$Pd(OAc)_2/S=PPh_3$ Accelerated Activation of *gem*-Dichloroalkenes for the Construction of 3-Arylchromones

Jianming Liu,[a,*] Weiwei Song,[a] Yuanyuan Yue,[a] Ren Liu,[a] Hong Yi,[b] Kelei Zhuo,[a] and Aiwen Lei[b,*]

[a]Collaborative Innovation Center of Henan Province for Green Manufacturing of Fine Chemicals, Key Laboratory of Green Chemical Media and Reactions, Ministry of Education, School of Chemistry and Chemical Engineering, Henan Normal University, Xinxiang, Henan 453007, P. R. China. Email: jmliu@htu.cn.

[b]College of Chemistry and Molecular Sciences, Wuhan University, Wuhan, 430072, P. R. China. Email: aiwenlei@whu.edu.cn.

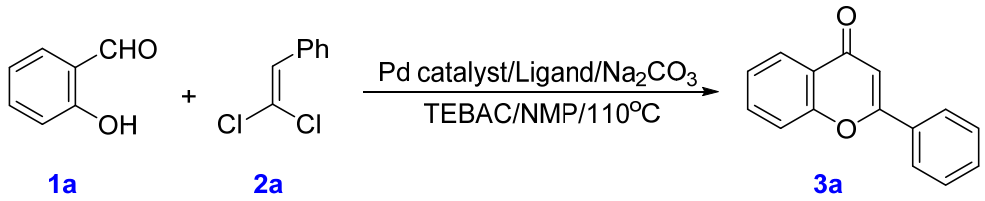
Contents

1. General information

All the chemicals and solvents were used as received without further purification. Silica gel was purchased from Qing Dao Hai Yang Chemical Industry Co. NMR spectra of the products were recorded using a Bruker Avance TM spectrometer operating at 400 MHz for ^{1}H and 101 MHz for ^{13}C in $CDCl_3$ unless otherwise noted. High resolution mass spectra (HRMS) of the products were obtained on a Bruker Daltonics micro TOF-Q spectrometer.

2. Experimental procedure for the $Pd(OAc)_2$/S=PPh_3 catalyzed the tandem reaction

Table S1 Optimization of the reaction conditions.[a]



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

Entry	Catalyst	Additive	Ligand	Base	Solvent	Yield (%)[b]
1	$Pd(OAc)_2$	TEBAC	1,10-phen	Na_2CO_3	NMP	N.R.
2	$Pd(OAc)_2$	TEBAC	Xantphos	Na_2CO_3	NMP	trace
3	$Pd(OAc)_2$	TEBAC	dppe	Na_2CO_3	NMP	50
4	$Pd(OAc)_2$	TEBAC	BINAP	Na_2CO_3	NMP	trace
5	$Pd(OAc)_2$	TEBAC	PCy_3	Na_2CO_3	NMP	N.R.
6	$Pd(OAc)_2$	TEBAC	dppp	Na_2CO_3	NMP	37
7	$Pd(OAc)_2$	TEBAC	dppf	Na_2CO_3	NMP	45
8	$Pd(OAc)_2$	TEBAC	dppb	Na_2CO_3	NMP	trace
9	$Pd(OAc)_2$	TEBAC	dppm	Na_2CO_3	NMP	45
10	$PdCl_2$	TEBAC	S=PPh_3	Na_2CO_3	NMP	67
11	$PdCl_2(CH_3CN)_2$	TEBAC	S=PPh_3	Na_2CO_3	NMP	62
12[c]	$Pd(OAc)_2$	TEBAC	S=PPh_3	Na_2CO_3	NMP	51
13[d]	$Pd(OAc)_2$	TEBAC	S=PPh_3	Na_2CO_3	NMP	65

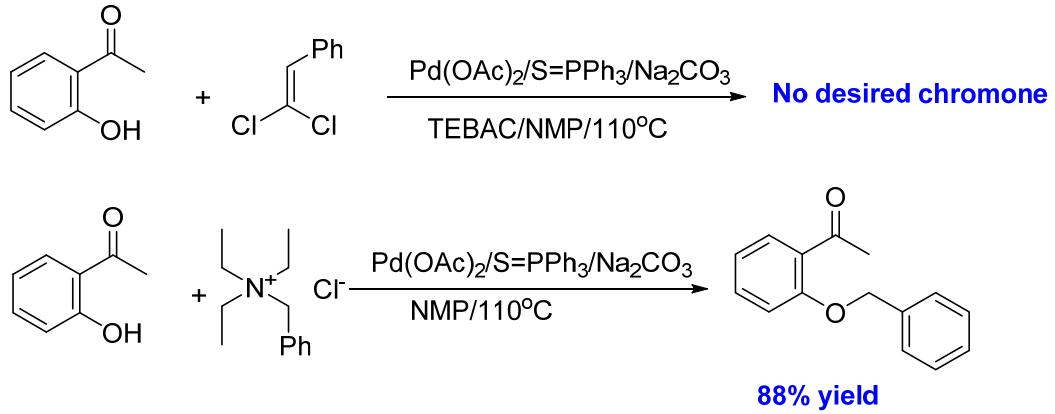
[a]Reaction conditions: **1a** (0.50 mmol), **2a** (1.0 mmol), Pd catalyst (5 mol%), ligand (10 mol%), TEBAC (1.0 mmol), Na_2CO_3 (1.5 mmol), NMP (2.0 mL), 110 °C, N_2, 24h. [b]Isolated yield.[c]**2a** (0.50 mmol), [d]**2a** (0.75 mmol).

(1) A mixture of salicylaldehyde (0.50 mmol), *gem*-dichloroalkene (1.0 mmol), $Pd(OAc)_2$ (5 mol%), S=PPh_3 (10 mol%), Na_2CO_3 (1.5 mmol), TEBAC (1.0 mmol) and NMP (2.0 mL) was

stirred at 110 ºC under N_2 atmosphere for 24 h. After cooling to room temperature, EtOAc (30 mL) was added and the aqueous phase was extracted by H_2O (3×30 mL). The organic phase was dried over Na_2SO_4, and concentrated in *vacuum*. The residue was purified by chromatography on silica gel with petroleum ether/ethyl acetate as eluent to afford the desired product.

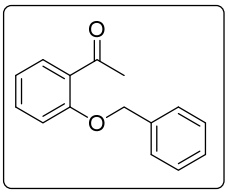
(2) A mixture of salicylaldehyde (0.50 mmol), *gem*-dichloroalkene (1.0 mmol), $Pd(OAc)_2$ (5 mol%), $S=PPh_3$ (10 mol%), Na_2CO_3 (1.5 mmol), TBAF (1.0 mmol) and NMP (2.0 mL) was stirred at 110 ºC under N_2 atmosphere for 24 h. After cooling to room temperature, EtOAc (30 mL) was added and the aqueous phase was extracted by H_2O (3×30 mL). The organic phase was dried over Na_2SO_4, and concentrated in *vacuum*. The residue was purified by chromatography on silica gel with petroleum ether/ethyl acetate as eluent to afford the desired product.

(3) A mixture of 2-hydroxyacetophenone (0.50 mmol), *gem*-dichloroalkene (1.0 mmol), $Pd(OAc)_2$ (5 mol%), $S=PPh_3$ (10 mol%), Na_2CO_3 (1.5 mmol), TEBAC (1.0 mmol) and NMP (2.0 mL) was stirred at 110 ºC under N_2 atmosphere for 24 h. After cooling to room temperature, EtOAc (30 mL) was added and the aqueous phase was extracted by H_2O (3×30 mL). The organic phase was dried over Na_2SO_4, and concentrated in *vacuum*. The residue was purified by chromatography on silica gel with petroleum ether/ethyl acetate as eluent to afford 1-(2-hydroxyphenyl)ethan-1-one in 86% yield (Scheme 1). In the process of the reaction, 2-hydroxyacetophenone coulpled with benzyltriethylammonium chloride (TEBAC).



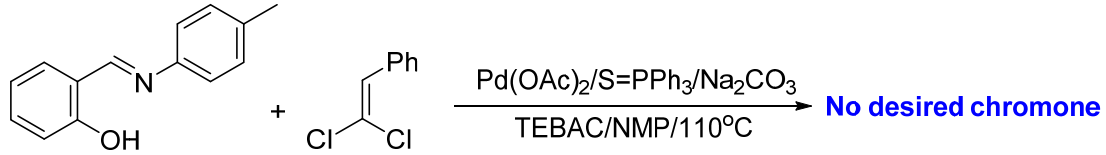
Scheme 1

1-(2-(benzyloxy)phenyl)ethan-1-one



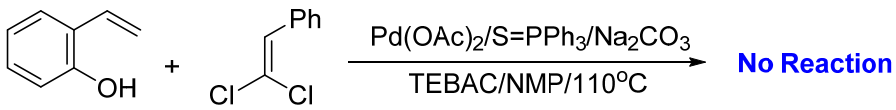
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.75 (d, *J* = 6.0 Hz, 1H), 7.45-7.41 (m, 3H), 7.39 (t, *J* = 6.0 Hz, 2H), 7.34 (t, *J* = 6.0 Hz, 1H), 7.02-6.99 (m, 2H), 5.15 (s, 2H), 2.59 (s, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 199.9, 158.1, 136.2, 133.6, 130.5, 128.8, 128.7, 128.3, 127.6, 120.9, 112.9, 70.7, 32.1. ESI-MS (M): 226.

(4) A mixture of 2-((p-tolylimino)methyl)phenol (0.50 mmol), *gem*-dichloroalkene (1.0 mmol), $Pd(OAc)_2$ (5 mol%), $S=PPh_3$ (10 mol%), Na_2CO_3 (1.5 mmol), TEBAC (1.0 mmol) and NMP (2.0 mL) was stirred at 110 ºC under N_2 atmosphere for 24 h. After cooling to room temperature, EtOAc (30 mL) was added and the aqueous phase was extracted by H_2O (3×30 mL). The organic phase was dried over Na_2SO_4, and concentrated in *vacuum*. The residue was detected by GC-MS, chromone was not detected (Scheme 2). 2-vinylphenol was destroyed and dichloroalkenes was converted into (2-chlorovinyl)benzene and 1, 4-diphenylbuta-1, 3-diyne.



Scheme 2

(5) A mixture of 2-vinylphenol (0.50 mmol), *gem*-dichloroalkene (1.0 mmol), $Pd(OAc)_2$ (5 mol%), $S=PPh_3$ (10 mol%), Na_2CO_3 (1.5 mmol), TEBAC (1.0 mmol) and NMP (2.0 mL) was stirred at 110 ºC under N_2 atmosphere for 24 h. After cooling to room temperature, EtOAc (30 mL) was added and the aqueous phase was extracted by H_2O (3×30 mL). The organic phase was dried over Na_2SO_4, and concentrated in *vacuum*. The residue was detected by GC-MS, no desired product was observed (Scheme 3). Regretfully, 2-(benzyloxy)benzaldehyde and N-benzyl-4-methylaniline were observed by GC-MS.

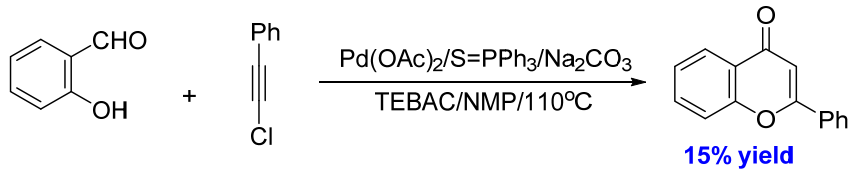


Scheme 3

3. Experiments on investigation of mechanism

3.1 The coupling between (chloroethynyl)benzene and salicylaldehyde

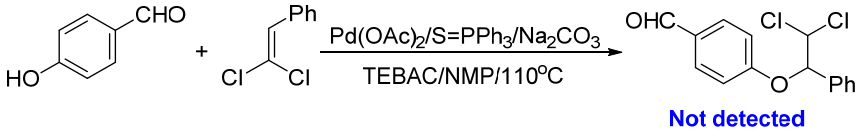
A mixture of salicylaldehyde (0.50 mmol), (chloroethynyl)benzene (1.0 mmol), $Pd(OAc)_2$ (5 mol%), $S=PPh_3$ (10 mol%), Na_2CO_3 (1.5 mmol), TEBAC (1.0 mmol) and NMP (2.0 mL) was stirred at 110 ºC under N_2 atmosphere for 24 h. After cooling to room temperature, EtOAc (30 mL) was added and the aqueous phase was extracted by H_2O (3×30 mL). The organic phase was dried over Na_2SO_4, and concentrated in *vacuum*. The residue was purified by chromatography on silica gel with petroleum ether/ethyl acetate as eluent to afford the desired product in 15 % yield (Scheme 4).



Scheme 4

3.2 The coupling between *p*-hydroxybenzaldehyde and *gem*-dichloroalkene

A mixture of *p*-hydroxybenzaldehyde (0.50 mmol), *gem*-dichloroalkene (1.0 mmol), $Pd(OAc)_2$ (5 mol%), $S=PPh_3$ (10 mol%), Na_2CO_3 (1.50 mmol), TEBAC (1.0 mmol) and NMP (2.0 mL) was stirred at 110 ºC under N_2 atmosphere for 24 h. After cooling to room temperature, EtOAc (30 mL) was added and the aqueous phase was extracted by H_2O (3×20 mL). The organic phase was dried over Na_2SO_4, and concentrated in vacuum. The residue was detected by GC-MS (Scheme 5).

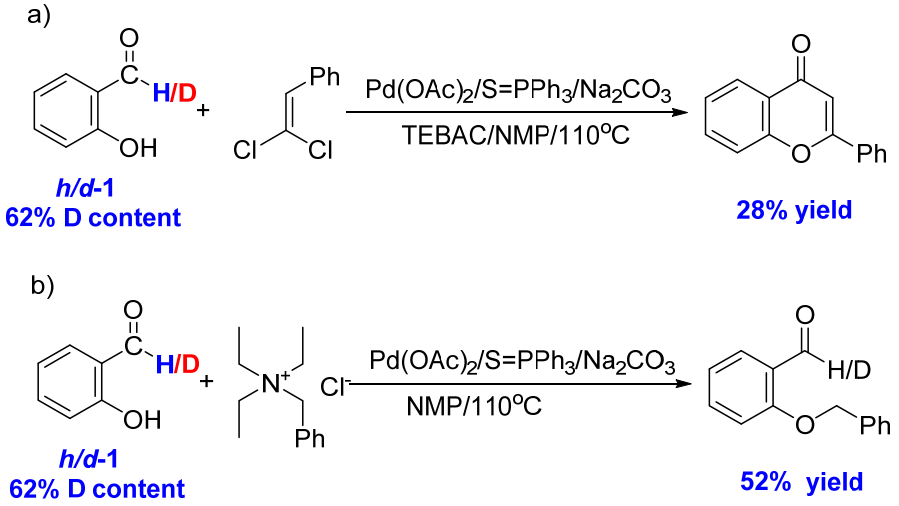


Scheme 5

3.3 The experiment between *gem*-dichloroalkene and salicylaldehyde contained 62% D content

A mixture of 62% D content of salicylic aldehyde (0.50 mmol),[1] *gem*-dichloroalkene (1.0 mmol), $Pd(OAc)_2$ (5 mol%), $S=PPh_3$ (10 mol%), Na_2CO_3 (1.5 mmol), TEBAC (1.0 mmol) and NMP (2.0 mL) was stirred at 110 ºC under N_2 atmosphere for 24 h. After cooling to room

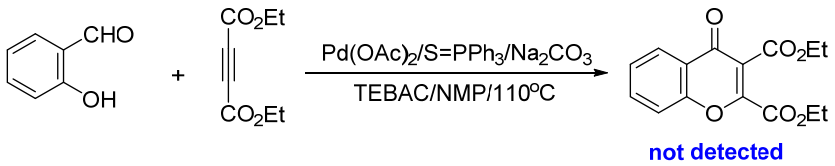
temperature, EtOAc (30 mL) was added and the aqueous phase was extracted by H_2O (3×30 mL). The organic phase was dried over Na_2SO_4, and concentrated in *vacuum*. The residue was purified by chromatography on silica gel with petroleum ether/ethyl acetate as eluent to afford the desired product in 28% yield. In the control experiment, 2-(benzyloxy)benzaldehyde was obtained in 52% yield (Scheme 6).



Scheme 6

3.4 The coupling between diethyl but-2-ynedioate and salicylaldehyde

A mixture of salicylaldehyde (0.50 mmol), diethyl but-2-ynedioate (1.0 mmol), $Pd(OAc)_2$ (5 mol%), $S=PPh_3$ (10 mol%), Na_2CO_3 (1.50 mmol), TEBAC (1.0 mmol) and NMP (2.0 mL) was stirred at 110 ºC under N_2 atmosphere for 24 h. After cooling to room temperature, EtOAc (30 mL) was added and the aqueous phase was extracted by H_2O (3×30 mL). The organic phase was dried over Na_2SO_4, and concentrated in *vacuum*. The residue was purified by chromatography on silica gel with petroleum ether/ethyl acetate as eluent to afford no desired product (Scheme 7).

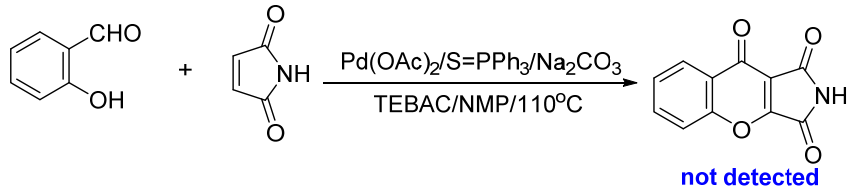


Scheme 7

3.5 The coupling between 1*H*-pyrrole-2, 5-dione and salicylaldehyde

A mixture of salicylaldehyde (0.50 mmol), 1*H*-pyrrole-2, 5-dione (1.0 mmol), $Pd(OAc)_2$ (5 mol%), $S=PPh_3$ (10 mol%), Na_2CO_3 (1.50 mmol), TEBAC (1.0 mmol) and NMP (2.0 mL) was

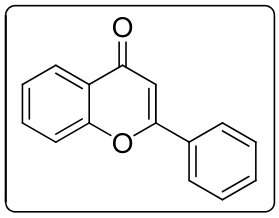
stirred at 110 ºC under N_2 atmosphere for 24 h. After cooling to room temperature, EtOAc (30 mL) was added and the aqueous phase was extracted by H_2O (3×30 mL). The organic phase was dried over Na_2SO_4, and concentrated in *vacuum*. The residue was purified by chromatography on silica gel with petroleum ether/ethyl acetate as eluent to afford no desired product (Scheme 8).



Scheme 8

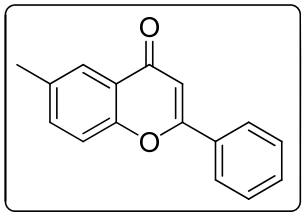
4. Characterization of Products

2-phenyl-4*H*-chromen-4-one 3a[2]



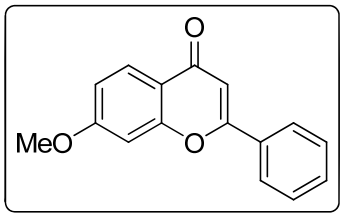
(m.p. 97-98°C) ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.24 (d, *J* = 8.0 Hz, 1H), 7.95-7.93 (m, 2H), 7.73-7.69 (m, 1H), 7.59-7.53 (m, 4H), 7.43 (t, *J* = 8.0 Hz, 1H), 6.84 (s, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 178.5, 163.4, 156.3, 133.8, 131.8, 131.6, 129.1, 126.3, 125.7, 125.3, 124.0, 118.1, 107.6. IR (neat, cm^{-1}): 3059, 2921, 2850, 1646, 1606, 1569, 1495, 1465, 1449, 1376, 1310, 1283, 1259, 1225, 1129. ESI-MS (M): 222.

6-methyl-2-phenyl-4H-chromen-4-one 3b[2]



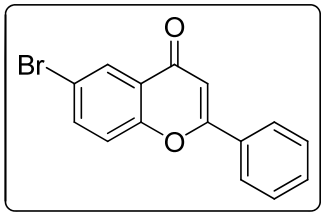
(m.p. 112 °C) ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.03 (s, 1H), 7.95-7.93 (m, 2H), 7.56-7.48 (m, 5H), 6.83 (s, 1H), 2.48 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 178.6, 163.3, 154.5, 135.2, 135.0, 131.9, 131.5, 129.0, 126.3, 125.1, 123.6, 117.9, 107.4, 21.0. IR (neat, cm^{-1}): 3064, 2920, 1645, 1615, 1569, 1494, 1483, 1450, 1431, 1361, 1302, 1255, 1223, 1139. ESI-MS (M): 236.

7-methoxy-2-phenyl-4*H*-chromen-4-one 3c



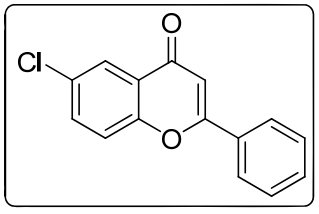
(m.p. 97 °C) ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.15 (d, *J* = 8.0 Hz, 1H), 7.93-7.91 (m, 2H), 7.54-7.52 (m, 3H), 7.02-6.98 (m, 2H), 6.78 (s, 1H), 3.94 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 177.9, 164.2, 163.0, 158.0, 131.9, 131.4, 129.0, 127.0, 126.2, 117.8, 114.4, 107.5, 100.4, 55.9. IR (neat, cm^{-1}): 3026, 3002, 2924, 2845, 1653, 1626, 1606, 1494, 1450, 1439, 1348, 1357, 1284, 1247, 1190, 1165, 1131. HRMS, calculated for $C_{16}H_{13}O_3$ ($M+H^+$): 253.0860, found: 253.0864.

6-bromo-2-phenyl-4*H*-chromen-4-one 3d



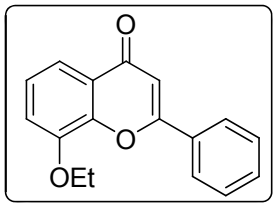
(m.p. 189 °C) ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.35 (d, *J* = 4.0 Hz, 1H), 7.92-7.90 (m, 2H), 7.78 (d, *J* = 8.0 Hz, 1H), 7.56-7.53 (m, 3H), 7.47 (d, *J* = 8.0 Hz, 1H), 6.83 (s, 1H).^{13}C NMR (101 MHz, $CDCl_3$) δ 177.1, 163.7, 155.0, 136.8, 131.9, 131.4, 129.2, 128.4, 126.4, 125.3, 120.1, 118.7, 107.6. IR (neat, cm^{-1}): 3083, 2920, 1648, 1614, 1597, 1563, 1494, 1456, 1434, 1350, 1304, 1271, 1253, 1210, 1133. HRMS, calculated for $C_{15}H_{10}BrO_2$ ($M+H^+$): 300.9859, found: 300.9858.

6-chloro-2-phenyl-4*H*-chromen-4-one 3e



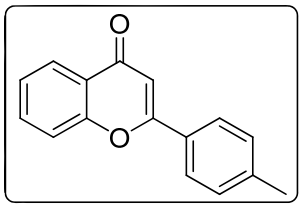
(m.p. 183°C) ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.19 (d, *J* = 2.6 Hz, 1H), 7.91 (dd, *J* = 8.0, 4.0 Hz, 2H), 7.64 (dd, *J* = 8.0, 4.0 Hz, 1H), 7.57-7.53 (m, 4H), 6.83 (s, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 177.2, 163.7, 154.6, 134.0, 131.9, 131.4, 131.2, 129.1, 126.3, 125.2, 124.9, 119.8, 107.5. IR (neat, cm^{-1}): 3085, 1648, 1615, 1601, 1566, 1494, 1456, 1436, 1353, 1306, 1291, 1272, 1253, 1132. HRMS, calculated for $C_{15}H_9ClNaO_2$ ($M+Na^+$): 279.0183, found: 279.0183.

