

Supporting Information

Facile Synthesis of Selenocarbamyl Fluorides, Selenoureas and Their Derivatives with [Me₄N][SeCF₃]

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1. General information.

All reactions were carried out under a nitrogen atmosphere. Unless otherwise specified, NMR spectra were recorded in CDCl₃ on a 500 MHz (for ¹H), 471 MHz (for ¹⁹F), and 126 MHz (for ¹³C) spectrometer. All chemical shifts were reported in ppm relative to TMS (0 ppm for ¹H NMR) or PhCF₃ (-63.5 ppm for ¹⁹F NMR) as an internal or external standard. The HPLC experiments were carried out on a Wufeng LC-100 II instrument (column: Shodex, C18, 5 μm, 4.6 × 250 mm), and the yields of product were determined by using the corresponding pure compound as an external standard. The coupling constants were reported in Hertz (Hz). The following abbreviations were used to explain the multiplicities: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, brs = broad singlet. Melting points were measured and uncorrected. MS experiments were performed on a TOF-Q ESI instrument. [Me₄N][SeCF₃] was synthesized according to the literature.¹ Dibenzylamine derivatives (**1b-f**) were prepared according to the literatures.² Solvents were purified according to the literature.³ Other reagents used in the reactions were all purchased from the commercial sources and used without further purification.

2. Screening of the optimal conditions for the reactions of dibenzylamine (**1a**) with [Me₄N][SeCF₃].

Table S1. Additive-free reactions of **1a** with [Me₄N][SeCF₃] in different solvents.

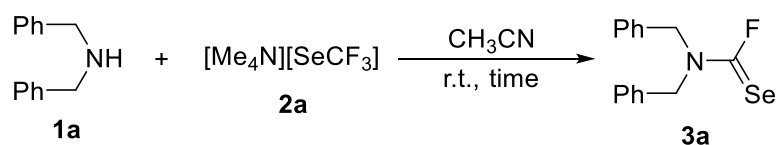
Reaction scheme: **1a** (dibenzylamine) + **2a** ([Me₄N][SeCF₃]) $\xrightarrow[\text{r.t., 12 h}]{\text{solvent}}$ **3a** (N-(dibenzyl)seleconium fluoride)

Entry ^a	Solvent	Yield (3a , %) ^b
1	CH ₃ CN	96 (93)
2	THF	80
3	DCM	90
4	1,4-dioxane	68
5	NMP	66
6	DMF	60
7	DMAc	56
8	DCE	48

9	DME	42
10	<i>m</i> -xylene	42
11	chlorobenzene	37
12	toluene	32

^a Reaction conditions: **1a** (0.20 mmol), **2a** (0.24 mmol), solvent (2 mL), N₂, room temperature. ^b Yields were determined by HPLC using **3a** as an external standard (*t_R* = 9.53 min, λ_{max} = 284 nm, water / methanol = 20 / 80 (v / v)). Isolated yield is depicted in the parenthesis.

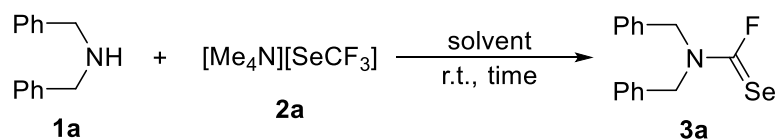
Table S2. Reactions of **1a** and [Me₄N][SeCF₃] with different times.



Entry ^a	Time	Yield (3a , %) ^b
1	2 min	82
2	5 min	87
3	10 min	93
4	30 min	96
5	1 h	96
6	2 h	98
7	4 h	96
8	12 h	96

^a Reaction conditions: **1a** (0.20 mmol), **2a** (0.24 mmol), CH₃CN (2 mL), N₂, room temperature. ^b Yields were determined by HPLC using **3a** as an external standard (*t_R* = 9.53 min, λ_{max} = 284 nm, water / methanol = 20 / 80 (v / v)).

Table S3. Reactions of **1a** and [Me₄N][SeCF₃] with different molar ratios.



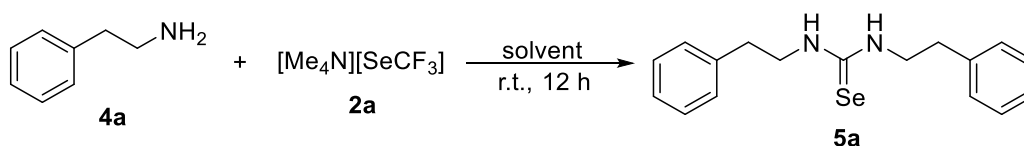
Entry ^a	Time	1a:2a	Yield (3a , %) ^b
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1	10 min	1:1.2	93
2	1 h	1:1.2	96
3	12 h	1:1.2	96
4	10 min	1:1	82
5	1 h	1:1	84
6	12 h	1:1	90
7	10 min	1:1.1	82
8	1 h	1:1.1	82
9	12 h	1:1.1	92
10	12 h	1.5:1	99 ^c
11	12 h	2:1	> 99 ^c

^a Reaction conditions: **1a** (0.20, 0.30, or 0.40 mmol), **2a** (0.20, 0.22, or 0.24 mmol), CH₃CN (2 mL), N₂, room temperature. ^b Yields were determined by HPLC using **3a** as an external standard ($t_R = 9.53$ min, $\lambda_{max} = 284$ nm, water / methanol = 20 / 80 (v / v)). ^c Isolated yield.

3. Screening of the optimal conditions for the reactions of phenethylamine (**4a**) with [Me₄N][SeCF₃].

Table S4. Additive-free reactions of **4a** with [Me₄N][SeCF₃] in different solvents.

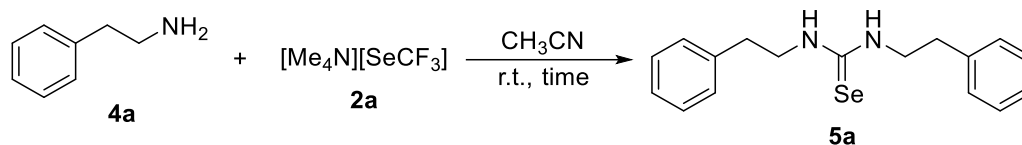


Entry ^a	Solvent	Yield (5a , %) ^b
1	CH ₃ CN	96
2	THF	89
3	DME	89
4	1,4-dioxane	73
5	DCM	58
6	DMF	49
7	DMSO	40

^a Reaction conditions: **4a** (0.40 mmol), **2a** (0.20 mmol), solvent (2 mL), N₂, room

temperature. ^b Yields were determined by HPLC using **5a** as an external standard ($t_R = 6.30$ min, $\lambda_{max} = 218$ nm, water / methanol = 20 / 80 (v / v)).

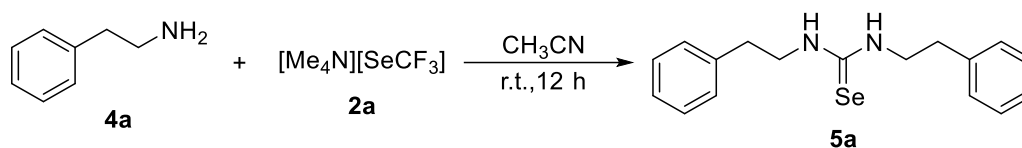
Table S5. Reactions of **4a** and $[\text{Me}_4\text{N}][\text{SeCF}_3]$ with different times.



Entry ^a	Time	Yield (5a , %) ^b
1	4 h	77
2	6 h	81
3	8 h	98 (93)
4	12 h	96
5	24 h	98

^a Reaction conditions: **4a** (0.40 mmol), **2a** (0.20 mmol), CH_3CN (2 mL), N_2 , room temperature. ^b Yields were determined by HPLC using **5a** as an external standard ($t_R = 6.30$ min, $\lambda_{max} = 218$ nm, water / methanol = 20 / 80 (v / v)). Isolated yield is depicted in the parenthesis.

Table S6. Reactions of **4a** and $[\text{Me}_4\text{N}][\text{SeCF}_3]$ with different molar ratios.



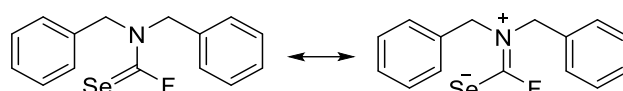
Entry ^a	1a:2a	Yield (5a , %) ^b
1	1:1	66%, 70%
2	1:1.5	66%, 72%
3	1:2	66%, 71%
4	1.5:1	79%, 82%
5	2:1	93%

^a Reaction conditions: **4a** (0.20, 0.30 or 0.40 mmol), **2a** (0.20, 0.30 or 0.40 mmol), CH_3CN (2 mL), N_2 , room temperature. ^b Isolated yield.

4. General procedure for the synthesis of selenocarbamoyl fluorides (**3**).

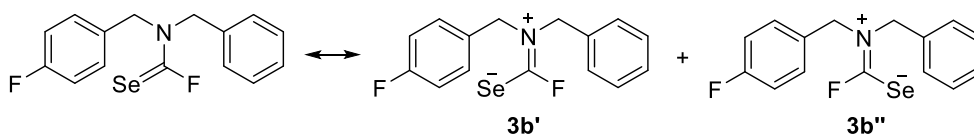
In a nitrogen-filled glovebox, a tube was charged with secondary aliphatic amines (**1**, 0.20 mmol), [Me₄N][SeCF₃] (**2a**, 53.2 mg, 0.24 mmol), and MeCN (2 mL) with stirring. The mixture was reacted at room temperature for 30 min and concentrated to dryness under reduced pressure. The residue was purified by column chromatography on silica gel using dichloromethane or a mixture of petroleum ether and ethyl acetate as eluents to give the title compound (**3**).

Dibenzylcarbamoseleoic fluoride (**3a**)



Light yellow oil (57.1 mg, 93%), a mixture of petroleum ether/ethyl acetate = 10/1 (v/v) as eluents for column chromatography. ¹H NMR (500 MHz, CDCl₃) δ 7.41-7.35 (m, 7.85H), 7.18 (d, *J* = 6.7 Hz, 2.00H), 5.07 (s, 2H), 4.58 (s, 2H). ¹⁹F NMR (471 MHz, CDCl₃) δ 27.2 (m). ¹³C NMR (126 MHz, CDCl₃) δ 188.6 (d, *J* = 350 Hz), 133.4, 133.2, 129.2, 129.1, 128.7, 128.7, 128.3, 127.9, 57.5 (d, *J* = 7.0 Hz), 51.3 (d, *J* = 7.4 Hz). IR (KBr): 3063, 3030, 2926, 2852, 1605, 1586, 1517, 1496, 1453, 1358, 1262, 1232, 1202, 1170, 1082, 1072, 1029, 1001, 939, 887, 824, 752, 735, 700, 629 cm⁻¹. HRMS-ESI (m/z) calcd. for [C₁₅H₁₅FNSe]⁺ ([M + H]⁺): 308.0348; found: 308.0356.

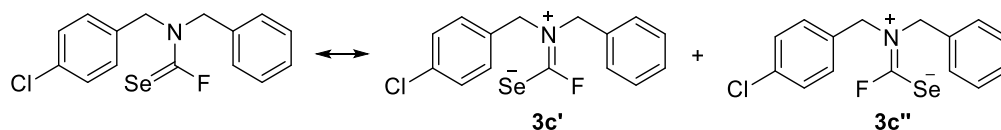
Benzyl(4-fluorobenzyl)carbamoseleoic fluoride (**3b**)



Light yellow oil (50.7 mg, 78%), a mixture of petroleum ether/ethyl acetate = 10/1 (v/v) as eluents for column chromatography. ¹H NMR (500 MHz, CDCl₃, **3b'** + **3b''**) δ 7.39-7.37 (m, 4.94H), 7.18-7.13 (m, 2.00H), 7.09-7.05 (m, 2.01H), 5.06 (s) + 5.03 (s) (2H), 4.57 (s) + 4.55 (s) (2H). ¹⁹F NMR (471 MHz, CDCl₃, **3b'** + **3b''**) δ 27.4 (m), 27.3 (m), -112.6 (m), -112.7 (m). ¹³C NMR (126 MHz, CDCl₃, **3b'** + **3b''**) δ 187.1 (d, *J* = 350.8 Hz), 187.1 (d, *J* = 350.2 Hz), 162.9 (d, *J* = 248.1 Hz), 162.8 (d, *J* = 248.5 Hz), 133.2, 133.0, 130.3 (d, *J* = 8.3 Hz), 129.9 (d, *J* = 8.4 Hz), 129.3, 129.3 (d, *J* = 3.1 Hz), 129.1, 129.0 (d, *J* = 3.3 Hz), 128.8, 128.8, 128.3, 127.9, 116.2 (d, *J* = 21.5 Hz), 116.1 (d, *J* = 21.5 Hz), 57.6 (d, *J* = 7.0 Hz), 56.8 (d, *J* = 6.8 Hz), 51.3 (d, *J* = 7.4 Hz), 50.6 (d, *J* = 7.5 Hz). IR (KBr): 3064, 3032, 2928, 2854, 1606, 1510, 1446, 1416, 1357, 1261, 1225, 1203, 1170, 1158, 1100, 1081, 1015, 995, 932, 851, 823, 748, 701, 667

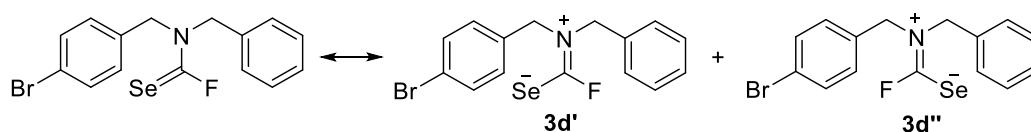
cm⁻¹. HRMS-ESI (m/z) calcd. for [C₁₅H₁₄F₂NSe]⁺ ([M + H]⁺): 327.0288; found: 327.0285.

Benzyl(4-chlorobenzyl)carbamoselenoic fluoride (**3c**)



Light yellow oil (55.9 mg, 82%), a mixture of petroleum ether/ethyl acetate = 10/1 (v/v) as eluents for column chromatography. ¹H NMR (500 MHz, CDCl₃, **3c'** + **3c''**) δ 7.40-7.31 (m, 6.88H), 7.17 (d, *J* = 7.2 Hz, 1.13H), 7.10 (d, *J* = 8.3 Hz, 0.94H), 5.05 (s) + 5.02 (s) (2H), 4.57 (s) + 4.54 (s) (2H). ¹⁹F NMR (471 MHz, CDCl₃, **3c'** + **3c''**) δ 27.7 (m), 27.3 (m). ¹³C NMR (126 MHz, CDCl₃, **3c'** + **3c''**) δ 187.3 (d, *J* = 350.8 Hz), 187.1 (d, *J* = 350.8 Hz), 134.7, 134.7, 133.2, 132.9, 131.9, 131.7, 129.7, 129.4, 129.3, 129.3, 129.2, 128.8, 128.8, 128.3, 127.9, 57.7 (d, *J* = 7.0 Hz), 56.8 (d, *J* = 6.8 Hz), 51.5 (d, *J* = 7.4 Hz), 50.6 (d, *J* = 7.5 Hz). IR (KBr): 3063, 3030, 2928, 2853, 1598, 1586, 1514, 1493, 1444, 1408, 1356, 1282, 1229, 1168, 1087, 1015, 995, 934, 891, 842, 800, 727, 700, 669 cm⁻¹. HRMS-ESI (m/z) calcd. for [C₁₅H₁₄FCINSe]⁺ ([M + H]⁺): 341.9959; found: 341.9952.

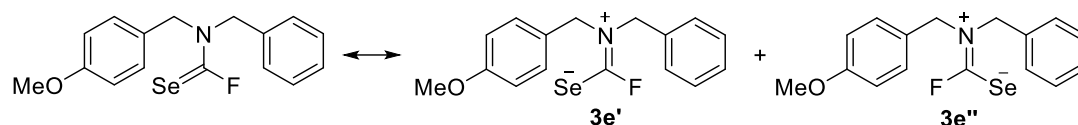
Benzyl(4-bromobenzyl)carbamoselenoic fluoride (**3d**)



Light yellow oil (55.3 mg, 77%), a mixture of petroleum ether/ethyl acetate = 10/1 (v/v) as eluents for column chromatography. ¹H NMR (500 MHz, CDCl₃, **3d'** + **3d''**) δ 7.51 (dd, *J* = 8.4, 2.7 Hz, 1.89H), 7.39-7.37 (m, 3.86H), 7.26 (d, *J* = 8.3 Hz, 1.19H), 7.17 (d, *J* = 6.5 Hz, 1.09H), 7.04 (d, *J* = 8.3 Hz, 0.94H), 5.05 (s) + 5.00 (s) (2H), 4.57 (s) + 4.52 (s) (2H). ¹⁹F NMR (471 MHz, CDCl₃, **3d'** + **3d''**) δ 27.7 (m), 27.3 (m). ¹³C NMR (126 MHz, CDCl₃, **3d'** + **3d''**) δ 187.3 (d, *J* = 350.6 Hz), 187.2 (d, *J* = 350.3 Hz), 133.2, 132.9, 132.4, 132.4, 132.2, 132.2, 130.0, 129.6, 129.3, 129.2, 128.8, 128.8, 128.3, 127.9, 122.8, 122.8, 57.7 (d, *J* = 7.0 Hz), 56.9 (d, *J* = 6.8 Hz), 51.5 (d, *J* = 7.4 Hz), 50.7 (d, *J* = 7.5 Hz). IR (KBr): 3062, 3030, 2928, 2853, 1604, 1514, 1441, 1405, 1355, 1280, 1229, 1169, 1107, 1072, 1012, 934, 891, 839, 795, 748, 724, 701, 669, 642 cm⁻¹. HRMS-ESI (m/z) calcd. for [C₁₅H₁₄FBrNSe]⁺ ([M + H]⁺): 385.9453; found:

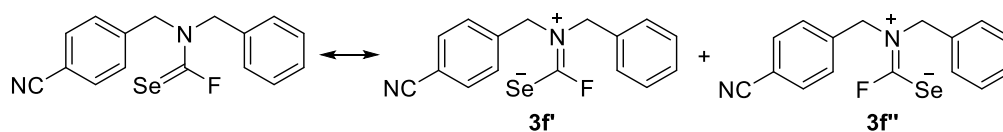
385.9450.

Benzyl(4-methoxybenzyl)carbamoseleoic fluoride (**3e**)



Light yellow oil (62.7 mg, 93%), a mixture of petroleum ether/ethyl acetate = 5/1 (v/v) as eluents for column chromatography. ^1H NMR (500 MHz, CDCl_3 , **3e'** + **3e''**) δ 7.41-7.35 (m, 3.94H), 7.34 (d, $J = 8.6$ Hz, 1.01H), 7.18 (d, $J = 6.8$ Hz, 1.00H), 7.11 (d, $J = 8.5$ Hz, 1.00H), 6.91 (m, 2.00H), 5.04 (s) + 5.00 (s) (2H), 4.56 (s) + 4.51 (s) (2H), 3.82 (s) + 3.82 (s) (3H). ^{19}F NMR (471 MHz, CDCl_3 , **3e'** + **3e''**) δ 27.2 (m), 26.6 (m). ^{13}C NMR (126 MHz, CDCl_3 , **3e'** + **3e''**) δ 186.8 (d, $J = 350.1$ Hz), 186.7 (d, $J = 350.6$ Hz), 160.0, 159.9, 133.5, 133.2, 129.9, 129.6, 129.2, 129.1, 128.7, 128.6, 128.3, 127.9, 125.4, 125.0, 114.6, 114.4, 57.2 (d, $J = 6.9$ Hz), 57.0 (d, $J = 7.1$ Hz), 55.4, 55.4, 51.0 (d, $J = 7.3$ Hz), 50.8 (d, $J = 7.5$ Hz). IR (KBr): 3063, 3031, 3003, 2955, 2933, 2835, 1611, 1585, 1514, 1441, 1421, 1356, 1304, 1250, 1230, 1167, 1113, 1081, 1031, 993, 931, 889, 846, 816, 739, 700, 667 cm^{-1} . HRMS-ESI (m/z) calcd. for $[\text{C}_{16}\text{H}_{17}\text{FNOSe}]^+$ ($[\text{M} + \text{H}]^+$): 338.0454; found: 338.0463.

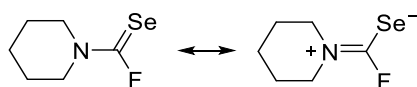
Benzyl(4-cyanobenzyl)carbamoseleoic fluoride (**3f**)



White solid (61.8 mg, 93%), a mixture of petroleum ether/ethyl acetate = 5/1 (v/v) as eluents for column chromatography. M.p.: 69.9-71.5 $^{\circ}\text{C}$. ^1H NMR (500 MHz, CDCl_3 , **3f'** + **3f''**) δ 7.66 (dd, $J = 8.2, 1.7$ Hz, 1.95H), 7.46 (d, $J = 8.2$ Hz, 1.15H), 7.40-7.34 (m, 3.82H), 7.25 (d, $J = 8.2$ Hz, 0.95H), 7.17 (m, 1.16H), 5.10 (s) + 5.08 (s) (2H), 4.63 (s) + 4.61 (s) (2H). ^{19}F NMR (471 MHz, CDCl_3 , **3f'** + **3f''**) δ 28.8 (m), 27.5 (m). ^{13}C NMR (126 MHz, CDCl_3 , **3f'** + **3f''**) δ 187.7 (d, $J = 351.5$ Hz), 187.4 (d, $J = 351.1$ Hz), 138.7, 138.6, 132.9, 132.8, 132.6, 129.4, 129.2, 129.0, 129.0, 128.7, 128.4, 128.3, 128.0, 118.3, 118.1, 112.6, 112.5, 58.4 (d, $J = 7.0$ Hz), 57.1 (d, $J = 6.4$ Hz), 52.3 (d, $J = 7.2$ Hz), 51.0 (d, $J = 7.3$ Hz). IR (KBr): 3058, 3027, 2958, 2918, 2848, 2226, 1609, 1586, 1522, 1438, 1414, 1355, 1293, 1242, 1170, 1094, 1018, 959, 941, 921, 898, 838, 810, 752, 723, 696, 677 cm^{-1} . HRMS-ESI (m/z) calcd. for $[\text{C}_{16}\text{H}_{14}\text{FN}_2\text{Se}]^+$ ($[\text{M} + \text{H}]^+$):

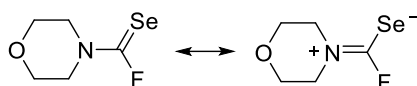
333.0301; found: 338.0302.

Piperidine-1-carboselenoyl fluoride (**3g**)



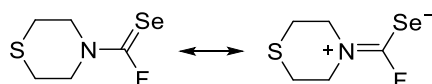
Yellow brown oil (24.9 mg, 64%), a mixture of petroleum ether/ethyl acetate = 40/1 (v/v) as eluents for column chromatography. ¹H NMR (500 MHz, CDCl₃) δ 4.05 (t, *J* = 5.6 Hz, 2H), 3.69 (t, *J* = 5.7 Hz, 2H), 1.79 (m, 2H), 1.74-1.65 (m, 4H). ¹⁹F NMR (471 MHz, CDCl₃) δ 24.0 (m). ¹³C NMR (126 MHz, CDCl₃) δ 183.4 (d, *J* = 350.0 Hz), 54.4 (d, *J* = 7.8 Hz), 48.1 (d, *J* = 8.2 Hz), 25.6, 24.8, 23.6. IR (KBr): 2933, 2884, 2851, 1636, 1531, 1484, 1462, 1441, 1402, 1368, 1284, 1254, 1200, 1134, 1108, 1086, 1024, 1003, 949, 906, 871, 853, 794 cm⁻¹. HRMS-ESI (*m/z*) calcd. for [C₆H₁₁FNSe]⁺ ([M + H]⁺): 196.0035; found: 196.0034.

Morpholine-4-carboselenoyl fluoride (**3h**)



Light yellow solid (25.6 mg, 70%), a mixture of petroleum ether/ethyl acetate = 5/1 (v/v) as eluents for column chromatography. M.p.: 48.5-49.5 °C. ¹H NMR (500 MHz, CDCl₃) δ 4.11 (t, *J* = 4.8 Hz, 2H), 3.84 (t, *J* = 4.8 Hz, 2H), 3.76-3.71 (m, 4H). ¹⁹F NMR (471 MHz, CDCl₃) δ 24.0 (m). ¹³C NMR (126 MHz, CDCl₃): δ 184.5 (d, *J* = 350.4 Hz), 65.7, 65.6, 52.7 (d, *J* = 7.1 Hz), 47.4 (d, *J* = 6.7 Hz). IR (KBr): 2973, 2924, 2870, 1526, 1446, 1436, 1389, 1364, 1305, 1273, 1245, 1218, 1204, 1119, 1104, 1067, 1025, 932, 884, 847, 834 cm⁻¹. HRMS-ESI (*m/z*) calcd. for [C₅H₉FNSe]⁺ ([M + H]⁺): 197.9828; found: 197.9832.

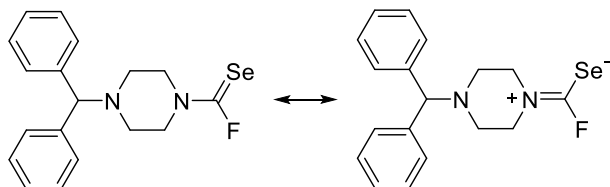
Thiomorpholine-4-carboselenoyl fluoride (**3i**)



White solid (36.9 mg, 87%), a mixture of petroleum ether/ethyl acetate = 5/1 (v/v) as eluents for column chromatography. M.p.: 45.9-46.6 °C. ¹H NMR (500 MHz, CDCl₃) δ 4.37 (m, 2H), 4.00 (m, 2H), 2.85 (t, *J* = 4.9 Hz, 2H), 2.72 (m, 2H). ¹⁹F NMR (471 MHz, CDCl₃) δ 25.9 (m). ¹³C NMR (126 MHz, CDCl₃) δ 184.6 (d, *J* = 350.3 Hz), 55.8 (d, *J* = 6.9 Hz), 49.8 (d, *J* = 7.6 Hz), 27.5, 26.8. IR (KBr): 3009, 2970, 2920,

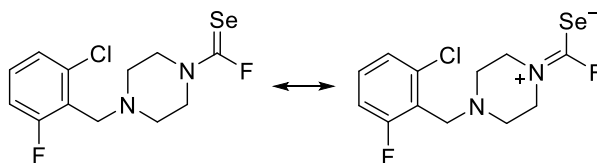
2904, 1539, 1446, 1419, 1364, 1311, 1291, 1258, 1229, 1209, 1163, 1096, 1032, 948, 853 cm^{-1} . HRMS-ESI (m/z) calcd. for $[\text{C}_5\text{H}_9\text{FNSSe}]^+$ ($[\text{M} + \text{H}]^+$): 213.9599; found: 213.9607.

4-Benzhydrylpiperazine-1-carboselenoyl fluoride (**3j**)



White solid (43.3 mg, 60%), a mixture of petroleum ether/ethyl acetate = 40/1 (v/v) as eluents for column chromatography. M.p.: 128.6-129.3 °C. ^1H NMR (500 MHz, CDCl_3) δ 7.41 (d, $J = 7.5$ Hz, 4H), 7.30 (t, $J = 7.5$, 4H), 7.22 (t, $J = 7.4$ Hz, 2H), 4.30 (s, 1H), 4.10 (t, $J = 5.2$ Hz, 2H), 3.73 (t, $J = 5.1$ Hz, 2H), 2.57 (t, $J = 5.1$ Hz, 2H), 2.46 (t, $J = 5.2$ Hz, 2H). ^{19}F NMR (471 MHz, CDCl_3) δ 23.8 (m). ^{13}C NMR (126 MHz, CDCl_3) δ 183.9 (d, $J = 349.4$ Hz), 141.4, 128.8, 127.8, 127.5, 75.5, 53.0 (d, $J = 7.2$ Hz), 50.8, 50.5, 47.2 (d, $J = 7.1$ Hz). IR (KBr): 3059, 3026, 2950, 2914, 2806, 2763, 1541, 1519, 1491, 1446, 1286, 1248, 1144, 1112, 1092, 1076, 1025, 989, 826, 759, 747, 706, 696 cm^{-1} . HRMS-ESI (m/z) calcd. for $[\text{C}_{18}\text{H}_{20}\text{FN}_2\text{Se}]^+$ ($[\text{M} + \text{H}]^+$): 363.0770; found: 363.0772.

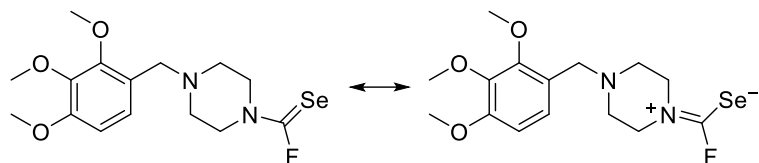
4-(2-Chloro-6-fluorobenzyl)piperazine-1-carboselenoyl fluoride (**3k**)



Light yellow solid (34.9 mg, 52%), a mixture of petroleum ether/ethyl acetate = 5/1 (v/v) as eluents for column chromatography. M.p.: 68.3-68.8 °C. ^1H NMR (500 MHz, CDCl_3) δ 7.25-7.20 (m, 2H), 7.01 (m, 1H), 4.08 (t, $J = 5.2$ Hz, 2H), 3.77 (d, $J = 2.2$ Hz, 2H), 3.72 (t, $J = 5.2$ Hz, 2H), 2.73 (t, $J = 5.2$ Hz, 2H), 2.61 (t, $J = 5.2$ Hz, 2H). ^{19}F NMR (471 MHz, CDCl_3) δ 23.9 (m), -112.1 (s). ^{13}C NMR (126 MHz, CDCl_3) δ 183.9 (d, $J = 349.6$ Hz), 162.0 (d, $J = 249.8$ Hz), 136.6 (d, $J = 5.6$ Hz), 129.8 (d, $J = 9.7$ Hz), 125.6 (d, $J = 3.5$ Hz), 122.8 (d, $J = 17.8$ Hz), 114.2 (d, $J = 23.3$ Hz), 52.9 (d, $J = 7.2$ Hz), 51.8 (d, $J = 1.2$ Hz), 51.5, 51.2, 47.1 (d, $J = 7.2$ Hz). IR (KBr): 3023, 2948, 2919, 2821, 2783, 1606, 1577, 1528, 1445, 1402, 1367, 1348, 1296, 1280, 1266, 1244, 1197,

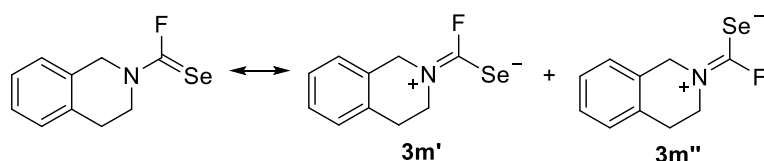
1168, 1139, 1096, 1036, 993, 858, 826, 776, 721 cm^{-1} . HRMS-ESI (m/z) calcd. for $[\text{C}_{12}\text{H}_{14}\text{ClF}_2\text{N}_2\text{Se}]^+$ ($[\text{M} + \text{H}]^+$): 338.9973; found: 338.9981.

4-(2,3,4-Trimethoxybenzyl)piperazine-1-carboselenoyl fluoride (**3l**)



White solid (60.1 mg, 80%), a mixture of petroleum ether/ethyl acetate = 5/1 (v/v) as eluents for column chromatography. M.p.: 84.8-85.9 °C. ^1H NMR (500 MHz, CDCl_3) δ 6.93 (d, $J = 8.5$ Hz, 1H), 6.64 (d, $J = 8.5$ Hz, 1H), 4.09 (t, $J = 5.1$ Hz, 2H), 3.87 (s, 3H), 3.87 (s, 3H), 3.87 (s, 3H), 3.72 (t, $J = 5.2$ Hz, 2H), 3.52 (s, 2H), 2.63 (t, $J = 5.1$ Hz, 2H), 2.52 (t, $J = 5.2$ Hz, 2H). ^{19}F NMR (471 MHz, CDCl_3) δ 23.9 (m). ^{13}C NMR (126 MHz, CDCl_3) δ 183.9 (d, $J = 349.7$ Hz), 153.4, 152.7, 142.4, 125.2, 125.7, 107.1, 61.2, 60.8, 56.0, 56.0, 53.0 (d, $J = 7.1$ Hz), 51.6, 51.3, 47.2 (d, $J = 7.1$ Hz). IR (KBr): 3050, 3003, 2950, 2922, 2848, 1599, 1539, 1506, 1419, 1397, 1320, 1285, 1258, 1231, 1200, 1159, 1132, 1084, 1032, 1015, 983, 954, 899, 858, 798, 771, 739, 690 cm^{-1} . HRMS-ESI (m/z) calcd. for $[\text{C}_{15}\text{H}_{22}\text{FN}_2\text{O}_3\text{Se}]^+$ ($[\text{M} + \text{H}]^+$): 377.0774; found: 377.0790.

