Supporting Information

Gold-catalyzed chemo- and diastereo-selective C(sp^2)-H functionalization of enaminones for the synthesis of Pyrrolo[3,4-*c*]-quinolin-1-one derivatives

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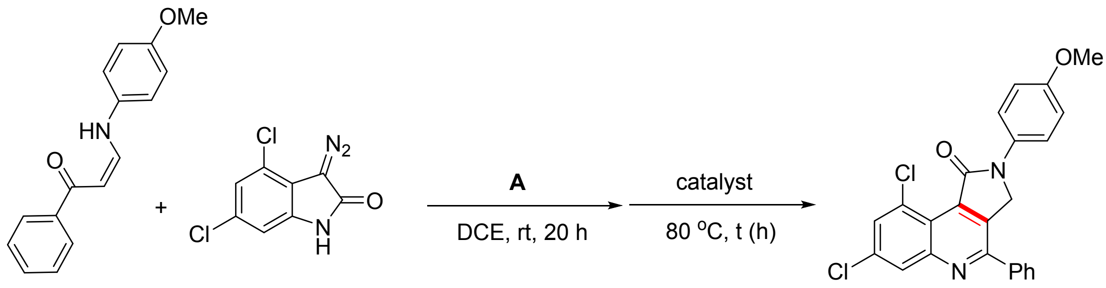
General Methods. All reactions were carried out under air except noted. Anhydrous CH_2ClCH_2Cl were prepared by distillation from CaH_2. Toluene was distilled from sodium and benzophenone. Unless noted, all commercial reagents were used without further purification. Ph_3PAuCl[1] and PPh_3AuNTf_2[2] were prepared according to the published methods. (Acetonitrile)[(2-biphenyl)di-tert-butylphosphine]gold(I) hexafluoroantimonate was purchased from Aldrich Chemical Company. $AgNTf_2$ was purchased from Sinocompound Technology Company. 3-Diazooxindoles[3] were synthesized according to literature procedures. Enaminones[4] were prepared according to the published methods.

Reactions were monitored by thin layer chromatography using UV light to visualize the course of reaction. Purification of reaction products was carried out by flash chromatography on silica gel or filtration. Chemical yields refer to pure isolated substances. ^1H and ^{13}C NMR spectra were obtained using a Bruker DPX-400 or 500 spectrometer. Chemical shifts are reported in ppm from $CDCl_3$, CD_2Cl_2 or $(CD_3)_2SO$ with the solvent resonance as the internal standard. The following abbreviations were used to designate chemical shift multiplicities: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, br = broad. High-resolution mass spectra was obtained by using Waters Micromass GCT, Bruker Daltonics, Inc. APEXIII 7.0 TESLA FTMS, IonSpec 4.7 Tesla FTMS or Agilent Technologies 6224 TOF LC/MS mass spectrometer. Single crystal X-ray diffraction data was collected in Bruker SMARTAPEX diffractiometers with molybdenum cathodes.

Optimization of one-pot reaction conditions.[a]

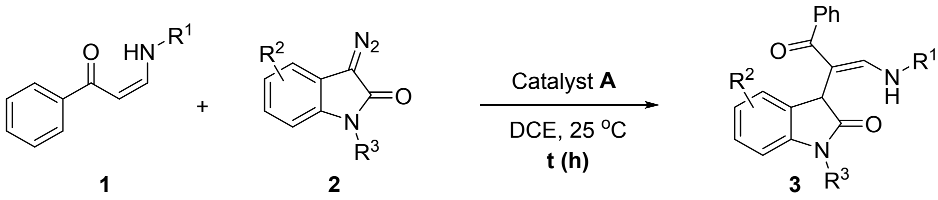


Entry	Catalyst	Additive	Time [h]	Yield [%][b]
1	TsOH	-	4.0	83
2	CF_3CO_2H	-	7.5	79
3	$FeCl_3$	-	4.5	-
4	TsOH	4Å MS	7.0	trace
5	TsOH	-	13.0	62[c]
6	TsOH	-	4.0	78[d]
7	TsOH	-	4.0	82[e]

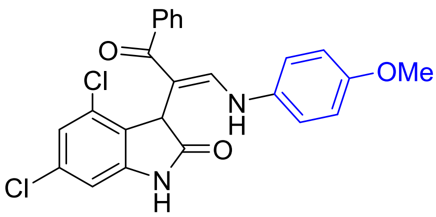
[a] Unless otherwise noted, reactions were carried out with **1** (0.25 mmol), **2** (0.25 mmol), and **A** (4 mol%), in DCE (2.5 mL) for 20 h. After the first step, catalyst (20 mol%) was added. [b] Isolated yield. [c] 10 mol% of TsOH was used. [d] 2.0 mL of DCE was used. [e] The reaction was carried out under N2 atmosphere.

After complete investigation of the two separate reactions, to build a one-pot reaction became feasible. With the best separate reaction conditions in hand, the one-pot reaction of **1a** with **2a** was carried out with 4 mol% of catalyst **A** under ambient temperature. After 20 hours, 20 mol% of TsOH was added for another 4.0 h at 80 °C, and the desired polycyclic compound **4a** could be isolated with the best yield of 83% (entry 1). When the TsOH was replaced with CF_3CO_2H, **4a** could only be obtained with 79% yield (entry 2). No target product was obtained with $FeCl_3$ as the catalyst (entry 3). When the 4 Å molecular sieve and TsOH were added to the reaction together, only trace amount of **4a** was obtained (entry 4). No better results could be achieved by reduce the usage of catalyst or solvent (entries 5-6). And no significant difference could be found, when the reaction was carried out under nitrogen atmosphere. (entry 7).

A typical procedure for the synthesis of 3.

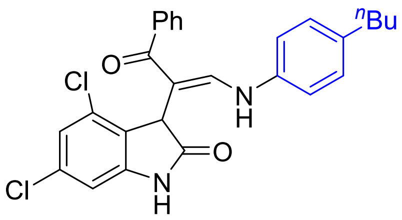


Under an atmosphere of air, to a vial were added enaminone **1** (0.25 mmol), diazooxindole **2** (0.25 mmol) and JohnPhos(MeCN)$AuSbF_6$ (**A**) (4 mol%), followed by anhydrous CH_2ClCH_2Cl (2.5 mL). The resulting mixture was stirred at ambient temperature till diazooxindole **2a** disappeared by TLC analysis. Then, 5 mL petroleum ether was added to the mixture, and kept stirring for 15 min. The mixture was filtered, and the filter cake was washed with petroleum ether (5 mL). The pure filter cake **3a** was collected and dried. (If the filtrate still containing a small amount of **3a** in individual substrates judging by TLC, the small amount of product could be recovered by column chromatographic purification using CH_2Cl_2/acetone (from 10/1 to 5/1, v/v) as the eluent.)



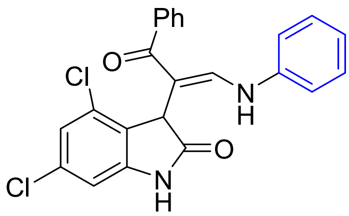
3a

(**3a**): Yellow solid, 96% yield (112 mg, t = 20 h), mp 167-168 °C; ^1H NMR (500 MHz, DMSO): δ 10.78 (s, 1H) , 9.73 (d, J = 13.5 Hz, 1H), 7.67 (d, J = 13. 5Hz, 1H), 7.49-7.44 (m, 5H), 7.02-6.84 (m, 6H), 4.93 (s, 1H) 3.70 (s, 3H); ^{13}C NMR (125 MHz, DMSO): δ 192.30, 176.83, 155.37, 148.19, 146.22, 139.80, 134.49, 132.32, 130.20, 128.99, 128.31, 128.26, 126.23, 120.48, 117.49, 115.03, 108.00, 107.88, 55.29, 43.62; HRMS (ESI) calcd for $C_{24}H_{19}Cl_2N_2O_3$ $[M+H]^+$: 453.0767, found 453.0769.



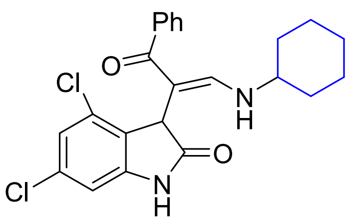
3b

(**3b**): Yellowish solid, 94% yield (113 mg, t = 12 h), mp 148-149 ºC; ^{1}H NMR (400 MHz, DMSO): δ 10.79 (s, 1H) , 9.77 (d, *J* = 13.6 Hz, 1H), 7.73 (d, *J* = 13.6 Hz, 1H), 7.54-7.44 (m, 5H), 7.14 (d, *J* = 8.4 Hz, 2H), 7.03 (d, *J* = 2.0 Hz, 1H), 6.93 (d, *J* = 8.4 Hz, 2H), 6.85 (d, *J* = 2.0 Hz, 1H), 4.97 (s, 1H), 2.52-2.48 (m, 2H), 1.52-1.47 (m, 2H), 1.30-1.23 (m, 2H), 0.87 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, DMSO): δ 193.19, 177.38, 148.02, 146.70, 140.12, 139.29, 137.40, 132.76, 130.73, 129.93, 129.39, 128.74, 128.68, 126.55, 120.85, 116.25, 108.73, 108.32, 43.63, 33.98, 33.10, 21.52, 13.62; HRMS (ESI) calcd for $C_{27}H_{24}Cl_2N_2NaO_2$ [M+Na]$^+$: 501.1107, found 501.1095.



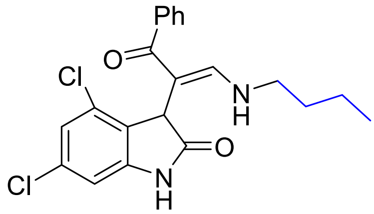
3c

(**3c**): Yellow solid, 86% yield (91 mg, t = 20 h), mp 162-163 ºC; ^{1}H NMR (400 MHz, DMSO): δ 10.80 (s, 1H) , 9.81 (d, *J* = 13.2 Hz, 1H), 7.77 (d, *J* = 13.6 Hz, 1H), 7.56-7.42 (m, 5H), 7.36-7.28 (m, 2H), 7.06-6.97 (m, 4H), 6.85 (s, 1H), 4.99 (s, 1H); ^{13}C NMR (100 MHz, DMSO): δ 193.38, 177.33, 147.66, 146.70, 141.50, 140.02, 132.81, 130.84, 130.20, 129.41, 128.79, 128.73, 126.49, 123.19, 120.88, 116.19, 109.26, 108.36, 43.66; HRMS (ESI) calcd for $C_{23}H_{16}Cl_2N_2NaO_2$ [M+Na]$^+$: 445.0481, found 445.0494.



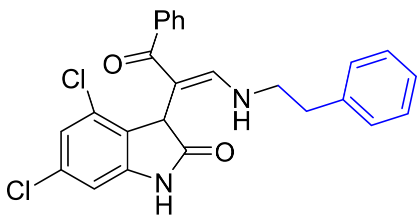
3d

(**3d**): White solid, 89% yield (95 mg, t = 20 h), mp 147-149 ºC; ^{1}H NMR (400 MHz, DMSO): δ 10.63 (s, 1H) , 7.70-7.60 (m, 1H), 7.46-7.34 (m, 3H), 7.34-7.21 (m, 3H), 6.98 (s, 1H), 6.78 (s, 1H), 4.64 (s, 1H), 3.10 (s, br, 1H), 1.85-1.75 (m, 2H), 1.69 (s, br, 2H), 1.60-1.50 (m, 1H), 1.40-1.35 (m, 4H), 1.10-1.00 (m, 1H); ^{13}C NMR (100 MHz, DMSO): δ 191.62, 178.02, 155.32, 146.75, 141.06, 132.35, 129.80, 129.25, 128.45, 127.27, 120.65, 107.98, 103.78, 57.29, 43.38, 34.12, 33.30, 24.76, 24.33; HRMS (ESI) calcd for $C_{23}H_{22}Cl_2N_2NaO_2$ $[M+Na]^+$: 451.0951, found 451.0970.



3e

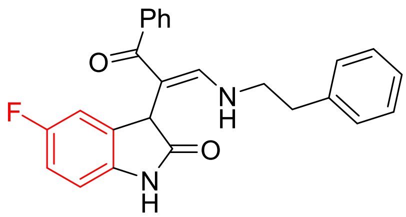
(**3e**): Yellowish solid, 90% yield (91 mg, t = 20 h), mp 227-228 °C; ^{1}H NMR (400 MHz, DMSO): δ 10.63 (s, 1H) , 7.81-7.70 (m, 1H), 7.45-7.35 (m, 3H), 7.35-7.26 (m, 2H), 7.21 (d, J = 14.0 Hz, 1H), 6.99 (d, J = 1.6 Hz, 1H), 6.78 (d, J = 1.6 Hz, 1H), 4.55 (s, 1H), 3.25-3.15 (m, 2H), 1.50-1.41 (m, 2H), 1.40-1.25 (m, 2H), 0.87 (t, J = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, DMSO): δ 191.53, 177.86, 157.07, 146.77, 141.00, 132.37, 129.85, 129.16, 128.45, 128.41, 127.19, 120.62, 108.01, 103.87, 47.76, 43.34, 32.77, 18.74, 13.39; HRMS (ESI) calcd for $C_{21}H_{20}Cl_2N_2NaO_2$ $[M+Na]^+$: 425.0794, found 425.0804.



3f

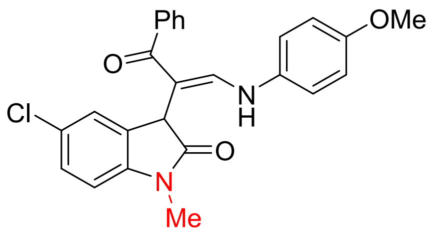
(**3f**): White solid, 89% yield (101 mg, t = 20 h), mp 145-146 °C; ^{1}H NMR (400 MHz, DMSO): δ 10.60 (s, 1H) , 7.90-7.77 (m, 1H), 7.41-7.23 (m, 8H), 7.07-6.93 (m, 4H), 6.77 (d, J = 1.6 Hz, 1H), 4.50 (s, 1H), 3.60-3.37 (m, 2H), 2.90-2.68 (m, 2H); ^{13}C NMR (100 MHz, DMSO): δ 191.33, 177.80, 157.03, 146.73, 140.64, 139.03, 132.32, 129.83, 129.55, 129.25, 128.74, 128.58, 128.29, 127.19, 126.65, 120.52, 107.93, 103.71, 49.66, 43.37, 36.96;

HRMS (ESI) calcd for $C_{25}H_{20}Cl_2N_2NaO_2$ $[M+Na]^+$: 473.0794, found 473.0809.



3g

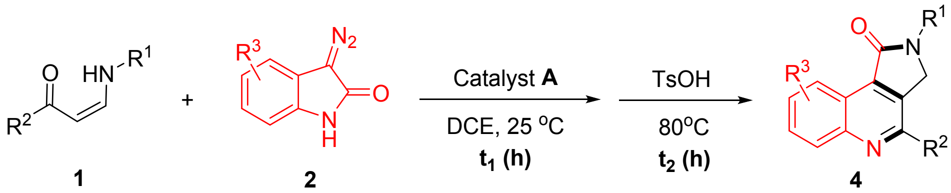
(**3g**): Yellowish solid, 85% yield (85 mg, t = 10 h), mp 125-126 °C; ^{1}H NMR (400 MHz, DMSO): δ 10.22 (s, 1H) , 7.69 (s, br, 1H), 7.44-7.19 (m, 8H), 7.05-6.86 (m, 4H), 6.79-6.70 (m, 1H), 6.64 (d, J = 8.4 Hz, 1H), 4.40 (s, 1H), 3.60-3.35 (m, 2H), 2.85-2.70 (m, 2H); ^{13}C NMR (100 MHz, DMSO): δ 191.28, 178.59, 158.21 (d, $J_{C\text{-}F}$ = 253.2 Hz), 156.89, 140.66, 139.91, 139.26, 133.17 (d, $J_{C\text{-}F}$ = 7.9 Hz), 129.81, 129.69, 128.82, 128.45, 128.28, 126.75, 113.12 (d, $J_{C\text{-}F}$ = 22.6 Hz), 110.38 (d, $J_{C\text{-}F}$ = 24.1 Hz), 109.34 (d, $J_{C\text{-}F}$ = 8.1 Hz), 105.63, 49.80, 44.04, 36.77; HRMS (ESI) calcd for $C_{25}H_{21}FN_2NaO_2$ $[M+Na]^+$: 423.1479, found 423.1489.



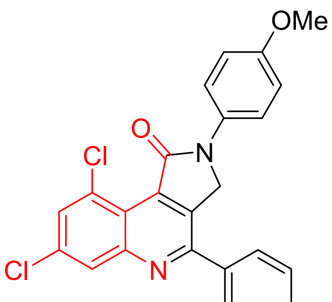
3h

(**3h**): Yellow solid, 87% yield (94 mg, t = 12 h), mp 110-112 °C; ^{1}H NMR (400 MHz, DMSO): δ 9.73 (d, J = 13.2 Hz, 1H) , 7.70 (d, J = 13.2 Hz, 1H), 7.53-7.42 (m, 5H), 7.31 (dd, J = 8.4 Hz, 2.4 Hz 1H), 7.16 (s, 1H), 7.05-6.90 (m, 5H), 4.85 (s, 1H), 3.71 (s, 3H), 3.20 (s, 3H); ^{13}C NMR (100 MHz, DMSO): δ 192.75, 176.32, 155.86, 148.69, 144.11, 140.13, 135.00, 132.01, 130.65, 128.74, 128.58, 127.37, 125.97, 122.95, 118.00, 115.29, 109.73, 109.45, 55.35, 43.45, 26.29; HRMS (ESI) calcd for $C_{25}H_{21}ClN_2NaO_3$ $[M+Na]^+$: 455.1133, found 455.1128.

A typical procedure for the synthesis of 4.

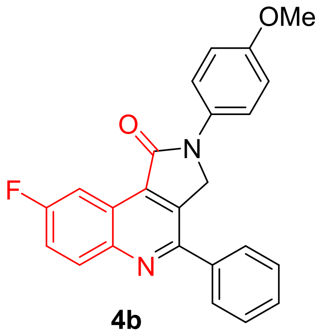


Under an atmosphere of air, to a vial were added enaminone **1** (0.25 mmol), diazooxindole **2** (0.25 mmol) and JohnPhos(MeCN)AuSbF$_6$ (**A**) (4 mol%), followed by anhydrous CH_2ClCH_2Cl (2.5 mL). The resulting mixture was stirred at ambient temperature till diazooxindole **2** disappeared by TLC analysis. Then, 20 mol% of TsOH was added, and the reaction temperature was raised to 80 °C till most of the intermediate **3** disappeared by TLC analysis. Subsequently, the reaction was cooled to room temperature, and the mixture was subjected to column chromatography for purification directly, using petroleum ether/CH_2Cl_2 (from 2/1 to 0/1, v/v) as the eluent.



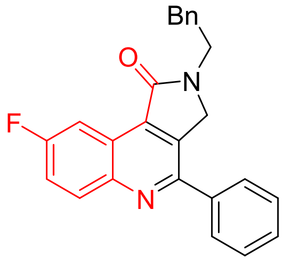
4a

(**4a**):Yellow solid, 83% yield (91 mg, t_1 = 20 h, t_2 = 4 h), mp 221-222°C; ^{1}H NMR (400 MHz, CD_2Cl_2): δ 8.14 (d, *J* = 2.0 Hz, 1H), 7.92-7.88 (m, 2H), 7.72 (d, *J* = 2.0 Hz, 1H), 7.65 (d, *J* = 8.8 Hz, 2H), 7.60-7.57 (m, 3H), 6.91 (d, *J* = 8.8 Hz, 2H), 4.97 (s, 2H), 3.79 (s, 3H); ^{13}C NMR (100 MHz, CD_2Cl_2): δ164.85, 158.26, 156.66, 151.16, 138.42, 138.21, 136.66, 135.66, 132.86, 132.07, 131.56, 131.20, 129.99, 129.45, 123.15, 121.74, 115.05, 56.07, 51.29; HRMS (ESI) calcd for $C_{24}H_{17}Cl_2N_2O_2$ [M+H]$^+$: 435.0662, found 435.0663.



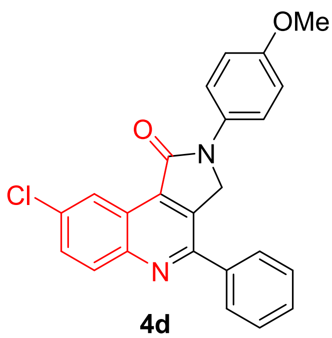
4b

(**4b**): Yellowish solid, 64% yield (62 mg, t_1 = 18 h, t_2 = 0.6 h), mp 208-209°C; ^{1}H NMR (400 MHz, CD_3Cl): δ 8.75 (dd, *J* = 9.6 Hz, 2.8 Hz, 1H), 8.23-8.19 (m, 1H), 7.90 (d, *J* = 7.2 Hz, 2H), 7.70 (d, *J* = 8.8 Hz, 2H), 7.59-7.49 (m, 4H), 6.89 (d, J = 9.2 Hz, 2H), 4.99 (s, 2H), 3.79 (s, 3H); ^{13}C NMR (100 MHz, CD_3Cl): δ167.03, 162.34 (d, J_{C-F} = 250.5 Hz), 157.78, 153.34, 146.22, 138.71, 136.36 (d, J_{C-F} = 6.0 Hz), 133.58, 132.72 (d, J_{C-F} = 9.4 Hz), 132.28, 130.32, 129.68 , 128.77, 123.91 (d, J_{C-F} = 11.7 Hz), 122.21, 121.01 (d, J_{C-F} = 26.1 Hz), 114.88, 107.84 (d, J_{C-F} = 23.9 Hz), 55.70, 51.28; HRMS (ESI) calcd for $C_{24}H_{18}FN_2O_2$ $[M+H]^+$: 385.1347, found 385.1345.



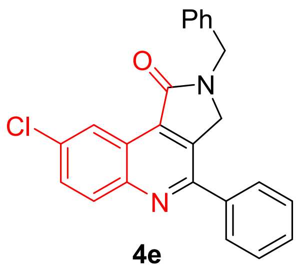
4c

(**4c**): White solid, 65% yield (63 mg, t_1 = 14 h, t_2 = 0.6 h), mp 156-158 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.77 (dd, *J* = 9.6 Hz, 3.2 Hz, 1H) , 8.24 (dd, *J* = 9.6 Hz, 5.6 Hz, 1H), 7.85-7.74 (m, 2H), 7.59-7.47 (m, 4H), 7.34-7.21 (m, 5H), 4.46 (s, 2H), 3.95 (t, *J* = 7.2 Hz, 2H), 3.06 (t, *J* = 7.6 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 168.18, 162.06 (d, J_{C-F} = 250.3 Hz), 153.23 (d, J_{C-F} = 2.9 Hz), 145.87, 138.84, 138.58, 136.06 (d, J_{C-F} = 6.1 Hz), 134.43, 132.39 (d, J_{C-F} = 9.3 Hz), 129.98, 129.34, 129.14, 129.09, 128.50, 127.09, 123.77 (d, J_{C-F} = 11.8 Hz), 120.72 (d, J_{C-F} = 26.0 Hz), 107.66 (d, J_{C-F} = 27.3 Hz), 50.65, 44.25, 34.85; HRMS (ESI) calcd for $C_{25}H_{19}FN_2NaO$ $[M+Na]^+$: 405.1374, found 405.1385.



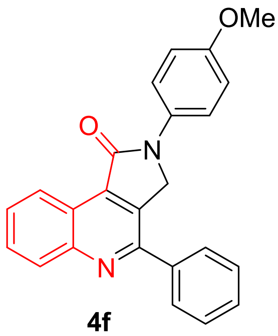
4d

(**4d**): Yellow solid, 63% yield (64 mg, t_1 = 24 h, t_2 = 0.8 h), mp 209-211°C; ^{1}H NMR (400 MHz, CD_3Cl): δ 9.15 (s, 1H), 8.16 (d, *J* = 8.8 Hz, 1H), 7.92 (d, *J* = 7.2 Hz, 2H), 7.72 (d, *J* = 8.8 Hz, 3H), 7.60-7.53 (m, 3H), 6.92 (d, *J* = 8.8 Hz, 2H), 5.03 (s, 2H), 3.81 (s, 3H); ^{13}C NMR (100 MHz, CD_3Cl): δ166.91, 157.82, 154.29, 147.43, 138.62, 136.01, 135.01, 133.67, 132.23, 131.85, 131.71, 130.48, 129.73, 128.81, 123.66, 123.11, 122.28, 114.90, 55.73, 51.33; HRMS (ESI) calcd for $C_{24}H_{18}ClN_2O_2$ $[M+H]^+$: 401.1051, found 401.1049.



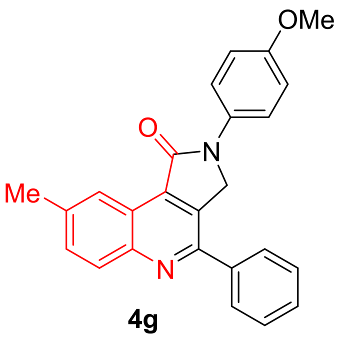
4e

(**4e**): White solid, 70% yield (68 mg, t_1 = 12 h, t_2 = 1 h), mp 138-139 ºC; ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.20 (d, *J* = 2.4 Hz, 1H) , 8.18 (d, *J* = 8.8 Hz, 1H), 7.86-7.79 (m, 2H), 7.74 (dd, *J* = 9.2 Hz, 2.4 Hz, 1H), 7.56-7.48 (m, 3H), 7.38-7.29 (m, 5H), 4.90 (s, 2H), 4.58 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 168.33, 154.45, 147.38, 138.71, 137.19, 135.72, 134.94, 134.79, 131.84, 131.68, 130.37, 129.62, 129.61, 128.81, 128.73, 128.59, 123.82, 123.22, 49.87, 46.71; HRMS (ESI) calcd for $C_{24}H_{17}ClN_2NaO$ $[M+Na]^+$: 407.0922, found 407.0914.



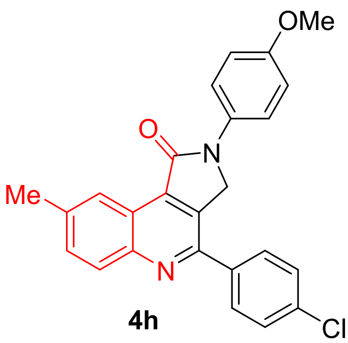
4f

(**4f**): Yellow solid, 69% yield (64 mg, t_1 = 48 h, t_2 = 2 h), mp 197-198ºC; ^{1}H NMR (400 MHz, CD_3Cl): δ 9.15 (d, *J* = 8.0 Hz, 1H), 8.23 (d, *J* = 8.4 Hz, 1H), 7.91 (d, *J* = 7.2 Hz, 2H), 7.81-7.64 (m, 4H), 7.58-7.51 (m, 3H), 6.90 (d, *J* = 8.8 Hz, 2H), 4.99 (s, 2H), 3.78 (s, 3H); ^{13}C NMR (100 MHz, CD_3Cl): δ167.40, 157.67, 154.09, 149.05, 138.98, 136.62, 132.88, 132.41, 130.72, 130.24, 130.22, 129.63, 128.84, 128.71, 123.99, 123.19, 122.27, 114.82, 55.68, 51.26; HRMS (ESI) calcd for $C_{24}H_{19}N_2O_2$ [M+H]$^+$: 367.1441, found 367.1442.



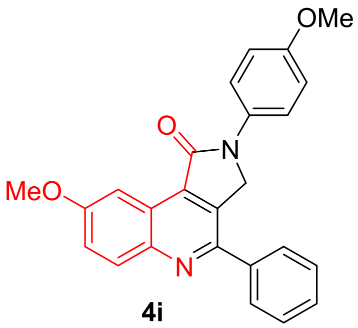
4g

(**4g**): Yellow solid, 66% yield (63 mg, t_1 = 23 h, t_2 = 0.6 h), mp 152-154ºC; ^{1}H NMR (400 MHz, CD_3Cl): δ 8.95 (s, 1H), 8.13 (d, *J* = 8.8 Hz, 1H), 7.91 (d, *J* = 7.2 Hz, 2H), 7.76 (d, *J* = 8.8 Hz, 2H), 7.65-7.49 (m, 4H), 6.95 (d, *J* = 8.8 Hz, 2H), 5.02 (s, 2H), 3.82 (s, 3H), 2.60 (s, 3H); ^{13}C NMR (100 MHz, CD_3Cl): δ167.66, 157.72, 153.12, 147.77, 139.25, 139.15, 135.91, 133.06, 132.86, 132.58, 130.06, 129.95, 129.62, 128.81, 123.26, 122.86, 122.31, 114.89, 55.74, 51.29, 22.10; HRMS (ESI) calcd for $C_{25}H_{21}N_2O_2$ [M+H]$^+$: 381.1598, found 381.1599.



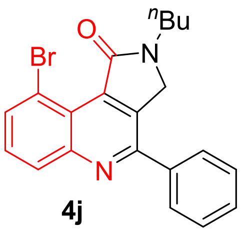
4h

(**4h**): Yellow solid, 62% yield (64 mg, t_1 = 12 h, t_2 = 1 h), mp 226-228 ºC; ^{1}H NMR (400 MHz, CD_2Cl_2): δ 8.93 (s, 1H) , 8.09 (d, *J* = 8.4 Hz, 1H), 7.96-7.90 (m, 2H), 7.80-7.73 (m, 2H), 7.66 (dd, *J* = 8.4 Hz, 2.0 Hz, 1H), 7.60-7.52 (m, 2H), 7.03-6.95 (m, 2H), 5.02 (s, 2H), 3.83 (s, 3H), 2.61 (s, 3H); ^{13}C NMR (100 MHz, CD_2Cl_2): δ 167.49, 158.01, 151.75, 147.85, 139.73, 137.85, 136.28, 136.20, 133.25, 132.96, 132.81, 130.40, 130.07, 129.80, 123.46, 122.85, 122.54, 114.97, 55.87, 51.53, 22.05; HRMS (ESI) calcd for $C_{25}H_{20}ClN_2O_2$ [M+H]$^+$: 415.1208, found 415.1214.



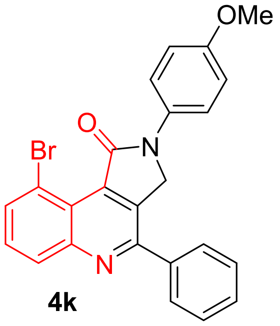
4i

4i): yellowish solid, 64% yield (63 mg, t_1 = 20 h, t_2 = 2 h), mp 75-77 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.46 (d, J = 2.8 Hz, 1H) , 8.09 (d, J = 9.2 Hz, 1H), 7.89 (d, J = 7.2 Hz, 2H), 7.73 (d, J = 9.2 Hz, 2H),7.59-7.45 (m, 3H), 7.41 (dd, J = 9.2, 2.8 Hz, 1H), 6.93 (d, J = 8.8 Hz, 2H), 4.98 (s, 2H), 3.98 (s, 3H), 3.81 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 167.57, 159.77, 157.47, 150.89, 145.06, 138.89, 134.80, 132.72, 132.25, 131.43, 129.62, 129.34, 128.47, 124.33, 123.62, 122.05, 114.61, 100.96, 55.77, 55.49, 51.00; HRMS (ESI) calcd for $C_{25}H_{21}N_2O_3$ $[M+H]^+$: 397.1547, found 397.1551.



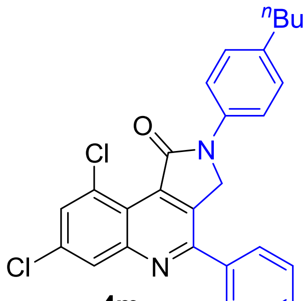
4j

(**4j**): White solid, 61% yield (60 mg, t_1 = 20 h, t_2 = 1 h), mp 142-143 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.25-8.18 (m, 1H) , 8.04-7.98 (m, 1H), 7.90-7.84 (m, 2H), 7.62-7.52 (m, 4H), 4.59 (s, 2H), 3.67 (t, J = 7.2 Hz, 2H), 1.75-1.65 (m, 2H), 1.45-1.35 (m, 2H), 0.96 (t, J = 7.6 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 166.26, 154.94, 150.50, 138.02, 137.73, 136.63, 135.50, 130.34, 130.30, 130.07, 129.42, 128.72, 124.40, 117.00, 49.56, 42.88, 30.32, 20.07, 13.62; HRMS (ESI) calcd for $C_{21}H_{19}BrN_2NaO$ $[M+Na]^+$: 417.0573, found 417.0566.



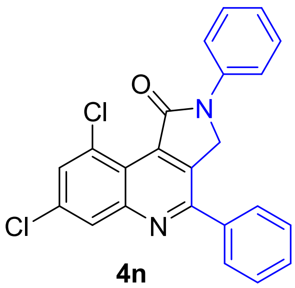
4k

(**4k**): Yellow solid, 70% yield (78 mg, t_1 = 21 h, t_2 = 6.0 h), mp 238-240°C; ^{1}H NMR (400 MHz, CD_3Cl): δ 8.22 (d, J = 8.0 Hz, 1H), 8.01 (d, J = 7.6 Hz, 1H), 7.89 (d, J = 6.8 Hz, 2H), 7.69 (d, J = 8.8 Hz, 2H), 7.62-7.54 (m, 4H), 6.92 (d, J = 9.2 Hz, 2H), 4.99 (s, 2H), 3.80 (s, 3H); ^{13}C NMR (100 MHz, CD_3Cl): δ165.02, 157.83, 155.10, 150.79, 138.01, 137.80, 135.88, 132.42, 130.64, 130.61, 130.47, 129.72, 128.92, 124.37, 122.96, 117.18, 114.83, 55.73, 50.86; HRMS (ESI) calcd for $C_{24}H_{18}BrN_2O_2$ [M+H]$^+$: 445.0546, found 445.0553.



