Synthesis of 3-cyano-2-(1*H*-indol-3-yl)-6-(9-butylcarbazol-3-yl)pyridine derivatives by a multicomponent reaction under microwave irradiation

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Preparation of a series of 3-cyano-2-(1*H*-indol-3-yl)-6-(9-butylcarbazol-3-yl)pyridine derivatives through the one-pot four-component coupling of aromatic aldehydes, 1-(9-butylcarbazol-3-yl)ethanone, 3-(cyanoacetyl)indole and ammonium acetate is reported. All these compounds were obtained in good yield and their structures were confirmed by ^{1}H NMR and IR spectroscopy and by elemental analysis.

Keywords: multicomponent reaction, 3-cyanoacetylindole, 1-(9-butylcarbazol-3-yl)ethanone, pyridine derivatives, microwave irradiation

During the past few years, the synthesis and application of carbazole derivatives have been of great interest because of their intrinsic photophysical and redox properties. They exhibit relatively intense luminescence and undergo reversible oxidation processes which make them suitable as hole carriers.[1] Moreover, carbazole can be easily functionalised at its 3-, 6-, or 9-positions and the carbazole moiety is beneficial for raising the glass transition temperature and thermal stability.[2] Carbazole derivatives are widely used as building blocks for potential organic semiconductors[3,4] and organic light-emitting diodes.[5,6] However, further progress in these fields requires an intensification of the research effort focused on the design and synthesis of new compounds with electrochemical, optical, and electronic properties specifically tailored for each type of application.

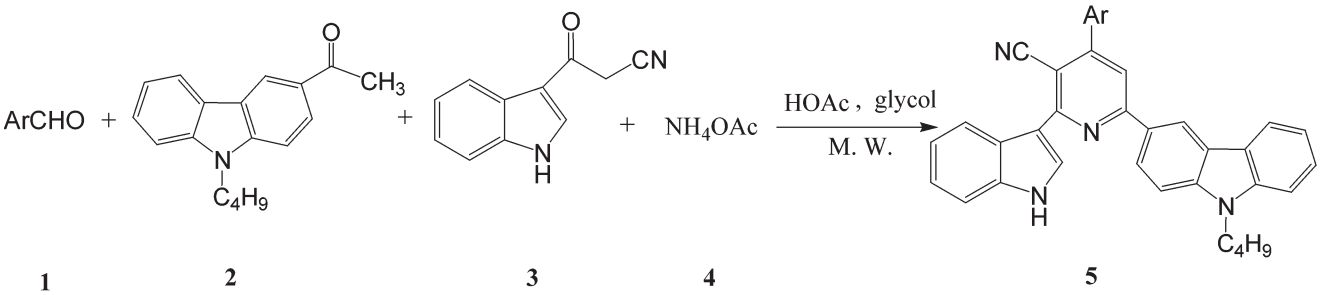
Since the publication of the Strecker reaction in 1850 considered to be the beginning of the multicomponent reaction (MCR) story,[7] there has been considerable interest in one-pot MCRs due their diversity, efficiency and rapid access to complex and highly functionalised organic molecules.[8,9] In the past decade, there have been many developments in three- and four-component reactions[10,11] and great efforts are being made to find and develop new MCRs.[12–14]