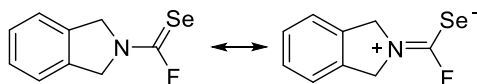
3,4-Dihydroisoquinoline-2(1H)-carboselenoyl fluoride (**3m**)



Colorless oil (30.0 mg, 62%), a mixture of petroleum ether/ethyl acetate = 10/1 (v/v) as eluents for column chromatography. M.p.: 68.8-69.3 °C. ^1H NMR (500 MHz, CDCl_3 , **3m'** + **3m''**) δ 7.29-7.11 (m, 4H), 5.07 (s, 1.04H), 4.81 (s, 0.92H), 4.18 (t, $J = 6.1$ Hz, 0.93H), 3.92 (t, $J = 6.0$ Hz, 1.07H), 3.06 (t, $J = 6.0$ Hz, 0.93H), 2.96 (t, $J = 6.1$ Hz, 1.07H). ^{19}F NMR (471 MHz, CDCl_3 , **3m'** + **3m''**) δ 27.6 (m), 25.3 (m). ^{13}C NMR (126 MHz, CDCl_3 , **3m'** + **3m''**) δ 184.3 (d, $J = 349.7$ Hz), 134.0, 133.2, 130.7, 130.2, 128.5, 128.2, 127.9, 127.6, 127.3, 127.2, 126.6, 126.2, 54.2 (d, $J = 7.9$ Hz), 51.0 (d, $J = 7.6$ Hz), 48.3 (d, $J = 8.1$ Hz), 45.1 (d, $J = 6.8$ Hz), 28.4, 27.7. IR (KBr): 3065, 3026, 2926, 2900, 2850, 1588, 1518, 1507, 1493, 1453, 1371, 1345, 1284, 1250, 1235, 1217, 1173, 1090, 1048, 975, 926, 824, 749, 673 cm^{-1} . HRMS-ESI (m/z) calcd. for

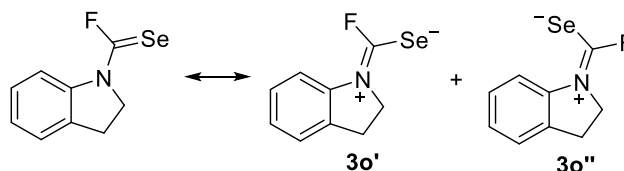
$[\text{C}_{10}\text{H}_{11}\text{FNSe}]^+$ ($[\text{M} + \text{H}]^+$): 242.0043; found: 242.0050.

Isoindoline-2-carboselenoyl fluoride (**3n**)



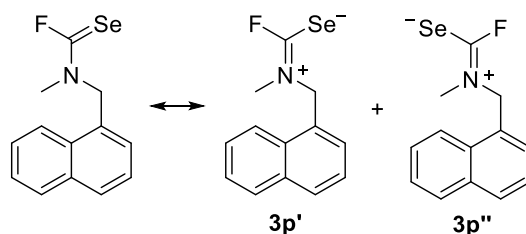
White solid (17.8 mg, 39%), a mixture of petroleum ether/ethyl acetate = 5/1 (v/v) as eluents for column chromatography. M.p.: 131.8-133.6 °C. ^1H NMR (500 MHz, CDCl_3): δ 7.39-7.33 (m, 3H), 7.26 (m, 1H), 5.03 (s, 2H), 4.94 (s, 2H). ^{19}F NMR (471 MHz, CDCl_3) δ 30.3 (m). ^{13}C NMR (126 MHz, CDCl_3) δ 182.9 (d, $J = 350.8$ Hz), 134.0, 134.0, 128.6, 128.5, 122.9, 122.7, 60.1 (d, $J = 6.4$ Hz), 54.7 (d, $J = 2.2$ Hz). IR (KBr): 3074, 3022, 2925, 2867, 1801, 1791, 1594, 1518, 1488, 1454, 1351, 1327, 1270, 1224, 1187, 1177, 1153, 1093, 1075, 1024, 990, 920, 855, 816, 752, 738 cm^{-1} . HRMS-ESI (m/z) calcd. for $[\text{C}_9\text{H}_9\text{FNSe}]^+$ ($[\text{M} + \text{H}]^+$): 229.9879; found: 229.9884.

Indoline-1-carboselenoyl fluoride (**3o**)



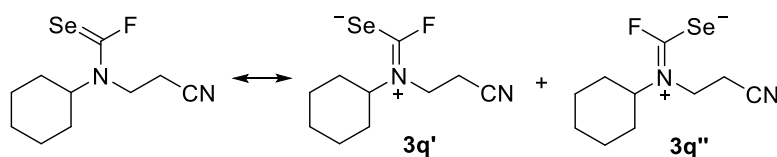
Light yellow solid (35.7 mg, 78%), a mixture of petroleum ether/ethyl acetate = 8/1 (v/v) as eluents for column chromatography. M.p.: 86.1-87.4 °C. ^1H NMR (500 MHz, CDCl_3 , **3o'** + **3o''**) δ 9.18 (d, $J = 8.1$ Hz, 0.12H), 7.63 (dd, $J = 8.1, 3.2$ Hz, 0.85H), 7.37-7.28 (m, 1.86H), 7.21 (t, $J = 7.5$ Hz, 0.88H), 4.38 (t, $J = 8.0$ Hz, 1.73H), 4.34 (m, 0.26H), 3.26 (t, $J = 8.1$ Hz, 1.73H), 3.21 (t, $J = 8.1$ Hz, 0.27H). ^{19}F NMR (471 MHz, CDCl_3 , **3o'** + **3o''**) δ 50.6 (m), 25.8 (m). ^{13}C NMR (126 MHz, CDCl_3 , **3o'** + **3o''**) δ 180.2 (d, $J = 354.3$ Hz), 139.8, 139.7, 133.5, 128.4 (d, $J = 3.1$ Hz), 127.3, 127.3, 126.6, 126.2, 125.2, 117.9, 117.7, 117.6 (d, $J = 2.1$ Hz), 56.3 (d, $J = 7.8$ Hz), 52.0 (d, $J = 3.6$ Hz), 26.8, 26.4. IR (KBr): 3068, 2962, 2922, 2848, 1601, 1498, 1456, 1325, 1261, 1226, 1191, 1165, 1132, 1065, 990, 934, 852, 752, 721, 699 cm^{-1} . HRMS-ESI (m/z) calcd. for $[\text{C}_9\text{H}_9\text{FNSe}]^+$ ($[\text{M} + \text{H}]^+$): 229.9879; found: 229.9888.

Methyl(naphthalen-1-ylmethyl)carbamosenoic fluoride (**3p**)



White solid (45.5 mg, 81%), a mixture of petroleum ether/ethyl acetate = 10/1 (v/v) as eluents for column chromatography. M.p.: 81.9-82.8 °C. ^1H NMR (500 MHz, CDCl_3 , **3p'** + **3p''**) δ 8.04 (d, $J = 8.3$ Hz, 0.58H), 7.93-7.87 (m, 1.98H), 7.82 (d, $J = 8.2$ Hz, 0.42H), 7.61-7.54 (m, 2.04H), 7.50-7.46 (m, 1.00H), 7.43 (d, $J = 6.8$ Hz, 0.57H), 7.29 (d, $J = 7.0$ Hz, 0.41H), 5.53 (s, 1.18H), 5.19 (s, 0.84H), 3.34 (s, 1.24H), 2.92 (d, $J = 2.8$ Hz, 1.76H). ^{19}F NMR (471 MHz, CDCl_3 , **3p'** + **3p''**) δ 29.6 (m), 25.7 (m). ^{13}C NMR (126 MHz, CDCl_3 , **3p'** + **3p''**) δ 186.4 (d, $J = 350.5$ Hz), 185.7 (d, $J = 349.8$ Hz), 134.0, 133.9, 131.6, 130.8, 129.7, 129.6, 129.2, 129.0, 128.8, 128.4, 127.7, 127.3, 127.3, 126.5, 126.5, 126.0, 125.4, 125.3, 123.5, 122.0, 59.1 (d, $J = 7.6$ Hz), 52.9 (d, $J = 7.9$ Hz), 42.9 (d, $J = 7.2$ Hz), 35.1 (d, $J = 8.4$ Hz). IR (KBr): 3003, 2962, 2928, 2897, 2850, 2829, 1602, 1537, 1496, 1444, 1413, 1369, 1347, 1315, 1296, 1273, 1202, 1143, 1097, 1041, 1029, 994, 946, 931, 901, 815, 795, 754, 687, 671, 660 cm^{-1} . HRMS-ESI (m/z) calcd. for $[\text{C}_{13}\text{H}_{13}\text{FNSe}]^+$ ($[\text{M} + \text{H}]^+$): 282.0192; found: 282.0191.

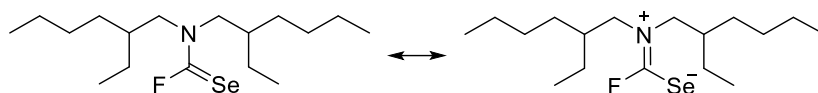
(2-Cyanoethyl)(cyclohexyl)carbamoseleonic fluoride (**3q**)



Light yellow oil (42.9 mg, 82%), a mixture of petroleum ether/ethyl acetate = 8/1 (v/v) as eluents for column chromatography. ^1H NMR (500 MHz, CDCl_3 , **3p'** + **3p''**) δ 4.80 (m, 0.28H), 4.10 (tm, $J = 12.2$ Hz, 0.72H), 4.00 (t, $J = 7.2$ Hz, 1.43H), 3.65 (td, $J = 7.6$, 2.4 Hz, 0.56H), 3.03 (t, $J = 7.2$ Hz, 1.44H), 2.70 (t, $J = 7.6$ Hz, 0.56H), 2.02-1.88 (m, 4H), 1.75-1.54 (m, 2H), 1.48-1.34 (m, 2.92H), 1.19-1.11 (m, 1.07H). ^{19}F NMR (471 MHz, CDCl_3 , **3p'** + **3p''**) δ 32.3 (m), 28.2 (m). ^{13}C NMR (126 MHz, CDCl_3 , **3p'** + **3p''**) δ 186.6 (d, $J = 353.5$ Hz), 186.3 (d, $J = 351.0$ Hz), 117.1, 116.0, 65.2 (d, $J = 8.6$ Hz), 62.1 (d, $J = 5.6$ Hz), 47.5 (d, $J = 5.4$ Hz), 40.5 (d, $J = 6.9$ Hz), 30.8, 29.5, 25.5, 25.4, 25.0, 24.8, 18.0 (d, $J = 2.2$ Hz), 15.8 (d, $J = 2.8$ Hz). IR (KBr): 2924, 2854, 2250, 1776, 1721, 1668, 1506, 1432, 1375, 1339, 1307, 1253, 1237, 1177, 1141, 1088, 1048, 1004, 942, 897, 759, 699 cm^{-1} . HRMS-ESI (m/z) calcd. for $[\text{C}_{10}\text{H}_{16}\text{FN}_2\text{Se}]^+$ ($[\text{M}$

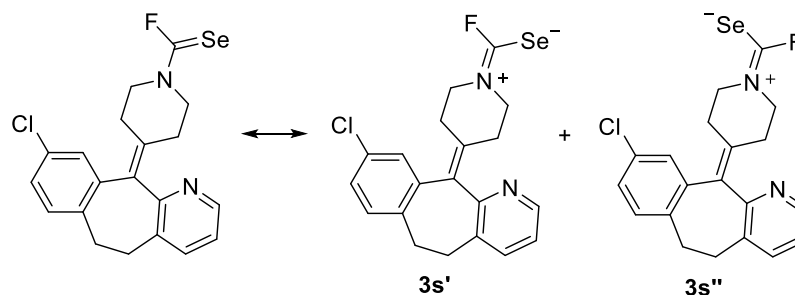
+ H⁺): 263.0457; found: 263.0457.

Di(octan-3-yl)carbamoseleonic fluoride (**3r**)



Light yellow oil (68.8 mg, 98%), a mixture of petroleum ether/ethyl acetate = 40/1 (v/v) as eluents for column chromatography. ¹H NMR (500 MHz, CDCl₃) δ 3.72 (m, 2H), 3.40 (m, 2H), 2.07 (m, 1H), 1.72 (m, 1H), 1.42-1.20 (m, 16H), 0.93-0.87 (m, 12H). ¹⁹F NMR (471 MHz, CDCl₃) δ 29.7 (m). ¹³C NMR (126 MHz, CDCl₃): δ 185.9 (d, *J* = 349.6 Hz), 58.8 (d, *J* = 7.2 Hz), 54.0 (d, *J* = 5.9 Hz), 37.8, 36.5, 30.4, 30.3 (d, *J* = 1.0 Hz), 28.5, 28.4 (d, *J* = 1.6 Hz), 23.8, 23.7, 23.0, 22.9, 14.0, 14.0, 10.6, 10.5. IR (KBr): 2960, 2930, 2873, 2860, 1686, 1606, 1522, 1457, 1427, 1381, 1266, 1237, 1214, 1184, 1110, 1080, 1011, 961, 768, 728, 686 cm⁻¹. HRMS-ESI (*m/z*) calcd. for [C₁₇H₃₅FNSe]⁺ ([M + H]⁺): 352.1913; found: 352.1924.

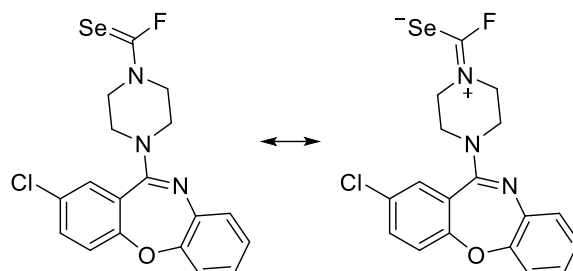
4-(9-Chloro-5,6-dihydro-11*H*-benzo[5,6]cyclohepta[1,2-*b*]pyridin-11-ylidene)piperidine-1-carboselenoyl fluoride (**3s**)



Light yellow oil (47.8 mg, 57%), dichloromethane as eluent for column chromatography. ¹H NMR (500 MHz, CDCl₃, **3S'** + **3S''**) δ 8.40 (t, *J* = 6.0 Hz, 1H), 7.45 (d, *J* = 7.6 Hz, 1H), 7.19 (s, 1H), 7.16-7.08 (m, 3H), 4.32 (m, 1H), 3.96 (m, 1H), 3.83 (m, 1H), 3.54 (m, 1H), 3.33 (m, 2H), 2.90-2.39 (m, 6H). ¹⁹F NMR (471 MHz, CDCl₃, **3S'** + **3S''**) δ 24.6 (m), 24.5 (m). ¹³C NMR (126 MHz, CDCl₃, **3S'** + **3S''**) δ 183.9 (d, *J* = 350.0 Hz), 183.9 (d, *J* = 349.5 Hz), 156.1, 156.0, 146.8, 146.8, 139.7, 139.6, 138.0, 137.2, 137.1, 136.4, 136.4, 133.8, 133.7, 133.5, 133.4, 133.4, 133.3, 130.1, 130.1, 129.1, 126.4, 126.4, 122.7, 122.7, 53.6 (d, *J* = 6.8 Hz), 53.6 (d, *J* = 6.7 Hz), 47.1 (d, *J* = 7.3 Hz), 31.6 (d, *J* = 4.3 Hz), 29.9, 29.6, 29.2, 28.9. IR (KBr): 3044, 2921, 2856, 2217, 1784, 1654, 1590, 1520, 1478, 1456, 1439, 1367, 1330, 1273, 1233, 1159, 1090, 1073, 988, 926, 910, 888, 830, 785, 731, 645 cm⁻¹. MS-ESI (*m/z*) calcd.

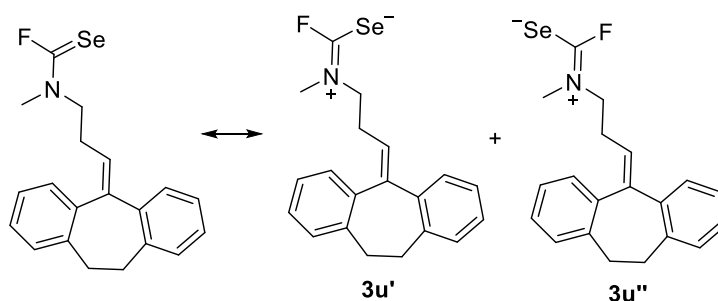
for $[C_{20}H_{19}ClFN_2Se]^+$ ($[M + H]^+$): 423.0382; found: 423.0382.

4-(2-Chlorodibenzo[*b,f*][1,4]oxazepin-11-yl)piperazine-1-carboselenoyl fluoride (**3t**)



White solid (42.9 mg, 51%), a mixture of petroleum ether/ethyl acetate = 10/1 (v/v) as eluents for column chromatography. M.p.: 182.1-182.8 °C. 1H NMR (500 MHz, $CDCl_3$) δ 7.43 (dd, $J = 8.7, 2.4$ Hz, 1H), 7.31 (d, $J = 2.5$ Hz, 1H), 7.21 (d, $J = 8.7$ Hz, 1H), 7.16-7.10 (m, 3H), 7.04 (m, 1H), 4.21 (s, 2H), 3.86 (s, 2H), 3.69 (s, 2H), 3.63 (s, 2H). ^{19}F NMR (471 MHz, $CDCl_3$) δ 25.0 (m). ^{13}C NMR (126 MHz, $CDCl_3$) δ 184.6 (d, $J = 350.1$ Hz), 159.4, 158.1, 151.7, 139.4, 133.2, 130.7, 128.6, 127.2, 126.0, 125.5, 124.4, 123.0, 120.3, 52.2 (d, $J = 7.0$ Hz), 46.5 (d, $J = 6.8$ Hz), 46.4. IR (KBr): 3064 2923, 2835, 1606, 1592, 1562, 1538, 1471, 1445, 1402, 1284, 1257, 1235, 1208, 1185, 1167, 1144, 1098, 1012, 961, 936, 907, 871, 830, 799, 776, 752, 675 cm^{-1} . RMS-ESI (m/z) calcd. for $[C_{18}H_{16}ClFN_3OSe]^+$ ($[M + H]^+$): 424.0126; found: 424.0132.

(3-(10,11-Dihydro-5*H*-dibenzo[*a,d*][7]annulen-5-ylidene)propyl)(methyl) carbamoselenoic fluoride (**3u**)



Yellow oil (67.8 mg, 91%), a mixture of petroleum ether/ethyl acetate = 10/1 (v/v) as eluents for column chromatography. 1H NMR (500 MHz, $CDCl_3$, **3u'** + **3u''**) δ 7.33-7.31 (m, 0.47H), 7.28-7.15 (m, 6H), 7.08 (t, $J = 7.0$ Hz, 1.54H), 5.90 (t, $J = 7.6$ Hz, 0.43H), 5.79 (t, $J = 7.7$ Hz, 0.57H), 3.93 (t, $J = 7.3$ Hz, 0.88H), 3.60 (m, 1.16H), 3.40-3.32 (m, 1.91H), 3.27 (s, 1.75H), 2.98, (m, 2.33H), 2.81 (m, 0.93H), 2.62 (q, $J = 7.4$ Hz, 0.94H), 2.50 (m, 1.13H). ^{19}F NMR (471 MHz, $CDCl_3$, **3u'** + **3u''**) δ 29.3 (m),

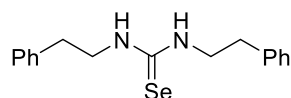
24.6 (m). ^{13}C NMR (126 MHz, CDCl_3 , **3u'** + **3u''**) δ 185.5 (d, $J = 350.3$ Hz), 185.3 (d, $J = 349.0$ Hz), 147.0, 146.2, 140.5, 140.4, 139.4, 139.4, 139.2, 137.1, 137.1, 130.3, 130.2, 128.6, 128.4, 128.4, 128.3, 128.0, 128.0, 127.9, 127.7, 127.6, 127.5, 126.2, 126.2, 126.0, 126.0, 125.5, 124.9, 57.0 (d, $J = 7.1$ Hz), 51.5 (d, $J = 5.8$ Hz), 43.3 (d, $J = 7.0$ Hz), 36.5 (d, $J = 8.7$ Hz), 33.8, 33.8, 32.1, 32.0, 27.5, 26.0. IR (KBr): 3059, 3016, 2923, 2854, 2831, 1735, 1541, 1485, 1443, 1404, 1360, 1329, 1261, 1198, 1127, 1098, 1038, 948, 890, 865, 777, 768, 757, 719, 634 cm^{-1} . HRMS-ESI (m/z) calcd. for $[\text{C}_{20}\text{H}_{21}\text{FNSe}]^+$ ($[\text{M} + \text{H}]^+$): 374.0818; found: 374.0820.

5. General procedures for the synthesis of selenoureas (**5**) from amines and $[\text{Me}_4\text{N}][\text{SeCF}_3]$.

Procedure A: In a nitrogen-filled glovebox, a tube was charged with primary amine (**4**, 0.4 mmol), $[\text{Me}_4\text{N}][\text{SeCF}_3]$ (**2a**, 44.3 mg, 0.2 mmol), and MeCN (2 mL) with stirring. The mixture was reacted at room temperature for 8 h and concentrated to dryness under reduced pressure. The residue was purified by column chromatography on silica gel using a mixture of petroleum ether and ethyl acetate as eluents to give the title compound (**5**).

Procedure B: In a nitrogen-filled glovebox, a tube was charged with $[\text{Me}_4\text{N}][\text{SeCF}_3]$ (**2a**, 44.3 mg, 0.2 mmol), MeCN (2 mL) and aniline (**4j**, 0.2 mmol) with stirring. After 0.5 hour, **4f** (0.2 mmol) or **1a** (0.2 mmol) was added. The mixture was reacted at room temperature for another 8 h and concentrated to dryness under reduced pressure. The residue was purified by column chromatography on silica gel using a mixture of petroleum ether and ethyl acetate as eluents to give the title compound (**5**).

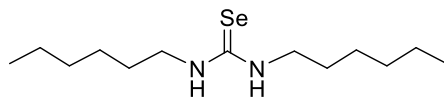
1,3-Diphenethylselenourea (**5a**)



White solid (61.7 mg, 93%), a mixture of petroleum ether/ethyl acetate =5/1 (v/v) as eluents for column chromatography. M.p.: 148.9-149.6 °C. ^1H NMR (500 MHz, CDCl_3) δ 7.29 (t, $J = 7.0$ Hz, 4H), 7.23 (t, $J = 7.3$ Hz, 2H), 7.16 (d, $J = 7.0$ Hz, 4H), 6.62 (brs), 3.66 (brs, 4H), 2.84 (s, 4H). ^{13}C NMR (126 MHz, CDCl_3) δ 179.3, 138.0, 128.9, 128.7, 126.9, 47.7 (brs), 35.0. IR (KBr): 3276, 3209, 3061, 3028, 2926, 2870, 2855, 1570, 1496, 1454, 1352, 1276, 1199, 1147, 1083, 1035, 1013, 908, 748, 698,

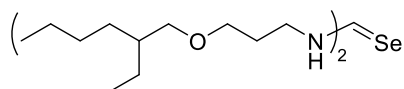
661 cm^{-1} . HRMS-ESI (m/z) calcd. for $[\text{C}_{17}\text{H}_{21}\text{N}_2\text{Se}]^+$ ($[\text{M} + \text{H}]^+$): 333.0864; found: 333.0872.

1,3-Dihexylselenourea (**5b**)



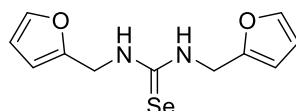
Light yellow oil (49.6 mg, 85%), a mixture of petroleum ether/ethyl acetate = 5/1 (v/v) as eluents for column chromatography. ^1H NMR (500 MHz, CDCl_3) δ 6.39 (brs, 2H), 3.43 (brs, 4H), 1.59 (m, 4H), 1.33 (m, 4H), 1.30-1.26 (m, 8H), 0.86 (t, $J = 6.9$ Hz, 6H). ^{13}C NMR (126 MHz, CDCl_3) δ 178.7, 48.0 (brs), 31.4, 28.9, 26.5, 22.5, 14.0. IR (KBr): 3201, 3094, 2956, 2926, 2857, 1570, 1513, 1471, 1454, 1430, 1372, 1293, 1256, 1228, 1195, 1129, 1087, 1014, 893, 804, 731, 629 cm^{-1} . HRMS-ESI (m/z) calcd. for $[\text{C}_{13}\text{H}_{29}\text{N}_2\text{Se}]^+$ ($[\text{M} + \text{H}]^+$): 293.1490; found: 293.1491.

N-(3-((2-ethylhexyl)oxy)propyl)methaneselenoamide (**5c**)



Colorless oil (52.4 mg, 94%), a mixture of petroleum ether/ethyl acetate = 3/1 (v/v) as eluents for column chromatography. ^1H NMR (500 MHz, CDCl_3) δ 6.79 (brs, 2H), 3.74-3.26 (m, 12H), 1.85 (m, 4H), 1.49 (m, 2H), 1.38-1.26 (m, 16H), 0.89-0.84 (m, 12H). ^{13}C NMR (126 MHz, CDCl_3) δ 178.3, 74.4, 69.6 (brs), 47.9 (brs), 39.6, 30.5, 29.1, 28.6, 23.8, 23.1, 14.1, 11.0. IR (KBr): 3231, 3058, 2957, 2928, 2859, 1558, 1495, 1464, 1378, 1299, 1271, 1242, 1205, 1107, 1042, 988, 919, 768 cm^{-1} . HRMS-ESI (m/z) calcd. for $[\text{C}_{23}\text{H}_{49}\text{N}_2\text{O}_2\text{Se}]^+$ ($[\text{M} + \text{H}]^+$): 465.2954; found: 465.5959.

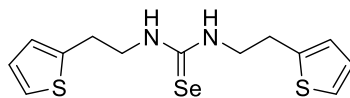
1,3-Bis(furan-2-ylmethyl)selenourea (**5d**)



Light yellow solid (39.7 mg, 70%), a mixture of petroleum ether/ethyl acetate = 3/1 (v/v) as eluents for column chromatography. M.p.: 97.9-98.8 $^{\circ}\text{C}$. ^1H NMR (500 MHz, CDCl_3) δ 7.33 (s, 2H), 6.90 (brs), 6.31 (m, 2H), 6.28 (m, 2H), 4.65 (brs, 4H). ^{13}C NMR (126 MHz, CDCl_3): δ 180.3, 149.6, 142.6, 110.7, 108.6, 43.5 (brs). IR (KBr): 3242, 3191, 3113, 3043, 2920, 2852, 1600, 1570, 1540, 1503, 1420, 1356, 1336, 1296,

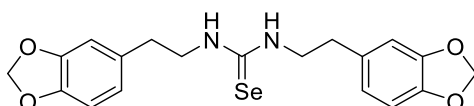
1258, 1187, 1147, 1072, 1009, 948, 907, 884, 813, 740, 720, 702, 666 cm^{-1} . HRMS-ESI (m/z) calcd. for $[\text{C}_{11}\text{H}_{13}\text{N}_2\text{O}_2\text{Se}]^+$ ($[\text{M} + \text{H}]^+$): 285.0137; found: 285.0144.

1,3-Bis(2-(thiophen-2-yl)ethyl)selenourea (**5e**)



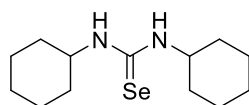
Light yellow solid (59.1 mg, 86%), a mixture of petroleum ether/ethyl acetate = 3/1 (v/v) as eluents for column chromatography. M.p.: 115.9-116.9 °C. ^1H NMR (500 MHz, CDCl_3) δ 7.16 (d, $J = 5.1$ Hz, 2H), 6.93 (m, 2H), 6.83 (d, $J = 3.1$ Hz, 2H), 6.51 (brs), 3.69 (brs, 4H), 3.08 (t, $J = 6.0$ Hz, 4H). ^{13}C NMR (126 MHz, CDCl_3) δ 179.8 (brs), 140.3, 127.3, 125.8, 124.4, 47.6 (brs), 29.3. IR (KBr): 3256, 3081, 2925, 2852, 1559, 1504, 1451, 1424, 1361, 1342, 1322, 1276, 1255, 1230, 1188, 1105, 980, 886, 846, 741 cm^{-1} . HRMS-ESI (m/z) calcd. for $[\text{C}_{13}\text{H}_{17}\text{N}_2\text{S}_2\text{Se}]^+$ ($[\text{M} + \text{H}]^+$): 344.9993; found: 344.9998.

1,3-Bis(2-(benzo[d][1,3]dioxol-5-yl)ethyl)selenourea (**5f**)



White solid (52.9 mg, 63%), a mixture of petroleum ether/ethyl acetate = 3/1 (v/v) as eluents for column chromatography. M.p.: 130.0-131.6 °C. ^1H NMR (500 MHz, CDCl_3) δ 6.72 (d, $J = 7.8$ Hz, 2H), 6.64 (s, 2H), 6.60 (d, $J = 7.6$ Hz, 2H), 5.91 (s, 4H), 3.66 (brs, 4H), 2.76 (s, 4H). ^{13}C NMR (126 MHz, CDCl_3) δ 179.4 (brs), 148.1, 146.6, 131.6 (brs), 121.7, 109.0, 108.6, 101.1, 48.3 (brs), 34.7. IR (KBr): 3276, 3207, 3059, 2930, 2897, 1607, 1570, 1505, 1493, 1445, 1348, 1271, 1250, 1189, 1121, 1101, 1043, 1014, 925, 869, 813, 776, 670 cm^{-1} . HRMS-ESI (m/z) calcd. for $[\text{C}_{19}\text{H}_{21}\text{N}_2\text{O}_4\text{Se}]^+$ ($[\text{M} + \text{H}]^+$): 421.0661; found: 421.0665.

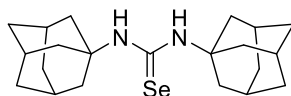
1,3-Dicyclohexylselenourea (**5g**)⁴



White solid (39.7 mg, 69%), a mixture of petroleum ether/ethyl acetate = 3/1 (v/v) as eluents for column chromatography. ^1H NMR (500 MHz, CDCl_3) δ 5.99 (brs, 2H), 3.87 (brs, 2H), 3.31 (s, 4H), 1.72 (m, 4H), 1.62 (m, 2H), 1.37 (m, 4H), 1.26-1.14 (m,

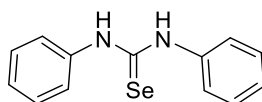
6H). ^{13}C NMR (126 MHz, CDCl_3) δ 175.9, 54.5 (brs), 32.7, 25.3, 24.6.

1,3-Di((3*S*,5*S*,7*S*)-adamantan-1-yl)selenourea (**5h**)



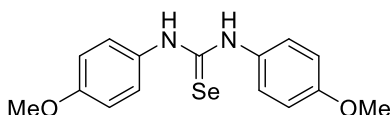
White solid (31.4 mg, 40%), a mixture of petroleum ether/ethyl acetate = 10/1 (v/v) as eluents for column chromatography. M.p.: 144.2-145.6 °C. ^1H NMR (500 MHz, CDCl_3) δ 2.12 (s, 6H), 2.02 (s, 12H), 1.66 (m, 12H). ^{13}C NMR (126 MHz, CDCl_3) δ 59.2, 43.3, 34.5, 29.1. IR (KBr): 3348, 2913, 2852, 2157, 2109, 2039, 1990, 1626, 1554, 1451, 1353, 1344, 1303, 1265, 1186, 1102, 1077, 933, 812, 735, 636 cm^{-1} . HRMS-ESI (m/z) calcd. for $[\text{C}_{21}\text{H}_{33}\text{N}_2\text{Se}]^+$ ($[\text{M} + \text{H}]^+$): 393.1803; found: 393.1809.

1,3-Diphenylselenourea (**5i**)⁵



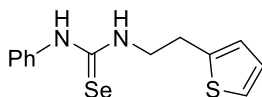
White solid (51.8 mg, 94%). The residue (crude product) was washed with DCM (ca. 5 mL) and water (ca. 5 mL) and dried to give the pure product (without column chromatography). ^1H NMR (500 MHz, $\text{DMSO}-d_6$) δ 10.15 (s, 2H), 7.39 (m, 4H), 7.33 (m, 4H), 7.17 (m, 2H). ^{13}C NMR (126 MHz, $\text{DMSO}-d_6$) δ 179.1, 140.2, 129.0, 125.7, 125.1.

1,3-Bis(4-methoxyphenyl)selenourea (**5j**)⁵



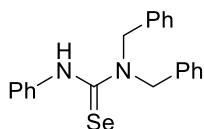
White solid (53.7 mg, 80%). The residue (crude product) was washed with DCM (ca. 5 mL) and water (ca. 5 mL) and dried to give the pure product (without column chromatography). ^1H NMR (500 MHz, $\text{DMSO}-d_6$) δ 9.75 (s, 2H), 7.25 (d, $J = 8.4$ Hz, 4H), 6.90 (d, $J = 8.5$ Hz, 4H), 3.74 (s, 6H). ^{13}C NMR (126 MHz, $\text{DMSO}-d_6$) δ 179.2, 157.5, 133.0, 127.4, 114.2, 55.7.

1-Phenyl-3-(2-(thiophen-2-yl)ethyl)selenourea (**5k**)



Light yellow solid (19.8 mg, 32%), a mixture of petroleum ether/ethyl acetate = 10/1 (v/v) as eluents for column chromatography. M.p.: 66.8-67.9 °C. ¹H NMR (500 MHz, CDCl₃) δ 8.48 (brs, 1H), 7.35 (t, *J* = 7.8 Hz, 2H), 7.28 (t, *J* = 7.3 Hz, 1H), 7.14 (d, *J* = 5.1 Hz, 1H), 7.04 (d, *J* = 7.7 Hz, 2H), 6.89 (t, *J* = 3.6 Hz, 1H), 6.76 (s, 1H), 6.36 (brs, 1H), 3.97 (q, *J* = 6.0 Hz, 2H), 3.17 (t, *J* = 6.4 Hz, 2H). ¹³C NMR (126 MHz, CDCl₃) δ 178.8, 140.6, 135.5, 130.3, 127.8, 127.1, 125.8, 125.2, 124.3, 49.0, 29.2. IR (KBr): 3259, 3146, 2993, 2958, 2925, 2112, 2066, 1626, 1591, 1551, 1523, 1496, 1449, 1414, 1316, 1243, 1218, 1164, 1137, 1095, 1058, 1006, 936, 912, 811, 782, 726, 692 cm⁻¹. HRMS-ESI (*m/z*) calcd. for [C₁₃H₁₅N₂SSe]⁺ ([M + H]⁺): 311.0116; found: 311.0122.