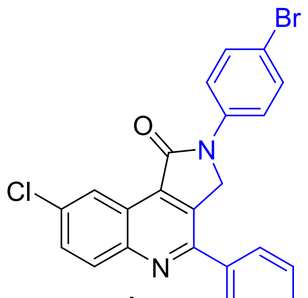
4m

(**4m**): Yellow solid, 75% yield (86 mg, t_1 = 7 h, t_2 = 6 h), mp 172-174°C; ^{1}H NMR (400 MHz, CD_3Cl): δ8.17 (d, J = 1.6 Hz, 1H), 7.89-7.86 (m, 2H), 7.74(d, J = 2.0 Hz, 1H), 7.66 (d, J = 8.4 Hz, 2H),7.60-7.57 (m, 3H),7.20 (d, J = 8.4 Hz, 2H), 5.01 (s, 2H), 2.59 (t, J = 7.6 Hz, 2H), 1.60-1.54 (m, 2H), 1.38-1.32 (m, 2H), 0.93 (t, J = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, CD_3Cl): δ164.70, 156.32, 150.81, 140.98, 138.18, 137.78, 136.85, 136.01, 135.60, 131.96, 131.42, 130.88, 129.75, 129.68, 129.06, 128.96, 121.44, 121.16, 50.65, 35.21, 33.71, 22.39, 14.00; HRMS (ESI) calcd for $C_{27}H_{23}Cl_2N_2O$ [M+H]$^+$: 461.1182, found 461.1184.



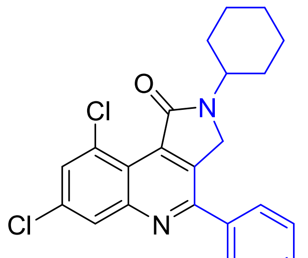
4n

(**4n**): Yellowish solid, 64% yield (65 mg, t_1 = 12 h, t_2 = 11 h), mp 230-232°C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.17 (s, 1H), 7.87 (d, J = 6.0 Hz, 2H), 7.79-7.73 (m, 3H), 7.60-7.57 (m, 3H), 7.41-7.36 (m, 2H), 7.20-7.16 (m, 1H), 5.02 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 164.78, 156.34, 150.79, 139.25, 138.03, 137.72, 135.97, 135.66, 132.01, 131.36, 130.91, 129.78, 129.08, 128.94, 125.95, 121.36, 120.94, 50.45; HRMS (EI) calcd for $C_{23}H_{14}Cl_2N_2O$ [M^+]: 404.0483, found 404.0481.



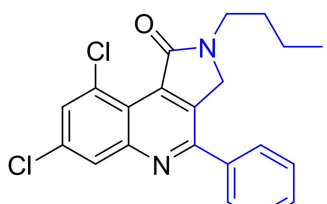
4o

(**4o**): Yellow solid, 51% yield (57 mg, t_1 = 19 h, t_2 = 0.5 h), mp 249-251°C; ^1H NMR (400 MHz, CD_2Cl_2): δ 9.15 (s, 1H), 8.20 (d, *J* = 9.2 Hz, 1H), 7.97 (d, *J* = 6.8 Hz, 2H), 7.85 (d, *J* = 8.8 Hz, 2H), 7.80-7.77 (m, 1H), 7.63-7.57 (m, 5H), 5.13 (s, 2H); ^{13}C NMR (100 MHz, CD_2Cl_2): δ 167.64, 154.83, 147.96, 139.10, 138.99, 136.06, 135.46, 134.26, 133.24, 132.37, 132.23, 130.96, 130.06, 129.31, 123.95, 123.34, 122.05, 118.82, 51.29; HRMS (ESI) calcd for $C_{23}H_{15}BrClN_2O$ [M+H]$^+$: 449.0051, found 449.0051.



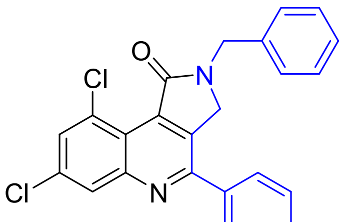
4p

(**4p**): Yellowish solid, 92% yield (94 mg, t_1 = 36 h, t_2 = 5 h), mp 180-182°C; ^1H NMR (400 MHz, CD_3Cl): δ 8.17 (d, *J* = 1.6 Hz, 1H), 7.86-7.83 (m, 2H), 7.74 (d, *J* = 1.6 Hz, 1H), 7.60-7.57 (m, 3H), 4.55 (s, 2H), 4.30 (s, br, 1H), 1.92-1.87 (m, 4H), 1.75-1.71 (m, 1H), 1.54-1.38 (m, 4H), 1.26-1.16 (m, 1H); ^{13}C NMR (100 MHz, CD_3Cl): δ165.24, 156.04, 150.32, 138.10, 137.70, 136.84, 134.96, 131.38, 131.12, 130.43 , 129.37, 128.68, 121.29, 51.43, 45.76, 31.08, 25.44, 25.26; HRMS (ESI) calcd for $C_{23}H_{21}Cl_2N_2O$ [M+H]$^+$: 411.1025, found 411.1026.



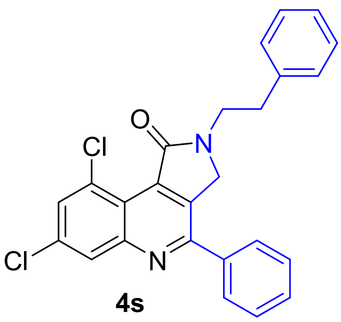
4q

(**4q**): Yellowish solid, 84% yield (81 mg, t_1 = 5 h, t_2 = 4 h), mp 142-144°C; ^1H NMR (400 MHz, CD_3Cl): δ 8.16 (d, *J* = 2.0 Hz, 1H), 7.86-7.83 (m, 2H), 7.73 (d, *J* = 1.6 Hz, 1H), 7.59-7.56 (m, 3H), 4.60 (s, 2H), 3.66 (t, J = 7.6 Hz, 2H), 1.72-1.64 (m, 2H), 1.43-1.36 (m, 2H), 0.96 (t, J = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, CD_3Cl): δ 166.06, 156.28, 150.70, 138.31, 137.96, 136.90, 135.37, 131.74, 131.46, 130.76, 129.67, 128.95, 121.63, 49.87, 43.19, 30.51, 20.29, 13.84; HRMS (ESI) calcd for $C_{21}H_{19}Cl_2N_2O$ [M+H]$^+$: 385.0869, found 385.0869.



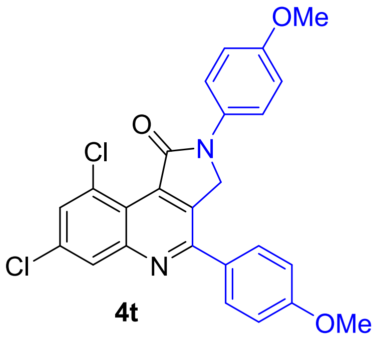
4r

(**4r**): White solid, 68% yield (71 mg, t_1 = 20 h, t_2 = 3.5 h), mp 179-181°C; ^{1}H NMR (400 MHz, CD_3Cl): δ8.15 (d, J= 1.2 Hz, 1H), 7.77-7.74 (m, 3H), 7.53-7.51 (m, 3H), 7.34-7.28 (m, 5H), 4.85 (s, 2H), 4.50 (s, 2H); ^{13}C NMR (100 MHz, CD_3Cl): δ 165.96, 156.09, 150.43, 137.57, 136.91, 136.72, 135.22, 131.57, 131.17, 130.51, 129.40, 129.30, 128.77, 128.67, 128.59, 128.28, 121.35, 49.07, 46.96; HRMS (ESI) calcd for $C_{24}H_{17}Cl_2N_2O$ [M+H]$^+$: 419.0712, found 419.0715.



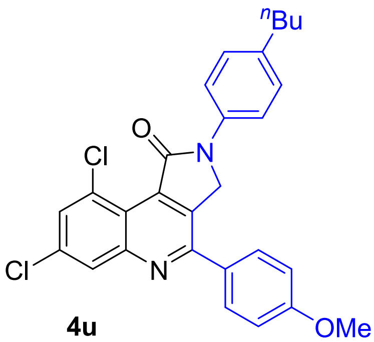
4s

(**4s**): White solid, 71% yield (78 mg, t_1 = 20 h, t_2 = 1 h), mp 197-198°C; ^{1}H NMR (400 MHz, CD_3Cl): δ 8.15 (d, J = 1.6 Hz, 1H), 7.72 (s, 3H), 7.54-7.53 (m, 3H), 7.32-7.24 (m, 5H), 4.38 (s, 2H), 3.88 (t, J = 7.2 Hz, 2H), 3.02 (t, J = 7.2 Hz, 2H); ^{13}C NMR (100 MHz, CD_3Cl): δ 165.84, 156.03, 150.41, 138.91, 137.78, 137.57, 136.78, 135.14, 131.50, 131.16, 130.48, 129.36, 129.12, 129.09, 128.75, 128.64, 127.01, 121.35, 50.19, 45.01, 34.50; HRMS (ESI) calcd for $C_{25}H_{19}Cl_2N_2O$ $[M+H]^+$: 433.0869, found 433.0870.



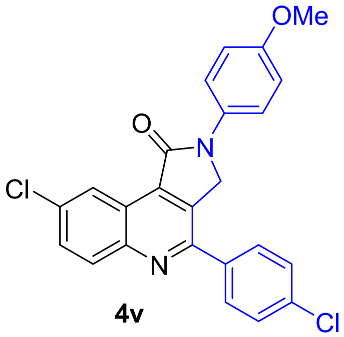
4t

(**4t**): Yellow solid, 57% yield (67 mg, t_1 = 7 h, t_2 = 3 h), mp 255-256°C; ^{1}H NMR (400 MHz, CD_2Cl_2): δ8.16 (s, 1H), 7.92 (d, *J* = 8.4 Hz, 2H), 7.75-7.68 (m, 3H), 7.10 (d, *J* = 8.8 Hz, 2H), 6.98 (d, *J* = 8.8 Hz, 2H), 5.04 (s, 2H), 3.91 (s, 3H), 3.83 (s, 3H); ^{13}C NMR (100 MHz, CD_2Cl_2): δ165.06, 162.62, 158.41, 156.21, 151.22, 138.44, 136.56, 135.66, 132.93, 131.75, 131.63, 131.04, 130.54, 129.22, 123.47, 121.53, 115.33, 115.17, 56.18, 56.14, 51.68; HRMS (ESI) calcd for $C_{25}H_{19}Cl_2N_2O_3$ $[M+H]^+$: 465.0767, found 465.0781.



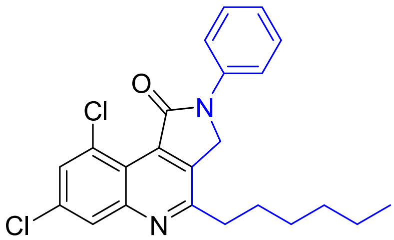
4u

(**4u**): Yellow solid, 59% yield (73 mg, t_1 = 36 h, t_2 = 4.5 h), mp 146-148°C; ^{1}H NMR (400 MHz, CD_3Cl): δ 7.97 (d, *J* = 1.6 Hz, 1H), 7.78 (d, *J* = 8.4 Hz, 2H), 7.58-7.55 (m, 3H), 7.08 (d, *J* = 8.4 Hz, 2H), 6.99 (d, *J* = 8.4 Hz, 2H), 4.81 (s, 2H), 3.84 (s, 3H), 2.51 (t, *J* = 7.6 Hz, 2H), 1.57-1.48 (m, 2H), 1.37-1.28 (m, 2H), 0.92 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, CD_3Cl): δ164.25, 161.70, 154.92, 150.18, 140.27, 137.40, 136.48, 135.33, 134.94, 131.08, 130.76, 130.17, 129.60, 129.19, 128.46, 120.52, 120.23, 114.61, 55.39, 50.29, 34.84, 33.34, 22.15, 13.74; HRMS (ESI) calcd for $C_{28}H_{25}Cl_2N_2O_2$ $[M+H]^+$: 491.1288, found 491.1288.



4v

(**4v**): Yellow solid, 73% yield (80 mg, t_1 = 5.5 h, t_2 = 0.5 h), mp 225-227°C; ^{1}H NMR (400 MHz, CD_3Cl): δ9.14 (s, 1H), 8.13 (d, *J* = 9.2 Hz, 1H), 7.88 (d, *J* = 8.4 Hz, 2H), 7.74-7.69 (m, 3H), 7.55 (d, *J* = 8.4 Hz, 2H), 6.93 (d, *J* = 9.2 Hz, 2H), 5.01 (s, 2H), 3.82 (s, 3H); ^{13}C NMR (100 MHz, CD_3Cl): δ166.53, 157.76, 152.67, 147.20, 136.82, 136.61, 136.00, 135.09, 133.13, 131.92, 131.83, 131.47, 129.94, 129.74, 123.51, 122.93, 122.17, 114.77, 55.54, 51.11; HRMS (ESI) calcd for $C_{24}H_{17}Cl_2N_2O_2$ [M+H]+: 435.0662, found 435.0672.



4w

4w): White solid, 49% yield (51 mg, t_1 = 24 h, t_2 = 6 h), mp 106-108 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.08 (d, *J* = 2.0 Hz, 1H), 7.83 (d, *J* = 8.0 Hz, 2H), 7.70 (d, *J* = 2.0 Hz, 1H), 7.47-7.36 (m, 2H), 7.25-7.15 (m, 1H), 4.90 (s, 2H), 2.99 (t, *J* = 7.6 Hz, 2H), 1.96-1.80 (m, 2H), 1.51-1.41 (m, 2H), 1.40-1.30 (m, 4H), 0.91 (t, *J* = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 164.94, 159.58, 150.66, 139.25, 136.51, 136.26, 134.96, 131.16, 131.02, 129.60, 128.44, 125.67, 121.05, 120.73, 48.88, 34.92, 31.60, 29.23, 27.88, 22.45, 13.93; HRMS (ESI) calcd for $C_{23}H_{22}Cl_2N_2NaO$ [M+Na]$^+$: 435.1001, found 435.0999.

References:

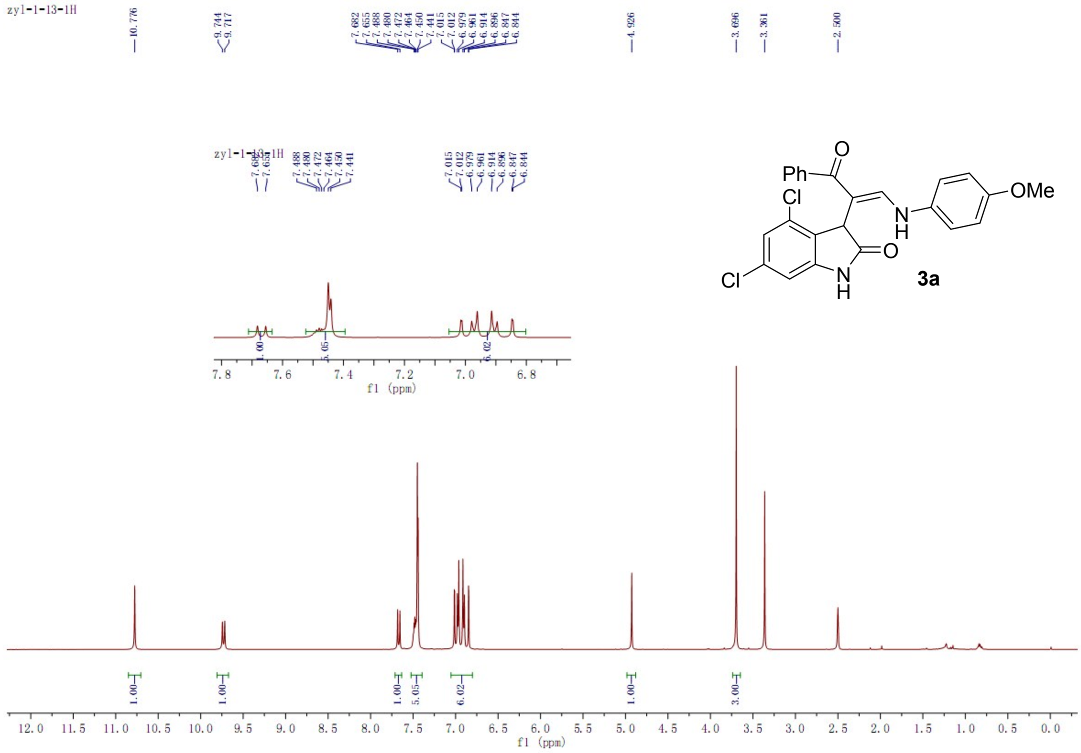
[1] P. Braunstein, H. Lehner, D. Matt, *Inorg. Synth.* **1990**, *27*, 218.

[2] N. Mé zailles, L. Ricard, F. Gagosz, *Org. Lett.* **2005**, *7*, 4133.

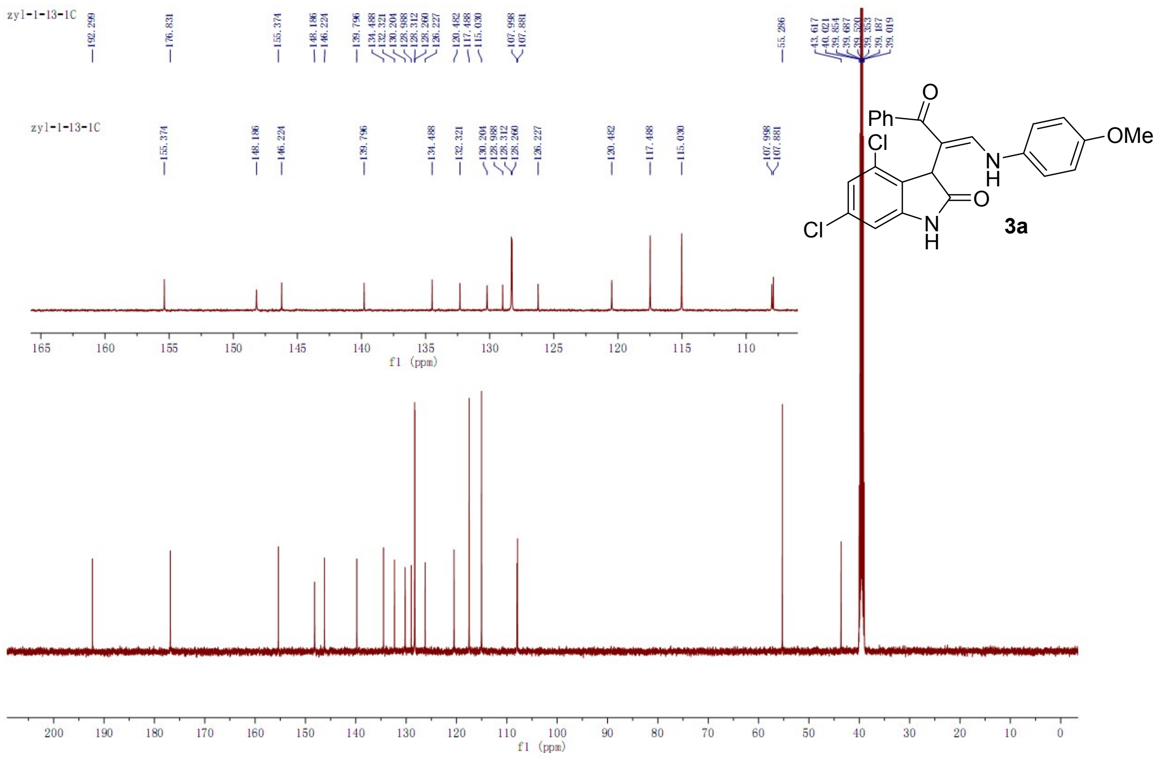
[3] R. Augusti, C. Kascheres, *J. Org. Chem.* **1993**, *58*, 7079.

[4] J. Yang, C. Wang, X. Xie, H. Li, Y. Li, *E. J. Org. Chem.***2010**, 4189.

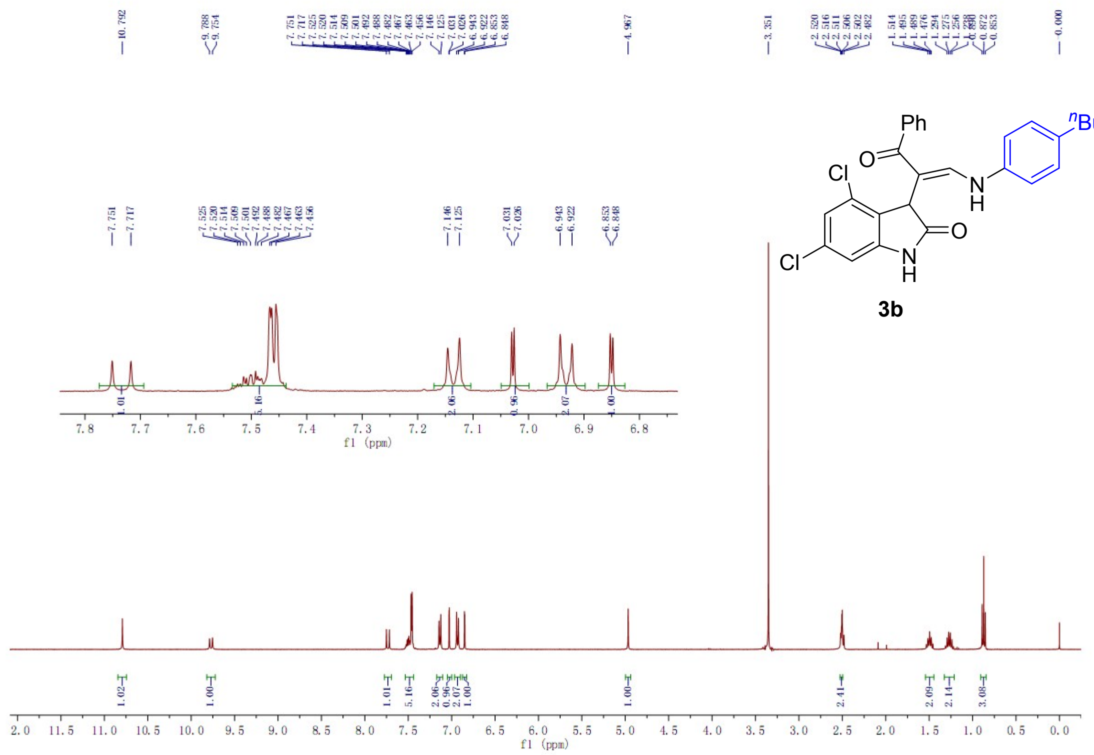
zyl-1-13-h



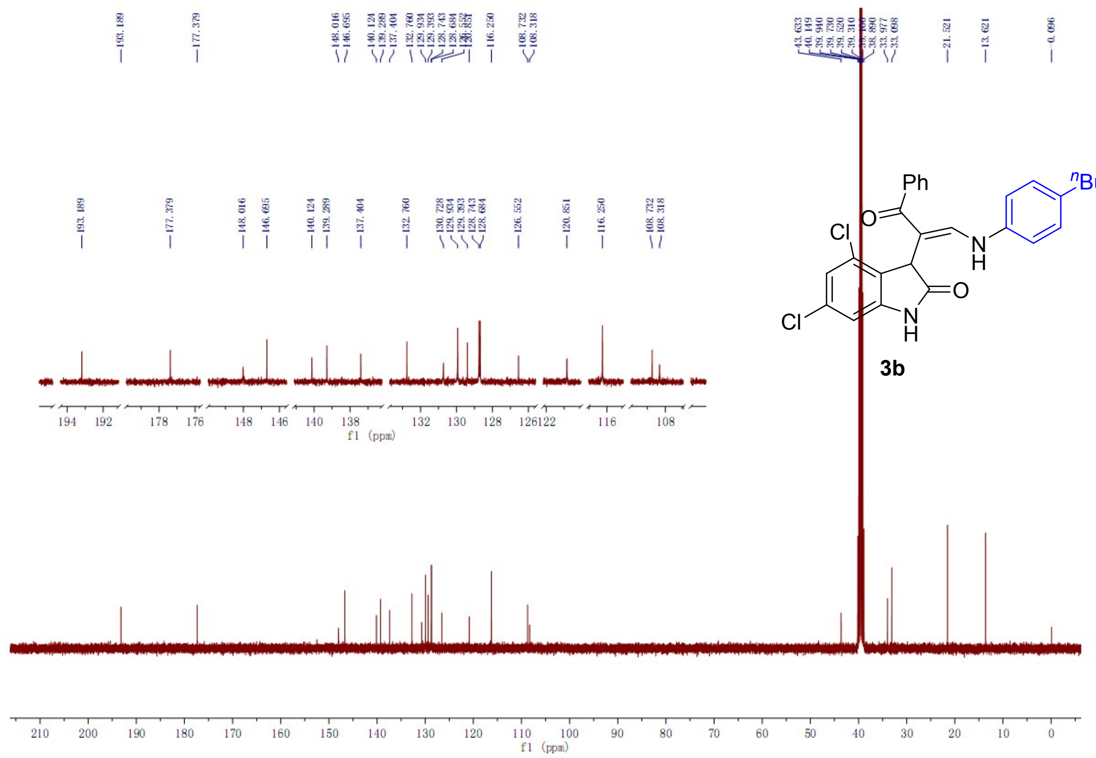
zyl-1-13-c



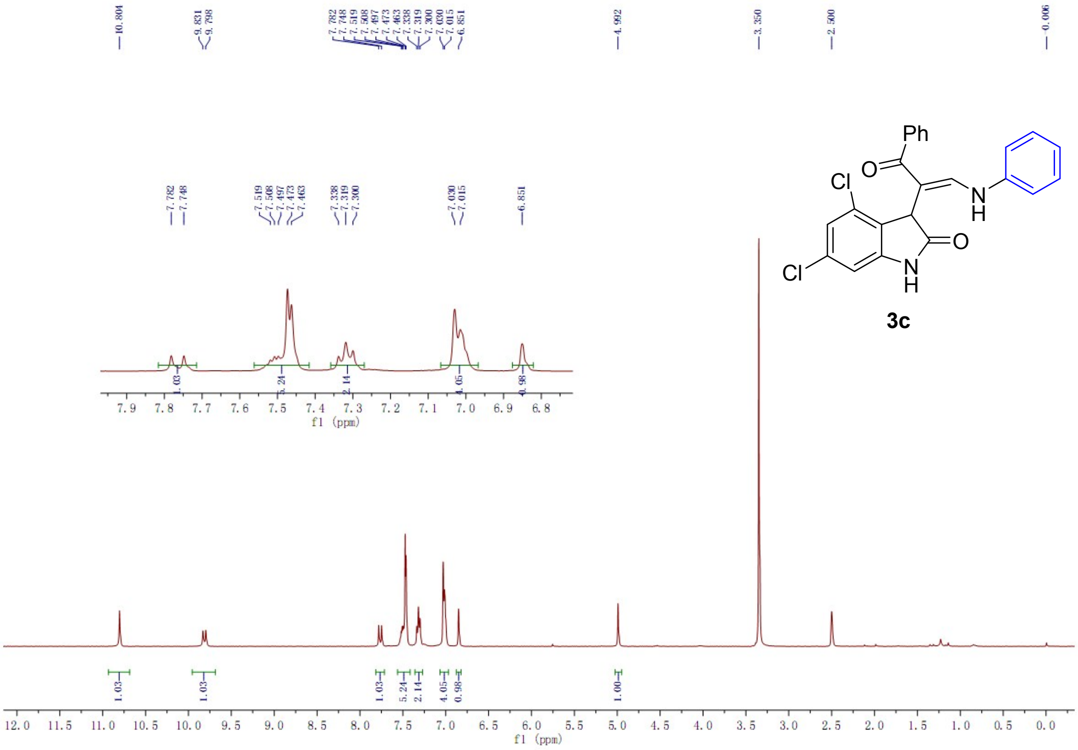
Zyl-3-90-h



Zyl-3-90-c



Zyl-3-64-h



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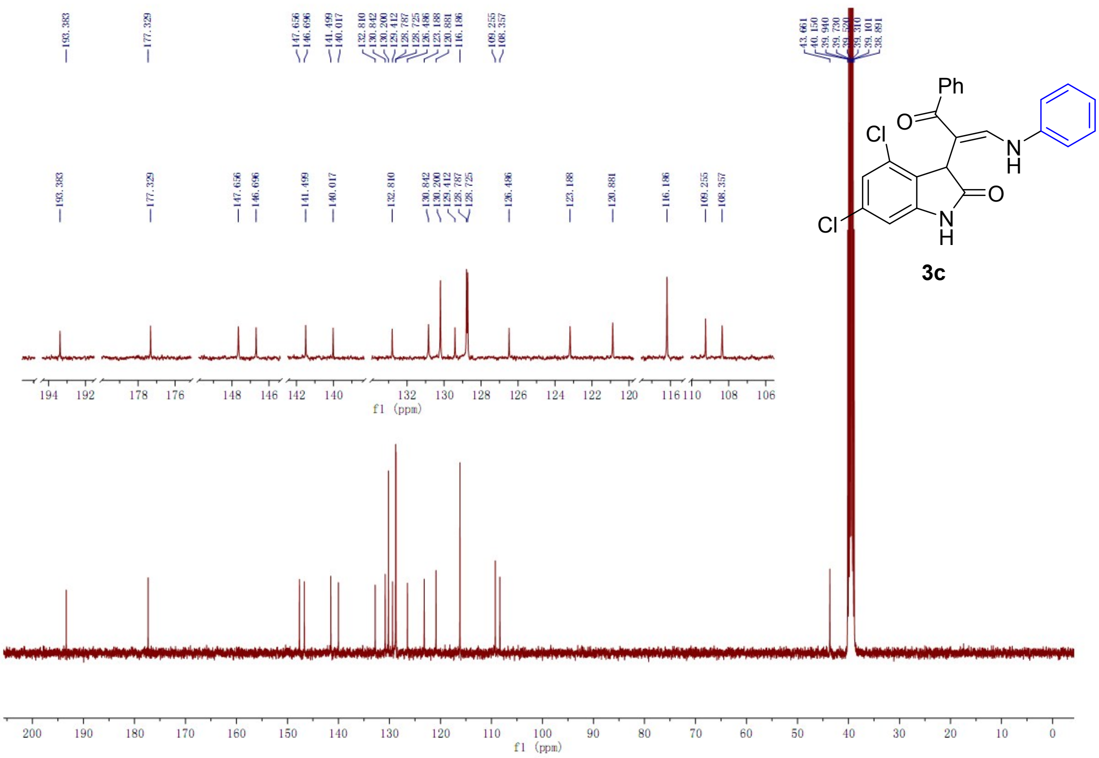
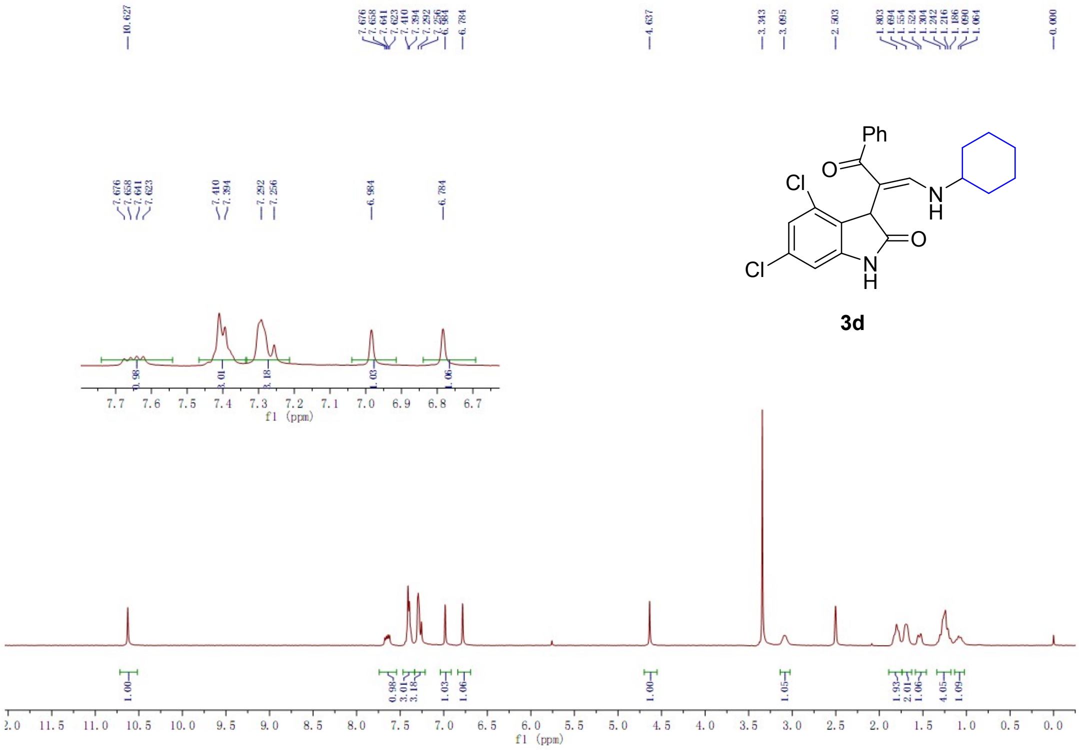


