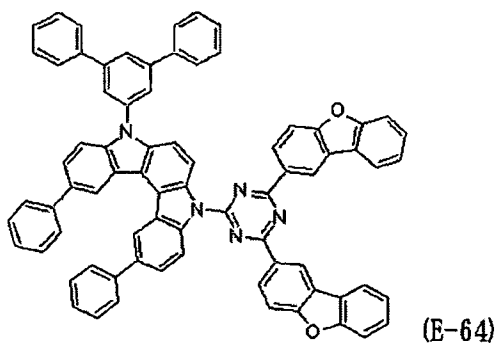


(E-56)

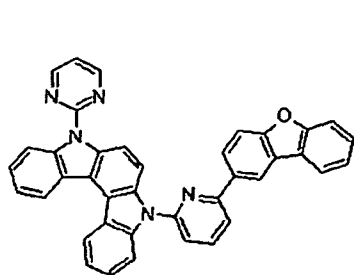


(E-57)

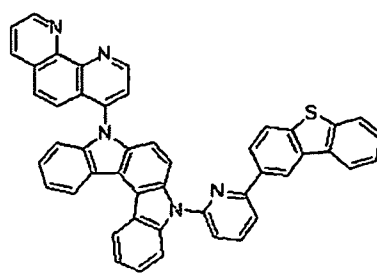




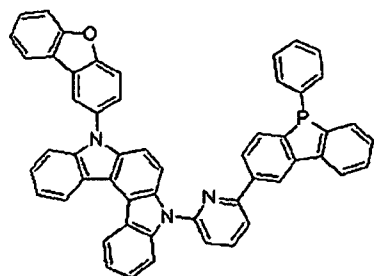
【0059】



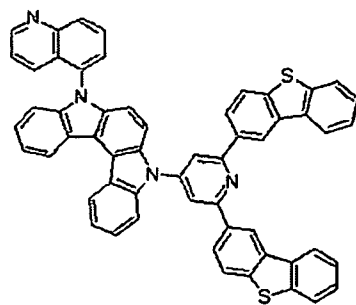
(F-1)



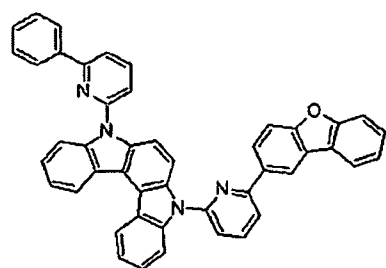
(F-2)



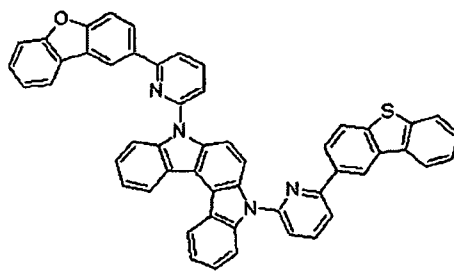
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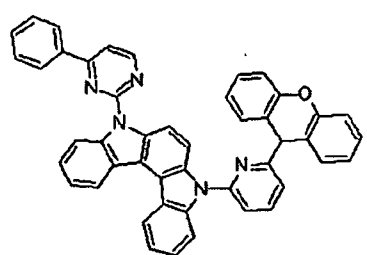
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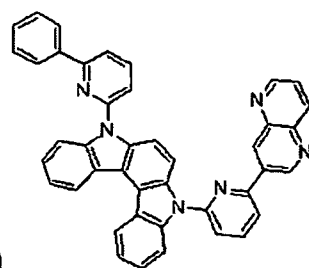
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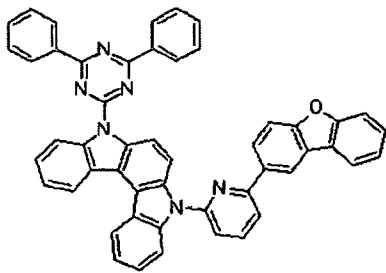
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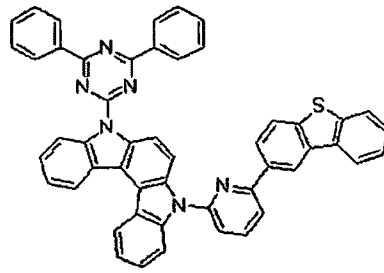
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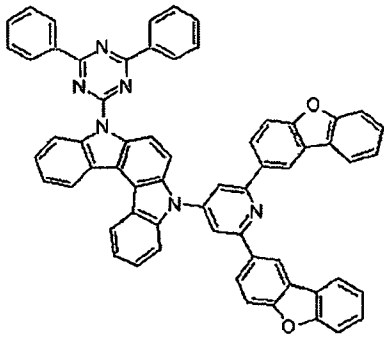
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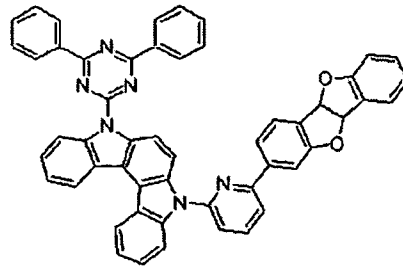
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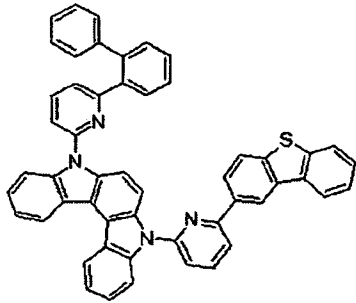
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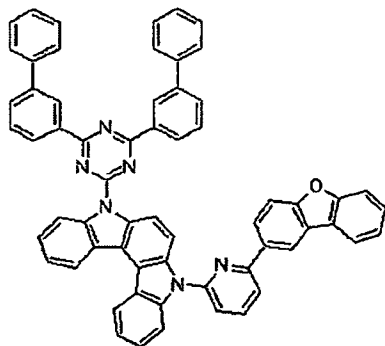
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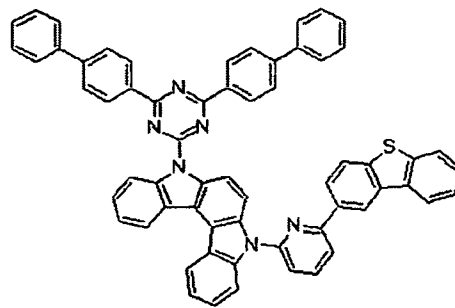
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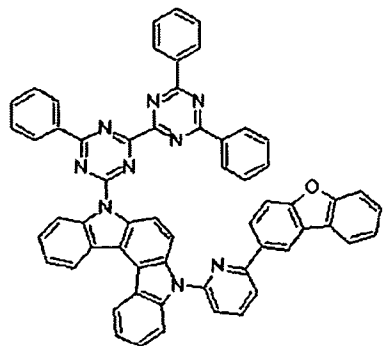
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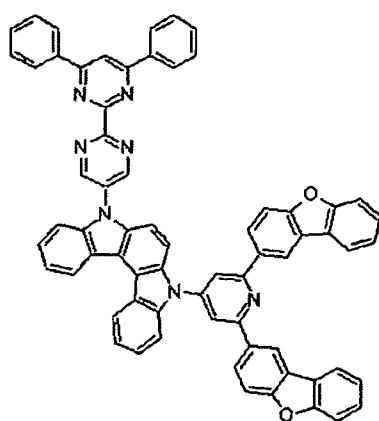
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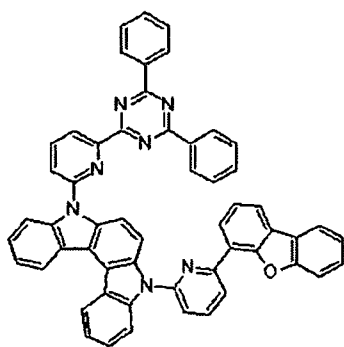
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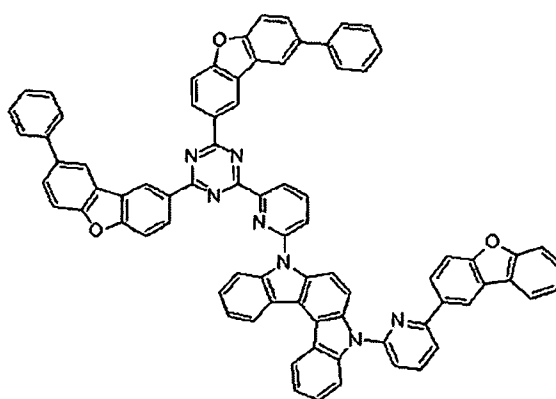
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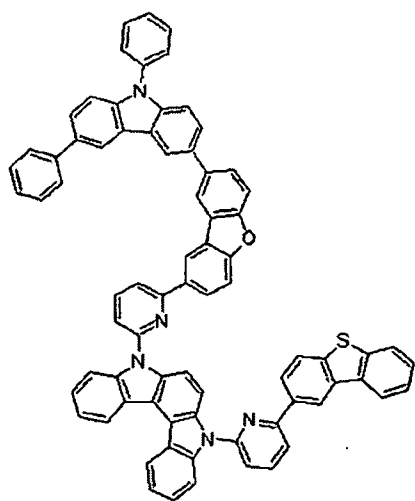
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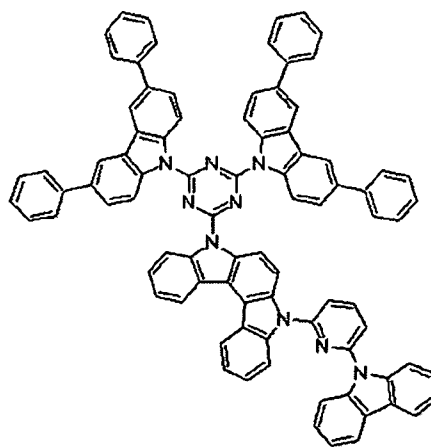
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(F-19)

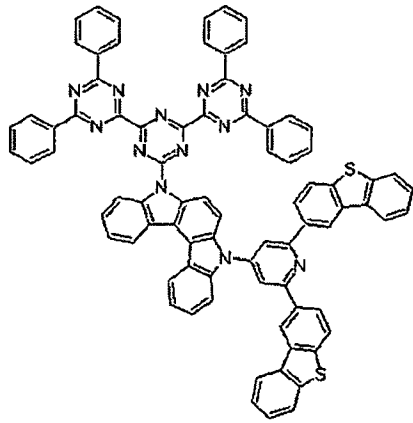


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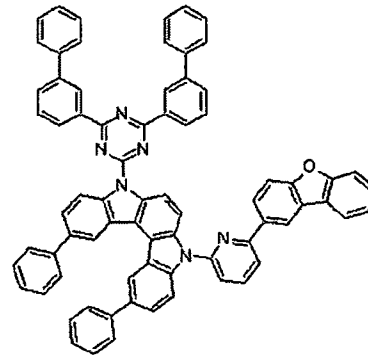


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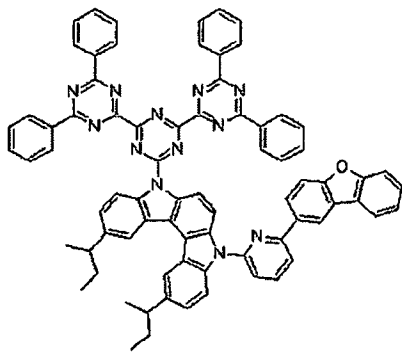




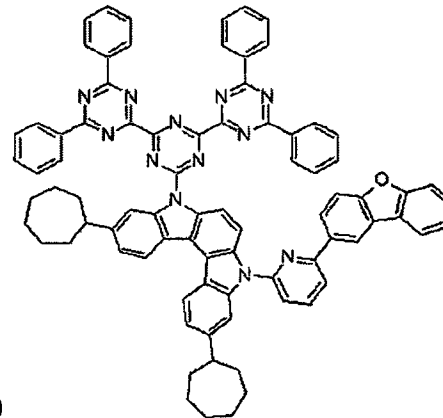
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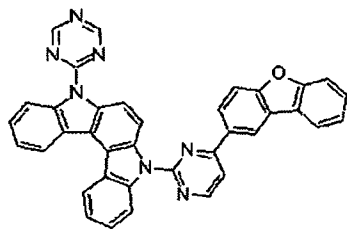
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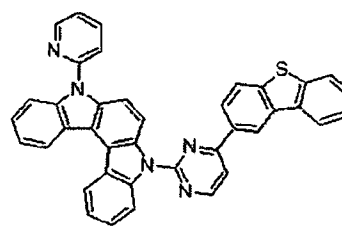
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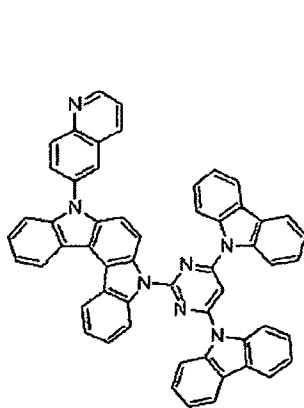
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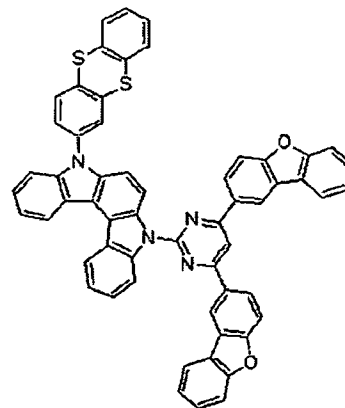
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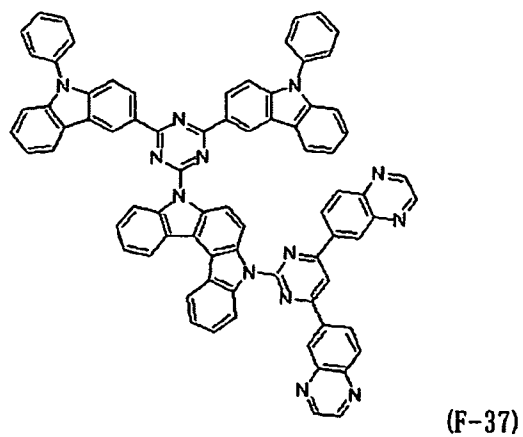
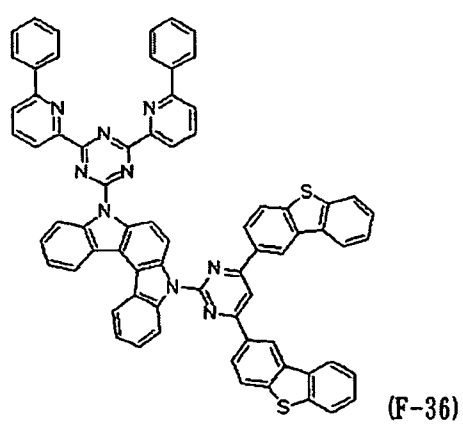
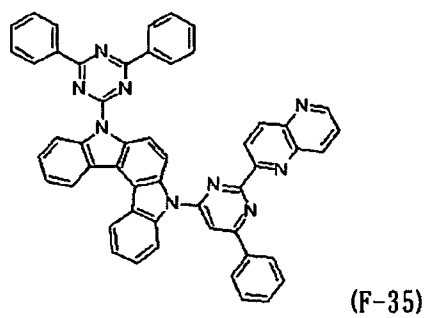
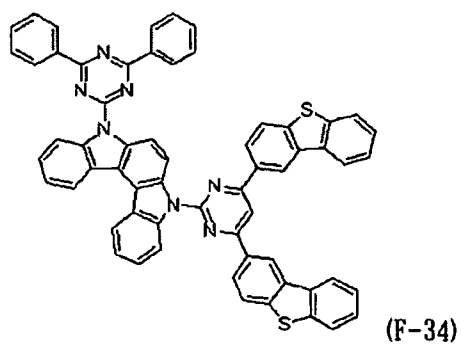
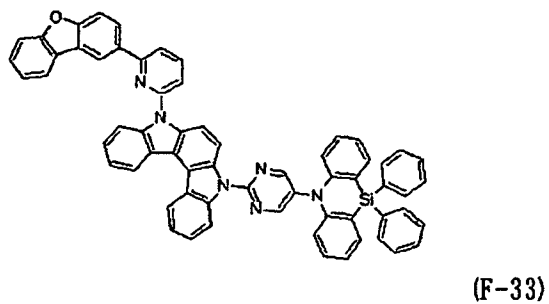
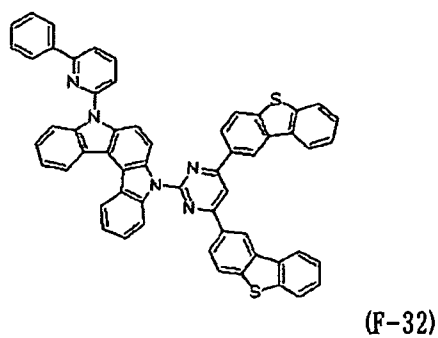
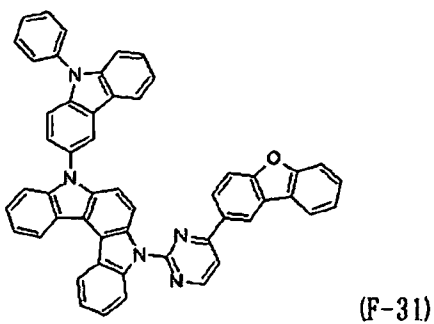
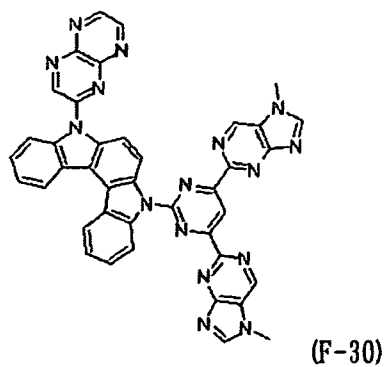
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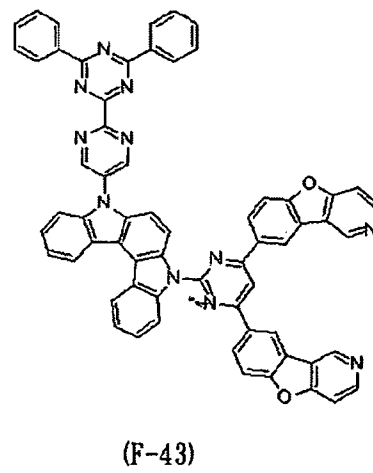
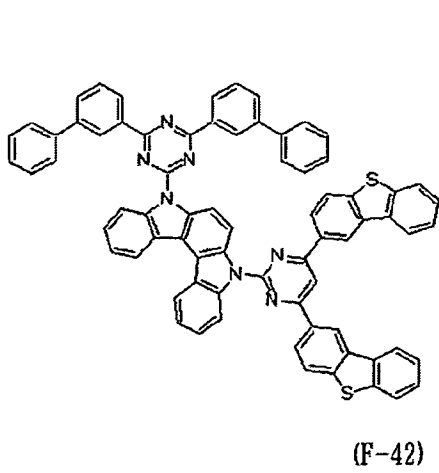
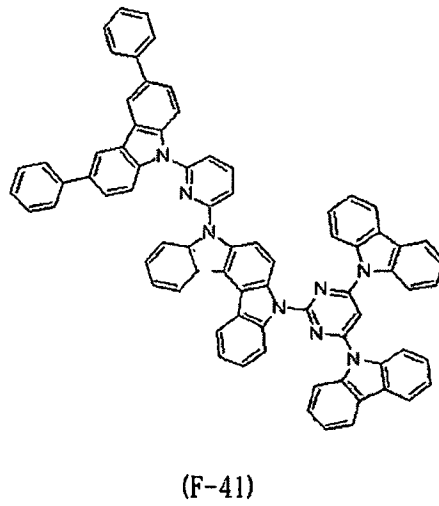
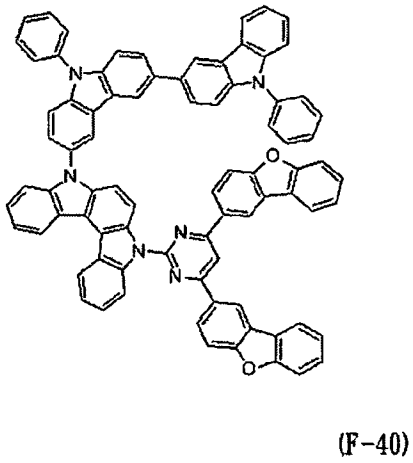
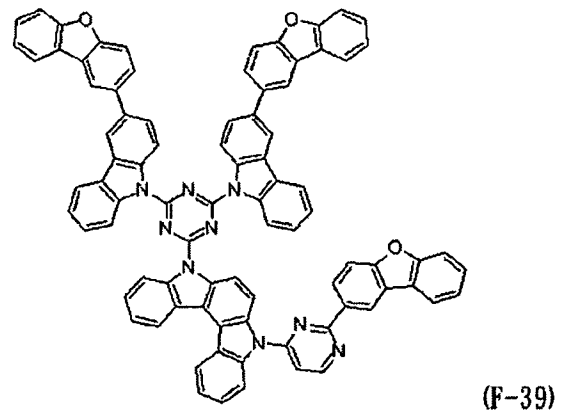
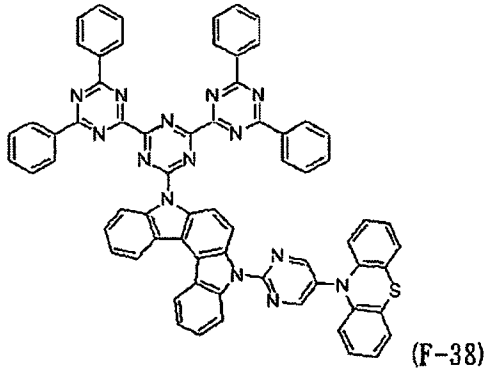