Results and discussion

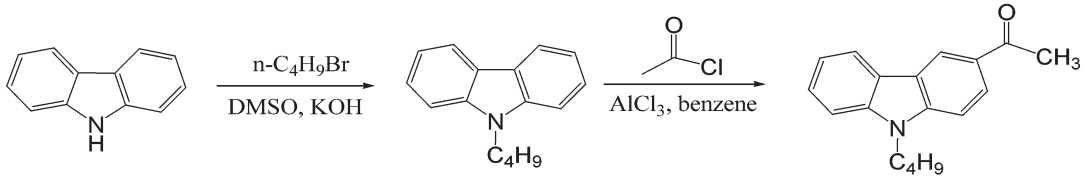
Other groups have described the synthesis of 3-cyano-2-(1*H*-indole-3-yl)pyridines, for example the 4-aryl-6-(1*H*-indole-3-yl)-2,2-bispyridine derivatives[15] and 4-aryl-2,6-di(1*H*-indole-3-yl)pyridine derivatives.[16] Our group has reported a new and efficient synthesis of 3-cyano-2-(1*H*-indol-3-yl)pyridine derivatives by MCRs in good yields.[17,18] In continuing our interest in the multicomponent syntheses and in multi-functional derivatives, we now report a simple and efficient protocol for the preparation of a series of 3-cyano-2-(1*H*-indol-3-yl)-6-(9-butylcarbazol-3-yl)pyridine derivatives. The target compounds were synthesised by the four-component reaction of aromatic aldehydes, 1-(9-butylcarbazol-3-yl)ethanone, 3-(cyanoacetyl)-indole and ammonium acetate under microwave irradiation (Scheme 1). To realise our objective, the starting compound 3-(cyanoacetyl)indole was prepared according to the procedure described by Bergman *et al.*[19] 9-Butylcarbazole[20] was obtained by the reaction of carbazole with 1-bromobutane in DMSO catalysed by KOH, subsequent acylation with acetyl chloride in benzene provided 1-(9-butylcarbazol-3-yl) ethanone[21] (Scheme 2).

Because an appropriate solvent is of crucial importance for the successful microwave-assisted synthesis, our initial studies were carried out under various reaction conditions. Both solvents and microwave power were tested in the one pot four-component synthesis of the target compounds. We found that AcOH and ethane-1,2-diol (glycol) (1:2) and irradiation at 300 W was optimal to maximise yields. Under these conditions, a series of new 3-cyano-2-(1*H*-indol-3-yl)-6-(9-butylcarbazol-3-yl)pyridine derivatives were synthesised in good yields. The optimised results are summarised in Table 1.

As can been seen from Table 1, the reaction proceeded smoothly with a series of substituted aromatic aldehydes. In addition, we explored the synthesis of **5b** under classical



Scheme 1



Scheme 2

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Table 1 Synthesis of compounds **5a–k** under microwave irradiation

Entry	Ar	Product	Time/min	Yield[a]/%	M.p./°C
1	C_6H_5	**5a**	4	82	232.7–233.4
2	4-$CH_3C_6H_4$	**5b**	4	85	285.3–286.2
3	4-$CH_3OC_6H_4$	**5c**	4	86	256.5–257.3
4	3-$CH_3OC_6H_4$	**5d**	4	84	251.8–252.4
5	4-$(CH_3)_2CHC_6H_4$	**5e**	4	84	273.5–274.1
6	4-$(CH_3)_3CC_6H_4$	**5f**	4	82	>300
7	4-$(CH_3)_2NC_6H_4$	**5g**	4	83	250.9–251.7
8	4-HOC_6H_4	**5h**	4	77	281.5–282.2
9	1-Naphthyl	**5i**	4	75	272.0–273.2
10	4-ClC_6H_4	**5j**	4	81	>300
11	4-BrC_6H_4	**5k**	4	76	282.5–283.3

[a]Isolated yield.

heating conditions. After refluxing for 2 h (AcOH-glycol, 1:2), the desired product **5b** was obtained in 56% yield. However, under microwave irradiation, the yield of **5b** was up to 85% after just four minutes (entry 2). Therefore, microwave irradiation exhibited several advantages over the conventional heating by significantly reducing the reaction times and improving the product yield.

The structures of **5a–k** were confirmed by spectral studies and elemental analysis as exemplified for compound **5c** as follows: The IR spectrum of **5c** showed an absorption at 2212 cm^{-1} for the CN group. The signals at δ 11.82 and δ 3.89 confirmed the presence of the indolyl NH and OCH_3 groups respectively in the ^1H NMR spectrum.

Based on the above results, a possible mechanism was proposed (Scheme 3). 3-(Cyanoacetyl)indole reacts with ammonia from ammonium acetate to give intermediate **7**, which further reacts with the corresponding chalcones **6**, to yield **8**. Michael addition product **8** was then cyclised to afford the Hantzsch dihydropyridine derivative **9** with elimination of water. Subsequent dehydrogenation of **9** leads to formation of the highly substituted pyridine derivative **5**.

