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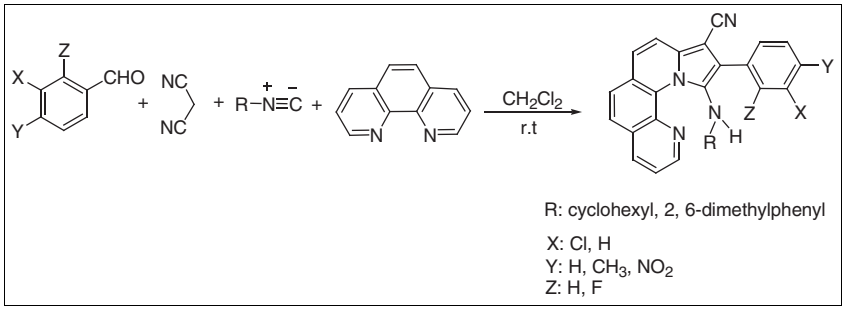

1,10-Phenanthroline reacts with malonitrile and aldehydes in the presence of isocyanides as domino-Knoevenagel-nucleophilic cycloaddition for generation of a new class of 10-(aryl)-11-(alkyl- or arylamino-) pyrrolo[1,2-*a*][1,10]phenanthroline-9-carbonitrile compounds in excellent yield. All compounds are fully characterized with one structurally authenticated by a single X-ray diffraction study.

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INTRODUCTION

Extensive researches revolving around the biological, medicinal, photographic, and other useful applications of substituted indolizines have been made, resulting in frequent reviews of the chemistry, synthesis, and properties of this system and its analogs [1–5]. The increasing demand for mild, new, and efficient synthetic methods for indolizine heterocycles is due to their importance in the fields of biology and pharmacology [6–13].

Although there are a number of reports regarding new indolizines in recent years [14–20], the present article describes the synthesis of new indolizines with helical structure. In continuation of our interest in multicomponent reactions [21–23], we have developed an efficient condensation reaction of four components involving benzaldehyde derivatives **1**, malonitrile **2**, and isocyanides **3** in the presence of phenanthroline **4** to access unsymmetrical pyrroles [24]. We have established a proof of concept for such multicomponent condensation to access this class of compounds. Such an approach could be of interest to organic chemists in general. We deployed aldehyde with electron withdrawing groups and two different types of isocyanides bearing cyclohexyl or dimethylphenyl substituents.

RESULTS AND DISCUSSION

Herein, we now describe the results of the condensation reaction between benzaldehyde derivatives **1**, malonitrile **2**, isocyanides **3**, and phenanthroline **4**, which lead to compounds **5**, in excellent yields, shown in Scheme 1.

Compounds **5** are stable solids whose structures are fully characterized and supported by elemental analysis and spectroscopic data of FTIR, ^{1}H NMR and ^{13}C NMR, and mass spectroscopy. For instance, the mass spectrum of **5a** displayed the molecular ion [M^+] signal at $m/z = 450$ that is consistent with the product structure. The IR spectrum of **5a** showed absorption at 3421 and 2207 cm^{-1} indicating the presence of NH and CN functional groups.

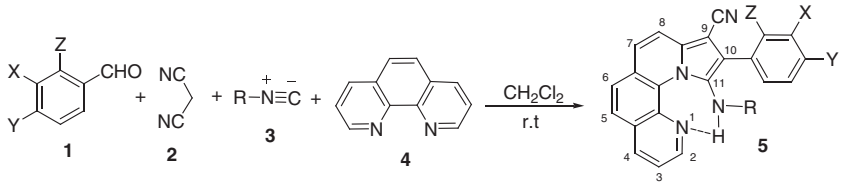
The ^{1}H NMR spectrum of **5a** exhibited multiplet signals arising from five methylene protons of the cyclohexyl group at $\delta = 0.52–1.19$ ppm and a multiplet at $\delta = 2.24$ ppm from NCH of the cyclohexyl group.

Characteristic signals of NH proton in ^{1}H NMR spectrum of **5a** are observed at $\delta = 6.09$ ppm as a broad singlet. The aromatic protons of **5a** resonated at $\delta = 7.31–9.10$ ppm in the aromatic region.