Zyl-3-59-h



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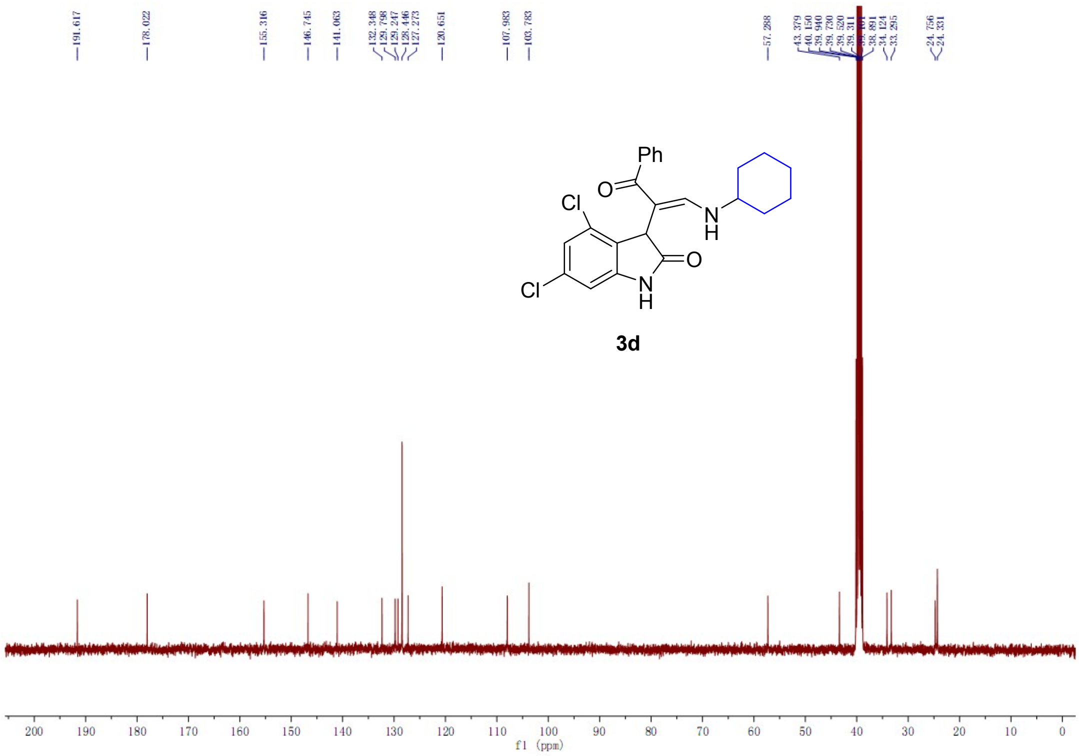
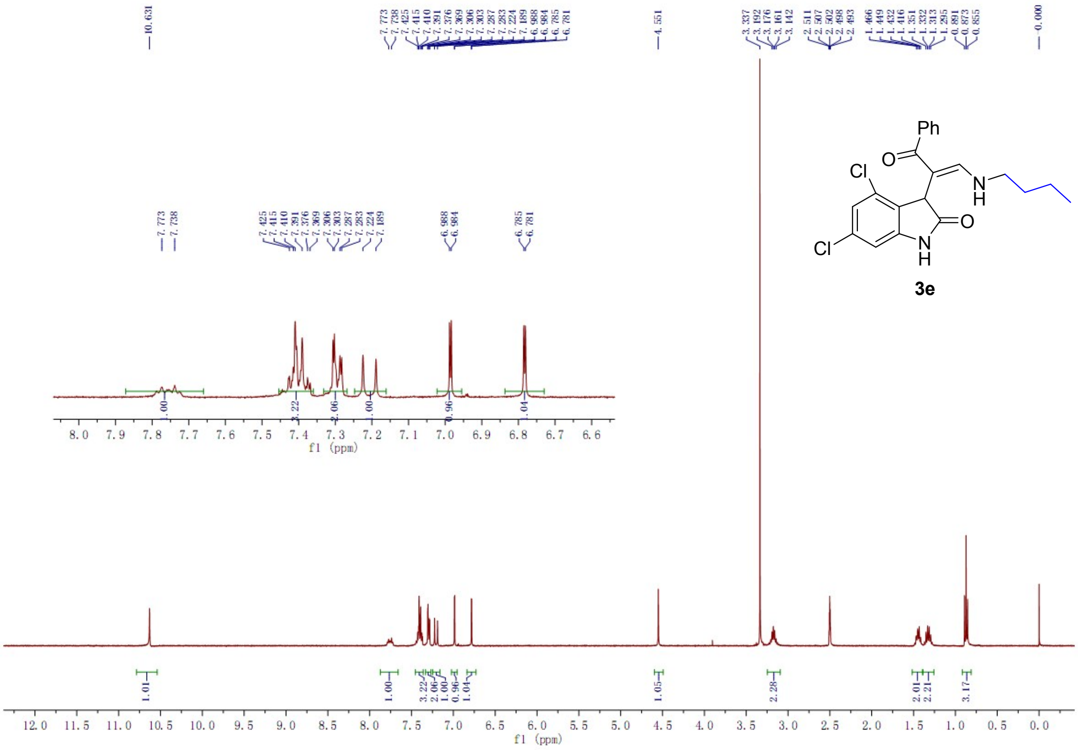


Zyl-3-86-h



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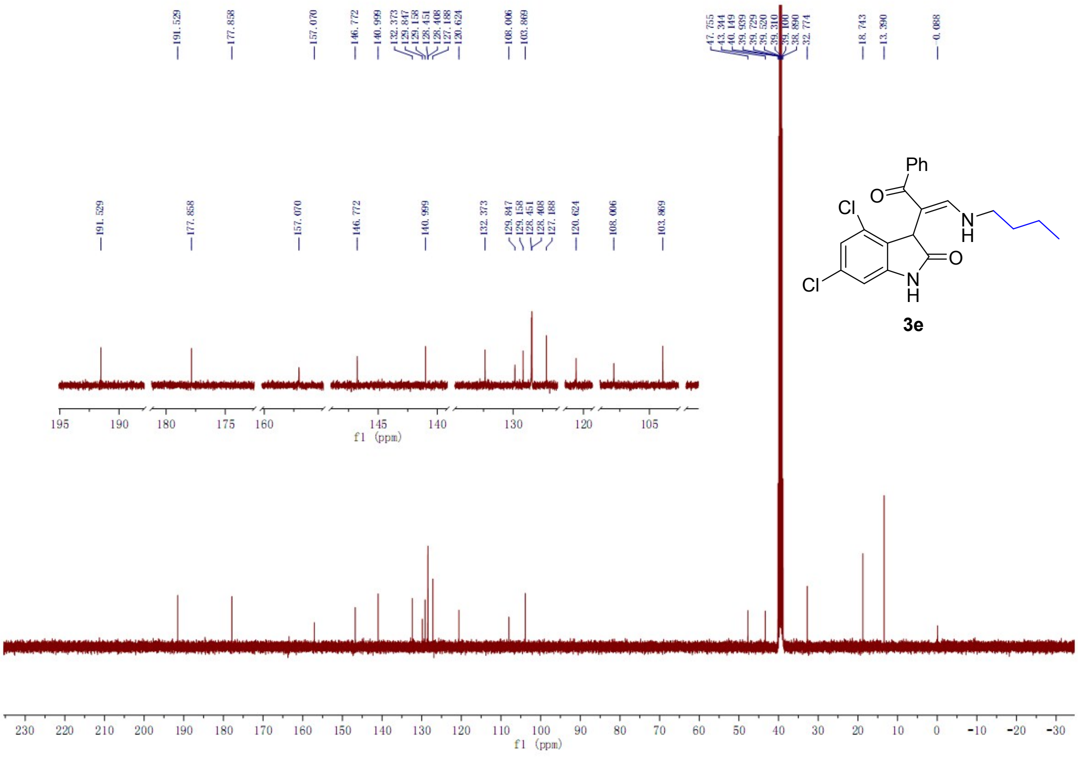
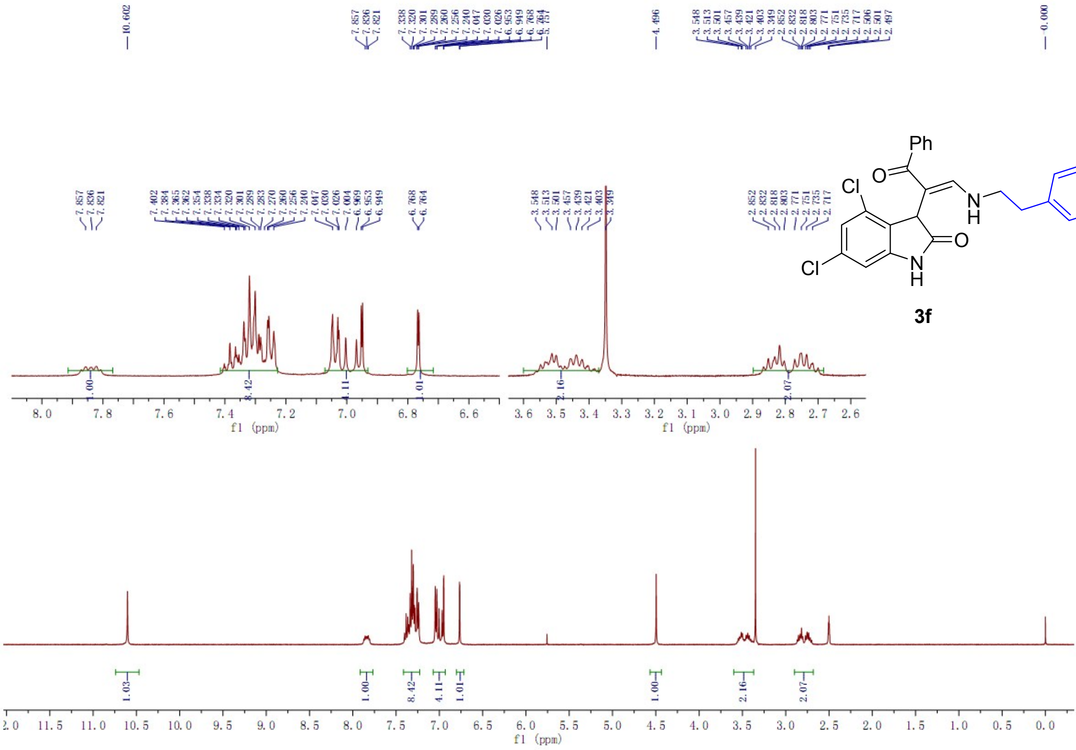
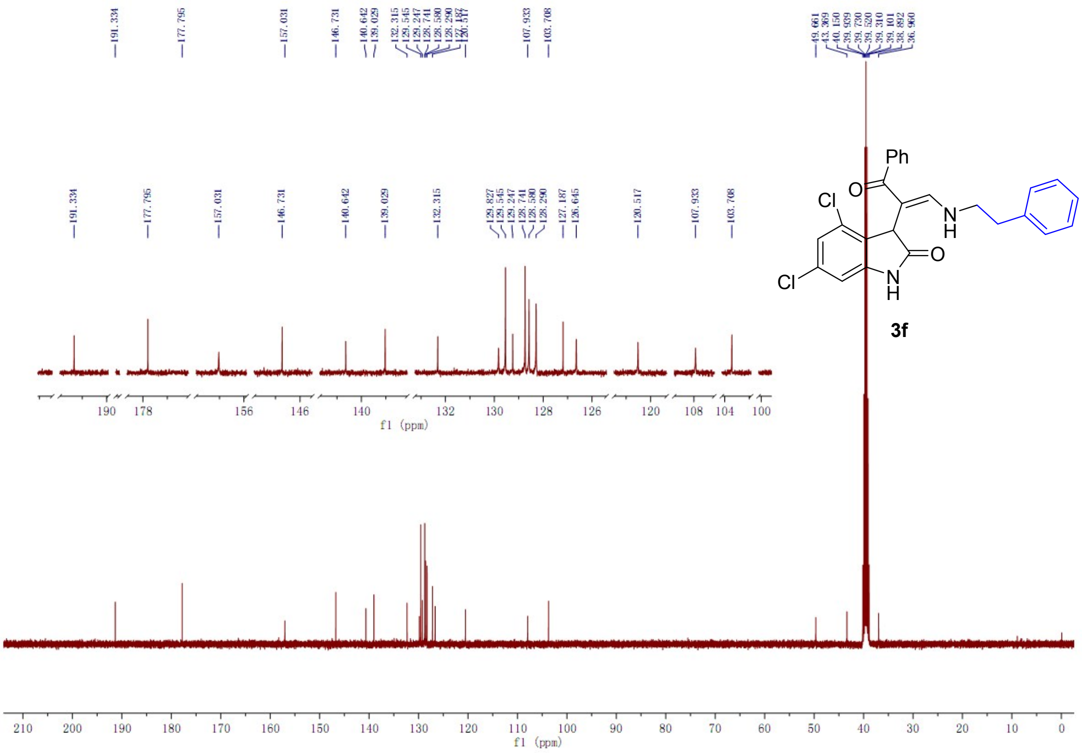


Zyl-3-87-h



Zyl-3-87-c



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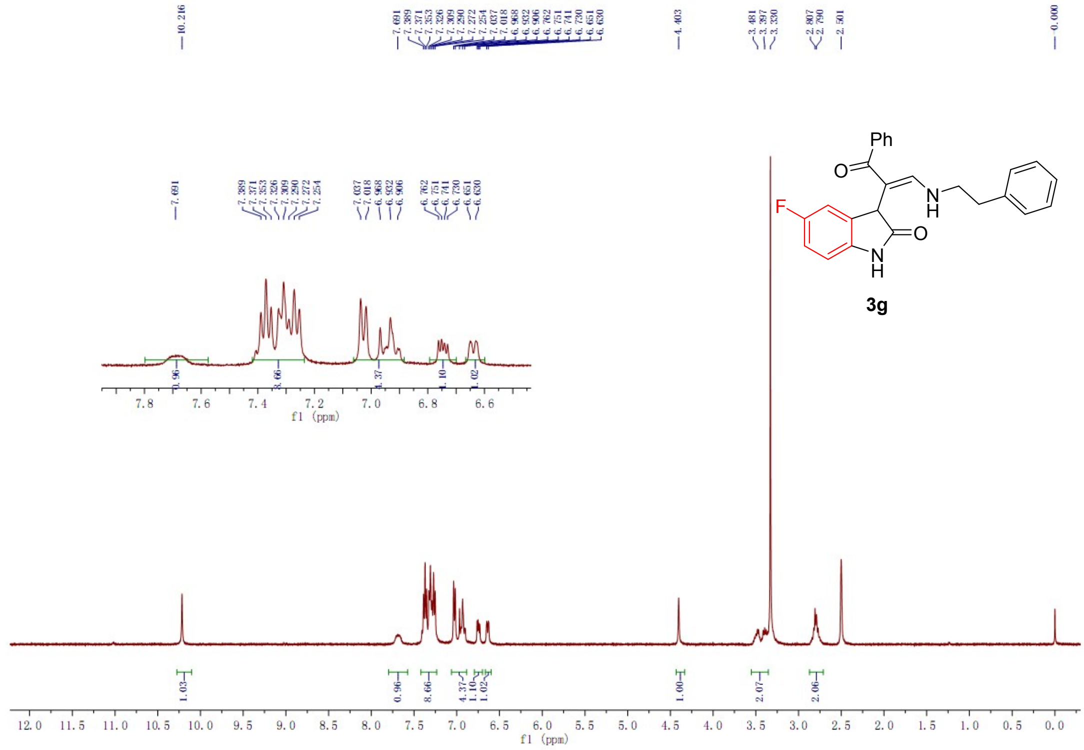

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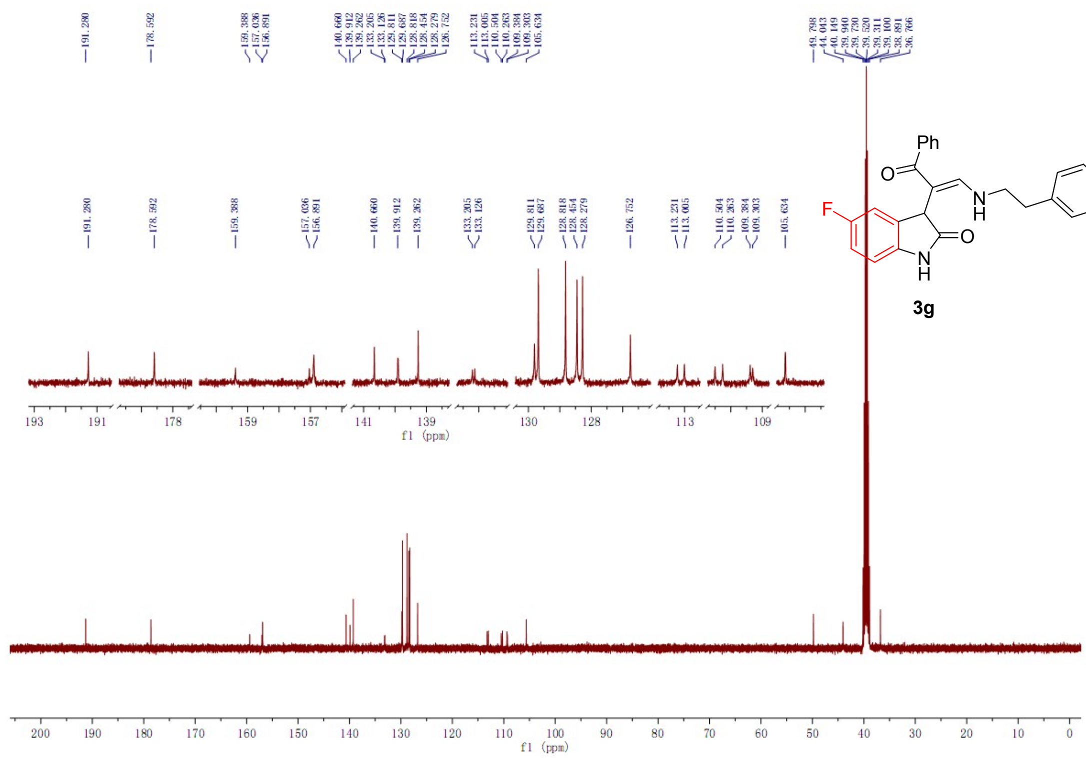

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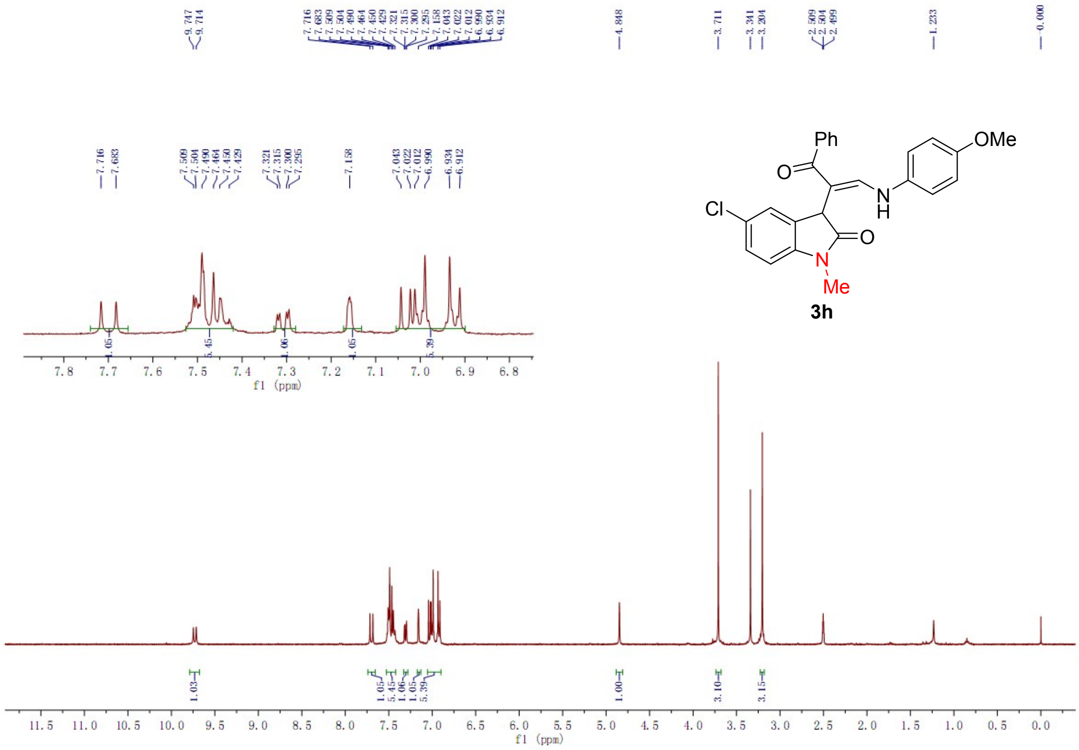
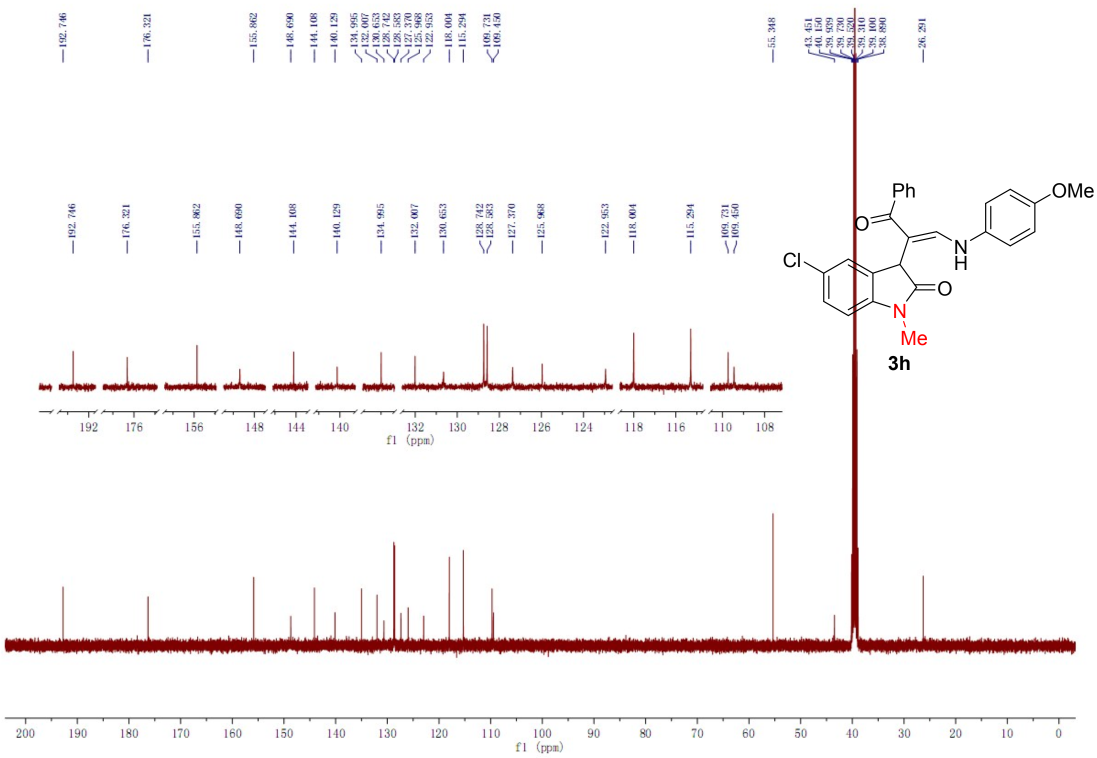


Zyl-3-96-c



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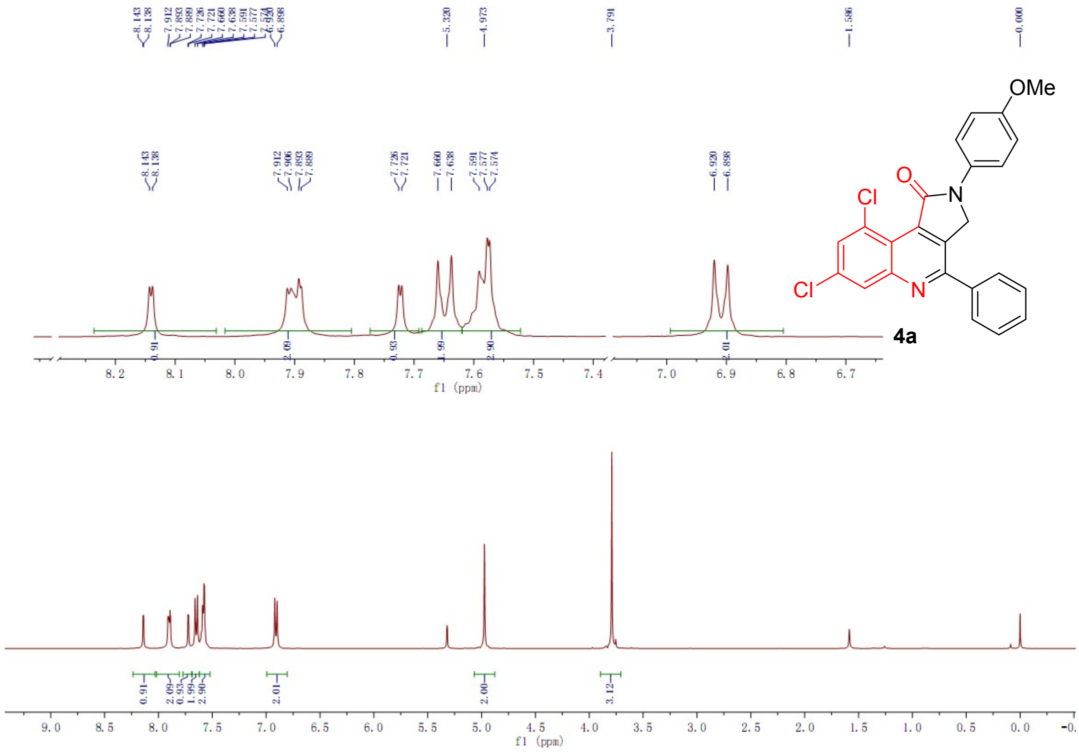

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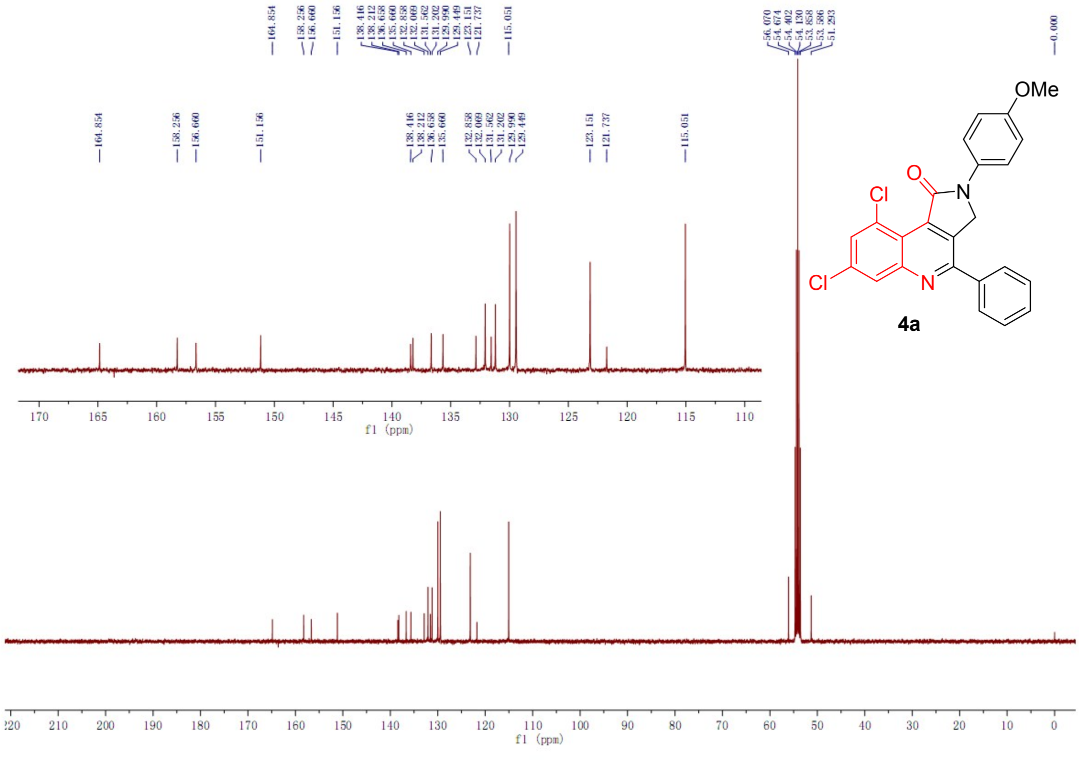

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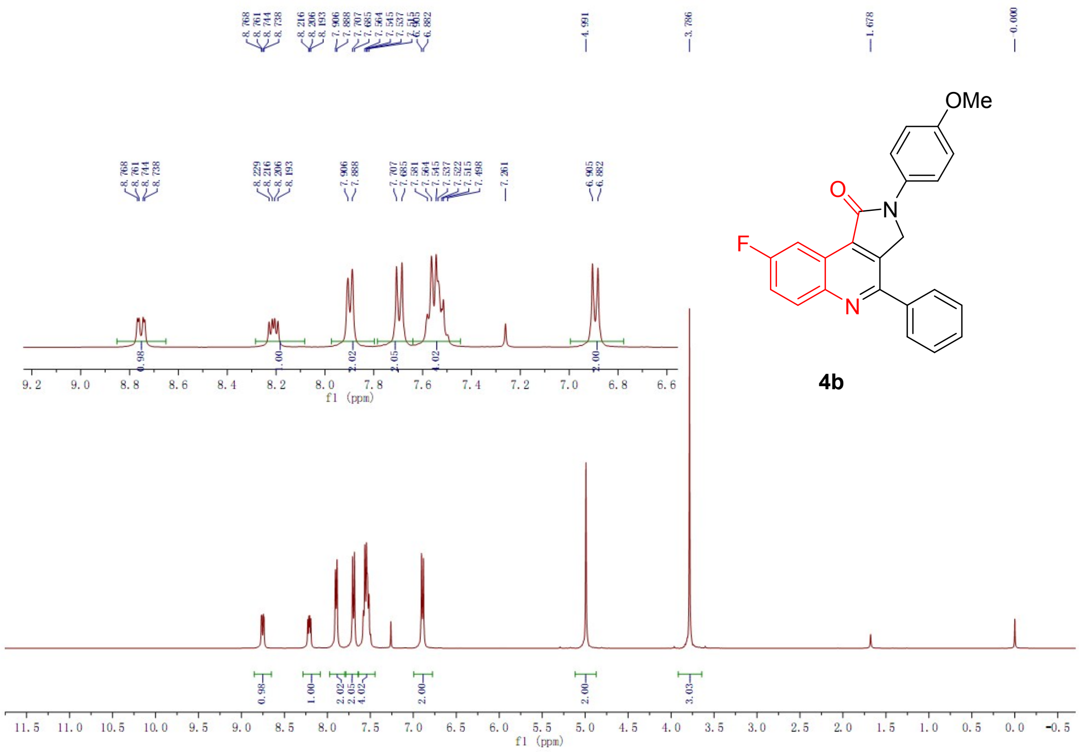

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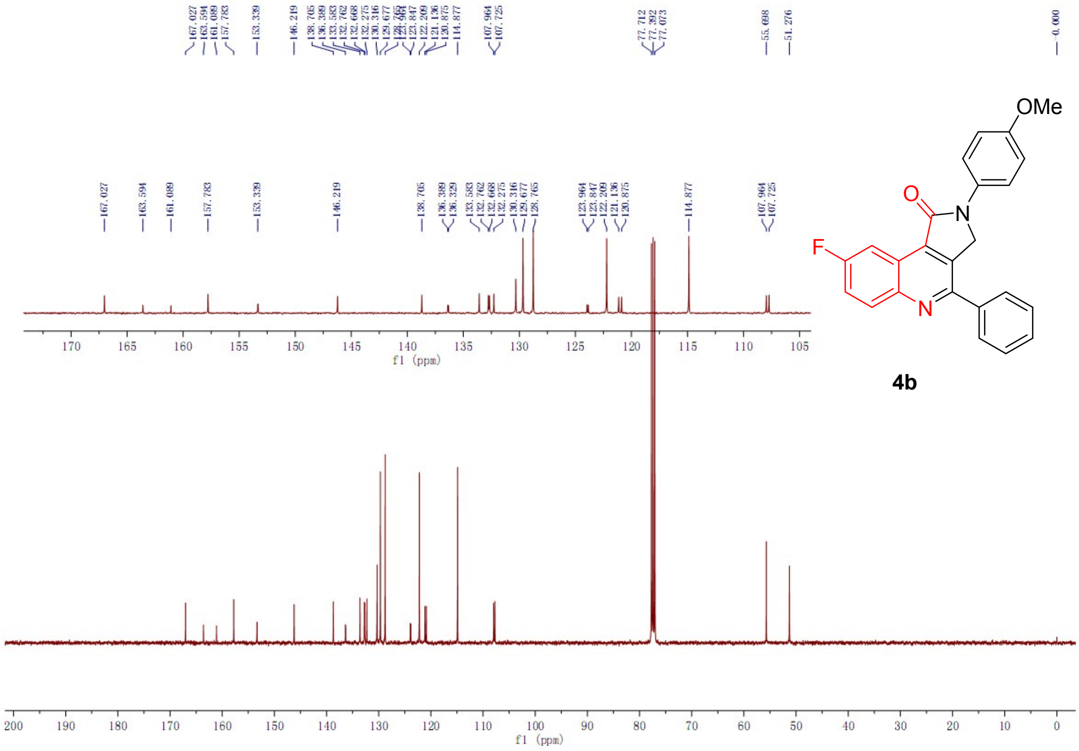

