Supporting Information for

Rh(II)-Catalyzed Synthesis of 5*H*-Isochromeno[3,4-*b*]indolizines from 4-Diazoisochroman-3-imines and Pyridines

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Content

General Information:

The 0.1 mmol scale reactions were carried out at different temperatures by a parallel reactor with 35 mL pressure sealed tubes (Figure S1, left). The 1 mmol scale reactions were carried out at 80 ºC in oil bath with 100 mL pressure sealed tube (Figure S1, right). The reaction temperatures are the temperatures indicated by the thermometer in parallel reactor or oil bath. The maximum internal pressure that the pressure sealed tubes can withstand is 600 kPa. Unless otherwise mentioned, solvent and reagent were purchased from commercial sources and used as received. ^{1}H NMR spectra were obtained on 400 or 600 MHz in $CDCl_3$. The chemical shifts were quoted in parts per million (ppm) referenced to 0.0 ppm for tetramethylsilane (TMS) as an internal standard. ^{13}C NMR spectra were recorded on 100 or 150 MHz in $CDCl_3$. The chemical shifts were reported in ppm referenced to the internal solvent signals (77.0 ppm for $CDCl_3$). $^{13}C\{^{1}H\}$ for proton-decoupled carbon data was recorded. The following abbreviations were used to describe peak patterns where appropriate: b = broad, s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet. Coupling constants *J* were reported in hertz unit (Hz). Infrared spectra were obtained on an FTIR spectrometer. High-resolution mass spectra (HRMS) data were obtained by using ESI ionization. Melting points were measured with micro melting point apparatus. Flash column chromatography was performed employing 300-400 mesh silica gel. Thin layer chromatography (TLC) was performed on silica gel HSGF254.

Substrates **1** and **2** were prepared according to the published procedures.[1,2]

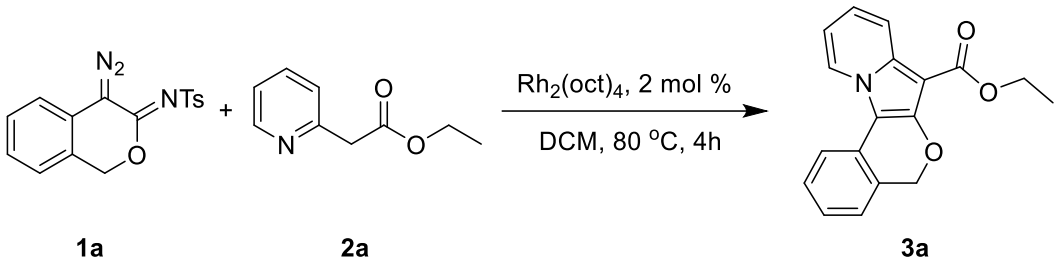
Figure S1. Pressure sealed tubes for reactions

References:

1) A. N. Ren, P. Lu and Y. G. Wang, *Chem. Commun.*, 2017, **53**, 3769-3772.

2) (a) B. Sahoo, M. N. Hopkinson and F. Glorius, *Angew. Chem. Int. Ed.,* 2015, **54**, 15545-15549. (b) Z. W. Chen, P. Liang, X. Y. Ma, H. Q. Luo, G. H. Xu, T. G. Liu, X. W. Wen, J. Zheng and H. Ye, *J. Org. Chem.*, 2019, **84**, 1630-1639.

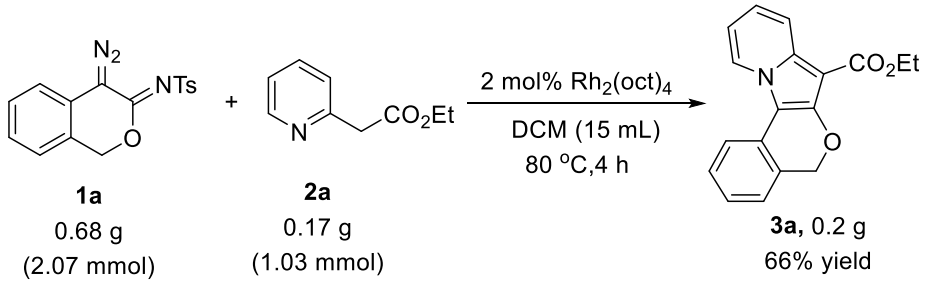
General Procedure for the Synthesis of 3:



1a **2a** **3a**

To a 35 mL pressure sealed tube equipped with a magnetic stirring bar was added sequentially **1** (0.2 mmol), **2** (0.1 mmol), $Rh_2(oct)_4$ (2 mol%) and DCM (1.5 mL). Then the mixture was stirred at 80 °C for 4 hours. Upon completion, the solvent was evaporated in vacuum. The residue was purified by column chromatography on silica (petroleum ether/ ethyl acetate/ dichloromethane = 10:1:2, v/v) to give the product **3**.

Preparation of Compound 3a at 1 mmole scale



1a
0.68 g
(2.07 mmol)

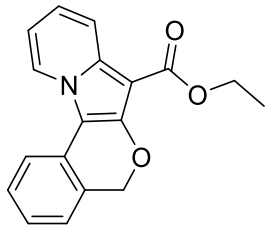
2a
0.17 g
(1.03 mmol)

3a, 0.2 g
66% yield

To a 100 mL pressure sealed tube equipped with a magnetic stirring bar was added sequentially **1** (2.07 mmol), **2** (1.03 mmol), $Rh_2(oct)_4$ (0.02 mmol) and DCM (15 mL). Then the mixture was stirred at 80 °C (oil bath) for 4 hours. Upon completion, the solvent was evaporated in vacuum. The residue was purified by column chromatography on silica (petroleum ether/ ethyl acetate/ dichloromethane = 10:1:2, v/v) to get 0.2 g of pure products **3a** as yellow oil.

Characterization data of Products 3

Ethyl 5*H*-isochromeno[3,4-*b*]indolizine-7-carboxylate (3a)



Yellow oil; Yield 70% (21 mg).

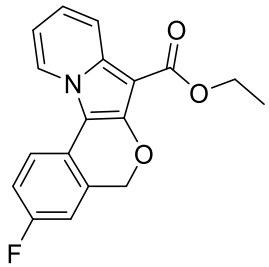
^{1}H NMR (400 MHz, Chloroform-d) δ 8.50 – 8.48 (m, 1H), 8.26 – 8.23 (m, 1H), 7.62 (d, *J* = 7.6 Hz, 1H), 7.38 – 7.33 (m, 1H), 7.24 (d, *J* = 7.2 Hz, 1H), 7.182 – 7.142 (m, 1H), 7.138 – 7.096 (m, 1H), 6.89 – 6.86 (m, 1H), 5.24 (s, 2H), 4.41 (q, *J* = 7.2 Hz, 2H), 1.43 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, Chloroform-d) δ 163.8, 150.0, 135.6, 128.6, 126.9, 126.1, 125.6, 125.2, 124.4, 122.6, 119.6, 115.8, 113.2, 109.9, 92.3, 71.2, 59.6, 14.6.

HRMS (ESI-TOF) calcd for $C_{18}H_{15}NNaO_3^+$ ([M+Na]$^+$): 316.0944; found: 316.0946.

IR (film): 2978, 1682, 1631, 1604, 1532, 1494, 1415, 1357, 1315, 1236, 1193, 1085, 1039, 928, 781, 717, 622 cm^{-1}.

Ethyl 3-fluoro-5*H*-isochromeno[3,4-*b*]indolizine-7-carboxylate (3b)



Light yellow solid; Yield 68% (21 mg); M.p. 150-151 ºC.

^{1}H NMR (400 MHz, Chloroform-d) δ 8.46 – 8.44 (m, 1H), 8.27 – 8.24 (m, 1H), 7.59 (dd, *J* = 8.8, 4.8 Hz, 1H), 7.16 – 7.12 (m, 1H), 7.11 – 7.06 (m, 1H), 7.01 (dd, *J* = 8.4, 2.8 Hz, 1H), 6.92 – 6.89 (m, 1H), 5.22 (s, 2H), 4.41 (q, *J* = 7.2 Hz, 2H), 1.43 (t, *J* = 7.2 Hz, 3H).

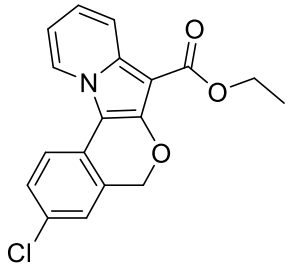
^{13}C NMR (100 MHz, Chloroform-d) δ 163.8, 160.4 (d, *J* = 244.5 Hz), 149.4, 135.3, 129.3 (d, *J* = 7.0 Hz), 124.0, 122.62, 122.59, 119.8, 117.2 (d, *J* = 7.7 Hz), 115.1 (d, *J* = 21.7 Hz), 113.42 (d, *J* = 22.8 Hz), 113.36, 109.5, 92.3, 70.8 (d, *J* = 1.9 Hz), 59.7, 14.7.

^{19}F NMR (376 MHz, Chloroform-d) δ -115.98.

HRMS (ESI-TOF) calcd for $C_{18}H_{14}FNNaO_3^+$ ([M+Na]$^+$): 334.0850; found: 334.0852.

IR (film): 2979, 1683, 1629, 1531, 1418, 1330, 1312, 1240, 1189, 1123, 1081, 1040, 966, 803, 735, 669 cm^{-1}.

Ethyl 3-chloro-5*H*-isochromeno[3,4-*b*]indolizine-7-carboxylate (3c)



White solid; Yield 74% (24 mg); M.p. 231-232 ºC.

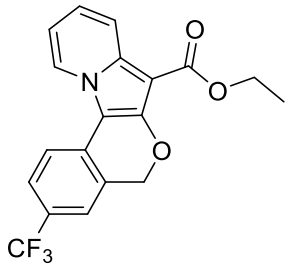
^{1}H NMR (400 MHz, Chloroform-d) δ 8.44 – 8.42 (m, 1H), 8.27 – 8.24 (m, 1H), 7.54 (d, *J* = 8.4 Hz, 1H), 7.33 (dd, *J* = 8.4, 2.0 Hz, 1H), 7.24 (d, *J* = 2.0 Hz, 1H), 7.18 – 7.13 (m, 1H), 6.93 – 6.89 (m, 1H), 5.21 (s, 2H), 4.41 (q, *J* = 7.2 Hz, 2H), 1.43 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, Chloroform-d) δ 163.7, 150.0, 135.8, 130.2, 128.54, 128.50, 125.9, 124.7, 124.3, 123.0, 119.8, 116.8, 113.5, 109.3, 92.4, 70.6, 59.7, 14.7.

HRMS (ESI-TOF) calcd for $C_{18}H_{14}ClNNaO_3^+$ ([M+Na]$^+$): 350.0554; found: 350.0555.

IR (film): 2912, 1683, 1568, 1527, 1495, 1404, 1315, 1226, 1192, 1126, 1097, 1018, 898, 779, 736, 677 cm^{-1}.

Ethyl 3-(trifluoromethyl)-5*H*-isochromeno[3,4-*b*]indolizine-7-carboxylate (3d)



White solid; Yield 84% (30 mg); M.p. 268-269 ºC.

^{1}H NMR (400 MHz, Chloroform-d) δ 8.50 (d, *J* = 6.8 Hz, 1H), 8.30 – 8.27 (m, 1H), 7.69 (d, *J* = 8.0 Hz, 1H), 7.62 (d, *J* = 8.0 Hz, 1H), 7.49 (s, 1H), 7.23 – 7.19 (m, 1H), 7.98 – 7.94

(m, 1H), 5.28 (s, 2H), 4.42 (q, *J* = 7.2 Hz, 2H), 1.43 (t, *J* = 7.2 Hz, 3H).

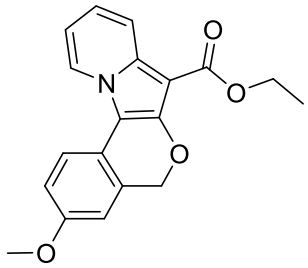
^{13}C NMR (100 MHz, Chloroform-d) δ 163.5, 151.3, 136.6, 129.3, 126.8, 126.5 (q, *J* = 32.7 Hz), 125.9 (q, *J* = 3.9 Hz), 124.6, 124.1 (q, *J* = 269.9 Hz), 123.7, 122.5 (q, *J* = 3.6 Hz), 119.9, 115.3, 113.7, 109.2, 92.6, 70.8, 59.8, 14.6.

^{19}F NMR (376 MHz, Chloroform-d) δ -62.19.

HRMS (ESI-TOF) calcd for $C_{19}H_{14}F_3NNaO_3^+$ ([M+Na]$^+$): 384.0818; found: 384.0821.

IR (film): 2918, 1684, 1631, 1504, 1417, 1315, 1233, 1167, 1130, 1086, 1020, 914, 813, 778, 732, 683 cm^{-1}.

Ethyl 3-methoxy-5*H*-isochromeno[3,4-*b*]indolizine-7-carboxylate (3e)



Light yellow solid; Yield 63% (20 mg); M.p. 180-181 ºC.

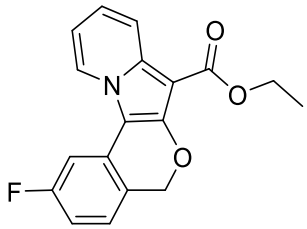
^{1}H NMR (400 MHz, Chloroform-d) δ 8.46 – 8.44 (m, 1H), 8.25 – 8.22 (m, 1H), 7.57 (d, *J* = 8.4 Hz, 1H), 7.12 – 7.08 (m, 1H), 6.91 (dd, *J* = 8.4, 2.8 Hz, 1H), 6.90 – 6.86 (m, 1H), 6.84 (d, *J* = 2.8 Hz, 1H), 5.23 (s, 2H), 4.42 (q, *J* = 7.2 Hz, 2H), 3.84 (s, 3H), 1.43 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, Chloroform-d) δ 163.9, 157.5, 148.7, 134.7, 129.2, 123.9, 121.90 , 119.6, 119.4, 117.3, 113.2, 113.1, 112.2, 110.0, 92.3, 71.2, 59.6, 55.4, 14.7.

HRMS (ESI-TOF) calcd for $C_{19}H_{17}NNaO_4^+$ ([M+Na]$^+$): 346.1050; found: 346.1053.

IR (film): 2920, 1682, 1577, 1532, 1504, 1418, 1314, 1247, 1193, 1081, 1043, 939, 780, 733, 677 cm^{-1}.

Ethyl 2-fluoro-5*H*-isochromeno[3,4-*b*]indolizine-7-carboxylate (3f)



White solid; Yield 66% (21 mg); M.p. 203-204 ºC.

^{1}H NMR (400 MHz, Chloroform-d) δ 8.45 – 8.42 (m, 1H), 8.29 – 8.26 (m, 1H), 7.32 (dd, *J* = 10.0, 2.4 Hz, 1H), 7.234 – 7.204 (m, 1H), 7.201 – 7.162 (m, 1H), 6.96 – 6.92 (m, 1H), 6.89 – 6.84 (m, 1H), 5.22 (s, 2H), 4.42 (q, *J* = 7.2 Hz, 2H), 1.43 (t, *J* = 7.2 Hz, 3H).

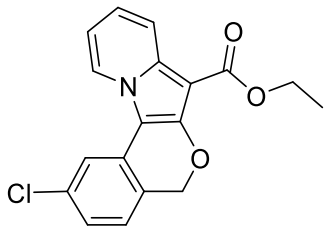
^{13}C NMR (100 MHz, Chloroform-d) δ 163.6, 163.1 (d, *J* = 243.4 Hz), 150.6, 136.1, 127.7 (d, *J* = 9.4 Hz), 127.0 (d, *J* = 9.2 Hz), 124.3, 123.2, 122.1 (d, *J* = 2.8 Hz), 119.7, 113.5, 111.2 (d, *J* = 21.9 Hz), 109.4 (d, *J* = 2.3 Hz), 103.3 (d, *J* = 25.3 Hz), 92.3, 70.8, 59.7, 14.6.

^{19}F NMR (376 MHz, Chloroform-d) δ -133.19.

HRMS (ESI-TOF) calcd for $C_{18}H_{14}FNNaO_3^+$ ([M+Na]$^+$): 344.0850; found: 334.0848.

IR (film): 2981, 2850, 1692, 1589, 1529, 1492, 1455, 1317, 1218, 1172, 1103, 1019, 927, 841, 738, 628 cm^{-1}.

Ethyl 2-chloro-5*H*-isochromeno[3,4-*b*]indolizine-7-carboxylate (3g)



White solid; Yield 73% (24 mg); M.p. 190-191 ºC.

^{1}H NMR (400 MHz, Chloroform-d) δ 8.48 – 8.45 (m, 1H), 8.29 – 8.26 (m, 1H), 7.59 (d, *J* = 2.0 Hz, 1H), 7.21 – 7.18 (m, 1H), 7.18 – 7.17 (m, 1H), 7.14 (dd, *J* = 8.0, 2.0 Hz, 1H), 6.98 – 6.94 (m, 1H), 5.21 (s, 2H), 4.41 (q, *J* = 7.2 Hz, 2H), 1.43 (t, *J* = 7.2 Hz, 3H).

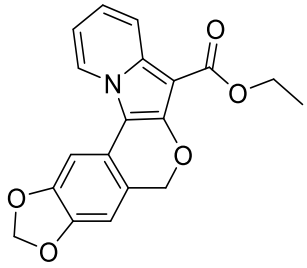
^{13}C NMR (100 MHz, Chloroform-d) δ 163.6, 150.7, 136.1, 134.6, 127.7, 126.8, 124.83, 124.76, 124.5, 123.3, 119.8, 115.8, 113.6, 109.0, 92.4, 70.76, 59.8, 14.7.

HRMS (ESI-TOF) calcd for $C_{18}H_{14}ClNNaO_3^+$ ([M+Na]$^+$): 350.0554; found: 350.0557.

IR (film): 2918, 1683, 1599, 1525, 1491, 1424, 1312, 1236, 1189, 1092, 1039, 927, 781,

734, 623 cm^{-1}.

Ethyl 5*H*-[1,3]dioxolo[4',5':6,7]isochromeno[3,4-*b*]indolizine-7-carboxylate (3h)



Yellow solid; Yield 54% (18 mg); M.p. 256-257 ºC.

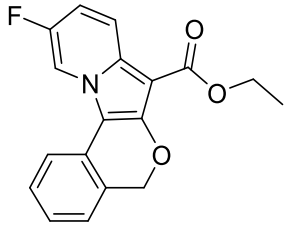
^{1}H NMR (400 MHz, Chloroform-d) δ 8.37 (d, *J* = 7.2 Hz, 1H), 8.24 (d, *J* = 8.8 Hz, 1H), 7.17 (s, 1H), 7.12 – 7.08 (m, 1H), 6.87 (t, *J* = 7.2 Hz, 1H), 6.78 (s, 1H), 5.99 (s, 2H), 5.14 (s, 2H), 4.41 (q, *J* = 7.2 Hz, 2H), 1.43 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, Chloroform-d) δ 163.8, 149.1, 147.9, 145.0, 135.0, 123.8, 122.1, 120.51, 120.50, 119.7, 113.2, 110.4, 107.2, 101.2, 97.9, 92.2, 71.3, 59.6, 14.7.

HRMS (ESI-TOF) calcd for $C_{19}H_{15}NNaO_5^+$ ($[M+Na]^+$): 360.0842; found: 360.0844.

IR (film): 3097, 2917, 1682, 1540, 1474, 1357, 1308, 1234, 1163, 1102, 1020, 911, 834, 779, 743, 684 cm^{-1}.

Ethyl 10-fluoro-5*H*-isochromeno[3,4-*b*]indolizine-7-carboxylate (3i)



Creamy white solid; Yield 67% (21 mg); M.p. 212-213 ºC.

^{1}H NMR (400 MHz, Chloroform-d) δ 8.42 (dd, *J* = 5.2, 2.0 Hz, 1H), 8.24 (dd, *J* = 9.6, 6.0 Hz, 1H), 7.61 (d, *J* = 7.6 Hz, 1H), 7.42 – 7.38 (m, 1H), 7.26 (d, *J* = 8.0 Hz, 1H), 7.23 – 7.19 (m, 1H), 7.06 – 7.01 (m, 1H), 5.25 (s, 2H), 4.41 (q, *J* = 7.2 Hz, 2H), 1.42 (t, *J* = 7.0 Hz, 3H).

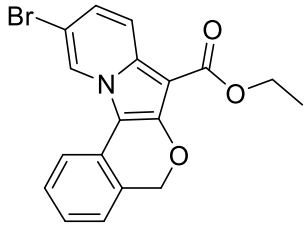
^{13}C NMR (100 MHz, Chloroform-d) δ 163.5, 154.0 (d, *J* = 235.2 Hz), 150.3 (d, *J* = 1.6 Hz), 132.6, 128.7, 127.2, 125.72 , 125.71 , 125.69 , 120.1 (d, *J* = 8.6 Hz), 115.9, 113.5 (d, *J* = 23.4 Hz), 111.6, 111.2, 93.0, 71.2, 59.8, 14.6.

^{19}F NMR (376 MHz, Chloroform-d) δ -138.33.

HRMS (ESI-TOF) calcd for $C_{18}H_{14}FNNaO_3^+$ ([M+Na]$^+$): 334.0850; found: 334.0852.

IR (film): 2924, 1682, 1606, 1539, 1497, 1334, 1262, 1214, 1153, 1106, 1082, 1040, 993, 804, 753, 649 cm^{-1}.

Ethyl 10-bromo-5*H*-isochromeno[3,4-*b*]indolizine-7-carboxylate (3j)



Creamy white solid; Yield 72% (27 mg); M.p. 214-215 ºC.

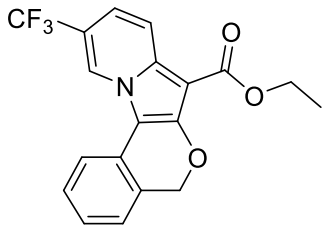
^{1}H NMR (400 MHz, Chloroform-d) δ 8.57 (s, 1H), 8.13 (d, *J* = 9.6 Hz, 1H), 7.58 (d, *J* = 8.0 Hz, 1H), 7.41 – 7.37 (m, 1H), 7.24 (d, *J* = 6.8 Hz, 1H), 7.21 – 7.17 (m, 1H), 7.15 (dd, *J* = 9.6, 2.0 Hz, 1H), 5.24 (s, 2H), 4.40 (q, *J* = 7.2 Hz, 2H), 1.42 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, Chloroform-d) δ 163.4, 149.9, 133.4, 128.7, 127.1, 125.8, 125.7, 125.5, 125.2, 124.1, 120.1, 116.1, 110.4, 107.8,93.3, 71.2, 59.8, 14.6.

HRMS (ESI-TOF) calcd for $C_{18}H_{14}BrNNaO_3^+$ ([M+Na]$^+$): 394.0049; found: 394.0052.

IR (film): 2978, 1695, 1607, 1530, 1494, 1446, 1418, 1332, 1237, 1181, 1087, 1070, 1042, 998, 807, 749, 640 cm^{-1}

Ethyl 10-(trifluoromethyl)-5*H*-isochromeno[3,4-*b*]indolizine-7-carboxylate (3k)



Yellow solid; Yield 56% (20 mg); M.p. 168-169 ºC.

^{1}H NMR (400 MHz, Chloroform-d) δ 8.81 (s, 1H), 8.34 (d, *J* = 9.6 Hz, 1H), 7.62 (d, *J* = 8.0 Hz, 1H), 7.45 – 7.41 (m, 1H), 7.28 (d, *J* = 5.6 Hz, 1H), 7.25 – 7.22 (m, 2H), 5.28 (s, 2H), 4.42 (q, *J* = 7.2 Hz, 2H), 1.43 (t, *J* = 7.2 Hz, 3H).

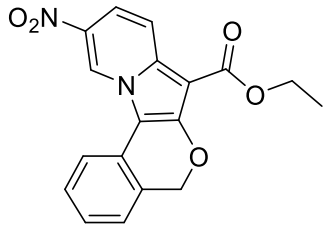
^{13}C NMR (100 MHz, Chloroform-d) δ 163.3, 150.8, 135.2, 128.9, 127.2, 126.2, 125.9, 125.3, 123.7 (q, *J* = 269.3 Hz), 122.6 (q, *J* = 5.7 Hz), 120.1, 117.7 (q, *J* = 2.6 Hz), 117.2 (q, *J* = 33.6 Hz), 116.2, 111.3, 94.5, 71.3, 60.0, 14.6.

^{19}F NMR (376 MHz, Chloroform-d) δ -62.15.

HRMS (ESI-TOF) calcd for $C_{19}H_{14}F_3NNaO_3^+$ ([M+Na]$^+$): 384.0817; found: 384.0819

IR (film): 2924, 1705, 1648, 1603, 1454, 1375, 1316, 1240, 1130, 1065, 969, 808, 752, 634 cm^{-1}.

Ethyl 10-nitro-5*H*-isochromeno[3,4-*b*]indolizine-7-carboxylate (3l)



Orange solid; Yield 75% (25 mg); M.p. 303-304 ºC.

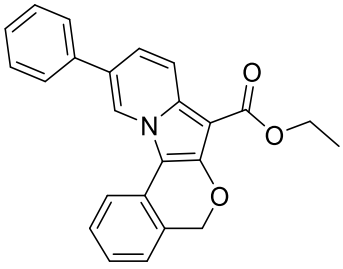
^{1}H NMR (400 MHz, Chloroform-d) δ 9.58 (d, *J* = 1.6 Hz, 1H), 8.31 (d, *J* = 9.6 Hz, 1H), 7.83 (dd, *J* = 10.0, 2.0 Hz, 1H), 7.69 (d, *J* = 8.0 Hz, 1H), 7.51 – 7.45 (m, 1H), 7.30 (s, 1H), 7.29 (s, 1H), 5.32 (s, 2H), 4.43 (q, *J* = 7.2 Hz, 2H), 1.44 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, Chloroform-d) δ 162.9, 151.8, 137.5, 135.3, 129.1, 127.2, 126.8, 125.9, 124.8, 123.9, 118.9, 116.5, 115.4, 112.1, 96.2, 71.4, 60.4, 14.5.

HRMS (ESI-TOF) calcd for $C_{18}H_{14}N_2NaO_5^+$ ([M+Na]$^+$): 361.0795; found: 361.0796.

IR (film): 2917, 1694, 1642, 1549, 1499, 1447, 1328, 1238, 1188, 1095, 1038, 997, 818, 750, 644 cm^{-1}.

Ethyl 10-phenyl-5*H*-isochromeno[3,4-*b*]indolizine-7-carboxylate (3m)



Yellow solid; Yield 74% (27 mg); M.p. 233-234 ºC.

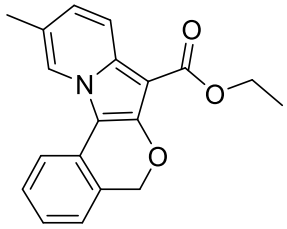
^{1}H NMR (400 MHz, Chloroform-d) δ 8.66 (s, 1H), 8.30 (d, *J* = 9.2 Hz, 1H), 7.68 (d, *J* = 8.0 Hz, 1H), 7.61 (d, *J* = 7.2 Hz, 2H), 7.52 – 7.48 (m, 2H), 7.42 (d, *J* = 7.2 Hz, 1H), 7.40 – 7.39 (m, 1H), 7.38 – 7.36 (m, 1H), 7.25 (d, *J* = 3.2 Hz, 1H), 7.18 (t, *J* = 7.6 Hz, 1H), 5.26 (s, 2H), 4.43 (q, *J* = 7.2 Hz, 2H), 1.44 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, Chloroform-d) δ 163.7, 150.4, 137.7, 134.6, 129.2, 128.7, 127.9, 127.6, 127.0, 126.9, 126.1, 125.7, 125.3, 122.9, 122.0, 119.5, 115.9, 110.3, 92.4, 71.3, 59.7, 14.7.