In conclusion, a series of novel 3-cyano-2-(1*H*-indol-3-yl)-6-(9-butylcarbazol-3-yl)pyridine compounds were synthesised through a one-pot four-component condensation reaction under microwave irradiation. This method has several unique merits, such as short reaction times, an easy workup procedure, and efficient yields.

Experimental

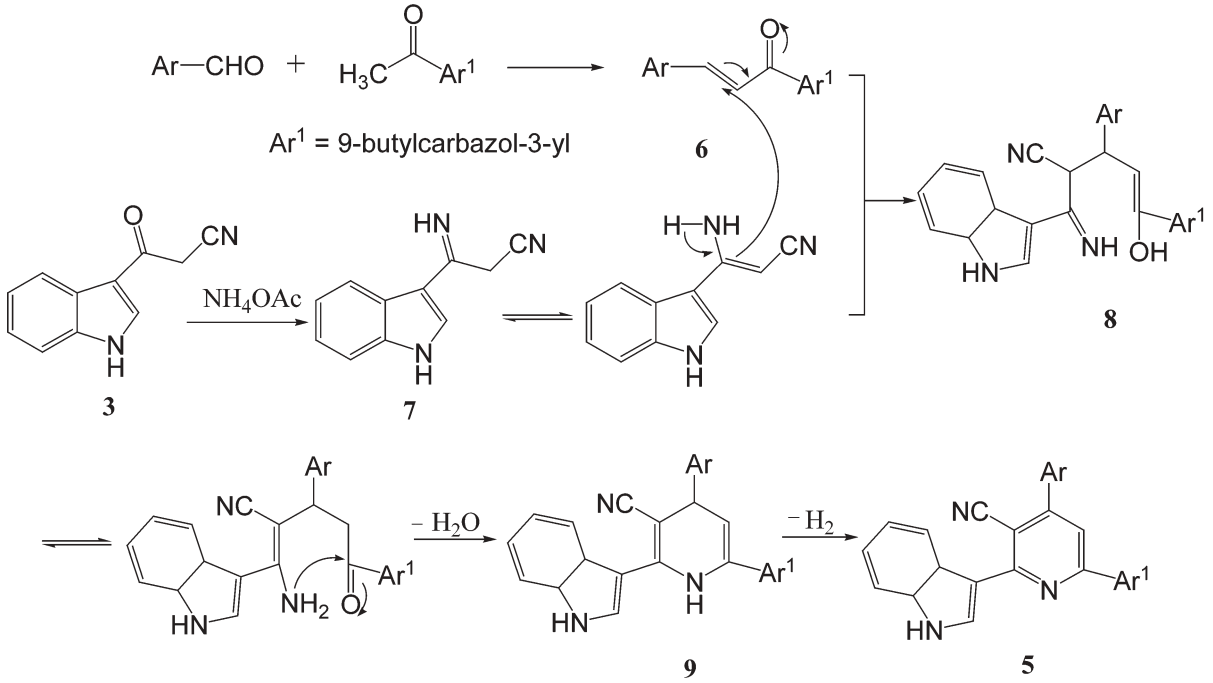
Melting points were recorded on an Electrothermal digital melting point apparatus and are uncorrected. ^1H NMR spectra were determined on a Varian VXP-500s spectrometer using DMSO-d_6 as solvent and tetramethylsilane (TMS) as internal reference. The IR spectra were obtained on a Nicolet 6700 spectrophotometer using KBr pellets. Elemental analyses were performed by a Heraeus CHN-O-RAPID analyser. Microwave reactions were carried out in a Xianghu XH-100B microwave oven. All chemicals were purchased and used without further purification.

General procedure

A mixture of aldehyde **1** (2 mmol), 1-(9-butylcarbazol-3-yl)ethanone **2** (2 mmol), 3-(cyanoacetyl)indole **3** (2 mmol), and ammonium acetate **4** (8 mmol) in HOAc 2 mL and glycol 4 mL was irradiated at 300 W (flask equipped with a condenser). After completion of the reaction (the reaction was followed by TLC), the mixture was allowed to cool to room temperature and extracted with dichloromethane, washed with H_2O (3 × 10 mL) and the organic layer dried over $MgSO_4$ and concentrated under reduced pressure. The crude product was chromatographed on silica gel (200–300 mesh) using a mixture of petroleum ether and dichloromethane as eluent to afford the pure product (**5a–k**).

