

Visible-light-induced cascade chromone cyclization/chalcogenation to access 3-chalcogenyl-chromones using elemental sulfur/selenium

Tao Guo,^a Chuanhu Gao,^a Zhonghui Li,^a Penghua Hu,^a Huan Chen,^b
Shulei Han,^{*b} Yunhui Zhao,^{*c} and Congjun Zhu^{*a}

^a *School of Chemistry and Chemical Engineering, Henan University of Technology,
Zhengzhou, Henan 450001, PR China.*

^b *China National Tobacco Quality Supervision and Test Center, Key Laboratory of Tobacco
Biological Effects, Zhengzhou, 450001, China*

^c *School of Chemistry and Chemical Engineering, Hunan University of Science and
Technology, Xiangtan 411201, PR China*

Supplementary Information

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1 General experimental

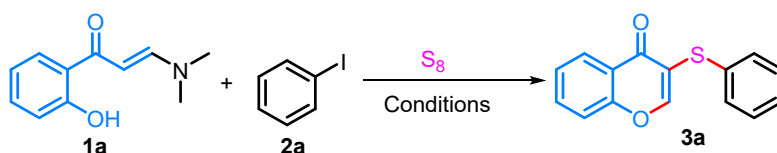
Unless otherwise mentioned, all materials were commercially obtained and used without further purification. Enaminones (**1**)¹ were synthesized according to previously described methods. The ¹H NMR, ¹³C NMR, and ¹⁹F NMR spectra were recorded at 500 MHz, 126 MHz, 471 MHz, respectively, on a Bruker AM500 MHz with chemical shift values in ppm relative to TMS (δ H 0.00 and δ C 0.0) as internal standard. The coupling constants *J*, are reported in Hertz (Hz). All melting points were determined on a SGW X-4A melting point instrument without correction. High-resolution mass spectra (HRMS) were recorded on Q-Exactive plus Orbitrap (ESI) or HP-5989A instrument.. Infrared spectra (IR) were recorded on Spectrum TWO. Reactions were monitored by thin layer chromatography (TLC), on glass plates coated with silica gel with Fluorescent indicator (Huanghai, HSGF254) and visualized with UV light at 254 nm. Flash chromatography was performed on silica gel (Huanghai, 300-400) using petroleum ether (PE)-ethyl acetate (EA) as eluent.

2 The reaction equipment of visible light catalysis

We use RLH-18 8-position Photo Reaction System, which manufactured by Beijing Rogertech Co.ltd base in Beijing PRC. This Photo reactor we used have equipped 8 blue light 10W LED. This blue light 10 WLED's energy peak wavelength is 454.9 nm, peak width at half-height is 22.9 nm, irradiance@10 W is 172 mW/cm². Irradiation vessel is borosilicate glass test tube, LED irradiate through a high-reflection channel to the test tube, path length is 2 cm. No filter between LED and test tube.

3 Optimization of the reaction conditions

Table S1. Optimization of the sulfenylation conditions^a

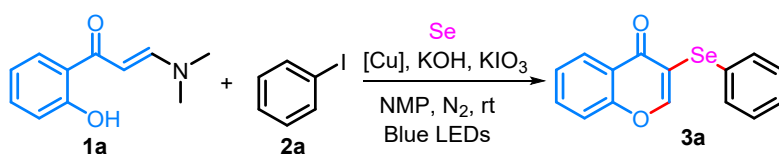


Entry	Catalyst (10 mol%)	Oxidant (2 equiv.)	Base (equiv.)	Solvent (mL)	Yield ^b (%)
1	CuI	KIO ₃	Cs ₂ CO ₃ (3)	DMF (1)	40
2	CuI	KIO ₃	^t BuOK (3)	DMF (1)	61
3	CuI	KIO ₃	K ₂ CO ₃ (3)	DMF (1)	48
4	CuI	KIO ₃	KOH (3)	DMF (1)	65
5	CuI	KIO ₃	KOH (2)	DMF (1)	56
6	CuI	KIO ₃	KOH (3)	DMSO (1)	61
7	CuI	KIO ₃	KOH (3)	DCM (1)	0
8	CuI	KIO ₃	KOH (3)	NMP (1)	74

9	CuI	KIO ₃	KOH (3)	CH ₃ CN (1)	65
10	CuI	KIO ₃	KOH (3)	H ₂ O (1)	0
11	--	KIO ₃	KOH (3)	NMP (1)	0
12	CuI	--	KOH (3)	NMP (1)	Trace
13	Cu(OAc) ₂	KIO ₃	KOH (3)	NMP (1)	52
14	CuBr ₂	KIO ₃	KOH (3)	NMP (1)	64
15	Cu ₂ S	KIO ₃	KOH (3)	NMP (1)	83
16	CuBr	KIO ₃	KOH (3)	NMP (1)	53
17	CuCl	KIO ₃	KOH (3)	NMP (1)	47
18	FeCl ₃	KIO ₃	KOH (3)	NMP (1)	0
19	CoCl ₂	KIO ₃	KOH (3)	NMP (1)	0
20	Cu ₂ S	KMnO ₄	KOH (3)	NMP (1)	59
21	Cu ₂ S	TBHP	KOH (3)	NMP (1)	51
22	Cu ₂ S	KIO ₃	KOH (3)	NMP (2)	76
23	Cu ₂ S	KIO ₃	KOH (3)	NMP (0.5)	62
24 ^c	Cu ₂ S	KIO ₃	KOH (3)	NMP (1)	Trace
25 ^d	Cu ₂ S	KIO ₃	KOH (3)	NMP (1)	43
26 ^e	Cu ₂ S	KIO ₃	KOH (3)	NMP (1)	68

^a Reaction conditions: **1a** (0.2 mmol), S₈ (0.6 mmol), **2a** (0.6 mmol), catalyst (0.1 equiv.), oxidant (2 equiv.), base, solvent (1 mL), N₂ atmosphere, Blue LEDs (10 W, λ_{max} = 455 nm), 24 h. ^b Isolated yield. ^c Under air. ^d Under O₂. ^e KIO₃ (1.2 equiv.) was used.

Table S2. Optimization of the selenation conditions^a

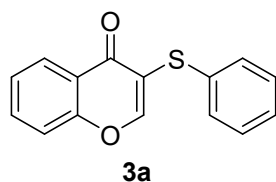


Entry	Catalyst (10 mol%)	Yield ^b (%)
1	Cu(OAc) ₂	trace
2	CuBr ₂	32
3	Cu ₂ S	70
4	CuI	75
5	CuBr	45
6	CuCl	29

^a Reaction conditions: **1a** (0.2 mmol), Se (0.6 mmol), **2a** (0.6 mmol), catalyst (0.1 equiv.), KIO₃ (2 equiv.), KOH (3 equiv.), NMP (1 mL), N₂ atmosphere, Blue LEDs (10 W, λ_{max} = 455 nm), 24 h. ^b Isolated yield.

4 General procedure for the synthesis of 3-chalcogenyl-chromones 3 and 5

Under nitrogen, 1 mL NMP, **1** (0.2 mmol), **2** or **4** (0.6 mmol), S₈ (0.6 mmol, 19.2 mg)/Cu₂S (0.02 mmol, 3.2 mg) or Se (0.6 mmol, 47.3 mg)/CuI (0.02 mmol, 3.8 mg), KOH (0.6 mmol, 33.7 mg), and KIO₃ (0.4 mmol, 85.6 mg) were added into the dry thick-walled glass pressure tube. The mixture was stirred at room temperature under 10W blue LEDs. After the reaction was complete, the mixture was diluted with 20 mL ethyl acetate and washed with 10 mL H₂O. The aqueous layer was extracted twice with ethyl acetate (5 mL) and the combined organic phase was dried over Na₂SO₄. After evaporation of the solvents the residue was purified by flash column chromatography (silica gel, PE–EA) to afford the desired products **3** and **5**.



3-(phenylthio)-4H-chromen-4-one²

Purified by flash chromatograph column (PE : EtOAc = 15 : 1).

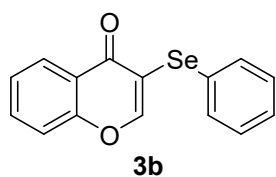
Yellow solid (42.0 mg, 83% yield); mp 103-104 °C.

¹H NMR (500 MHz, CDCl₃) δ 8.24 (dd, *J* = 8.0, 1.5 Hz, 1H), 8.14 (s, 1H), 7.70 – 7.67 (m, 1H), 7.47 – 7.39 (m, 4H), 7.28 (t, *J* = 7.5 Hz, 2H), 7.21 (t, *J* = 7.5 Hz, 1H).

¹³C NMR (126 MHz, CDCl₃) δ 175.2, 157.5, 156.4, 134.1, 129.9, 129.3, 127.2, 126.5, 125.8, 123.7, 120.0, 118.3.

IR (KBr) 2922, 1643, 1609, 1458, 1305, 1083, 763, 732, 686 cm⁻¹.

HRMS for C₁₅H₁₁O₂S⁺(M⁺⁺H): calcd. 255.04743, found 255.04776.



3-(phenylselanyl)-4H-chromen-4-one³

Purified by flash chromatograph column (PE : EtOAc = 10 : 1).

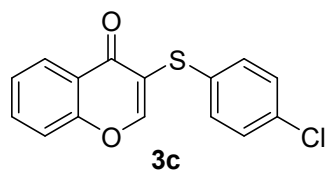
Yellow solid (45.3 mg, 75% yield); mp 69-70 °C.

¹H NMR (500 MHz, CDCl₃) δ 8.22 (d, *J* = 8.0 Hz, 1H), 7.88 (s, 1H), 7.67 – 7.63 (m, 1H), 7.60 – 7.58 (m, 2H), 7.42 – 7.38 (m, 2H), 7.30 – 7.27 (m, 3H).

¹³C NMR (126 MHz, CDCl₃) δ 175.2, 156.4, 155.8, 133.9, 133.9, 129.6, 128.2, 126.3, 125.6, 123.2, 118.1, 117.9.

IR (KBr) 2921, 1609, 1461, 1308, 1067, 876, 755, 688 cm⁻¹.

HRMS for $C_{15}H_{11}O_2Se^+(M^+H)$: calcd. 302.99188, found 302.99176.



3-((4-chlorophenyl)thio)-4H-chromen-4-one²

Purified by flash chromatograph column (PE : EtOAc = 15 : 1).

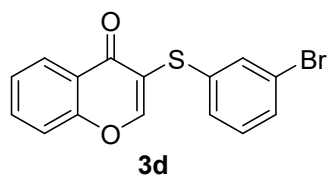
Yellow solid (48.7 mg, 85% yield), mp 164-166 °C.

¹H NMR (500 MHz, CDCl₃) δ 8.24 – 8.22 (m, 2H), 7.72 – 7.69 (m, 1H), 7.48 (d, *J* = 8.0 Hz, 1H), 7.44 (d, *J* = 7.5 Hz, 1H), 7.32 (d, *J* = 8.5 Hz, 2H), 7.26 – 7.23 (m, 2H).

¹³C NMR (126 MHz, CDCl₃) δ 175.1, 158.0, 156.5, 134.3, 133.2, 132.9, 131.0, 129.4, 126.6, 126.0, 123.8, 119.3, 118.3.

IR (KBr) 3050, 1636, 1464, 1311, 1087, 756 cm⁻¹.

HRMS for $C_{15}H_{10}ClO_2S^+(M^+H)$: calcd. 289.00845, found 289.00894.



3-((3-bromophenyl)thio)-4H-chromen-4-one

Purified by flash chromatograph column (PE : EtOAc = 15 : 1).

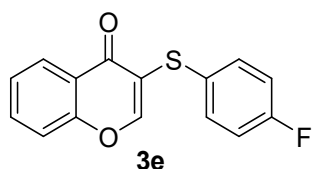
Yellow solid (52.9 mg, 80% yield); mp 118-120 °C.

¹H NMR (500 MHz, CDCl₃) δ 8.27 (s, 1H), 8.23 (dd, *J* = 8.0, 1.5 Hz, 1H), 7.73 – 7.69 (m, 1H), 7.50 – 7.48 (m, 1H), 7.46 – 7.42 (m, 2H), 7.31 – 7.27 (m, 2H), 7.11 (dd, *J* = 8.0, 1.5 Hz, 1H).

¹³C NMR (126 MHz, CDCl₃) δ 174.9, 158.7, 156.4, 136.9, 134.3, 131.3, 130.4, 129.9, 127.5, 126.5, 126.0, 123.8, 123.0, 118.4, 118.3.

IR (KBr) 2922, 1655, 1607, 1558, 1456, 1305, 1080, 764 cm⁻¹.

HRMS for $C_{15}H_{10}BrO_2S^+(M^+H)$: calcd. 332.95794, found 332.95856.



3-((4-fluorophenyl)thio)-4H-chromen-4-one⁶

Purified by flash chromatograph column (PE : EtOAc = 15 : 1).

Yellow solid (46.1 mg, 85% yield); mp 134-135 °C.

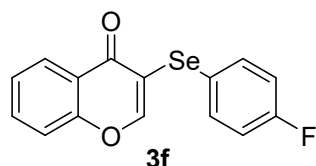
¹H NMR (500 MHz, CDCl₃) δ 8.23 (dd, *J* = 8.0, 1.5 Hz, 1H), 8.14 (s, 1H), 7.70 – 7.67 (m, 1H), 7.47 – 7.41 (m, 4H), 7.01 – 6.97 (m, 2H).

¹³C NMR (126 MHz, CDCl₃) δ 175.2, 162.4 (d, ¹*J*_{C-F} = 246.3 Hz), 157.1, 156.4, 134.2, 132.9 (d, ³*J*_{C-F} = 8.3 Hz), 128.9 (d, ⁴*J*_{C-F} = 3.3 Hz), 126.5, 125.9, 123.7, 120.6, 118.3, 116.4 (d, ²*J*_{C-F} = 22.5 Hz).

¹⁹F NMR (471 MHz, CDCl₃) δ -114.0.

IR (KBr) 2906, 1635, 1489, 1216, 1085, 756, 625 cm⁻¹.

HRMS for C₁₅H₁₀FO₂S⁺(M⁺+H): calcd.273.03801, found 273.03760.



3-((4-fluorophenyl)selanyl)-4H-chromen-4-one⁵

Purified by flash chromatograph column (PE : EtOAc = 10 : 1).

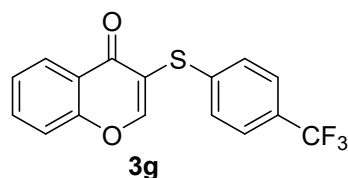
colorless oil (40.4 mg, 63% yield).

¹H NMR (500 MHz, CDCl₃) δ 8.23 (dd, *J* = 8.0, 1.0 Hz, 1H), 7.90 (s, 1H), 7.69 – 7.61 (m, 3H), 7.42 (t, *J* = 8.5 Hz, 2H), 7.02 – 6.99 (m, 2H).

¹³C NMR (126 MHz, CDCl₃) δ 175.3, 163.1 (d, ¹*J*_{C-F} = 246.4 Hz), 156.5, 155.7, 136.5 (d, ³*J*_{C-F} = 7.9 Hz), 134.0, 126.4, 125.7, 123.2, 122.6 (d, ⁴*J*_{C-F} = 3.4 Hz), 118.2, 118.1, 116.8 (d, ²*J*_{C-F} = 21.4 Hz).

IR (KBr) 2972, 1631, 1462, 1214, 1047, 755 cm⁻¹.

HRMS for C₁₅H₁₀FO₂Se⁺(M⁺+H): calcd. 320.98246, found 320.98242.



3-((4-(trifluoromethyl)phenyl)thio)-4H-chromen-4-one

Purified by flash chromatograph column (PE : EtOAc = 15 : 1).

Yellow solid (40.5 mg, 63% yield); mp 165-166 °C.

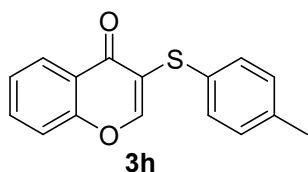
¹H NMR (500 MHz, CDCl₃) δ 8.29 (s, 1H), 8.18 (dd, *J* = 8.0, 1.5 Hz, 1H), 7.68 – 7.65 (m, 1H), 7.46 – 7.38 (m, 4H), 7.30 (d, *J* = 8.0 Hz, 2H).

¹³C NMR (126 MHz, CDCl₃) δ 174.9, 159.5, 156.5, 140.2, 134.5, 128.4 (q, *J* = 32.9 Hz), 127.7, 126.6, 126.2, 125.9 (q, *J* = 3.5 Hz), 124.1 (q, *J* = 272.7 Hz), 123.9, 118.4, 117.4.

¹⁹F NMR (471 MHz, CDCl₃) δ -62.5.

IR (KBr) 2920, 1643, 1463, 1324, 1105, 822, 753 cm⁻¹.

HRMS for C₁₆H₁₀F₃O₂S⁺(M⁺+H): calcd. 323.03481, found 323.03473.



3-(p-tolylthio)-4H-chromen-4-one²

Purified by flash chromatograph column (PE : EtOAc = 15 : 1).

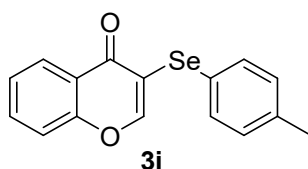
Yellow solid (47.9 mg, 89% yield); mp 106-108 °C.

¹H NMR (500 MHz, CDCl₃) δ 8.23 (d, *J* = 8.0 Hz, 1H), 8.03 (s, 1H), 7.68- 7.65 (m, 1H), 7.45 – 7.39 (m, 2H), 7.34 (d, *J* = 8.5 Hz, 2H), 7.10 (d, *J* = 8.0 Hz, 2H), 2.30 (s, 3H).

¹³C NMR (126 MHz, CDCl₃) δ 175.2, 156.4, 137.7, 134.0, 131.1, 130.1, 129.9, 126.4, 125.7, 123.6, 121.2, 118.2, 21.2.

IR (KBr) 2921, 1644, 1555, 1462, 1305, 1080, 755 cm⁻¹.

HRMS for C₁₆H₁₃O₂S⁺(M⁺+H): calcd. 269.06308, found 269.06351.



3-(p-tolylselanyl)-4H-chromen-4-one⁴

Purified by flash chromatograph column (PE : EtOAc = 5 : 1).

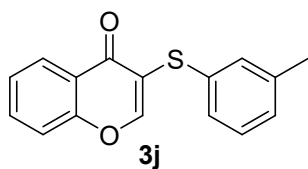
Yellow solid (46.6 mg, 74% yield); mp 90-92 °C.

¹H NMR (500 MHz, CDCl₃) δ 8.22 (dd, *J* = 8.5, 1.5 Hz, 1H), 7.77 (s, 1H), 7.64 (td, *J* = 8.0, 1.5 Hz, 1H), 7.52 (d, *J* = 8.0 Hz, 2H), 7.41 – 7.38 (m, 2H), 7.12 (d, *J* = 8.0 Hz, 2H), 2.33 (s, 3H).

¹³C NMR (126 MHz, CDCl₃) δ 175.4, 156.5, 155.0, 138.6, 134.8, 133.9, 130.6, 126.4, 125.6, 124.0, 123.2, 118.7, 118.1, 21.3.

IR (KBr) 2921, 2852, 1655, 1455, 1303, 1080, 765, 685 cm⁻¹.

HRMS for C₁₆H₁₃O₂Se⁺(M⁺+H): calcd. 317.00753, found 317.00748.



3-(m-tolylthio)-4H-chromen-4-one⁷

Purified by flash chromatograph column (PE : EtOAc = 5 : 1).

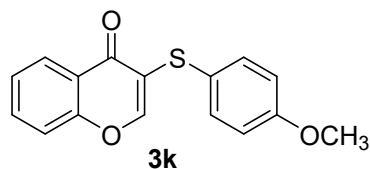
Yellow solid (38.6 mg, 72% yield); mp 109-111 °C.

¹H NMR (500 MHz, CDCl₃) δ 8.23 (dd, *J* = 8.0, 1.0 Hz, 1H), 8.11 (s, 1H), 7.69 – 7.66 (m, 1H), 7.46 – 7.40 (m, 2H), 7.22 – 7.14 (m, 3H), 7.02 (d, *J* = 7.0 Hz, 1H), 2.29 (s, 3H).

^{13}C NMR (126 MHz, CDCl_3) δ 175.2, 157.2, 156.4, 139.2, 134.0, 133.7, 130.7, 129.1, 128.2, 127.2, 126.5, 125.8, 123.8, 120.3, 118.2, 21.4.

IR (KBr) 2920, 2359, 1655, 1606, 1457, 1304, 1113, 763, 685 cm^{-1} .

HRMS for $\text{C}_{16}\text{H}_{13}\text{O}_2\text{S}^+(\text{M}^+\text{+H})$: calcd. 269.06308, found 269.06317.



3-((4-methoxyphenyl)thio)-4H-chromen-4-one⁶

Purified by flash chromatograph column (PE : EtOAc = 5 : 1).

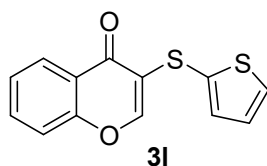
Yellow solid (45.8 mg, 81% yield); mp 108-110 $^{\circ}\text{C}$.

^1H NMR (500 MHz, CDCl_3) δ 8.21 (dd, J = 8.0, 1.0 Hz, 1H), 7.89 (s, 1H), 7.66 – 7.63 (m, 1H), 7.47 (d, J = 9.0 Hz, 2H), 7.42 – 7.37 (m, 2H), 6.85 (d, J = 9.0 Hz, 2H), 3.77 (s, 3H).

^{13}C NMR (126 MHz, CDCl_3) δ 175.1, 159.9, 156.3, 155.0, 134.3, 133.8, 126.3, 125.5, 123.4, 123.1, 122.6, 118.1, 115.0, 55.4.

IR (KBr) 2957, 2923, 1647, 1491, 1462, 1243, 825, 755 cm^{-1} .

HRMS for $\text{C}_{16}\text{H}_{13}\text{O}_3\text{S}^+(\text{M}^+\text{+H})$: calcd. 285.05799, found 285.05792.



3-(thiophen-2-ylthio)-4H-chromen-4-one⁷

Purified by flash chromatograph column (PE : EtOAc = 3 : 1).

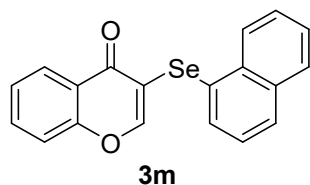
Yellow solid (34.5 mg, 66% yield); mp 82-84 $^{\circ}\text{C}$.

^1H NMR (500 MHz, CDCl_3) δ 8.25 – 8.23 (m, 1H), 7.87 (s, 1H), 7.68 – 7.65 (m, 1H), 7.43 – 7.40 (m, 3H), 7.36 (d, J = 3.0 Hz, 1H), 7.03 – 7.02 (m, 1H).

^{13}C NMR (126 MHz, CDCl_3) δ 174.9, 156.3, 154.6, 135.9, 134.0, 131.2, 130.1, 128.0, 126.3, 125.7, 123.4, 123.2, 118.2.

IR (KBr) 2920, 1643, 1461, 1344, 1211, 754, 692 cm^{-1} .

HRMS for $\text{C}_{13}\text{H}_9\text{O}_2\text{S}_2^+(\text{M}^+\text{+H})$: calcd. 261.00385, found 261.00388.



3-(naphthalen-1-ylselanyl)-4H-chromen-4-one⁷

Purified by flash chromatograph column (PE : EtOAc = 10 : 1).

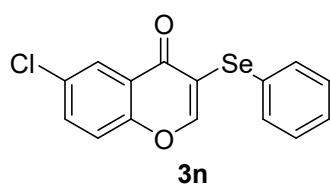
Yellow solid (48.7 mg, 69% yield); mp 79-80 °C.

¹H NMR (500 MHz, CDCl₃) δ 8.39 (d, *J* = 8.0 Hz, 1H), 8.23 (dd, *J* = 9.0, 1.5 Hz, 1H), 7.95 (dd, *J* = 7.0, 0.5 Hz, 1H), 7.89 – 7.84 (m, 2H), 7.63 – 7.59 (m, 1H), 7.56 – 7.50 (m, 2H), 7.42 – 7.37 (m, 2H), 7.33 – 7.31 (m, 2H).

¹³C NMR (126 MHz, CDCl₃) δ 175.5, 156.3, 153.4, 135.4, 134.4, 134.3, 133.8, 130.3, 128.9, 127.8, 127.5, 126.7, 126.2, 126.2, 126.0, 125.5, 122.8, 118.4, 118.1.

IR (KBr) 2921, 1627, 1459, 1312, 1107, 1067, 755, 690 cm⁻¹.

HRMS for C₁₉H₁₃O₂Se⁺(M⁺+H): calcd. 353.00753, found 353.00751.



6-chloro-3-(phenylselenanyl)-4H-chromen-4-one⁵

Purified by flash chromatograph column (PE : EtOAc = 10 : 1).

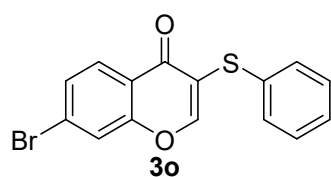
Yellow solid (45.5 mg, 68% yield); mp 110-111 °C.

¹H NMR (500 MHz, CDCl₃) δ 8.08 (d, *J* = 2.5 Hz, 1H), 7.74 (s, 1H), 7.52 – 7.49 (m, 3H), 7.29 (d, *J* = 9.0 Hz, 1H), 7.23 – 7.21 (m, 3H).

¹³C NMR (126 MHz, CDCl₃) δ 174.2, 155.5, 154.8, 134.3, 134.2, 131.6, 129.8, 128.5, 127.7, 125.8, 124.0, 120.0, 118.3.

IR (KBr) 2921, 1640, 1478, 1308, 1115, 1066, 741, 691 cm⁻¹.

HRMS for C₁₅H₁₀ClO₂Se⁺(M⁺+H): calcd. 336.95291, found 336.95258.



7-bromo-3-(phenylthio)-4H-chromen-4-one⁷

Purified by flash chromatograph column (PE : EtOAc = 15 : 1).

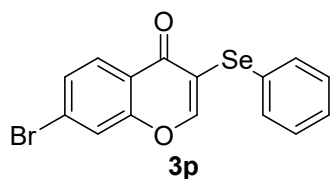
Yellow solid (48.2 mg, 73% yield); mp 135-137 °C.

¹H NMR (500 MHz, CDCl₃) δ 8.08 (d, *J* = 8.5 Hz, 1H), 8.04 (s, 1H), 7.65 (d, *J* = 1.0 Hz, 1H), 7.53 (dd, *J* = 8.5, 1.5 Hz, 1H), 7.40 (d, *J* = 7.0 Hz, 2H), 7.30 (t, *J* = 7.0 Hz, 2H), 7.24 (t, *J* = 7.0 Hz, 1H).

¹³C NMR (126 MHz, CDCl₃) δ 174.4, 156.7, 156.4, 133.4, 130.5, 129.4, 129.4, 128.4, 127.9, 127.5, 122.5, 121.3, 121.1.

IR (KBr) 2921, 1645, 1600, 1417, 1117, 777, 735, 685 cm⁻¹.

HRMS for C₁₅H₁₀BrO₂S⁺(M⁺+H): calcd. 332.95794, found 332.95798.



7-bromo-3-(phenylselanyl)-4H-chromen-4-one⁵

Purified by flash chromatograph column (PE : EtOAc = 10 : 1).

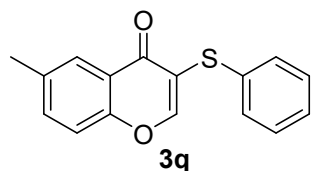
Yellow solid (48.7 mg, 64% yield); mp 115-116 °C.

¹H NMR (500 MHz, CDCl₃) δ 8.06 (d, *J* = 8.5 Hz, 1H), 7.76 (s, 1H), 7.61 – 7.59 (m, 3H), 7.50 (dd, *J* = 9.5, 1.5 Hz, 1H), 7.31 – 7.30 (m, 3H).

¹³C NMR (126 MHz, CDCl₃) δ 174.7, 156.5, 155.1, 134.4, 129.8, 129.3, 128.5, 128.3, 127.9, 127.6, 122.0, 121.3, 118.9.

IR (KBr) 2924, 2360, 1616, 1435, 1238, 1078, 1022, 852, 739 cm⁻¹.

HRMS for C₁₅H₁₀BrO₂Se⁺(M⁺+H): calcd. 380.90239, found 380.90204.



6-methyl-3-(phenylthio)-4H-chromen-4-one⁷

Purified by flash chromatograph column (PE : EtOAc = 15 : 1).

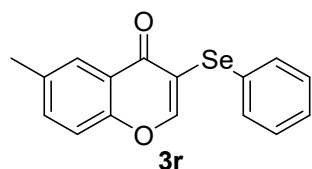
Yellow solid (43.3 mg, 81% yield); mp 102-103 °C.

¹H NMR (500 MHz, CDCl₃) δ 8.10 (s, 1H), 7.98 (s, 1H), 7.45 (dd, *J* = 8.5, 1.5 Hz, 1H), 7.36 – 7.31 (m, 3H), 7.24 (t, *J* = 7.5 Hz, 2H), 7.17 (t, *J* = 7.5 Hz, 1H), 2.40 (s, 3H).

¹³C NMR (126 MHz, CDCl₃) δ 175.2, 157.5, 154.6, 135.9, 135.3, 134.3, 129.7, 129.2, 127.0, 125.7, 123.4, 119.5, 117.9, 21.0.

IR (KBr) 2920, 1639, 1558, 1478, 1307, 1122, 820, 748 cm⁻¹.

HRMS for C₁₆H₁₃O₂S⁺(M⁺+H): calcd. 269.06308, found 269.06351.



6-methyl-3-(phenylselanyl)-4H-chromen-4-one⁵

Purified by flash chromatograph column (PE : EtOAc = 10 : 1).

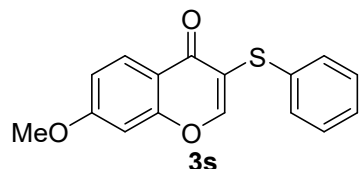
Yellow solid (45.4 mg, 72% yield); mp 100-102 °C.

¹H NMR (500 MHz, CDCl₃) δ 7.91 (s, 1H), 7.79 (s, 1H), 7.50 – 7.48 (m, 2H), 7.37 (dd, *J* = 9.0, 2.0 Hz, 1H), 7.22 – 7.18 (m, 4H), 2.34 (s, 3H).

^{13}C NMR (126 MHz, CDCl_3) δ 175.3, 156.0, 154.7, 135.7, 135.2, 133.7, 129.6, 128.5, 128.1, 125.7, 122.9, 117.9, 117.5, 21.0.

IR (KBr) 2922, 2359, 1643, 1475, 1311, 1117, 1066, 820, 742 cm^{-1} .

HRMS for $\text{C}_{16}\text{H}_{13}\text{O}_2\text{Se}^+(\text{M}^+\text{+H})$: calcd. 317.00753, found 317.00742.



7-methoxy-3-(phenylthio)-4H-chromen-4-one⁷

Purified by flash chromatograph column (PE : EtOAc = 15 : 1).

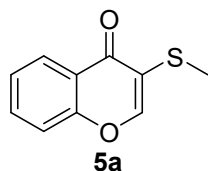
Yellow solid (45.0 mg, 79% yield); mp 103-105 °C.

^1H NMR (500 MHz, CDCl_3) δ 8.11 (d, J = 9.0 Hz, 1H), 8.05 (s, 1H), 7.37 (d, J = 7.5 Hz, 2H), 7.26 (t, J = 7.5 Hz, 2H), 7.19 (t, J = 7.5 Hz, 1H), 6.95 (dd, J = 9.0, 2.0 Hz, 1H), 6.81 (d, J = 7.0 Hz, 1H), 3.87 (s, 3H).

^{13}C NMR (126 MHz, CDCl_3) δ 174.4, 164.4, 158.2, 157.0, 134.4, 129.8, 129.2, 127.9, 127.1, 119.8, 117.6, 115.1, 100.4, 56.0.

IR (KBr) 2959, 1616, 1437, 1270, 1235, 1081, 831, 688 cm^{-1} .

HRMS for $\text{C}_{16}\text{H}_{13}\text{O}_3\text{S}^+(\text{M}^+\text{+H})$: calcd. 285.05799, found 285.05774.



3-(methylthio)-4H-chromen-4-one²

Purified by flash chromatograph column (PE : EtOAc = 10 : 1).

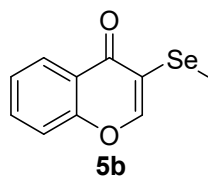
Yellow solid (28.7 mg, 75% yield); mp 87-92 °C.

^1H NMR (500 MHz, CDCl_3) δ 8.26 (dd, J = 8.0, 1.5 Hz, 1H), 8.06 (s, 1H), 7.70 – 7.66 (m, 1H), 7.47 – 7.41 (m, 2H), 2.41 (s, 3H).

^{13}C NMR (126 MHz, CDCl_3) δ 175.8, 156.4, 154.1, 133.9, 126.3, 125.6, 123.3, 122.0, 118.2, 16.4.

IR (KBr) 3070, 2922, 1597, 1463, 1253, 1109, 754, 693 cm^{-1} .

HRMS for $\text{C}_{10}\text{H}_9\text{O}_2\text{S}^+(\text{M}^+\text{+H})$: calcd. 193.03178, found 193.03192.



3-(methylselanyl)-4H-chromen-4-one

Purified by flash chromatograph column (PE : EtOAc = 10 : 1).

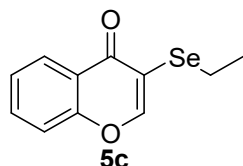
Yellow solid (31.8 mg, 66% yield); mp 44-45 °C.

¹H NMR (500 MHz, CDCl₃) δ 8.25 (d, *J* = 9.0 Hz, 1H), 8.06 (s, 1H), 7.70 – 7.66 (m, 1H), 7.46 – 7.41 (m, 2H), 2.41 (s, 3H).

¹³C NMR (126 MHz, CDCl₃) δ 175.8, 156.4, 154.0, 133.9, 126.2, 125.6, 123.3, 122.0, 118.2, 16.4.

IR (KBr) 2920, 2851, 1625, 1597, 1463, 1080, 892, 756 cm⁻¹.

HRMS for C₁₀H₉O₂Se⁺(M⁺+H): calcd.240.97623, found 240.97635.



3-(ethylselanyl)-4H-chromen-4-one

Purified by flash chromatograph column (PE : EtOAc = 5 : 1).

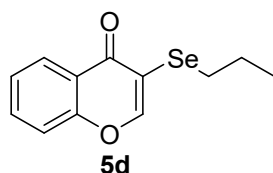
Yellow solid (31.4 mg, 62% yield); mp 42-43 °C.

¹H NMR (500 MHz, CDCl₃) δ 8.24 (dd, *J* = 8.0, 1.5 Hz, 1H), 8.19 (s, 1H), 7.70 – 7.66 (m, 1H), 7.46 – 7.41 (m, 2H), 2.92 (q, *J* = 2.5 Hz, 2H), 1.40 (t, *J* = 7.5 Hz, 3H).

¹³C NMR (126 MHz, CDCl₃) δ 176.0, 156.6, 156.5, 133.9, 126.4, 125.6, 123.3, 118.2, 114.5, 19.9, 15.5.

IR (KBr) 2958, 2922, 1610, 1502, 1308, 1069, 769, 689 cm⁻¹.

HRMS for C₁₁H₁₁O₂Se⁺(M⁺+H): calcd.254.99188, found 254.99178.



3-(propylselanyl)-4H-chromen-4-one

Purified by flash chromatograph column (PE : EtOAc = 5 : 1).

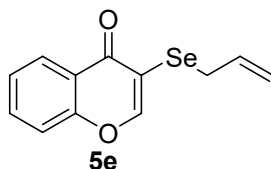
Yellow solid (36.1 mg, 67% yield); mp 41-42 °C.

¹H NMR (500 MHz, CDCl₃) δ 8.25 (dd, *J* = 8.0, 1.5 Hz, 1H), 8.19 (s, 1H), 7.80 – 7.66 (m, 1H), 7.46 – 7.41 (m, 2H), 2.89 (t, *J* = 7.5 Hz, 2H), 1.73 – 1.66 (m, 2H), 1.00 (t, *J* = 7.5 Hz, 3H).

¹³C NMR (126 MHz, CDCl₃) δ 176.1, 156.5, 133.8, 126.5, 125.6, 123.4, 118.2, 114.8, 28.5, 23.5, 14.4.

IR (KBr) 2924, 2869, 1607, 1554, 1461, 1342, 1058, 752 cm⁻¹.

HRMS for C₁₂H₁₃O₂Se⁺(M⁺+H): calcd. 269.00753, found 269.00766.



3-(allylselanyl)-4H-chromen-4-one

Purified by flash chromatograph column (PE : EtOAc = 5 : 1).

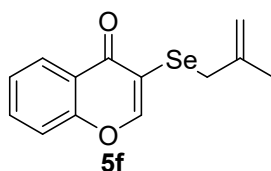
Yellow solid (41.6 mg, 78% yield); mp 56-57 °C.

¹H NMR (500 MHz, CDCl₃) δ 8.17 (dd, *J* = 8.0, 1.0 Hz, 1H), 8.08 (s, 1H), 7.63 – 7.59 (m, 1H), 7.39 – 7.35 (m, 2H), 5.85 – 5.76 (m, 1H), 4.86 – 4.79 (m, 2H), 3.44 (d, *J* = 7.5 Hz, 2H).

¹³C NMR (126 MHz, CDCl₃) δ 175.9, 157.9, 156.5, 134.2, 133.9, 126.5, 125.7, 123.5, 118.2, 117.4, 114.1, 28.7.

IR (KBr) 2922, 1608, 1461, 1307, 1108, 1070, 755, 691 cm⁻¹.

HRMS for C₁₂H₁₁O₂Se⁺(M⁺+H): calcd. 266.99188, found 266.99197.



3-((2-methylallyl)selanyl)-4H-chromen-4-one

Purified by flash chromatograph column (PE : EtOAc = 5 : 1).

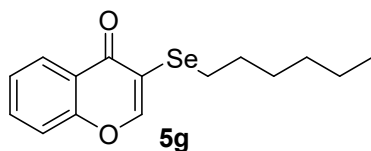
Yellow solid (40.9 mg, 73% yield); mp 59-60 °C.

¹H NMR (500 MHz, CDCl₃) δ 8.17 (dd, *J* = 8.0, 1.5 Hz, 1H), 8.05 (s, 1H), 7.62 – 7.59 (m, 1H), 7.38 – 7.34 (m, 2H), 4.63 (s, 1H), 4.52 (s, 1H), 3.43 (s, 2H), 1.77 (s, 3H).

¹³C NMR (126 MHz, CDCl₃) δ 175.7, 157.8, 156.5, 141.4, 133.8, 126.4, 125.6, 123.5, 118.2, 114.5, 114.2, 33.9, 21.1.

IR (KBr) 2922, 1608, 1459, 1305, 1059, 759, 689 cm⁻¹.

HRMS for C₁₃H₁₃O₂Se⁺(M⁺+H): calcd. 281.00753, found 281.00754.



3-(hexylselanyl)-4H-chromen-4-one

Purified by flash chromatograph column (PE : EtOAc = 5 : 1).

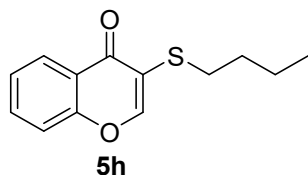
Yellow solid (42.5 mg, 69% yield); mp 78-80 °C.

¹H NMR (500 MHz, CDCl₃) δ 8.23 (dd, *J* = 8.0, 1.5 Hz, 1H), 8.17 (s, 1H), 7.69 – 7.65 (m, 1H), 7.45 – 7.40 (m, 2H), 2.88 (t, *J* = 7.5 Hz, 2H), 1.69 – 1.63 (m, 2H), 1.42 – 1.36 (m, 2H), 1.29 – 1.26 (m, 4H), 0.86 (t, *J* = 7.0 Hz, 3H).

^{13}C NMR (126 MHz, CDCl_3) δ 175.9, 156.4, 156.2, 133.7, 126.3, 125.5, 123.2, 118.1, 114.8, 31.3, 30.1, 29.4, 26.3, 22.6, 14.0.

IR (KBr) 2922, 2853, 1705, 1606, 1461, 1069, 751, 689 cm^{-1} .

HRMS for $\text{C}_{15}\text{H}_{19}\text{O}_2\text{Se}^+(\text{M}^+\text{+H})$: calcd. 311.05448, found 311.05444.



3-(butylthio)-4H-chromen-4-one⁶

Purified by flash chromatograph column (PE : EtOAc = 10 : 1).

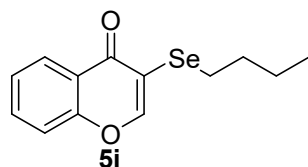
Yellow solid (34.8 mg, 74% yield); mp 48-50 $^{\circ}\text{C}$.

^1H NMR (500 MHz, CDCl_3) δ 8.25 (dd, $J = 8.0, 1.5$ Hz, 1H), 8.15 (s, 1H), 7.70 – 7.67 (m, 1H), 7.47 – 7.42 (m, 2H), 2.86 (t, $J = 7.0$ Hz, 2H), 1.59 – 1.53 (m, 2H), 1.46 – 1.39 (m, 2H), 0.90 (t, $J = 7.5$ Hz, 3H).

^{13}C NMR (126 MHz, CDCl_3) δ 176.1, 156.4, 156.1, 133.9, 126.3, 125.6, 123.5, 119.8, 118.2, 32.5, 31.5, 21.8, 13.7.

IR (KBr) 2953, 2868, 1609, 1462, 1352, 1112, 751, 691 cm^{-1} .