HRMS (ESI-TOF) calcd for $C_{24}H_{19}NNaO_3^+$ ([M+Na]$^+$): 392.1257; found: 392.1260.

IR (film): 2923, 1689, 1633, 1604, 1543, 1518, 1489, 1339, 1220, 1168, 1089, 811, 751, 699, 628 cm^{-1}.

Ethyl 10-methyl-5*H*-isochromeno[3,4-*b*]indolizine-7-carboxylate (3n)



Yellow solid; Yield 60% (18 mg); M.p. 196-197 ºC.

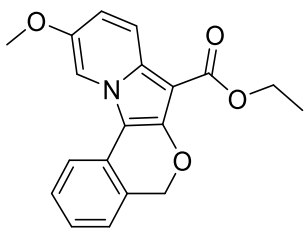
^{1}H NMR (400 MHz, Chloroform-d) δ 8.29 (s, 1H), 8.14 (d, *J* = 9.2 Hz, 1H), 7.64 (d, *J* = 8.0 Hz, 1H), 7.39 – 7.35 (m, 1H), 7.24 (d, *J* = 7.2 Hz, 1H), 7.18 – 7.14 (m, 1H), 6.99 (d, *J* = 9.2 Hz, 1H), 5.23 (s, 2H), 4.40 (q, *J* = 7.2 Hz, 2H), 2.37 (s, 3H), 1.42 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, Chloroform-d) δ 163.8, 149.9, 134.3, 128.6, 126.9, 126.3, 125.6, 125.6, 125.0, 122.8, 122.5, 119.0, 115.8, 109.7, 91.8, 71.2, 59.5, 18.6, 14.7.

HRMS (ESI-TOF) calcd for $C_{19}H_{17}NNaO_3^+$ ([M+Na]$^+$): 330.1101; found: 330.1102

IR (film): 2925, 1682, 1605, 1541, 1494, 1419, 1313, 1274, 1220, 1085, 1040, 938, 803, 753, 646 cm^{-1}.

Ethyl 10-methoxy-5*H*-isochromeno[3,4-*b*]indolizine-7-carboxylate (3o)



Creamy white solid; Yield 55% (18 mg); M.p. 227-228 ºC.

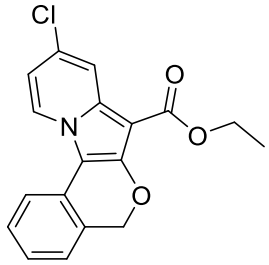
^{1}H NMR (400 MHz, Chloroform-d) δ 8.15 (dd, *J* = 9.6, 2.0 Hz, 1H), 8.08 – 8.07 (m, 1H), 7.63 (dd, *J* = 8.0, 2.8 Hz, 1H), 7.40 – 7.35 (m, 1H), 7.25 (d, *J* = 7.6 Hz, 1H), 7.19 – 7.15 (m, 1H), 6.96 – 6.92 (m, 1H), 5.22 (s, 2H), 4.40 (q, *J* = 7.2 Hz, 2H), 3.89 (s, 3H), 1.42 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, Chloroform-d) δ 163.7, 149.9, 131.9, 128.6, 127.1, 126.2, 125.7, 125.1, 119.8, 116.3, 115.5, 110.7, 108.1, 99.9, 92.0, 71.2, 59.6, 56.2, 14.7.

HRMS (ESI-TOF) calcd for $C_{19}H_{17}NNaO_4^+$ ([M+Na]$^+$): 346.1050; found: 346.1051.

IR (film): 2917, 1691, 1639, 1577, 1531, 1494, 1277, 1215, 1154, 1104, 1027, 853,778, 762, 645 cm^{-1}.

Ethyl 9-chloro-5*H*-isochromeno[3,4-*b*]indolizine-7-carboxylate (3p)



Light yellow solid; Yield 40% (13 mg); M.p. 241-242 ºC.

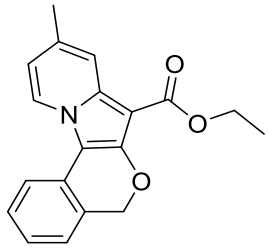
^{1}H NMR (400 MHz, Chloroform-d) δ 8.39 (d, *J* = 7.6 Hz, 1H), 8.25 (d, *J* = 2.4 Hz, 1H), 7.55 (d, *J* = 7.6 Hz, 1H), 7.39 – 7.35 (m, 1H), 7.24 (d, *J* = 7.2 Hz, 1H), 7.19 (t, *J* = 7.2 Hz, 1H), 6.85 (dd, *J* = 7.6, 2.4 Hz, 1H), 5.25 (s, 2H), 4.41 (q, *J* = 7.2 Hz, 2H), 1.43 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, Chloroform-d) δ 163.4, 150.3, 135.4, 129.0, 128.7, 126.9, 125.7 , 125.6, 124.9, 118.6, 115.9, 114.4, 110.2, 92.7, 71.3, 59.9, 14.6.

HRMS (ESI-TOF) calcd for $C_{18}H_{14}ClNNaO_3^+$ ([M+Na]$^+$): 350.0554; found: 350.0556.

IR (film): 2916, 1682, 1646, 1516, 1489, 1440, 1401, 1336, 1272, 1191, 1107, 971, 868, 749, 683 cm^{-1}.

Ethyl 9-methyl-5*H*-isochromeno[3,4-*b*]indolizine-7-carboxylate (3q)



Yellow solid; Yield 56% (17 mg); M.p. 160-161 ºC.

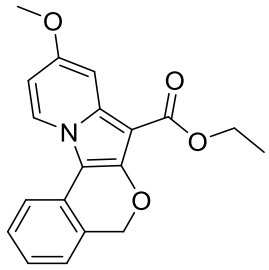
^{1}H NMR (400 MHz, Chloroform-d) δ 8.40 (d, *J* = 6.8 Hz, 1H), 8.06 (m, 1H), 7.58 (d, *J* = 7.6 Hz, 1H), 7.36 (t, *J* = 7.6 Hz, 1H), 7.24 (d, *J* = 7.2 Hz, 1H), 7.17 – 7.13 (m, 1H), 6.73 (dd, *J* = 7.2, 2.0 Hz, 1H), 5.23 (s, 2H), 4.41 (q, *J* = 7.2 Hz, 2H), 2.42 (s, 3H), 1.43 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, Chloroform-d) δ 164.0, 150.1, 136.3, 133.9, 128.6, 126.6, 126.4, 125.6, 124.8, 124.1, 118.5, 115.6, 115.5, 109.3, 91.1, 71.3, 59.6, 21.4, 14.7.

HRMS (ESI-TOF) calcd for $C_{19}H_{17}NNaO_3^+$ ([M+Na]$^+$): 330.1101; found: 330.1104.

IR (film): 2975, 1682, 1604, 1533, 1495, 1409, 1348, 1240, 1195, 1110, 1041, 993, 933, 752, 609 cm^{-1}.

Ethyl 9-methyoxy-5*H*-isochromeno[3,4-*b*]indolizine-7-carboxylate (3r)



Light yellow solid; Yield 42% (14 mg); M.p. 227-228 ºC.

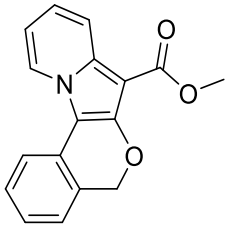
^{1}H NMR (600 MHz, Chloroform-*d*) δ 8.36 (d, *J* = 7.8 Hz, 1H), 7.69 (d, *J* = 3.0 Hz, 1H), 7.51 (d, *J* = 7.8 Hz, 1H), 7.35 – 7.33 (m, 1H), 7.23 (d, *J* = 7.2 Hz, 1H), 7.14 – 7.12 (m, 1H), 6.60 (dd, *J* = 7.8, 3.0 Hz, 1H), 5.22 (s, 2H), 4.40 (q, *J* = 7.2 Hz, 2H), 3.91 (s, 3H), 1.42 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, Chloroform-d) δ 164.2, 156.5, 150.0, 138.4, 128.6, 126.4, 126.10, 126.09, 125.6, 124.5, 115.0, 108.4, 107.2, 97.9, 91.0, 71.3, 59.5, 55.5, 14.7.

HRMS (ESI-TOF) calcd for $C_{19}H_{17}NNaO_4^+$ ([M+Na]$^+$): 346.1050; found: 346.1051.

IR (film): 2919, 1679, 1600, 1540, 1433, 1286, 1242, 1211, 1188, 1107, 1021, 974, 837, 745, 683 cm^{-1}.

Methyl 5*H*-isochromeno[3,4-*b*]indolizine-7-carboxylate (3s)



Yellow solid; Yield 72% (20 mg); M.p. 191-192 ºC.

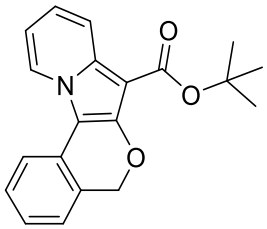
^{1}H NMR (600 MHz, Chloroform-d) δ 8.52 (d, *J* = 7.2 Hz, 1H), 8.29 (d, *J* = 9.0 Hz, 1H), 7.65 (d, *J* = 7.8 Hz, 1H), 7.40 – 7.37 (m, 1H), 7.26 (d, *J* = 7.8 Hz, 1H), 7.20 – 7.18 (m, 1H), 7.16 – 7.13 (m, 1H), 6.92 – 6.90 (m, 1H), 5.26 (s, 2H), 3.94 (s, 3H).

^{13}C NMR (100 MHz, Chloroform-d) δ 164.2, 149.7, 135.8, 128.7, 126.8, 126.1, 125.6, 125.3, 124.4, 122.8, 119.6, 115.9, 113.4, 110.0, 91.9, 71.3, 51.0.

HRMS (ESI-TOF) calcd for $C_{17}H_{13}NNaO_3$ ($[M+Na]^+$): 302.0788; found: 302.0790.

IR (film): 2919, 2849, 1688, 1532, 1494, 1444, 1404, 1316, 1237, 1195, 1087, 1034, 972, 781, 718, 622 cm^{-1}.

tert-Butyl 5*H*-isochromeno[3,4-*b*]indolizine-7-carboxylate (3t)



Yellow oil; Yield 57% (18 mg).

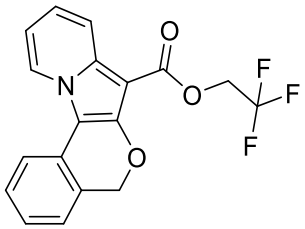
^{1}H NMR (400 MHz, Chloroform-d) δ 7.51 (d, *J* = 7.6 Hz, 1H), 8.16 (d, *J* = 8.8 Hz, 1H), 7.64 (d, *J* = 8.0 Hz, 1H), 7.39 – 7.35 (m, 1H), 7.25 (d, *J* = 7.6 Hz, 1H), 7.19 – 7.16 (m, 1H), 7.12 – 7.08 (m, 1H), 6.89 – 6.85 (m, 1H), 5.25 (s, 2H), 1.65 (s, 9H).

^{13}C NMR (100 MHz, Chloroform-d) δ 163.2, 150.7, 135.1, 128.6, 127.0, 126.2, 125.7 , 125.1, 124.4, 122.2, 119.7, 115.7, 112.9, 109.9, 93.8, 80.1, 71.2, 28.6.

HRMS (ESI-TOF) calcd for $C_{20}H_{19}NNaO_3$ ($[M+Na]^+$): 344.1257; found:344.1259.

IR (film): 2919, 1683, 1530, 1494, 1409, 1364, 1317, 1242, 1171, 1124, 1084, 1036, 963, 782, 734, 621 cm^{-1}.

2,2,2-Trifluoroethyl 5*H*-isochromeno[3,4-*b*]indolizine-7-carboxylate (3u)



Yellow solid; Yield 65% (23 mg); M.p. 153-154 ºC.

^{1}H NMR (400 MHz, Chloroform-d) δ 8.55 – 8.53 (m, 1H), 8.19 – 8.16 (m, 1H), 7.64 (d, *J* = 7.6 Hz, 1H), 7.41 – 7.36 (m, 1H), 7.26 (dd, *J* = 7.6, 1.2 Hz 1H), 7.22 – 7.18 (m, 2H), 6.97 – 6.93 (m, 1H), 5.27 (s, 2H), 4.74 (q, *J* = 8.8 Hz, 2H).

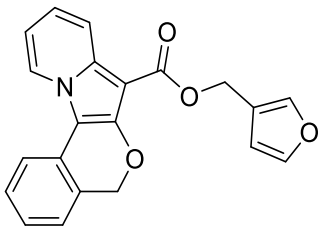
^{13}C NMR (100 MHz, Chloroform-d) δ 161.5, 150.6, 135.8, 128.7, 126.9, 125.9, 125.7, 125.6, 124.7, 123.6, 123.5 (q, *J* = 275.6 Hz), 119.5, 115.9, 113.8, 110.5, 90.5, 71.3, 59.5 (q, *J* = 36.0 Hz).

^{19}F NMR (376 MHz, Chloroform-d) δ -73.44.

HRMS (ESI-TOF) calcd for $C_{18}H_{12}F_3NNaO_3^+$ ($[M+Na]^+$): 370.0661; found: 370.0661.

IR (film): 2924, 1704, 1605, 1524, 1495, 1445, 1416, 1316, 1279, 1170, 1111, 983, 757, 671, 545 cm^{-1}.

Furan-3-ylmethyl 5*H*-isochromeno[3,4-*b*]indolizine-7-carboxylate (3v)



Yellow oil; Yield 51% (18 mg).

^{1}H NMR (400 MHz, Chloroform-d) δ 8.50 (d, *J* = 6.8 Hz, 1H), 8.22 (d, *J* = 9.2 Hz, 1H), 7.63 (d, *J* = 7.6 Hz, 1H), 7.58 (s, 1H), 7.42 – 7.41 (m, 1H), 7.39 – 7.35 (m, 1H), 7.25 (d, *J* = 7.2

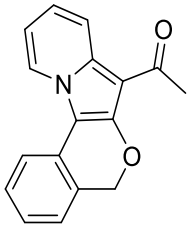
Hz, 1H), 7.19 – 7.16 (m, 1H), 7.22 – 7.18 (m, 1H), 6.91 – 6.87 (m, 1H), 6.56 (s, 1H), 5.27 (s, 2H), 5.25 (s, 2H).

^{13}C NMR (100 MHz, Chloroform-d) δ 163.5, 150.3, 143.2, 141.3, 135.7, 128.6, 126.9, 126.1, 125.7, 125.3, 124.5, 122.8, 121.3, 119.6, 115.8, 113.3, 110.6, 110.1, 92.0, 71.3, 57.07.

HRMS (ESI-TOF) calcd for $C_{21}H_{15}NNaO_4^+$([M+Na]$^+$): 368.0893; found: 368.0896.

IR (film): 2918, 1683, 1631, 1532, 1494, 1414, 1316, 1234, 1192, 1084, 1019, 874, 781, 719, 600 cm^{-1}.

1-(5*H*-Isochromeno[3,4-*b*]indolizin-7-yl)ethan-1-one (3w)



Yellow solid; Yield 39% (10 mg); M.p. 189-190 ºC.

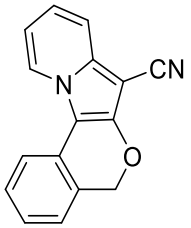
^{1}H NMR (400 MHz, Chloroform-d) δ 8.59 – 8.56 (m, 1H), 8.54 – 8.51 (m, 1H), 7.65 (d, *J* = 7.6 Hz, 1H), 7.42 – 7.38 (m, 1H), 7.28 (d, *J* = 6.8 Hz, 1H), 7.23 – 7.21 (m, 1H), 7.20 – 7.18 (m, 1H), 6.98 – 6.94 (m, 1H), 5.24 (s, 2H), 2.59 (s, 3H).

^{13}C NMR (100 MHz, Chloroform-d) δ 192.5, 150.1, 135.4, 128.8, 126.7, 126.3, 125.7, 125.2, 124.24, 124.18, 120.5, 116.0, 114.2, 109.8, 101.8, 71.2, 30.2.

HRMS (ESI-TOF) calcd for $C_{17}H_{13}NNaO_2^+$ ([M+Na]$^+$): 286.0838; found: 286.0840.

IR (film): 2918, 1620, 1603, 1525, 1490, 1404, 1406, 1362, 1227, 1147, 1018, 900, 717, 631 cm^{-1}.

5*H*-Isochromeno[3,4-*b*]indolizine-7-carbonitrile (3x)



Light yellow solid; Yield 46% (11 mg); M.p. 295-296 ºC.

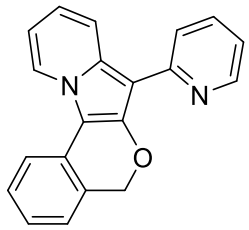
^{1}H NMR (600 MHz, Chloroform-d) δ 8.52 – 8.50 (m, 1H), 7.632 – 7.625 (m, 1H), 7.62 – 7.61 (m, 1H) 7.42 –7.39 (m, 1H), 7.27 – 7.25 (m, 1H), 7.24 – 7.21 (m, 1H), 7.16 – 7.13 (m, 1H), 6.95 – 6.92 (m, 1H), 5.25 (s, 2H).

^{13}C NMR (100 MHz, Chloroform-d) δ 150.4, 136.5, 128.9, 127.0, 125.9, 125.6, 125.0, 122.6, 117.6, 116.3, 114.2, 113.7, 109.8, 72.0, 71.4.

HRMS (ESI-TOF) calcd for $C_{16}H_{10}N_2NaO^+$ ([M+Na]$^+$): 269.0685; found:269.0684.

IR (film): 2917, 2210, 1605, 1532, 1495, 1437, 1349, 1315, 1240, 1141, 1011, 920, 740, 717, 692 cm^{-1}.

7-(Pyridin-2-yl)-5*H*-isochromeno[3,4-*b*]indolizine (3y)



Yellow oil; Yield 60% (18 mg).

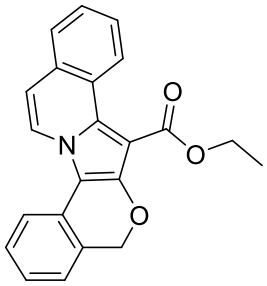
^{1}H NMR (600 MHz, Chloroform-d) δ 8.78 – 8.76 (m, 1H), 8.65 – 8.64 (m, 1H), 8.48 (d, *J* = 7.2 Hz, 1H), 7.98 – 7.97 (m, 1H), 7.70 (d, *J* = 7.8 Hz, 1H), 7.69 – 7.66 (m, 1H), 7.41 – 7.38 (m, 1H), 7.28 (d, *J* = 7.2 Hz, 1H), 7.18 – 7.16 (m, 1H), 7.04 – 7.03 (m, 1H), 7.02 – 7.00 (m, 1H), 6.81 –6.78 (m, 1H), 5.24 (s, 2H).

^{13}C NMR (150 MHz, Chloroform-d) δ 154.0, 148.8, 148.0, 135.9, 132.6, 128.7, 127.2, 126.8, 125.6, 124.6, 123.8, 122.1, 120.53, 120.46, 119.1, 116.1, 112.2, 109.5, 99.9, 71.2.

HRMS (ESI-TOF) calcd for $C_{20}H_{15}N_2O^+$ ([M+H]$^+$): 299.1179; found: 299.1181.

IR (film): 3062, 2918, 2843, 1601, 1586, 1532, 1456, 1356, 1314, 1252, 1142, 1013, 908, 784, 721, 642 cm^{-1}.

Ethyl 5*H*-isochromeno[3',4':4,5]pyrrolo[2,1-*a*]isoquinoline-7-carboxylate (3z)



Yellow oil; Yield 53% (18 mg).

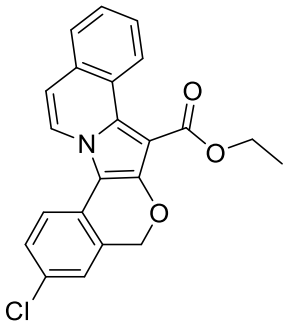
^{1}H NMR (400 MHz, Chloroform-d) δ 9.21 (dd, *J* = 8.4, 1.2 Hz, 1H), 8.37 (d, *J* = 7.2 Hz, 1H), 7.67 – 7.64 (m, 2H), 7.57 – 7.53 (m, 1H), 7.51 – 7.47 (m, 1H), 7.41 – 7.37 (m, 1H), 7.27 (d, *J* = 7.2 Hz, 1H), 7.22 – 7.18 (m, 1H), 7.09 (d, *J* = 7.2 Hz, 1H), 5.21 (s, 2H), 4.49 (q, *J* = 7.2 Hz, 2H), 1.46 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, Chloroform-d) δ 165.1, 148.8, 130.7, 128.7, 128.6, 127.7, 127.44, 127.41, 126.9, 126.3, 126.0, 125.8, 125.5, 125.2, 122.3, 116.6, 113.4, 111.4, 98.3, 71.4, 60.6, 14.5.

HRMS (ESI-TOF) calcd for $C_{22}H_{17}NNaO_3^+$ ([M+Na]$^+$): 366.1101; found: 366.1101.

IR (film): 2920, 1694, 1605, 1544, 1494, 1457, 1346, 1269, 1195, 1104, 1048, 988, 911, 759, 733 cm^{-1}.

Ethyl 3-chloro-5*H*-isochromeno[3',4':4,5]pyrrolo[2,1-*a*]isoquinoline-7-carboxylate (3A)



Yellow solid; Yield 61% (23 mg); M.p. 250-251 ºC.

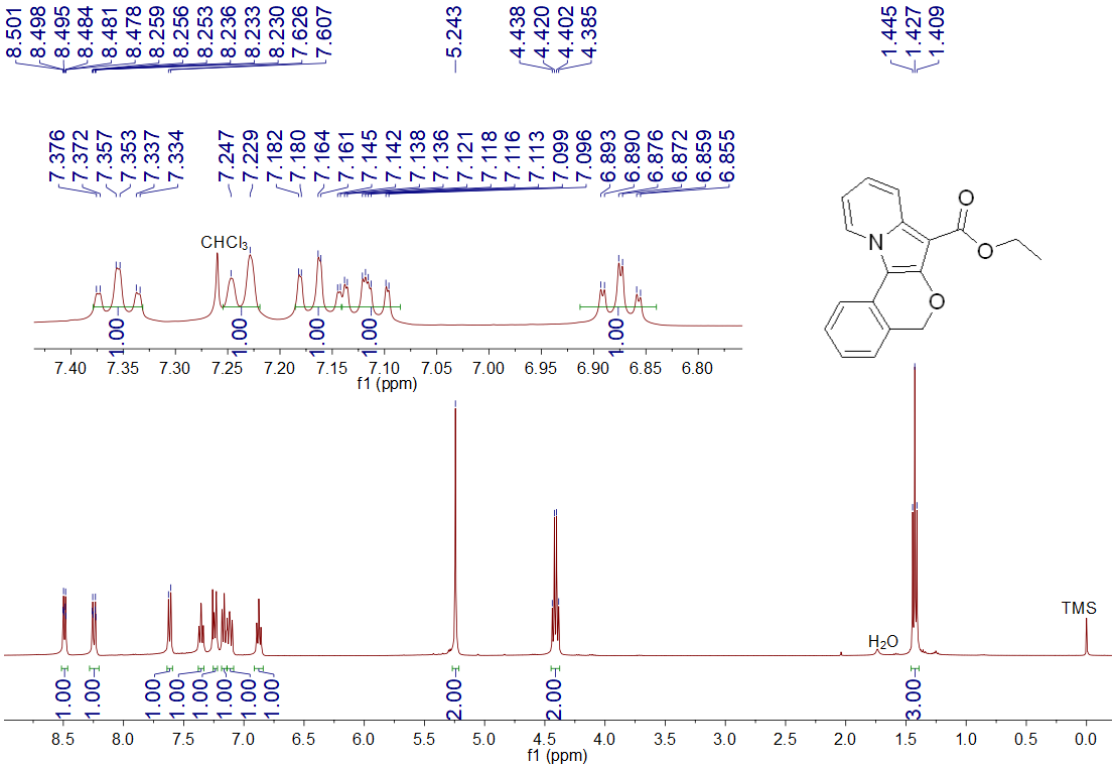
^{1}H NMR (400 MHz, Chloroform-d) δ 9.20 (dd, *J* = 8.4, 1.2 Hz, 1H), 8.26 (d, *J* = 7.2 Hz, 1H), 7.64 (dd, *J* = 8.0, 1.6 Hz, 1H), 7.57 – 7.55 (m, 1H), 7.54 – 7.53 (m, 1H), 7.52 – 7.47 (m, 1H)7.34 (dd, *J* = 8.4, 2.0 Hz, 1H), 7.24 (d, *J* = 2.0 Hz, 1H), 7.08 (d, *J* = 7.6 Hz, 1H), 5.16 (s, 2H), 4.49 (q, *J* = 7.2 Hz, 2H), 1.46 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, Chloroform-d) δ 164.9, 148.8, 131.0, 130.6, 129.3, 128.6, 128.6, 127.6, 127.6, 126.9, 126.4, 126.0, 125.1, 124.5, 122.1, 117.6, 113.7, 110.7, 98.4, 70.8, 60.6, 14.5.

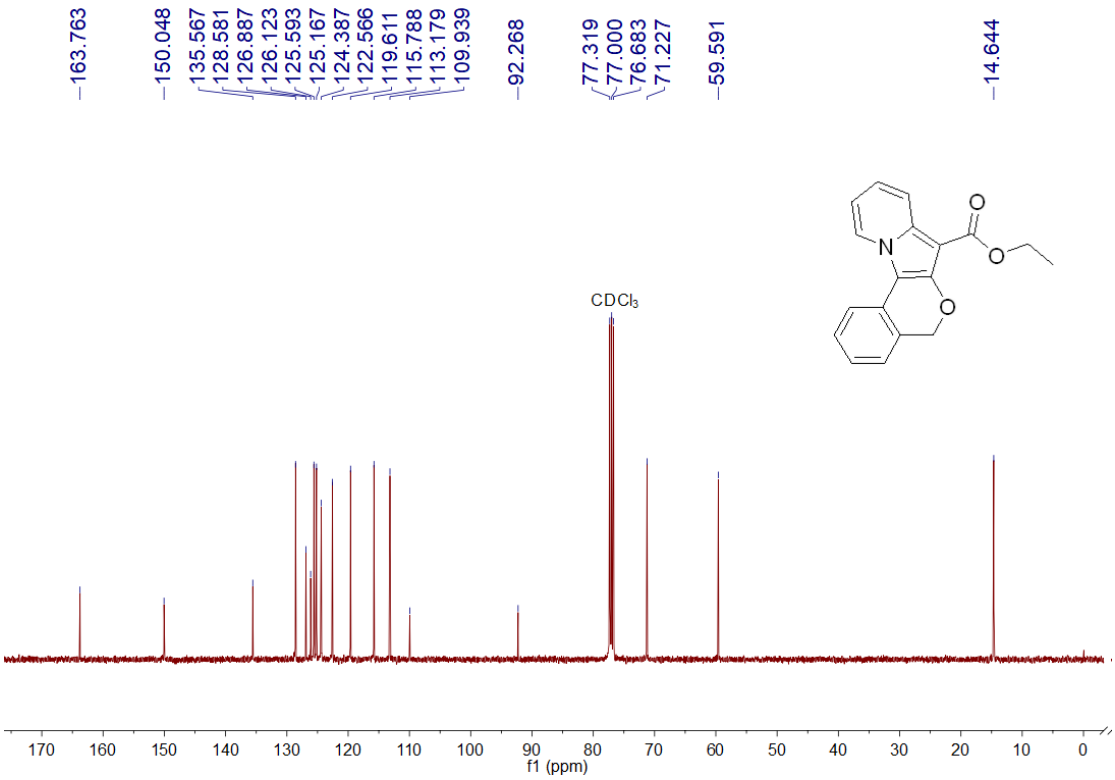
HRMS (ESI-TOF) calcd for $C_{22}H_{16}ClNNaO_3^+$ ($[M+Na]^+$): 400.0711; found: 400.0714.