*3-Cyano-2-(1*H*-indol-3-yl)-4-phenyl-6-(9-butylcarbazol-3-yl)pyridine* (**5a**): Pale yellow powder; IR (KBr) ν_{max} 3428, 3053, 2955, 2927, 2870, 2212 (CN), 1570, 1526, 1481, 1428, 1384, 1363, 1241, 1210, 1133, 1104, 746 cm^{-1}; ^1H NMR (500 MHz, DMSO-d_6): δ 11.82 (s, 1H), 9.21 (s, 1H), 8.56 (dd, 1H, *J* = 1.5 Hz and *J* = 8.5 Hz), 8.53–8.51 (m, 1H), 8.39 (d, 1H, *J* = 3.0 Hz), 8.27 (d, 1H, *J* = 8.0 Hz), 8.12 (s, 1H), 7.85–7.80 (m, 3H), 7.66–7.57 (m, 5H), 7.50 (t, 1H, *J* = 7.5 Hz), 7.30–7.25 (m, 3H), 4.46 (t, 2H, *J* = 7.0 Hz), 1.82–1.77 (m, 2H), 1.37–1.30 (m, 2H), 0.90 (t, 3H, *J* = 7.5 Hz). Anal. Calcd for $C_{36}H_{28}N_4$, C, 83.69; H, 5.46; N, 10.84. Found: C, 83.62; H, 5.33; N, 10.98%.

*3-Cyano-2-(1*H*-indol-3-yl)-4-(4-methylphenyl)-6-(9-butylcarbazol-3-yl)pyridine* (**5b**): Pale yellow powder; IR (KBr) ν_{max} 3426, 3136, 3050, 2956, 2928, 2871, 2215 (CN), 1573, 1525, 1479, 1427, 1365, 1323, 1240, 1218, 1134, 1021, 811, 747 cm^{-1}; ^1H NMR (500 MHz, DMSO-d_6): δ 11.82 (s, 1H), 9.20 (s, 1H), 8.55 (dd, 1H, *J* = 1.5 Hz and



Scheme 3

J = 9.0 Hz), 8.51–8.50 (m, 1H), 8.38 (d, 1H, *J* = 3.0 Hz), 8.27 (d, 1H, *J* = 7.5 Hz), 8.09 (s, 1H), 7.82 (d, 1H, *J* = 9.0 Hz), 7.75 (d, 2H, *J* = 8.0 Hz), 7.67 (d, 1H, *J* = 8.5 Hz), 7.59–7.57 (m, 1H), 7.51 (t, 1H, *J* = 7.5 Hz), 7.45 (d, 2H, *J* = 8.0 Hz), 7.30–7.26 (m, 3H), 4.47 (t, 2H, *J* = 7.0 Hz), 2.50 (t, 3H, *J* = 1.5 Hz), 1.84–1.78 (m, 2H), 1.38–1.31 (m, 2H), 0.91 (t, 3H, *J* = 7.5 Hz). Anal. Calcd for $C_{37}H_{30}N_4$, C, 83.74; H, 5.70; N, 10.56. Found: C, 83.72; H, 5.83; N, 10.68%.

*3-Cyano-2-(1*H-*indol-3-yl)-4-(4-methoxyphenyl)-6-(9-butylcarbazol-3-yl)pyridine* (**5c**): Yellow powder; IR (KBr) ν_{max} 3310, 3053, 2950, 2927, 2870, 2212 (CN), 1609, 1570, 1524, 1511, 1489, 1457, 1365, 1253, 1211, 1178, 1135, 1032, 834, 745 cm^{-1}; ^1H NMR (500 MHz, DMSO-d_6): δ 11.82 (s, 1H), 9.21 (s, 1H), 8.55 (dd, 1H, *J* = 1.5 Hz and *J* = 8.5 Hz), 8.53–8.51 (m, 1H), 8.40 (d, 1H, *J* = 2.5 Hz), 8.28 (d, 1H, *J* = 8.0 Hz), 8.08 (s, 1H), 7.84–7.80 (m, 3H), 7.66 (d, 1H, *J* = 8.0 Hz), 7.60–7.58 (m, 1H), 7.51 (t, 1H, *J* = 8.0 Hz), 7.31–7.28 (m, 3H), 7.21–7.19 (m, 2H), 4.46 (t, 2H, *J* = 7.0 Hz), 3.89 (s, 3H), 1.83–1.77 (m, 2H), 1.38–1.31 (m, 2H), 0.91 (t, 3H, *J* = 7.5 Hz). Anal. Calcd for $C_{37}H_{30}N_4O$, C, 81.29; H, 5.53; N, 10.25. Found: C, 81.32; H, 5.42; N, 10.38%.