1,1-Dibenzyl-3-phenylselenourea (**5l**)

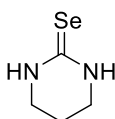


Light yellow solid (22.8 mg, 30%), a mixture of petroleum ether/ethyl acetate = 10/1 (v/v) as eluents for column chromatography. M.p.: 129.3-130.1 °C. ¹H NMR (500 MHz, CDCl₃) δ 7.42-7.34 (m, 10H), 7.31 (t, *J* = 7.6 Hz, 2H), 7.22 (t, *J* = 7.4 Hz, 1H), 7.17 (d, *J* = 8.0 Hz, 2H), 5.16 (brs, 4H). ¹³C NMR (126 MHz, CDCl₃) δ 184.2, 140.4, 134.9 (brs), 129.2, 128.7, 128.3, 127.2, 126.7, 126.5, 56.1 (brs). IR (KBr): 3206, 3061, 3029, 2938, 2847, 1590, 1526, 1495, 1449, 1403, 1364, 1337, 1301, 1223, 1076, 1029, 1009, 961, 936, 898, 852, 791, 759, 730, 693 cm⁻¹. HRMS-ESI (*m/z*) calcd. for [C₂₁H₂₁N₂Se]⁺ ([M + H]⁺): 381.0864; found: 381.0868.

6. General procedure for the synthesis of cycloselenoureas and their derivatives (**7**) from [Me₄N][SeCF₃].

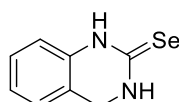
In a nitrogen-filled glovebox, a tube was charged with diamine, aminoalcohol, aminothiols, dithiol or diol (**6**, 0.2 mmol), [Me₄N][SeCF₃] (**2a**, 44.3 mg, 0.2 mmol), and MeCN (2 mL) with stirring. The mixture was reacted at room temperature for 8 h and concentrated to dryness under reduced pressure. The residue was purified by column chromatography on silica gel using a mixture of petroleum ether and ethyl acetate as eluents to give the title compound (**7**).

Tetrahydropyrimidine-2(1*H*)-selenone (**7a**)⁶



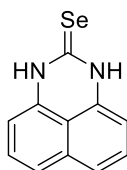
Light pink solid (7.9 mg, 24%), a mixture of petroleum ether/ethyl acetate = 3/1 (v/v) as eluents for column chromatography. ¹H NMR (500 MHz, DMSO-*d*₆) δ 8.23 (s, 2H), 3.07 (t, *J* = 6.0 Hz, 4H), 1.74 (m, 2H). ¹³C NMR (126 MHz, DMSO-*d*₆) δ 169.6, 40.5, 19.2.

3,4-Dihydroquinazoline-2(1*H*)-selenone (**7b**)⁶



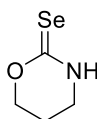
Yellow solid (39.8 mg, 94%), a mixture of petroleum ether/ethyl acetate = 3/1 (v/v) as eluents for column chromatography. ¹H NMR (500 MHz, DMSO-*d*₆) δ 10.69 (s, 1H), 9.05 (s, 1H), 7.17 (t, *J* = 7.4 Hz, 1H), 7.07 (d, *J* = 7.3 Hz, 1H), 7.01-6.97 (m, 2H), 4.36 (s, 2H). ¹³C NMR (126 MHz, DMSO-*d*₆) δ 171.2, 134.9, 128.6, 126.7, 124.0, 117.7, 114.6, 43.9.

1*H*-Perimidine-2(3*H*)-selenone (**7c**)⁷



Pale yellow solid (36.7 mg, 78%), a mixture of petroleum ether/ethyl acetate = 5/1 (v/v) as eluents for column chromatography. ¹H NMR (500 MHz, DMSO-*d*₆) δ 11.75 (s, 2H), 7.24 (t, *J* = 7.6 Hz, 2H), 7.20 (d, *J* = 8.2 Hz, 2H), 6.67 (d, *J* = 7.1 Hz, 2H). ¹³C NMR (126 MHz, DMSO-*d*₆) δ 170.0, 135.2, 134.5, 128.8, 120.0, 117.3, 105.4.

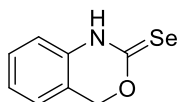
1,3-Oxazinane-2-selenon (**7d**)



Yellow solid (16.2 mg, 49%), a mixture of petroleum ether/ethyl acetate = 1/1 (v/v) as eluents for column chromatography. M.p.: 135.9-141.5 °C. ¹H NMR (500 MHz, DMSO-*d*₆) δ 10.17 (s, 1H), 4.28 (t, *J* = 5.2 Hz, 2H), 3.13 (t, *J* = 5.8 Hz, 2H), 1.96 (m,

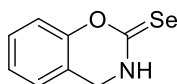
2H). ^{13}C NMR (126 MHz, $\text{DMSO-}d_6$) δ 184.6, 68.7, 40.4, 19.7. IR (KBr): 3439, 3246, 3187, 3044, 2909, 2840, 1571, 1503, 1420, 1336, 1296, 1147, 1072, 1010, 948, 907, 813, 740, 720 cm^{-1} . HRMS-ESI (m/z) calcd. for $[\text{C}_4\text{H}_8\text{NOSe}]^+$ ($[\text{M} + \text{H}]^+$): 165.9766; found: 165.9766.

1,4-Dihydro-2H-benzo[*d*][1,3]oxazine-2-selenone (**7e**)



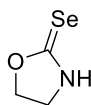
White solid (37.4 mg, 88%), a mixture of petroleum ether/ethyl acetate = 3/1 (v/v) as eluents for column chromatography. M.p.: 151.3-152.2 $^{\circ}\text{C}$. ^1H NMR (500 MHz, $\text{DMSO-}d_6$) δ 12.72 (s, 1H), 7.32 (t, $J = 7.4$ Hz, 1H), 7.22 (d, $J = 7.4$ Hz, 1H), 7.17 (t, $J = 7.4$ Hz, 1H), 7.08 (d, $J = 7.8$ Hz, 1H), 5.40 (s, 2H). ^{13}C NMR (126 MHz, $\text{DMSO-}d_6$) δ 186.1, 132.7, 129.7, 125.6, 125.3, 119.1, 114.4, 69.1. IR (KBr): 3172, 3131, 3105, 3010, 2964, 2938, 1625, 1602, 1534, 1494, 1450, 1423, 1323, 1235, 1222, 1143, 1101, 953, 943, 887, 838, 757, 707 cm^{-1} . HRMS-ESI (m/z) calcd. for $[\text{C}_8\text{H}_8\text{NOSe}]^+$ ($[\text{M} + \text{H}]^+$): 213.9766; found: 213.9772.

3,4-Dihydro-2H-benzo[*e*][1,3]oxazine-2-selenone (**7f**)



White solid (21.3 mg, 50%), a mixture of petroleum ether/ethyl acetate = 3/1 (v/v) as eluents for column chromatography. M.p.: 121.5-123.1 $^{\circ}\text{C}$. ^1H NMR (500 MHz, $\text{DMSO-}d_6$) δ 11.0 (s, 1H), 7.34 (t, $J = 7.0$ Hz, 1H), 7.25-7.20 (m, 2H), 7.14 (d, $J = 8.0$ Hz, 1H), 4.39 (s, 2H). ^{13}C NMR (126 MHz, $\text{DMSO-}d_6$) δ 182.1, 148.0, 129.6, 127.3, 126.0, 117.2, 115.9, 41.9. IR (KBr): 3157, 3101, 3050, 2968, 2917, 2854, 1718, 1627, 1595, 1577, 1487, 1461, 1454, 1425, 1351, 1269, 1223, 1188, 1145, 1094, 1007, 935, 913, 773, 749 cm^{-1} . HRMS-ESI (m/z) for $[\text{C}_8\text{H}_8\text{NOSe}]^+$ ($[\text{M} + \text{H}]^+$): 213.9766; found: 213.9766.

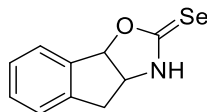
Oxazolidine-2-selenone (**7g**)⁸



Light pink solid (13.6 mg, 45%), ethyl acetate as eluent for column chromatography.

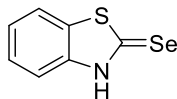
^1H NMR (500 MHz, $\text{DMSO-}d_6$) δ 10.65 (s, 1H), 4.61 (t, $J = 9.0$ Hz, 2H), 3.62 (t, $J = 9.0$ Hz, 2H). ^{13}C NMR (126 MHz, $\text{DMSO-}d_6$) δ 187.3, 71.7, 45.2.

3,3a,4,8b-Tetrahydro-2*H*-indeno[2,1-*d*]oxazole-2-selenone (**7h**)



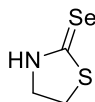
Light brown solid (31.0 mg, 65%), a mixture of petroleum ether/ethyl acetate = 1/2 (v/v) as eluents for column chromatography. M.p.: 187.3-188.9 °C. ^1H NMR (500 MHz, CDCl_3) δ 8.74 (s, 1H), 7.35 (t, $J = 7.3$ Hz, 1H), 7.31-7.27 (m, 3H), 5.74 (m, 1H), 5.37 (d, $J = 7.7$ Hz, 1H), 3.51 (m, 2H). ^{13}C NMR (126 MHz, CDCl_3) δ 188.6, 139.3, 137.7, 130.1, 128.3, 125.8, 124.8, 88.9, 66.1, 38.9. IR (KBr): 3297, 3068, 2962, 2924, 2853, 1784, 1735, 1645, 1485, 1471, 1425, 1372, 1274, 1246, 1210, 1177, 1136, 1013, 997, 928, 902, 840, 829, 752, 713, 642 cm^{-1} . HRMS-ESI (m/z) calcd. for $[\text{C}_{10}\text{H}_{10}\text{NOSe}]^+$ ($[\text{M} + \text{H}]^+$): 239.9922; found: 239.9931. *Note:* This compound is unstable under an ambient atmosphere.

Benzo[*d*]thiazole-2(3*H*)-selenone (**7i**)⁹



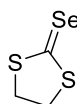
Yellow solid (13.7 mg, 32%), a mixture of petroleum ether/ethyl acetate = 5/1 (v/v) as eluents for column chromatography. ^1H NMR (500 MHz, CDCl_3) δ 7.96 (d, $J = 8.2$ Hz, 1H), 7.80 (d, $J = 8.0$ Hz, 1H), 7.46 (t, $J = 7.6$ Hz, 1H), 7.36 (t, $J = 7.6$ Hz, 1H). ^{13}C NMR (126 MHz, CDCl_3) δ 158.7, 155.2, 137.8, 126.4, 125.2, 122.6, 121.3.

Thiazolidine-2-selenone (**7j**)¹⁰



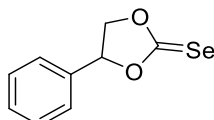
Light yellow solid (32.0 mg, 96%), a mixture of petroleum ether/ethyl acetate = 1/1 (v/v) as eluents for column chromatography. ^1H NMR (500 MHz, $\text{DMSO-}d_6$) δ 10.85 (s, 1H), 3.83 (t, $J = 8.1$ Hz, 2H), 3.46 (t, $J = 8.1$ Hz, 2H). ^{13}C NMR (126 MHz, $\text{DMSO-}d_6$) δ 195.2, 54.2, 34.4.

1,3-Dithiolane-2-selenone (**7k**)¹¹



Light pink solid (17.3 mg, 47%), a mixture of petroleum ether/ethyl acetate = 5/1 (v/v) as eluents for column chromatography. ^1H NMR (500 MHz, $\text{DMSO-}d_6$) δ 4.0 (s, 4H). ^{13}C NMR (126 MHz, $\text{DMSO-}d_6$) δ 228.2, 47.1.

4-Phenyl-1,3-dioxolane-2-selenone (**71**)



Colorless oil (9.1 mg, 20%), a mixture of petroleum ether/ethyl acetate = 1/1 (v/v) as eluents for column chromatography. ^1H NMR (500 MHz, CDCl_3) δ 7.44 (d, $J = 7.5$ Hz, 2H), 7.38 (t, $J = 7.4$ Hz, 2H), 7.34 (t, $J = 7.4$ Hz, 1H), 5.34 (t, $J = 6.9$ Hz, 1H), 4.64 (m, 1H), 4.46 (t, $J = 8.7$ Hz, 1H). ^{13}C NMR (126 MHz, CDCl_3) δ 170.8, 137.2, 129.3, 128.8, 127.7, 75.1, 50.7. IR (KBr): 3061, 3032, 2954, 2925, 2893, 2848, 1747, 1723, 1694, 1491, 1460, 1452, 1372, 1277, 1243, 1196, 1180, 1066, 1039, 1013, 968, 851, 761, 702, 691 cm^{-1} . HRMS-ESI (m/z) calcd. for $[\text{C}_9\text{H}_9\text{O}_2\text{Se}]^+$ ($[\text{M} + \text{H}]^+$): 228.9762; found: 228.9775.

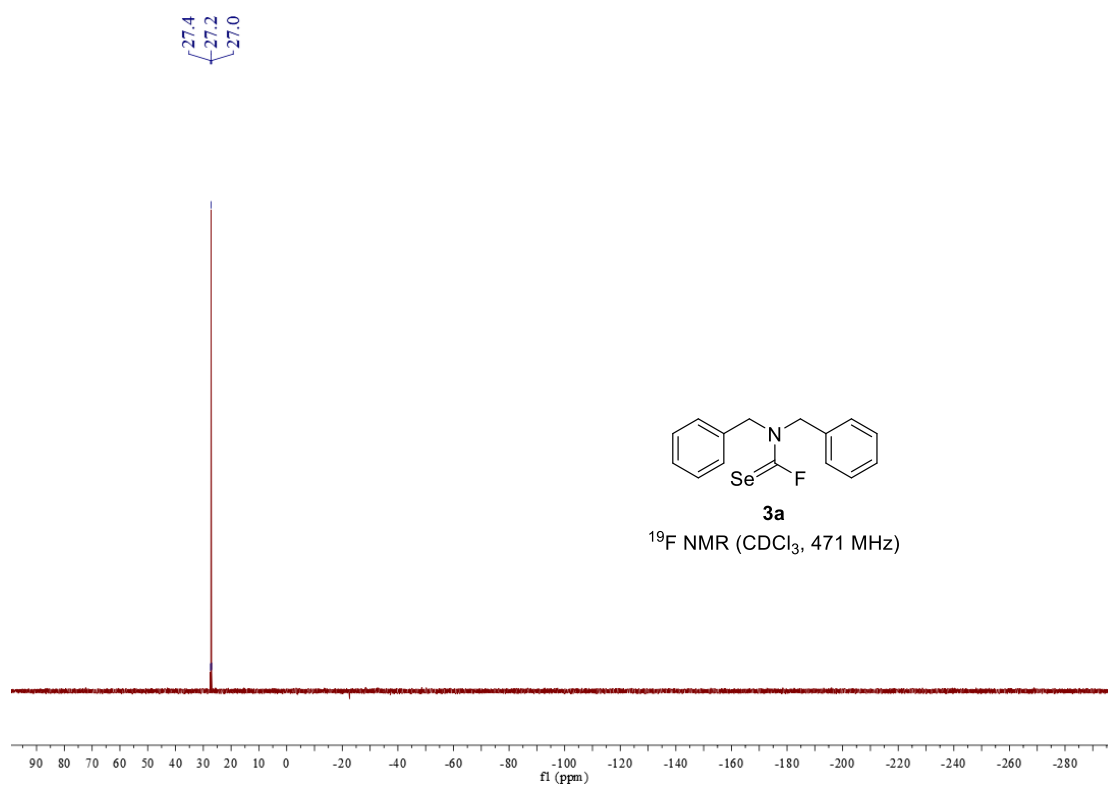
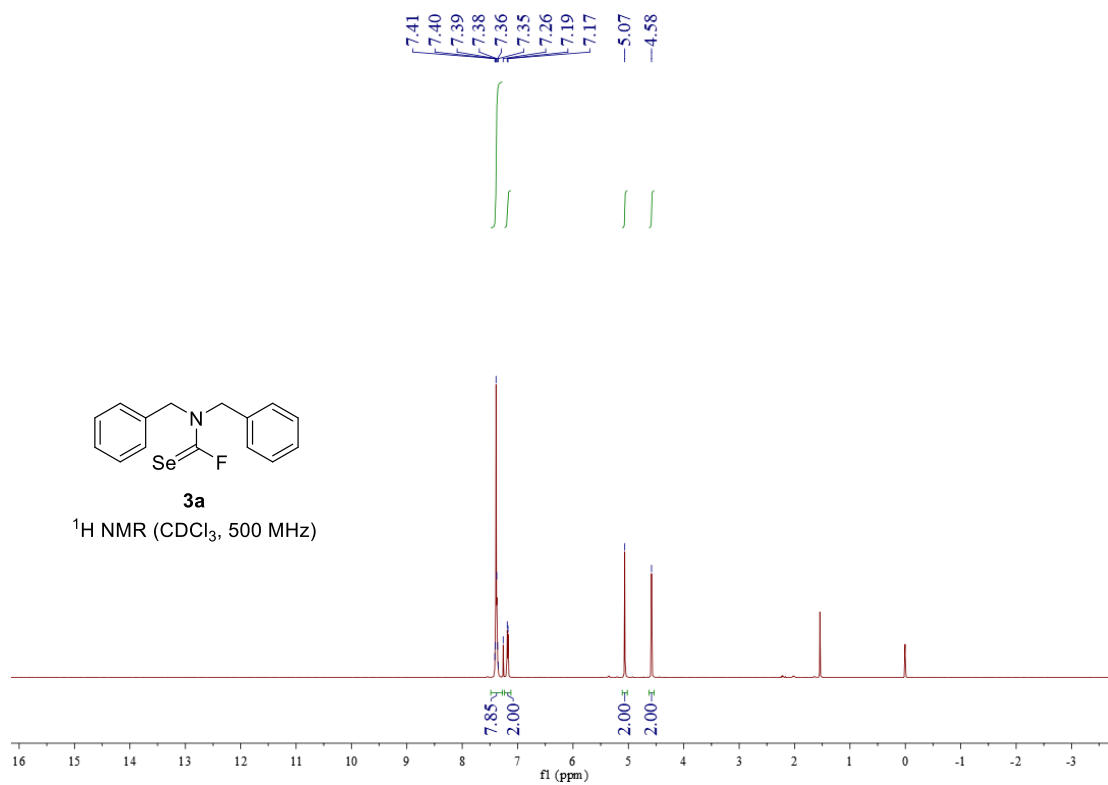
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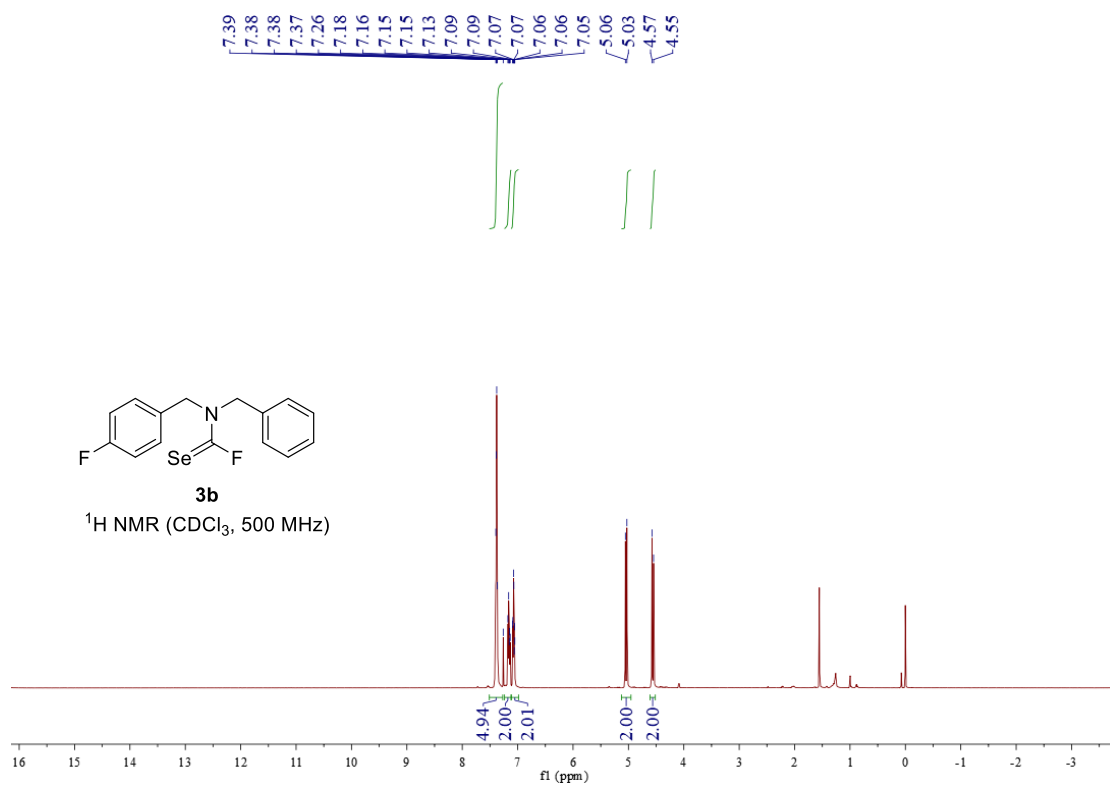
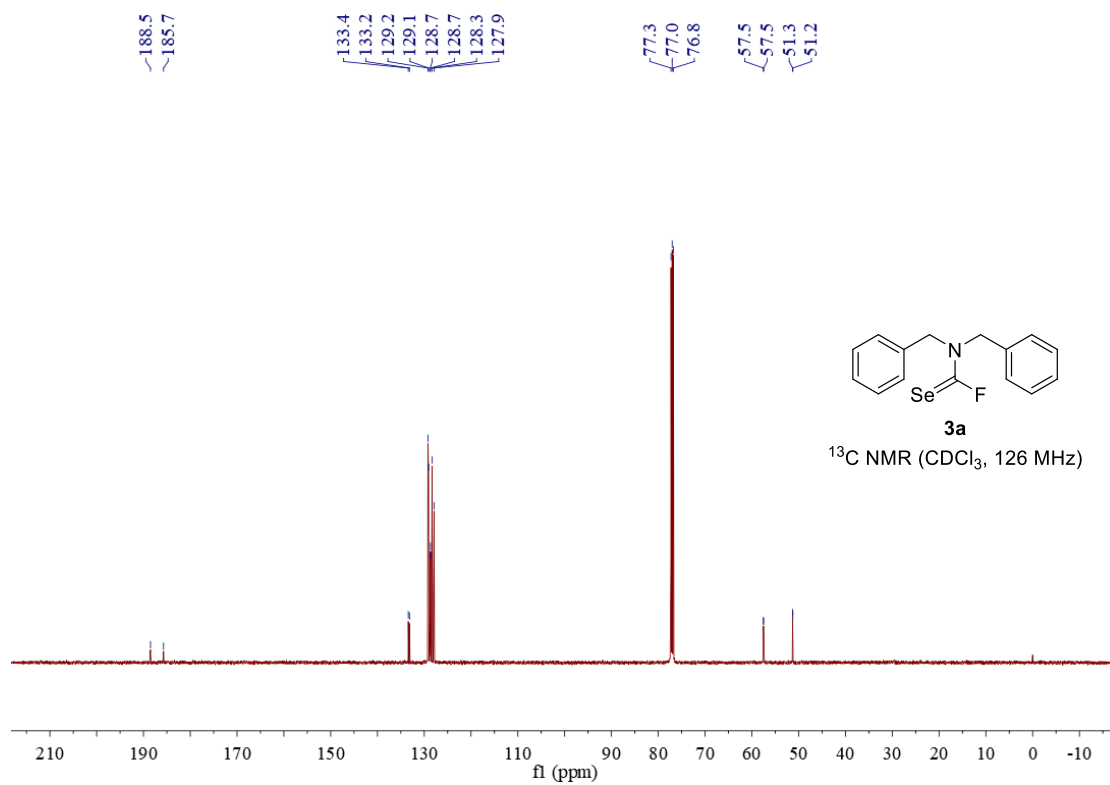
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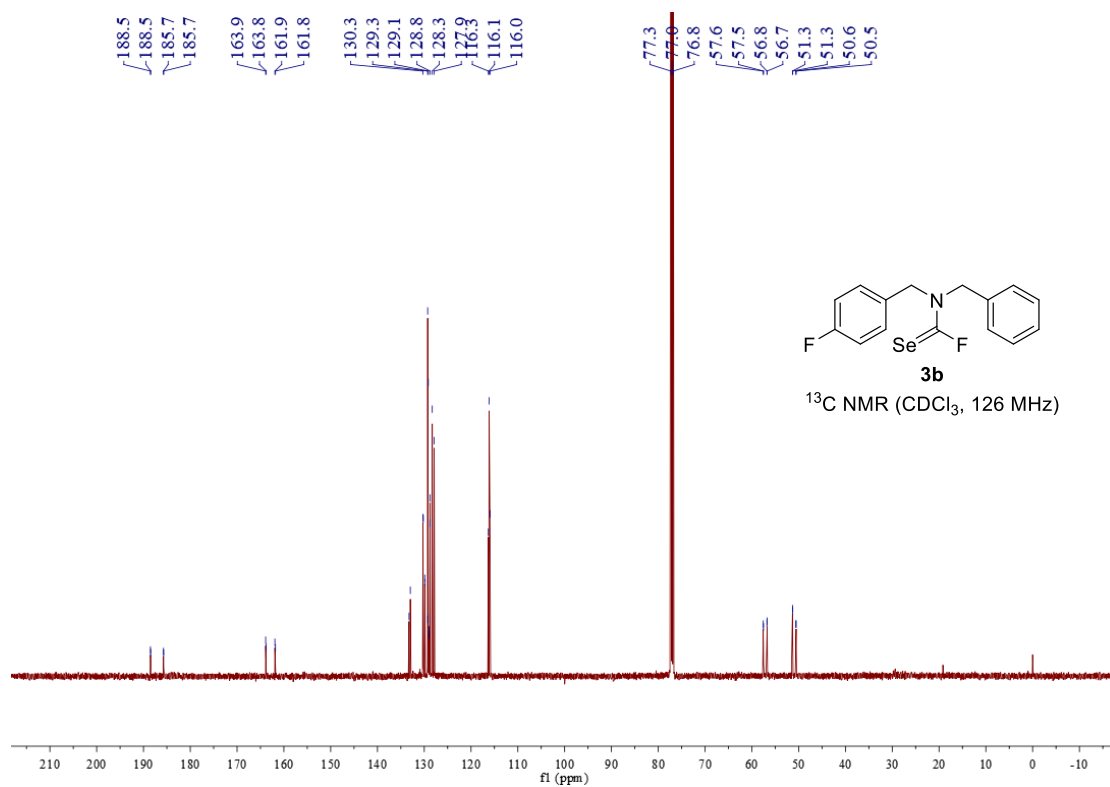
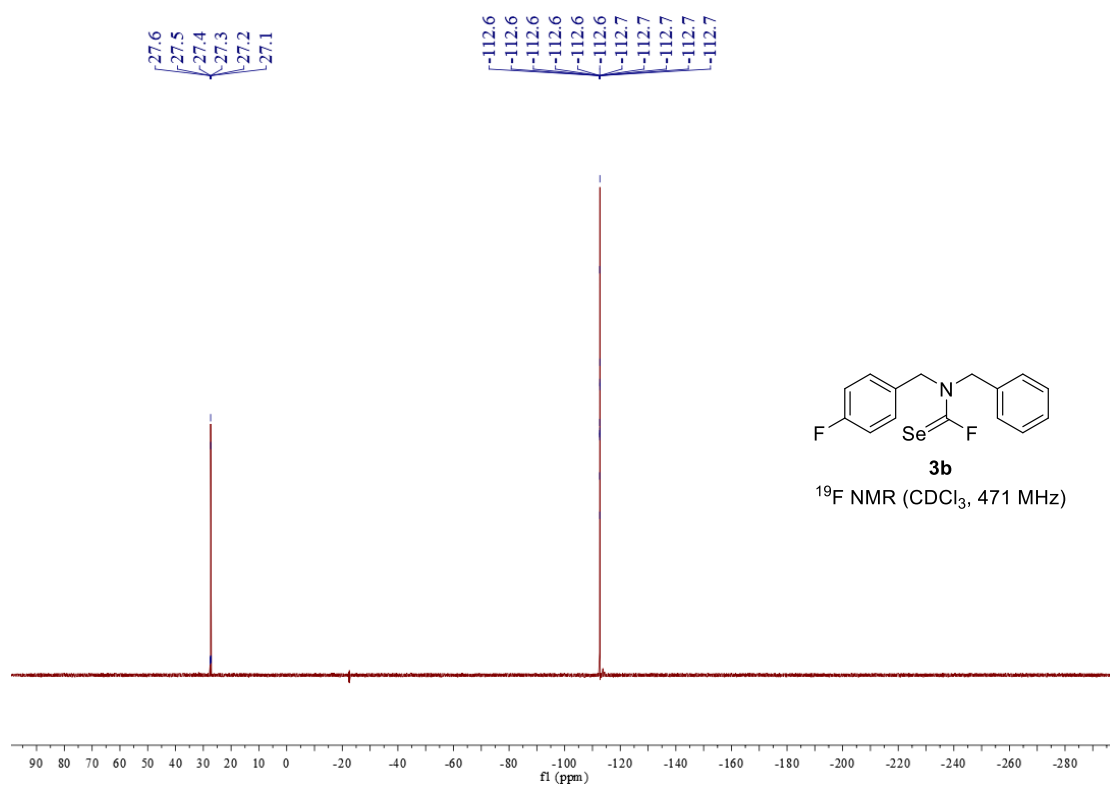
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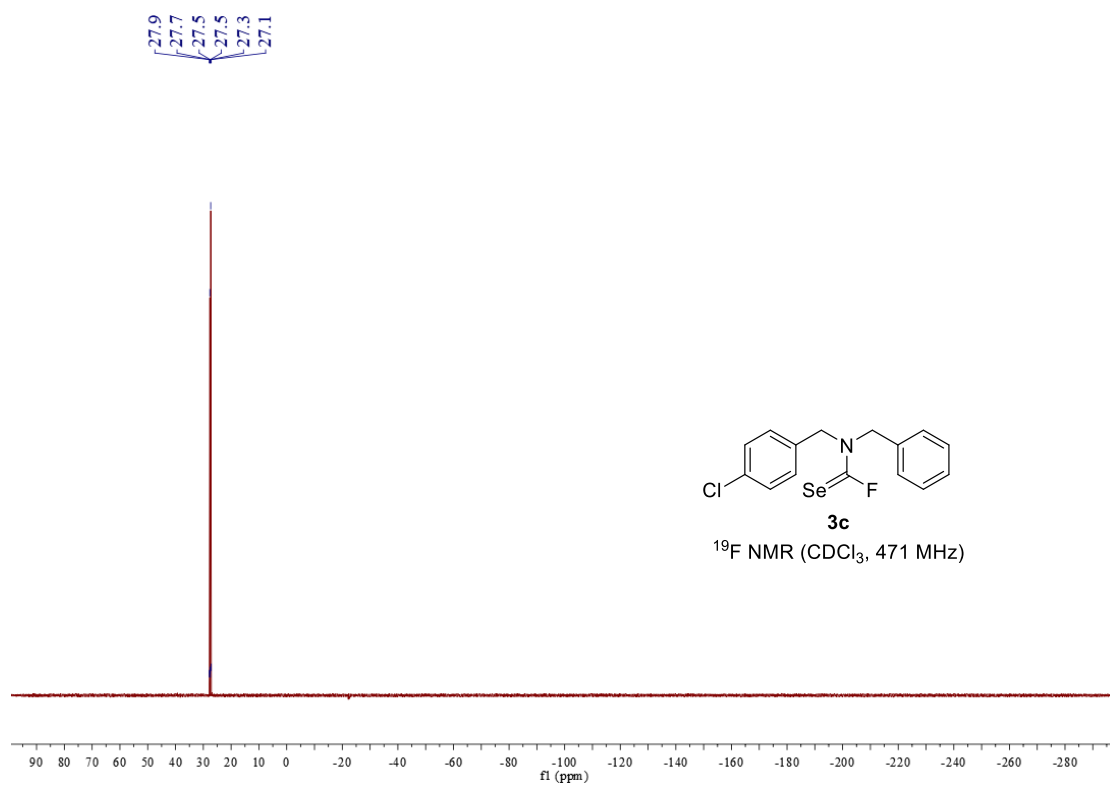
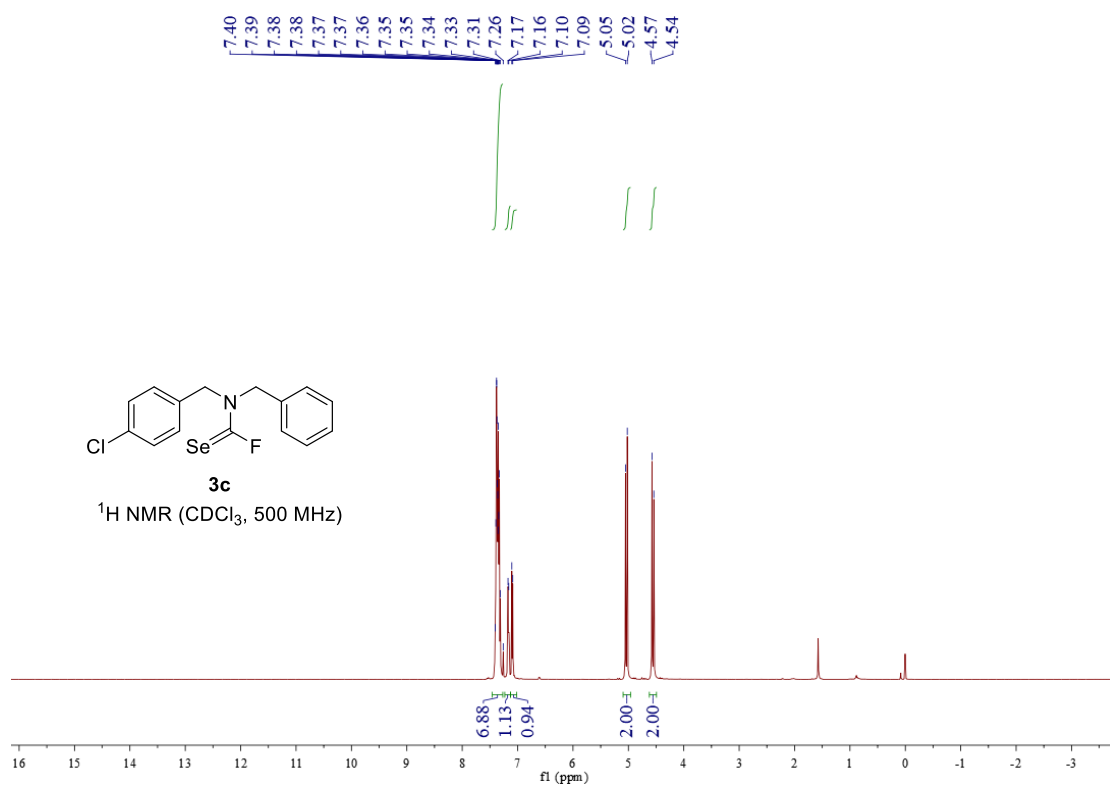
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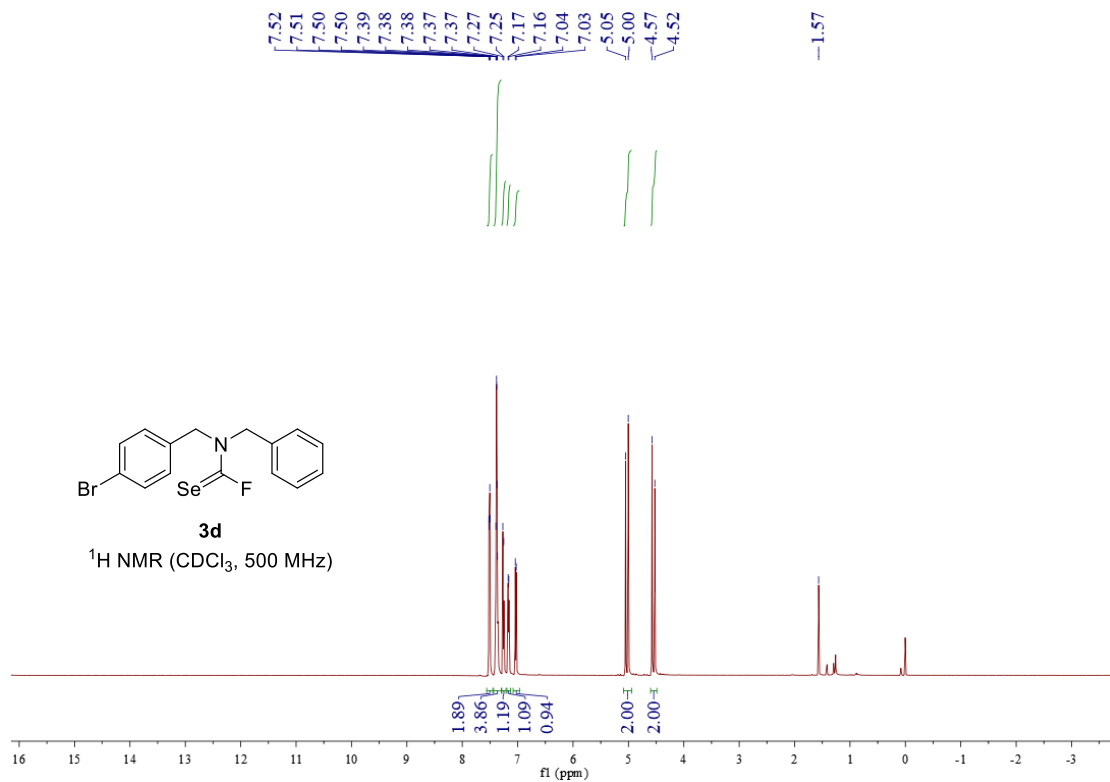
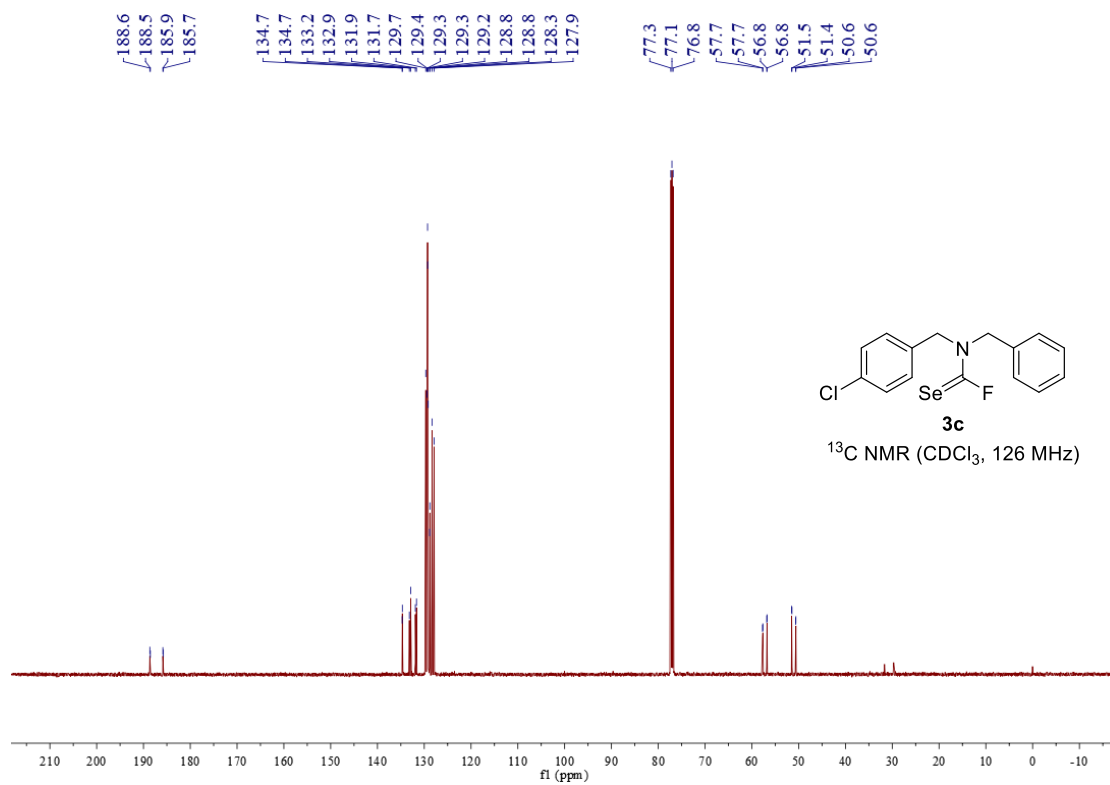
7. NMR spectra of the products