IR (film): 2920, 1694, 1571, 1539, 1508, 1451, 1342, 1269, 1193, 1108, 1033, 902, 871, 786, 732 cm^{-1}.

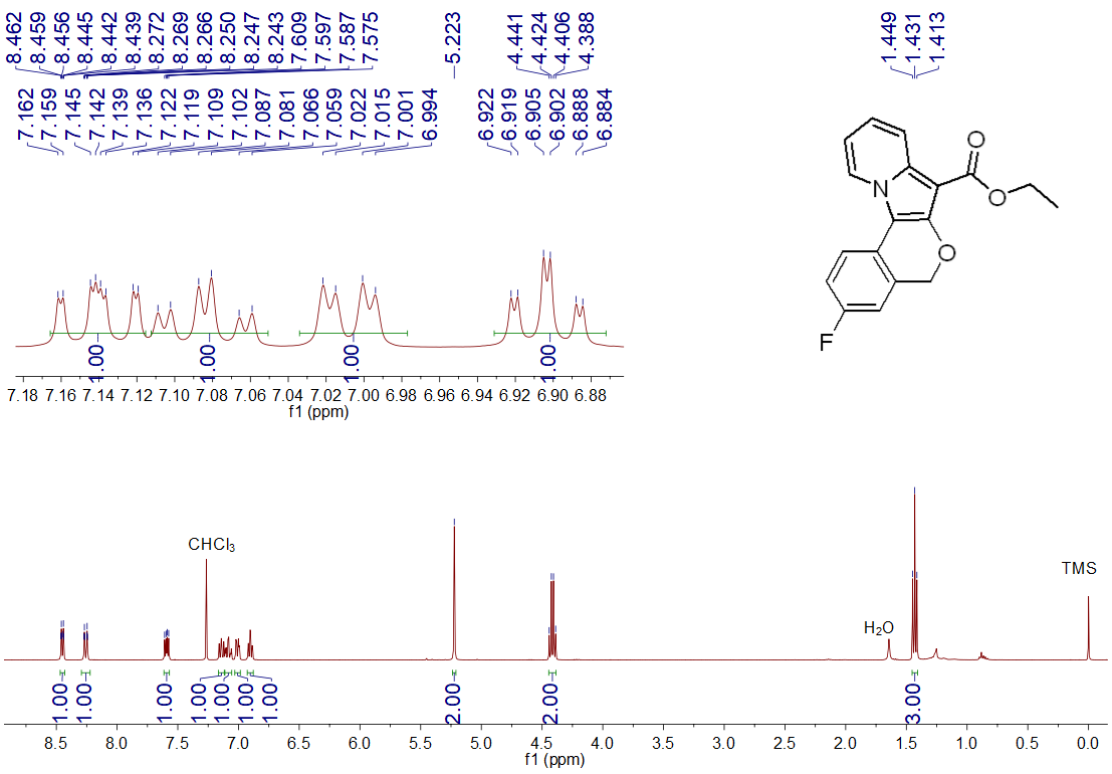
Copies of NMR Spectra



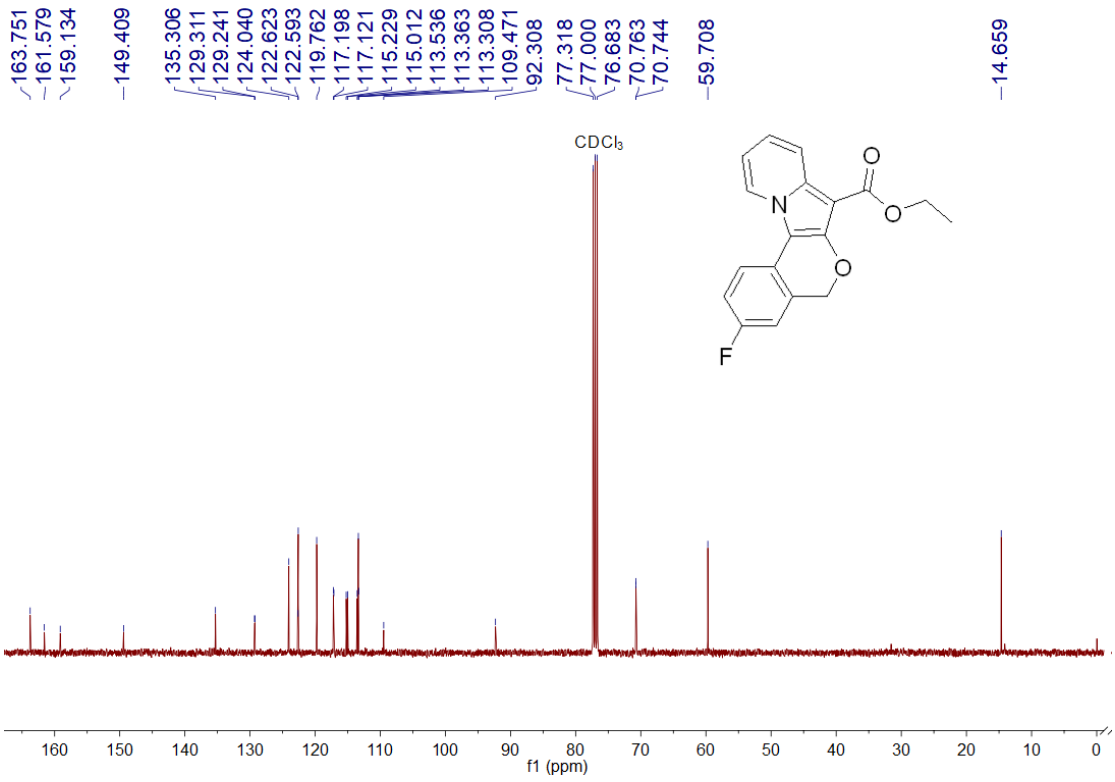
^{1}H NMR of **3a** ($CDCl_3$, 400 MHz)



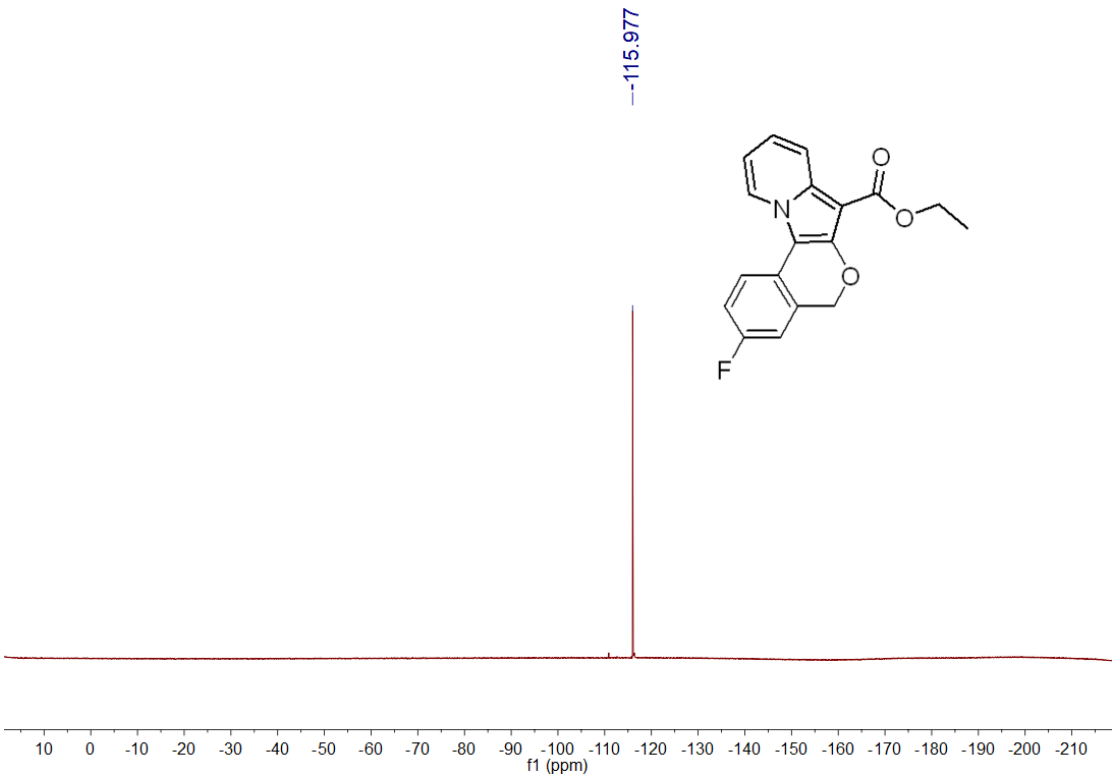
^{13}C{^{1}H} NMR of **3a** ($CDCl_3$, 100 MHz)



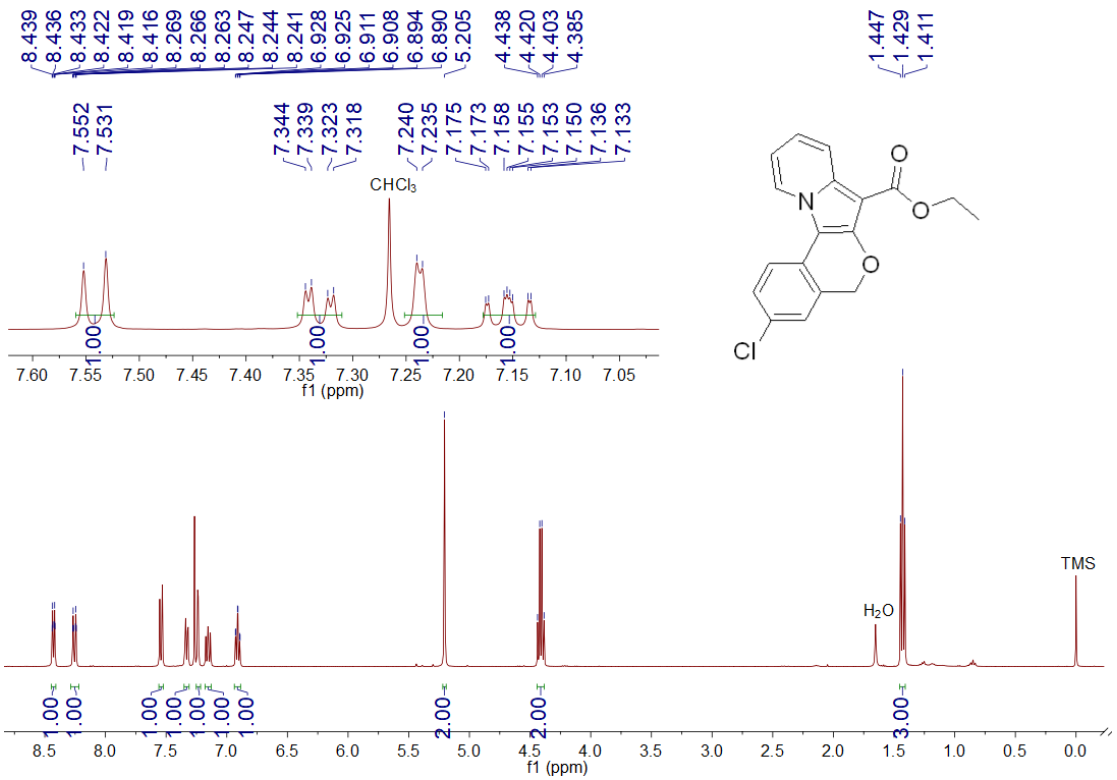
^{1}H NMR of **3b** ($CDCl_3$, 400 MHz)



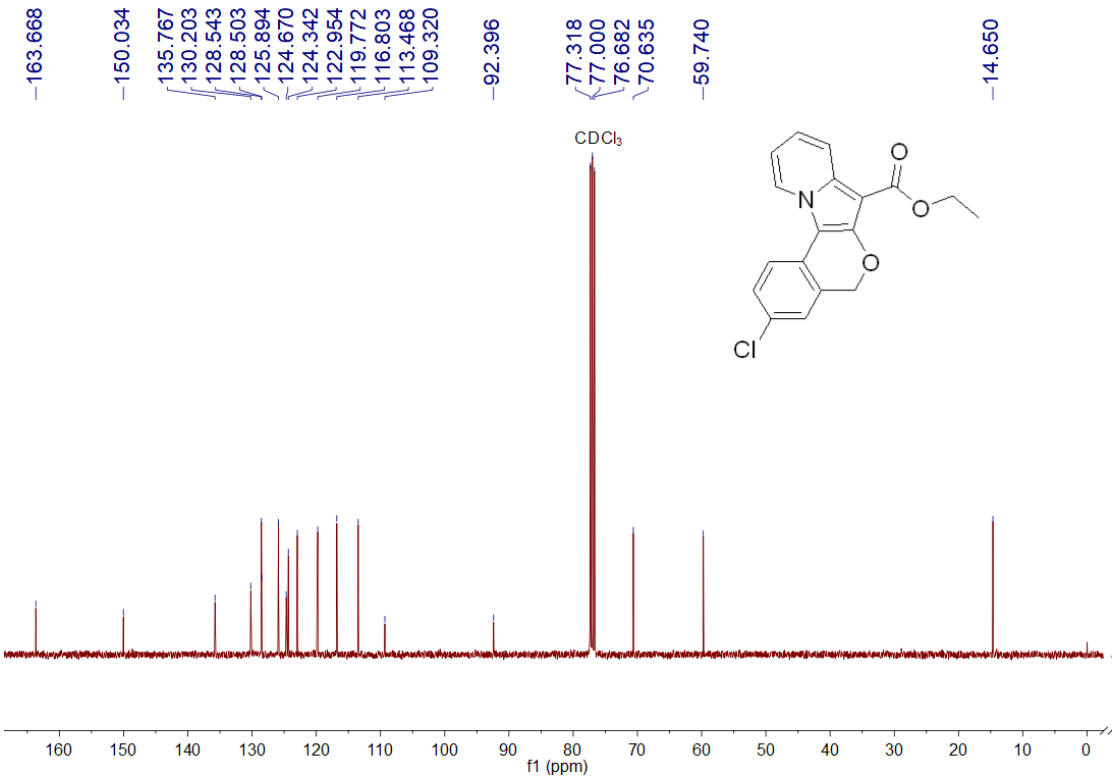
^{13}C{^{1}H} NMR of **3b** ($CDCl_3$, 100 MHz)



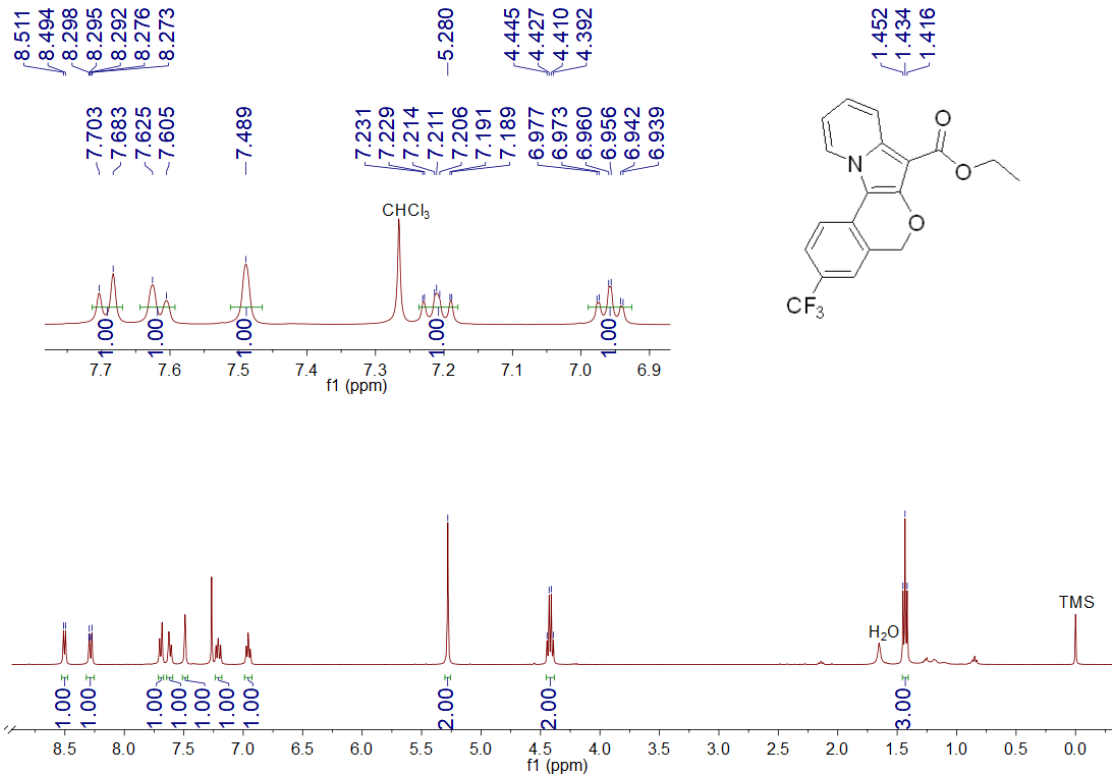
^{19}F NMR of **3b** ($CDCl_3$, 376 MHz)



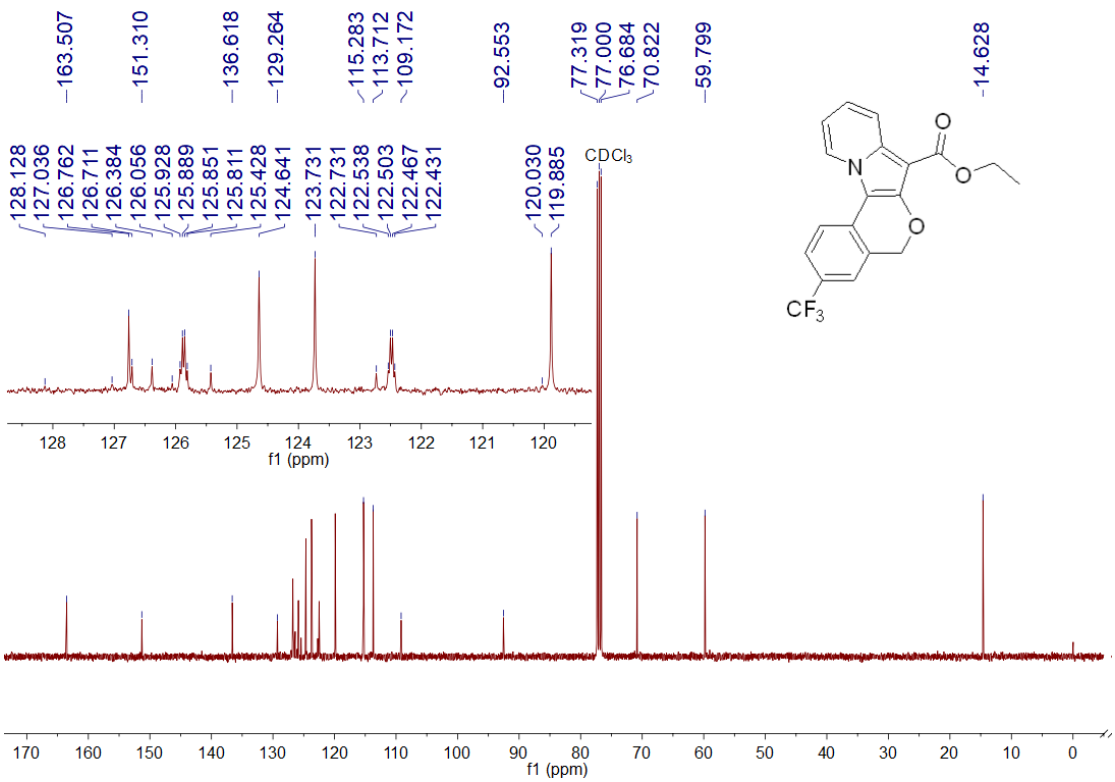
^{1}H NMR of **3c** ($CDCl_3$, 400 MHz)



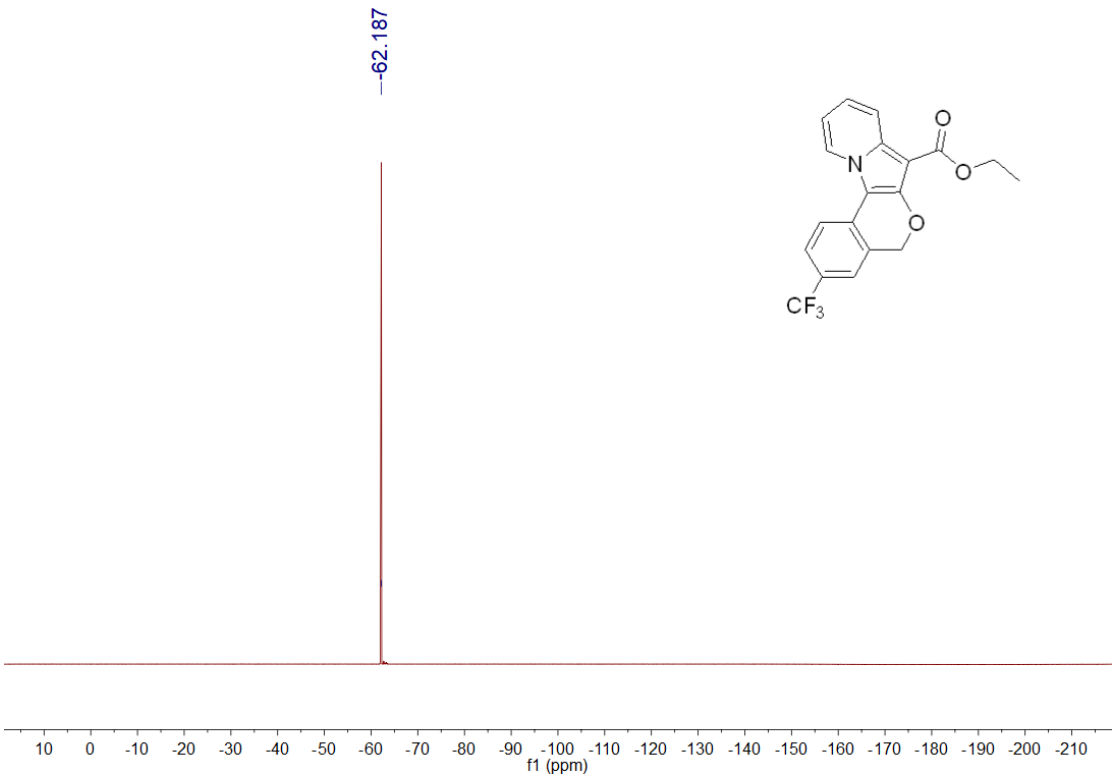
^{13}C{^{1}H} NMR of **3c** ($CDCl_3$, 100 MHz)



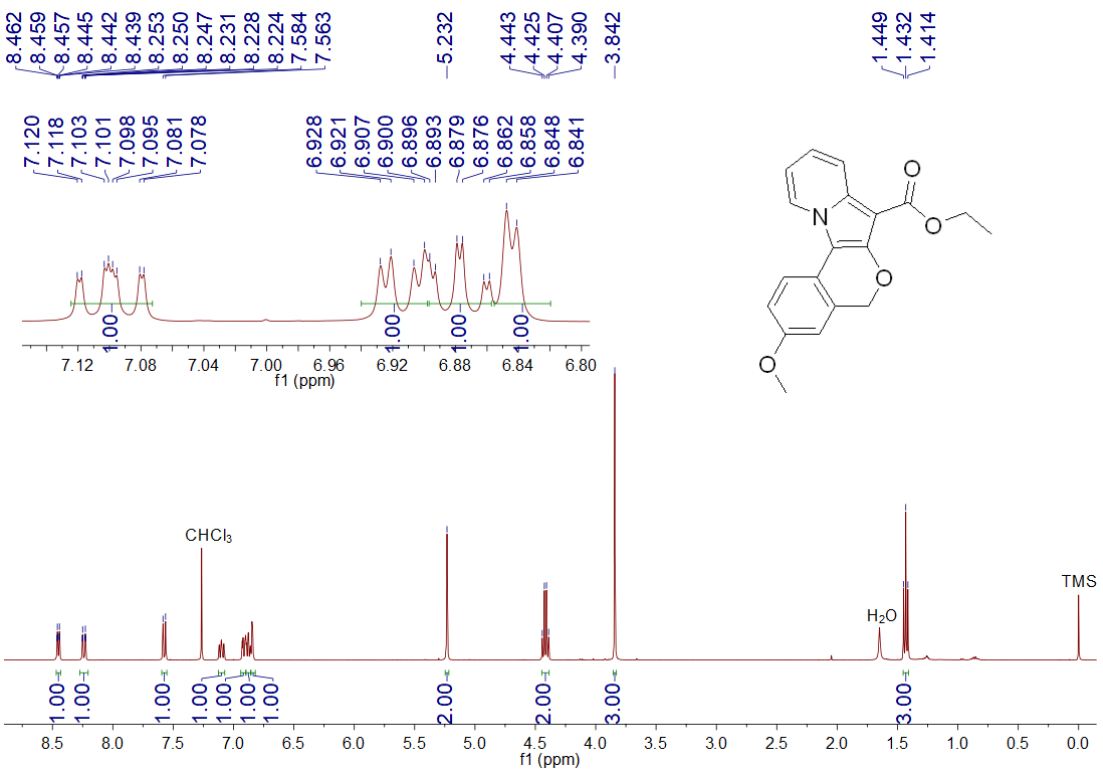
^{1}H NMR of **3d** ($CDCl_3$, 400 MHz)



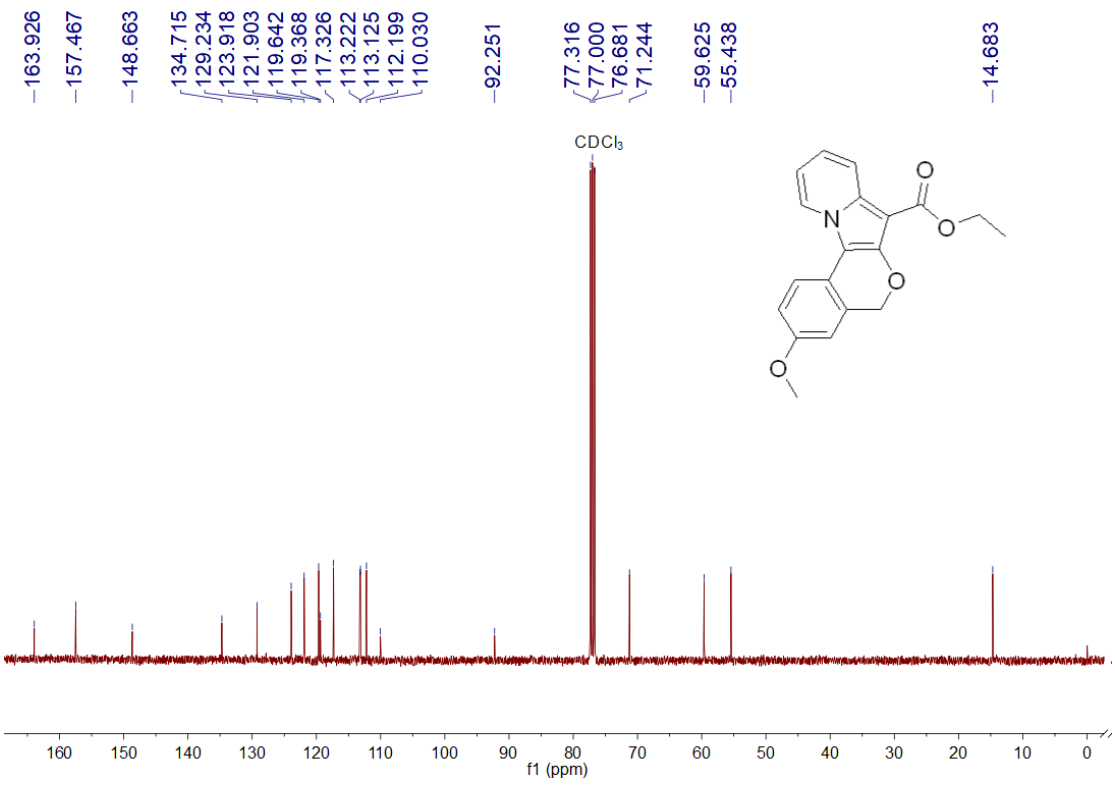
^{13}C{^{1}H} NMR of **3d** ($CDCl_3$, 100 MHz)