8-ethoxy-2-phenyl-4*H*-chromen-4-one 3f



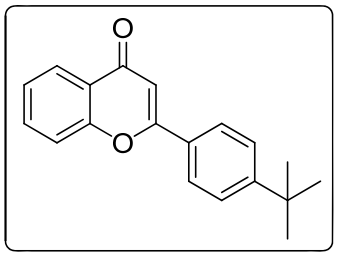
(m.p. 105 °C) ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.00-7.97 (m, 2H), 7.78 (dd, *J* = 8.0, 4.0 Hz, 1H), 7.55-7.53 (m, 3H), 7.32 (t, *J* = 8.0 Hz, 1H), 7.19 (d, *J* = 8.0 Hz, 1H), 6.86 (s, 1H), 4.24 (q, *J* = 8.0 Hz, 2H), 1.58 (t, *J* = 8.0 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 178.6, 162.9, 148.5, 146.9, 131.9, 131.5, 129.0, 126.3, 125.0, 124.8, 116.4, 115.7, 107.3, 65.0, 14.8. IR (neat, cm^{-1}): 3068, 2975, 2934, 2896, 1576, 1496, 1473, 1451, 1379, 1325, 1310, 1279, 1221, 1196, 1178, 1150. HRMS, calculated for $C_{17}H_{14}NaO_3$ ($M+Na^+$): 289.0835, found: 289.0835.

2-p-tolyl-4*H*-chromen-4-one 3g[1]



(m.p. 111 °C) ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.24 (d, *J* = 8.0 Hz, 1H), 7.84 (d, *J* = 8.0 Hz, 2H), 7.71 (t, *J* = 8.0 Hz, 1H), 7.58 (d, *J* = 8.0 Hz, 1H), 7.43 (t, *J* = 8.0 Hz, 1H), 7.34 (d, *J* = 8.0 Hz, 2H), 6.81 (d, *J* = 4.0 Hz, 1H), 2.45 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 178.5, 163.6, 156.3, 142.3, 133.7, 129.8, 129.0, 126.2, 125.7, 125.1, 124.0, 118.1, 107.0, 21.6. IR (neat, cm^{-1}): 3035, 2918, 1638, 1568, 1510, 1466, 1414, 1372, 1313, 1281, 1255, 1228. ESI-MS (M): 236.

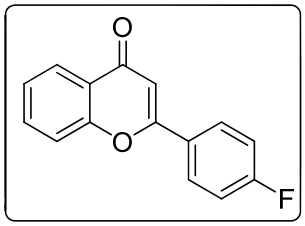
2-(4-tert-butylphenyl)-4*H*-chromen-4-one 3h



(m.p. 80 °C) ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.24 (d, *J* = 8.0 Hz, 1H), 7.88 (dd, *J* = 8.0, 4.0 Hz, 2H), 7.73-7.69 (m, 1H), 7.57 (t, *J* = 8.0 Hz, 3H), 7.43 (t, *J* = 8.0 Hz, 1H), 6.83 (d, *J* = 1.4 Hz, 1H),

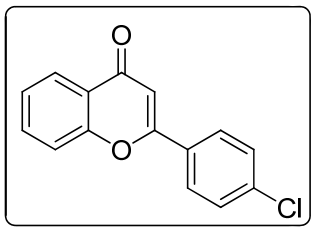
1.38 (s, 9H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 178.5, 163.6, 156.3, 155.3, 133.7, 128.9, 126.2, 126.1, 125.7, 125.1, 124.0, 118.1, 107.1, 35.0, 31.1. IR (neat, cm^{-1}): 3065, 2960, 2902, 2867, 1645, 1573, 1514, 1466, 1415, 1374, 1336, 1330, 1306, 1283, 1269, 1239, 1227, 1202. HRMS, calculated for $C_{19}H_{18}NaO_2$ ($M+Na^+$): 301.1199, found: 301.1196.

2-(4-fluorophenyl)-4*H*-chromen-4-one 3i



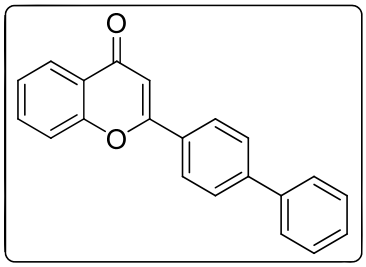
(m.p. 140 °C) ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.25 (d, *J* = 8.0 Hz, 1H), 7.97-7.94 (m, 2H), 7.73 (d, *J* = 8.0 Hz, 1H), 7.58 (d, *J* = 8.0 Hz, 1H), 7.47-7.43 (m, 1H), 7.34 (t, *J* = 8.0 Hz, 2H), 6.79 (s, 1H). ^{13}C NMR (101 MHz, CDCl3) δ 178.2, 166.0, 162.9 (d, J_{CF} = 120 Hz), 156.1, 133.8, 128.5 (d, J_{CF} = 10 Hz), 128.0, 125.5 (d, J_{CF} = 40 Hz), 123.8, 118.0, 116.3 (d, J_{CF} = 20 Hz), 107.3. ^{19}F NMR (376 MHz, $CDCl_3$) δ -107.42 ppm. IR (neat, cm^{-1}): 3077, 1653, 1574, 1508, 1466, 1417, 1378, 1332, 1302, 1284, 1233, 1163, 1133. HRMS, calculated for $C_{15}H_{10}FO_2$ ($M+H^+$): 241.0660, found: 241.0663.

2-(4-chlorophenyl)-4*H*-chromen-4-one 3j



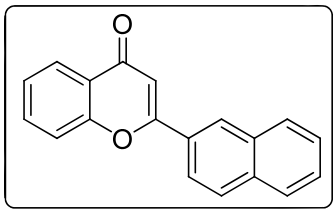
(m.p. 187 °C) ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.21 (d, *J* = 8.0 Hz, 1H), 7.84 (d, *J* = 8.0 Hz, 2H), 7.72-7.68 (m, 1H), 7.55 (d, *J* = 8.0 Hz, 1H), 7.50-7.47 (m, 2H), 7.42 (t, *J* = 8.0 Hz, 1H), 6.78 (s, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 178.2, 162.2, 156.1, 137.9, 133.9, 130.2, 129.4, 127.5, 125.7, 125.4, 123.9, 118.0, 107.7. IR (neat, cm^{-1}): 3090, 1668, 1622, 1607, 1593, 1575, 1480, 1467, 1408, 1375, 1332, 1278, 1220, 1132. HRMS, calculated for $C_{15}H_9ClNaO_2$ ($M+Na^+$): 279.0183, found: 279.0183.

2-(biphenyl-4-yl)-4*H*-chromen-4-one 3k



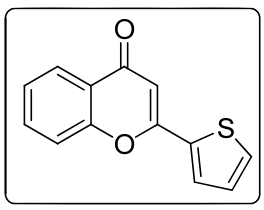
(m.p. 158 °C) ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.25 (d, *J* = 8.0 Hz, 1H), 8.02 (d, *J* = 8.0 Hz, 2H), 7.78-7.70 (m, 3H), 7.66 (d, *J* = 8.0 Hz, 2H), 7.61 (d, *J* = 8.0 Hz, 1H), 7.52-7.42 (m, 4H), 6.89 (s, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 178.4, 163.1, 156.3, 144.4, 139.7, 133.8, 130.5, 129.0, 128.2, 127.6, 127.1, 126.7, 125.7, 125.2, 124.0, 118.1, 107.4. IR (neat, cm^{-1}): 3062, 2361, 1637, 1571, 1519, 1487, 1475, 1464, 1411, 1376, 1335, 1316, 1284, 1261, 1220, 1129. HRMS, calculated for $C_{21}H_{15}O_2$ ($M+H^+$): 299.1067, found: 299.1071.

2-(naphthalen-2-yl)-4*H*-chromen-4-one 3l



(m.p. 160 °C) ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.49 (s, 1H), 8.26 (d, *J* = 8.0 Hz, 1H), 7.98-7.91 (m, 4H), 7.74-7.71 (m, 1H), 7.66-7.58 (m, 3H), 7.45 (t, *J* = 8.0 Hz, 1H), 6.97 (s, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 178.4, 163.2, 156.3, 134.6, 133.8, 132.9, 129.0, 128.9, 128.0, 127.8, 127.1, 126.9, 125.7, 125.2, 122.5, 118.1, 107.8. IR (neat, cm^{-1}): 3070, 1637, 1567, 1503, 1463, 1437, 1380, 1347, 1330, 1283, 1225, 1202, 1131. HRMS, calculated for $C_{19}H_{12}NaO_2$ ($M+Na^+$): 295.0730, found: 295.0734.

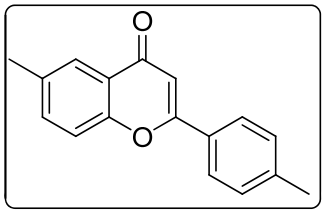
2-(thiophen-2-yl)-4*H*-chromen-4-one 3m



(m.p. 90-92 °C) ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.21 (d, *J* = 8.0 Hz, 1H), 7.74-7.67 (m, 2H), 7.59 (d, *J* = 4.0 Hz, 1H), 7.54 (d, *J* = 4.0 Hz, 1H), 7.42 (t, *J* = 8.0 Hz, 1H), 7.21-7.18 (m, 1H), 6.71 (d, *J*

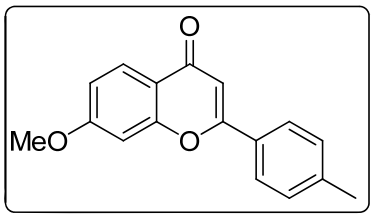
= 1.1 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 177.9, 159.0, 155.9, 135.2, 133.7, 130.3, 128.5, 128.4, 125.7, 125.3, 124.0, 117.9, 106.2. IR (neat, cm^{-1}): 3066, 1619, 1567, 1461, 1423, 1384, 1352, 1255, 1126. HRMS, calculated for $C_{13}H_8NaO_2S$ ($M+Na^+$): 251.0137, found: 251.0142.

6-methyl-2-p-tolyl-4*H*-chromen-4-one 3n



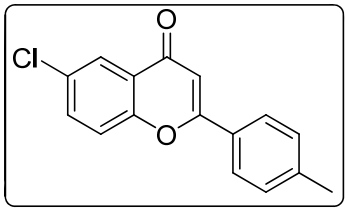
(m.p. 142 °C) ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.04 (s, 1H), 7.85 (d, *J* = 8.0 Hz, 2H), 7.51 (q, *J* = 8.0 Hz, 2H), 7.35 (d, *J* = 8.0 Hz, 2H), 6.81 (d, *J* = 4.0 Hz, 1H), 2.49 (s, 3H), 2.47 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 178.6, 163.5, 154.6, 142.2, 135.1, 134.9, 129.8, 129.1, 126.2, 125.1, 123.7, 117.9, 106.9, 21.6, 21.0. IR (neat, cm^{-1}): 3039, 2919, 2850, 1645, 1614, 1578, 1563, 1509, 1484, 1435, 1364, 1310, 1296, 1223, 1138. HRMS, calculated for $C_{17}H_{14}NaO_2$ ($M+Na^+$): 273.0886, found: 273.0883.

7-methoxy-2-p-tolyl-4*H*-chromen-4-one 3o



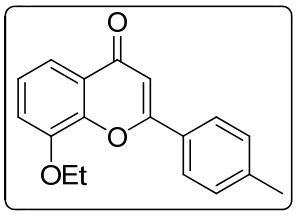
(m.p. 127 °C) ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.14 (d, *J* = 8.0 Hz, 1H), 7.81 (d, *J* = 8.0 Hz, 2H), 7.33 (d, *J* = 8.0 Hz, 2H), 6.99 (d, *J* = 8.0 Hz, 2H), 6.75 (s, 1H), 3.94 (s, 3H), 2.45 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 177.9, 164.1, 163.2, 158.0, 142.0, 129.7, 129.1, 127.0, 126.1, 117.9, 114.3, 106.9, 100.4, 55.8, 21.5. IR (neat, cm^{-1}): 2920, 1653, 1510, 1472, 1440, 1414, 1377, 1274, 1189, 1160. HRMS, calculated for $C_{17}H_{14}NaO_3$ ($M+Na^+$): 289.0835, found: 289.0836.

6-chloro-2-p-tolyl-4*H*-chromen-4-one 3p



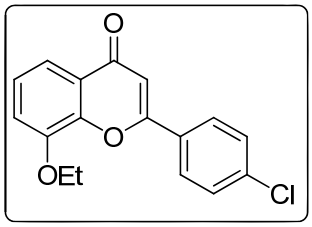
(m.p. 190 °C) ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.20 (d, *J* = 2.6 Hz, 1H), 7.82 (d, *J* = 8.0 Hz, 2H), 7.64 (dd, *J* = 8.0, 4.0 Hz, 1H), 7.54 (d, *J* = 8.0 Hz, 1H), 7.34 (d, *J* = 8.0 Hz, 2H), 6.81 (s, 1H), 2.46 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 177.2, 163.9, 154.5, 142.6, 133.8, 131.1, 129.8, 128.5, 126.2, 125.1, 124.9, 119.8, 106.8, 21.6. IR (neat, cm^{-1}): 3033, 2917, 1641, 1614, 1568, 1512, 1479, 1466, 1437, 1359, 1290, 1273, 1256. HRMS, calculated for $C_{16}H_{11}ClNaO_2$ ($M+Na^+$): 293.0340, found: 293.0339.

8-ethoxy-2-p-tolyl-4*H*-chromen-4-one 3q



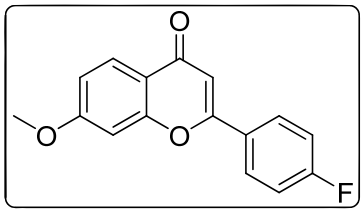
(m.p. 155 °C) ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.87 (d, *J* = 8.0 Hz, 2H), 7.77 (d, *J* = 8.0 Hz, 1H), 7.33 (d, *J* = 8.0 Hz, 3H), 7.18 (d, *J* = 8.0 Hz, 1H), 6.83 (d, *J* = 4.0 Hz, 1H), 4.24 (q, *J* = 6.8 Hz, 2H), 2.45 (s, 3H), 1.57 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 178.6, 163.1, 148.4, 146.8, 142.1, 129.8, 129.1, 126.2, 125.0, 124.7, 116.4, 115.6, 106.6, 65.0, 21.6, 14.8. IR (neat, cm^{-1}): 3030, 2991, 2941, 1646, 1612, 1601, 1579, 1510, 1491, 1466, 1413, 1376, 1351, 1281, 1247, 1217, 1191, 1180, 1149. HRMS, calculated for $C_{18}H_{16}NaO_3$ ($M+Na^+$): 303.0992, found: 303.0993.

2-(4-chlorophenyl)-8-ethoxy-4*H*-chromen-4-one 3r



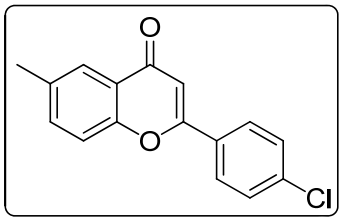
(m.p. 168 °C) ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.89 (dd, *J* = 8.0, 4.0 Hz, 2H), 7.75 (d, *J* = 8.0 Hz, 1H), 7.50 (dd, *J* = 8.0, 4.0 Hz, 2H), 7.30 (t, *J* = 8.0 Hz, 1H), 7.18 (dd, *J* = 8.0, 4.0 Hz, 1H), 6.80 (s, 1H), 4.22 (q, *J* = 8.0 Hz, 2H), 1.57 (t, *J* = 6.0 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 178.4, 161.7, 148.4, 146.6, 137.8, 130.3, 129.4, 127.5, 125.0, 124.9, 116.3, 115.6, 107.3, 64.9, 14.8. IR (neat, cm^{-1}): 3071, 2983, 2934, 2361, 1638, 1595, 1578, 1493, 1471, 1444, 1412, 1397, 1375, 1353, 1277, 1253, 1221, 1178, 1148. HRMS, calculated for $C_{17}H_{13}ClNaO_3$ ($M+Na^+$): 323.0445, found: 323.0444.

2-(4-fluorophenyl)-7-methoxy-4*H*-chromen-4-one 3s



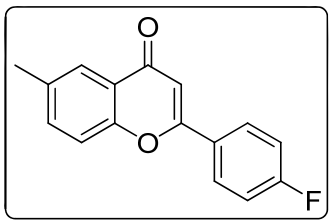
(m.p. 160 °C) ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.16 (d, J = 8.0 Hz, 1H), 7.96-7.92 (m, 2H), 7.24 (t, J = 8.0 Hz, 2H), 7.04-6.99 (m, 2H), 6.74 (s, 1H), 3.97 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 177.7, 165.9, 163.8 (d, J_{CF} = 81 Hz), 162.0, 157.9, 128.4 (d, J_{CF} = 10 Hz), 128.1(J_{CF} = 10 Hz), 127.1, 117.7, 116.3 (d, J_{CF} = 30 Hz), 114.5, 107.3, 100.4, 55.9. ^{19}F NMR (376 MHz, $CDCl_3$) δ -107.77 ppm. IR (neat, cm^{-1}): 3073, 3013, 2957, 2849, 1660, 1607, 1509, 1471, 1442, 1417, 1377, 1287, 1252, 1232, 1203, 1192, 1164. HRMS, calculated for $C_{16}H_{11}FNaO_3$ (M+Na$^+$): 293.0584, found: 293.0585.

2-(4-chlorophenyl)-6-methyl-4*H*-chromen-4-one 3t



(m.p. 194 °C) ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.01 (s, 1H), 7.86 (d, J = 8.0 Hz, 2H), 7.53-7.45 (m, 4H), 6.78 (s, 1H), 2.47 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 178.3, 162.0, 154.4, 137.8, 135.4, 135.1, 130.3, 129.3, 127.5, 125.1, 123.5, 117.8, 107.5, 21.0. IR (neat, cm^{-1}): 3427, 3063, 3028, 2922, 2856, 1642, 1622, 1594, 1577, 1490, 1453, 1407, 1378, 1364, 1285, 1228. HRMS, calculated for $C_{16}H_{11}ClNaO_2$ (M+Na$^+$): 293.0340, found: 293.0340.

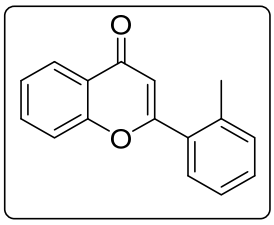
2-(4-fluorophenyl)-6-methyl-4*H*-chromen-4-one 3u



(m.p. 150-154 °C) ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.01 (d, J = 0.9 Hz, 1H), 7.93 (q, J = 8.0, 2H), 7.51 (dd, J = 8.0, 4.0 Hz, 1H), 7.45 (d, J = 8.0 Hz, 1H), 7.21 (t, J = 8.0 Hz, 2H), 6.75 (s, 1H), 2.47

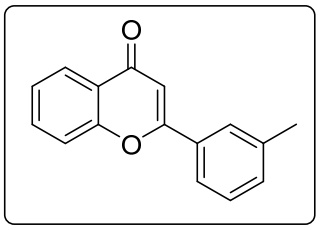
(s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 178.4, 166.0, 162.8(d, J_{CF} = 81 Hz), 154.4, 135.3, 135.0, 128.5 (d, J_{CF} = 10 Hz), 128.1(d, J_{CF} = 3.0 Hz), 125.1, 123.5, 117.8, 116.3 (d, J_{CF} = 30 Hz), 107.2, 21.0. ^{19}F NMR (565 MHz, $CDCl_3$) δ -107.58 ppm. IR (neat, cm^{-1}): 3426, 3063, 2925, 1645, 1602, 1507, 1484, 1453, 1433, 1363, 1299, 1235, 1196, 1162, 1139. HRMS, calculated for $C_{16}H_{11}FNaO_2$ ($M+Na^+$): 277.0635, found: 277.0635.

2-(o-tolyl)-4*H*-chromen-4-one 3v



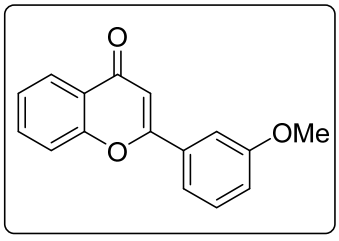
(m.p. 105 °C) ^{1}H NMR (400 MHz, $CDCl_3$) 8.27 (d, *J* = 8.0, 1H), 7.70 (t, *J* = 8.0, 1H), 7.55-7.49 (m, 2H), 7.46-7.41 (m, 2H), 7.33 (t, *J* = 8.0, 2H), 6.50 (s, 1H), 2.49 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 178.3, 166.1, 156.5, 136.8, 133.8, 132.7, 131.3, 130.8, 129.2, 126.2, 125.8, 125.3, 123.8, 118.1, 112.0, 20.6. IR (neat, cm^{-1}): 2926, 1652, 1571, 1465, 1370, 1220, 1130. ESI-MS (M): 236.

2-(m-tolyl)-4*H*-chromen-4-one[2] 3w



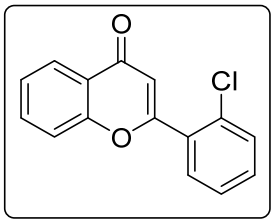
(m.p. 107 °C) ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.24 (dd, *J* = 4.0, 8.0 Hz, 1H), 7.73-7.68 (m, 3H), 7.58 (d, *J* = 8.0 Hz, 1H), 7.44-7.39 (m, 2H), 7.35 (d, *J* = 8.0 Hz, 1H), 6.82 (s, 1H), 2.46 (s, 3H).^{13}C NMR (101 MHz, $CDCl_3$) δ 178.5, 163.6, 156.3, 138.8, 133.7, 132.4, 131.7, 128.9, 126.8, 125.7, 125.2, 124.0, 123.5, 118.1, 107.5, 21.5. IR (neat, cm^{-1}): 3068, 2921, 1637, 1603, 1569, 1488, 1467, 1433, 1368, 1332, 1301, 1269, 1225. ESI-MS (M): 236.

2-(3-methoxyphenyl)-4*H*-chromen-4-one[2] 3x



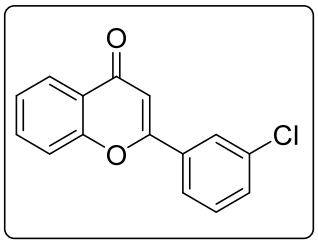
(m.p. 127 °C) ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.27 (d, *J* = 8.0 Hz, 1H), 7.70 (t, *J* = 8.0 Hz, 1H), 7.57 (d, *J* = 8.0 Hz, 1H), 7.50 (d, *J* = 8.0 Hz, 1H), 7.45-7.41 (m, 3H), 7.07 (d, *J* = 4.0 Hz, 1H), 6.83 (s, 1H), 3.89 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 178.5, 163.2, 160.0, 156.2, 133.8, 133.1, 130.1, 125.7, 125.3, 123.9, 118.7, 118.1, 117.2, 111.8, 107.8, 55.5. IR (neat, cm^{-1}): 3078, 3000, 2922, 2842, 1653, 1606, 1572, 1491, 1469, 1446, 1434, 1369, 1346, 1330, 1295, 1275, 1249, 1228, 1213, 1192, 1130. ESI-MS (M): 252.

2-(2-chlorophenyl)-4*H*-chromen-4-one 3y



(m.p. 118-120 °C) ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.26 (d, *J* = 12.0 Hz, 1H), 7.71 (t, *J* = 9.0 Hz, 1H), 7.64 (d, *J* = 12.0 Hz, 1H), 7.53 (q, *J* = 8.0 Hz, 2H), 7.48-7.40 (m, 3H), 6.66 (s, 1H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 178.2, 162.7, 156.6, 133.9, 133.0, 132.0, 131.8, 130.8, 130.7, 127.1, 125.8, 125.4, 123.9, 118.2, 113.0. IR (neat, cm^{-1}): 3064, 2924, 2360, 2341, 1653, 1572, 1467, 1370, 1220, 1129. ESI-MS (M): 256.