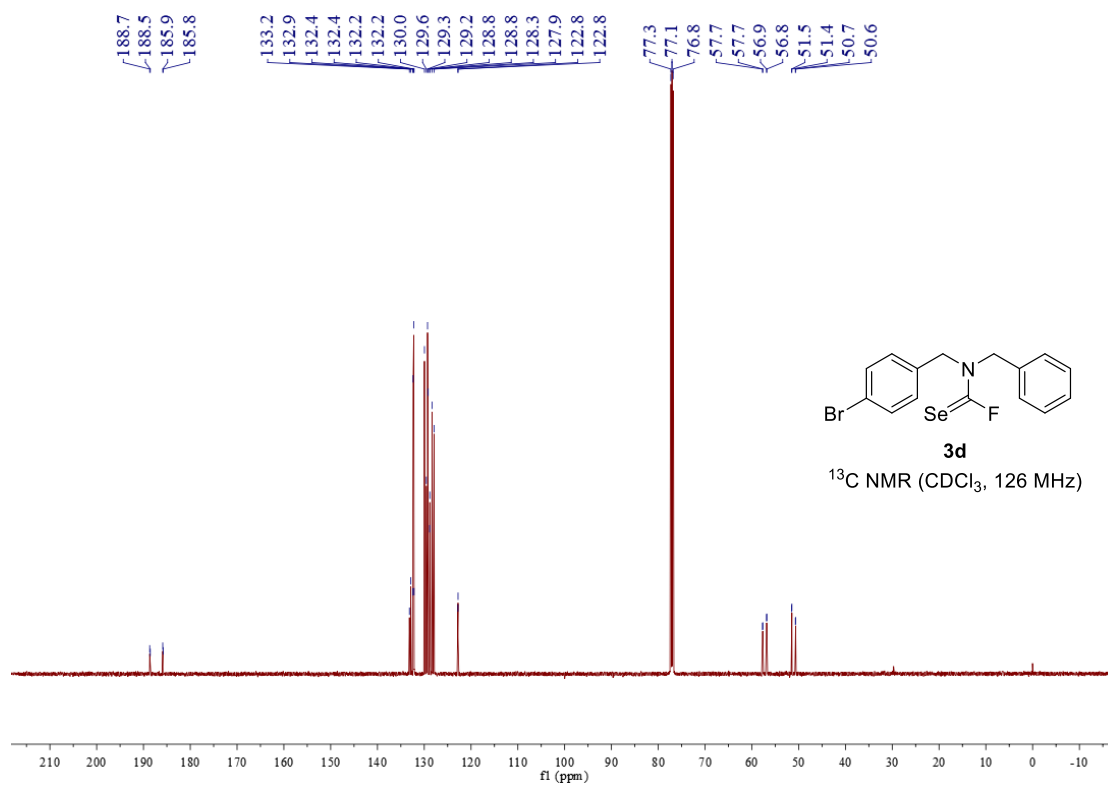
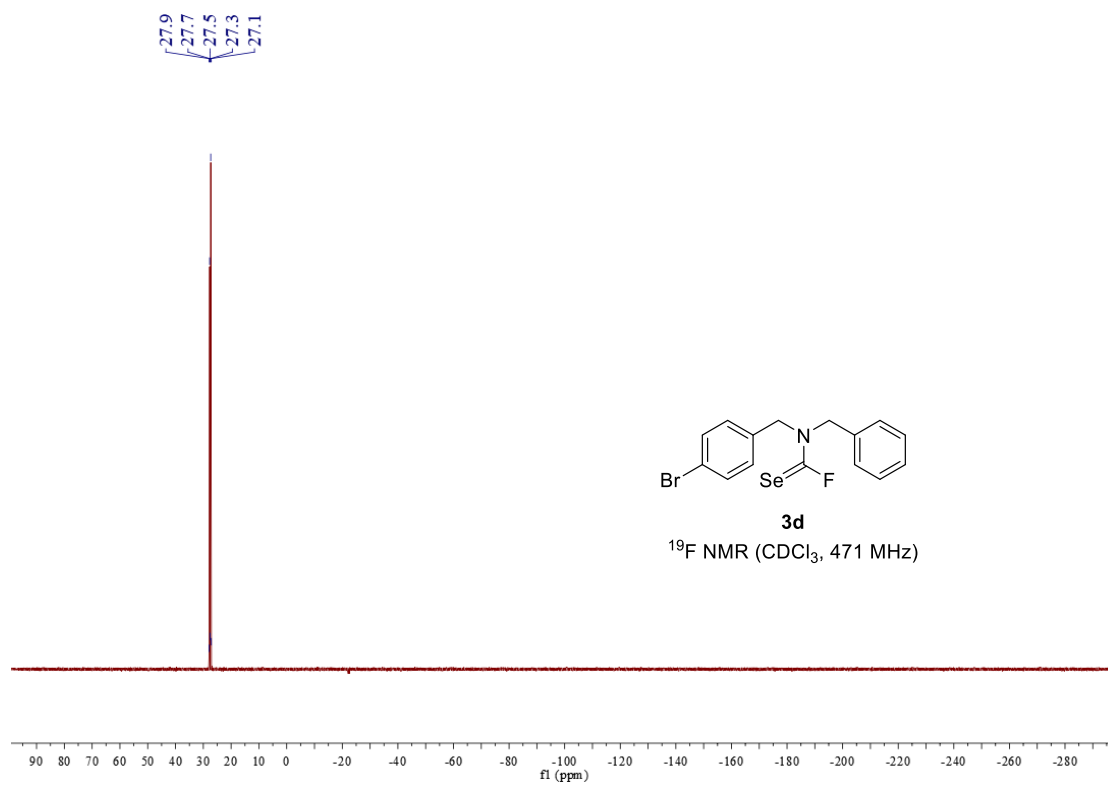


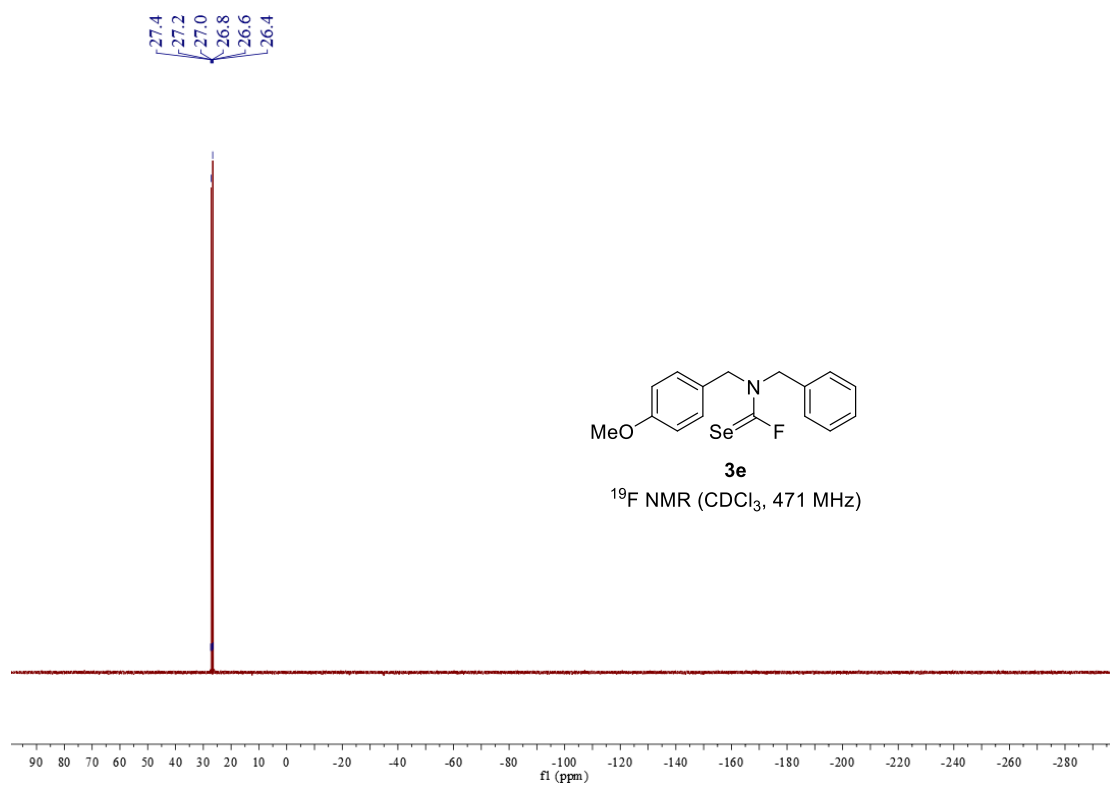
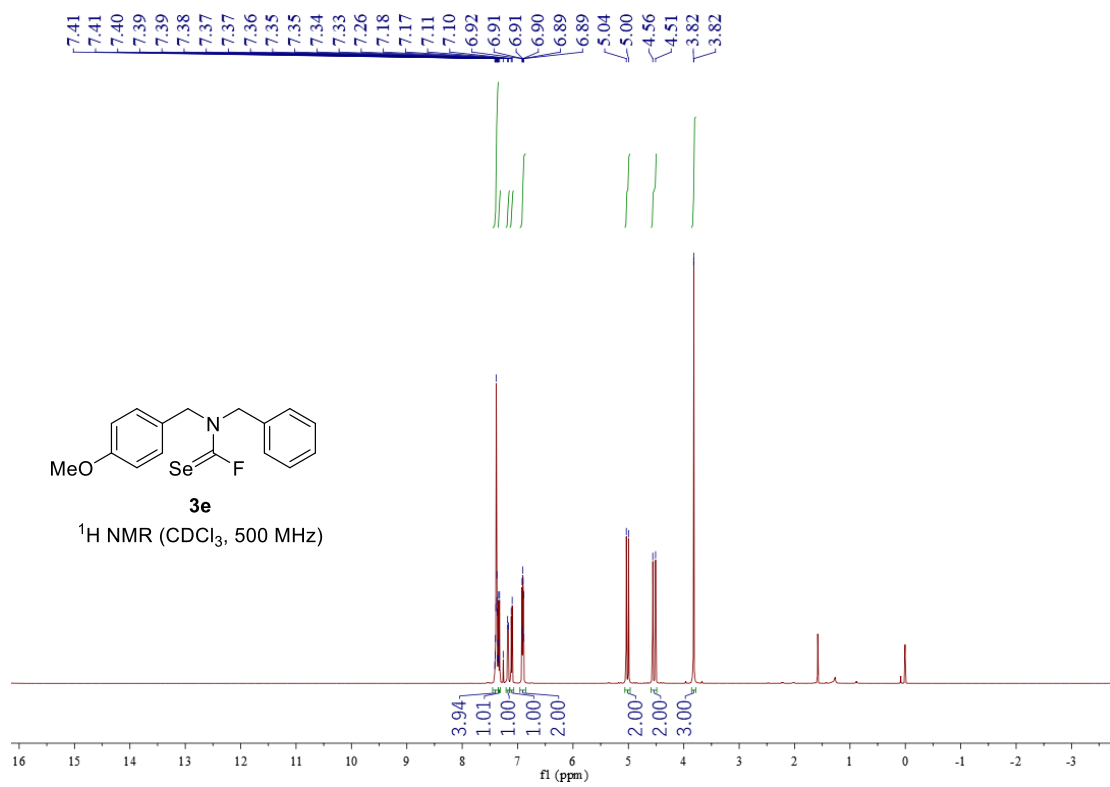


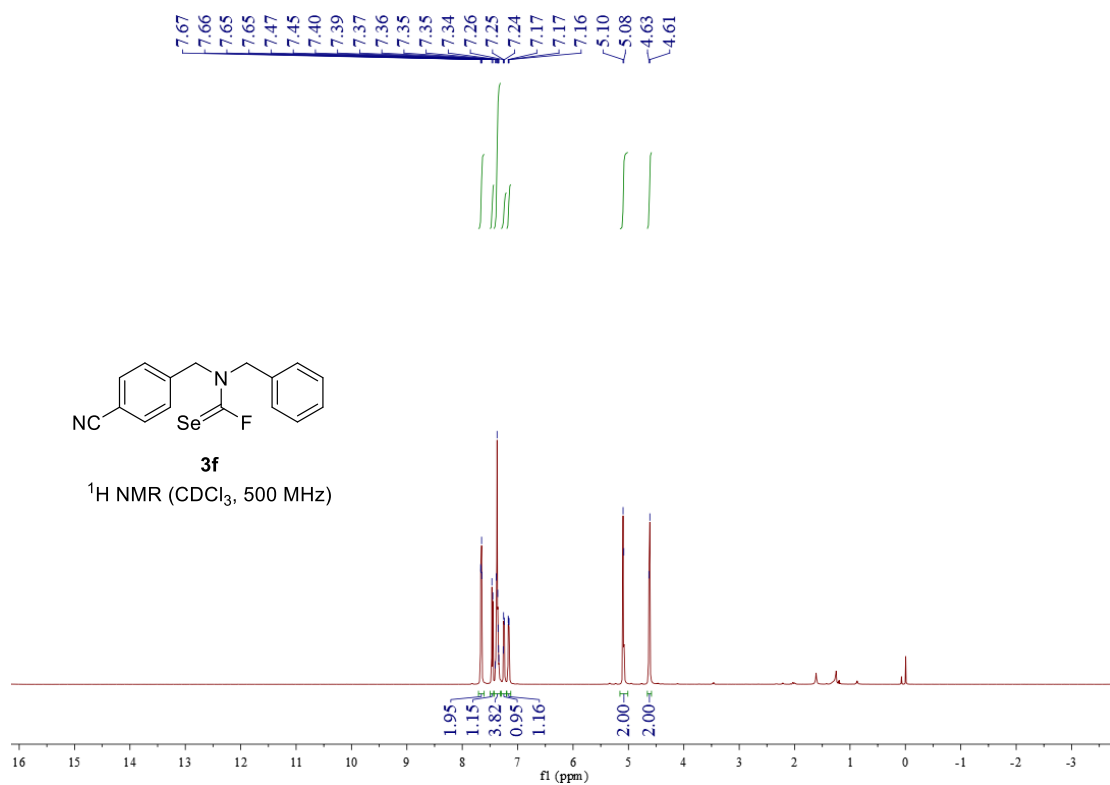
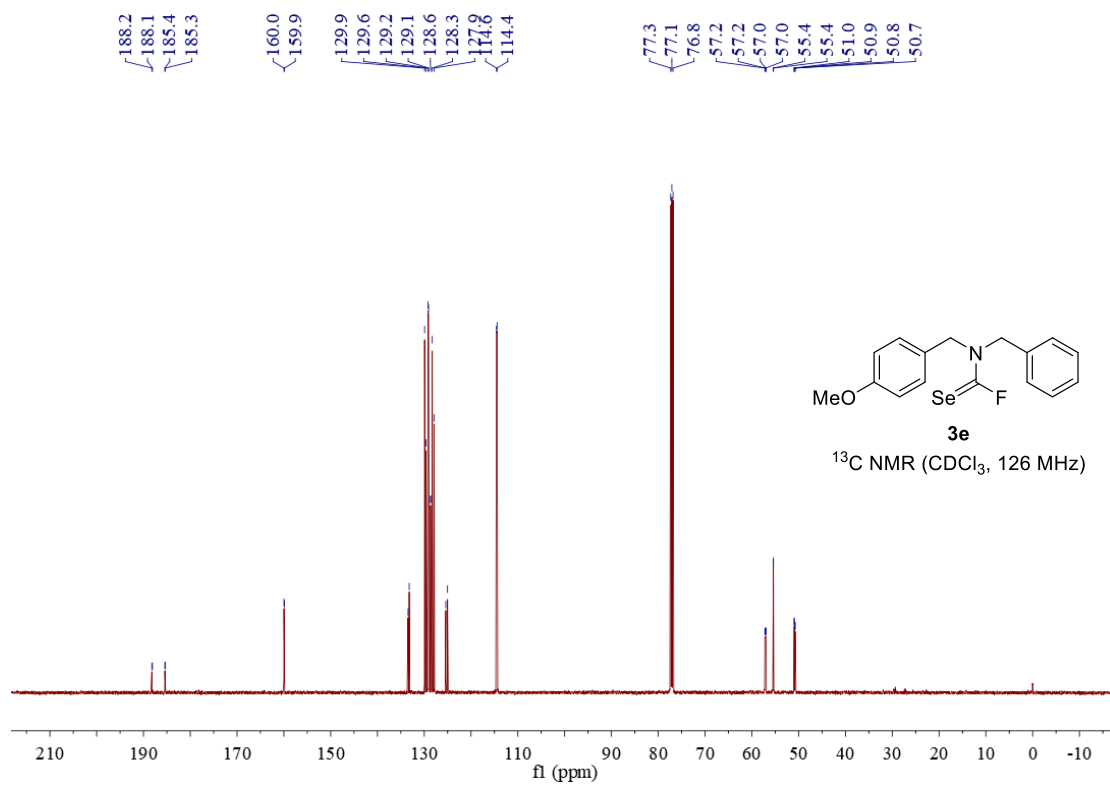


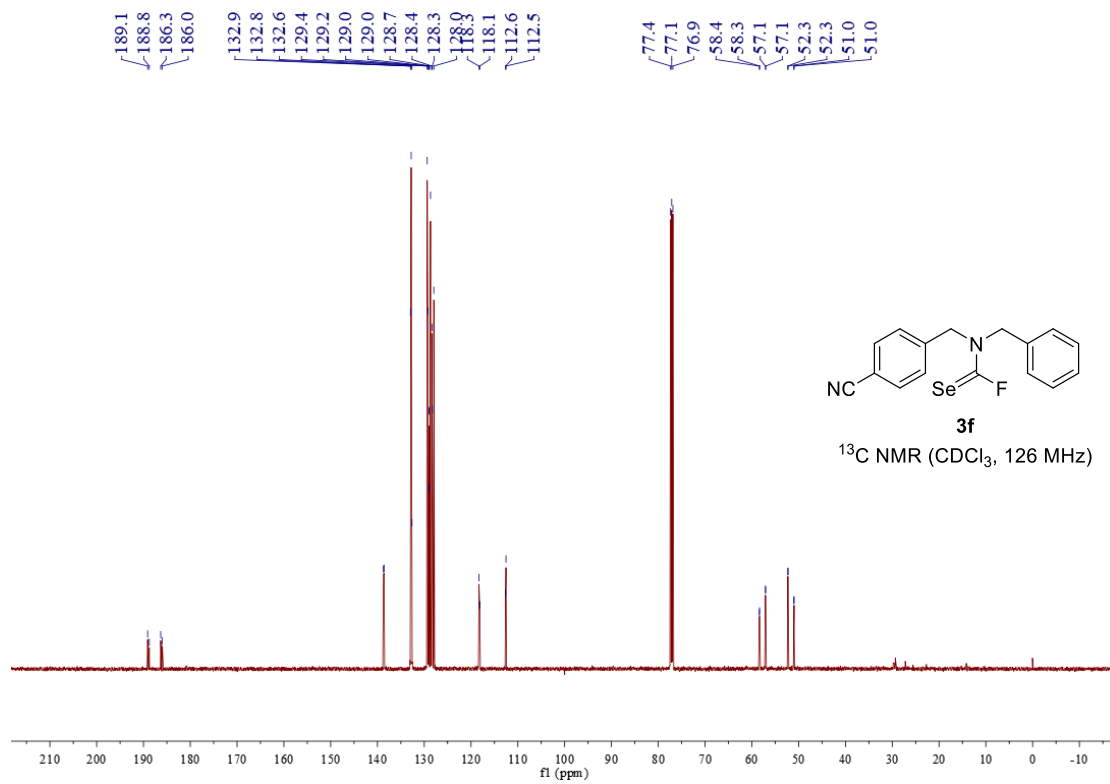
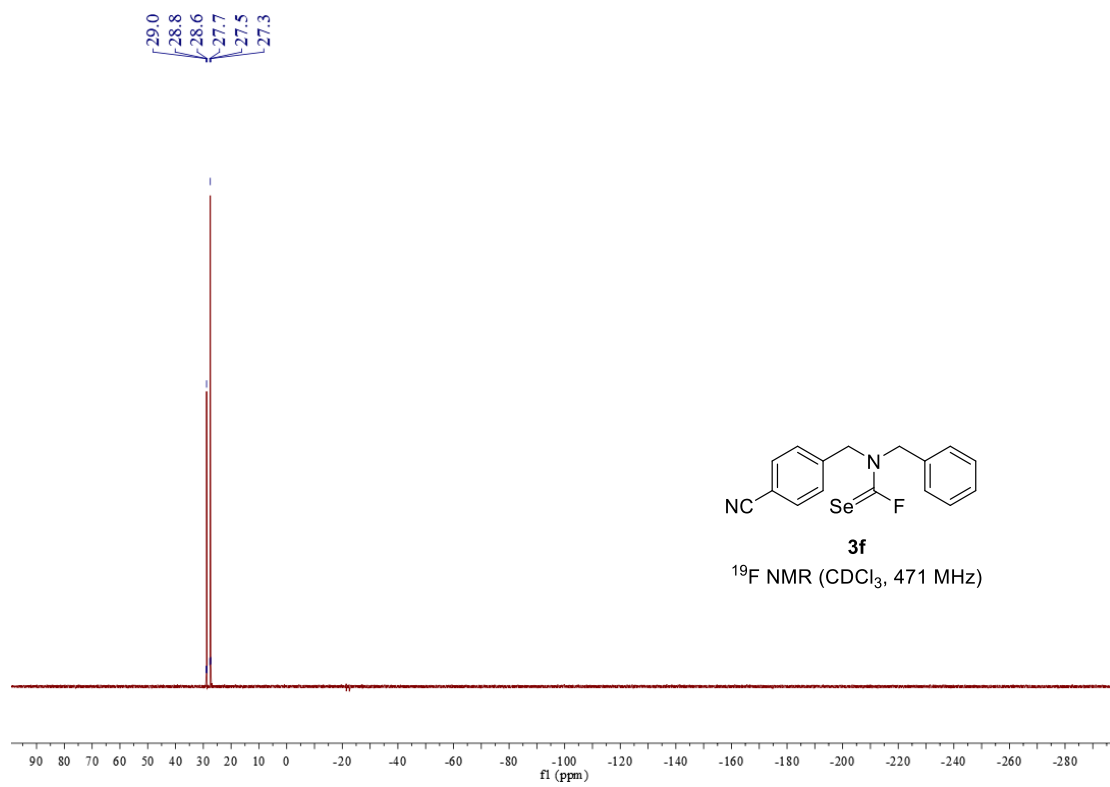


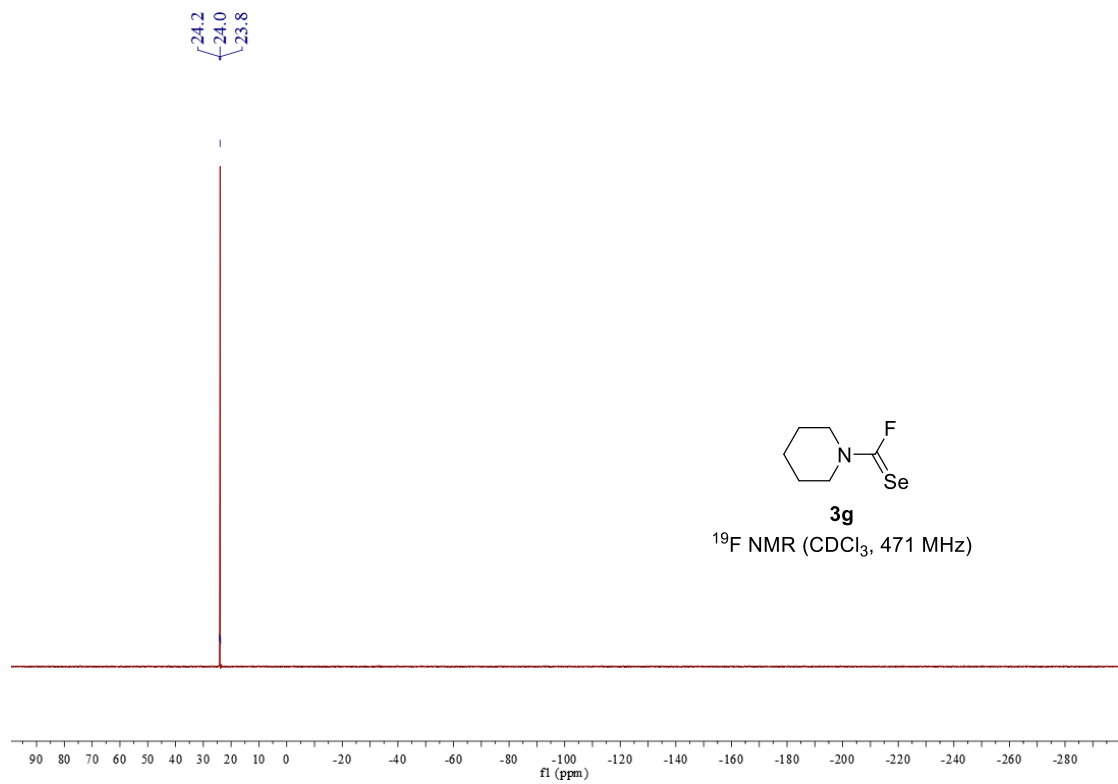
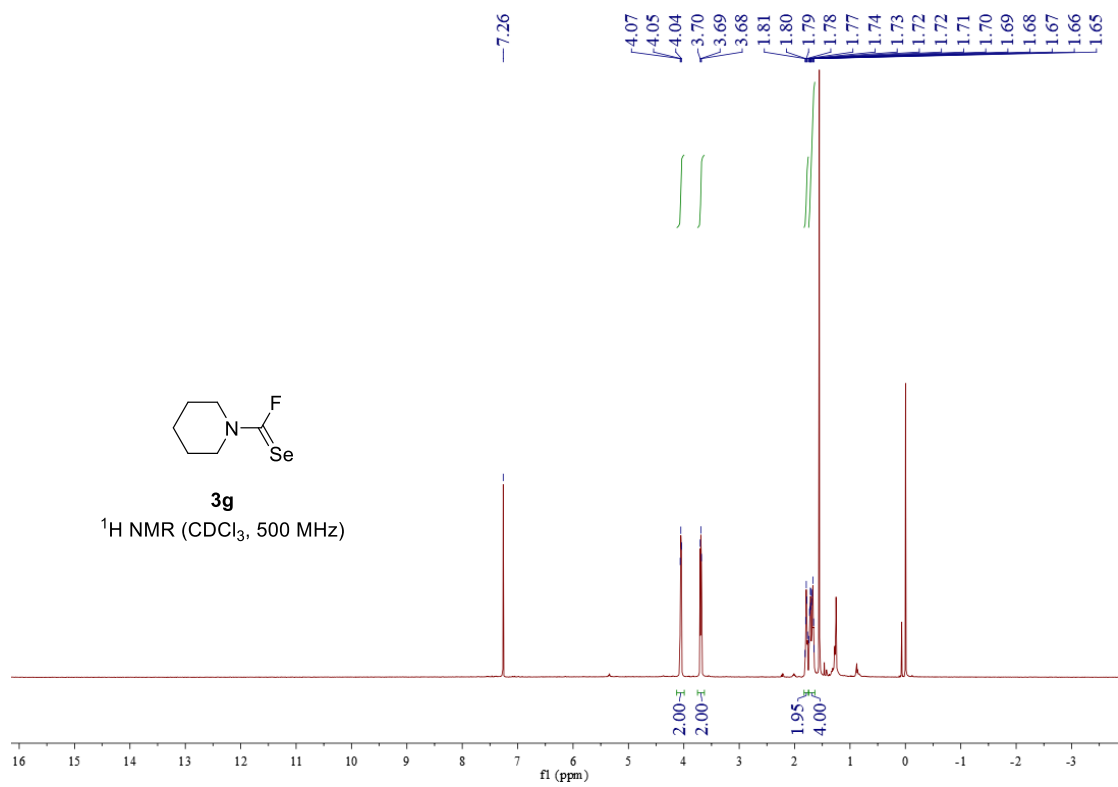