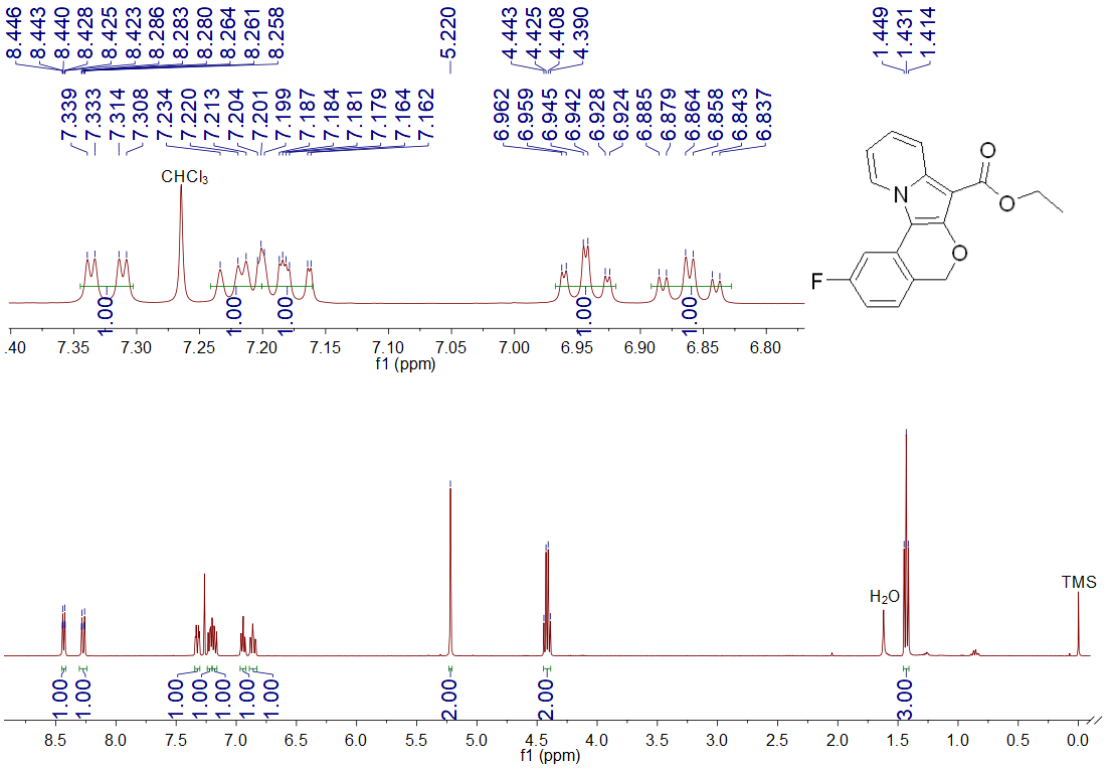
^{19}F NMR of **3d** ($CDCl_3$, 376 MHz)



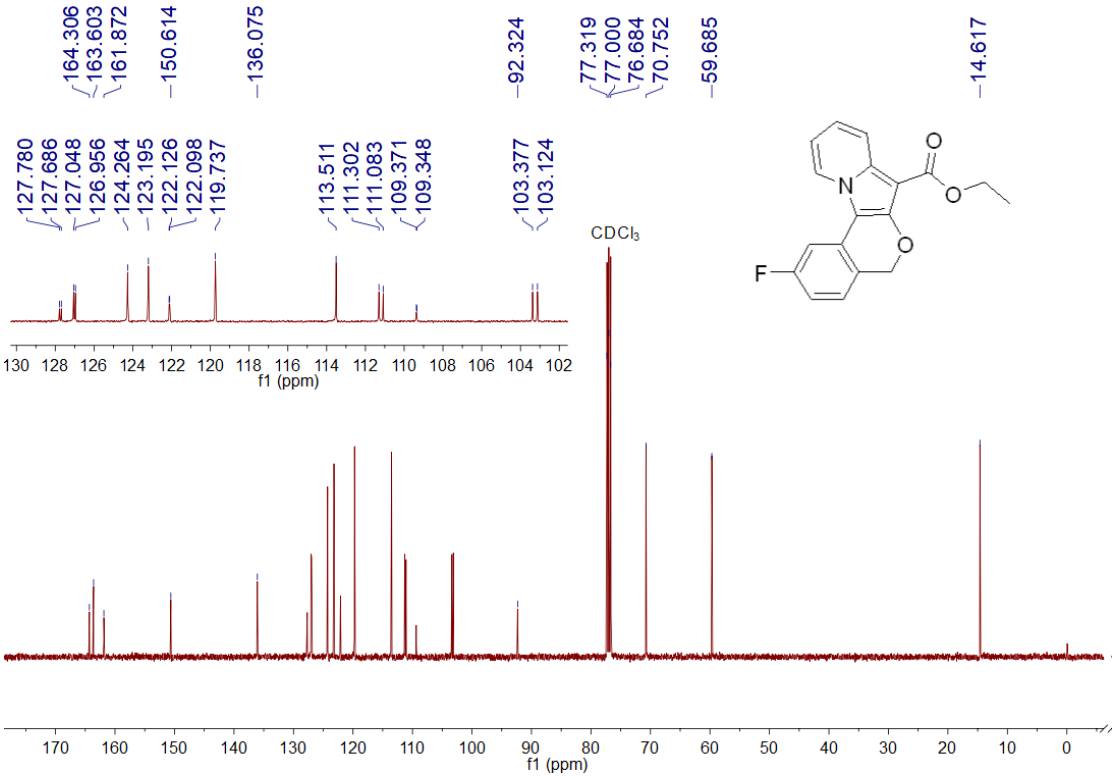
^{1}H NMR of **3e** ($CDCl_3$, 400 MHz)



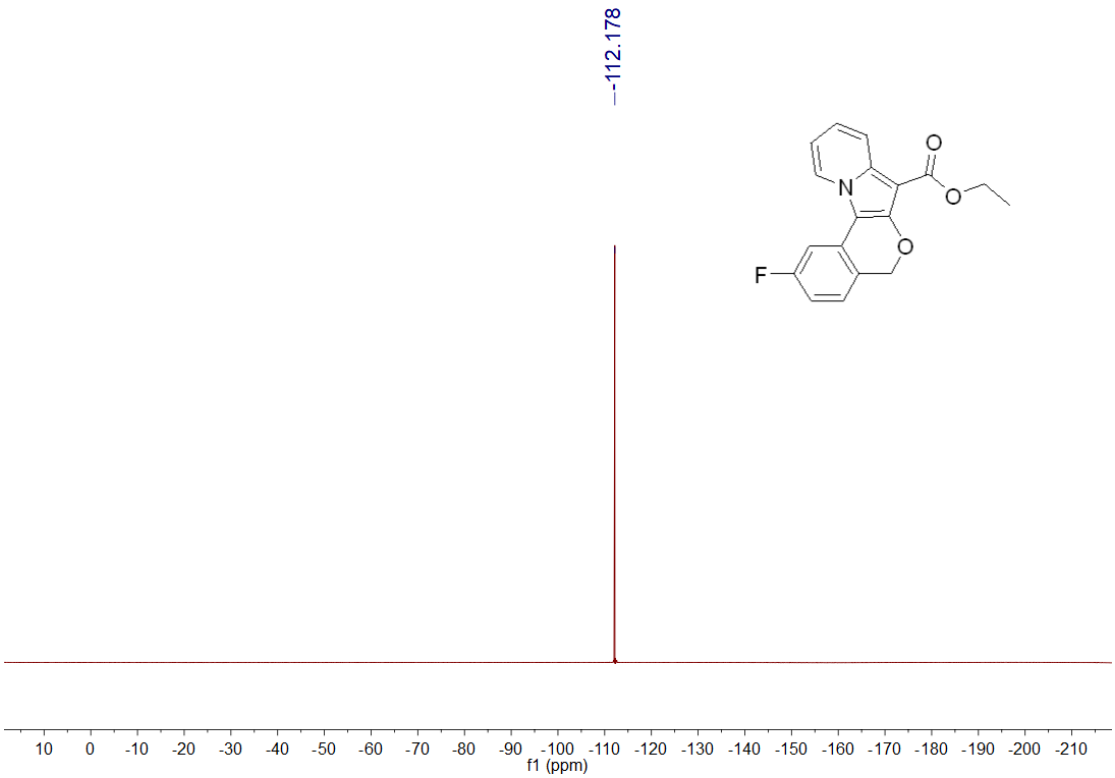
^{13}C{^{1}H} NMR of **3e** ($CDCl_3$, 100 MHz)



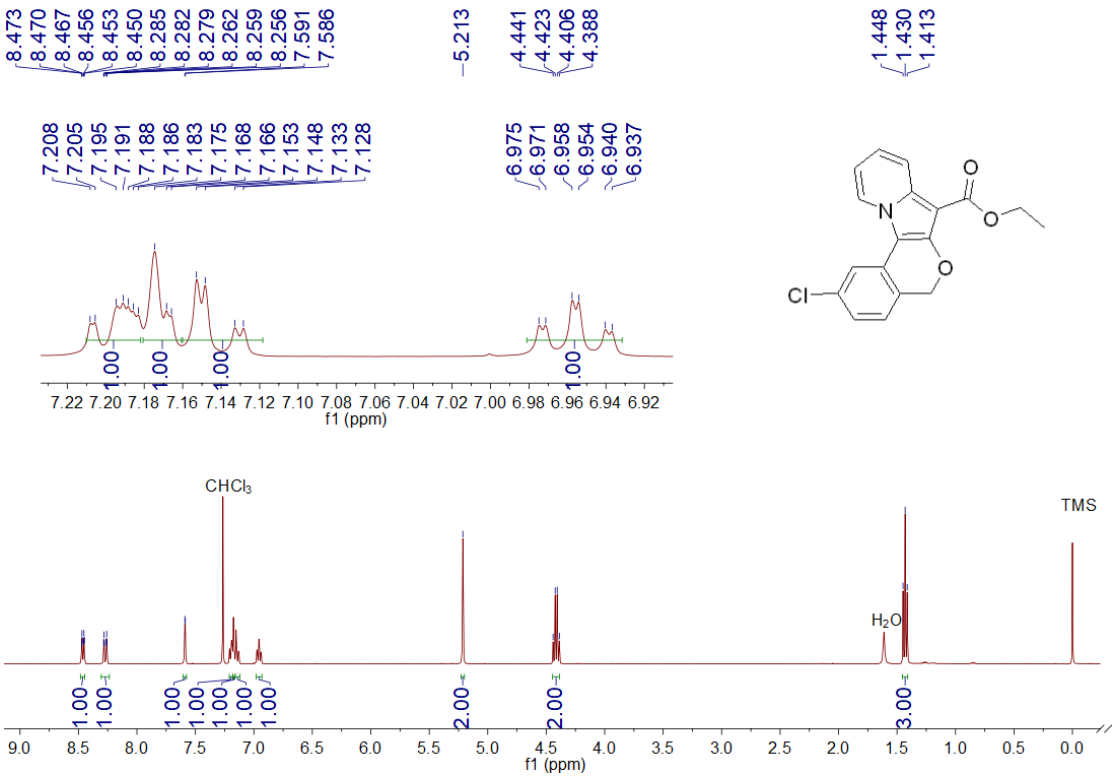
^{1}H NMR of **3f** ($CDCl_3$, 400 MHz)



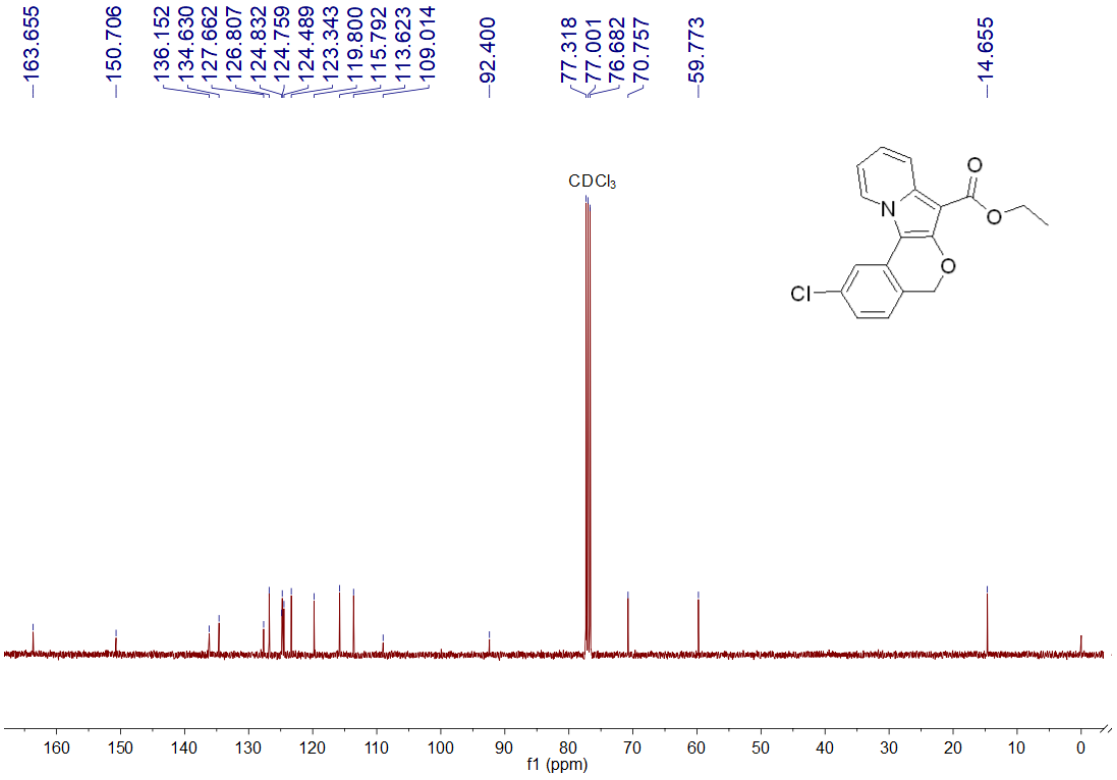
^{13}C{^{1}H} NMR of **3f** ($CDCl_3$, 100 MHz)



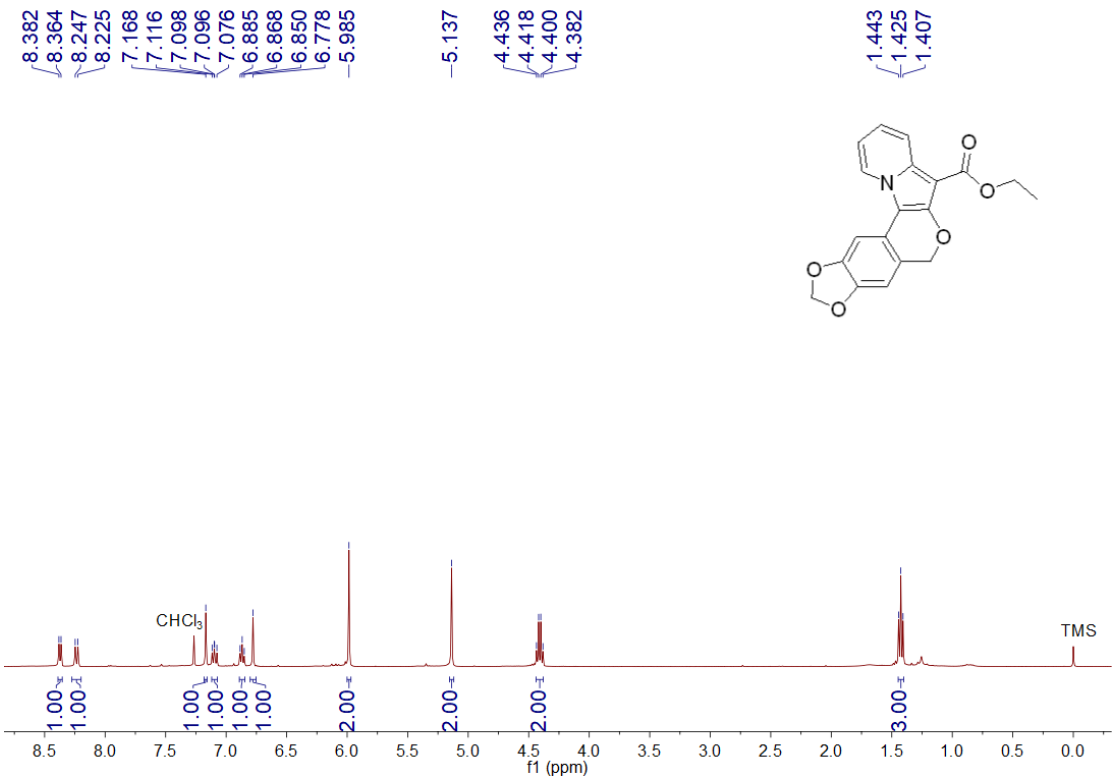
^{19}F NMR of **3f** ($CDCl_3$, 376 MHz)



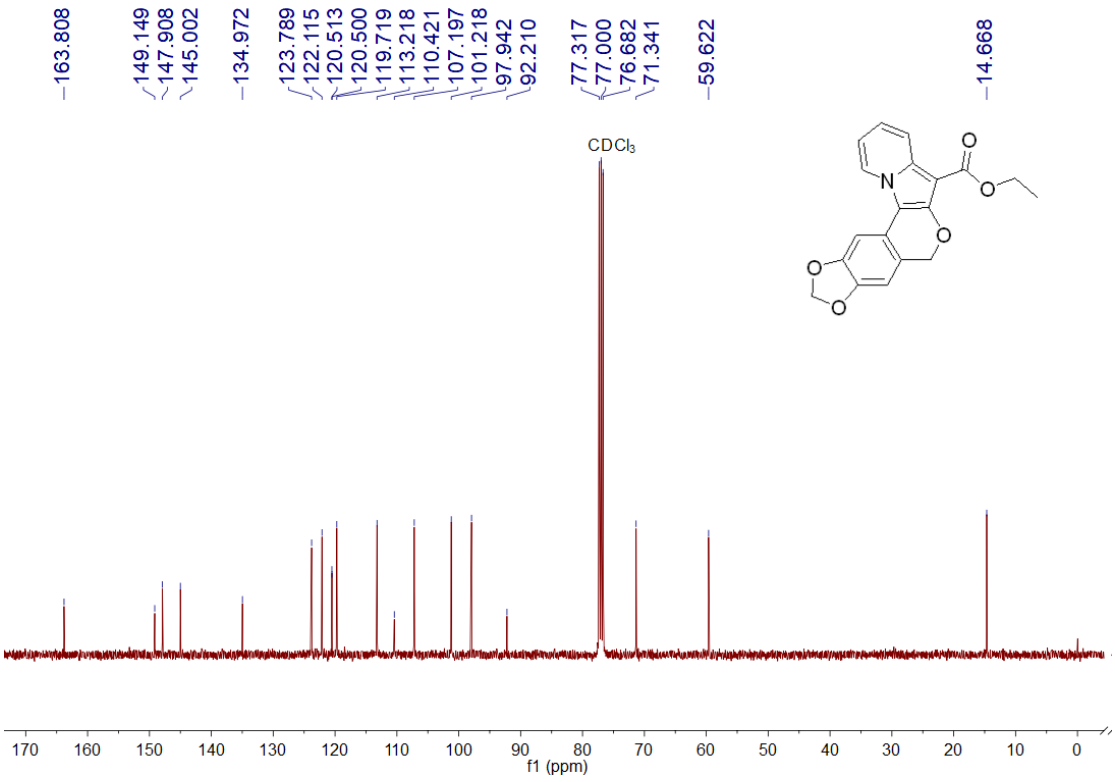
^{1}H NMR of **3g** ($CDCl_3$, 400 MHz)



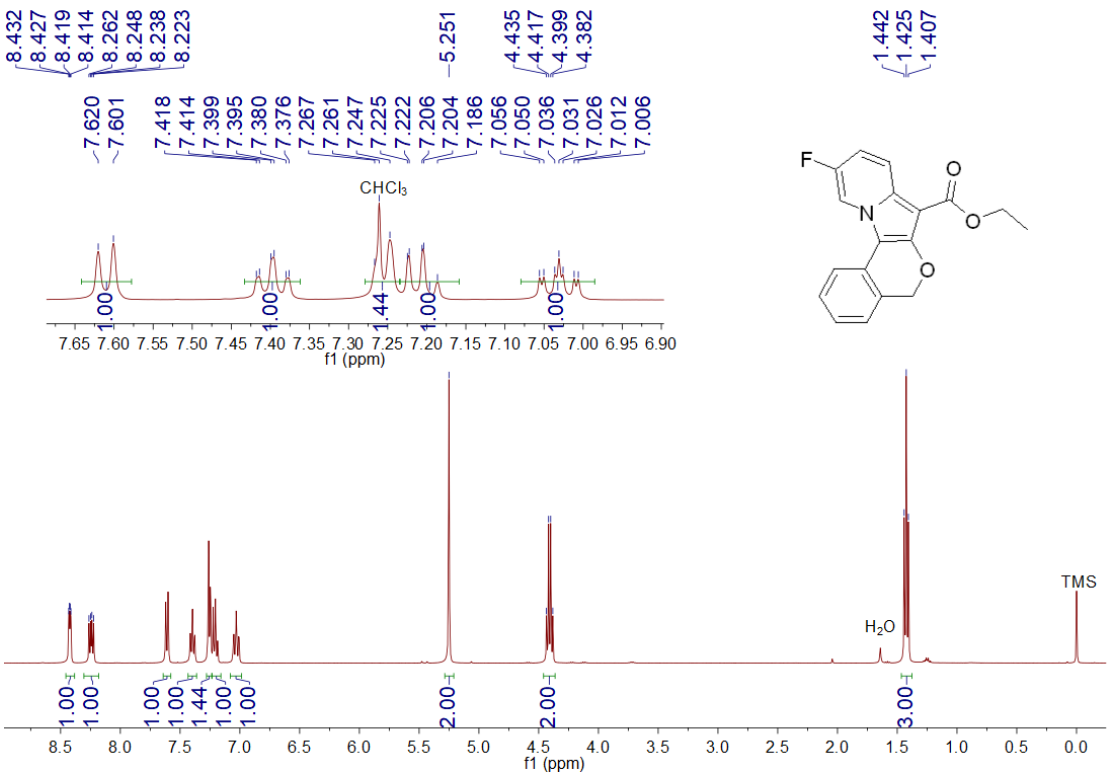
^{13}C{^{1}H} NMR of **3g** ($CDCl_3$, 100 MHz)



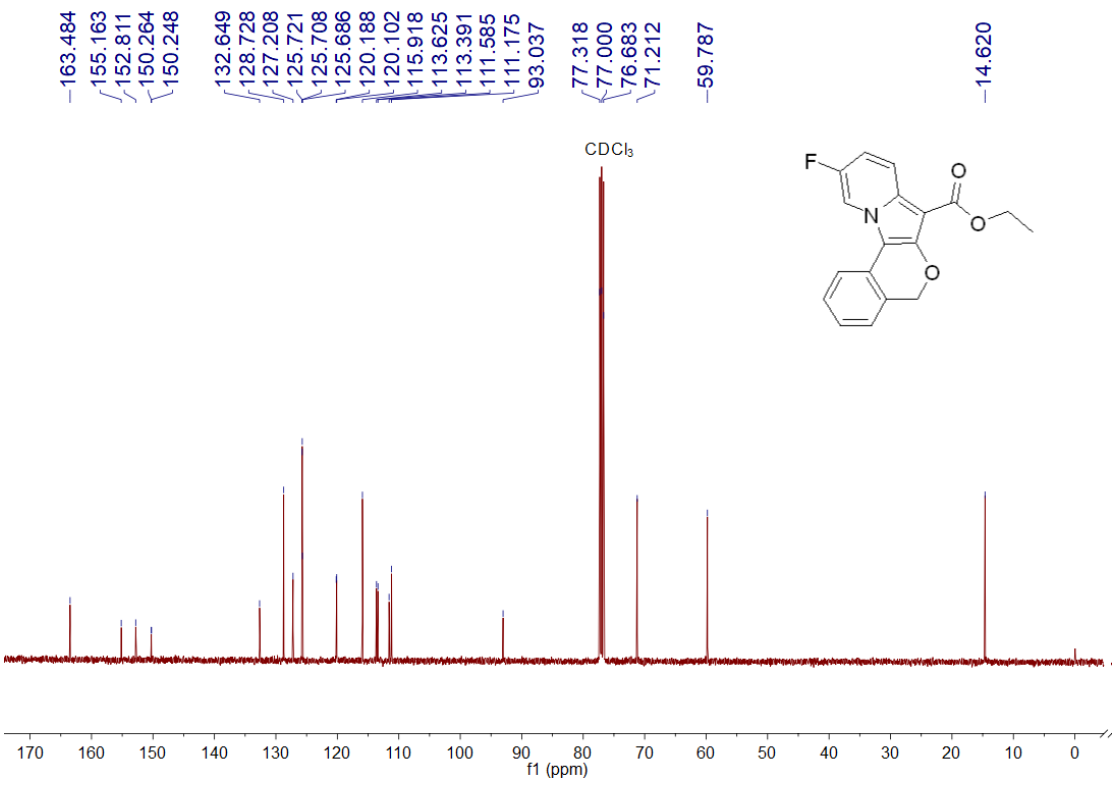
^{1}H NMR of **3h** ($CDCl_3$, 400 MHz)



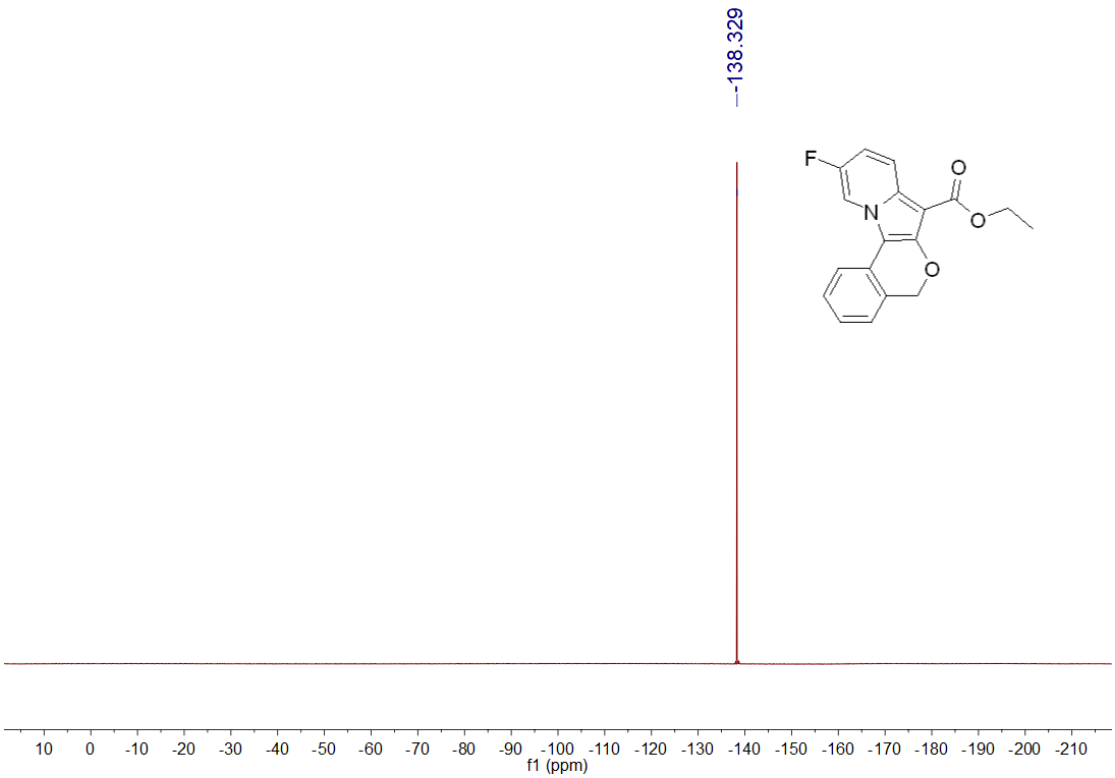
^{13}C{^{1}H} NMR of **3h** ($CDCl_3$, 100 MHz)