The ^{13}C NMR spectrum of compound **5a** showed characteristic signals for the carbon containing the cyanide group at $\delta = 84.2$ ppm, whereas the carbon of the cyanide group resonance appears at $\delta = 116.9$ ppm. The detailed spectroscopic information of compound **5a** is available in the experimental section.

Single-crystal X-ray diffraction [25] was used to authenticate structure of compound **5b**. Prismatic dark-red crystal of **5b** was prepared by slow evaporation of a saturated

Scheme 1. Synthesis of 10-(aryl)-11-(alkyl- or arylamino-)pyrrolo[1,2-a][1,10]phenanthroline-9-carbonitrile derivatives.



5	R	X	Y	Z	Yield%
a	cyclohexyl	Cl	H	H	93
b	2, 6-dimethylphenyl	Cl	H	H	97
c	cyclohexyl	H	NO_2	H	94
d	cyclohexyl	H	CH_3	H	93
e	cyclohexyl	H	H	F	96

solution of dichloromethane. Compound **5b** crystallizes in the triclinic space group, $Z = 2$, with the asymmetric unit comprising one molecule, shown in Figure 1.

Proton shift and HCN elimination for aromatization occur as proposed in the mechanism [26,27] shown in Scheme 2. Such a mechanism is consistent with the general structure of compound **5a** and is supported by spectroscopic data.

Helical structure and some interesting conformational parameters such as bond length, bond angels, and torsion angels of structure **5b** are shown in Figure 2.

In conclusion, we believe that the recent method benefits from a mild, simple, efficient, and one-pot synthetic procedure for the preparation of substituted phenanthroline derivatives with the probable pharmaceutical properties in the multicomponent reaction involving isocyanides. The products were obtained in quantitative yield without any prior activation or modifications. It seems that its ease of work-up and mild reaction conditions make it a useful approach to modern synthetic routes.

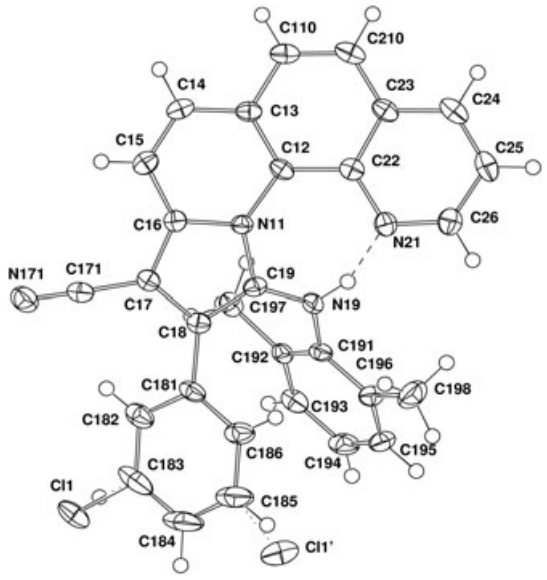


Figure 1. Molecular structure of the compound **5b**. Bonds to the disordered components are shown as dotted lines. The intramolecular H bond is indicated as a dashed line.