2-(3-chlorophenyl)-4*H*-chromen-4-one 3z



(m.p. 118 °C) ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.24 (d, *J* = 8.0 Hz, 1H), 7.93 (t, *J* = 2.0 Hz, 1H), 7.80 (d, *J* = 8.0 Hz, 1H), 7.73 (dt, *J* = 4.0, 8.0 Hz, 1H), 7.60 (d, *J* = 12 Hz, 1H), 7.54-7.43 (m, 3H), 6.82 (s, 1H).^{13}C NMR (101 MHz, $CDCl_3$) δ 178.2, 161.8, 156.2, 135.3, 134.0, 133.6, 131.5, 130.3, 126.4, 125.8, 125.5, 124.4, 123.9, 118.1, 108.2. IR (neat, cm^{-1}): 3085, 1645, 1565, 1466, 1422, 1372, 1335, 1304, 1261, 1226, 1131. ESI-MS (M): 256.

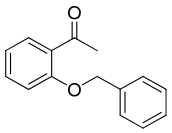
Reference and notes

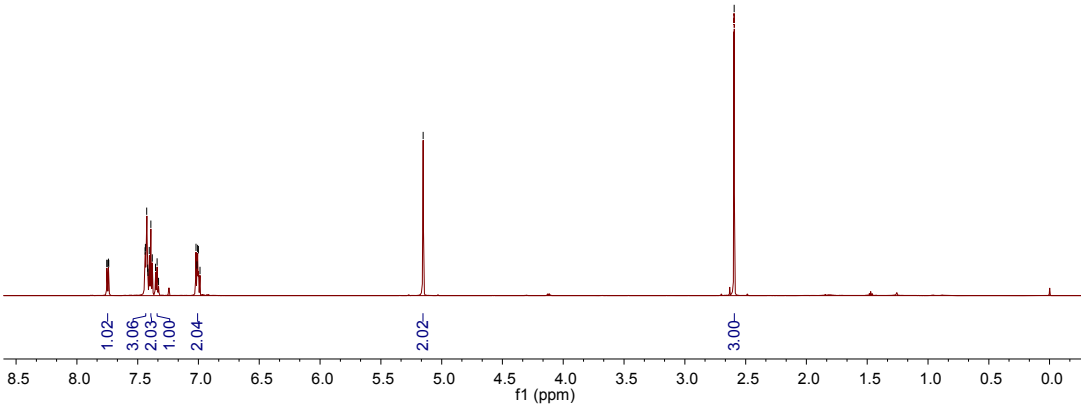
1. M. von Delius, C. M. Le, V. M. Dong, *J. Am. Chem. Soc.*, 2012, **134**, 15022.

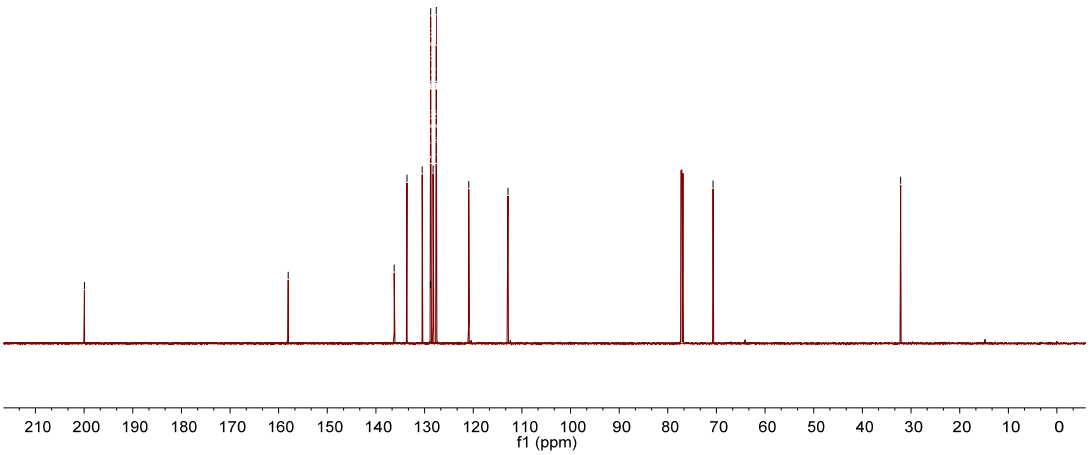
2. (a) J. M. Liu, M. W. Liu, Y. Y. Yue, N. F. Zhang, Y. L. Zhang, K. L. Zhuo, *Tetrahedron Lett.*

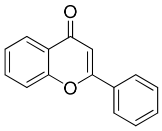
2013, **54**, 1802; (b) Q. Yang, H. Alper, *J. Org. Chem.* 2010, **75**, 948; (c) X.-F. Wu, H. Neumann, M. Beller, *Chem. Eur. J.* 2012, **18**, 1259; (d) Z. Y. Du, H. F. Ng, K. Zhang, H. Q. Zeng, J. Wang, *Org. Biomol. Chem.*, 2011, **9**, 6930.

NMR Spectra of Products





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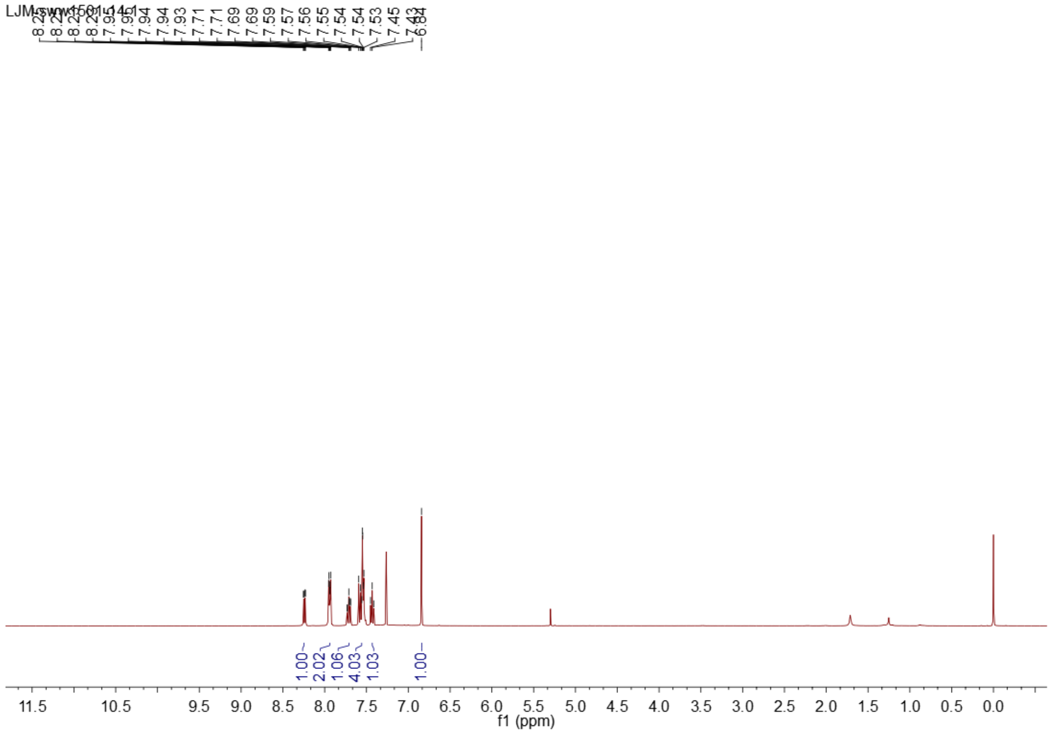
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7.59
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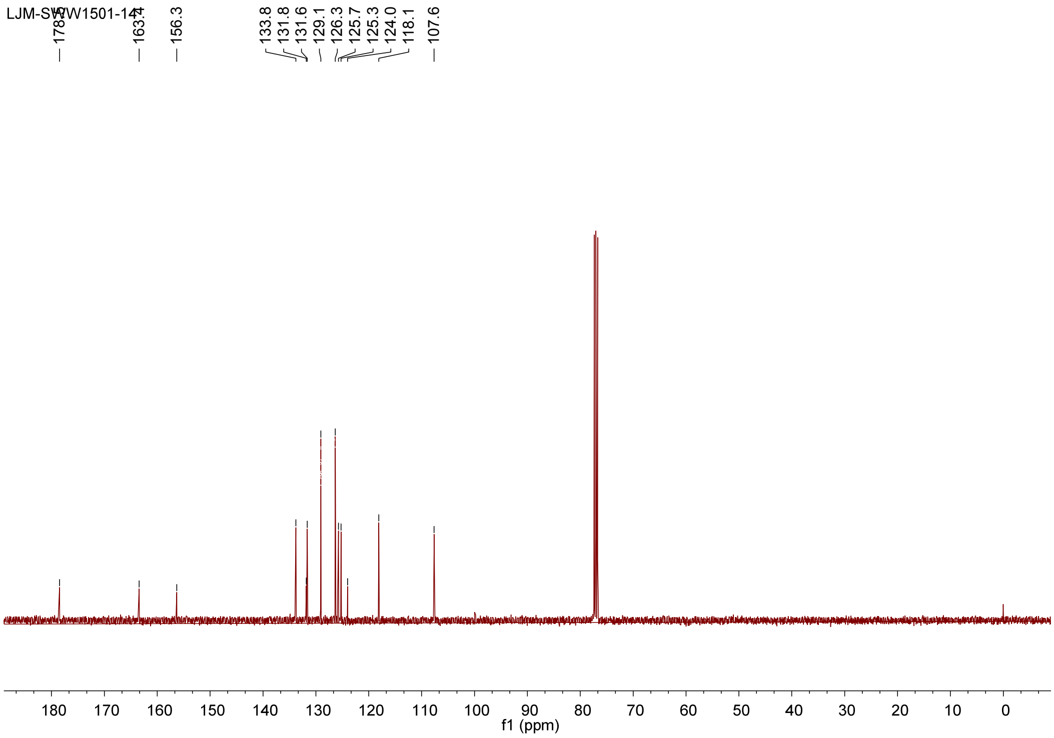
7.54
7.53
7.45
6.84
1.00
2.02
1.06
4.03
1.03
1.00
11.5 10.5 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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118.1
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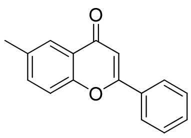
f1 (ppm)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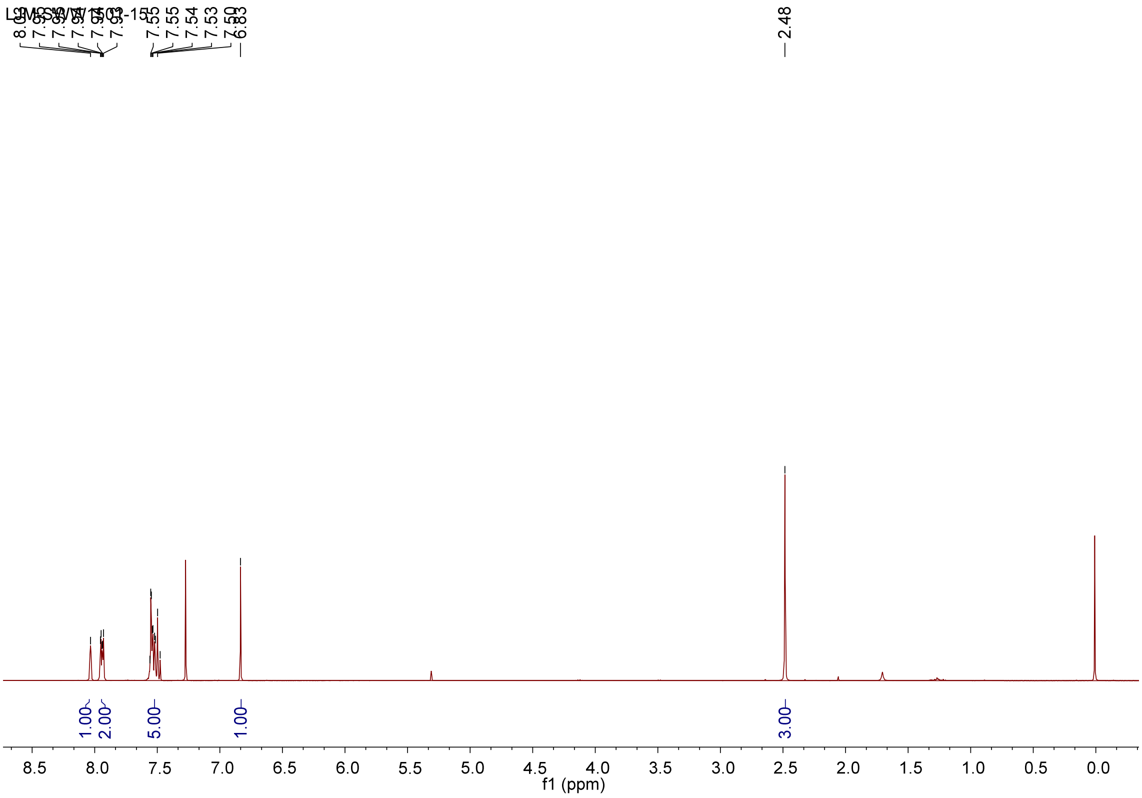
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5.00
1.00
3.00
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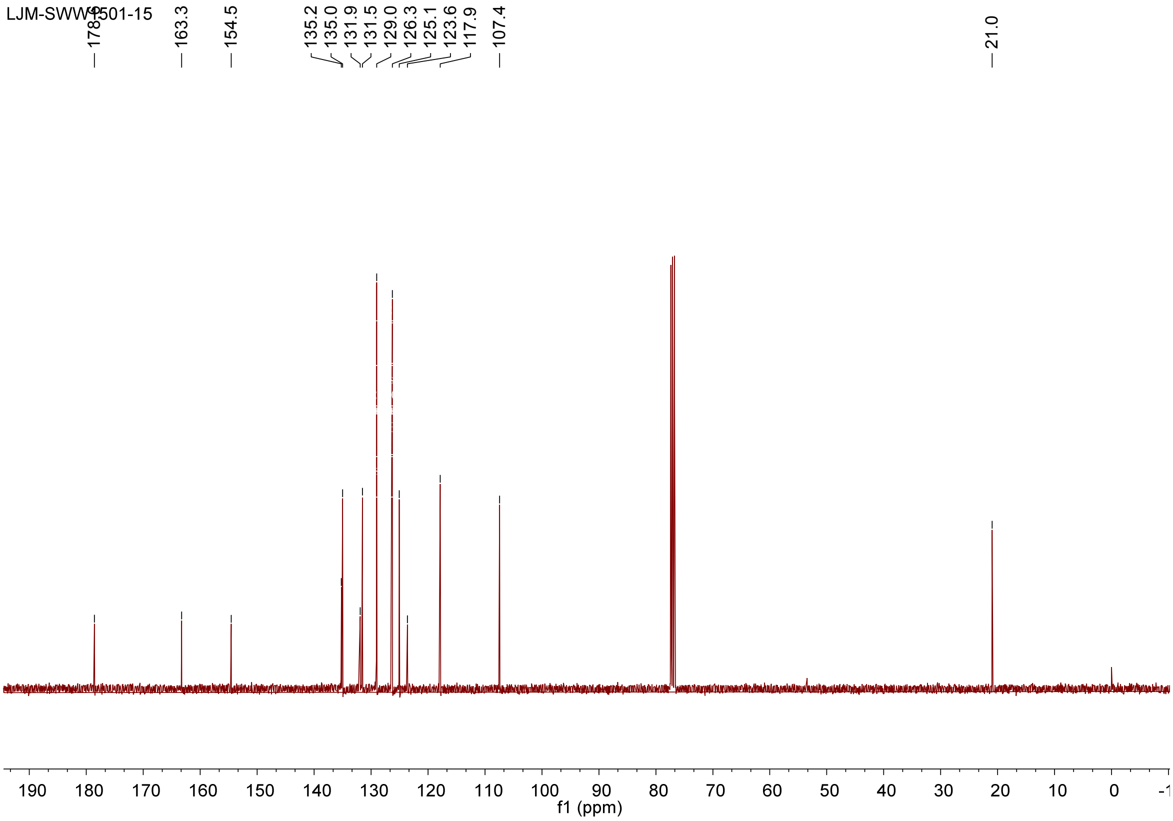
f1 (ppm)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117.9
107.4
21.0
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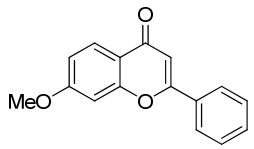
f1 (ppm)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MeO
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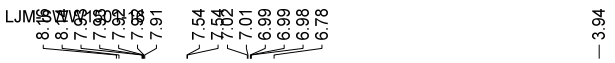
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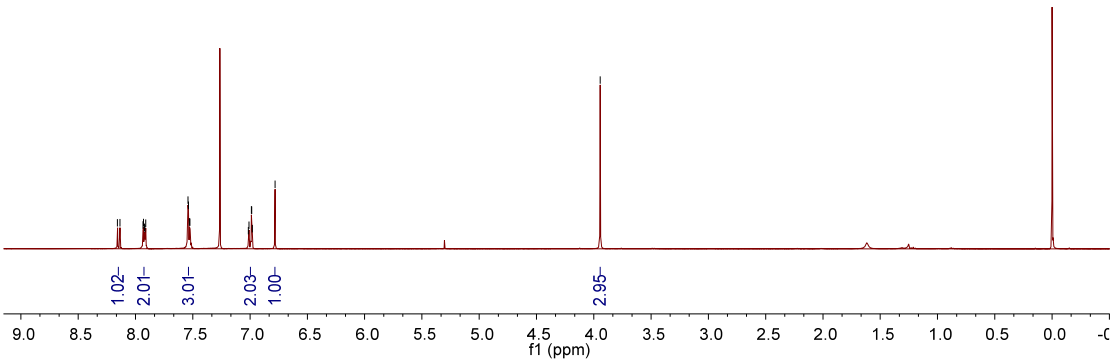
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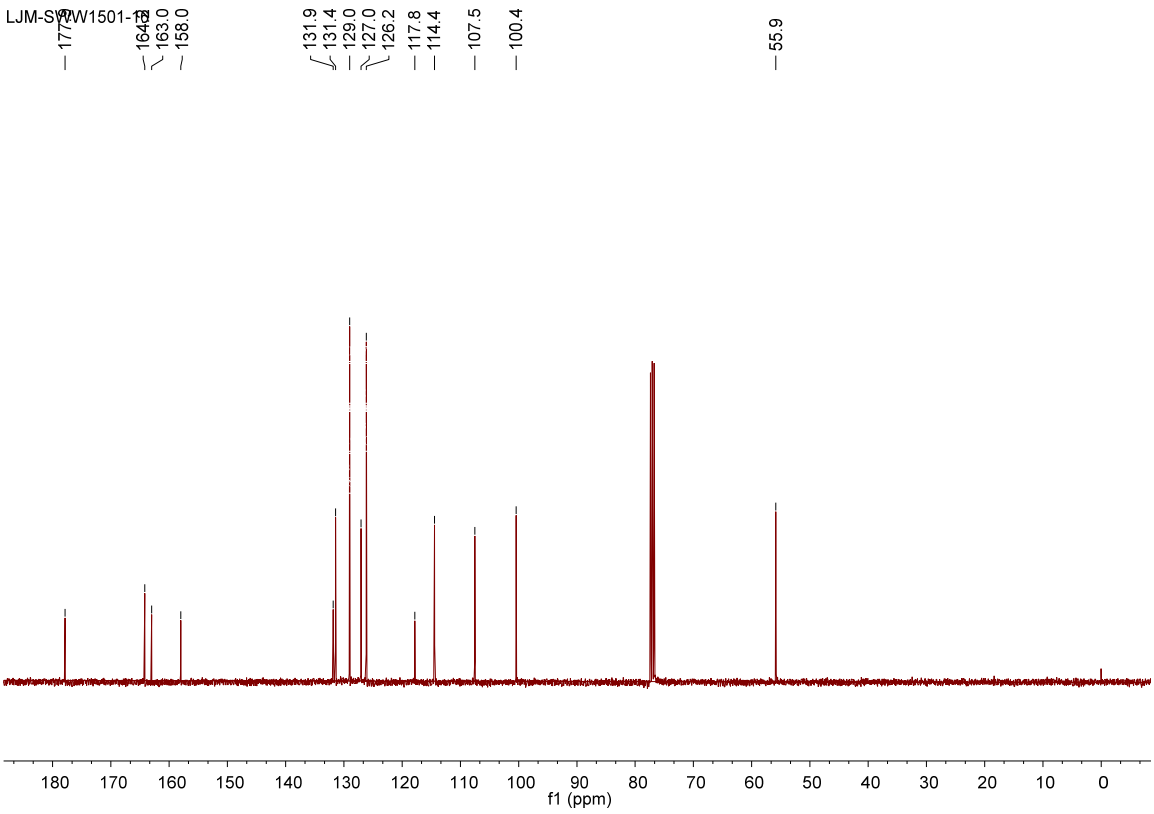
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117.8
114.4
107.5
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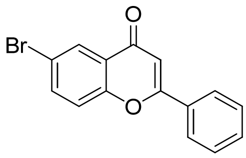
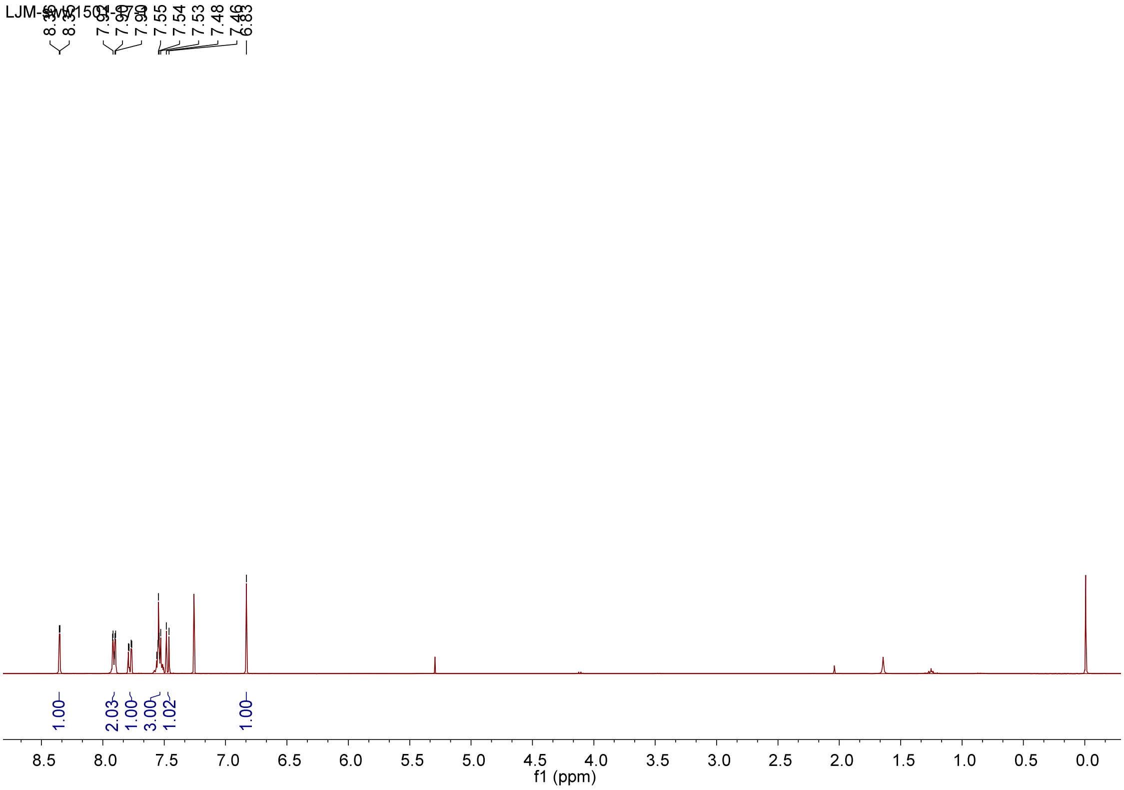
55.9
f1 (ppm)