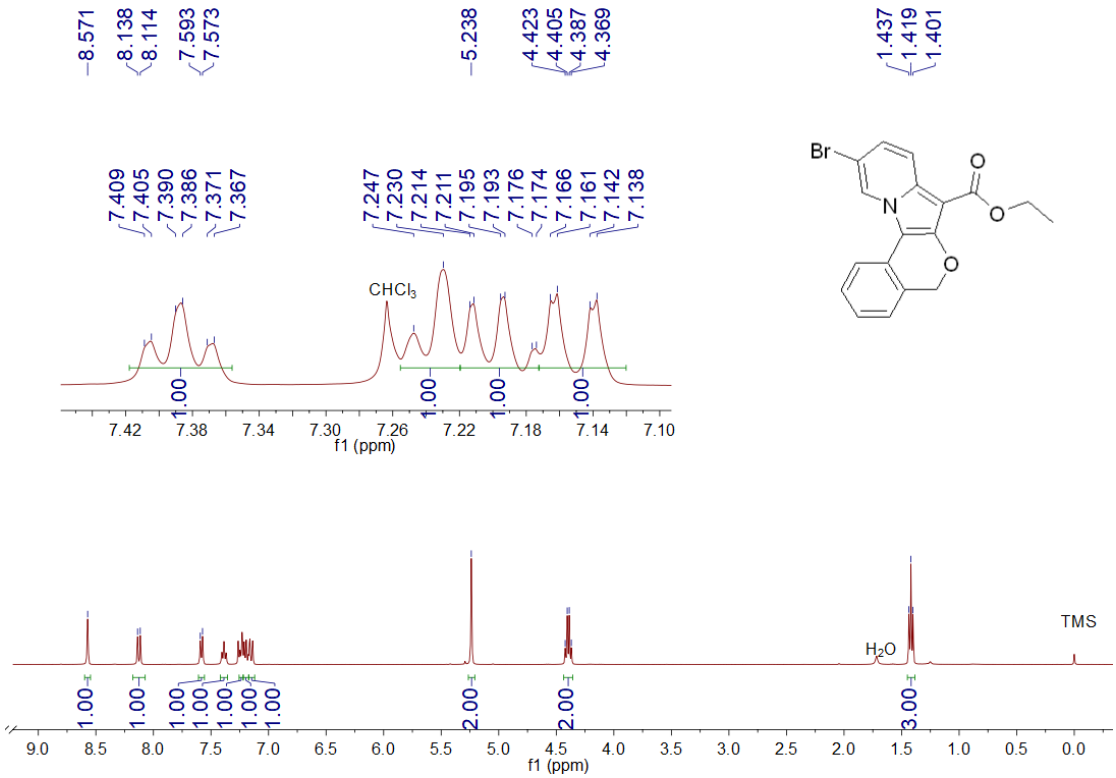
^{1}H NMR of **3i** ($CDCl_3$, 400 MHz)



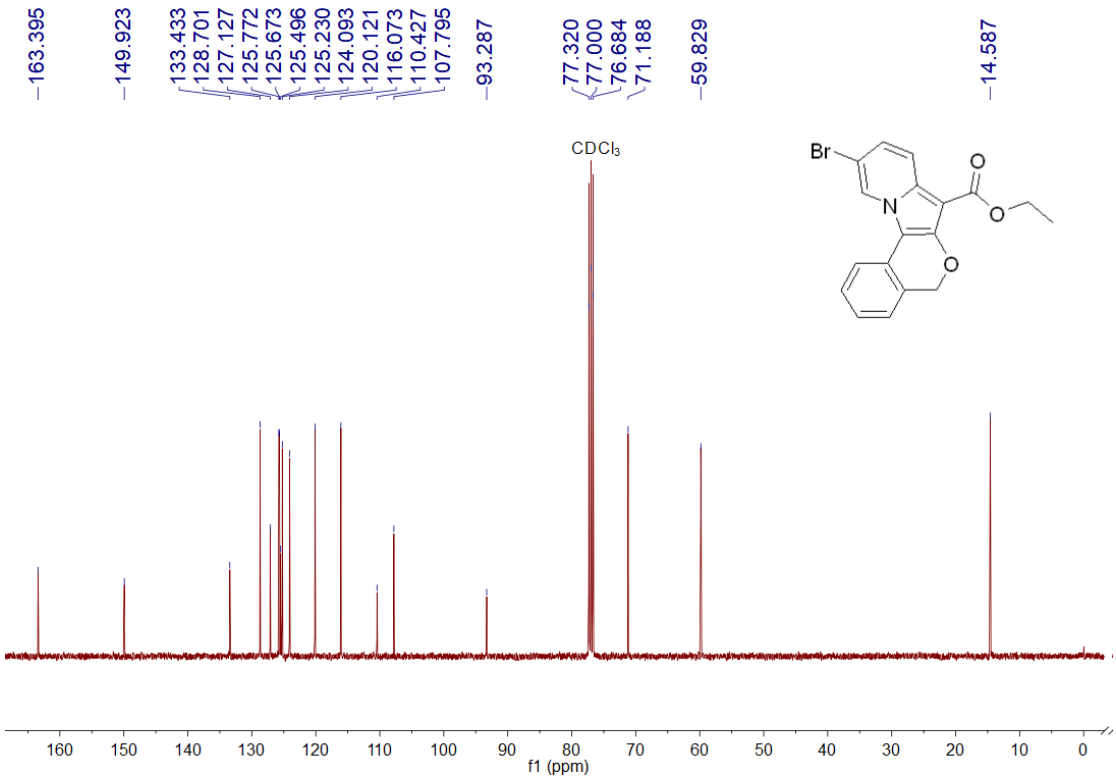
^{13}C{^{1}H} NMR of **3i** ($CDCl_3$, 100 MHz)



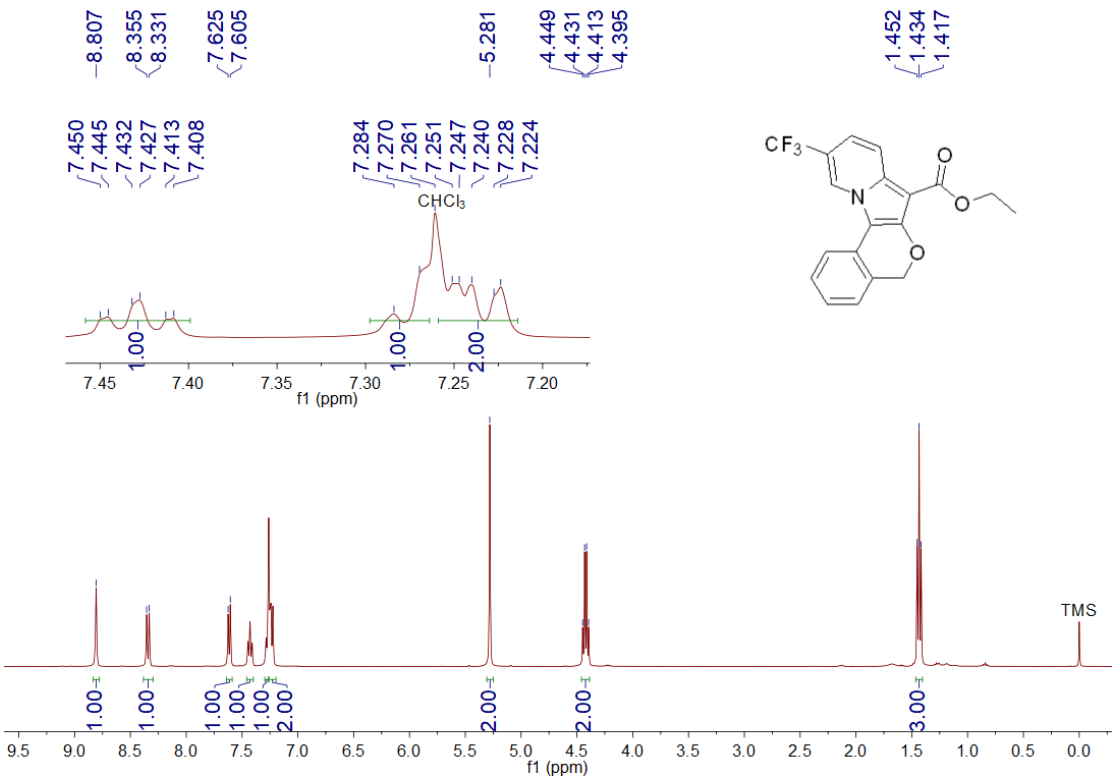
^{19}F NMR of **3i** ($CDCl_3$, 376 MHz)



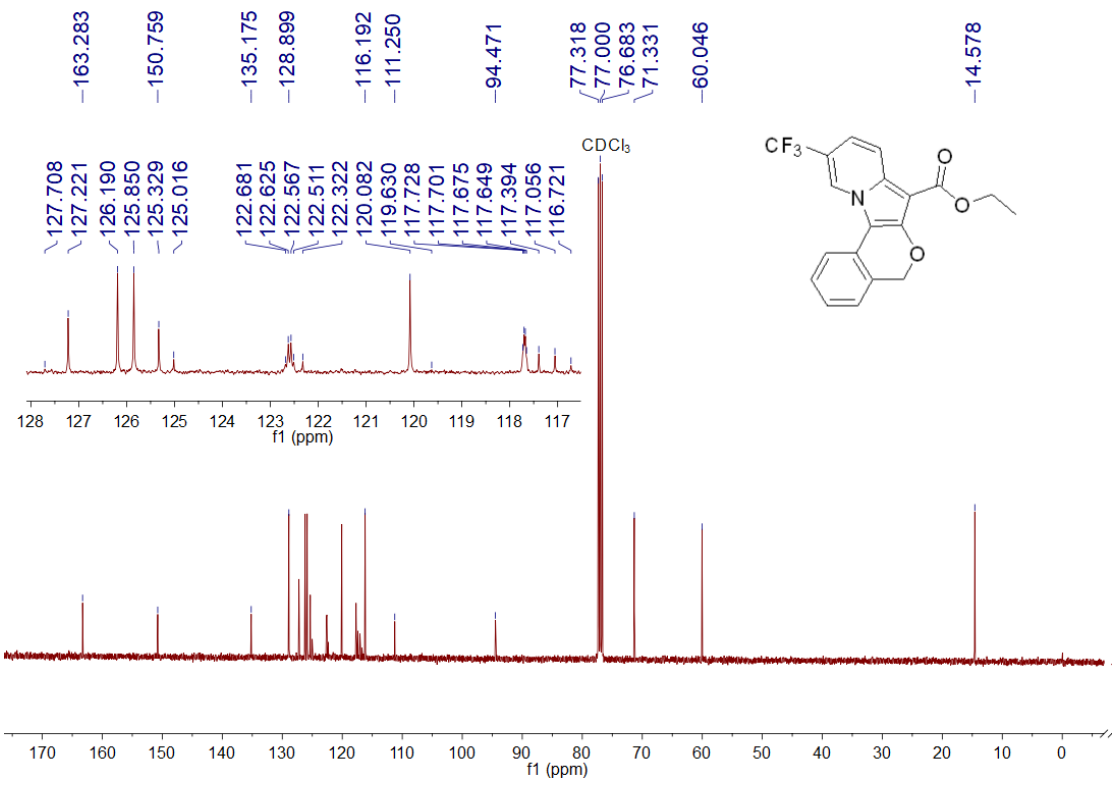
^{1}H NMR of **3j** ($CDCl_3$, 400 MHz)



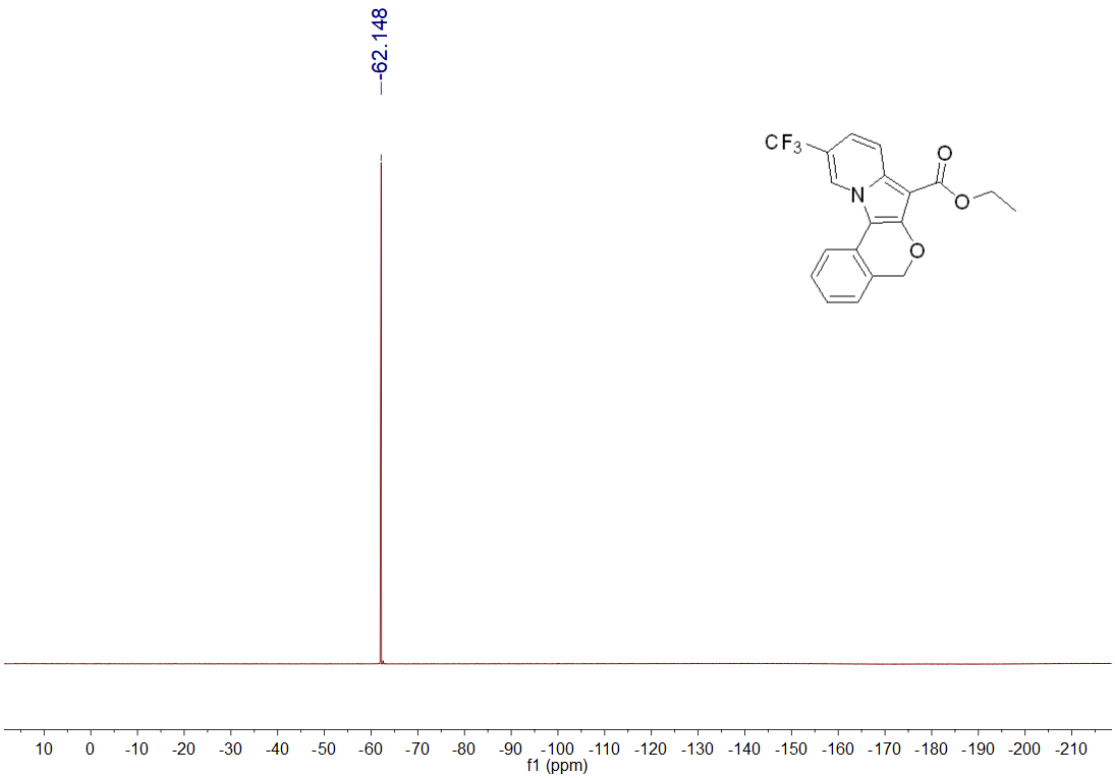
^{13}C{^{1}H} NMR of **3j** ($CDCl_3$, 100 MHz)



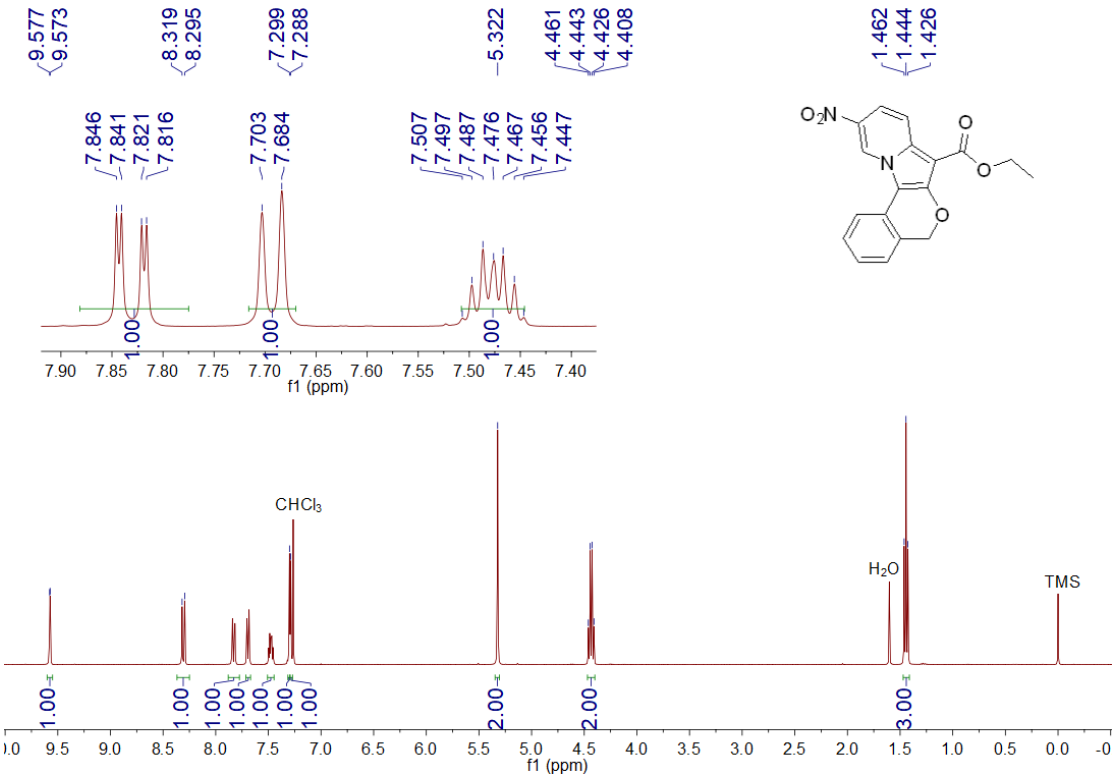
^{1}H NMR of **3k** ($CDCl_3$, 400 MHz)



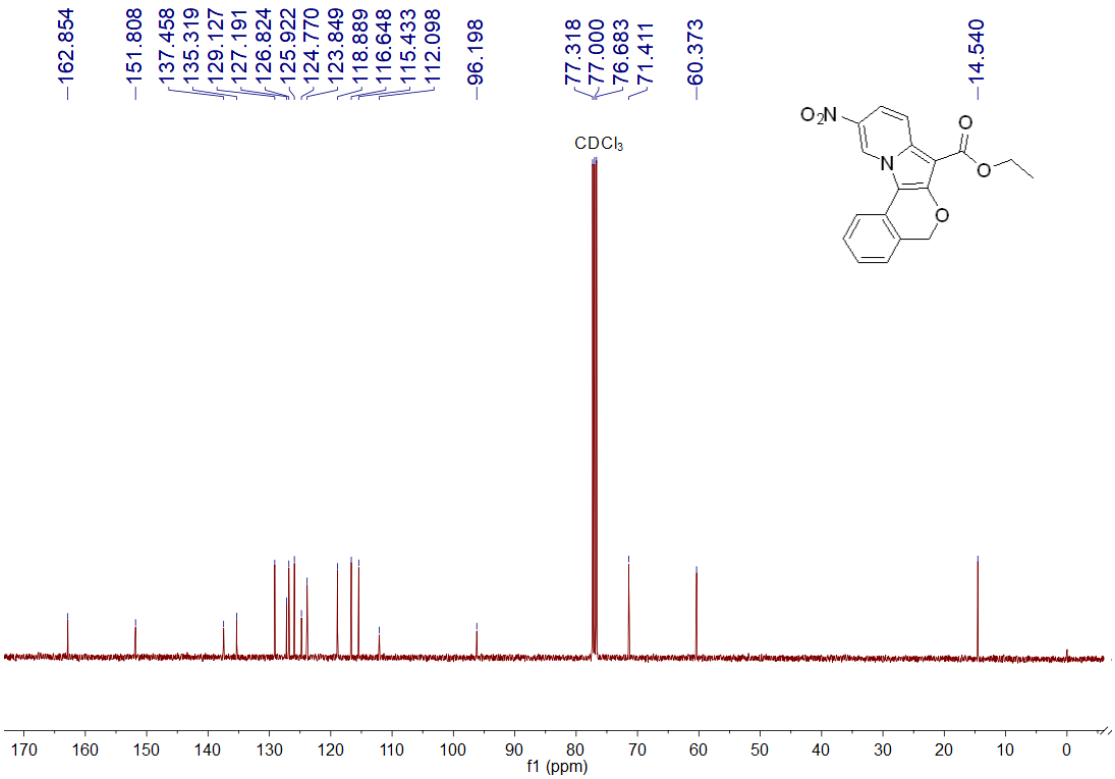
^{13}C{^{1}H} NMR of **3k** ($CDCl_3$, 100 MHz)



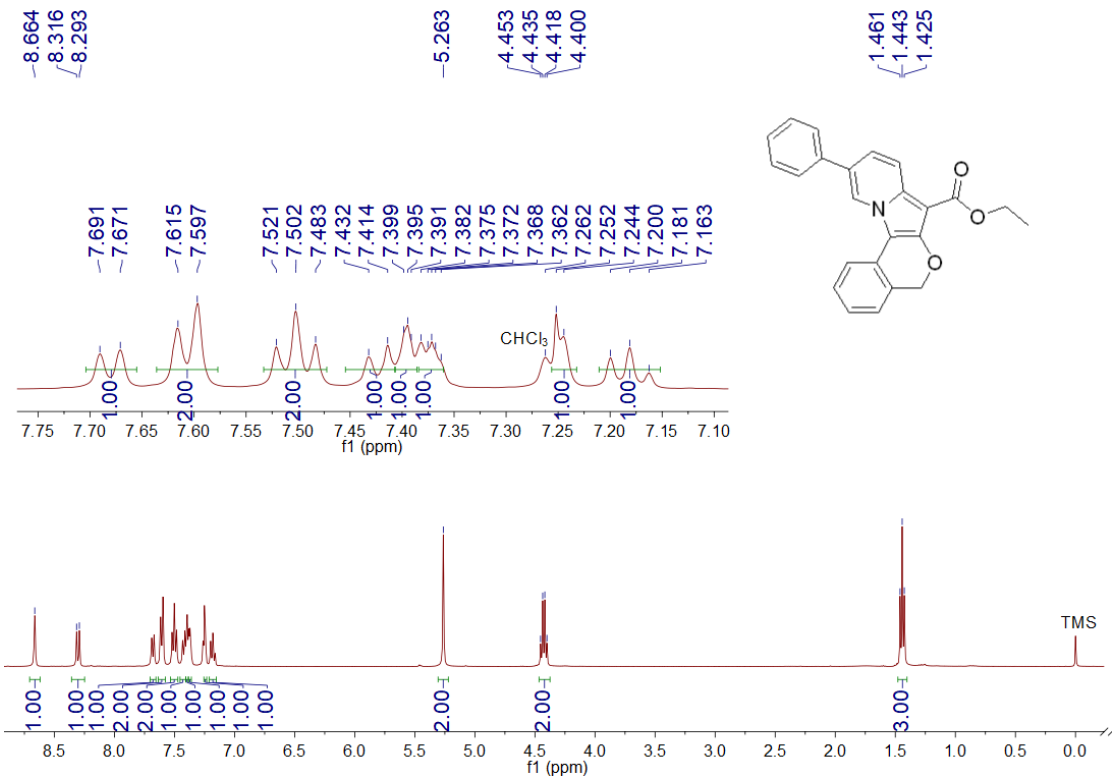
^{19}F NMR of **3k** ($CDCl_3$, 376 MHz)



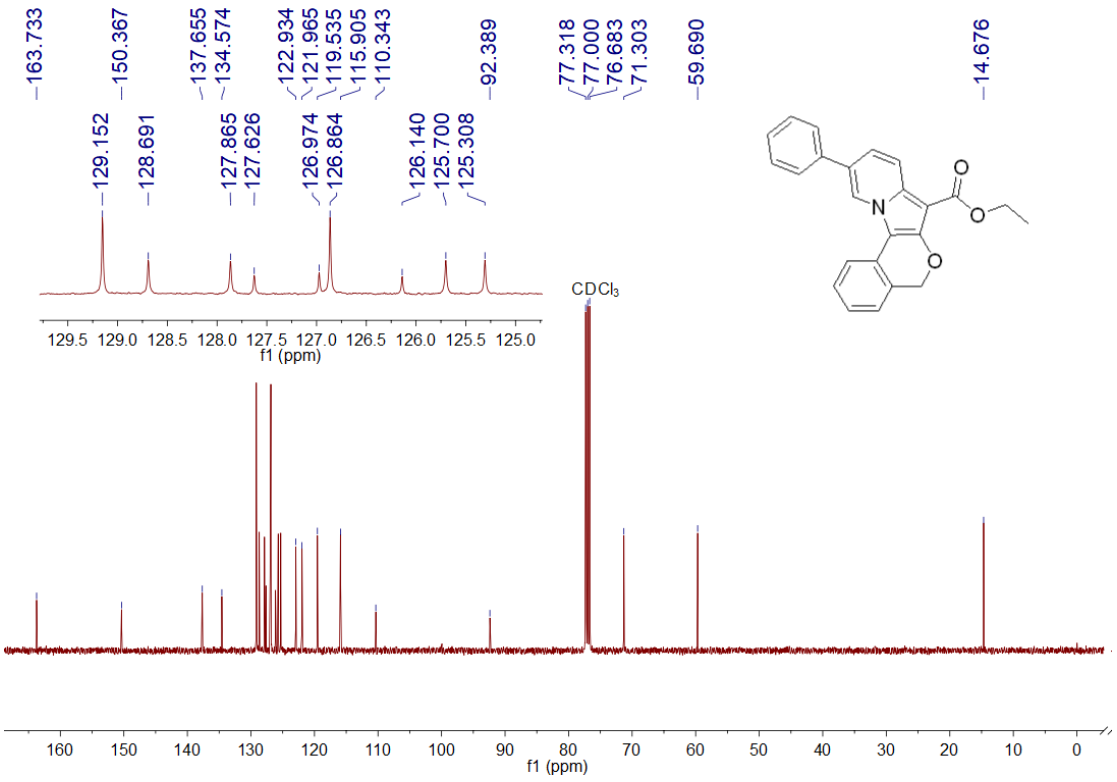
^{1}H NMR of **3l** ($CDCl_3$, 400 MHz)



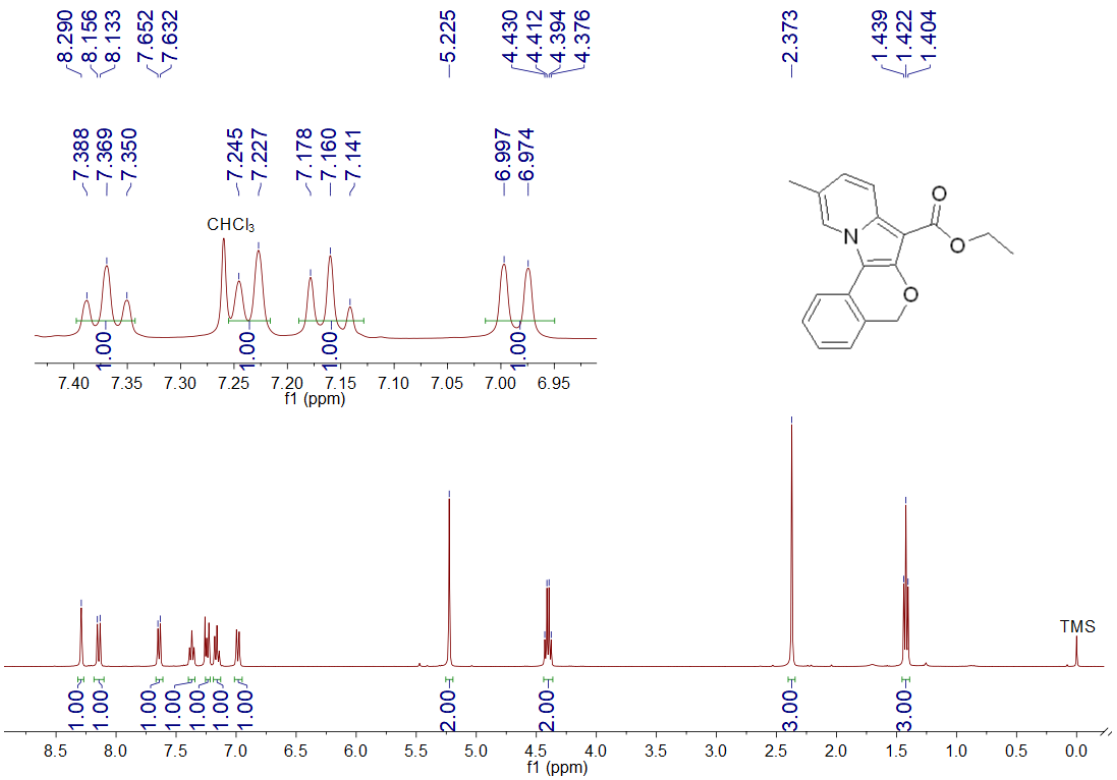
^{13}C{^{1}H} NMR of **3l** ($CDCl_3$, 100 MHz)