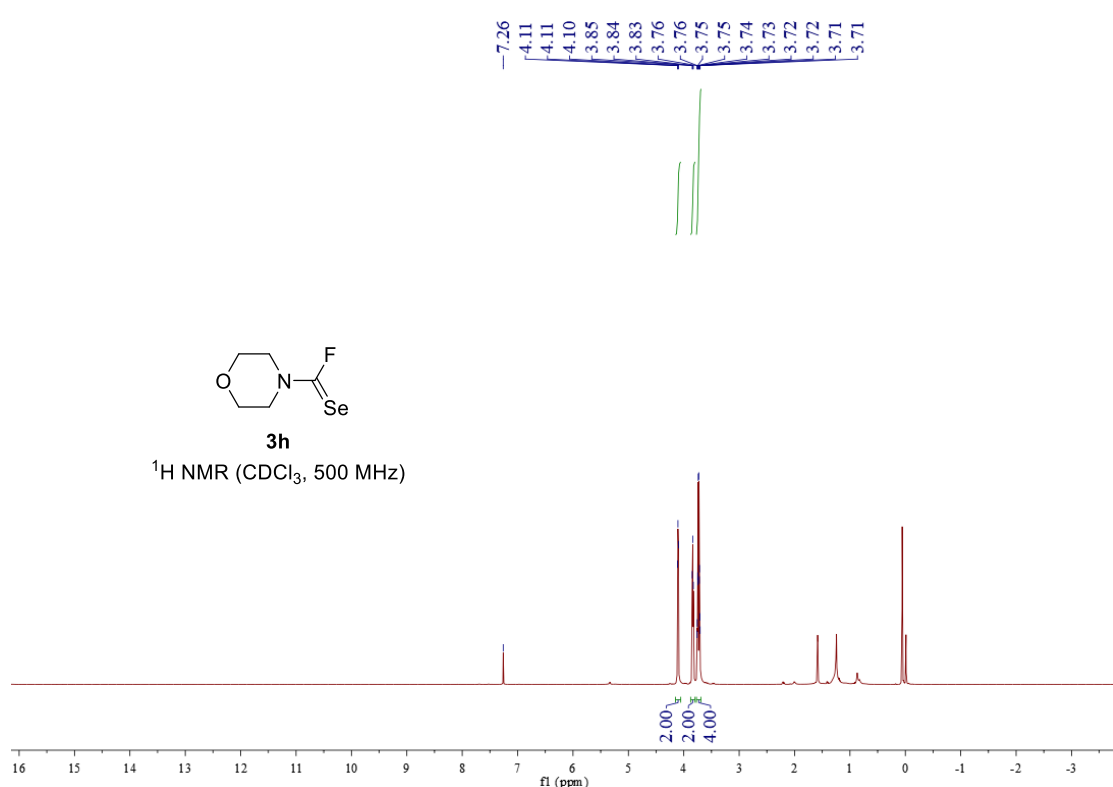
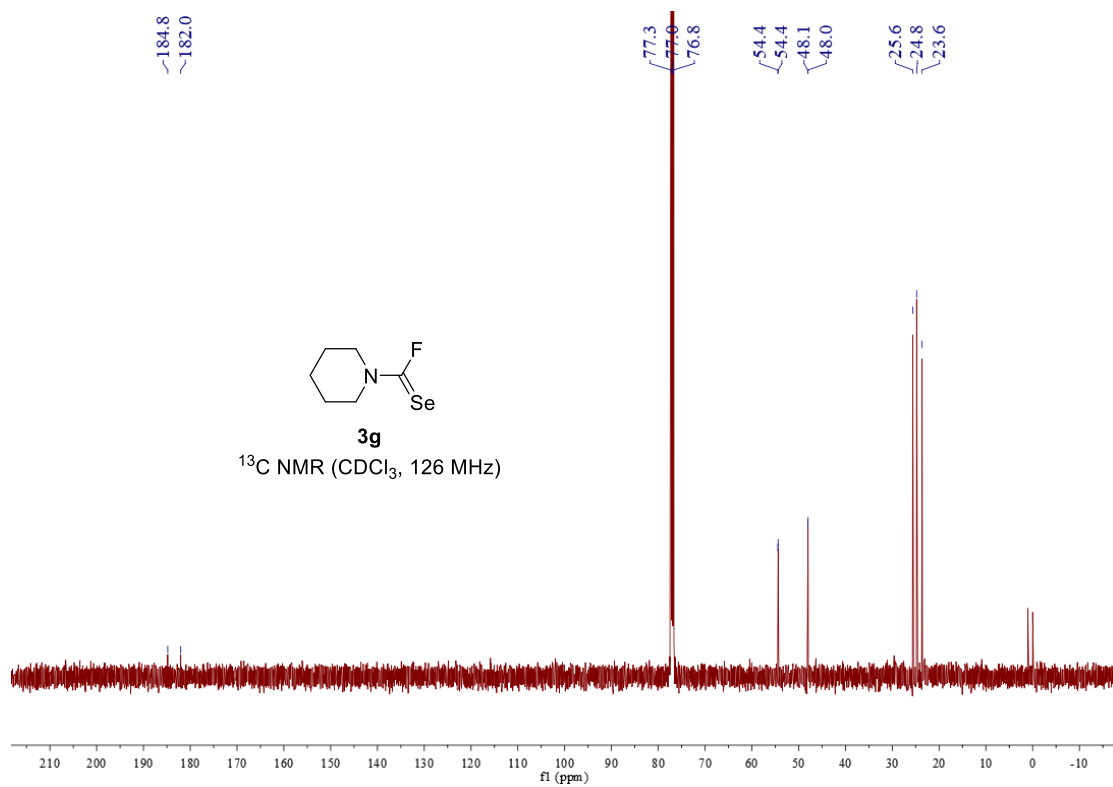


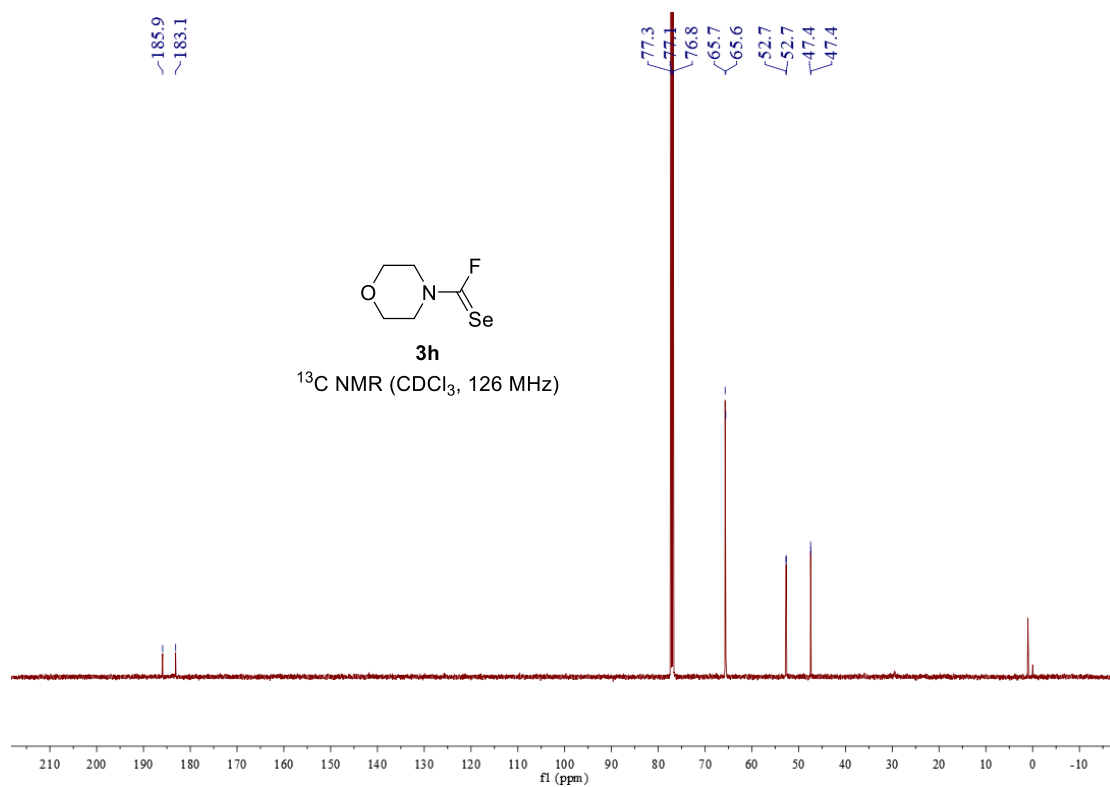
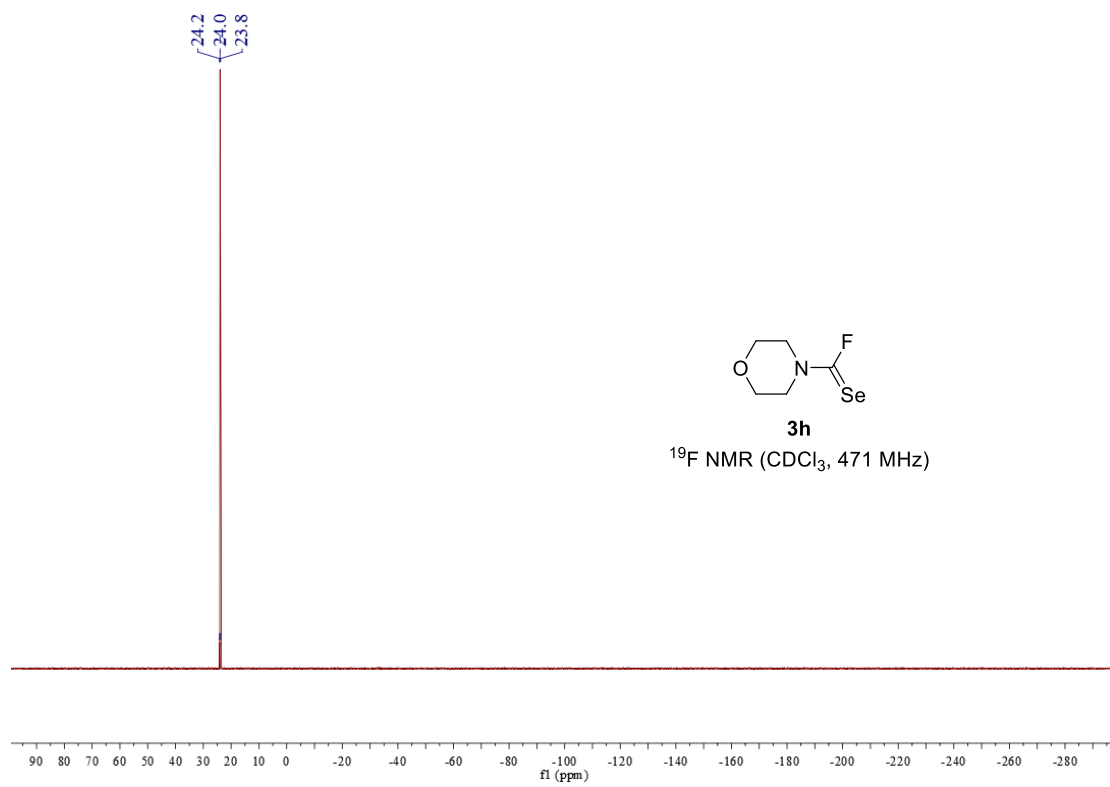


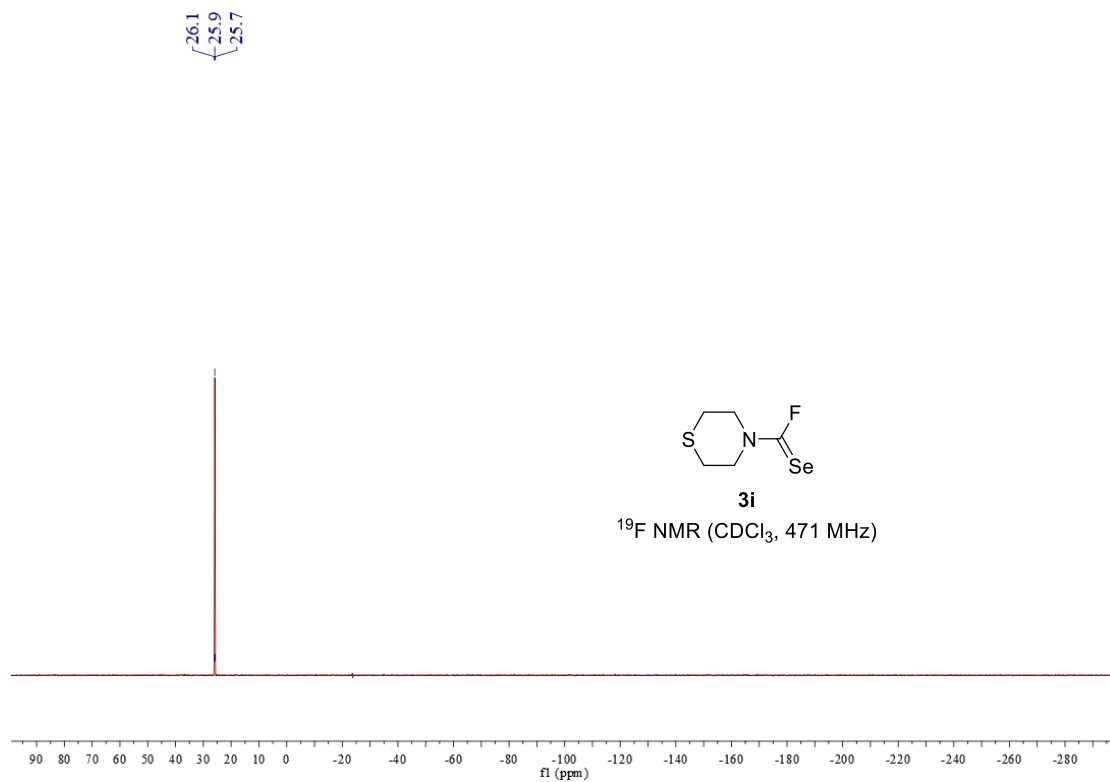
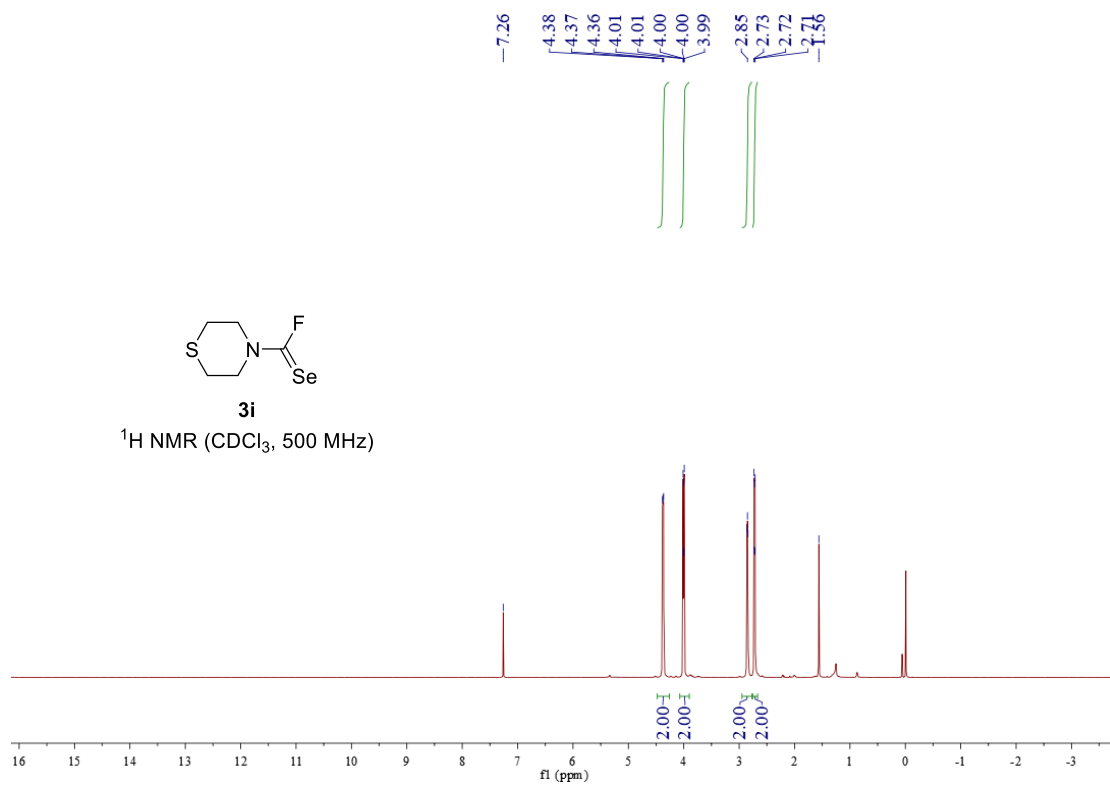


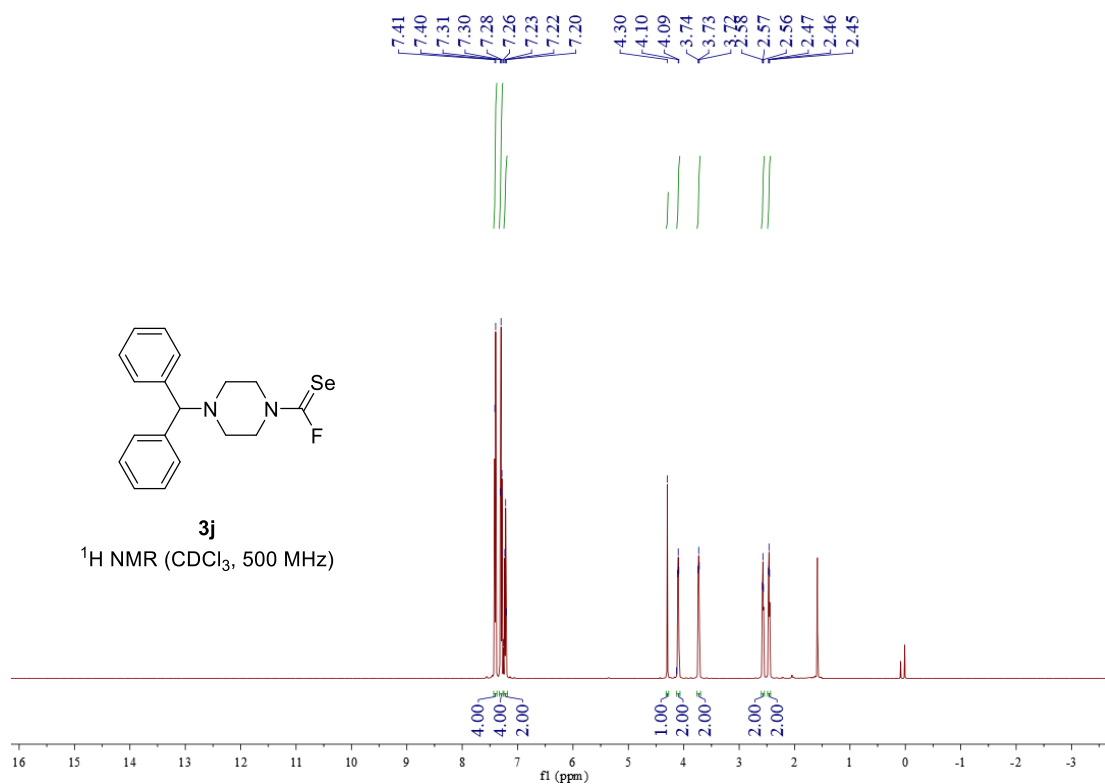
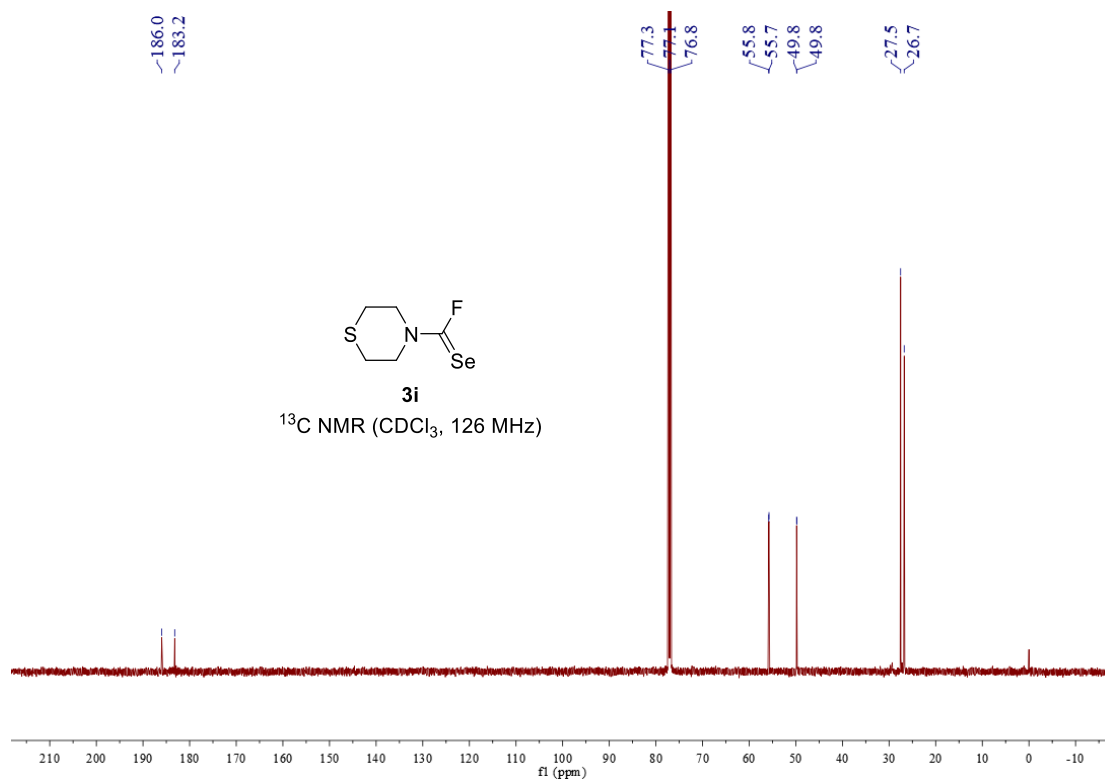


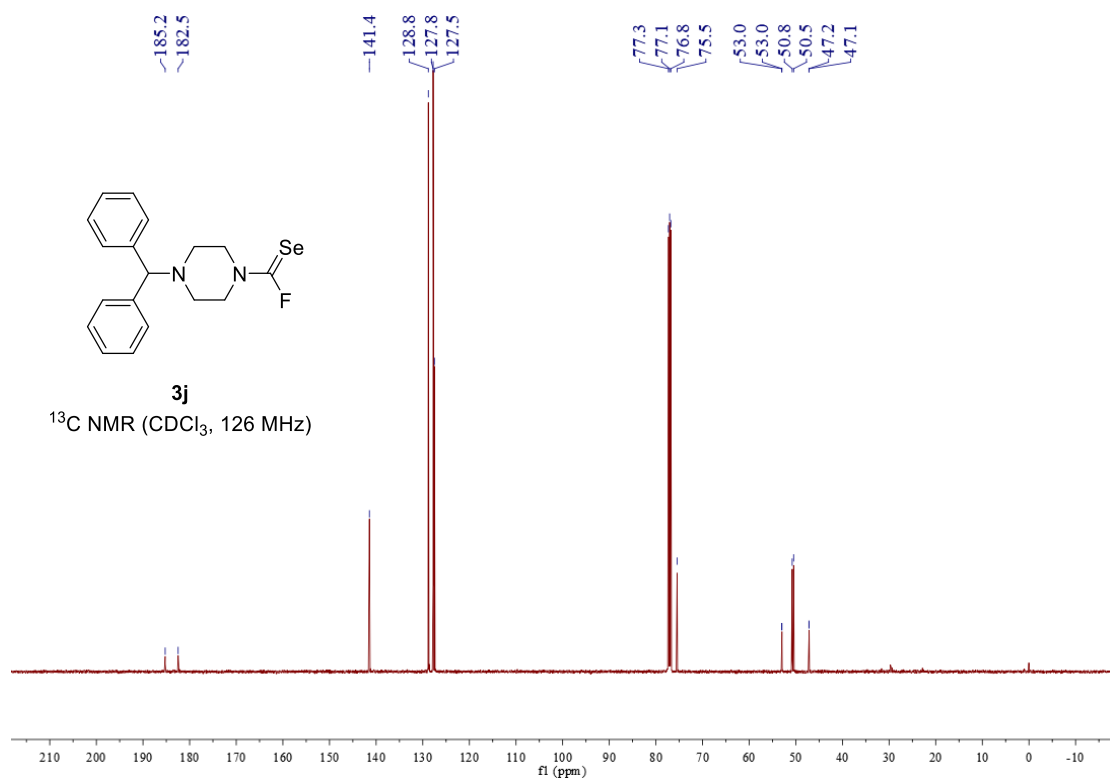
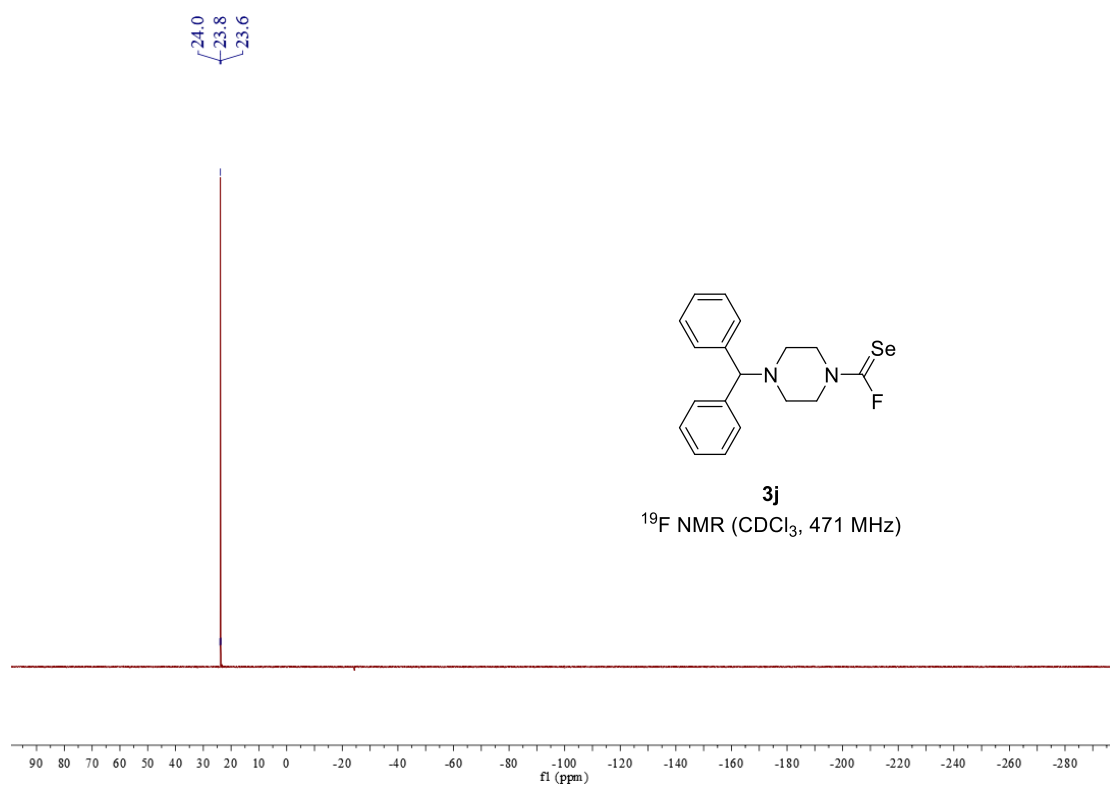


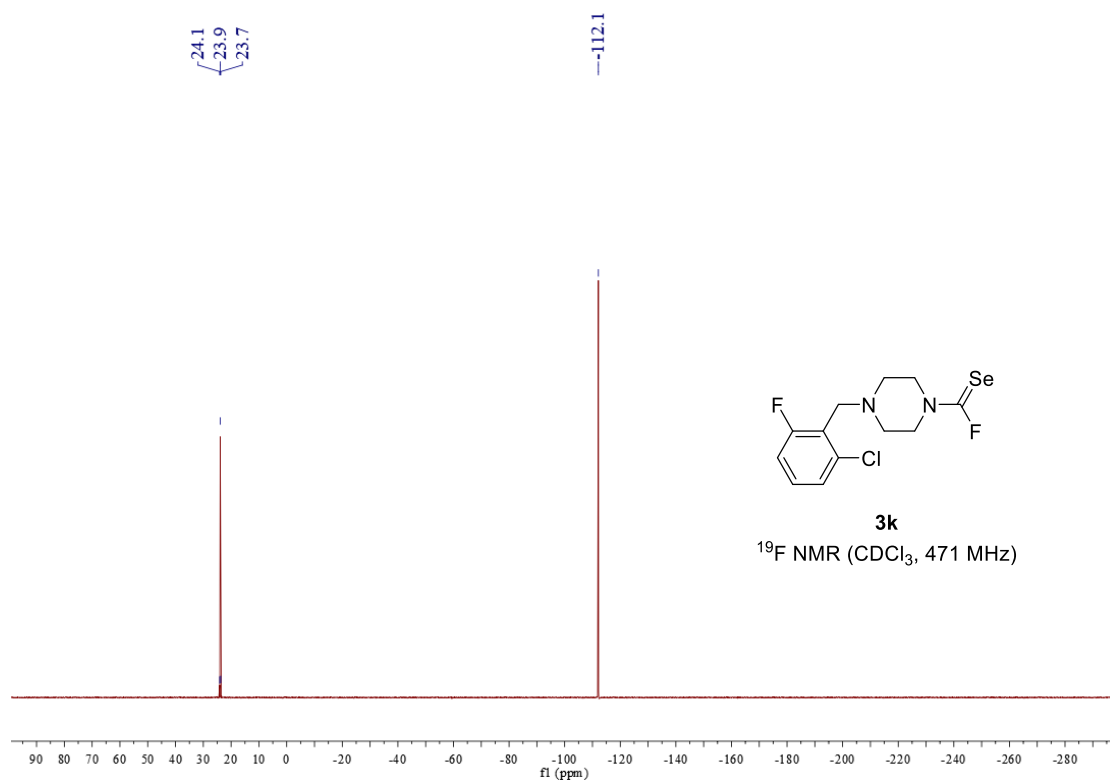
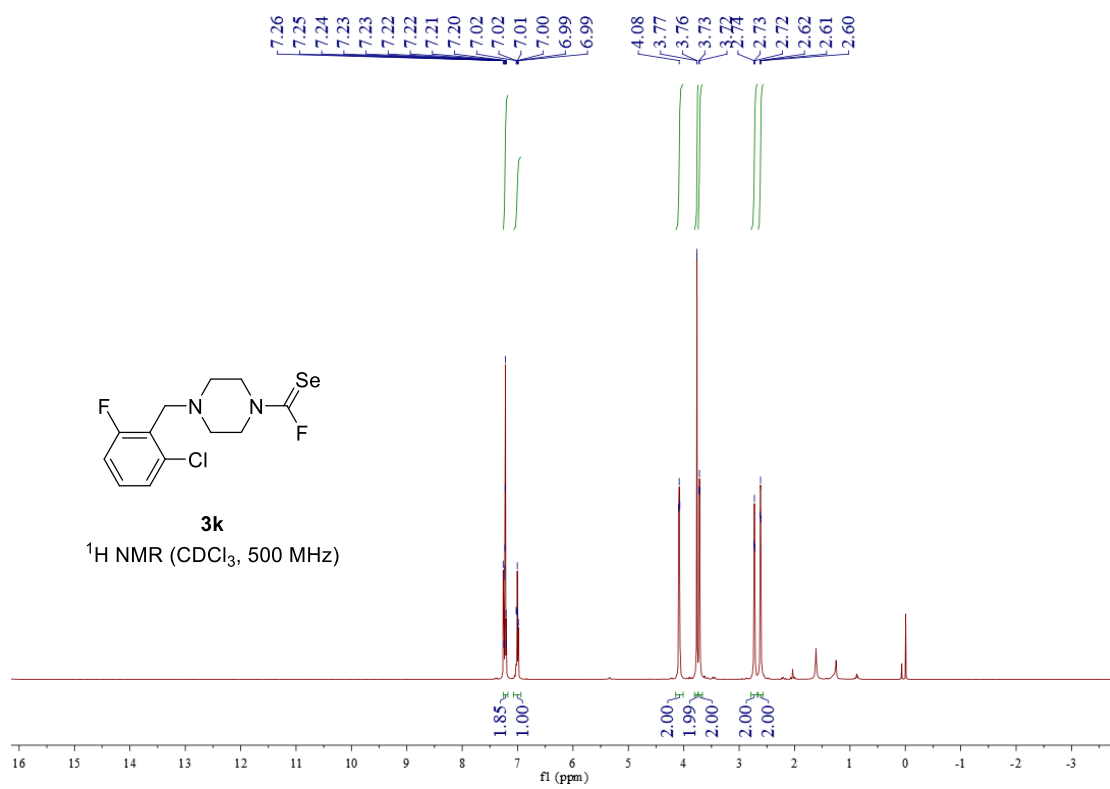