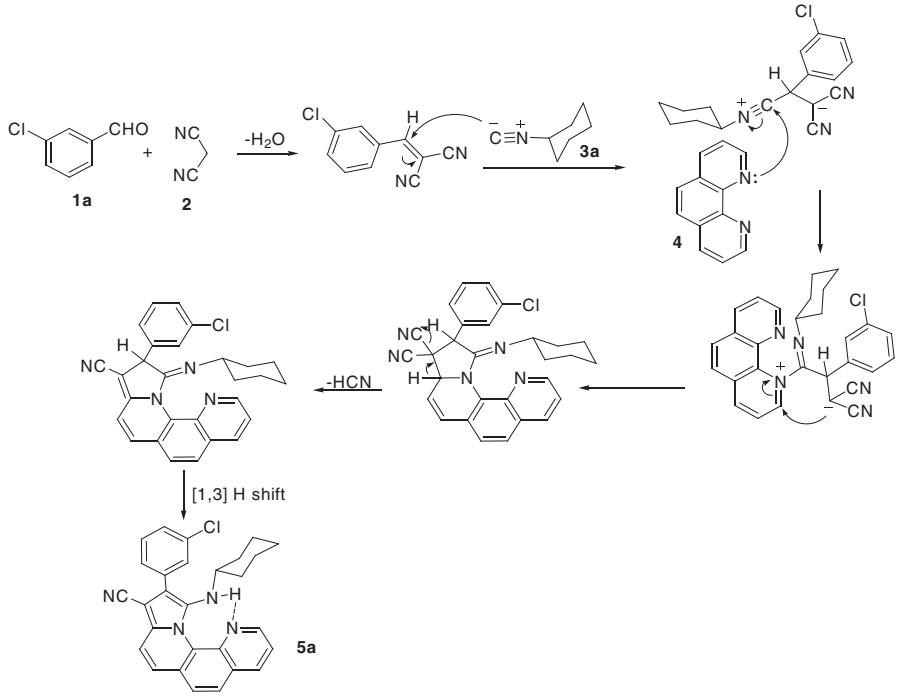
EXPERIMENTAL

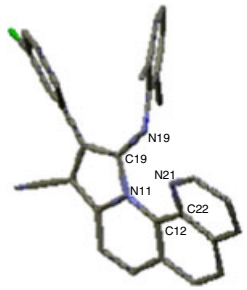
Melting points and IR spectra of all compounds were measured on an Electrothermal 9100 apparatus (Mount Holly, NJ) and a JASCO FTIR spectrometer (Easton, MD), respectively. Also, the ^{1}H NMR and ^{13}C NMR spectra were recorded on a BRUKER DRX-500 AVANCE instrument (Rheinstetten, Germany) using $CDCl_3$ as the applied solvent and TMS as internal standard at 500.1 and 125.8 MHz, respectively. Elemental analyses for C, H, and N were performed using a Heraeus CHN-O-Rapid analyzer (Banau, Germany). In addition, the mass spectra were recorded on Shimadzu GCMS-QP5050A mass spectrometer (Kyoto, Japan) operating at an ionization potential of 70 eV. Phenanthroline, malonitrile, isocyanide, and aldehyde derivatives were purchased from Fluka (Buchs, Switzerland), Merck (Darmstadt, Germany), and Aldrich (Milwaukee, WI) companies, respectively, and used without further purification.

General procedure for synthesis of compounds 5 (exemplified by 5a). To a stirred solution of 3-cholorobenzaldehyde (1 mmol), malonitrile (1 mmol), and phenanthroline (1 mmol) in CH_2Cl_2 (10 mL) was added, dropwise, a mixture of cyclohexyl isocyanide (1.1 mmol) in CH_2Cl_2 (3 mL) over 5 min at ambient temperature. The reaction mixture was stirred at ambient temperature for 15–48 h. The solvent was removed under reduced pressure, and the crude product was washed with diethyl ether (2×3 mL) and recrystallized by slow evaporation method (CH_2Cl_2/*n*-Hexane), then the crystalline solid was dried to afford compound **5a**.

10-(3-Chlorophenyl)-11-(cyclohexylamino)pyrrolo[1,2-a][1,10]phenanthroline-9-carbonitrile (5a). Red crystals; (0.42 g), yield 93%; mp 213–216°C, IR (KBr) (ν_{max}, cm^{-1}): 3421 (NH), 2207 (CN), 1638 (C=N). MS, *m/z* (%) = 452 ($M^+ + 2$, 33), 451 ($M^+ + 1$, 33), 450 (M^+, 85), 367 (100), 353 (4), 340 (7), 332 (24), 306 (4), 230 (2), 83 (4), 55 (18), 41 (15). *Anal*. Calcd for $C_{28}H_{23}ClN_4$ (450.96): C, 74.57; H, 5.14; N, 12.42. Found: C, 74.50; H, 4.99; N, 12.51. ^{1}H NMR (500.1 MHz, $CDCl_3$): 0.52–

Scheme 2. Proposed mechanism for synthesis of 10-(aryl)-11-(alkyl- or arylamino-)pyrrolo[1,2-*a*][1,10]phenanthroline-9-carbonitriles.





