Supplementary Material

Tf_2O-Mediated mild synthesis of 6*H*-chromeno[4,3-*b*]quinolines

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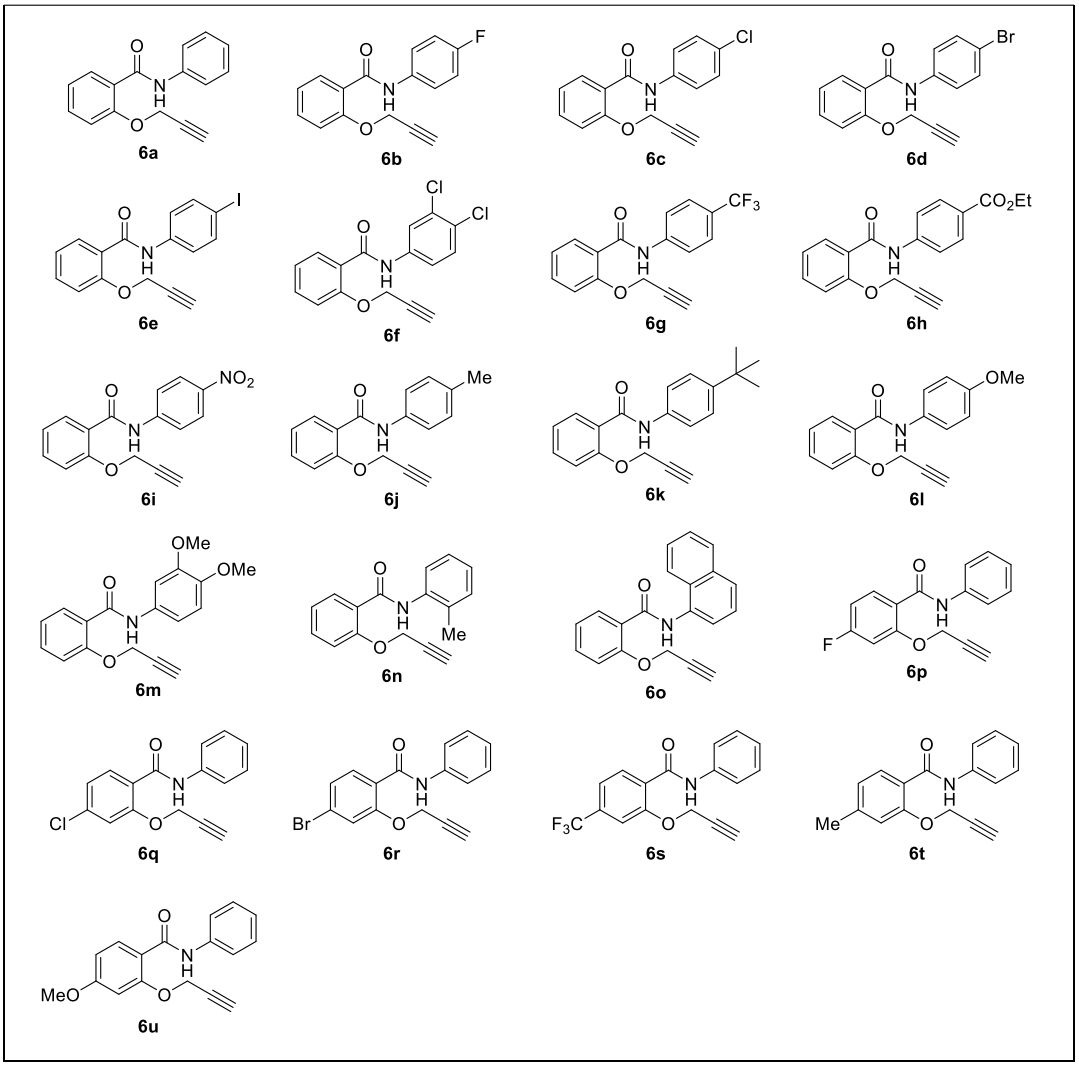
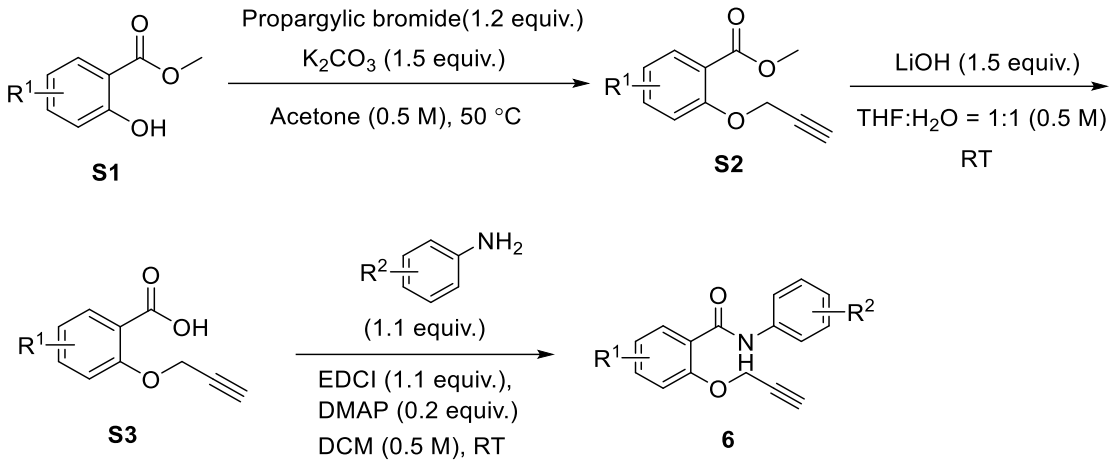
The structures of all amides used



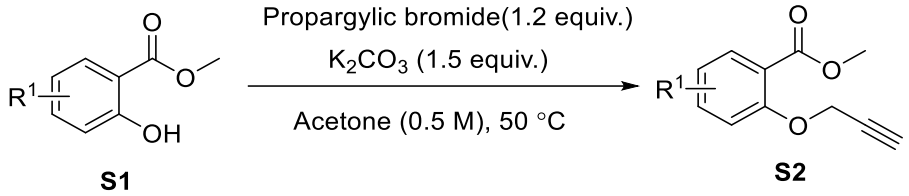
1. Preparation of amides 6

Amides **6** were prepared according to the procedures described in the literatures.[1,2] Amides **6a**,[3] **6b**,[4] **6c**,[5] **6i,**[5] **6j,**[5] **6k,**[5] **6l,**[5] **6m,**[5] **6n,**[4] **6o**[6] have been previously reported, and their spectroscopic data matched those reported in the literatures. All new amides have been characterized by ^{1}H NMR, ^{13}C NMR, and HRMS.



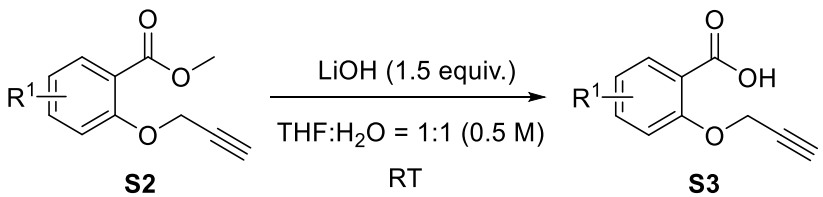
Scheme S1 The synthesis of *N*-phenyl *ortho*-propynyloxy benzamides **6**

1.1 General Procedure A for the synthesis of *ortho*-propynyloxy benzoic acid methyl ester



A mixture of the salicylate **S1** (10 mmol), K_2CO_3 (15 mmol) and the propargylic bromide (12 mmol) in acetone (20 mL) was stirred at 50 ºC for 24 h. Then the reaction was diluted with water (20 mL) and extracted with CH_2Cl_2 (20 mL × 3). The organic phases were combined and dried over anhydrous Na_2SO_4, filtered, and concentrated under reduced pressure. The residue was purified by flash column chromatography on silica gel (300-400 mesh) eluting with ethyl EtOAc/petroleum ether to give the desired product **S2**.

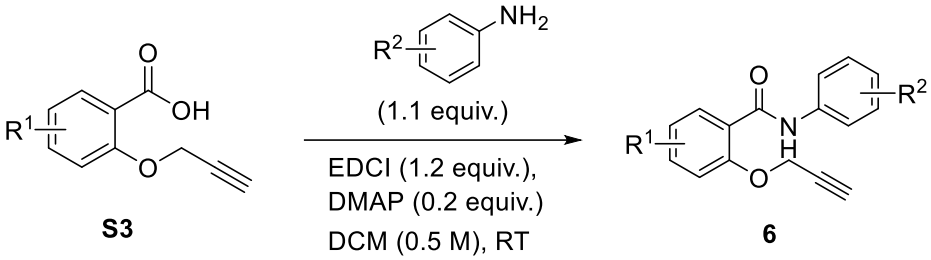
1.2 General Procedure B for the synthesis *ortho*-propynyloxy benzoic acid



A mixture of the ester **S2** (9 mmol), LiOH (13.5 mmol) in THF : H_2O (1:1, 18 mL) was stirred at room temperature for 24 h. Then the reaction mixture was acidified to pH

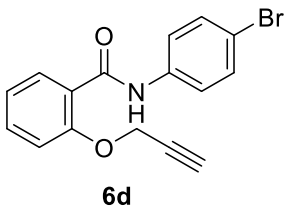
= 3 using 2 N HCl and extracted with CH_2Cl_2 (20 mL × 3). The organic phases were combined and dried over anhydrous Na_2SO_4, filtered, and concentrated under reduced pressure. The residue was purified by flash column chromatography on silica gel (300-400 mesh) eluting with ethyl EtOAc/petroleum ether to give the desired product.

1.3 General Procedure C for the synthesis of amides



To a solution of the acid **S3** (5 mmol), 4-dimethylaminopyridine (DMAP, 1.0 mmol) and *N*-ethyl-*N*'-(3-dimethylaminopropyl)carbodiimide hydrochloride (EDCI, 6.0 mmol) in CH_2Cl_2 (10.0 mL) aniline was added. The reaction was stirred at room temperature for 12 h. To the reaction mixture CH_2Cl_2 (50 mL) and water (50 mL) were added. The organic layer was separated and washed successively with 1 N HCl (50 mL × 3), and sat. aqueous $NaHCO_3$ (50 mL). The aqueous phase was extracted with CH_2Cl_2 (20 mL × 3). The organic phases were combined and dried over anhydrous Na_2SO_4, filtered, and concentrated under reduced pressure. The residue was purified by flash column chromatography on silica gel (300-400 mesh) eluting with ethyl EtOAc/petroleum ether to to give the desired amide **6**.

***N*-(4-Bromophenyl)-2-(prop-2-yn-1-yloxy)benzamide**.

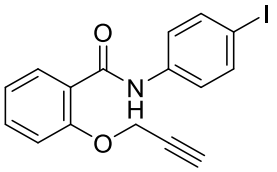


6d

Following general procedure C, amide **6d** was obtained as a white solid (1.35 g, 82%). Mp: 124-126 °C; IR (film) ν_{max}: 3358, 3290, 2918, 1660, 1600, 1544, 1488, 1395, 821, 748 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 9.81 (s, 1H), 8.29 (dd, J = 7.9, 1.8 Hz, 1H),

7.62 (d, *J* = 8.8 Hz, 2H), 7.54 – 7.44 (m, 3H), 7.19 (t, *J* = 7.6 Hz, 1H), 7.06 (d, *J* = 8.3 Hz, 1H), 4.91 (d, *J* = 2.4 Hz, 2H), 2.70 (t, *J* = 2.4 Hz, 1H) ppm; ^{13}C NMR (101 MHz, $CDCl_3$) δ 163.06, 155.54, 137.75, 133.57, 132.97, 132.14 (2C), 122.94, 122.38, 121.97 (2C), 116.83, 113.10, 77.41 (2C), 57.63 ppm; HRMS (ESI) m/z: calcd for $C_{16}H_{13}BrNO_2^+$ $[M + H]^+$: 330.0124, found: 330.0124.

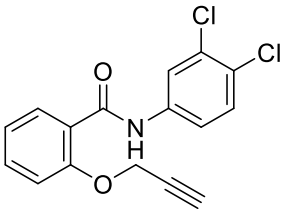
***N*-(4-Iodophenyl)-2-(prop-2-yn-1-yloxy)benzamide.**



6e

Following general procedure C, amide **6e** was obtained as a green-yellow solid (1.49 g, 79%). Mp: 142-144 °C; IR (film) ν_{max}: 3357, 3284, 2955, 1660, 1594, 1538, 1485, 1011, 821, 806, 749 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.79 (s, 1H), 8.25 (dd, *J* = 7.8, 1.8 Hz, 1H), 7.63 (d, *J* = 8.7 Hz, 2H), 7.52 – 7.45 (m, 3H), 7.19 – 7.13 (m, 1H), 7.03 (dd, *J* = 8.3, 0.9 Hz, 1H), 4.87 (d, *J* = 2.4 Hz, 2H), 2.70 (t, *J* = 2.4 Hz, 1H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.02, 155.45, 138.37, 138.00 (2C), 133.52, 132.80, 122.78, 122.21 (3C), 113.01, 87.35, 77.38, 77.17, 57.54 ppm; HRMS (ESI) m/z: calcd for $C_{16}H_{13}INO_2^+$ $[M + H]^+$: 377.9986, found: 377.9985.

***N*-(3,4-Dichlorophenyl)-2-(prop-2-yn-1-yloxy)benzamide.**

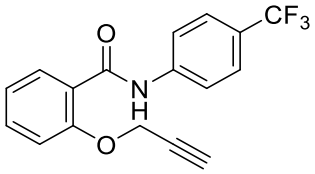


6f

Following general procedure C, amide **6f** was obtained as a white gummy solid (1.32 g, 83%). Mp: 172-174 °C; IR (film) ν_{max}: 3344, 3300, 2925, 1665, 1592, 1475, 1222, 1006, 855, 749 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.87 (s, 1H), 8.28 (dd, *J* = 7.9, 1.9 Hz, 1H), 7.95 (d, *J* = 2.4 Hz, 1H), 7.56 – 7.50 (m, 2H), 7.40 (d, *J* = 8.7 Hz, 1H), 7.22 – 7.17 (m, 1H), 7.06 (d, *J* = 8.3 Hz, 1H), 4.91 (d, *J* = 2.4 Hz, 2H), 2.72 (t, *J* = 2.4 Hz, 1H)

ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.13, 155.57, 138.16, 133.80, 132.99, 132.91, 130.68, 127.34, 123.01, 122.02 (2C), 119.60, 113.12, 77.44, 77.14, 57.70 ppm; HRMS (ESI) m/z: calcd for $C_{16}H_{12}Cl_2NO_2^+$ $[M + H]^+$: 320.0240, found: 320.0240.

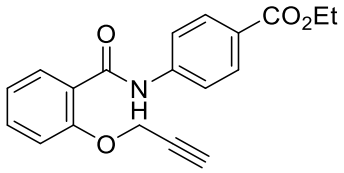
2-(Prop-2-yn-1-yloxy)-*N*-(4-(trifluoromethyl)phenyl)benzamide.



6g

Following general procedure C, amide **6g** was obtained as a pale-yellow crystalline solid (1.15 g, 72%). Mp: 139-142 °C; IR (film) ν_{max}: 3353, 3319, 2920, 1670, 1603, 1335, 1227, 1106, 839, 750 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.98 (s, 1H), 8.28 (dd, J = 7.9, 1.9 Hz, 1H), 7.82 (d, J = 8.4 Hz, 2H), 7.59 (d, J = 8.5 Hz, 2H), 7.52 –7.48 (m, 1H), 7.20 – 7.12 (m, 1H), 7.04 (d, J = 8.3 Hz, 1H), 4.89 (d, J = 2.4 Hz, 2H), 2.72 (t, J = 2.4 Hz, 1H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.30, 155.57, 141.71, 133.78, 132.94, 126.39 (q, $J_{C\text{-}F}$ = 4.0 Hz, 2C) , 125.91 (q, $J_{C\text{-}F}$ = 33.0 Hz), 124.39 (q, $J_{C\text{-}F}$ = 270.0 Hz), 122.89, 121.93, 119.98 (2C), 113.05, 77.43, 77.13, 57.62 ppm; HRMS (ESI) m/z: calcd for $C_{17}H_{13}F_3NO_2^+$ $[M + H]^+$: 320.0893, found: 320.0891.

Ethyl 4-(2-(prop-2-yn-1-yloxy)benzamido)benzoate.

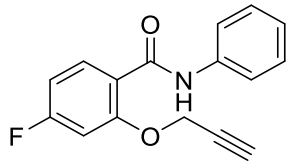


6h

Following general procedure C, amide **6h** was obtained as a pale-yellow solid (1.49 g, 92%). Mp: 149-152 °C; IR (film) ν_{max}: 3328, 2995, 2938, 1700, 1656, 1600, 1276, 1223, 998, 765 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.00 (s, 1H), 8.30 (dd, J = 7.9, 1.8 Hz, 1H), 8.05 (d, J = 8.7 Hz, 2H), 7.79 (d, J = 8.7 Hz, 2H), 7.53 – 7.49 (m, 1H), 7.23 – 7.15 (m, 1H), 7.06 (d, J = 8.3 Hz, 1H), 4.91 (d, J = 2.4 Hz, 2H), 4.37 (q, J = 7.1 Hz, 2H), 2.72 (t, J = 2.4 Hz, 1H), 1.40 (t, J = 7.2 Hz, 3H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ

166.42, 163.22, 155.57, 142.75, 133.73, 132.99, 130.97 (2C), 125.96, 122.91, 122.22, 119.49 (2C), 113.07, 77.48, 77.15, 61.00, 57.64, 14.57 ppm; HRMS (ESI) m/z: calcd for $C_{19}H_{18}NO_4^+$ $[M + H]^+$: 324.1230, found: 324.1230.

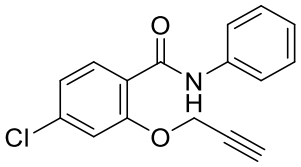
4-Fluoro-*N*-phenyl-2-(prop-2-yn-1-yloxy)benzamide.



6p

Following general procedure C, amide **6p** was obtained as a white gummy solid (1.18 g, 88%). Mp: 120-121 °C; IR (film) ν_{max}: 3370, 3300, 3055, 2925, 1661, 1603, 1555, 1279, 966, 759 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 9.58 (s, 1H), 8.31 (t, J = 7.8 Hz, 1H), 7.68 (d, J = 7.9 Hz, 2H), 7.36 (t, J = 7.7 Hz, 2H), 7.13 (t, J = 7.3 Hz, 1H), 6.92 – 6.84 (m, 1H), 6.79 (dd, J = 10.2, 2.5 Hz, 1H), 4.88 (d, J = 2.6 Hz, 2H), 2.73 (t, J = 2.6 Hz, 1H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.51 (d, J_{C-F} = 252.0 Hz), 162.15, 156.64 (d, J_{C-F} = 10.6 Hz), 138.45, 134.94 (d, J_{C-F} = 10.3 Hz), 129.20, 124.47, 120.50, 118.91 (d, J_{C-F} = 2.9 Hz), 109.74 (d, J_{C-F} = 21.1 Hz), 101.15 (d, J_{C-F} = 26.3 Hz), 77.90, 76.61, 57.78 ppm; HRMS (ESI) m/z: calcd for $C_{16}H_{13}FNO_2^+$ $[M + H]^+$: 270.0925, found: 270.0924.