HRMS for $\text{C}_{13}\text{H}_{15}\text{O}_2\text{S}^+(\text{M}^+\text{+H})$: calcd. 235.07873, found 235.07903.



3-(butylselanyl)-4H-chromen-4-one⁸

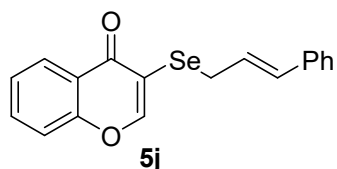
Purified by flash chromatograph column (PE : EtOAc = 5 : 1).

Yellow solid (37.7 mg, 67% yield); mp 50-51 $^{\circ}\text{C}$.

^1H NMR (500 MHz, CDCl_3) δ 8.24 (dd, $J = 8.0, 1.5$ Hz, 1H), 8.18 (s, 1H), 7.70 – 7.66 (m, 1H), 7.46 – 7.41 (m, 2H), 2.89 (t, $J = 7.5$ Hz, 2H), 1.68 – 1.62 (m, 2H), 1.46 – 1.38 (m, 2H), 0.90 (t, $J = 7.5$ Hz, 3H)

^{13}C NMR (126 MHz, CDCl_3) δ 176.1, 156.4, 133.9, 126.5, 125.6, 123.4, 118.2, 114.9, 36.6, 32.3, 26.1, 22.9, 13.7.

IR (KBr) 2954, 2868, 1723, 1607, 1462, 1113, 750 691 cm^{-1} .



3-(cinnamylselanyl)-4H-chromen-4-one

Purified by flash chromatograph column (PE : EtOAc = 5 : 1).

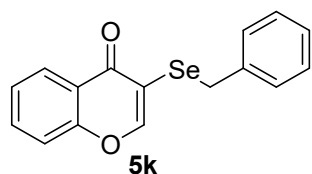
Yellow solid (44.2 mg, 65% yield); mp 82-83 °C.

¹H NMR (500 MHz, CDCl₃) δ 8.24 (dd, *J* = 8.0, 1.5 Hz, 1H), 7.79 (s, 1H), 7.66 – 7.63 (m, 1H), 7.44 – 7.35 (m, 4H), 7.29 (t, *J* = 7.5 Hz, 2H), 7.29 (t, *J* = 7.5 Hz, 1H), 6.53 (d, *J* = 16.0 Hz, 1H), 6.38 – 6.32 (m, 1H), 3.40 (d, *J* = 7.0 Hz, 2H).

¹³C NMR (126 MHz, CDCl₃) δ 177.6, 156.6, 152.8, 137.2, 133.6, 132.4, 128.6, 127.4, 126.4, 126.3, 126.0, 125.1, 123.9, 123.4, 118.2, 29.1.

IR (KBr) 2958, 2922, 1609, 1452, 1259, 1071, 745, 689 cm⁻¹.

HRMS for C₁₈H₁₅O₂Se⁺(M⁺+H): calcd. 343.02318, found 343.02252.



3-(benzylselanyl)-4H-chromen-4-one⁵

Purified by flash chromatograph column (PE : EtOAc = 5 : 1).

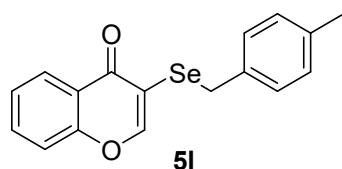
Yellow solid (49.4 mg, 78% yield); mp 103-105 °C.

¹H NMR (500 MHz, CDCl₃) δ 8.27 (dd, *J* = 8.0, 2.0 Hz, 1H), 7.88 (s, 1H), 7.70 – 7.66 (m, 1H), 7.46 – 7.41 (m, 2H), 7.24 – 7.15 (m, 5H), 4.11 (s, 2H).

¹³C NMR (126 MHz, CDCl₃) δ 176.0, 157.8, 156.5, 138.6, 133.9, 129.1, 128.6, 127.0, 126.5, 125.8, 123.5, 118.2, 114.4, 29.9.

IR (KBr) 2920, 1701, 1610, 1463, 1346, 1111, 1072, 754, 690 cm⁻¹.

HRMS for C₁₆H₁₃O₂Se⁺(M⁺+H): calcd. 317.00753, found 317.00751.



3-((4-methylbenzyl)selanyl)-4H-chromen-4-one

Purified by flash chromatograph column (PE : EtOAc = 5 : 1).

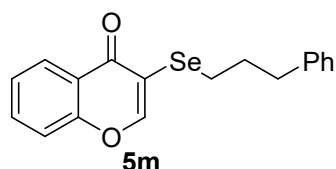
Yellow solid (48.0 mg, 73% yield); mp 101-103 °C

¹H NMR (500 MHz, CDCl₃) δ 8.18 (dd, *J* = 8.0, 1.5 Hz, 1H), 7.81 (s, 1H), 7.60 – 7.57 (m, 1H), 7.37 – 7.32 (m, 2H), 7.01 – 6.93 (m, 4H), 4.00 (s, 2H), 2.19 (s, 3H).

¹³C NMR (126 MHz, CDCl₃) δ 175.9, 157.5, 156.4, 136.6, 135.4, 133.8, 129.2, 129.0, 126.4, 125.7, 123.4, 118.2, 114.5, 29.6, 21.2.

IR (KBr) 2919, 1628, 1461, 1343, 1072, 752, 689 cm⁻¹

HRMS for C₁₇H₁₅O₂Se⁺(M⁺+H): calcd. 331.02318, found 331.02298.



3-(3-phenylpropylselanyl)-4H-chromen-4-one

Purified by flash chromatograph column (PE : EtOAc = 5 : 1).

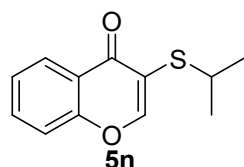
Yellow oil (40.1 mg, 58% yield).

¹H NMR (500 MHz, CDCl₃) δ 8.24 (dd, *J* = 8.0, 1.5 Hz, 1H), 8.16 (s, 1H), 7.70 – 7.66 (m, 1H), 7.46 – 7.41 (m, 2H), 7.26 (t, *J* = 7.5 Hz, 2H), 7.19 – 7.15 (m, 3H), 2.90 (t, *J* = 7.5 Hz, 2H), 2.73 (t, *J* = 7.5 Hz, 2H), 2.02 – 1.96 (m, 2H).

¹³C NMR (126 MHz, CDCl₃) δ 176.0, 156.6, 156.5, 141.4, 133.9, 128.6, 128.5, 126.5, 126.1, 125.7, 123.4, 118.2, 114.6, 35.8, 31.8, 25.7

IR (KBr) 2922, 1612, 1458, 1340, 1136, 1050, 742, 698 cm⁻¹.

HRMS for C₁₈H₁₇O₂Se⁺(M⁺+H): calcd. 345.03883, found 345.03882.



3-(isopropylthio)-4H-chromen-4-one

Purified by flash chromatograph column (PE : EtOAc = 10 : 1).

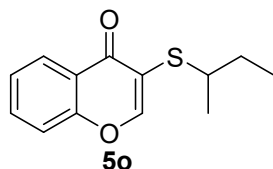
Yellow solid (31.2 mg, 71% yield); mp 45-46 °C.

¹H NMR (500 MHz, CDCl₃) δ 8.26 – 8.25 (m, 2H), 7.71 – 7.67 (m, 1H), 7.48 – 7.42 (m, 2H), 3.35 – 3.27 (m, 1H), 1.35 (d, *J* = 7.0 Hz, 6H).

¹³C NMR (126 MHz, CDCl₃) δ 175.6, 156.4, 155.9, 134.1, 126.3, 125.8, 123.6, 123.2, 118.3, 41.8, 22.6.

IR (KBr) 2920, 2861, 1610, 1544, 1442, 1339, 1050, 748 cm⁻¹

HRMS for C₁₂H₁₃O₂S⁺(M⁺+H): calcd. 221.06308, found 221.06300.



3-(sec-butylthio)-4H-chromen-4-one

Purified by flash chromatograph column (PE : EtOAc = 10 : 1).

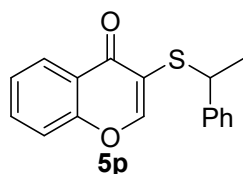
Yellow oil (27.3 mg, 58% yield).

¹H NMR (500 MHz, CDCl₃) δ 8.26 – 8.25 (m, 2H), 7.71 – 7.68 (m, 1H), 7.48 – 7.42 (m, 2H), 3.11 – 3.05 (m, 1H), 1.80 – 1.73 (m, 1H), 1.62 – 1.54 (m, 1H), 1.35 (d, *J* = 7.5 Hz, 3H), 0.98 (t, *J* = 7.5 Hz, 3H).

¹³C NMR (126 MHz, CDCl₃) δ 175.6, 156.4, 155.7, 134.1, 126.3, 125.8, 123.5, 123.2, 118.3, 48.5, 29.0, 20.0, 11.5.

IR (KBr) 2925, 1612, 1452, 1335, 1101, 1032, 744, 702 cm⁻¹.

HRMS for C₁₃H₁₅O₂S⁺(M⁺+H): calcd. 235.07873, found 235.07904.



3-((1-phenylethyl)thio)-4H-chromen-4-one

Purified by flash chromatograph column (PE : EtOAc = 10 : 1).

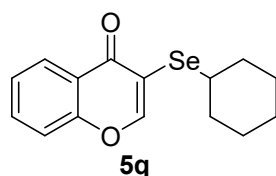
Yellow oil (28.9 mg, 51% yield).

¹H NMR (500 MHz, CDCl₃) δ 8.23 (dd, *J* = 8.0, 1.5 Hz, 1H), 7.81 (s, 1H), 7.69 – 7.65 (m, 1H), 7.44 – 7.41 (m, 2H), 7.36 (d, *J* = 7.5 Hz, 2H), 7.26 – 7.23 (m, 2H), 7.18 (t, *J* = 7.0 Hz, 1H), 4.91 (q, *J* = 7.0 Hz, 1H), 1.72 (d, *J* = 7.0 Hz, 3H).

¹³C NMR (126 MHz, CDCl₃) δ 175.4, 156.2, 156.2, 141.8, 134.0, 128.6, 128.0, 127.7, 126.3, 125.7, 123.6, 122.1, 118.2, 50.6, 20.9.

IR (KBr) 2953, 2916, 1642, 1609, 1555, 1462, 755, 691 cm⁻¹.

HRMS for C₁₇H₁₅O₂S⁺(M⁺+H): calcd. 283.07873, found 283.07693.



3-(cyclohexylselanyl)-4H-chromen-4-one

Purified by flash chromatograph column (PE : EtOAc = 10 : 1).

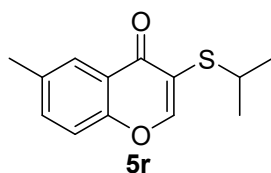
Yellow oil (33.0 mg, 54% yield).

¹H NMR (500 MHz, CDCl₃) δ 8.26 – 8.24 (m, 2H), 7.70 – 7.67 (m, 1H), 7.47 – 7.42 (m, 2H), 7.46 – 3.41 (m, 1H), 2.01 – 1.99 (m, 2H), 1.74 – 1.71 (m, 2H), 1.61 – 1.59 (m, 1H), 1.52 – 1.45 (m, 2H), 1.34 – 1.24 (m, 3H).

¹³C NMR (126 MHz, CDCl₃) δ 176.3, 158.1, 156.6, 133.9, 126.6, 125.7, 123.6, 118.2, 114.2, 41.4, 34.2, 26.8, 25.8.

IR (KBr) 2924, 2848, 1610, 1453, 1342, 1214, 755, 699 cm⁻¹.

HRMS for C₁₅H₁₇O₂Se⁺(M⁺+H): calcd. 309.03883, found 309.03876.



3-(isopropylthio)-6-methyl-4H-chromen-4-one

Purified by flash chromatograph column (PE : EtOAc = 10 : 1).

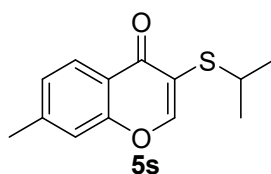
Yellow solid (33.5 mg, 72% yield); mp 47-48 °C.

¹H NMR (500 MHz, CDCl₃) δ 8.23 (s, 1H), 8.02 (s, 1H), 7.49 (dd, *J* = 8.5, 2.0 Hz, 1H), 7.36 (d, *J* = 8.5 Hz, 1H), 3.35 – 3.27 (m, 1H), 2.46 (s, 3H), 1.35 (d, *J* = 7.0 Hz, 6H).

¹³C NMR (126 MHz, CDCl₃) δ 175.7, 156.0, 154.7, 135.9, 135.3, 125.5, 123.3, 122.8, 118.0, 41.8, 22.6, 21.1.

IR (KBr) 2924, 1607, 1548, 1452, 1324, 1035, 735 cm⁻¹.

HRMS for C₁₃H₁₅O₂S⁺(M⁺+H): calcd. 235.07873, found 235.07878.



3-(isopropylthio)-7-methyl-4H-chromen-4-one

Purified by flash chromatograph column (PE : EtOAc = 10 : 1).

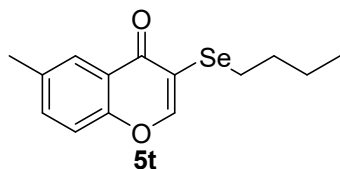
Yellow solid (33.4 mg, 71% yield); mp 45-46 °C.

¹H NMR (500 MHz, CDCl₃) δ 8.15 – 8.12 (m, 2H), 7.25 – 7.23 (m, 2H), 3.58 – 3.50 (m, 1H), 2.49 (s, 3H), 1.25 (d, *J* = 7.0 Hz, 6H).

¹³C NMR (126 MHz, CDCl₃) δ 176.3, 157.5, 156.6, 145.4, 127.2, 126.2, 121.5, 119.0, 117.9, 36.2, 23.1, 21.9.

IR (KBr) 2958, 2922, 2863, 1620, 1550, 1419, 1347, 776, 693 cm⁻¹.

HRMS for C₁₃H₁₅O₂S⁺(M⁺+H): calcd. 235.07873, found 235.07899.



3-(butylselanyl)-6-methyl-4H-chromen-4-one

Purified by flash chromatograph column (PE : EtOAc = 5 : 1).

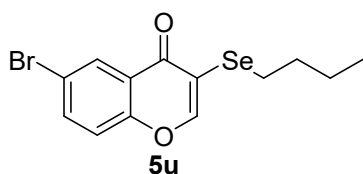
Yellow solid (40.7 mg, 69% yield); mp 39-40 °C.

¹H NMR (500 MHz, CDCl₃) δ 8.16 (s, 1H), 8.02 (s, 1H), 7.48 (dd, *J* = 8.5, 1.5 Hz, 1H), 7.34 (d, *J* = 8.5 Hz, 1H), 2.88 (t, *J* = 7.5 Hz, 2H), 2.46 (s, 3H), 1.67 – 1.61 (m, 2H), 1.45 – 1.37 (m, 2H), 0.90 (t, *J* = 7.5 Hz, 3H).

¹³C NMR (126 MHz, CDCl₃) δ 176.1, 156.4, 154.8, 135.6, 135.1, 125.7, 123.0, 117.9, 114.5, 32.3, 26.1, 22.9, 21.1, 13.7.

IR (KBr) 2955, 2868, 1615, 1480, 1307, 1068, 924, 771 cm⁻¹

HRMS for C₁₄H₁₇O₂Se⁺(M⁺+H): calcd. 297.03883, found 297.03888.



6-bromo-3-(butylselanyl)-4H-chromen-4-one

Purified by flash chromatograph column (PE : EtOAc = 20 : 1).

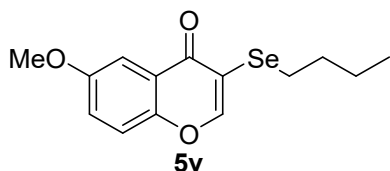
Yellow solid (45.8 mg, 64% yield); mp 74-76 °C.

¹H NMR (500 MHz, CDCl₃) δ 8.28 (s, 1H), 8.09 (s, 1H), 7.67 (d, *J* = 8.5 Hz, 1H), 7.28 (d, *J* = 9.0 Hz, 1H), 2.81 (t, *J* = 7.0 Hz, 2H), 1.60 – 1.54 (m, 2H), 1.38 – 1.30 (m, 2H), 0.83 (t, *J* = 7.0 Hz, 3H).

¹³C NMR (126 MHz, CDCl₃) δ 174.8, 156.3, 155.2, 136.8, 129.0, 124.5, 120.2, 119.0, 115.1, 32.2, 26.2, 22.9, 13.7.

IR (KBr) 2926, 2865, 1622, 1549, 1457, 1363, 1117, 1067, 893, 813 cm⁻¹.

HRMS for C₁₃H₁₄BrO₂Se⁺(M⁺+H): calcd. 360.93369, found 360.93347.



3-(butylselanyl)-6-methoxy-4H-chromen-4-one

Purified by flash chromatograph column (PE : EtOAc = 5 : 1).

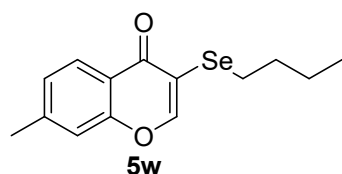
Yellow solid (45.6 mg, 73% yield); mp 44-46 °C.

¹H NMR (500 MHz, CDCl₃) δ 8.17 (s, 1H), 7.58 (d, *J* = 3.0 Hz, 1H), 7.38 (d, *J* = 9.5 Hz, 1H), 7.26 (dd, *J* = 9.0, 3.0 Hz, 1H), 3.90 (s, 3H), 2.89 (t, *J* = 7.5 Hz, 2H), 1.68 – 1.62 (m, 2H), 1.45 – 1.38 (m, 2H), 0.90 (t, *J* = 7.0 Hz, 3H).

¹³C NMR (126 MHz, CDCl₃) δ 175.9, 157.2, 156.3, 151.3, 124.0, 123.9, 119.6, 113.8, 105.3, 56.0, 32.3, 26.1, 22.9, 13.7.

IR (KBr) 2927, 2867, 1610, 1480, 1307, 1067, 825, 715 cm⁻¹.

HRMS for C₁₄H₁₇O₃Se⁺(M⁺+H): calcd. 313.03374, found 313.03357.



3-(butylselanyl)-7-methyl-4H-chromen-4-one

Purified by flash chromatograph column (PE : EtOAc = 5 : 1).

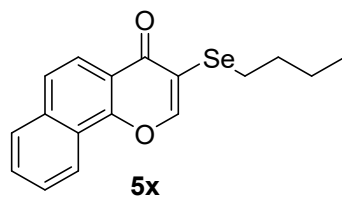
Yellow oil (36.5 mg, 62% yield).

¹H NMR (500 MHz, CDCl₃) δ 8.13 – 8.11 (m, 2H), 7.24 – 7.22 (m, 2H), 2.88 (t, *J* = 7.5 Hz, 2H), 2.48 (s, 3H), 1.67 – 1.63 (m, 2H), 1.43 – 1.39 (m, 2H), 0.90 (t, *J* = 7.5 Hz, 3H).

¹³C NMR (126 MHz, CDCl₃) δ 175.9, 156.6, 156.2, 145.2, 127.1, 126.2, 121.1, 117.9, 114.7, 32.3, 26.1, 22.9, 21.9, 13.7.

IR (KBr) 2955, 1641, 1609, 1550, 1462, 1306, 1112, 843, 751cm⁻¹.

HRMS for C₁₄H₁₇O₂Se⁺(M⁺+H): calcd. 297.03883, found 297.03864 .



3-(butylselanyl)-4H-benzo[h]chromen-4-one

Purified by flash chromatograph column (PE : EtOAc = 5 : 1).

Yellow solid (34.3 mg, 52% yield); mp 84-85 °C.

¹H NMR (500 MHz, CDCl₃) δ 8.47 (d, *J* = 8.5 Hz, 1H), 8.33 (s, 1H), 8.18 (d, *J* = 9.0 Hz, 1H), 7.94 (d, *J* = 7.5 Hz, 1H), 7.79 (d, *J* = 8.5 Hz, 1H), 7.74 – 7.67 (m, 2H), 2.95 (t, *J* = 7.5 Hz, 2H), 1.72 – 1.66 (m, 2H), 1.48 – 1.41 (m, 2H), 0.92 (t, *J* = 7.5 Hz, 3H).

¹³C NMR (126 MHz, CDCl₃) δ 175.9, 155.0, 154.0, 135.9, 129.6, 128.3, 127.4, 125.8, 124.1, 122.4, 121.3, 119.5, 116.8, 32.3, 25.9, 23.0, 13.7.

IR (KBr) 2922, 2853, 1720, 1462, 1354, 1080, 759, 690 cm⁻¹.

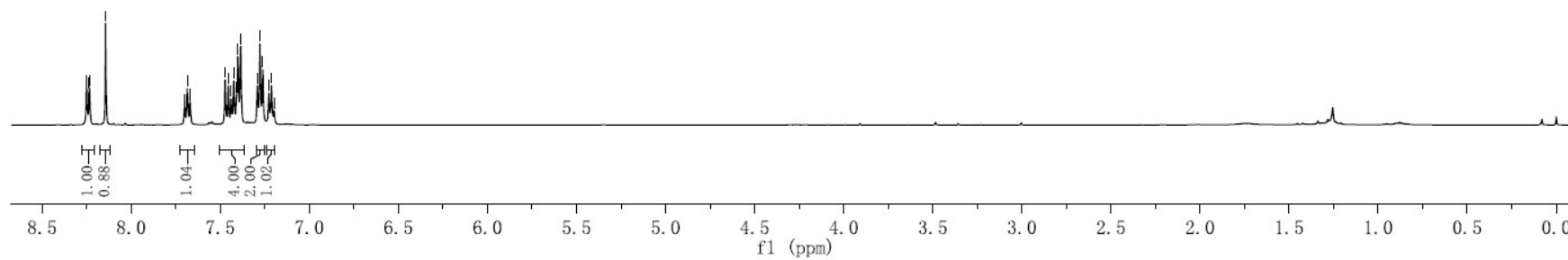
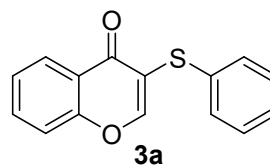
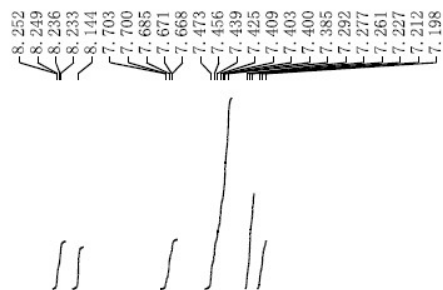
HRMS for C₁₇H₁₇O₂Se⁺(M⁺+H): calcd. 333.03883, found 333.03888.

References

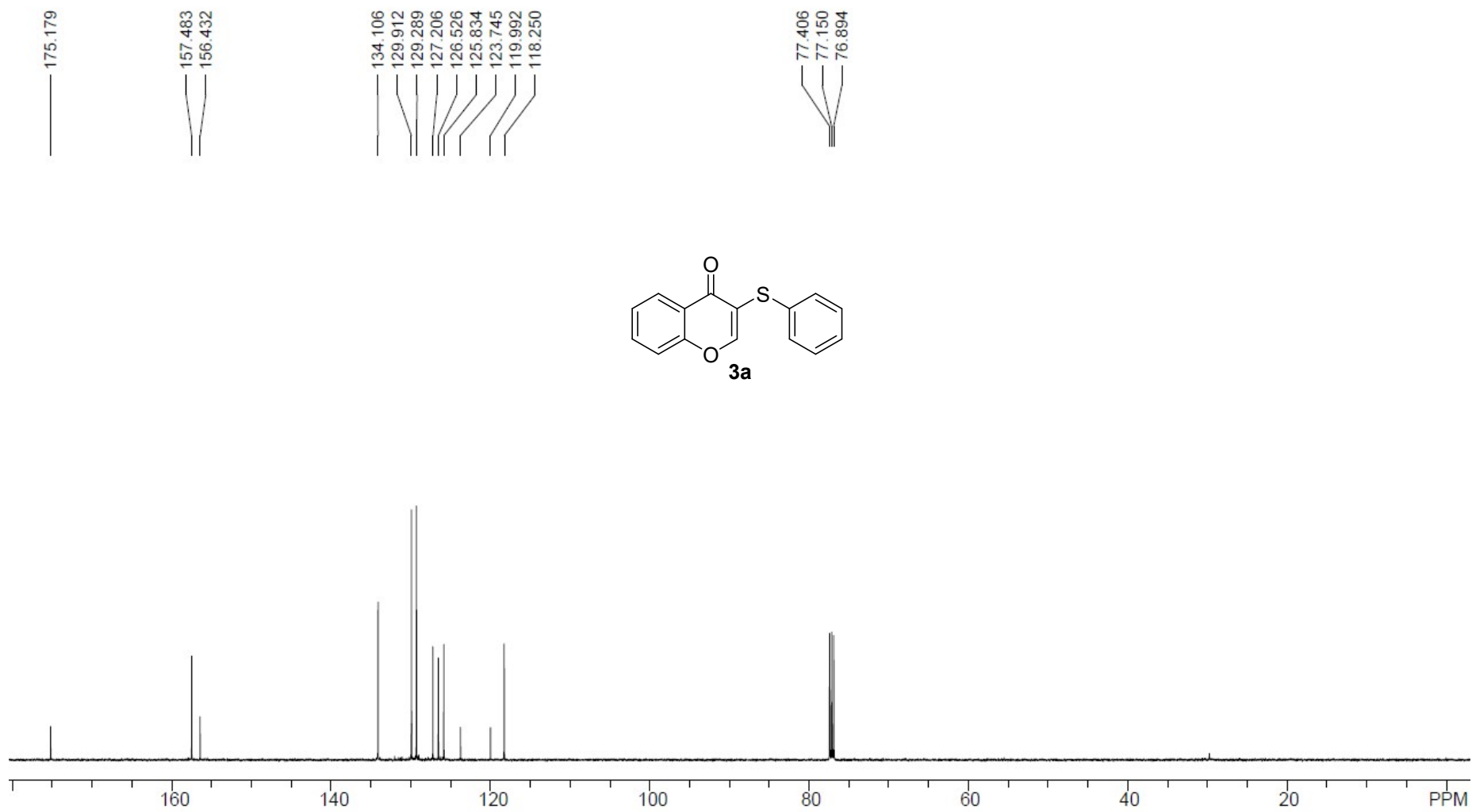
1. H.-Y. Liu, J.-R. Zhang, G.-B. Huang, Y.-H. Zhou, Y.-Y. Chen, Y.-L. Xu, *Adv. Synth. Catal.* **2021**, *363*, 1656- 1661.
2. Q. Tang, Z. Bian, W. Wu, J. Wang, P. Xie, C. U. Pittman, Jr., A. Zhou, *J. Org. Chem.* **2017**, *82*, 10617-10622.
3. J. Zhu, B. Xu, J. Yu, Y. Ren, J. Wang, P. Xie, C. U. Pittman, Jr., A. Zhou, *Org. Biomol. Chem.*, **2018**, *16*, 5999-6005.
4. H.-Y. Liu, J.-R. Zhang, G.-B. Huang, Y.-H. Zhou, Y.-Y. Chen, Y.-L. Xu, *Adv. Synth. Catal.* **2021**, *363*, 1656-1661
5. C. Ding, Y. Yu, Q. Yu, Z. Xie, Y. Zhou, J. Zhou, G. Liang, Z. Song, *ChemCatChem*, **2018**, *10*, 5397-5401.
6. P. Xu, Z. Zhong, H. Huang, W. Pan, Y. Zhang, A. Zhou, *Tetrahedron*, **2022**, *124*, 133018.
7. T. Guo, *Synthetic Commun.*, **2017**, *47*, 2053–2061
8. C. V. Doerner, J. S. S. Neto, C. R. Cabreira, S. Saba, L. P. Sandjo, J. Rafique, A. L. Braga, F. F. de Assis, *New J. Chem.*, **2023**, *47*, 5598–5602

5 Copies of ^1H and ^{13}C NMR Spectra

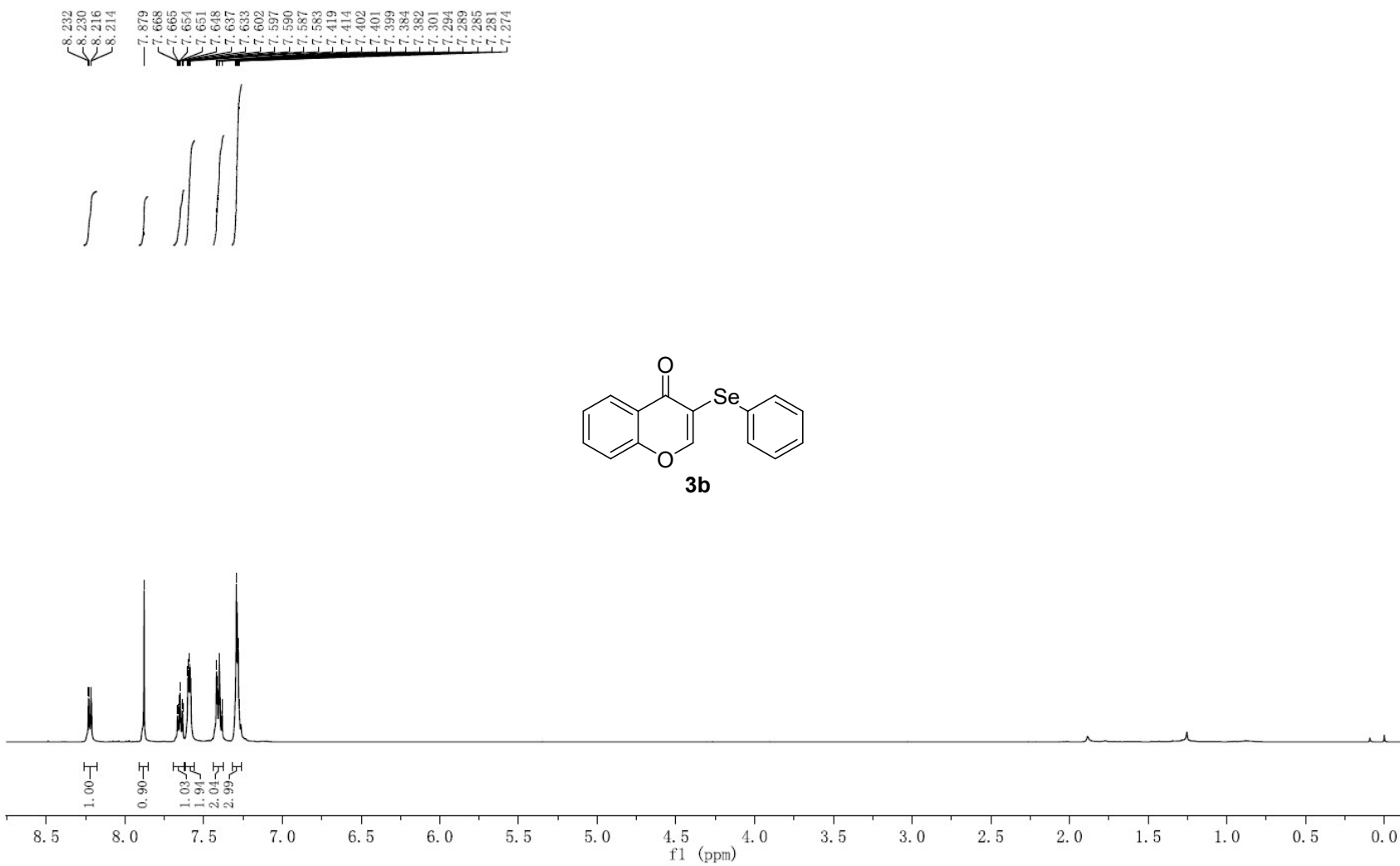
^1H NMR (500 MHz, CDCl_3) of **3a**



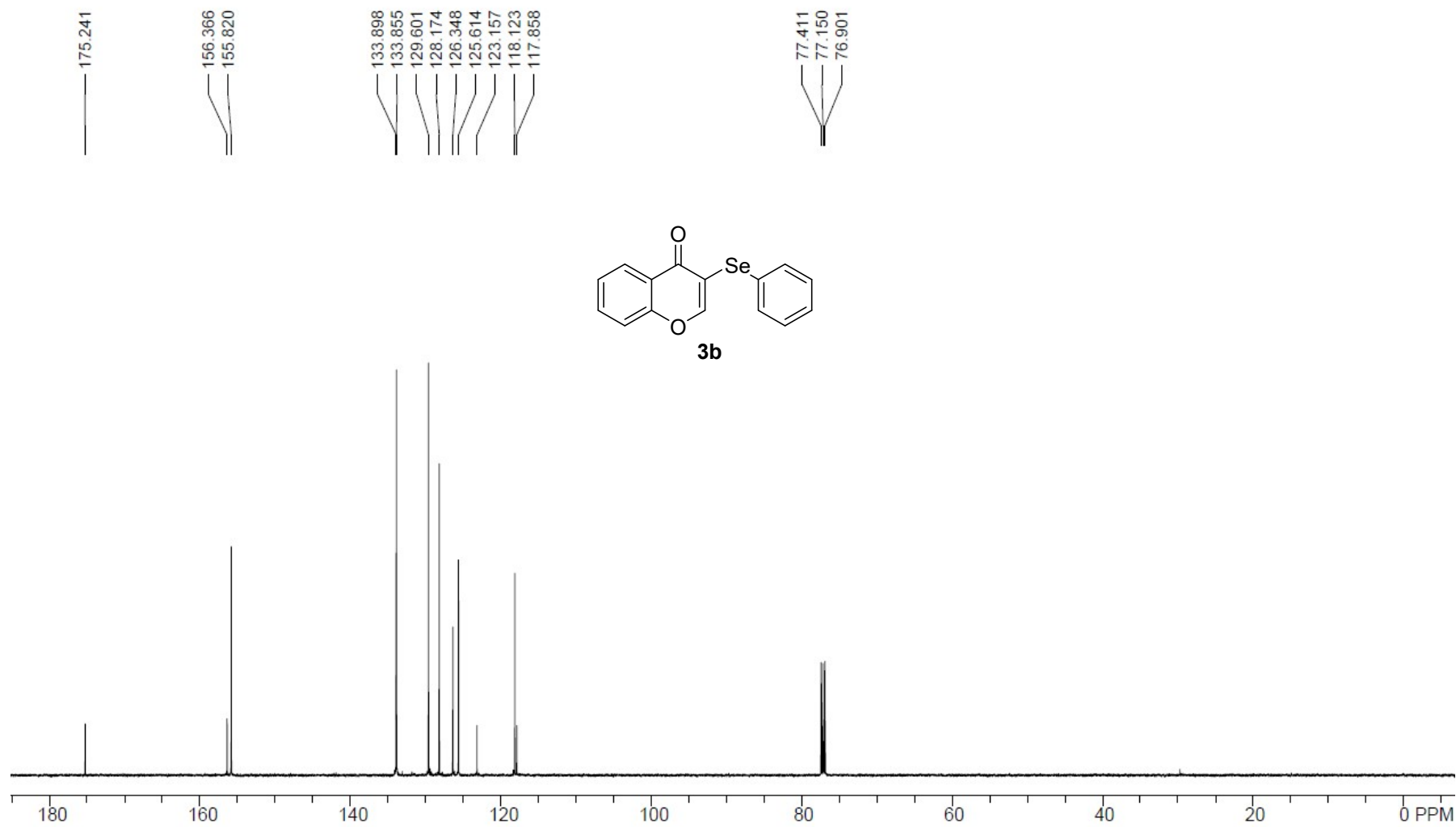
^{13}C NMR (126 MHz, CDCl_3) of **3a**



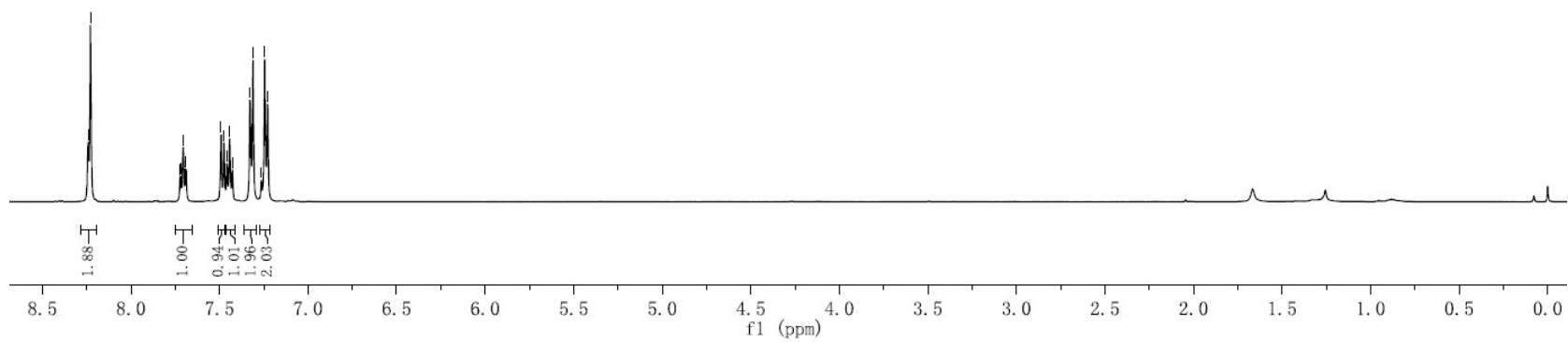
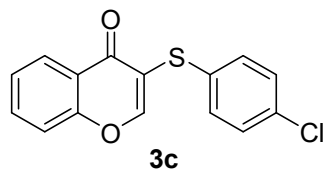
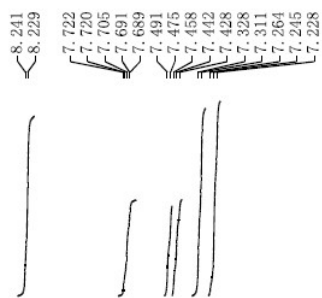
^1H NMR (500 MHz, CDCl_3) of **3b**



