

# 2,7(3,6)-Diaryl(arylamino)-substituted Carbazoles

Subjects: Biochemical Research Methods

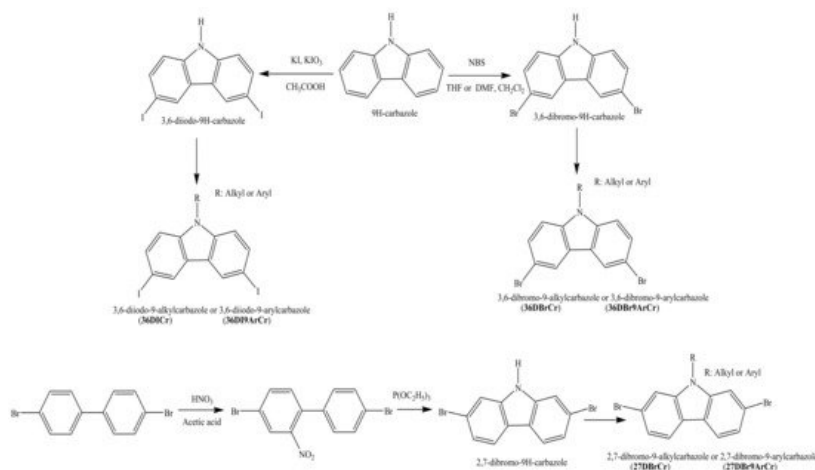
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The carbazole is an electron-rich heterocyclic compound, whose structure can be easily modified by rather simple reactions in order to obtain 2,7(3,6)-diaryl(arylamino)-substituted carbazoles. The substituted derivatives are widely used for the formation of Organic light emitting diode (OLED) due to their good charge carrier injection and transfer characteristics, electroluminescence, thermally activated delayed fluorescence, improved thermal and morphological stability as well as their thin film forming characteristics.

Keywords: substituted carbazole ; organic light emitting diode

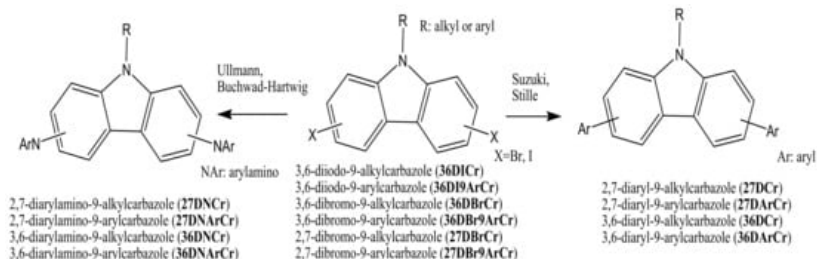
## 1. Synthesis of 2,7(3,6)-Diiodo(dibromo)carbazoles and 2,7(3,6)-Diaryl(diarylamino)-substituted Objective Carbazoles

Both the 3,6-Diiodo-9H-carbazoles and 3,6(2,7)-dibromo-9H-carbazoles are intermediate materials for the preparation of objective carbazole-based derivatives for OLEDs. Structures of the halogenated derivatives are shown in [Scheme 1](#). The bromination reaction of 9H-carbazole with N-bromosuccinimide (NBS) <sup>[1]</sup> or its Tucker iodination reaction <sup>[2]</sup> correspondingly yields 3,6-dibromocarbazole or 3,6-diiodocarbazole. On the other hand, 2,7-dibromocarbazole is obtained by a two-step synthesis, as shown in the [Scheme 1](#) <sup>[3]</sup>. The starting material 4,4'-dibromo-1,1'-biphenyl is firstly nitrified. The obtained 4,4'-dibromo-2-nitro-1,1'-biphenyl is then reacted with triethylphosphate to get 2,7-dibromo-9H-carbazole. The nitrogen atom of the halogenated carbazoles can be then functionalized by different alkyl or aryl groups in order to obtain the key starting materials: 3,6-diiodo-9-alkylcarbazoles (**36DICr**), 3,6-diiodo-9-arylcarbazoles (**36DI9ArCr**), 3,6-dibromo-9-alkylcarbazoles (**36DBrCr**), 3,6-dibromo-9-arylcarbazoles (**36DBr9ArCr**), 2,7-dibromo-9-alkylcarbazoles (**27DBrCr**), and 2,7-dibromo-9-arylcarbazoles (**27DBr9ArCr**), which are shown in the [Scheme 1](#). The alkylation reactions with bromo or iodo alkanes under basic conditions are rather simple and widely described in the literature <sup>[4][5][6]</sup>. The di-halogenated 9-arylcarbazoles are prepared by Ullmann or Buchwald–Hartwig reactions <sup>[7][8][9]</sup>.