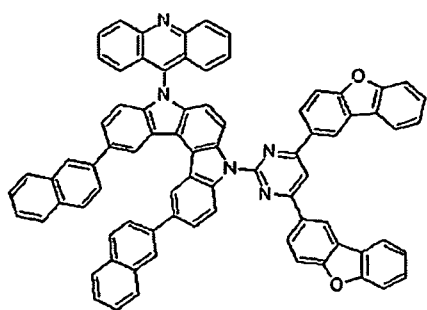
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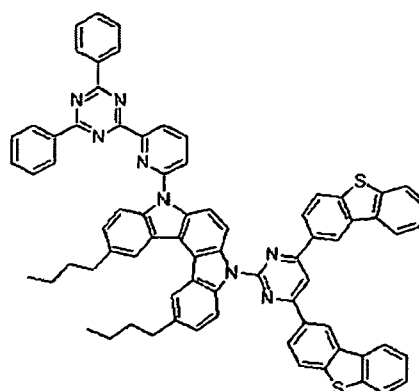
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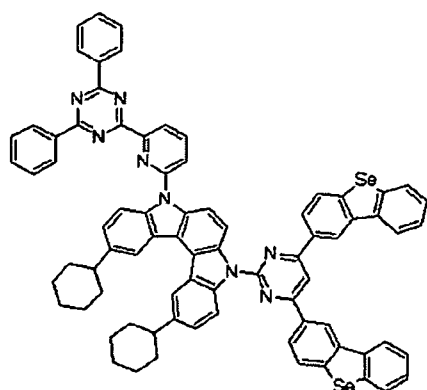




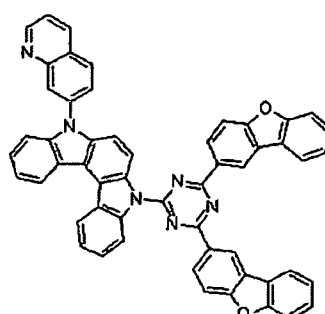
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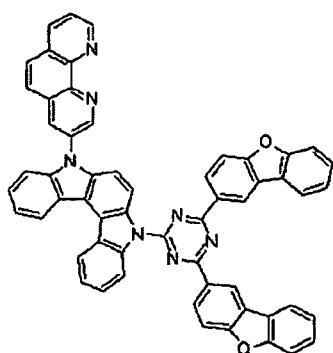
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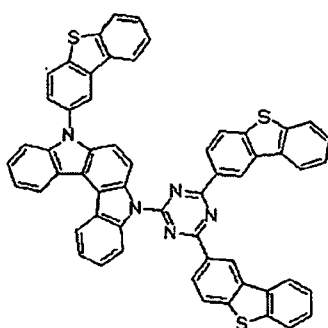
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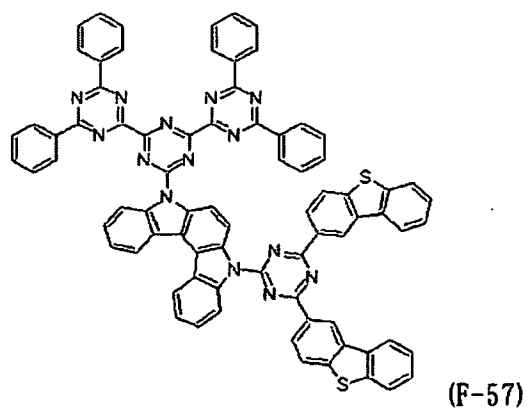
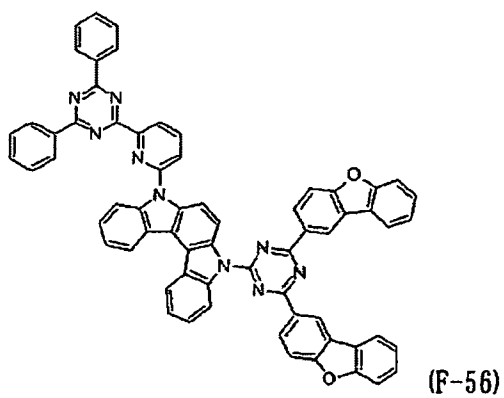
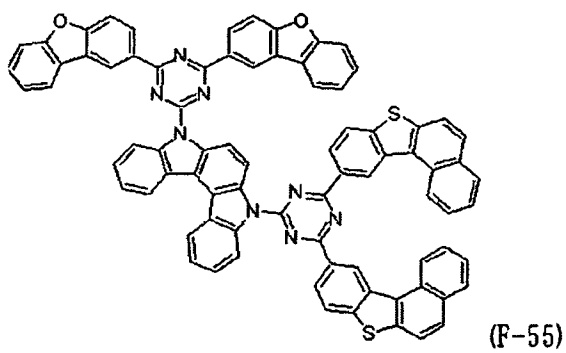
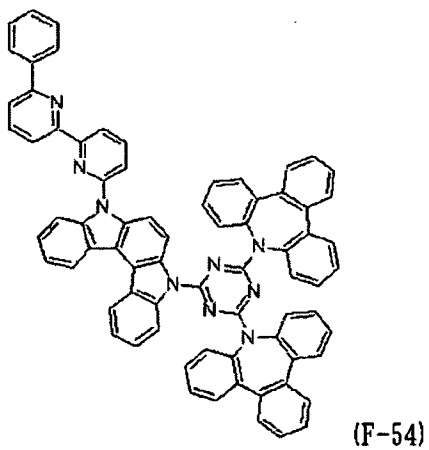
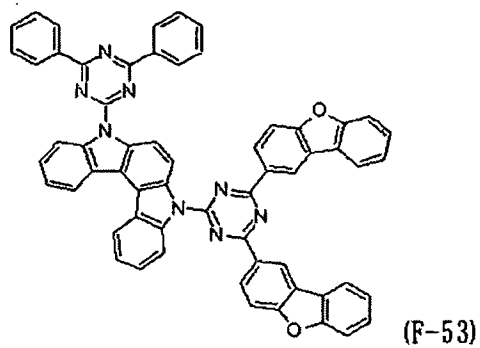
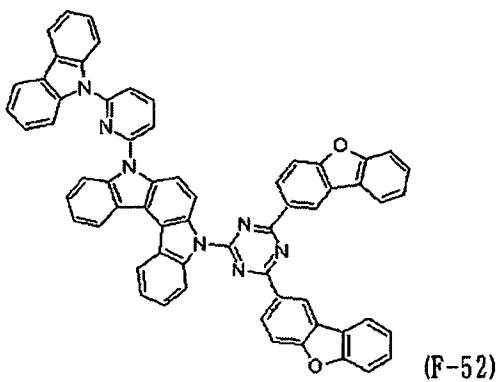
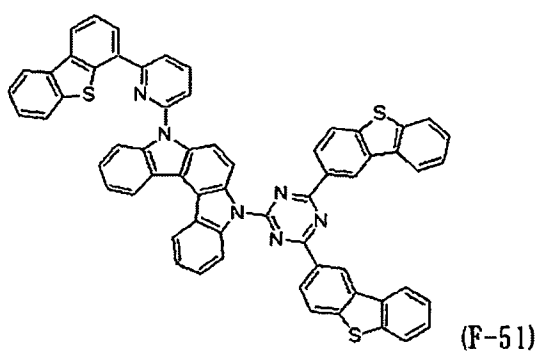
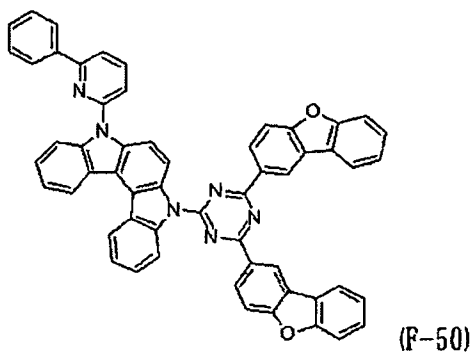
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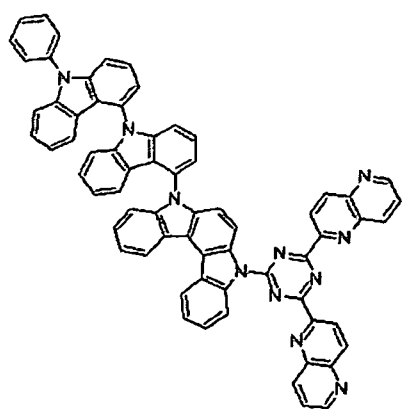


(F-48)

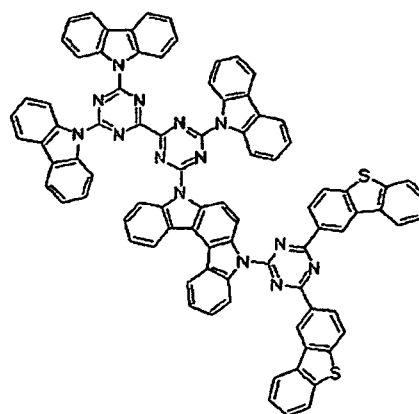


(F-49)

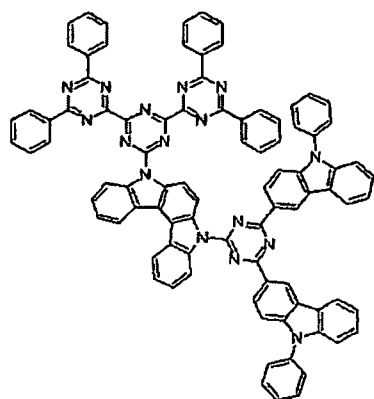




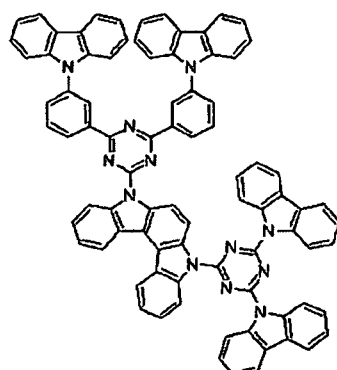
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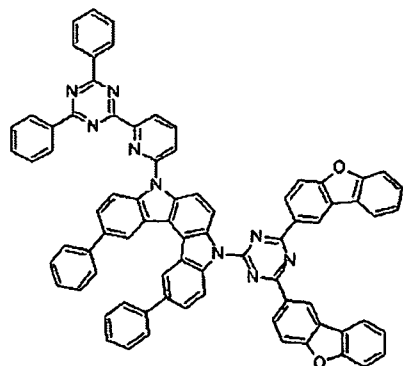
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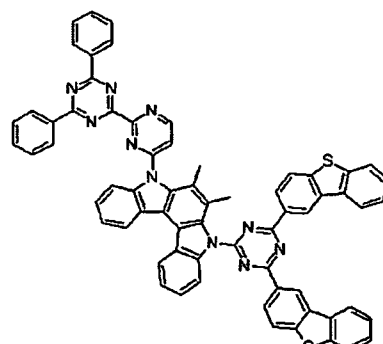
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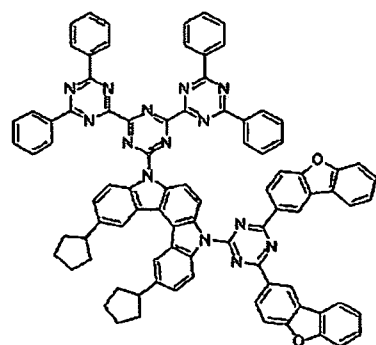
(F-61)



(F-62)

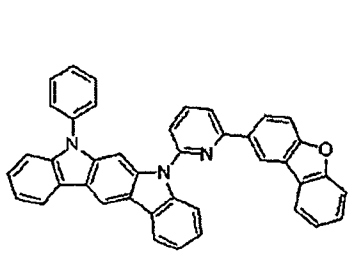


(F-63)

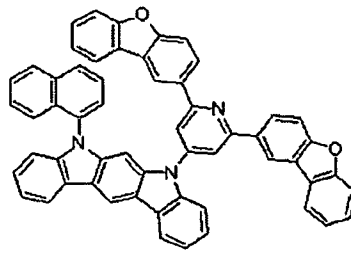


(F-64)

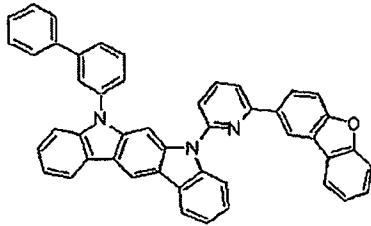
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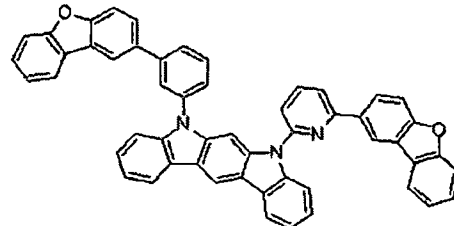
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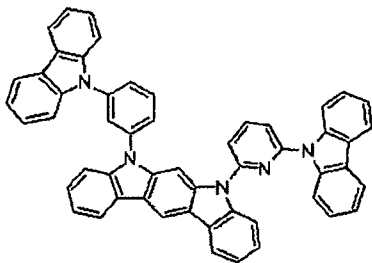
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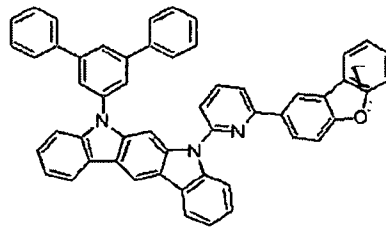
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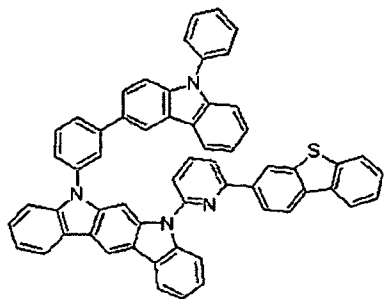
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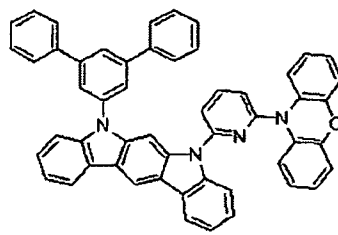
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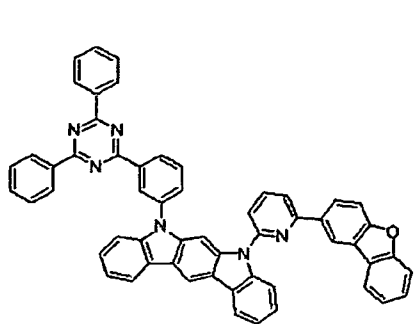
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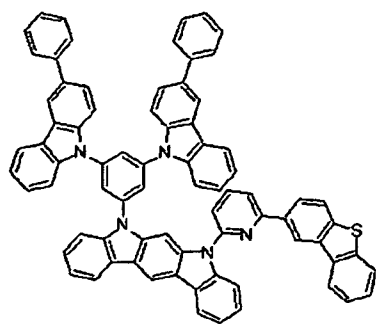
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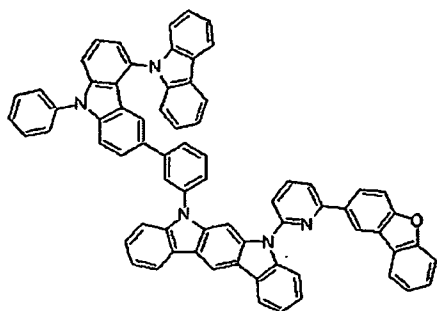
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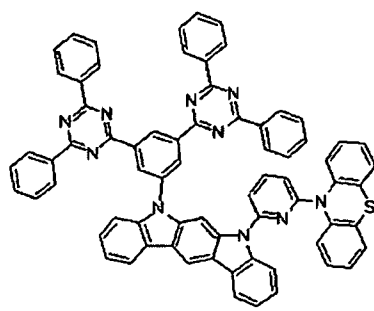
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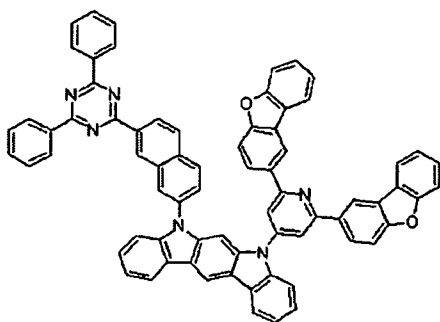
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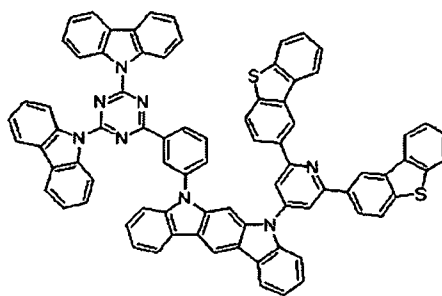
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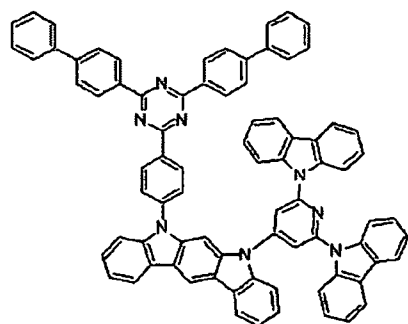
(G-12)



(G-13)

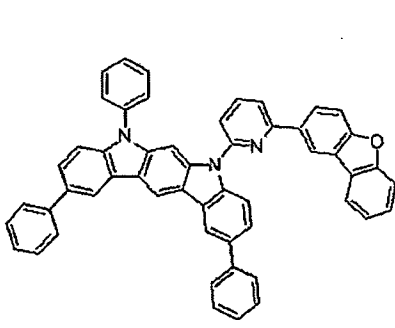


(G-14)

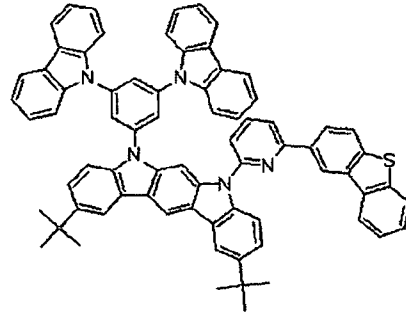


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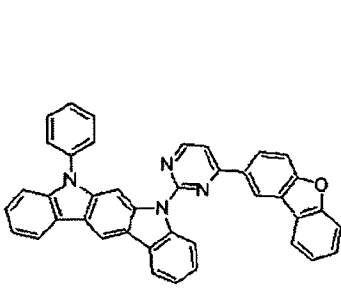




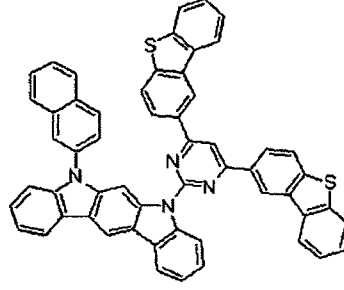
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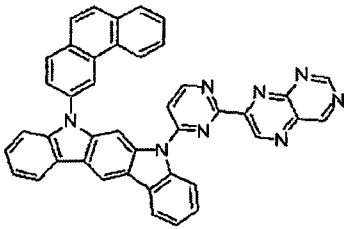
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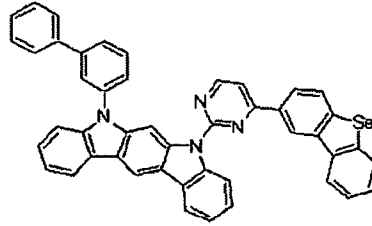
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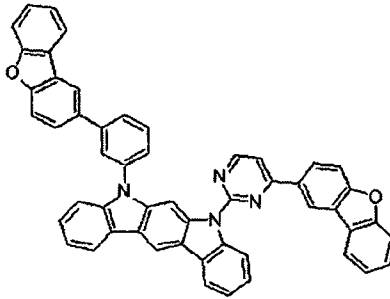
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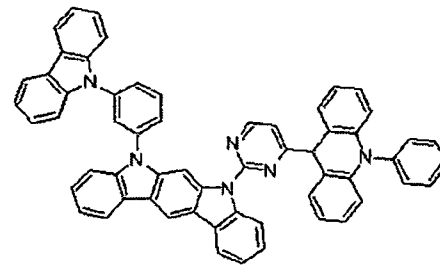
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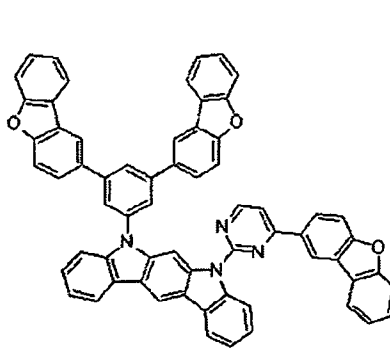
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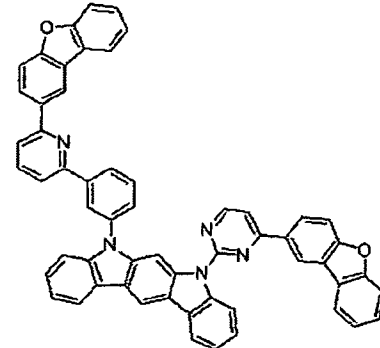
(G-22)