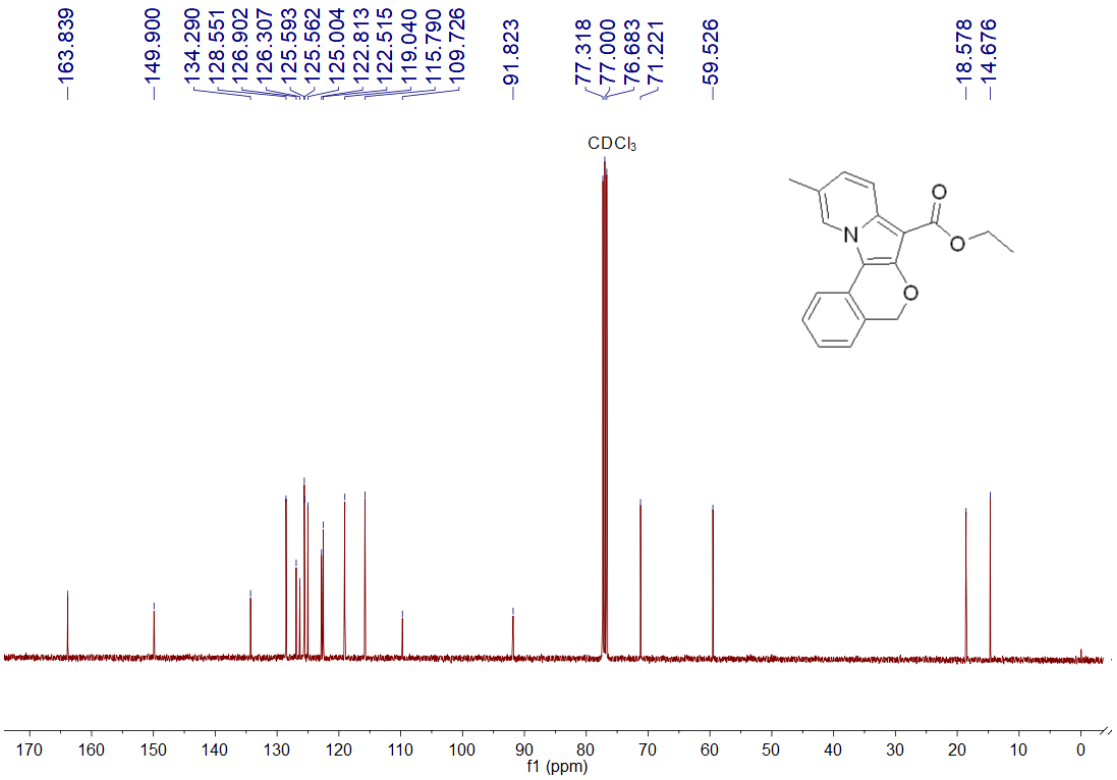
^{1}H NMR of **3m** ($CDCl_3$, 400 MHz)



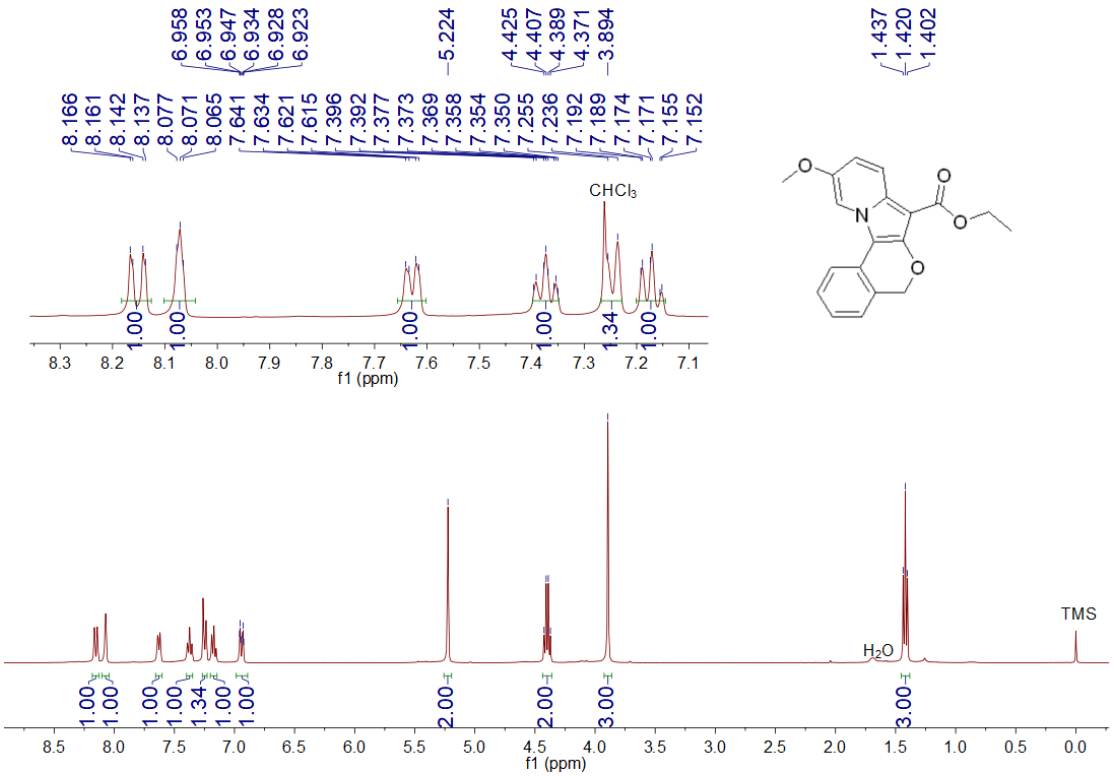
^{13}C{^{1}H} NMR of **3m** ($CDCl_3$, 100 MHz)



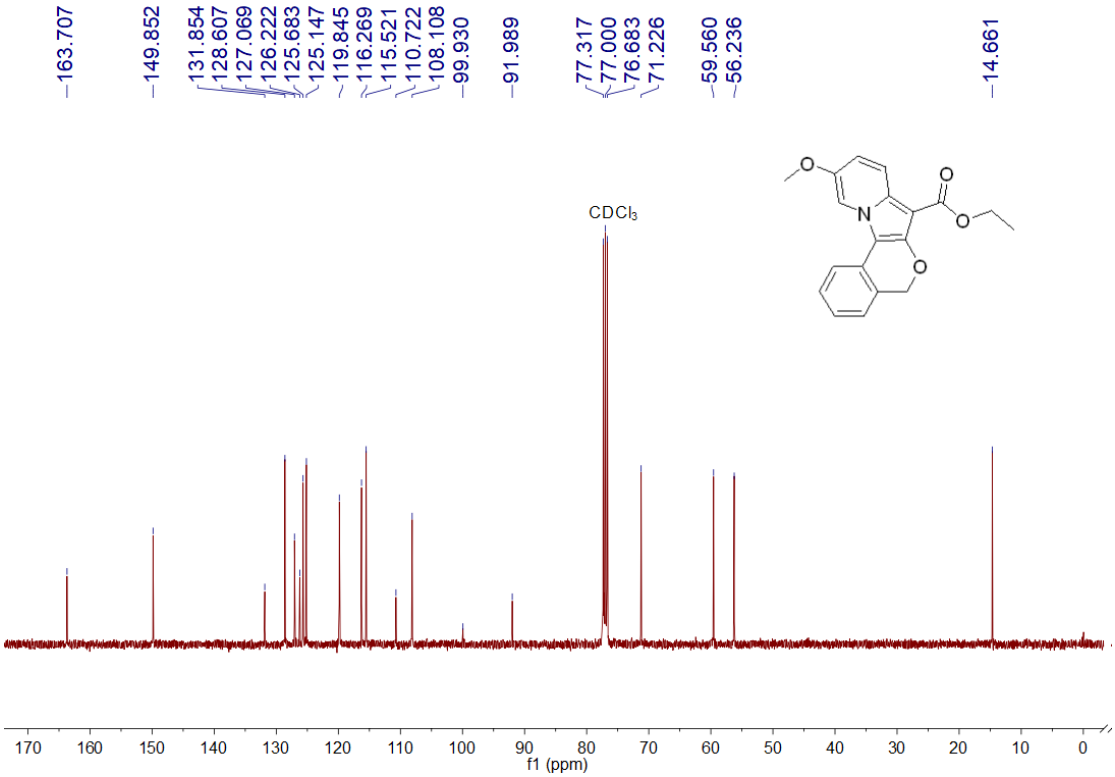
^{1}H NMR of **3n** ($CDCl_3$, 400 MHz)



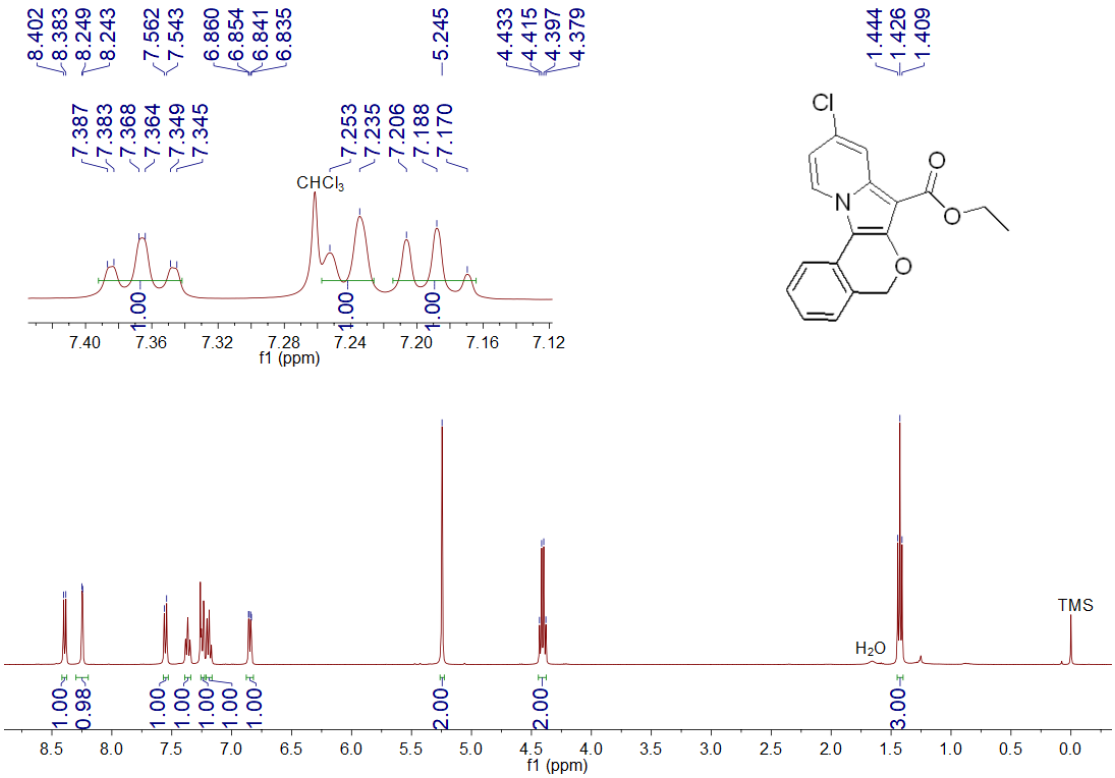
^{13}C{^{1}H} NMR of **3n** ($CDCl_3$, 100 MHz)



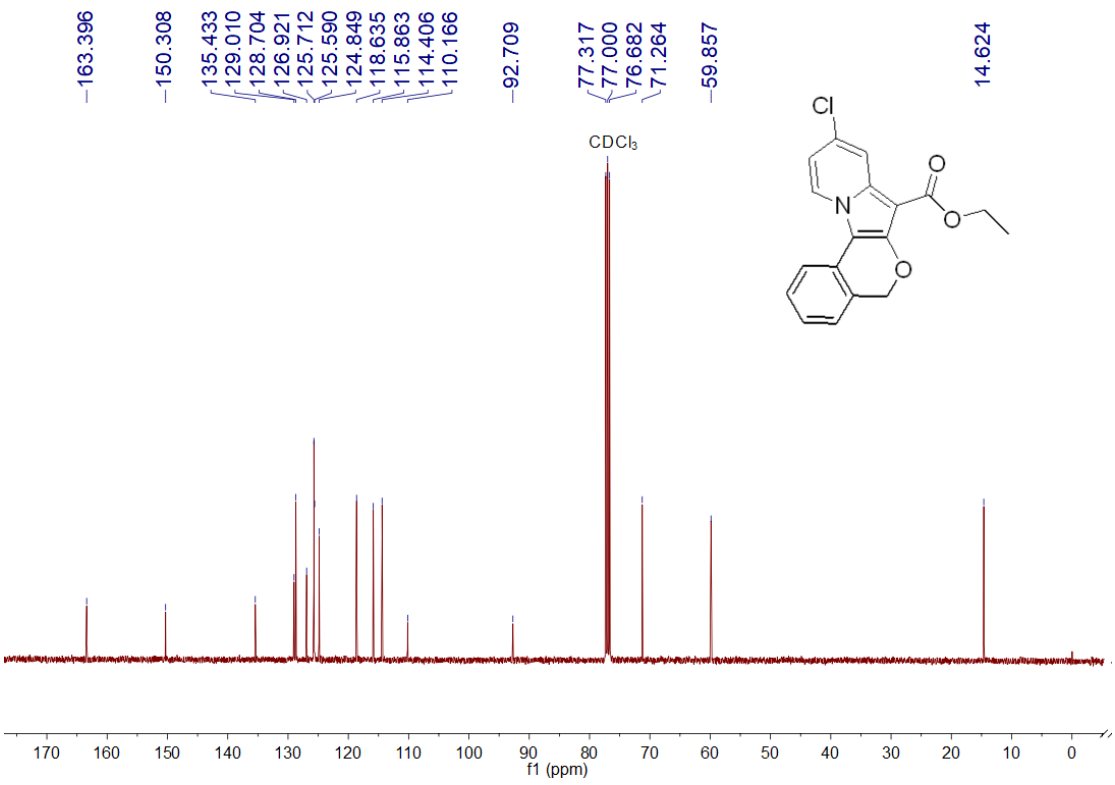
^{1}H NMR of **3o** ($CDCl_3$, 400 MHz)



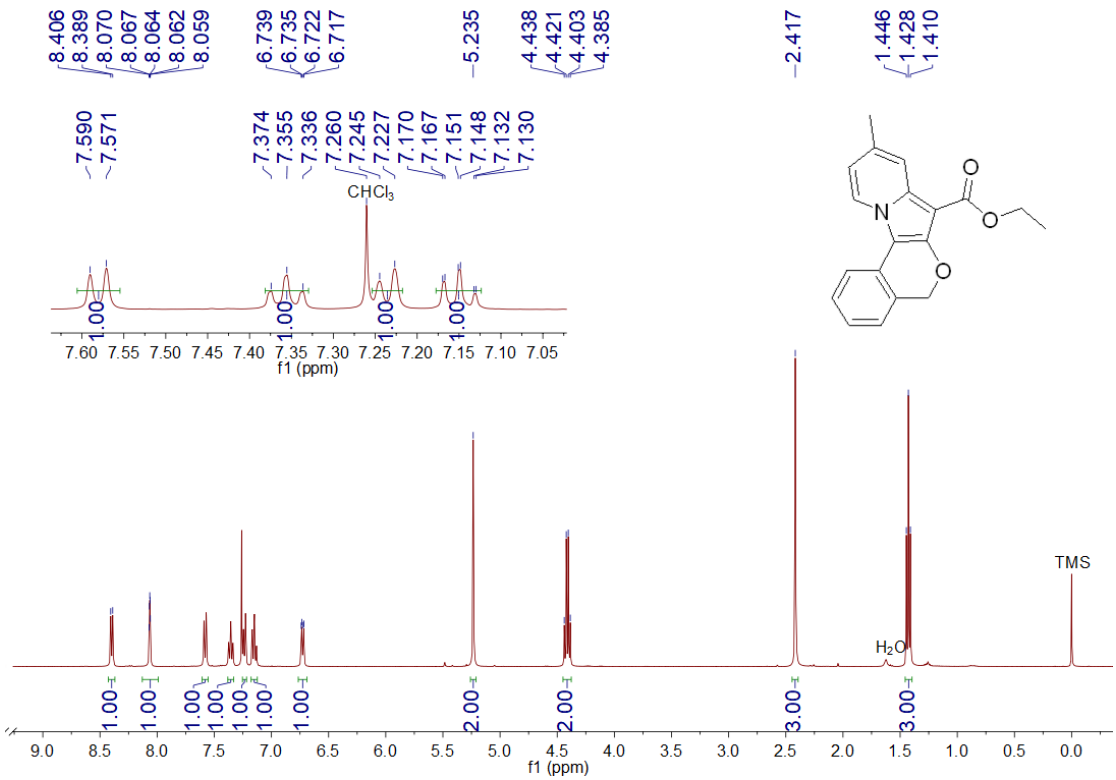
^{13}C{^{1}H} NMR of **3o** ($CDCl_3$, 100 MHz)



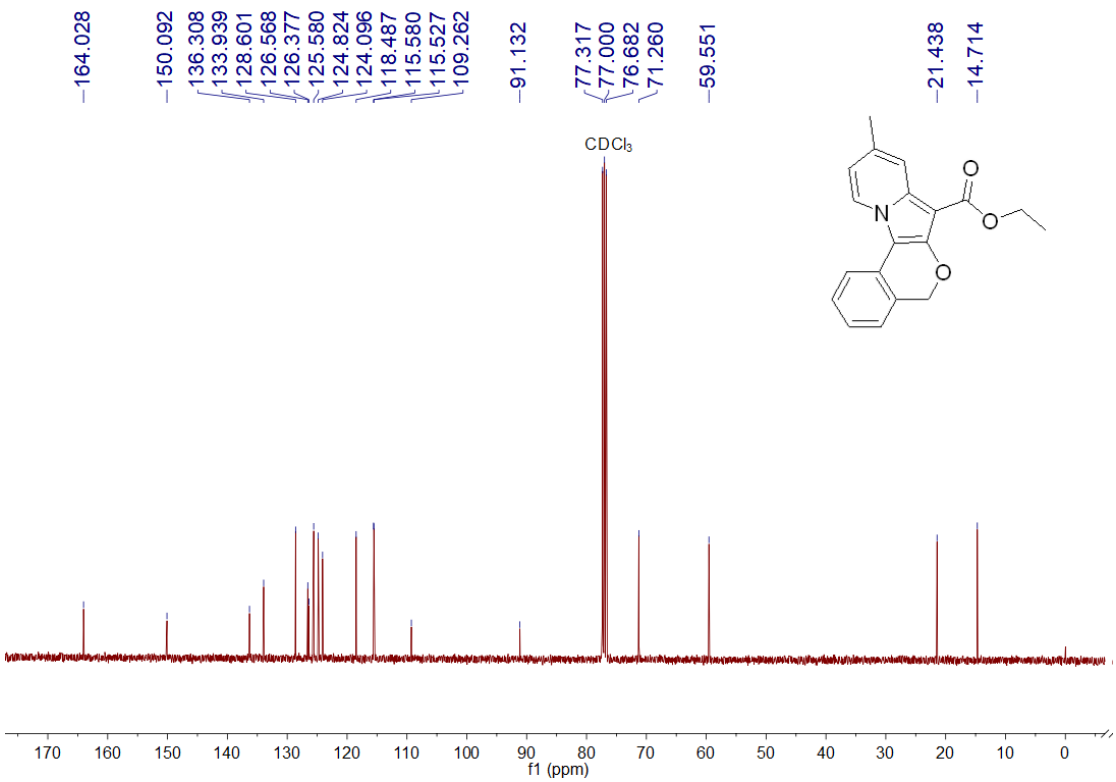
^{1}H NMR of **3p** ($CDCl_3$, 400 MHz)



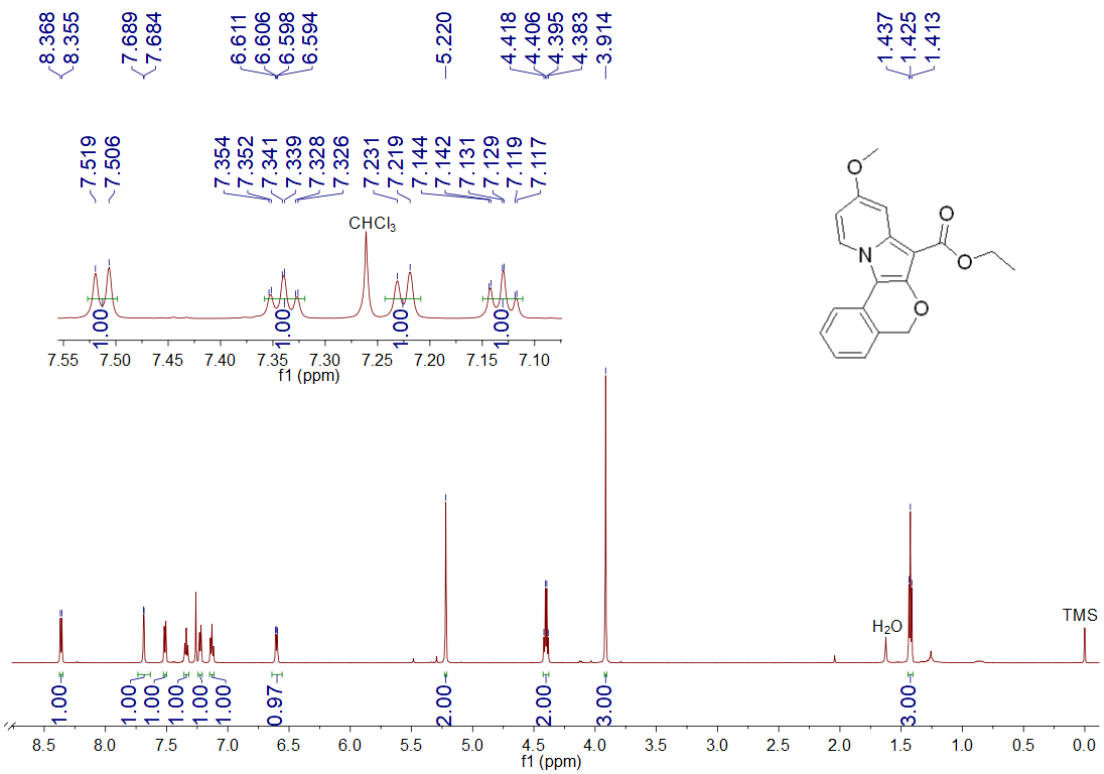
^{13}C{^{1}H} NMR of **3p** ($CDCl_3$, 100 MHz)



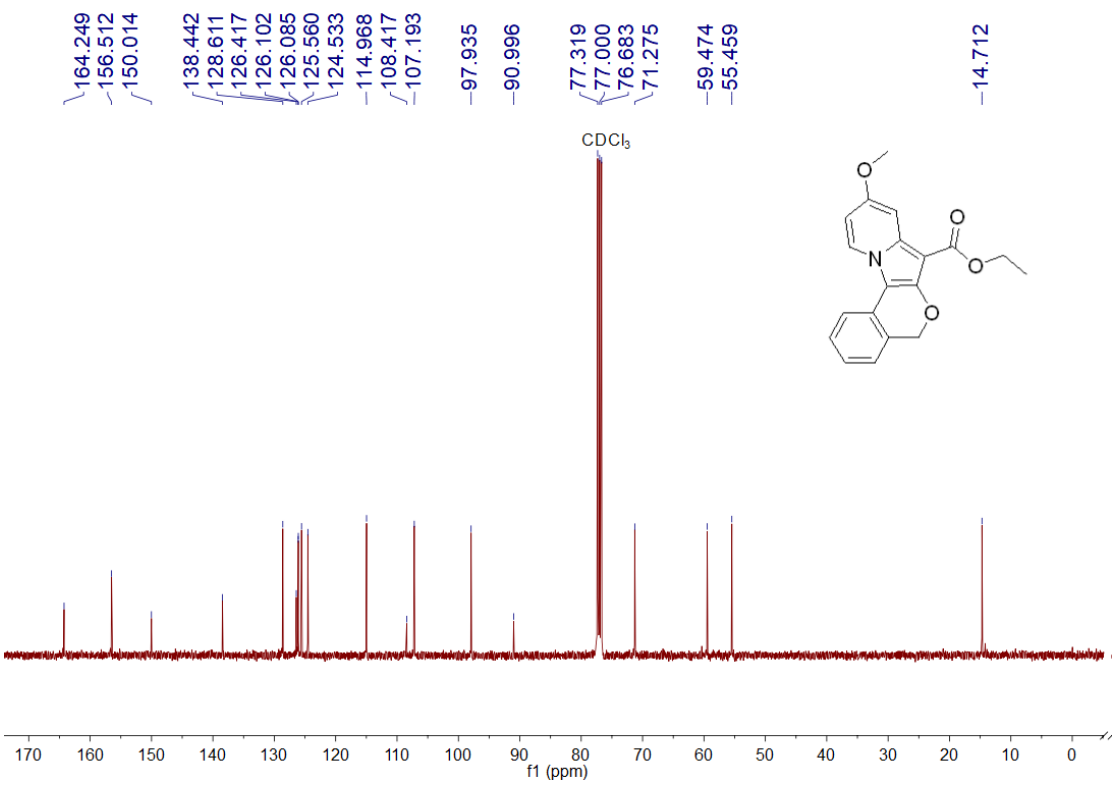
^{1}H NMR of **3q** ($CDCl_3$, 400 MHz)



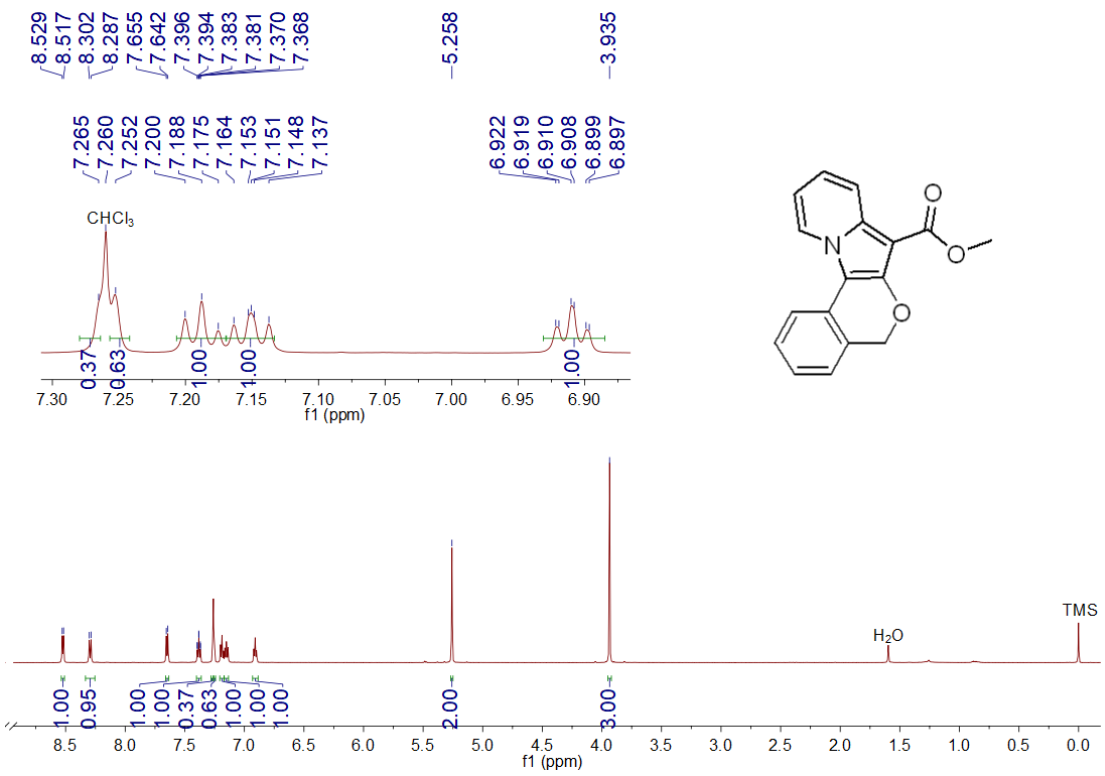
^{13}C{^{1}H} NMR of **3q** ($CDCl_3$, 100 MHz)