**Scheme 1.** Synthesis of starting compounds: 3,6-diiodo(dibromo)-9-alkyl(aryl)carbazoles and 2,7-dibromo-9-alkyl(aryl)carbazoles.

The key starting di-halogenated compounds **36DICr**, **36DI9ArCr**, **36DBrCr**, **36DBr9ArCr**, **27DBrCr**, and **27DBr9ArCr** can then be used in the following amination or C-C coupling reactions to obtain the target compounds: 2,7-diarylamino-9-alkyl(aryl)carbazoles (**27DNCr** and **27DNArCr**), 3,6-diarylamino-9-alkyl(aryl)carbazoles (**36DNCr** and **36DNArCr**), 2,7-diaryl-9-alkyl(aryl)carbazoles (**27DCr** and **27DArCr**), or 3,6-diaryl-9-alkyl(aryl)carbazoles (**36DCr** and **36DArCr**) ([Scheme 2](#)). Most of the objective carbazole-based derivatives, which are used later as electroactive components for the production of OLED devices, are obtained from the di-halogenated carbazoles by the Ullmann, Suzuki, Stille, or Buchwald–Hartwig reactions.

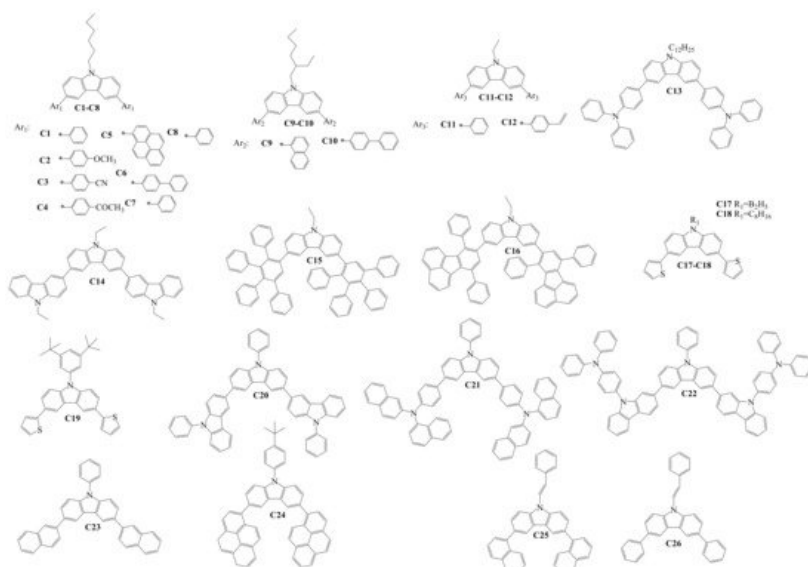


**Scheme 2.** Synthetic pathway of the 2,7(3,6)-diaryl(arylamino)-substituted objective 9-alkyl(aryl)carbazoles.

The Ullmann reaction is a coupling reaction between aryl halides and aromatic amines or aromatic heterocyclic compounds in the presence of copper-based catalysts. The mechanism of the Ullmann reaction was extensively studied for many years. Complications arise because the reactions are often heterogeneous, especially those catalyzed with metallic copper. [10][11]. Suzuki cross-coupling procedures, developed by Nobel laureate Akira Suzuki, are among the most widely investigated reactions in the formation of carbon–carbon bonds in aromatic compounds. These reactions were generally catalyzed by inorganic catalysts, such as soluble palladium (Pd) complexes having various ligands, and more recently, also in aqueous media [12][13]. The Buchwald–Hartwig amination procedure is a chemical reaction used in organic chemistry for the formation of carbon–nitrogen bonds via palladium-catalyzed coupling reactions of aromatic amines with aryl halides [14]. Other reactions as the Stille coupling [15][16], Diels Alder [17][18][19], and Friedel–Craft [20] have also been used to obtain the target carbazole compounds, but in rarer cases. In the general case, diarylamino carbazoles (**27DNArCr**, **27DNArCr**, **36DNArCr**, and **36DNArCr**) are usually obtained by Ullmann or Buchwald–Hartwig reactions. On the other hand, diaryl carbazoles (**27DCr**, **27DArCr**, **36DCr**, and **36DArCr**) are usually prepared by Suzuki or Stille reactions, as presented in [Scheme 2](#).