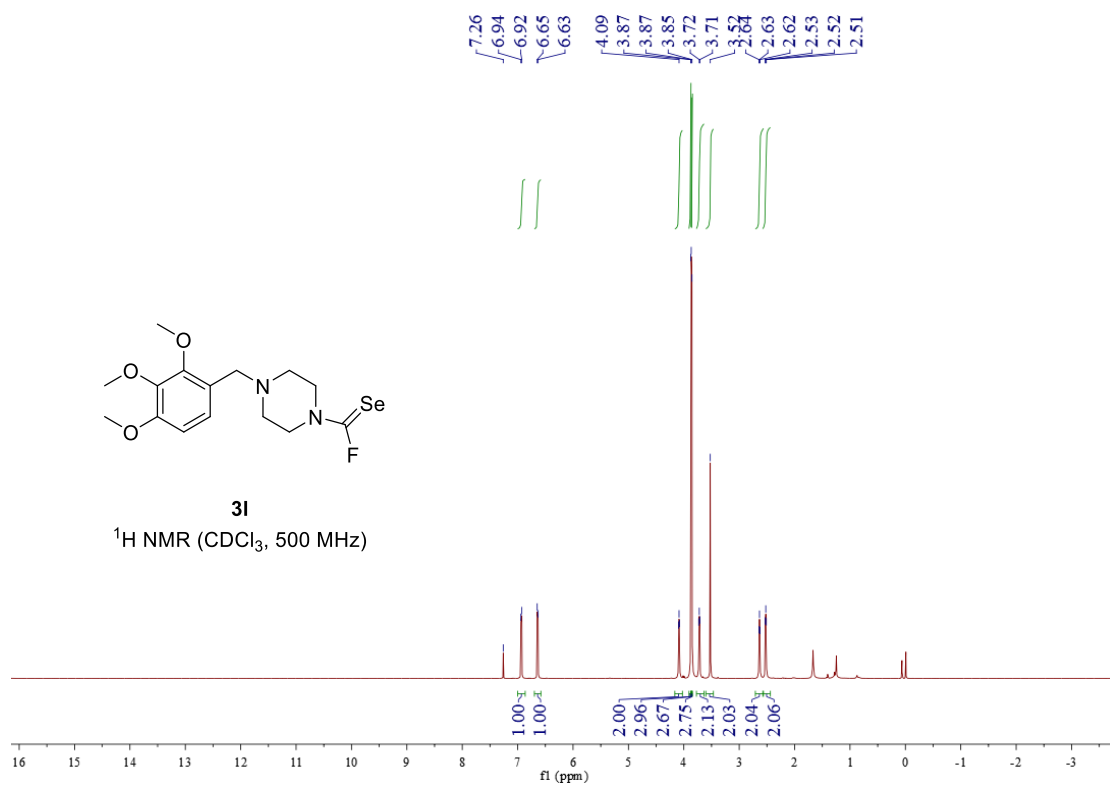
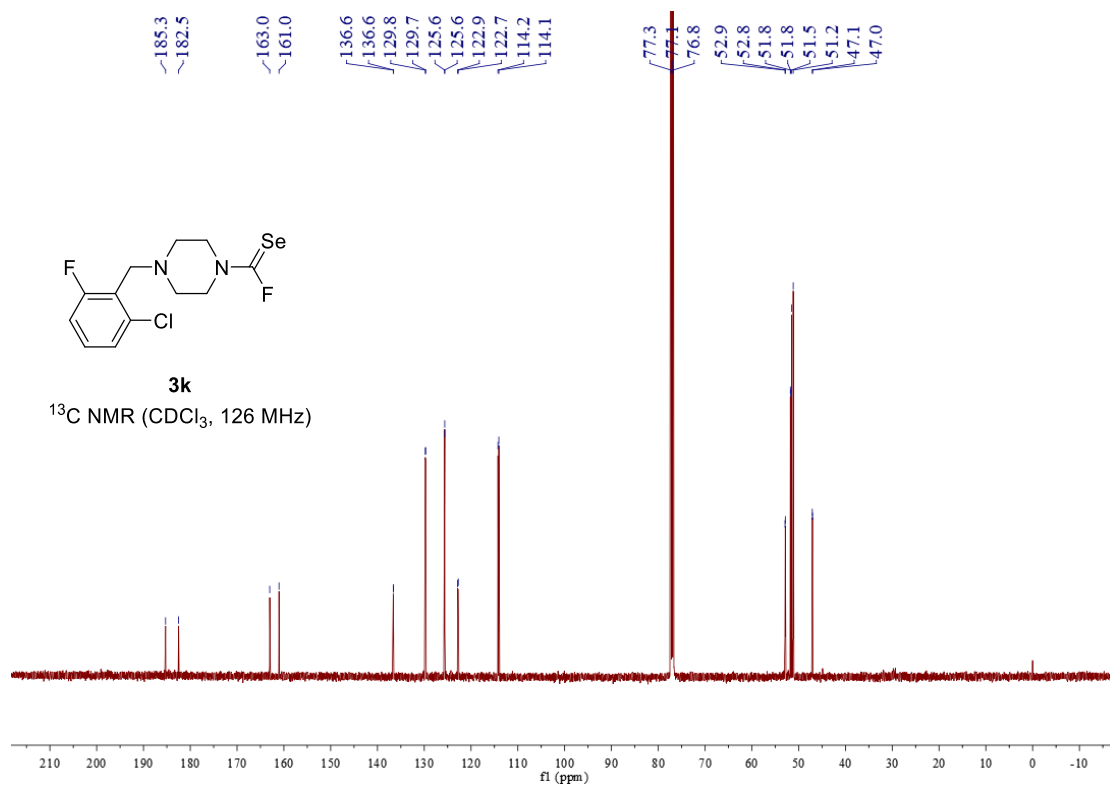


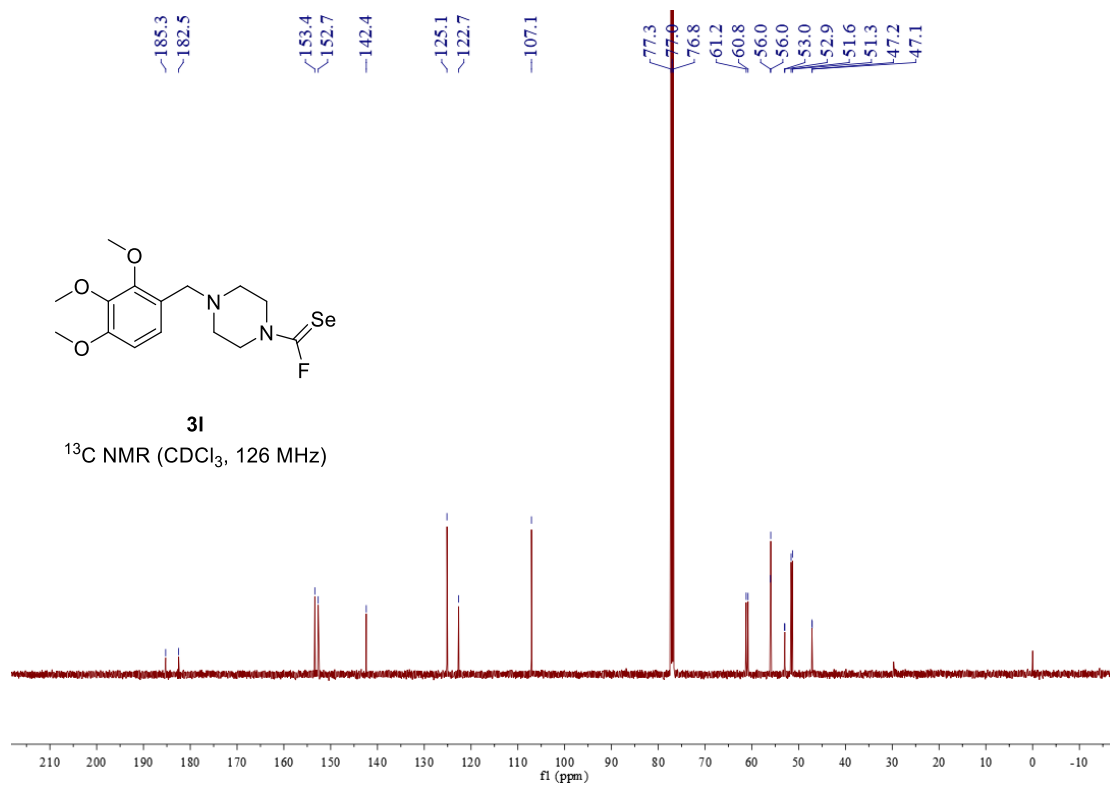
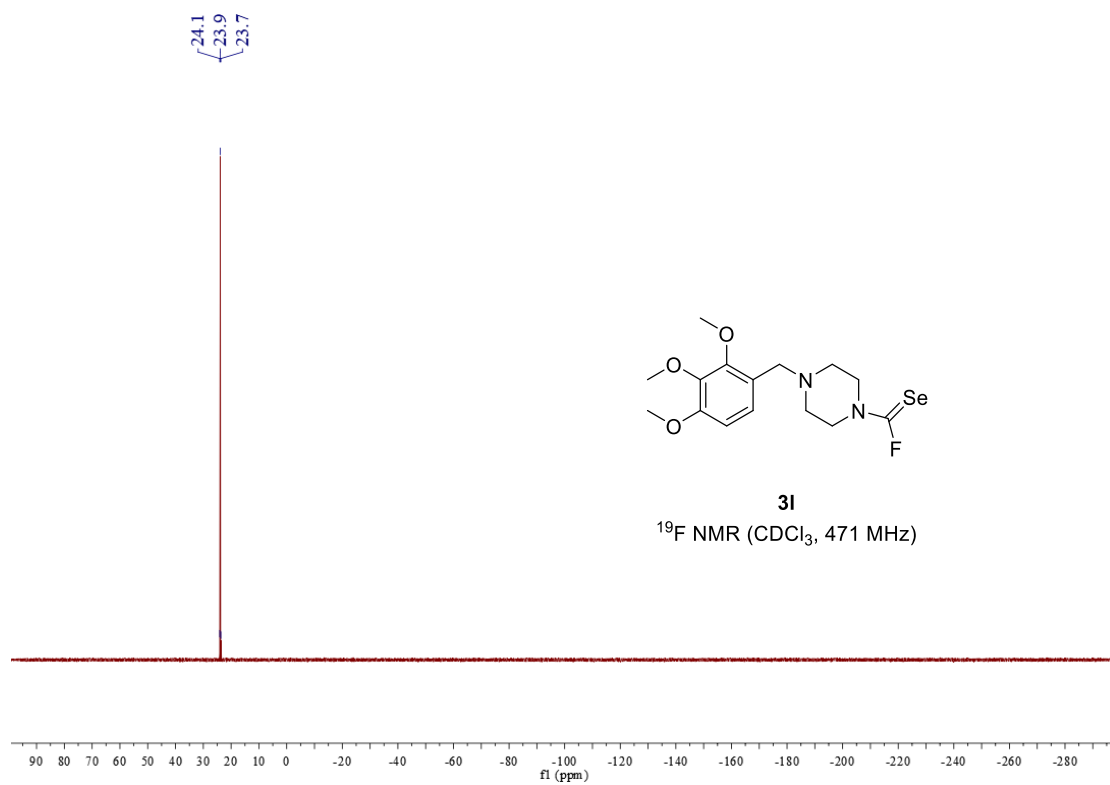




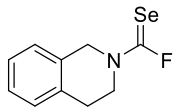






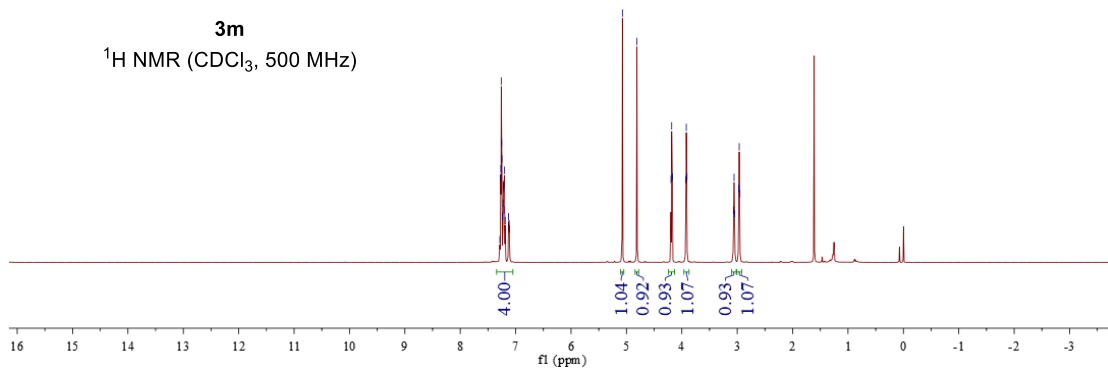


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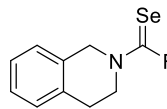


3m

¹H NMR (CDCl₃, 500 MHz)

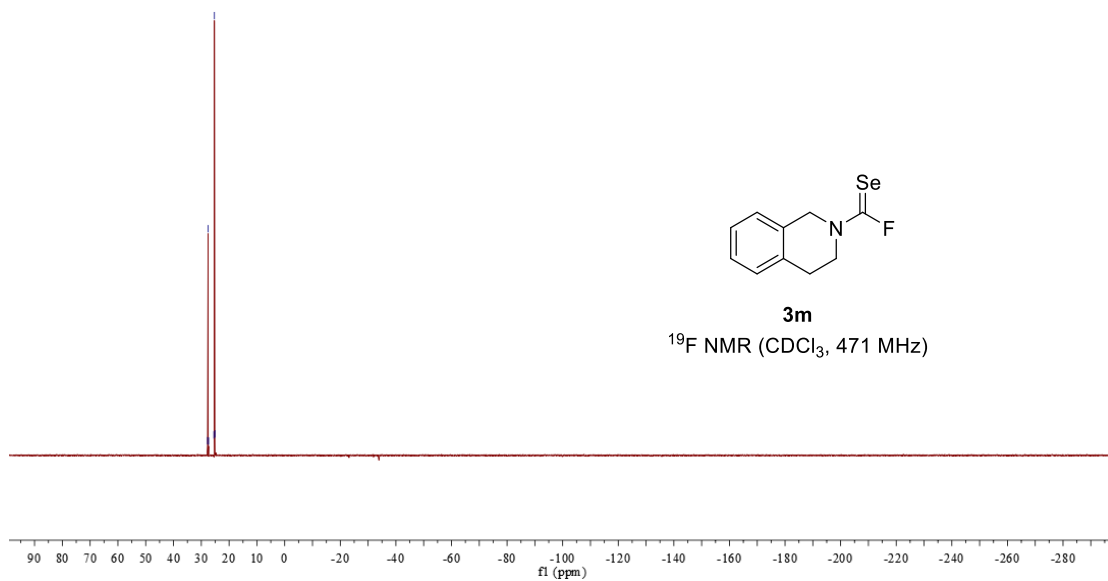


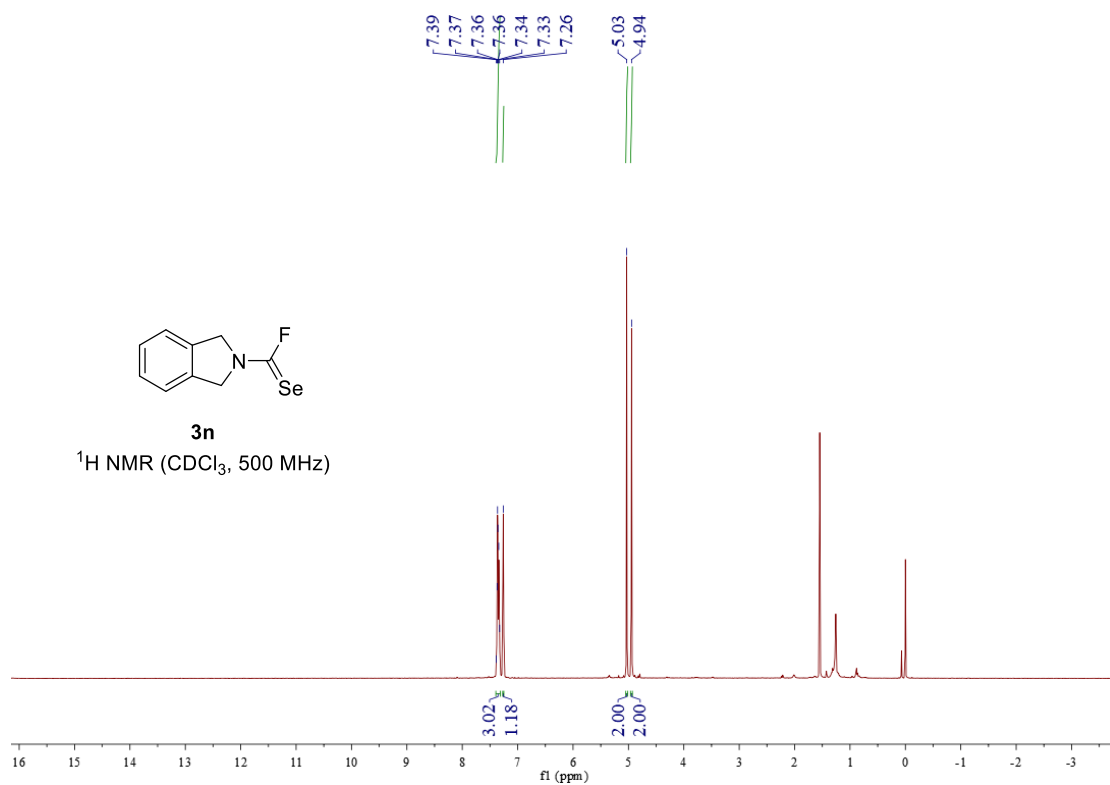
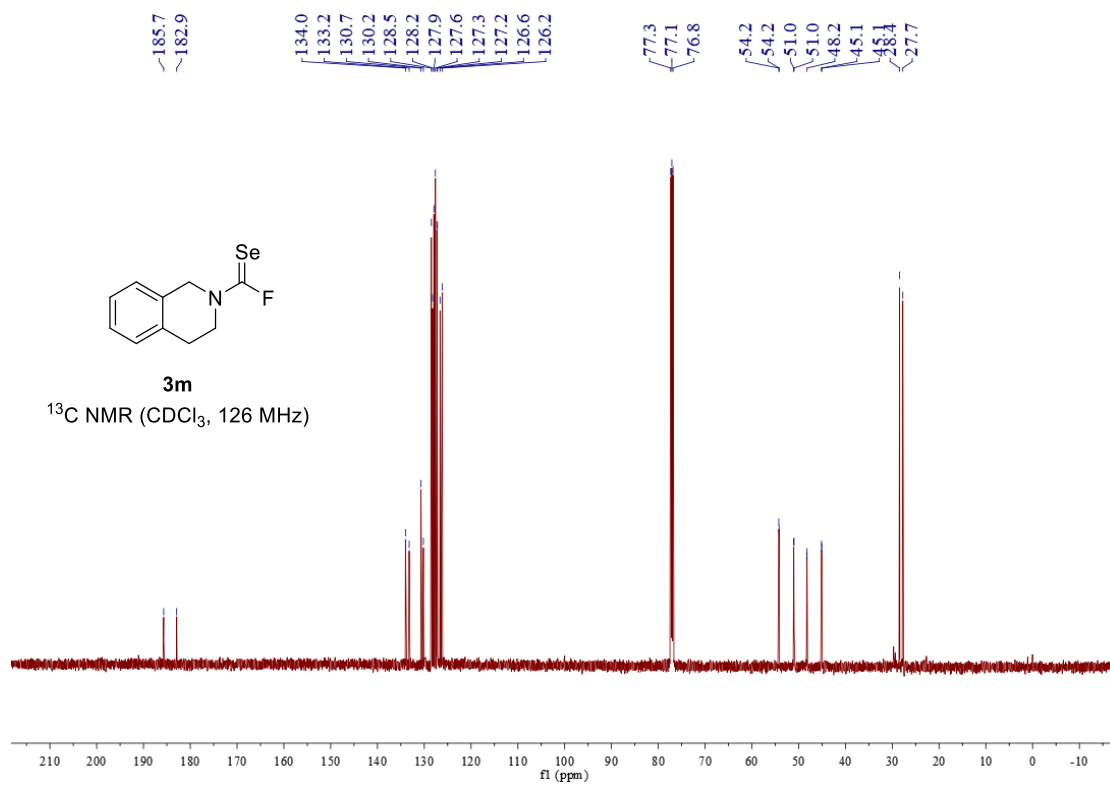
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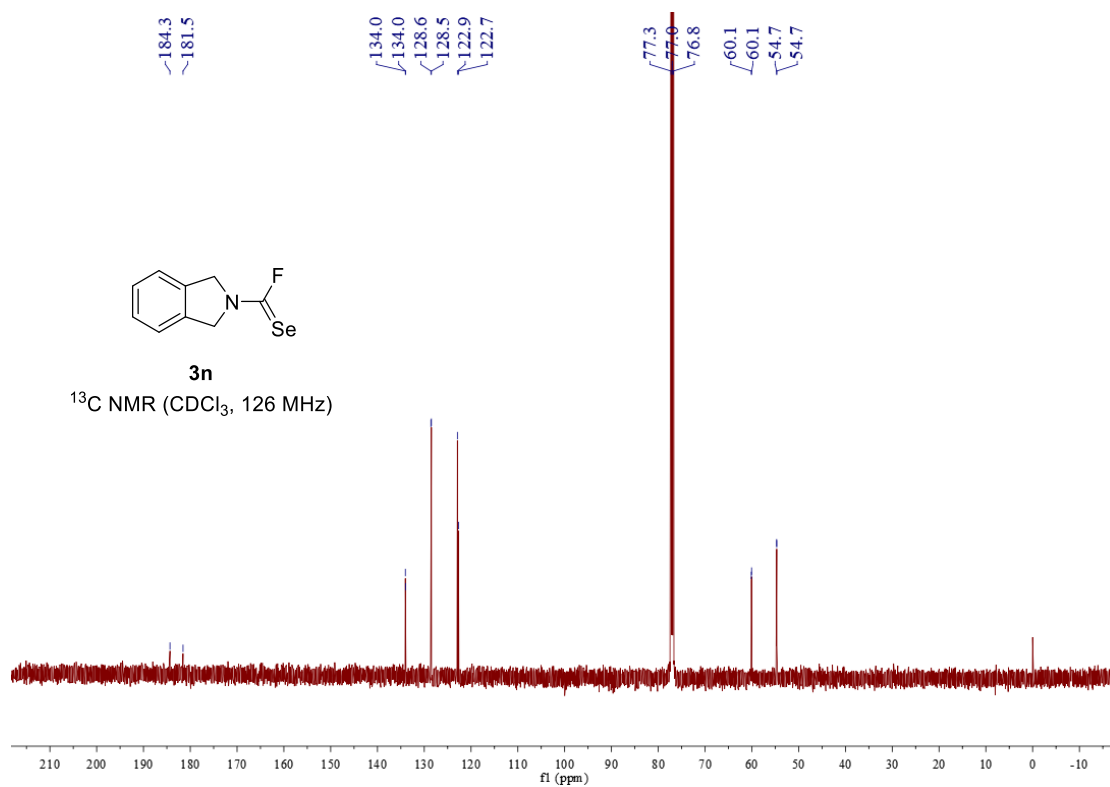
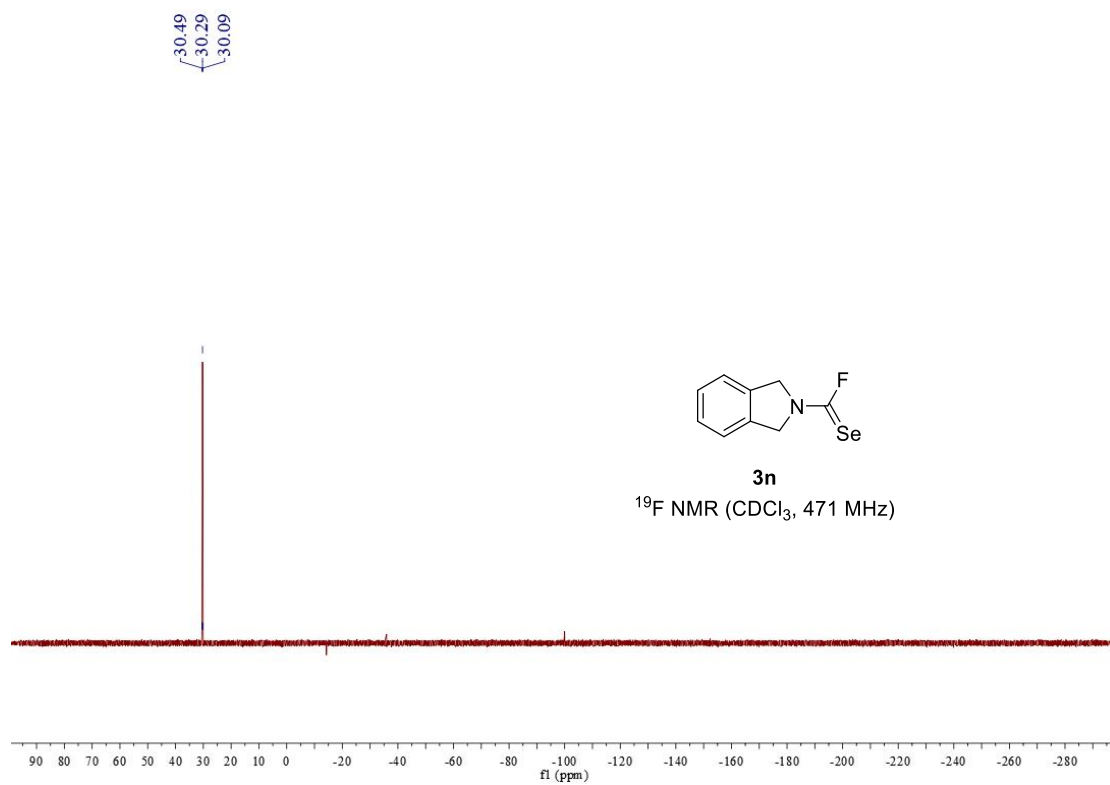