Br
O
O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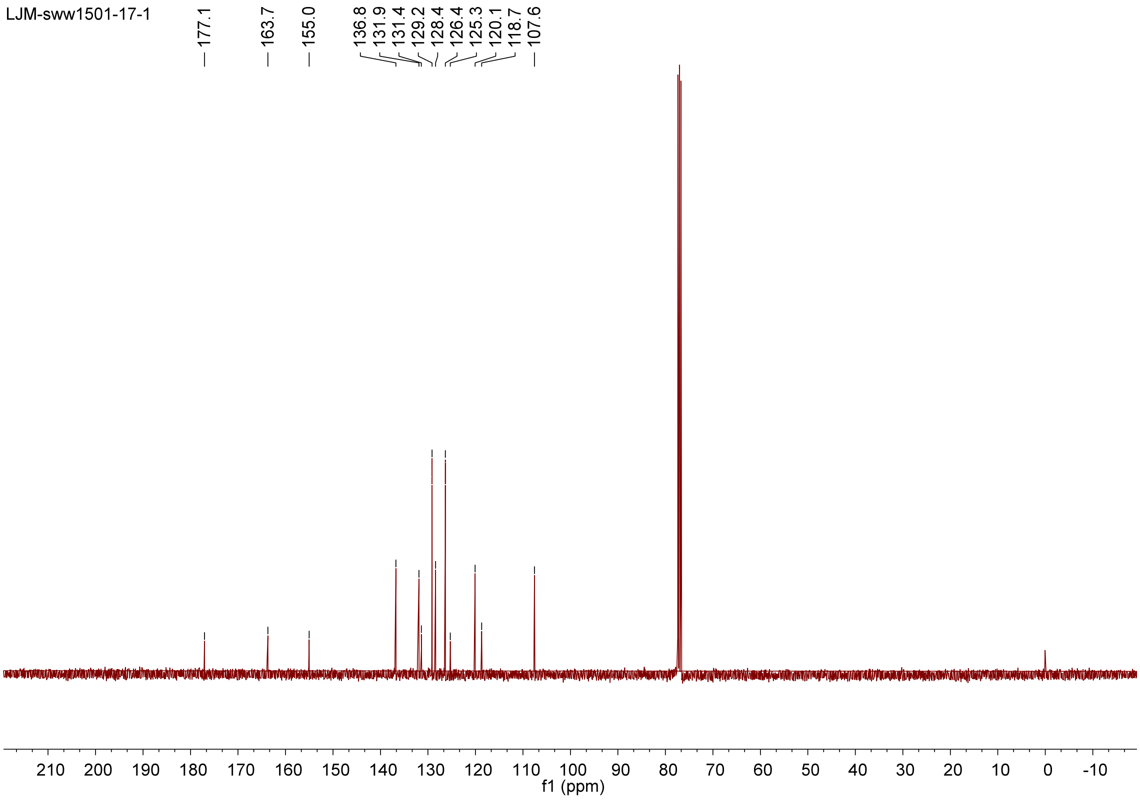
1.00
f1 (ppm)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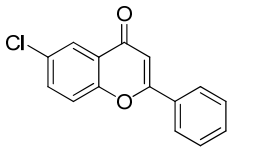
118.7
107.6
f1 (ppm)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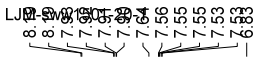
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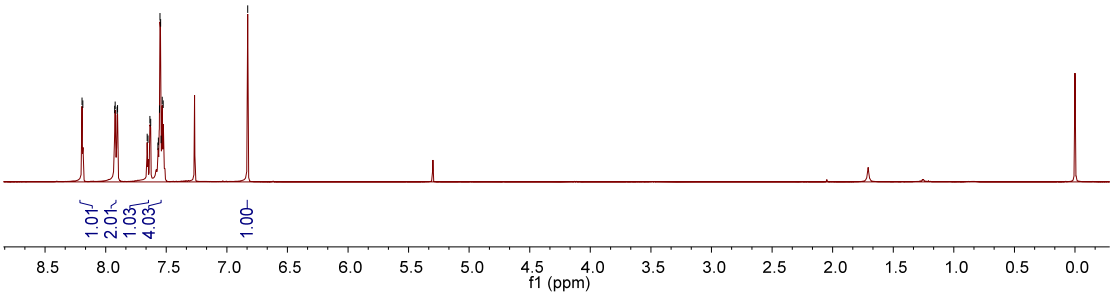
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3.5
3.0
2.5
2.0
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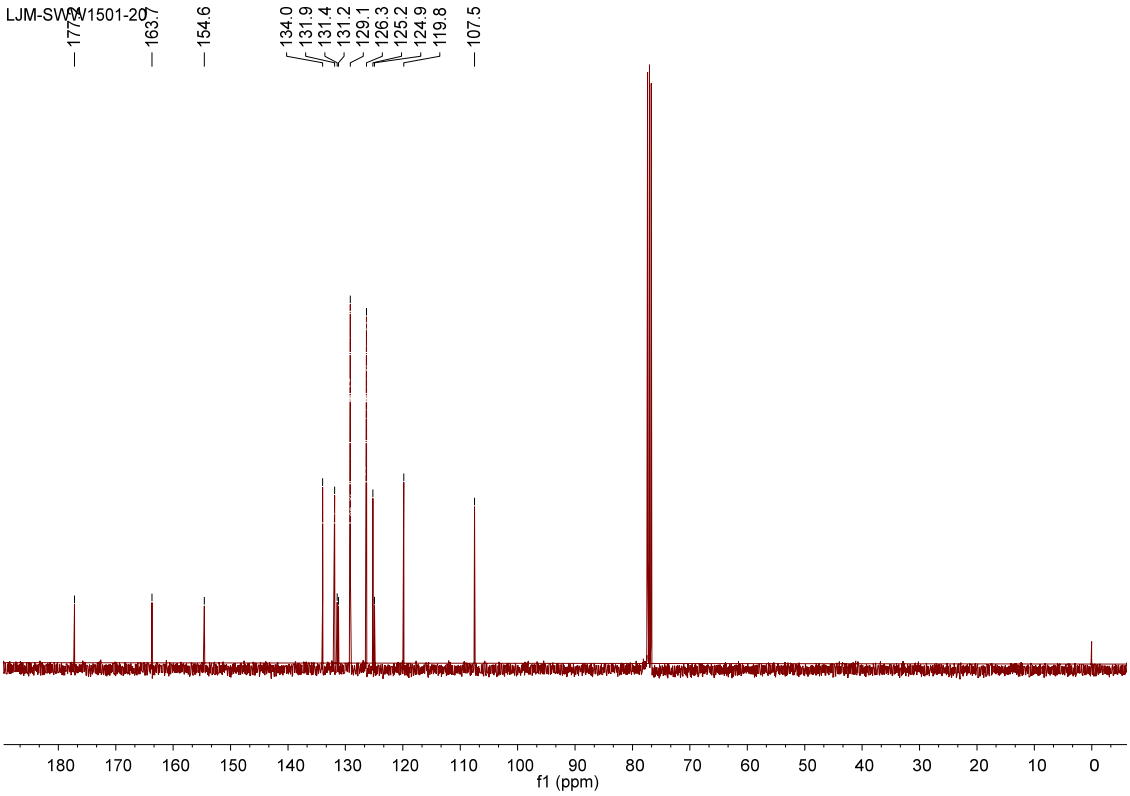
1.0
0.5
0.0
f1 (ppm)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131.4
131.2
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119.8
107.5
180
170
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150
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130
120
110
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50
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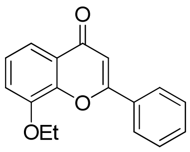
10
0
f1 (ppm)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O
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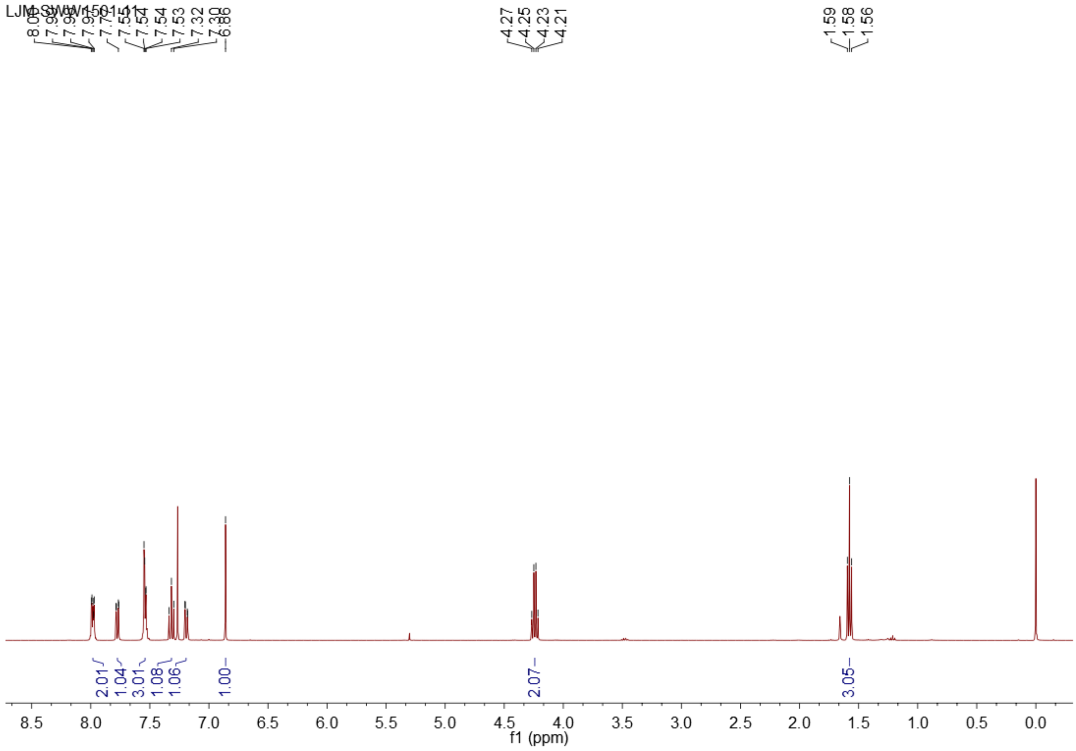
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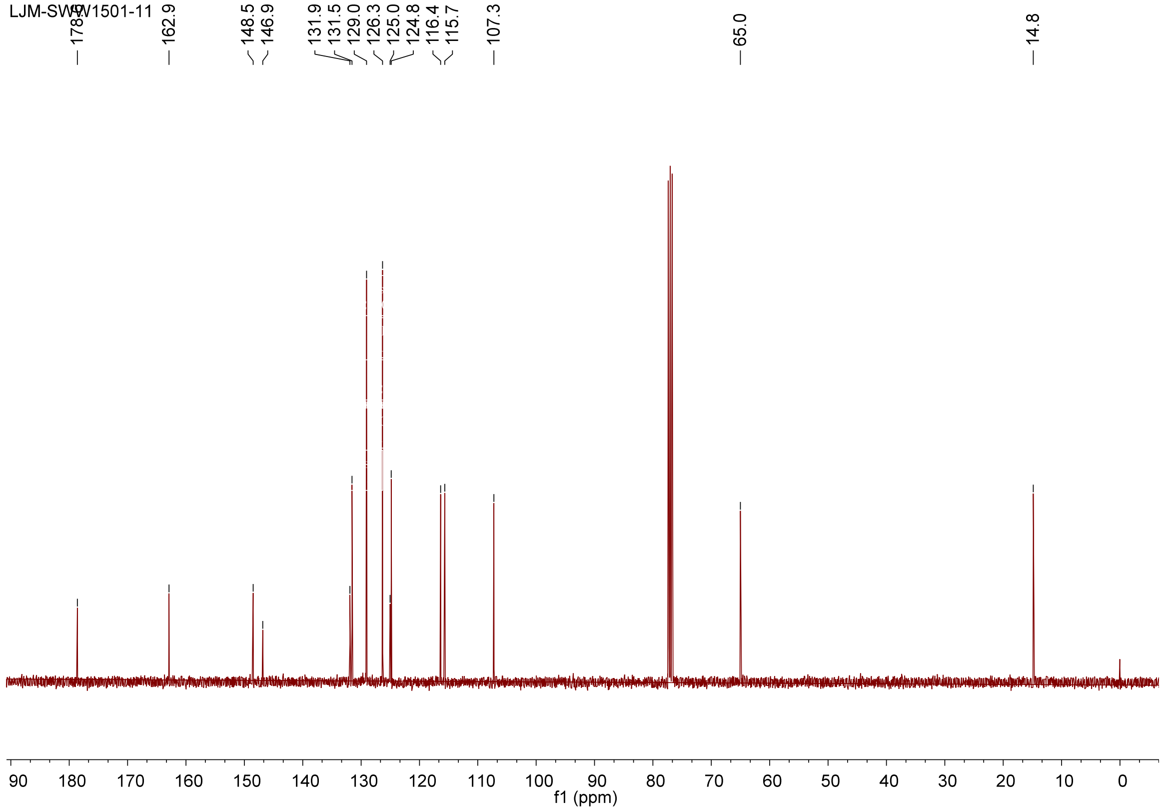
3.05
f1 (ppm)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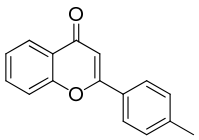
f1 (ppm)

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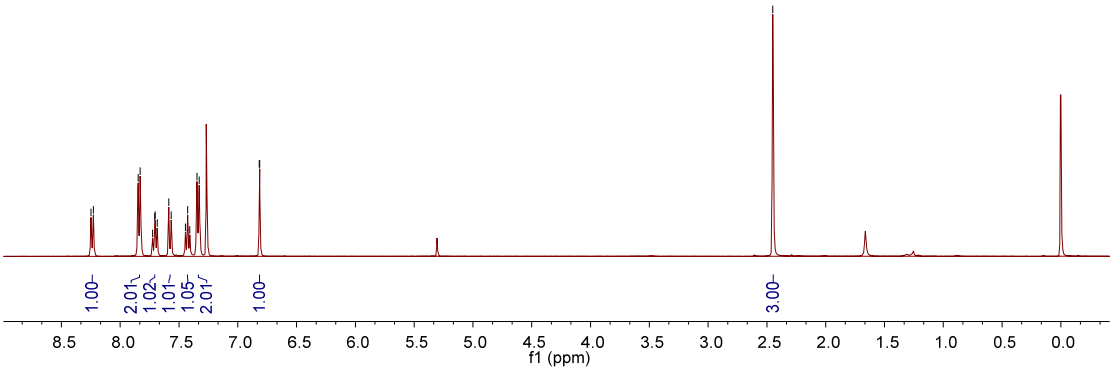
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6.0
5.5
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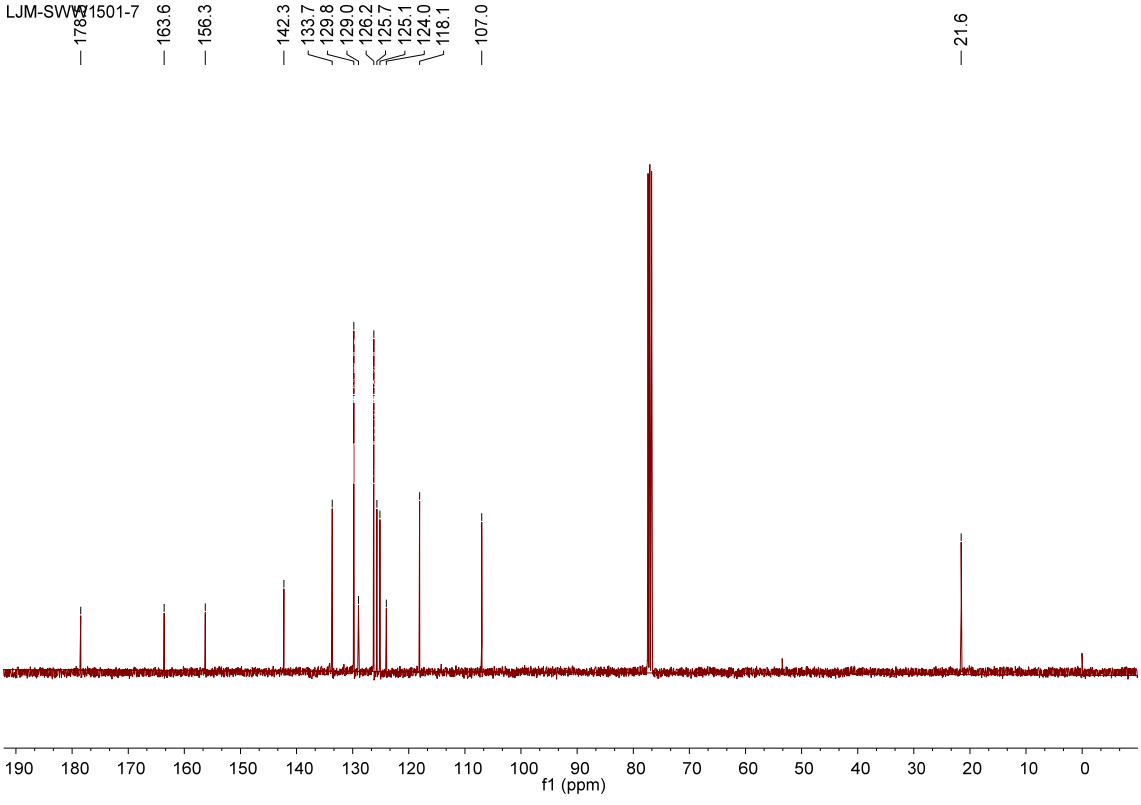
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124.0
118.1
107.0
21.6
190
180
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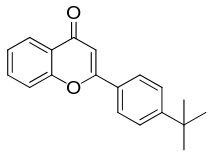
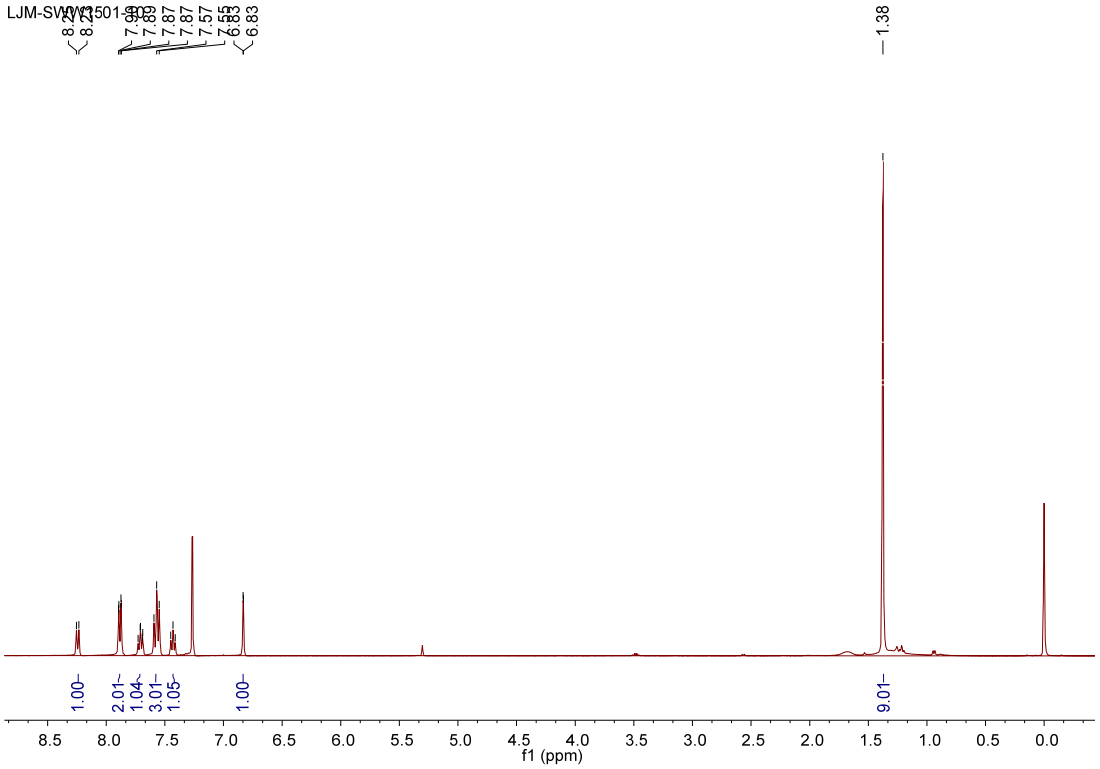
f1 (ppm)

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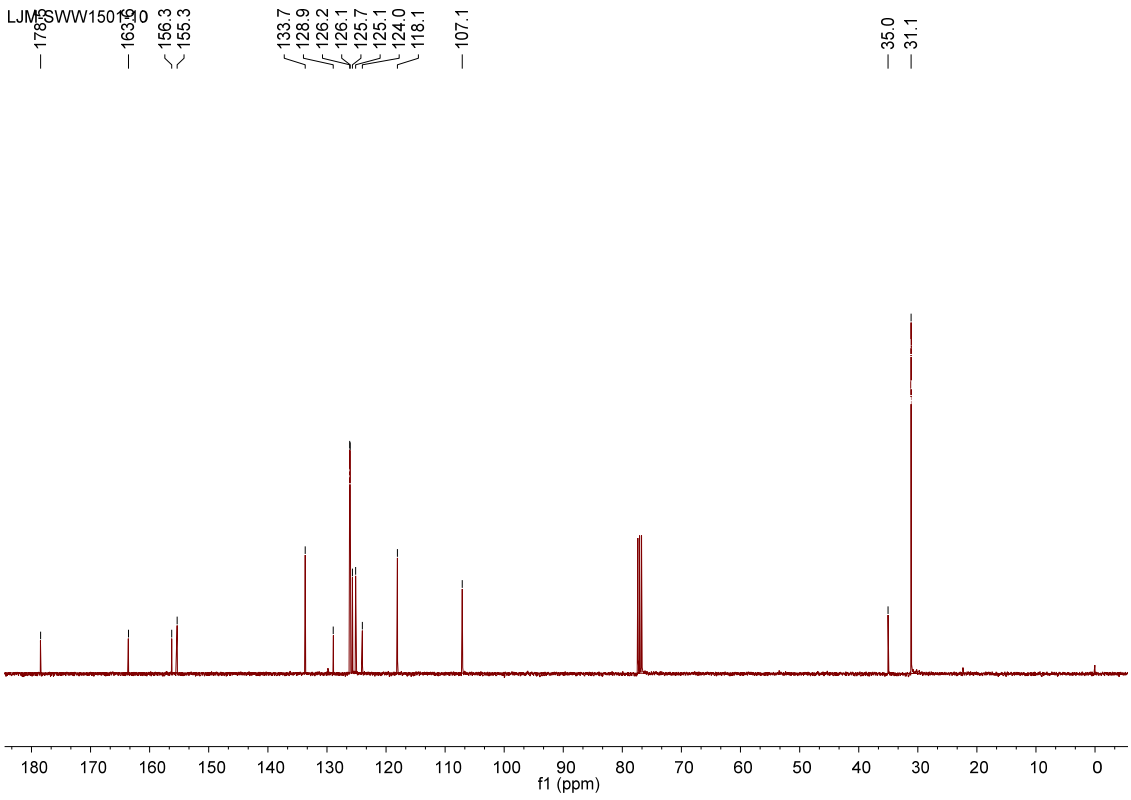
f1 (ppm)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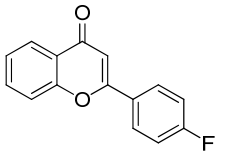
f1 (ppm)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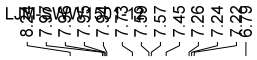
F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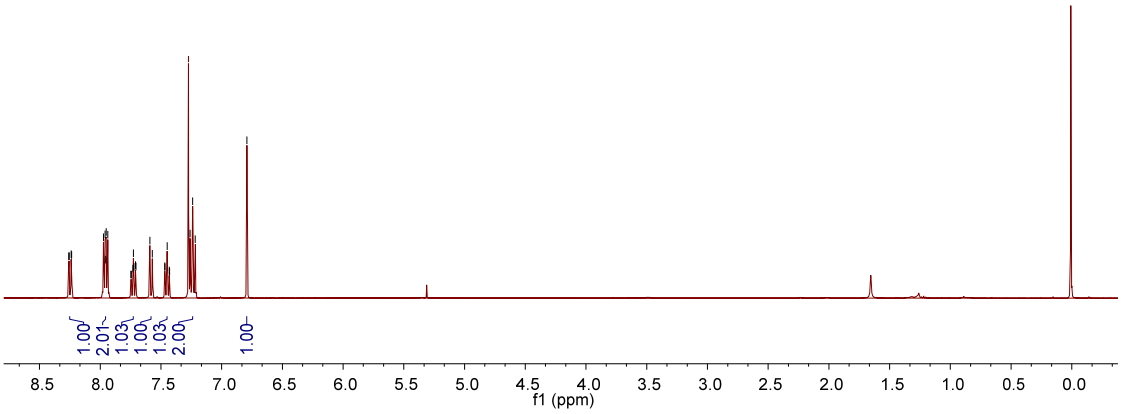
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5.5
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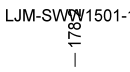
f1 (ppm)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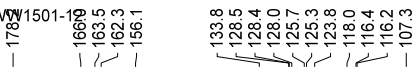
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116.4
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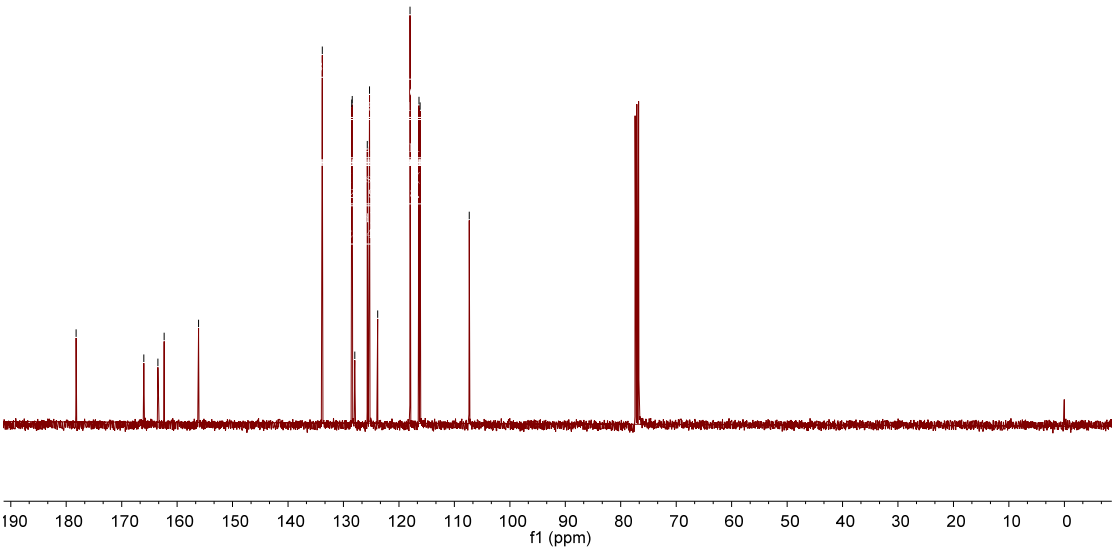
107.3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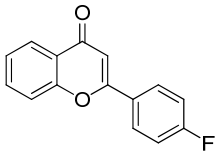
f1 (ppm)