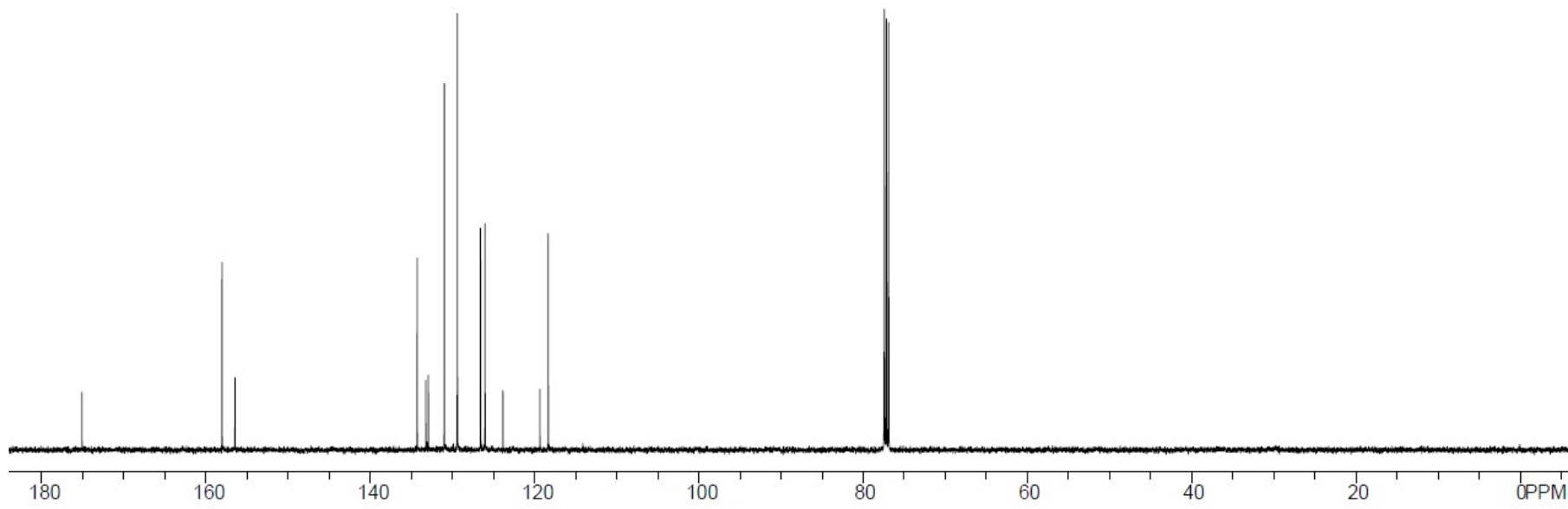
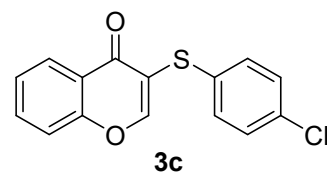
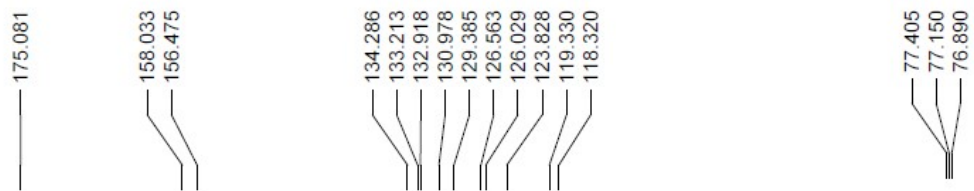
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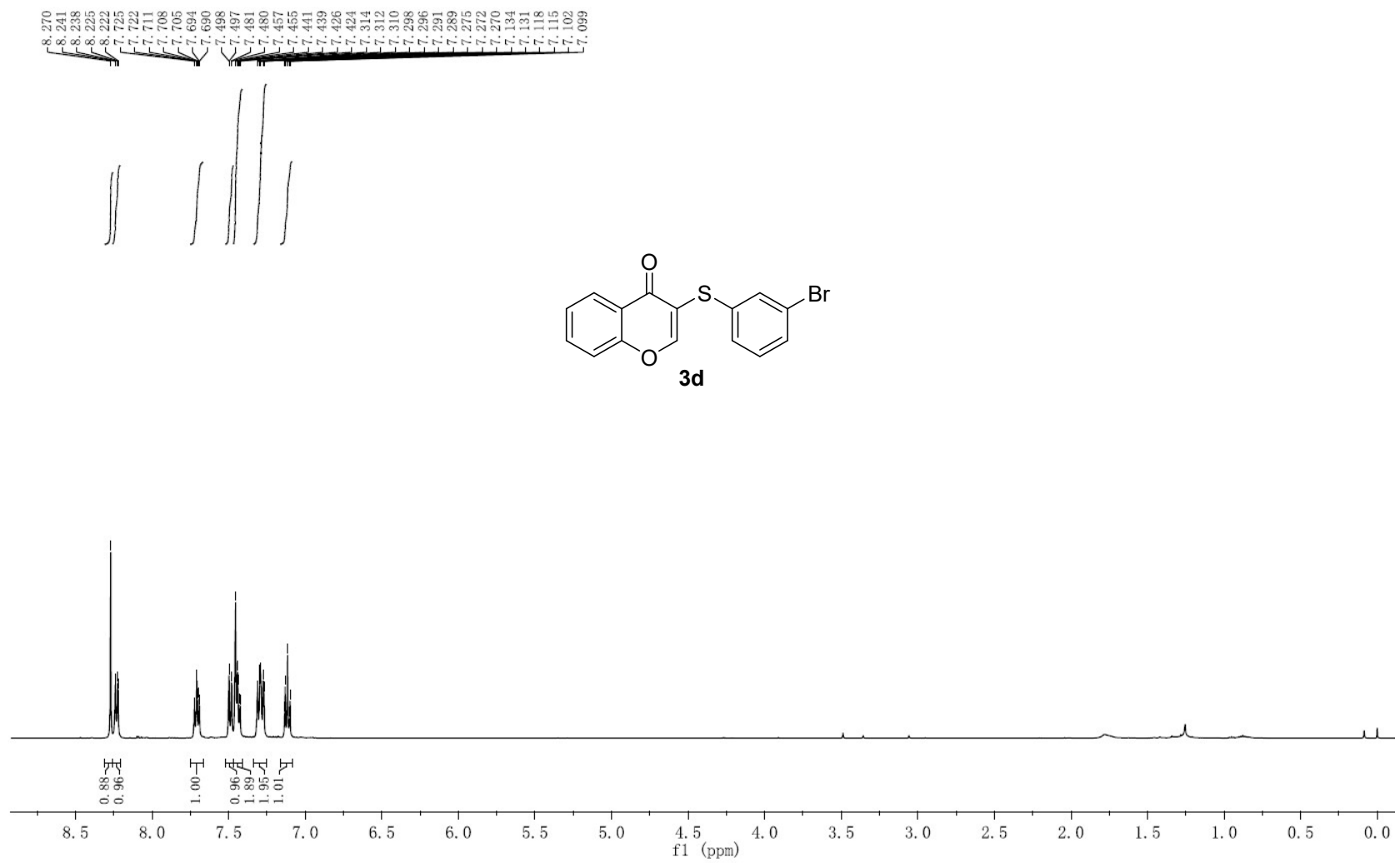
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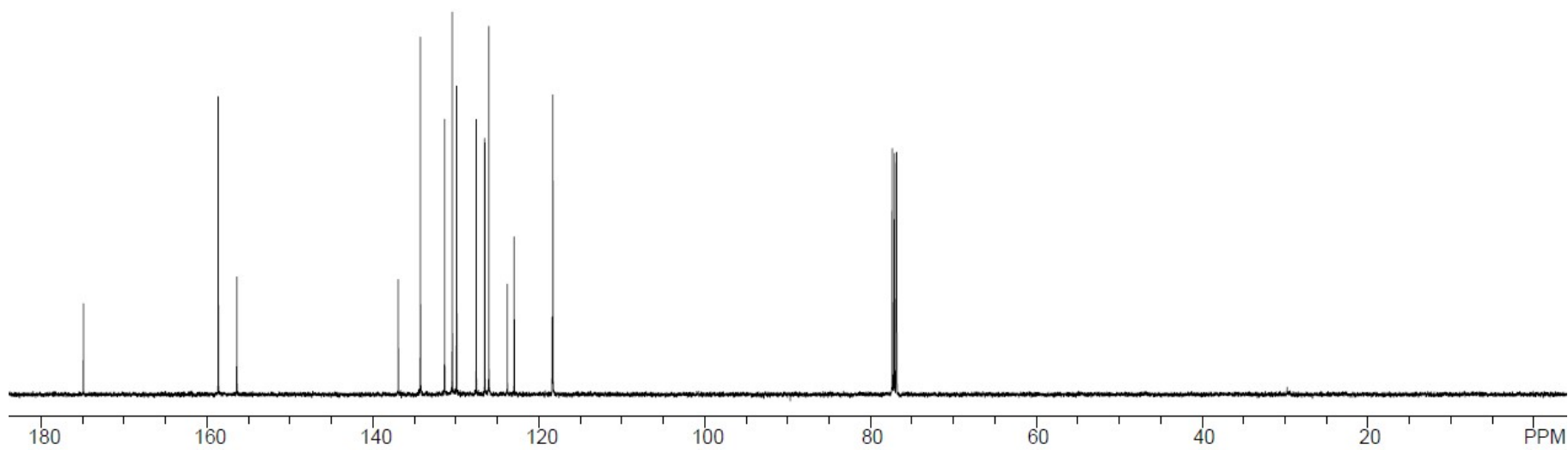
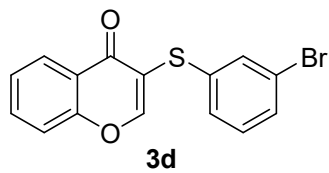
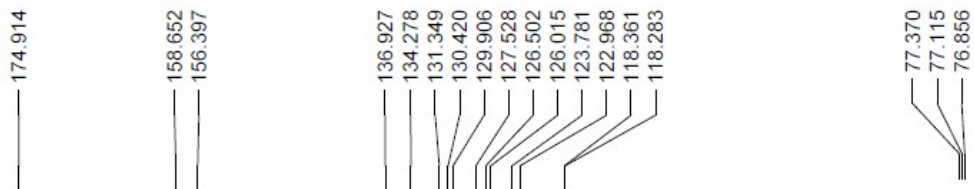
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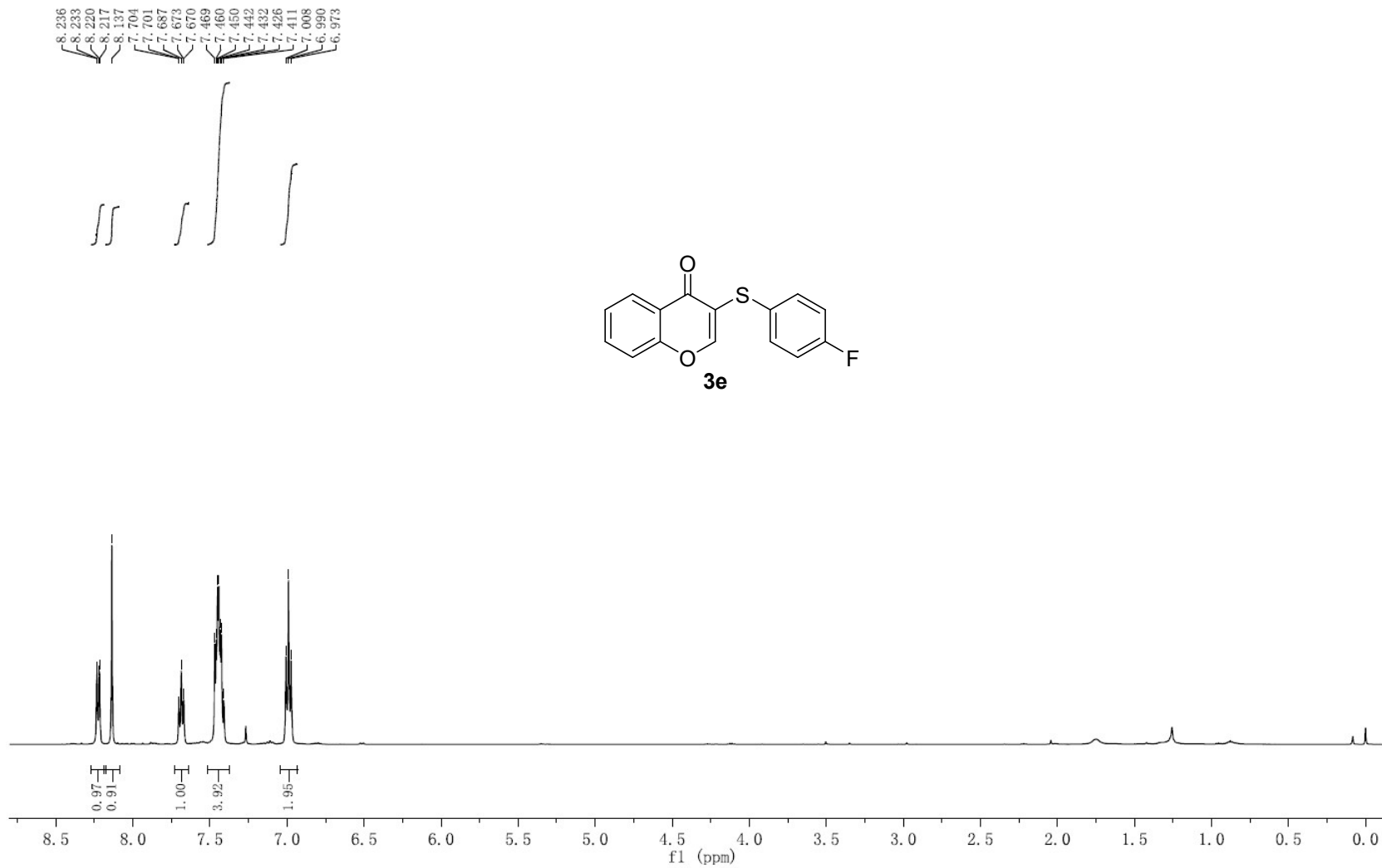
^1H NMR (500 MHz, CDCl_3) of **3d**



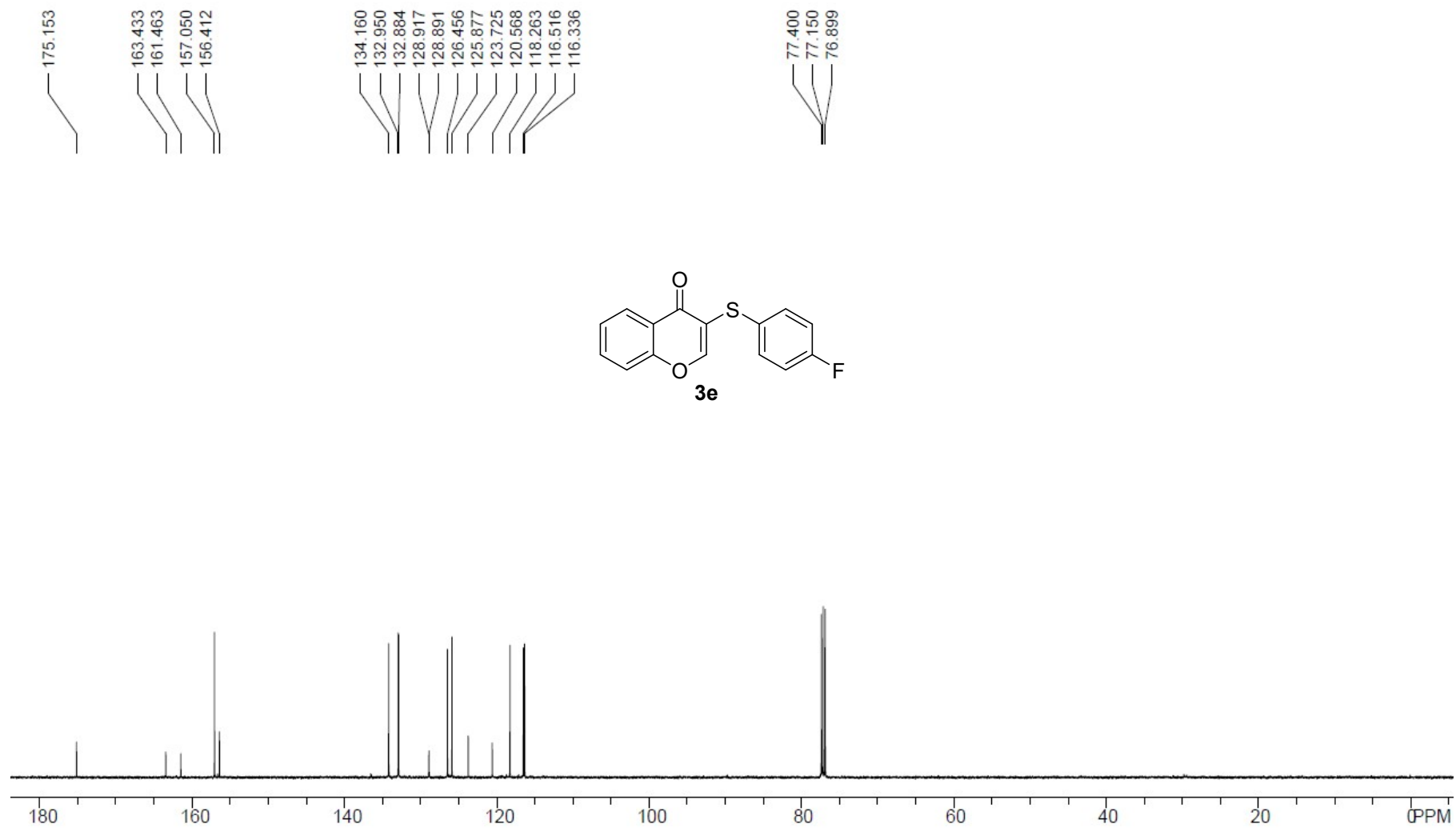
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^1H NMR (500 MHz, CDCl_3) of **3e**

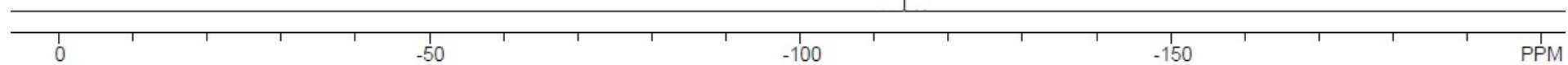
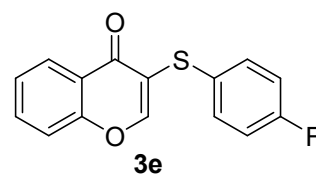


^{13}C NMR (126 MHz, CDCl_3) of **3e**

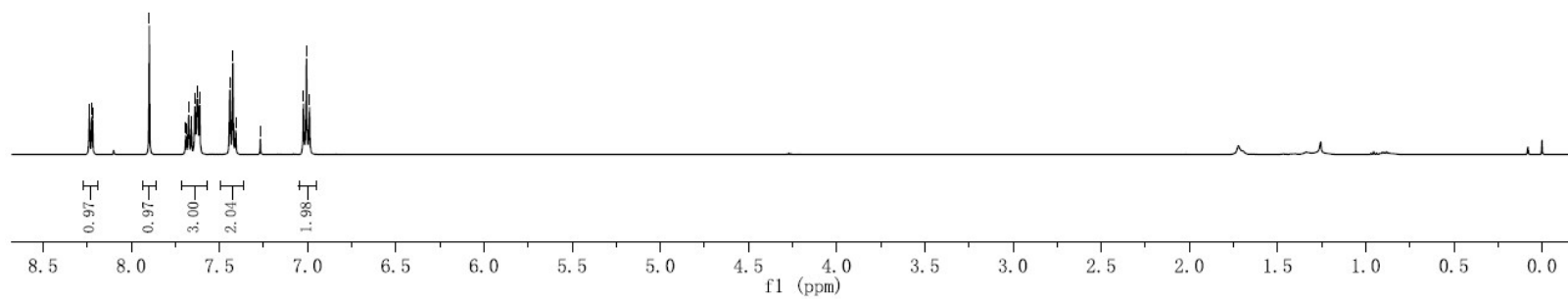
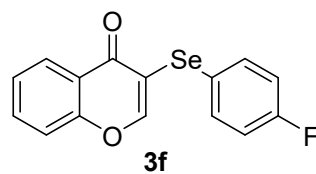
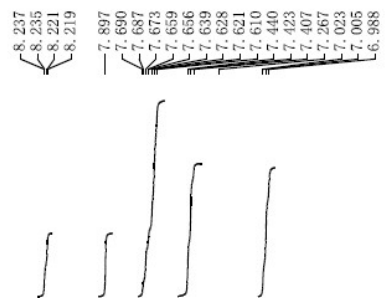


^{19}F NMR (471 MHz, CDCl_3) of **3e**

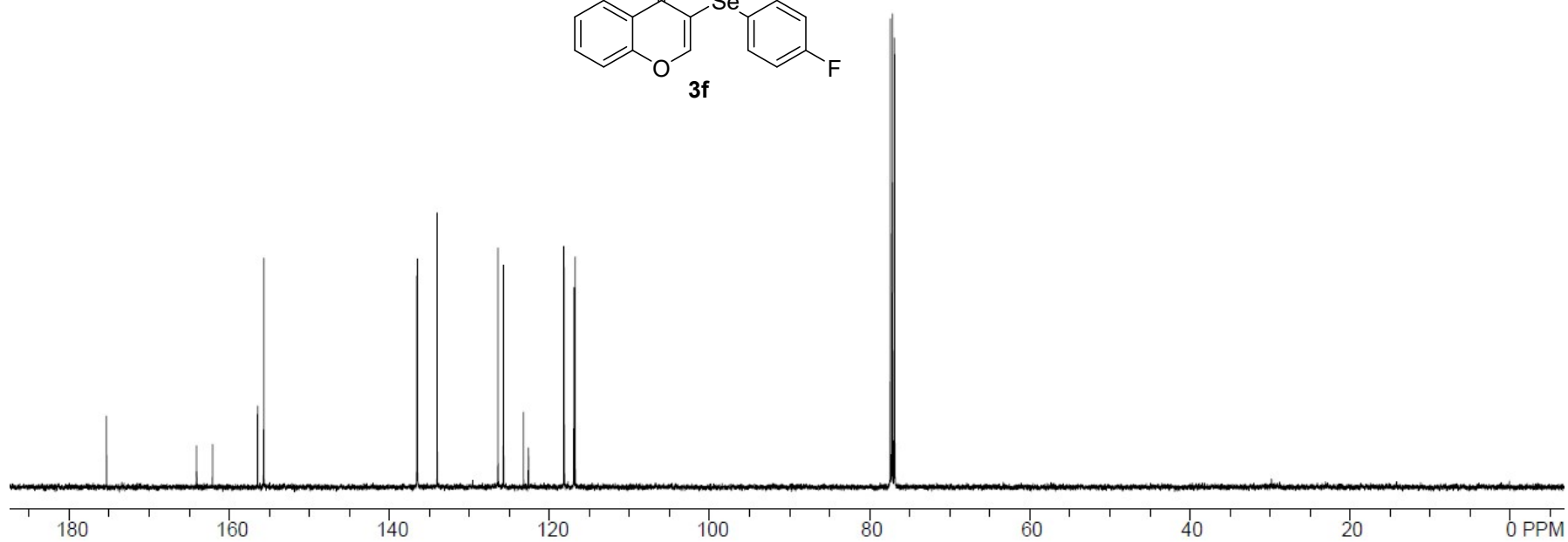
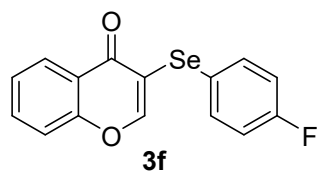
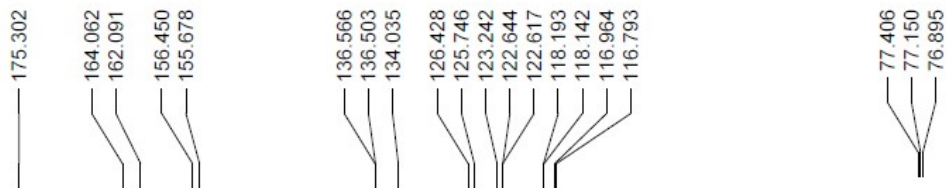
114.049



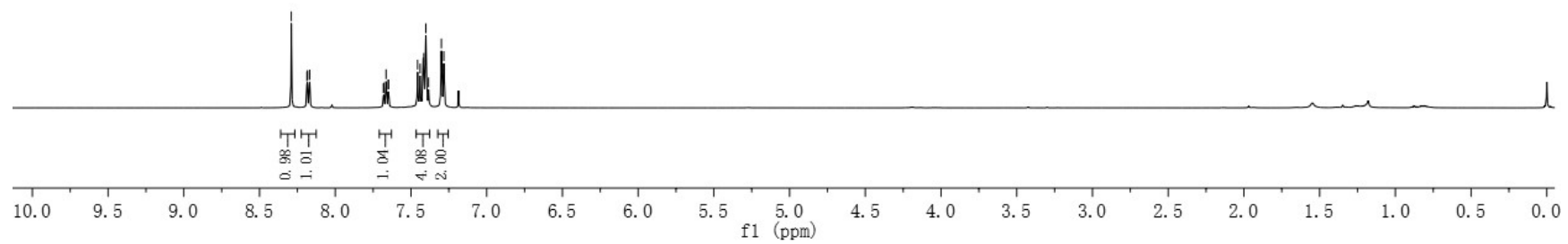
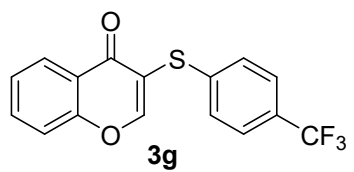
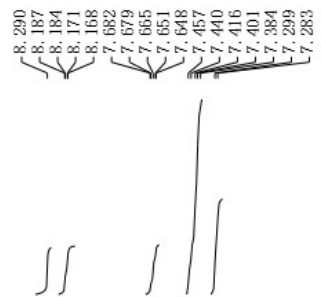
^1H NMR (500 MHz, CDCl_3) of **3f**



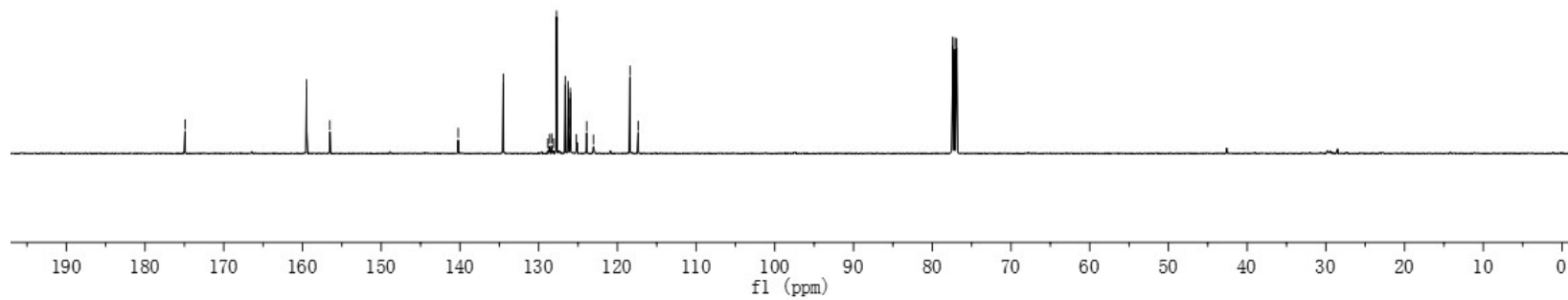
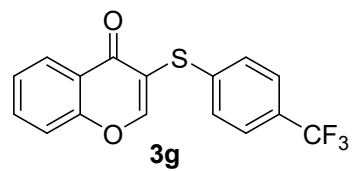
^{13}C NMR (126 MHz, CDCl_3) of **3f**



^1H NMR (500 MHz, CDCl_3) of **3g**

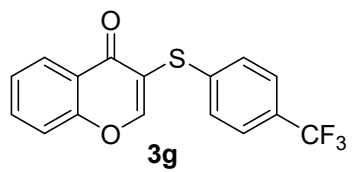


^{13}C NMR (126 MHz, CDCl_3) of **3g**



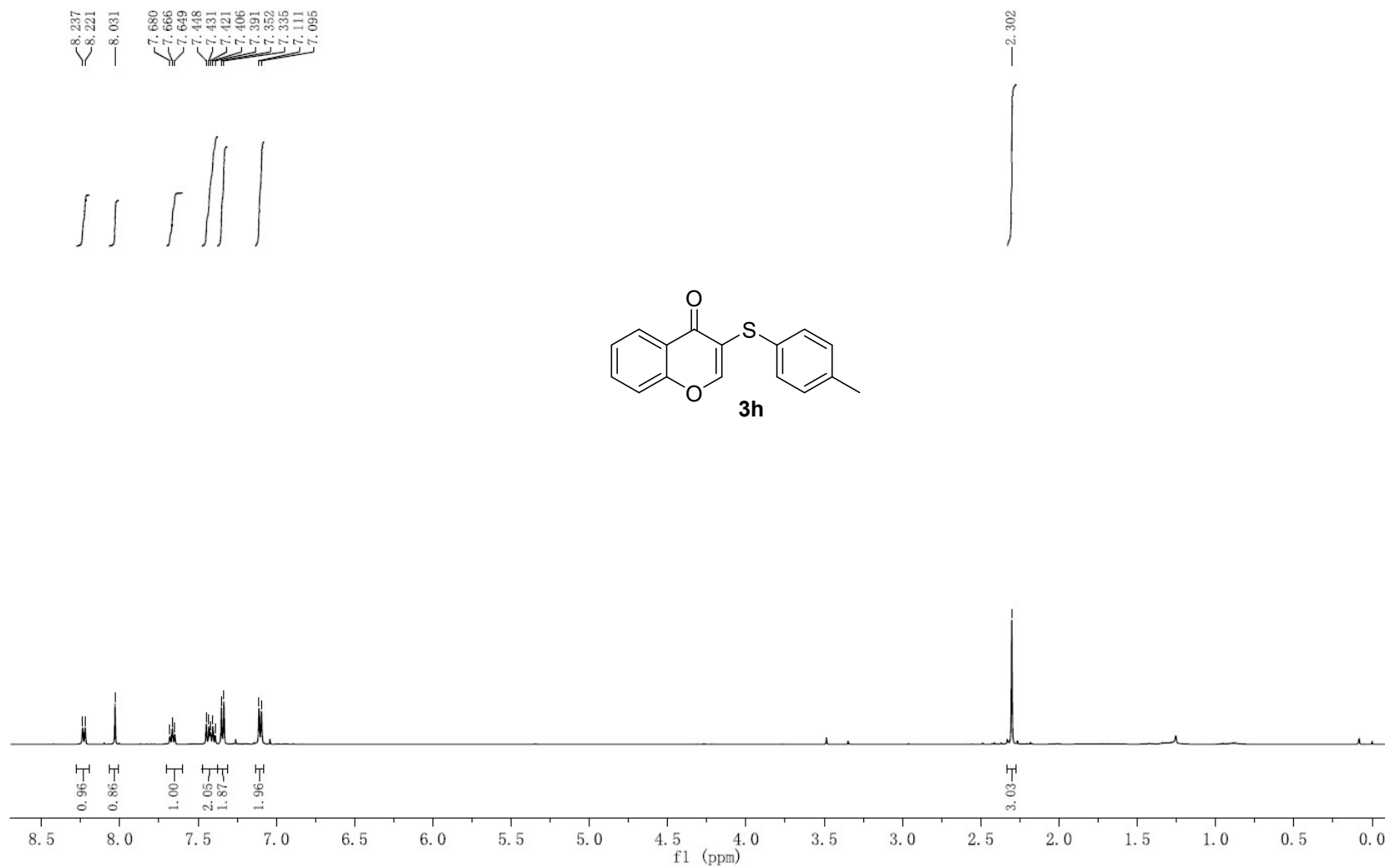
^{19}F NMR (471 MHz, CDCl_3) of **3g**

62.496

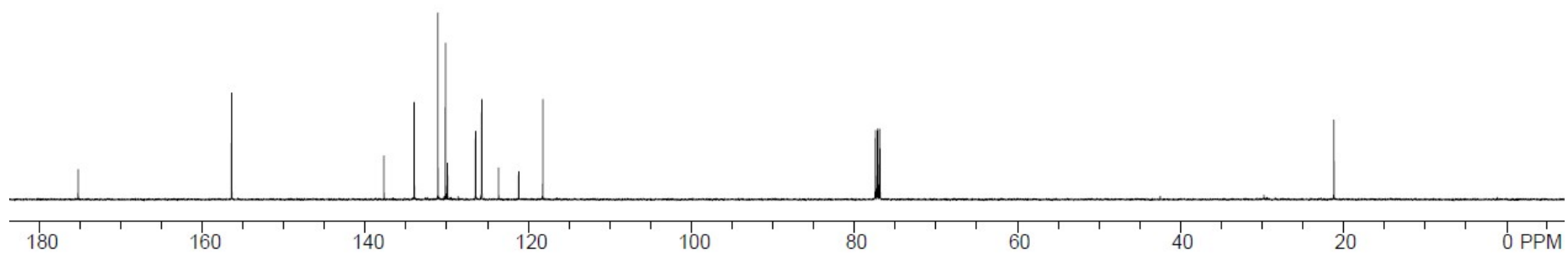
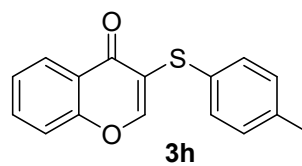


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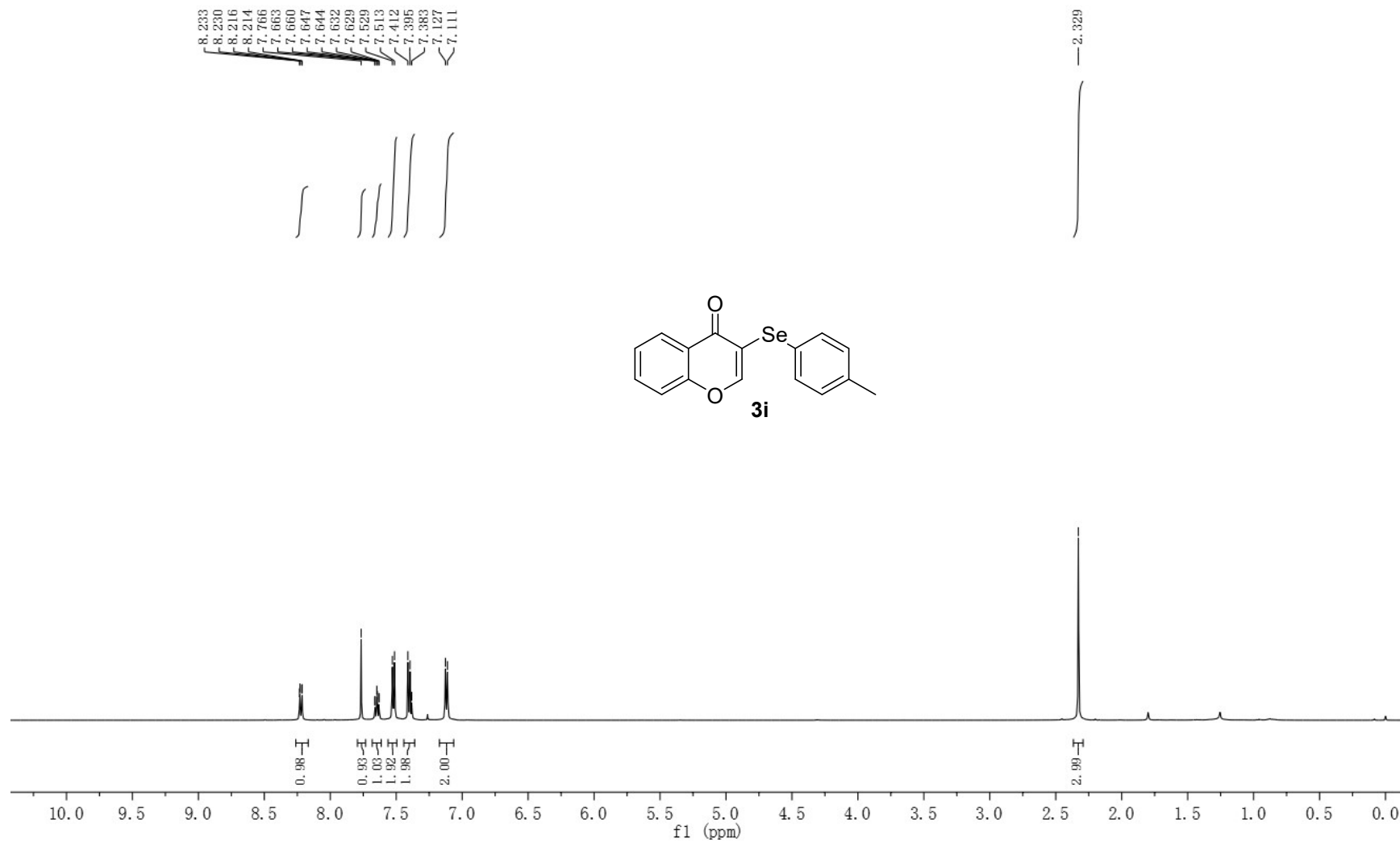
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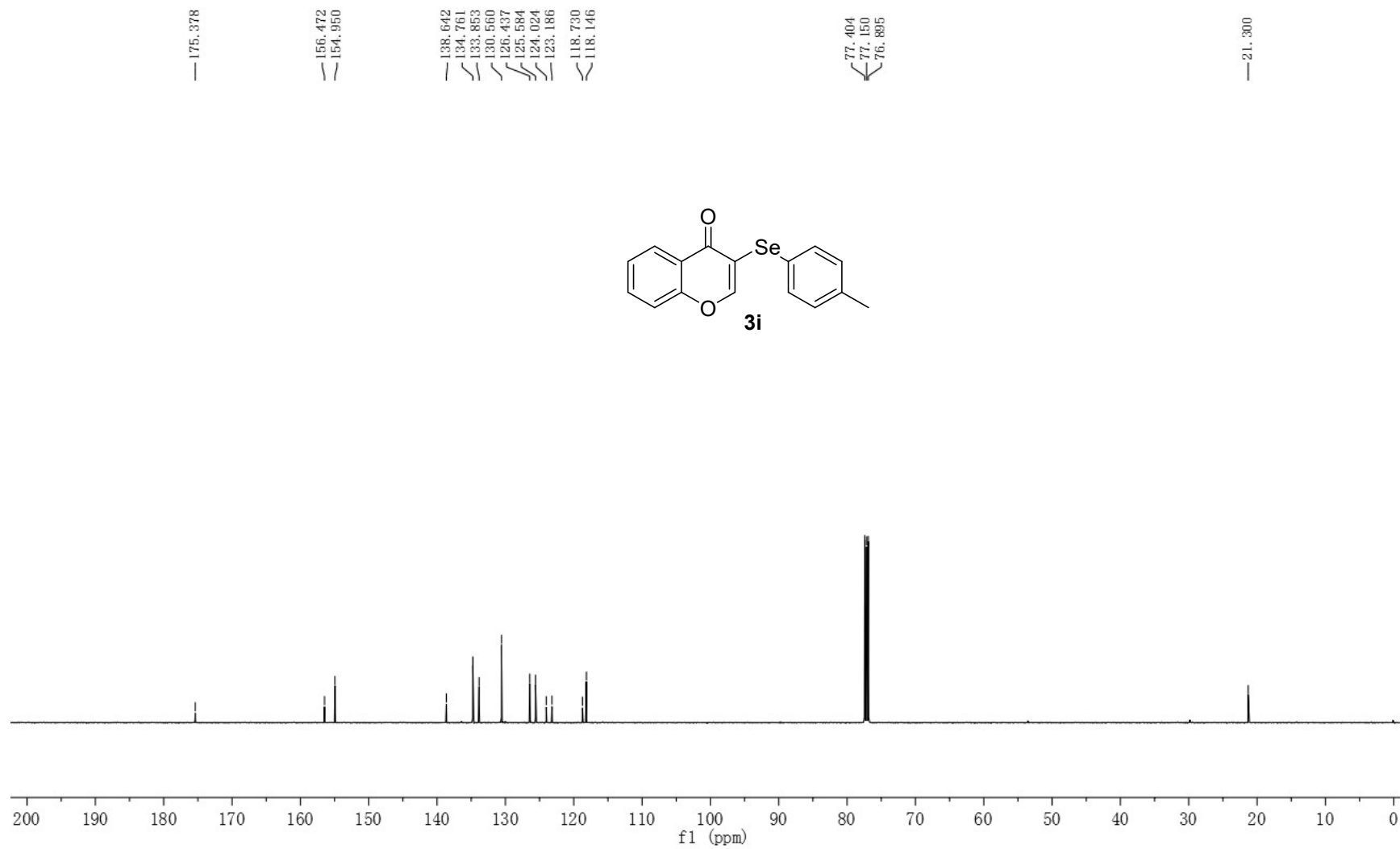
^{13}C NMR (126 MHz, CDCl_3) of **3h**



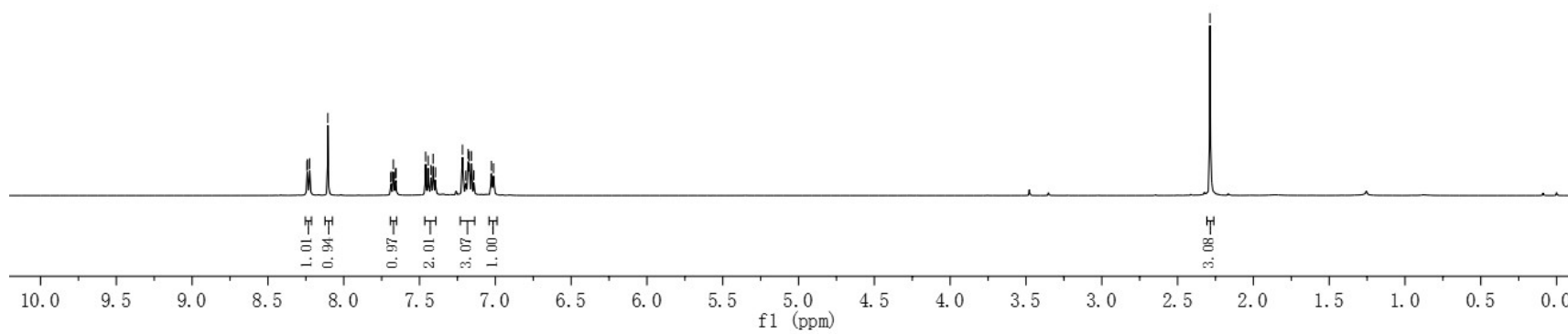
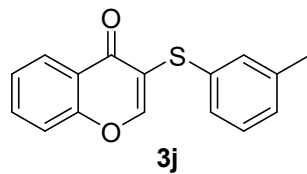
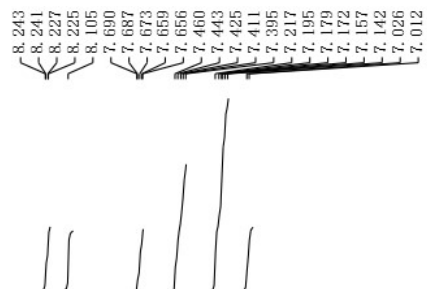
^1H NMR (500 MHz, CDCl_3) of **3i**



^{13}C NMR (126 MHz, CDCl_3) of **3i**



^1H NMR (500 MHz, CDCl_3) of **3j**



¹³C NMR (126 MHz, CDCl₃) of **3j**

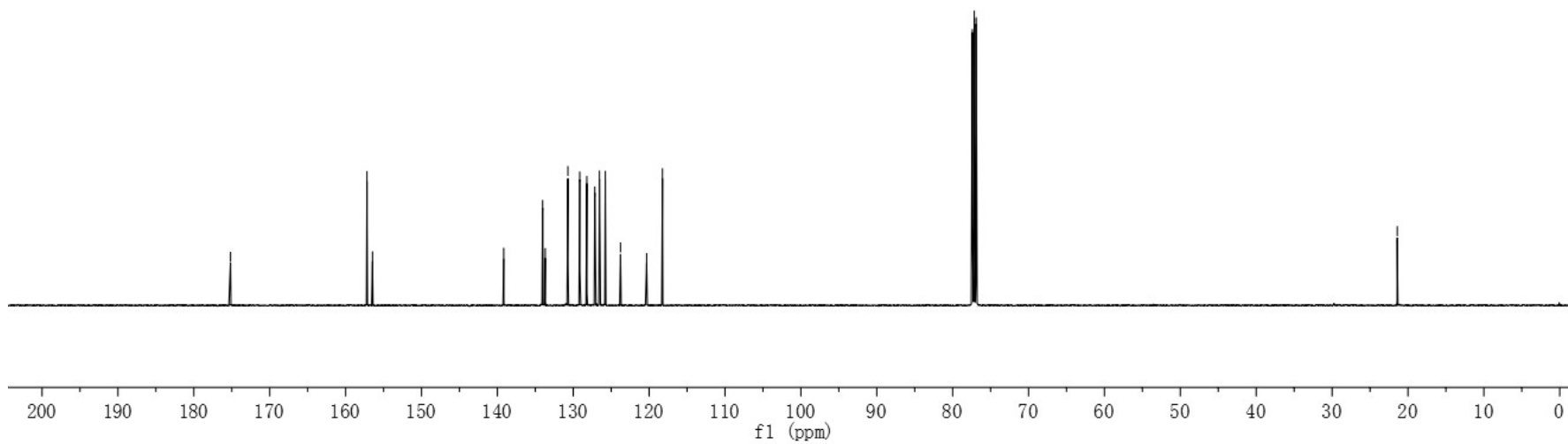
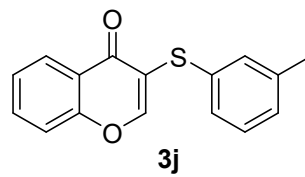
175.166