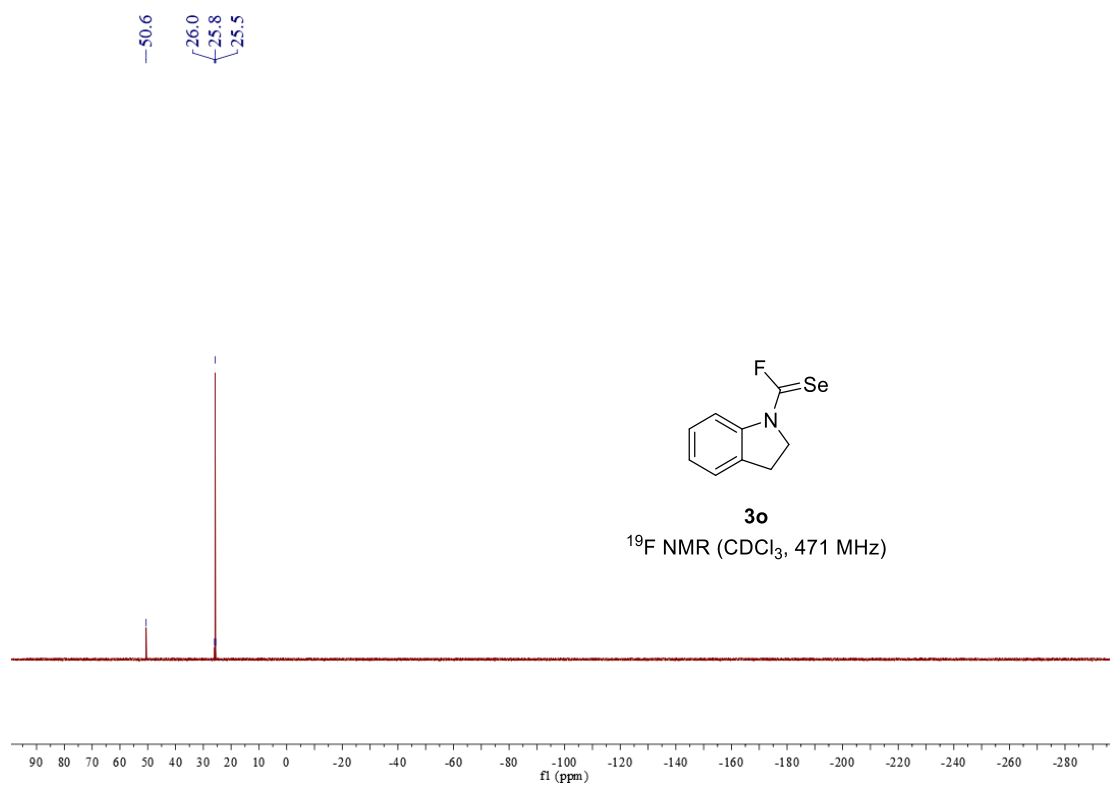
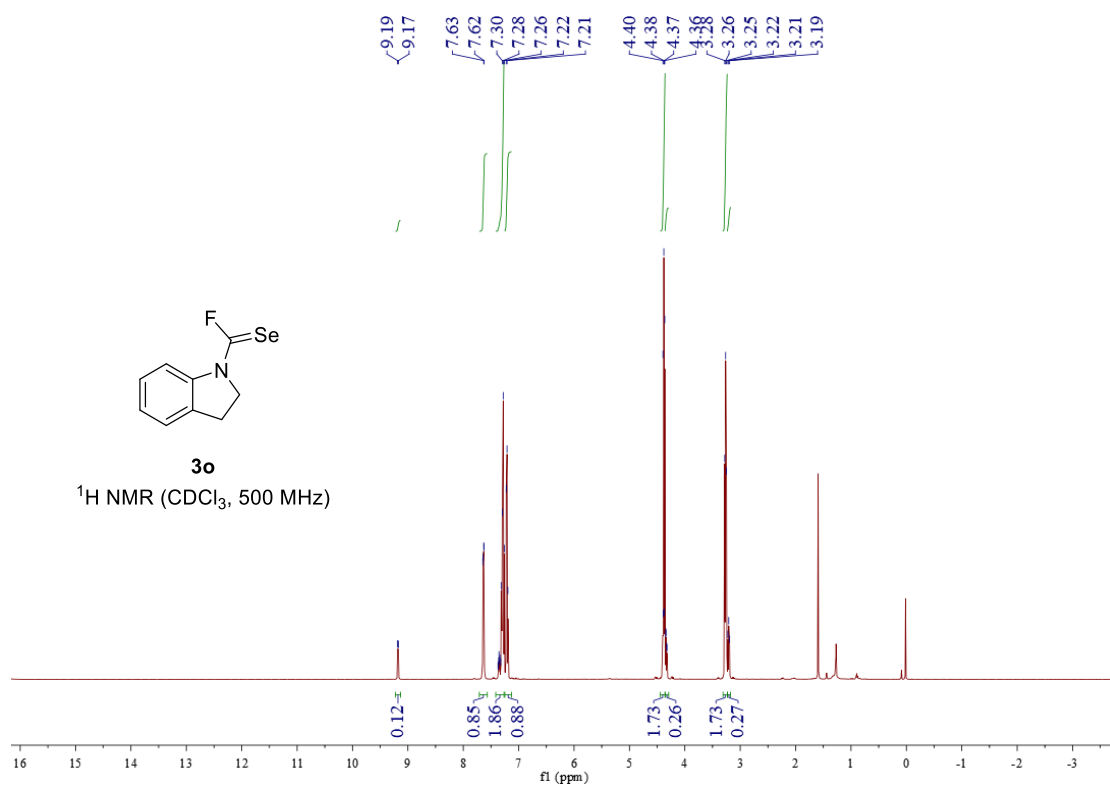
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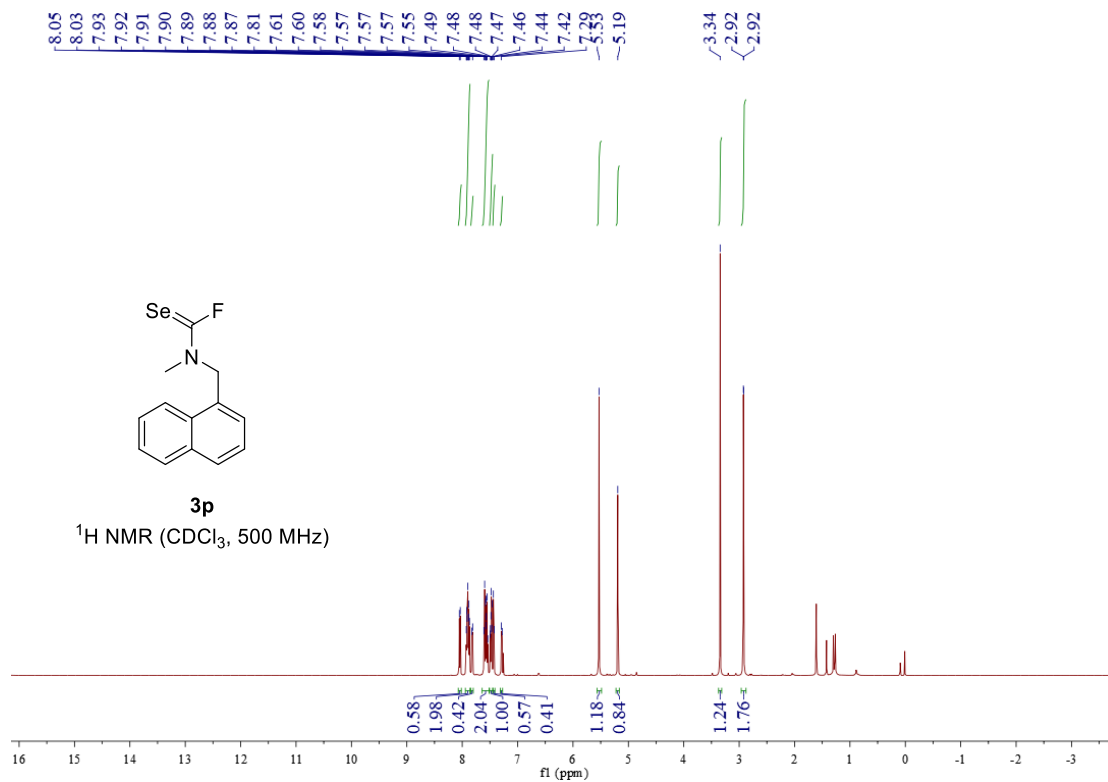
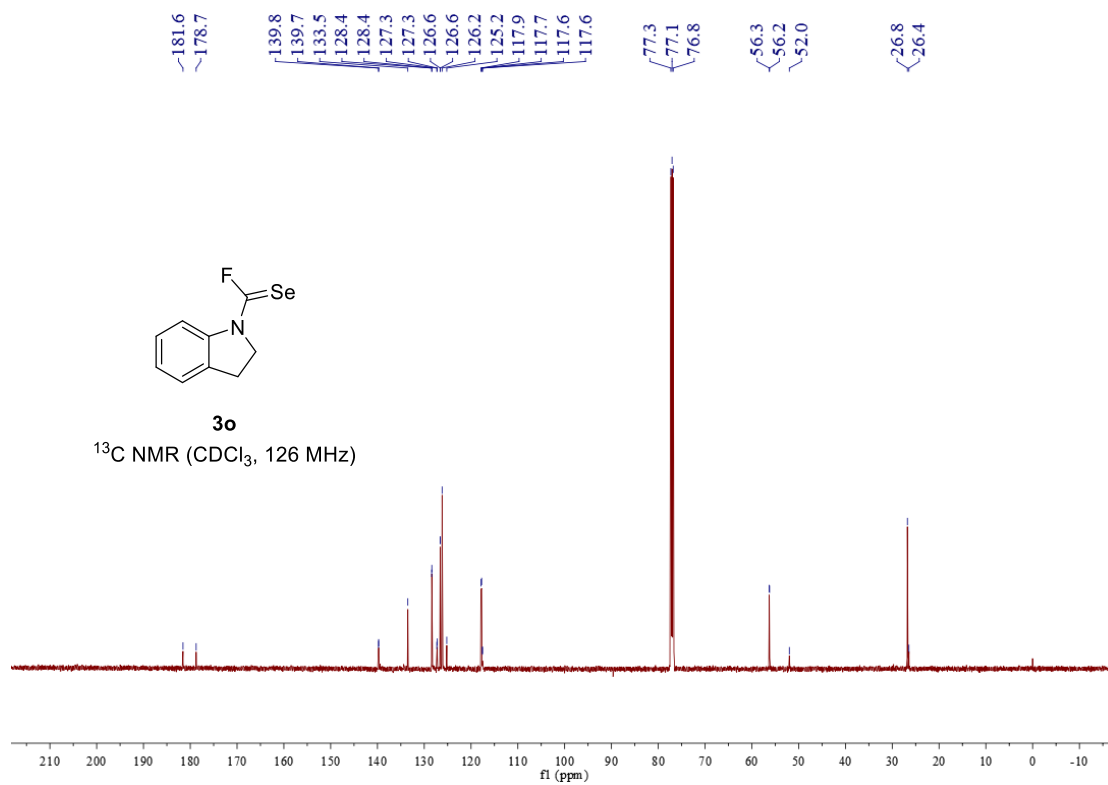
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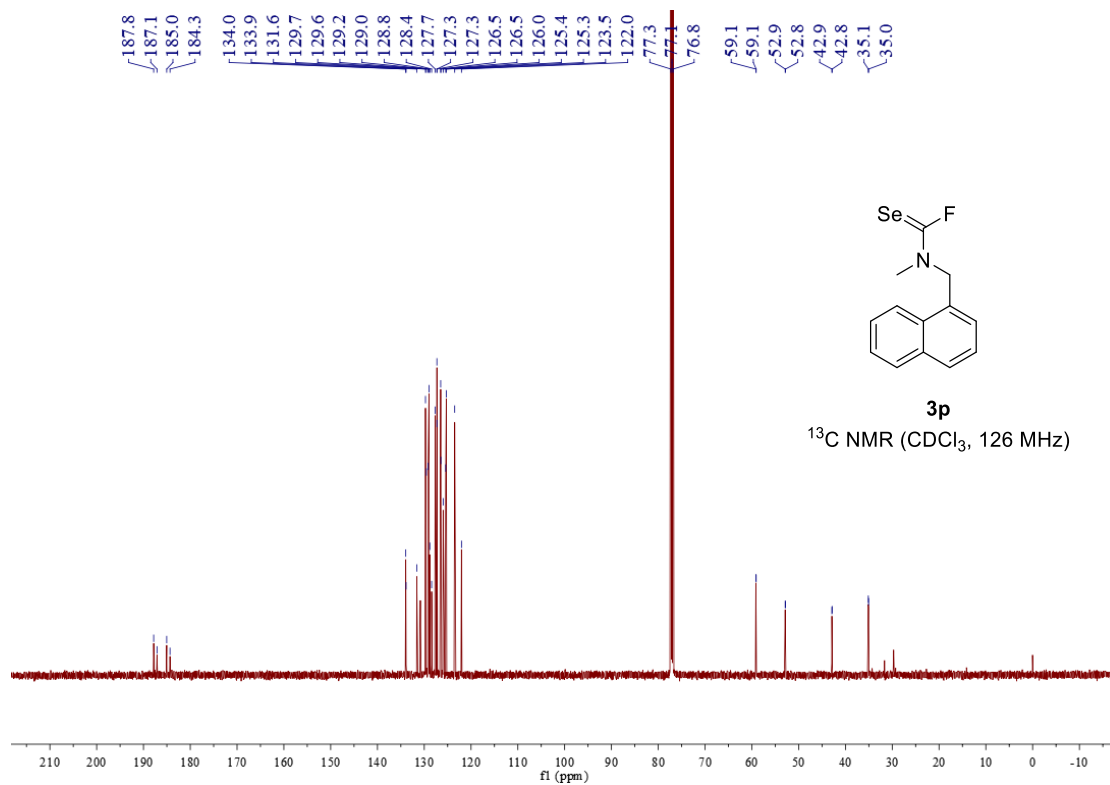
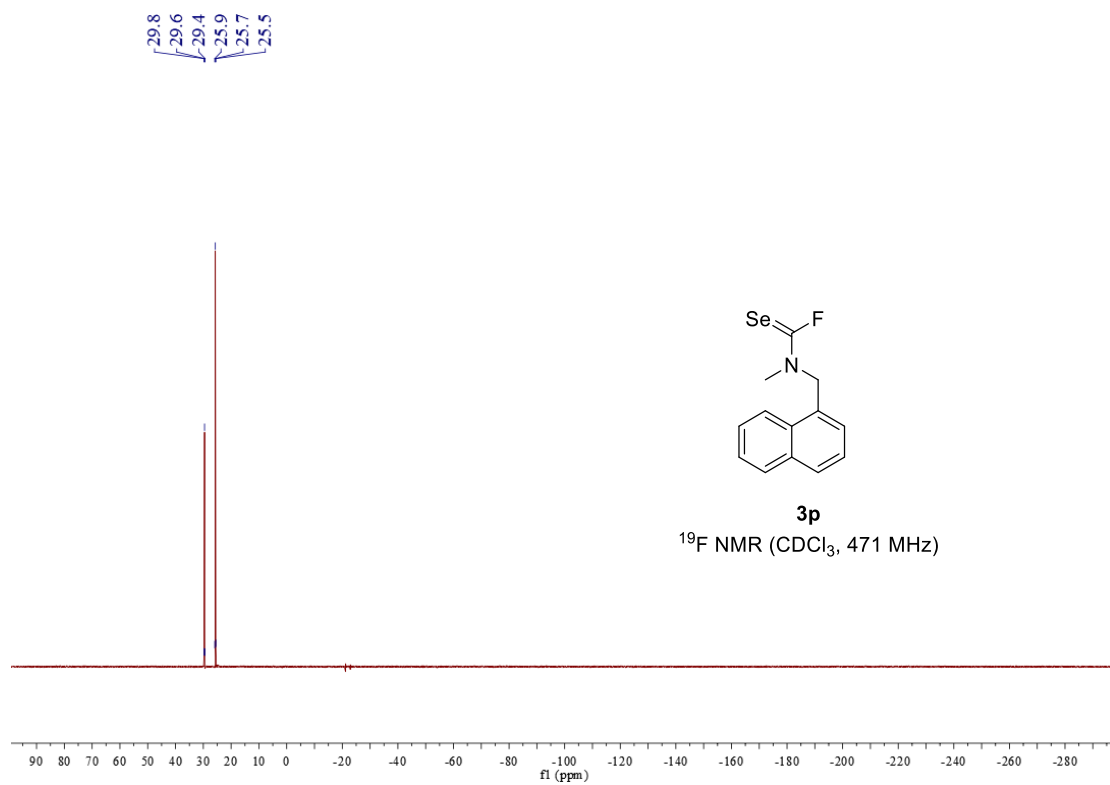


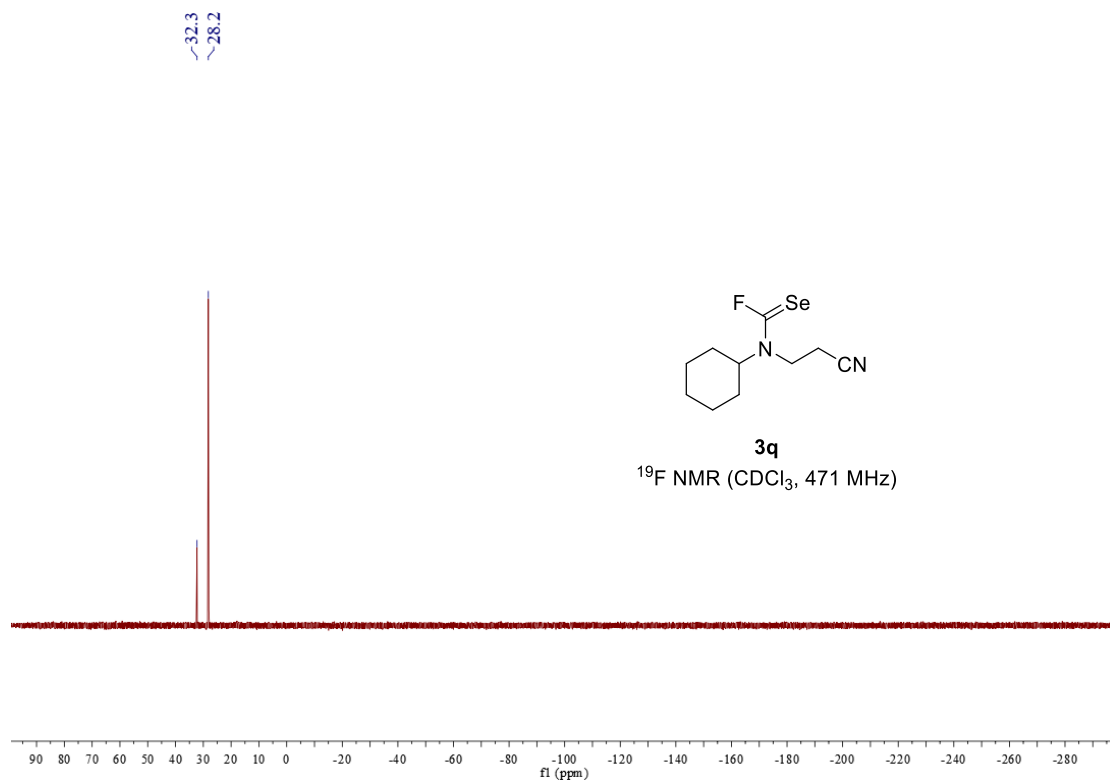
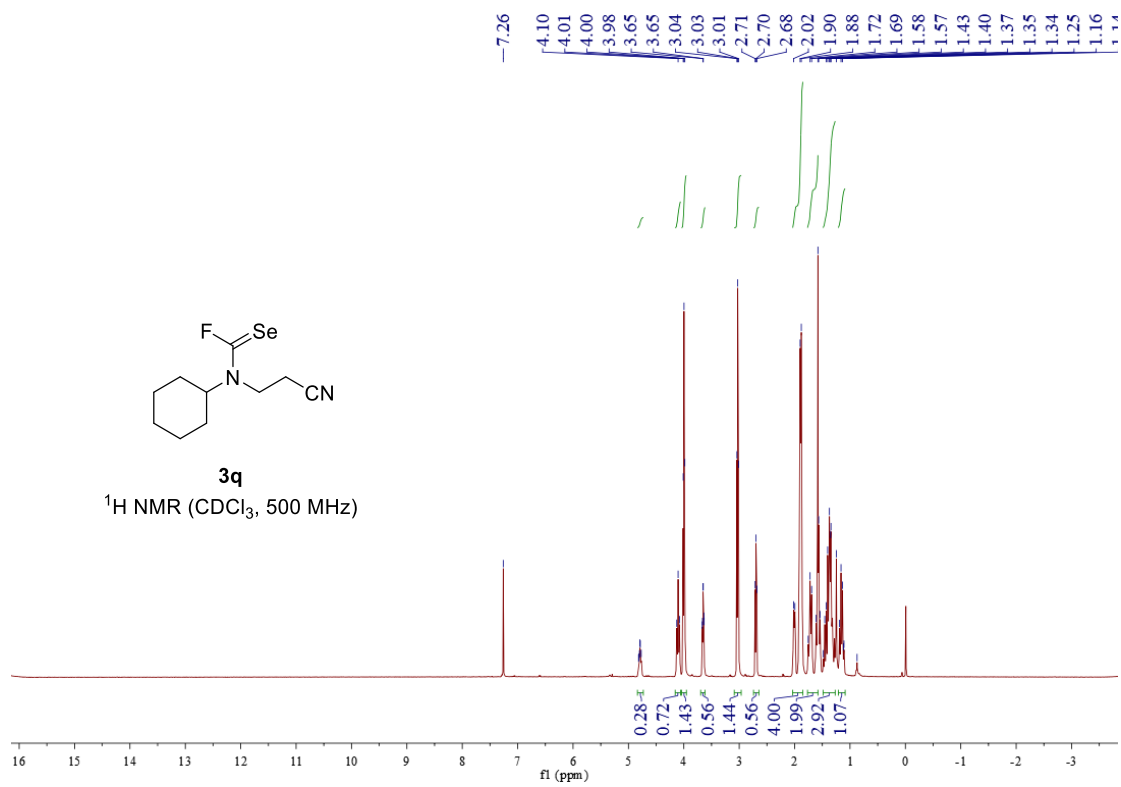


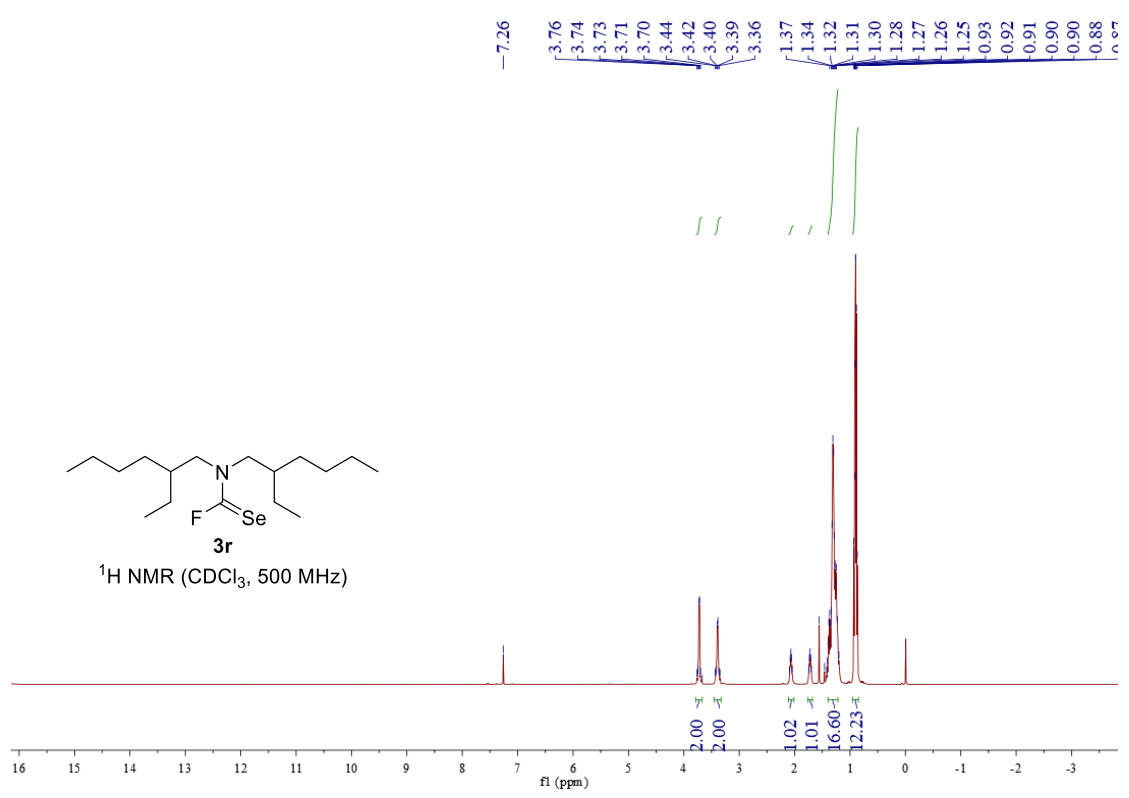
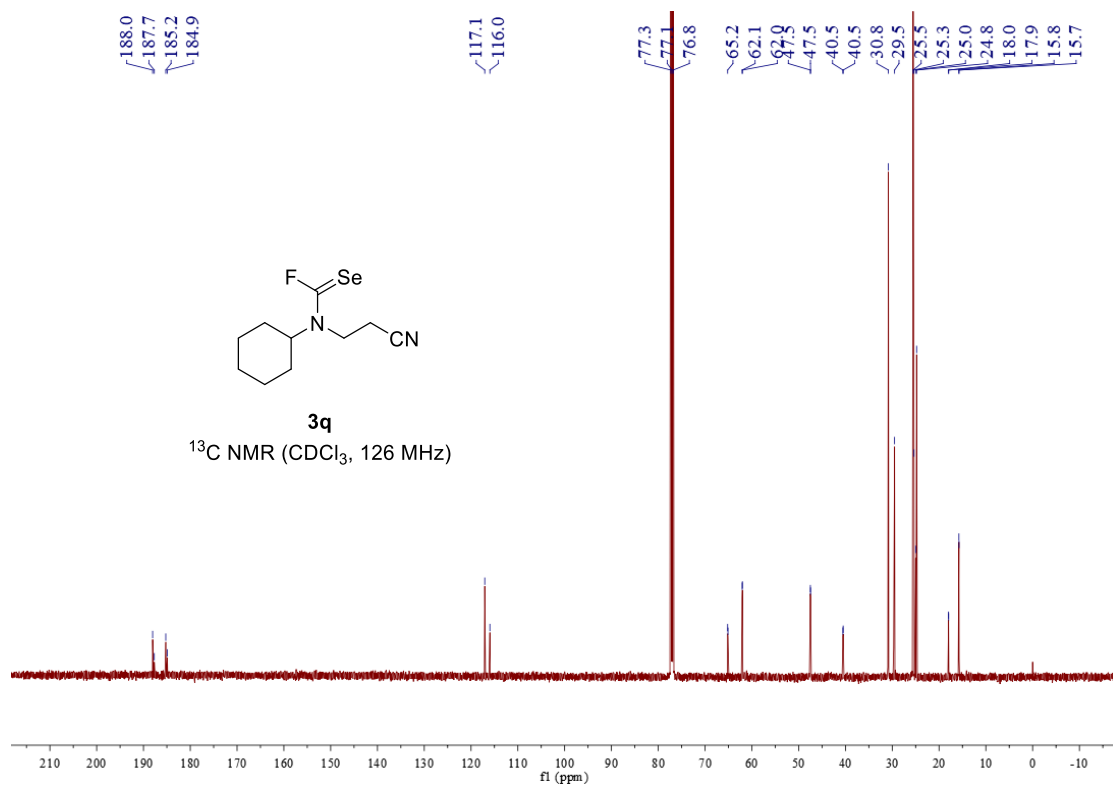


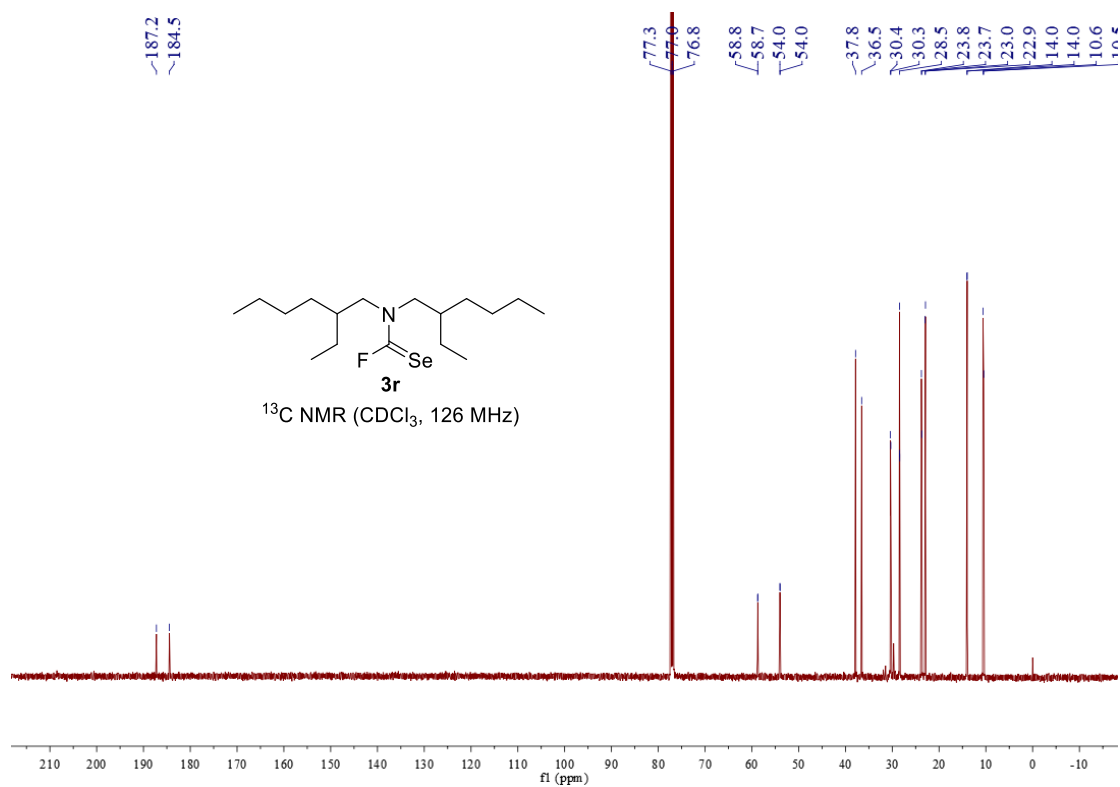
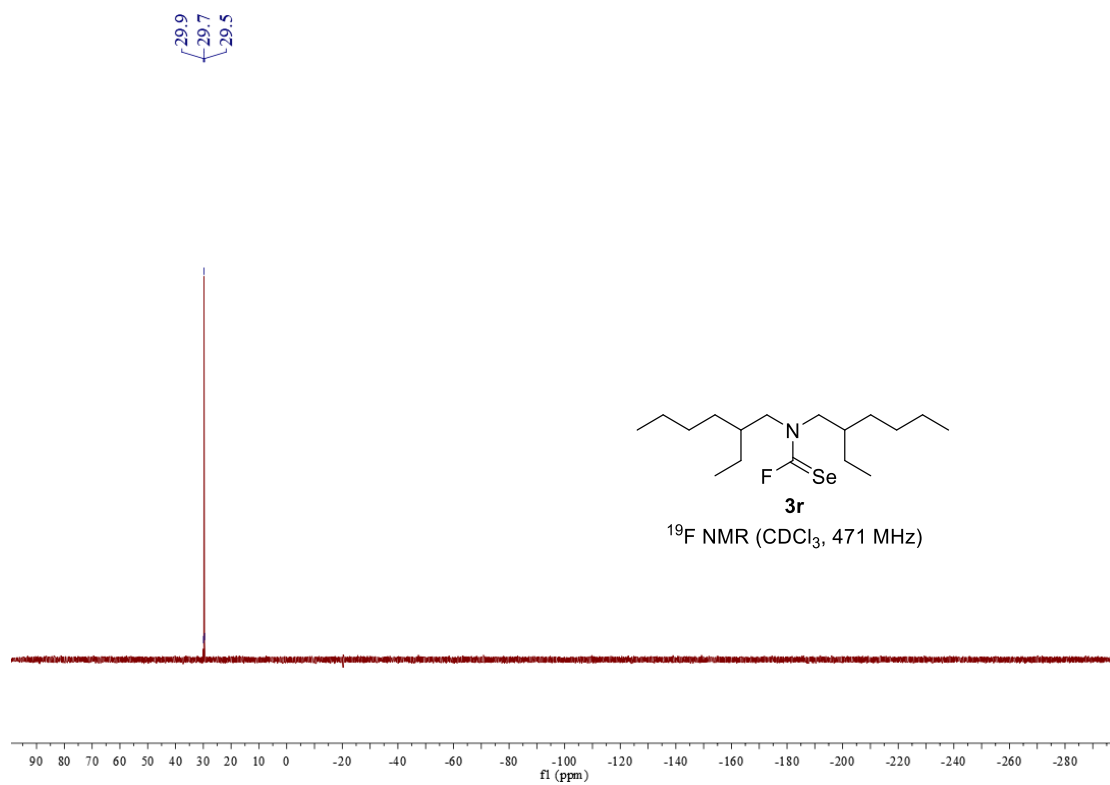


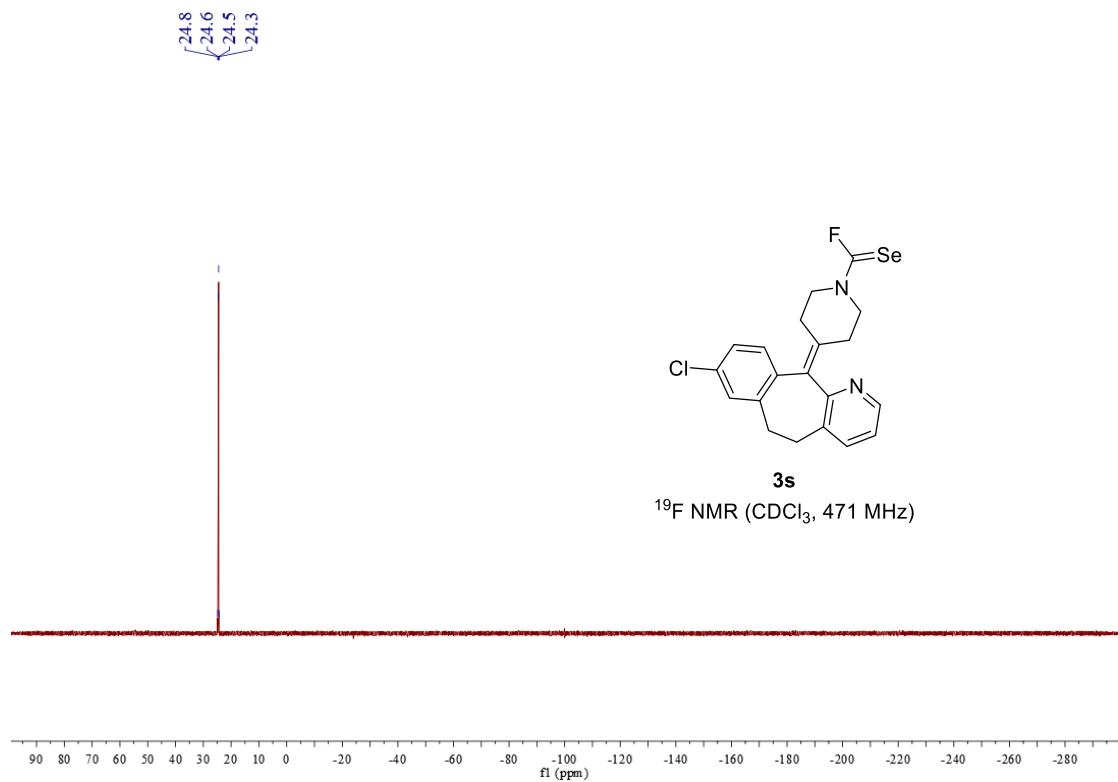
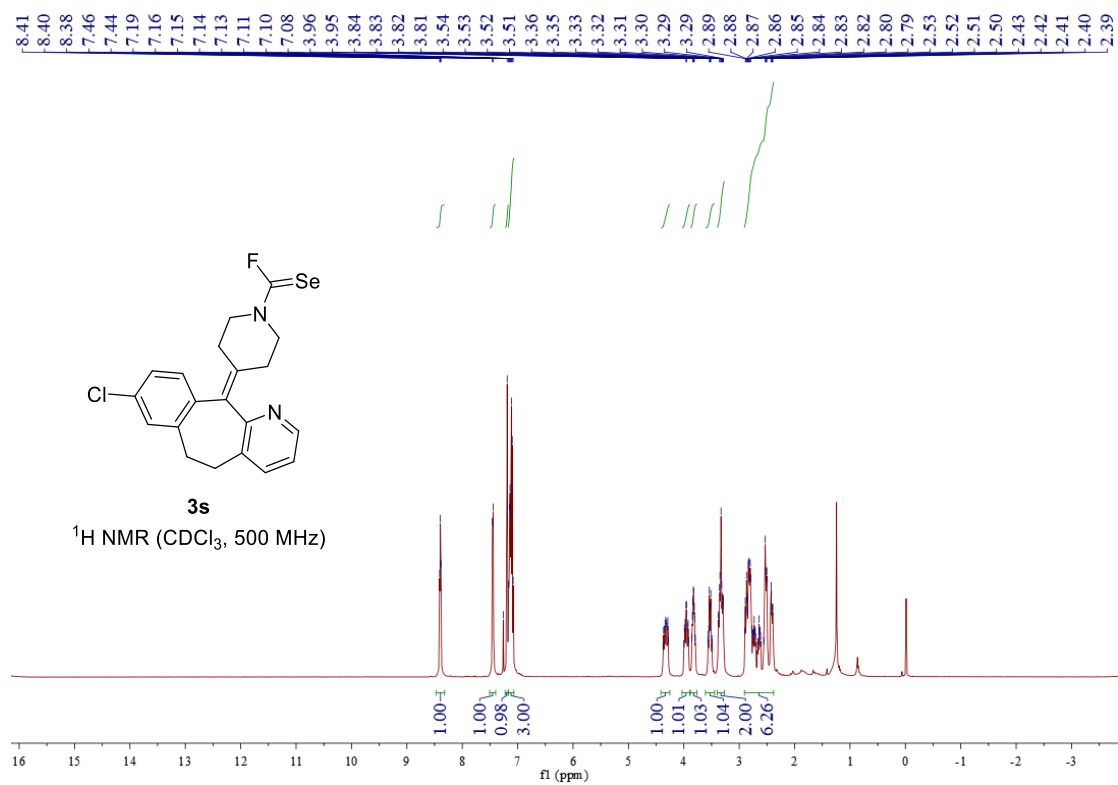