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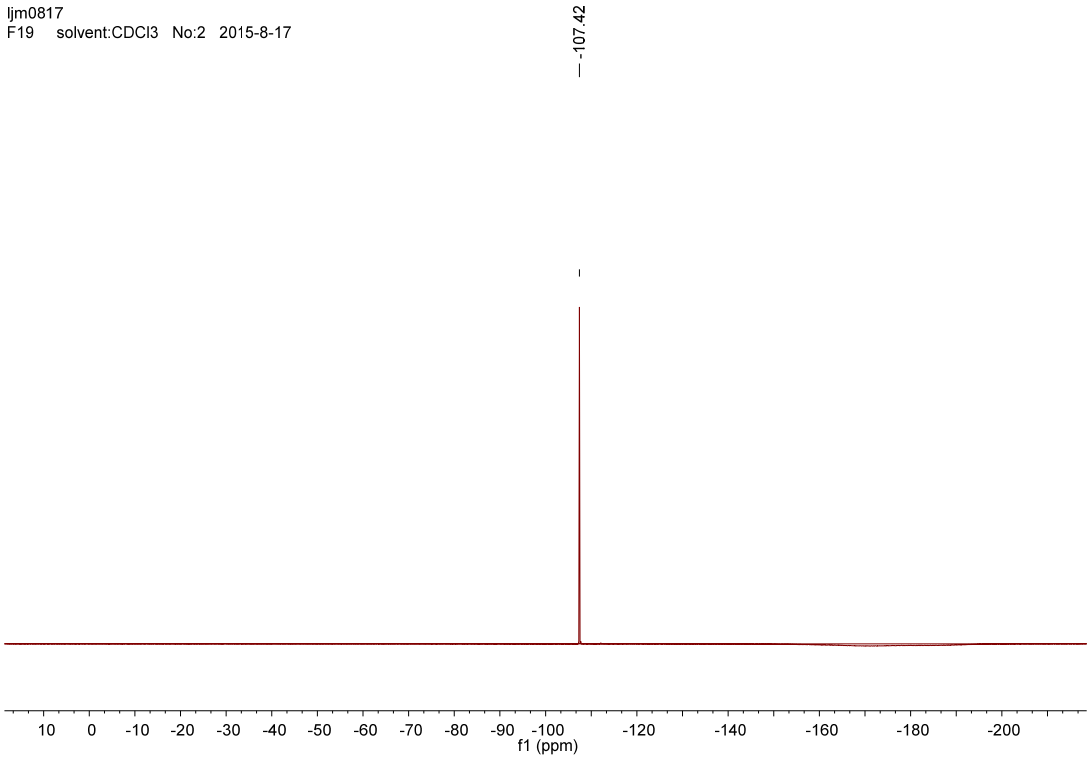

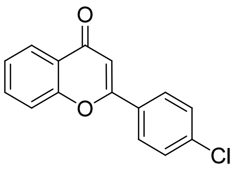
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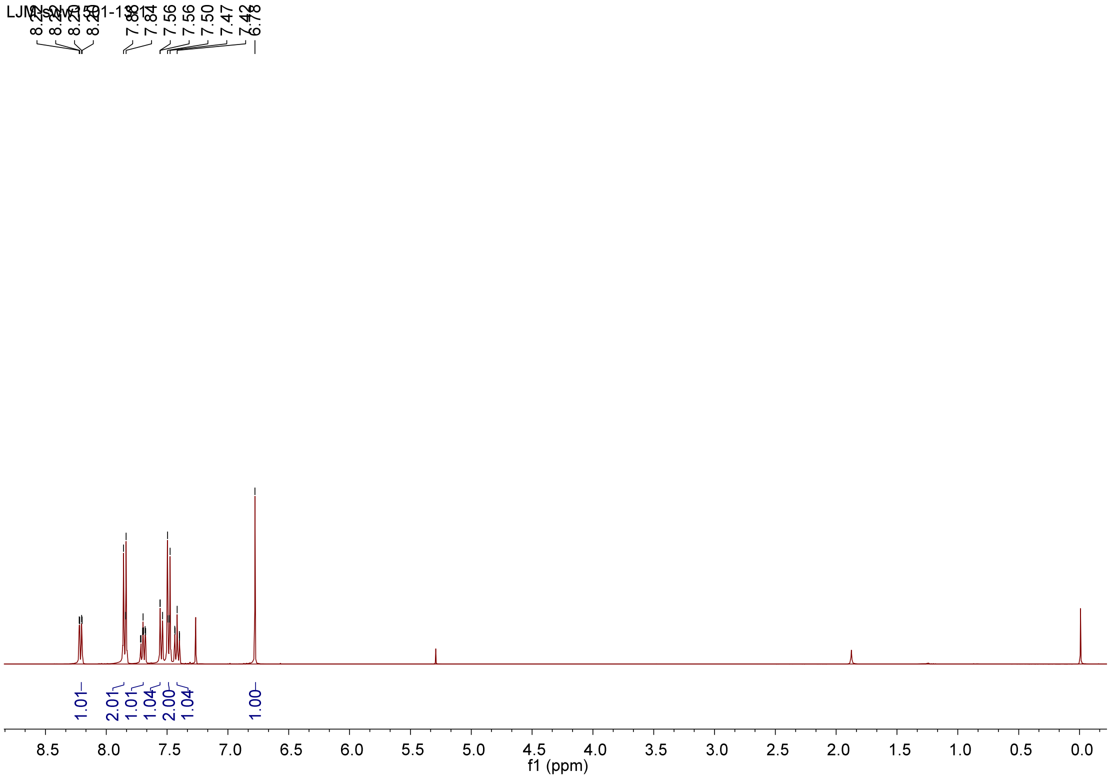
O
Cl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1.01
1.04
2.00
1.04
1.00
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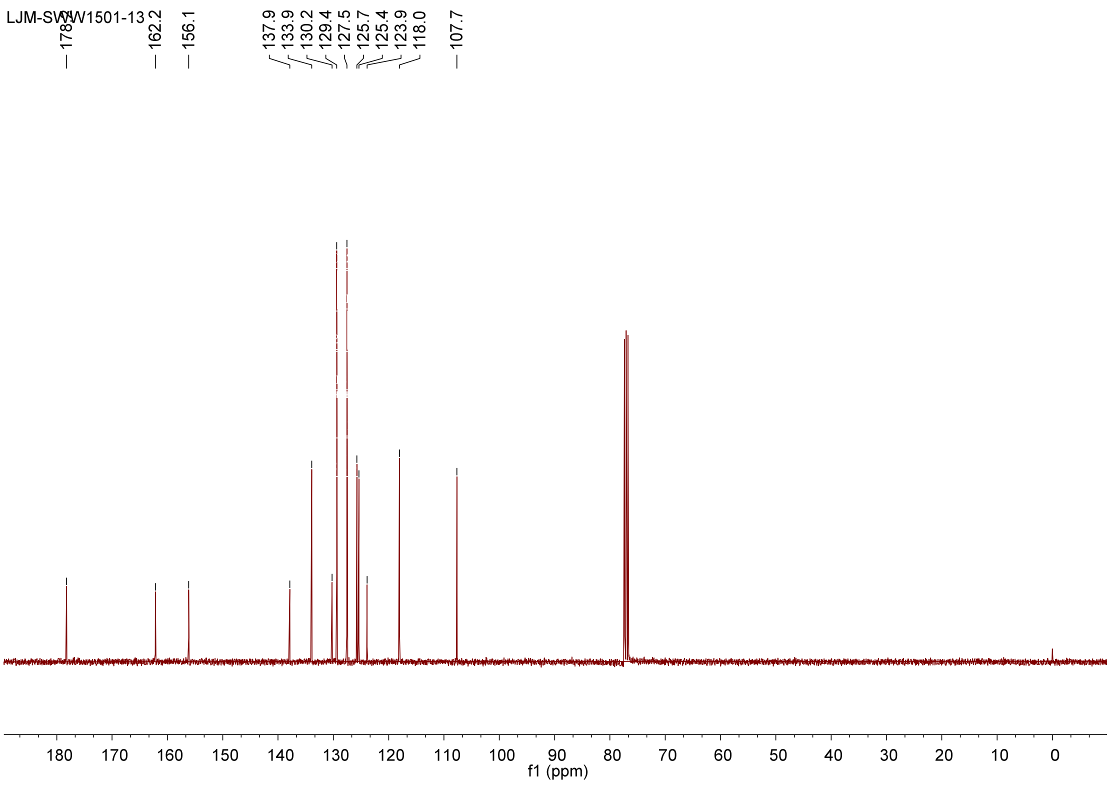
f1 (ppm)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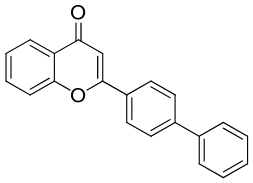
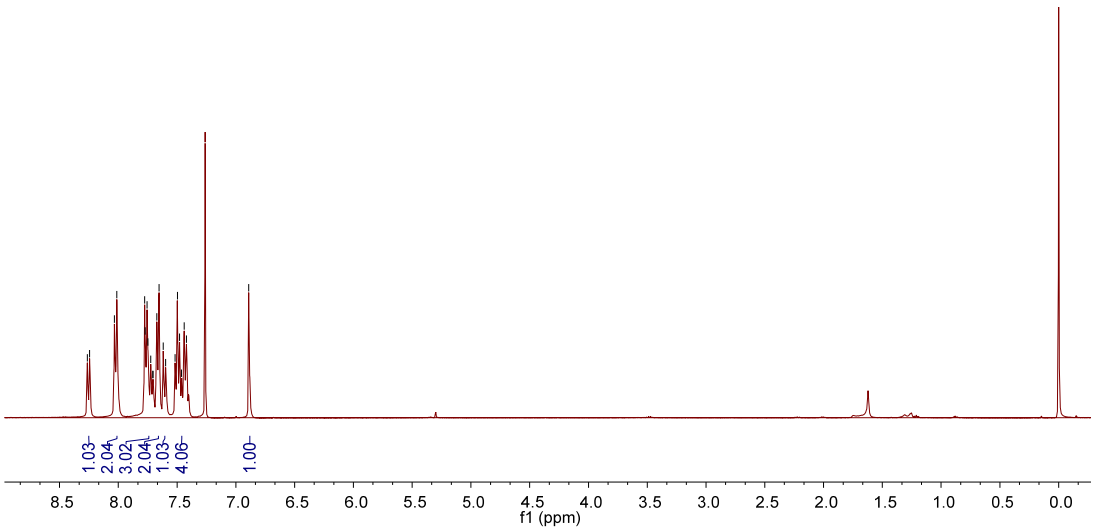
f1 (ppm)

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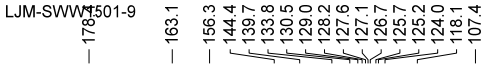
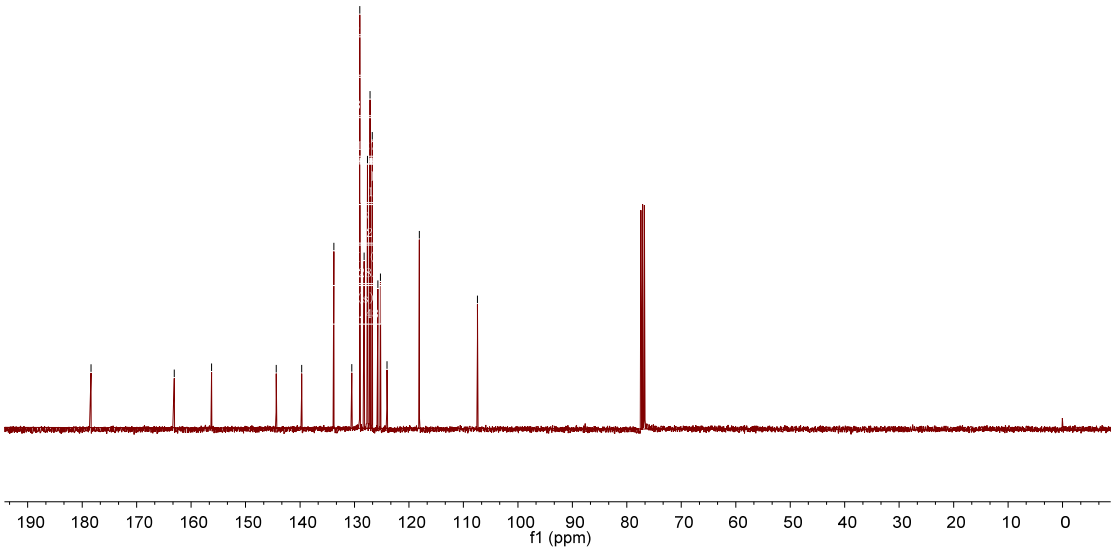
2.04
1.03
4.06
1.00
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

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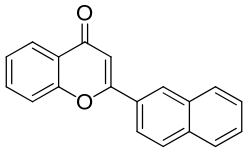
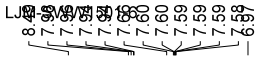
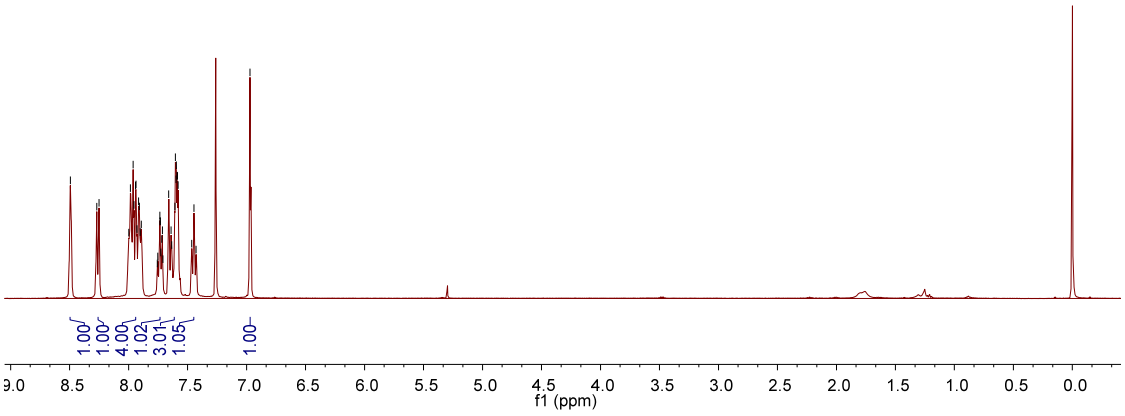
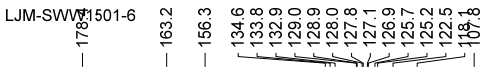
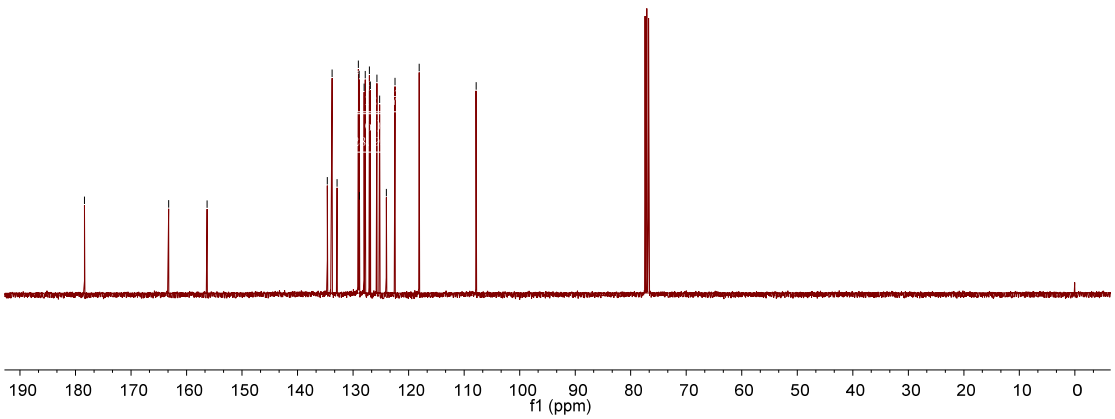
f1 (ppm)

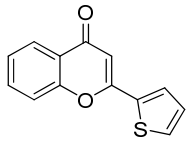


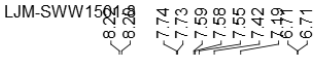

7.60
7.59
7.59
7.59
7.58

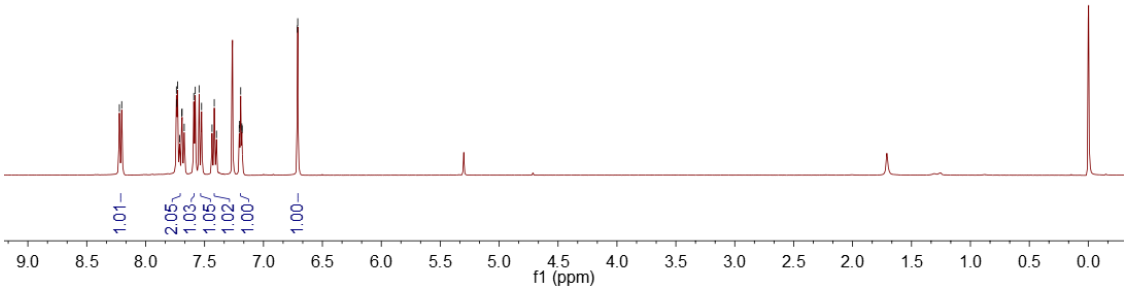

1.00
1.00
4.00
1.02
3.01
1.05
1.00
f1 (ppm)

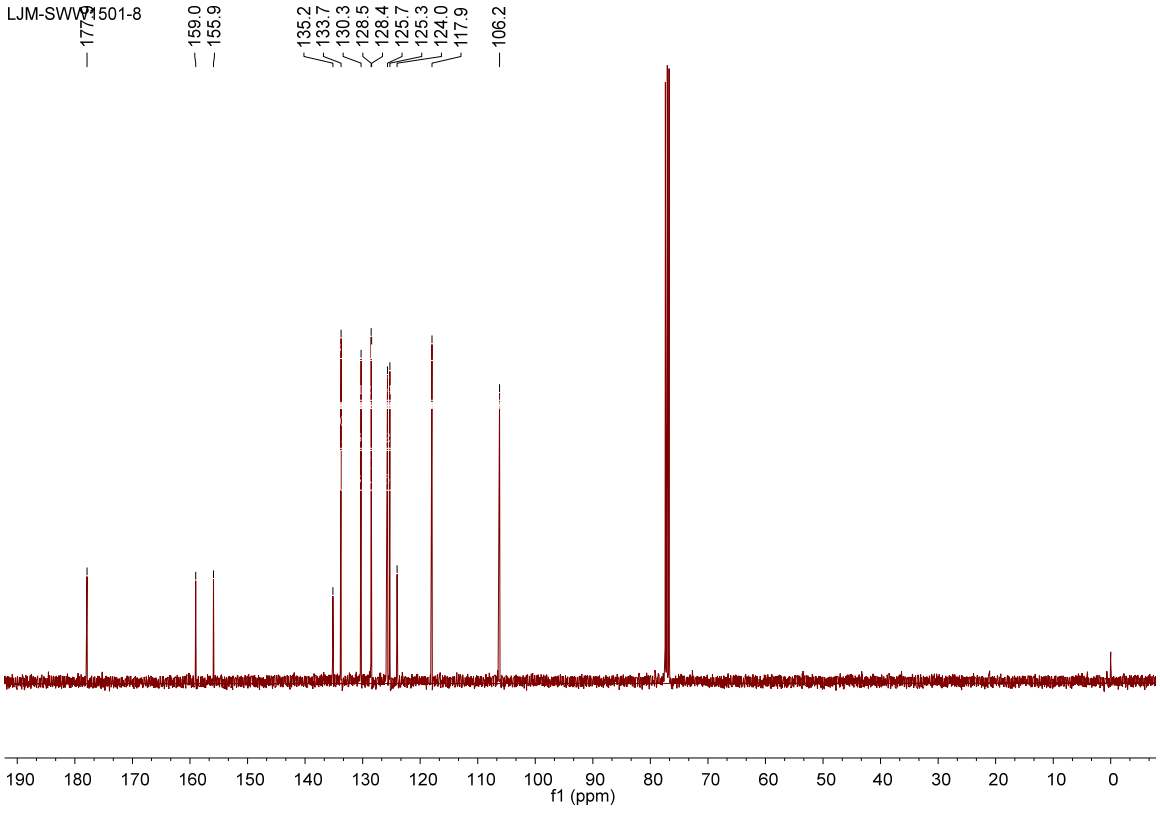

LJM-SWV-1501-6
163.2
156.3
134.6
133.8
132.9
129.0
128.9
128.0
127.8
127.1
126.9
125.7
125.2
122.5

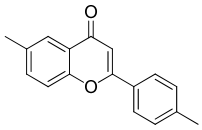

f1 (ppm)