4-Chloro-*N*-phenyl-2-(prop-2-yn-1-yloxy)benzamide.

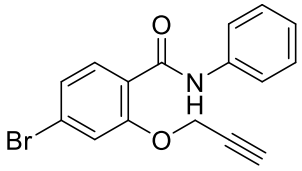


6q

Following general procedure C, amide **6q** was obtained as a white solid (1.24 g, 87%). Mp: 92-95 °C; IR (film) ν_{max}: 3368, 3299, 3100, 1660, 1556, 1445, 1403, 1229, 907, 751 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 9.60 (s, 1H), 8.23 (d, J = 8.4 Hz, 1H), 7.68 (dd, J = 8.6, 1.2 Hz, 2H), 7.35 (dd, J = 8.5, 7.3 Hz, 2H), 7.17 – 7.10 (m, 2H), 7.05 (d, J = 1.9 Hz, 1H), 4.88 (d, J = 2.4 Hz, 2H), 2.73 (t, J = 2.4 Hz, 1H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.07, 155.71, 138.89, 138.37, 134.01, 129.20 (2C), 124.54, 123.06, 121.23, 120.47 (2C), 113.64, 77.90, 76.61, 57.84 ppm; HRMS (ESI) m/z: calcd for

$C_{16}H_{13}ClNO_2^+$ [M + H]$^+$: 286.0629, found: 286.0629.

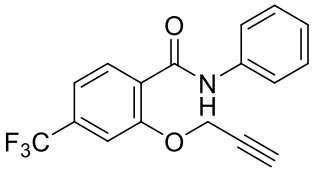
4-Bromo-*N*-phenyl-2-(prop-2-yn-1-yloxy)benzamide.



6r

Following general procedure C, amide **6r** was obtained as a white gummy solid (1.5 g, 91%). Mp: 84-86 °C; IR (film) ν_{max}: 3367, 3298, 2914, 1661, 1587, 1542, 1442, 1403, 892, 751 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.59 (s, 1H), 8.15 (d, J = 8.4 Hz, 1H), 7.67 (dd, J = 8.6, 1.2 Hz, 2H), 7.38 – 7.29 (m, 3H), 7.20 (d, J = 1.8 Hz, 1H), 7.16 – 7.10 (m, 1H), 4.88 (d, J = 2.4 Hz, 2H), 2.73 (t, J = 2.4 Hz, 1H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.14, 155.62, 138.34, 134.10, 129.19 (2C), 127.09, 126.06, 124.54, 121.67, 120.46 (2C), 116.53, 77.90, 76.60, 57.87 ppm; HRMS (ESI) m/z: calcd for $C_{16}H_{13}BrNO_2^+$ [M + H]$^+$: 330.0124, found: 330.0124.

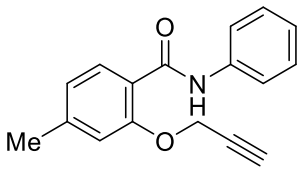
***N*-Phenyl-2-(prop-2-yn-1-yloxy)-4-(trifluoromethyl)benzamide.**



6s

Following general procedure C, amide **6s** was obtained as a white solid (1.48 g, 93%). Mp: 96-97 °C; IR (film) ν_{max}: 3352, 3229, 2934, 1670, 1446, 1333, 1131, 1006, 853, 770 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.63 (s, 1H), 8.41 – 8.37 (m, 1H), 7.69 (dd, J = 8.4, 1.3 Hz, 2H), 7.42 (d, J = 8.2 Hz, 1H), 7.36 (t, J = 7.9 Hz, 2H), 7.29 (s, 1H), 7.18 – 7.12 (m, 1H), 4.94 (d, J = 2.4 Hz, 2H), 2.73 (t, J = 2.4 Hz, 1H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ 161.70, 155.33, 138.17, 134.75 (q, J_{C-F} = 33.0 Hz), 133.71, 129.26 (2C), 125.92, 124.80, 123.46 (q, J_{C-F} = 274.0 Hz), 120.55 (2C), 119.41 (q, J_{C-F} = 3.0 Hz), 110.23 (q, J_{C-F} = 4.0 Hz), 78.09, 76.45, 57.92 ppm; HRMS (ESI) m/z: calcd for $C_{17}H_{13}F_3NO_2^+$ [M + H]$^+$: 320.0893, found: 320.0891.

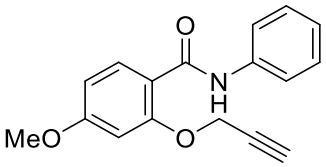
4-Methyl-*N*-phenyl-2-(prop-2-yn-1-yloxy)benzamide.



6t

Following general procedure C, amide **6t** was obtained as a white floppy solid (1.19 g, 90%). Mp: 114-116 °C; IR (film) ν_{max}: 3439, 3354, 2923, 1657, 1601, 1549, 1445, 1402, 910, 891 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 9.77 (s, 1H), 8.19 (d, J = 8.0 Hz, 1H), 7.71 (d, J = 7.8 Hz, 2H), 7.35 (t, J = 7.9 Hz, 2H), 7.11 (t, J = 7.4 Hz, 1H), 6.99 (d, J = 8.0 Hz, 1H), 6.84 (s, 1H), 4.88 (d, J = 2.4 Hz, 2H), 2.69 (t, J = 2.4 Hz, 1H), 2.41 (s, 3H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.12, 155.50, 144.35, 138.79, 132.86, 129.16 (2C), 124.19, 123.69, 120.41 (2C), 119.95, 113.73, 77.38, 57.51, 21.96 ppm; HRMS (ESI) m/z: calcd for $C_{17}H_{16}NO_2^+$ $[M + H]^+$: 266.1176, found: 266.1175.

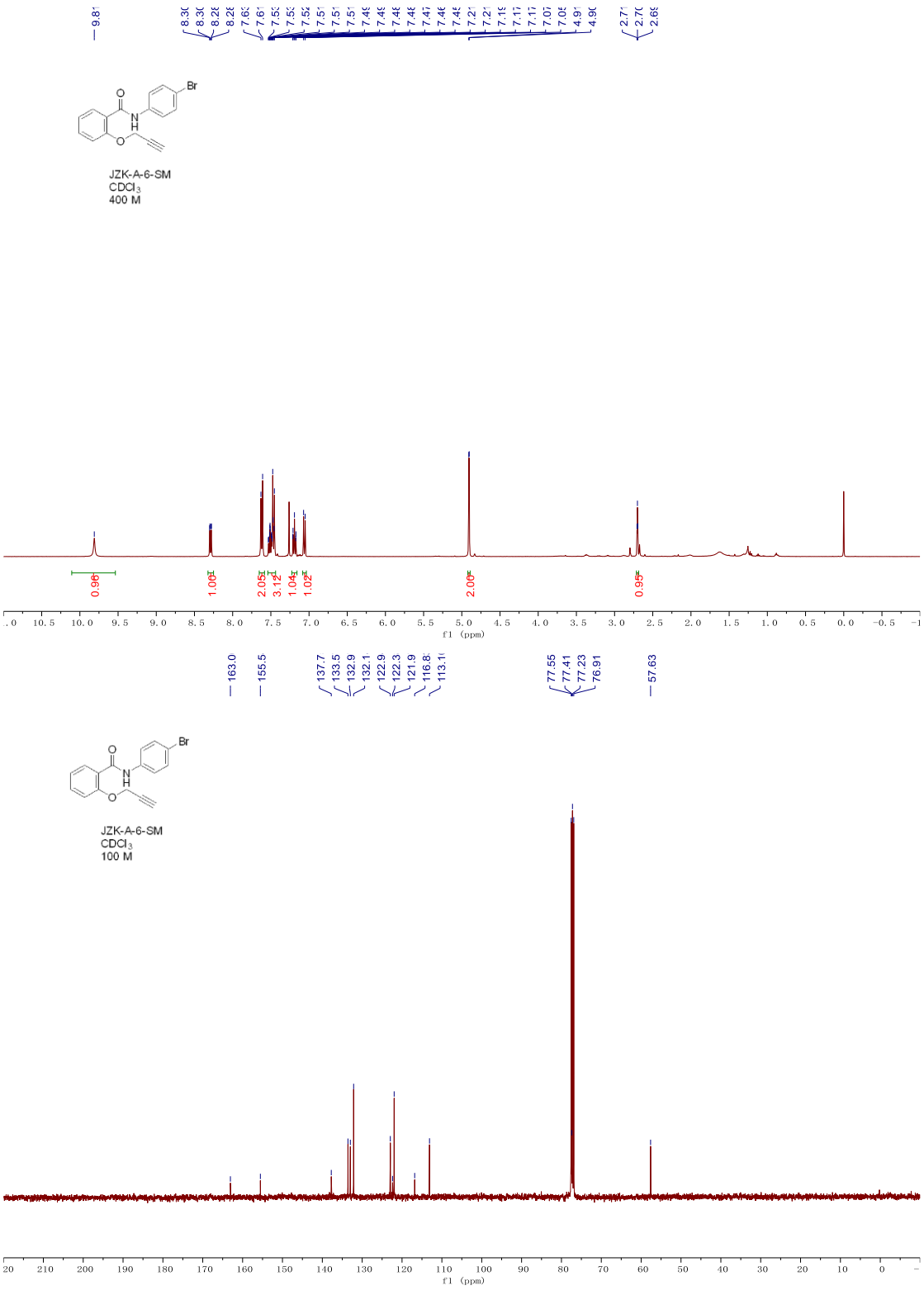
4-Methoxy-*N*-phenyl-2-(prop-2-yn-1-yloxy)benzamide.



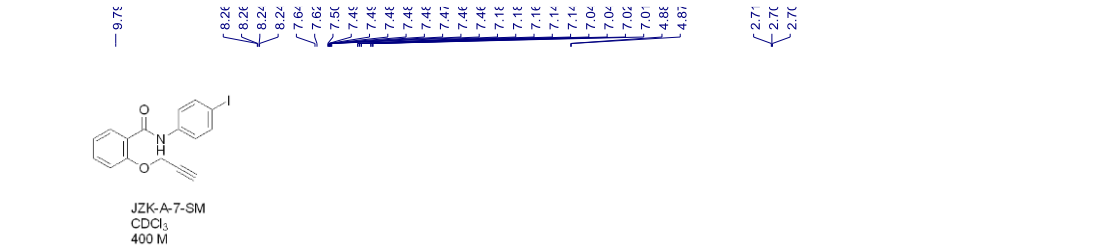
6u

Following general procedure C, amide **6u** was obtained as a white solid (1.29 g, 92%). Mp: 123-125 °C; IR (film) ν_{max}: 3377, 3254, 3059, 2977, 2120, 1597, 1446, 1251, 838, 753 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 9.65 (s, 1H), 8.21 (d, J = 8.8 Hz, 1H), 7.67 (dd, J = 8.4, 1.3 Hz, 2H), 7.31 (t, J = 7.9 Hz, 2H), 7.11 – 7.02 (m, 1H), 6.61 (dd, J = 8.8, 2.3 Hz, 1H), 6.47 (d, J = 2.3 Hz, 1H), 4.78 (d, J = 2.4 Hz, 2H), 3.77 (s, 3H), 2.70 (t, J = 2.4 Hz, 1H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.56, 162.76, 156.62, 138.76, 134.19, 128.96, 128.96, 123.86, 120.13, 120.13, 115.03, 106.73, 99.74, 77.26, 77.08, 57.27, 55.60 ppm; HRMS (ESI) m/z: calcd for $C_{17}H_{16}NO_3^+$ $[M + H]^+$: 282.1125, found: 282.1125.

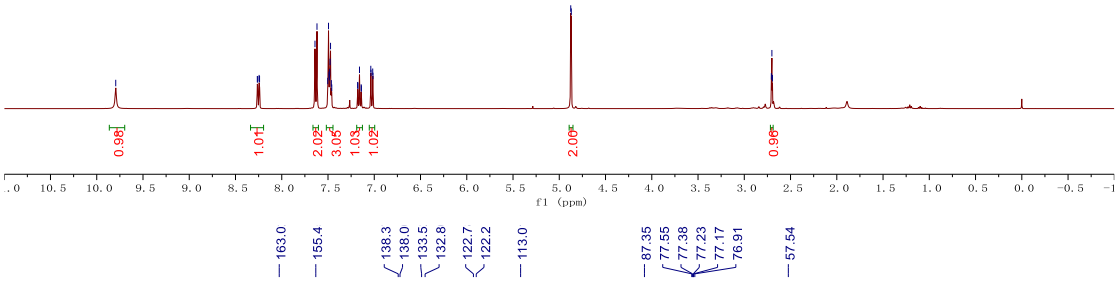
^{1}H and ^{13}C NMR spectra of compound 6d