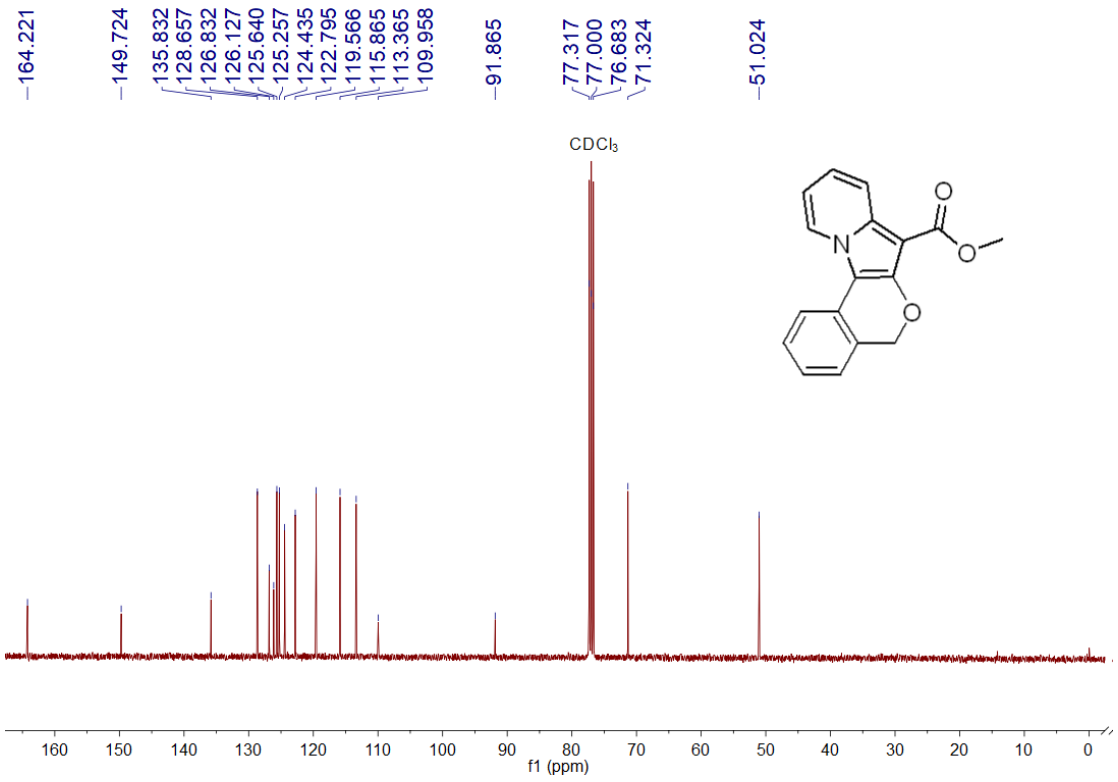
^{1}H NMR of **3r** ($CDCl_3$, 600 MHz)



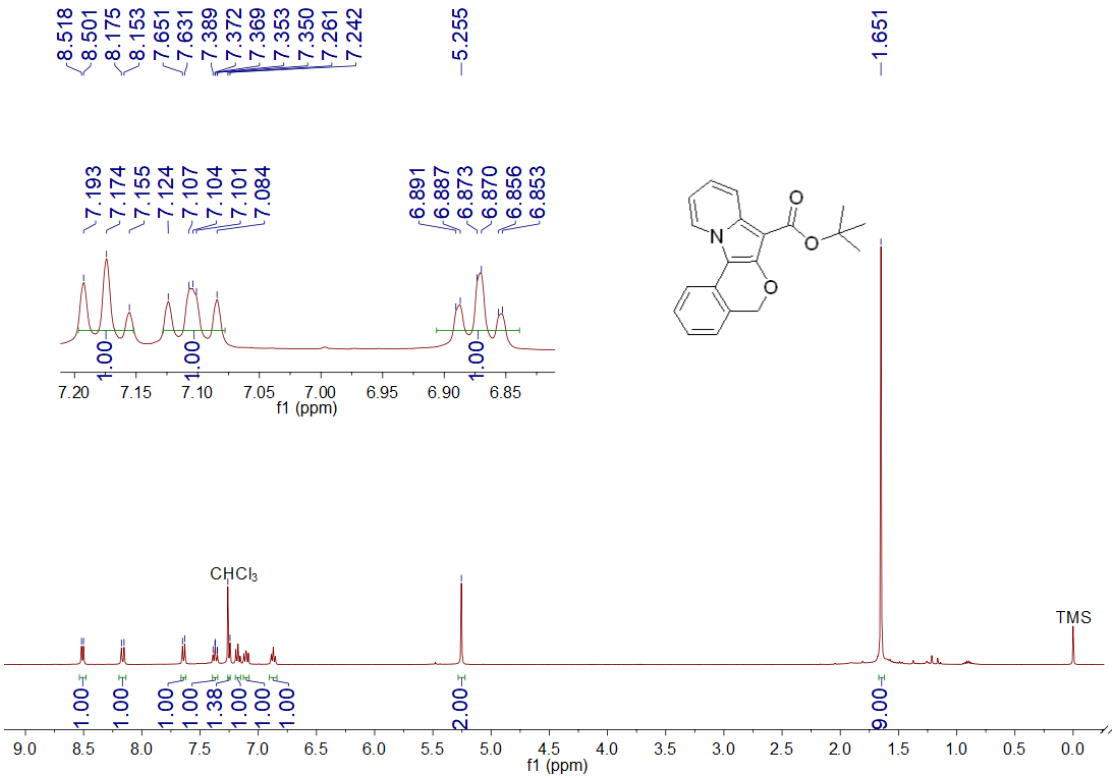
^{13}C{^{1}H} NMR of **3r** ($CDCl_3$, 100 MHz)



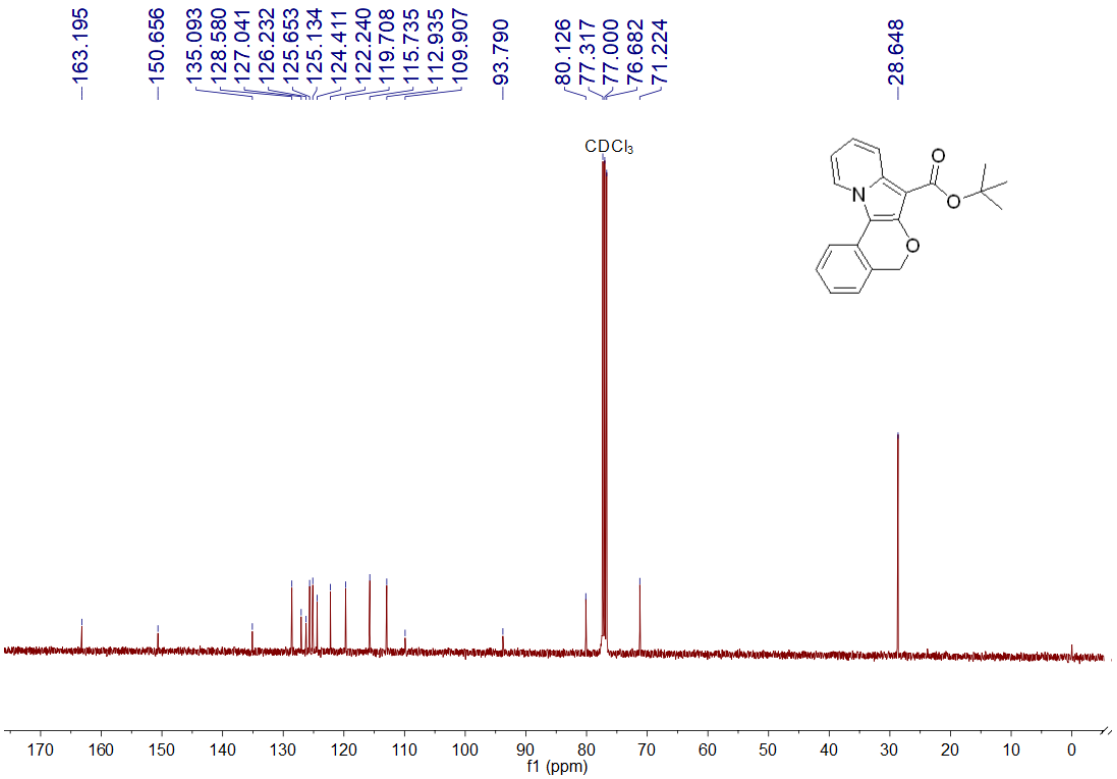
^{1}H NMR of **3s** ($CDCl_3$, 600 MHz)



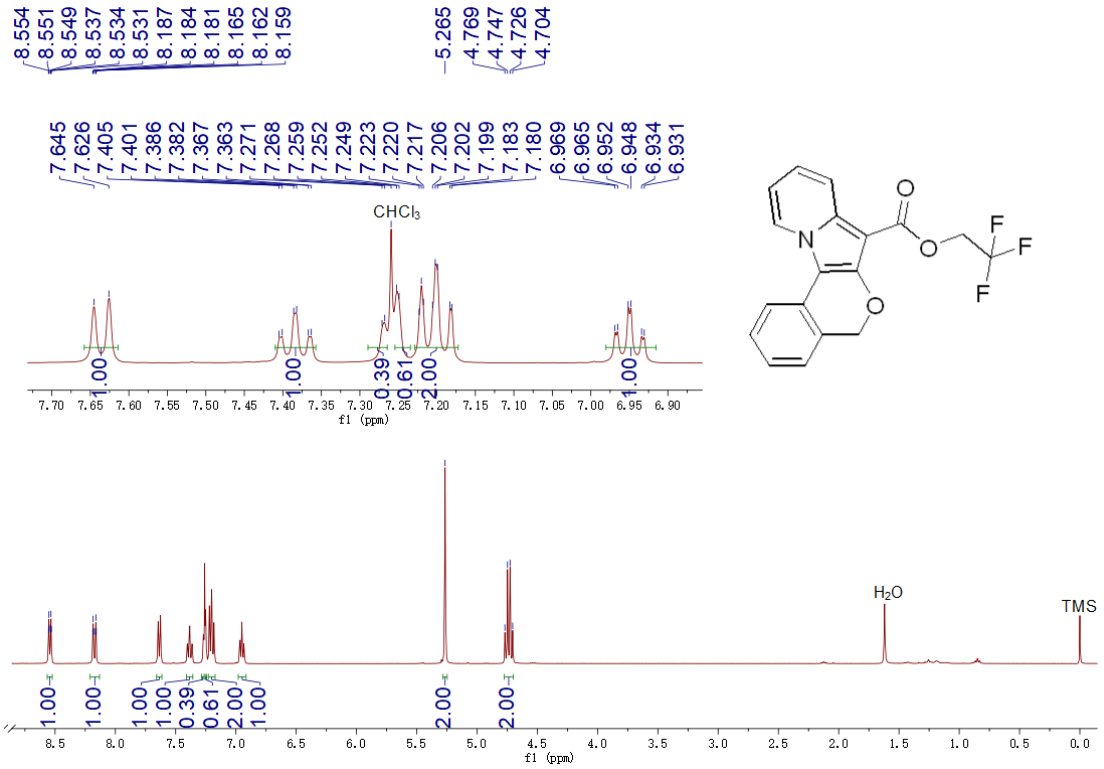
^{13}C{^{1}H} NMR of **3s** ($CDCl_3$, 100 MHz)



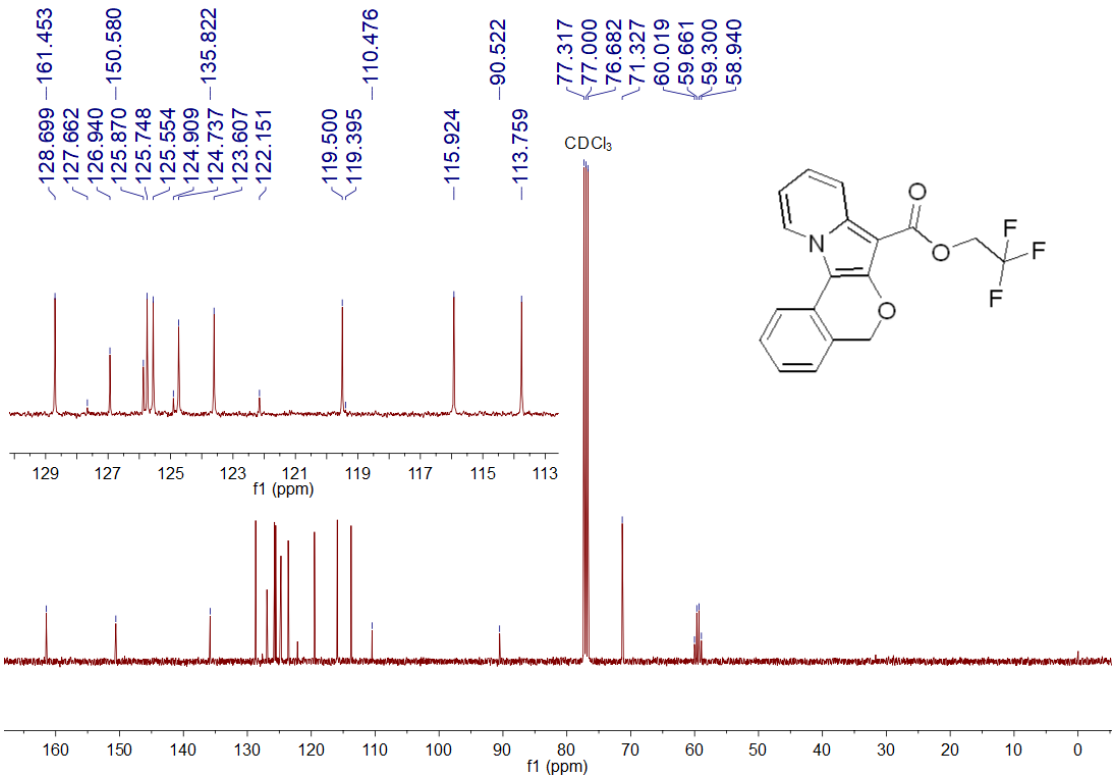
^{1}H NMR of **3t** ($CDCl_3$, 400 MHz)



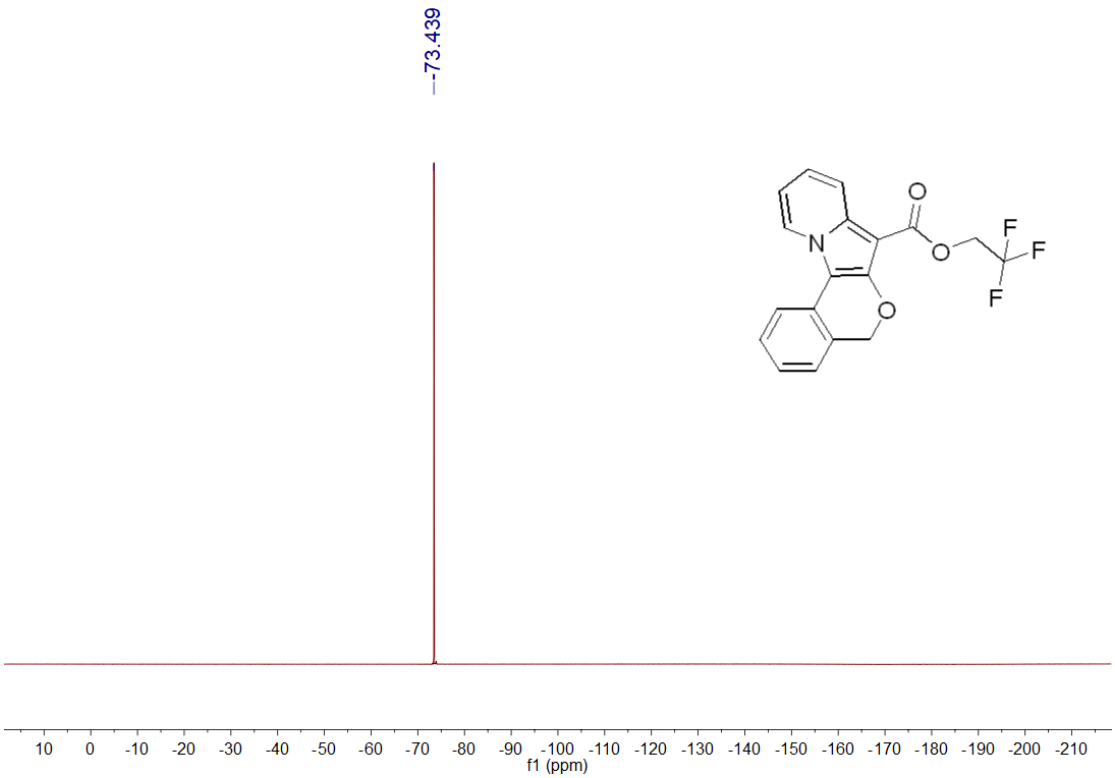
^{13}C{^{1}H} NMR of **3t** ($CDCl_3$, 100 MHz)



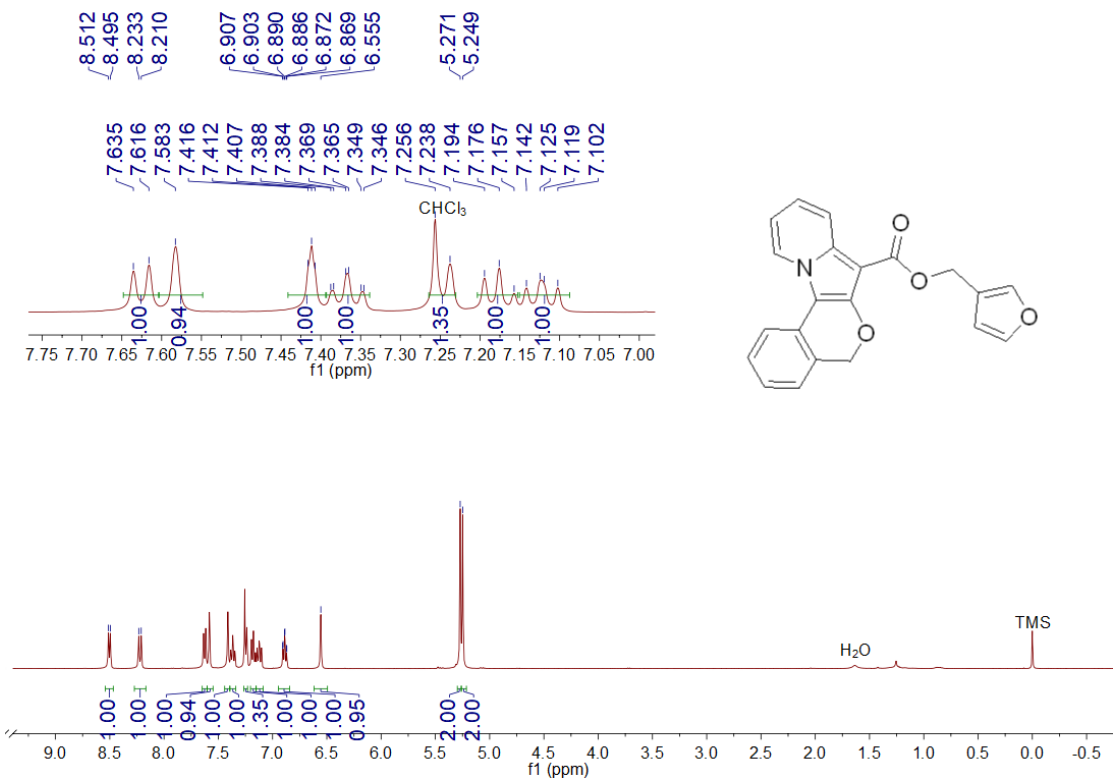
^{1}H NMR of **3u** ($CDCl_3$, 400 MHz)



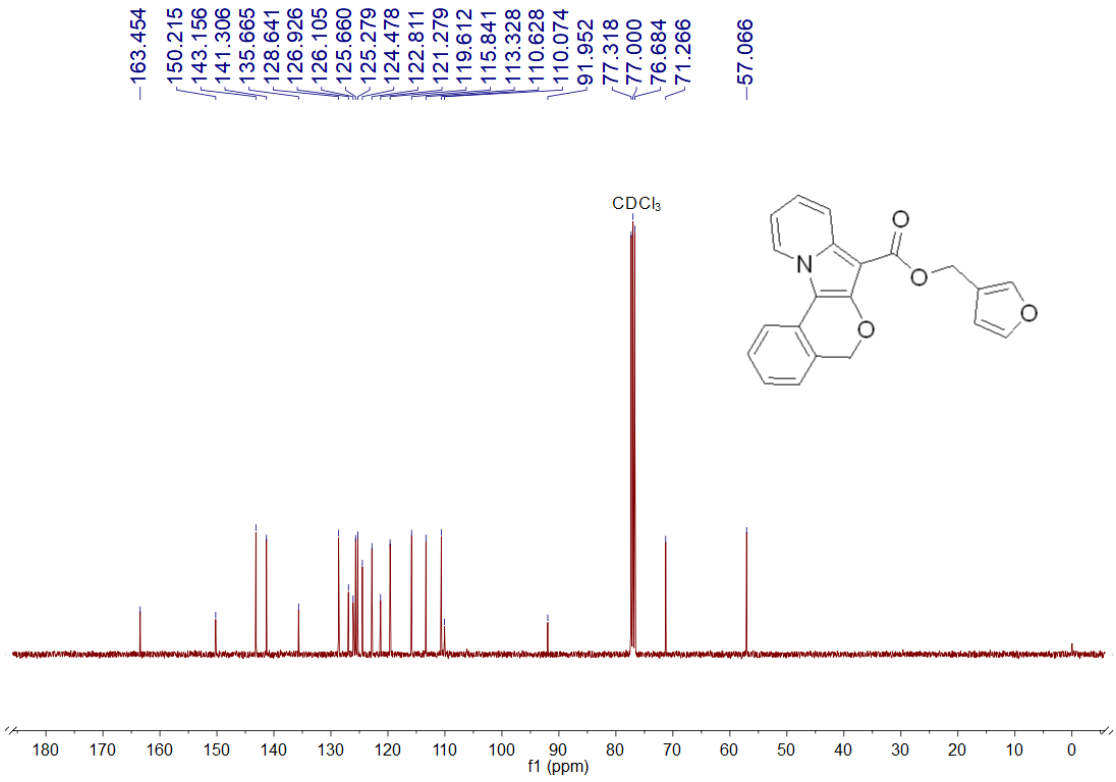
^{13}C{^{1}H} NMR of **3u** ($CDCl_3$, 100 MHz)



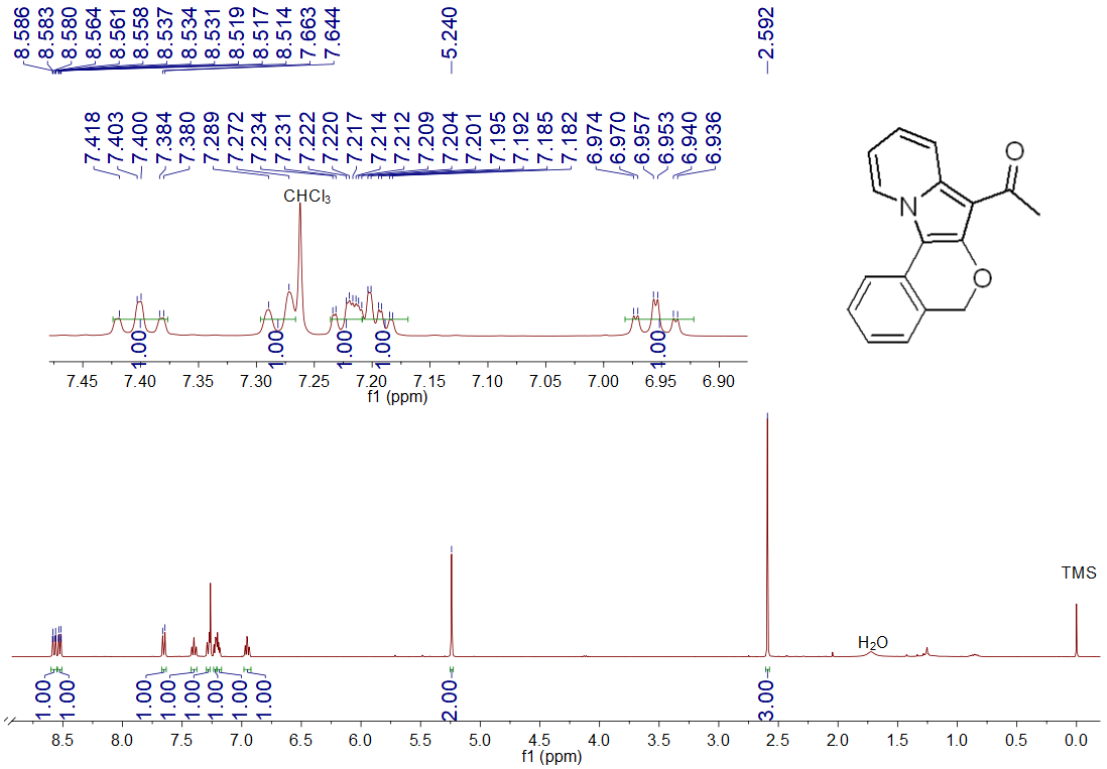
^{19}F NMR of **3u** ($CDCl_3$, 376 MHz)