157.184
156.446

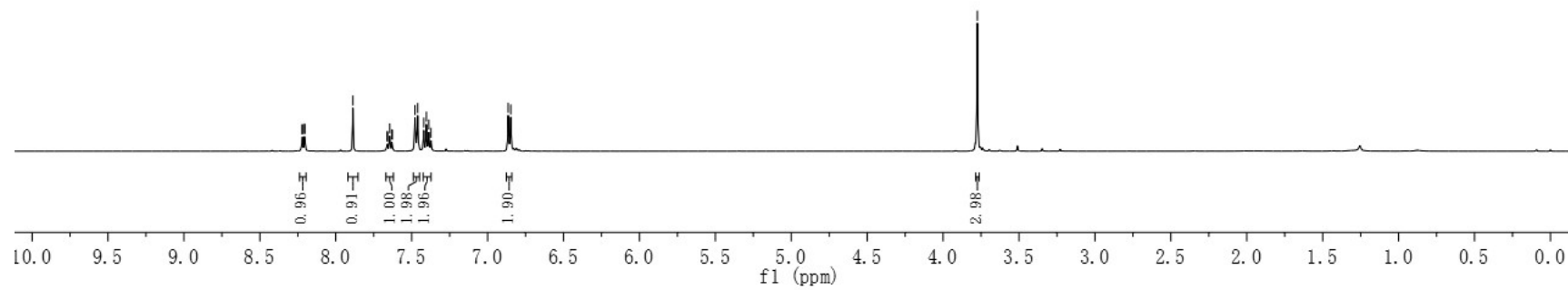
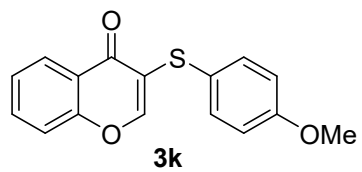
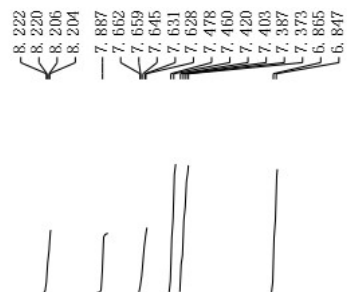
139.160
134.085
133.690
130.701
129.141
128.210
127.161
126.549
125.776
123.761
120.324
118.243

77.404
77.150
76.896

21.407



¹H NMR (500 MHz, CDCl₃) of **3k**



^{13}C NMR (126 MHz, CDCl_3) of **3k**

175.101

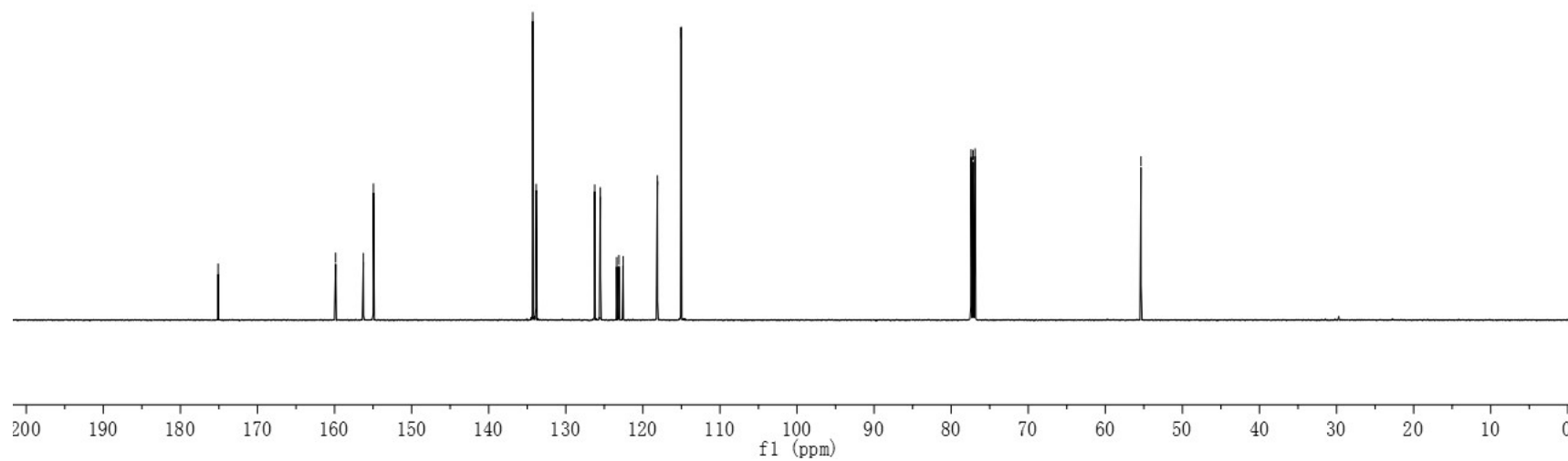
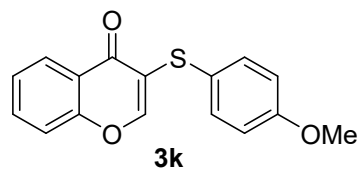
159.863
156.262
154.952

134.287
133.838

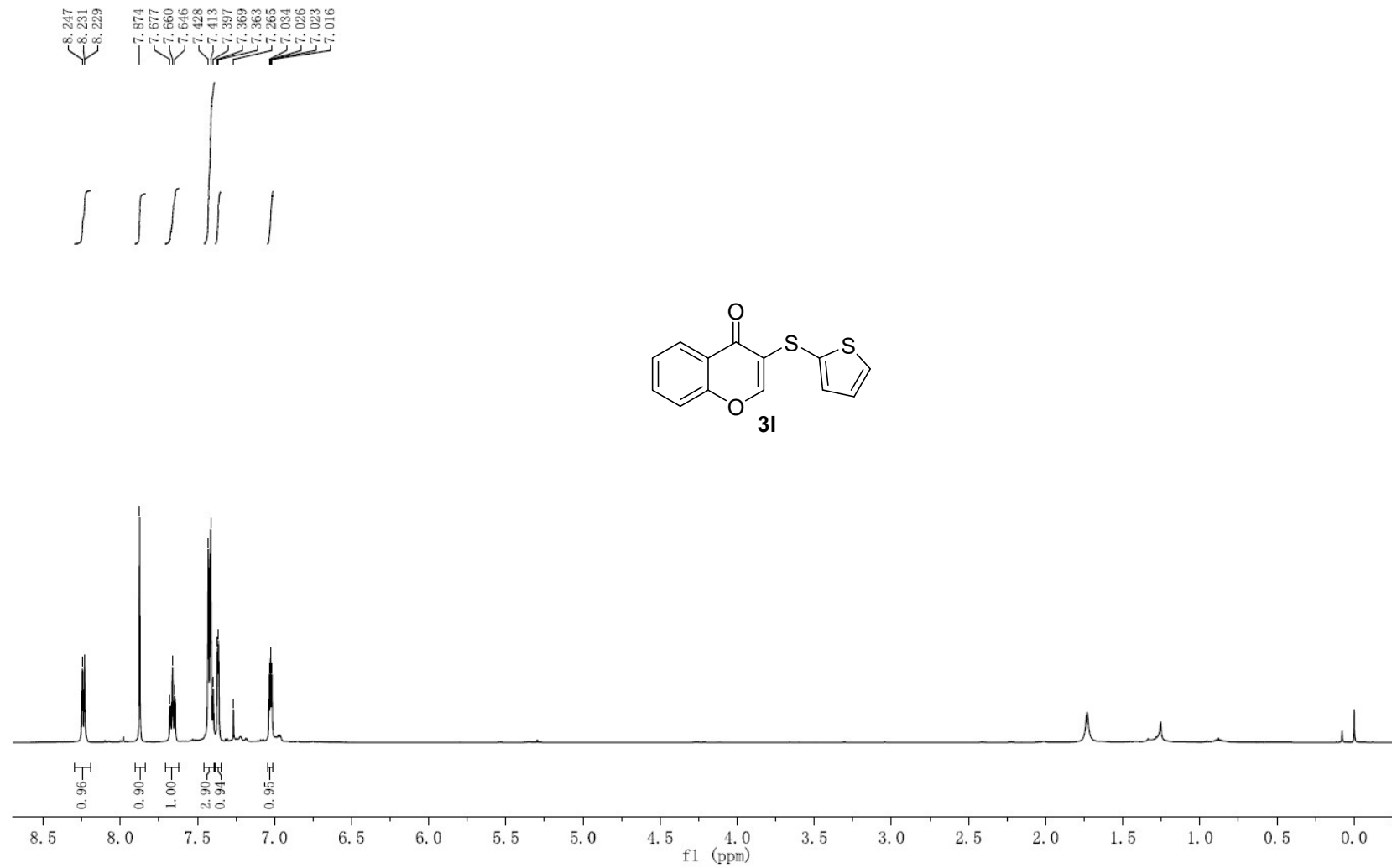
126.247
125.517
123.405
123.110
122.564
118.116
115.034

77.405
77.150
76.895

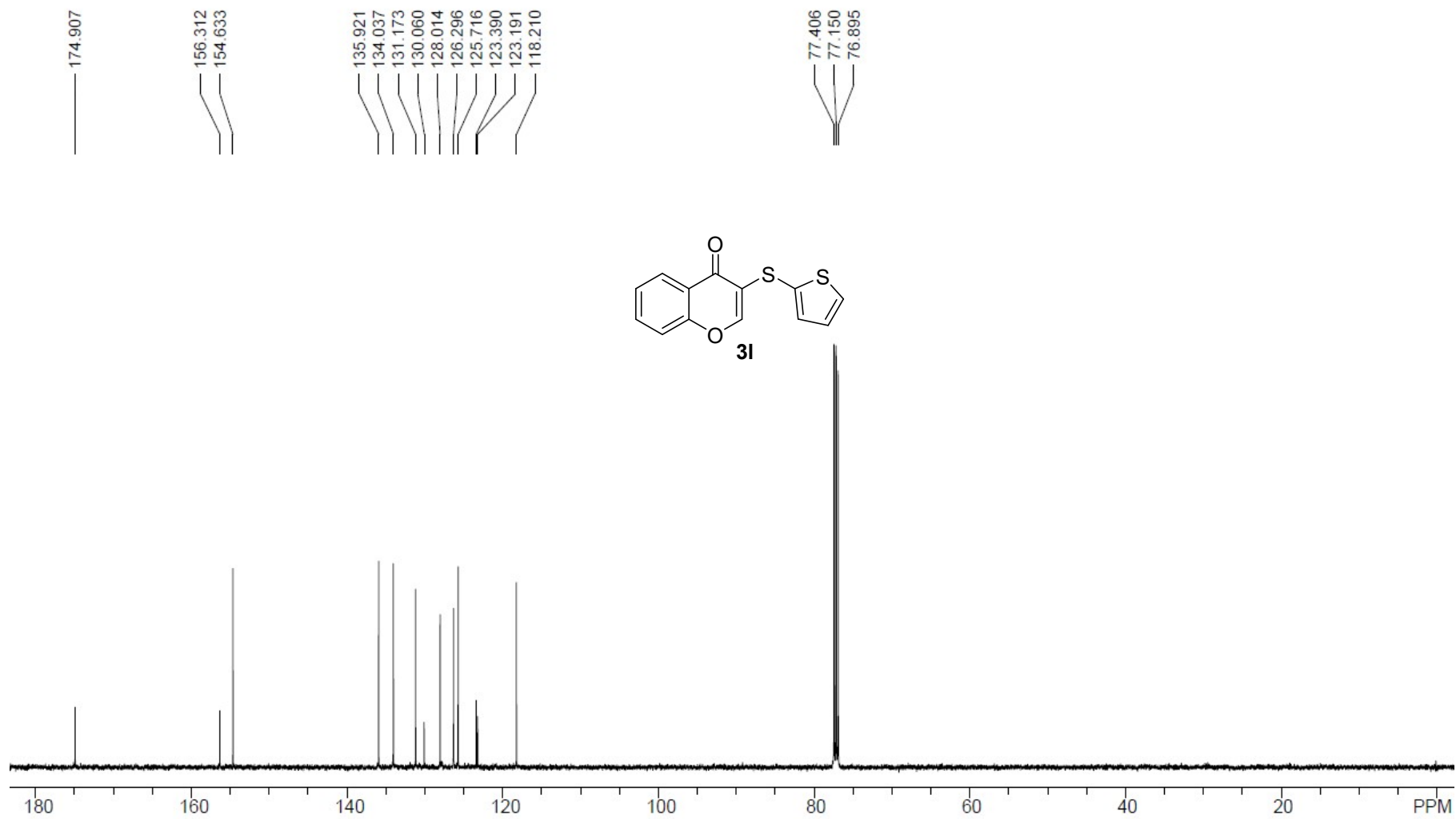
55.379



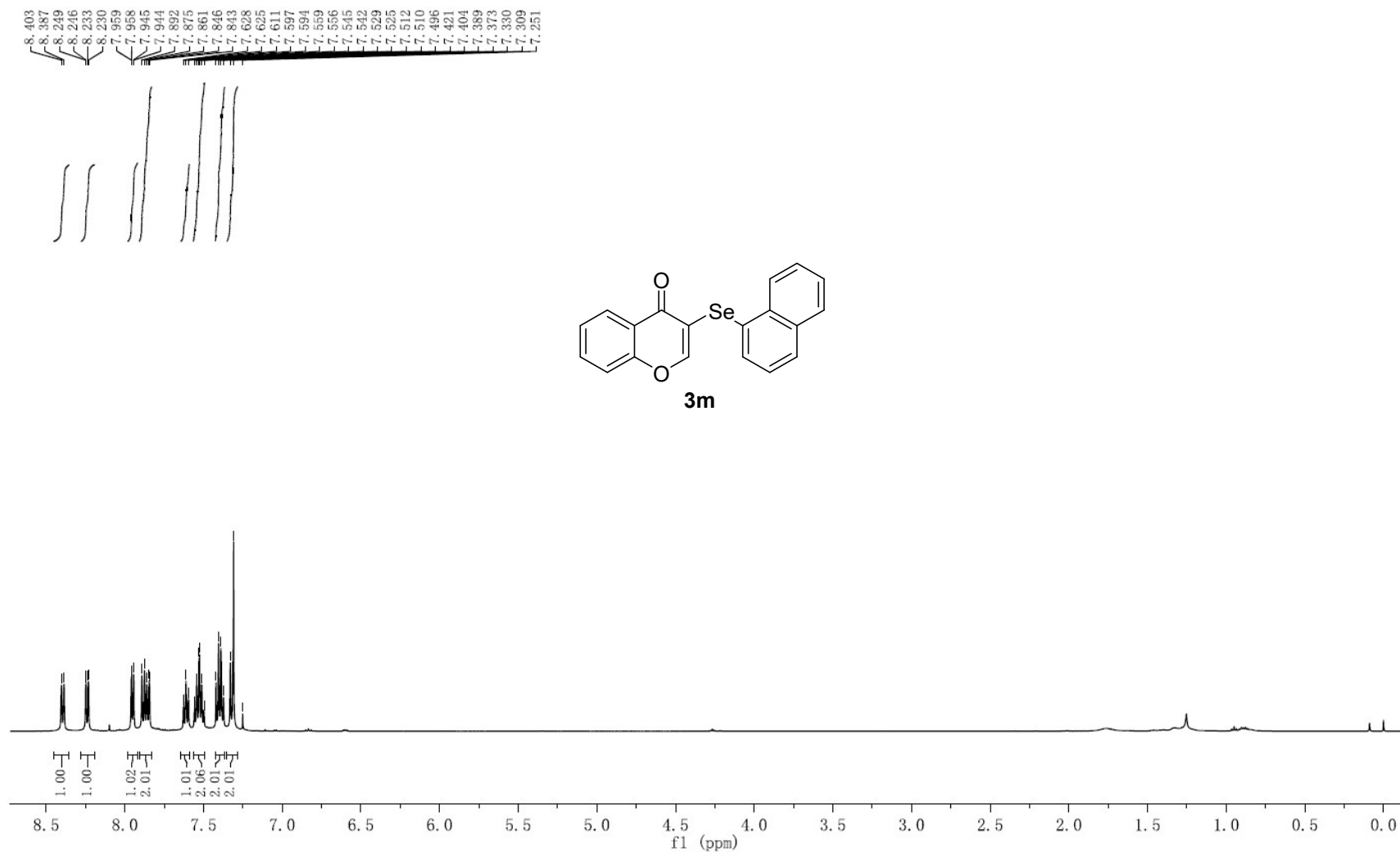
^1H NMR (500 MHz, CDCl_3) of **31**



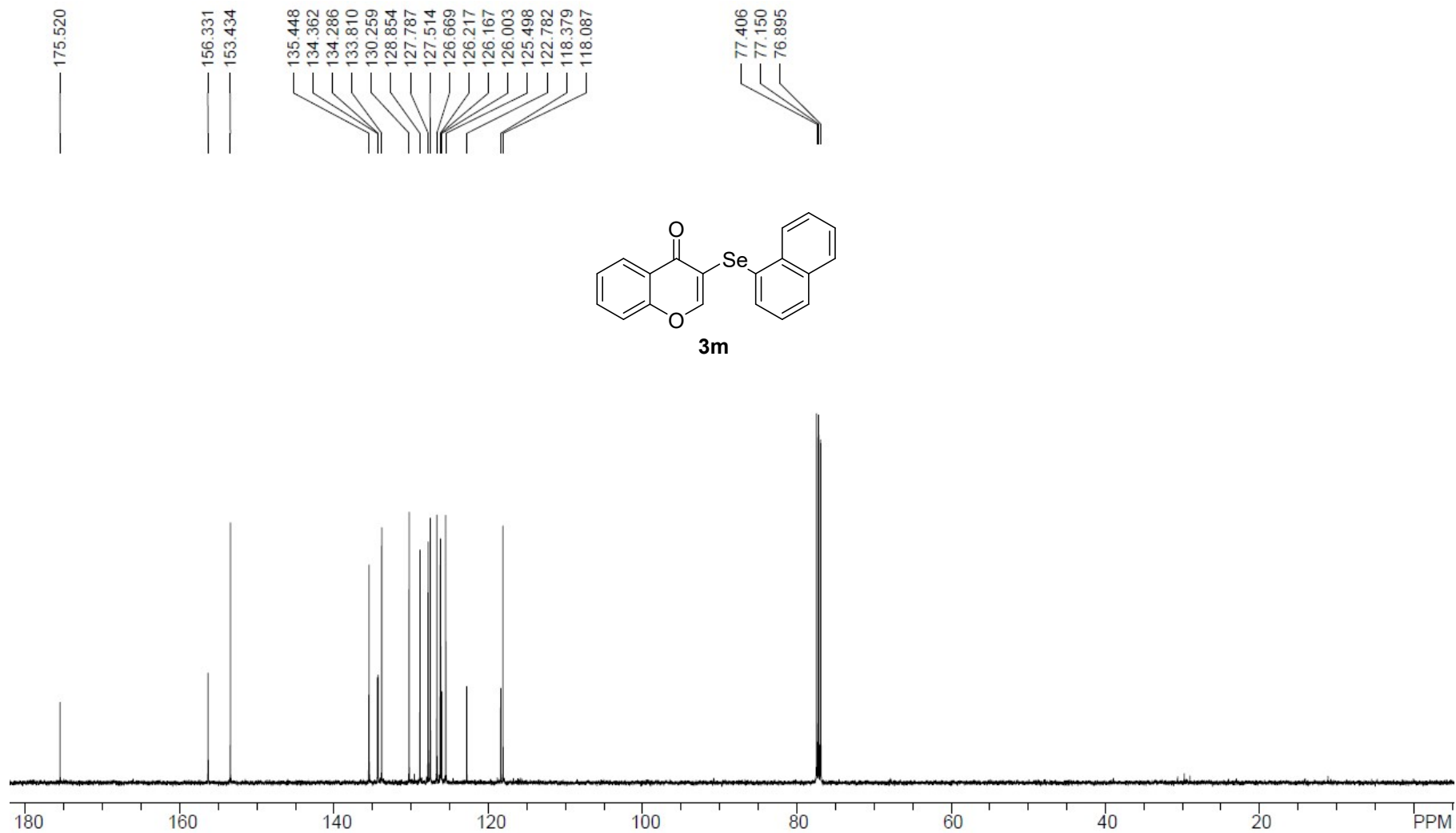
^{13}C NMR (126 MHz, CDCl_3) of **3I**



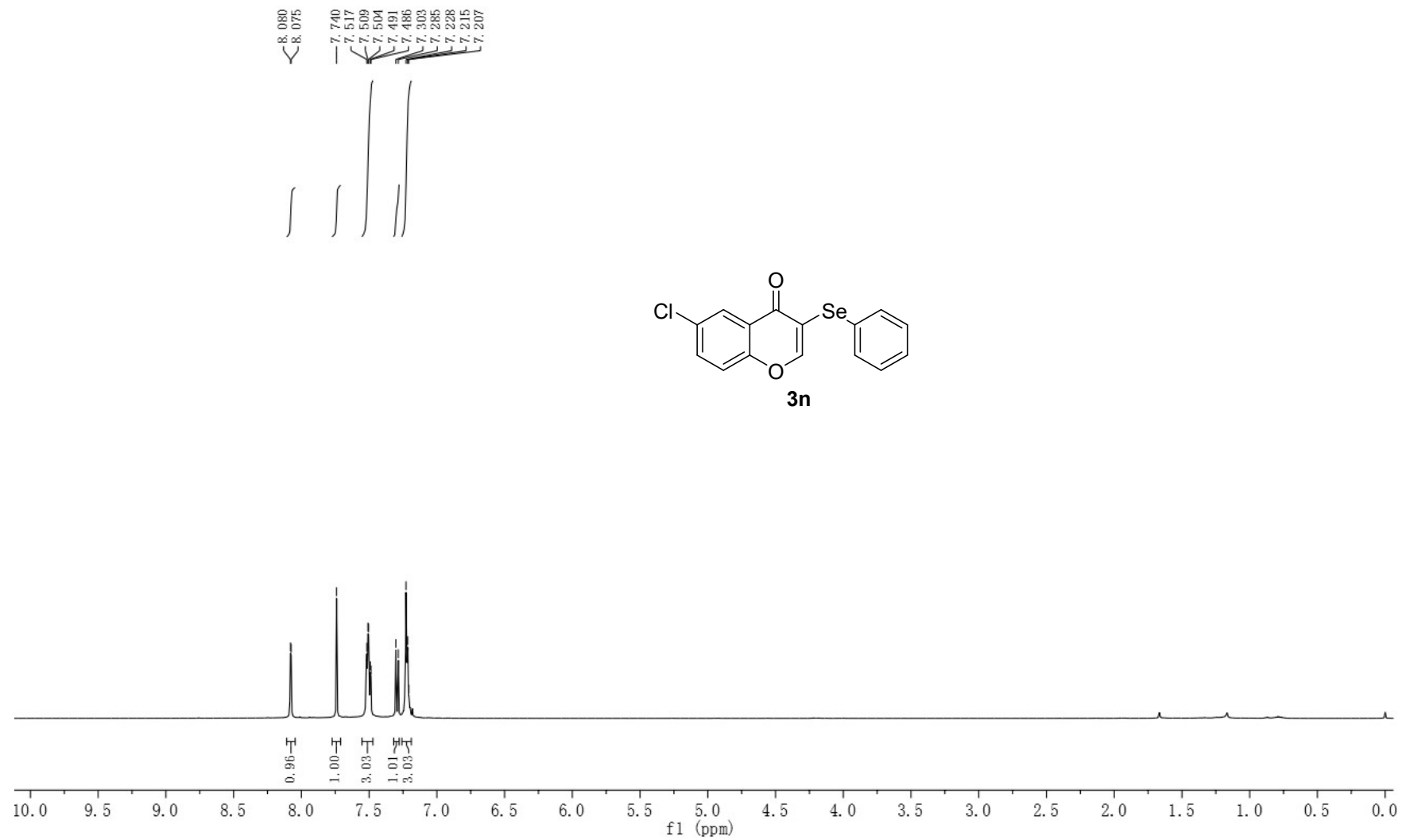
^1H NMR (500 MHz, CDCl_3) of **3m**



^{13}C NMR (126 MHz, CDCl_3) of **3m**



^1H NMR (500 MHz, CDCl_3) of **3n**



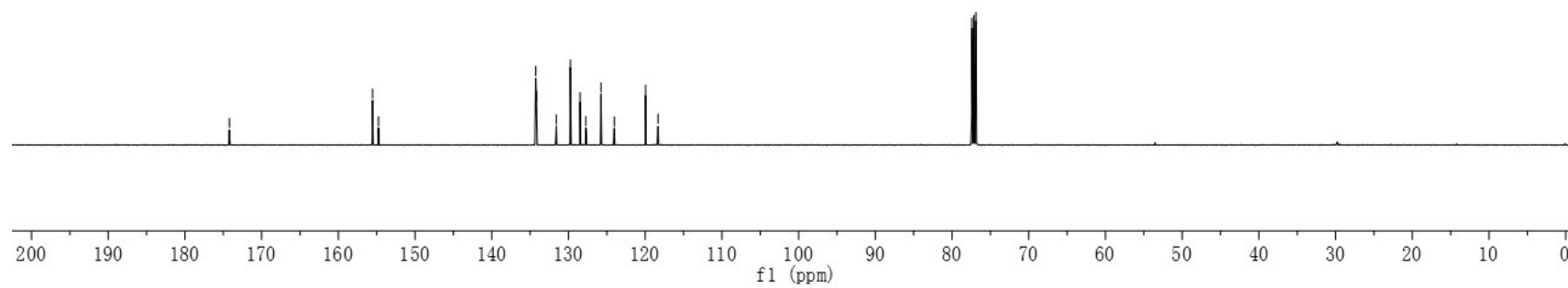
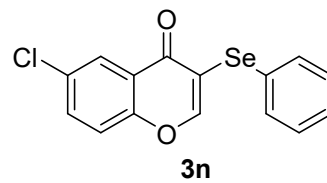
^{13}C NMR (126 MHz, CDCl_3) of **3n**

174.20

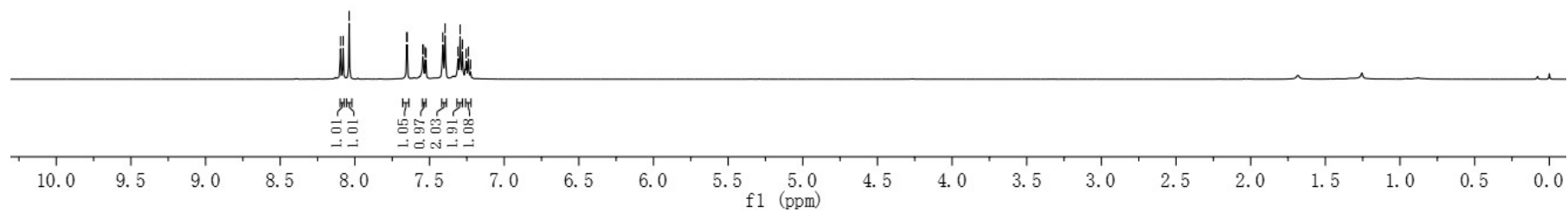
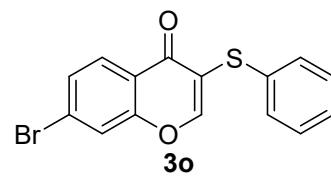
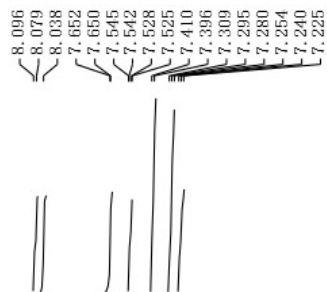
155.54
154.77

134.26
134.17
131.58
129.75
128.49
127.74
126.76
124.03
119.95
118.33

77.40
77.15
76.90



^1H NMR (500 MHz, CDCl_3) of **3o**



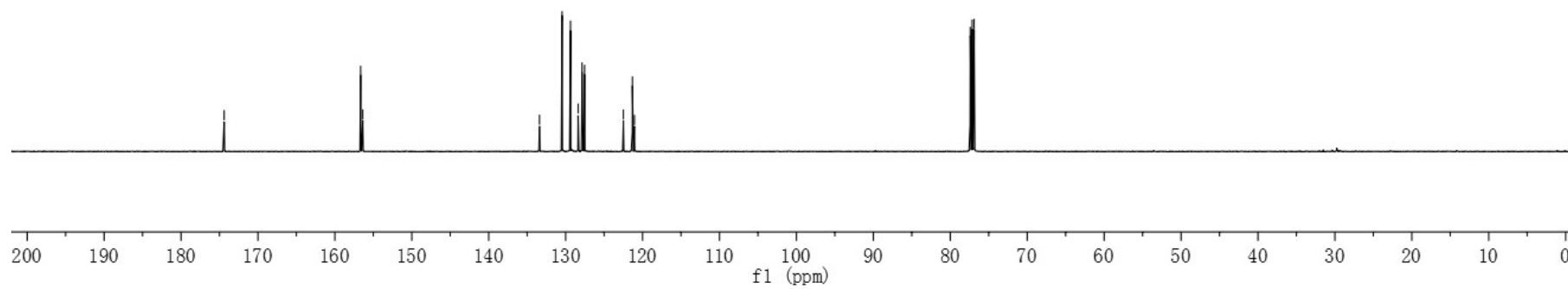
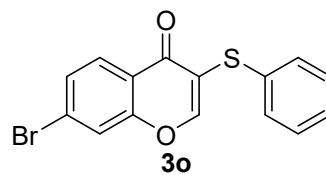
^{13}C NMR (126 MHz, CDCl_3) of **3o**

174.387

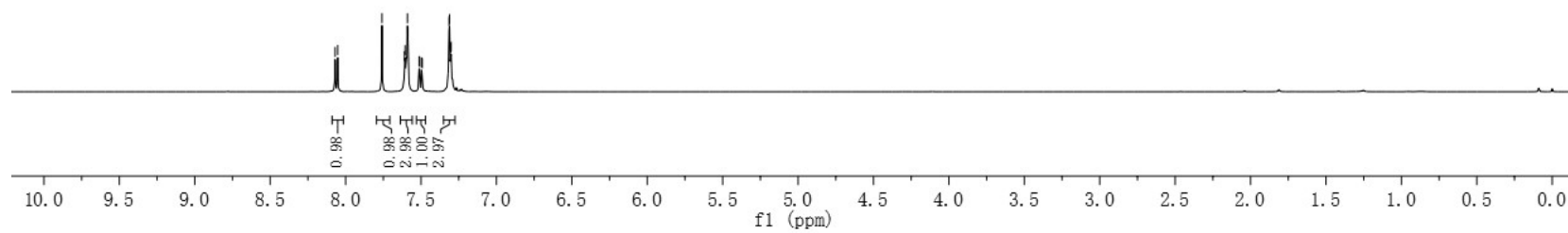
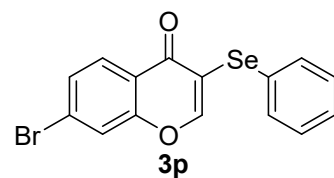
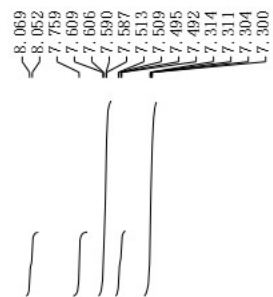
156.650
156.376

133.397
130.461
129.415
129.368
128.375
127.870
127.538
122.496
121.317
121.057

77.404
77.150
76.896



^1H NMR (500 MHz, CDCl_3) of **3p**



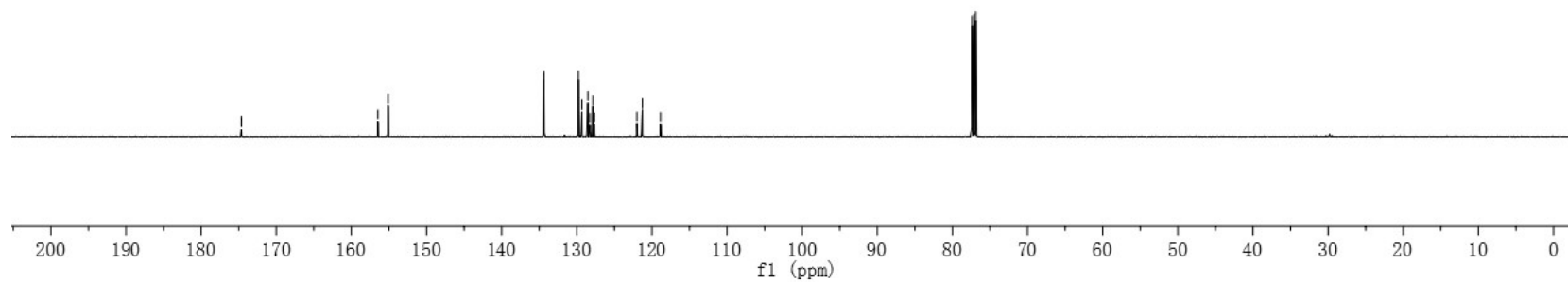
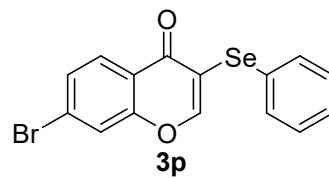
^{13}C NMR (126 MHz, CDCl_3) of **3p**

174.649

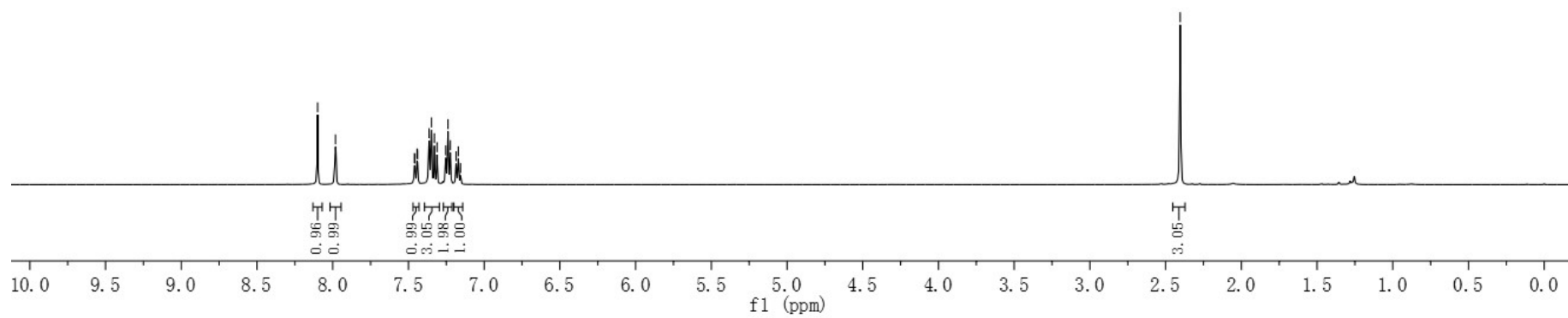
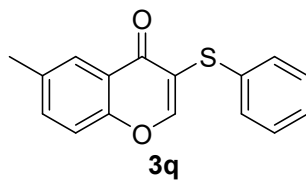
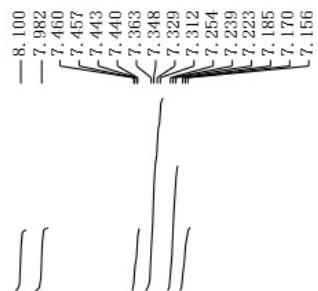
156.467
155.117

134.368
129.775
129.323
128.532
128.252
127.861
127.636
122.004
121.259
118.848

77.405
77.150
76.836



^1H NMR (500 MHz, CDCl_3) of **3q**



¹³C NMR (126 MHz, CDCl₃) of **3q**

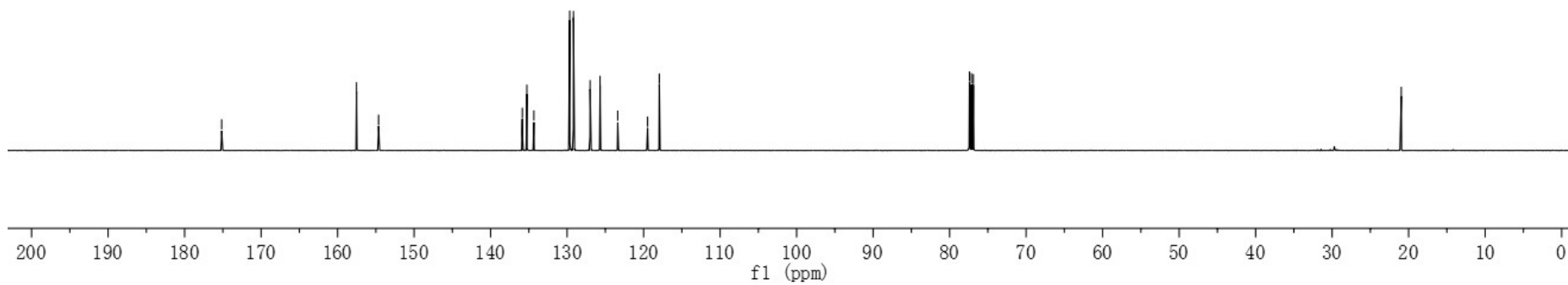
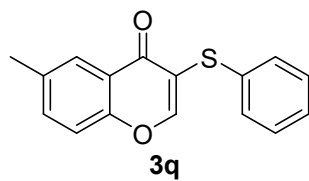
175.150

157.535
154.646

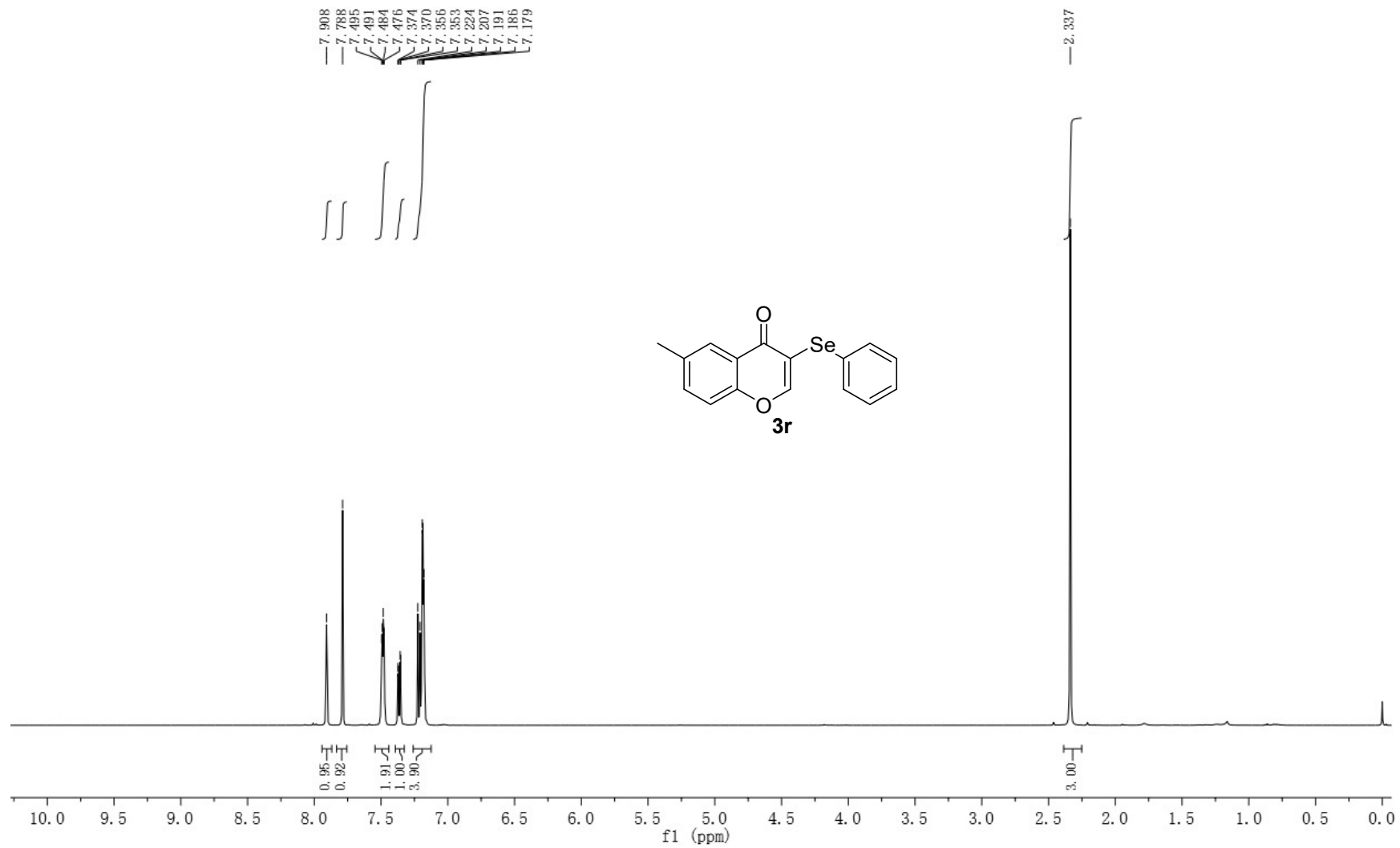
135.852
135.263
134.343
129.657
129.166
126.993
125.693
123.381
119.480
117.949