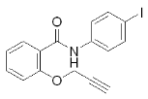


^{1}H and ^{13}C NMR spectra of compound 6e

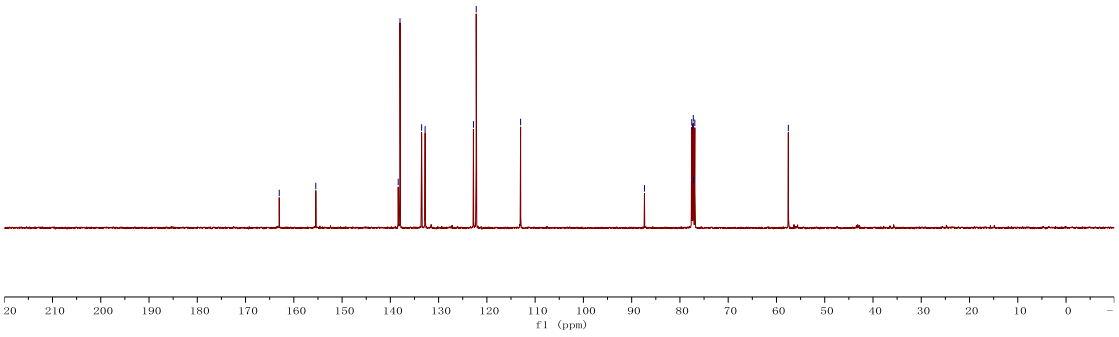


JZK-A-7-SM
$CDCl_3$
400 M

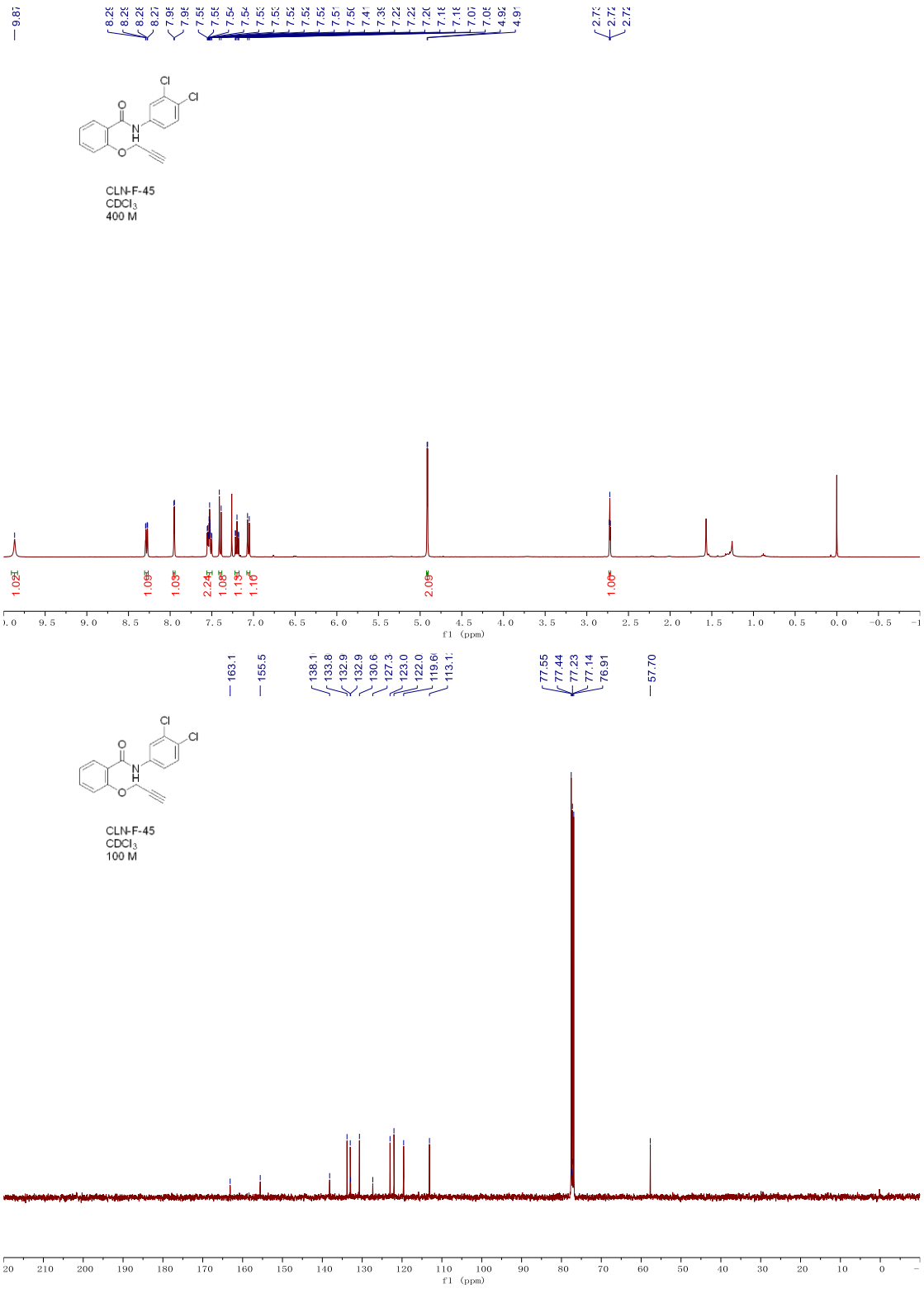




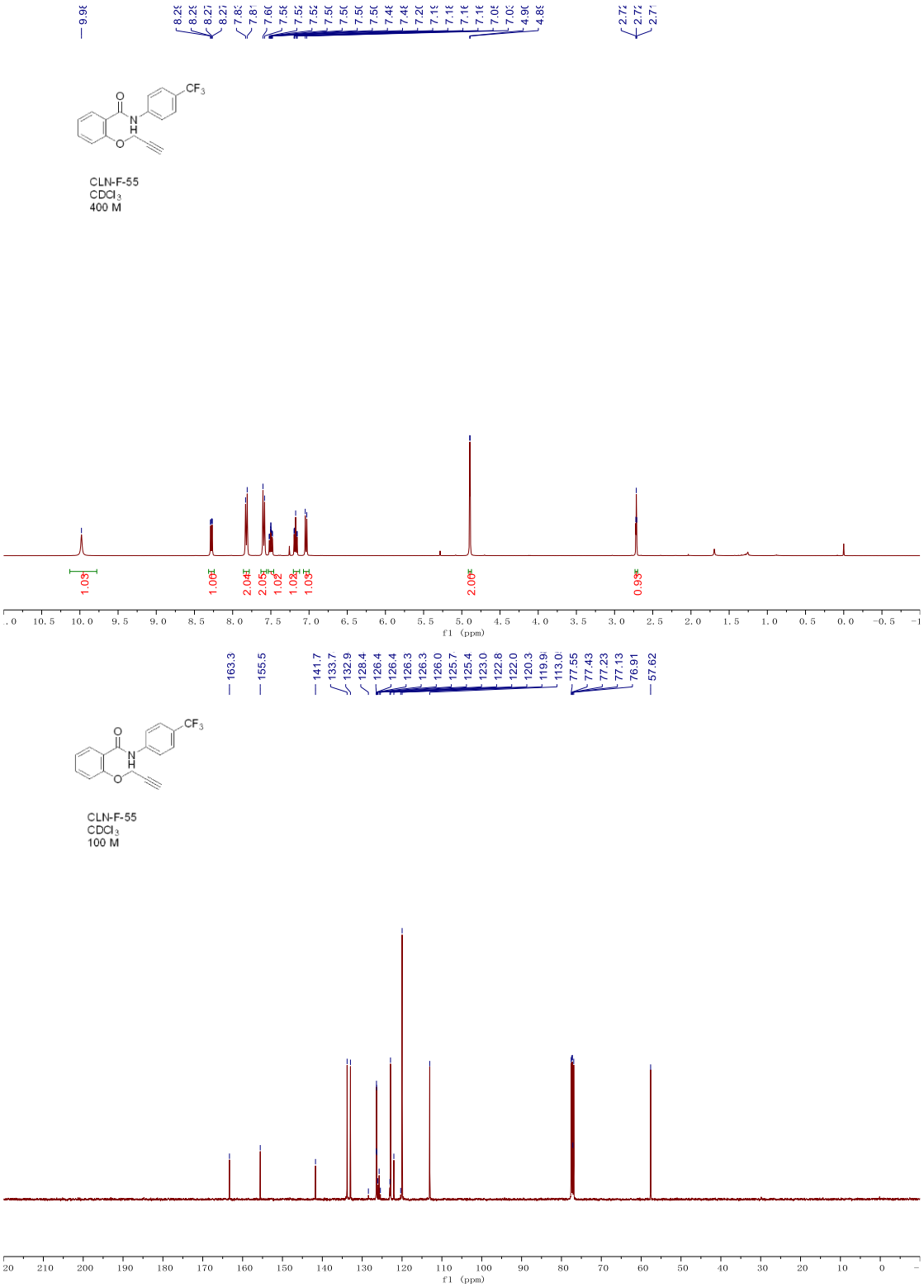
JZK-A-7-SM
$CDCl_3$
100 M



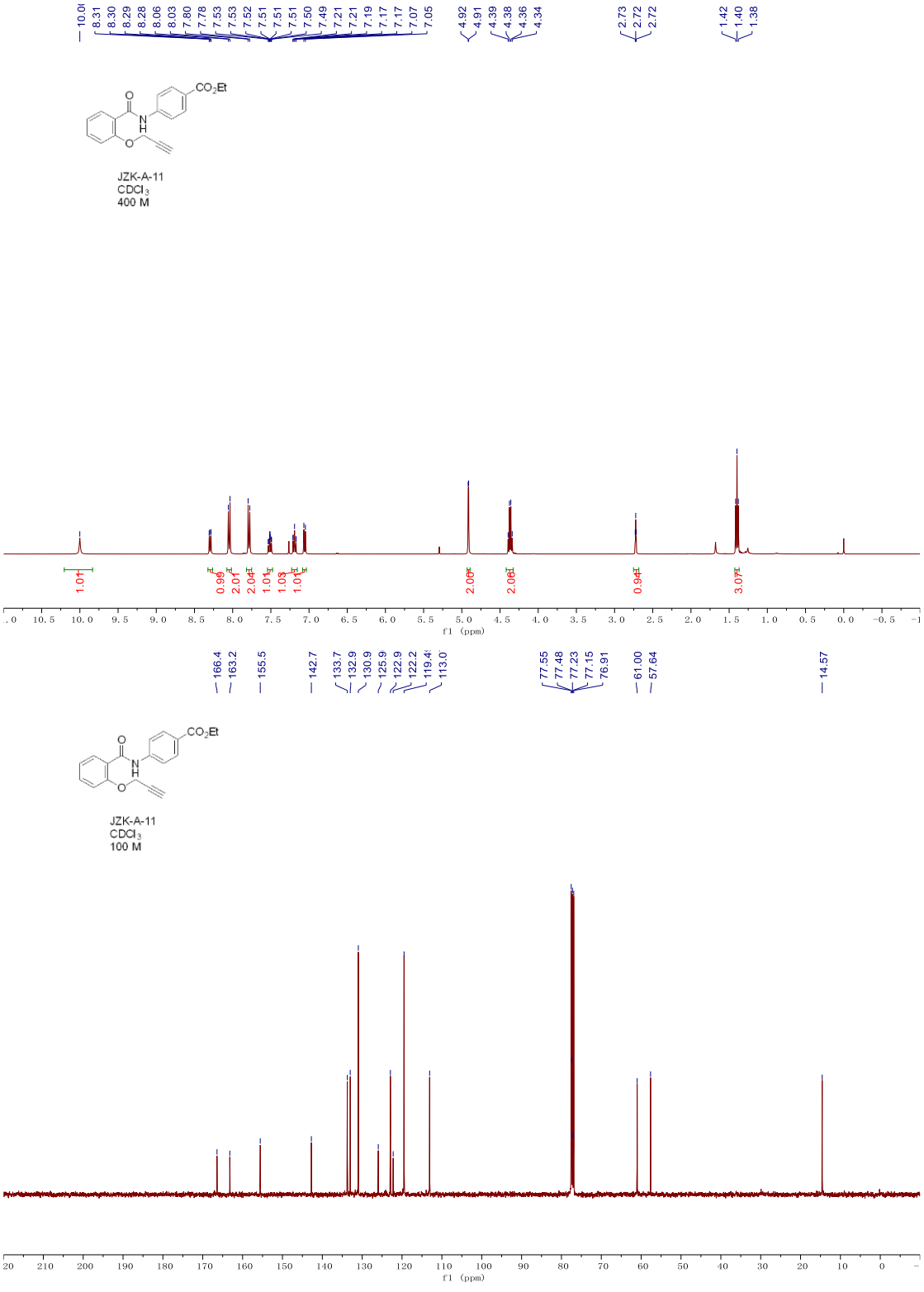
^{1}H and ^{13}C NMR spectra of compound 6f



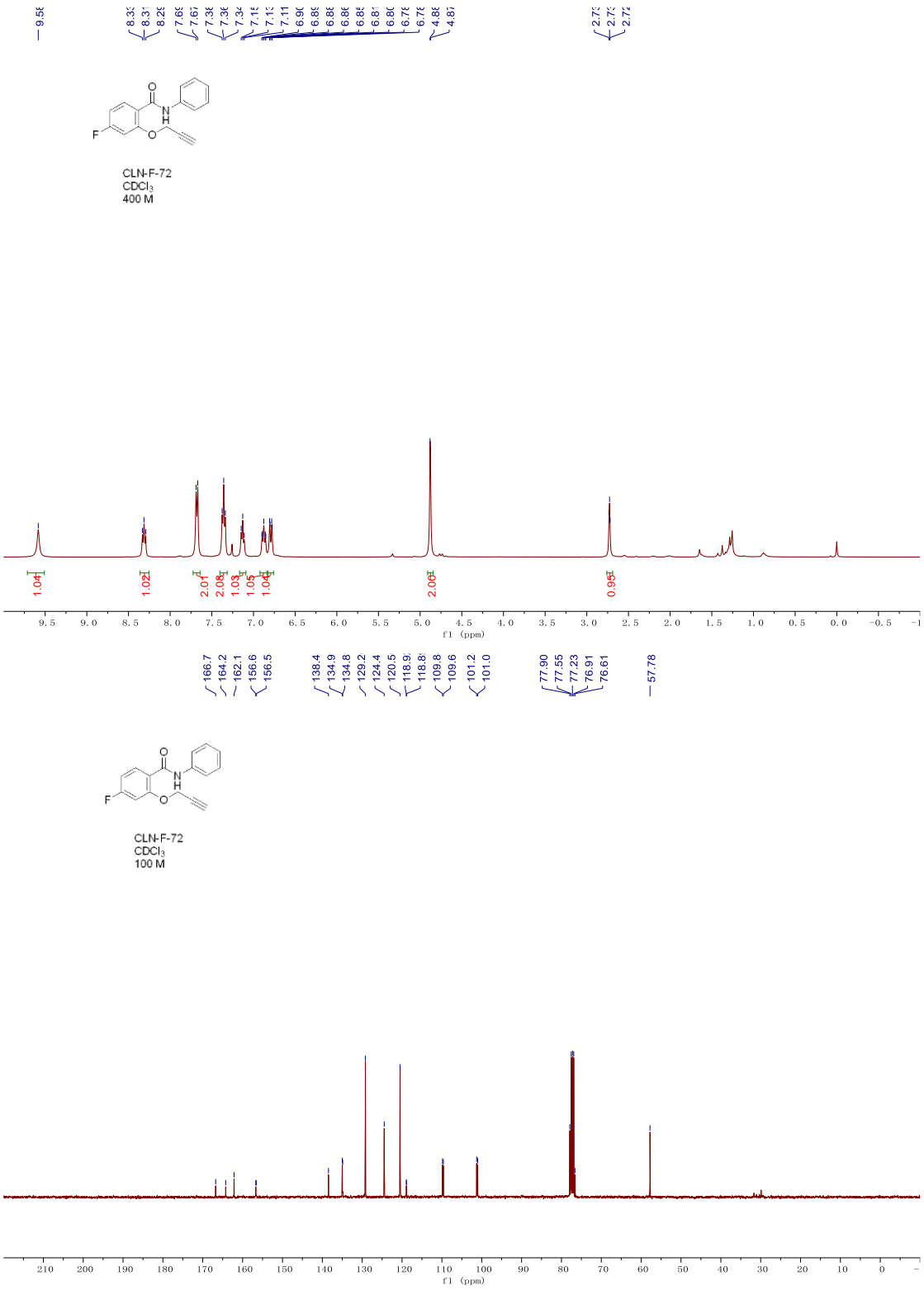
^{1}H and ^{13}C NMR spectra of compound 6g



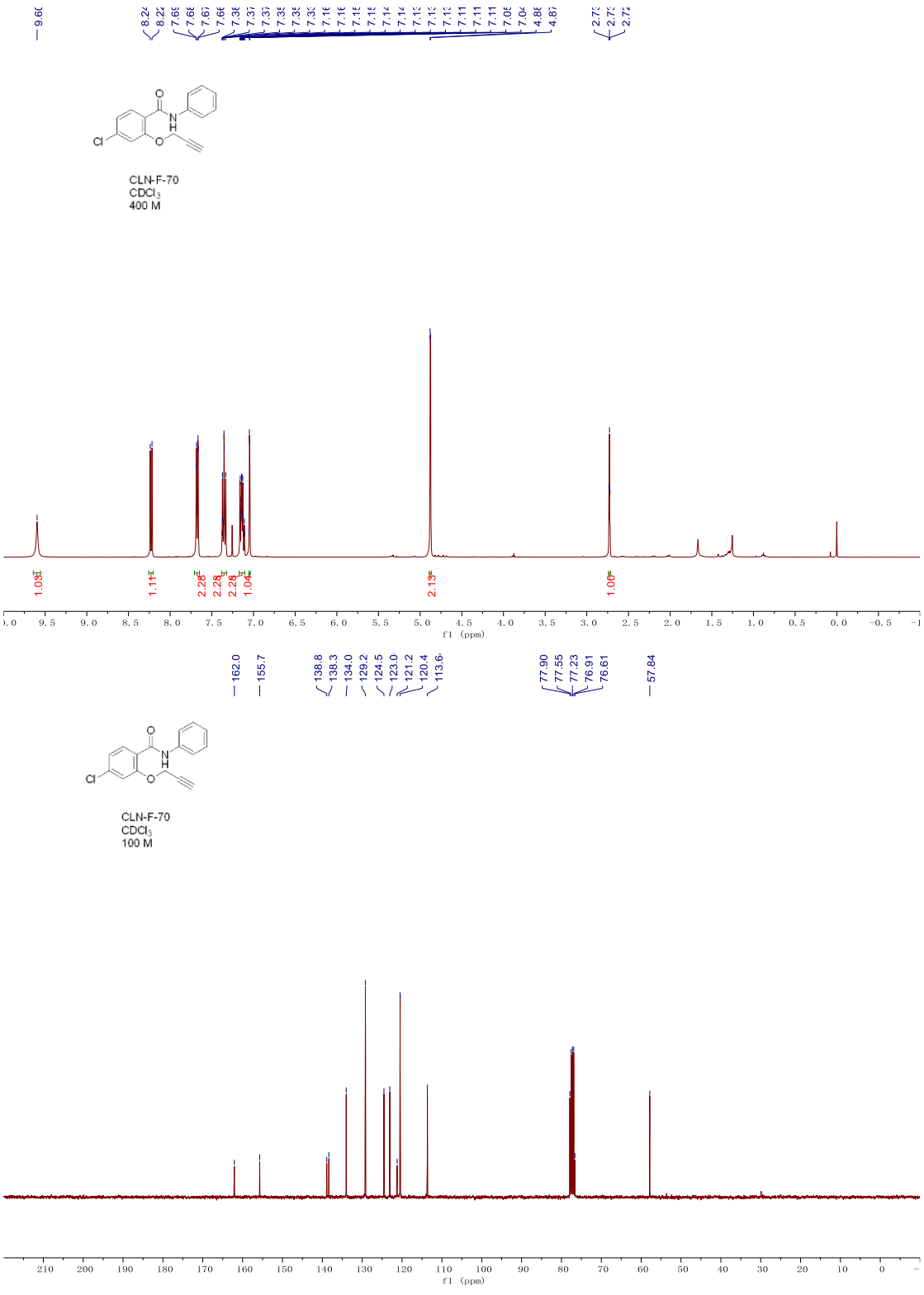
^{1}H and ^{13}C NMR spectra of compound 6h



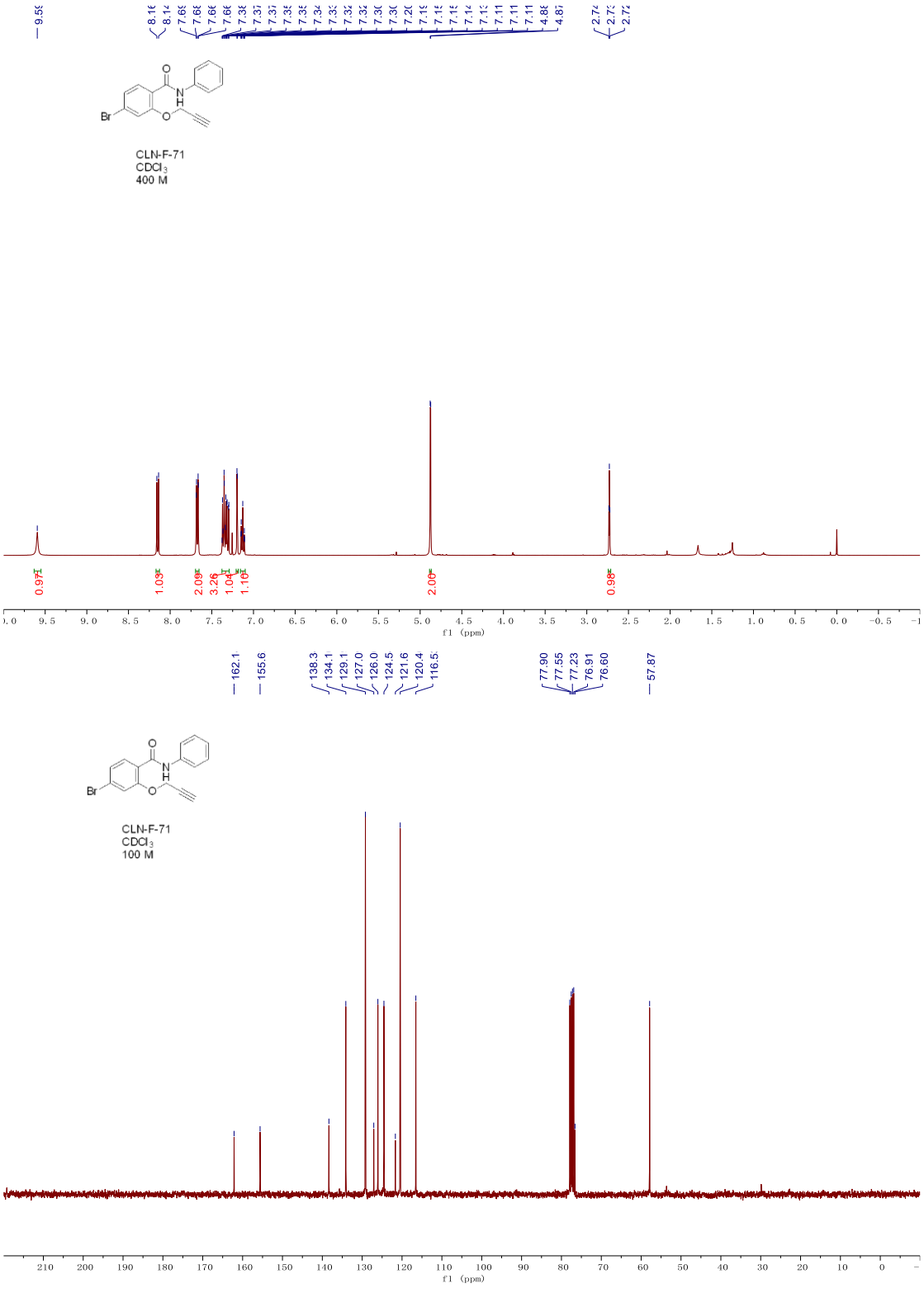
^{1}H and ^{13}C NMR spectra of compound 6p