Entry	Bond length	(Å)	Entry	Atom1	Atom2	Atom3	Angle (°)	Entry	Atom1	Atom2	Atom3	Atom4	Angle (°)
1	N21–C22	1.366(2)	1	N21	C22	C12	120.8(2)	1	N11	C12	C22	N21	-11.6(3)
2	C12–C22	1.439(3)	2	N11	C12	C22	125.2(1)	2	C19	N11	C12	C22	-20.5(3)
3	N11–C12	1.426(2)	3	C12	N11	C19	132.6(1)	3	C12	N11	C19	N19	-9.2(3)
4	N11–C19	1.409(2)	4	N11	C19	N19	121.9(1)						
5	C19–N19	1.370(2)											

Figure 2. Selected bond lengths (Å), bond angles (°), and torsion angles (°) in helical structure of compound **5b**.[Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

1.19 (10H, m, 5 CH_2 of cyclohexyl), 2.24 (1H, m, NC*H* of cyclohexyl), 6.09 (1H, br s, N*H*), 7.31 (1H, dq, $J_1 = 0.7$, $J_2 = 7.8$ Hz, C$H_{chlorophen}$), 7.38 (1H, d, $J = 9.0$ Hz, C5-*H*), 7.39 (1H, t, $J = 8.2$ Hz, C$H_{chlorophen}$), 7.59 (1H, dd, $J_1 = 4.3$, $J_2 = 8.2$ Hz, C3-*H*), 7.79 (1H, d, $J = 9.0$ Hz, C6-*H*), 7.83 (1H, d, $J = 8.5$ Hz, C8-*H*), 7.86 (1H, d, $J = 8.5$ Hz, C7-*H*), 7.93 (1H, dd, $J_1 = 7.8$, $J_2 = 8.0$ Hz, C$H_{chlorophen}$), 8.19 (1H, t, $J = 1.8$ Hz, C$H_{chlorophen}$), 8.36 (1H, dd, $J_1 = 1.8$, $J_2 = 8.2$ Hz, C4-*H*), 9.10 (1H, dd, $J_1 = 1.8$, $J_2 = 4.3$ Hz, C2-*H*). ^{13}C NMR (125.8 MHz, $CDCl_3$): 24.2, 25.5, 33.4 and 56.6 (5 CH_2 and HN*C* of cyclohexyl), 84.2 (*C*9), 116.9 (-*C*N), 118.7, 121.0 (2 CH_{phen}), 122.2 and 125.4 (2 C$H_{chlorophen}$), 127.1 (CH_{phen}), 127.4 (C$H_{chlorophen}$), 127.5 (CH_{phen}), 128.1 (*C*10), 129.0, 129.4, 129.6 and 130.1 (2 C_{phen} and 2 CH_{phen}), 134.1 and 135.4 (2 $C_{chlorophen}$), 136.5 (C_{phen}), 137.1 (C$H_{chlorophen}$), 137.9 (C_{phen}), 139.8 (*C*11), 147.8 and 150.2 (C_{phen} and CH_{phen}).

10-(3-Chlorophenyl)-11-(2,6-dimethylphenylamino)pyrrolo[1,2-a][1,10]phenanthroline-9-carbonitrile (5b). Red crystals; (0.46 g), yield 97%; mp 267–270°C (decomposed), IR (KBr) (ν_{max}, cm^{-1}): 3422 (NH), 2207 (CN), 1664 (C=N). MS, *m/z* (%) = 474 ($M^+ + 2$, 7), 473 ($M^+ + 1$, 7), 472 (M^+, 18), 376 (58), 367 (18), 328 (39), 272 (100), 199 (11), 180 (66). *Anal*. Calcd for $C_{30}H_{21}ClN_4$ (472.97): C, 76.18; H, 4.48; N, 11.85. Found: C, 76.24; H, 4.43; N, 11.92. ^{1}H NMR (500.1 MHz, $CDCl_3$): 1.83 (6H, s, ArMe_2), 6.50 (1H, t, $J = 7.5$ Hz, Ar-*H*), 6.65 (2H,