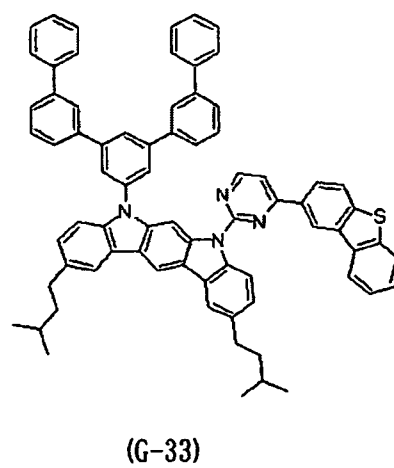
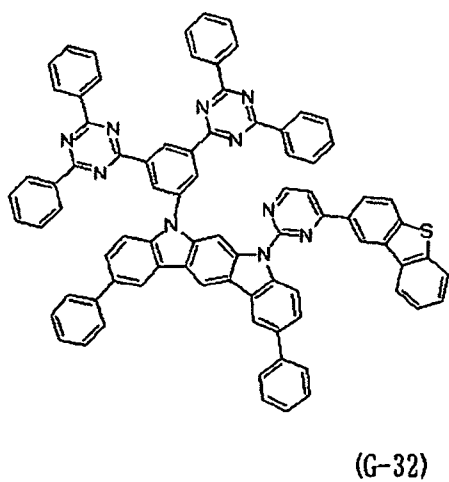
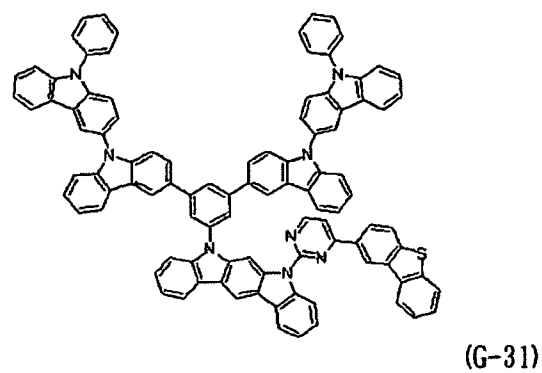
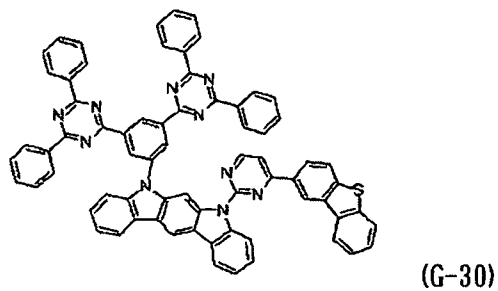
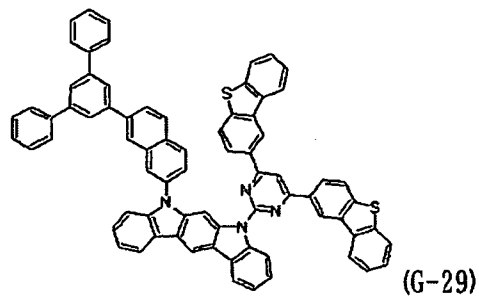
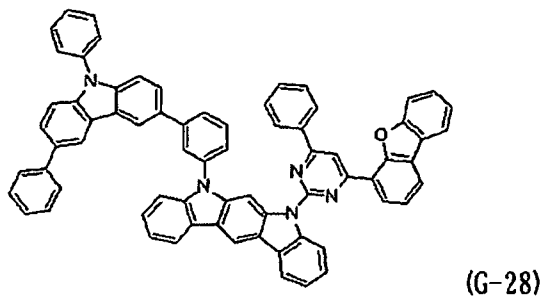
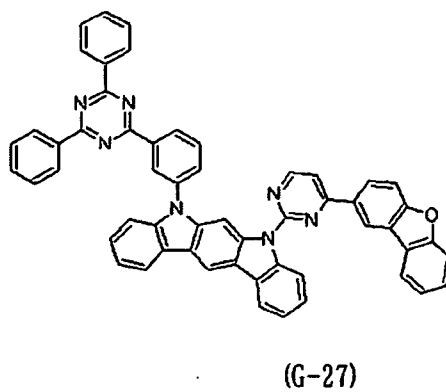
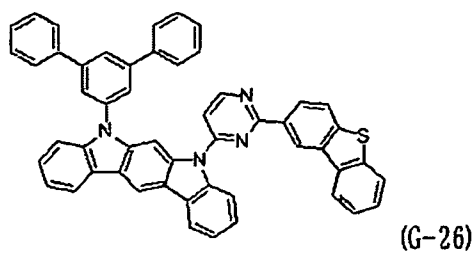
(G-23)

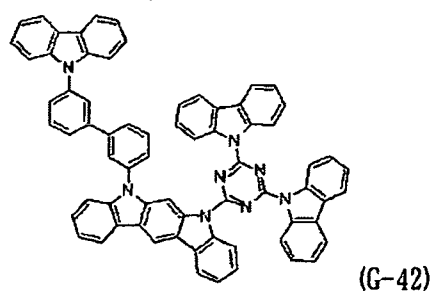
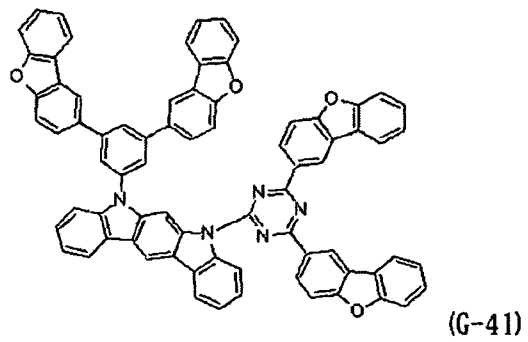
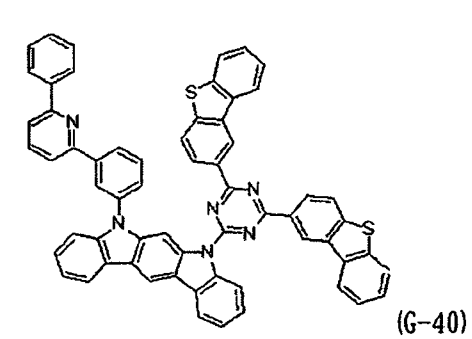
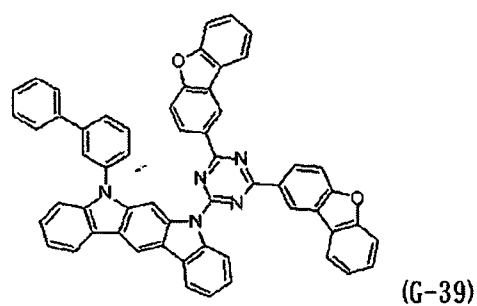
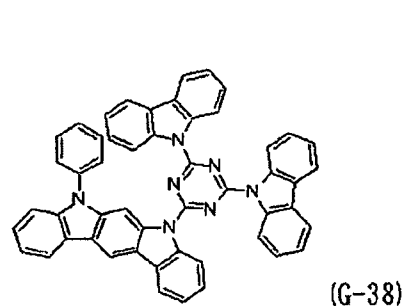
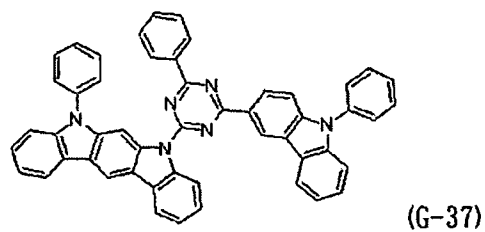
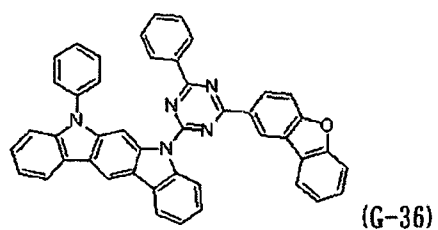
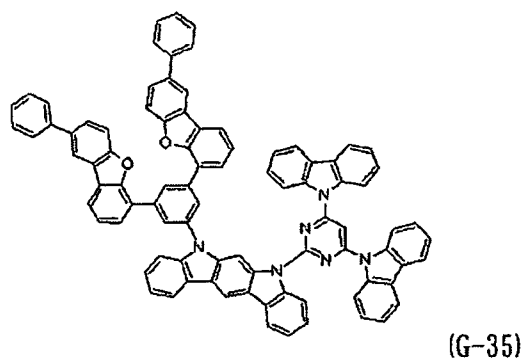
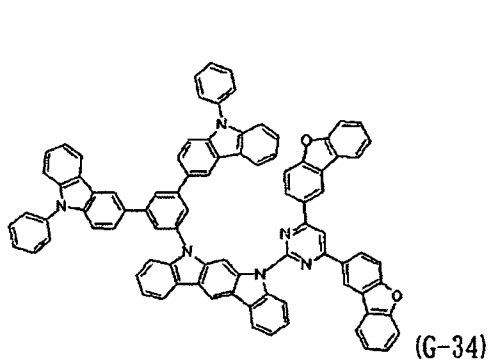


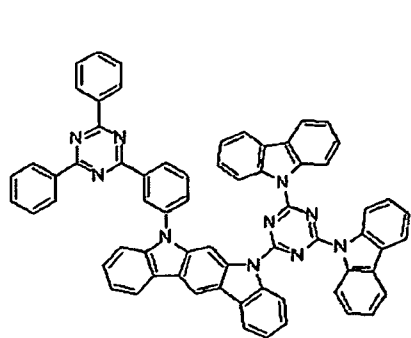
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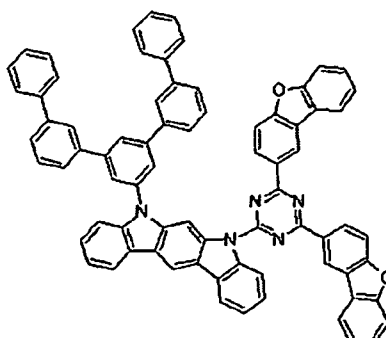
(G-25)



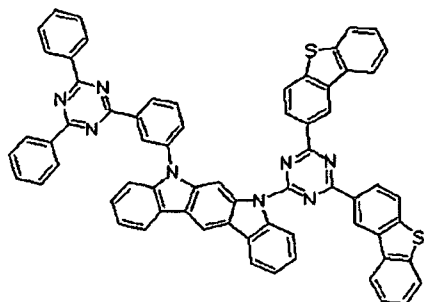




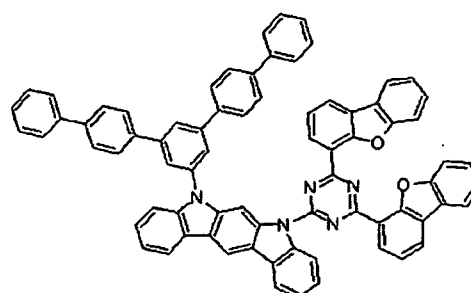
(G-43)



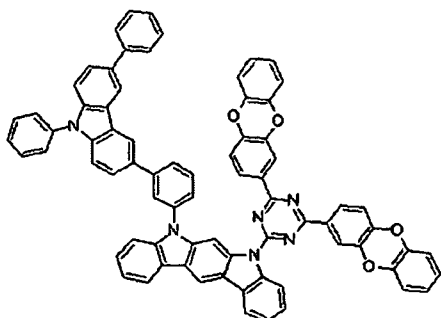
(G-44)



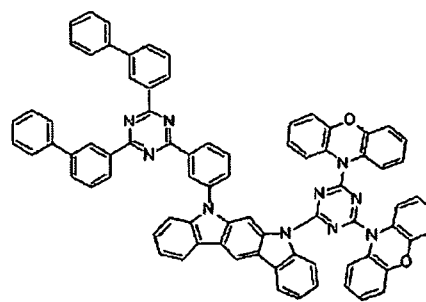
(G-45)



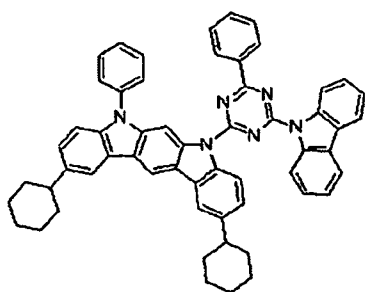
(G-46)



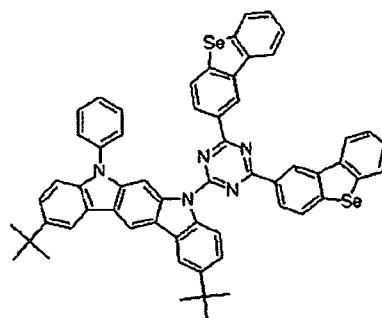
(G-47)



(G-48)

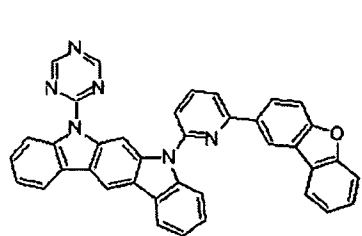


(G-49)

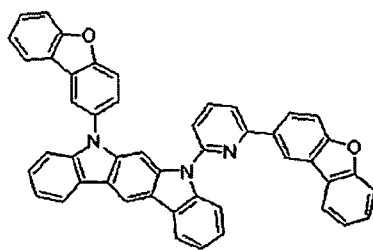


(G-50)

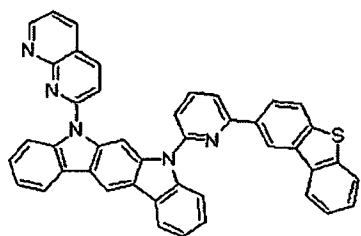
## 【0061】



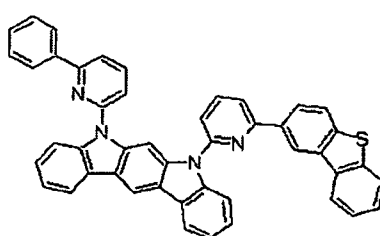
(H-1)



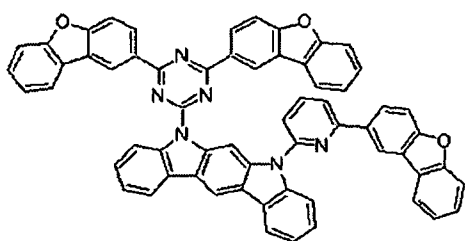
(H-2)



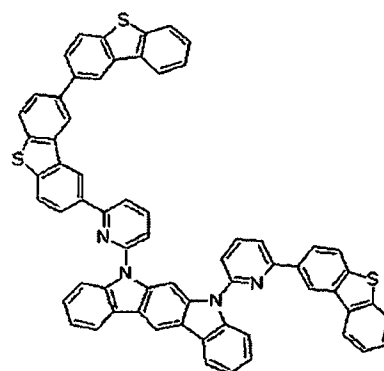
(H-3)



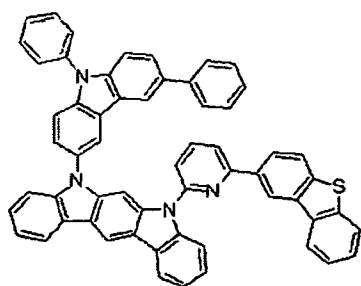
(H-4)



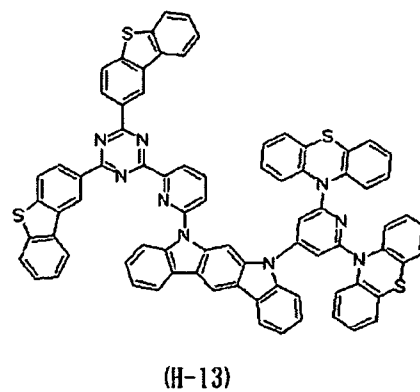
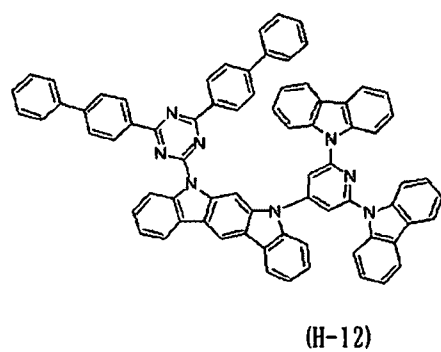
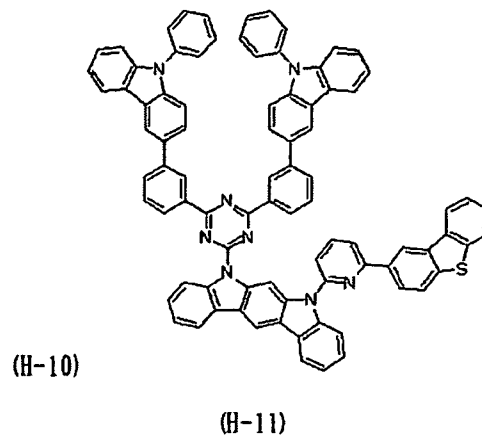
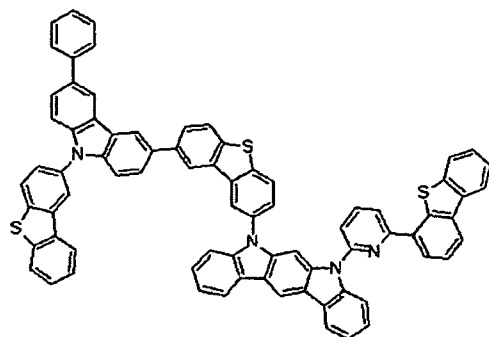
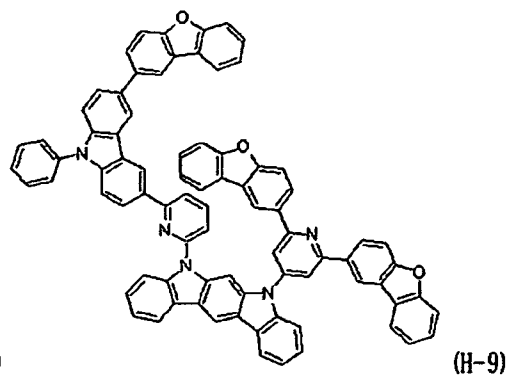
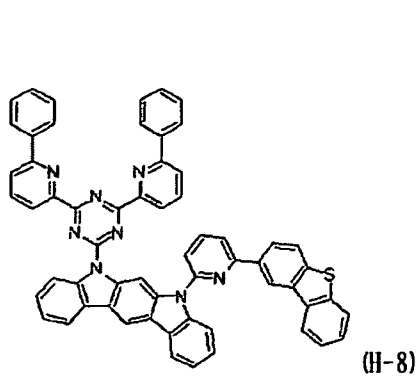
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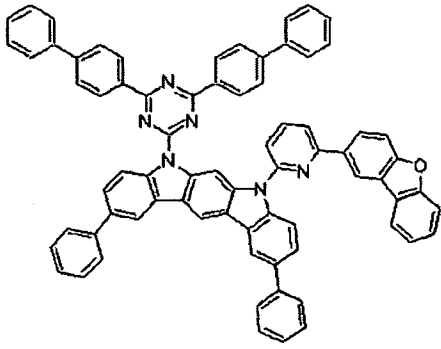


(H-6)

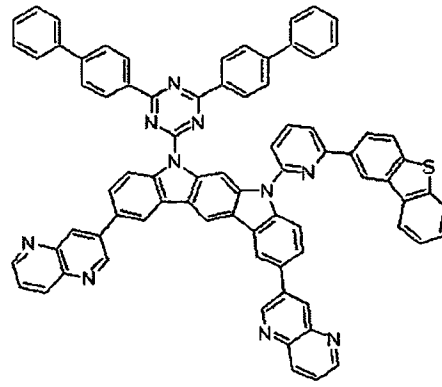


(H-7)

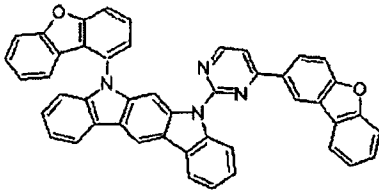




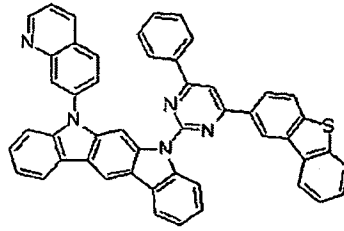
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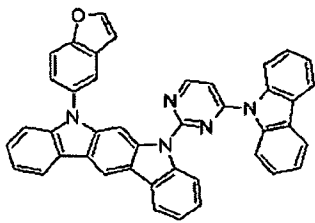
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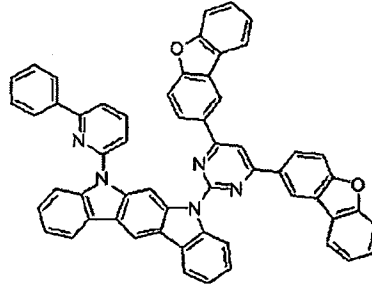
(H-16)



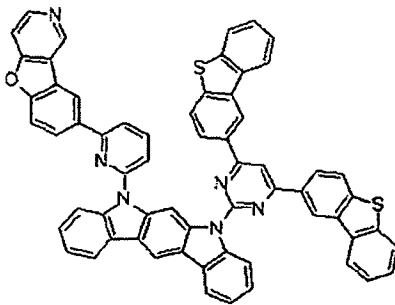
(H-17)



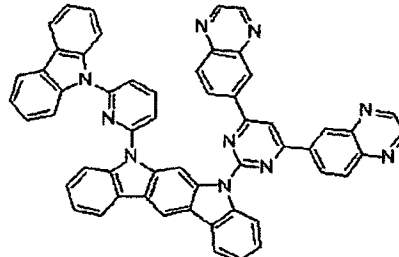
(H-18)



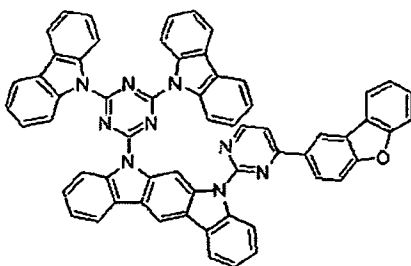
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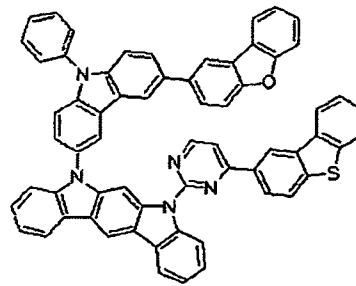
(H-20)



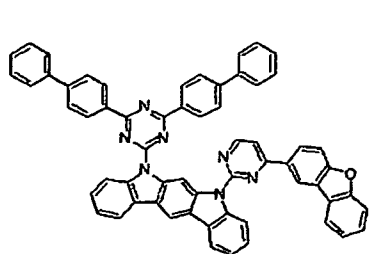
(H-21)



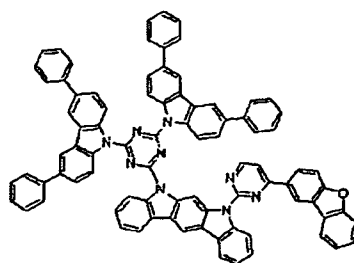
(H-22)



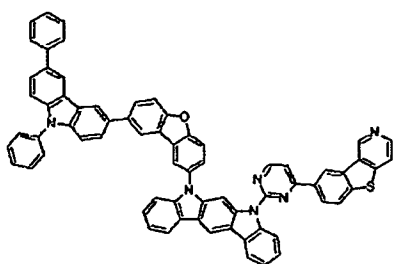
(H-23)



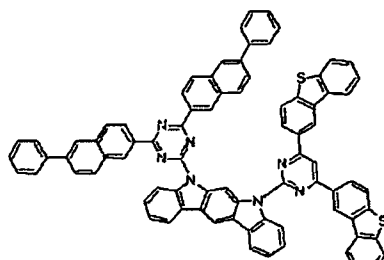
(H-24)



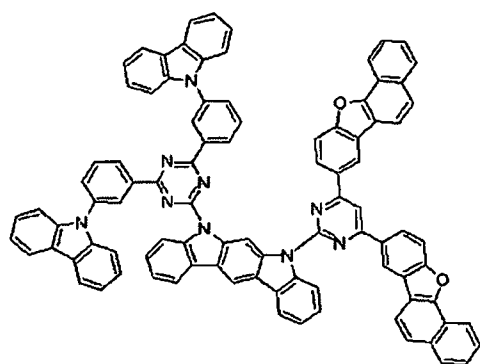
(H-25)