77.404
77.150
76.895

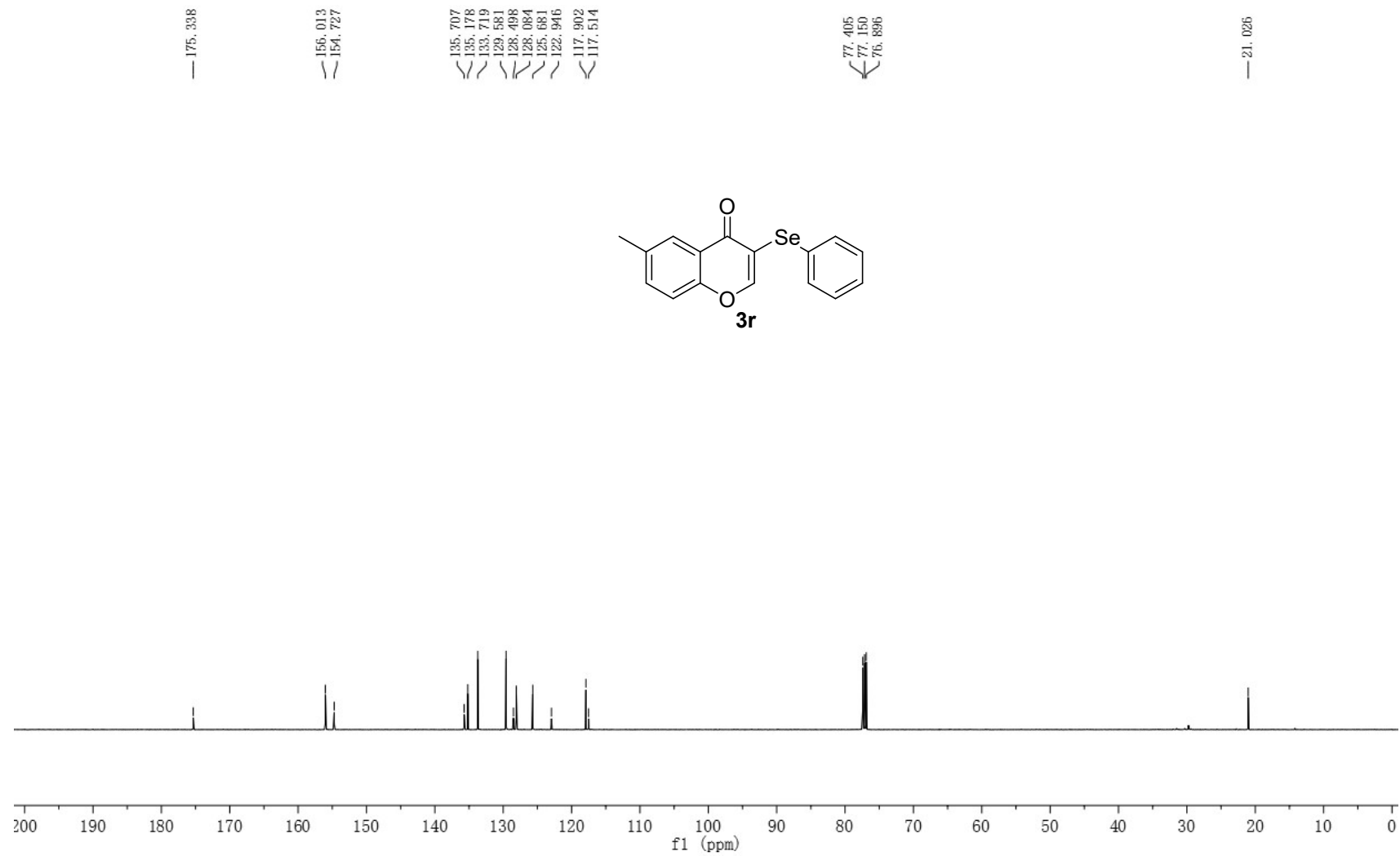
20.966



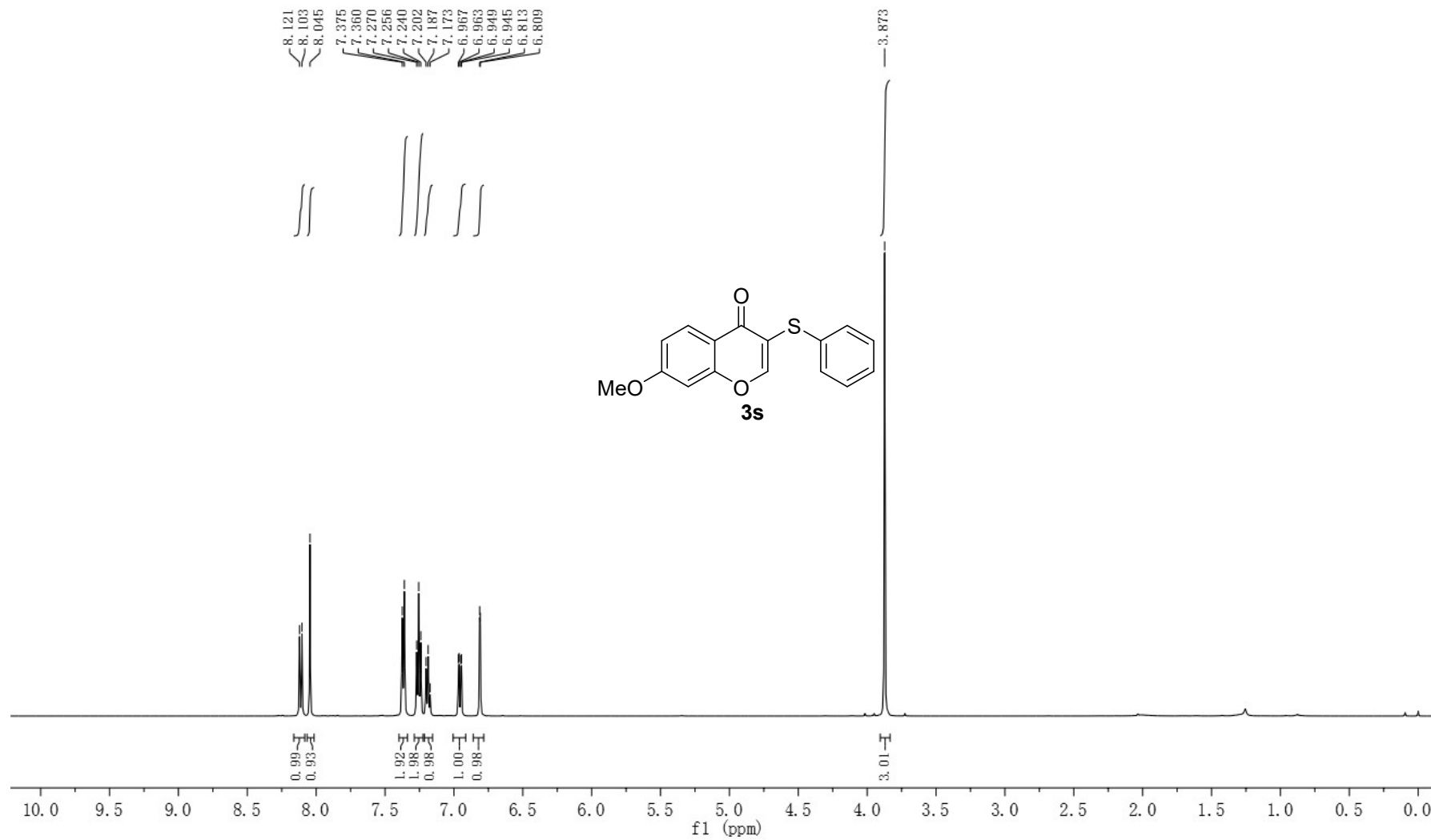
^1H NMR (500 MHz, CDCl_3) of **3r**



^{13}C NMR (126 MHz, CDCl_3) of **3r**

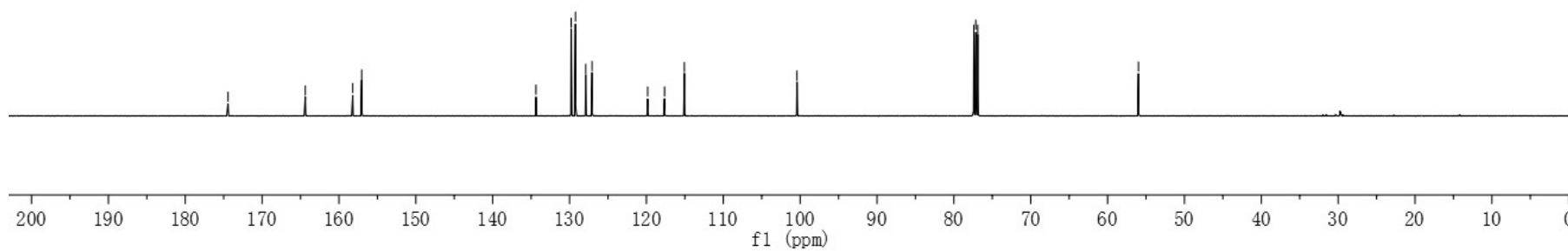
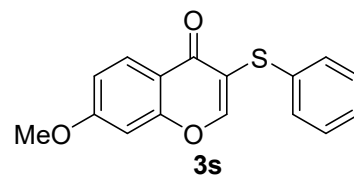


^1H NMR (500 MHz, CDCl_3) of **3s**

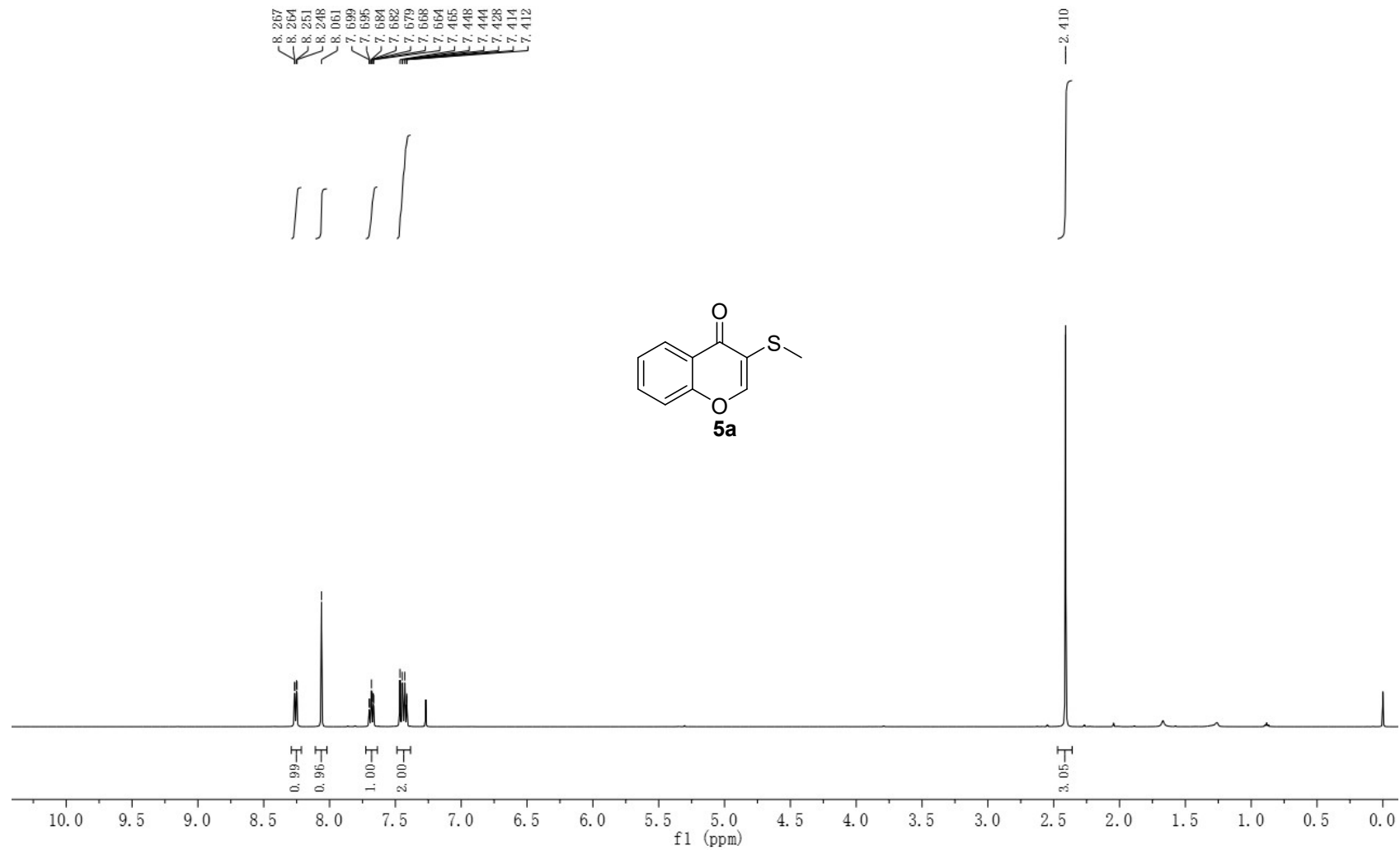


^{13}C NMR (126 MHz, CDCl_3) of **3s**

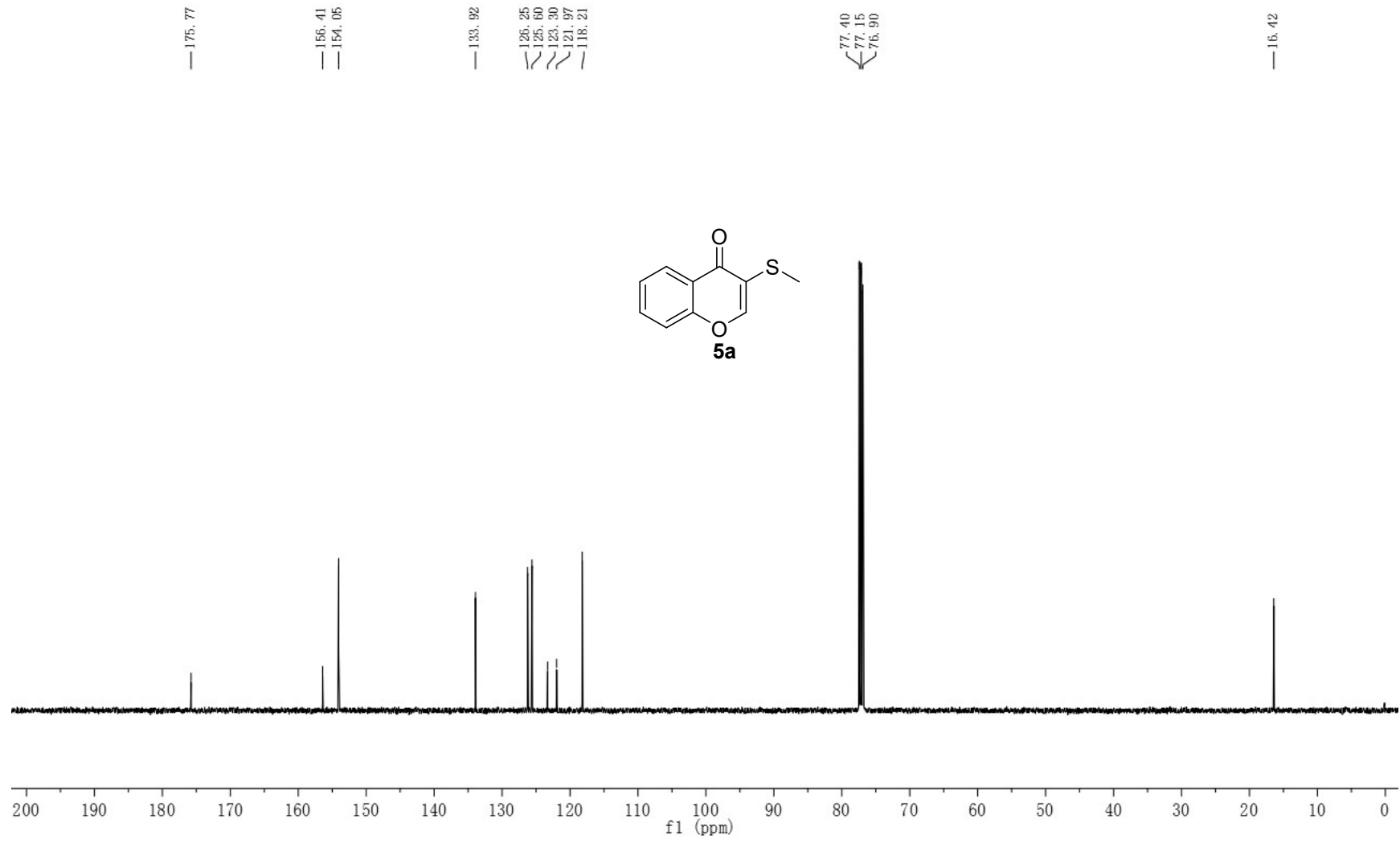
174.435
164.384
158.217
157.046
134.370
129.792
129.228
127.900
127.069
119.840
117.643
115.066
100.415
77.404
77.150
76.896
55.982



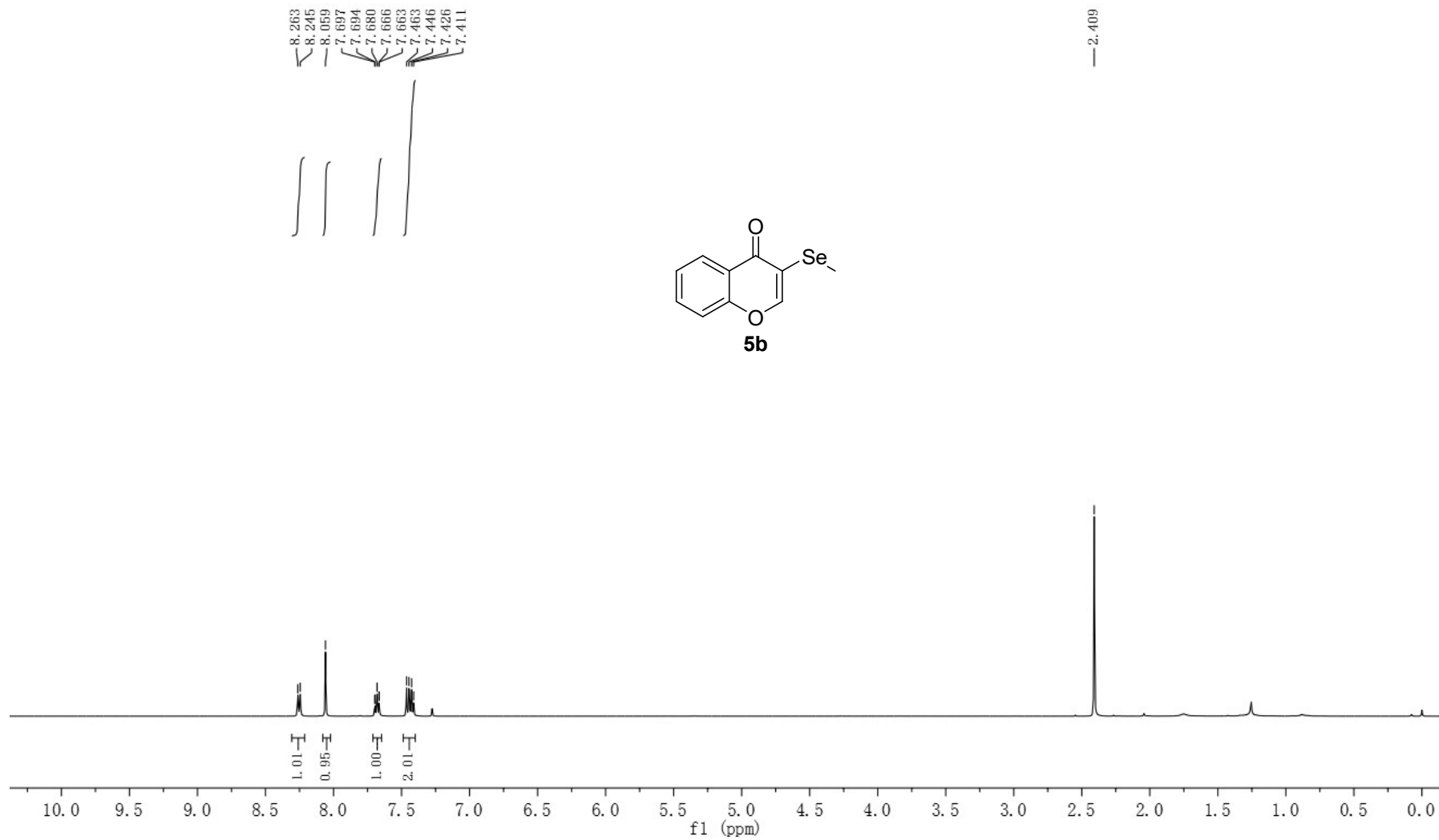
¹H NMR (500 MHz, CDCl₃) of **5a**



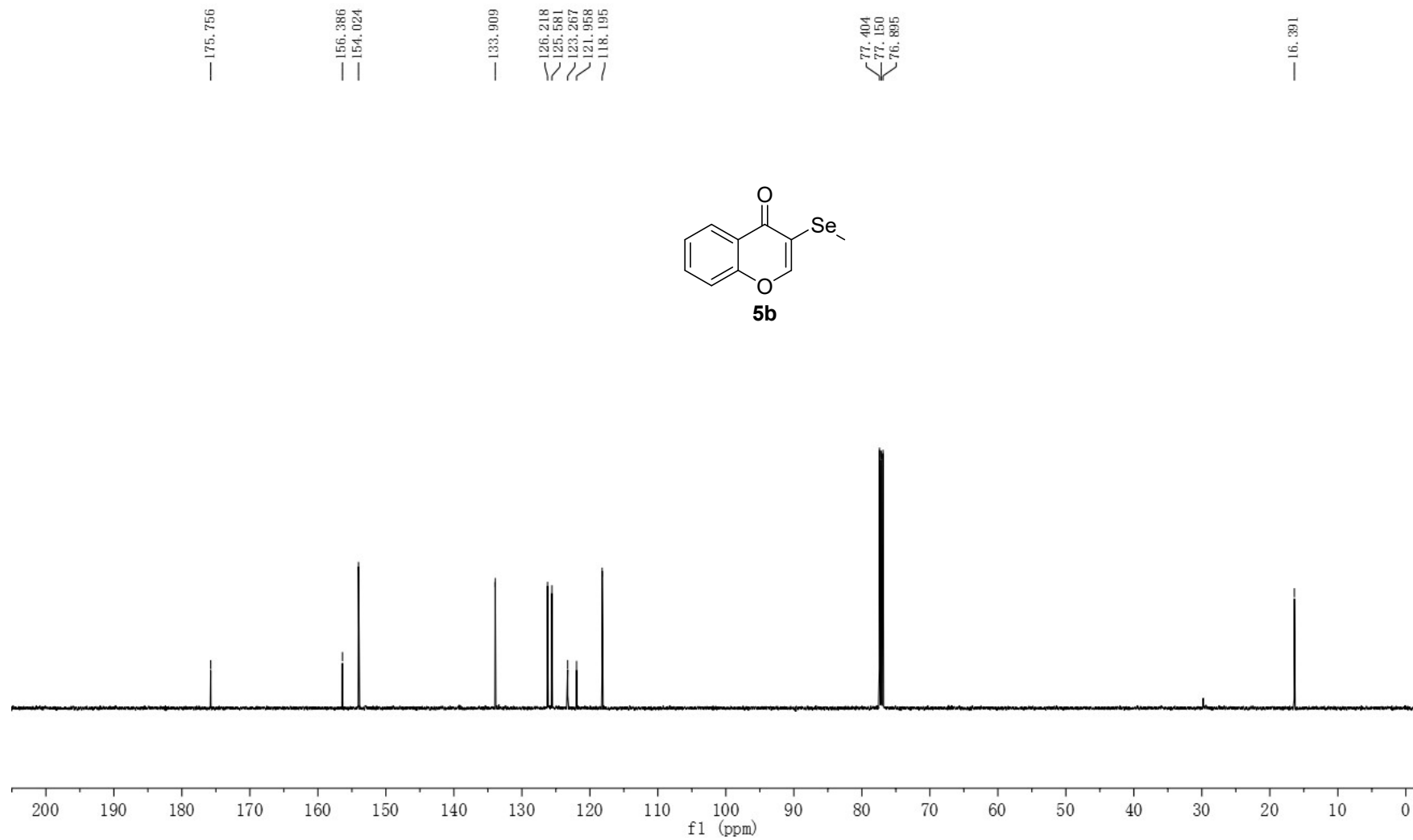
^{13}C NMR (126 MHz, CDCl_3) of **5a**



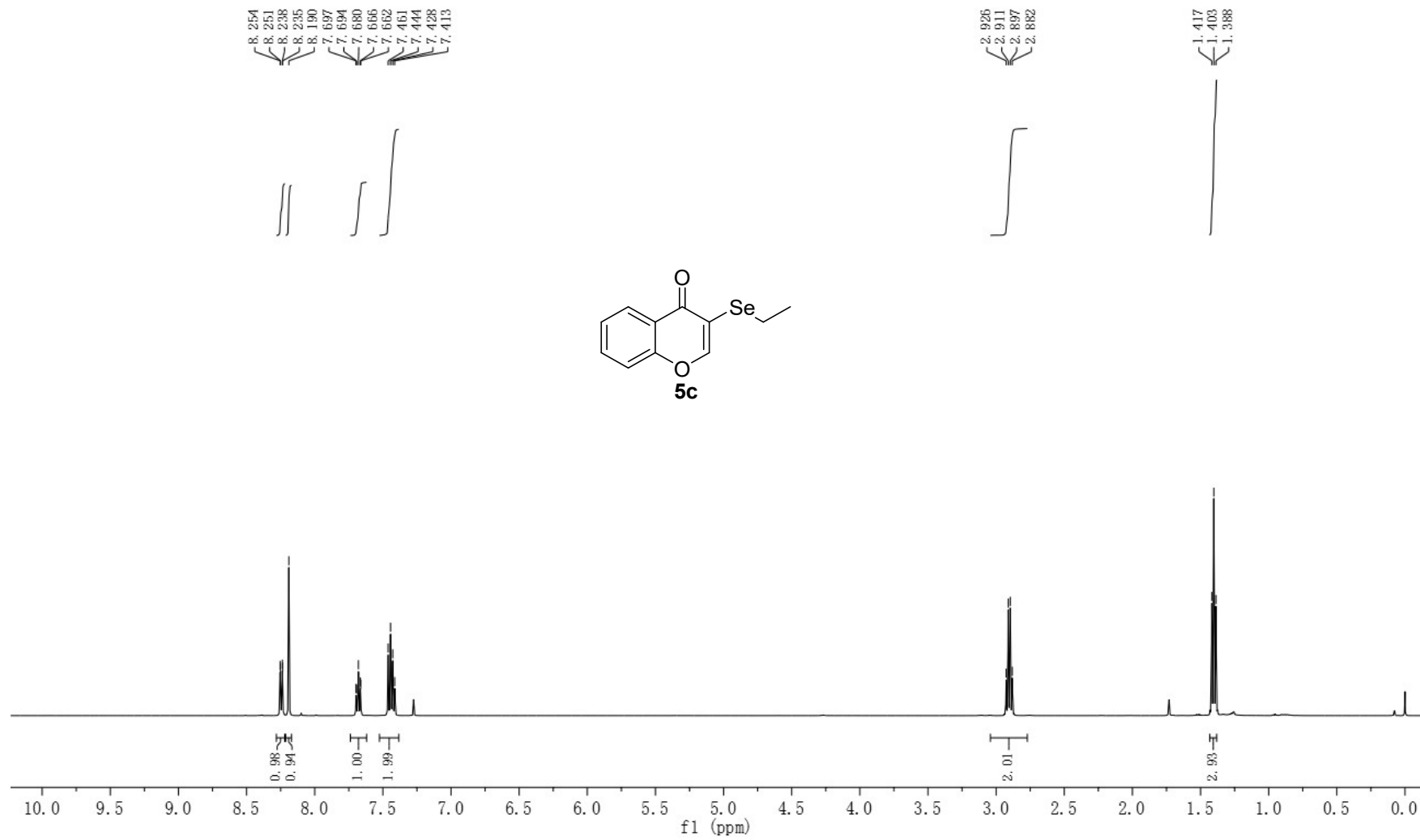
¹H NMR (500 MHz, CDCl₃) of **5b**



^{13}C NMR (126 MHz, CDCl_3) of **5b**



^1H NMR (500 MHz, CDCl_3) of **5c**



^{13}C NMR (126 MHz, CDCl_3) of **5c**

176.02

156.62
156.48

133.85

126.42

125.62

123.32

118.16

114.48

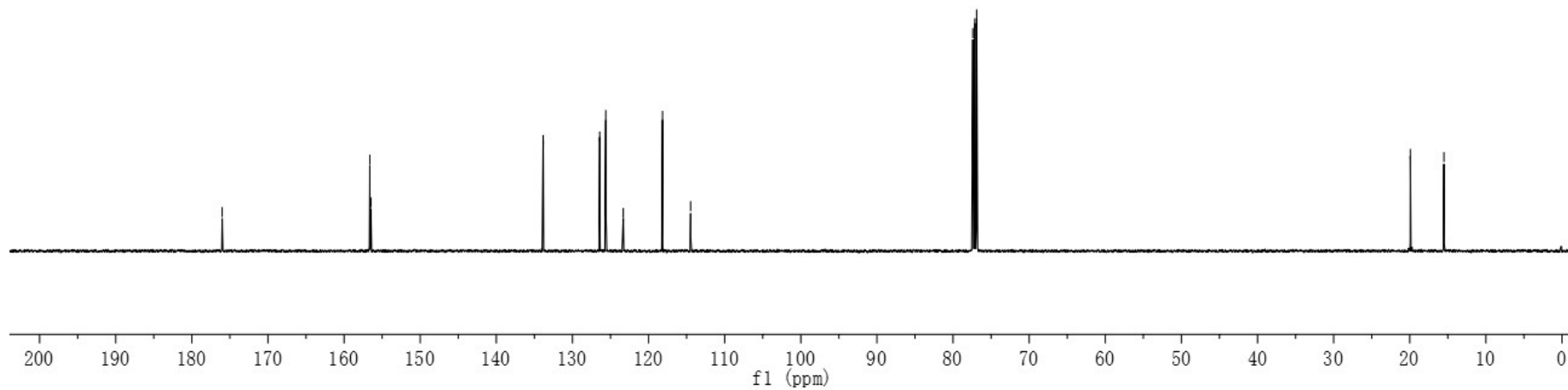
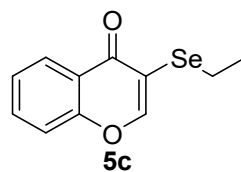
77.40

77.15

76.90

19.92

15.51



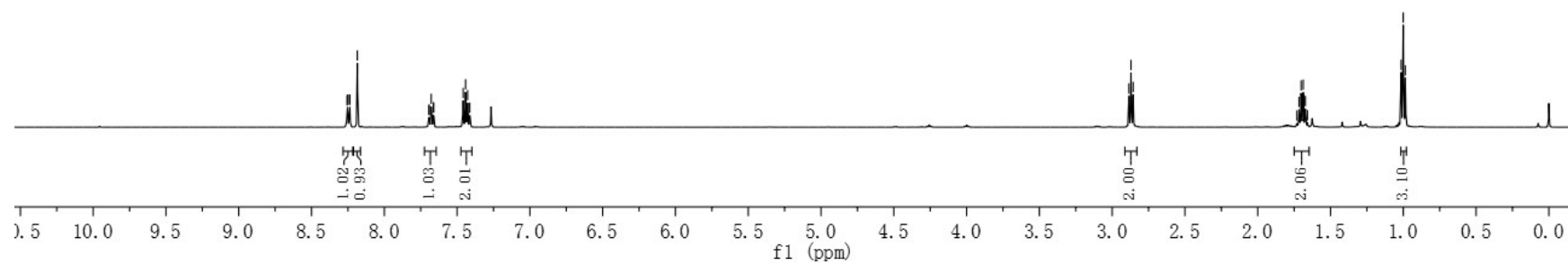
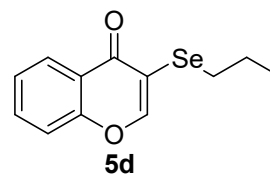
^1H NMR (500 MHz, CDCl_3) of **5d**

8.256
8.253
8.240
8.237
8.185
7.695
7.692
7.678
7.664
7.661
7.458
7.442
7.428
7.413

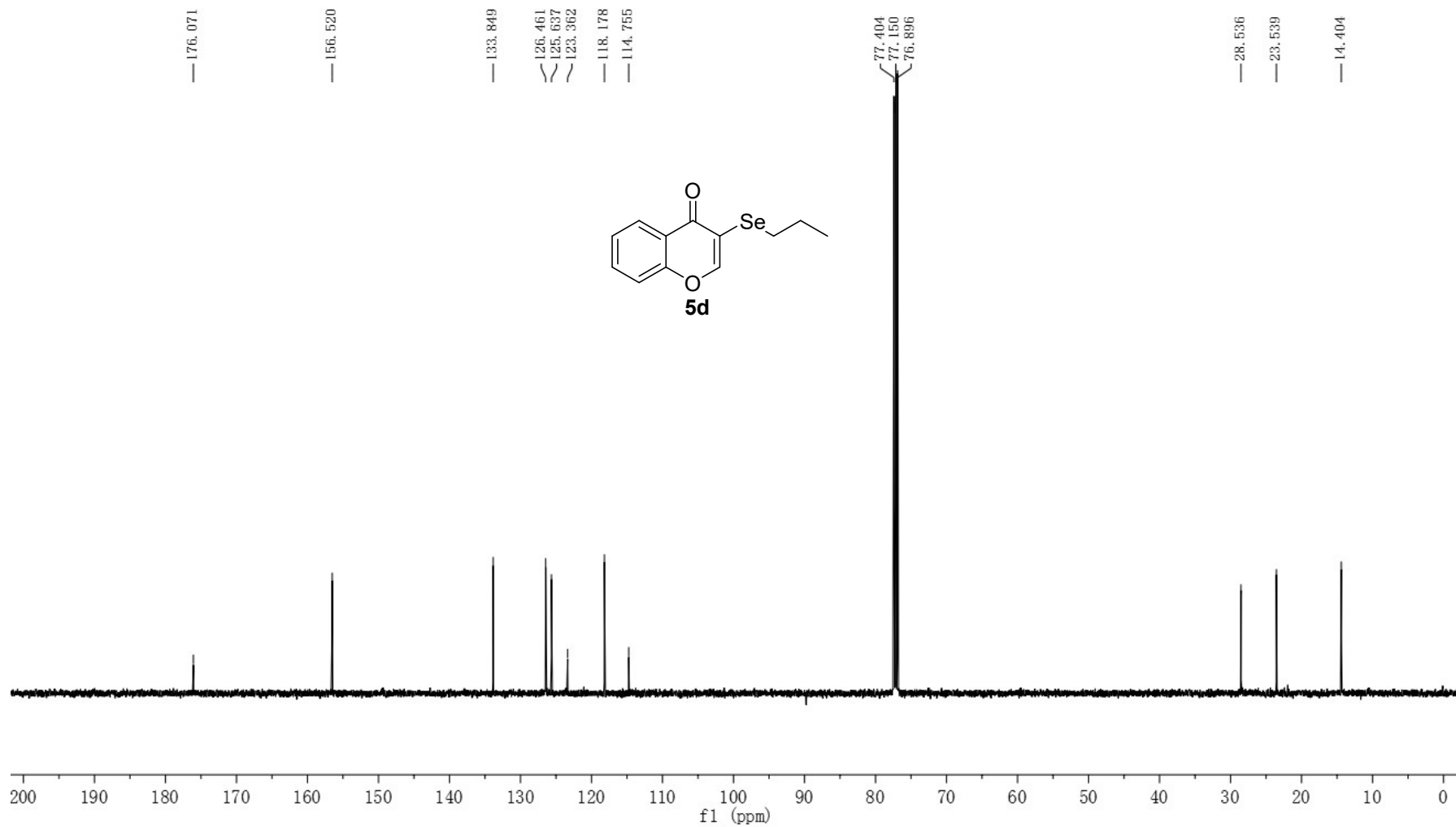
2.885
2.871
2.856

1.731
1.717
1.702
1.687
1.673
1.658

1.015
1.000
0.986



^{13}C NMR (126 MHz, CDCl_3) of **5d**



176.071

156.520

133.849

126.461

125.637

123.362

118.178

114.755

77.404

77.150

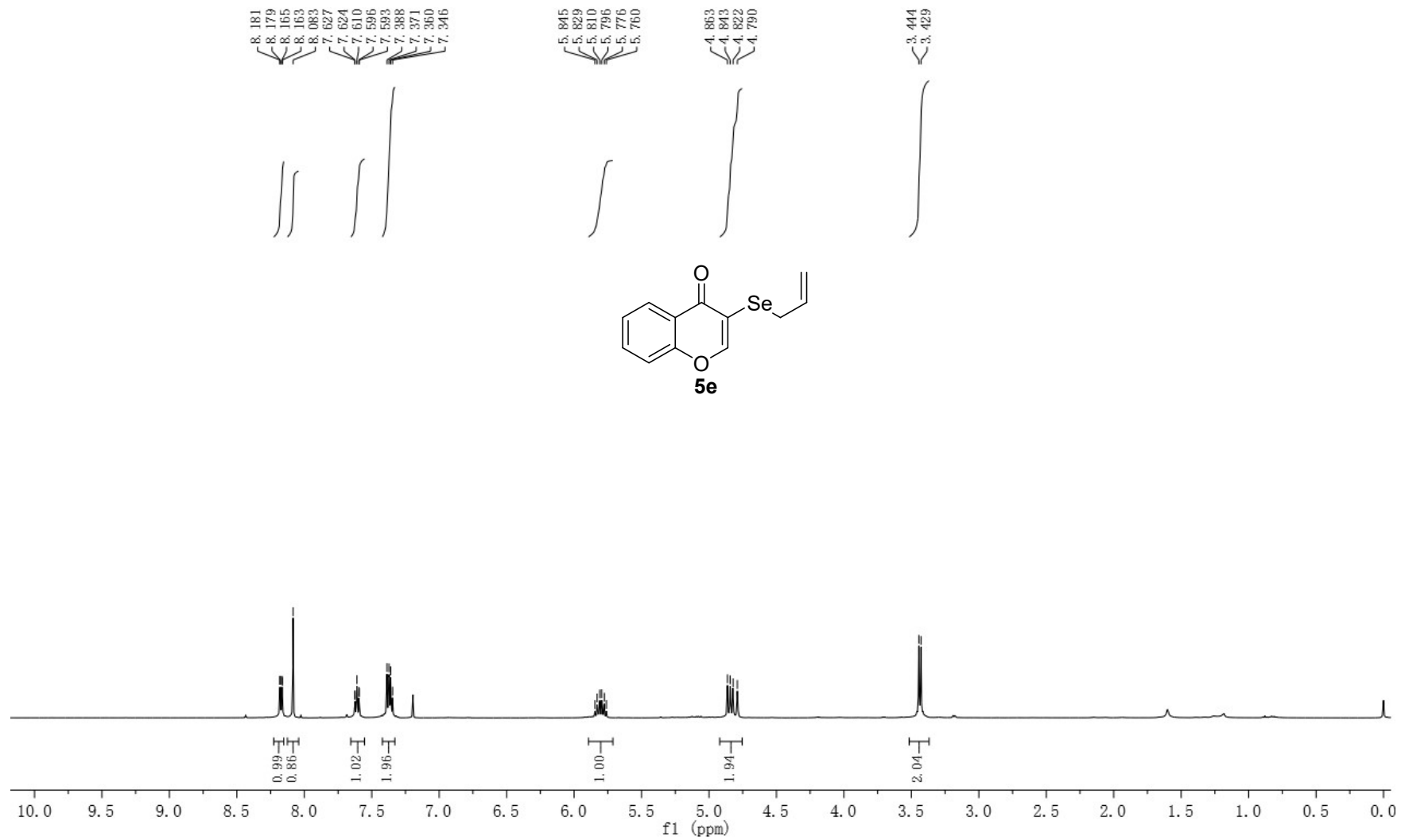
76.896

28.536

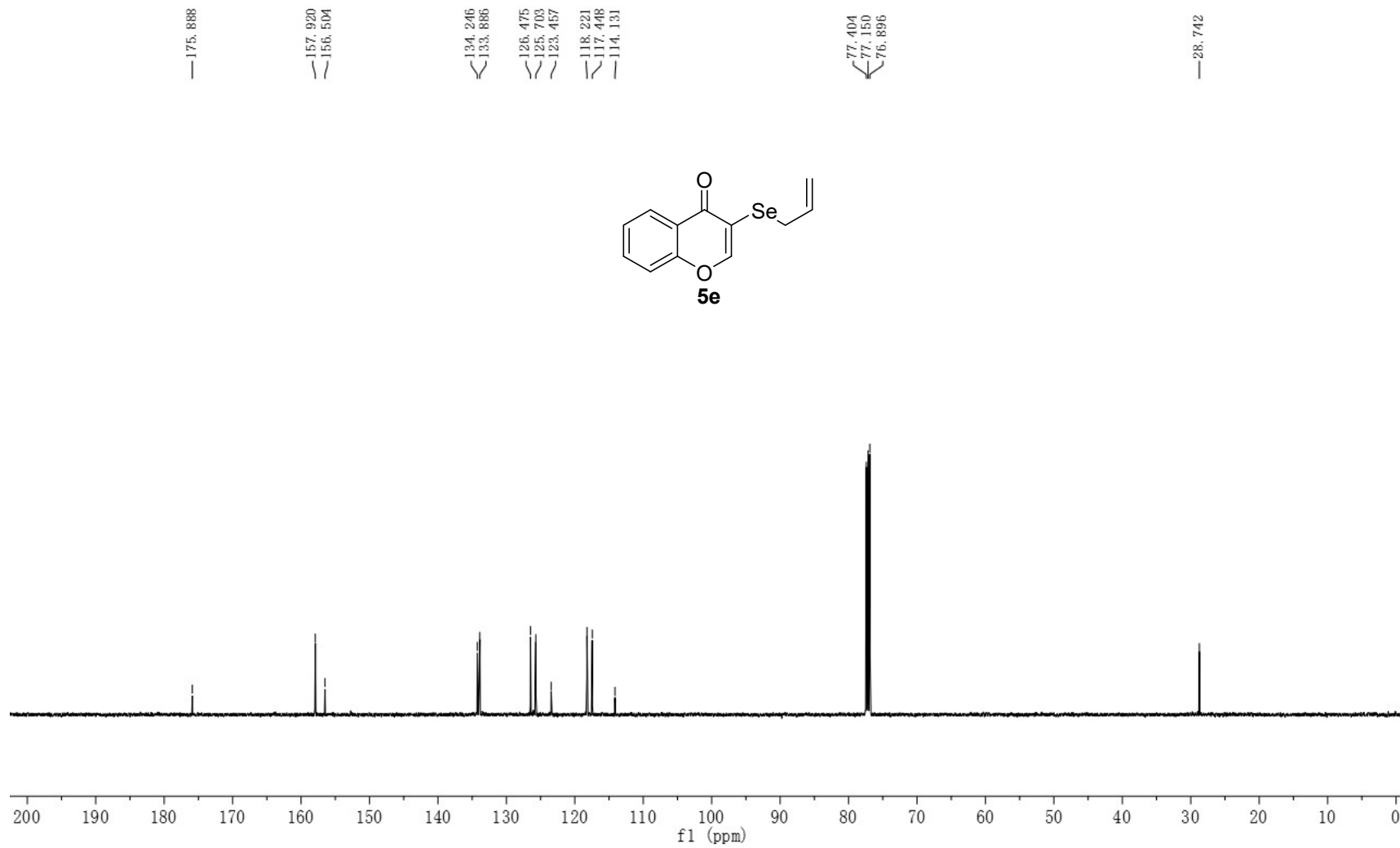
23.539

14.404

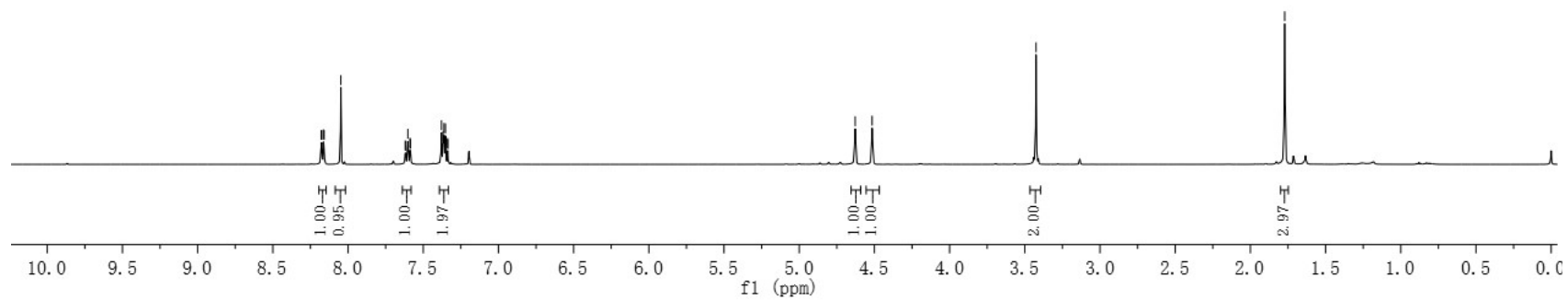
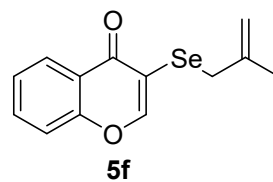
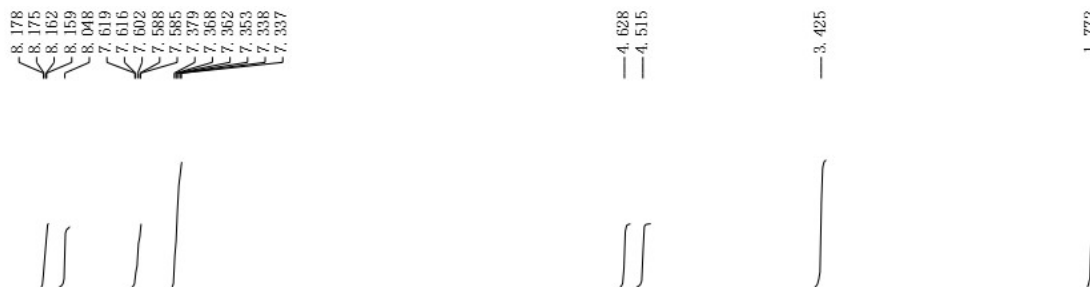
^1H NMR (500 MHz, CDCl_3) of **5e**