O
O
S

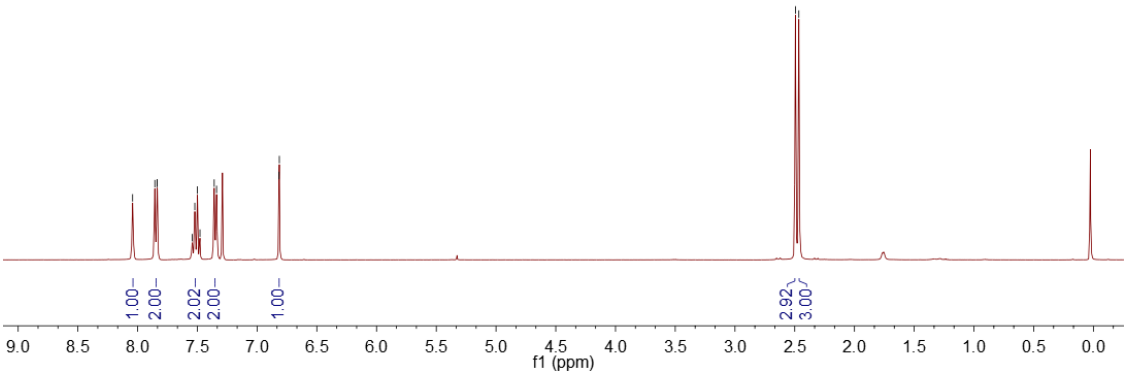

LJM-SWW1501-8
8.22
8.20
7.74
7.73
7.59
7.58
7.55
7.42
7.19
6.71
6.71


1.01
2.05
1.03
1.05
1.02
1.00
1.00
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

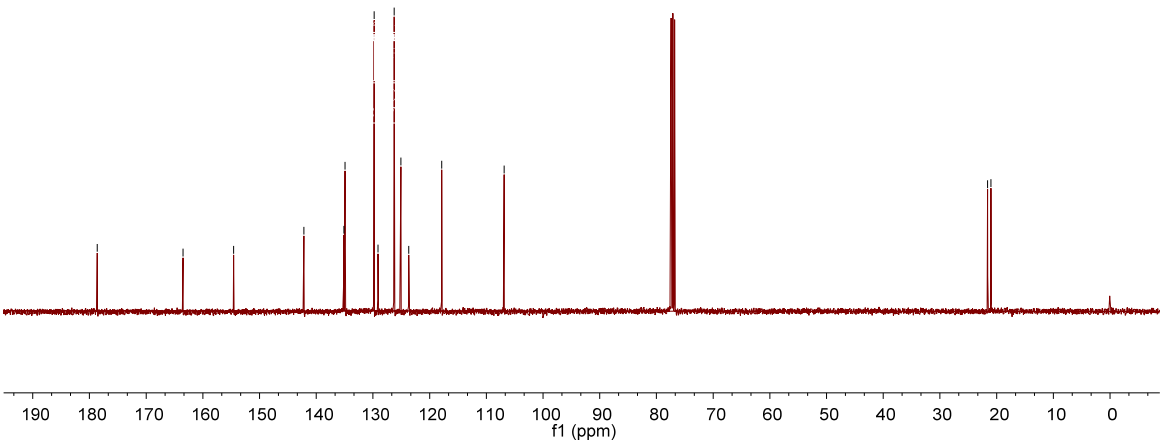

LJM-SWW1501-8
177.1
159.0
155.9
135.2
133.7
130.3
128.5
128.4
125.7
125.3
124.0
117.9
106.2
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

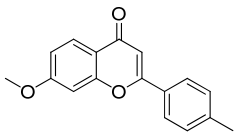



LJM-SWW1501-2
8.00
7.85
7.84
7.54
7.52
7.50
7.48
7.36
7.34
6.82
6.81
2.49
2.47


1.00
2.00
2.02
2.00
1.00
2.92
3.00
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

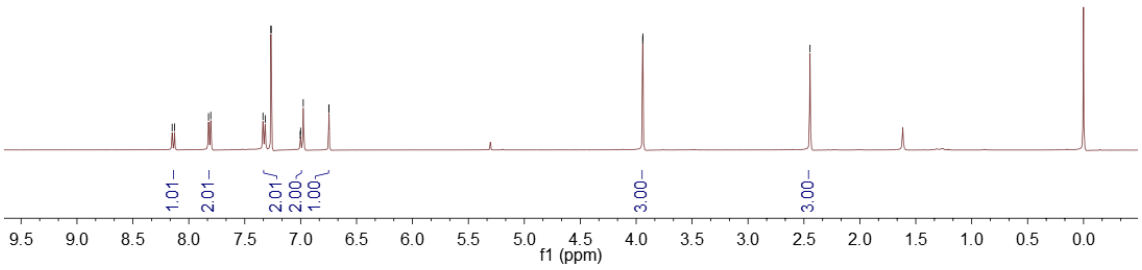

LJM-SWW1501-2
178.5
163.5
154.6
142.2
135.1
134.9
129.8
129.1
126.2
125.1
123.7
117.9
106.9
21.6
21.0


190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)



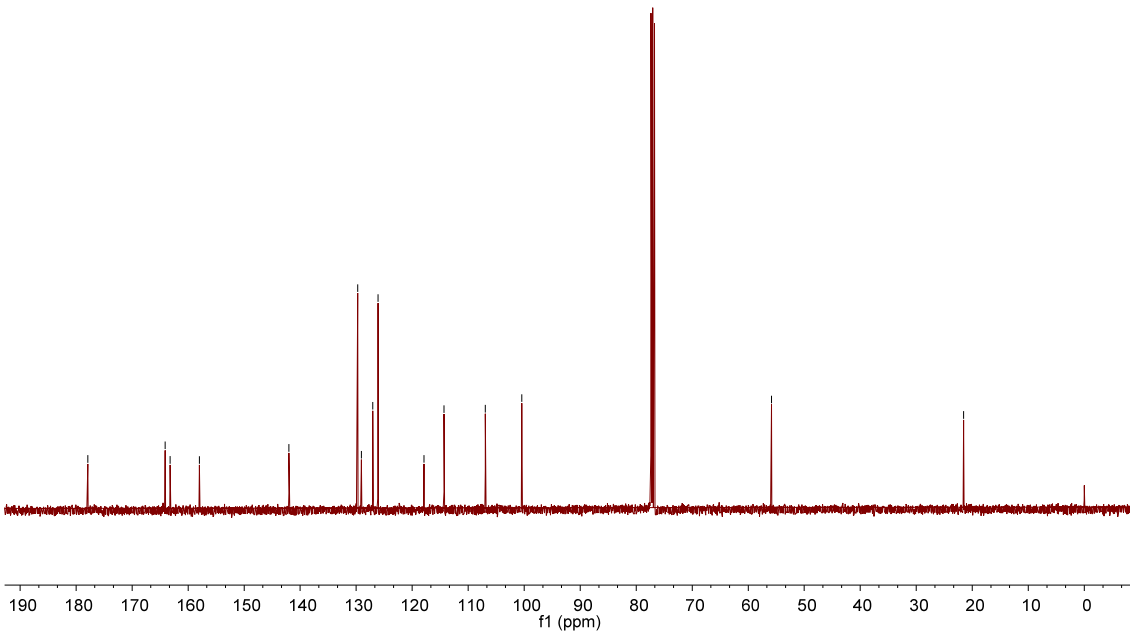
LJM-SWW1501-1

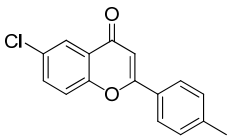
8.15
8.13
7.82
7.80
7.34
7.32
7.27
7.26
7.00
7.00
6.98
6.75
6.75
3.94
3.94
2.45



LJM-SWW1501-1

177.3
164.1
163.2
158.0
142.0
129.7
129.1
127.0
126.1
117.9
114.3
106.9
100.4
55.8
21.5




O
Cl
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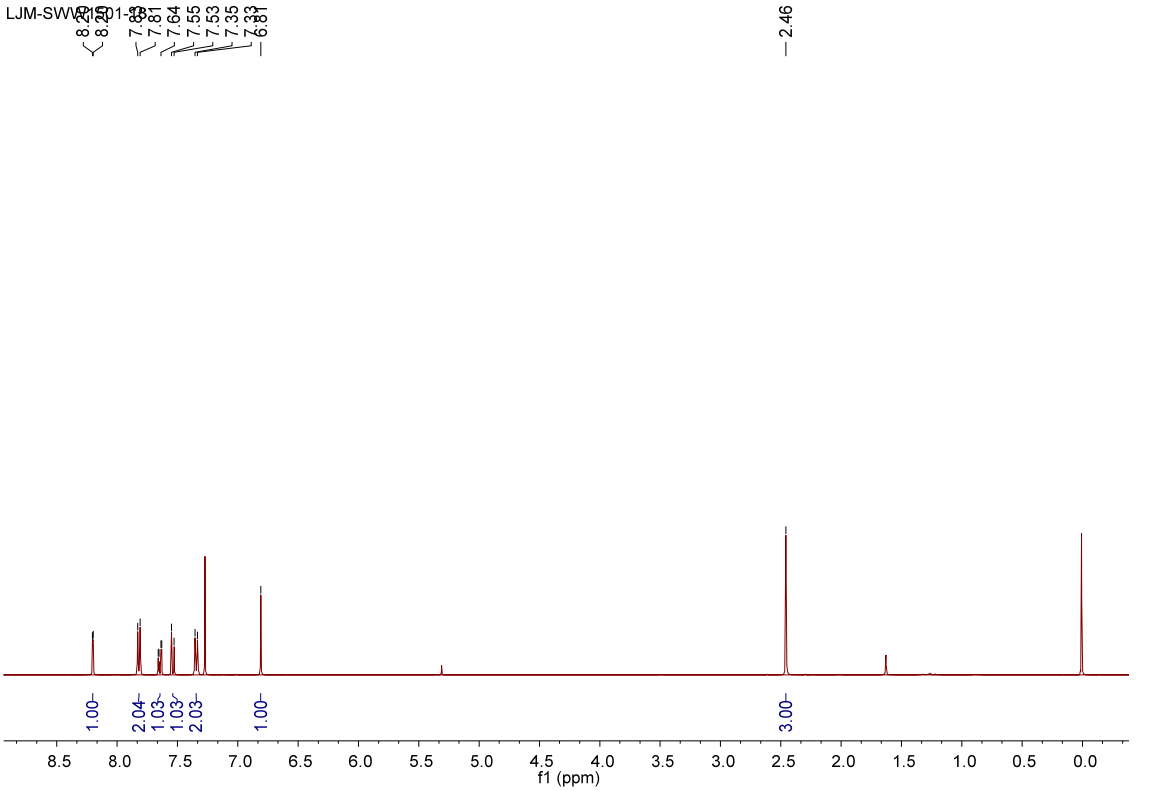
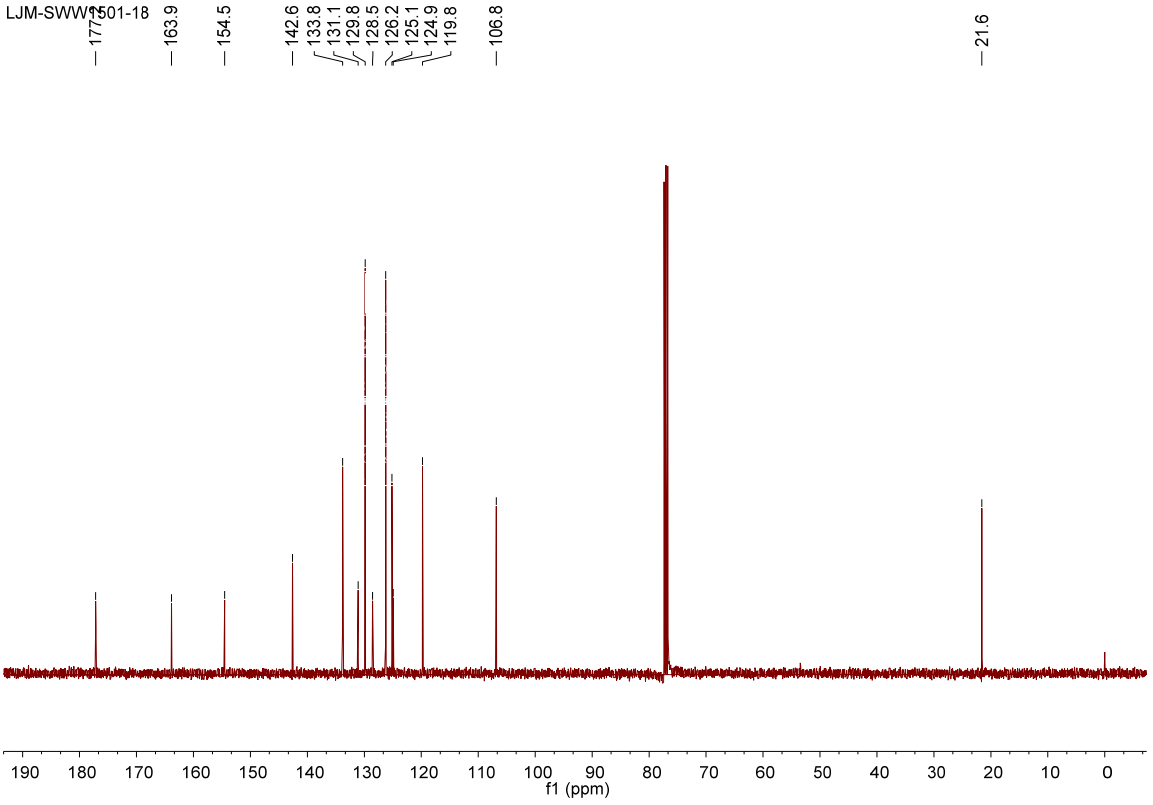
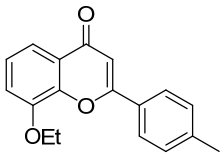

LJM-SWW-1501-18
8.21
8.20
7.83
7.81
7.64
7.55
7.53
7.35
7.33
6.81
2.46
1.00
2.04
1.03
1.03
2.03
1.00
3.00
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


LJM-SWW-1501-18
177.2
163.9
154.5
142.6
133.8
131.1
129.8
128.5
126.2
125.1
124.9
119.8
106.8
21.6
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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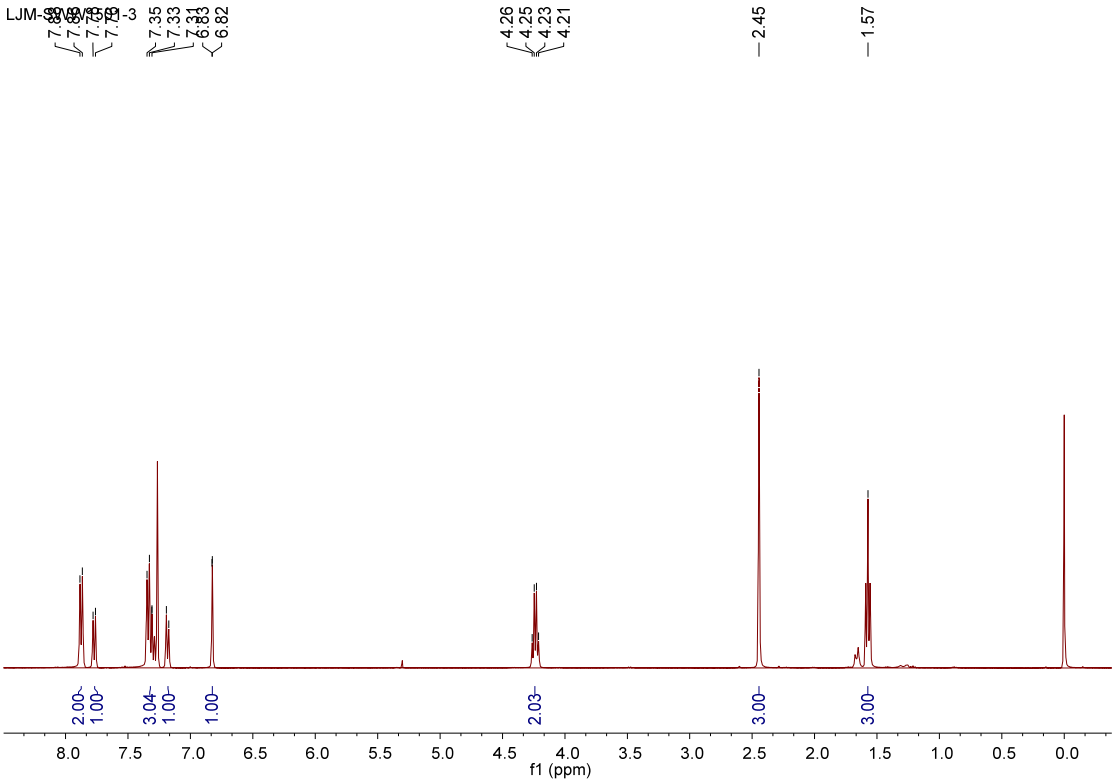
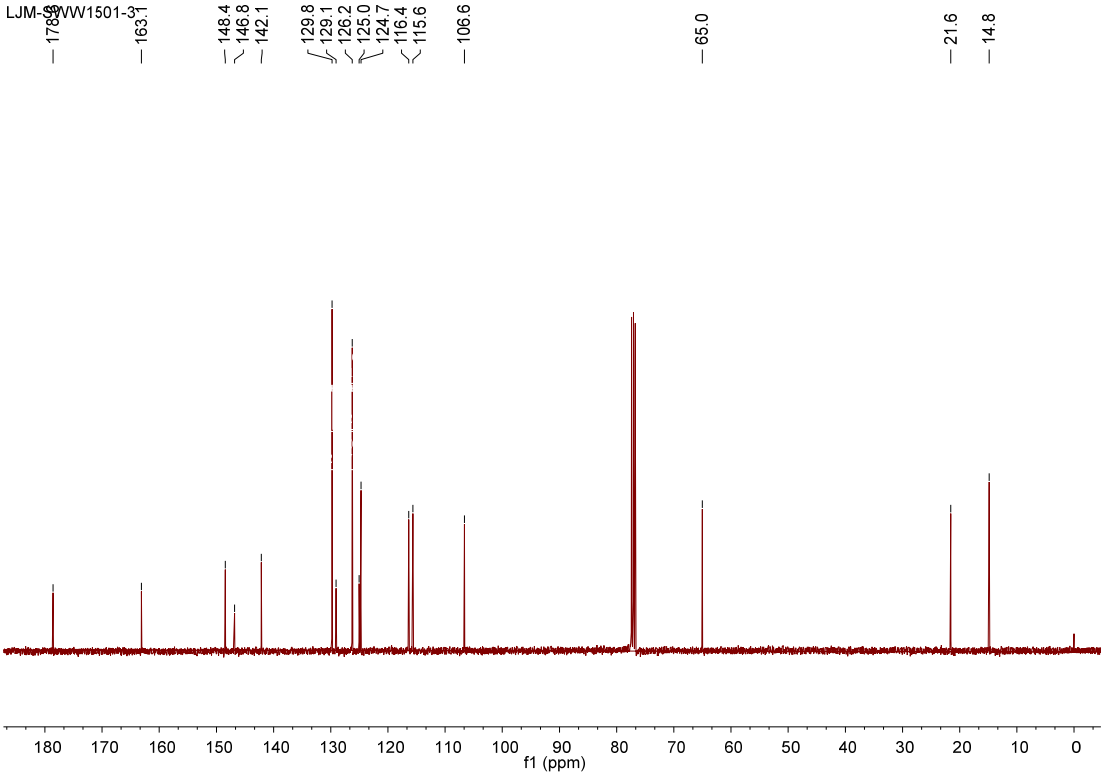
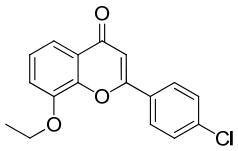
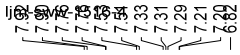

LJM-SWW1501-3
7.35
7.33
7.31
6.83
6.82
4.26
4.25
4.23
4.21
2.45
1.57
2.00
1.00
3.04
1.00
1.00
2.03
3.00
3.00
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

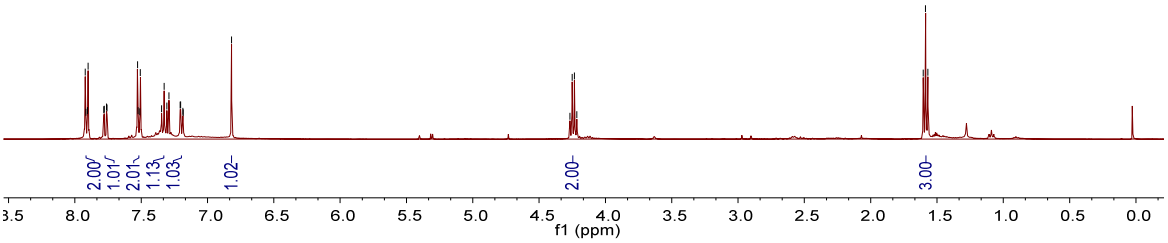

LJM-SWW1501-3
178.9
163.1
148.4
146.8
142.1
129.8
129.1
126.2
125.0
124.7
116.4
115.6
106.6
65.0
21.6
14.8
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