## 2. Diaryl(diarylamino)-substituted Carbazoles as Charge-Transporting Layer Materials for OLEDs

Structures of the diaryl-substituted compounds (**Cx**), which were used to form the hole-transporting layers (HTLs) in OLED devices, are shown in [Scheme 3](#). The target compounds **C1–C14** [21][22][23][24][25] and **C17–C26** [26][27][28][29][30][31] were obtained under the conditions of the Suzuki reaction. Compounds **C15–C16** were generated by the Diels–Alder reaction between conjugated diene and substituted alkene, forming the substituted cyclohexene fragments [32]. Authors of the research studied properties of the derivatives and used these materials for the formation of HTLs in OLED devices. Thermal properties were examined for compounds **C1–C13**, **C17–C19**, **C21–C22**, and **C24–C26**. The reported material **C22** demonstrated the highest thermal stability in this group, with a very high thermal decomposition temperature ( $T_d$ ) of 575 °C, as well as the highest glass transition temperature ( $T_g$ ) of 260 °C. The values of ionization potentials ( $I_p$ ) for compounds **C9–C11**, **C17–C20**, and **C23** were 5.65 eV, 5.55 eV, 5.8 eV, 5.38 eV, 5.42 eV, 5.19 eV, and 5.50 eV, respectively, and confirmed suitable hole-injecting properties for thin layers of many of the materials. HOMO and LUMO levels of the materials **C1–C8**, **C13–C17**, **C19–C22**, and **C24–C26** are different and depend on the nature of the substituents. The HOMO level of the diarylcarbazoles varied between –4.93 and –6.02 eV, and the LUMO level was in a broad range between –0.87 and –2.93 eV due to different electron-withdrawing or donating substituents at the carbazole core.



### Scheme 3. Structures of 3,6-diarylcarbazoles used for hole-transporting films in OLEDs.

Positive charge drift mobility ( $\mu_h$ ) in the thin layers of the derivatives **C12**, **C17–C19**, **C21–C22**, and **C25–C26** were reported. The compounds demonstrated rather high hole-drift mobility in their amorphous films ranging from  $5 \times 10^{-5} \text{ cm}^2 \cdot \text{V}^{-1} \cdot \text{s}^{-1}$  to  $1.5 \times 10^{-4} \text{ cm}^2 \cdot \text{V}^{-1} \cdot \text{s}^{-1}$  at high electrical fields. The charge-injecting/transporting properties of these materials confirmed that they are suitable hole-transporting layer materials in OLEDs.