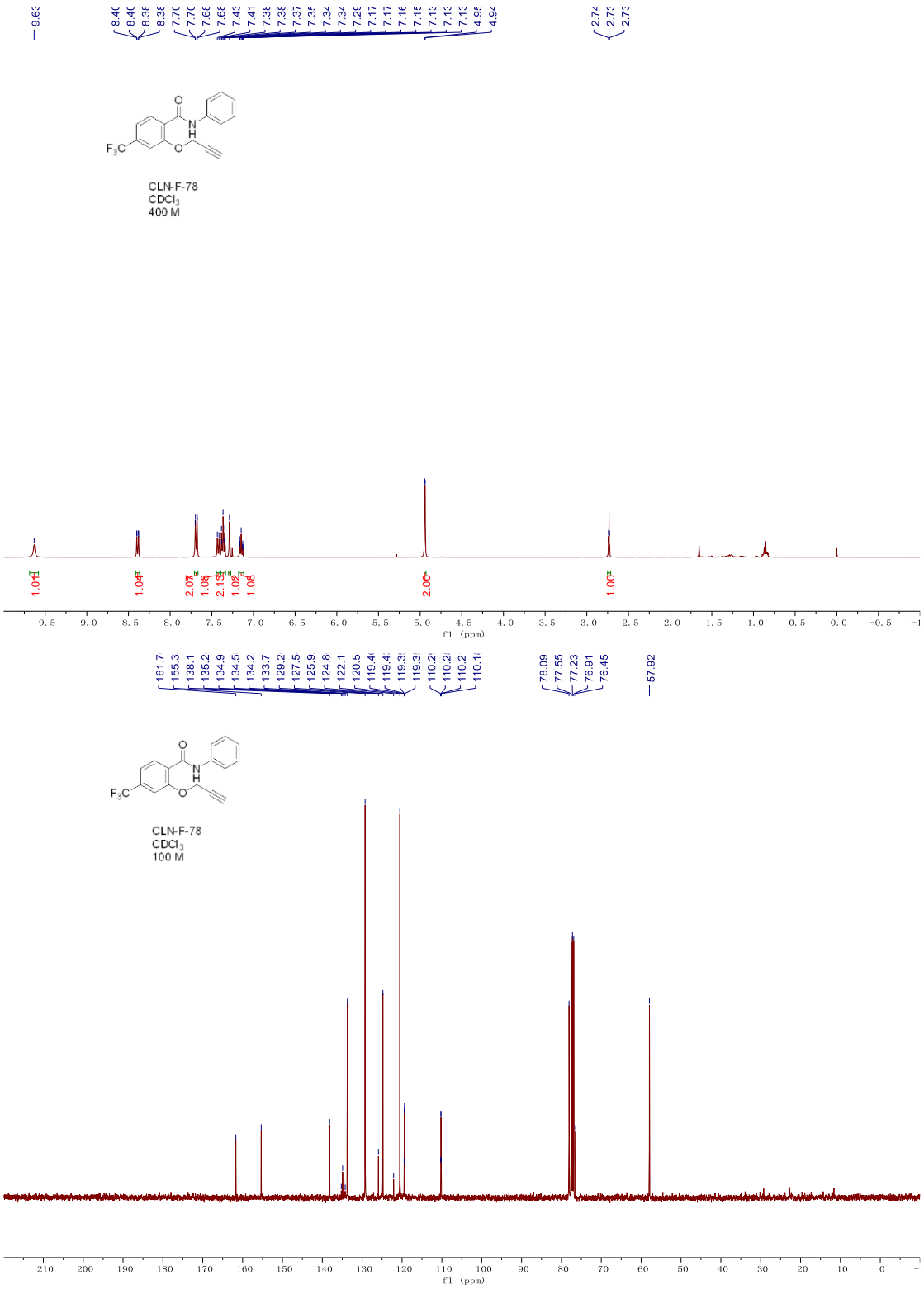
^{1}H and ^{13}C NMR spectra of compound 6q



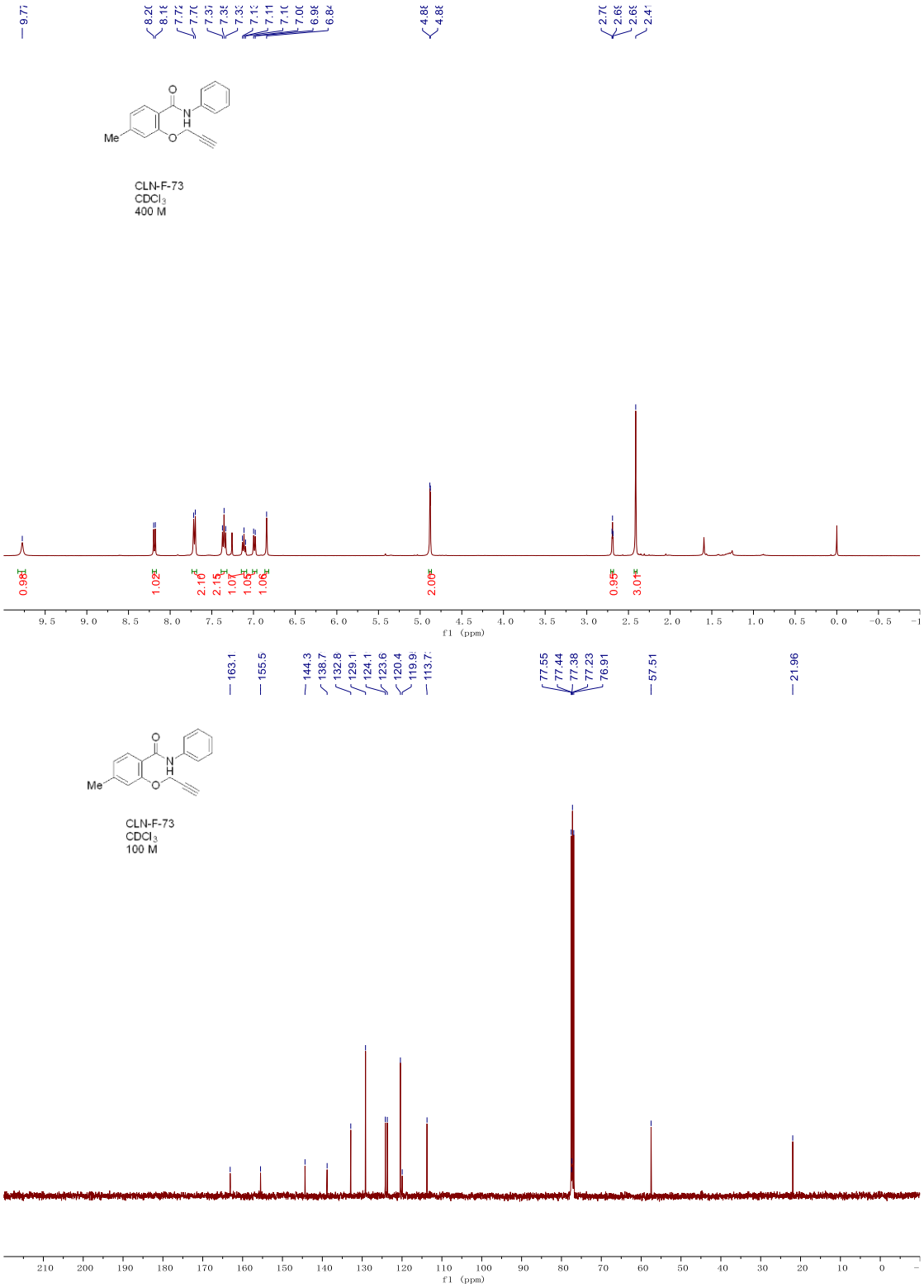
^{1}H and ^{13}C NMR spectra of compound 6r



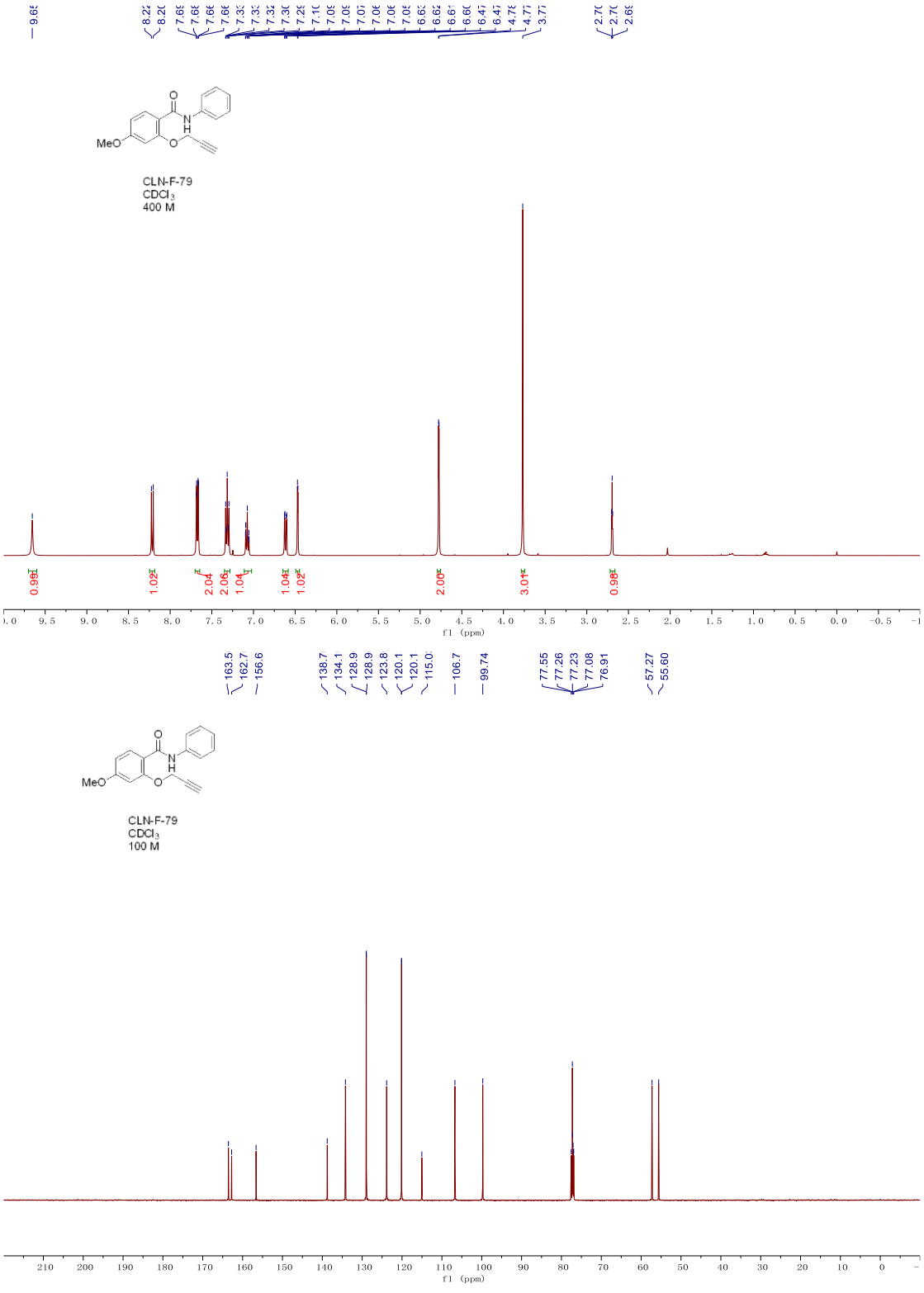
^{1}H and ^{13}C NMR spectra of compound 6s



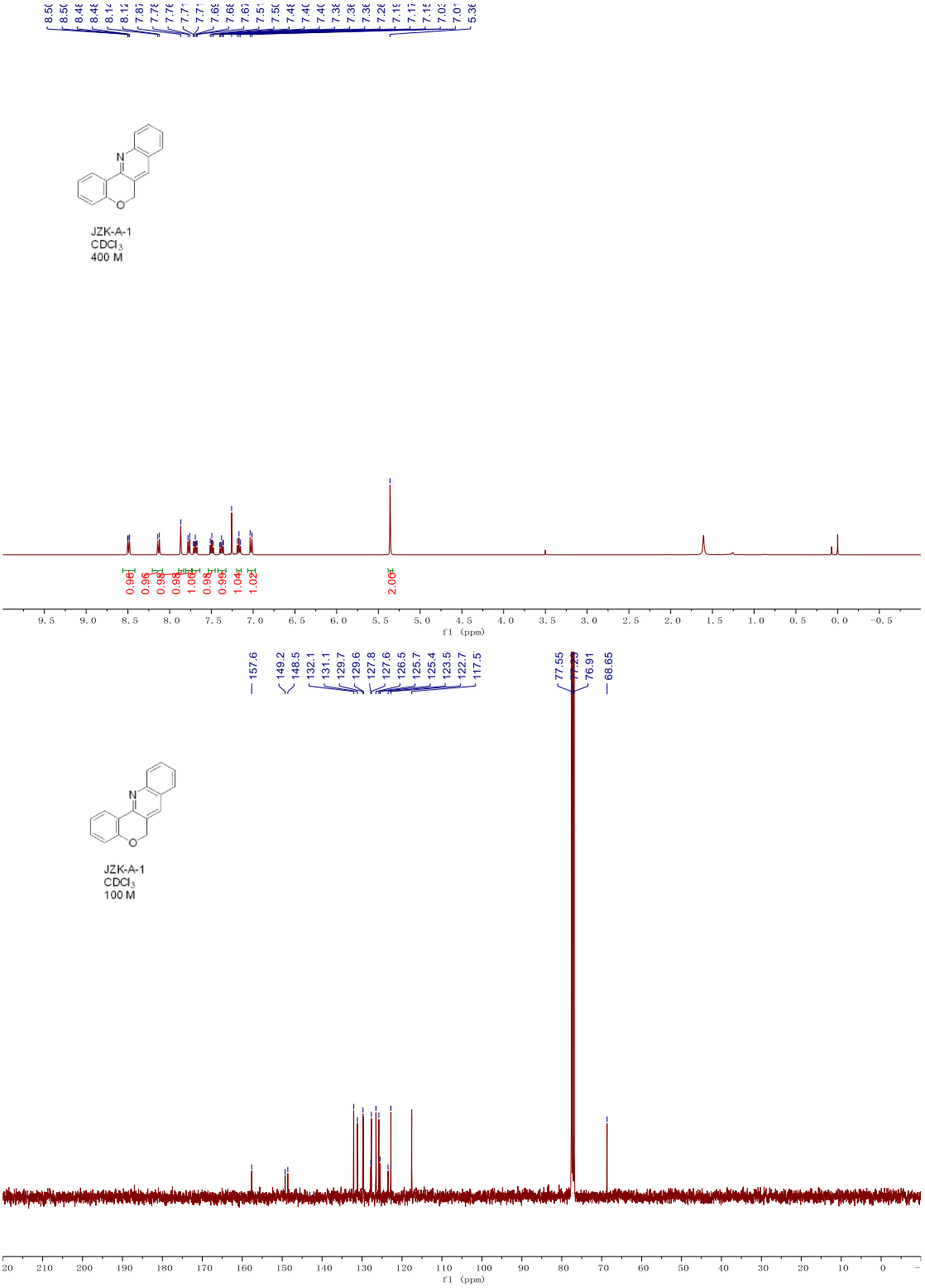
^{1}H and ^{13}C NMR spectra of compound 6t



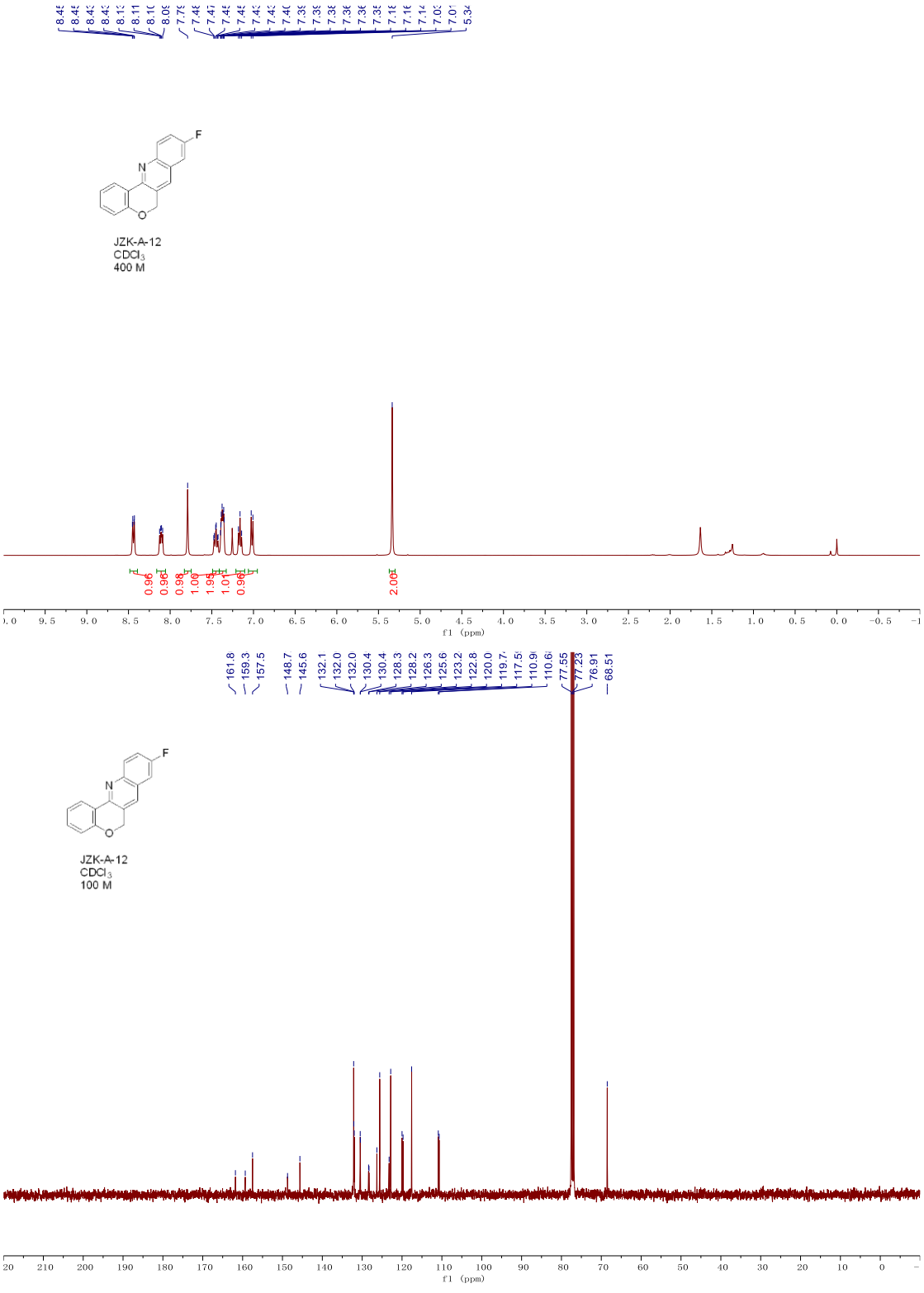
^{1}H and ^{13}C NMR spectra of compound 6u