O
O
O
Cl

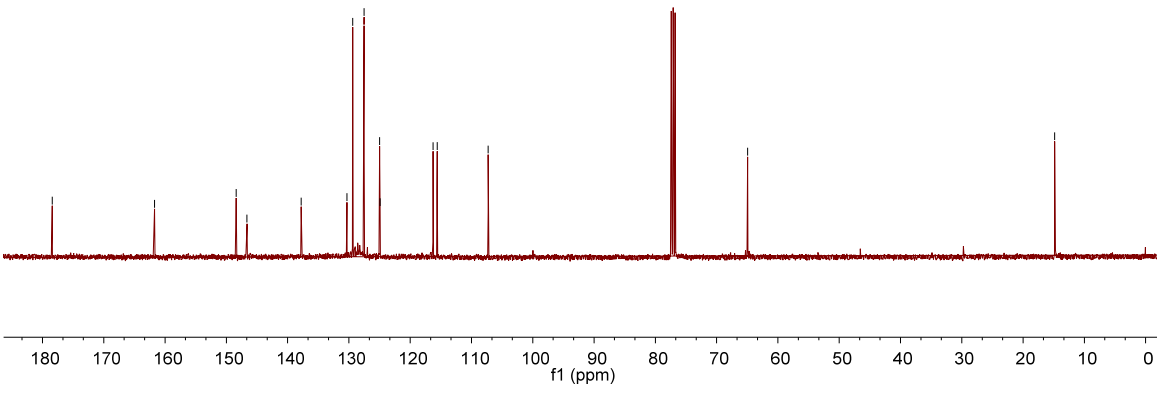

7.82
7.33
7.31
7.29
7.21
7.20
6.82

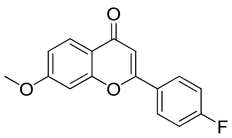

4.27
4.25
4.23
4.22

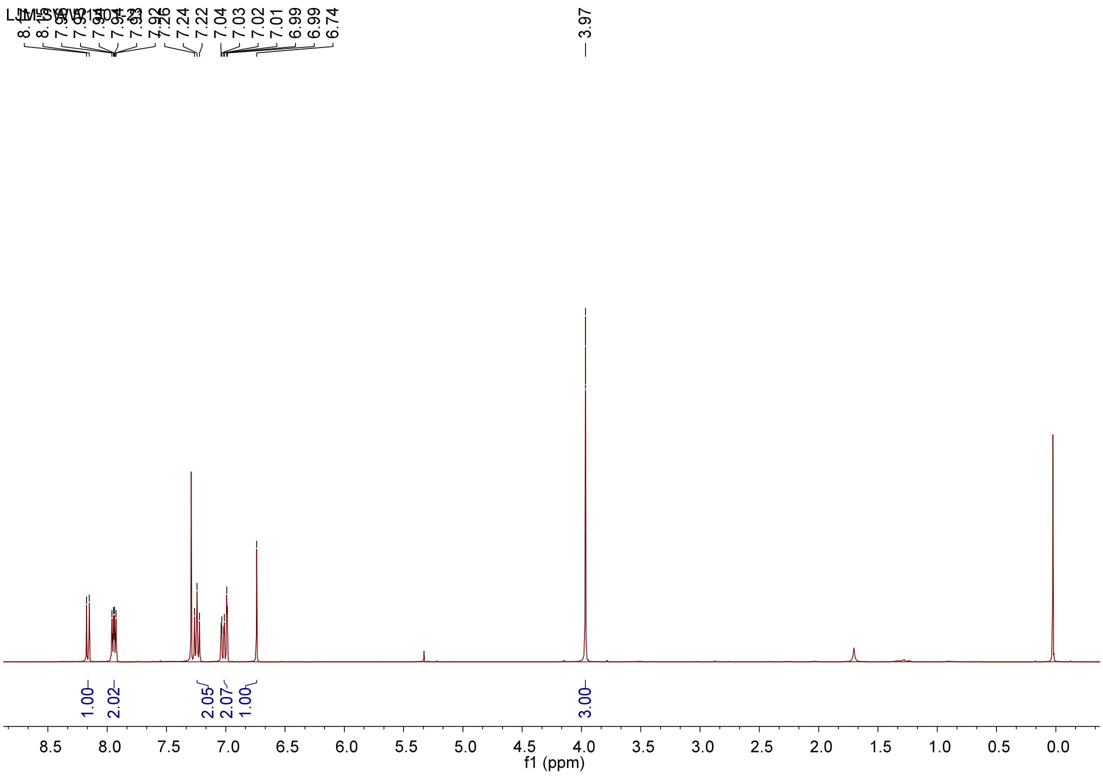

1.60
1.59
1.57

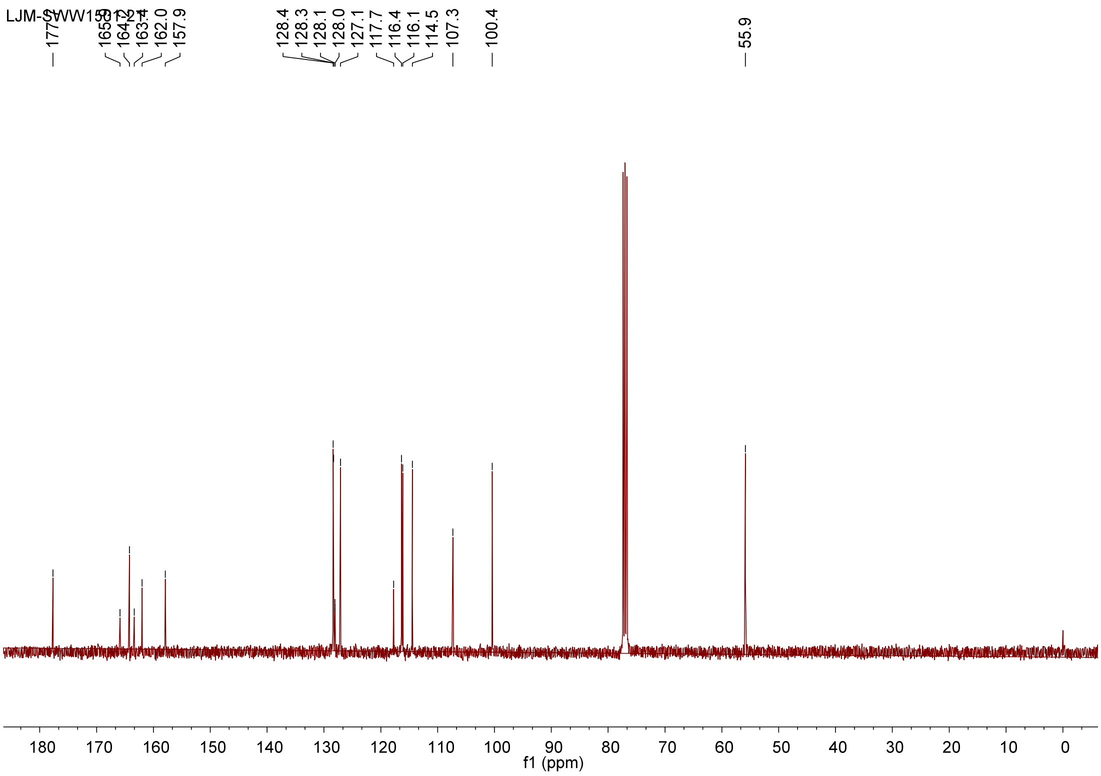

2.00
1.01
2.01
1.13
1.03
1.02
2.00
3.00
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

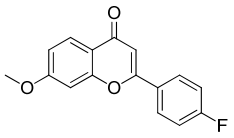

ljm-sww-516-4
178.4
161.7
148.4
146.6
137.8
130.3
129.4
127.5
125.0
124.9
116.3
115.6
107.3
64.9
14.8


180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

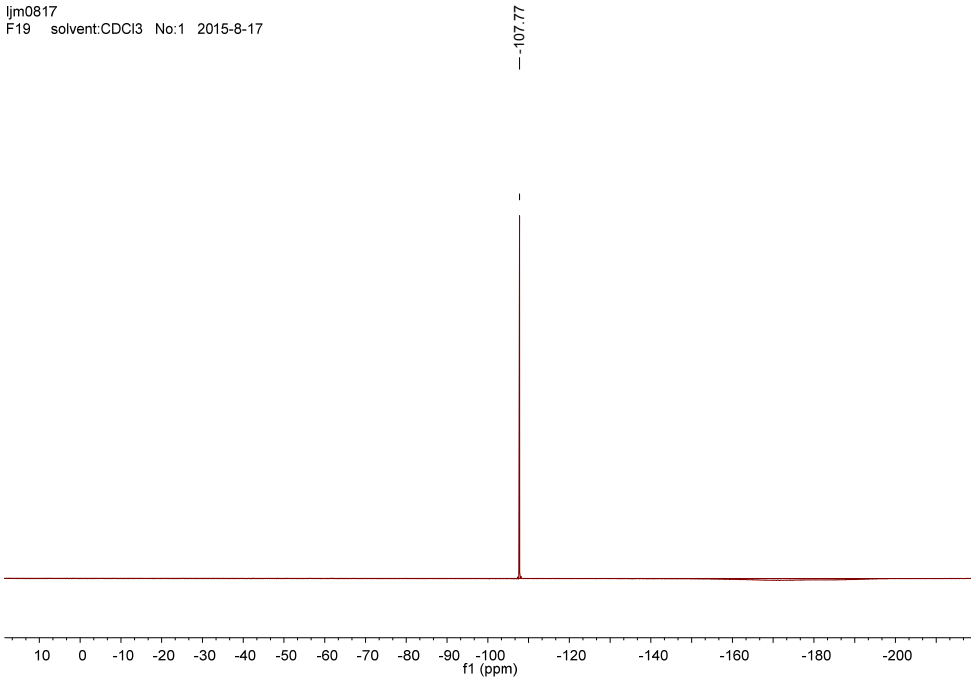

O
O
O
F

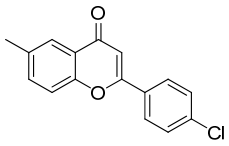

7.26
7.24
7.22
7.04
7.03
7.02
7.01
6.99
6.99
6.74
3.97
1.00
2.02
2.05
2.07
1.00
3.00
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

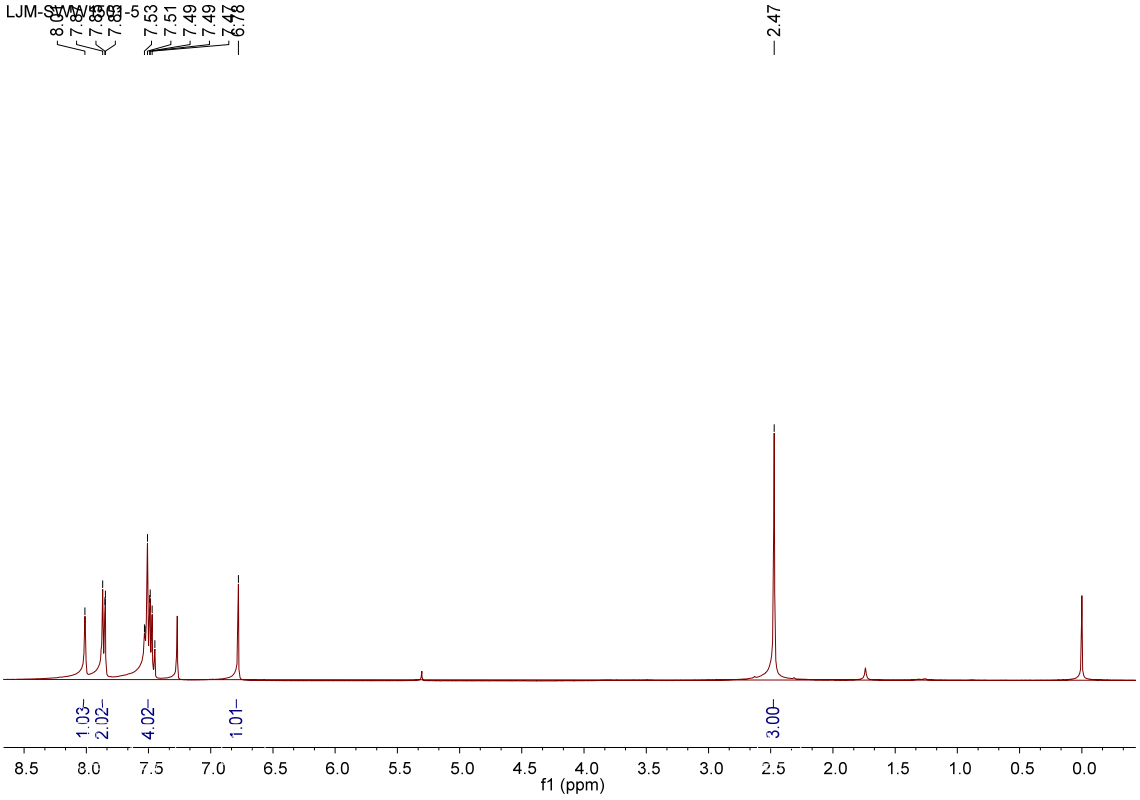

162.0
157.9
128.4
128.3
128.1
128.0
127.1
117.7
116.4
116.1
114.5
107.3
100.4
55.9
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

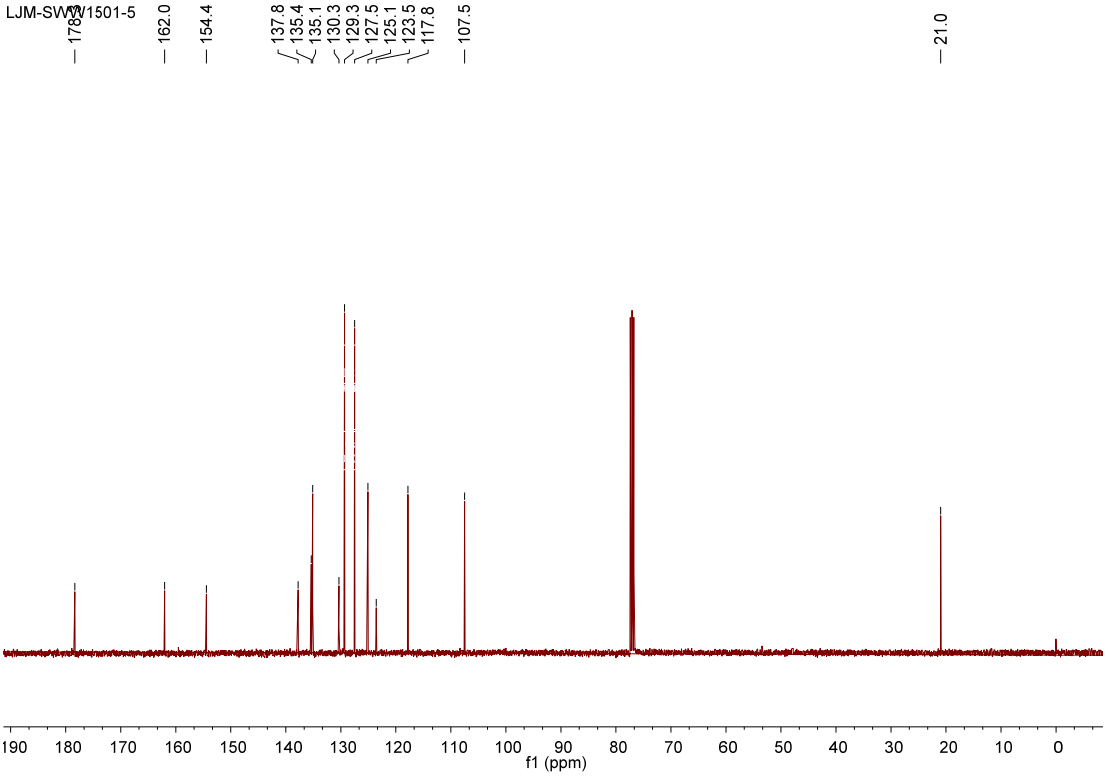


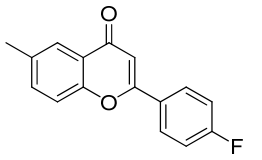
ljm0817
F19 solvent:CDCl3 No:1 2015-8-17

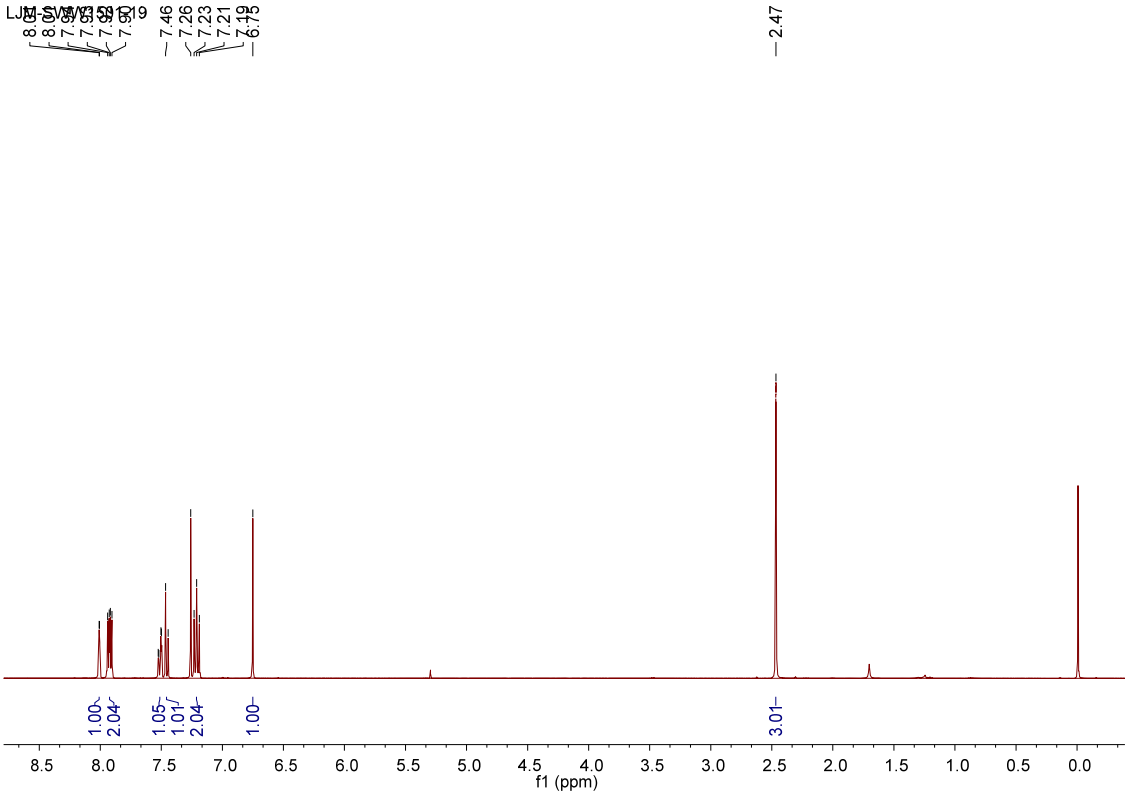


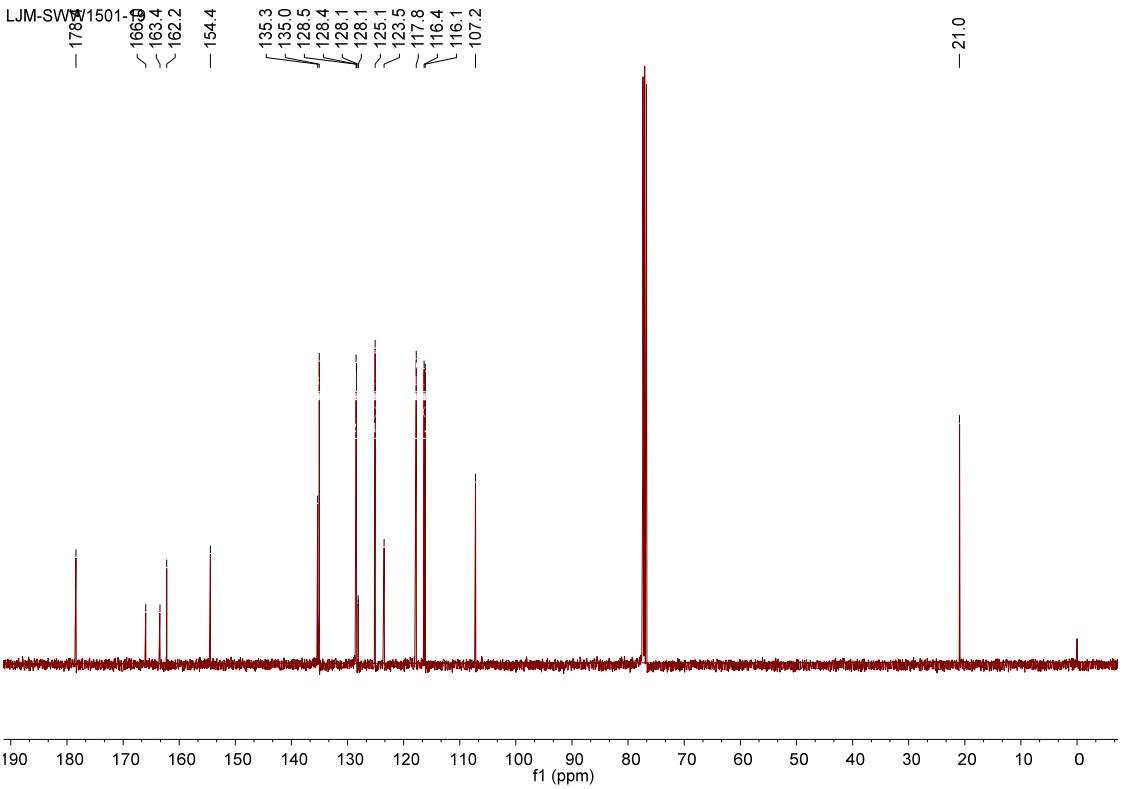

O
O
Cl

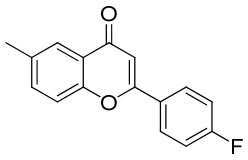

LJM-SWW1501-5
8.04
7.85
7.83
7.53
7.51
7.49
7.49
7.47
6.78
2.47
1.03
2.02
4.02
1.01
3.00
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


LJM-SWW1501-5
178.3
162.0
154.4
137.8
135.4
135.1
130.3
129.3
127.5
125.1
123.5
117.8
107.5
21.0
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

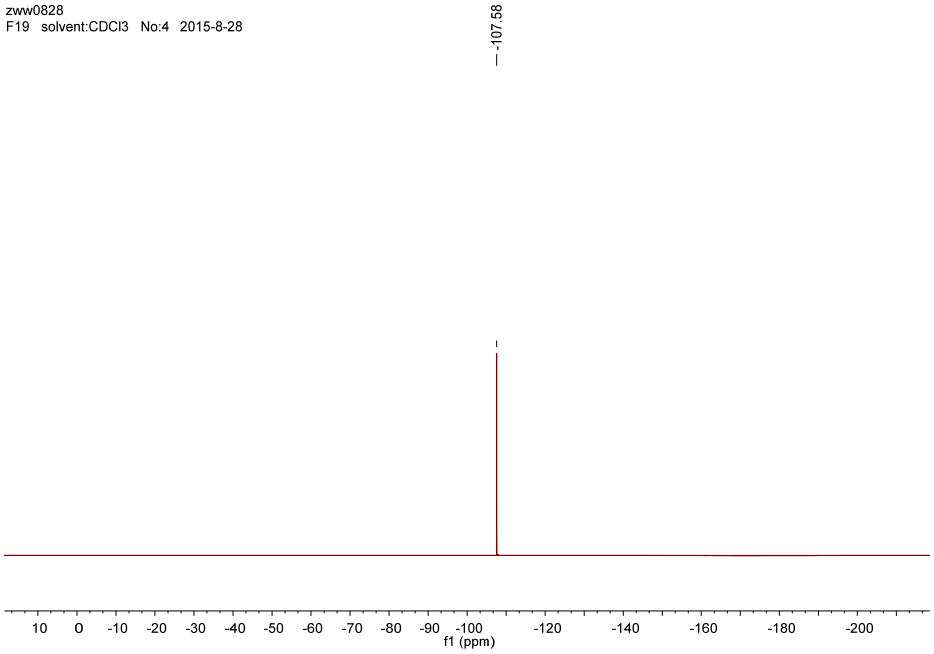

O
O
F

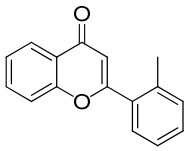

7.46
7.26
7.23
7.21
2.47
1.00
2.04
1.05
1.01
2.04
1.00
3.01
f1 (ppm)

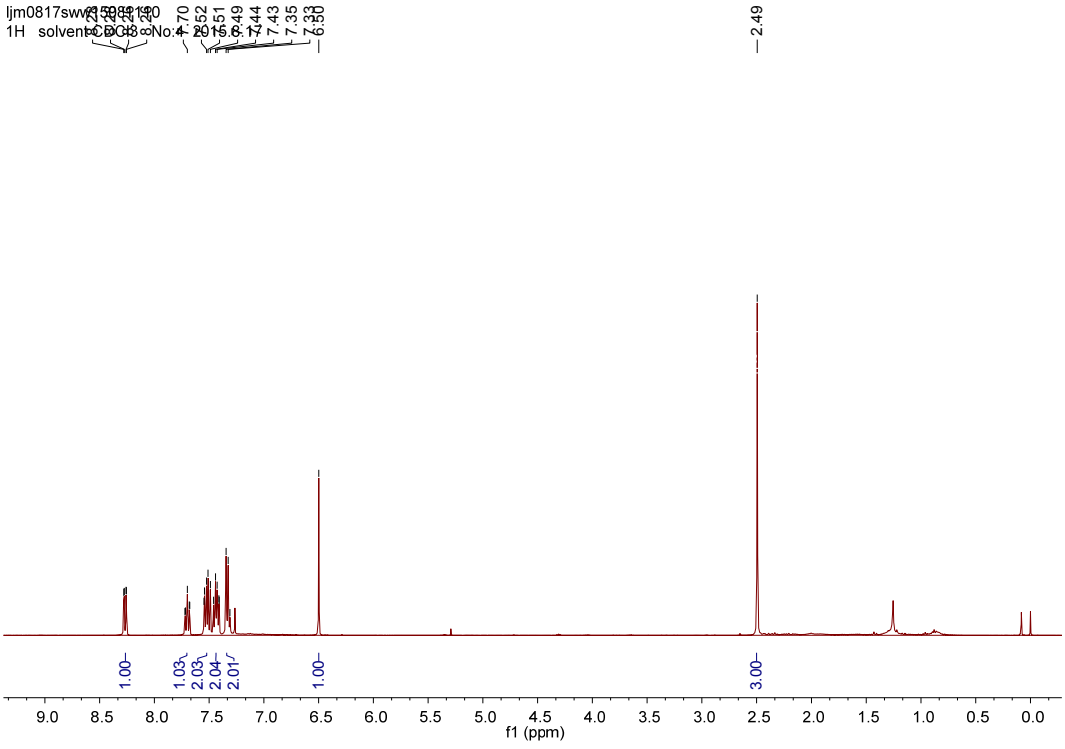

178.4
166.9
163.4
162.2
154.4
135.3
135.0
128.5
128.4
128.1
128.1
125.1
123.5
117.8
116.4
116.1
107.2
21.0
f1 (ppm)