d, $J = 7.5$ Hz, Ar-*H*), 7.05 (1H, d, $J = 8.2$ Hz, C$H_{chlorophen}$), 7.11 (1H, t, $J = 7.9$ Hz, C$H_{chlorophen}$), 7.36 (1H, t, $J = 7.6$ Hz, C5-*H*), 7.40 (1H, br s, C6-*H*), 7.46 (1H, d, $J = 8.9$ Hz, C7-*H*), 7.61 (1H, dd, $J_1 = 4.2$, $J_2 = 8.1$ Hz, C3-*H*), 7.88 (1H, d, $J = 8.9$ Hz, C8-*H*), 7.91 (2H, br s, 2 C$H_{chlorophen}$), 8.38 (1H, dd, $J_1 = 1.5$, $J_2 = 8.1$ Hz, C4-*H*), 8.49 (1H, br s, N*H*), 9.12 (1H, dd, $J_1 = 1.5$, $J_2 = 4.2$ Hz, C2-*H*). ^{13}C NMR (125.8 MHz, $CDCl_3$): 18.0 (Ar*Me*$_2$), 83.7 (*C*9), 116.2 (-*C*N), 118.3, 120.6 (2 *C*H$_{phen}$), 121.4 and 121.8 (2 *C*H$_{chlorophen}$), 125.5 (*C*H$_{aryl}$), 126.3 and 126.6 (2 *C*H$_{phen}$), 127.0 (*C*10), 127.7 (*C*H$_{phen}$), 128.2 (*C*$_{phen}$), 128.3 (2 *C*H$_{aryl}$ and *C*H$_{chlorophen}$), 128.6 (*C*H$_{phen}$), 129.1 (*C*$_{phen}$), 133.1 (2 *C*$_{aryl}$), 133.1 and 133.9 (2 *C*$_{phen}$), 136.2 and 137.2 (2 *C*$_{chlorophen}$), 139.4 (*C*11), 140.2 (*C*$_{aryl}$), 147.9 and 150.1 (*C*$_{phen}$ and *C*H$_{phen}$).

11-(Cyclohexylamino)-10-(4-nitrophenyl)H-pyrrolo[1,2-a][1,10]phenanthroline-9-carbonitrile (5c). Pale white crystals; (0.43 g), yield 94%; mp 236–239°C. IR (KBr) (ν_{max}, cm^{-1}): 3213 (NH), 2209 (CN), 1662 (C═N). MS, *m/z* (%) = 462 ($M^+ + 1$, 14), 461 (M^+, 44), 378 (87), 331 (45), 306 (7), 180 (100), 98 (6), 83 (12), 55 (21), 41 (14). *Anal*. Calcd for $C_{28}H_{23}N_5O_2$ (461.51): C, 72.87; H, 5.02; N, 15.17. Found: C, 72.93; H, 5.07; N, 15.09. ^{1}H NMR (500.1 MHz, $CDCl_3$): 0.54–1.24 (10H, m, 5 CH_2 of cyclohexyl), 2.25 (1H, m, NC*H* of cyclohexyl), 7.46 (1H, d, $J = 8.9$ Hz, C5-*H*), 7.64 (1H, dd, $J_1 = 4.2$, $J_2 = 7.9$ Hz, C3-*H*), 7.84 (1H, d, $J = 8.9$ Hz, C6-*H*), 7.89 (1H, d, $J = 8.5$ Hz, C7-*H*), 7.92 (1H, d, $J = 8.5$ Hz, C$H_{nitrophen}$), 8.29 (1H, d, $J = 8.5$ Hz, C8-*H*), 8.31–8.39 (3H, m, 3 C$H_{nitrophen}$), 8.42 (1H, dd, $J_1 = 1.7$, $J_2 = 7.9$ Hz, C4-*H*), 9.13 (1H, dd, $J_1 = 1.7$, $J_2 = 4.2$ Hz, C2-*H*), 9.18 (1H, br s, N*H*). ^{13}C NMR (125.8 MHz, $CDCl_3$): 24.2, 25.4, 33.4 and 57.1 (5 *C*H_2 and HN*C* of cyclohexyl), 83.8 (*C*9), 116.8 (-*C*N), 118.6, 121.2 (2 *C*H$_{phen}$), 123.1 (*C*H$_{phen}$), 123.7 (2 *C*H$_{nitrophen}$), 125.8 (*C*H$_{phen}$), 126.2 (*C*$_{phen}$), 127.5 (*C*H$_{phen}$), 128.2 (*C*10), 129.7 (*C*H$_{phen}$), 130.1, 136.1 and 137.1 (2 *C*$_{phen}$), 137.4 (2 *C*H$_{nitrophen}$), 139.6 (*C*$_{nitrophen}$), 140.7 (*C*11), 146.4 (*C*$_{nitrophen}$), 147.9 and 150.1 (*C*$_{phen}$ and *C*H$_{phen}$).