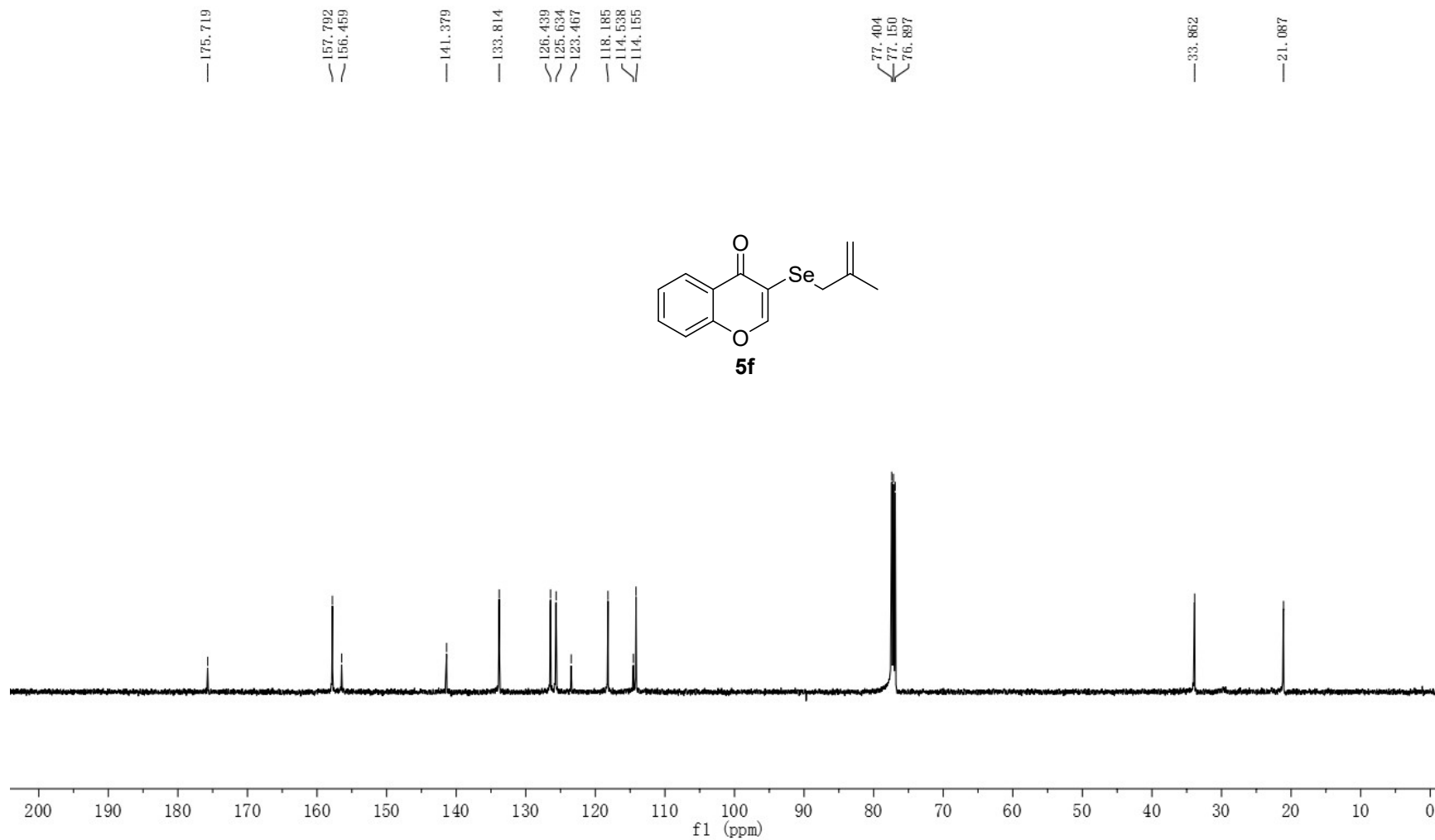
^{13}C NMR (126 MHz, CDCl_3) of **5e**



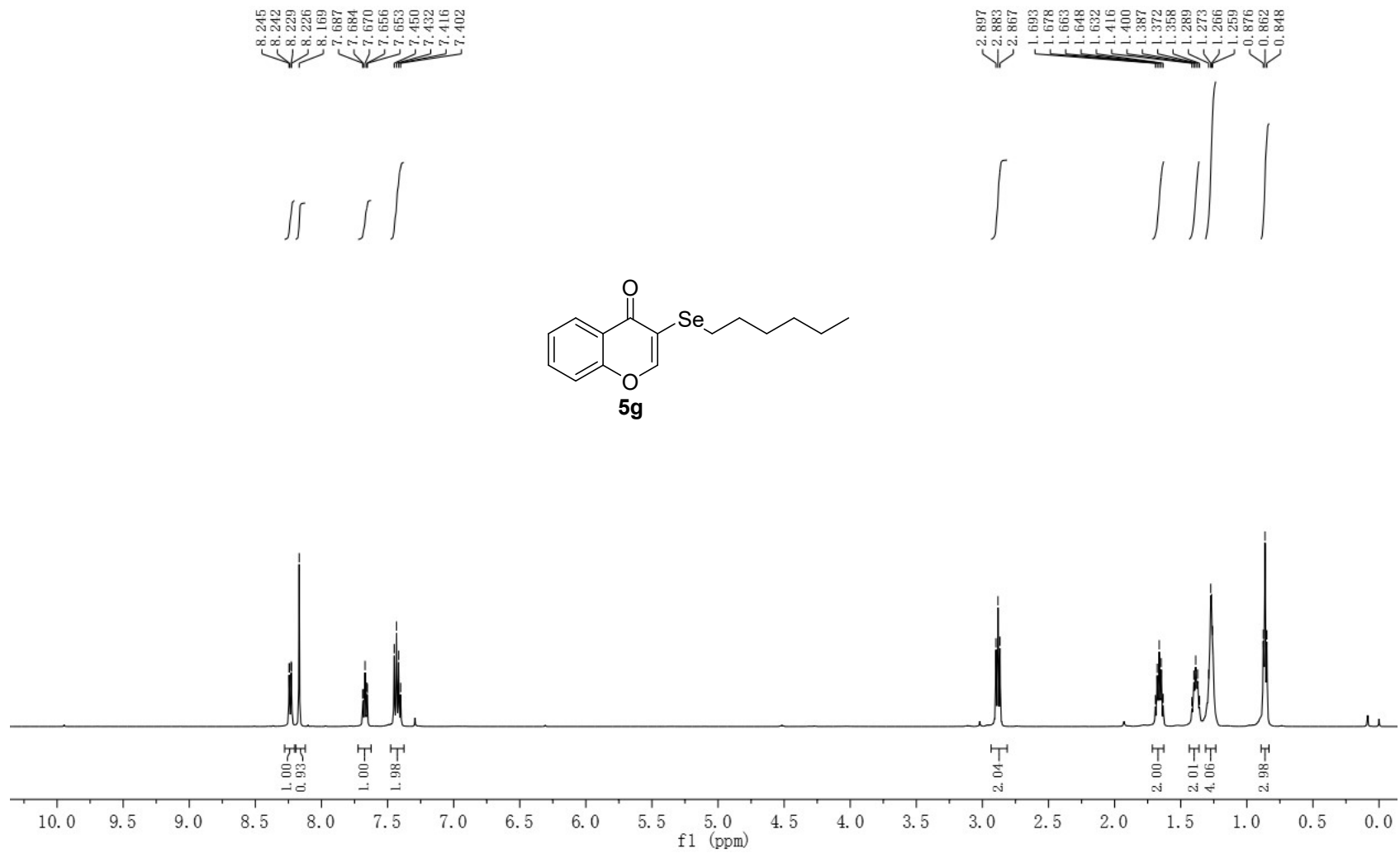
^1H NMR (500 MHz, CDCl_3) of **5f**



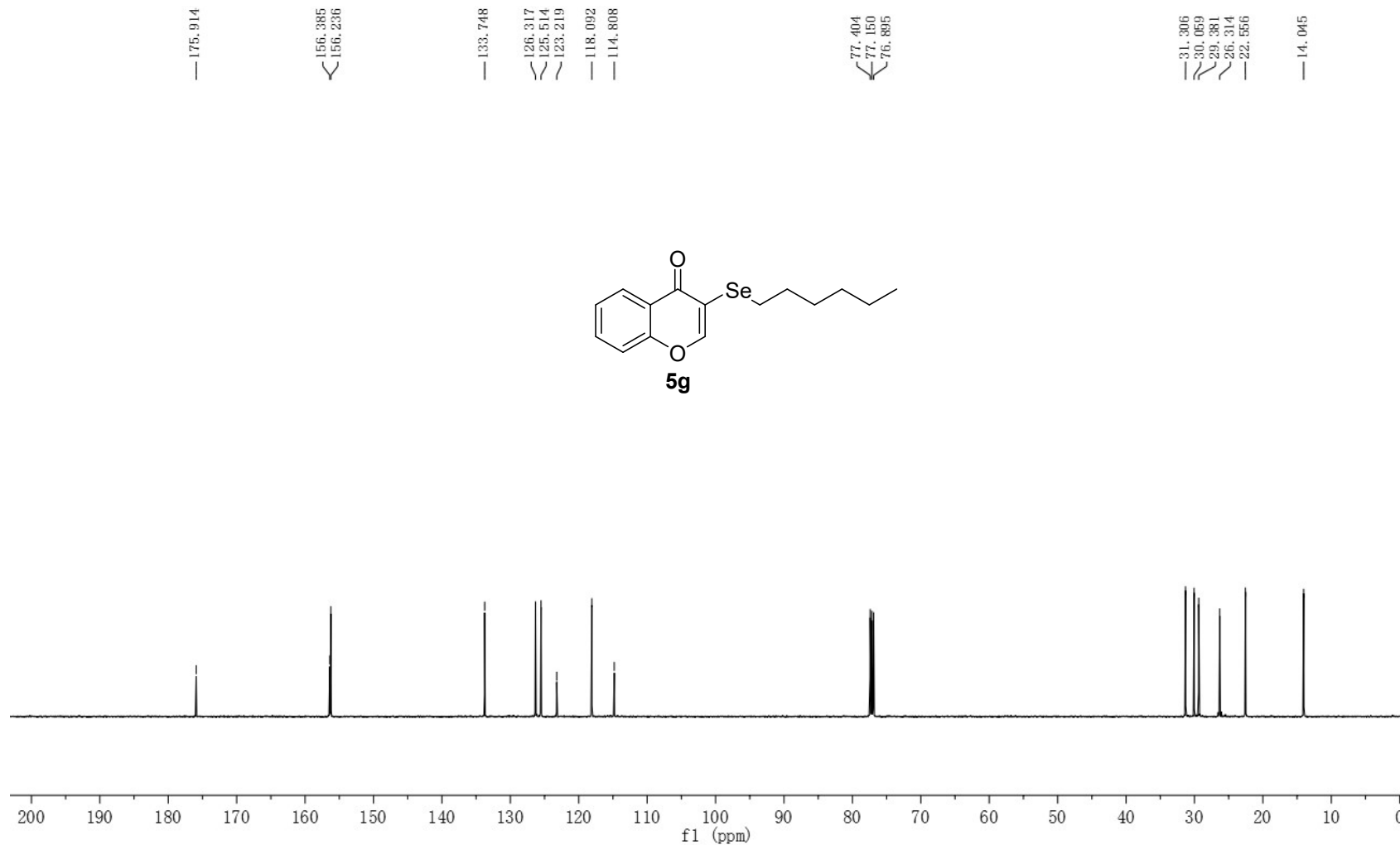
^{13}C NMR (126 MHz, CDCl_3) of **5f**



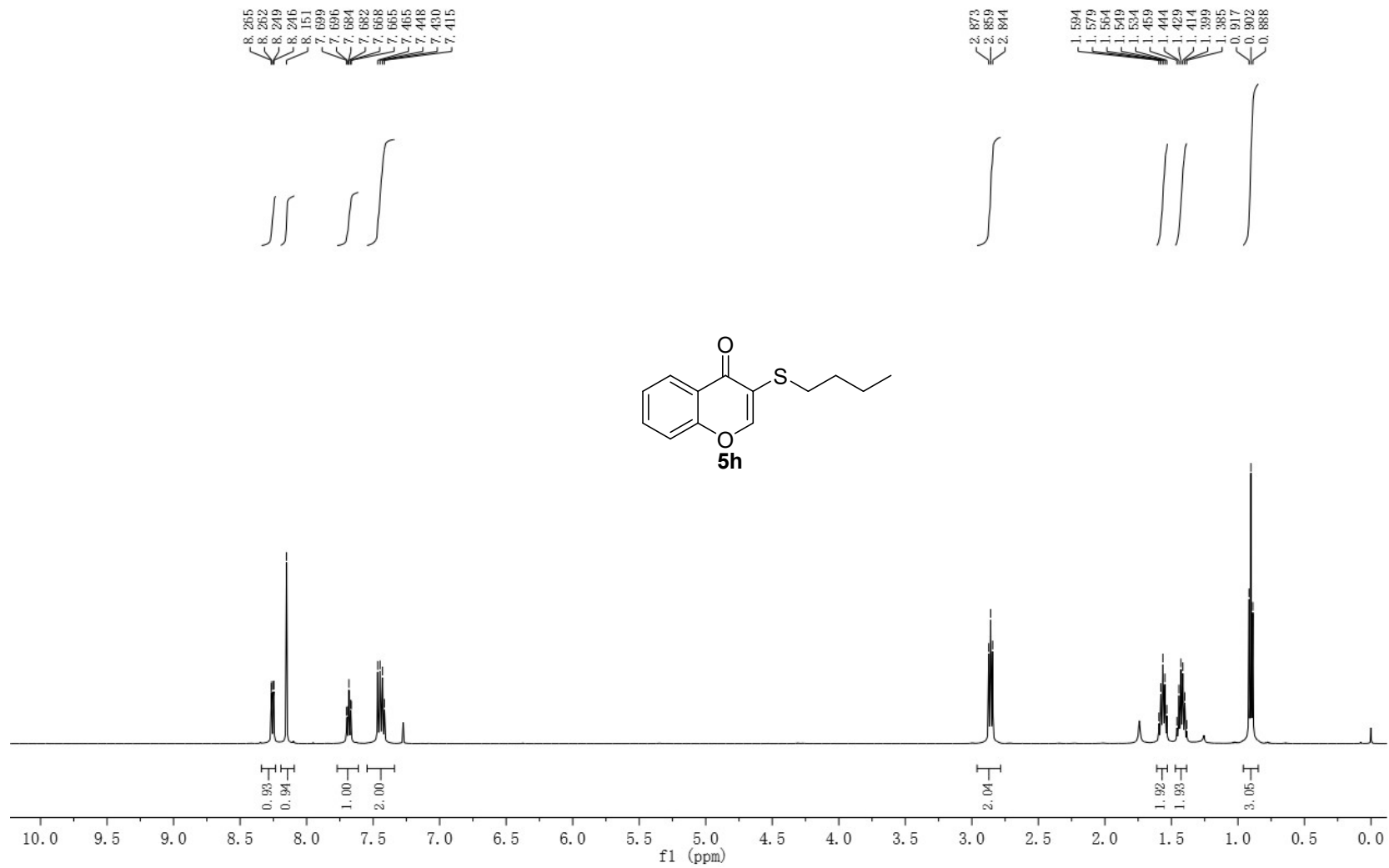
^1H NMR (500 MHz, CDCl_3) of **5g**



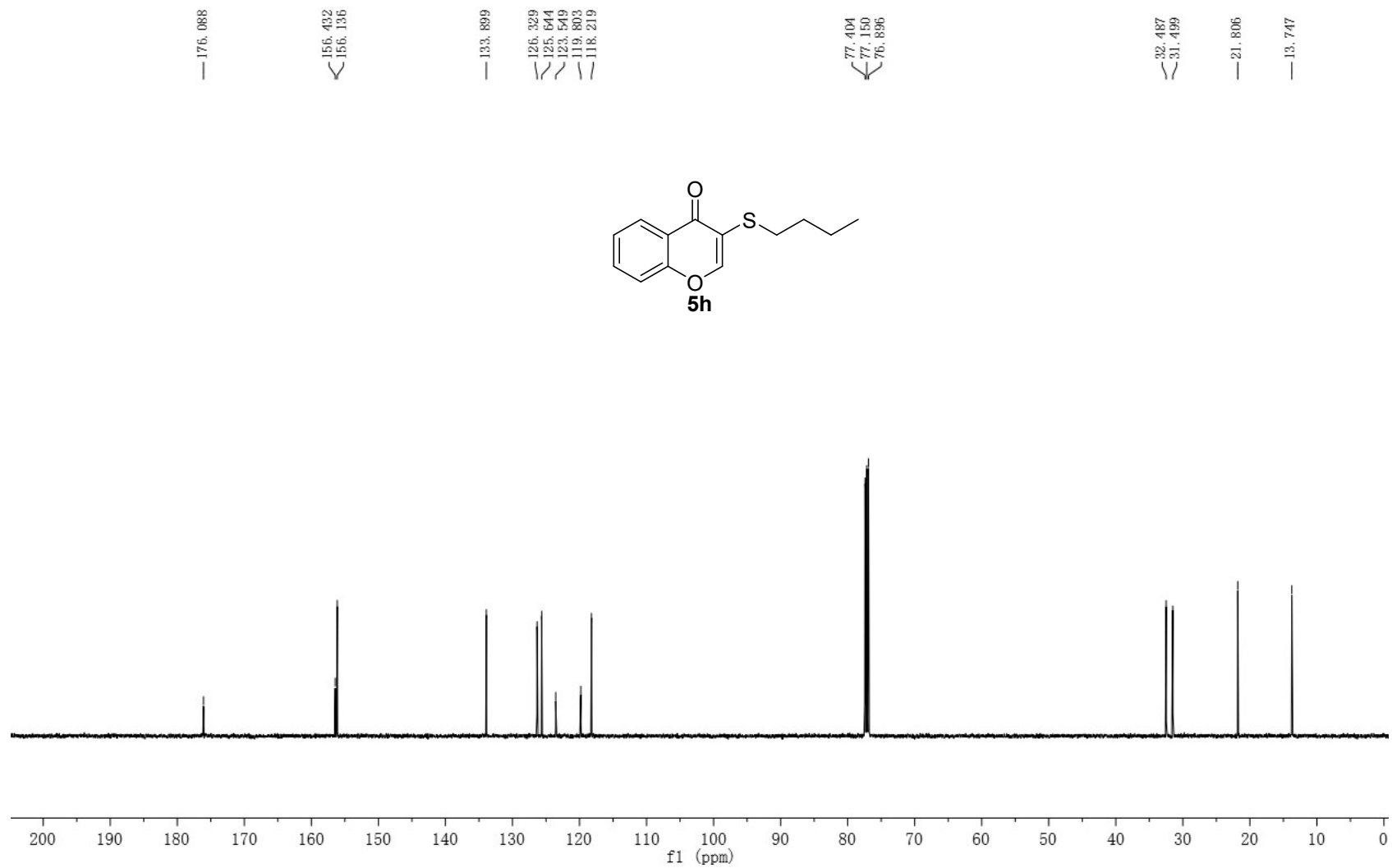
^{13}C NMR (126 MHz, CDCl_3) of **5g**



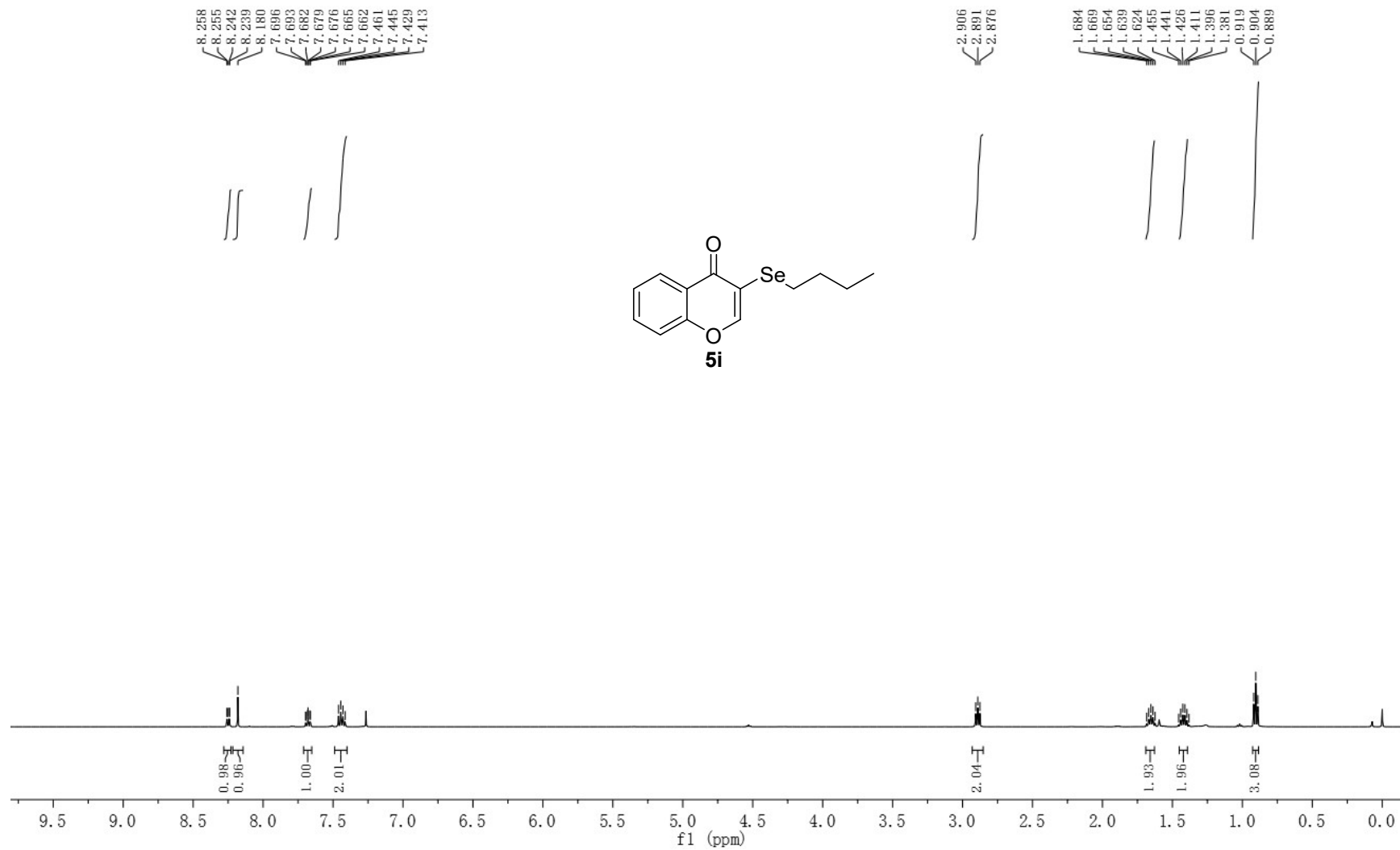
^1H NMR (500 MHz, CDCl_3) of **5h**



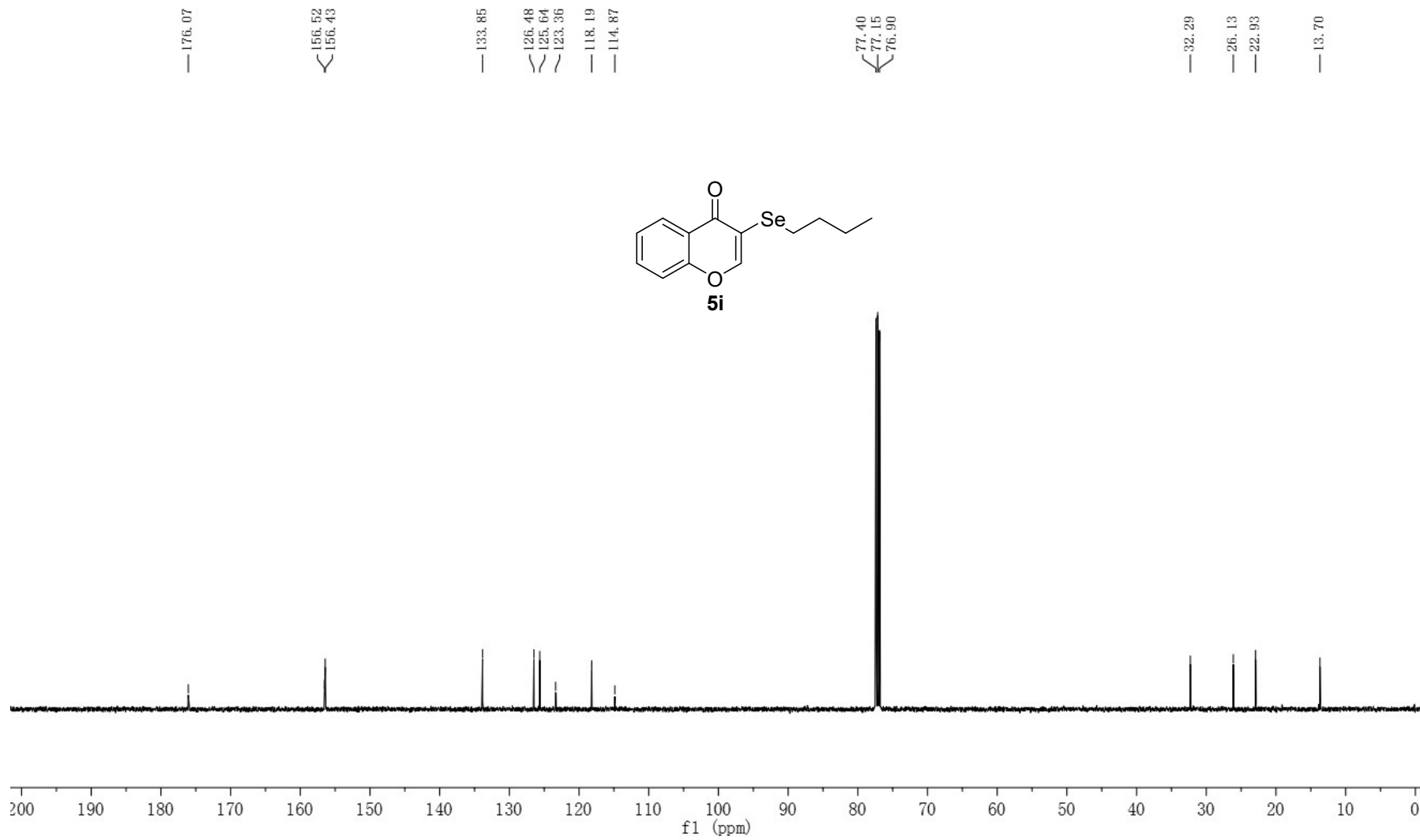
^{13}C NMR (126 MHz, CDCl_3) of **5h**



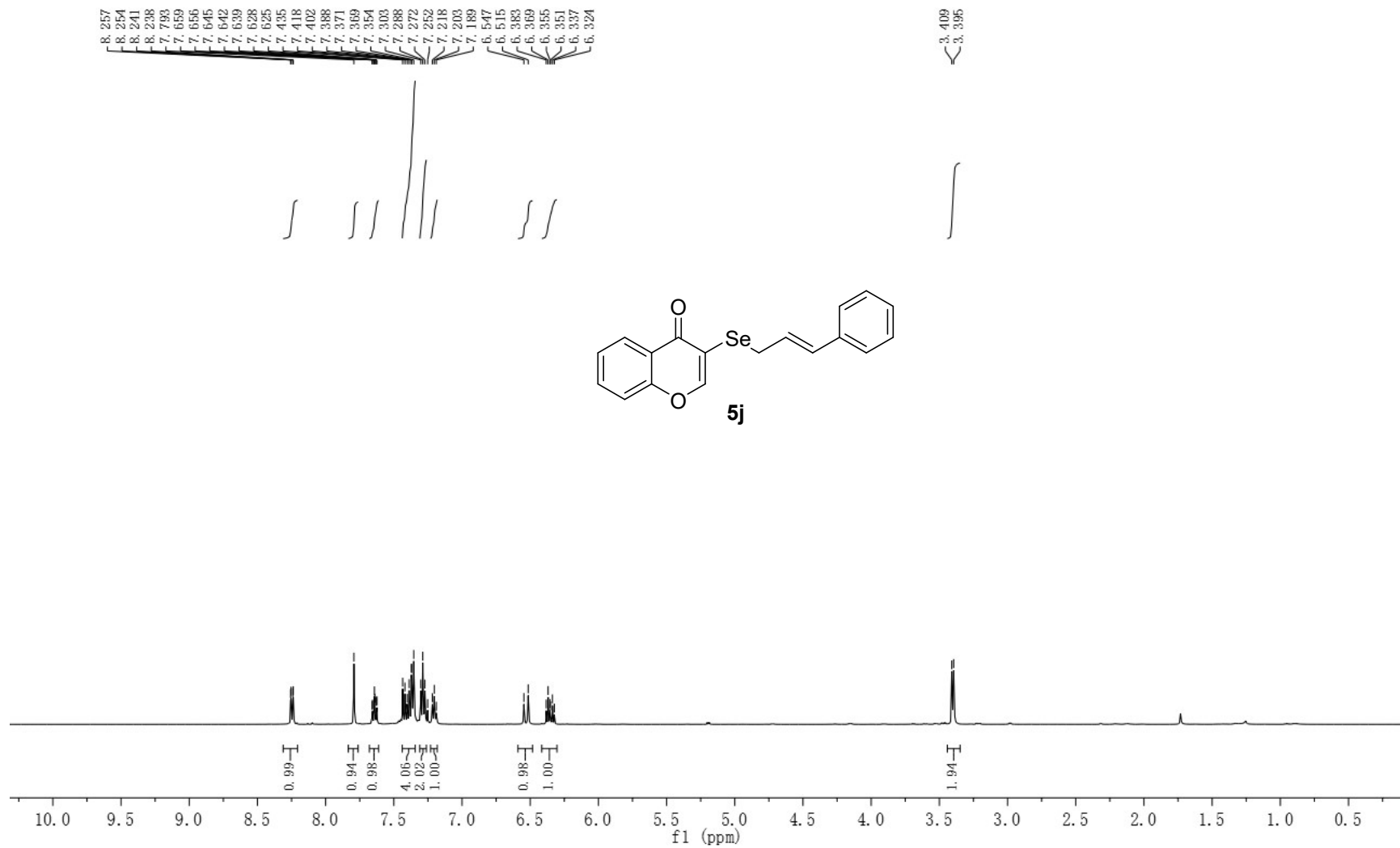
^1H NMR (500 MHz, CDCl_3) of **5i**



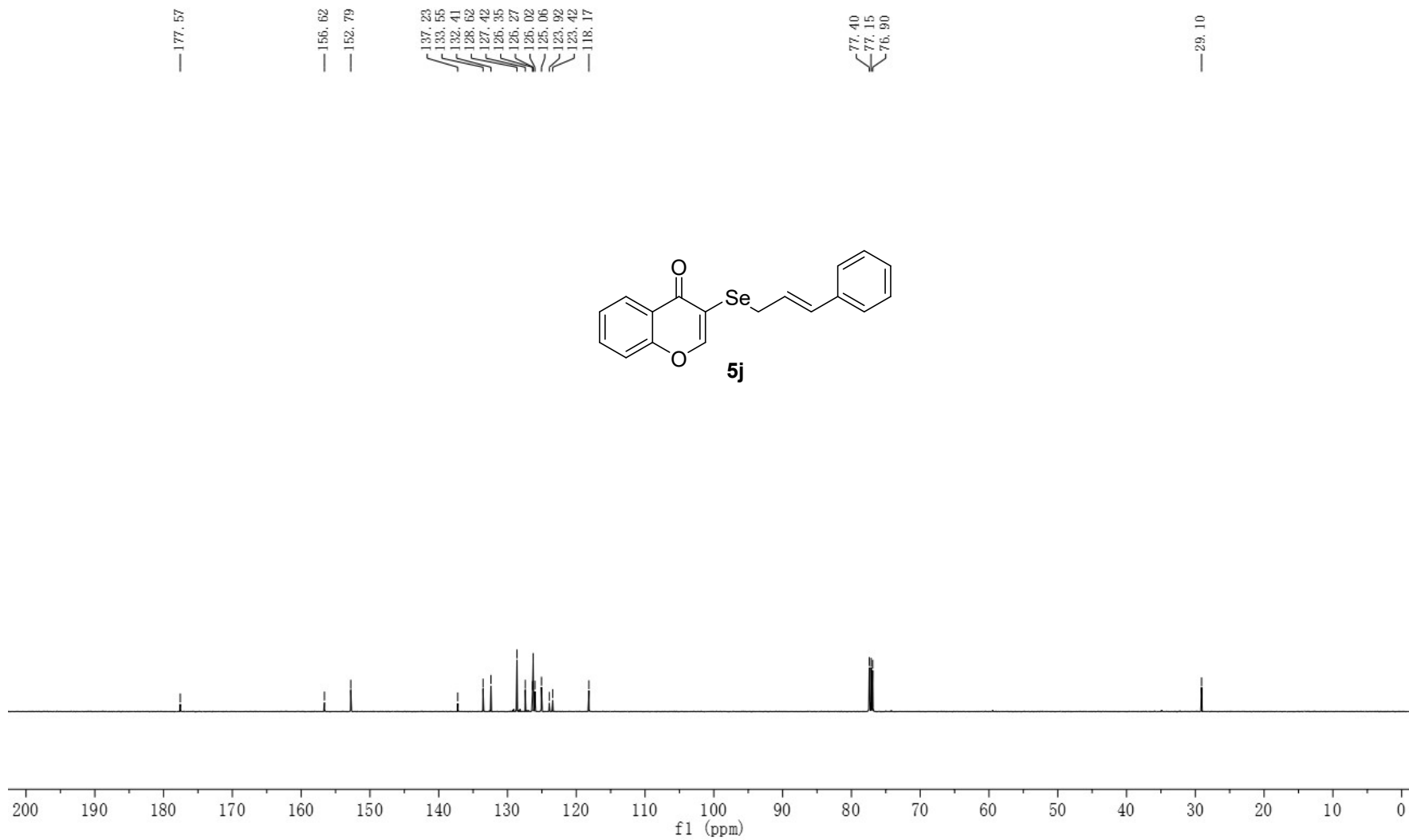
^{13}C NMR (126 MHz, CDCl_3) of **5i**



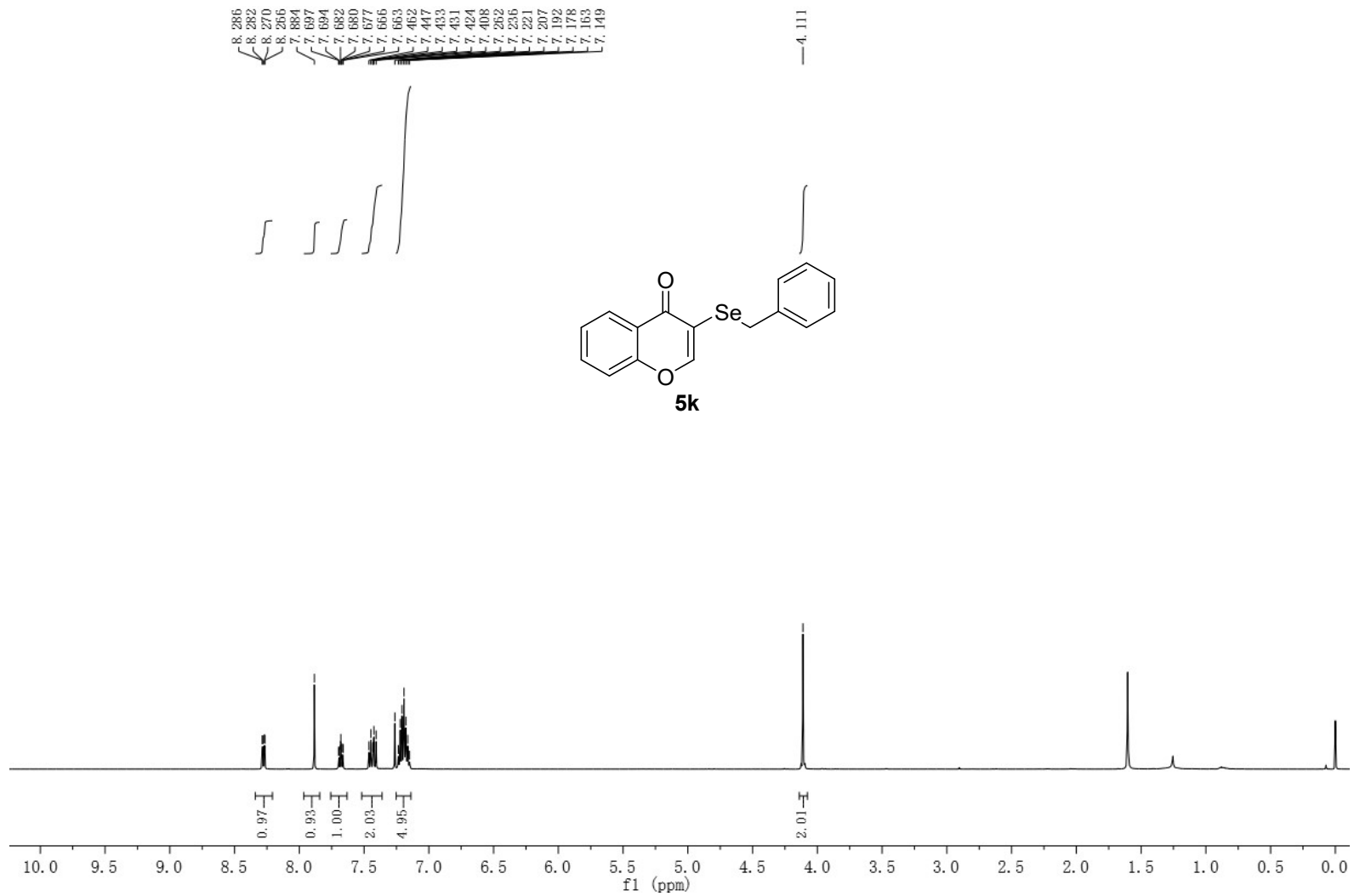
¹H NMR (500 MHz, CDCl₃) of **5j**



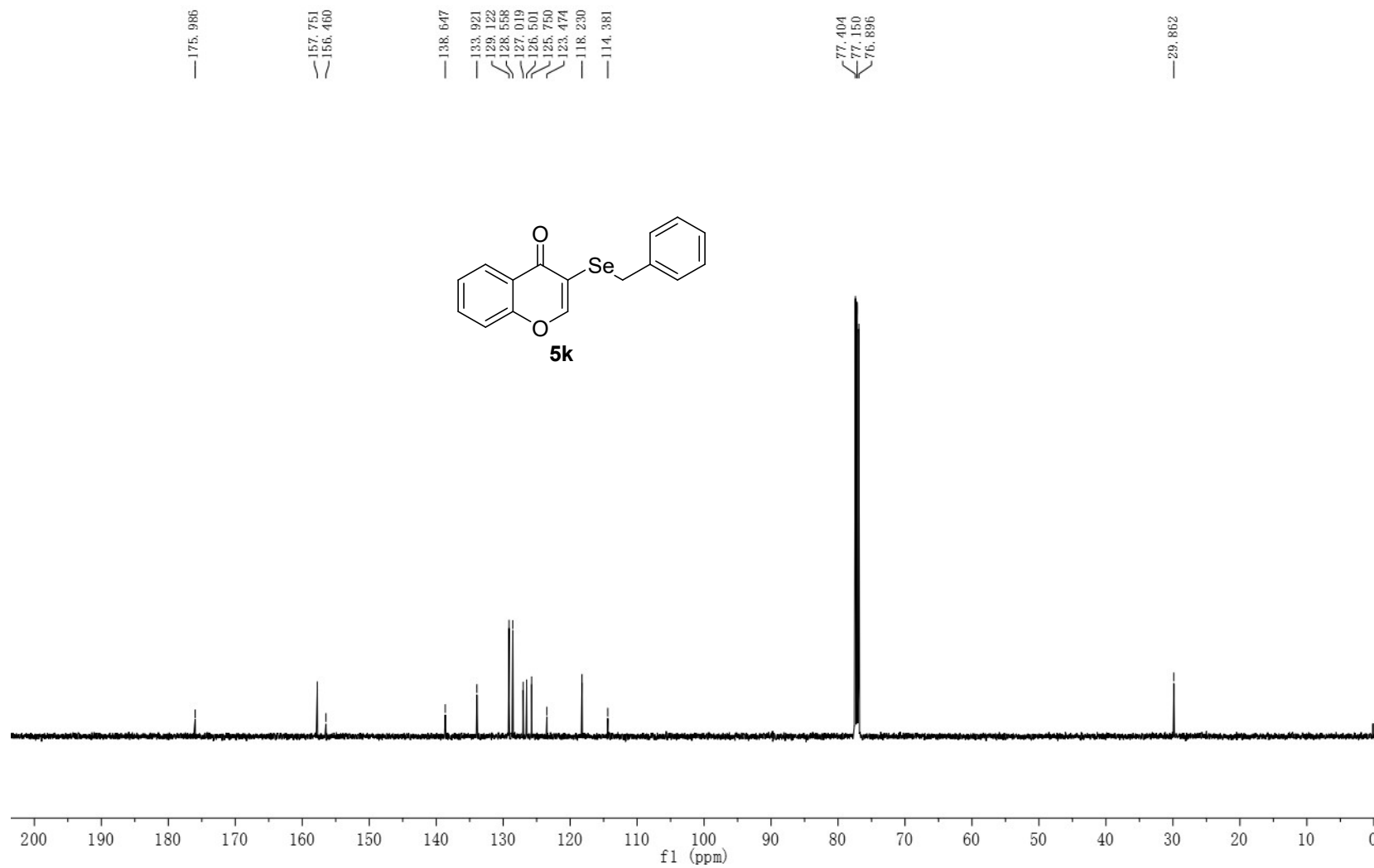
^{13}C NMR (126 MHz, CDCl_3) of **5j**



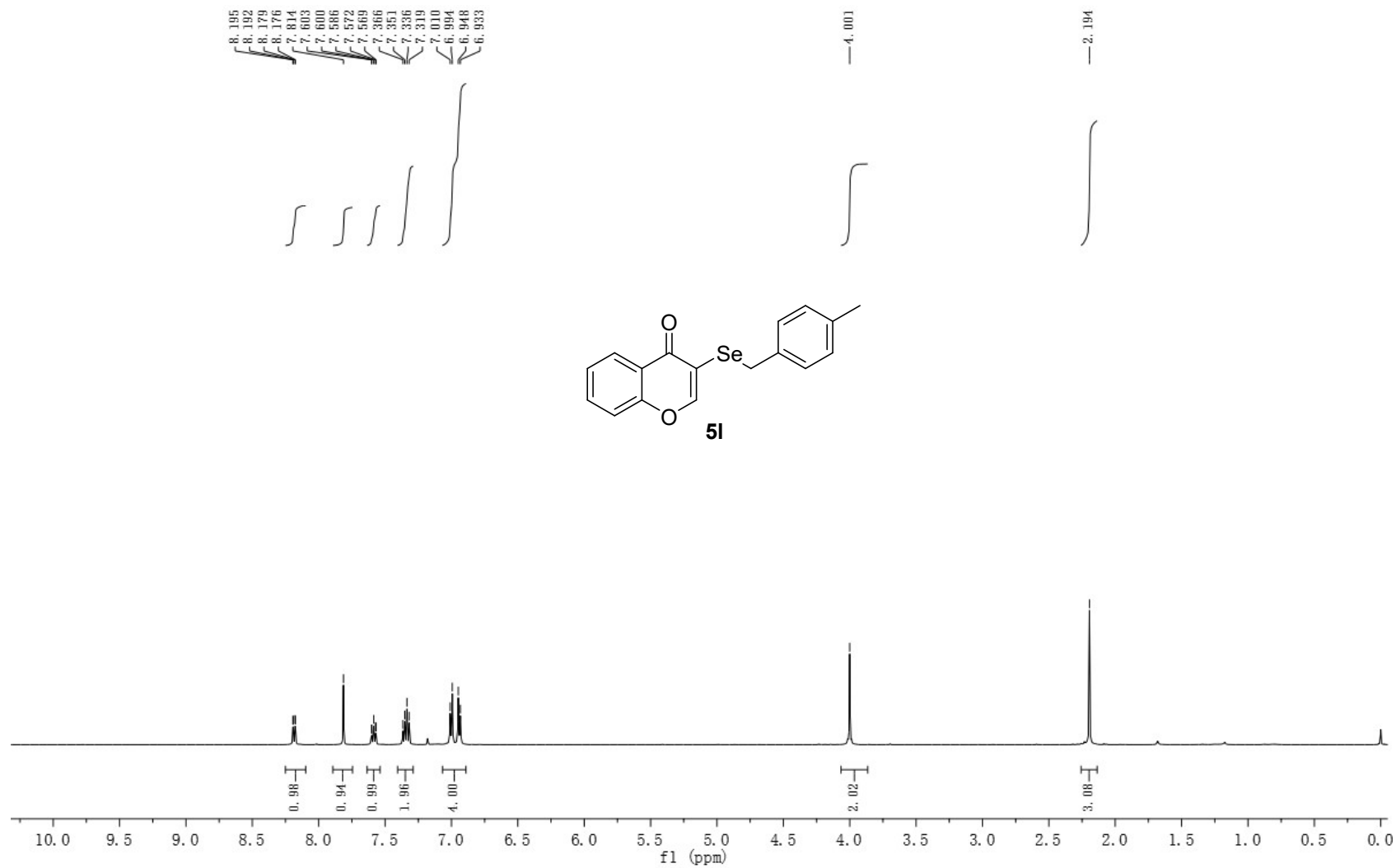