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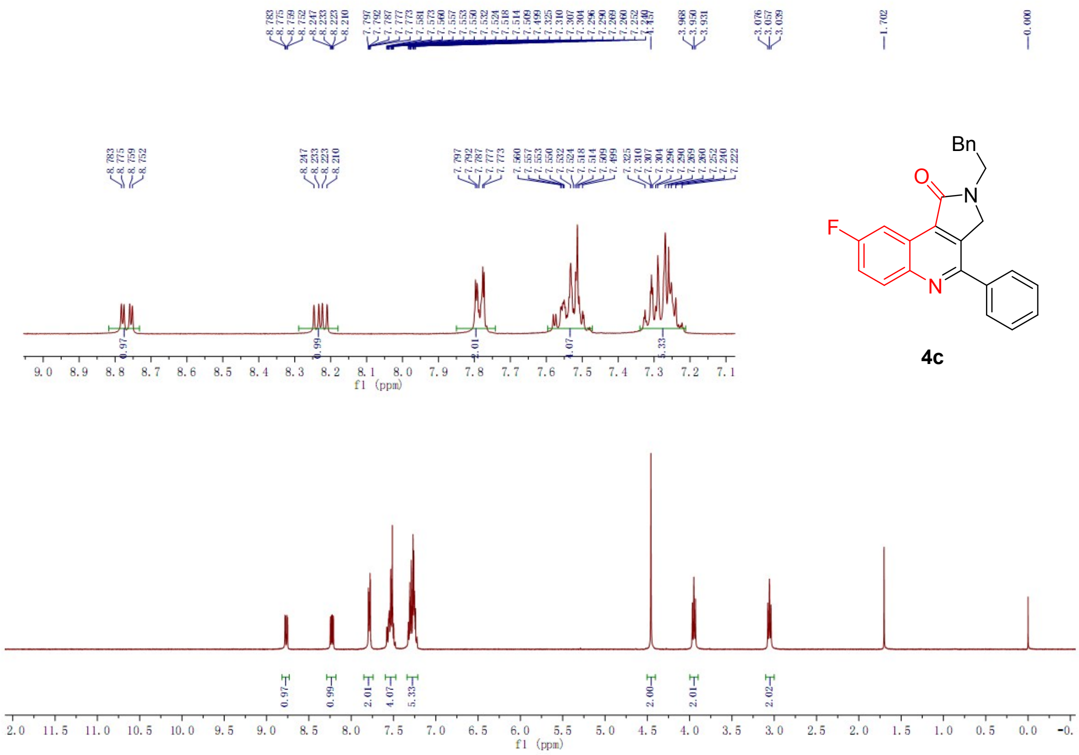

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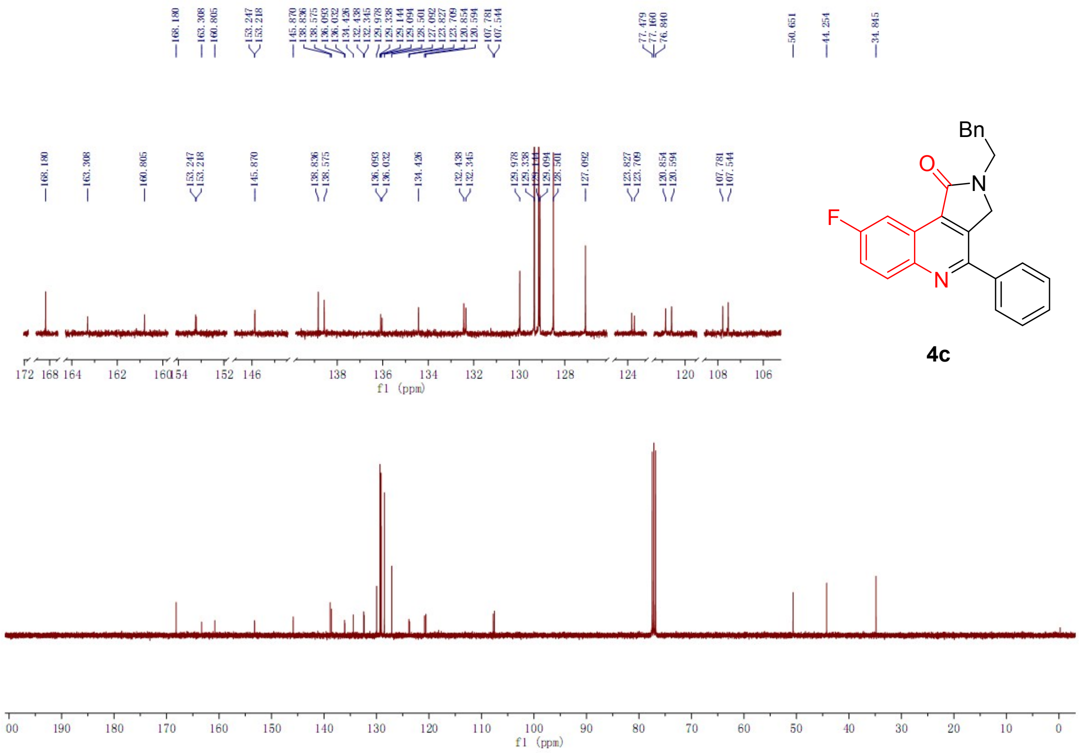

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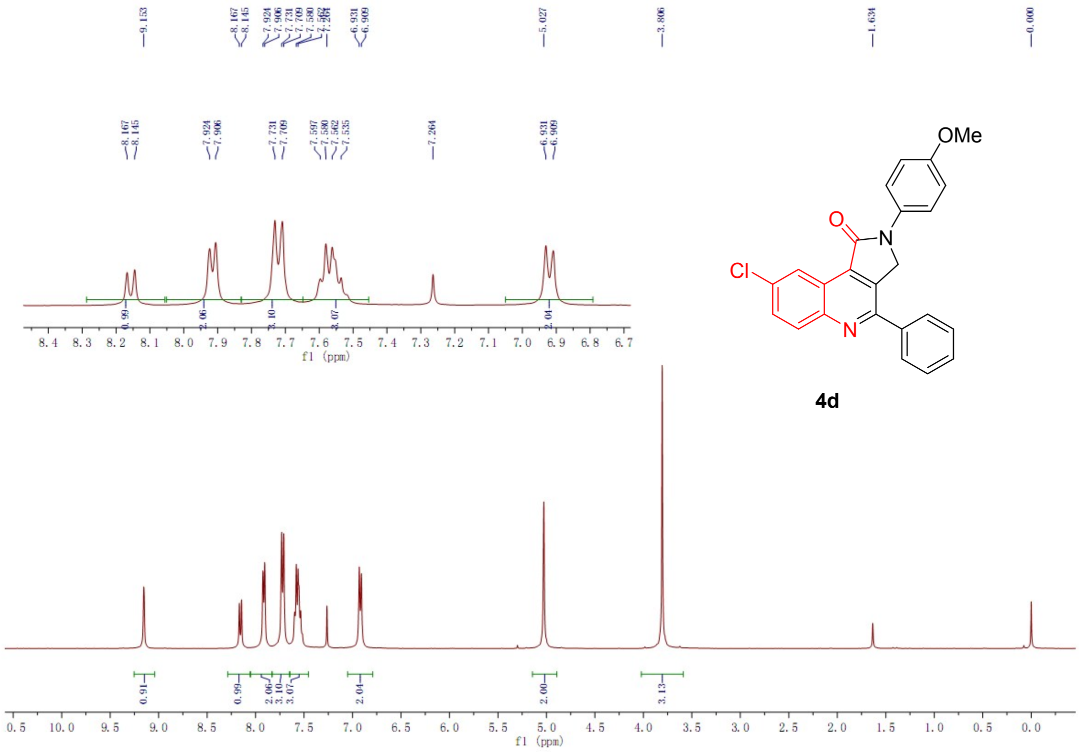

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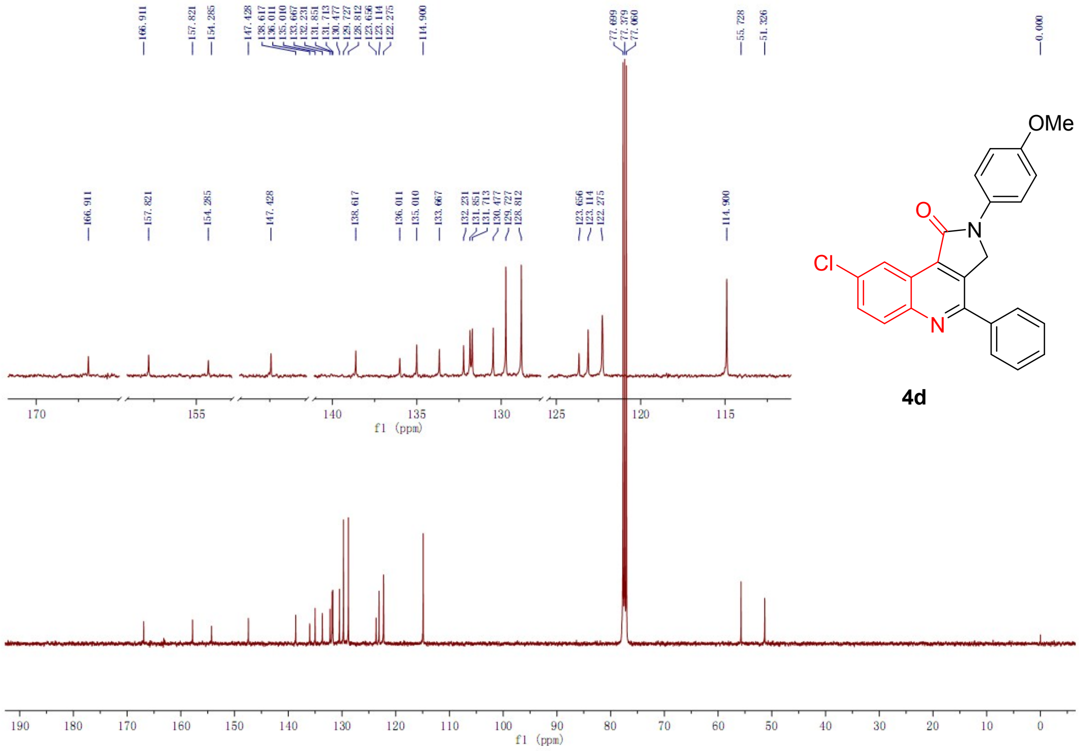
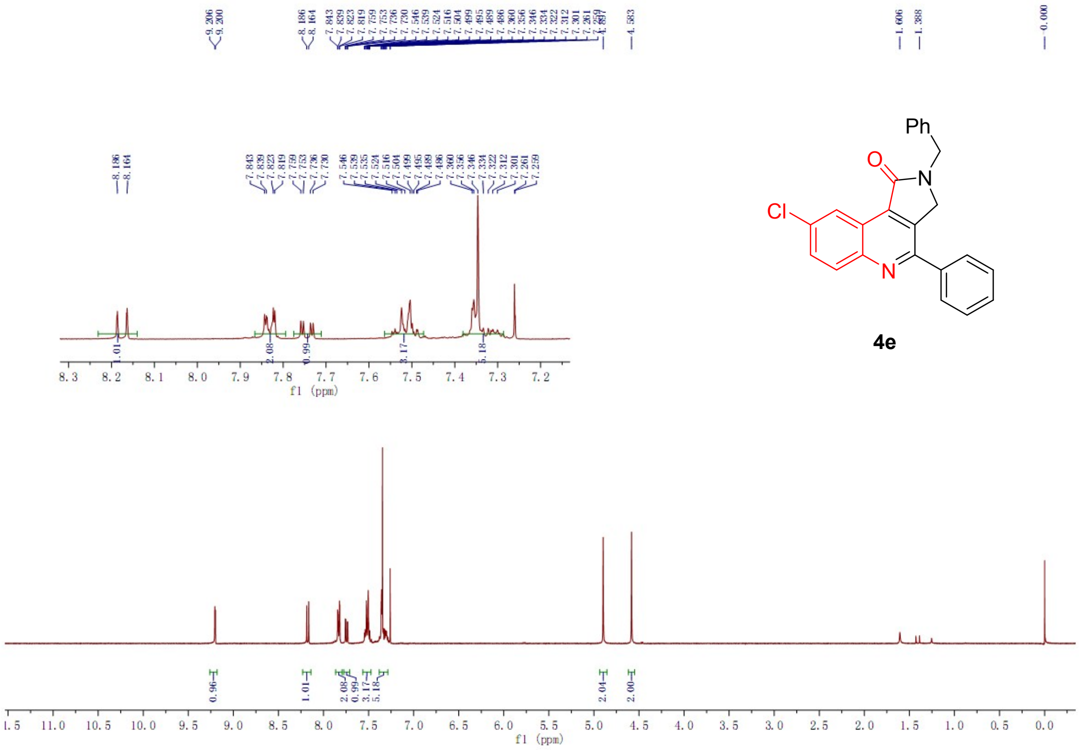
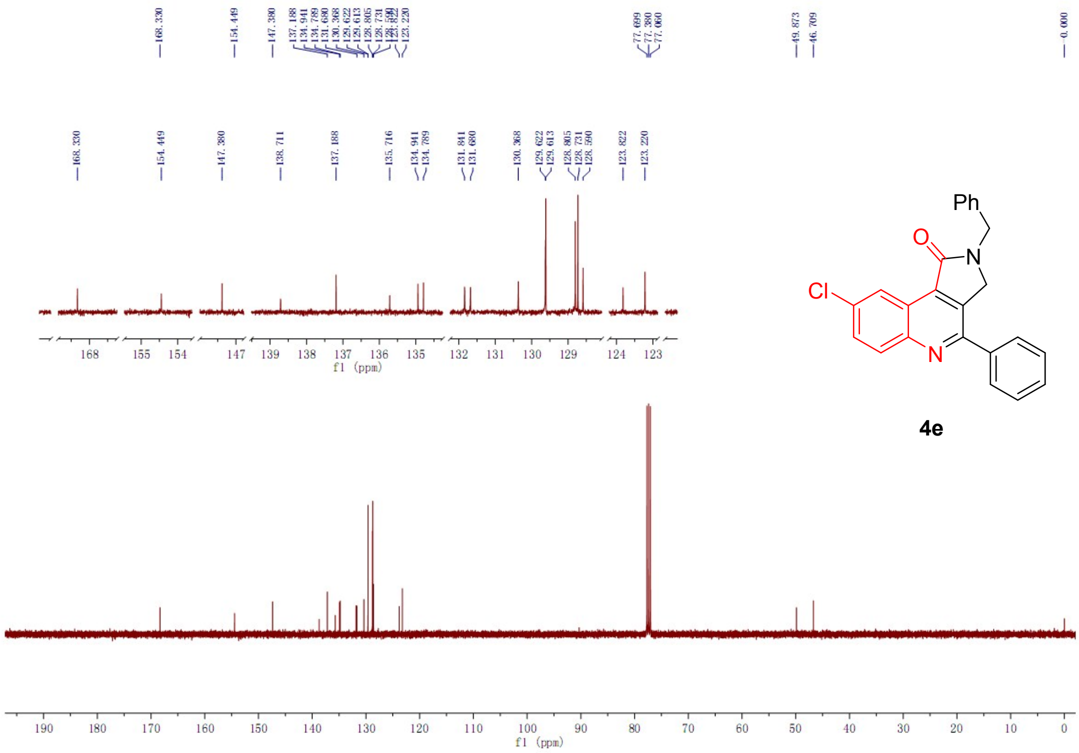


Zyl-3-91-h



Zyl-3-91-c



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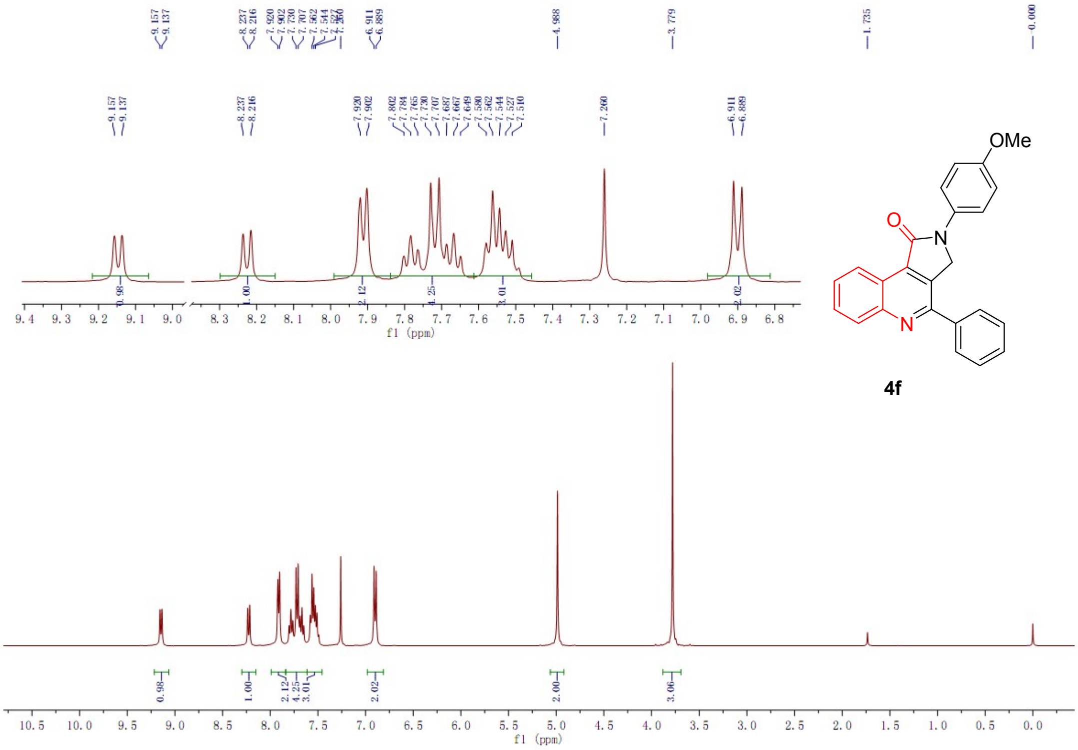

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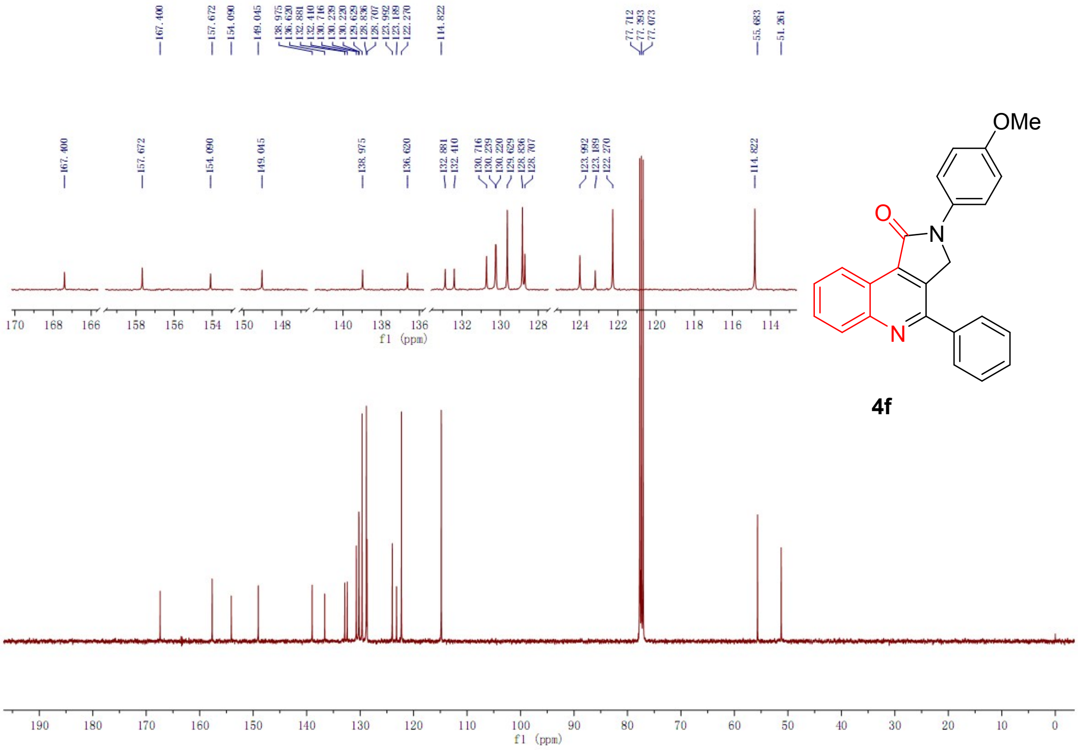

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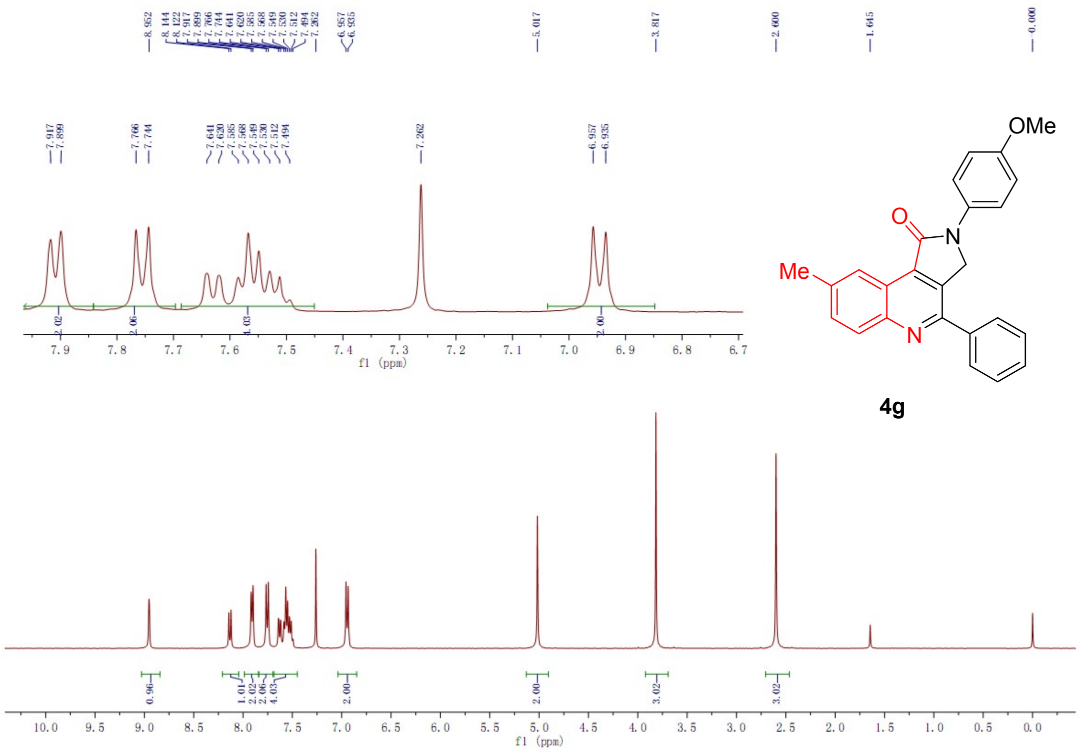

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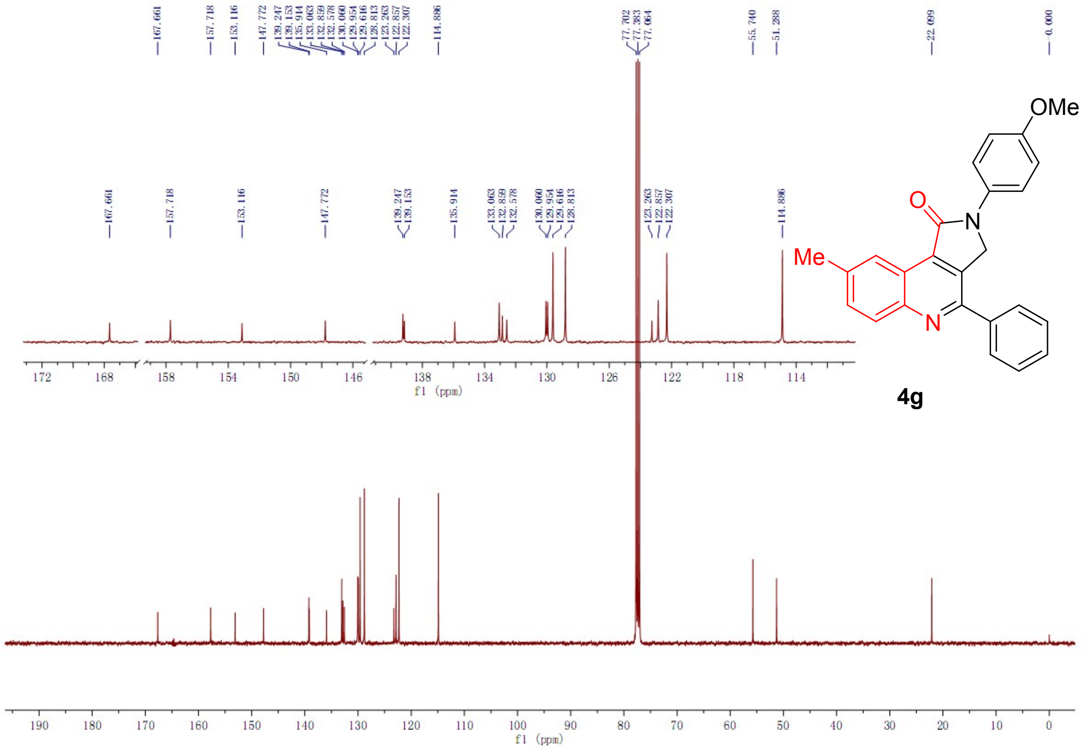
zyl-1-110-c



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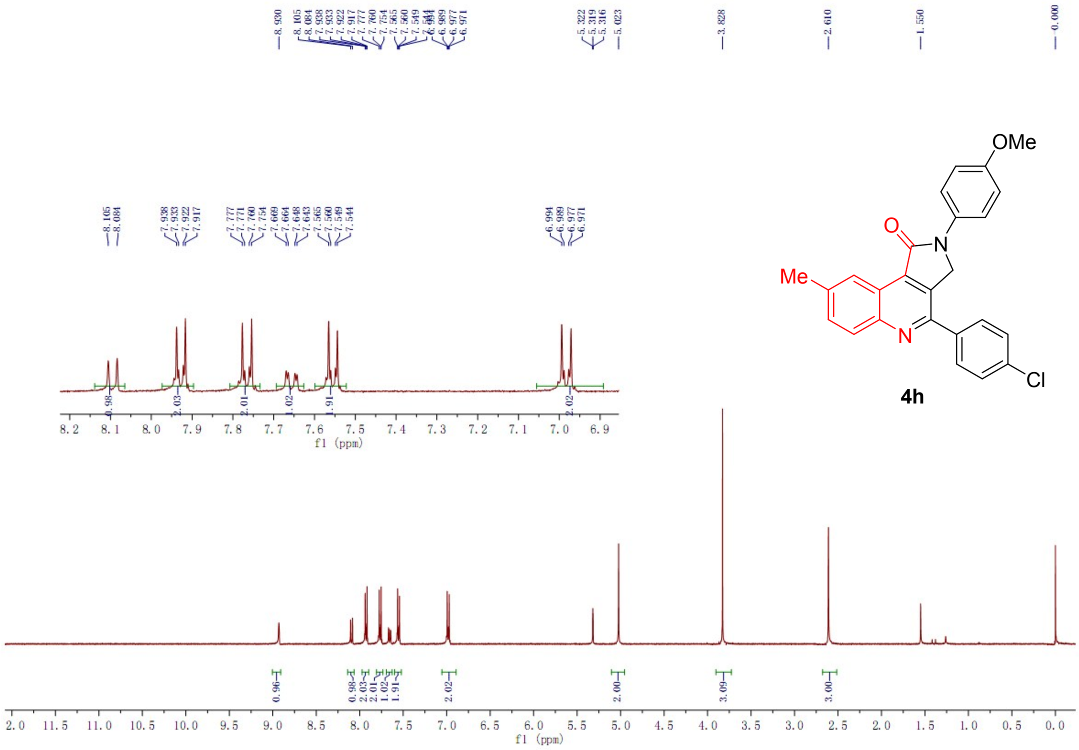
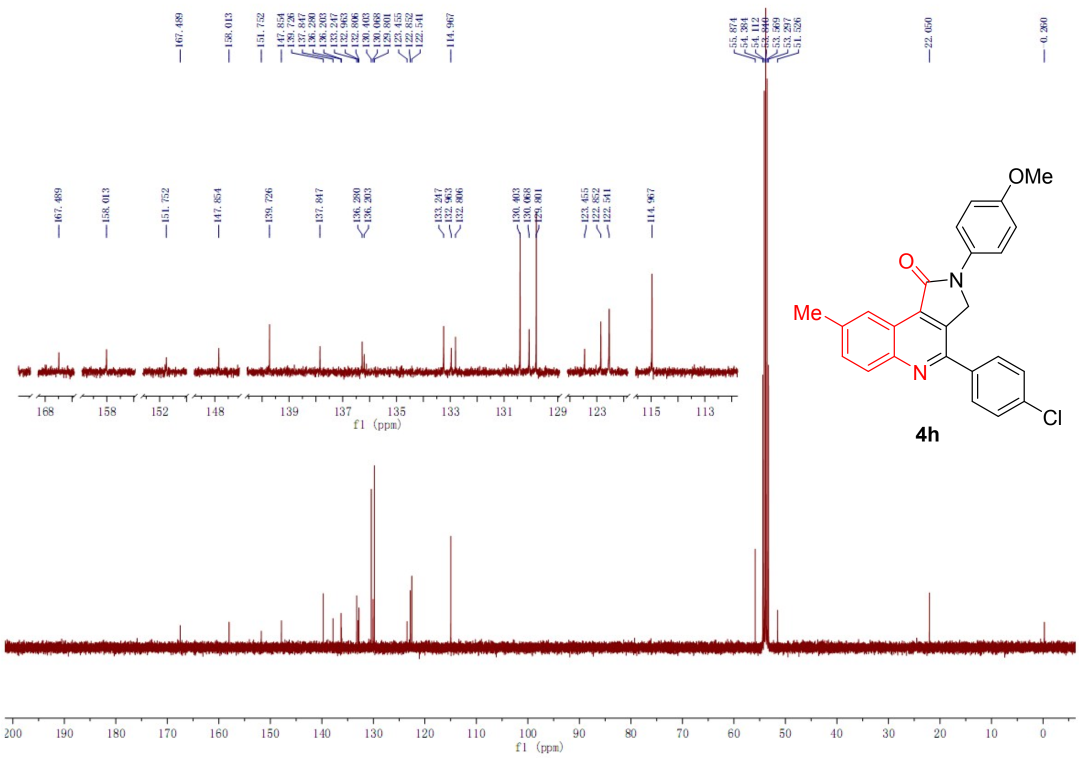
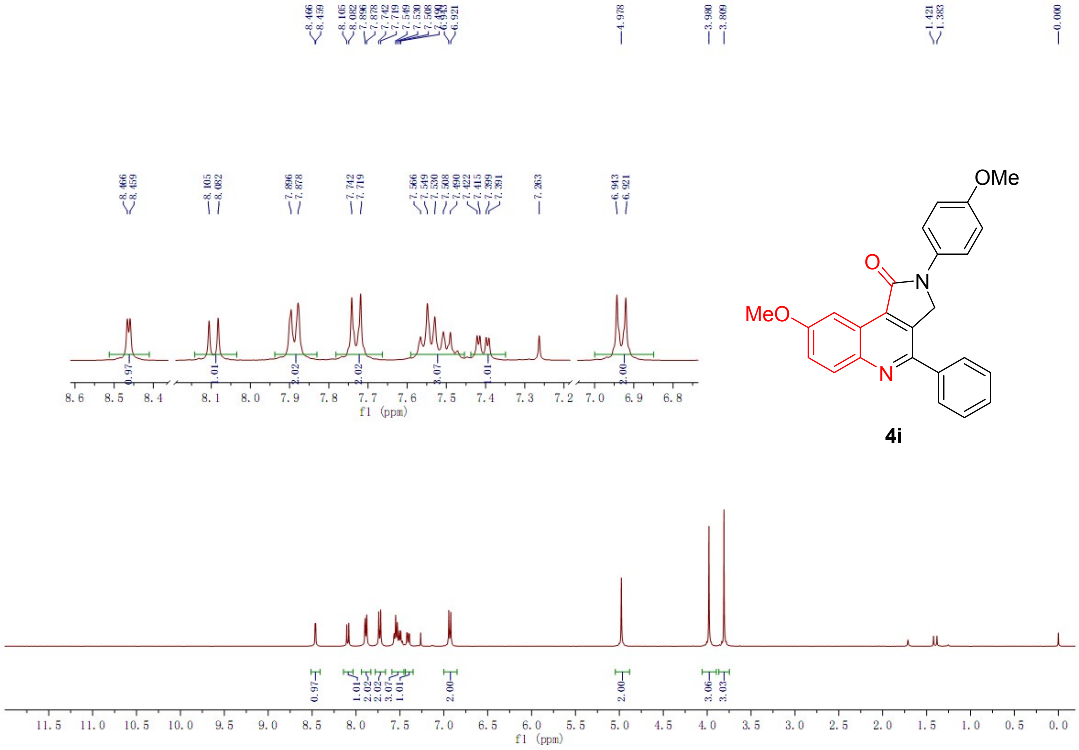