11-(Cyclohexylamino)-10-(p-tolyl)pyrrolo[1,2-a][1,10]phenanthroline-9-carbonitrile (5d). Red crystals; (0.42 g), yield 93%; mp 167–170°C. IR (KBr) (ν_{max}, cm^{-1}): 3373 (NH), 2204 (CN), 1649 (C═N). MS, *m/z* (%) = 431 ($M^+ + 1$, 6), 430 (M^+, 20), 347 (77), 180 (100), 154 (18), 119 (18), 83 (13), 55 (25), 41 (19). *Anal*. Calcd for $C_{29}H_{26}N_4$ (430.54): C, 80.90; H, 6.09; N, 13.01. Found: C, 80.96; H, 5.98; N, 12.93. ^{1}H NMR (500.1 MHz, $CDCl_3$): 0.55–1.28 (10H, m, 5 CH_2 of cyclohexyl), 2.28 (1H, m, NC*H* of cyclohexyl), 2.31 (3H, s, CH_3), 6.07 (1H, br s, N*H*), 7.32 (2H, d, $J = 8.0$ Hz, 2 CH_{tolyl}), 7.41 (1H, d, $J = 8.9$ Hz, C5-*H*), 7.60 (1H, dd, $J_1 = 4.3$, $J_2 = 8.1$ Hz, C3-*H*), 7.85 (1H, d, $J = 8.9$ Hz, C6-*H*), 7.87 (2H, br s, C7-*H*, C8-*H*), 7.87 (2H, d, $J = 8.0$ Hz, 2 CH_{tolyl}), 8.38 (1H, dd, $J_1 = 1.8$, $J_2 = 8.1$ Hz, C4-*H*), 9.12 (1H, dd, $J_1 = 1.8$, $J_2 = 4.3$ Hz, C2-*H*). ^{13}C NMR (125.8 MHz, $CDCl_3$): 21.2 (*C*H_3), 24.1, 25.4, 33.2 and 56.0 (5 *C*H_2 and HN*C* of cyclohexyl), 84.4 (*C*9), 117.2 (-*C*N), 118.5, 120.7 (2 *C*H$_{phen}$), 121.5 and 125.0 (2 *C*H$_{phen}$), 127.3 (*C*H$_{phen}$), 127.9 (*C*$_{tolyl}$), 128.8 (*C*$_{phen}$), 128.9 and 129.2 (4 *C*H$_{tolyl}$), 129.9 (*C*10), 130.4 (*C*$_{phen}$), 136.2 (*C*$_{tolyl}$), 136.8 (*C*11), 136.9 (*C*H$_{phen}$), 137.2 (*C*$_{phen}$), 139.8 (*C*$_{phen}$), 147.5 and 150.2 (*C*H$_{phen}$ and *C*$_{phen}$).