*3-Cyano-2-(1*H-*indol-3-yl)-4-(3-methoxyphenyl)-6-(9-butylcarbazol-3-yl)pyridine* (**5d**): Pale yellow powder; IR (KBr) ν_{max} 3282, 3046, 2953, 2227 (CN), 1565, 1526, 1488, 1455, 1378, 1365, 1205, 1140, 1051, 815, 752, 742 cm^{-1}; ^1H NMR (500 MHz, DMSO-d_6): δ 11.82 (s, 1H), 9.21 (s, 1H), 8.56 (d, 1H, *J* = 8.5 Hz), 8.51 (t, 1H, *J* = 5.0 Hz), 8.39 (d, 1H, *J* = 3.0 Hz), 8.28 (d, 1H, *J* = 8.0 Hz), 8.13 (s, 1H), 7.82 (d, 1H, *J* = 8.5 Hz), 7.67 (d, 1H, *J* = 8.0 Hz), 7.59–7.49 (m, 3H), 7.40 (d, 2H, *J* = 7.0 Hz), 7.29–7.27 (m, 3H), 7.18–7.16 (m, 1H), 4.47 (t, 2H, *J* = 7.0 Hz), 3.90 (s, 3H), 1.81 (t, 2H, *J* = 8.0 Hz), 1.37–1.32 (m, 2H), 0.91 (t, 3H, *J* = 7.5 Hz). Anal. Calcd for $C_{37}H_{30}N_4O$, C, 81.29; H, 5.53; N, 10.25. Found: C, 81.25; H, 5.63; N, 10.18%.

*3-Cyano-2-(1*H-*indol-3-yl)-4-(4-isopropylphenyl)-6-(9-butylcarbazol-3-yl)pyridine* (**5e**): Pale yellow powder; IR (KBr) ν_{max} 3300, 3051, 2952, 2869, 2220 (CN), 1562, 1524, 1494, 1442, 1379, 1320, 1247, 1201, 1140, 810, 741 cm^{-1}; ^1H NMR (500 MHz, DMSO-d_6): δ 11.82 (s, 1H), 9.21 (s, 1H), 8.57–8.55 (m, 1H), 8.51 (t, 1H, *J* = 4.5 Hz), 8.39 (d, 1H, *J* = 3.0 Hz), 8.27 (d, 1H, *J* = 8.0 Hz), 8.11 (s, 1H), 7.82–7.77 (m, 3H), 7.66 (d, 1H, *J* = 8.0 Hz), 7.59–7.58 (m, 1H), 7.52–7.49 (m, 3H), 7.29–7.25 (m, 3H), 4.46 (t, 2H, *J* = 7.0 Hz), 3.08–3.00 (m, 1H), 1.83–1.77 (m, 2H), 1.36–1.30 (m, 8H), 0.90 (t, 3H, *J* = 7.5 Hz). Anal. Calcd for $C_{39}H_{34}N_4$, C, 83.84; H, 6.13; N, 10.03. Found: C, 83.68; H, 6.26; N, 10.11%.