Zyl-3-92-c



Zyl-4-125-h



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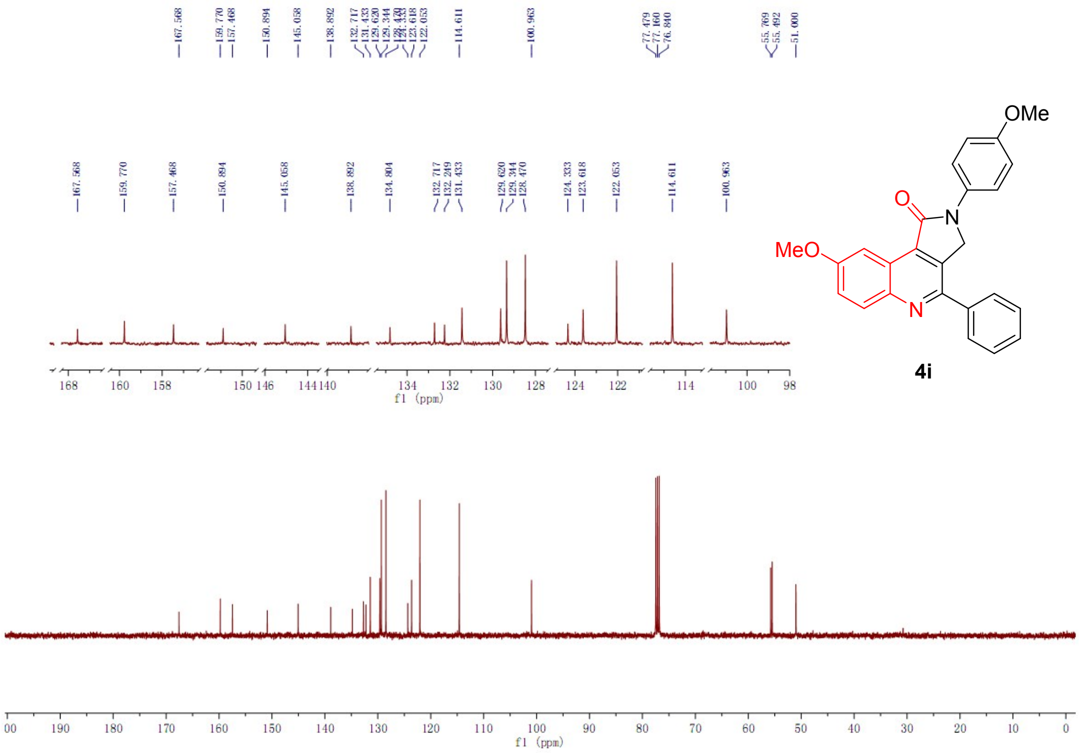
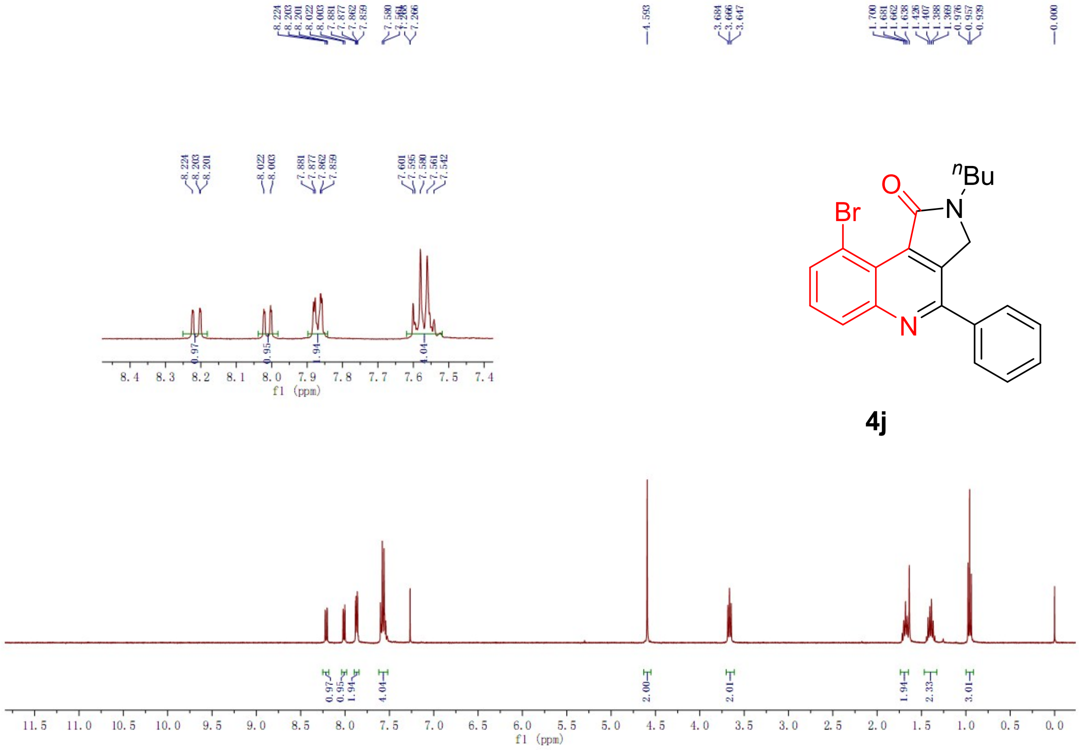
Zyl-4-125-c



4i

Zyl-3-89-h



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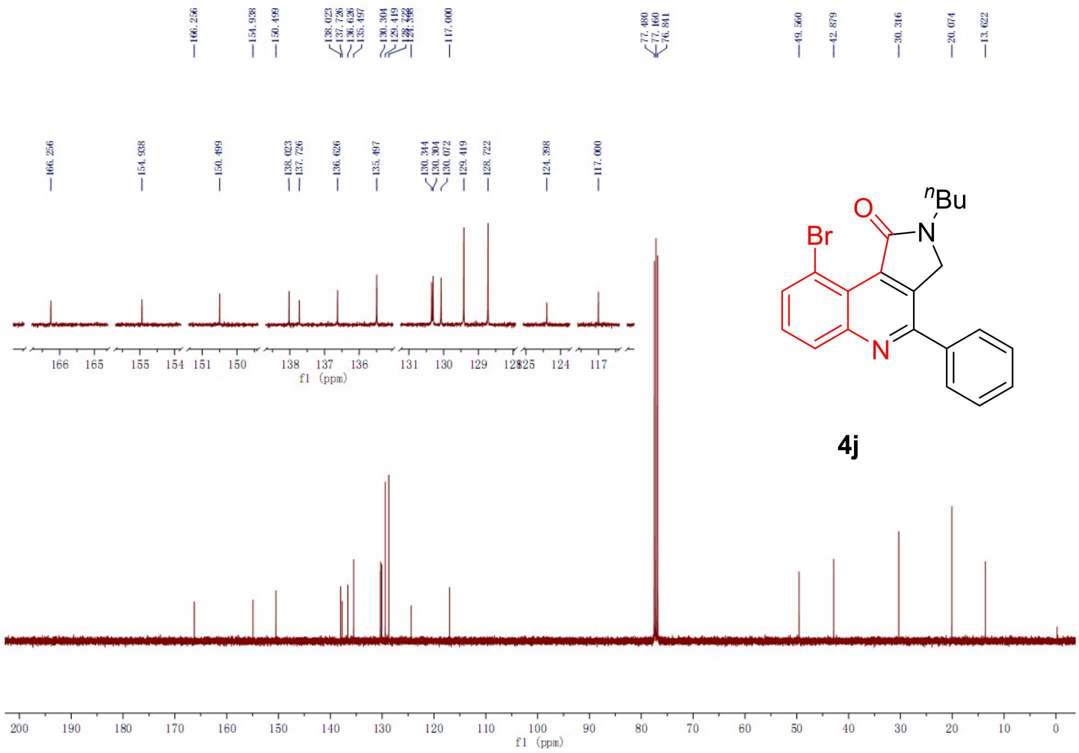

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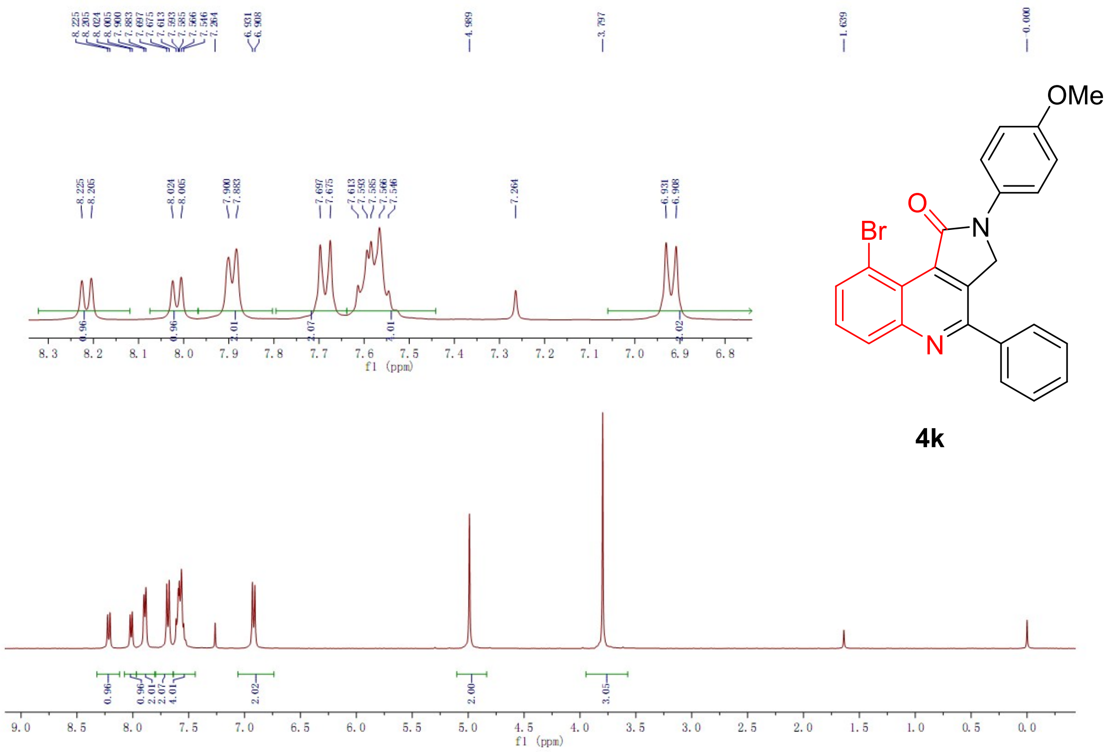

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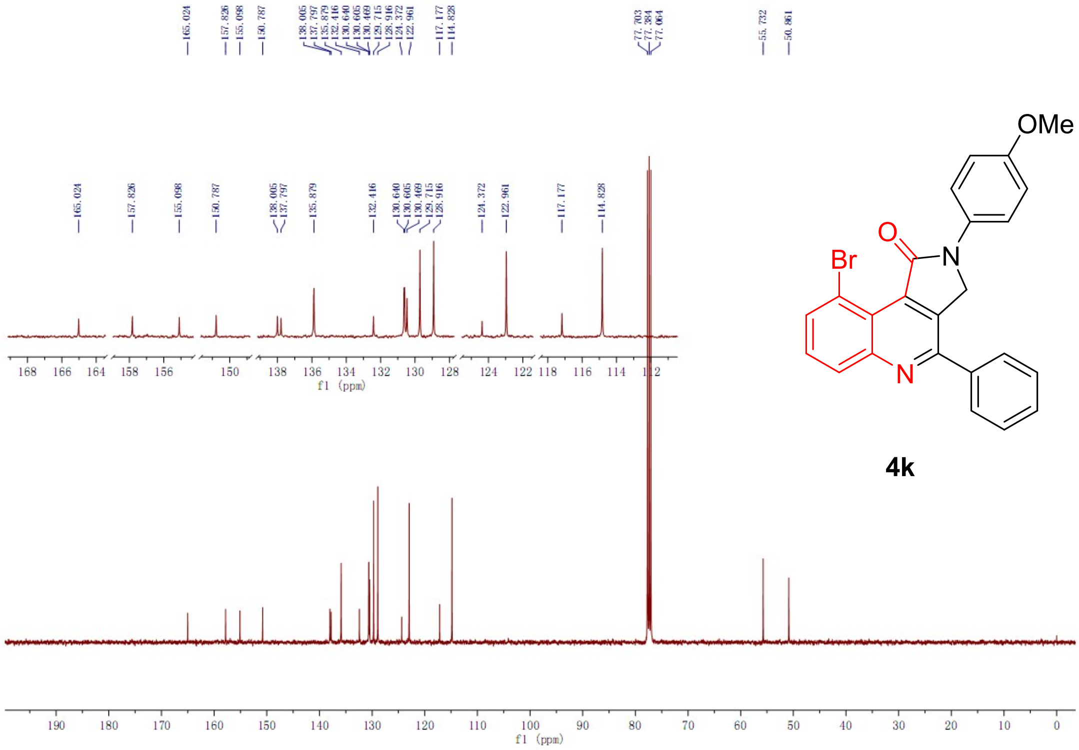

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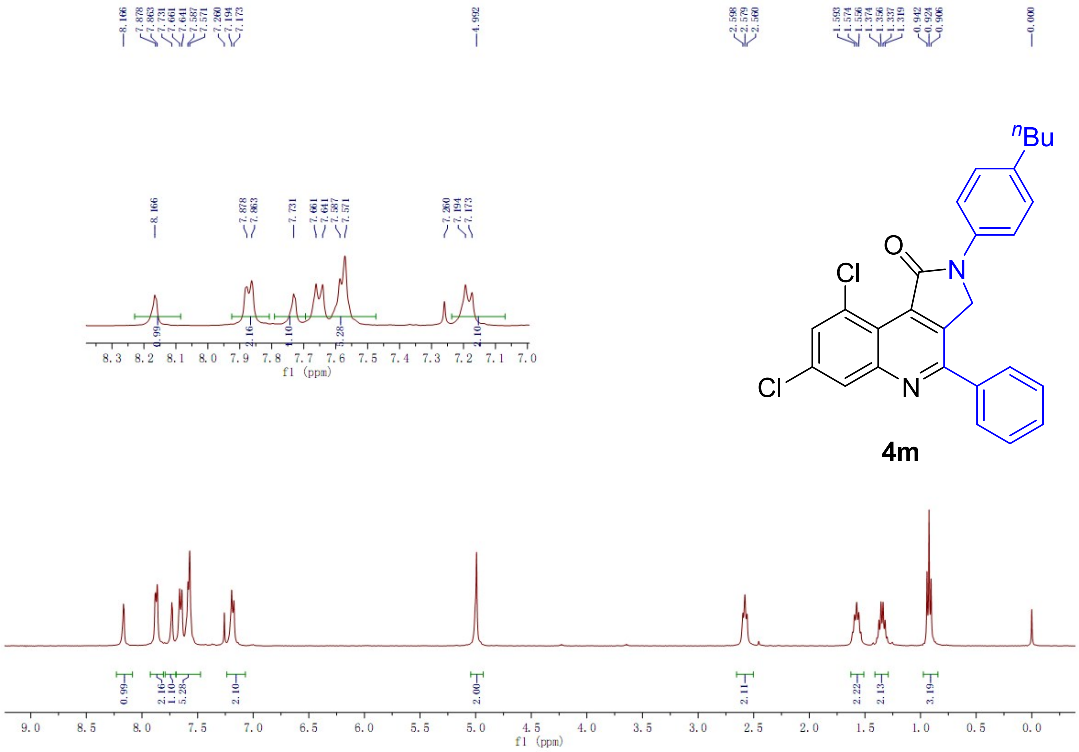
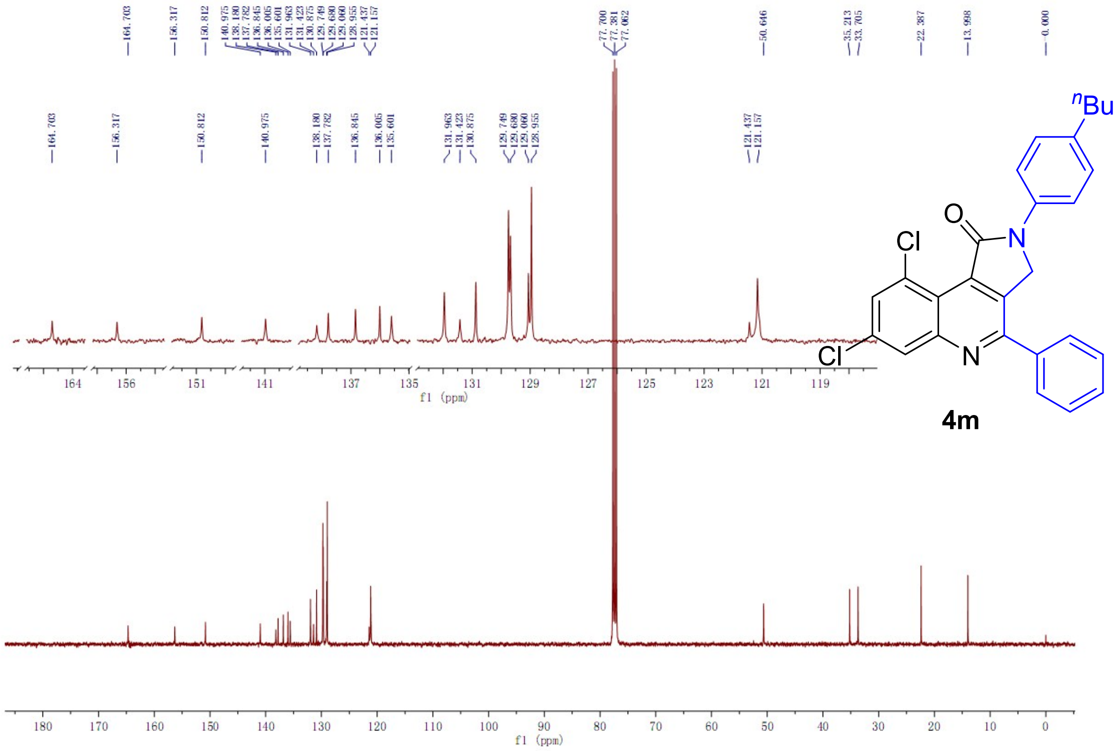


zyl-1-132-c



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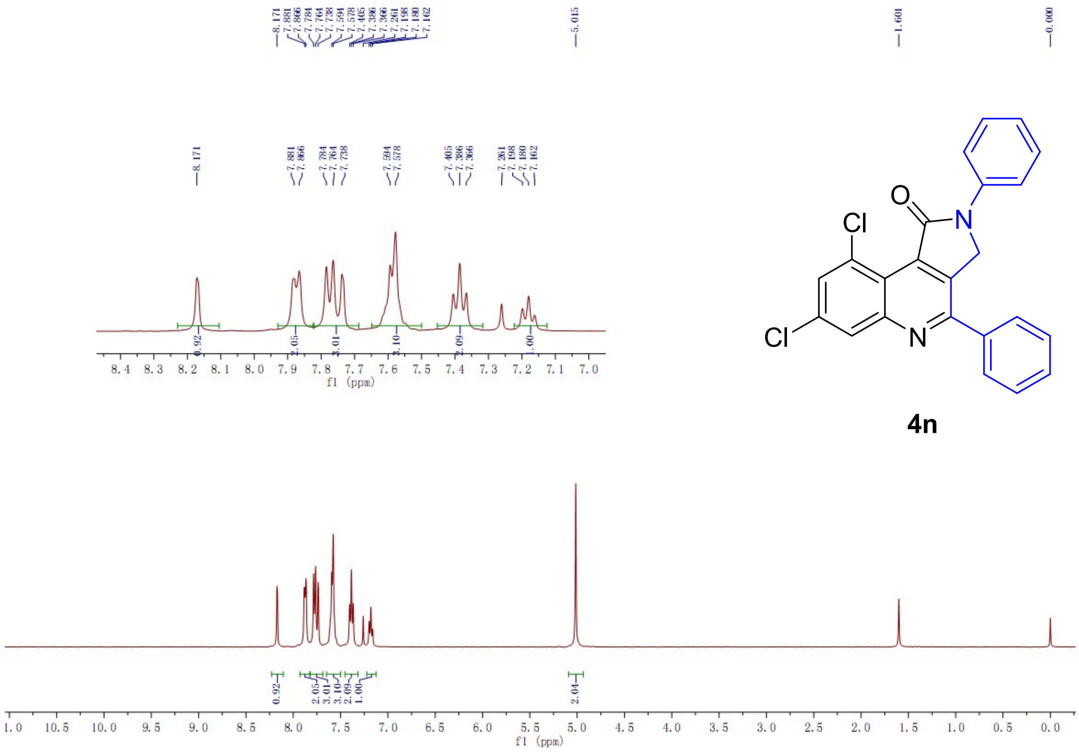

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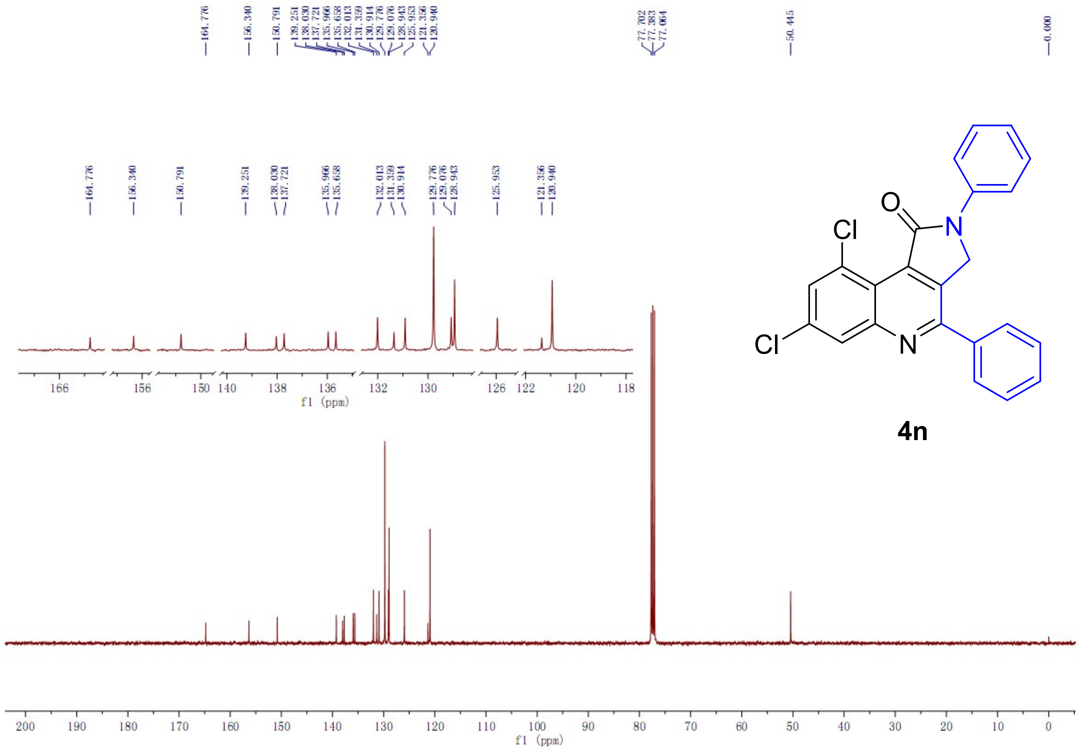

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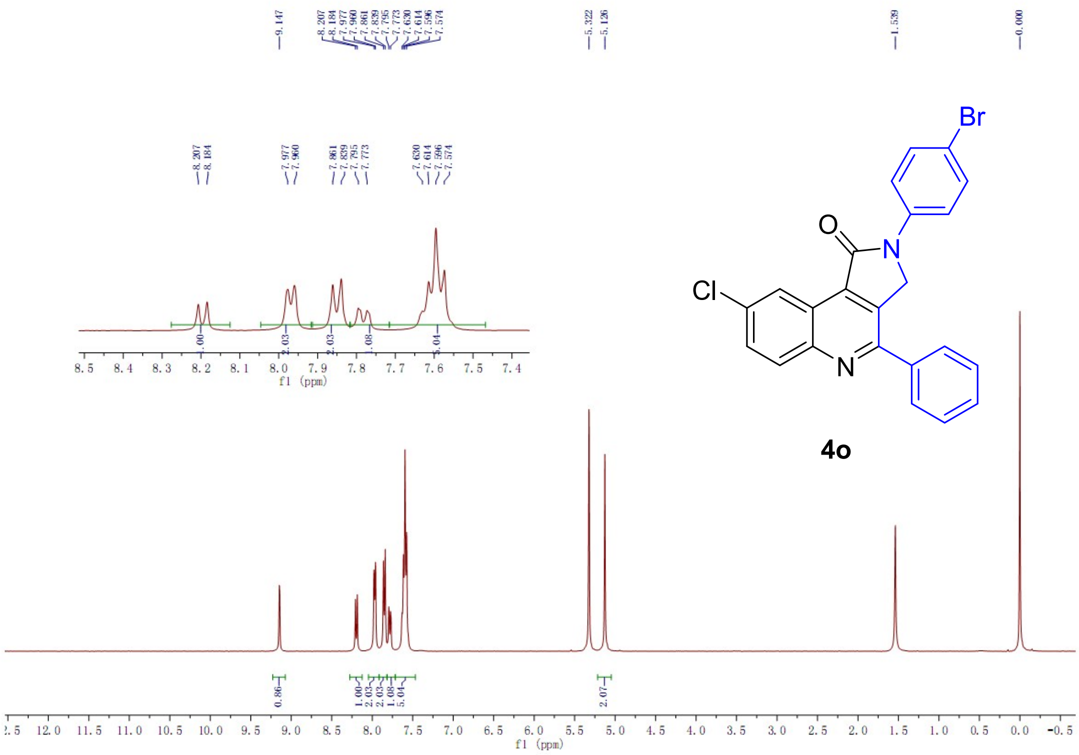

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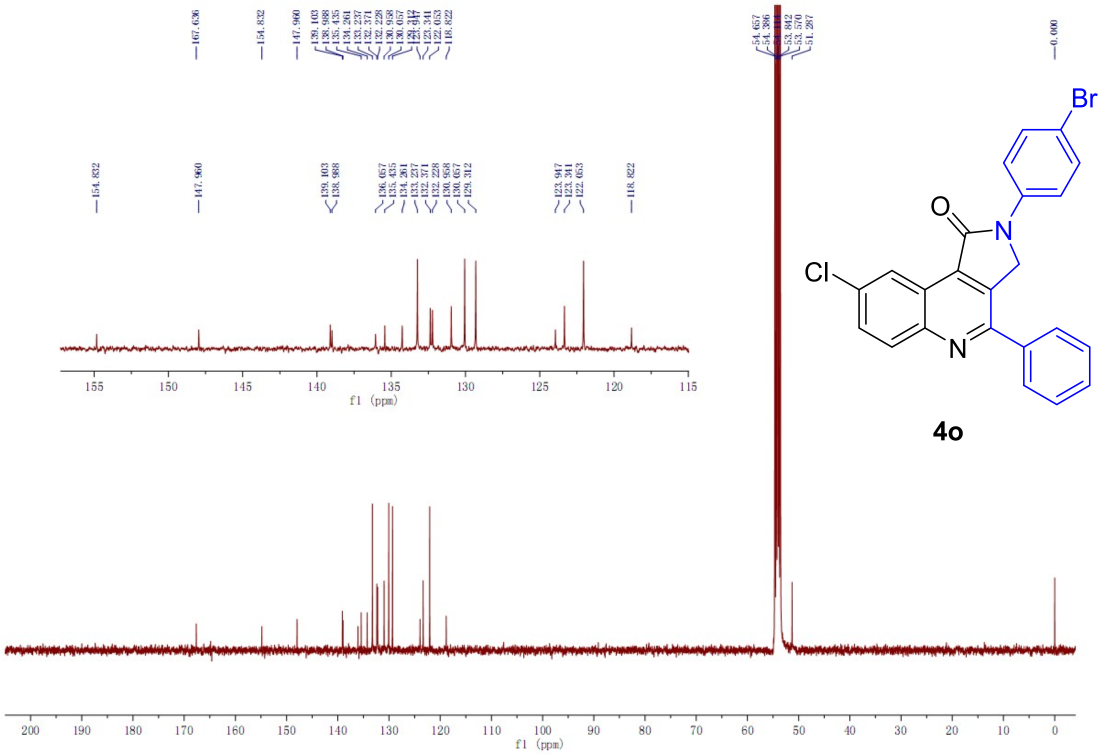
zyl-1-147-C



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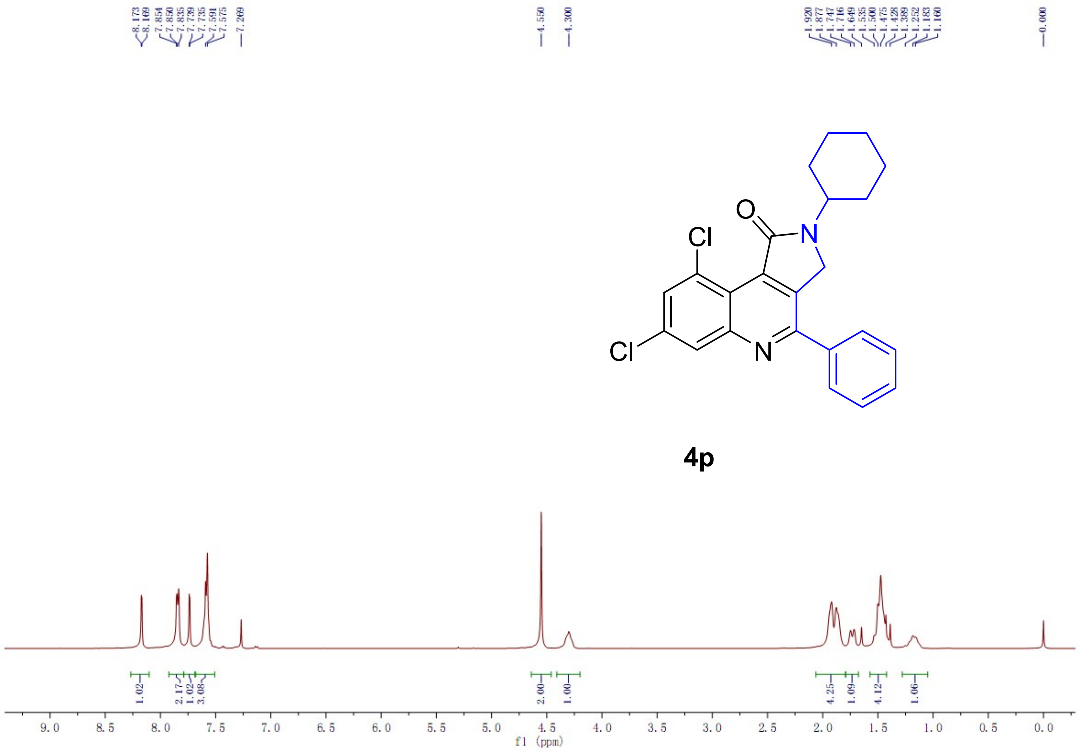

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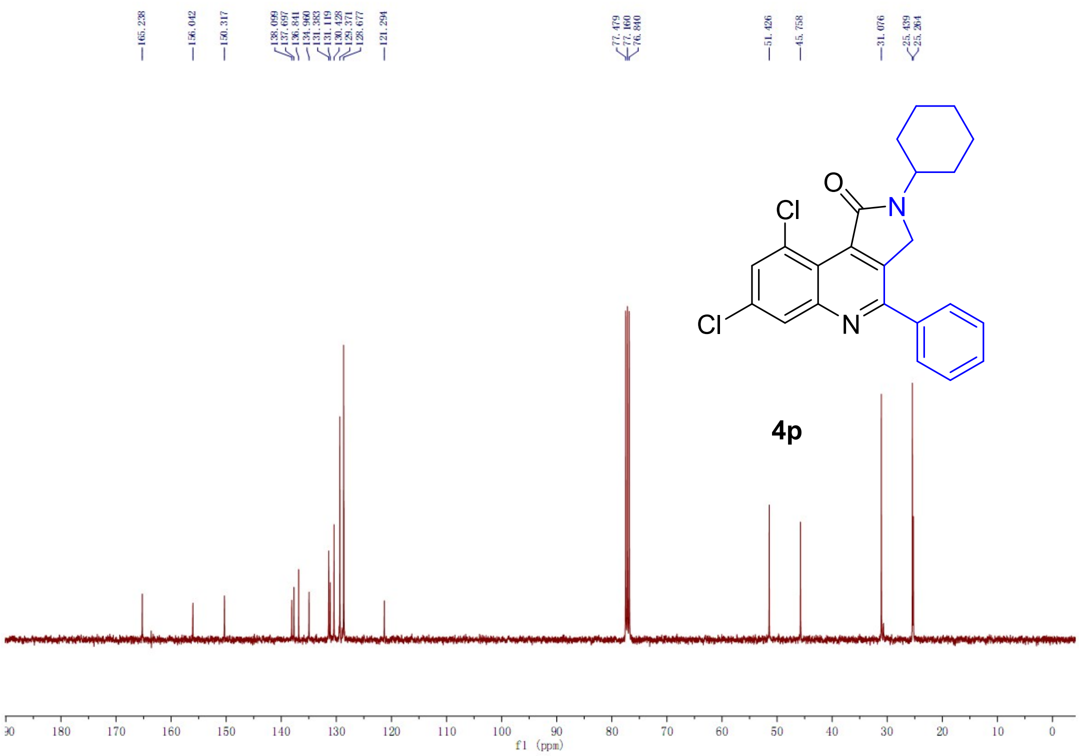