^1H NMR (500 MHz, CDCl_3) of **5k**



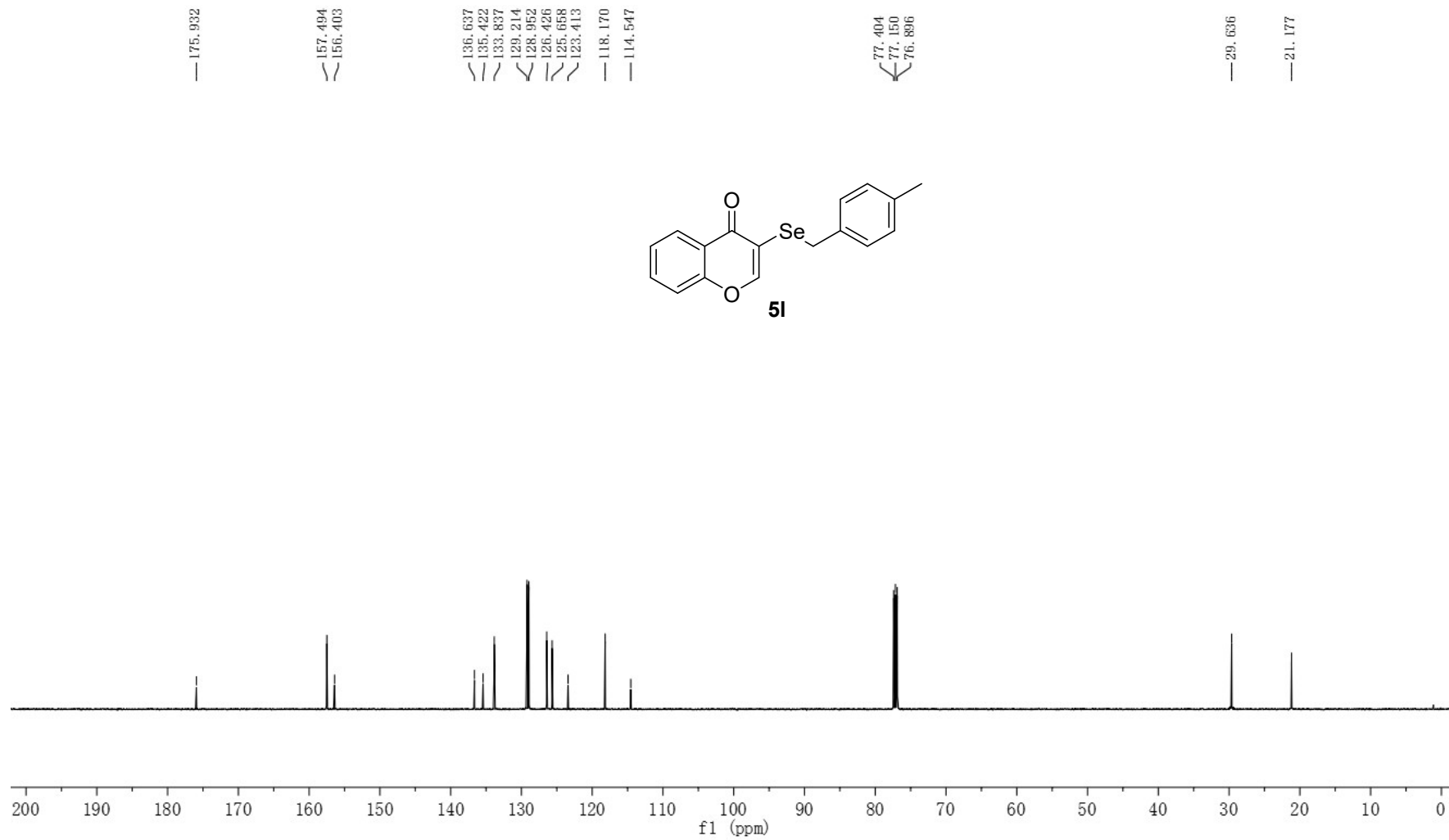
^{13}C NMR (126 MHz, CDCl_3) of **5k**



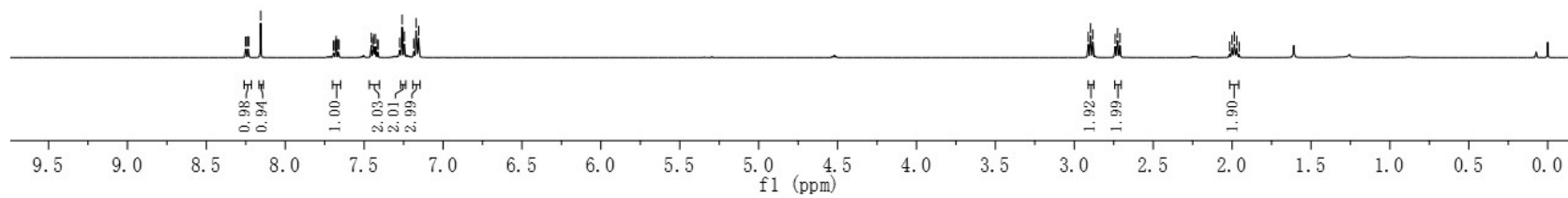
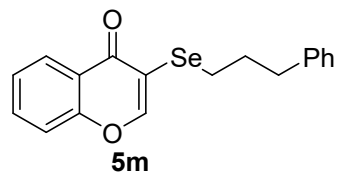
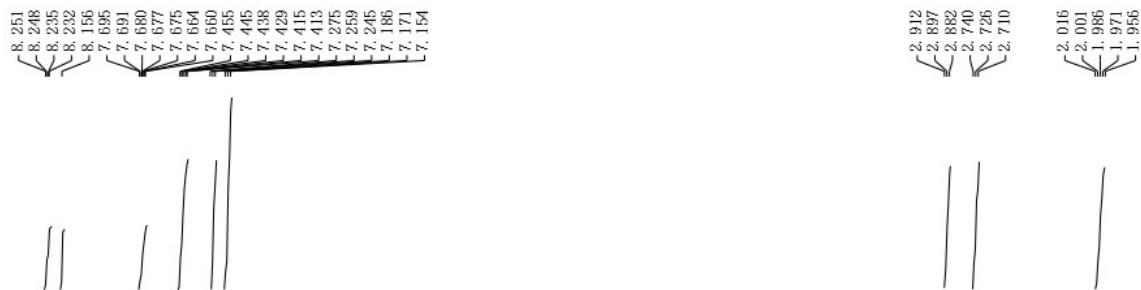
¹H NMR (500 MHz, CDCl₃) of **51**



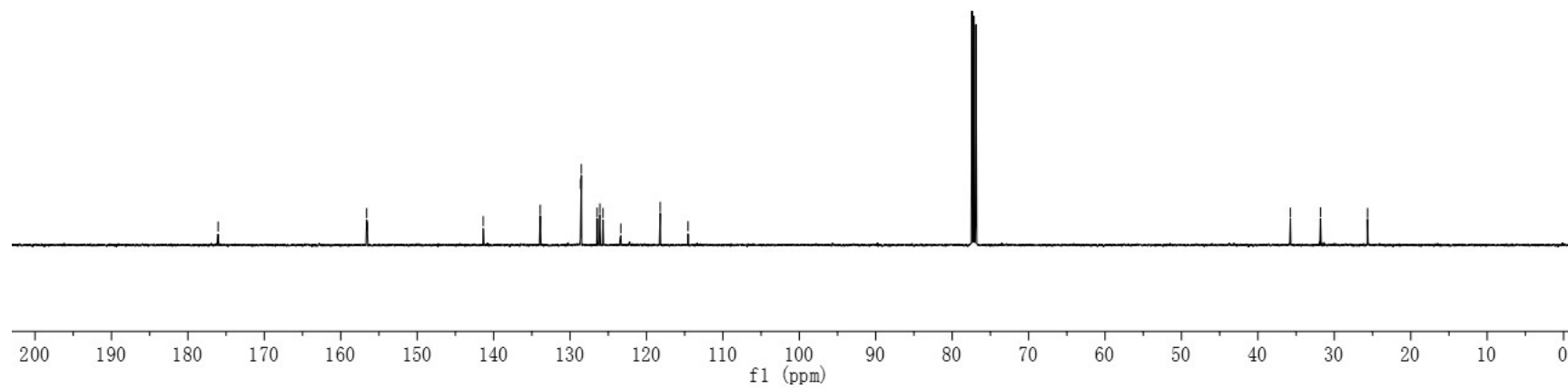
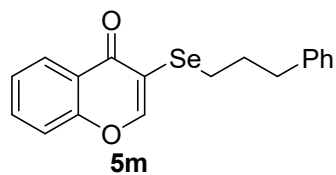
^{13}C NMR (126 MHz, CDCl_3) of **5I**



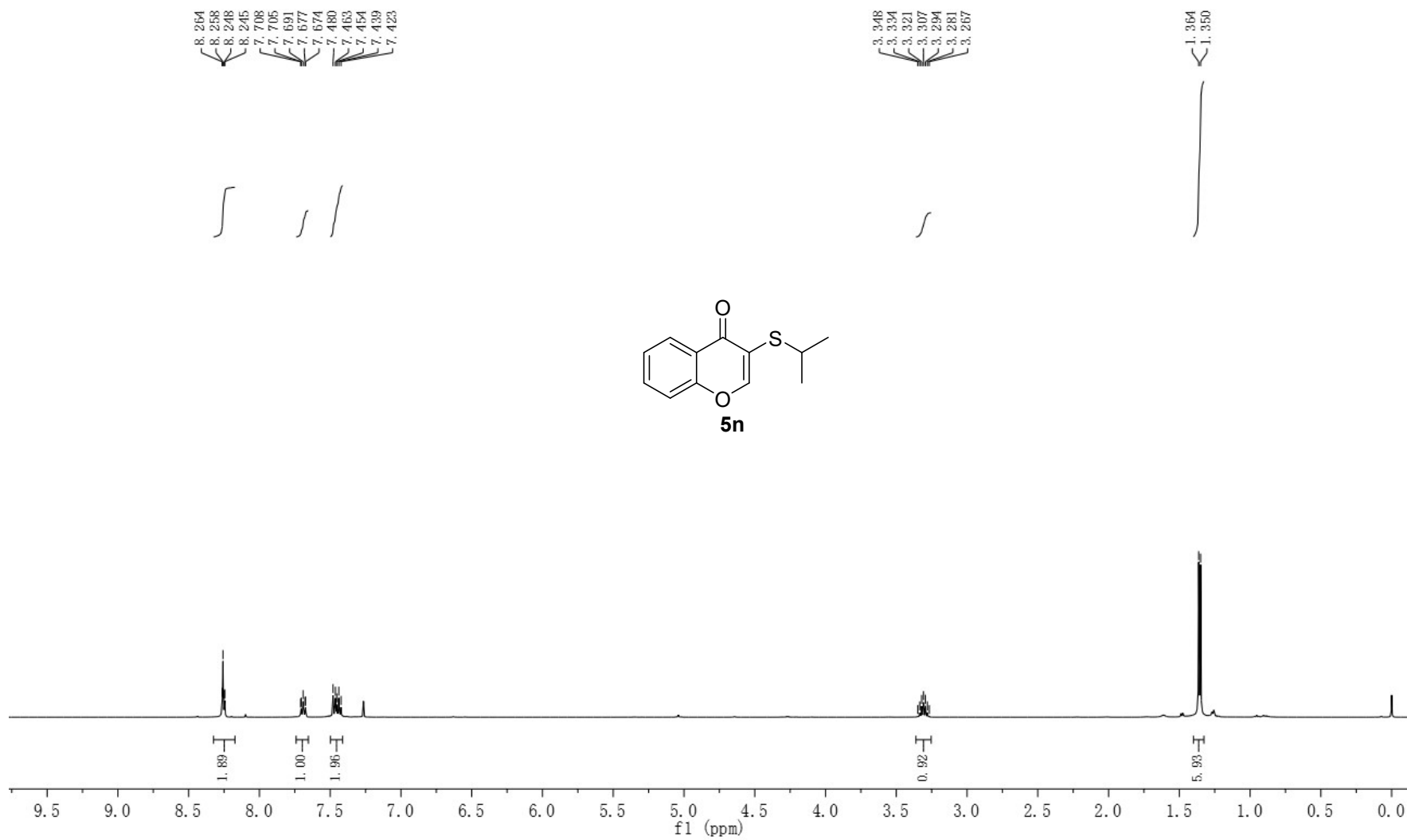
^1H NMR (500 MHz, CDCl_3) of **5m**



^{13}C NMR (126 MHz, CDCl_3) of **5m**



^1H NMR (500 MHz, CDCl_3) of **5n**



^{13}C NMR (126 MHz, CDCl_3) of **5n**

— 175.63

— 156.44
— 155.92

— 134.09

— 126.31

— 125.80

— 123.59

— 123.15

— 118.31

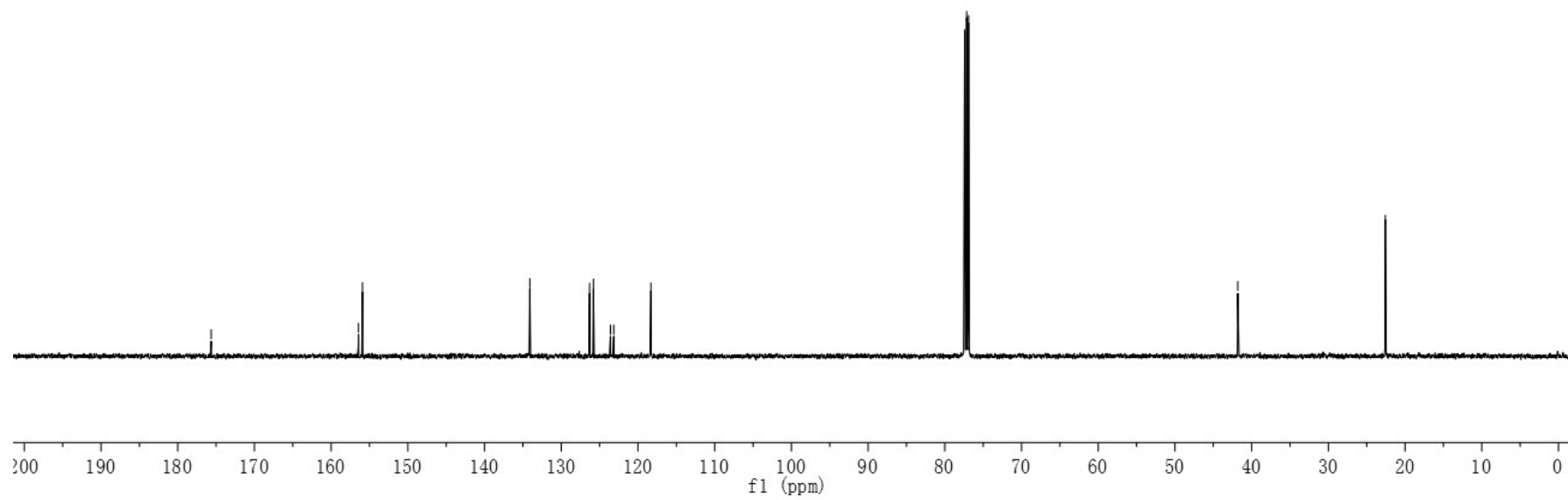
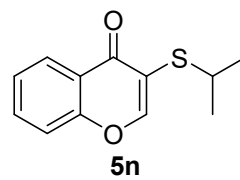
— 77.40

— 77.15

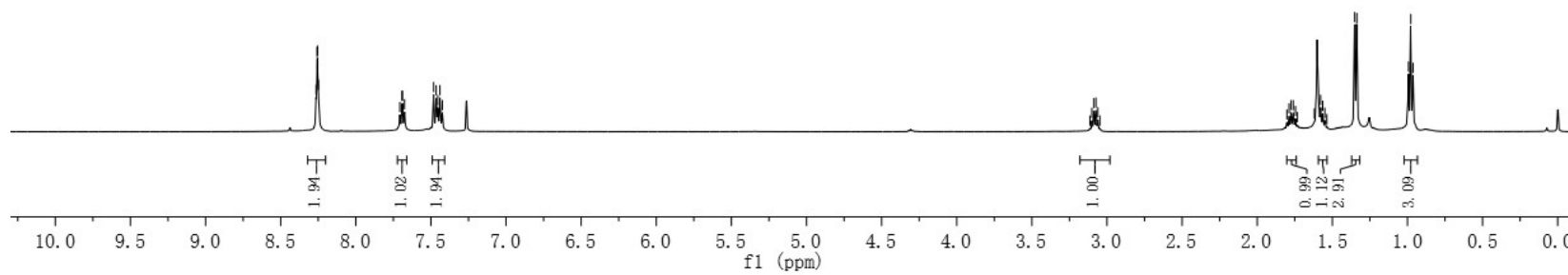
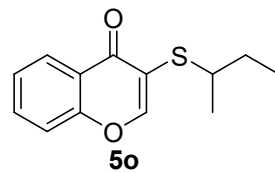
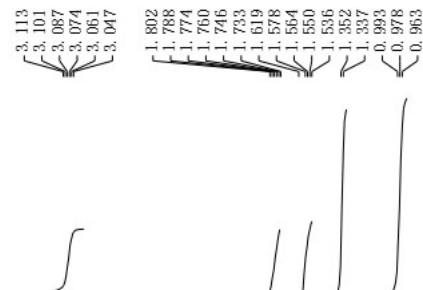
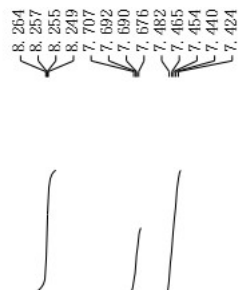
— 76.90