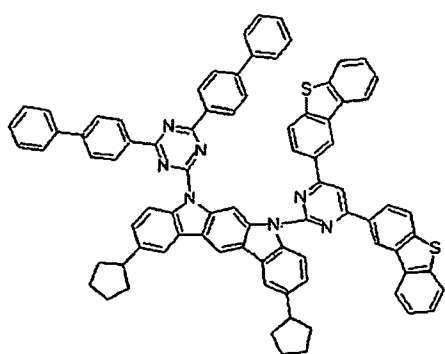
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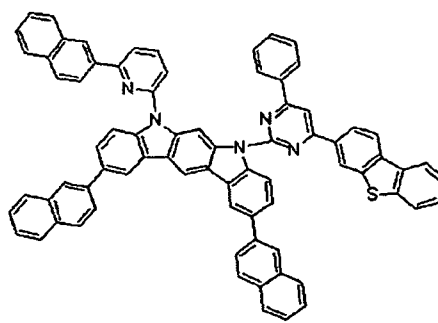
(H-27)



(H-28)

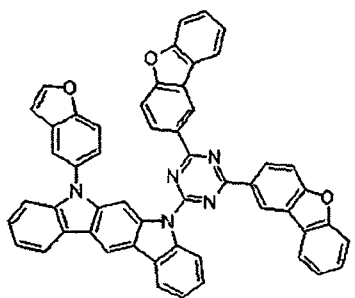


(H-29)

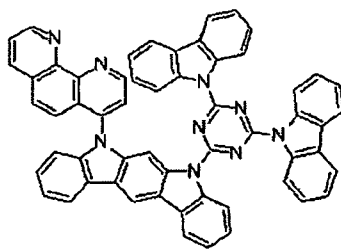


(H-30)

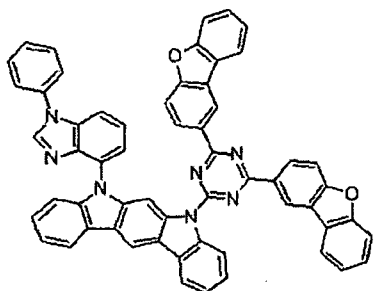




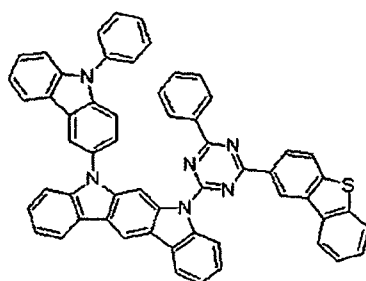
(H-31)



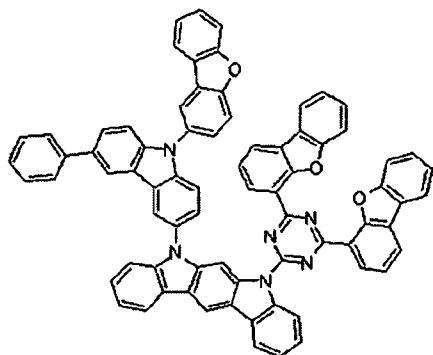
(H-32)



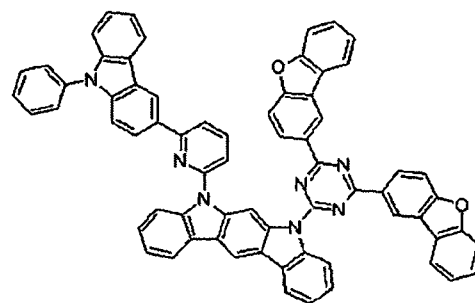
(H-33)



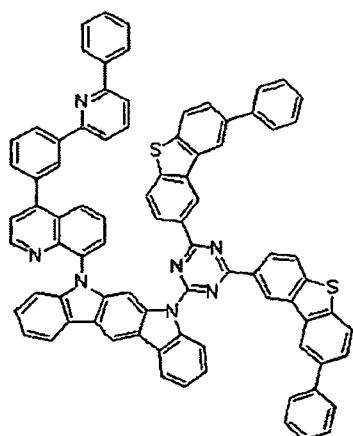
(H-34)



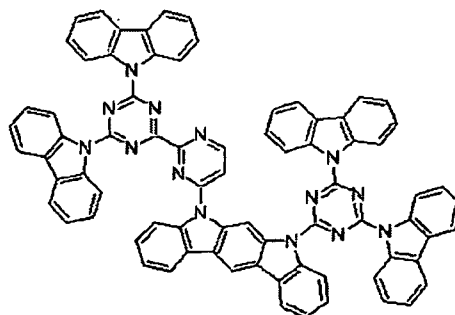
(H-35)



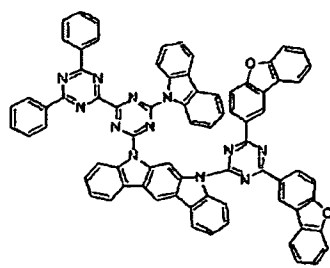
(H-36)



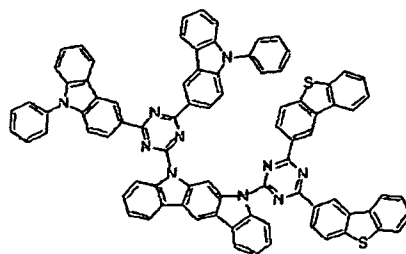
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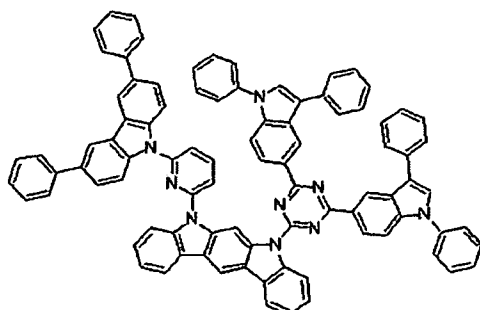
(H-38)



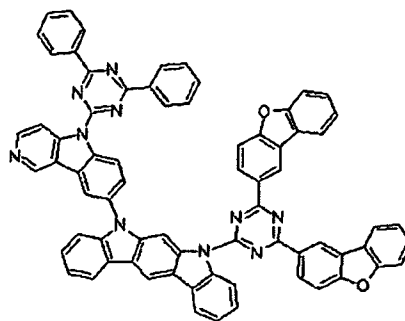
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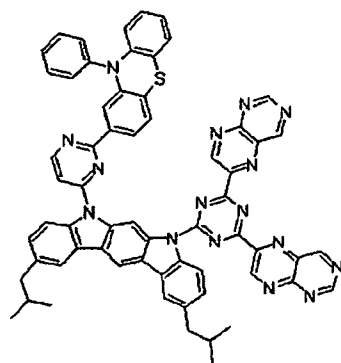
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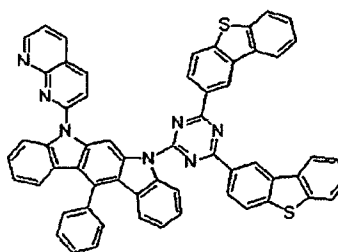
(H-41)



(H-42)

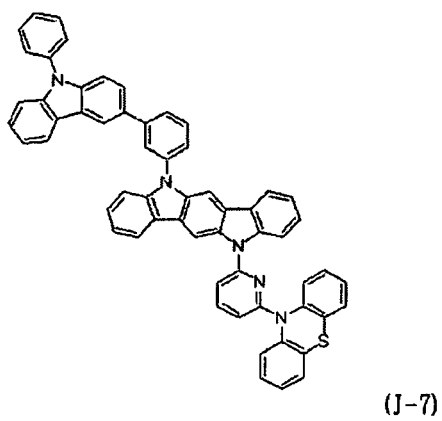
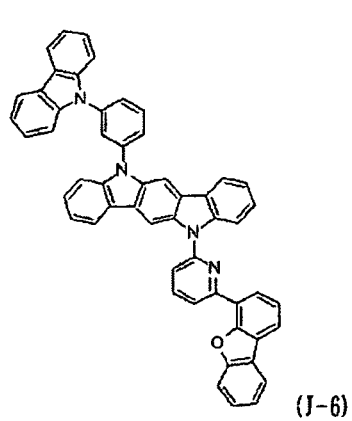
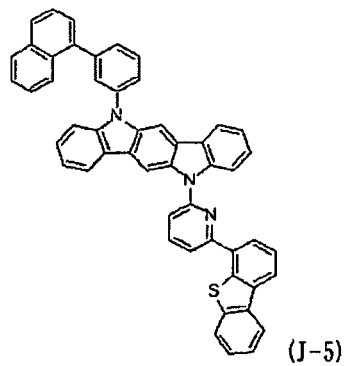
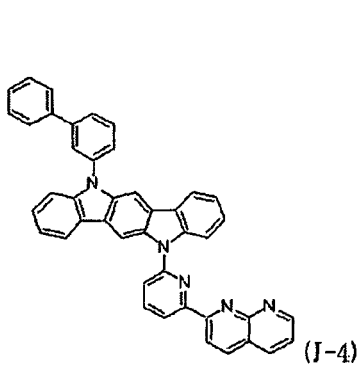
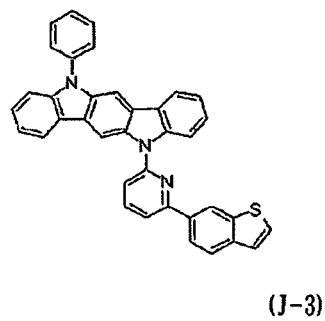
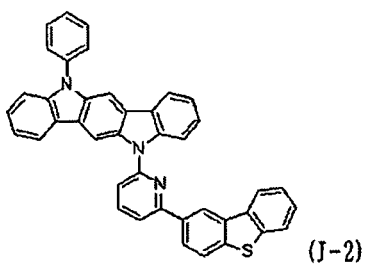
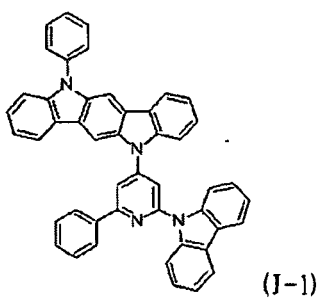


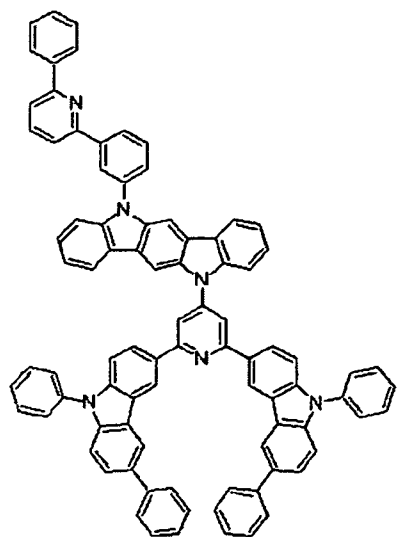
(H-43)



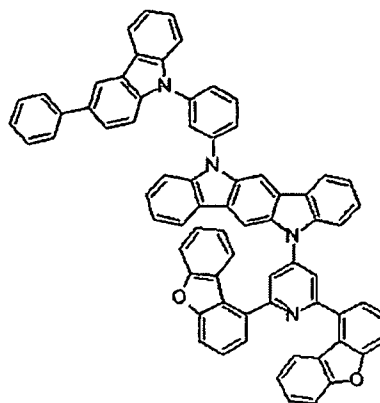
(H-44)

【0062】

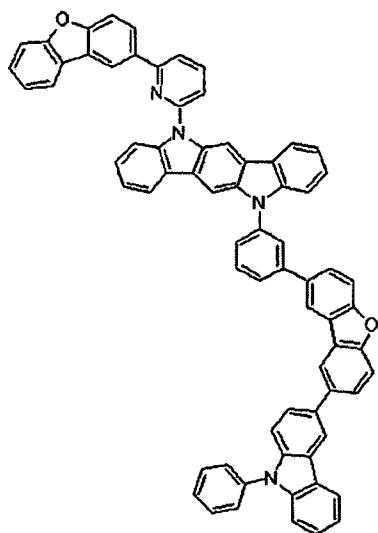




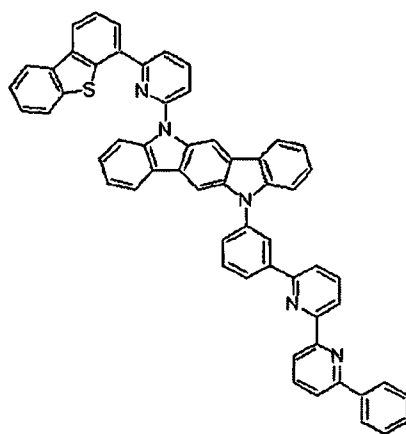
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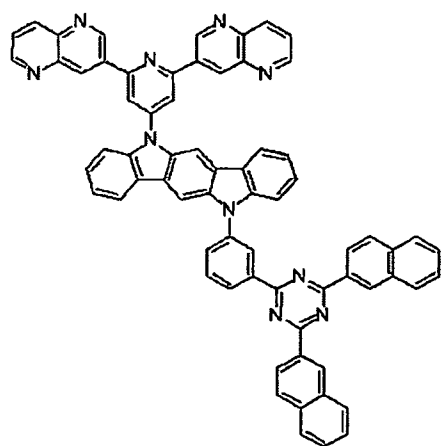
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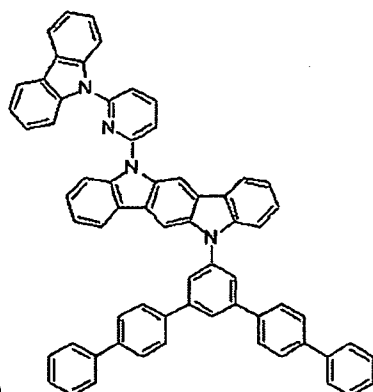
(J-10)



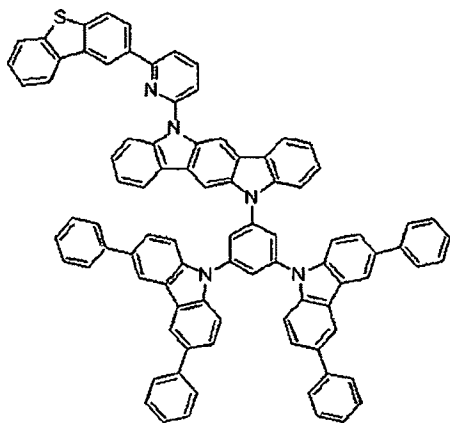
(J-11)



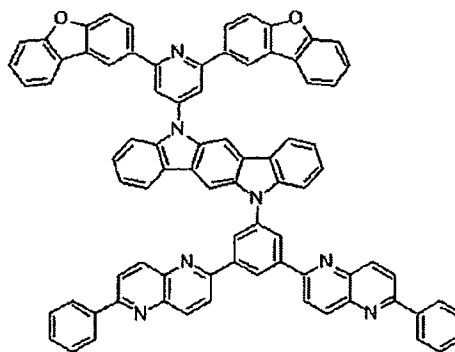
(J-12)



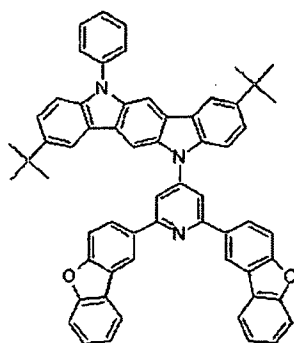
(J-13)



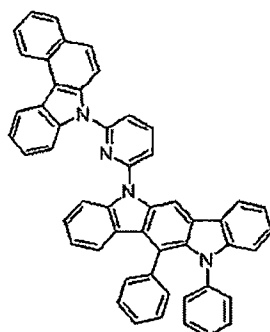
(J-14)



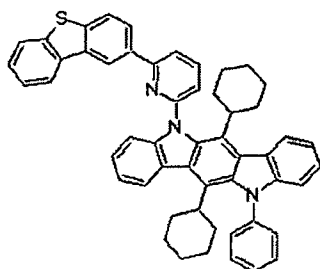
(J-15)



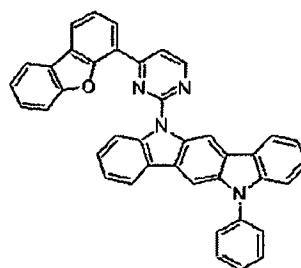
(J-16)



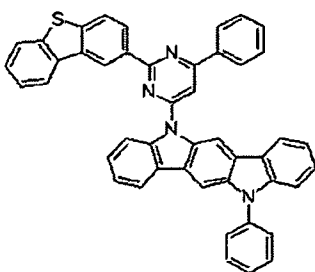
(J-17)



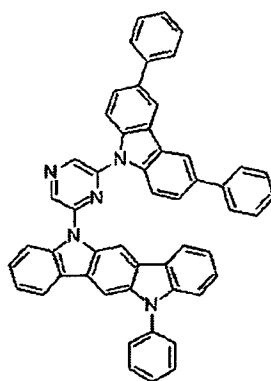
(J-18)



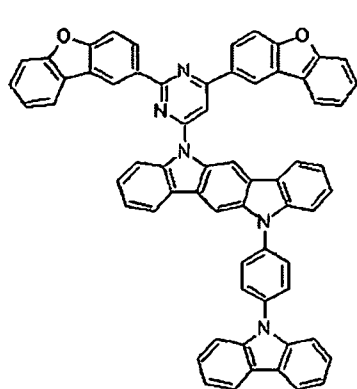
(J-19)



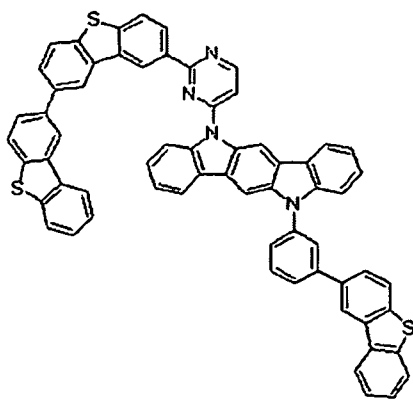
(J-20)



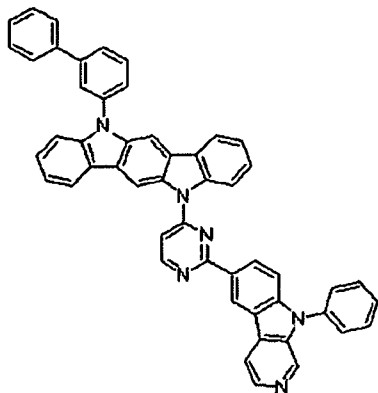
(J-21)



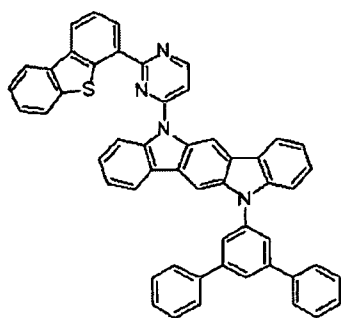
(J-22)



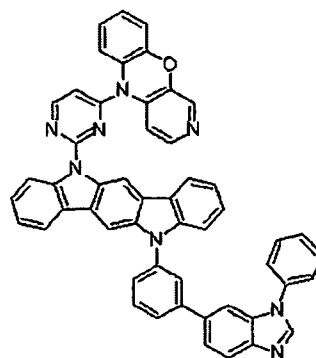
(J-23)



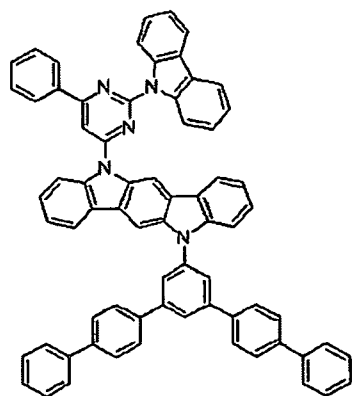
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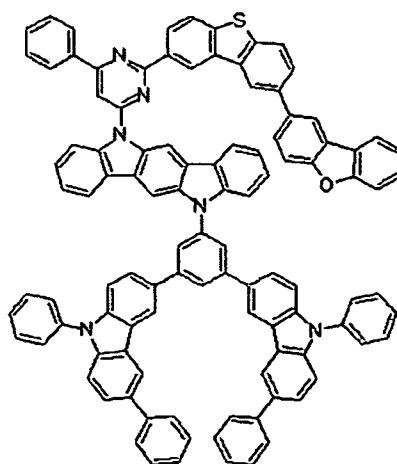
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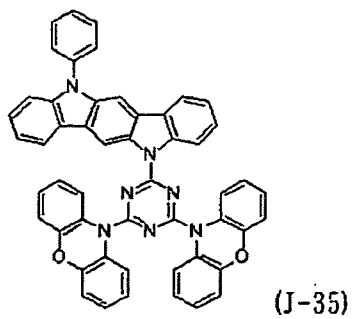
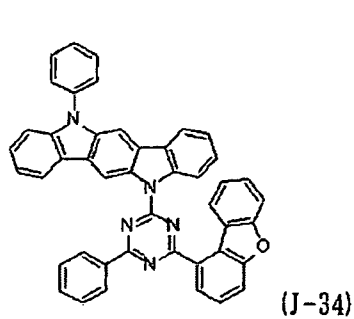
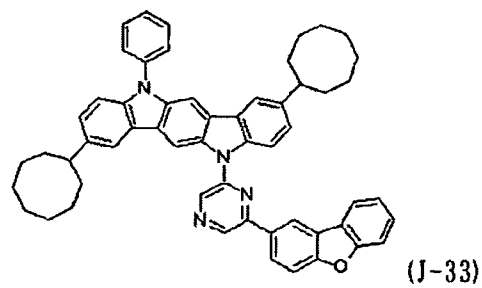
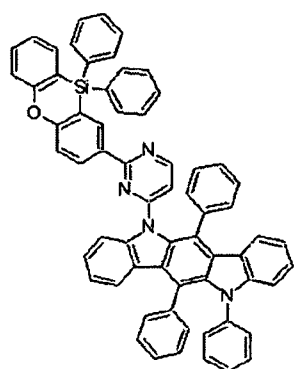
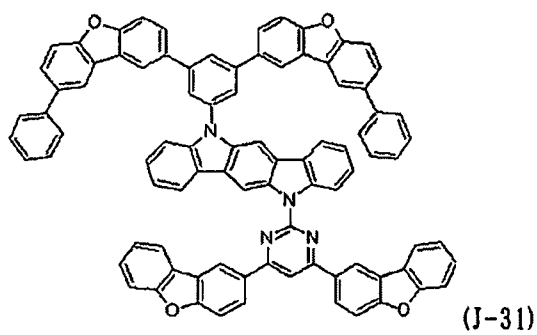
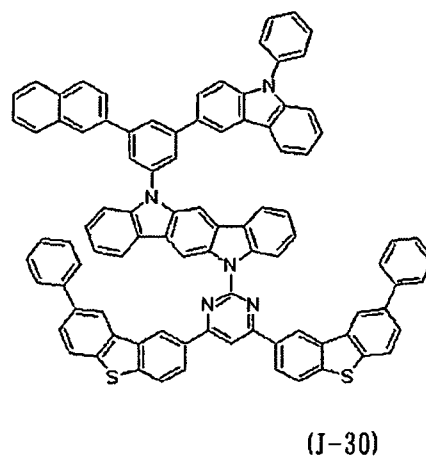
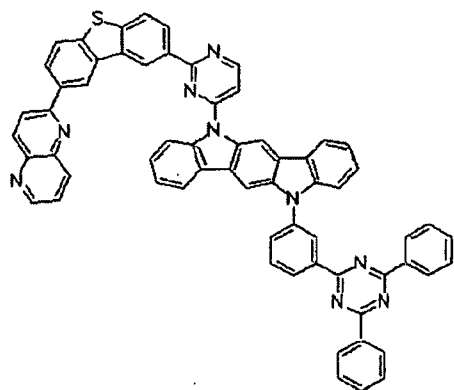
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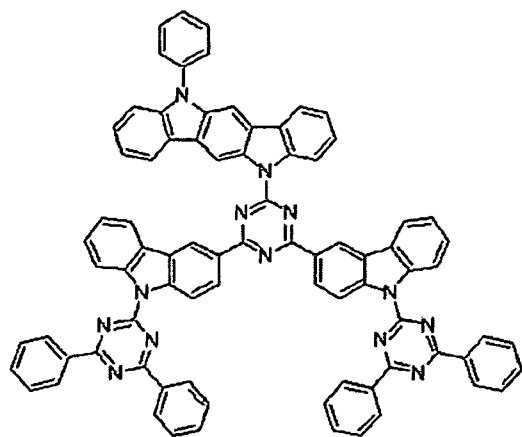