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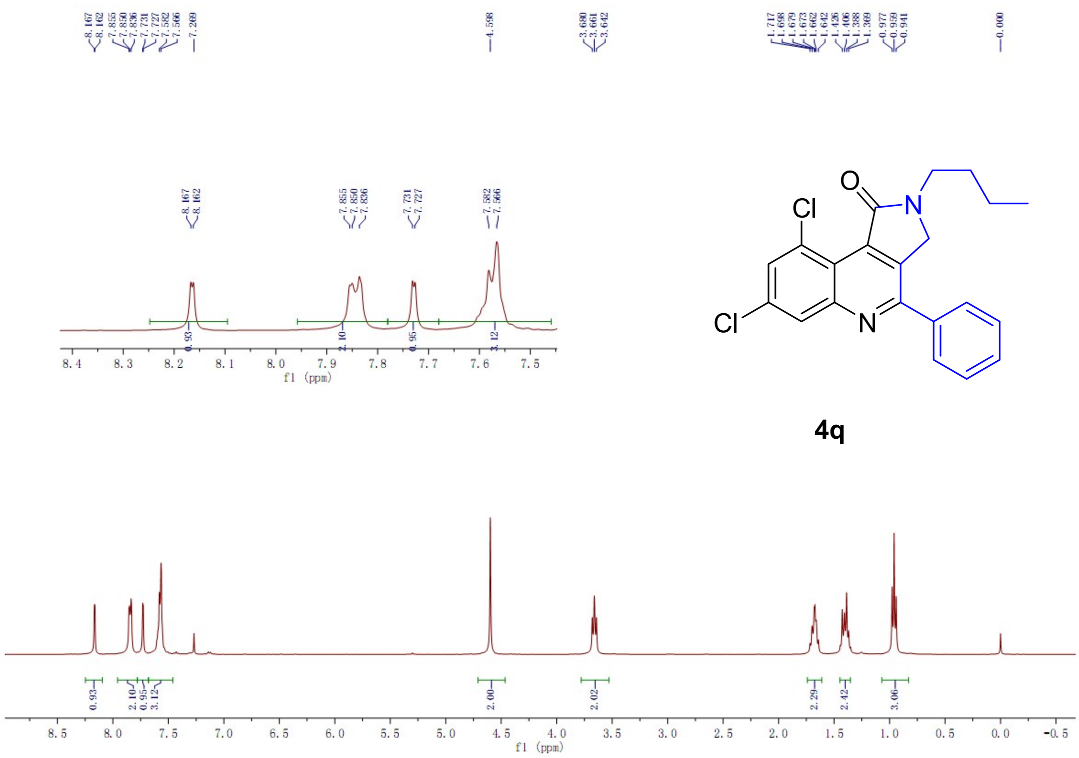
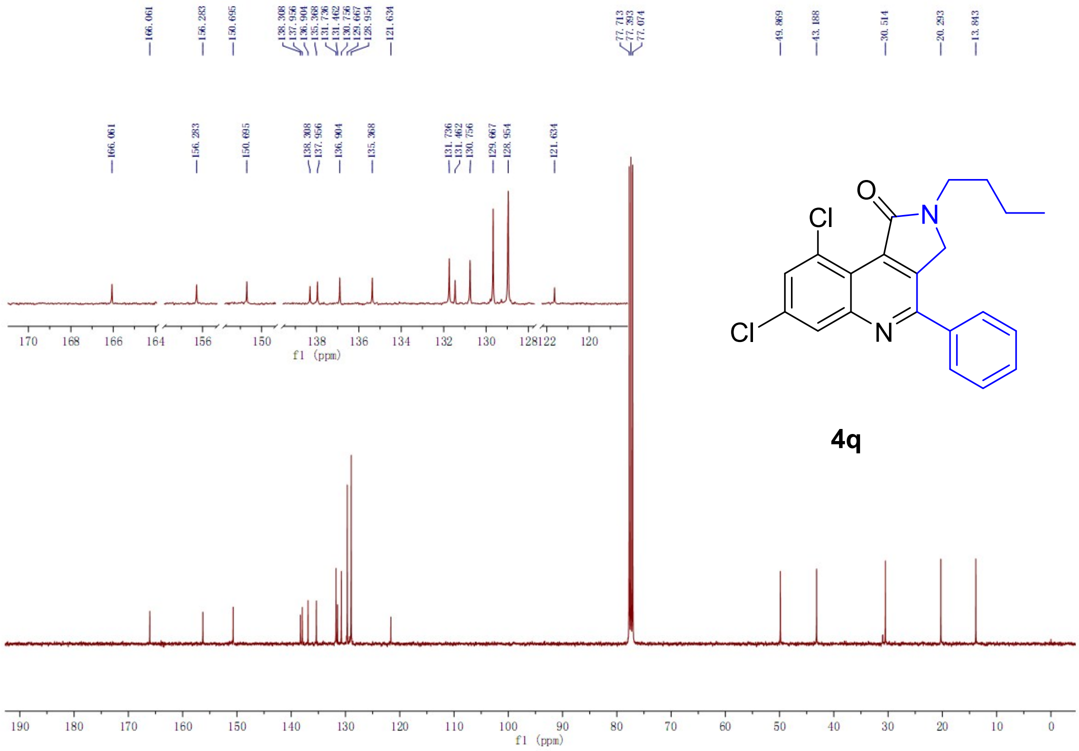


4q

zyl-1-126-C



4q

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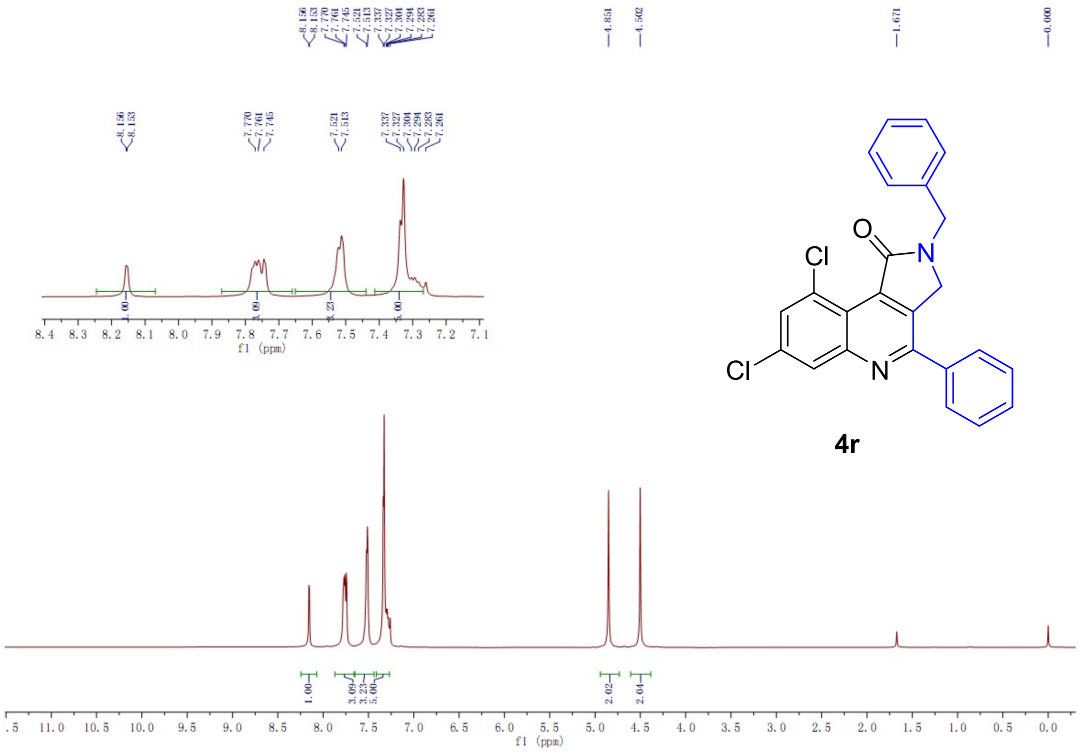
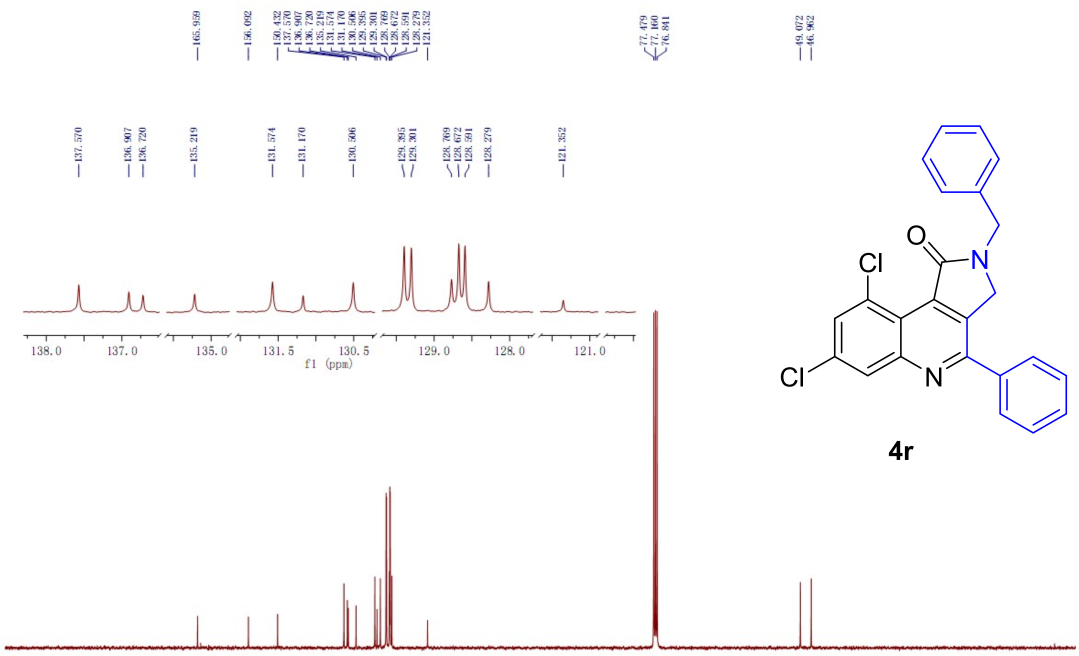
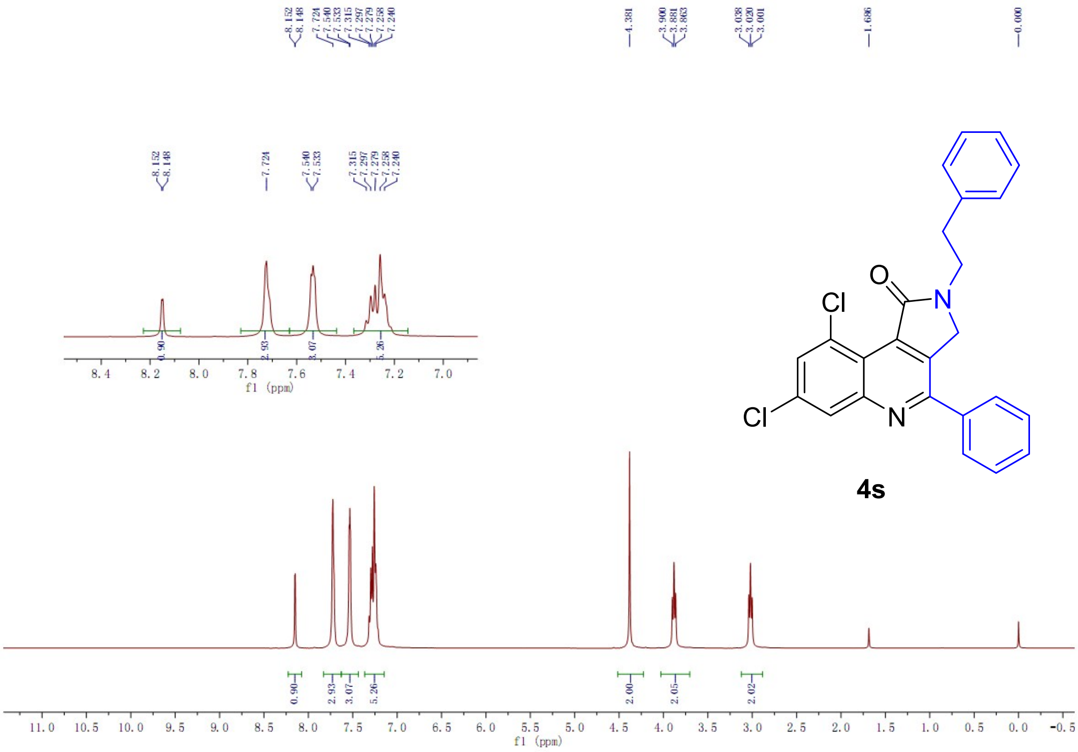


zyl-1-119-c



zyl-1-140-h



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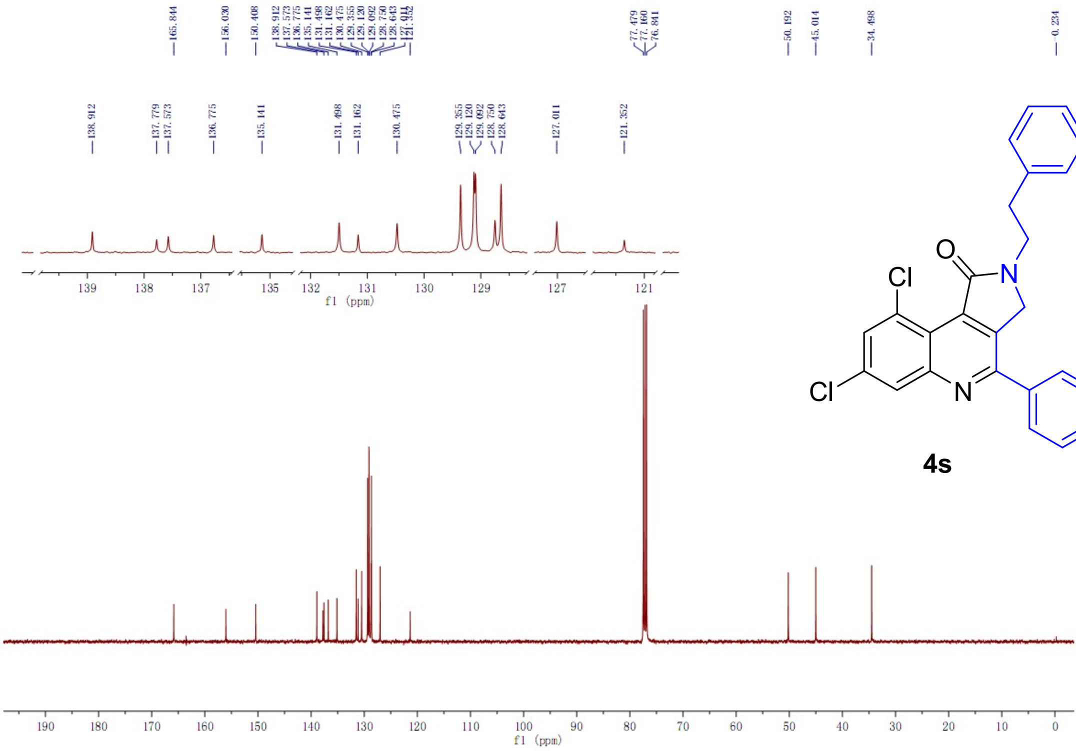

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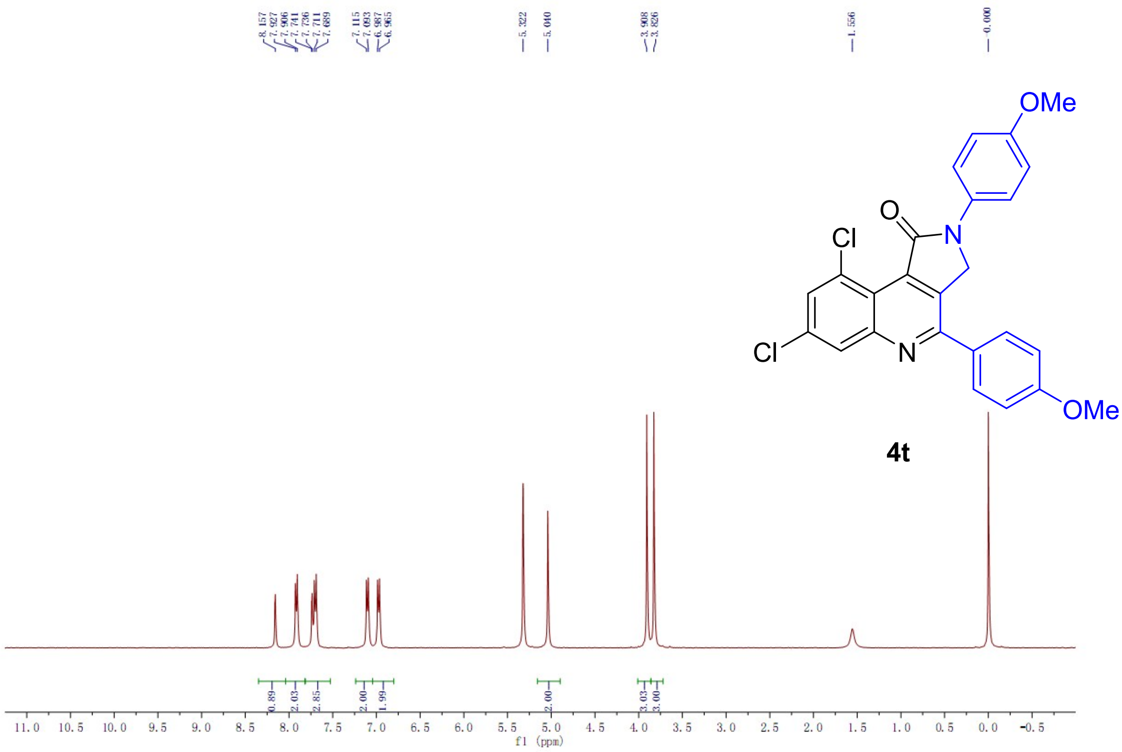

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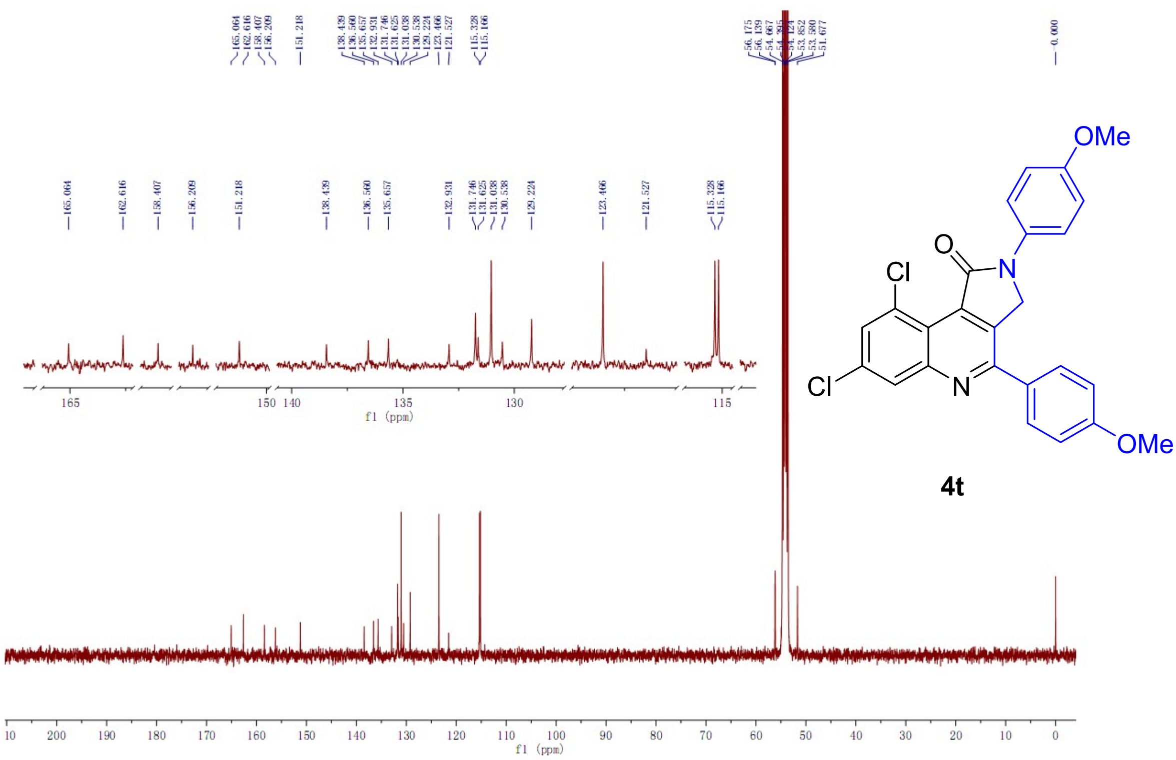

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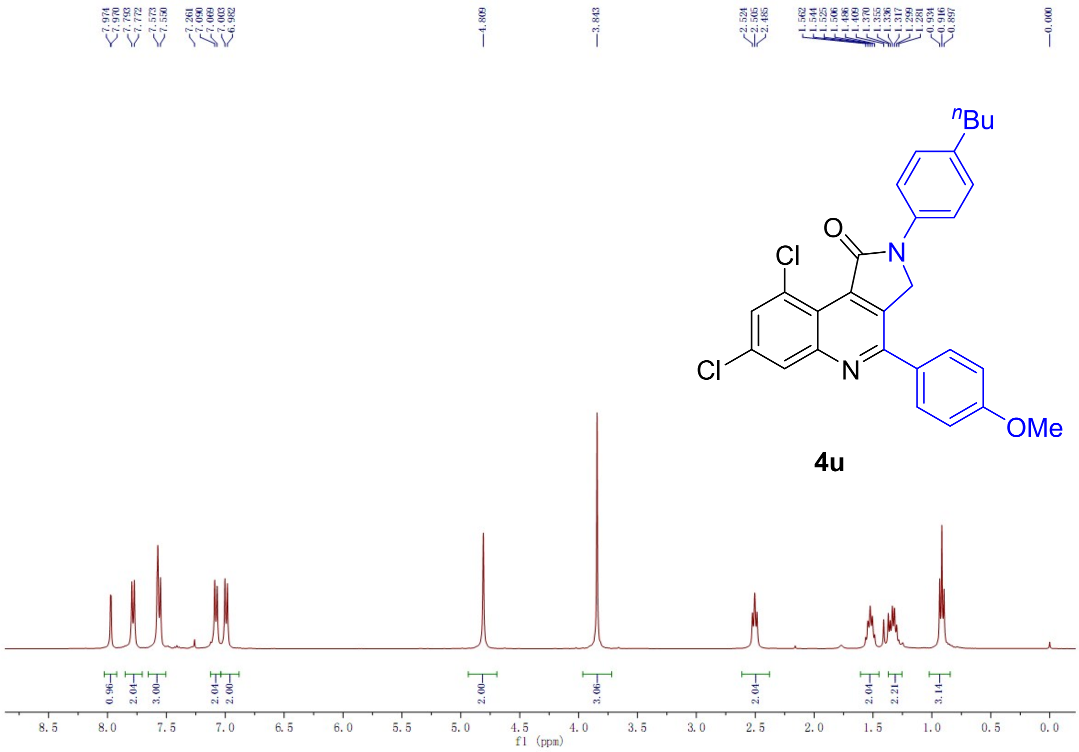

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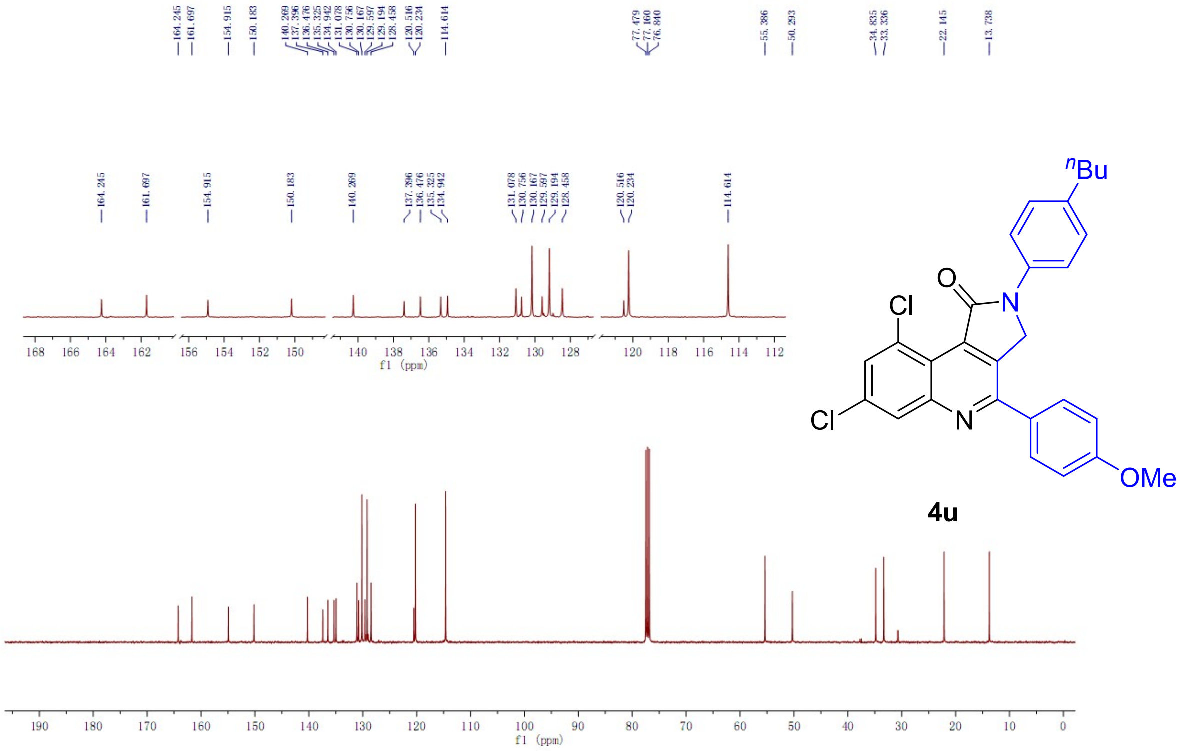

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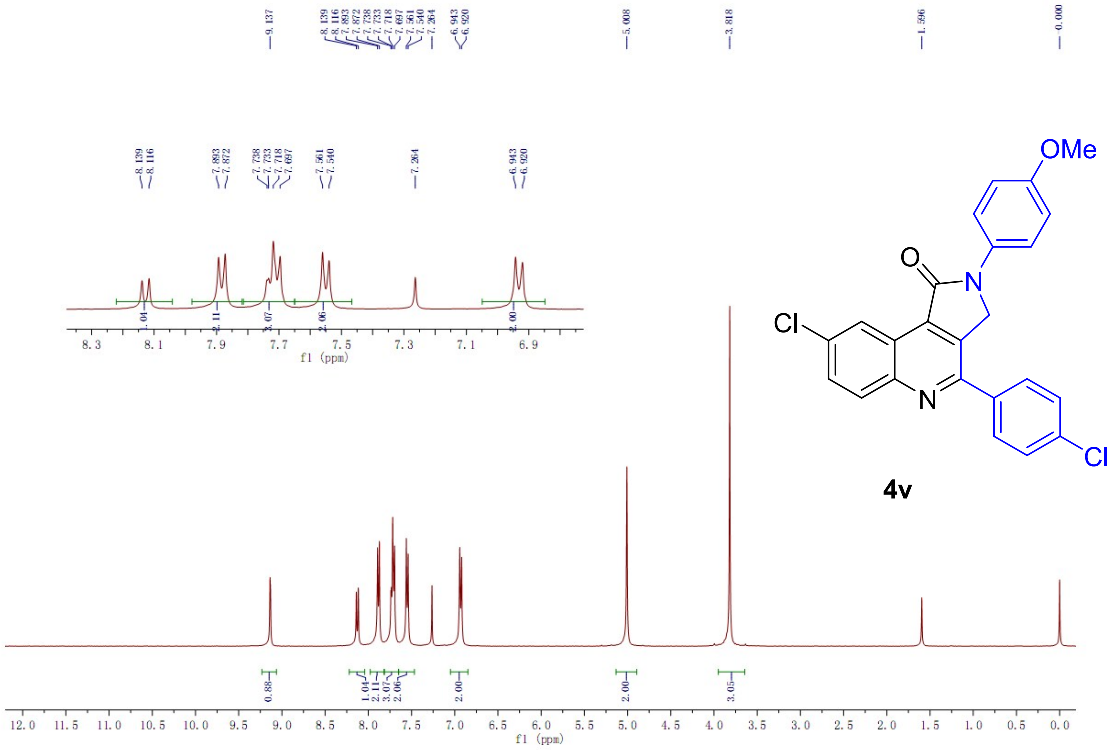

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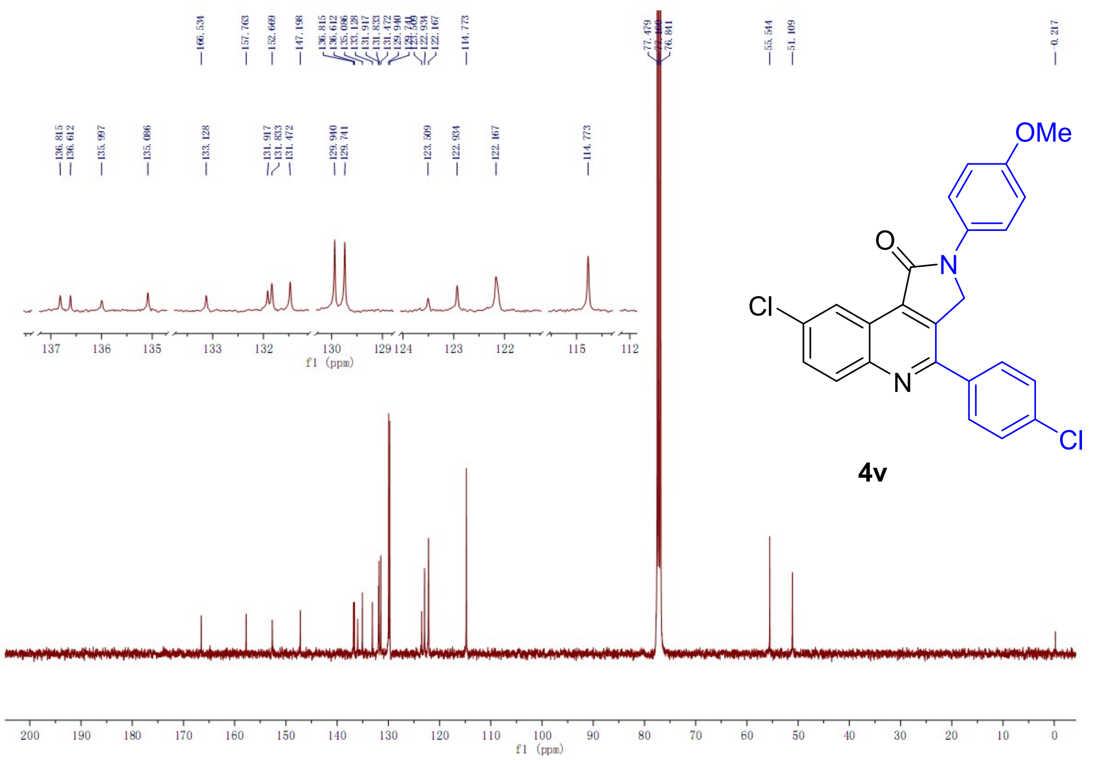