*3-Cyano-2-(1*H-*indol-3-yl)-4-(4-*tert*-butylphenyl)-6-(9-butylcarbazol-3-yl)pyridine* (**5f**): Pale yellow powder; IR (KBr) ν_{max} 3308, 3048, 2959, 2869, 2221 (CN), 1565, 1537, 1470, 1379, 1245, 1212, 1140, 1122, 838, 809, 739 cm^{-1}; ^1H NMR (500 MHz, DMSO-d_6): δ 11.82 (s, 1H), 9.22 (s, 1H), 8.57–8.52 (m, 2H), 8.40 (d, 1H, *J* = 3.0 Hz), 8.27 (d, 1H, *J* = 8.0 Hz), 8.11 (s, 1H), 7.80–7.77 (m, 3H), 7.66–7.63 (m, 3H), 7.60–7.58 (m, 1H), 7.50 (t, 1H, *J* = 8.0 Hz), 7.29–7.25 (m, 3H), 4.45 (t, 2H, *J* = 7.0 Hz), 1.82–1.76 (m, 2H), 1.38 (s, 9H), 1.36–1.31 (m, 2H), 0.90 (t, 3H, *J* = 7.5 Hz). Anal. Calcd for $C_{40}H_{36}N_4$, C, 83.88; H, 6.34; N, 9.78. Found: C, 83.71; H, 6.47; N, 9.69%.

*3-Cyano-2-(1*H-*indol-3-yl)-4-(4-dimethylaminophenyl)-6-(9-butylcarbazol-3-yl)pyridine* (**5g**): Yellow powder; IR (KBr) ν_{max} 3408, 3297, 3052, 2924, 2214 (CN), 1609, 1568, 1526, 1517, 1493, 1440, 1360, 1211, 1136, 1123, 813, 744 cm^{-1}; ^1H NMR (500 MHz, DMSO-d_6): δ 11.79 (s, 1H), 9.18 (s, 1H), 8.54–8.52 (m, 1H), 8.50–8.48 (m, 1H), 8.37 (d, 1H, *J* = 3.0 Hz), 8.28 (d, 1H, *J* = 7.5 Hz), 8.04 (s, 1H), 7.80 (d, 1H, *J* = 9.0 Hz), 7.75 (d, 2H, *J* = 8.5 Hz), 7.66 (d, 1H, *J* = 8.5 Hz), 7.58–7.56 (m, 1H), 7.52–7.49 (m, 1H), 7.29–7.26 (m, 3H), 6.92 (d, 2H, *J* = 9.0 Hz), 4.47 (t, 2H, *J* = 7.0 Hz), 3.04 (s, 6H), 1.83–1.77 (m, 2H), 1.38–1.30 (m, 2H), 0.90 (t, 3H, *J* = 7.5 Hz). Anal. Calcd for $C_{38}H_{33}N_5$, C, 81.54; H, 5.94; N, 12.51. Found: C, 81.60; H, 5.83; N, 12.68%.

*3-Cyano-2-(1*H-*indol-3-yl)-4-(4-hydroxyphenyl)-6-(9-butylcarbazol-3-yl)pyridine* (**5h**): Yellow powder; IR (KBr) ν_{max} 3405, 3378, 3322, 3059, 2955, 2873, 2218 (CN), 1569, 1515, 1435, 1355, 1238, 1211, 1130, 1113, 837, 745 cm^{-1}; ^1H NMR (500 MHz, DMSO-d_6): δ 11.81 (s, 1H), 10.00 (s, 1H), 9.19 (s, 1H), 8.55–8.49 (m, 2H), 8.38 (d, 1H, *J* = 2.5 Hz), 8.28 (d, 1H, *J* = 7.5 Hz), 8.06 (s, 1H), 7.80 (d, 1H, *J* = 8.5 Hz), 7.72 (d, 2H, *J* = 8.5 Hz), 7.66 (d, 1H, *J* = 8.5 Hz), 7.59–7.57 (m, 1H), 7.51 (t, 1H, *J* = 7.0 Hz), 7.29–7.26 (m, 3H), 7.01 (d, 2H, *J* = 8.5 Hz), 4.46 (t, 2H, *J* = 7.0 Hz), 1.83–1.77 (m, 2H), 1.38–1.30 (m, 2H), 0.90 (t, 3H, *J* = 7.0 Hz). Anal. Calcd for $C_{36}H_{28}N_4O$, C, 81.18; H, 5.30; N, 10.52. Found: C, 81.09; H, 5.43; N, 10.48%.