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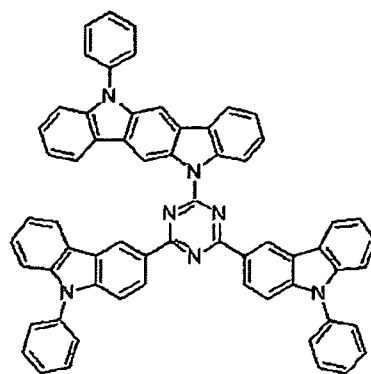


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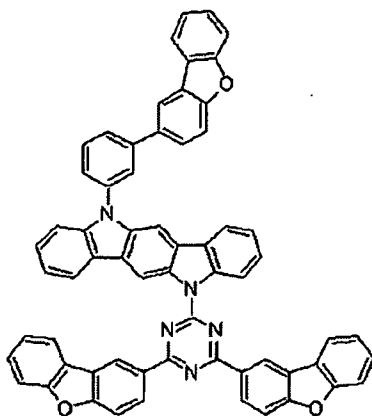




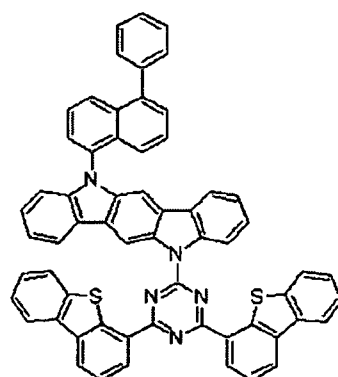
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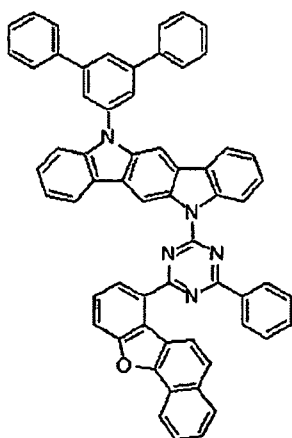
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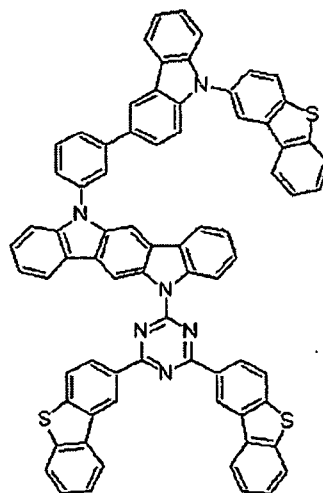
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(J-39)

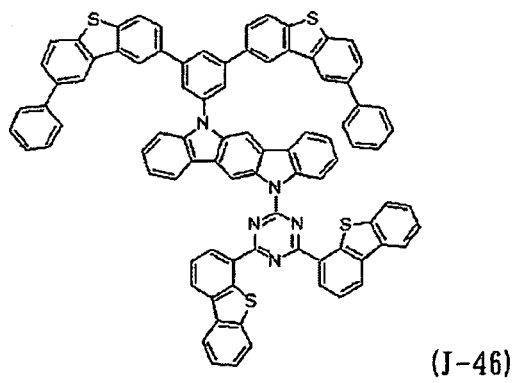
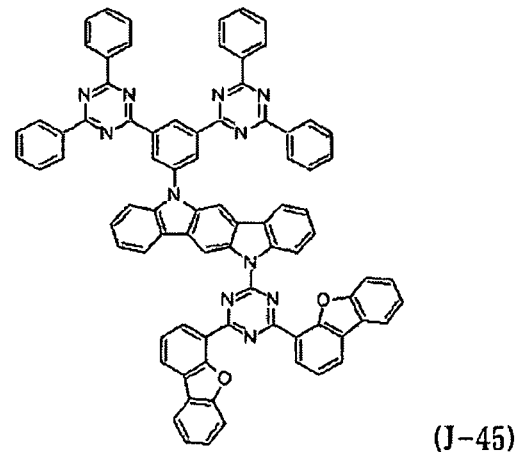
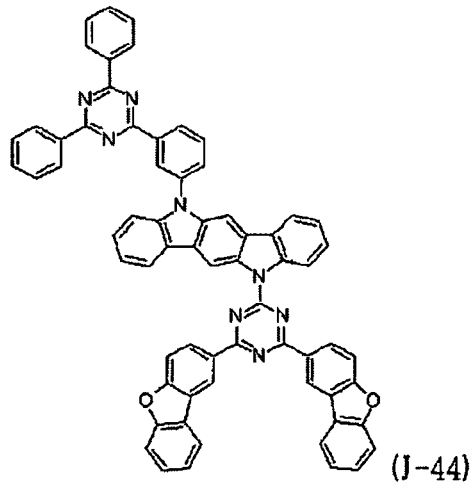
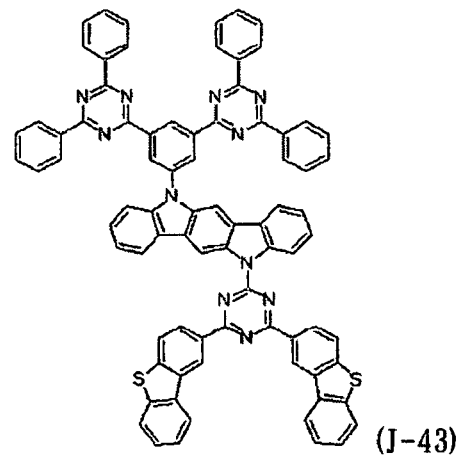
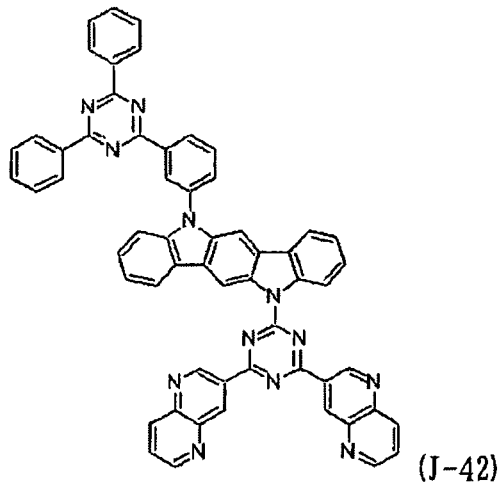


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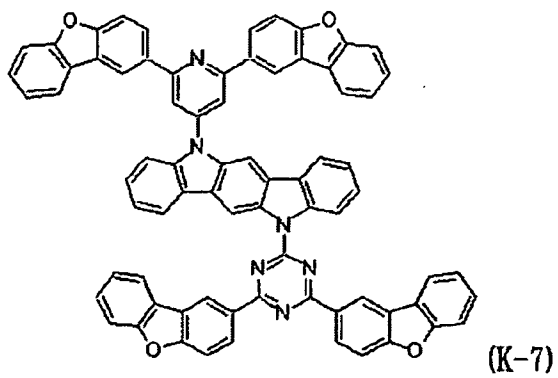
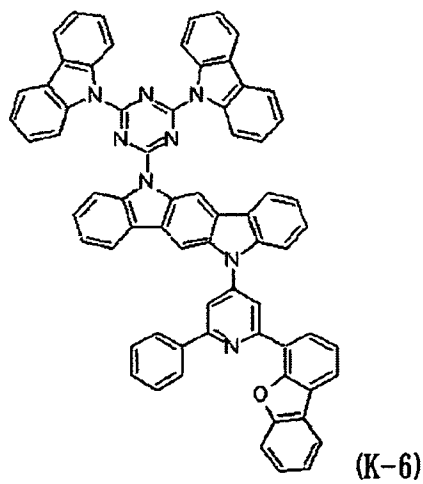
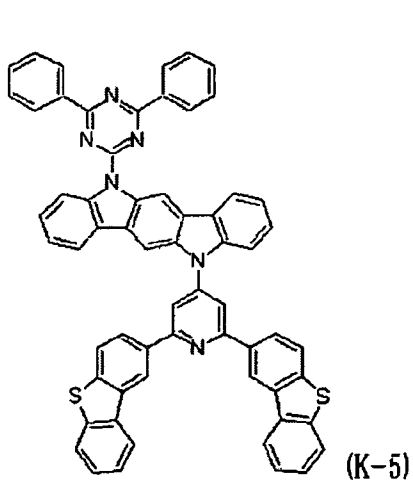
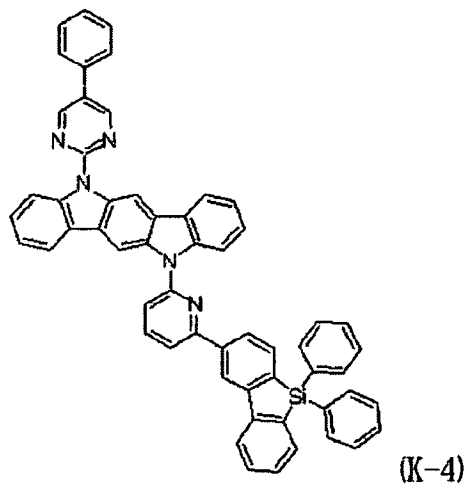
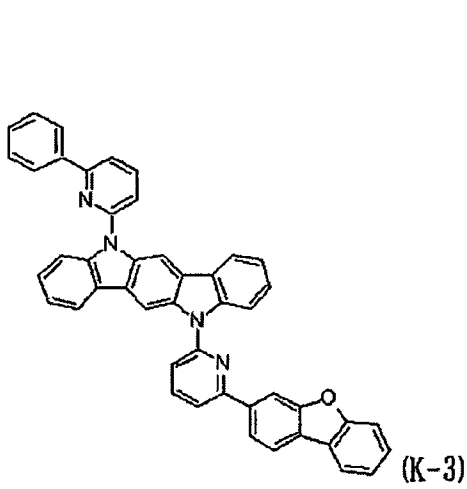
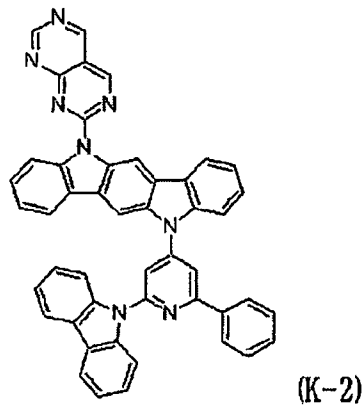
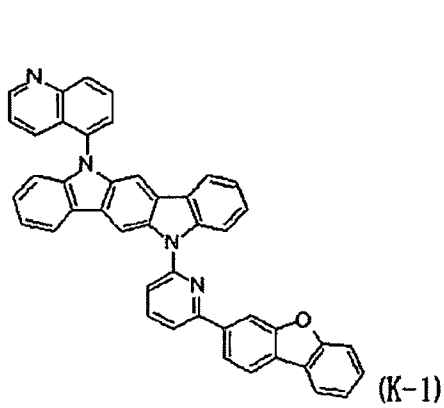
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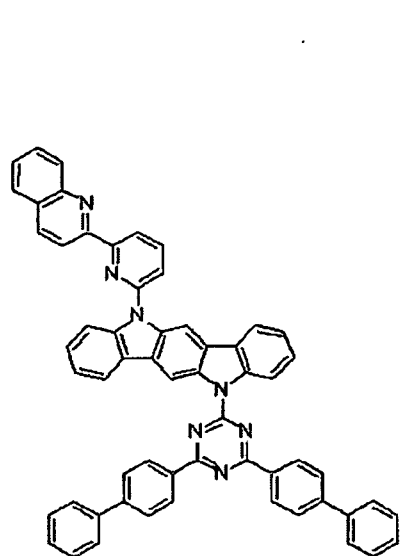




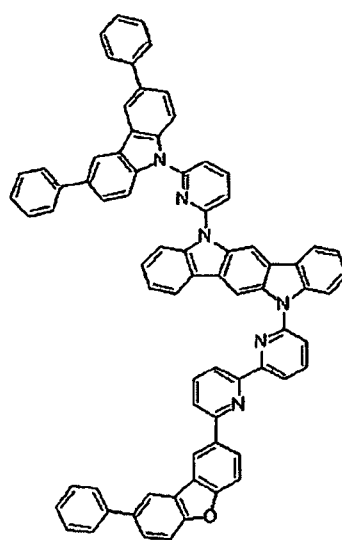


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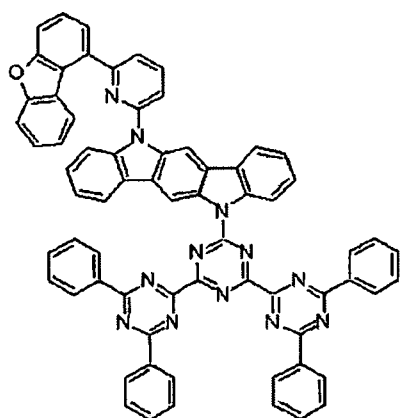




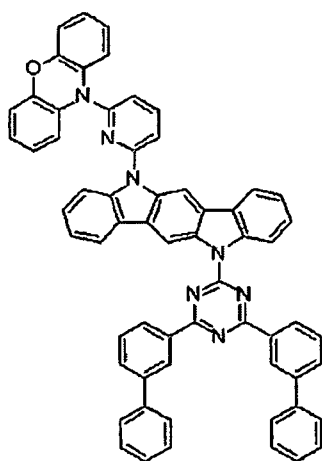
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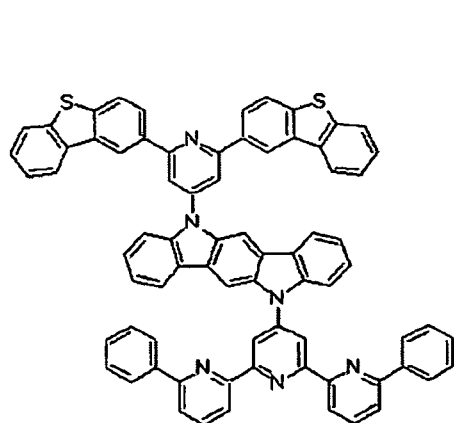
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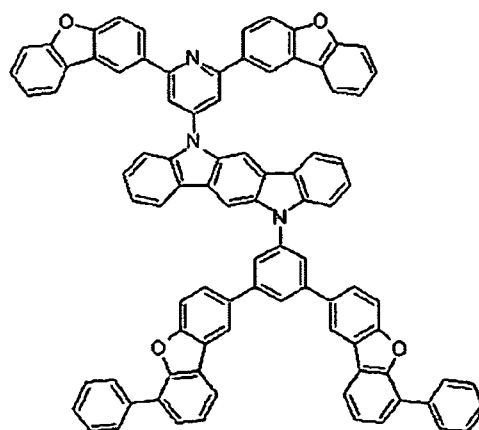
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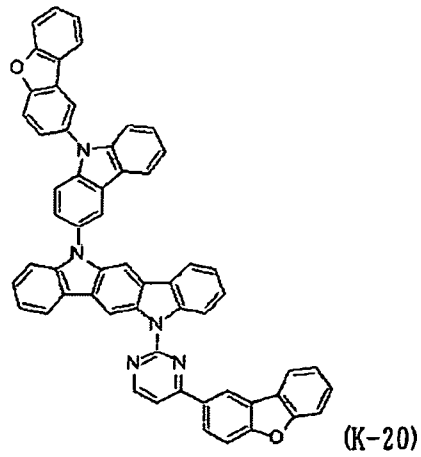
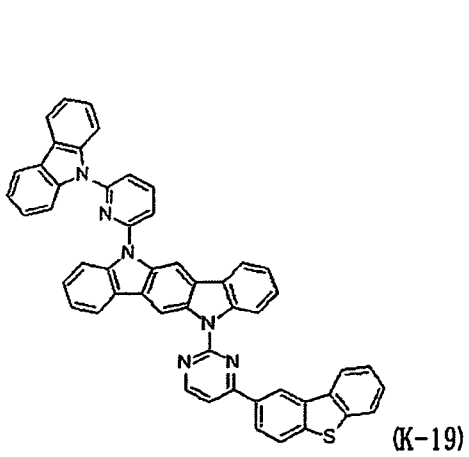
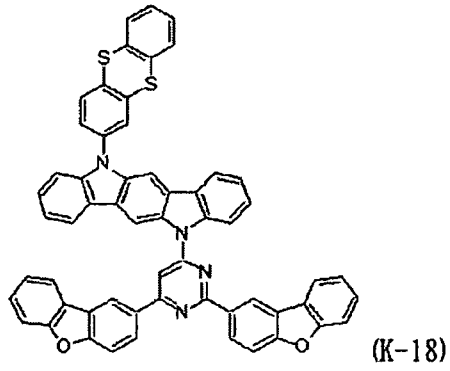
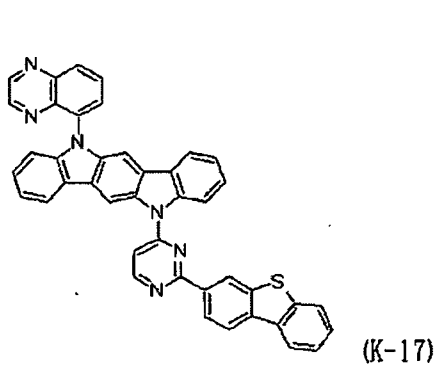
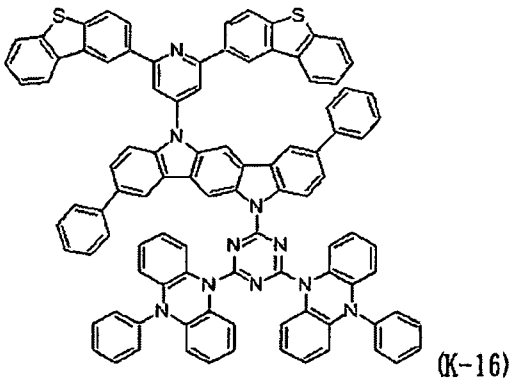
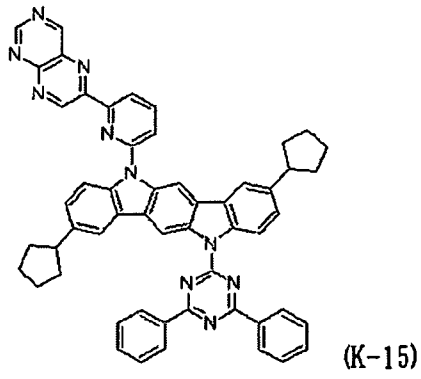
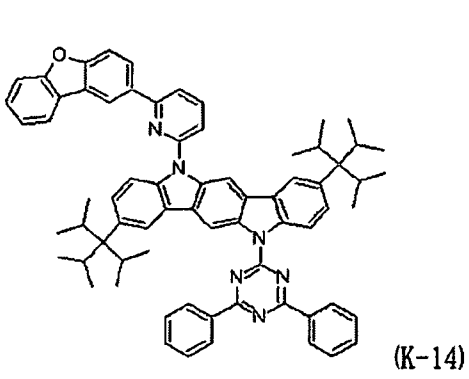
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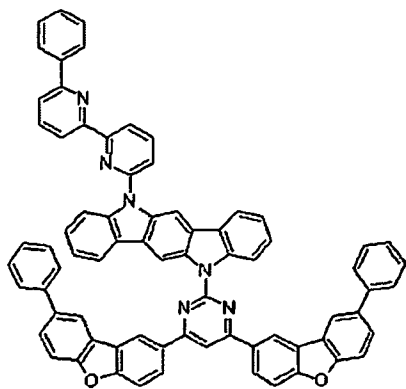


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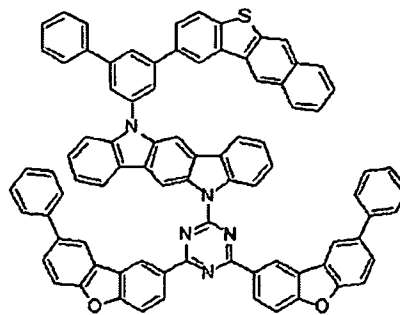


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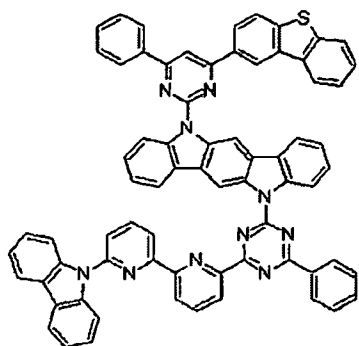