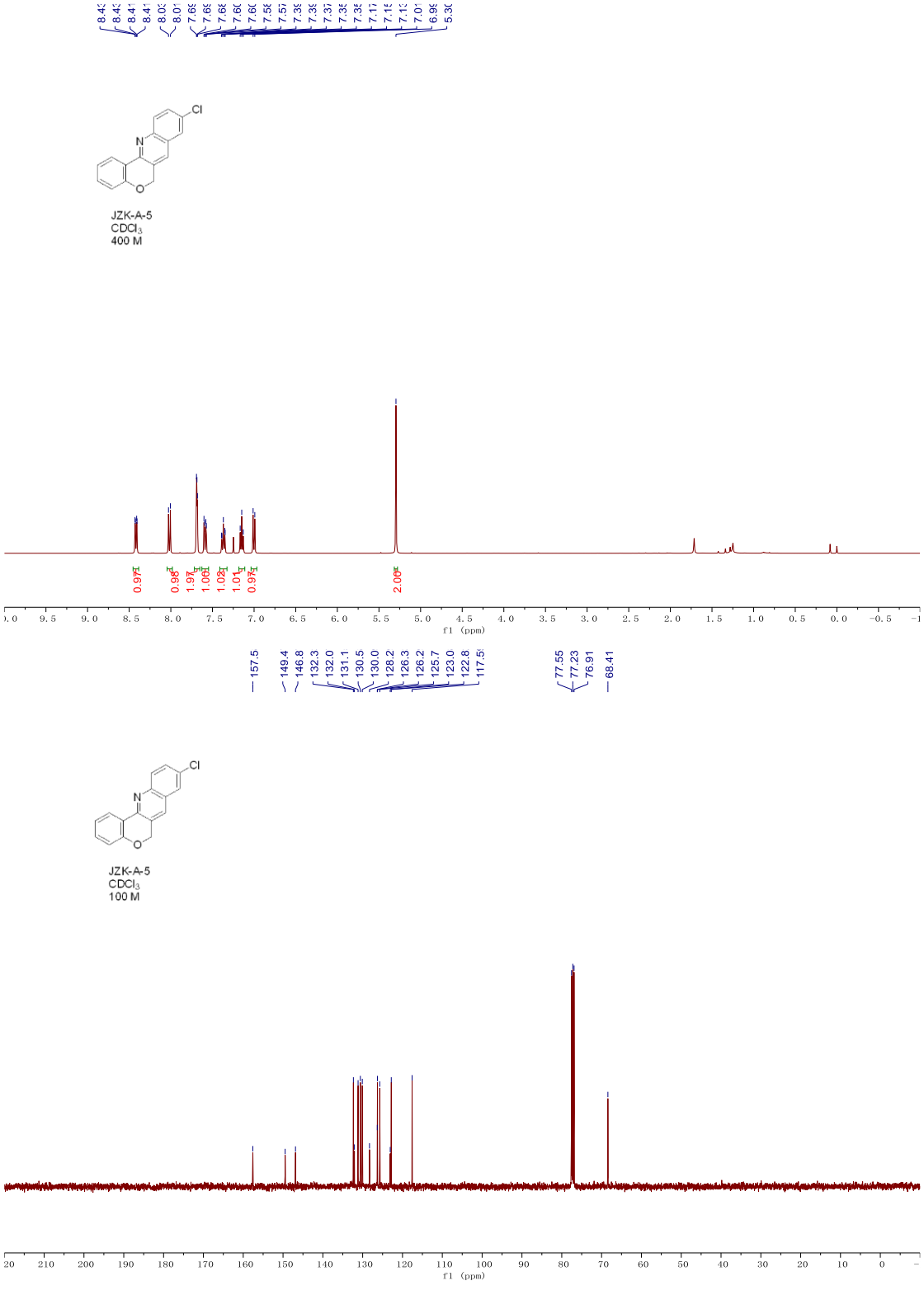
^{1}H and ^{13}C NMR spectra of compound 7a



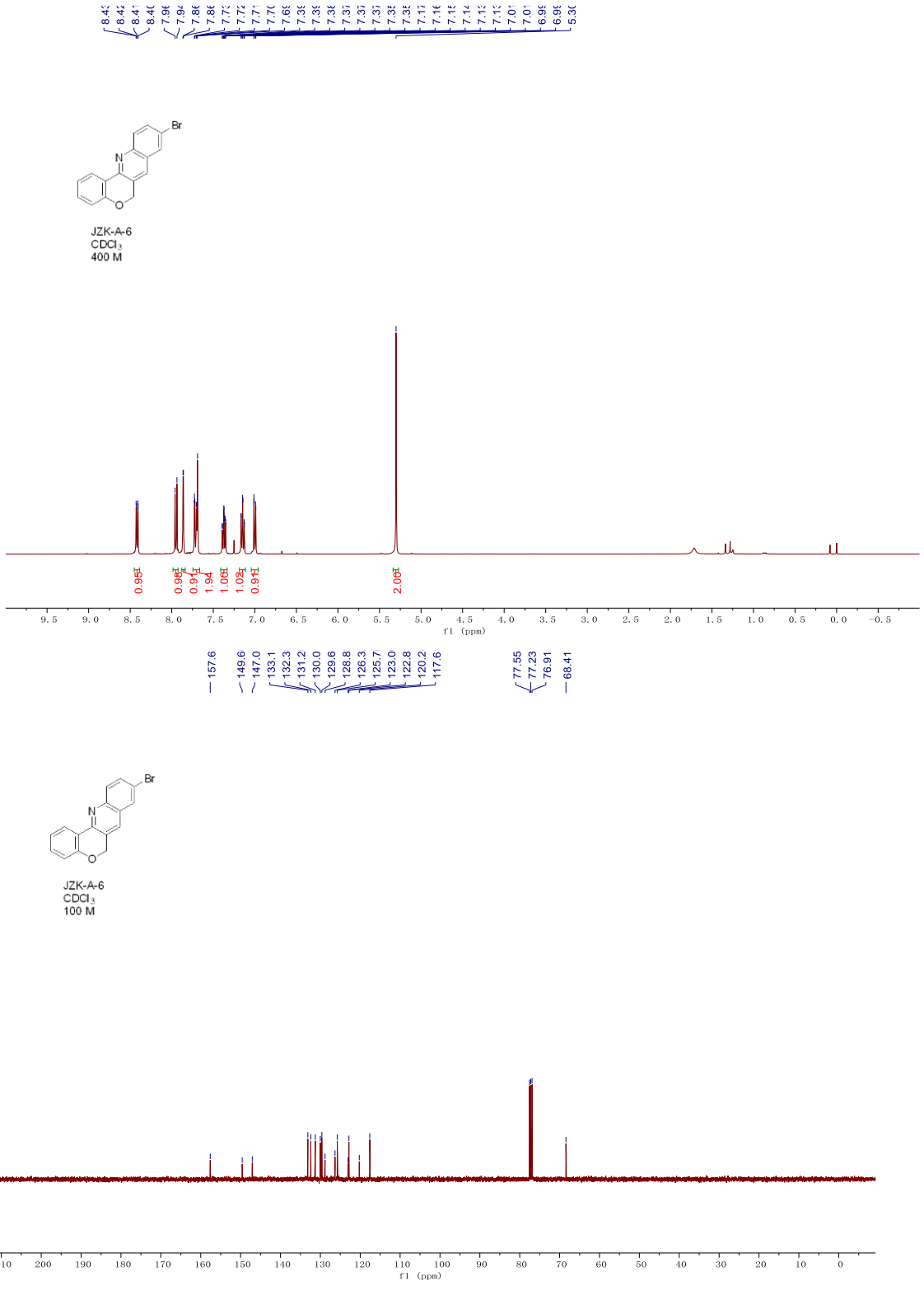
^{1}H and ^{13}C NMR spectra of compound 7b



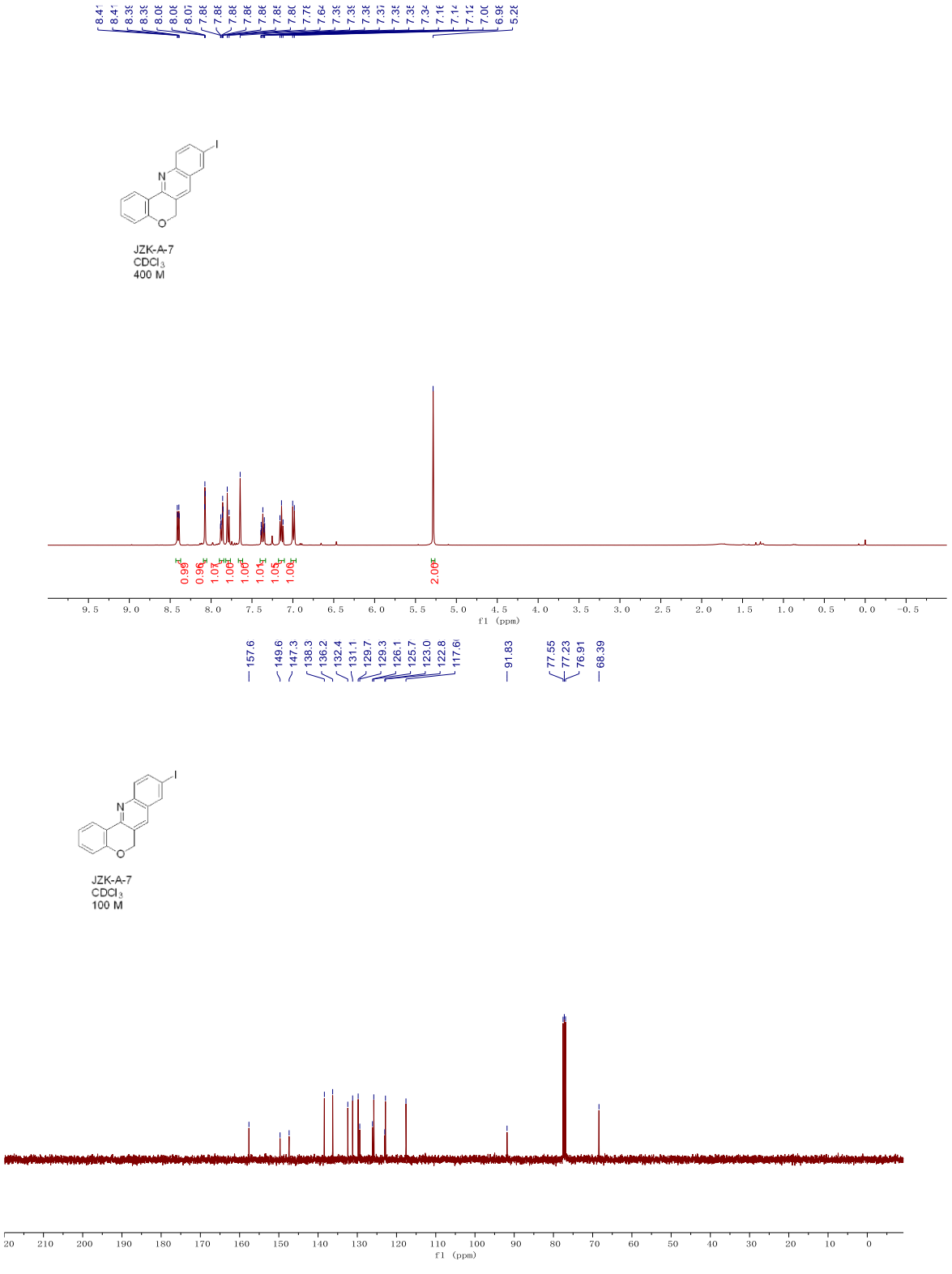
^{1}H and ^{13}C NMR spectra of compound 7c



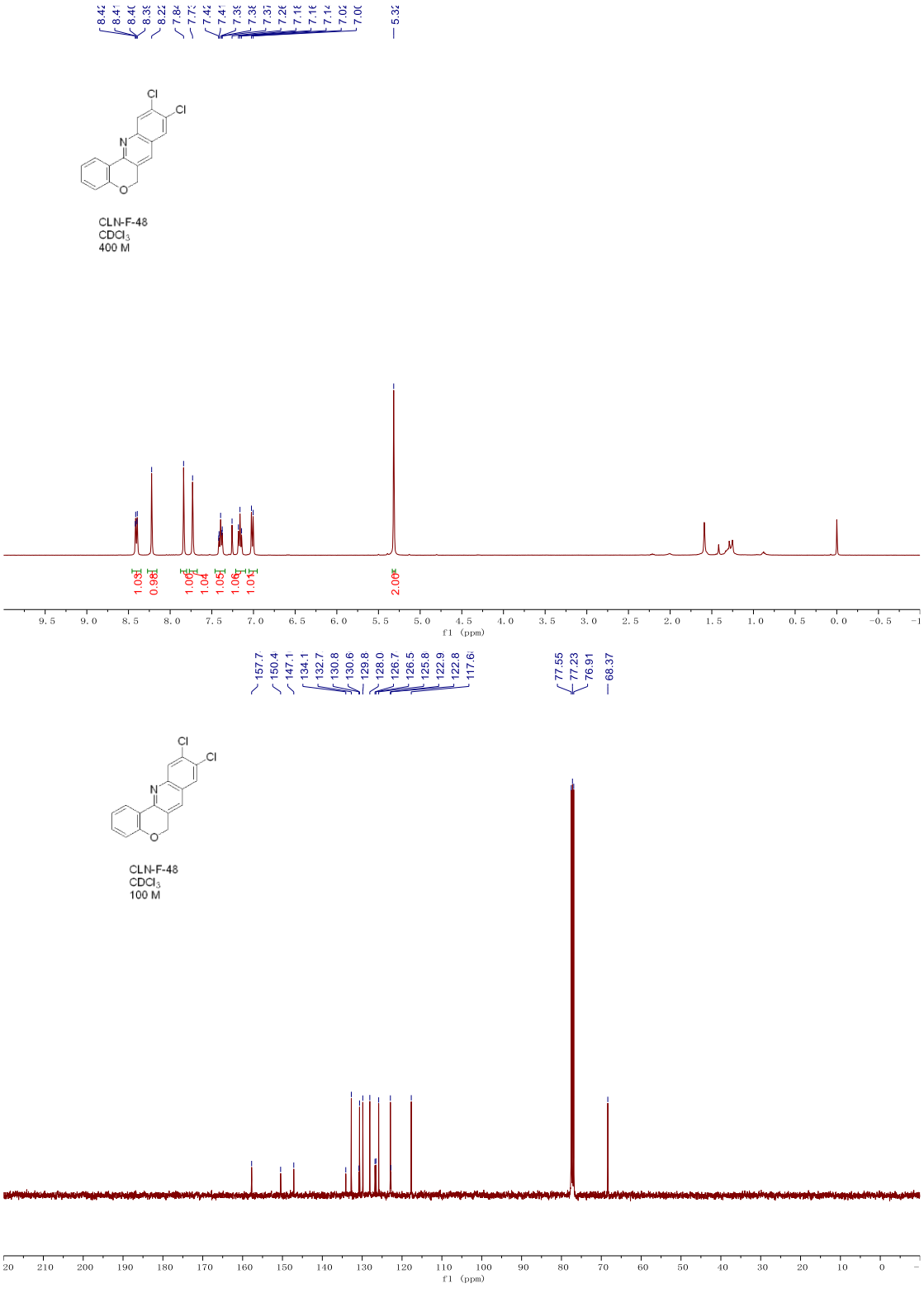
^{1}H and ^{13}C NMR spectra of compound 7d



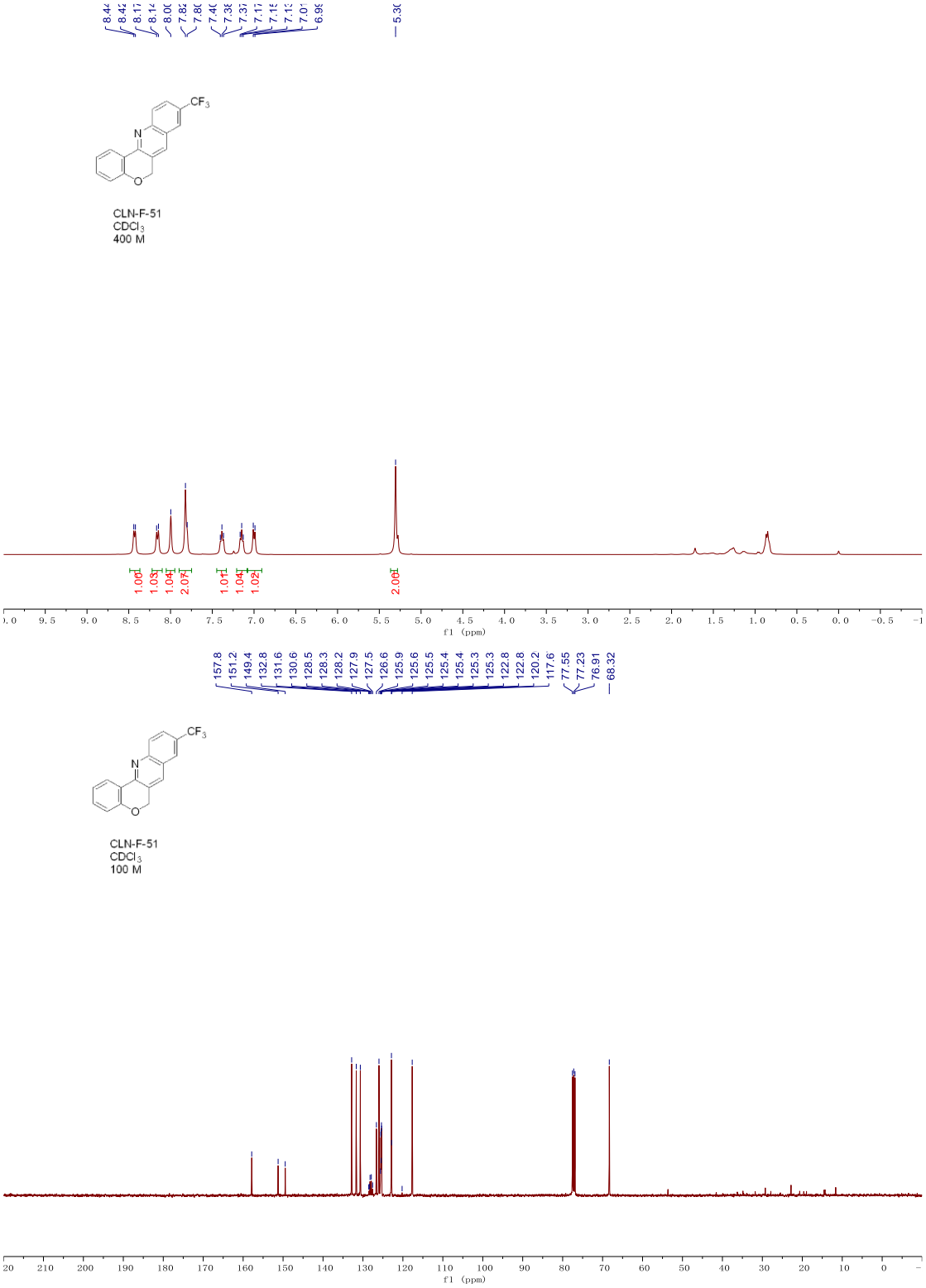
^{1}H and ^{13}C NMR spectra of compound 7e