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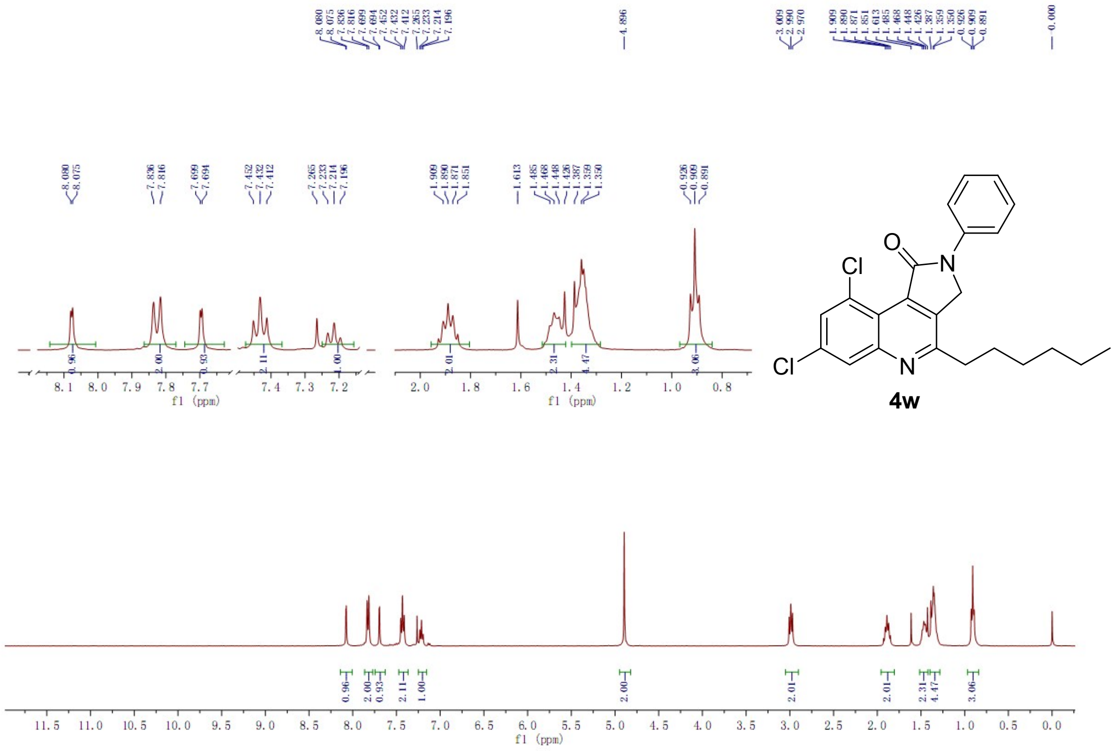
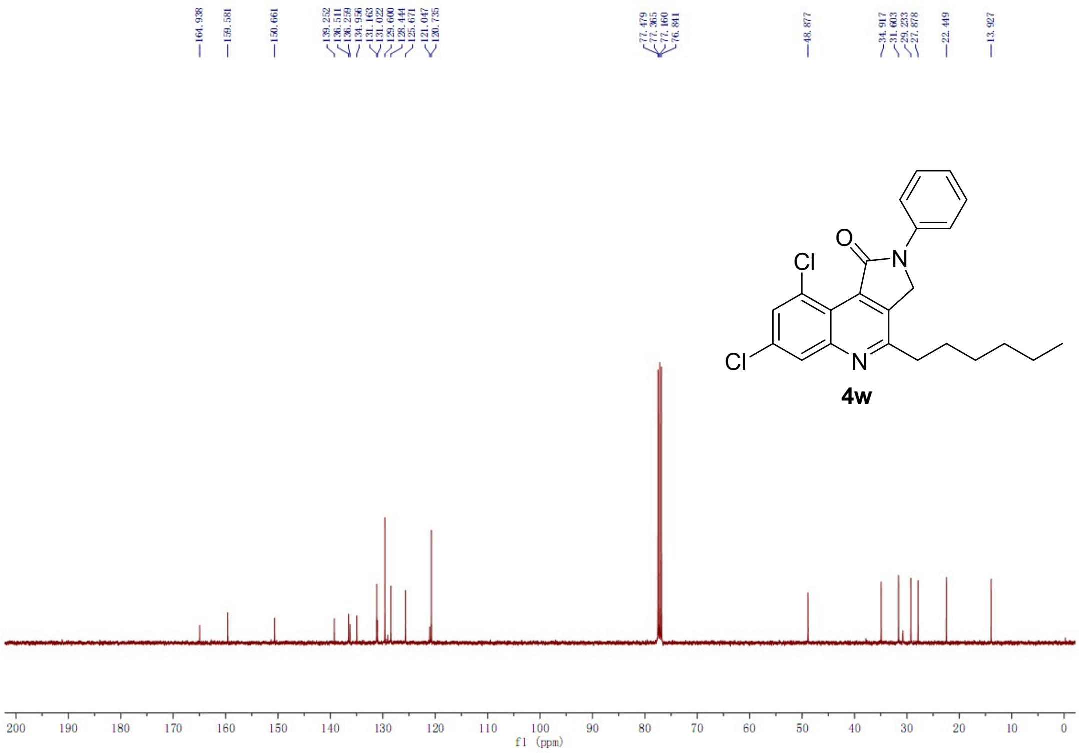
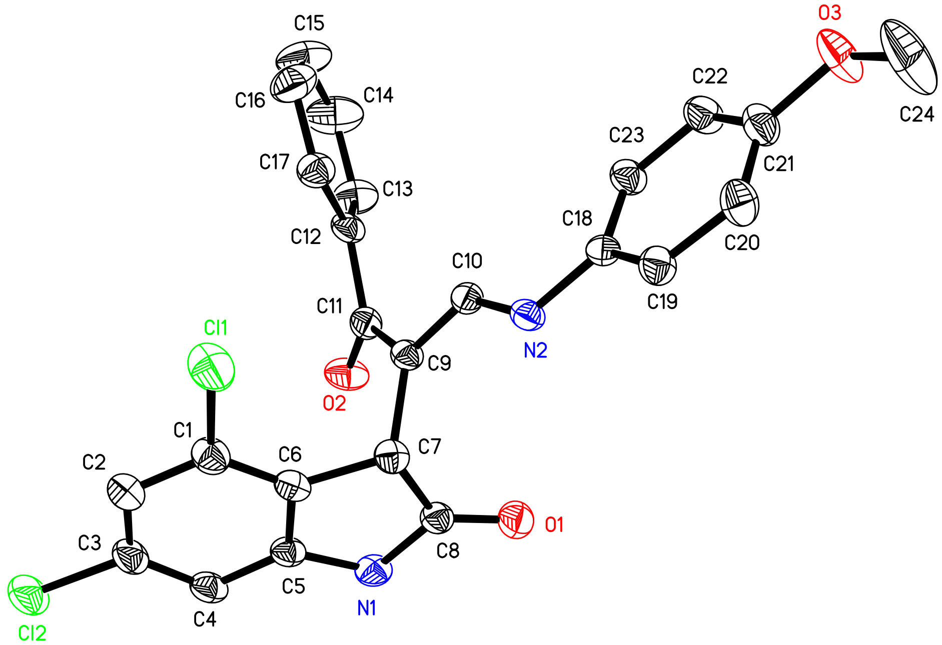


Zyl-4-128-c



X-ray crystal structure of **3a**



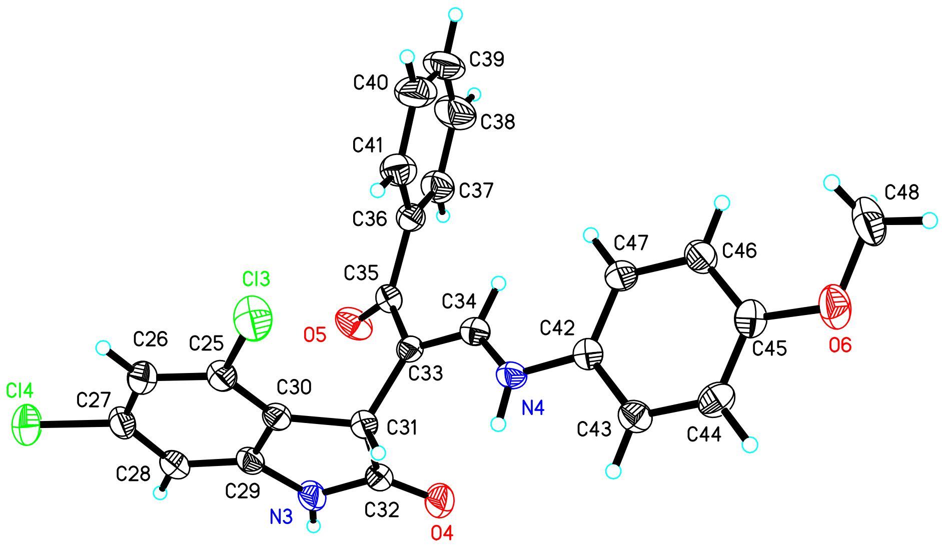


Table 1. Crystal data and structure refinement for **3a**.

Identification code	cd15125	
Empirical formula	C51 H42 Cl4 N4 O7	
Formula weight	964.68	
Temperature	293(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P -1	
Unit cell dimensions	a = 11.980(3) Å	α= 83.296(5)°.
	b = 13.777(3) Å	β= 80.970(5)°.
	c = 14.492(3) Å	γ = 87.489(5)°.
Volume	2345.2(9) $Å^3$	
Z	2	
Density (calculated)	1.366 Mg/m^3	
Absorption coefficient	0.310 mm^{-1}	
F(000)	1000	
Crystal size	0.180 x 0.150 x 0.080 mm^3	
Theta range for data collection	1.947 to 25.049°.	
Index ranges	-14<=h<=12, -14<=k<=16, -17<=l<=17	
Reflections collected	13088	
Independent reflections	8312 [R(int) = 0.0533]	
Completeness to theta = 25.242°	97.7 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.7456 and 0.6048	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	8312 / 0 / 615	
Goodness-of-fit on F^2	1.005	
Final R indices [I>2sigma(I)]	R1 = 0.0787, wR2 = 0.1858	
R indices (all data)	R1 = 0.1326, wR2 = 0.2211	
Extinction coefficient	n/a	
Largest diff. peak and hole	0.468 and -0.310 e.$Å^{-3}$	

Table 2. Atomic coordinates (x10^4) and equivalent isotropic displacement parameters ($Å^2$x10^3) for **3a**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Cl(1)	76(1)	4168(1)	3895(1)	74(1)
Cl(2)	-2094(1)	1761(1)	2178(1)	77(1)
Cl(3)	8573(1)	374(1)	10110(1)	73(1)
Cl(4)	4287(1)	1783(1)	10670(1)	89(1)
N(1)	-865(3)	854(3)	5440(3)	45(1)
N(2)	1492(3)	3457(3)	6553(3)	40(1)
N(3)	7452(3)	3328(3)	8164(3)	53(1)
N(4)	11373(3)	1515(3)	8068(3)	41(1)
O(1)	19(3)	892(2)	6722(2)	57(1)
O(2)	1632(3)	1143(2)	4415(2)	57(1)
O(3)	4213(3)	4926(3)	8798(3)	75(1)
O(4)	9173(3)	3656(3)	7295(3)	66(1)
O(5)	8895(3)	3639(2)	9782(3)	63(1)
O(6)	15875(3)	566(2)	6834(3)	64(1)
O(7)	6616(5)	4052(5)	6392(4)	146(2)
C(1)	-520(4)	3041(3)	3892(4)	50(1)
C(2)	-1035(4)	2875(4)	3136(4)	55(1)
C(3)	-1504(4)	1977(4)	3158(3)	53(1)
C(4)	-1510(4)	1254(3)	3886(3)	48(1)
C(5)	-987(4)	1451(3)	4620(3)	41(1)
C(6)	-483(3)	2339(3)	4632(3)	41(1)
C(7)	59(4)	2295(3)	5504(3)	40(1)
C(8)	-237(4)	1272(3)	5979(3)	44(1)
C(9)	1328(3)	2462(3)	5302(3)	38(1)
C(10)	1881(4)	2995(3)	5809(3)	37(1)
C(11)	1982(4)	1916(3)	4610(3)	43(1)
C(12)	3102(4)	2270(3)	4112(3)	43(1)
C(13)	3955(4)	1591(4)	3857(4)	62(1)
C(14)	4969(5)	1885(5)	3338(5)	89(2)
C(15)	5141(5)	2855(5)	3060(5)	85(2)
C(16)	4308(5)	3536(4)	3299(4)	72(2)
C(17)	3292(4)	3245(3)	3823(3)	51(1)
C(18)	2186(4)	3852(3)	7117(3)	38(1)
C(19)	1703(4)	4464(3)	7759(3)	45(1)
C(20)	2338(4)	4840(3)	8341(3)	53(1)
C(21)	3484(4)	4604(3)	8267(3)	51(1)
C(22)	3966(4)	3992(3)	7628(3)	51(1)
C(23)	3315(4)	3605(3)	7063(3)	45(1)
C(24)	3790(6)	5575(5)	9448(5)	116(3)

C(25)	7552(4)	1243(3)	9837(3)	46(1)
C(26)	6464(4)	1173(4)	10318(4)	56(1)
C(27)	5659(4)	1851(4)	10066(3)	53(1)
C(28)	5886(4)	2602(3)	9354(3)	49(1)
C(29)	6988(4)	2650(3)	8902(3)	44(1)
C(30)	7836(4)	1995(3)	9139(3)	40(1)
C(31)	8929(3)	2257(3)	8523(3)	40(1)
C(32)	8578(4)	3168(3)	7918(3)	48(1)
C(33)	9915(4)	2404(3)	9024(3)	37(1)
C(34)	10986(4)	2055(3)	8766(3)	40(1)
C(35)	9707(4)	3053(3)	9754(3)	43(1)
C(36)	10471(4)	3004(3)	10478(3)	48(1)
C(37)	10819(5)	3867(4)	10742(4)	66(2)
C(38)	11506(6)	3815(6)	11439(5)	97(2)
C(39)	11787(6)	2933(7)	11880(5)	96(2)
C(40)	11429(5)	2093(6)	11641(4)	84(2)
C(41)	10771(4)	2120(4)	10939(4)	64(1)
C(42)	12523(4)	1258(3)	7810(3)	38(1)
C(43)	12861(4)	960(3)	6931(3)	46(1)
C(44)	13979(4)	735(3)	6631(3)	52(1)
C(45)	14791(4)	796(3)	7195(3)	48(1)
C(46)	14459(4)	1080(3)	8078(3)	50(1)
C(47)	13335(4)	1308(3)	8377(3)	50(1)
C(48)	16701(5)	508(5)	7429(5)	86(2)
C(49)	6280(8)	2778(6)	5520(8)	187(6)
C(50)	6572(5)	3765(5)	5623(5)	87(2)
C(51)	6751(9)	4438(5)	4803(6)	135(3)

Table 3. Bond lengths [Å] and angles [°] for **3a**.

Cl(1)-C(1)	1.738(4)
Cl(2)-C(3)	1.742(5)
Cl(3)-C(25)	1.732(5)
Cl(4)-C(27)	1.738(5)
N(1)-C(8)	1.355(5)
N(1)-C(5)	1.388(6)
N(1)-H(1)	0.87(4)
N(2)-C(10)	1.327(5)
N(2)-C(18)	1.418(5)
N(2)-H(2A)	0.83(4)
N(3)-C(32)	1.355(6)
N(3)-C(29)	1.397(6)
N(3)-H(3)	0.77(5)
N(4)-C(34)	1.338(5)
N(4)-C(42)	1.410(5)
N(4)-H(4A)	0.84(4)
O(1)-C(8)	1.219(5)
O(2)-C(11)	1.239(5)
O(3)-C(21)	1.369(5)
O(3)-C(24)	1.397(7)
O(4)-C(32)	1.215(5)
O(5)-C(35)	1.235(5)
O(6)-C(45)	1.360(5)
O(6)-C(48)	1.406(7)
O(7)-C(50)	1.235(8)
C(1)-C(6)	1.363(6)
C(1)-C(2)	1.385(6)
C(2)-C(3)	1.377(7)
C(2)-H(2)	0.9300
C(3)-C(4)	1.363(7)
C(4)-C(5)	1.373(6)
C(4)-H(4)	0.9300
C(5)-C(6)	1.391(6)
C(6)-C(7)	1.502(6)
C(7)-C(8)	1.524(6)
C(7)-C(9)	1.525(6)
C(7)-H(7)	0.9800
C(9)-C(10)	1.356(5)
C(9)-C(11)	1.437(6)
C(10)-H(10)	0.9300
C(11)-C(12)	1.493(6)
C(12)-C(17)	1.378(6)

C(12)-C(13)	1.391(7)
C(13)-C(14)	1.375(8)
C(13)-H(13)	0.9300
C(14)-C(15)	1.364(8)
C(14)-H(14)	0.9300
C(15)-C(16)	1.372(8)
C(15)-H(15)	0.9300
C(16)-C(17)	1.378(7)
C(16)-H(16)	0.9300
C(17)-H(17)	0.9300
C(18)-C(23)	1.373(6)
C(18)-C(19)	1.373(6)
C(19)-C(20)	1.377(6)
C(19)-H(19)	0.9300
C(20)-C(21)	1.387(7)
C(20)-H(20)	0.9300
C(21)-C(22)	1.369(7)
C(22)-C(23)	1.380(6)
C(22)-H(22)	0.9300
C(23)-H(23)	0.9300
C(24)-H(24A)	0.9600
C(24)-H(24B)	0.9600
C(24)-H(24C)	0.9600
C(25)-C(30)	1.374(6)
C(25)-C(26)	1.380(7)
C(26)-C(27)	1.374(7)
C(26)-H(26)	0.9300
C(27)-C(28)	1.376(7)
C(28)-C(29)	1.379(6)
C(28)-H(28)	0.9300
C(29)-C(30)	1.386(6)
C(30)-C(31)	1.496(6)
C(31)-C(33)	1.513(6)
C(31)-C(32)	1.528(6)
C(31)-H(31)	0.9800
C(33)-C(34)	1.362(6)
C(33)-C(35)	1.449(6)
C(34)-H(34)	0.9300
C(35)-C(36)	1.490(6)
C(36)-C(41)	1.381(7)
C(36)-C(37)	1.391(7)
C(37)-C(38)	1.393(8)
C(37)-H(37)	0.9300
C(38)-C(39)	1.360(10)

C(38)-H(38)	0.9300
C(39)-C(40)	1.351(9)
C(39)-H(39)	0.9300
C(40)-C(41)	1.378(8)
C(40)-H(40)	0.9300
C(41)-H(41)	0.9300
C(42)-C(47)	1.378(6)
C(42)-C(43)	1.379(6)
C(43)-C(44)	1.375(6)
C(43)-H(43)	0.9300
C(44)-C(45)	1.377(7)
C(44)-H(44)	0.9300
C(45)-C(46)	1.375(6)
C(46)-C(47)	1.382(6)
C(46)-H(46)	0.9300
C(47)-H(47)	0.9300
C(48)-H(48A)	0.9600
C(48)-H(48B)	0.9600
C(48)-H(48C)	0.9600
C(49)-C(50)	1.449(10)
C(49)-H(49A)	0.9600
C(49)-H(49B)	0.9600
C(49)-H(49C)	0.9600
C(50)-C(51)	1.414(9)
C(51)-H(51A)	0.9600
C(51)-H(51B)	0.9600
C(51)-H(51C)	0.9600
C(8)-N(1)-C(5)	112.0(4)
C(8)-N(1)-H(1)	122(3)
C(5)-N(1)-H(1)	125(3)
C(10)-N(2)-C(18)	124.3(4)
C(10)-N(2)-H(2A)	117(3)
C(18)-N(2)-H(2A)	119(3)
C(32)-N(3)-C(29)	111.8(4)
C(32)-N(3)-H(3)	116(4)
C(29)-N(3)-H(3)	132(4)
C(34)-N(4)-C(42)	124.1(4)
C(34)-N(4)-H(4A)	124(3)
C(42)-N(4)-H(4A)	111(3)
C(21)-O(3)-C(24)	118.1(4)
C(45)-O(6)-C(48)	118.4(4)
C(6)-C(1)-C(2)	121.0(4)
C(6)-C(1)-Cl(1)	119.8(3)
C(2)-C(1)-Cl(1)	119.1(4)

C(3)-C(2)-C(1)	117.6(5)
C(3)-C(2)-H(2)	121.2
C(1)-C(2)-H(2)	121.2
C(4)-C(3)-C(2)	123.9(4)
C(4)-C(3)-Cl(2)	119.1(4)
C(2)-C(3)-Cl(2)	117.1(4)
C(3)-C(4)-C(5)	116.4(4)
C(3)-C(4)-H(4)	121.8
C(5)-C(4)-H(4)	121.8
C(4)-C(5)-N(1)	128.5(4)
C(4)-C(5)-C(6)	122.5(4)
N(1)-C(5)-C(6)	109.0(4)
C(1)-C(6)-C(5)	118.6(4)
C(1)-C(6)-C(7)	132.5(4)
C(5)-C(6)-C(7)	108.9(4)
C(6)-C(7)-C(8)	101.9(3)
C(6)-C(7)-C(9)	113.5(4)
C(8)-C(7)-C(9)	113.2(4)
C(6)-C(7)-H(7)	109.3
C(8)-C(7)-H(7)	109.3
C(9)-C(7)-H(7)	109.3
O(1)-C(8)-N(1)	125.1(4)
O(1)-C(8)-C(7)	126.8(4)
N(1)-C(8)-C(7)	108.0(4)
C(10)-C(9)-C(11)	118.6(4)
C(10)-C(9)-C(7)	124.0(4)
C(11)-C(9)-C(7)	116.9(3)
N(2)-C(10)-C(9)	130.1(4)
N(2)-C(10)-H(10)	114.9
C(9)-C(10)-H(10)	114.9
O(2)-C(11)-C(9)	120.9(4)
O(2)-C(11)-C(12)	118.7(4)
C(9)-C(11)-C(12)	120.4(4)
C(17)-C(12)-C(13)	118.3(4)
C(17)-C(12)-C(11)	122.2(4)
C(13)-C(12)-C(11)	119.2(4)
C(14)-C(13)-C(12)	120.9(5)
C(14)-C(13)-H(13)	119.5
C(12)-C(13)-H(13)	119.5
C(15)-C(14)-C(13)	119.8(6)
C(15)-C(14)-H(14)	120.1
C(13)-C(14)-H(14)	120.1
C(14)-C(15)-C(16)	120.2(6)
C(14)-C(15)-H(15)	119.9

C(16)-C(15)-H(15)	119.9
C(15)-C(16)-C(17)	120.2(5)
C(15)-C(16)-H(16)	119.9
C(17)-C(16)-H(16)	119.9
C(12)-C(17)-C(16)	120.5(5)
C(12)-C(17)-H(17)	119.7
C(16)-C(17)-H(17)	119.7
C(23)-C(18)-C(19)	119.4(4)
C(23)-C(18)-N(2)	121.7(4)
C(19)-C(18)-N(2)	118.9(4)
C(18)-C(19)-C(20)	120.9(4)
C(18)-C(19)-H(19)	119.5
C(20)-C(19)-H(19)	119.5
C(19)-C(20)-C(21)	119.3(4)
C(19)-C(20)-H(20)	120.3
C(21)-C(20)-H(20)	120.3
C(22)-C(21)-O(3)	114.7(4)
C(22)-C(21)-C(20)	119.8(4)
O(3)-C(21)-C(20)	125.5(4)
C(21)-C(22)-C(23)	120.2(4)
C(21)-C(22)-H(22)	119.9
C(23)-C(22)-H(22)	119.9
C(18)-C(23)-C(22)	120.3(4)
C(18)-C(23)-H(23)	119.8
C(22)-C(23)-H(23)	119.8
O(3)-C(24)-H(24A)	109.5
O(3)-C(24)-H(24B)	109.5
H(24A)-C(24)-H(24B)	109.5
O(3)-C(24)-H(24C)	109.5
H(24A)-C(24)-H(24C)	109.5
H(24B)-C(24)-H(24C)	109.5
C(30)-C(25)-C(26)	120.8(4)
C(30)-C(25)-Cl(3)	119.4(4)
C(26)-C(25)-Cl(3)	119.8(4)
C(27)-C(26)-C(25)	118.7(5)
C(27)-C(26)-H(26)	120.6
C(25)-C(26)-H(26)	120.6
C(26)-C(27)-C(28)	123.1(5)
C(26)-C(27)-Cl(4)	119.5(4)
C(28)-C(27)-Cl(4)	117.4(4)
C(27)-C(28)-C(29)	116.1(4)
C(27)-C(28)-H(28)	122.0
C(29)-C(28)-H(28)	122.0
C(28)-C(29)-C(30)	123.2(4)

C(28)-C(29)-N(3)	128.2(4)
C(30)-C(29)-N(3)	108.6(4)
C(25)-C(30)-C(29)	118.1(4)
C(25)-C(30)-C(31)	132.2(4)
C(29)-C(30)-C(31)	109.7(4)
C(30)-C(31)-C(33)	116.0(4)
C(30)-C(31)-C(32)	101.7(4)
C(33)-C(31)-C(32)	114.1(3)
C(30)-C(31)-H(31)	108.2
C(33)-C(31)-H(31)	108.2
C(32)-C(31)-H(31)	108.2
O(4)-C(32)-N(3)	124.5(5)
O(4)-C(32)-C(31)	127.2(4)
N(3)-C(32)-C(31)	108.2(4)
C(34)-C(33)-C(35)	119.1(4)
C(34)-C(33)-C(31)	124.2(4)
C(35)-C(33)-C(31)	116.3(4)
N(4)-C(34)-C(33)	128.7(4)
N(4)-C(34)-H(34)	115.7
C(33)-C(34)-H(34)	115.7
O(5)-C(35)-C(33)	120.2(4)
O(5)-C(35)-C(36)	120.1(4)
C(33)-C(35)-C(36)	119.8(4)
C(41)-C(36)-C(37)	119.4(5)
C(41)-C(36)-C(35)	121.1(4)
C(37)-C(36)-C(35)	119.4(4)
C(36)-C(37)-C(38)	119.0(6)
C(36)-C(37)-H(37)	120.5
C(38)-C(37)-H(37)	120.5
C(39)-C(38)-C(37)	120.2(6)
C(39)-C(38)-H(38)	119.9
C(37)-C(38)-H(38)	119.9
C(40)-C(39)-C(38)	121.0(6)
C(40)-C(39)-H(39)	119.5
C(38)-C(39)-H(39)	119.5
C(39)-C(40)-C(41)	120.1(6)
C(39)-C(40)-H(40)	119.9
C(41)-C(40)-H(40)	119.9
C(40)-C(41)-C(36)	120.2(6)
C(40)-C(41)-H(41)	119.9
C(36)-C(41)-H(41)	119.9
C(47)-C(42)-C(43)	117.9(4)
C(47)-C(42)-N(4)	123.8(4)
C(43)-C(42)-N(4)	118.3(4)

C(44)-C(43)-C(42)	120.7(4)
C(44)-C(43)-H(43)	119.7
C(42)-C(43)-H(43)	119.7
C(43)-C(44)-C(45)	121.3(4)
C(43)-C(44)-H(44)	119.3
C(45)-C(44)-H(44)	119.3
O(6)-C(45)-C(46)	124.5(4)
O(6)-C(45)-C(44)	117.0(4)
C(46)-C(45)-C(44)	118.5(4)
C(45)-C(46)-C(47)	120.1(4)
C(45)-C(46)-H(46)	120.0
C(47)-C(46)-H(46)	120.0
C(42)-C(47)-C(46)	121.6(4)
C(42)-C(47)-H(47)	119.2
C(46)-C(47)-H(47)	119.2
O(6)-C(48)-H(48A)	109.5
O(6)-C(48)-H(48B)	109.5
H(48A)-C(48)-H(48B)	109.5
O(6)-C(48)-H(48C)	109.5
H(48A)-C(48)-H(48C)	109.5
H(48B)-C(48)-H(48C)	109.5
C(50)-C(49)-H(49A)	109.5
C(50)-C(49)-H(49B)	109.5
H(49A)-C(49)-H(49B)	109.5
C(50)-C(49)-H(49C)	109.5
H(49A)-C(49)-H(49C)	109.5
H(49B)-C(49)-H(49C)	109.5
O(7)-C(50)-C(51)	119.1(8)
O(7)-C(50)-C(49)	123.1(8)
C(51)-C(50)-C(49)	117.7(8)
C(50)-C(51)-H(51A)	109.5
C(50)-C(51)-H(51B)	109.5
H(51A)-C(51)-H(51B)	109.5
C(50)-C(51)-H(51C)	109.5
H(51A)-C(51)-H(51C)	109.5
H(51B)-C(51)-H(51C)	109.5

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters (Å^{2}x10^3) for **3a**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2 a^{*2}U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Cl(1)	90(1)	43(1)	95(1)	3(1)	-40(1)	-15(1)
Cl(2)	60(1)	122(1)	56(1)	-26(1)	-17(1)	-16(1)
Cl(3)	65(1)	51(1)	96(1)	14(1)	-11(1)	4(1)
Cl(4)	46(1)	136(2)	76(1)	-3(1)	10(1)	-12(1)
N(1)	51(2)	35(2)	53(3)	-9(2)	-5(2)	-17(2)
N(2)	35(2)	44(2)	43(2)	-9(2)	-8(2)	-7(2)
N(3)	38(2)	62(3)	56(3)	13(2)	-14(2)	-3(2)
N(4)	41(2)	47(2)	38(2)	-11(2)	-10(2)	-3(2)
O(1)	64(2)	57(2)	53(2)	-2(2)	-14(2)	-14(2)
O(2)	54(2)	42(2)	77(2)	-23(2)	-5(2)	-15(2)
O(3)	72(3)	85(3)	85(3)	-44(2)	-39(2)	3(2)
O(4)	53(2)	79(2)	60(2)	15(2)	-6(2)	-14(2)
O(5)	52(2)	65(2)	79(3)	-29(2)	-23(2)	16(2)
O(6)	42(2)	72(2)	77(3)	-23(2)	3(2)	6(2)
O(7)	111(5)	233(7)	87(4)	14(4)	-24(3)	30(4)
C(1)	49(3)	43(3)	59(3)	-6(2)	-14(2)	-2(2)
C(2)	45(3)	65(3)	57(3)	-3(3)	-15(2)	-3(2)
C(3)	43(3)	78(4)	44(3)	-22(3)	-9(2)	-3(3)
C(4)	40(3)	53(3)	54(3)	-18(2)	-8(2)	-11(2)
C(5)	38(2)	42(2)	46(3)	-12(2)	-4(2)	-8(2)
C(6)	33(2)	44(3)	48(3)	-14(2)	-6(2)	-1(2)
C(7)	45(3)	36(2)	43(3)	-14(2)	-6(2)	-3(2)
C(8)	41(3)	50(3)	42(3)	-12(2)	-5(2)	-7(2)
C(9)	38(2)	36(2)	41(3)	-7(2)	-8(2)	-6(2)
C(10)	42(2)	34(2)	37(2)	-5(2)	-6(2)	-7(2)
C(11)	51(3)	39(2)	41(3)	-7(2)	-11(2)	-10(2)
C(12)	41(3)	45(3)	47(3)	-14(2)	-11(2)	-9(2)
C(13)	58(3)	47(3)	80(4)	-16(3)	3(3)	-6(2)
C(14)	58(4)	73(4)	128(6)	-32(4)	19(4)	0(3)
C(15)	62(4)	85(5)	101(5)	-21(4)	27(3)	-18(3)
C(16)	82(4)	62(3)	67(4)	-3(3)	7(3)	-23(3)
C(17)	54(3)	46(3)	53(3)	-10(2)	-7(2)	-8(2)
C(18)	41(3)	34(2)	39(2)	-2(2)	-6(2)	-12(2)
C(19)	46(3)	47(3)	42(3)	-13(2)	-6(2)	-1(2)
C(20)	61(3)	53(3)	48(3)	-18(2)	-14(2)	4(2)
C(21)	61(3)	48(3)	51(3)	-11(2)	-20(2)	-7(2)
C(22)	41(3)	60(3)	56(3)	-16(3)	-9(2)	-1(2)
C(23)	47(3)	45(3)	47(3)	-14(2)	-9(2)	-6(2)
C(24)	116(6)	132(6)	131(7)	-89(5)	-67(5)	24(5)

C(25)	42(3)	45(3)	51(3)	-4(2)	-10(2)	-2(2)
C(26)	53(3)	59(3)	53(3)	5(2)	-8(3)	-14(3)
C(27)	37(3)	68(3)	55(3)	-14(3)	-3(2)	-11(2)
C(28)	41(3)	53(3)	54(3)	-9(2)	-15(2)	-2(2)
C(29)	41(3)	48(3)	45(3)	-12(2)	-10(2)	-8(2)
C(30)	41(3)	40(2)	42(3)	-9(2)	-10(2)	-3(2)
C(31)	37(2)	43(2)	41(3)	-5(2)	-5(2)	-2(2)
C(32)	44(3)	58(3)	42(3)	-3(2)	-9(2)	-12(2)
C(33)	39(2)	38(2)	34(2)	0(2)	-6(2)	-5(2)
C(34)	44(3)	36(2)	41(3)	-2(2)	-10(2)	-2(2)
C(35)	35(2)	49(3)	45(3)	-5(2)	-6(2)	-1(2)
C(36)	42(3)	61(3)	41(3)	-13(2)	-5(2)	4(2)
C(37)	60(3)	79(4)	63(4)	-20(3)	-12(3)	-7(3)
C(38)	86(5)	138(7)	81(5)	-41(5)	-33(4)	-17(5)
C(39)	69(4)	174(8)	58(4)	-37(5)	-30(3)	17(5)
C(40)	70(4)	126(6)	59(4)	-15(4)	-21(3)	34(4)
C(41)	57(3)	78(4)	56(3)	-14(3)	-12(3)	17(3)
C(42)	41(3)	31(2)	44(3)	-4(2)	-5(2)	-1(2)
C(43)	46(3)	43(3)	51(3)	-11(2)	-11(2)	0(2)
C(44)	63(3)	49(3)	46(3)	-17(2)	-4(2)	4(2)
C(45)	47(3)	37(2)	56(3)	-8(2)	-1(2)	2(2)
C(46)	42(3)	56(3)	54(3)	-11(2)	-10(2)	-1(2)
C(47)	48(3)	62(3)	41(3)	-14(2)	-7(2)	-2(2)
C(48)	39(3)	117(5)	101(5)	-19(4)	-2(3)	7(3)
C(49)	171(10)	66(5)	275(14)	28(7)	79(9)	3(5)
C(50)	71(4)	111(6)	65(4)	13(4)	7(3)	20(4)
C(51)	208(10)	91(5)	95(6)	1(5)	1(6)	-12(6)

Table 5. Hydrogen coordinates (x10^4) and isotropic displacement parameters (Å^{2}x10^3) **3a**.

	x	y	z	U(eq)
H(1)	-1030(30)	240(30)	5550(30)	36(11)
H(2)	-1063	3354	2631	66
H(4)	-1849	660	3887	57
H(7)	-309	2781	5901	48
H(10)	2654	3043	5604	45
H(13)	3838	929	4039	75
H(14)	5536	1425	3176	106
H(15)	5826	3055	2708	102
H(16)	4429	4196	3107	87
H(17)	2731	3711	3982	61
H(19)	937	4627	7802	54
H(20)	2003	5249	8779	63
H(22)	4735	3837	7575	62
H(23)	3643	3174	6643	54
H(24A)	3395	6110	9143	174
H(24B)	4404	5820	9699	174
H(24C)	3279	5240	9950	174
H(26)	6278	676	10803	67
H(28)	5329	3050	9187	58
H(31)	9141	1738	8115	49
H(34)	11519	2213	9122	48
H(37)	10596	4470	10459	79
H(38)	11774	4385	11602	116
H(39)	12233	2907	12354	116
H(40)	11627	1496	11951	101
H(41)	10528	1540	10775	76
H(43)	12328	912	6537	55
H(44)	14190	538	6034	63
H(46)	14992	1118	8475	60
H(47)	13123	1500	8975	60
H(48A)	16790	1143	7617	129
H(48B)	17408	285	7101	129
H(48C)	16471	58	7977	129
H(49A)	6545	2332	6004	280
H(49B)	6628	2608	4915	280
H(49C)	5474	2740	5573	280
H(51A)	6111	4881	4791	203
H(51B)	6845	4090	4258	203
H(51C)	7418	4799	4802	203

H(2A)	800(40)	3520(30)	6690(30)	34(12)
H(4A)	10990(40)	1380(30)	7660(30)	43(13)
H(3)	7170(50)	3730(40)	7850(40)	68(19)

Table 6. Torsion angles [°] for **3a**.