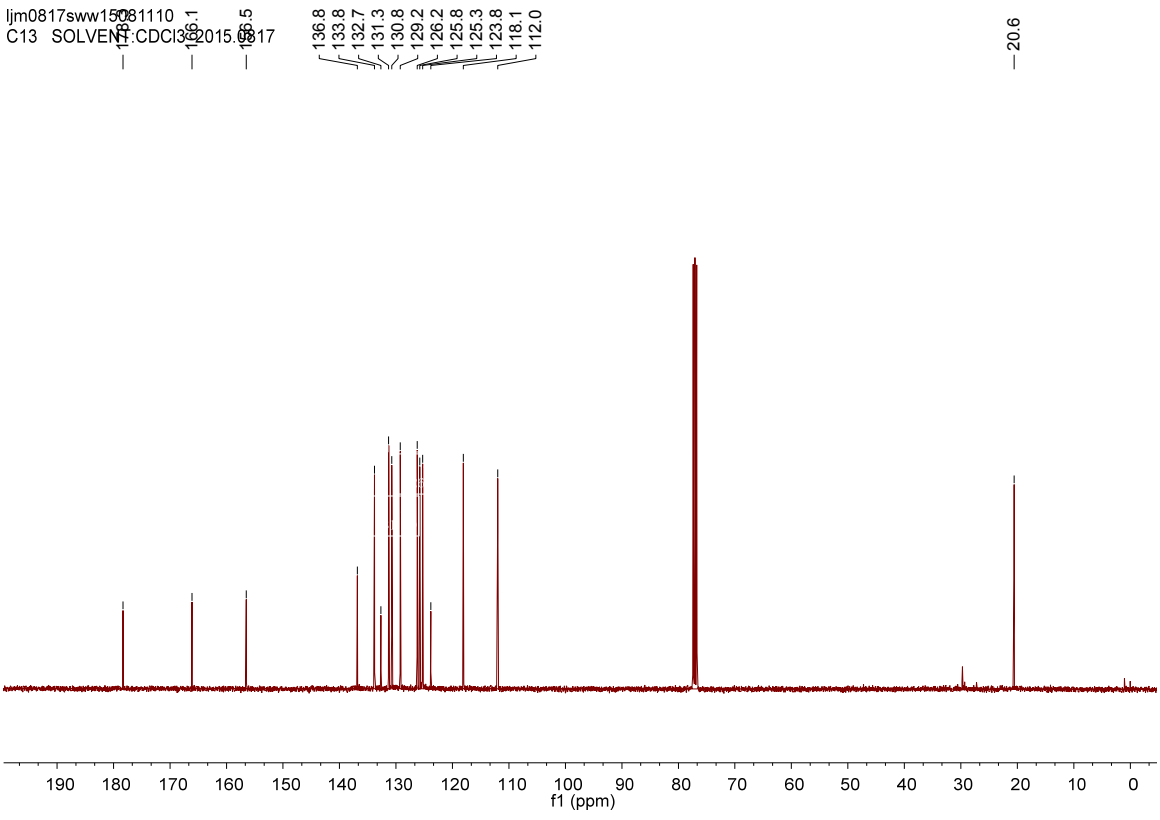


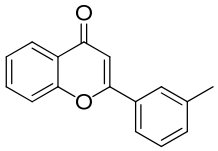
zww0828
F19 solvent:CDCl3 No:4 2015-8-28

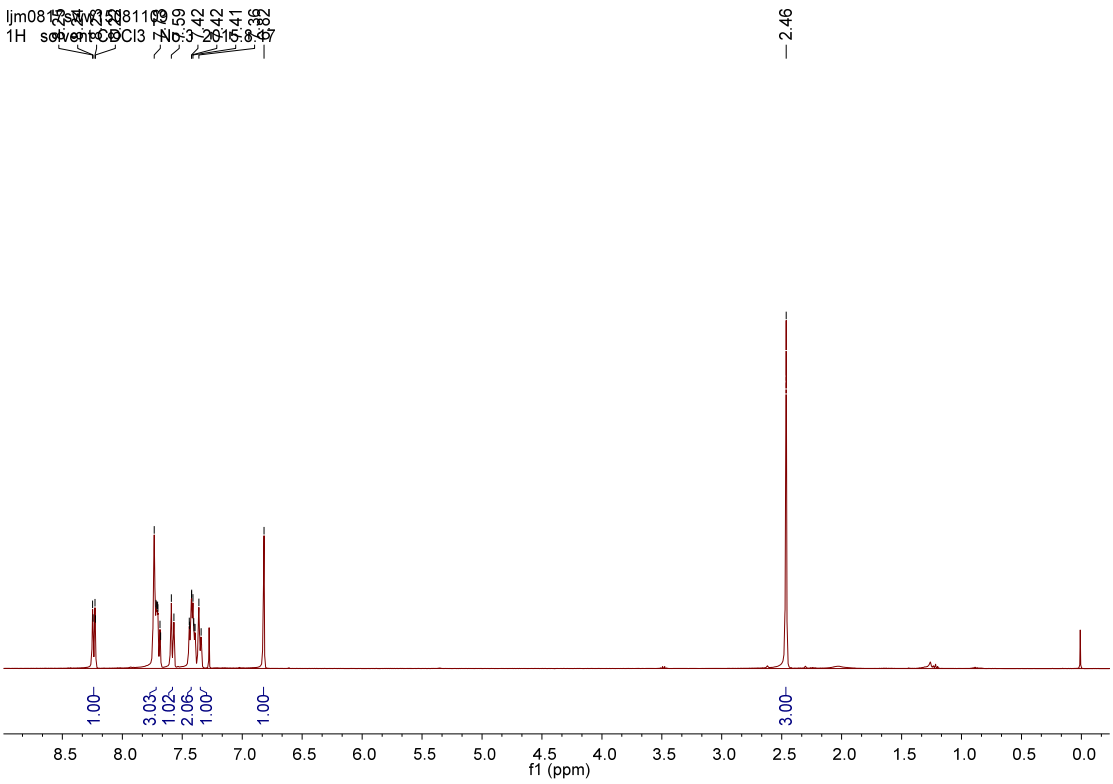


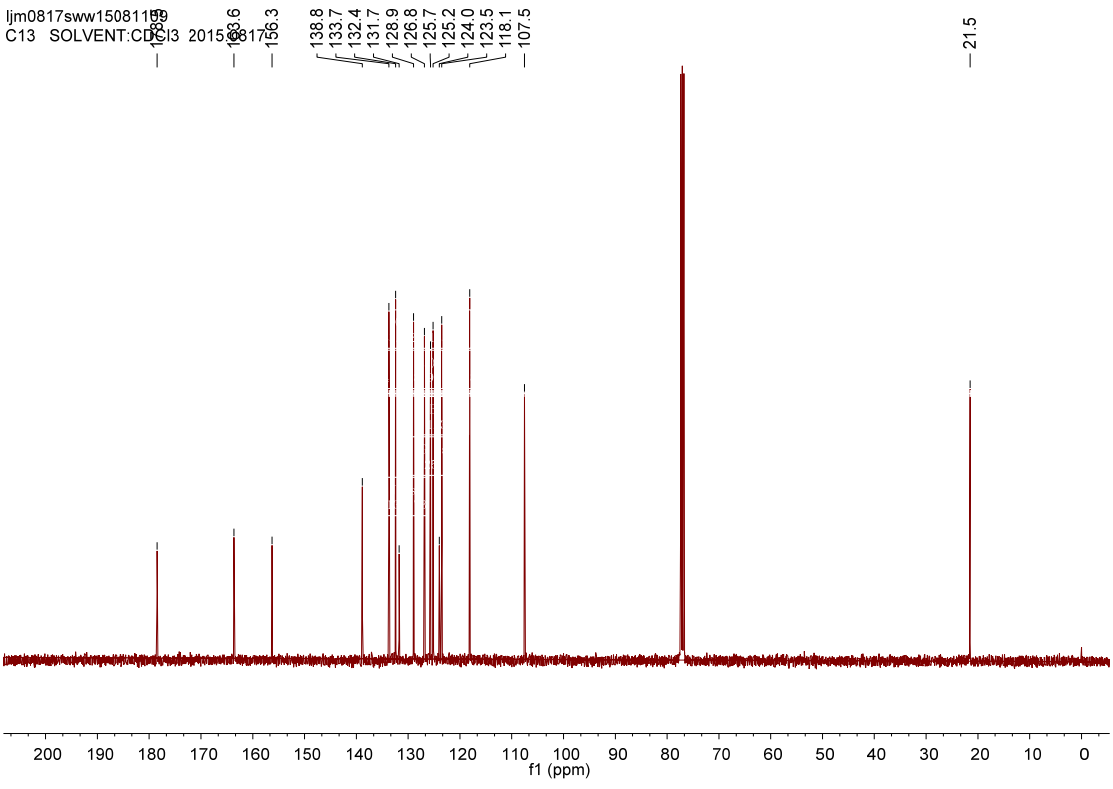


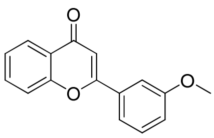

ljm0817sww15081110
1H solvent CDCl3 c No:4 2015.6.1
7.70
7.52
7.51
7.49
7.44
7.43
7.35
7.33
6.50
2.49
1.00
1.03
2.03
2.04
2.01
1.00
3.00
f1 (ppm)

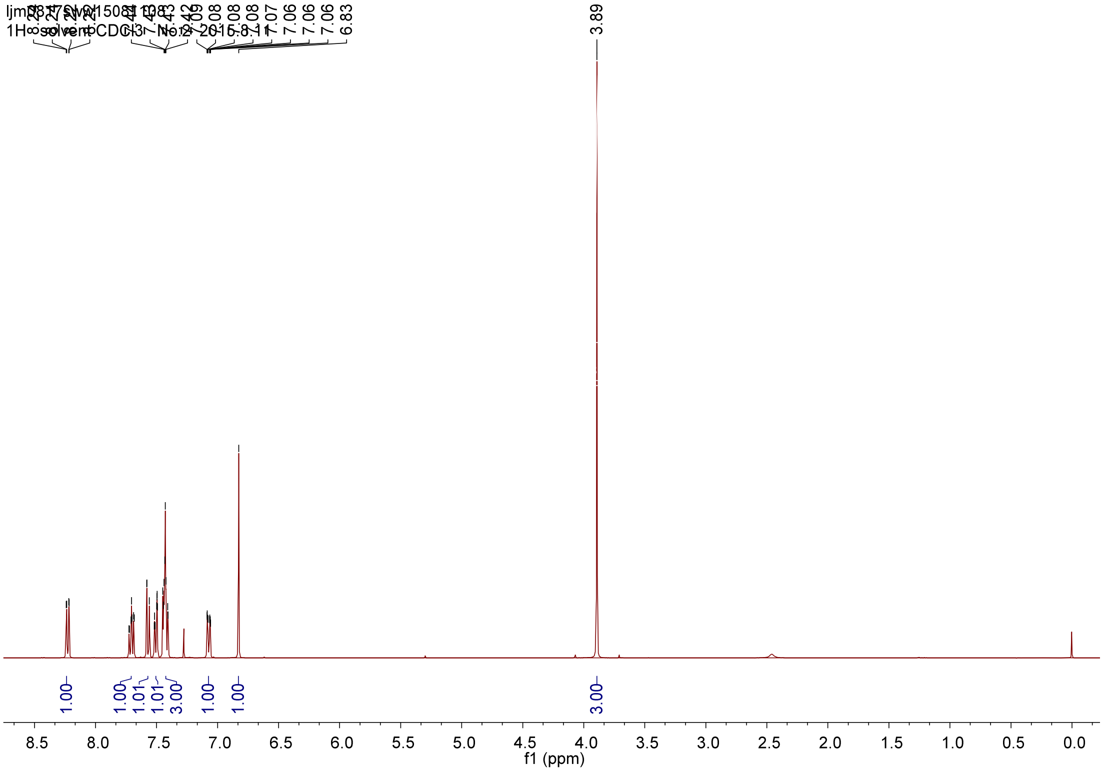

ljm0817sww15081110
C13 SOLVENT:CDCl3 2015.6.1
178.3
156.1
136.8
133.8
132.7
131.3
130.8
129.2
126.2
125.8
125.3
123.8
118.1
112.0
20.6
f1 (ppm)



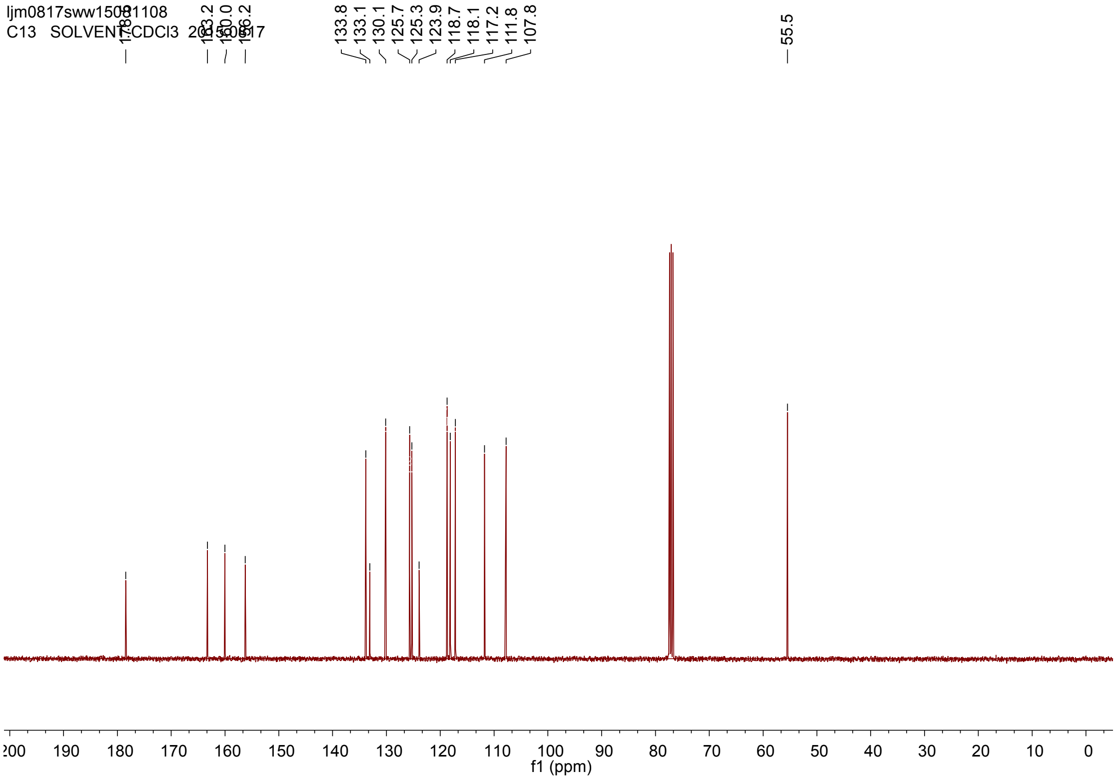

2.46
1.00
3.03
1.02
2.06
1.00
1.00
3.00
f1 (ppm)


C13 SOLVENT:CDCl3
159.9
153.6
156.3
138.8
133.7
132.4
131.7
128.9
126.8
125.7
125.2
124.0
123.5
118.1
107.5
21.5
f1 (ppm)

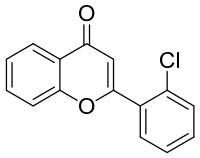


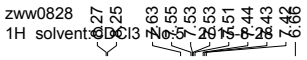


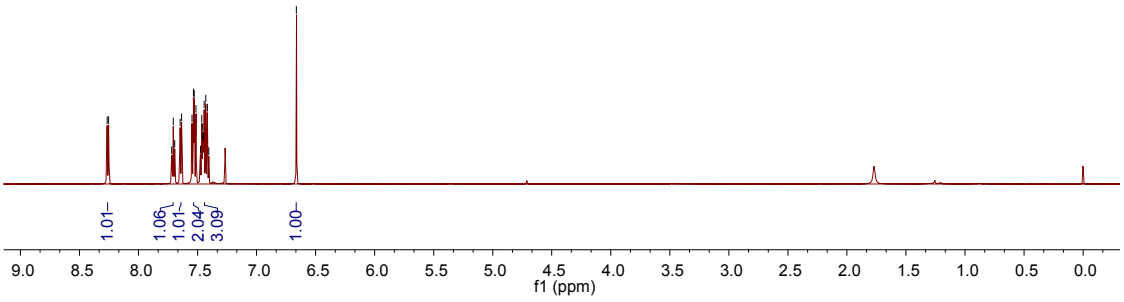
3.89
1.00
1.00
1.01
1.01
3.00
1.00
1.00
3.00
f1 (ppm)

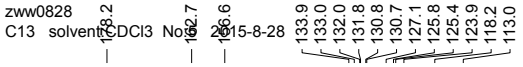


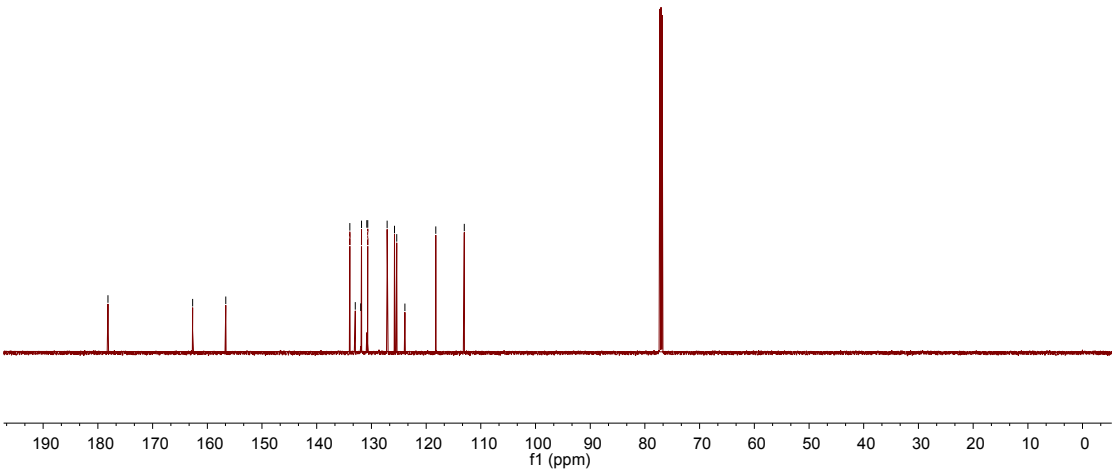
ljm0817sww15081108
C13 SOLVENT CDCl3
133.8
133.1
130.1
125.7
125.3
123.9
118.7
118.1
117.2
111.8
107.8
55.5
f1 (ppm)

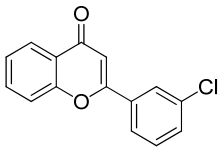

O
Cl
O

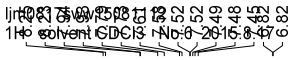

zww0828
1H solvent:CDCl3 No.5 7-2015-8-28
8.27
8.25
7.63
7.55
7.53
7.53
7.51
7.44
7.43
7.42
7.42
6.66

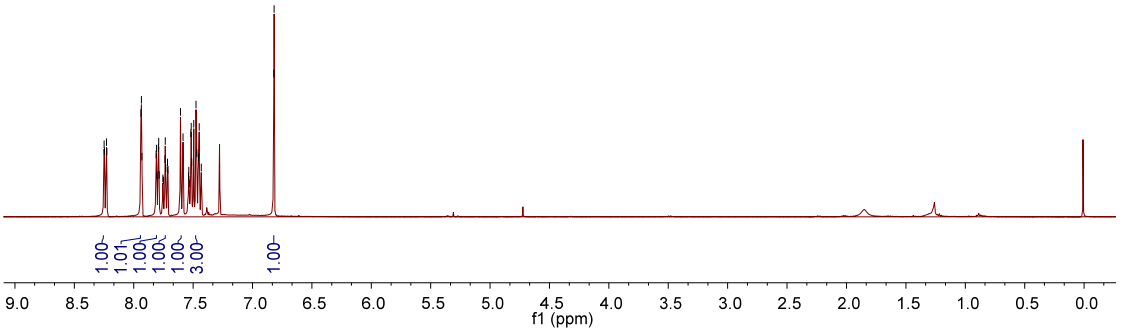

1.01
1.06
1.01
2.04
3.09
1.00
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

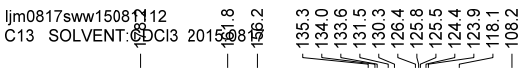

zww0828
C13 solvent:CDCl3 No.7 2015-8-28
178.2
162.7
156.6
133.9
133.0
132.0
131.8
130.8
130.7
127.1
125.8
125.4
123.9
118.2
113.0

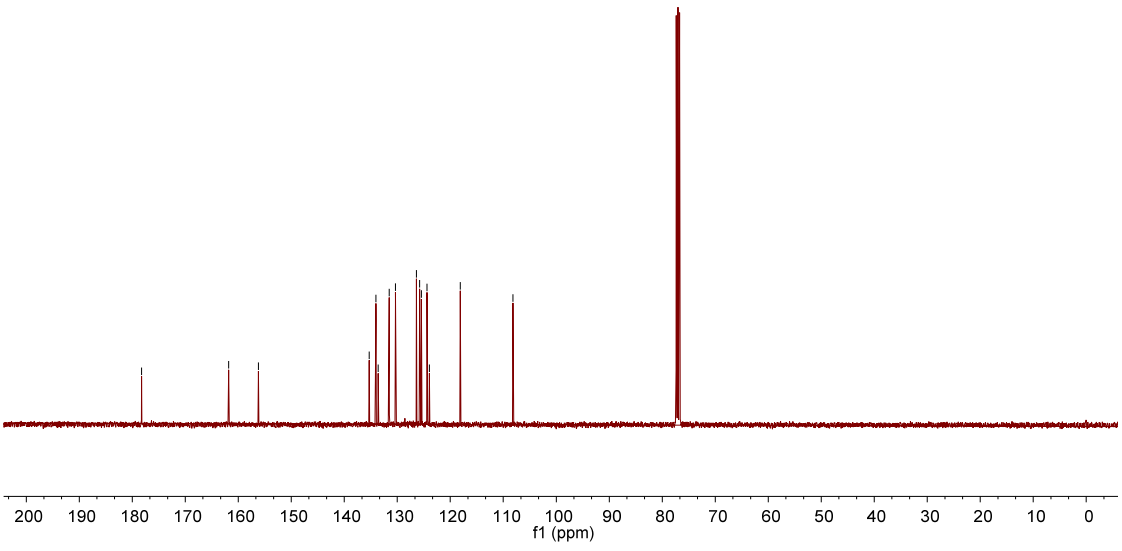

190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


O
O
Cl


1H solvent CDCl3


1.00
1.01
1.00
1.00
1.00
3.00
1.00
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


C13 SOLVENT:CDCl3
135.3
134.0
133.6
131.5
130.3
126.4
125.8
125.5
124.4
123.9
118.1
108.2


200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)