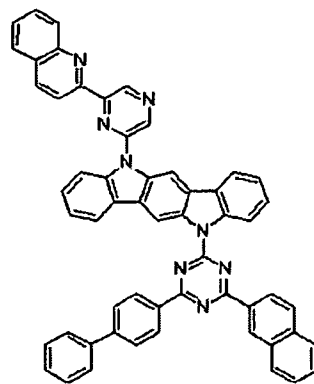
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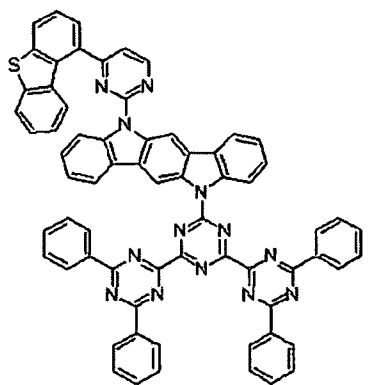
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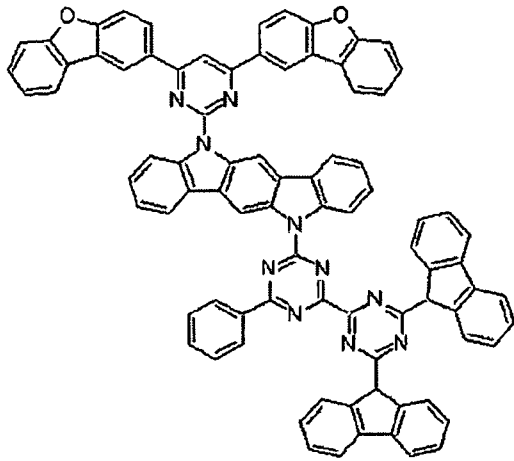
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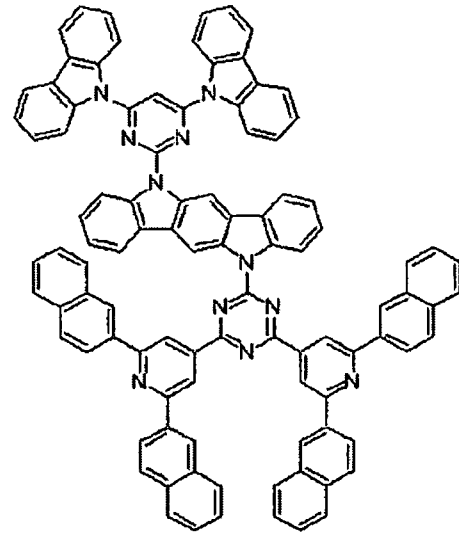
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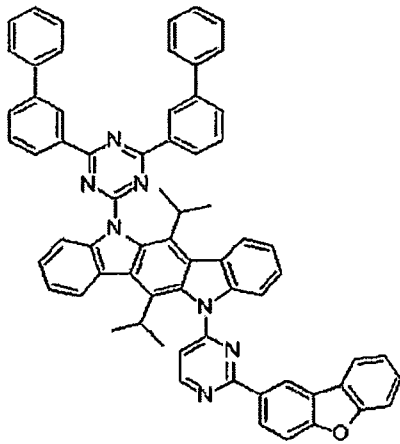
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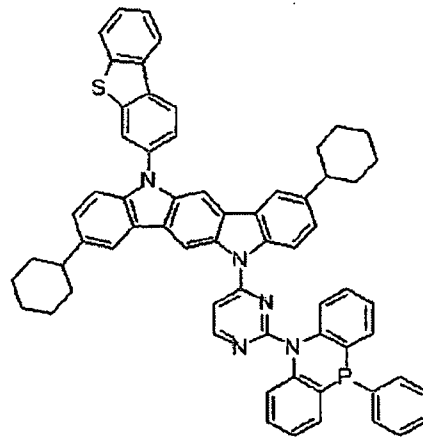
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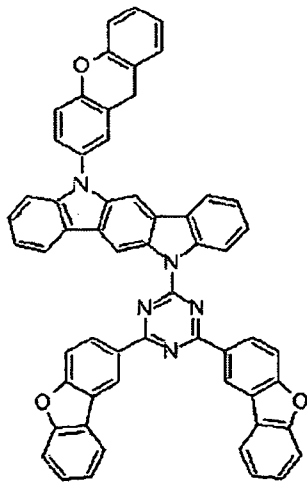
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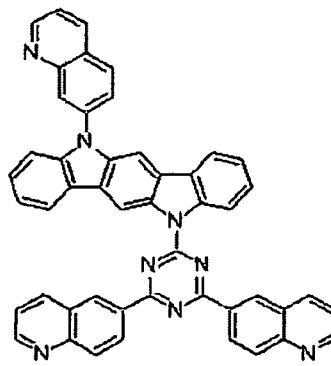
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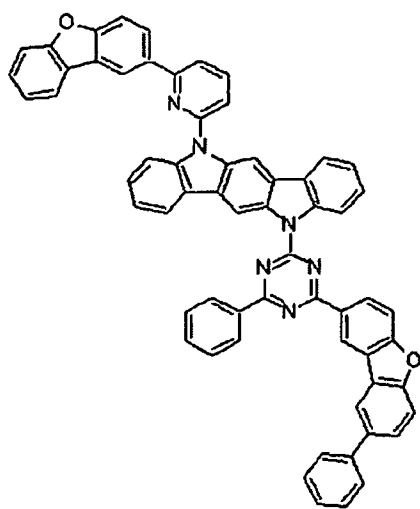
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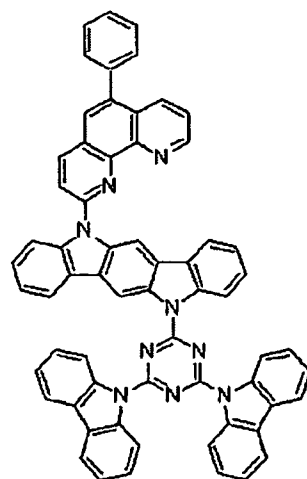
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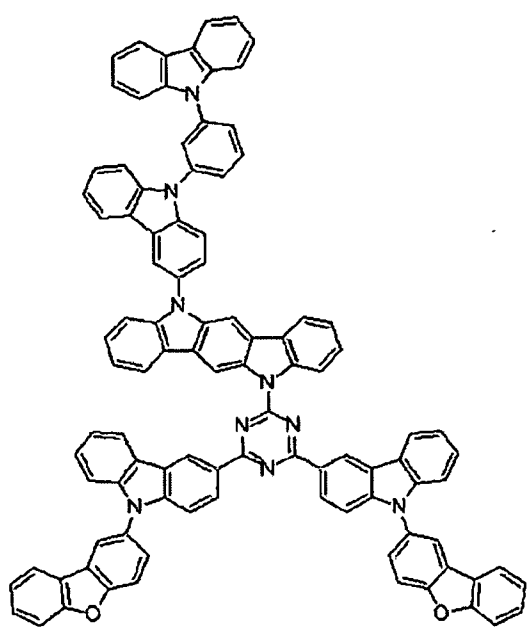
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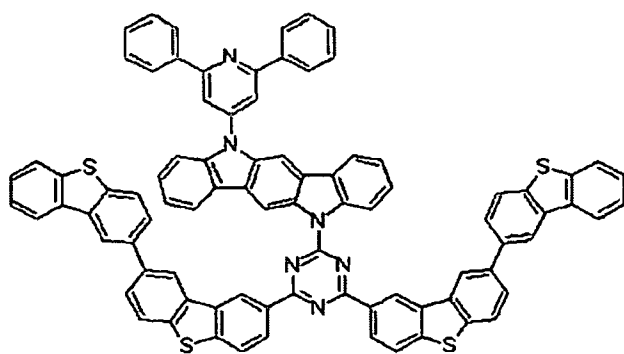
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(K-33)

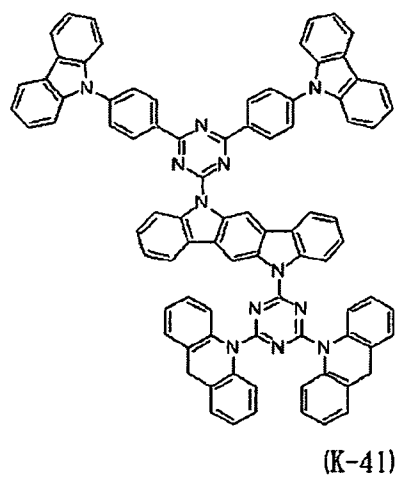
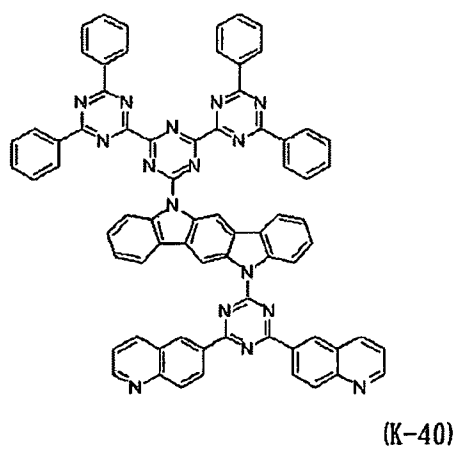
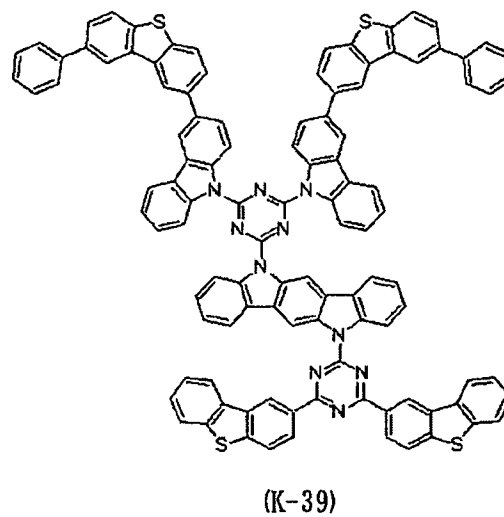
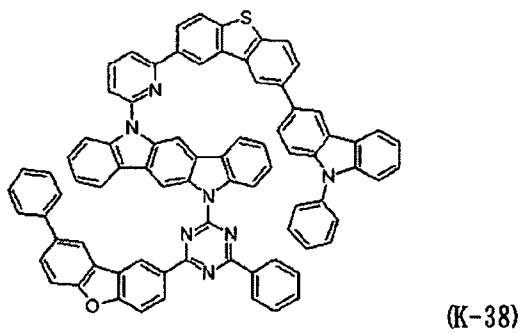
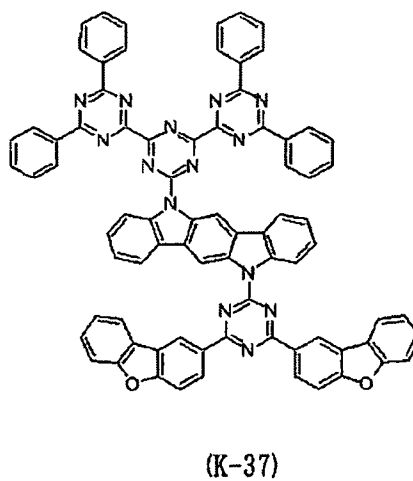
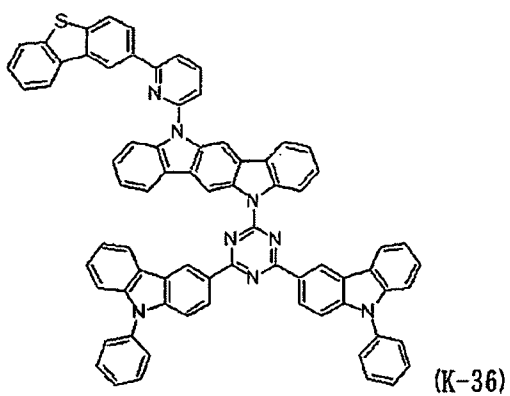


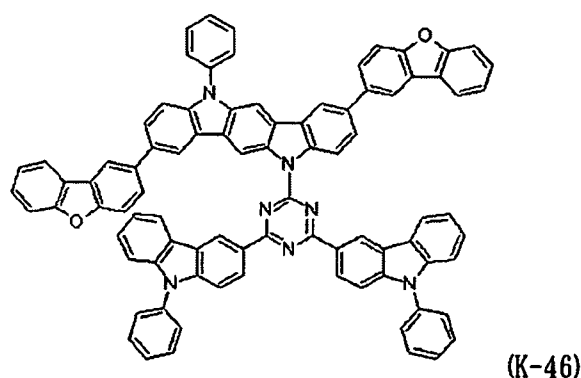
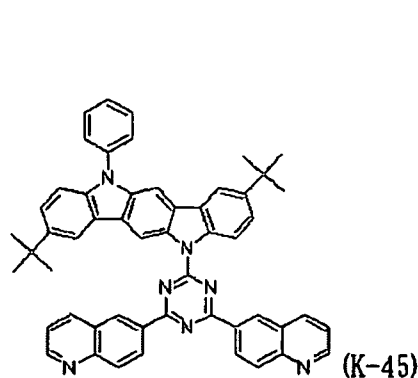
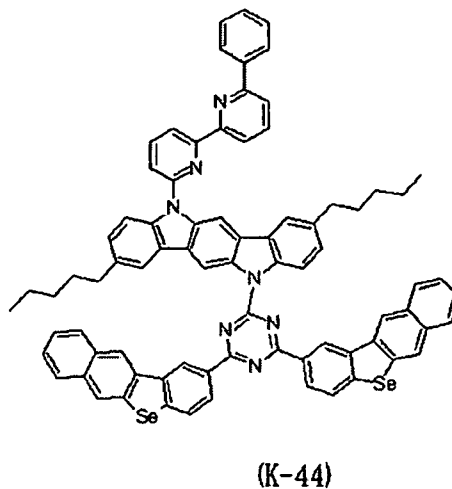
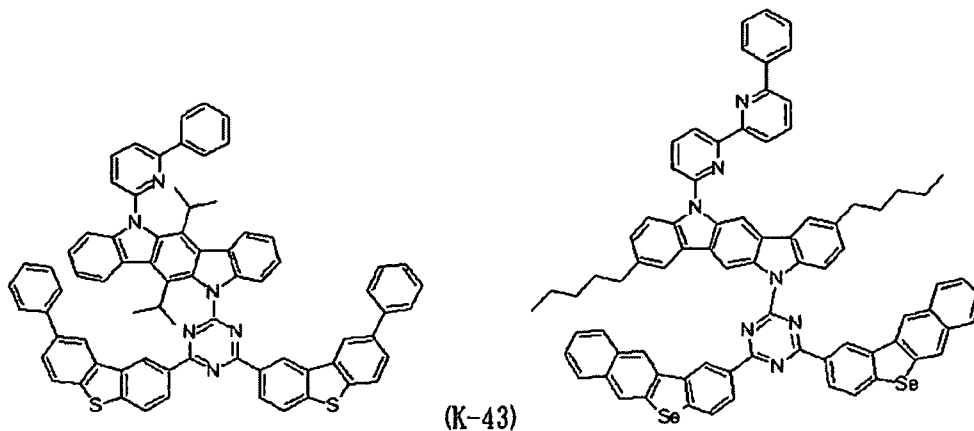
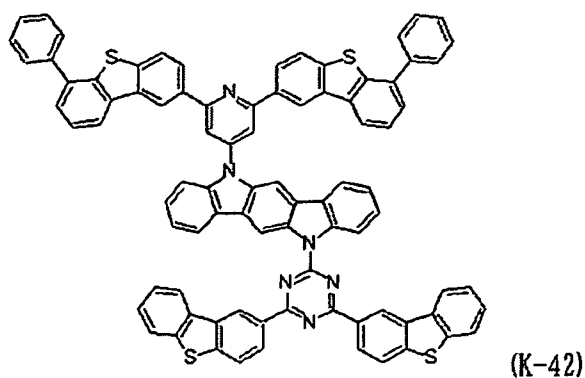
(K-34)



(K-35)







【0064】上述通式(1)所表示的吲哚并咪唑化合物含有於在基板上積層有陽極、多層有機層及陰極而成的有機 EL 元件的至少一層有機層中，藉此形成優異的有機電場發光元件。所含有的有機層合適的是磷光發光層、電洞傳輸層、電子傳輸層、電洞阻擋層或電子阻擋層。更佳為以作為含有磷光發光摻雜劑的發光層的主體材料而含有為宜。

【0065】 繼而，對本發明的有機 EL 元件加以說明。

【0066】 本發明的有機 EL 元件於積層於基板上的陽極與陰極之間具有至少一層包含發光層有機層，且至少一層有機層含有上述吡啶并吡嗪化合物。有利的是使本發明的有機電場發光元件用材料與磷光發光摻雜劑一併含有於發光層中。

【0067】 繼而，一面參照圖式一面對本發明的有機 EL 元件的結構加以說明，但本發明的有機 EL 元件的結構絲毫不限定於圖示的結構。

【0068】 圖 1 為表示本發明中所用的通常的有機 EL 元件的結構例的剖面圖，1 表示基板，2 表示陽極，3 表示電洞注入層，4 表示電洞傳輸層，5 表示發光層，6 表示電子傳輸層，7 表示陰極。於本發明的有機 EL 元件中，亦可與發光層鄰接而具有激子阻擋層，另外，亦可於發光層與電洞注入層之間具有電子阻擋層。激子阻擋層亦可於發光層的陽極側、陰極側之任一側插入，亦可於兩側同時插入。於本發明的有機 EL 元件中，具有基板、陽極、發光層及陰極作為必需層，於必需層以外的層中，以具有電洞注入傳輸層、電子注入傳輸層為宜，進而以於發光層與電子注入傳輸層之間具有電洞阻擋層為宜。再者，電洞注入傳輸層是指電洞注入層與電洞傳輸層中的任一者或兩者，電子注入傳輸層是指電子注入層與電子傳輸層中的任一者或兩者。

【0069】 再者，亦可為與圖 1 相反的結構，即於基板 1 上依序積層陰極 7、電子傳輸層 6、發光層 5、電洞傳輸層 4、陽極 2，於該

情形時，亦視需要可追加或省略層。

#### 【0070】 -基板-

本發明的有機 EL 元件較佳為由基板所支撐。該基板並無特別限制，只要為先前以來於有機 EL 元件中慣用者即可，例如可使用包含玻璃、透明塑膠、石英等的基板。

#### 【0071】 -陽極-

有機 EL 元件中的陽極可較佳地使用以功函數大的（4 eV 以上）金屬、合金、導電性化合物及該等的混合物作為電極物質者。此種電極物質的具體例可列舉：Au 等金屬、CuI、氧化銦錫（ITO）、 $\text{SnO}_2$ 、ZnO 等導電性透明材料。另外，亦可使用 IDIXO（ $\text{In}_2\text{O}_3$ -ZnO）等非晶質且可製作透明導電膜的材料。陽極是藉由蒸鍍或濺鍍等方法使該些電極物質形成薄膜，可利用光微影法來形成所需形狀的圖案，或於不大需要圖案精度的情形時（100  $\mu\text{m}$  以上左右），亦可於上述電極物質的蒸鍍或濺鍍時經由所需形狀的遮罩來形成圖案。或者，於使用有機導電性化合物般可進行塗佈的物質的情形時，亦可使用印刷方式、塗佈方式等濕式成膜法。於自該陽極取出發光的情形時，理想的是使透射率大於 10%，另外，作為陽極的片材電阻較佳為數百  $\Omega/\square$  以下。進而，膜厚亦取決於材料，但通常於 10 nm~1000 nm、較佳為 10 nm~200 nm 的範圍內選擇。

#### 【0072】 -陰極-

另一方面，陰極可使用以功函數小的（4 eV 以下）金屬（稱為電子注入性金屬）、合金、導電性化合物及該等的混合物作為電

極物質者。此種電極物質的具體例可列舉：鈉、鈉-鉀合金、鎂、鋰、鎂/銅混合物、鎂/銀混合物、鎂/鋁混合物、鎂/銦混合物、鋁/氧化鋁 ( $\text{Al}_2\text{O}_3$ ) 混合物、銦、鋰/鋁混合物、稀土金屬等。該等中，就電子注入性及對氧化等的耐久性的方面而言，較佳為電子注入性金屬與功函數的值較其更大的穩定金屬即第二金屬的混合物，例如鎂/銀混合物、鎂/鋁混合物、鎂/銦混合物、鋁/氧化鋁 ( $\text{Al}_2\text{O}_3$ ) 混合物、鋰/鋁混合物、鋁等。陰極可藉由以下方式製作：藉由蒸鍍或濺鍍等方法使該些電極物質形成薄膜。另外，作為陰極的片材電阻較佳為數百  $\Omega/\square$  以下，膜厚通常於  $10\text{ nm} \sim 5\ \mu\text{m}$ 、較佳為  $50\text{ nm} \sim 200\text{ nm}$  的範圍內選擇。再者，為了使所發出的光透射，若有機 EL 元件的陽極或陰極的任一者為透明或半透明，則發光亮度提高而合適。

【0073】 另外，於陰極上以  $1\text{ nm} \sim 20\text{ nm}$  的膜厚製作上述金屬後，於其上製作陽極的說明中列舉的導電性透明材料，藉此可製作透明或半透明的陰極，藉由應用上述電極，可製作陽極與陰極兩者具有透射性的元件。

#### 【0074】 -發光層-

發光層為磷光發光層，含有磷光發光摻雜劑及主體材料。磷光發光摻雜材料以含有以下有機金屬錯合物為宜，上述有機金屬錯合物含有選自鈮、銻、鈹、銀、銻、鐵、銻、鉑及金中的至少一種金屬。具體可列舉以下的專利公報中記載的化合物，但不限定於該些化合物。

【0075】 WO2009-073245 號公報、WO2009-046266 號公報、  
WO2007-095118 號公報、WO2008-156879 號公報、WO2008-140657  
號公報、US2008-261076 號公報、日本專利特表 2008-542203 號公  
報、WO2008-054584 號公報、日本專利特表 2008-505925 號公報、  
日本專利特表 2007-522126 號公報、日本專利特表 2004-506305 號  
公報、日本專利特表 2006-513278 號公報、日本專利特表  
2006-50596 號公報、WO2006-046980 號公報、WO2005113704 號  
公報、US2005-260449 號公報、US2005-2260448 號公報、  
US2005-214576 號公報、WO2005-076380 號公報、US2005-119485  
號公報、WO2004-045001 號公報、WO2004-045000 號公報、  
WO2006-100888 號公報、WO2007-004380 號公報、WO2007-023659  
號公報、WO2008-035664 號公報、日本專利特開 2003-272861 號  
公報、日本專利特開 2004-111193 號公報、日本專利特開  
2004-319438 號公報、日本專利特開 2007-2080 號公報、日本專利  
特開 2007-9009 號公報、日本專利特開 2007-227948 號公報、日本  
專利特開 2008-91906 號公報、日本專利特開 2008-311607 號公報、  
日本專利特開 2009-19121 號公報、日本專利特開 2009-46601 號公  
報、日本專利特開 2009-114369 號公報、日本專利特開 2003-253128  
號公報、日本專利特開 2003-253129 號公報、日本專利特開  
2003-253145 號公報、日本專利特開 2005-38847 號公報、日本專  
利特開 2005-82598 號公報、日本專利特開 2005-139185 號公報、  
日本專利特開 2005-187473 號公報、日本專利特開 2005-220136 號