C(6)-C(1)-C(2)-C(3)	-0.1(7)
Cl(1)-C(1)-C(2)-C(3)	-179.7(4)
C(1)-C(2)-C(3)-C(4)	1.4(8)
C(1)-C(2)-C(3)-Cl(2)	-177.3(4)
C(2)-C(3)-C(4)-C(5)	-1.5(7)
Cl(2)-C(3)-C(4)-C(5)	177.1(3)
C(3)-C(4)-C(5)-N(1)	-179.7(4)
C(3)-C(4)-C(5)-C(6)	0.4(7)
C(8)-N(1)-C(5)-C(4)	176.1(5)
C(8)-N(1)-C(5)-C(6)	-4.0(5)
C(2)-C(1)-C(6)-C(5)	-0.9(7)
Cl(1)-C(1)-C(6)-C(5)	178.7(3)
C(2)-C(1)-C(6)-C(7)	176.3(5)
Cl(1)-C(1)-C(6)-C(7)	-4.1(7)
C(4)-C(5)-C(6)-C(1)	0.7(7)
N(1)-C(5)-C(6)-C(1)	-179.1(4)
C(4)-C(5)-C(6)-C(7)	-177.1(4)
N(1)-C(5)-C(6)-C(7)	3.1(5)
C(1)-C(6)-C(7)-C(8)	-178.5(5)
C(5)-C(6)-C(7)-C(8)	-1.1(5)
C(1)-C(6)-C(7)-C(9)	-56.5(6)
C(5)-C(6)-C(7)-C(9)	120.9(4)
C(5)-N(1)-C(8)-O(1)	-178.2(4)
C(5)-N(1)-C(8)-C(7)	3.2(5)
C(6)-C(7)-C(8)-O(1)	-179.8(5)
C(9)-C(7)-C(8)-O(1)	58.0(6)
C(6)-C(7)-C(8)-N(1)	-1.2(5)
C(9)-C(7)-C(8)-N(1)	-123.5(4)
C(6)-C(7)-C(9)-C(10)	140.1(4)
C(8)-C(7)-C(9)-C(10)	-104.3(5)
C(6)-C(7)-C(9)-C(11)	-48.2(5)
C(8)-C(7)-C(9)-C(11)	67.3(5)
C(18)-N(2)-C(10)-C(9)	168.9(4)
C(11)-C(9)-C(10)-N(2)	-170.9(4)
C(7)-C(9)-C(10)-N(2)	0.7(7)
C(10)-C(9)-C(11)-O(2)	149.5(4)
C(7)-C(9)-C(11)-O(2)	-22.6(6)
C(10)-C(9)-C(11)-C(12)	-30.4(6)
C(7)-C(9)-C(11)-C(12)	157.5(4)
O(2)-C(11)-C(12)-C(17)	140.7(4)
C(9)-C(11)-C(12)-C(17)	-39.3(6)
O(2)-C(11)-C(12)-C(13)	-33.6(6)

C(9)-C(11)-C(12)-C(13)	146.4(5)
C(17)-C(12)-C(13)-C(14)	0.7(8)
C(11)-C(12)-C(13)-C(14)	175.2(5)
C(12)-C(13)-C(14)-C(15)	-0.6(10)
C(13)-C(14)-C(15)-C(16)	0.2(11)
C(14)-C(15)-C(16)-C(17)	0.1(10)
C(13)-C(12)-C(17)-C(16)	-0.4(7)
C(11)-C(12)-C(17)-C(16)	-174.8(5)
C(15)-C(16)-C(17)-C(12)	0.0(8)
C(10)-N(2)-C(18)-C(23)	-14.5(6)
C(10)-N(2)-C(18)-C(19)	168.4(4)
C(23)-C(18)-C(19)-C(20)	0.8(7)
N(2)-C(18)-C(19)-C(20)	178.0(4)
C(18)-C(19)-C(20)-C(21)	0.7(7)
C(24)-O(3)-C(21)-C(22)	178.3(6)
C(24)-O(3)-C(21)-C(20)	-2.4(8)
C(19)-C(20)-C(21)-C(22)	-0.9(7)
C(19)-C(20)-C(21)-O(3)	179.9(5)
O(3)-C(21)-C(22)-C(23)	178.9(4)
C(20)-C(21)-C(22)-C(23)	-0.4(7)
C(19)-C(18)-C(23)-C(22)	-2.1(7)
N(2)-C(18)-C(23)-C(22)	-179.2(4)
C(21)-C(22)-C(23)-C(18)	1.9(7)
C(30)-C(25)-C(26)-C(27)	2.1(7)
Cl(3)-C(25)-C(26)-C(27)	-177.7(4)
C(25)-C(26)-C(27)-C(28)	0.0(7)
C(25)-C(26)-C(27)-Cl(4)	-179.4(4)
C(26)-C(27)-C(28)-C(29)	-0.8(7)
Cl(4)-C(27)-C(28)-C(29)	178.6(3)
C(27)-C(28)-C(29)-C(30)	-0.3(6)
C(27)-C(28)-C(29)-N(3)	-179.9(4)
C(32)-N(3)-C(29)-C(28)	177.9(4)
C(32)-N(3)-C(29)-C(30)	-1.7(5)
C(26)-C(25)-C(30)-C(29)	-3.1(6)
Cl(3)-C(25)-C(30)-C(29)	176.7(3)
C(26)-C(25)-C(30)-C(31)	179.0(4)
Cl(3)-C(25)-C(30)-C(31)	-1.2(7)
C(28)-C(29)-C(30)-C(25)	2.3(6)
N(3)-C(29)-C(30)-C(25)	-178.1(4)
C(28)-C(29)-C(30)-C(31)	-179.4(4)
N(3)-C(29)-C(30)-C(31)	0.2(5)
C(25)-C(30)-C(31)-C(33)	-56.5(6)
C(29)-C(30)-C(31)-C(33)	125.5(4)
C(25)-C(30)-C(31)-C(32)	179.1(4)

C(29)-C(30)-C(31)-C(32)	1.1(4)
C(29)-N(3)-C(32)-O(4)	179.7(4)
C(29)-N(3)-C(32)-C(31)	2.4(5)
C(30)-C(31)-C(32)-O(4)	-179.3(4)
C(33)-C(31)-C(32)-O(4)	55.0(6)
C(30)-C(31)-C(32)-N(3)	-2.1(5)
C(33)-C(31)-C(32)-N(3)	-127.8(4)
C(30)-C(31)-C(33)-C(34)	137.0(4)
C(32)-C(31)-C(33)-C(34)	-105.4(5)
C(30)-C(31)-C(33)-C(35)	-50.2(5)
C(32)-C(31)-C(33)-C(35)	67.4(5)
C(42)-N(4)-C(34)-C(33)	174.5(4)
C(35)-C(33)-C(34)-N(4)	-174.4(4)
C(31)-C(33)-C(34)-N(4)	-1.8(7)
C(34)-C(33)-C(35)-O(5)	154.5(4)
C(31)-C(33)-C(35)-O(5)	-18.7(6)
C(34)-C(33)-C(35)-C(36)	-26.1(6)
C(31)-C(33)-C(35)-C(36)	160.7(4)
O(5)-C(35)-C(36)-C(41)	131.8(5)
C(33)-C(35)-C(36)-C(41)	-47.6(6)
O(5)-C(35)-C(36)-C(37)	-43.4(7)
C(33)-C(35)-C(36)-C(37)	137.2(5)
C(41)-C(36)-C(37)-C(38)	2.9(8)
C(35)-C(36)-C(37)-C(38)	178.2(5)
C(36)-C(37)-C(38)-C(39)	-3.0(10)
C(37)-C(38)-C(39)-C(40)	1.4(11)
C(38)-C(39)-C(40)-C(41)	0.2(11)
C(39)-C(40)-C(41)-C(36)	-0.2(9)
C(37)-C(36)-C(41)-C(40)	-1.4(8)
C(35)-C(36)-C(41)-C(40)	-176.5(5)
C(34)-N(4)-C(42)-C(47)	17.4(6)
C(34)-N(4)-C(42)-C(43)	-161.0(4)
C(47)-C(42)-C(43)-C(44)	-0.9(6)
N(4)-C(42)-C(43)-C(44)	177.7(4)
C(42)-C(43)-C(44)-C(45)	0.2(7)
C(48)-O(6)-C(45)-C(46)	7.2(7)
C(48)-O(6)-C(45)-C(44)	-172.6(5)
C(43)-C(44)-C(45)-O(6)	-179.5(4)
C(43)-C(44)-C(45)-C(46)	0.7(7)
O(6)-C(45)-C(46)-C(47)	179.4(4)
C(44)-C(45)-C(46)-C(47)	-0.8(7)
C(43)-C(42)-C(47)-C(46)	0.7(7)
N(4)-C(42)-C(47)-C(46)	-177.7(4)
C(45)-C(46)-C(47)-C(42)	0.1(7)

Symmetry transformations used to generate equivalent atoms:

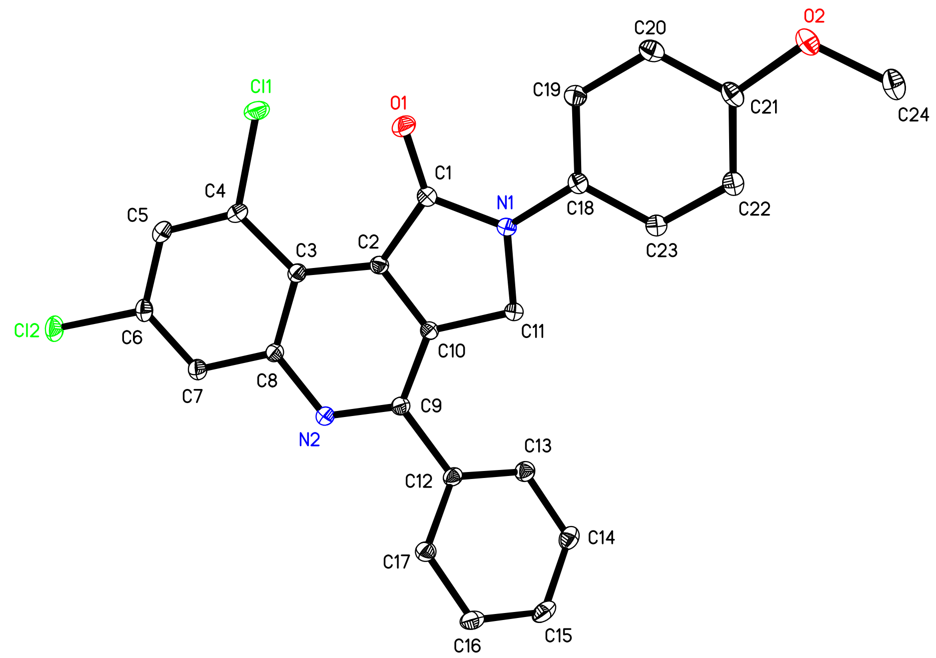
Table 7. Hydrogen bonds for **3a** [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
N(3)-H(3)...O(7)	0.77(5)	2.30(5)	2.952(7)	143(5)
N(4)-H(4A)...O(1)#1	0.84(4)	2.11(5)	2.946(5)	173(4)
N(2)-H(2A)...O(4)#2	0.83(4)	2.02(4)	2.831(5)	168(4)
C(31)-H(31)...O(1)#1	0.98	2.51	3.451(6)	160.3
C(22)-H(22)...O(7)	0.93	2.61	3.391(8)	141.4
C(7)-H(7)...O(4)#2	0.98	2.45	3.388(6)	159.1
C(22)-H(22)...O(7)	0.93	2.61	3.391(8)	141.4

Symmetry transformations used to generate equivalent atoms:

#1 x+1,y,z #2 x-1,y,z

X-ray crystal structure of **4a**



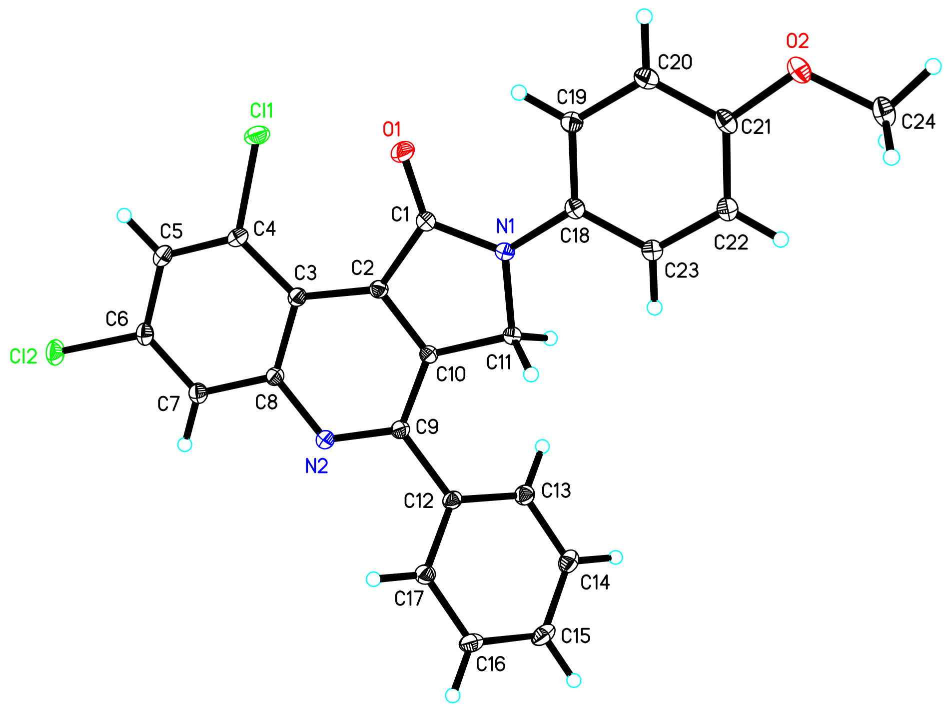


Table 1. Crystal data and structure refinement for **4a**.

Identification code	mo_dm14733_0m	
Empirical formula	C25 H17 Cl5 N2 O2	
Formula weight	554.65	
Temperature	130 K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P -1	
Unit cell dimensions	a = 7.0166(5) Å	α= 66.5140(10)°.
	b = 13.2800(9) Å	β= 85.3090(10)°.
	c = 13.9998(10) Å	γ = 89.3010(10)°.
Volume	1192.13(15) $Å^3$	
Z	2	
Density (calculated)	1.545 Mg/m^3	
Absorption coefficient	0.636 mm^{-1}	
F(000)	564	
Crystal size	0.35 x 0.2 x 0.18 mm^3	
Theta range for data collection	1.672 to 30.572°.	
Index ranges	-9<=h<=10, -18<=k<=17, -20<=l<=20	
Reflections collected	12148	
Independent reflections	7236 [R(int) = 0.0187]	
Completeness to theta = 25.242°	99.9 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.7461 and 0.6543	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	7236 / 0 / 308	
Goodness-of-fit on F^2	1.053	
Final R indices [I>2sigma(I)]	R1 = 0.0537, wR2 = 0.1357	
R indices (all data)	R1 = 0.0681, wR2 = 0.1467	
Extinction coefficient	n/a	
Largest diff. peak and hole	1.570 and -1.663 e.$Å^{-3}$	

Table 2. Atomic coordinates (x10^4) and equivalent isotropic displacement parameters (Å^{2}x10^3) for **4a**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Cl(1)	5913(1)	-3381(1)	4421(1)	29(1)
Cl(2)	6420(1)	-2944(1)	488(1)	27(1)
O(1)	7407(3)	-1931(1)	5252(1)	31(1)
O(2)	8276(2)	466(1)	8393(1)	28(1)
N(1)	7364(2)	-53(1)	4690(1)	17(1)
N(2)	7056(2)	416(1)	1252(1)	18(1)
C(1)	7236(3)	-1027(2)	4564(1)	18(1)
C(2)	6922(3)	-721(1)	3434(1)	16(1)
C(3)	6770(3)	-1347(1)	2810(1)	17(1)
C(4)	6467(3)	-2497(2)	3131(2)	19(1)
C(5)	6432(3)	-2975(2)	2424(2)	21(1)
C(6)	6620(3)	-2316(2)	1351(2)	20(1)
C(7)	6840(3)	-1206(2)	980(2)	20(1)
C(8)	6898(3)	-705(2)	1702(1)	17(1)
C(9)	7034(3)	963(2)	1857(1)	17(1)
C(10)	6976(3)	401(1)	2957(1)	16(1)
C(11)	7185(3)	901(1)	3725(1)	17(1)
C(12)	7197(3)	2177(2)	1331(1)	18(1)
C(13)	6037(3)	2856(2)	1661(2)	21(1)
C(14)	6183(3)	3988(2)	1121(2)	24(1)
C(15)	7489(3)	4445(2)	257(2)	25(1)
C(16)	8666(3)	3775(2)	-70(2)	24(1)
C(17)	8521(3)	2640(2)	468(1)	20(1)
C(18)	7659(3)	74(2)	5624(1)	17(1)
C(19)	7801(3)	-820(2)	6579(1)	19(1)
C(20)	8005(3)	-654(2)	7480(2)	22(1)
C(21)	8104(3)	402(2)	7453(2)	21(1)
C(22)	8021(3)	1297(2)	6507(2)	21(1)
C(23)	7790(3)	1130(2)	5605(2)	20(1)
C(24)	8286(3)	1536(2)	8404(2)	28(1)
Cl(3)	12182(2)	5337(1)	3020(1)	66(1)
Cl(4)	8701(2)	4283(1)	4191(1)	101(1)
Cl(5)	10334(2)	3716(1)	2526(1)	75(1)
C(25)	10806(4)	4126(2)	3538(2)	35(1)

Table 3. Bond lengths [Å] and angles [°] for **4a**.

Cl(1)-C(4)	1.7315(19)
Cl(2)-C(6)	1.7341(19)
O(1)-C(1)	1.215(2)
O(2)-C(21)	1.367(2)
O(2)-C(24)	1.427(3)
N(1)-C(1)	1.379(2)
N(1)-C(11)	1.451(2)
N(1)-C(18)	1.415(2)
N(2)-C(8)	1.367(2)
N(2)-C(9)	1.316(2)
C(1)-C(2)	1.504(2)
C(2)-C(3)	1.435(2)
C(2)-C(10)	1.367(2)
C(3)-C(4)	1.423(2)
C(3)-C(8)	1.438(2)
C(4)-C(5)	1.375(3)
C(5)-H(5)	0.9500
C(5)-C(6)	1.401(3)
C(6)-C(7)	1.360(3)
C(7)-H(7)	0.9500
C(7)-C(8)	1.417(2)
C(9)-C(10)	1.415(2)
C(9)-C(12)	1.483(2)
C(10)-C(11)	1.489(2)
C(11)-H(11A)	0.9900
C(11)-H(11B)	0.9900
C(12)-C(13)	1.394(3)
C(12)-C(17)	1.392(3)
C(13)-H(13)	0.9500
C(13)-C(14)	1.388(3)
C(14)-H(14)	0.9500
C(14)-C(15)	1.385(3)
C(15)-H(15)	0.9500
C(15)-C(16)	1.389(3)
C(16)-H(16)	0.9500
C(16)-C(17)	1.391(3)
C(17)-H(17)	0.9500
C(18)-C(19)	1.400(3)
C(18)-C(23)	1.396(3)
C(19)-H(19)	0.9500
C(19)-C(20)	1.383(3)
C(20)-H(20)	0.9500

C(20)-C(21)	1.391(3)
C(21)-C(22)	1.387(3)
C(22)-H(22)	0.9500
C(22)-C(23)	1.388(3)
C(23)-H(23)	0.9500
C(24)-H(24A)	0.9800
C(24)-H(24B)	0.9800
C(24)-H(24C)	0.9800
Cl(3)-C(25)	1.743(2)
Cl(4)-C(25)	1.728(3)
Cl(5)-C(25)	1.762(3)
C(25)-H(25)	1.0000
C(21)-O(2)-C(24)	117.12(17)
C(1)-N(1)-C(11)	112.59(14)
C(1)-N(1)-C(18)	126.84(15)
C(18)-N(1)-C(11)	120.56(15)
C(9)-N(2)-C(8)	119.12(16)
O(1)-C(1)-N(1)	124.63(17)
O(1)-C(1)-C(2)	129.16(17)
N(1)-C(1)-C(2)	106.18(15)
C(3)-C(2)-C(1)	133.31(16)
C(10)-C(2)-C(1)	107.28(15)
C(10)-C(2)-C(3)	119.13(16)
C(2)-C(3)-C(8)	114.58(15)
C(4)-C(3)-C(2)	129.50(17)
C(4)-C(3)-C(8)	115.90(16)
C(3)-C(4)-Cl(1)	122.99(14)
C(5)-C(4)-Cl(1)	114.72(14)
C(5)-C(4)-C(3)	122.06(17)
C(4)-C(5)-H(5)	120.1
C(4)-C(5)-C(6)	119.72(17)
C(6)-C(5)-H(5)	120.1
C(5)-C(6)-Cl(2)	118.14(14)
C(7)-C(6)-Cl(2)	119.90(15)
C(7)-C(6)-C(5)	121.85(17)
C(6)-C(7)-H(7)	120.5
C(6)-C(7)-C(8)	118.94(17)
C(8)-C(7)-H(7)	120.5
N(2)-C(8)-C(3)	124.19(16)
N(2)-C(8)-C(7)	114.41(16)
C(7)-C(8)-C(3)	121.39(16)
N(2)-C(9)-C(10)	120.70(16)
N(2)-C(9)-C(12)	116.99(16)
C(10)-C(9)-C(12)	122.19(16)

C(2)-C(10)-C(9)	121.90(16)
C(2)-C(10)-C(11)	111.20(15)
C(9)-C(10)-C(11)	126.55(16)
N(1)-C(11)-C(10)	102.62(14)
N(1)-C(11)-H(11A)	111.2
N(1)-C(11)-H(11B)	111.2
C(10)-C(11)-H(11A)	111.2
C(10)-C(11)-H(11B)	111.2
H(11A)-C(11)-H(11B)	109.2
C(13)-C(12)-C(9)	121.86(17)
C(17)-C(12)-C(9)	118.44(17)
C(17)-C(12)-C(13)	119.68(17)
C(12)-C(13)-H(13)	120.0
C(14)-C(13)-C(12)	120.04(18)
C(14)-C(13)-H(13)	120.0
C(13)-C(14)-H(14)	120.0
C(15)-C(14)-C(13)	120.09(19)
C(15)-C(14)-H(14)	120.0
C(14)-C(15)-H(15)	119.9
C(14)-C(15)-C(16)	120.23(18)
C(16)-C(15)-H(15)	119.9
C(15)-C(16)-H(16)	120.1
C(15)-C(16)-C(17)	119.83(19)
C(17)-C(16)-H(16)	120.1
C(12)-C(17)-H(17)	119.9
C(16)-C(17)-C(12)	120.13(18)
C(16)-C(17)-H(17)	119.9
C(19)-C(18)-N(1)	122.61(16)
C(23)-C(18)-N(1)	119.16(16)
C(23)-C(18)-C(19)	118.22(17)
C(18)-C(19)-H(19)	119.8
C(20)-C(19)-C(18)	120.44(18)
C(20)-C(19)-H(19)	119.8
C(19)-C(20)-H(20)	119.6
C(19)-C(20)-C(21)	120.78(18)
C(21)-C(20)-H(20)	119.6
O(2)-C(21)-C(20)	115.60(18)
O(2)-C(21)-C(22)	125.03(18)
C(22)-C(21)-C(20)	119.37(17)
C(21)-C(22)-H(22)	120.1
C(21)-C(22)-C(23)	119.86(18)
C(23)-C(22)-H(22)	120.1
C(18)-C(23)-H(23)	119.4
C(22)-C(23)-C(18)	121.30(18)

C(22)-C(23)-H(23)	119.4
O(2)-C(24)-H(24A)	109.5
O(2)-C(24)-H(24B)	109.5
O(2)-C(24)-H(24C)	109.5
H(24A)-C(24)-H(24B)	109.5
H(24A)-C(24)-H(24C)	109.5
H(24B)-C(24)-H(24C)	109.5
Cl(3)-C(25)-Cl(5)	109.46(14)
Cl(3)-C(25)-H(25)	108.4
Cl(4)-C(25)-Cl(3)	111.28(15)
Cl(4)-C(25)-Cl(5)	110.77(16)
Cl(4)-C(25)-H(25)	108.4
Cl(5)-C(25)-H(25)	108.4

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2$x 10^3) for **4a**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2 a^{*2}U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Cl(1)	43(1)	18(1)	22(1)	-3(1)	-3(1)	-7(1)
Cl(2)	33(1)	26(1)	31(1)	-19(1)	-2(1)	-1(1)
O(1)	55(1)	17(1)	20(1)	-5(1)	-10(1)	3(1)
O(2)	36(1)	32(1)	19(1)	-13(1)	-6(1)	-1(1)
N(1)	20(1)	16(1)	14(1)	-5(1)	-1(1)	0(1)
N(2)	22(1)	16(1)	18(1)	-7(1)	-1(1)	-1(1)
C(1)	20(1)	18(1)	17(1)	-7(1)	-1(1)	0(1)
C(2)	16(1)	16(1)	16(1)	-5(1)	0(1)	0(1)
C(3)	16(1)	15(1)	19(1)	-7(1)	-1(1)	0(1)
C(4)	19(1)	15(1)	21(1)	-4(1)	-2(1)	0(1)
C(5)	21(1)	16(1)	26(1)	-9(1)	-2(1)	1(1)
C(6)	19(1)	21(1)	24(1)	-14(1)	-1(1)	0(1)
C(7)	21(1)	20(1)	20(1)	-10(1)	-2(1)	1(1)
C(8)	18(1)	15(1)	18(1)	-7(1)	-1(1)	0(1)
C(9)	17(1)	16(1)	17(1)	-6(1)	0(1)	0(1)
C(10)	16(1)	16(1)	16(1)	-6(1)	1(1)	0(1)
C(11)	19(1)	15(1)	15(1)	-6(1)	0(1)	0(1)
C(12)	22(1)	15(1)	16(1)	-5(1)	-5(1)	-1(1)
C(13)	23(1)	19(1)	20(1)	-7(1)	-3(1)	1(1)
C(14)	27(1)	19(1)	27(1)	-10(1)	-8(1)	4(1)
C(15)	31(1)	14(1)	27(1)	-4(1)	-10(1)	-2(1)
C(16)	27(1)	19(1)	21(1)	-3(1)	-2(1)	-6(1)
C(17)	24(1)	19(1)	17(1)	-7(1)	-2(1)	-1(1)
C(18)	14(1)	20(1)	18(1)	-8(1)	-1(1)	1(1)
C(19)	19(1)	20(1)	19(1)	-7(1)	-1(1)	0(1)
C(20)	23(1)	25(1)	17(1)	-6(1)	-3(1)	0(1)
C(21)	18(1)	30(1)	18(1)	-12(1)	-2(1)	-1(1)
C(22)	21(1)	23(1)	22(1)	-11(1)	-1(1)	0(1)
C(23)	22(1)	19(1)	19(1)	-7(1)	-1(1)	0(1)
C(24)	28(1)	37(1)	27(1)	-20(1)	-4(1)	-1(1)
Cl(3)	82(1)	48(1)	57(1)	-9(1)	-9(1)	-35(1)
Cl(4)	69(1)	70(1)	119(1)	-1(1)	36(1)	23(1)
Cl(5)	124(1)	36(1)	54(1)	1(1)	-50(1)	-20(1)
C(25)	36(1)	23(1)	37(1)	-1(1)	-8(1)	-1(1)

Table 5. Hydrogen coordinates ($x10^4$) and isotropic displacement parameters ($Å^2 x 10^3$) for **4a**.

	x	y	z	U(eq)
H(5)	6281	-3749	2662	25
H(7)	6953	-773	249	24
H(11A)	8339	1385	3531	20
H(11B)	6047	1328	3781	20
H(13)	5146	2544	2257	25
H(14)	5385	4449	1345	29
H(15)	7579	5220	-113	30
H(16)	9568	4090	-659	29
H(17)	9326	2181	246	25
H(19)	7757	-1547	6607	23
H(20)	8078	-1269	8124	27
H(22)	8123	2022	6477	25
H(23)	7719	1747	4962	24
H(24A)	9457	1937	8017	43
H(24B)	8238	1471	9128	43
H(24C)	7170	1934	8074	43
H(25)	11557	3540	4047	42

Table 6. Torsion angles [°] for **4a**.

Cl(1)-C(4)-C(5)-C(6)	172.04(15)
Cl(2)-C(6)-C(7)-C(8)	176.68(14)
O(1)-C(1)-C(2)-C(3)	-1.5(4)
O(1)-C(1)-C(2)-C(10)	-175.2(2)
O(2)-C(21)-C(22)-C(23)	-178.17(18)
N(1)-C(1)-C(2)-C(3)	176.47(19)
N(1)-C(1)-C(2)-C(10)	2.8(2)
N(1)-C(18)-C(19)-C(20)	-177.12(17)
N(1)-C(18)-C(23)-C(22)	178.04(17)
N(2)-C(9)-C(10)-C(2)	-0.6(3)
N(2)-C(9)-C(10)-C(11)	171.95(17)
N(2)-C(9)-C(12)-C(13)	135.67(19)
N(2)-C(9)-C(12)-C(17)	-42.7(2)
C(1)-N(1)-C(11)-C(10)	-1.45(19)
C(1)-N(1)-C(18)-C(19)	-2.1(3)
C(1)-N(1)-C(18)-C(23)	178.75(18)
C(1)-C(2)-C(3)-C(4)	15.1(3)
C(1)-C(2)-C(3)-C(8)	-166.60(18)
C(1)-C(2)-C(10)-C(9)	169.77(16)
C(1)-C(2)-C(10)-C(11)	-3.9(2)
C(2)-C(3)-C(4)-Cl(1)	8.4(3)
C(2)-C(3)-C(4)-C(5)	-177.35(19)
C(2)-C(3)-C(8)-N(2)	-3.1(3)
C(2)-C(3)-C(8)-C(7)	177.78(16)
C(2)-C(10)-C(11)-N(1)	3.34(19)
C(3)-C(2)-C(10)-C(9)	-4.9(3)
C(3)-C(2)-C(10)-C(11)	-178.56(16)
C(3)-C(4)-C(5)-C(6)	-2.7(3)
C(4)-C(3)-C(8)-N(2)	175.41(17)
C(4)-C(3)-C(8)-C(7)	-3.7(3)
C(4)-C(5)-C(6)-Cl(2)	-176.10(15)
C(4)-C(5)-C(6)-C(7)	0.1(3)
C(5)-C(6)-C(7)-C(8)	0.6(3)
C(6)-C(7)-C(8)-N(2)	-177.83(17)
C(6)-C(7)-C(8)-C(3)	1.3(3)
C(8)-N(2)-C(9)-C(10)	4.2(3)
C(8)-N(2)-C(9)-C(12)	-179.61(16)
C(8)-C(3)-C(4)-Cl(1)	-169.93(14)
C(8)-C(3)-C(4)-C(5)	4.4(3)
C(9)-N(2)-C(8)-C(3)	-2.2(3)
C(9)-N(2)-C(8)-C(7)	176.95(17)
C(9)-C(10)-C(11)-N(1)	-169.93(17)

C(9)-C(12)-C(13)-C(14)	-177.39(18)
C(9)-C(12)-C(17)-C(16)	177.60(18)
C(10)-C(2)-C(3)-C(4)	-171.86(18)
C(10)-C(2)-C(3)-C(8)	6.4(2)
C(10)-C(9)-C(12)-C(13)	-48.2(3)
C(10)-C(9)-C(12)-C(17)	133.43(19)
C(11)-N(1)-C(1)-O(1)	177.40(19)
C(11)-N(1)-C(1)-C(2)	-0.7(2)
C(11)-N(1)-C(18)-C(19)	178.67(17)
C(11)-N(1)-C(18)-C(23)	-0.4(2)
C(12)-C(9)-C(10)-C(2)	-176.67(17)
C(12)-C(9)-C(10)-C(11)	-4.1(3)
C(12)-C(13)-C(14)-C(15)	-0.4(3)
C(13)-C(12)-C(17)-C(16)	-0.8(3)
C(13)-C(14)-C(15)-C(16)	-0.4(3)
C(14)-C(15)-C(16)-C(17)	0.5(3)
C(15)-C(16)-C(17)-C(12)	0.1(3)
C(17)-C(12)-C(13)-C(14)	1.0(3)
C(18)-N(1)-C(1)-O(1)	-1.8(3)
C(18)-N(1)-C(1)-C(2)	-179.97(16)
C(18)-N(1)-C(11)-C(10)	177.85(15)
C(18)-C(19)-C(20)-C(21)	-1.1(3)
C(19)-C(18)-C(23)-C(22)	-1.1(3)
C(19)-C(20)-C(21)-O(2)	179.07(18)
C(19)-C(20)-C(21)-C(22)	-0.8(3)
C(20)-C(21)-C(22)-C(23)	1.7(3)
C(21)-C(22)-C(23)-C(18)	-0.7(3)
C(23)-C(18)-C(19)-C(20)	2.0(3)
C(24)-O(2)-C(21)-C(20)	-176.96(18)
C(24)-O(2)-C(21)-C(22)	2.9(3)

Symmetry transformations used to generate equivalent atoms:

Table 7. Hydrogen bonds for **4a** [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)