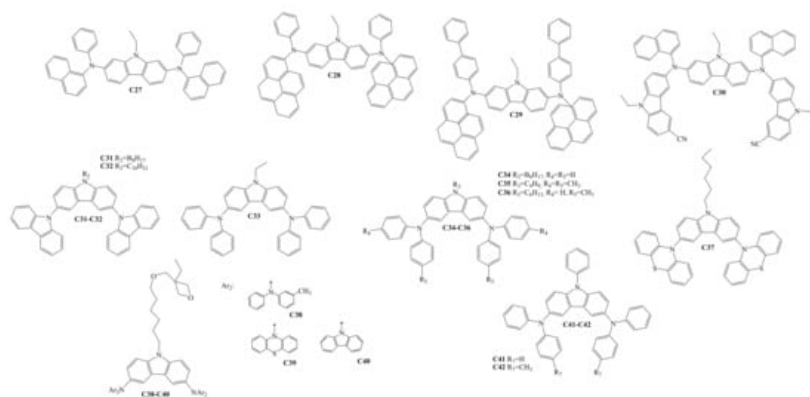
Agarwal fabricated the single-layer spin-coated OLEDs, in which the derivatives **C1–C8** were sandwiched between indium tin oxide (ITO) or ITO/PEDOT:PSS {poly(3,4-ethylene-dioxythiophene): poly(styrenesulfonate)} as the anode and a calcium (Ca) cathode, which was protected by a thick layer of Al. Multilayer OLEDs with the materials **C1–C8** were also fabricated. The structure of the devices was ITO/F<sub>4</sub>TCNQ (2,3,5,6-tetrafluoro-7,7',8,8'-tetracyano-p-quinodimethane)/TPD (N,N'-(bis(3-methylphenyl)-1,1'-biphenyl-4,4'-diamine))/CBP (N,N'-dicarbazolyl-4,4'-biphenyl)/**C3** or **C4**/BCP (4,7-diphenyl-1,10-phenanthroline)/LiF/Al. Brightness as high as 900–1000 cd/m<sup>2</sup> was achieved in the multilayer OLED devices using the HTLs of **C3** or **C4**. Green electro-phosphorescent devices ITO/PEDOT:PSS/**C9** or **C11**/CBP/Ir(ppy)<sub>3</sub> (tris(2-phenylpyridine)iridium(III))/TPBi (2,2',2''-(1,3,5-benzinetriyl)-tris(1-phenyl-1-H-benzimidazole))/LiF/Al were also presented. The device with **C11** demonstrated the best results, with a turn-on voltage of 5 V, a maximum brightness of 9800 cd/m<sup>2</sup>, and a maximum current efficiency of 22.5 cd/A. **C12** was tested as an electron-confining HTL for phosphorescent OLEDs with a red, green, or blue emitting layer. The power efficiency of the studied red device was increased from 8.5 to 13.5 lm/W, an increment of 59%, and the maximum luminance was enhanced from 13,000 to 19,000 cd/m<sup>2</sup>, an increment of 46%, as compared with an analogous device using commercial HTL material. For the blue device using **C12**, the power efficiency was increased from 6.9 to 8.9 lm/W, an increment of 29%, and the maximum brightness was enhanced from 9000 to 11,000 cd m<sup>-2</sup>, an increment of 22%. **C13** as an HTL was investigated by Kochapradist et al. Tris(8-hydroxyquinolino)aluminum (Alq<sub>3</sub>) emitter-based green OLEDs, with the structure of ITO/PEDOT:PSS/**C13**/Alq<sub>3</sub>/LiF/Al, were fabricated. The material **C13** showed excellent hole-transporting properties for the green device—a luminance (current) efficiency exceeding 5 cd/A was achieved. Lee and co-workers studied the hole-transporting properties of **C14** and formed the device with ITO /CuPc (copper(II)phthalocyanine)/**C14**/Alq<sub>3</sub>/LiF/Al. For an objective comparison of electroluminescent properties, a reference device was fabricated and NPB was used as the HTL material. The device with the compound **C14** showed a high power efficiency of 1.67 lm/W and a current efficiency of 6.12 cd/A, which were higher as compared with the NPB-based device. OLEDs with the structures ITO/2-TNATA (4,4',4''-tris{N-(2-naphthyl)-N-phenylamino}-triphenylamine)/**C15** or **C16**/Alq<sub>3</sub>/LiF/Al were fabricated by Park and co-workers. The devices showed pure performance, with their luminescence efficiency exceeding only 3 cd/A and a power efficiency exceeding 1 lm/W.