公報、日本專利特開 2006-63080 號公報、日本專利特開  
2006-104201 號公報、日本專利特開 2006-111623 號公報、日本專  
利特開 2006-213720 號公報、日本專利特開 2006-290891 號公報、  
日本專利特開 2006-298899 號公報、日本專利特開 2006-298900 號  
公報、WO2007-018067 號公報、WO2007/058080 號公報、  
WO2007-058104 號公報、日本專利特開 2006-131561 號公報、日  
本專利特開 2008-239565 號公報、日本專利特開 2008-266163 號公  
報、日本專利特開 2009-57367 號公報、日本專利特開 2002-117978  
號公報、日本專利特開 2003-123982 號公報、日本專利特開  
2003-133074 號公報、日本專利特開 2006-93542 號公報、日本專  
利特開 2006-131524 號公報、日本專利特開 2006-261623 號公報、  
日本專利特開 2006-303383 號公報、日本專利特開 2006-303394 號  
公報、日本專利特開 2006-310479 號公報、日本專利特開  
2007-88105 號公報、日本專利特開 2007-258550 號公報、日本專  
利特開 2007-324309 號公報、日本專利特開 2008-270737 號公報、  
日本專利特開 2009-96800 號公報、日本專利特開 2009-161524 號  
公報、WO2008-050733 號公報、日本專利特開 2003-73387 號公報、  
日本專利特開 2004-59433 號公報、日本專利特開 2004-155709 號  
公報、日本專利特開 2006-104132 號公報、日本專利特開  
2008-37848 號公報、日本專利特開 2008-133212 號公報、日本專  
利特開 2009-57304 號公報、日本專利特開 2009-286716 號公報、  
日本專利特開 2010-83852 號公報、日本專利特表 2009-532546 號

為第 102108503 號中文說明書無劃線修正本

修正日期:102 年 6 月 26 日

公報、日本專利特表 2009-536681 號公報、日本專利特表

2009-542026 號公報等。

【0076】較佳的磷光發光摻雜劑可列舉：具有 Ir 等貴金屬元素作為中心金屬的  $\text{Ir(ppy)}_3$  等錯合物類、 $\text{Ir(bt)}_2\cdot\text{acac}_3$  等錯合物類、 $\text{PtOEt}_3$  等錯合物類。以下示出該些錯合物類的具體例，但不限定於下述化合物。





【0078】 上述磷光發光摻雜劑含有於發光層中的量以處於 2 重量%~40 重量%、較佳為 5 重量%~30 重量%的範圍內為宜。

【0079】 發光層中的主體材料較佳為使用上述通式(1)所表示的吡啶并咪唑化合物。然而，於將該吡啶并咪唑化合物用於發光層以外的其他任一有機層中的情形時，發光層中使用的材料亦可為吡啶并咪唑化合物以外的其他主體材料。另外，亦可將吡啶并咪唑化合物與其他主體材料併用。進而，亦可將多種公知的主體材料併用使用。

【0080】 可使用的公知的主體化合物較佳為具有電洞傳輸能力、電子傳輸能力，且防止發光的長波長化，而且具有高的玻璃轉移溫度的化合物。

【0081】 此種其他主體材料已藉由大量的專利文獻等而為人所知，可自該等中選擇。主體材料的具體例並無特別限定，可列舉：吡啶衍生物、咪唑衍生物、三唑衍生物、噁唑衍生物、噁二唑衍生物、咪唑衍生物、聚芳基烷烴衍生物、吡啶啉衍生物、吡啶啉酮衍生物、伸苯基二胺衍生物、芳基胺衍生物、胺基取代查爾酮衍生物、苯乙烯基蔥衍生物、萘酮衍生物、腺衍生物、二苯乙烯衍生物、矽氮烷衍生物、芳香族第三級胺化合物、苯乙烯基胺化合物、芳香族二次甲基系化合物、吡啶系化合物、蔥醌二甲烷衍生物、蔥醌衍生物、二苯醌衍生物、二氧化噻喃衍生物、萘苊等的雜環四羧酸酐，酞菁衍生物、8-羥基喹啉衍生物的金屬錯合物或金屬酞菁、苯并噁唑或苯并噻唑衍生物的金屬錯合物所代表的

各種金屬錯合物，聚矽烷系化合物、聚(N-乙烷基咪唑)衍生物、苯胺系共聚物、噻吩低聚物、聚噻吩衍生物、聚苯衍生物、聚苯乙炔衍生物、聚萘衍生物等高分子化合物等。

**【0082】 -注入層-**

所謂注入層，是指爲了降低驅動電壓或提高發光亮度而設於電極與有機層之間的層，有電洞注入層與電子注入層，亦可存在於陽極與發光層或電洞傳輸層之間、及陰極與發光層或電子傳輸層之間。注入層可視需要而設置。

**【0083】 -電洞阻擋層-**

所謂電洞阻擋層，於廣義上具有電子傳輸層的功能，包含具有傳輸電子的功能且傳輸電洞的能力明顯小的電洞阻擋材料，藉由傳輸電子且阻擋電洞，可提高電子與電洞的再結合概率。

**【0084】** 於電洞阻擋層中較佳爲使用通式(1)所表示的吡啶并咪唑化合物，但於將吡啶并咪唑化合物用於其他任一有機層中的情形時，亦可使用公知的電洞阻擋層材料。另外，電洞阻擋層材料可視需要而使用後述電子傳輸層的材料。

**【0085】 -電子阻擋層-**

所謂電子阻擋層，包含具有傳輸電洞的功能且傳輸電子的能力明顯小的材料，藉由傳輸電洞且阻擋電子，可提高電子與電洞再結合的概率。

**【0086】** 電子阻擋層的材料可使用本發明的通式(1)所表示的吡啶并咪唑化合物，亦可視需要而使用後述電洞傳輸層的材料作爲

其他材料。電子阻擋層的膜厚較佳為 3 nm~100 nm，更佳為 5 nm~30 nm。

#### 【0087】 -激子阻擋層-

所謂激子阻擋層，是指用以阻擋藉由電洞與電子於發光層內再結合而生成的激子擴散至電荷傳輸層中的層，可藉由插入該層而將激子有效地封閉於發光層內，從而可提高元件的發光效率。激子阻擋層可與發光層鄰接而於陽極側、陰極側的任一側插入，亦可於兩側同時插入。

【0088】 激子阻擋層的材料可使用通式 (1) 所表示的吡啶并咪唑化合物，其他材料例如可列舉：1,3-二咪唑基苯 (mCP) 或雙(2-甲基-8-羥基喹啉)-4-苯基酚基鋁 (III) (BALq)。

#### 【0089】 -電洞傳輸層-

所謂電洞傳輸層，包含具有傳輸電洞的功能的電洞傳輸材料，電洞傳輸層可設置單層或多層。

【0090】 電洞傳輸材料具有電洞的注入或傳輸、電子的阻斷性的任一種，可為有機物、無機物的任一種。電洞傳輸層中較佳為使用通式 (1) 所表示的吡啶并咪唑化合物，但可自先前公知的化合物中選擇使用任意者。可使用的公知的電洞傳輸材料例如可列舉：三唑衍生物、噁二唑衍生物、咪唑衍生物、聚芳基烷烴衍生物、吡啶衍生物及吡啶酮衍生物、伸苯基二胺衍生物、芳基胺衍生物、胺基取代查爾酮衍生物、噁唑衍生物、苯乙烯基噻衍生物、萘酮衍生物、脞衍生物、二苯乙烯衍生物、矽氮烷衍生物、

苯胺系共聚物，另外可列舉導電性高分子低聚物、特別是噻吩低聚物等，較佳為使用吡啶化合物、芳香族三級胺化合物及苯乙烯基胺化合物，更佳為使用芳香族三級胺化合物。

### 【0091】 -電子傳輸層-

所謂電子傳輸層，包含具有傳輸電子的功能性的材料，電子傳輸層可設置單層或多層。

【0092】 電子傳輸材料（有時亦兼作電洞阻擋材料）只要具有將自陰極注入的電子傳至發光層中的功能即可。電子傳輸層中較佳為使用本發明的通式（1）所表示的材料，但可自先前公知的化合物中選擇使用任意者，例如可列舉：硝基取代萘衍生物、二苯醌衍生物、二氧化噻喃衍生物、碳二醯亞胺、伸萘基甲烷（fluorenylidene methane）衍生物、蔥醌二甲烷及蔥酮衍生物、噁二唑衍生物等。進而，上述噁二唑衍生物中，將噁二唑環的氧原子取代為硫原子的噻二唑衍生物、具有作為吸電子基而廣為人知的喹噁啉環的喹噁啉衍生物亦可用作電子傳輸材料。進而，亦可使用將該些材料導入至高分子鏈中、或以該些材料作為高分子的主鏈的高分子材料。

### [實施例]

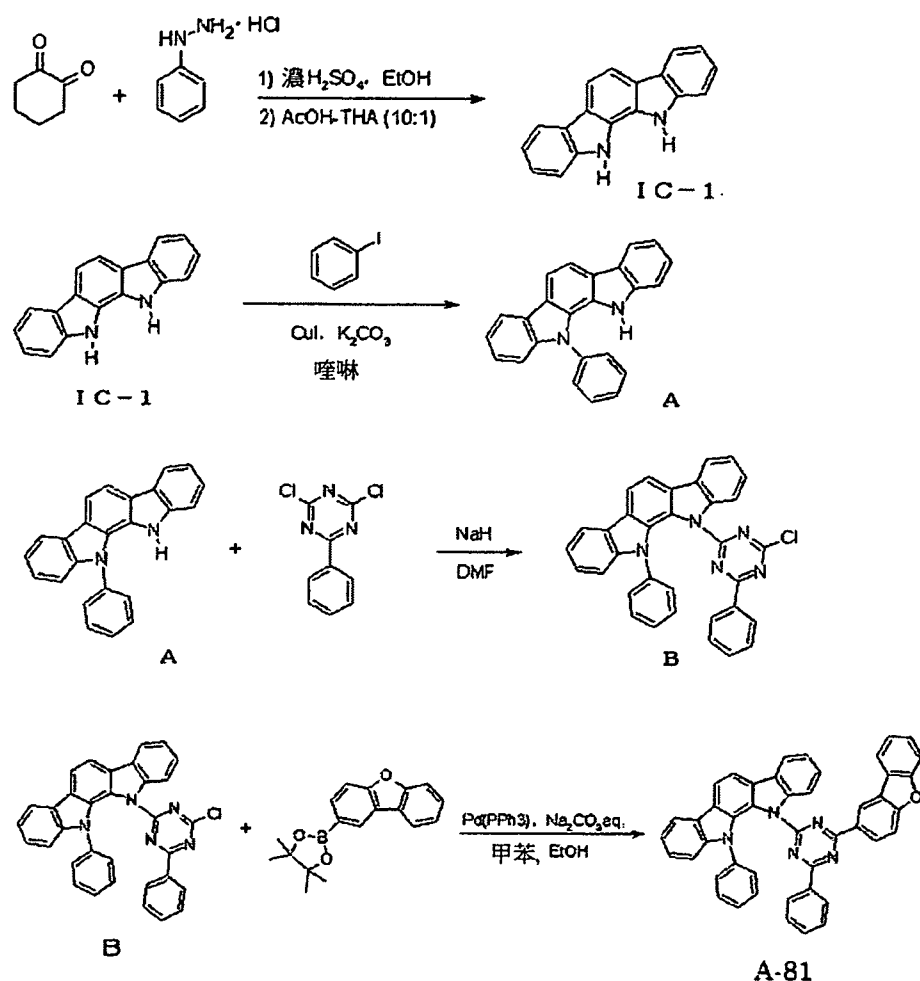
【0093】 以下，藉由實施例對本發明加以更詳細說明，但本發明當然不限定於該些實施例，只要不超出其主旨，則能以各種形態來實施。

【0094】 藉由以下所示的路徑來合成作為磷光發光元件用材料的

吡啶并咪唑化合物。另外，化合物編號與對上述例示化合物標註的編號相對應。

### 【0095】 合成例 1

(化合物 A-81) 的合成



【0096】 於氮氣環境下，一面於室溫下攪拌 1,2-環己二酮 33.3 g (0.30 mol)、苯基肼鹽酸鹽 86.0 g (0.60 mol) 及乙醇 1000 ml，一面用 5 分鐘滴加濃硫酸 3.0 g (0.031 mol) 後，一面於 65°C 下加熱一面攪拌 4 小時。將反應溶液冷卻至室溫為止後，濾取所析出的

結晶，使用乙醇（2×500 ml）進行清洗，獲得紫褐色結晶 80.0 g。將該結晶 72.0 g（0.28 mol）、三氟乙酸 72.0 g 及乙酸 720.0 g 一面於 100°C 下加熱一面攪拌 15 小時。將反應溶液冷卻至室溫為止後，濾取所析出的結晶，以乙酸（200 ml）進行清洗。進行再製漿純化，獲得作為白色結晶的 11,12-二氫吡啶并[2,3-a]吡啶（IC-1）30.0 g（0.12 mol，產率為 40%）。

【0097】 繼而，於經氮氣置換的 1000 ml 三口燒瓶中，添加上述所得的白色粉末 26.0 g（0.10 mol）、碘苯 122.7 g（0.60 mol）、碘化銅 54.7 g（0.29 mol）、碳酸鉀 66.7 g（0.48 mol）及喹啉 800 ml 並進行攪拌。其後，加熱至 190°C 為止並攪拌 72 小時。暫且冷卻至室溫為止後，添加水 500 ml 及二氯甲烷 500 ml，進行攪拌後，濾取所產生的黃色結晶。將濾液移至 2000 ml 分液漏斗中，分離為有機層與水層。利用 500 ml 的水將有機層清洗三次，其後利用硫酸鎂對所得的有機層進行脫水，暫且過濾分離硫酸鎂後，將溶劑減壓蒸餾去除。其後，利用管柱層析法（column chromatography）進行純化，獲得作為白色固體的中間體 A 13.7 g（0.041 mol，產率為 40.6%）。

【0098】 於氮氣環境下，添加 60%氫化鈉 8.46 g（0.21 mol）及脫水 N,N-二甲基甲醯胺 150 ml，進行攪拌。繼而，滴加使中間體 A 50.0 g（0.15 mol）溶解於脫水 N,N-二甲基甲醯胺 150 ml 中所得的溶液。然後繼續攪拌 1 小時。繼而將反應溶液冷卻至 -40°C，使 2,4-二氯-6-苯基-1,3,5-三嗪 37.4 g（0.17 mol）溶解於脫水 N,N-二甲基

甲醯胺 200 ml 中，滴加該溶液，於室溫下繼續攪拌 5 小時。繼而於該燒瓶內添加蒸餾水 1000 ml 及甲醇 200 ml，於室溫下攪拌 1 小時後，濾取所析出的黃色固體。使濾取的黃色固體溶解於二氯甲烷中，利用蒸餾水（100 ml×2 次）清洗後，利用無水硫酸鎂加以乾燥。將硫酸鎂過濾分離後，將溶劑減壓蒸餾去除。其後，利用甲醇 600 ml 對所得的固體進行再製漿純化，並加以乾燥，藉此獲得中間體 B 67.2 g（0.13 mol，產率為 61%）。

● **【0099】** 於氮氣環境下，添加中間體 B 30.0 g（0.057 mol）、2-(4,4,5,5-四甲基-1,3,2-二氮雜硼烷-2-基)二苯并呋喃 18.6 g(0.063 mol)、四(三苯基磷)鈮(0)0.73 g（0.00063 mol）、甲苯 300 ml 及乙醇 150 ml，於室溫下攪拌。其後，添加使碳酸鈉 53.4 g（0.50 mol）溶解於蒸餾水 300 ml 中所得的溶液，回流 14 小時。將反應溶液冷卻至室溫為止後，利用蒸餾水（2×150 ml）清洗有機層，利用無水硫酸鎂將有機層乾燥。其後，將硫酸鎂過濾分離後，將溶劑減壓蒸餾去除。將所得的固體溶解於四氫呋喃中，添加活性炭及漂白土（Fuller's earth）並於室溫下攪拌 1 小時。將活性炭及漂白土過濾分離後，將溶劑減壓蒸餾去除，對所得的殘渣進行矽膠管柱層析純化及再製漿純化，藉此獲得作為白色固體的化合物（A-81）26.5 g（0.041 mol，產率為 71%）。

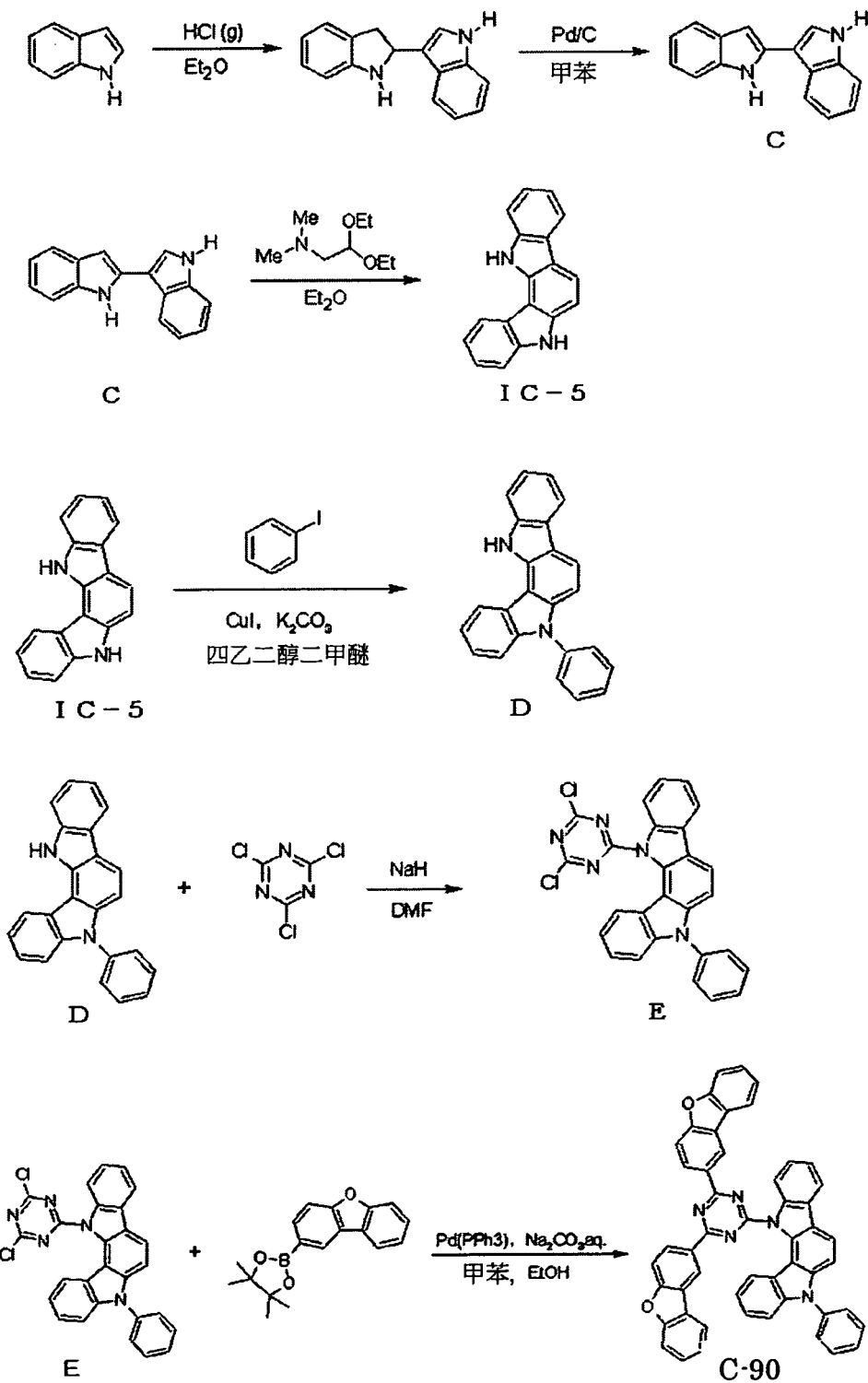
將常壓化學離子化飛行時間質譜（Atmospheric Pressure Chemical Ionization-Time-of-Flight Mass Spectrometry，APCI-TOFMS）， $m/z$  654  $[M+H]^+$ 、 $^1H$ -NMR 測定結果（測定溶劑：



THF-d8) 示於圖 2 中。

## 【0100】 合成例 2

(化合物 C-90) 的合成



【0101】於氮氣環境下，一面於室溫下攪拌吡啶 20.0 g (0.17 mol) 的脫水二乙醚 300 ml 溶液，一面吹入於濃硫酸 211.7 g (2.16 mol) 中用 1 小時滴加濃鹽酸 112.0 g (1.10 mol) 而產生的氯化氫氣體。將反應溶液於室溫下攪拌 15 小時後，添加乙酸乙酯 121.0 g 及飽和碳酸氫鈉水溶液 303.2 g。利用乙酸乙酯 (2×100 ml) 萃取水層後，利用飽和碳酸氫鈉水溶液 (100 ml) 及蒸餾水 (2×100 ml) 來清洗有機層。利用無水硫酸鎂將有機層加以乾燥後，將硫酸鎂過濾分離，將溶劑減壓蒸餾去除。將所得的殘渣溶解於甲苯 150 ml 中，添加鈀/活性炭 2.5 g 後，一面於 111°C 下加熱回流一面攪拌 3 小時。將反應溶液冷卻至室溫為止後，將鈀/活性炭過濾分離，將溶劑減壓蒸餾去除。藉由再結晶進行純化，獲得作為白色結晶的中間體 C 14.7 g (0.063 mol，產率為 37%)。