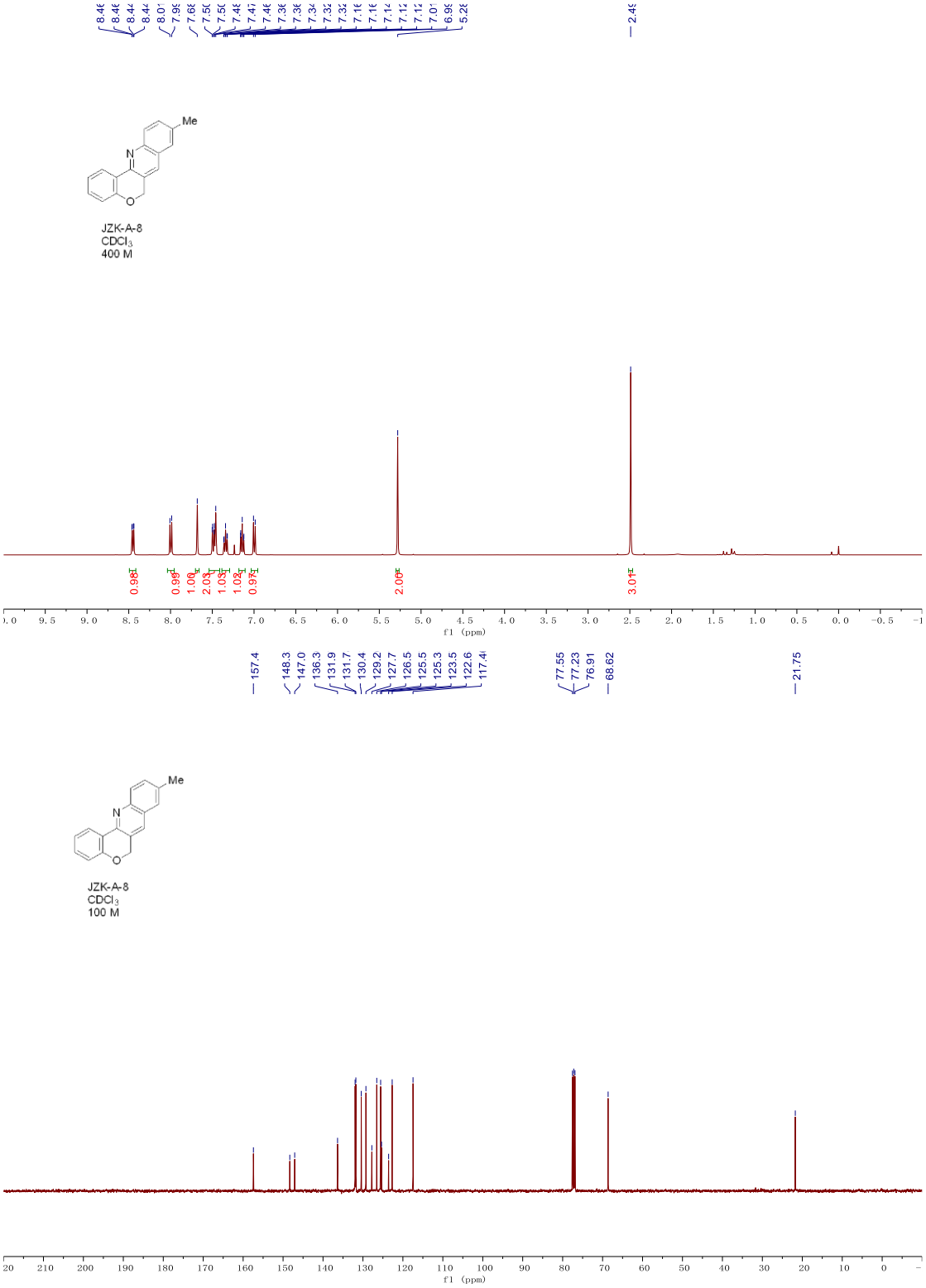
^{1}H and ^{13}C NMR spectra of compound 7f



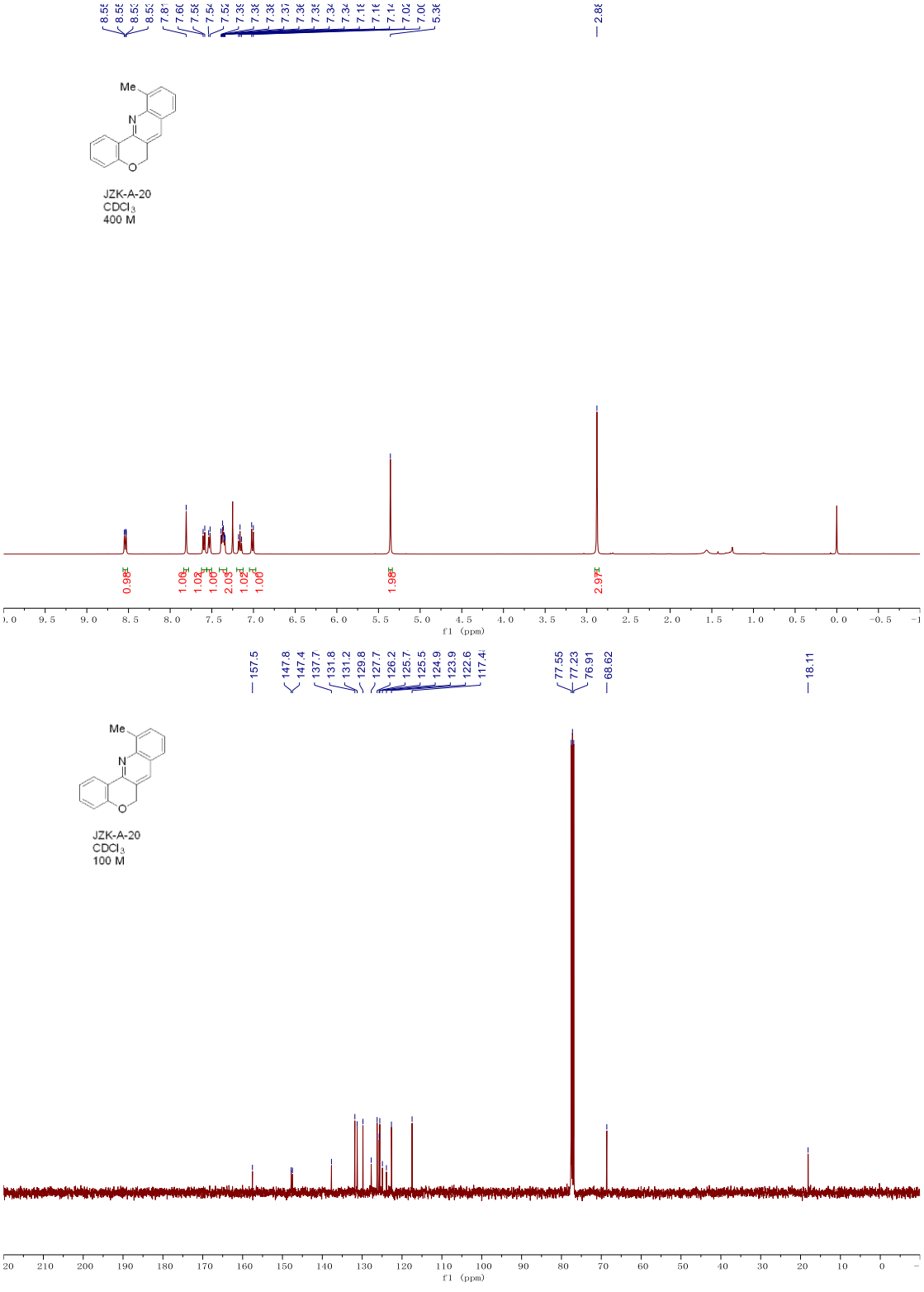
^{1}H and ^{13}C NMR spectra of compound 7g



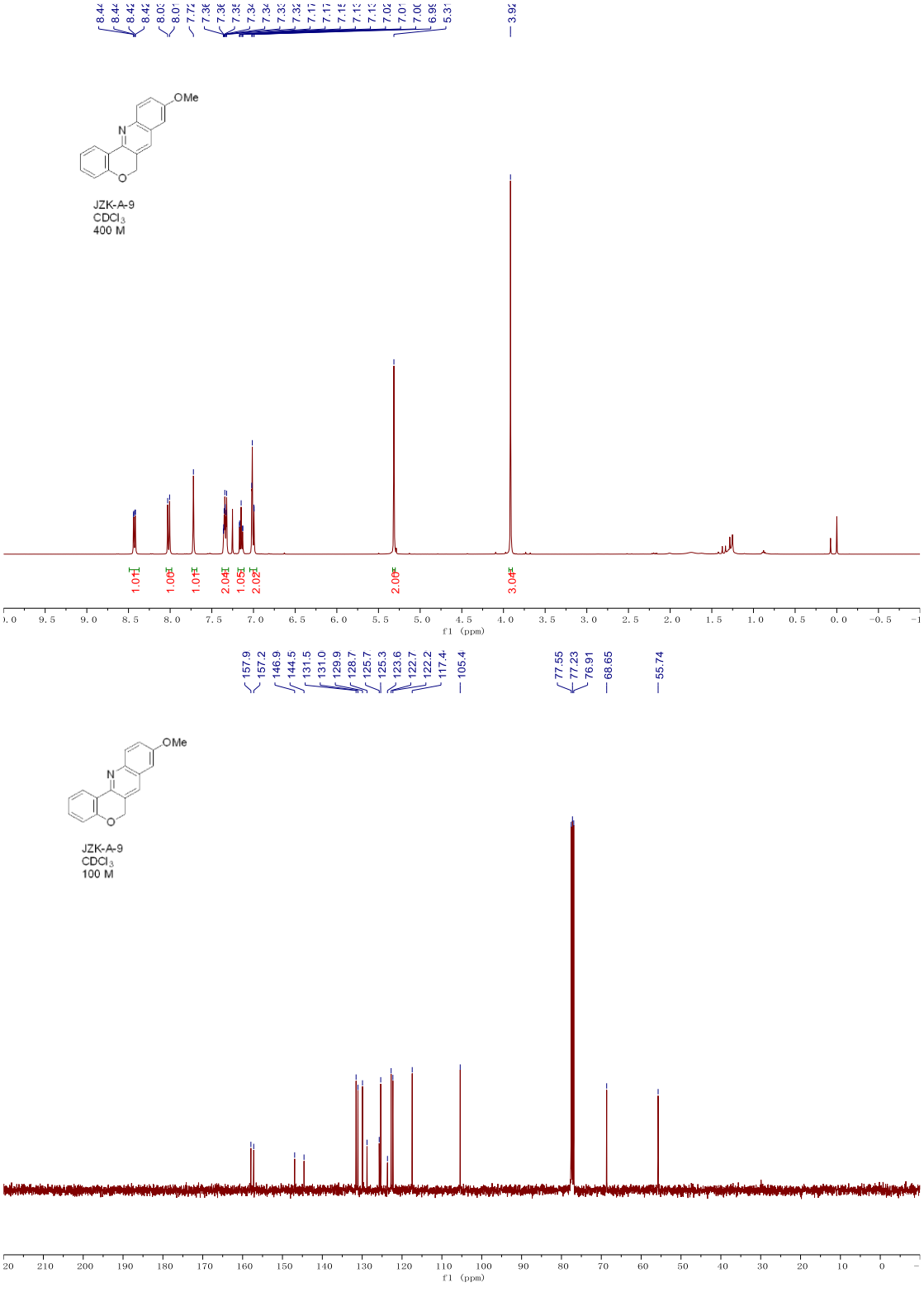
^{1}H and ^{13}C NMR spectra of compound 7j



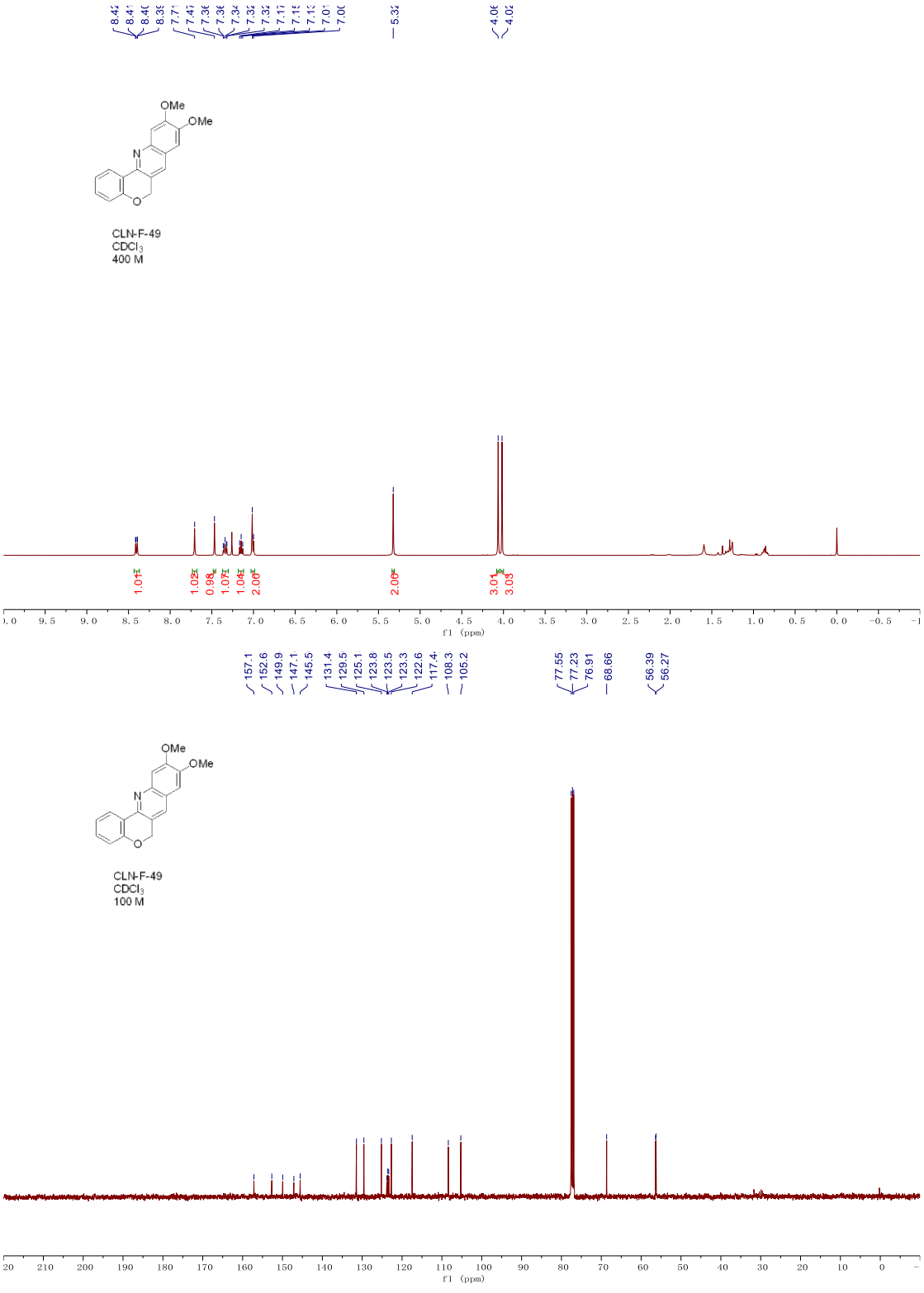
^{1}H and ^{13}C NMR spectra of compound 7n



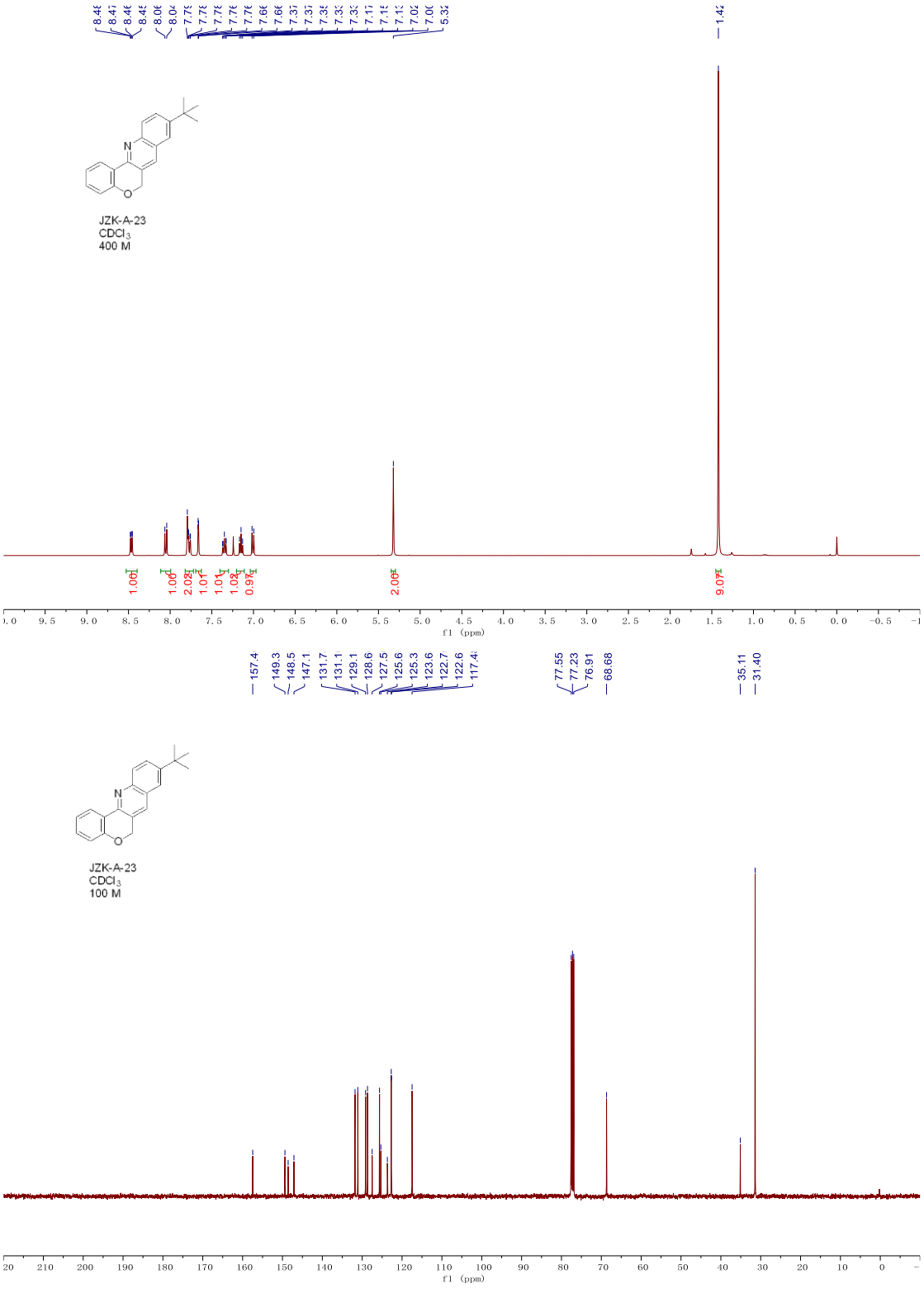
^{1}H and ^{13}C NMR spectra of compound 7l