*3-Cyano-2-(1*H-*indol-3-yl)-4-(1-naphthyl)-6-(9-butylcarbazol-3-yl)pyridine* (**5i**): Pale yellow powder; IR (KBr) ν 3297, 3052, 2954, 2928, 2870, 2225 (CN), 1561, 1542, 1522, 1485, 1430, 1358, 1323, 1213, 1123, 777, 742 cm^{-1}; ^1H NMR (500 MHz, DMSO-d_6): δ 11.83 (s, 1H), 9.21 (s, 1H), 8.63–8.60 (m, 2H), 8.38 (d, 1H, *J* = 3.0 Hz), 8.22 (d, 1H, *J* = 8.0 Hz), 8.19 (s, 1H), 8.17 (t, 1H, *J* = 6.0 Hz), 8.12 (d, 1H, *J* = 8.5 Hz), 7.82 (d, 1H, *J* = 9.0 Hz), 7.75–7.73 (m, 3H), 7.67–7.57 (m, 4H), 7.49 (t, 1H, *J* = 7.5 Hz), 7.31 (t, 2H, *J* = 3.5 Hz), 7.23 (t, 1H, *J* = 7.5 Hz), 4.47 (t, 2H, *J* = 7.0 Hz), 1.83–1.77 (m, 2H), 1.36–1.32 (m, 2H), 0.90 (t, 3H, *J* = 7.5 Hz). Anal. Calcd for $C_{40}H_{30}N_4$, C, 84.78; H, 5.34; N, 9.89. Found: C, 84.67; H, 5.41; N, 9.92%.

*3-Cyano-2-(1*H-*indol-3-yl)-4-(4-chlorophenyl)-6-(9-butylcarbazol-3-yl)pyridine* (**5j**): Pale yellow powder; IR (KBr) ν_{max} 3297, 3049, 2954, 2930, 2870, 2214 (CN), 1592, 1570, 1524, 1467, 1441, 1243, 1211, 1137, 1011, 747 cm^{-1}; ^1H NMR (500 MHz, DMSO-d_6): δ 11.84 (s, 1H), 9.21 (s, 1H), 8.57–8.55 (m, 1H), 8.52–8.50 (m, 1H), 8.39 (d, 1H, *J* = 3.0 Hz), 8.26 (d, 1H, *J* = 8.0 Hz), 8.13 (s, 1H), 7.87–7.81 (m, 5H), 7.67 (d, 1H, *J* = 8.5 Hz), 7.59–7.57 (m, 1H), 7.53–7.49 (m, 1H), 7.29–7.26 (m, 3H), 4.47 (t, 2H, *J* = 7.5 Hz), 1.84–1.78 (m, 2H), 1.38–1.31 (m, 2H), 0.91 (t, 3H, *J* = 7.0 Hz). Anal. Calcd for $C_{36}H_{27}ClN_4$, C, 78.46; H, 4.94; N, 10.17. Found: C, 78.52; H, 5.03; N, 10.29%.

*3-Cyano-2-(1*H-*indol-3-yl)-4-(4-bromophenyl)-6-(9-butylcarbazol-3-yl)pyridine* (**5k**): Pale yellow powder; IR (KBr) ν_{max} 3301, 3049, 2954, 2928, 2873, 2214 (CN), 1571, 1525, 1491, 1440, 1365, 1243, 1211, 1138, 1123, 1088, 799, 744 cm^{-1}; ^1H NMR (500 MHz, DMSO-d_6): δ 11.84 (s, 1H), 9.20 (s, 1H), 8.56–8.51 (m, 2H), 8.39 (d, 1H, *J* = 3.0 Hz), 8.26 (d, 1H, *J* = 7.5 Hz), 8.12 (s, 1H), 7.88 (d, 2H, *J* = 8.5 Hz), 7.81 (d, 1H, *J* = 8.5 Hz), 7.72–7.66 (m, 2H), 7.59–7.58 (m, 1H), 7.52–7.49 (m, 2H), 7.29–7.26 (m, 3H), 4.46 (t, 2H, *J* = 7.0 Hz), 1.83–1.77 (m, 2H), 1.38–1.30 (m, 2H), 0.90 (t, 3H, *J* = 7.0 Hz). Anal. Calcd for $C_{36}H_{27}BrN_4$, C, 72.61; H, 4.57; N, 9.41. Found: C, 72.69; H, 4.43; N, 9.38%.

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