【0102】於氮氣環境下，將中間體 C 14.1 g (0.061 mol)、N,N'-二甲基胺基乙醛二乙基縮醛 11.4 g (0.071 mol) 及乙酸 110.0 g 一面於 118°C 下加熱回流，一面攪拌 8 小時。將反應溶液冷卻至室溫為止後，濾取所析出的結晶，利用乙酸 (30 ml) 進行清洗。對所得的結晶進行再製漿純化，獲得作為白色結晶的 5,12-二氫吡啶并 [3,2-a] 吡啶 (IC-5) 10.4 g (0.041 mol，產率為 67%)。

【0103】於氮氣環境下，添加 (IC-5) 10.0 g (0.039 mol)、碘苯 39.8 g (0.20 mol)、銅 6.2g (0.098 mol)、碳酸鉀 8.1 g (0.059 mol) 及四乙二醇二甲醚 200 ml 並進行攪拌。其後加熱至 190°C 為止，

攪拌 24 小時。將反應溶液冷卻至室溫為止後，將銅、無機物過濾分離。於濾液中添加蒸餾水 200 ml 並進行攪拌，將析出的結晶過濾分離。將其減壓乾燥後，利用管柱層析法純化而獲得白色粉末的中間體 D 9.7 g (0.029 mol, 產率為 75.0%)。

【0104】於氮氣環境下，添加 60% 氫化鈉 1.3 g (0.033 mol) 及脫水 N,N-二甲基甲醯胺 50 ml，進行攪拌。繼而滴加使中間體 D 10.0 g (0.030 mol) 溶解於脫水 N,N-二甲基甲醯胺 100 ml 中所得的溶液。其後，繼續攪拌 30 分鐘。繼而，使 2,4,6-三氯-1,3,5-三嗪 5.5 g (0.030 mol) 溶解於脫水 N,N-二甲基甲醯胺 50 ml 中，於 -60°C 下滴加該溶液，於 -60°C 下攪拌 1 小時，於室溫下攪拌 1 小時。繼而於該燒瓶內添加蒸餾水 500 ml，於室溫下攪拌 1 小時後，濾取所析出的固體。使濾取的固體溶解於甲苯中，利用蒸餾水 (100 ml×2 次) 清洗後，利用無水硫酸鎂進行乾燥。將硫酸鎂過濾分離後，將溶劑減壓蒸餾去除。其後，利用管柱層析法將所得的固體純化，藉此獲得含有 50% 的中間體 D 的狀態的中間體 E 10.0 g (0.010 mol, 產率為 35%)。

【0105】於氮氣環境下，添加中間體 E 5.0 g (0.010 mol)、2-(4,4,5,5-四甲基-1,3,2-二氮雜硼烷-2-基)二苯并呋喃 7.35 g (0.025 mol)、四(三苯基膦)鈀(0) 0.58 g (0.00050 mol)、甲苯 100 ml 及乙醇 50 ml，於室溫下攪拌。其後，添加使碳酸鈉 21.2 g (0.20 mol) 溶解於蒸餾水 100 ml 中所得的溶液，於 90°C 下攪拌 3 小時。將反應溶液冷卻至室溫為止後，對所得的固體進行矽膠管柱層析純化

及再製漿純化，藉此獲得作為白色固體的化合物（C-90）1.3 g（0.0017 mol，產率為 17%）。

將 APCI-TOFMS， $m/z$  744  $[M+H]^+$ 、 $^1H$ -NMR 測定結果（測定溶劑：THF- $d_8$ ）示於圖 3 中。

#### 【0106】 實施例 1

於形成有膜厚為 110 nm 的包含 ITO 的陽極的玻璃基板上，利用真空蒸鍍法以  $4.0 \times 10^{-5}$  Pa 的真空度積層各薄膜。首先，於 ITO 上以 25 nm 的厚度形成銅酞菁（CuPC）。繼而，以 40 nm 的厚度形成 4,4'-雙[N-(1-萘基)-N-苯基氨基]聯苯（NPB）作為電洞傳輸層。繼而，於電洞傳輸層上，自不同的蒸鍍源將作為主體材料的化合物（A-81）與作為磷光發光摻雜劑的三(2-苯基吡啶)銱（III）（ $Ir(ppy)_3$ ）共蒸鍍，以 40 nm 的厚度形成發光層。發光層中的  $Ir(ppy)_3$  的濃度為 10.0 wt%。繼而，以 20 nm 的厚度形成三(8-羥基喹啉)鋁（III）（ $Alq_3$ ）作為電子傳輸層。進而，於電子傳輸層上，以 1.0 nm 的厚度形成氟化鋰（LiF）作為電子注入層。最後，於電子注入層上以 70 nm 的厚度形成鋁（Al）作為電極，製作有機 EL 元件。

【0107】 對所得的有機 EL 元件連接外部電源並施加直流電壓，結果確認到具有如表 1 般的發光特性。於表 1 中，亮度、電壓及發光效率表示 20 mA/cm<sup>2</sup> 下的值。元件發光光譜的極大波長為 520 nm，可知獲得了來自  $Ir(ppy)_3$  的發光。

#### 【0108】 實施例 2～實施例 17

與合成例 1 及合成例 2 同樣地合成化合物 A-24、化合物 B-17、化合物 B-46、化合物 C-40、化合物 C-113、化合物 D-92、化合物 E-64、化合物 F-15、化合物 G-8、化合物 H-11、化合物 H-38、化合物 J-37、化合物 K-35、化合物 K-37 及化合物 K-45。作為實施例 1 的發光層的主體材料，使用化合物 A-24、化合物 B-17、化合物 B-46、化合物 C-40、化合物 C-90、化合物 C-113、化合物 D-92、化合物 E-64、化合物 F-15、化合物 G-8、化合物 H-11、化合物 H-38、化合物 J-37、化合物 K-35、化合物 K-37 及化合物 K-45 代替化合物 A-81，除此以外，與實施例 1 同樣地製作有機 EL 元件。各元件發光光譜的極大波長為 520 nm，可知獲得了來自 Ir(ppy)<sub>3</sub> 的發光。將各元件的發光特性示於表 1 中。

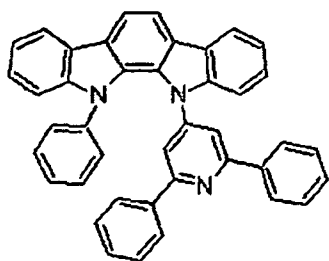
**【0109】 例 18 (比較)**

除了使用 CBP 作為發光層的主體材料以外，與實施例 1 同樣地製作有機 EL 元件。

**【0110】 例 19 (比較)**

除了使用下述化合物 (Ho-1) 作為發光層的主體材料以外，與實施例 1 同樣地製作有機 EL 元件。

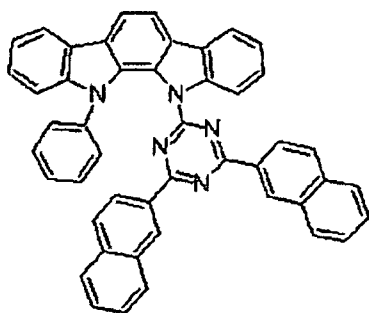
**【0111】**



(Ho-1)

## 【0112】 例 20 (比較)

除了使用下述化合物 (Ho-2) 作為發光層的主體材料以外，  
與實施例 1 同樣地製作有機 EL 元件。



(Ho-2)

【0113】 例 18~例 20 中製作的有機 EL 元件的元件發光光譜的極  
大波長均為 520 nm，可知獲得了來自 Ir(ppy)<sub>3</sub> 的發光。示出用作  
主體材料的化合物及作為 X 數的該化合物的式 (1b) 中的 X<sub>1</sub>~X<sub>4</sub>  
的合計值。將 20 mA/cm<sup>2</sup> 下的各發光特性示於表 1 中。X 數為 X<sub>1</sub>  
~X<sub>4</sub> 的總數。

【0114】 [表 1]

	化合物 (X 數)	亮度 (cd/m <sup>2</sup> )	電壓 (V)	視覺發光效率 (lm/W)
實施例 1	A-81 (1)	5680	6.7	13.3
實施例 2	A-24 (4)	5880	6.5	14.2
實施例 3	B-17 (5)	5850	6.7	13.7
實施例 4	B-46 (5)	5835	6.5	14.1
實施例 5	C-40 (5)	5870	6.4	14.4
實施例 6	C-90 (1)	5830	6.9	13.3
實施例 7	C-113 (4)	5860	6.5	14.2
實施例 8	D-92 (7)	5870	6.6	14.0
實施例 9	E-64 (3)	5760	6.9	13.1
實施例 10	F-15 (5)	5845	6.5	14.1
實施例 11	G-8 (3)	5580	7.0	12.5
實施例 12	H-11 (7)	5790	6.4	14.2
實施例 13	H-38 (4)	5800	6.5	14.0
實施例 14	J-37 (1)	5675	6.8	13.1
實施例 15	K-35 (3)	5720	6.8	13.2
實施例 16	K-37 (7)	5865	6.5	14.2
實施例 17	K-45 (1)	5590	6.8	12.9
例 18	CBP	4860	9.3	8.2
例 19	Ho-1	4713	7.4	10.0
例 20	Ho-2	3980	5.9	10.6

【0115】 由表 1 得知，相對於使用作為磷光主體而通常為人所知的 CBP 的情形，使用通式 (1) 所表示的吡啶并咪唑化合物的有機 EL 元件顯示良好的發光特性。另外，與使用作為於吡啶并咪唑的 N 上具有含氮 6 員環、進而於該含氮 6 員環上具有並非縮合雜環基的取代基的化合物的 Ho-1 及 Ho-2 的情形相比較，顯示良好的發光特性。由以上內容表明使用上述吡啶并咪唑化合物的有機 EL 元件的優越性。

[產業上之可利用性]

【0116】可認為，本發明的有機電場發光元件中使用的吡啶并咪唑化合物於吡啶并咪唑骨架的 2 個 N 上的一個上具有含氮 6 員環基，進而於該含氮 6 員環上具有縮合雜環基作為取代基，藉此保有高的 T1，顯示良好的電洞及電子的注入傳輸特性，且具有高的耐久性。尤其於縮合雜環基為二苯并咪喃基及二苯并噻吩基的情形時，於含氮 6 員環及二苯并咪喃環或二苯并噻吩環上，最低未佔分子軌道（Lowest Unoccupied Molecular Orbital, LUMO）的軌道變寬，可期待顯示良好的電子傳輸性。進而，藉由變更作為另一 N 上的取代基的芳香環的種類或數量，可實現電洞、電子移動速度的微調整以及 IP、EA、T1 的各種能量值的控制。尤其於將芳香環連結 4 個以上、較佳為 5 個以上的情形時，共軛系變廣，由此分子間的重疊變佳，不僅可期待分子的電子穩定性的提高，而且可提供移動度高的材料。

根據以上內容，使用該吡啶并咪唑化合物的有機 EL 元件可對發光層中的多種摻雜劑實現最適的載子平衡，結果可提供大幅度地改善了發光特性的有機 EL 元件。進而，該吡啶并咪唑化合物可於氧化、還原、激發的各活性狀態下提高穩定性，同時具有良好的非晶特性，故驅動壽命長，可實現耐久性高的有機 EL 元件。

【0117】本發明的有機 EL 元件於發光特性、驅動壽命及耐久性方面在實用上為可令人滿意的水準內，於對平板顯示器（行動電話顯示元件、車載顯示元件、辦公自動化（Office Automation, OA）電腦顯示元件或電視等）、發揮作為面發光體的特徵的光源（照



明、影印機的光源、液晶顯示器或量表類的背光光源)、顯示板或標識燈等的應用中，其技術價值大。

### 【符號說明】

#### 【0118】

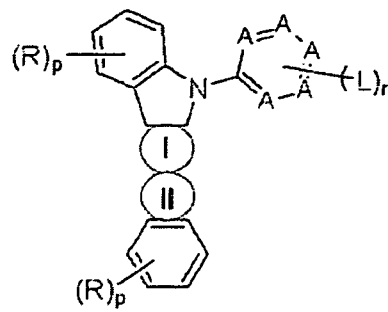
- 1：基板
- 2：陽極
- 3：電洞注入層
- 4：電洞傳輸層
- 5：發光層
- 6：電子傳輸層
- 7：陰極

為第 102108503 號中文專利範圍無劃線修正本

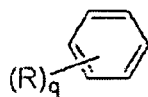
修正日期:104 年 12 月 9 日

## 申請專利範圍

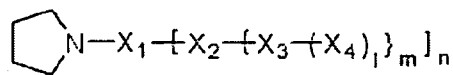
1. 一種有機電場發光元件，其是於基板上積層有陽極、包含磷光發光層的多層有機層及陰極而成，上述有機電場發光元件的特徵在於：於選自由磷光發光層、電洞傳輸層、電子傳輸層及電洞阻擋層所組成的群組中的至少一層有機層中，含有通式(1)所表示的吲哚并吡啶化合物，



(1)



(1a)



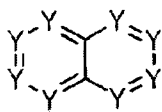
(1b)

此處，環 I 表示於任意的位置與鄰接環縮合的式(1a)所表示的芳香族烴環，環 II 表示於任意的位置與鄰接環縮合的式(1b)所表示的雜環；A 表示 C-R 或 N，但至少一個為 N；L 獨立地表示經取代或未經取代的碳數 6~18 的芳香族烴基或碳數 3~17 的芳香族雜環基，r 表示 1~4 的整數，L 的至少一個為下述式(2)或式(3)的任一個所表示的一價的縮合雜環基；通式(1)及式(1a)中，R 分別獨立地表示碳數 1~10 的脂肪族烴基、碳數 6~18 的

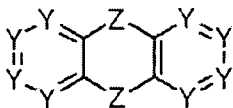
為第 102108503 號中文專利範圍無劃線修正本

修正日期:104 年 12 月 9 日

芳香族烴基或碳數 3~17 的芳香族雜環基，p 分別獨立地表示 0~4 的整數，q 表示 0~2 的整數；式 (1b) 中， $X_1 \sim X_4$  分別獨立地表示碳數 6~18 的芳香族烴基或碳數 3~17 的芳香族雜環基，l、m 及 n 分別獨立地表示 0~5 的整數；此處，於 l、m 或 n 為 2 以上的情形時， $X_2$ 、 $X_3$  及  $X_4$  分別可相同亦可不同，



(2)



(3)

此處，Y 分別獨立地表示次甲基、取代次甲基或氮，Y 的任一個為形成一價基的碳原子；Z 獨立地表示單鍵、-S-或-O-的任一個，於式 (2) 中 Y 的至少一個為雜原子，於式 (3) 中 Y 或 Z 的至少一個為雜原子。

2. 如申請專利範圍第 1 項所述的有機電場發光元件，其中於上述通式 (1) 中，至少一個 L 為上述式 (3) 所表示的一價芳香族雜環基。

3. 如申請專利範圍第 1 項所述的有機電場發光元件，其中於上述通式 (1) 中，至少一個 L 為經取代或未經取代的二苯并咪喃基或二苯并噻吩基。

4. 如申請專利範圍第 1 項所述的有機電場發光元件，其中於

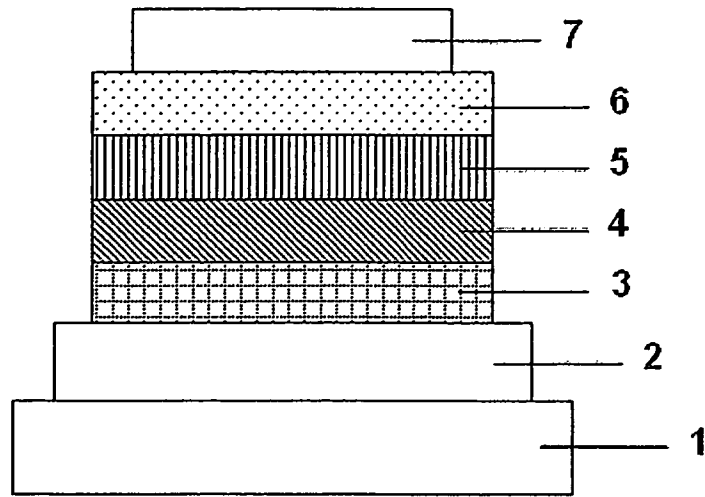
為第 102108503 號中文專利範圍無劃線修正本

修正日期:104 年 12 月 9 日

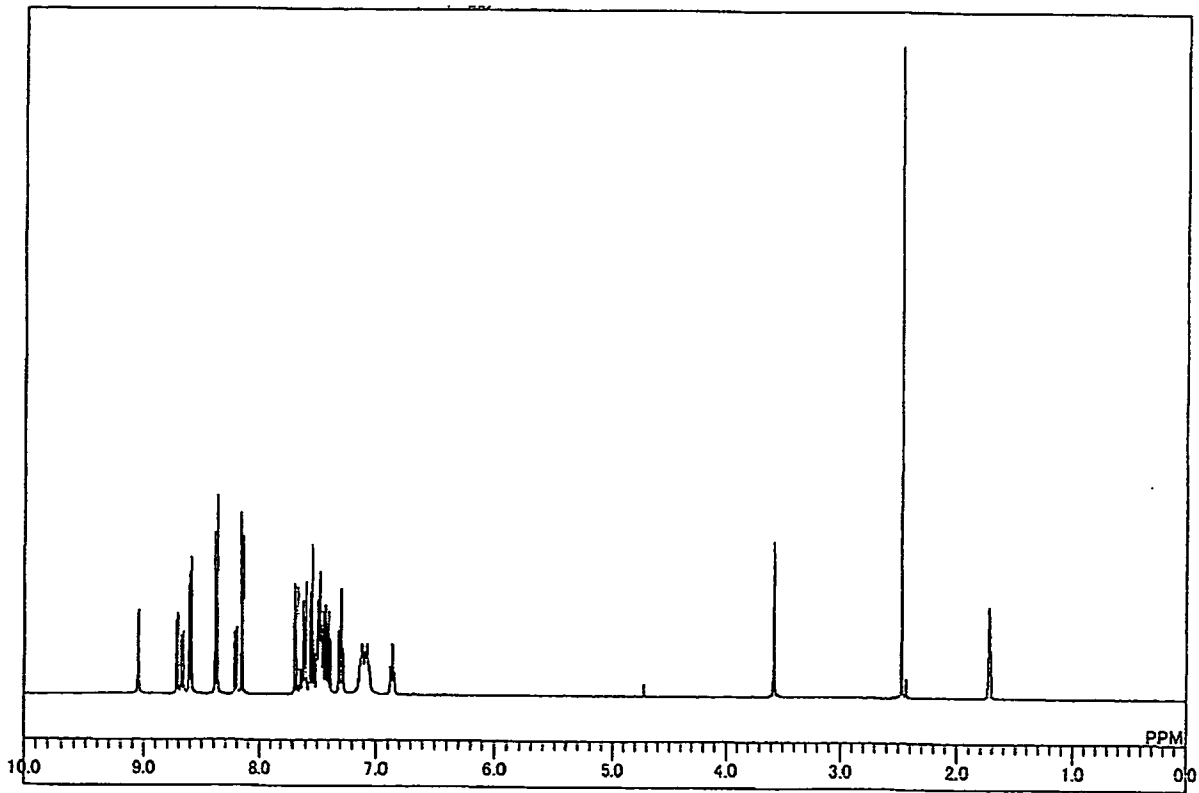
上述式 (1b) 中,  $X_1 \sim X_4$  的總數為 4~7。

5. 如申請專利範圍第 1 項至第 4 項中任一項所述的有機電場發光元件, 其中含有上述通式 (1) 所表示的吡啶并咪唑化合物的有機層為含有磷光發光摻雜劑的發光層。

# 圖式



## 圖 1



## 圖 2

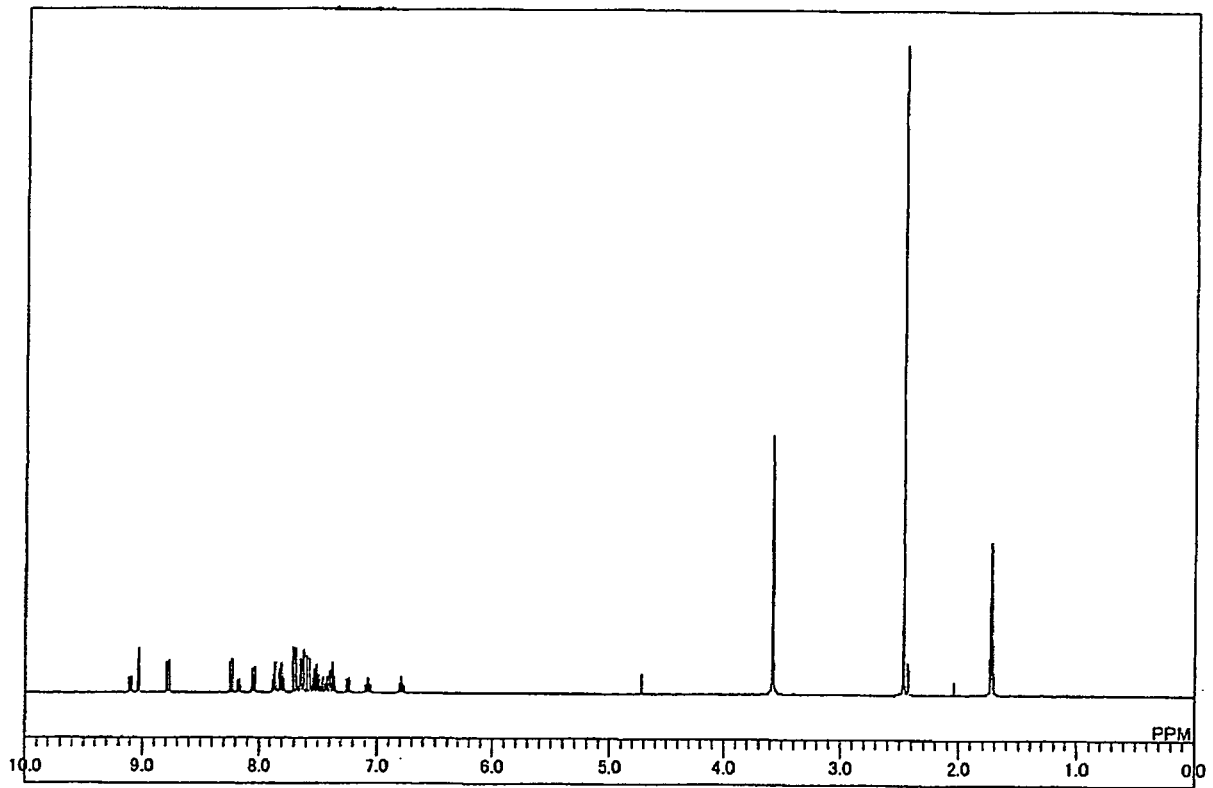


圖 3