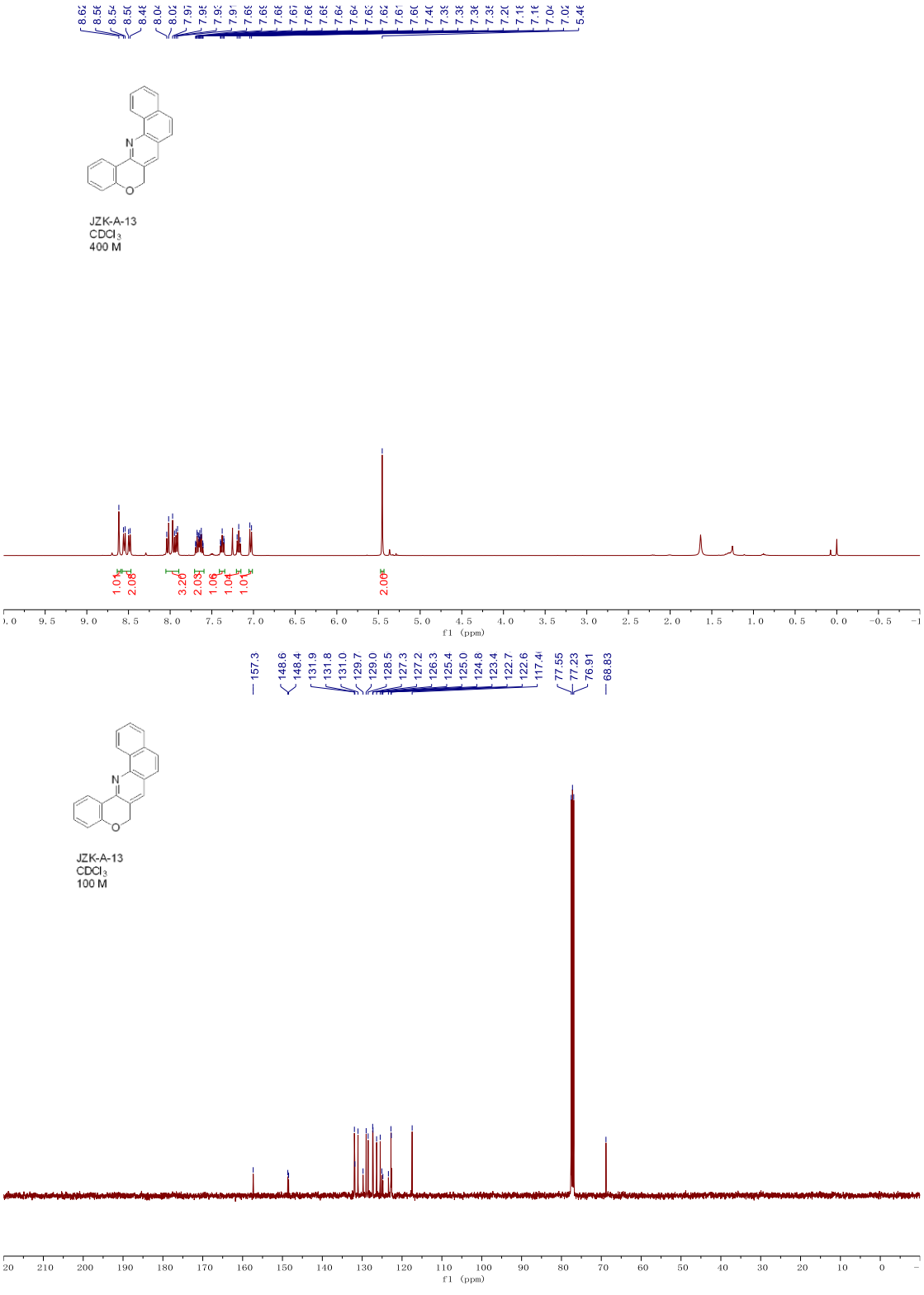
^{1}H and ^{13}C NMR spectra of compound 7m



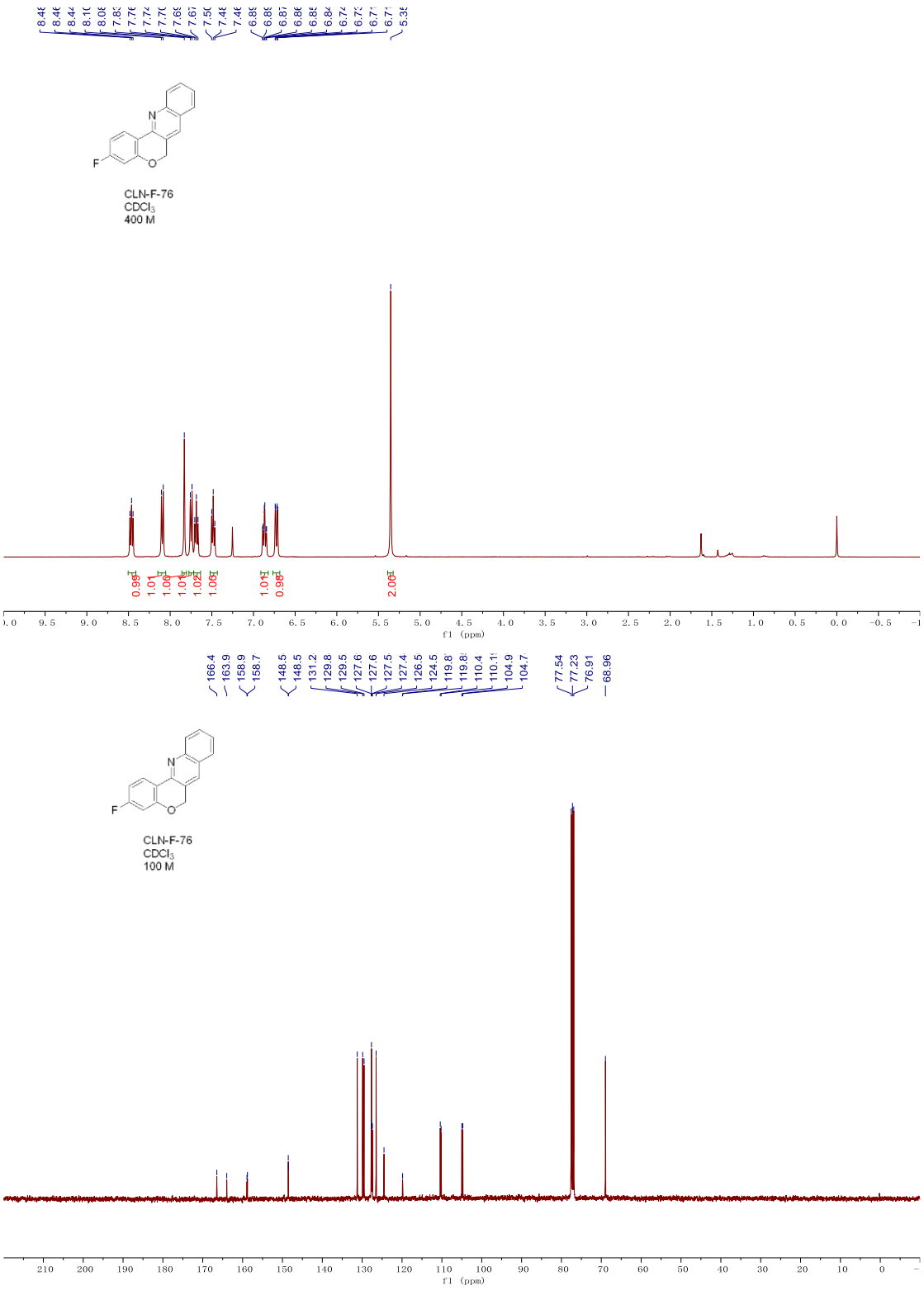
^{1}H and ^{13}C NMR spectra of compound 7k



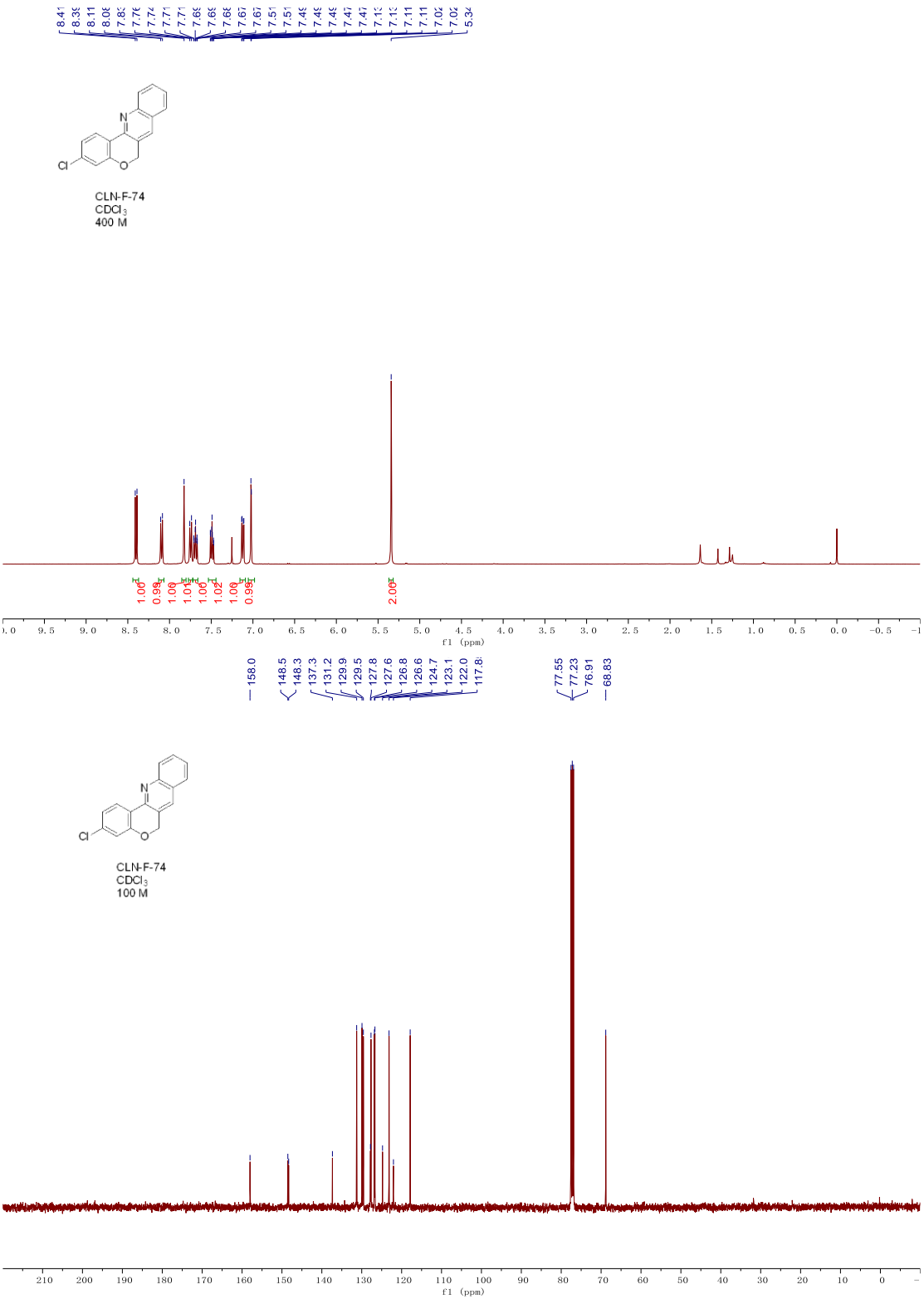
^{1}H and ^{13}C NMR spectra of compound 7o



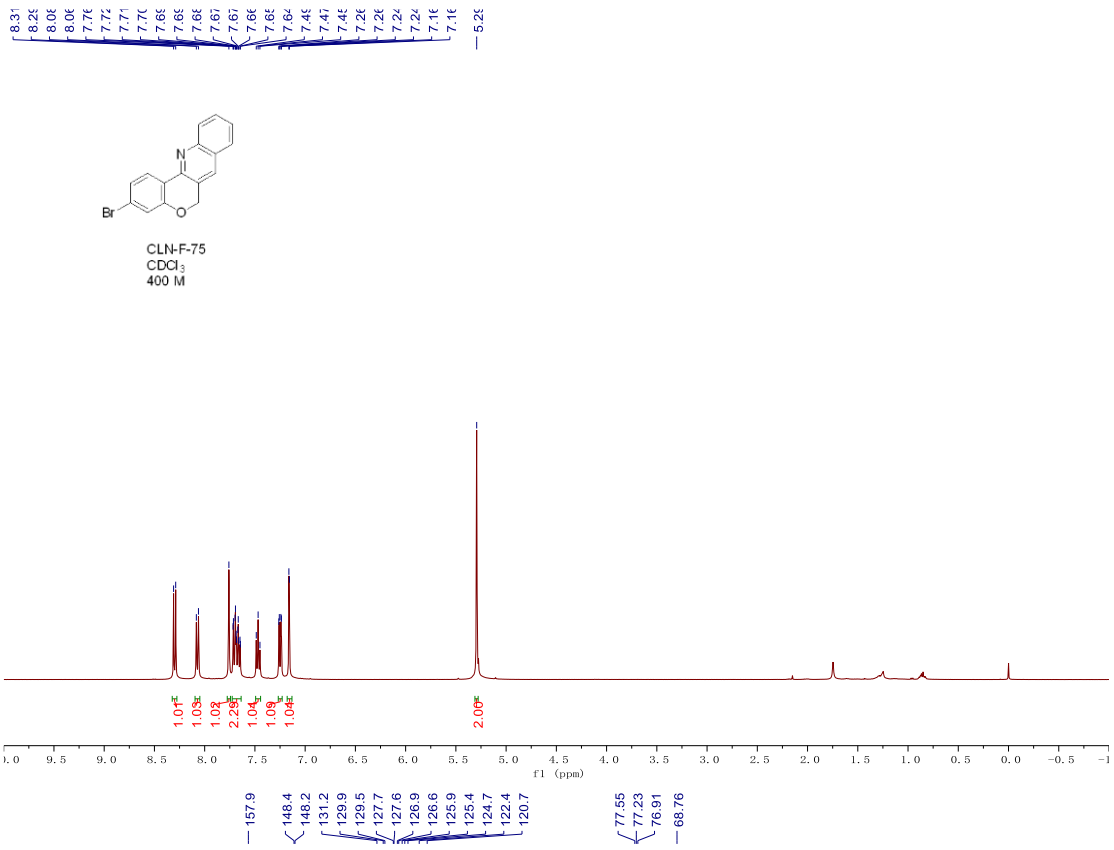
^{1}H and ^{13}C NMR spectra of compound 7p