— 41.80

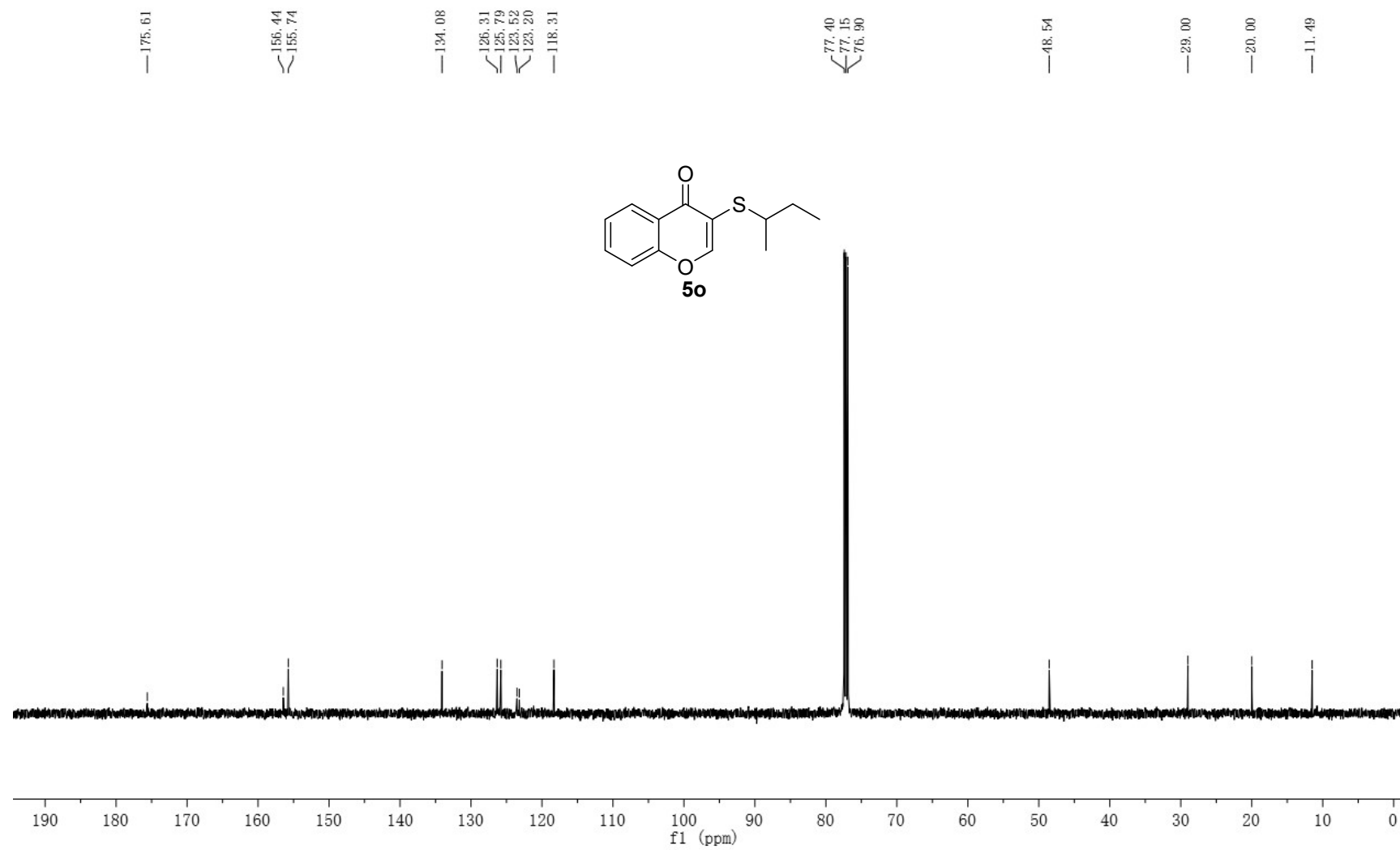
— 22.58



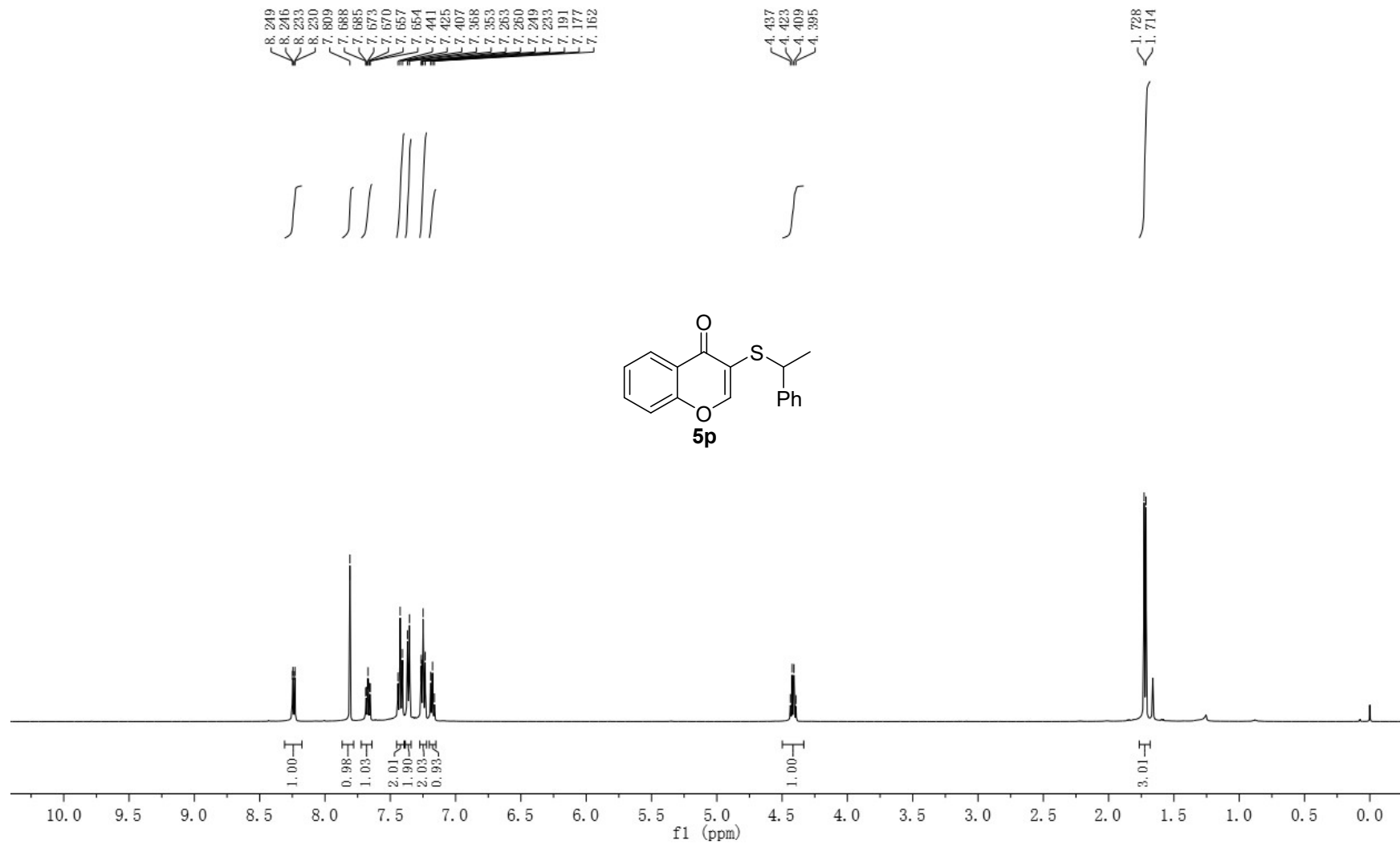
^1H NMR (500 MHz, CDCl_3) of **5o**



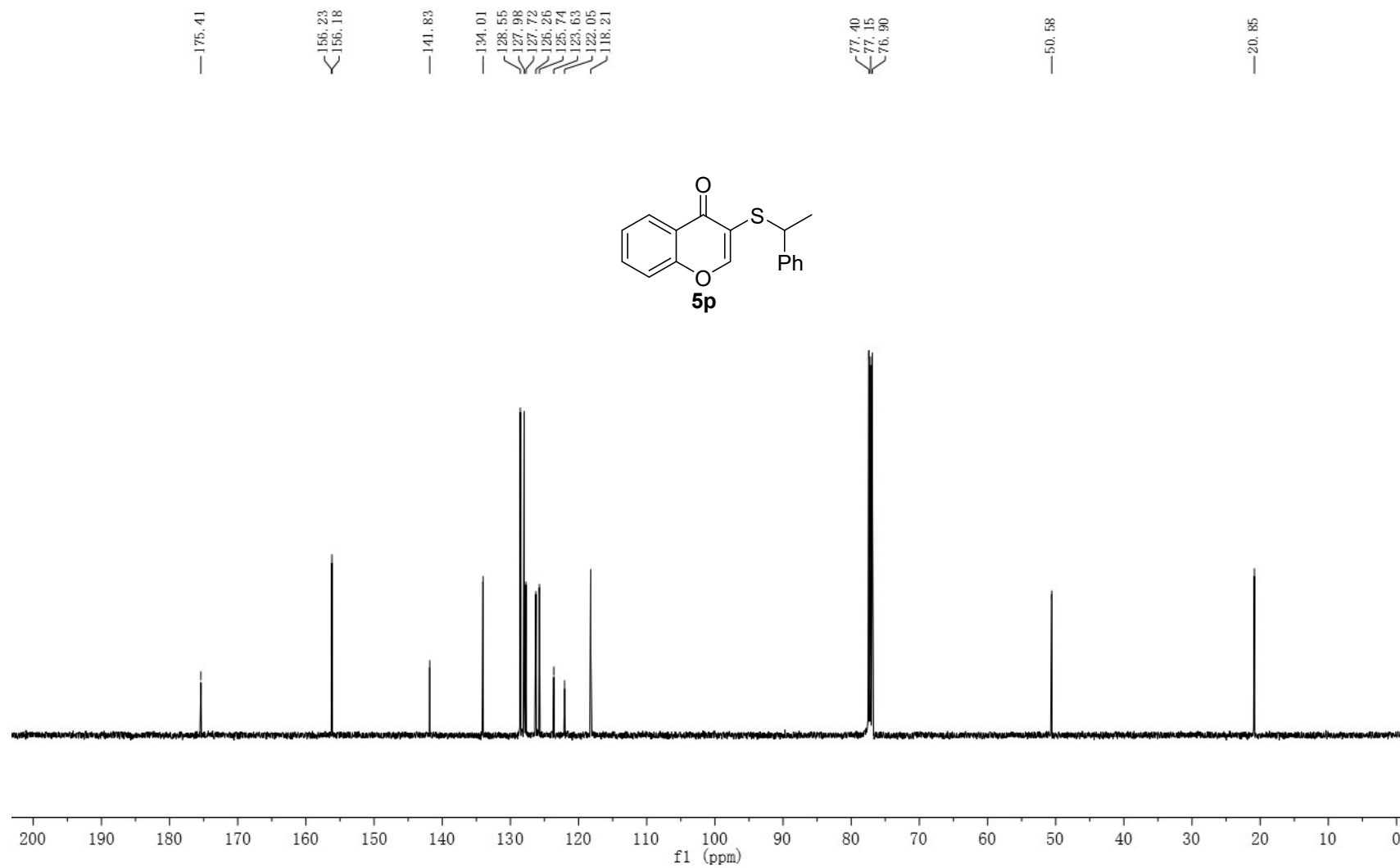
^{13}C NMR (126 MHz, CDCl_3) of **5o**



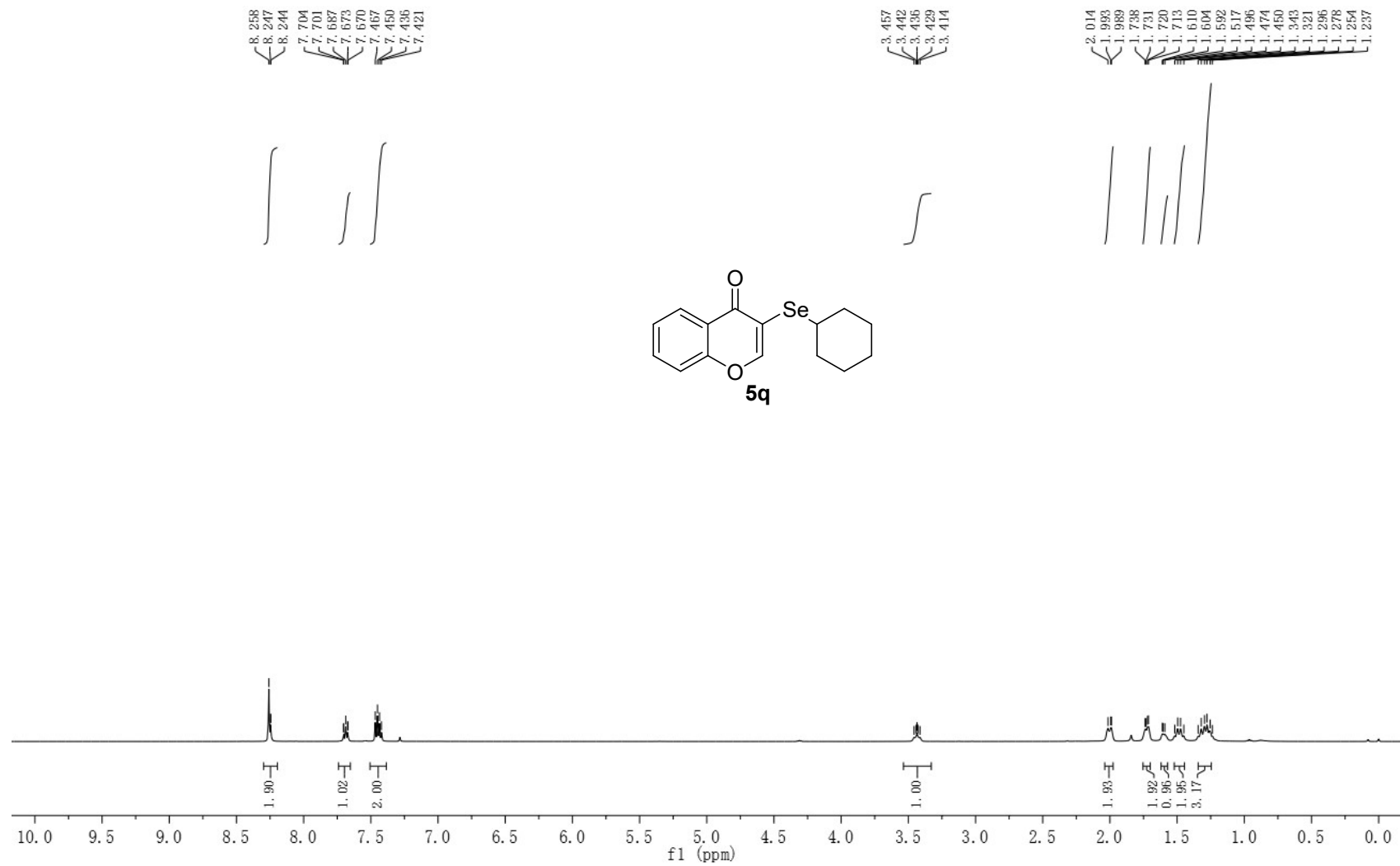
^1H NMR (500 MHz, CDCl_3) of **5p**



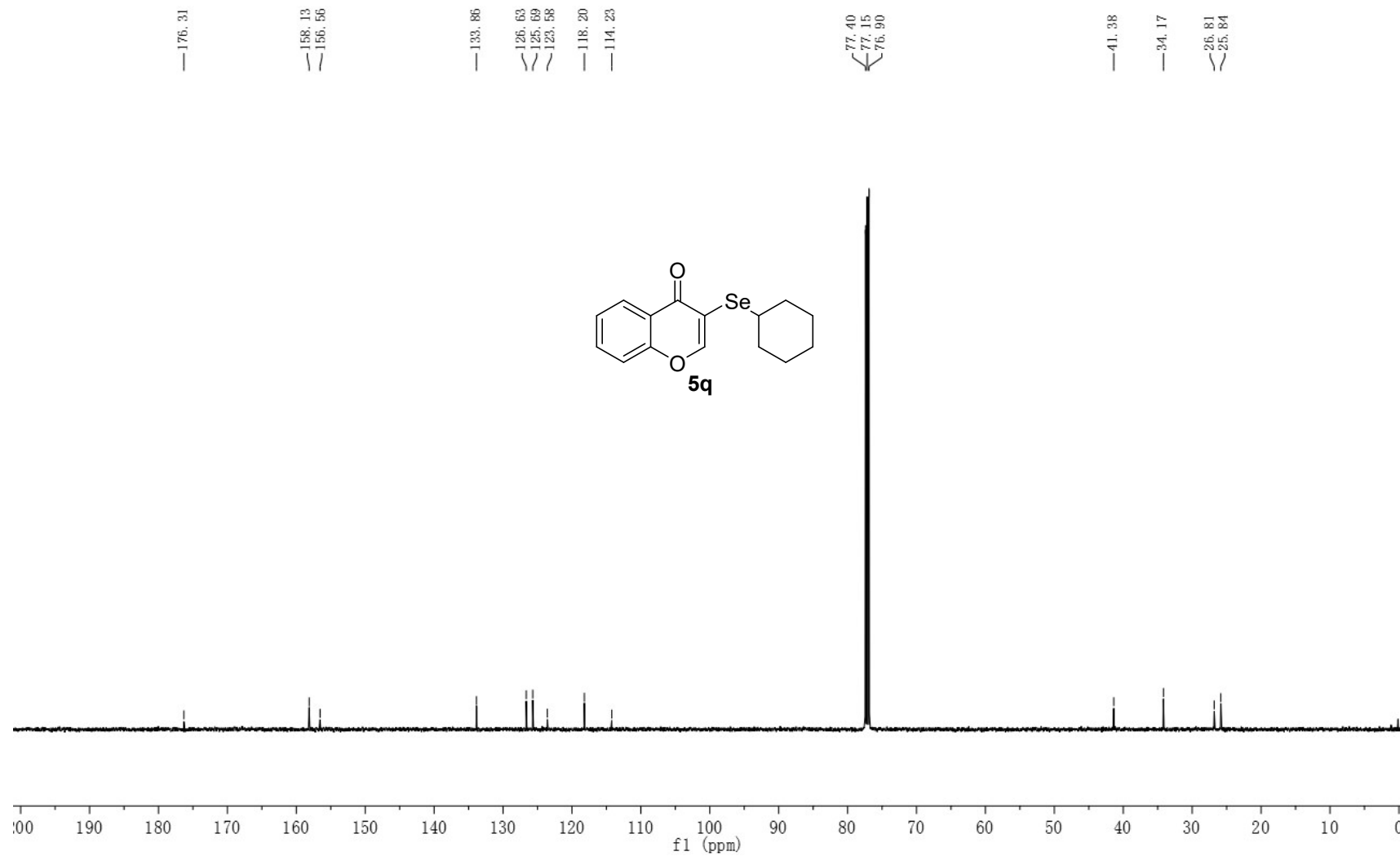
^{13}C NMR (126 MHz, CDCl_3) of **5p**



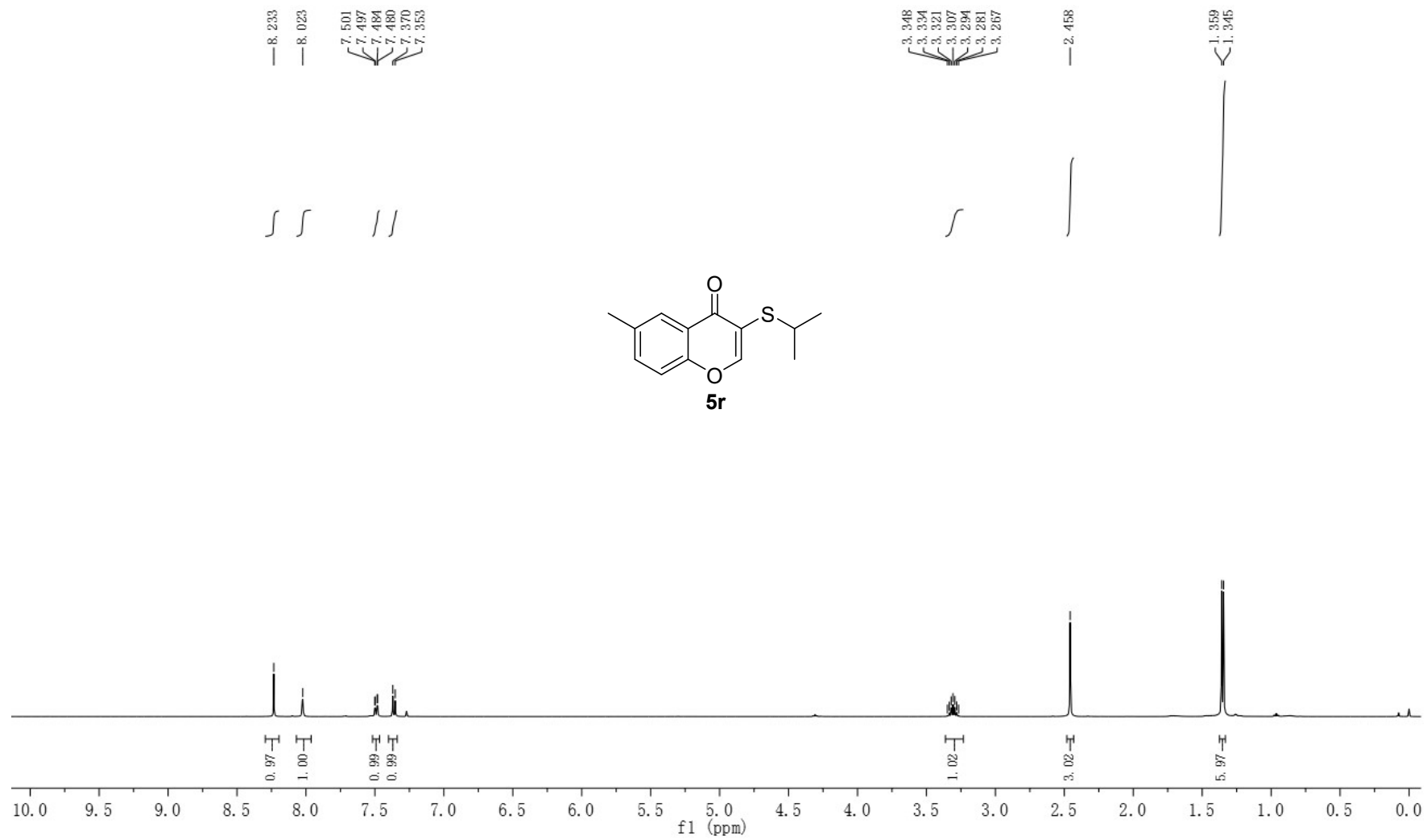
^1H NMR (500 MHz, CDCl_3) of **5q**



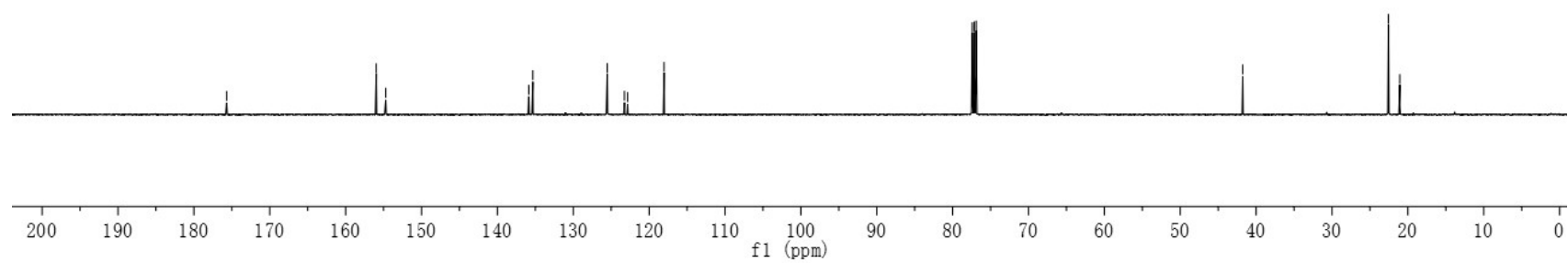
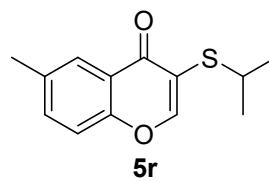
^{13}C NMR (126 MHz, CDCl_3) of **5q**



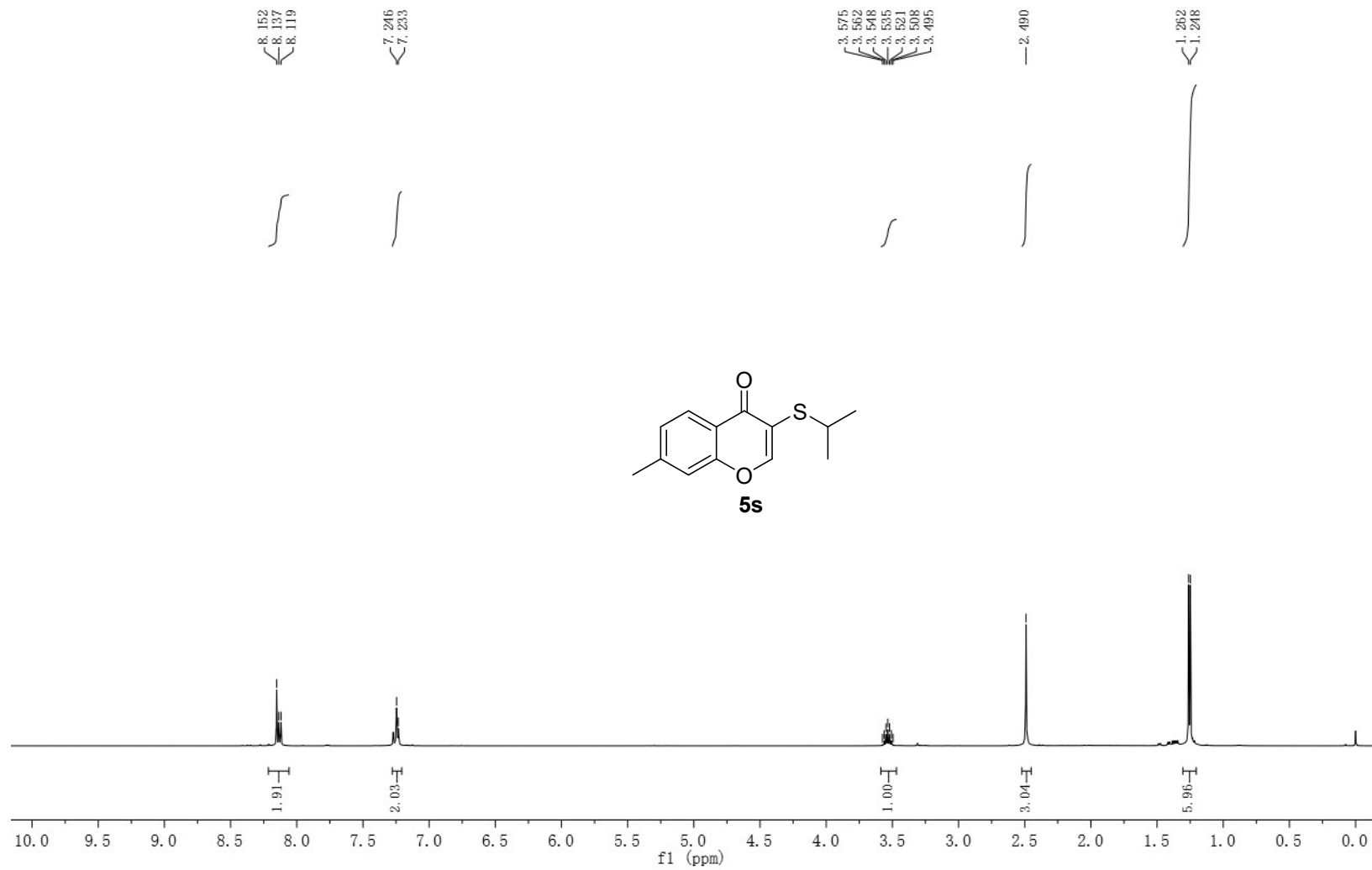
^1H NMR (500 MHz, CDCl_3) of **5r**



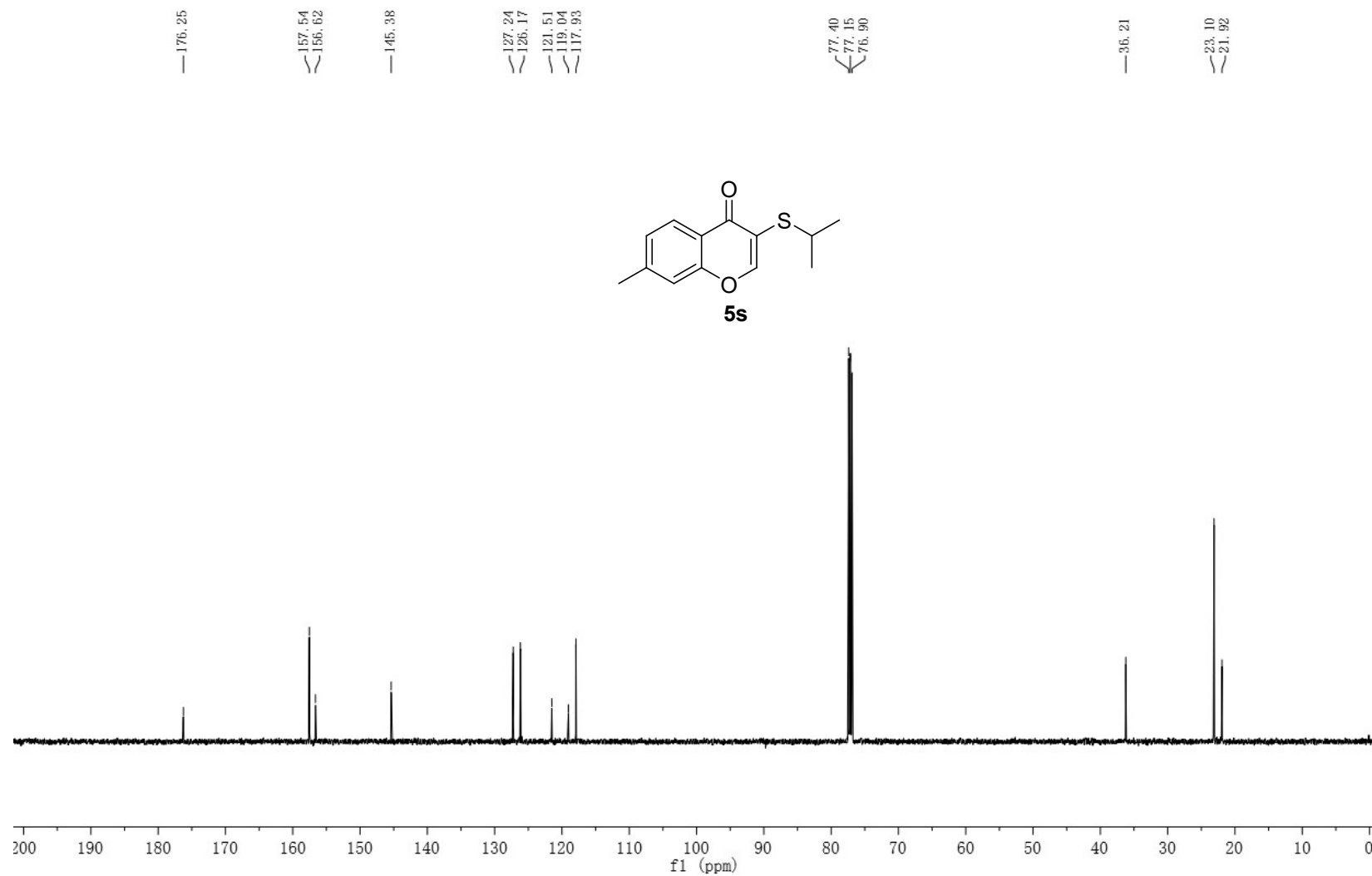
^{13}C NMR (126 MHz, CDCl_3) of **5r**



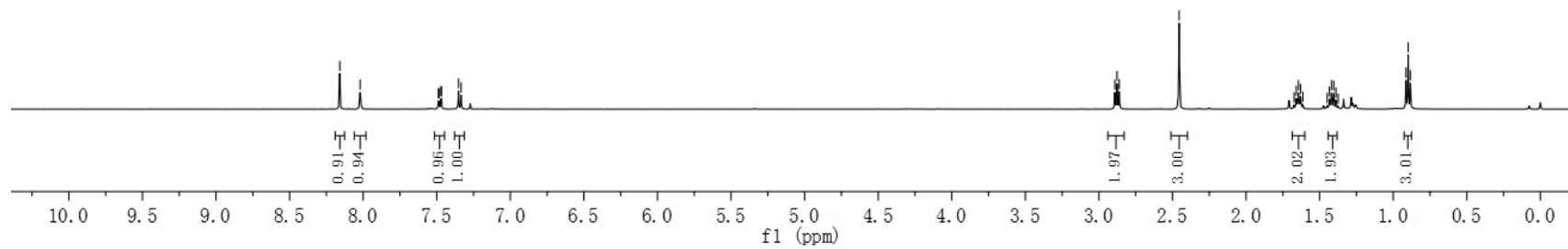
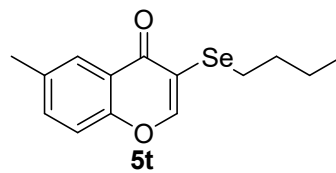
^1H NMR (500 MHz, CDCl_3) of **5s**



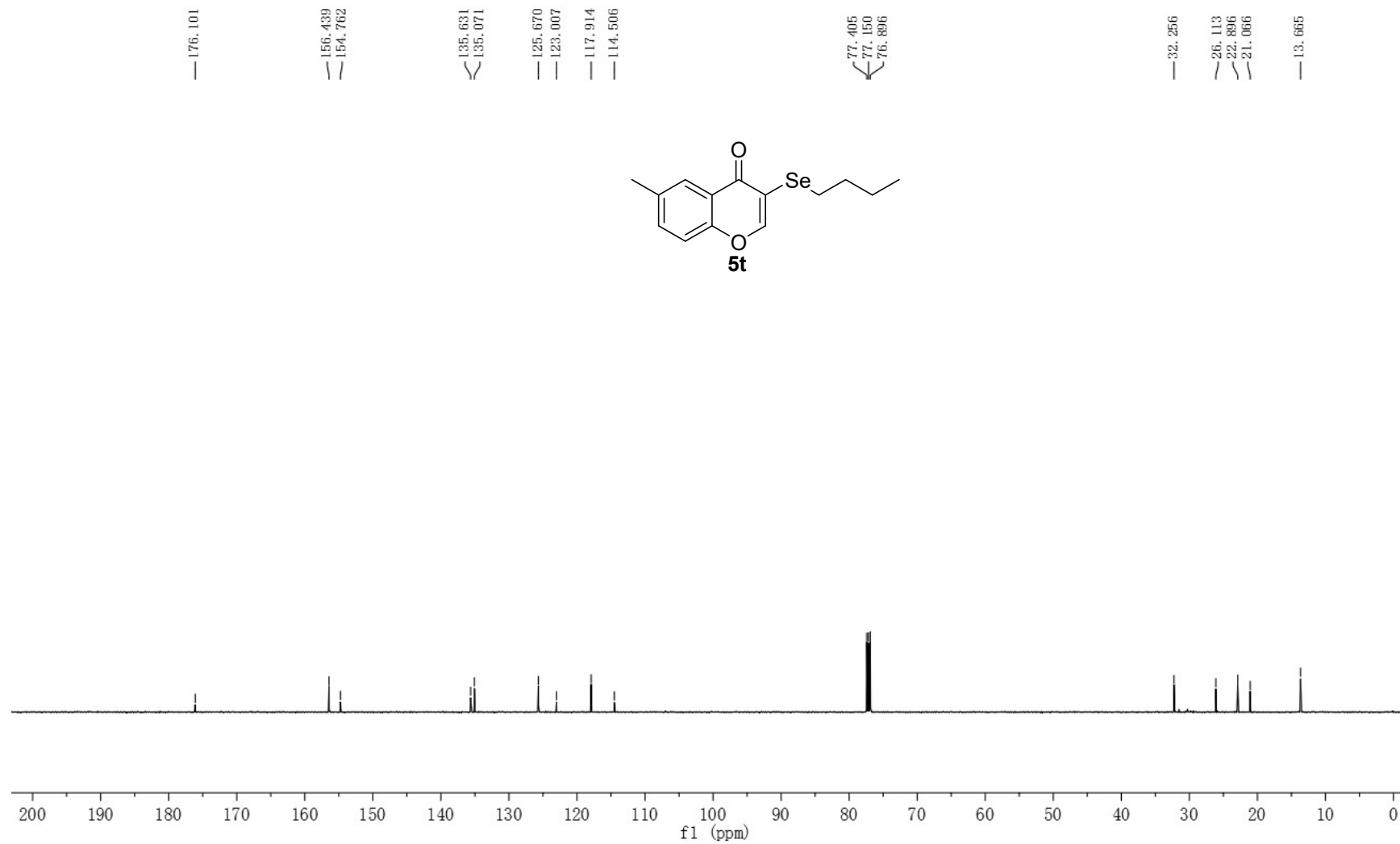
^{13}C NMR (126 MHz, CDCl_3) of **5s**



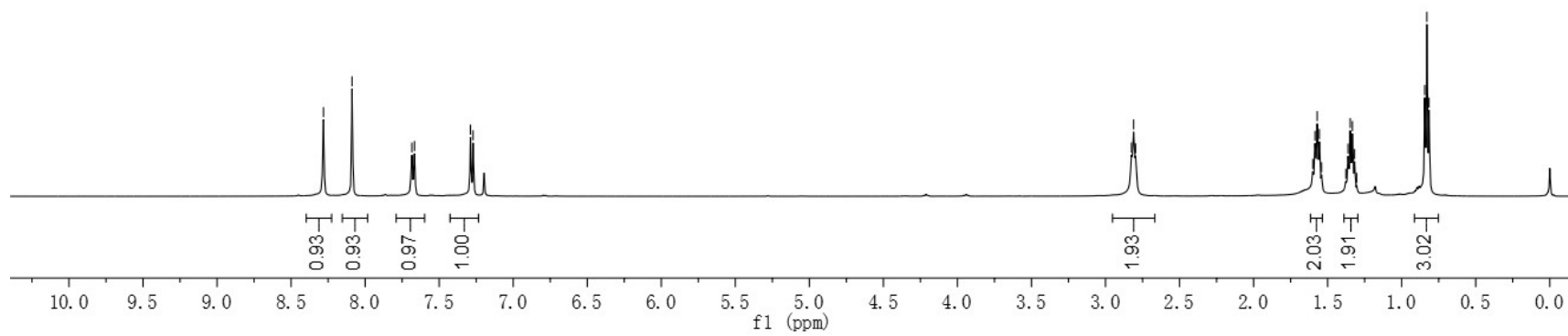
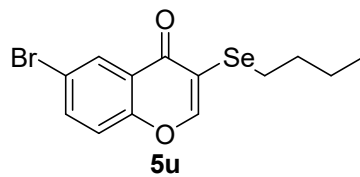
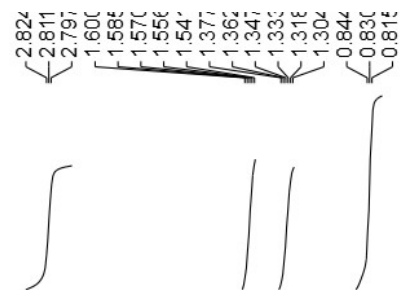
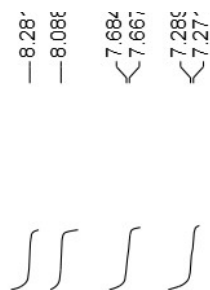
^1H NMR (500 MHz, CDCl_3) of **5t**



^{13}C NMR (126 MHz, CDCl_3) of **5t**



^1H NMR (500 MHz, CDCl_3) of **5u**



^{13}C NMR (126 MHz, CDCl_3) of **5u**

— 174.80

— 156.30
— 155.20

— 136.84

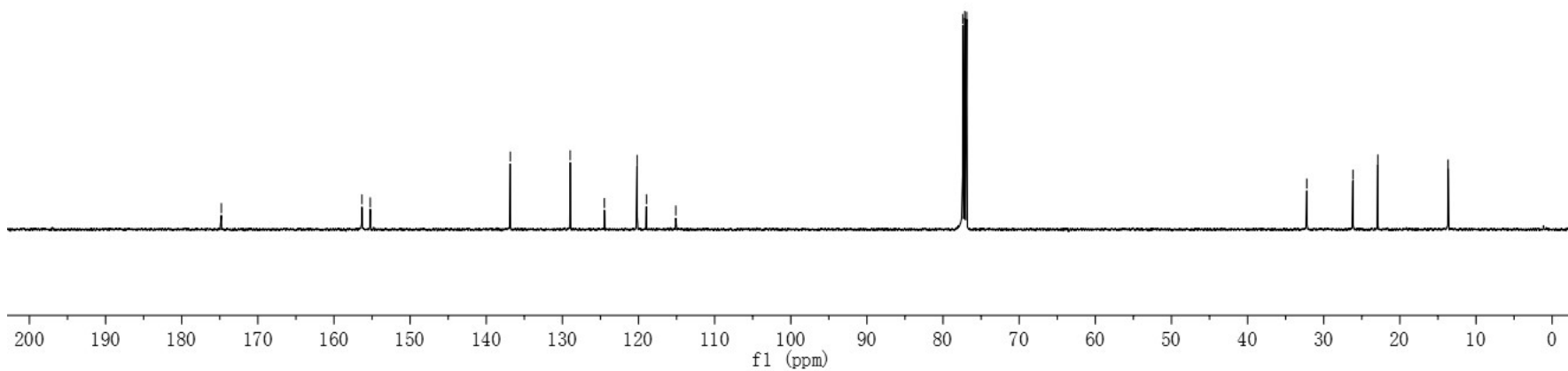
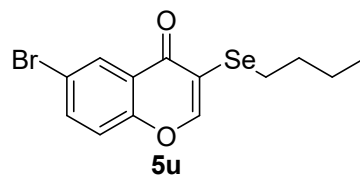
— 128.97
— 124.48
— 120.18
— 118.98
— 115.10

— 77.40
— 77.15
— 76.90

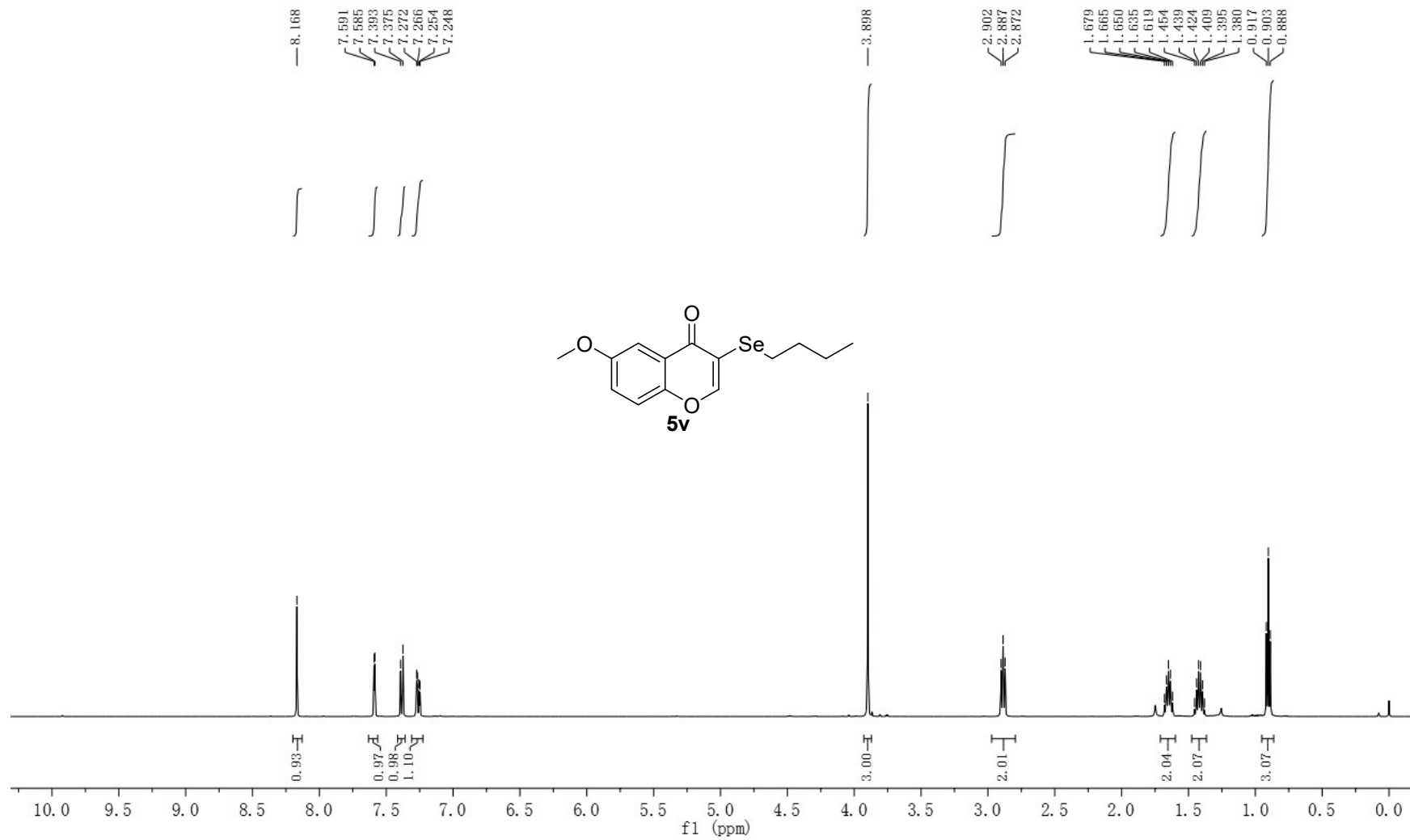
— 32.23

— 26.16
— 22.90

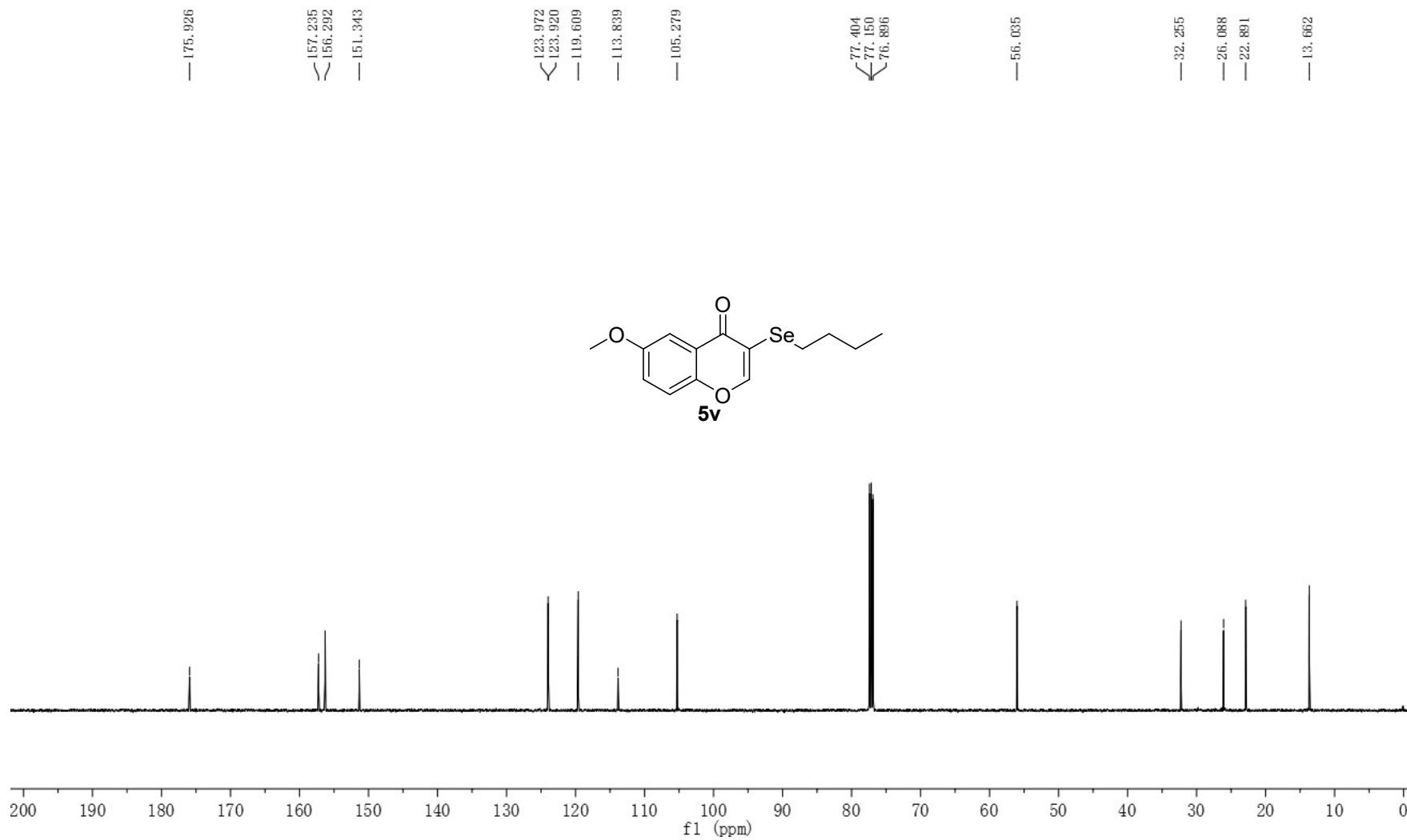
— 13.66



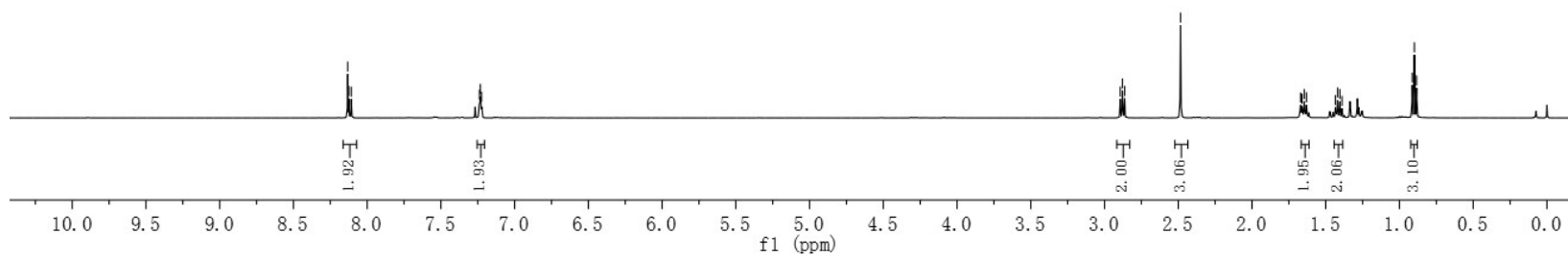
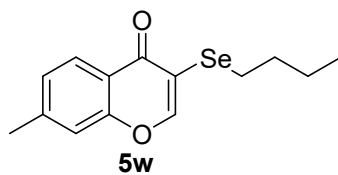
^1H NMR (500 MHz, CDCl_3) of **5v**



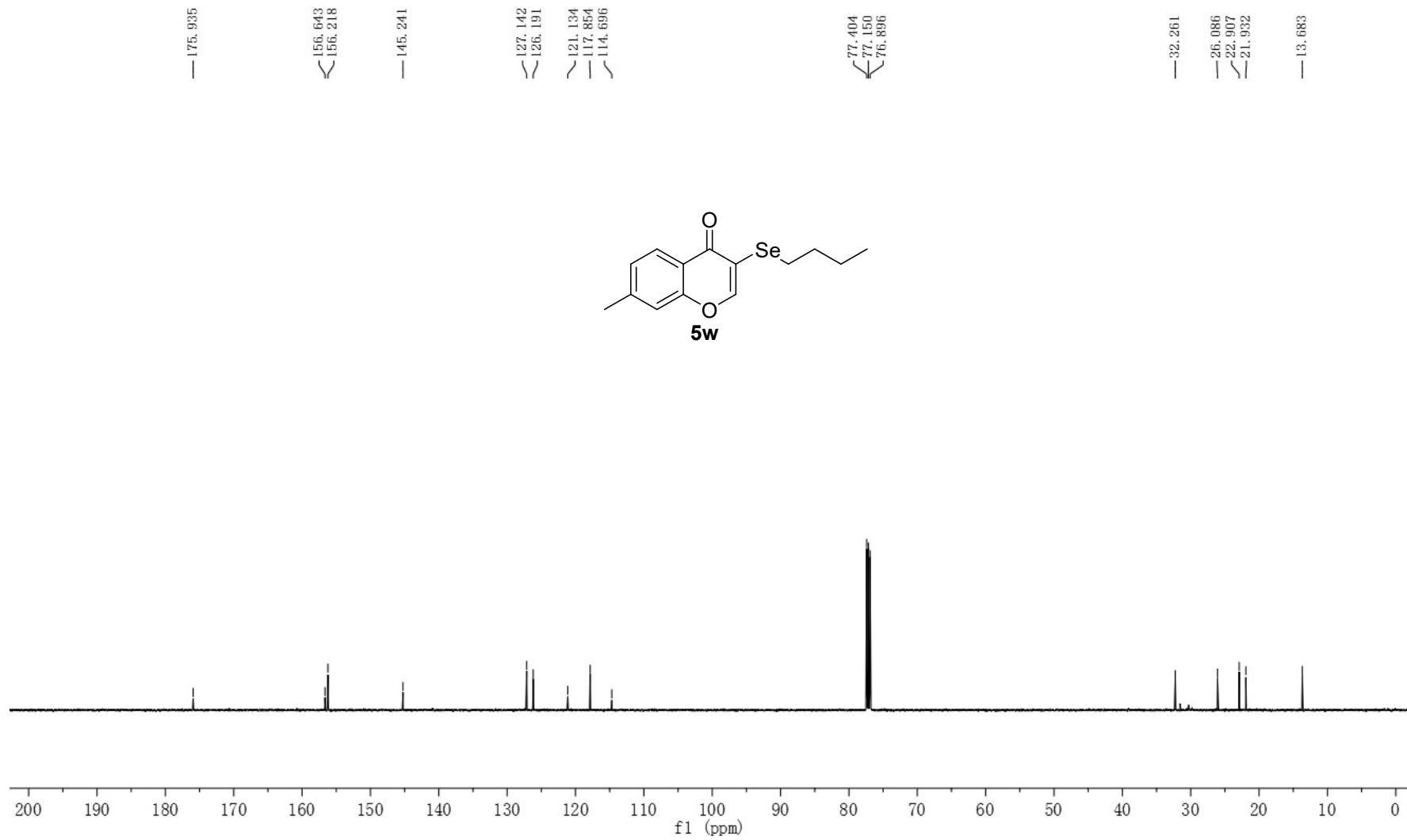
^{13}C NMR (126 MHz, CDCl_3) of **5v**



^1H NMR (500 MHz, CDCl_3) of **5w**



^{13}C NMR (126 MHz, CDCl_3) of **5w**

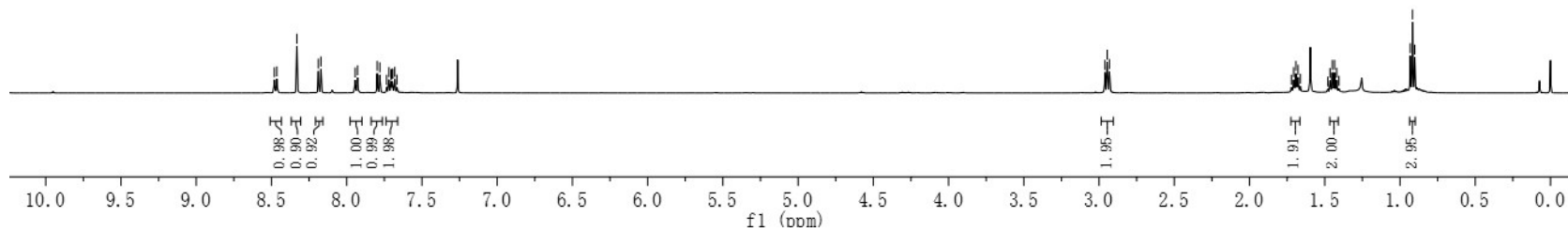
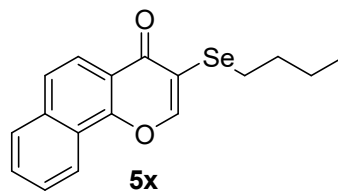
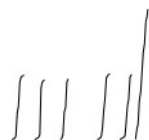


^1H NMR (500 MHz, CDCl_3) of **5x**

8.481
8.464
8.331
8.189
8.171
7.943
7.928
7.797
7.780
7.735
7.719
7.706
7.703
7.697
7.694
7.680
7.667

2.961
2.946
2.931

1.722
1.707
1.692
1.677
1.662
1.480
1.465
1.450
1.435
1.420
1.406
0.932
0.917
0.902



^{13}C NMR (126 MHz, CDCl_3) of **5x**