11-(Cyclohexylamino)-10-(2-fluorophenyl)pyrrolo[1,2-a][1,10]phenanthroline-9-carbonitrile (5e). Light red crystals; (0.42 g), yield 96%; mp 146–149°C. IR (KBr) (ν_{max}, cm^{-1}): 3417 (NH), 2204 (CN), 1656 (C═N). MS, *m/z* (%) = 435 ($M^+ + 1$, 9), 434 (M^+, 28), 351 (100), 324 (7), 180 (64), 83 (6), 55 (15), 41 (12). *Anal*. Calcd for $C_{28}H_{23}FN_4$ (434.51): C, 77.40; H, 5.34; N, 12.89. Found: C, 77.54; H, 5.29; N, 12.77. ^{1}H NMR (500.1 MHz, $CDCl_3$): 0.55–1.24 (10H, m, 5 CH_2 of cyclohexyl), 2.27 (1H, m, NC*H* of cyclohexyl), 6.18 (1H, br s, N*H*), 7.26 (1H, t, $J = 5.8$ Hz, C$H_{fluorophen}$), 7.30 (1H, t, $J = 7.5$ Hz, C$H_{fluorophen}$), 7.40 (1H, m, C$H_{fluorophen}$), 7.42 (1H, d, $J = 8.9$ Hz, C8-*H*), 7.63 (1H, dd, $J_1 = 4.2$, $J_2 = 8.1$ Hz, C3-*H*), 7.79 (1H, dt, $J_1 = 1.9$, $J_2 = 7.9$ Hz, C$H_{fluorophen}$), 7.85 (1H, d, $J = 8.9$ Hz, C7-*H*), 7.88 (2H, br s, C5-*H* and C6-*H*), 8.38 (1H, dd, $J_1 = 1.5$, $J_2 = 8.1$ Hz, C4-*H*), 9.10 (1H, dd, $J_1 = 1.5$, $J_2 = 4.2$ Hz, C2-*H*). ^{13}C NMR (125.8 MHz, $CDCl_3$): 24.1, 25.4, 33.2 and 55.7 (5 *C*H_2 and HN*C* of cyclohexyl), 85.9 (*C*9), 115.8 (d, $J_{CF} = 13.2$ Hz, *C*H$_{fluorophen}$), 116.0 (-*C*N), 118.6, 120.7 (2 *C*H$_{phen}$), 121.4 (d, $J_{CF} = 15.1$ Hz, *C*$_{fluorophen}$), 122.9 (*C*$_{phen}$), 123.8 (d, $J_{CF} = 3.4$ Hz, *C*H$_{fluorophen}$), 125.2 (*C*H$_{phen}$), 126.4 (*C*$_{phen}$), 127.2 (*C*H$_{phen}$), 127.9 (*C*H$_{phen}$), 128.8 (*C*10), 129.3 (d, $J_{CF} = 7.9$ Hz, *C*H$_{fluorophen}$), 129.9 (*C*$_{phen}$), 135.9 (d, $J_{CF} = 6.8$ Hz, *C*H$_{fluorophen}$), 138.1 (*C*$_{phen}$), 138.7 (*C*11), 138.9 (*C*H$_{phen}$), 147.6 and 150.1 (*C*H$_{phen}$ and *C*$_{phen}$), 160.3 (d, $J_{CF} = 248.4$ Hz, *C*H$_{fluorophen}$).

Crystal refinement details for compound 5b. $C_{30}H_{21}ClN_4$, $M = 472.96$, $F(000) = 492$, triclinic, P1, $Z = 2$, $T = 100(2)$ K, $a = 8.1485(6)$, $b = 12.1418(10)$, $c = 12.2529(9)$ Å, $\alpha = 77.113(7)$, $\beta = 85.161(6)$, $\gamma = 74.723(7)°$, $V = 1139.56(15)$ Å^3; $D_c = 1.378$ g cm^{-3}; $\mu_{Mo} = 0.196$ mm^{-1}; $\theta_{max} = 28.7°$; R_1 ($I > 2\sigma(I)$) = 0.0444, wR_2 (all data) = 0.0870, GOF = 0.864; $|\Delta\rho_{max}| = 0.31$ e Å^{-3}. Crystallographic data were collected at 100(2) K on an Oxford Diffraction Xcalibur diffractometer (Oxford Diffraction, Oxford, UK) fitted with graphite-monochromated Mo Kα radiation yielding 9062 reflections, these merging to 5049 unique after multiscan absorption corrections ($R_{int} = 0.0339$), with 2889 reflections having $I > 2\sigma(I)$. The structure was refined against F^2 with full-matrix least-squares using the program SHELXL-97 [25]. All H atoms were added at calculated positions and refined by use of a riding model with isotropic displacement parameters based on that of the parent atom. Anisotropic displacement parameters were employed throughout for the nonhydrogen atoms. The Ph ring 18n is rotationally disordered about the C181-C184 line resulting in the Cl atom being disordered over the 3-position and 5-position with occupancies refined to 0.655(1) and its complement for the two sites. The crystal structure for **5b** is depicted in Figure 1 where ellipsoids have been drawn at the 50% probability level. CCDC number 745666 contains the crystallographic data for compound **5b**. These data can be obtained free of charge at www.ccdc.cam.ac.uk/conts/retrieving.html or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: (internat.) +44-1223/336-033; Email: deposit@ccdc.cam.ac.uk.

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