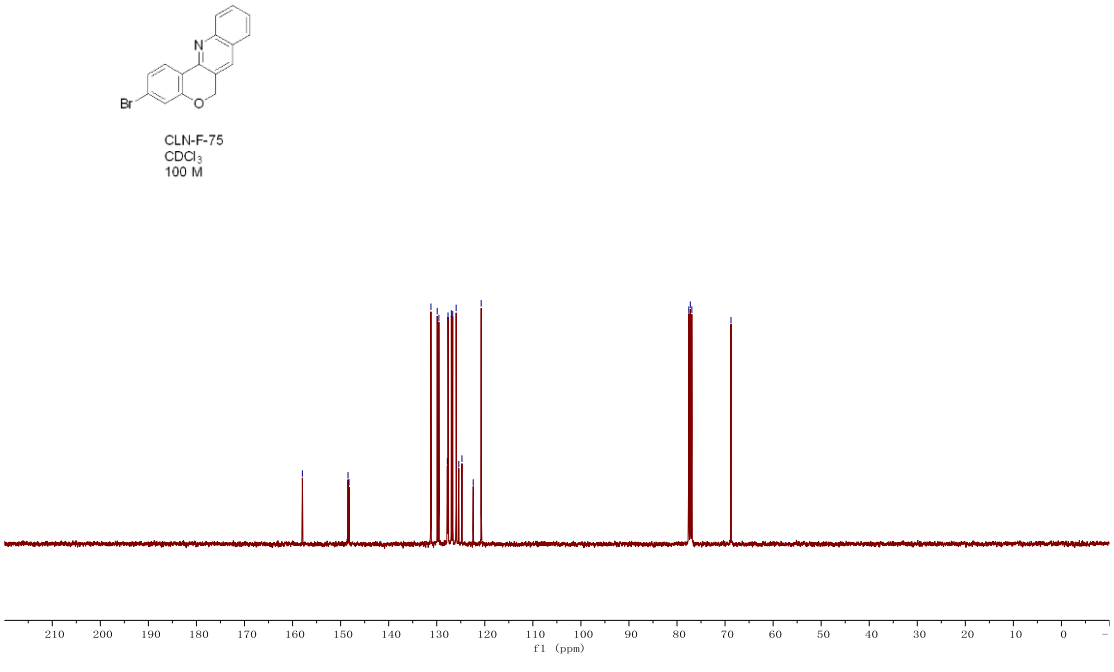


^{1}H and ^{13}C NMR spectra of compound 7q

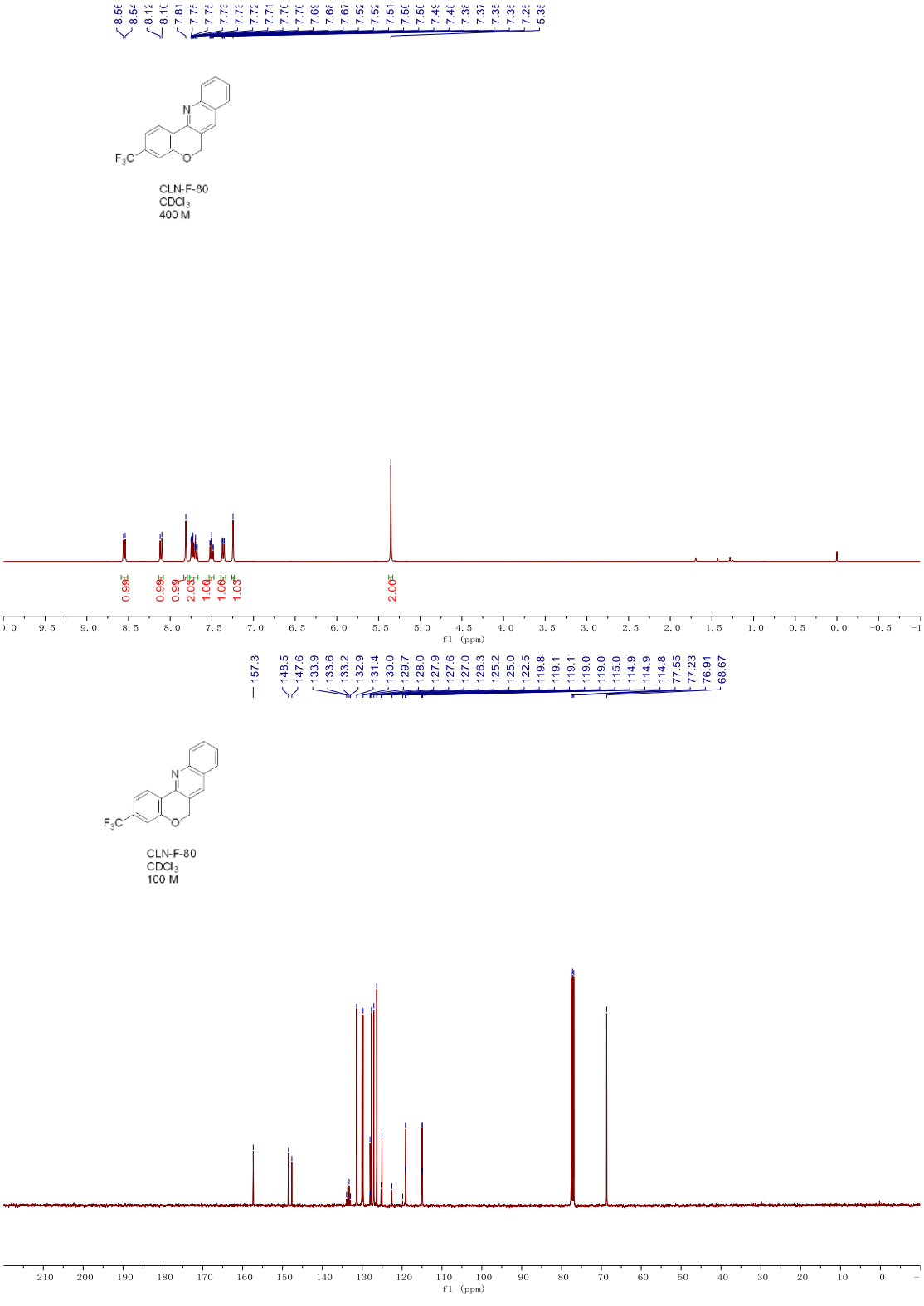


^{1}H and ^{13}C NMR spectra of compound 7r

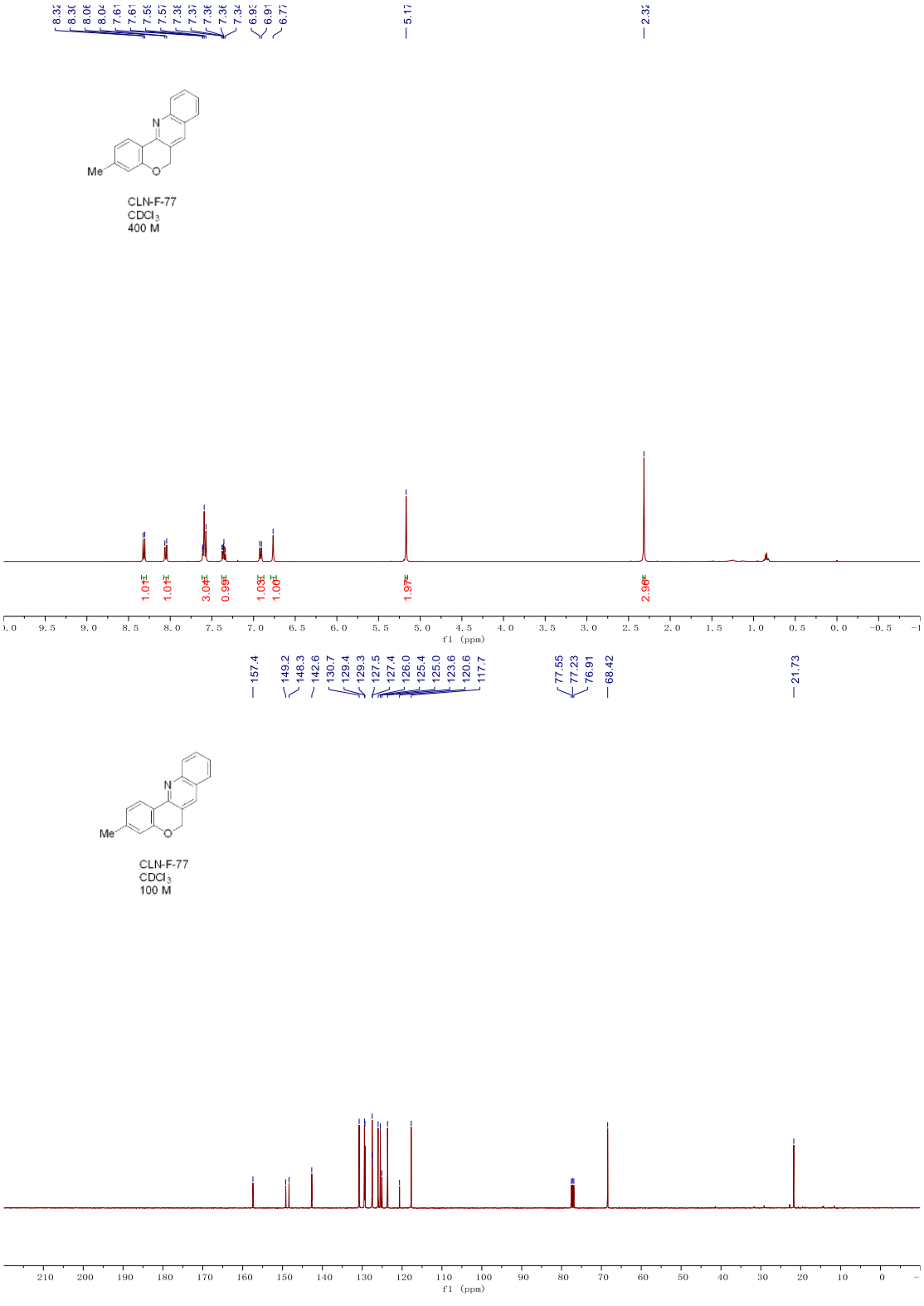




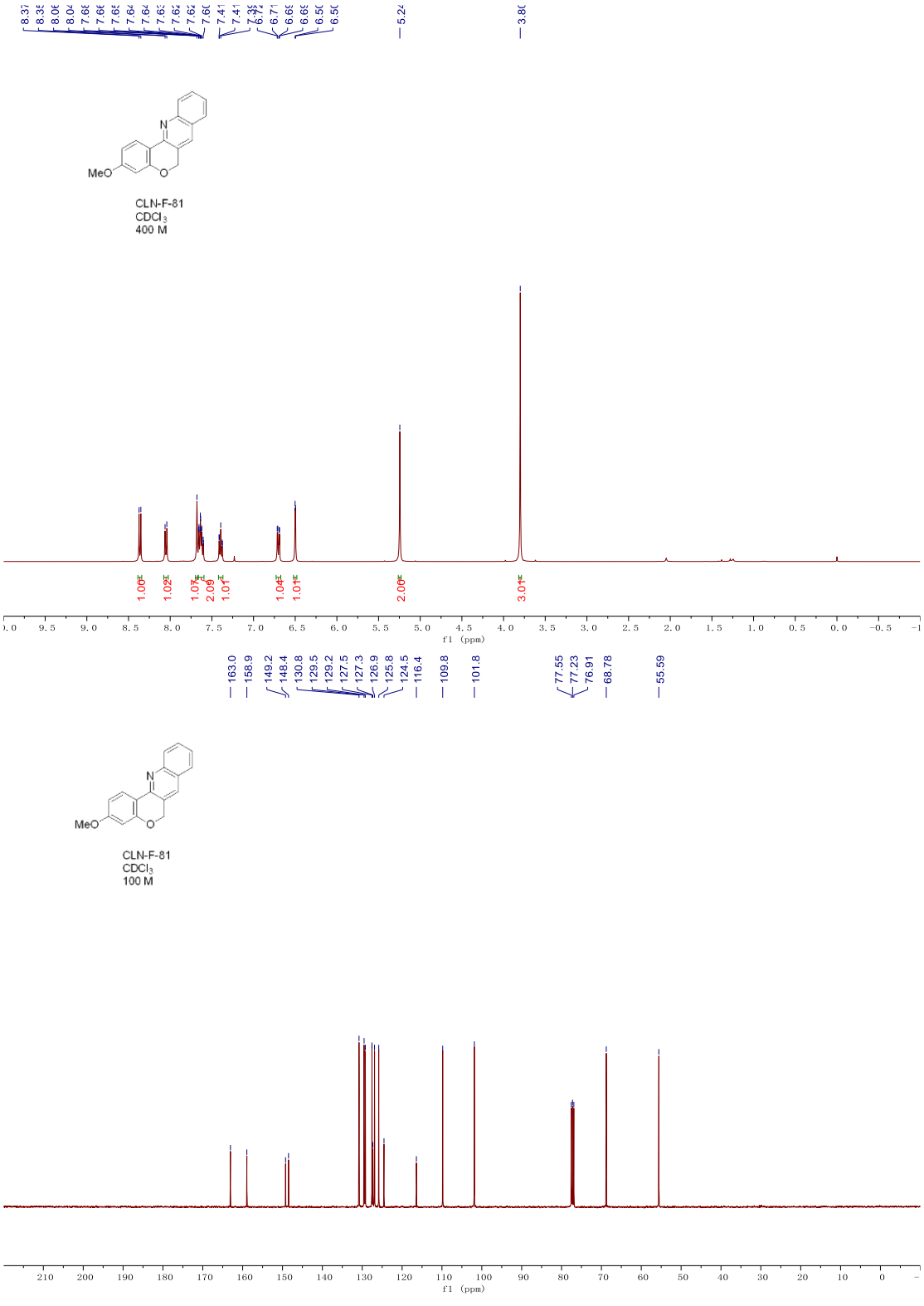
^{1}H and ^{13}C NMR spectra of compound 7s



^{1}H and ^{13}C NMR spectra of compound 7t



^{1}H and ^{13}C NMR spectra of compound 7u



CCDC 2335879, 2335878 contain the crystallographic data for compounds **7g**, **7u**. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/structures.

Single crystals of $C_{17}H_{10}F_3NO$ [**7g**] were obtained by slow evaporation of an CH_2Cl_2/petroleum ether solution of 7g. A suitable crystal was selected and mounted on a XtaLAB Synergy, Dualflex, HyPix diffractometer. The crystal was kept at 100.0(3) K during data collection. Using Olex2, the structure was solved with the olex2.solve structure solution program using Charge Flipping and refined with the olex2.refine refinement package using Gauss-Newton minimisation.

Crystal Data for $C_{17}H_{10}F_3NO$ [**7g**, **CCDC 2335879**] (M =301.270 g/mol): monoclinic, space group $P2_1/c$ (no. 14), a = 14.2286(3) Å, b = 6.9712(1) Å, c = 13.7710(3) Å, β = 108.636(2)°, V = 1294.33(5) Å^3, Z = 4, T = 100.0(3) K, μ(Cu Kα) = 1.083 mm^{-1}, *Dcalc* = 1.546 g/cm^3, 13628 reflections measured (6.56° ≤ 2Θ ≤ 155.04°), 2694 unique (R_{int} = 0.0339, R_{sigma} = 0.0266) which were used in all calculations. The final R_1 was 0.0428 (I>=2u(I)) and wR_2 was 0.1250 (all data).

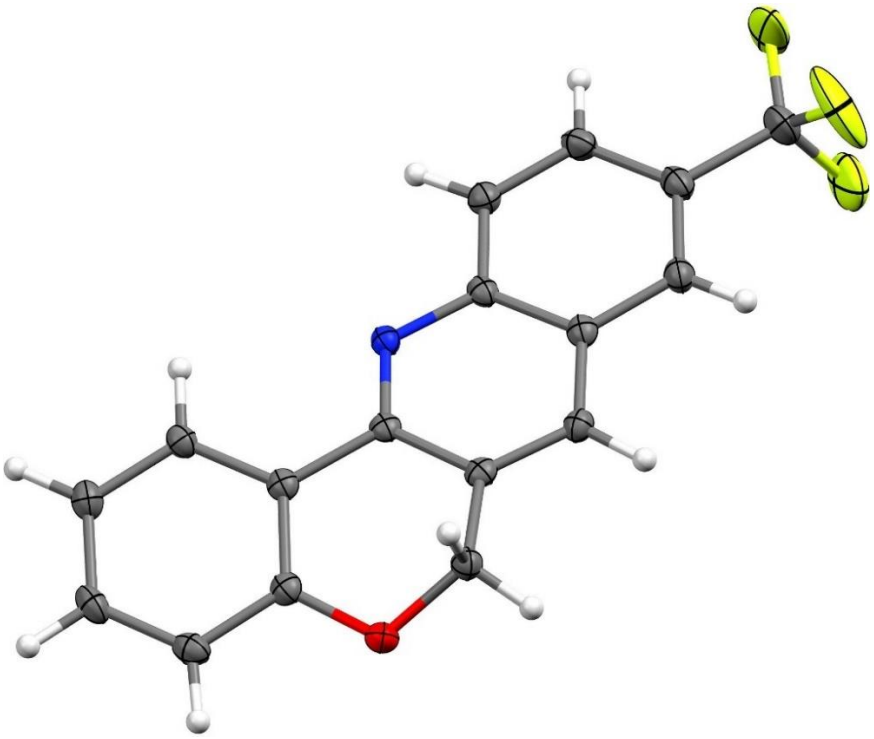


Figure S1. The single crystal X-ray structure of compound **7g** [**CCDC 2335879**]. Thermal ellipsoids are drawn at the 50% probability level.

Single crystals of $C_{17}H_{13}NO_2$ [**7u**] were obtained by slow evaporation of an CH_2Cl_2/petroleum ether solution of **7u**. A suitable crystal was selected and mounted on a XtaLAB Synergy, Dualflex, HyPix diffractometer. The crystal was kept at 128(20) K during data collection. Using Olex2, the structure was solved with the olex2.solve structure solution program using Charge Flipping and refined with the olex2.refine refinement package using Gauss-Newton minimisation.

Crystal Data for $C_{17}H_{13}NO_2$ [**7u**, **CCDC 2335878**] (M =263.298 g/mol): monoclinic, space group $P2_1/c$ (no. 14), a = 10.8552(5) Å, b = 12.8645(4) Å, c = 9.6121(4) Å, β = 107.657(5)°, V = 1279.06(10) Å^3, Z = 4, T = 128(20) K, μ(Cu Kα) = 0.725 mm^{-1}, *Dcalc* = 1.367 g/cm^3, 7370 reflections measured (8.54° ≤ 2Θ ≤ 154.42°), 2550 unique (R_{int} = 0.0397, R_{sigma} = 0.0338) which were used in all calculations. The final R_1 was 0.0626 (I>=2u(I)) and wR_2 was 0.2063 (all data).

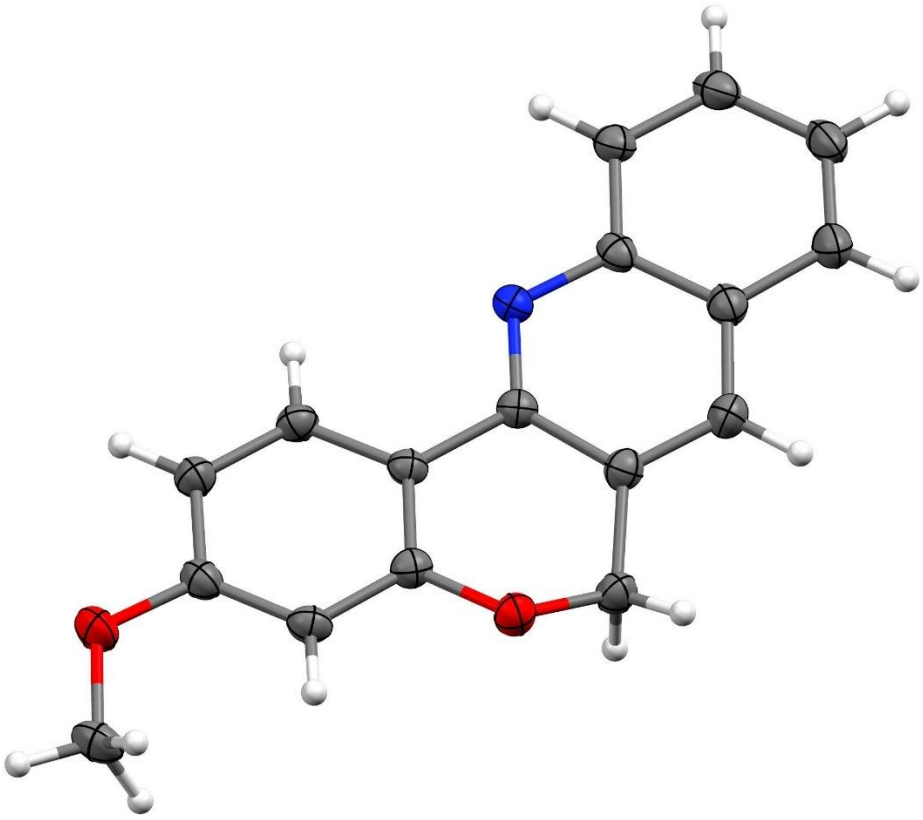


Figure S2. The single crystal X-ray structure of compound **7u** [CCDC 2335878]. Thermal ellipsoids are drawn at the 50% probability level.

References

1. Nolla-Saltiel, R.; Robles-Marín, E.; Porcel, S. *Tetrahedron Lett.* **2014**, *55,* 4484-4488.
2. Chen, H.; Wu, Z.-Z.; Shao, D.-Y.; Huang, P.-Q. *Sci. Adv.* **2022**, *8*, eade3431.
3. Ogiwara, Y.; Suzuki, Y.; Sato, K.; Sakai, N. *Org. Lett.* **2018**, *20*, 6965-6969.
4. Liu, B.; Qiu, Q.; Zhao, T.; Jiao, L.; Li, Y.; Huang, W.; Qian, H. *ChemMedChem*, **2014**, *10*, 336-344.
5. Pan, M.; Cui, J.; Jiao, L.; Ghaleb, H.; Liao, C.; Zhou, J.; Kairuki, M.; Lin, H.; Huang, W.; Qian, H. *Bioorg. Med. Chem.*, **2017**, *25*, 4194-4202.
6. Ibrahim, Y. A.; Moustafa, A. H. *J. Chem. Res. Synop.* **1999**, 254-255.