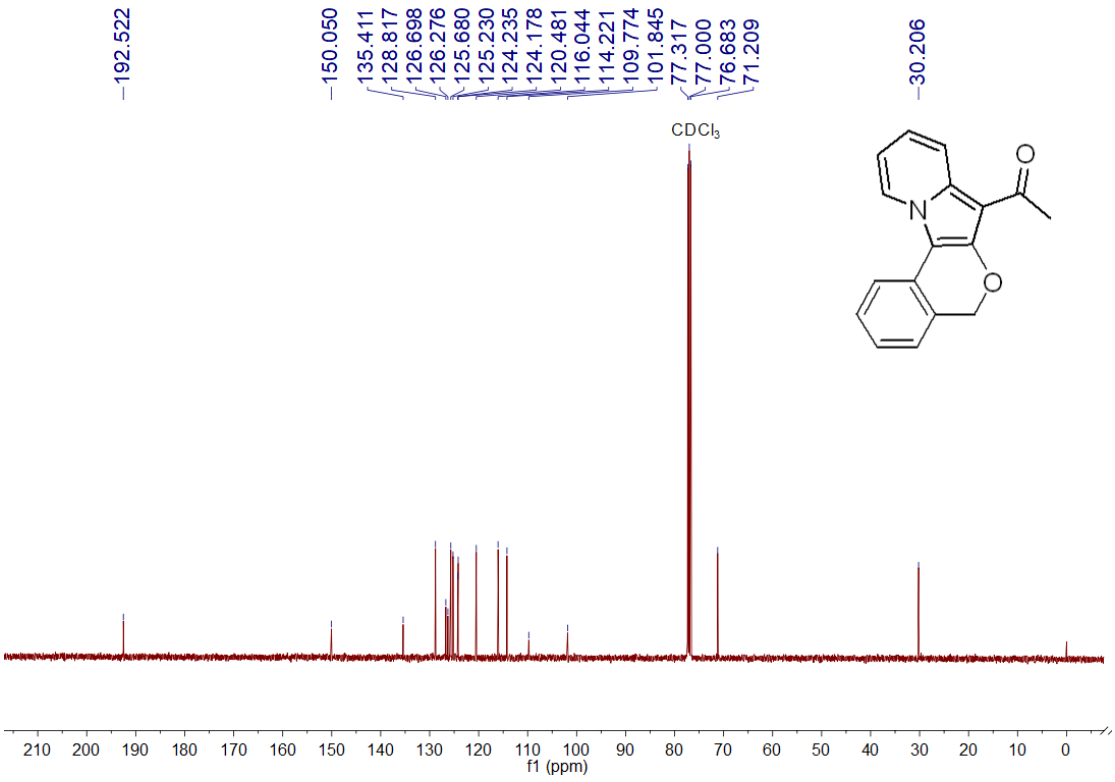
^{1}H NMR of **3v** ($CDCl_3$, 400 MHz)



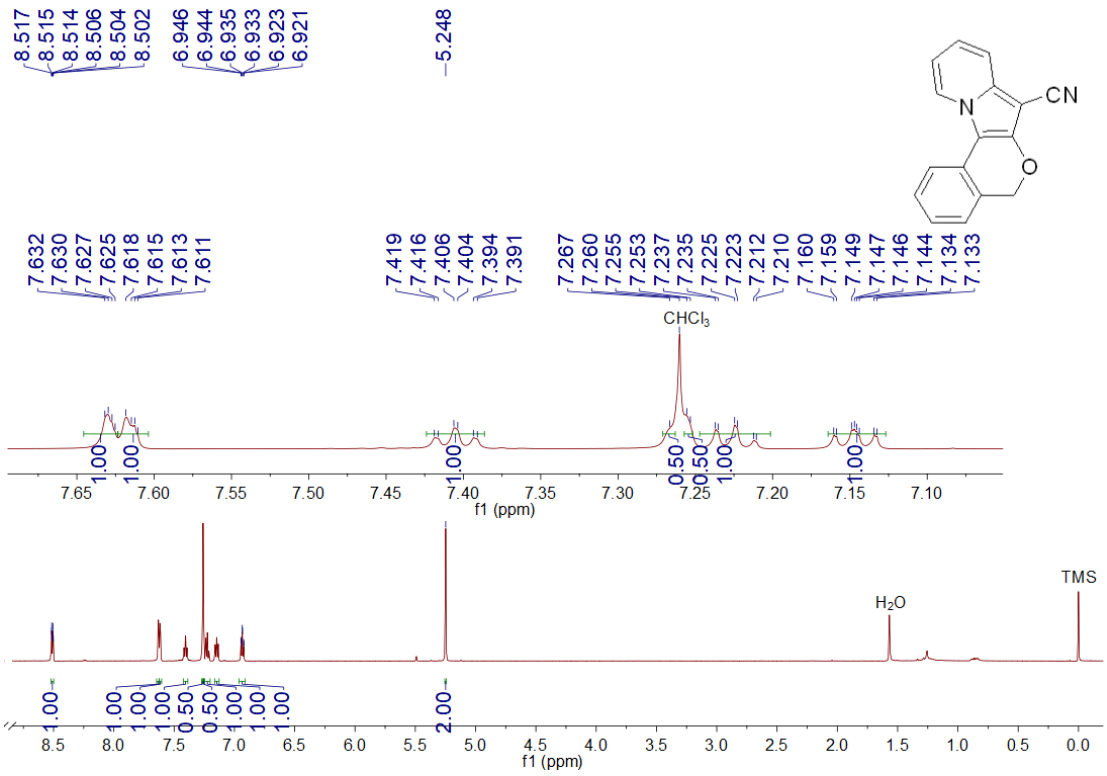
^{13}C{^{1}H} NMR of **3v** ($CDCl_3$, 100 MHz)



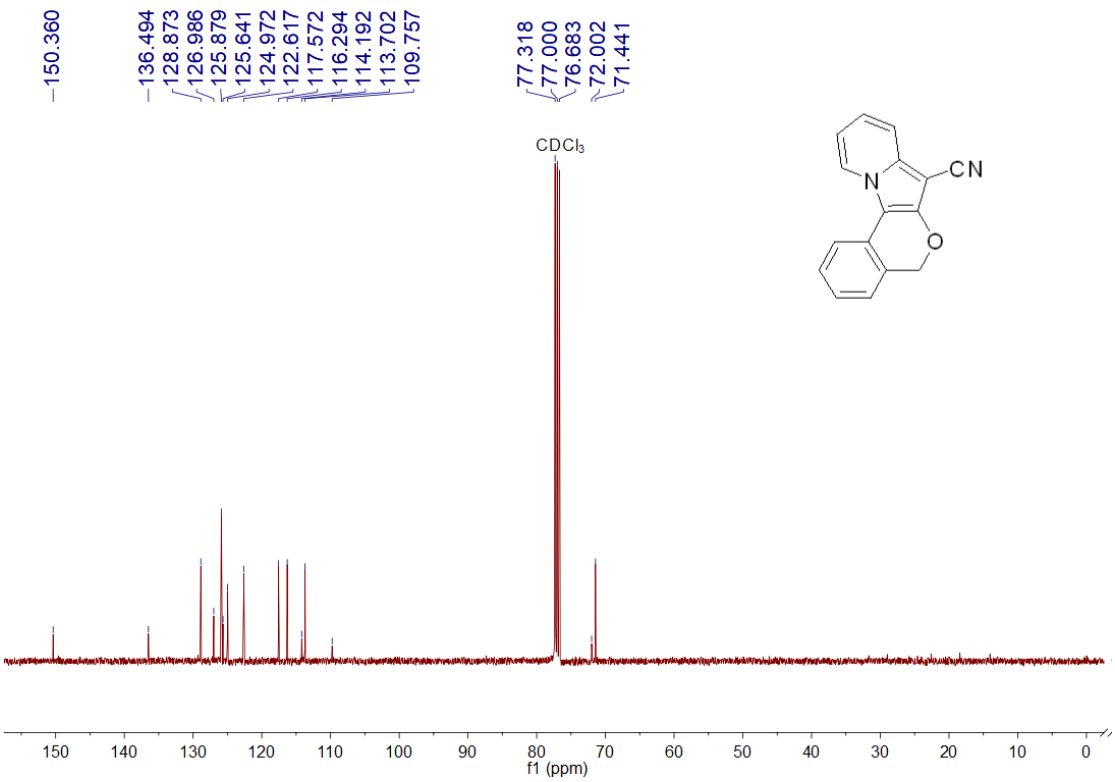
^{1}H NMR of **3w** ($CDCl_3$, 400 MHz)



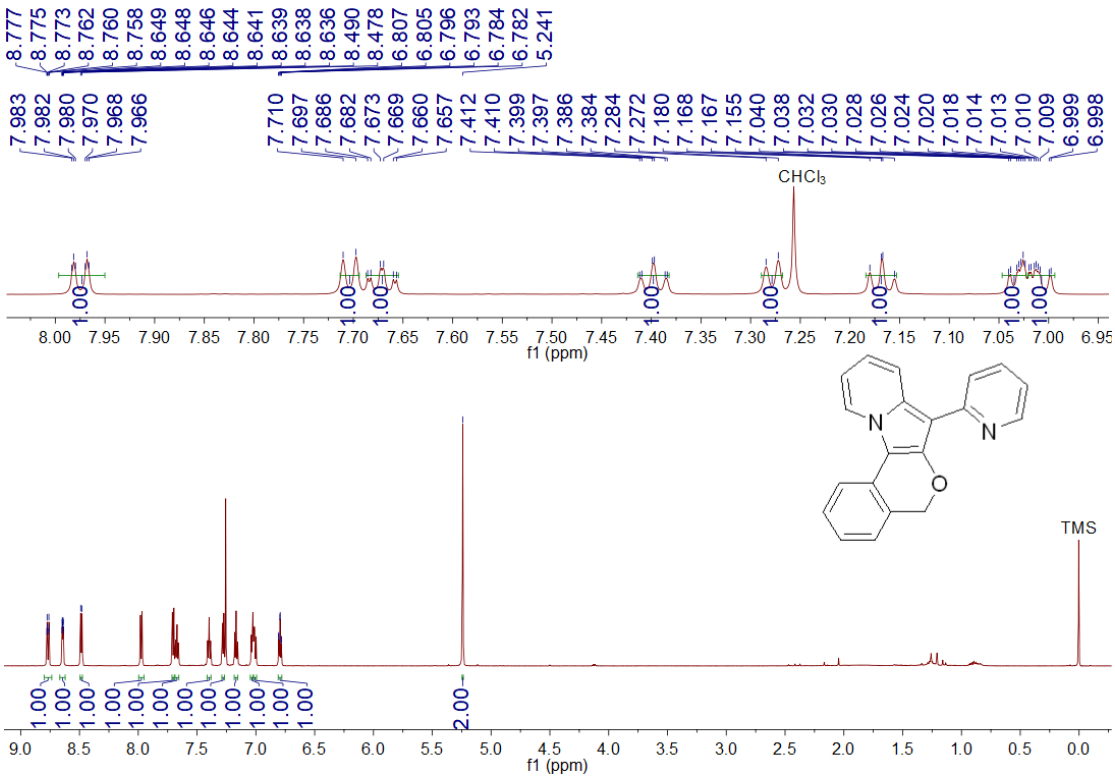
^{13}C{^{1}H} NMR of **3w** ($CDCl_3$, 100 MHz)



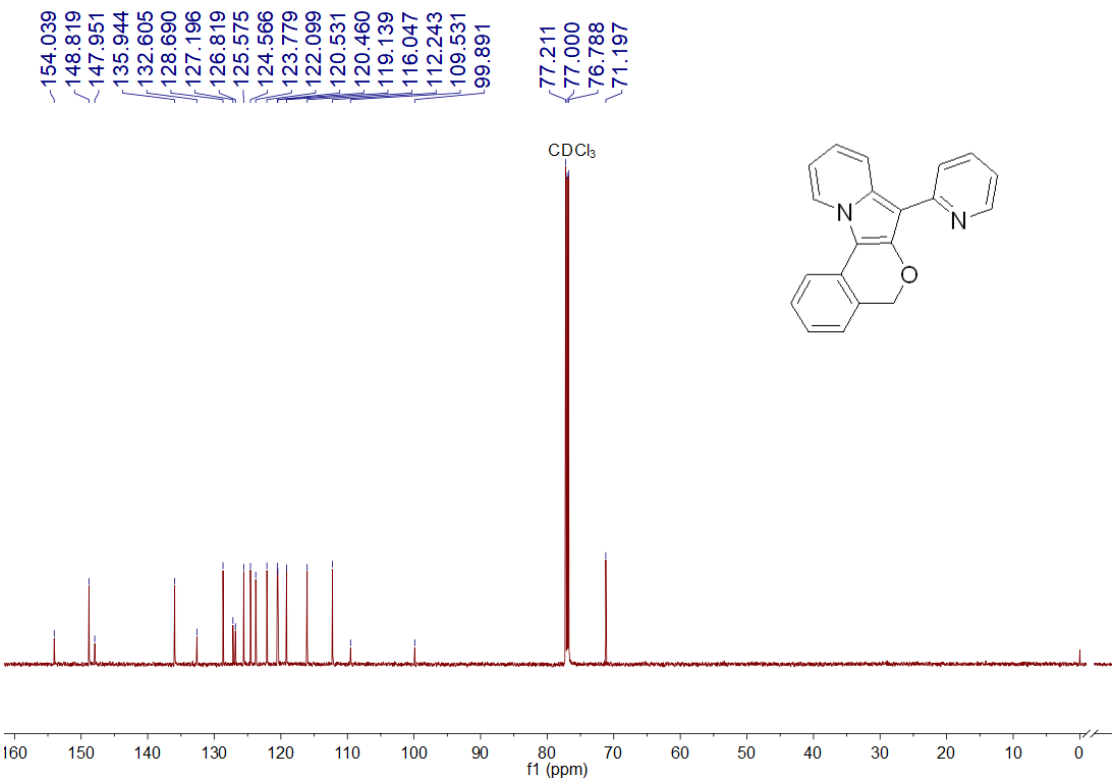
^{1}H NMR of **3x** ($CDCl_3$, 600 MHz)



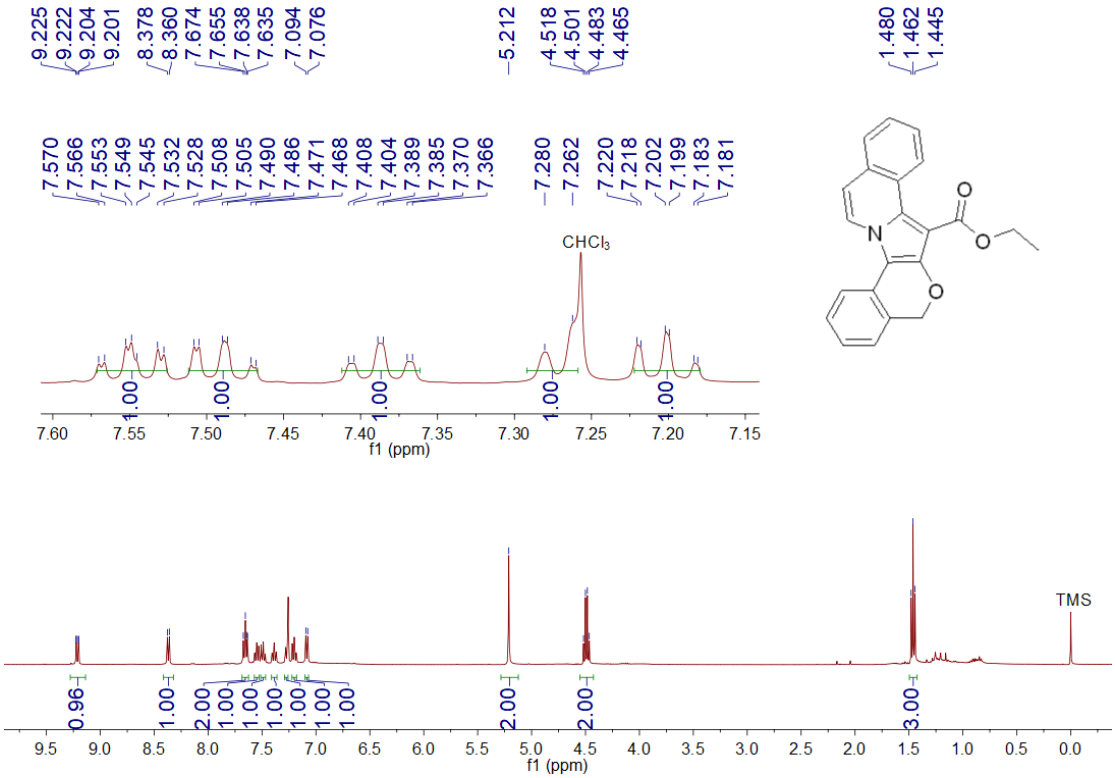
^{13}C{^{1}H} NMR of **3x** ($CDCl_3$, 100 MHz)



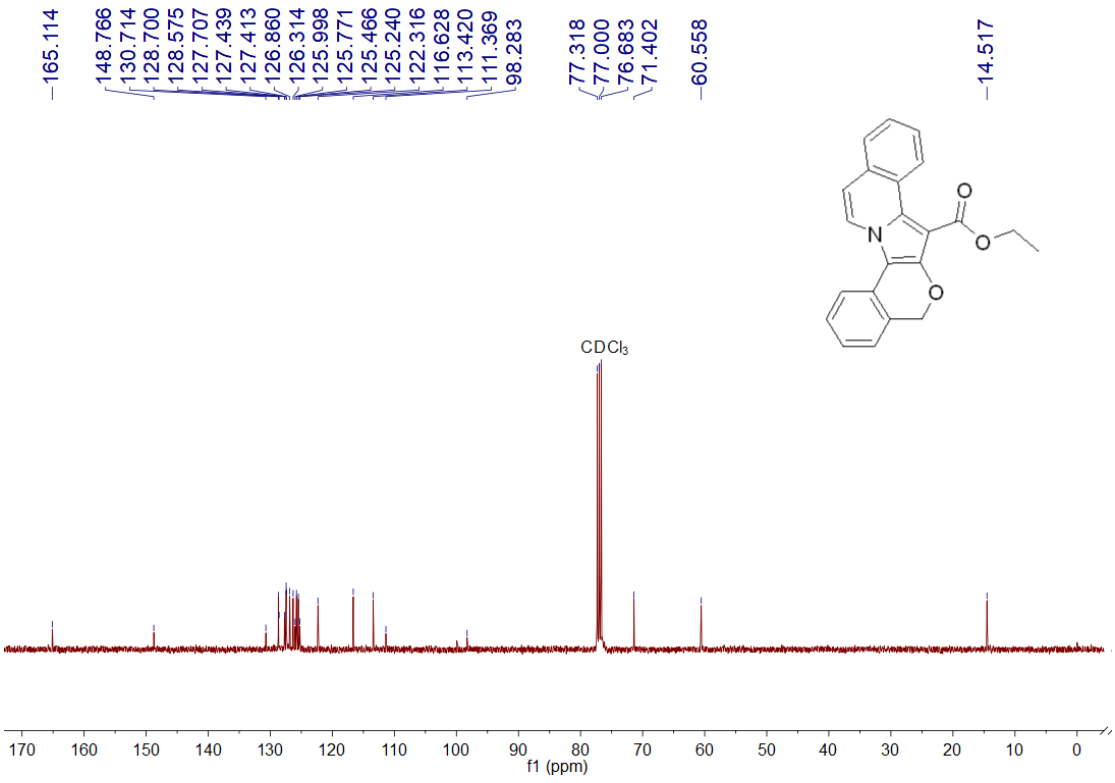
^{1}H NMR of **3y** ($CDCl_3$, 600 MHz)



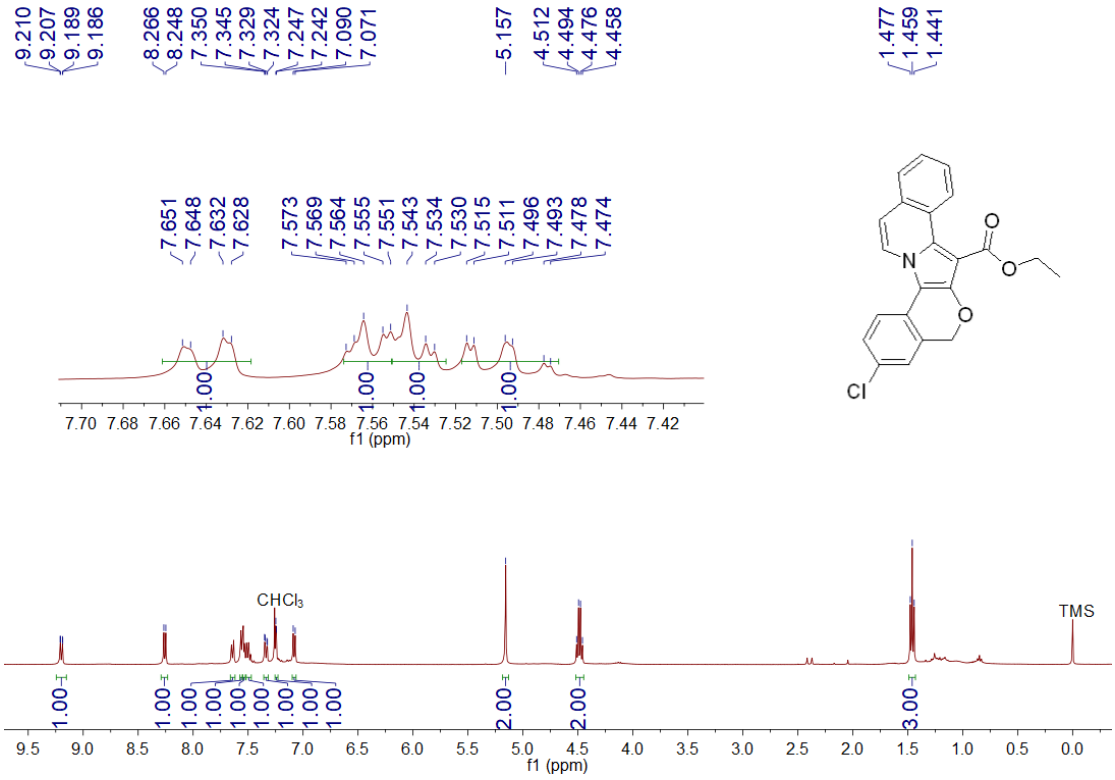
^{13}C{^{1}H} NMR of **3y** ($CDCl_3$, 150 MHz)



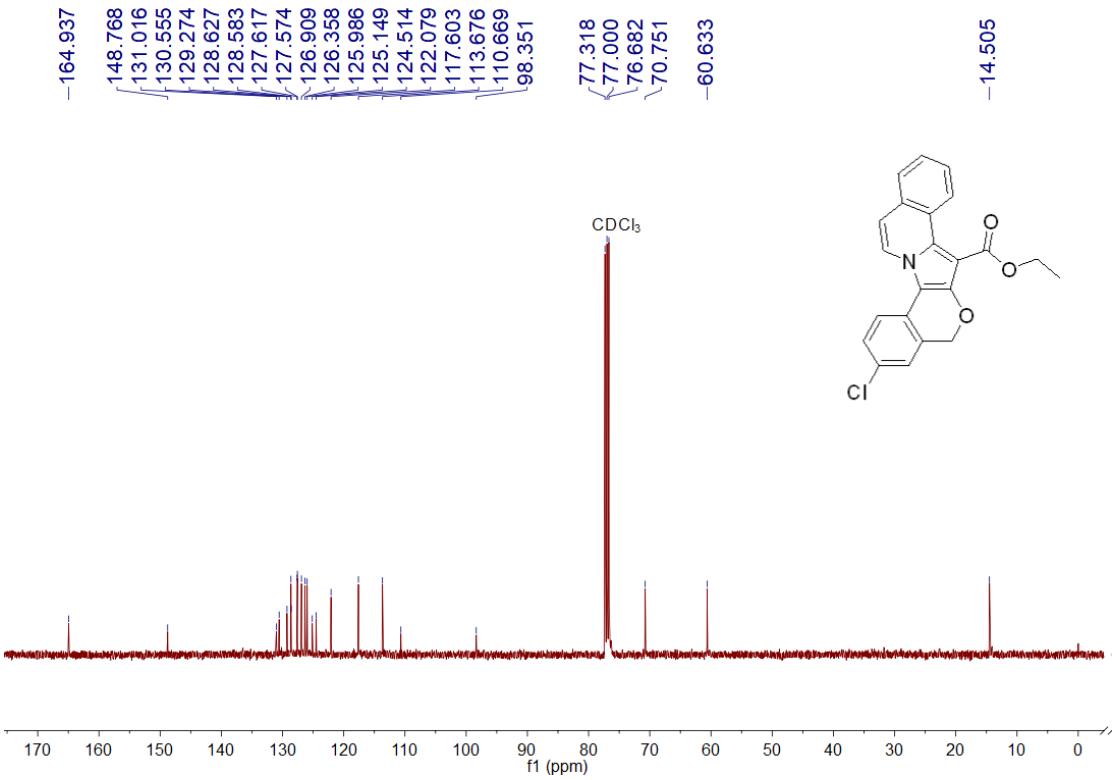
^{1}H NMR of **3z** ($CDCl_3$, 400 MHz)



^{13}C{^{1}H} NMR of **3z** ($CDCl_3$, 100 MHz)

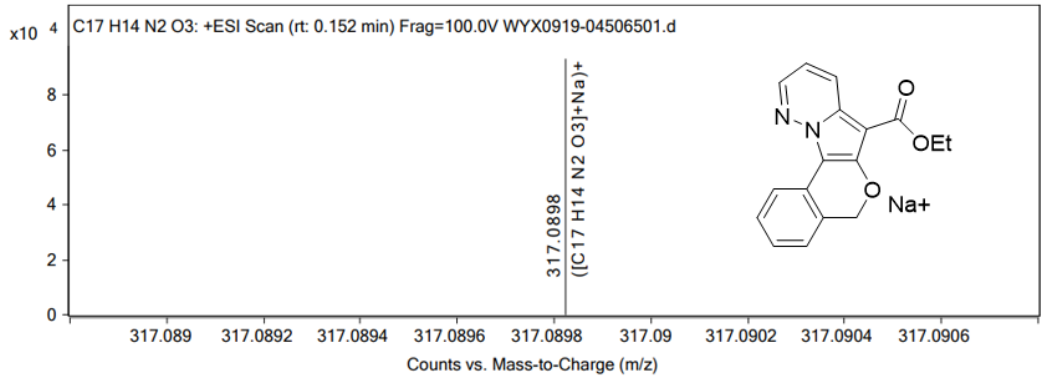


^{1}H NMR of **3A** ($CDCl_3$, 400 MHz)

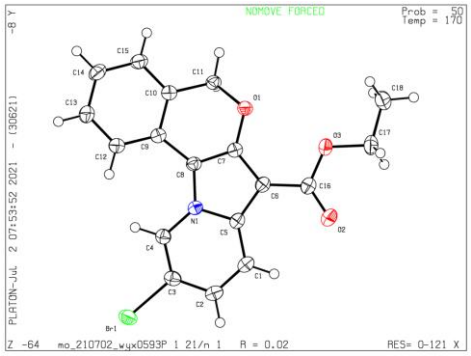


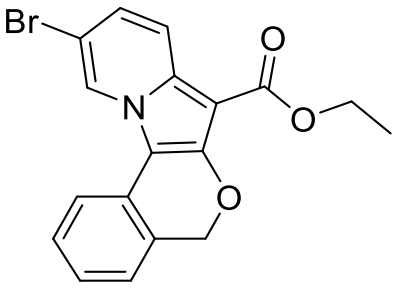
^{13}C{^{1}H} NMR of **3A** ($CDCl_3$, 100 MHz)

The High-resolution mass spectra data of 3B



The ORTEP and Crystal Parameters of 3j wherein thermal ellipsoids are drawn at 30% probability level





3j

Bond precision:	C-C = 0.0021 A		Wavelength=0.71073
Cell:	a=17.155(5)	b=4.3367(14)	c=21.762(7)
	alpha=90	beta=110.294(14)	gamma=90
Temperature:	170 K		

	Calculated	Reported
Volume	1518.5(8)	1518.5(8)
Space group	P 21/n	P 1 21/n 1
Hall group	-P 2yn	-P 2yn
Moiety formula	C18 H14 Br N O3	C18 H14 Br N O3
Sum formula	C18 H14 Br N O3	C18 H14 Br N O3
Mr	372.20	372.21
Dx,g cm-3	1.628	1.628
Z	4	4
Mu (mm-1)	2.722	2.722
F000	752.0	752.0
F000'	751.18	
h,k,lmax	21,5,27	21,5,27
Nref	3374	3362
Tmin,Tmax	0.770,0.849	0.457,0.746
Tmin'	0.319	

Correction method= # Reported T Limits: Tmin=0.457 Tmax=0.746
AbsCorr = MULTI-SCAN

Data completeness= 0.996 Theta(max)= 27.114

R(reflections)= 0.0204(3076) wR2(reflections)= 0.0545(3362)

S = 1.056 Npar= 209