In order to identify the charge-transporting properties of **C17**, multilayer OLED devices were fabricated by Kim et al. The green device ITO/CuPc/**C17**/Alq<sub>3</sub>/LiF /Al showed a current efficiency of 5.16 cd/A and a power efficiency of 2.35 lm/W. Braveenth and co-authors studied the properties of the compounds **C18–C19** as hole-transporting materials. Device configuration was ITO/DNTPD (4,4'-bis[N-[4-{N,N-bis(3-meth )-N-phenylamino]biphenyl})/**C18** or **C19** /Bebq<sub>2</sub> (bis(10-hydroxybenzo[h]quinolino)beryllium): Ir(mphmq)<sub>2</sub>(tmd) (bis[2,4-dimethyl-6-(4-methyl-2-quinolino)phenyl])(2,2,6,6-tetramethyl-3,5-heptanedionate)/Bebq<sub>2</sub>/LiF/Al. A **C18**-based phosphorescent device exhibited higher maximum current efficiency (24.6 cd/A) and higher maximum external quantum efficiency (23.2%) as compared with a **C18**-based OLED. In the double-layer device, ITO/**C20**/Alq<sub>3</sub>/LiF/Al, in which **C20** was used as the hole-transporting material, a yellowish-green color arising from the Alq<sub>3</sub> was observed. It was only reported that the luminance at the applied voltage of 10 V was about 9600 cd/m<sup>2</sup>. Devices with the structures of ITO/**C21**/TPBi/LiF/Al and ITO/NPB/**C21**/TPBi /LiF/Al were fabricated to evaluate the properties of **C21** as a hole-transporting light-emitting layer. A quantum efficiency of 2.21% was obtained for the **C21**-based device having an additional NPB layer.

Kumar and co-workers investigated the hole-transporting properties of **C25** and **C26** for fluorescent OLEDs ITO/PEDOT:PSS/NPB/**C25** or **C26**/Alq<sub>3</sub>/LiF/Al, as well as for phosphorescent devices ITO/PEDOT:PSS/NPB/**C25** or **C26** /Ir(ppy)<sub>3</sub>/CBP/TPBi/LiF/Al. At 1000 cd/m<sup>2</sup>, the fluorescent green device with **C26** showed a current efficiency of 4.0 cd/A, which was about 135% higher than that of the typical HTM NPB-based device. Green phosphorescent devices with **C25** showed a high current efficiency of 58.4 cd/A (power efficiency 54.8 lm/W and EQE 16.1%), while 45.1 cd/A (power efficiency 40.8 lm/W and EQE 12.5%) was measured in the **C26**-containing device.

Diaryl-amino-substituted carbazoles, which were used as HTLs in OLEDs, are shown in [Scheme 4](#). The target compounds **C27–C42** were all obtained by Ullmann amination reactions <sup>[33][34][35][36][37][38][39]</sup>. **C30–C42** demonstrated sufficient and high thermal stability, with the thermal degradation temperatures ( $T_d$ ) ranging from 263 °C to 490 °C. The derivatives **C30–C42** are also suitable for glass formation, having a  $T_g$  in the range of 42–217 °C. The Ip of the compounds **C30–C31** and **C37–C42** were reported and demonstrated the values of 5.80 eV, 5.81 eV, 5.50 eV, 5.24 eV,

5.43 eV, 5.67 eV, 5.28 eV, and 5.34 eV, respectively. HOMO and LUMO energy levels were reported for the materials **C27–C30**. The values of HOMO/LUMO for the derivatives **C27–C30** were  $-4.92/-1.91$  eV,  $-4.92/-2.24$  eV,  $-4.92/-2.33$  eV, and  $-4.83/-1.87$  eV, respectively.



**Scheme 4.** Structures of diarylamino-substituted carbazoles used for HTL formation.

The charge-transporting properties of the derivatives **C27–C28** and **C31–C42** were studied with the time-of-flight technique. The  $\mu_h$  values ranged from  $1.2 \times 10^{-8}$  to  $2 \times 10^{-3} \text{ cm}^2 \cdot \text{V}^{-1} \cdot \text{s}^{-1}$  at high electric fields. The  $\mu_h$  in the films of **C32** and **C40** dispersed in bisphenol Z polycarbonate (PC-Z) reached  $10^{-3} \text{ cm}^2 \cdot \text{V}^{-1} \cdot \text{s}^{-1}$  at high electrical fields. The charge injection/transport characteristics of some from the derivatives **C27–C28** and **C31–C42** showed that they are suitable as hole-transporting layers for OLED devices.

Shen and co-authors investigated two types of devices. The structure of device I was ITO/**C27–C30**/TPBi/Mg:Ag, and that of device II was ITO/**C27–C30**/(Alq<sub>3</sub>)/Mg:Ag. In type I, the derivatives functioned as both hole-transporting and emitting materials. In the type II devices, light was emitted from either the disubstituted carbazole layers or from Alq<sub>3</sub>. Device II with **C30** reached a maximum external quantum efficiency of 1.3%.

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