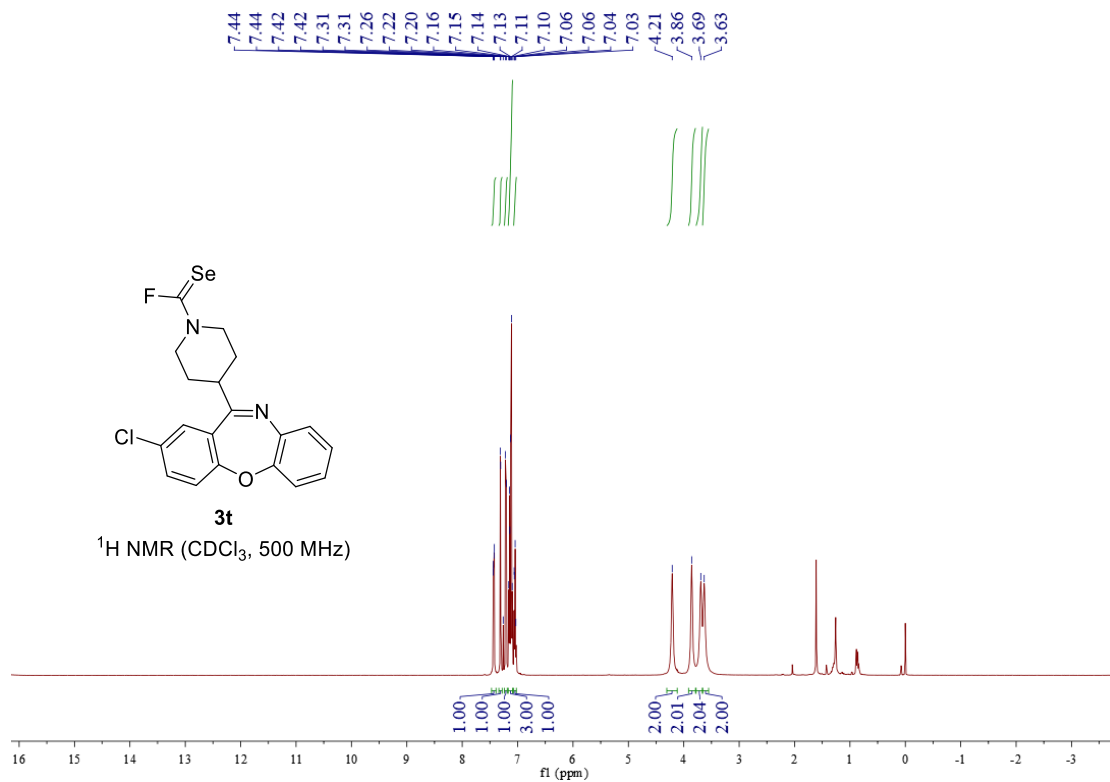
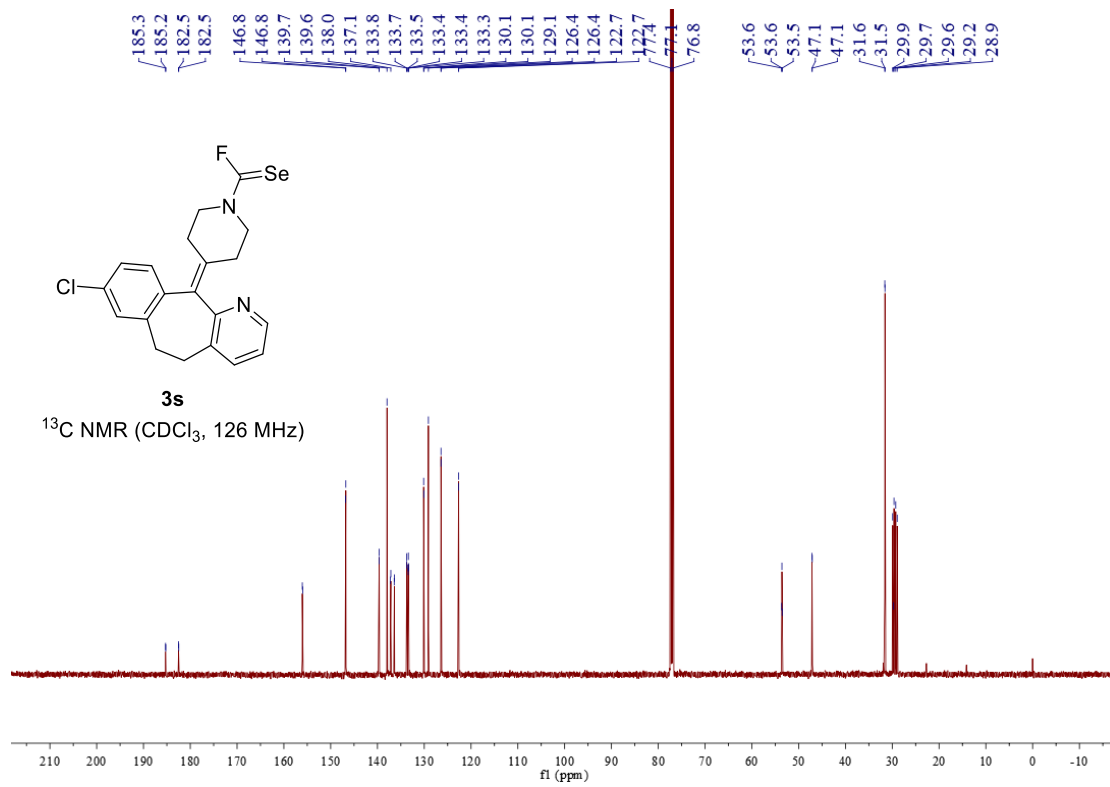


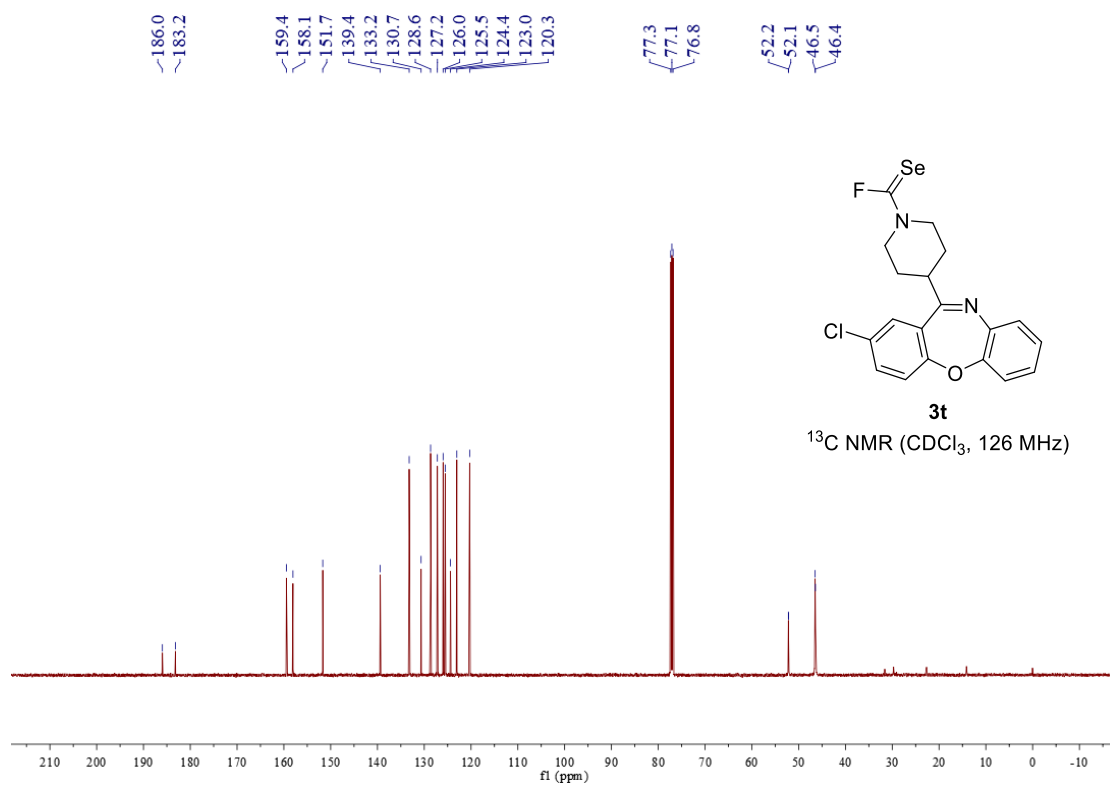
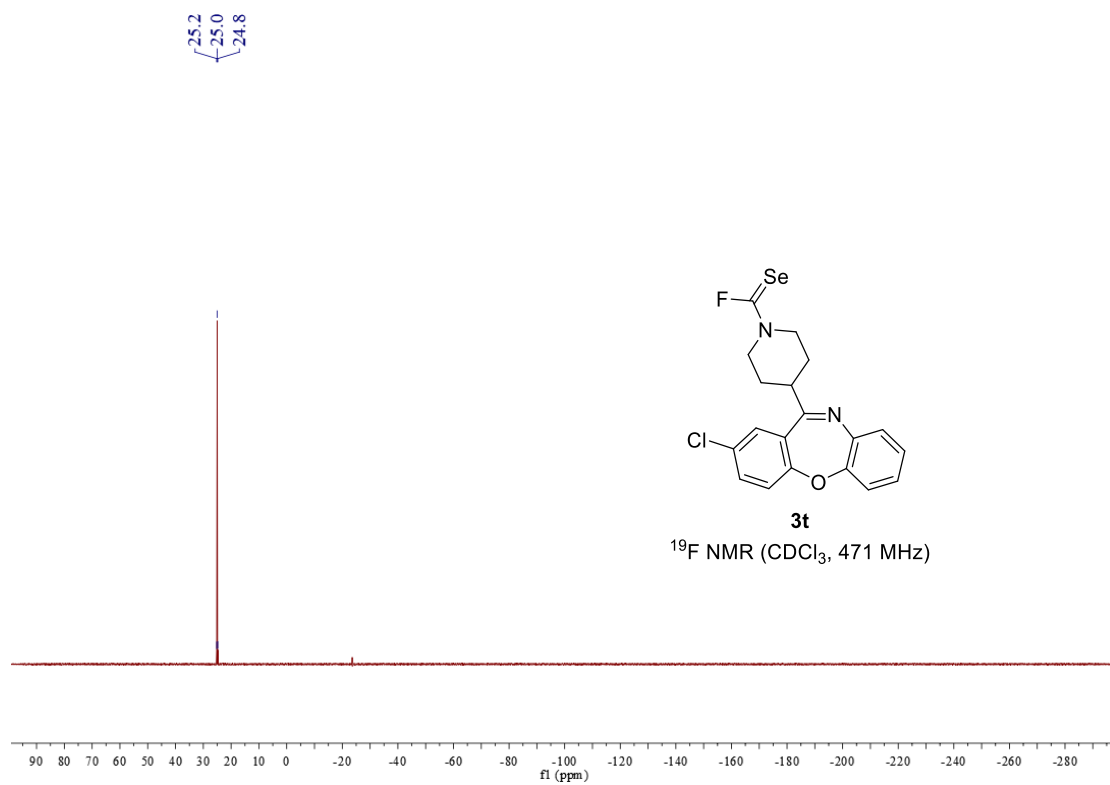


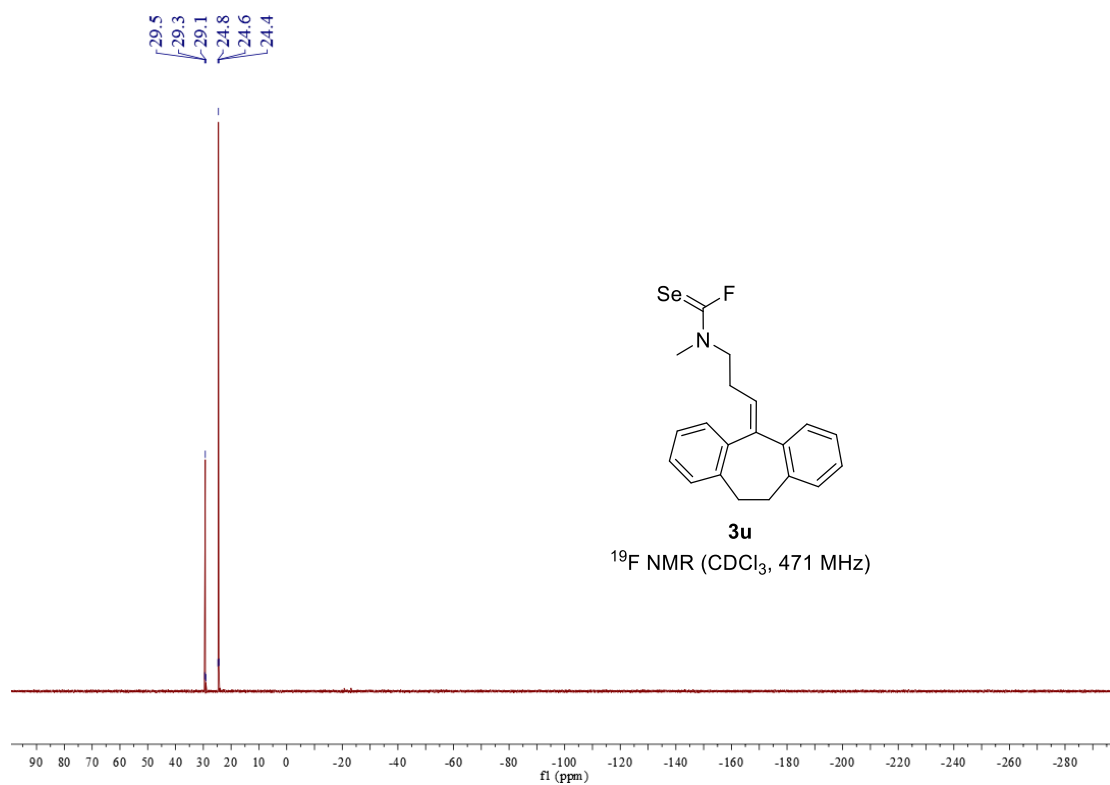
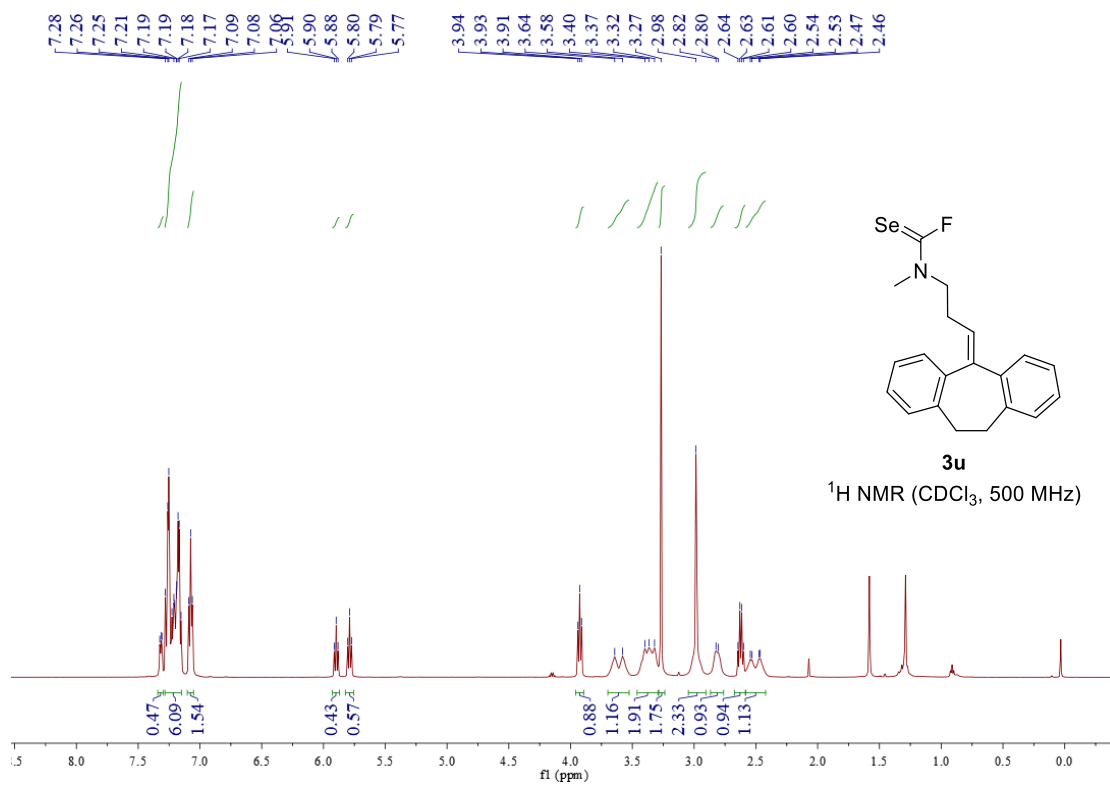


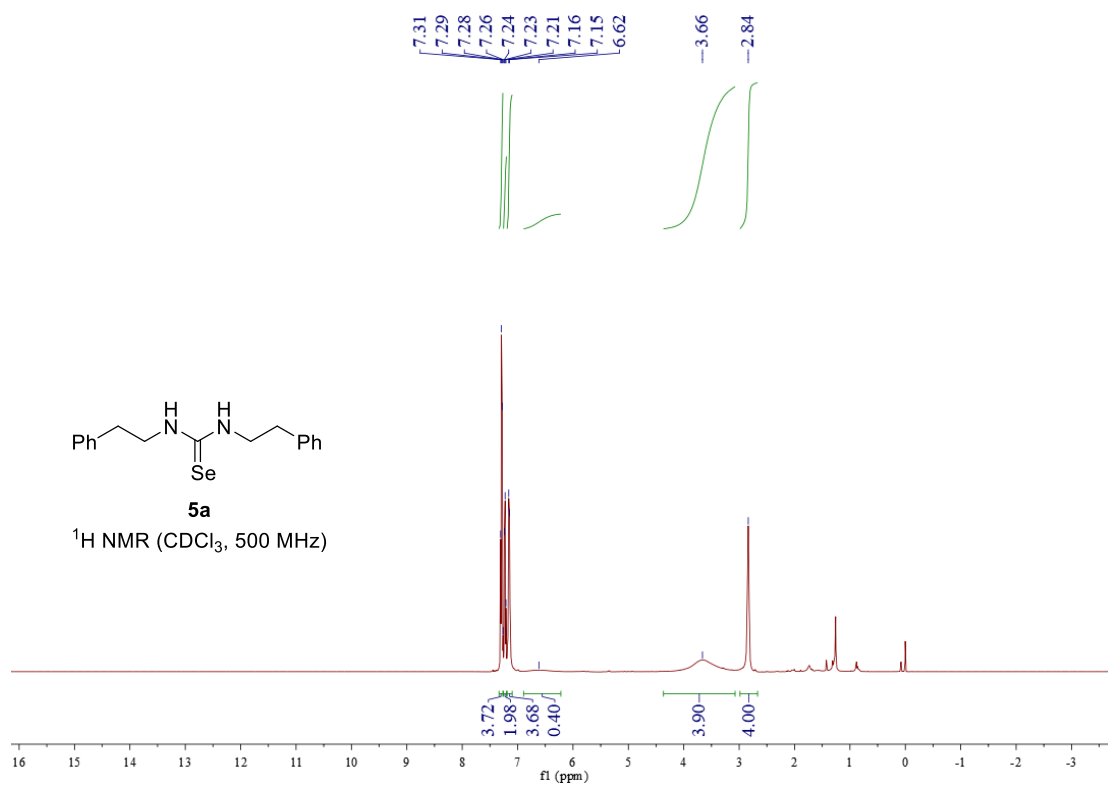
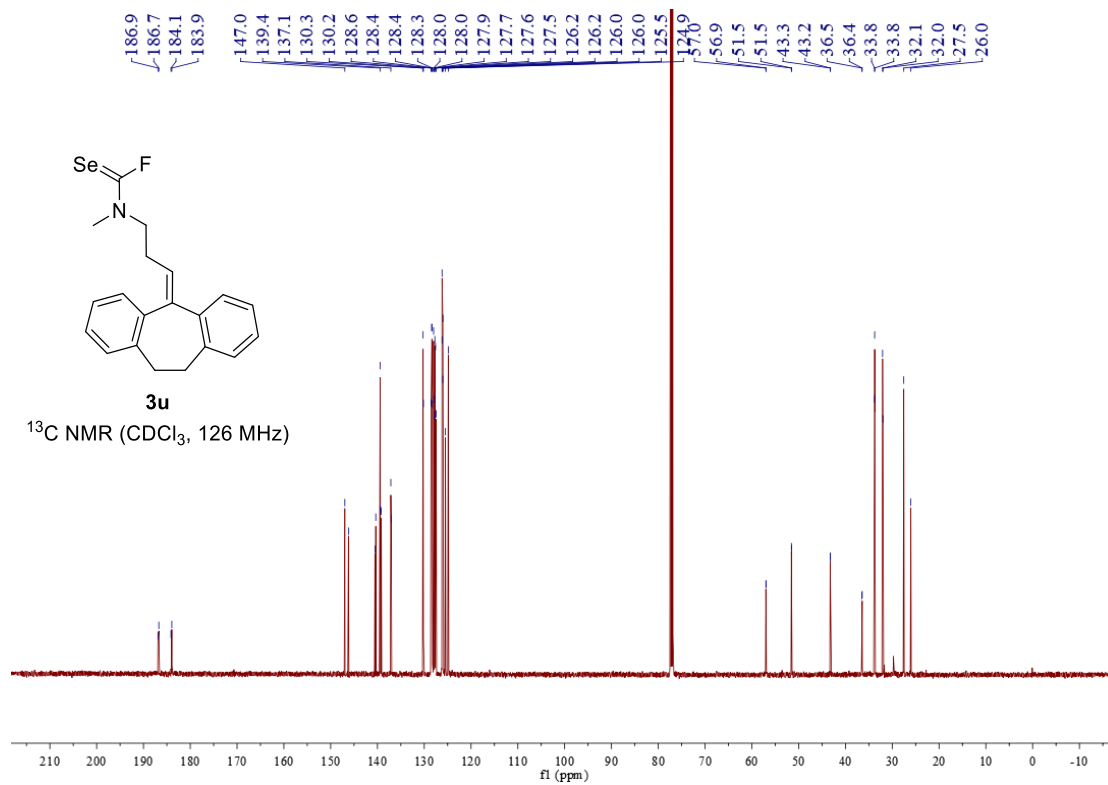


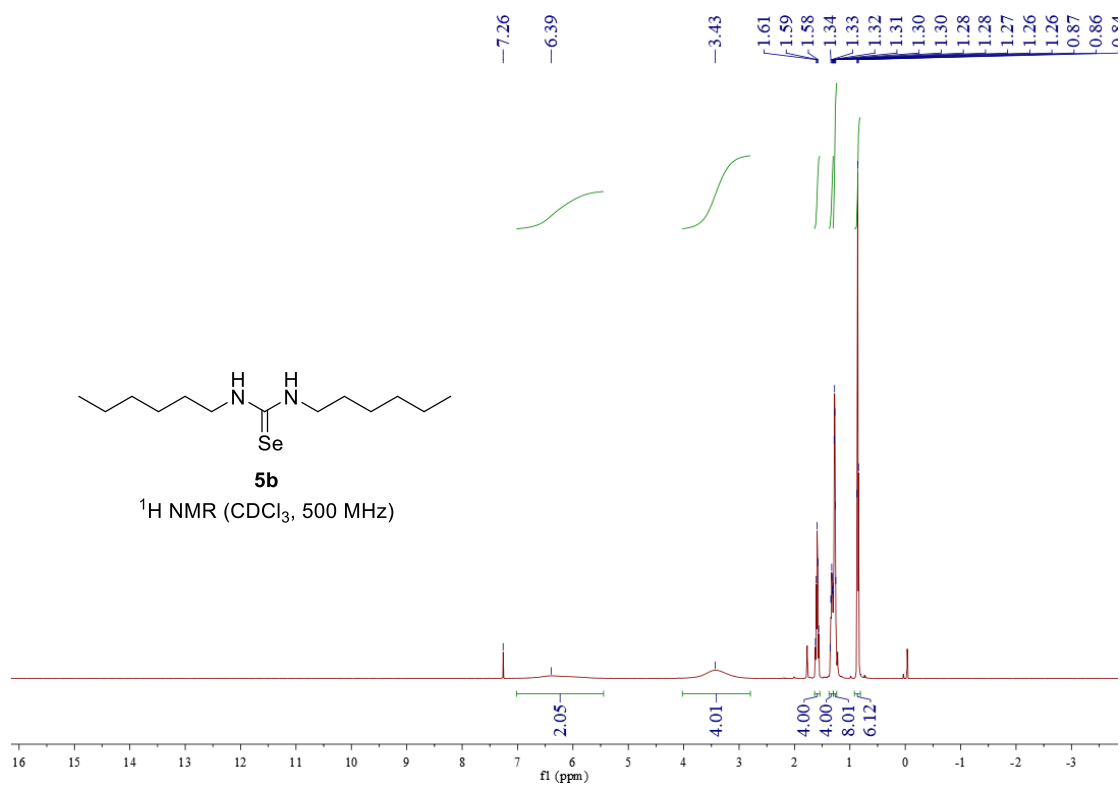
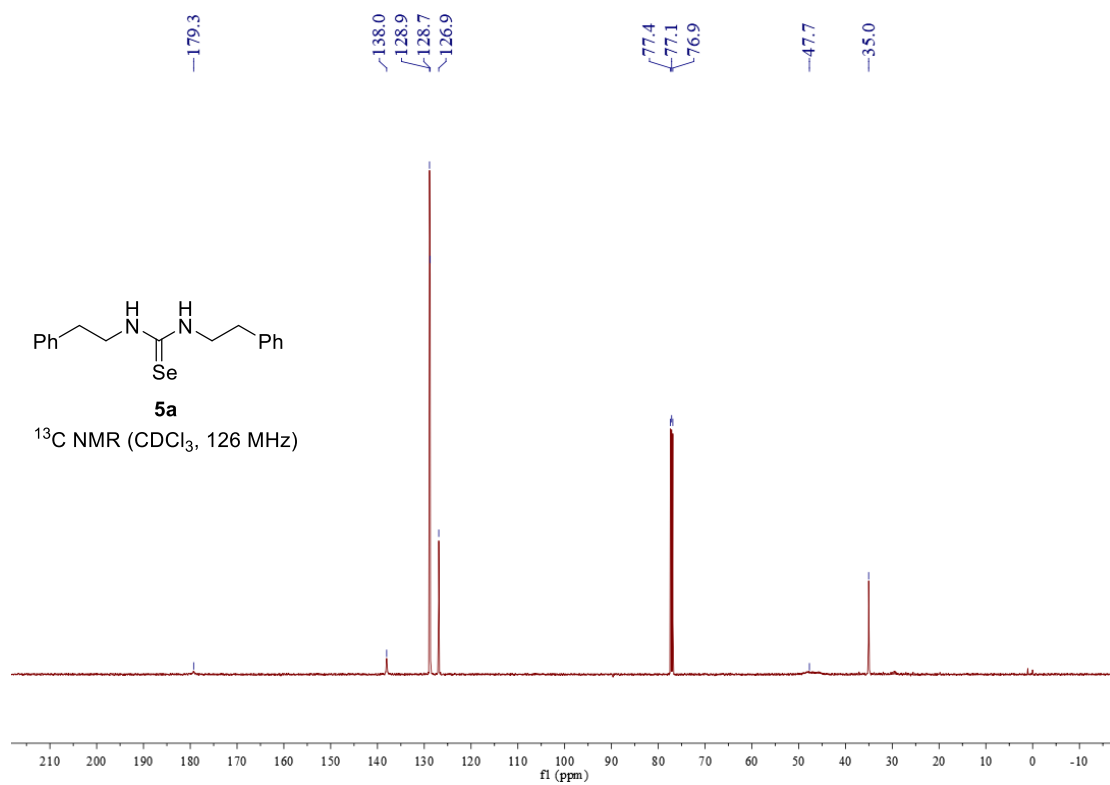


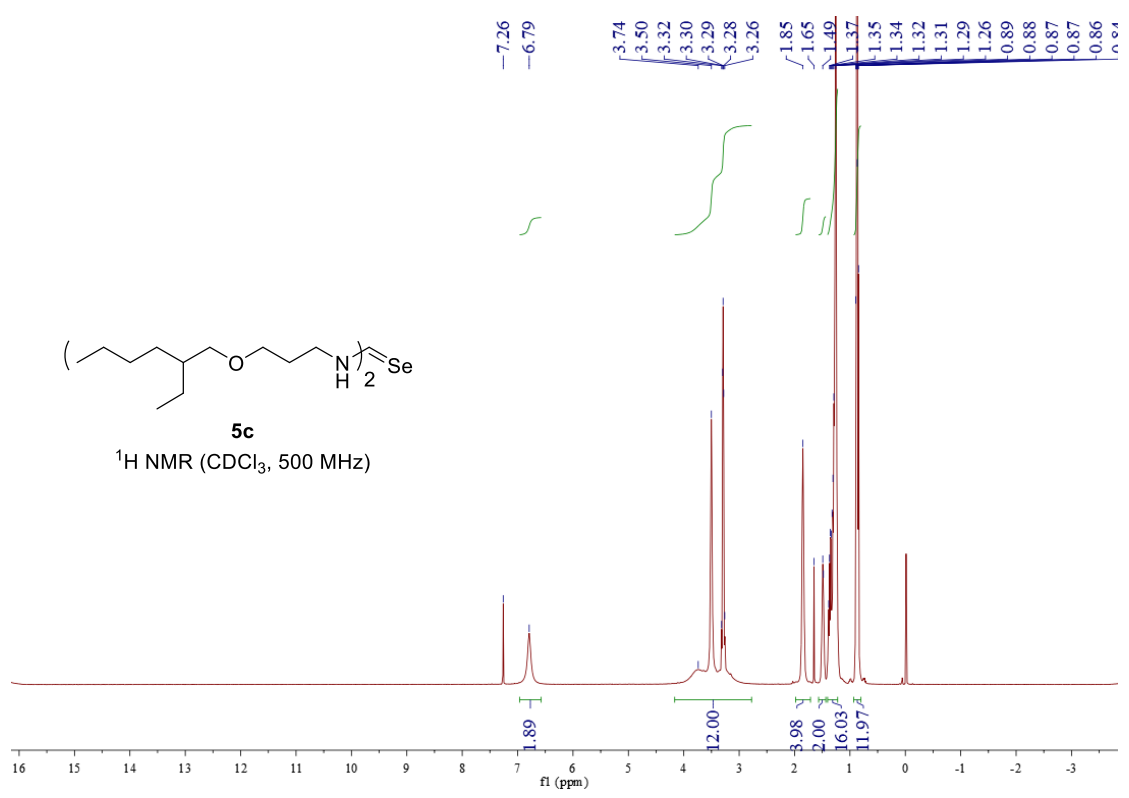
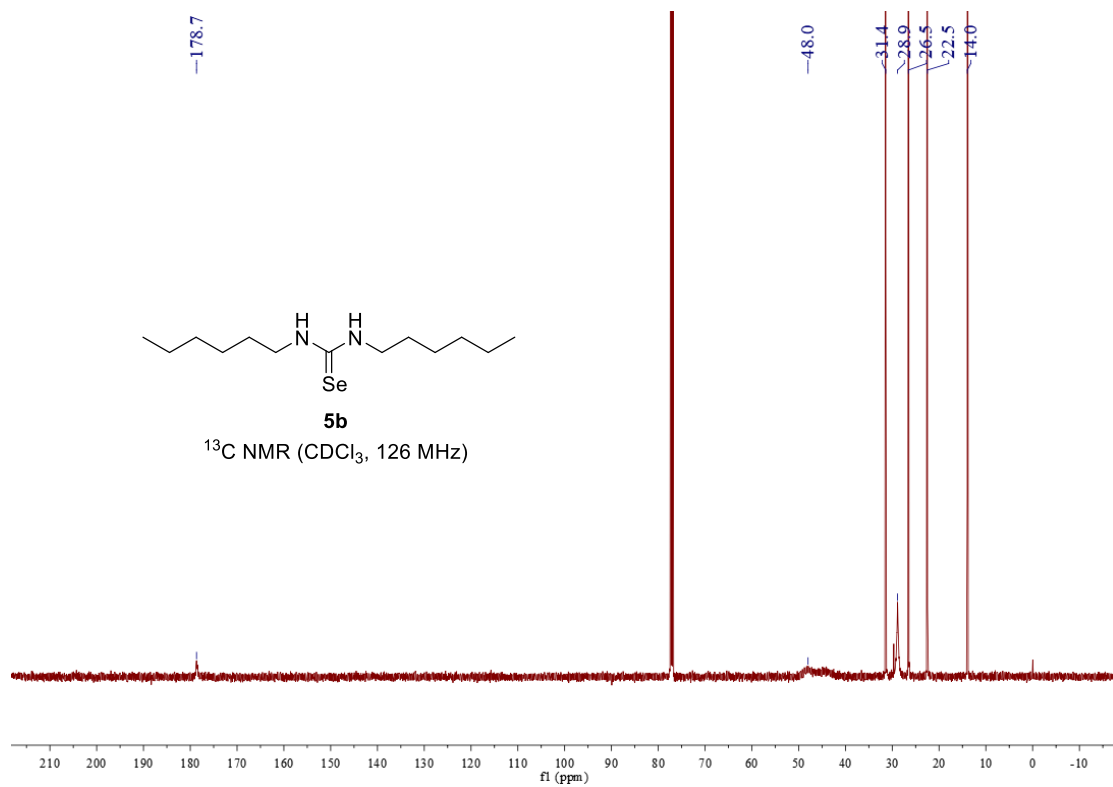


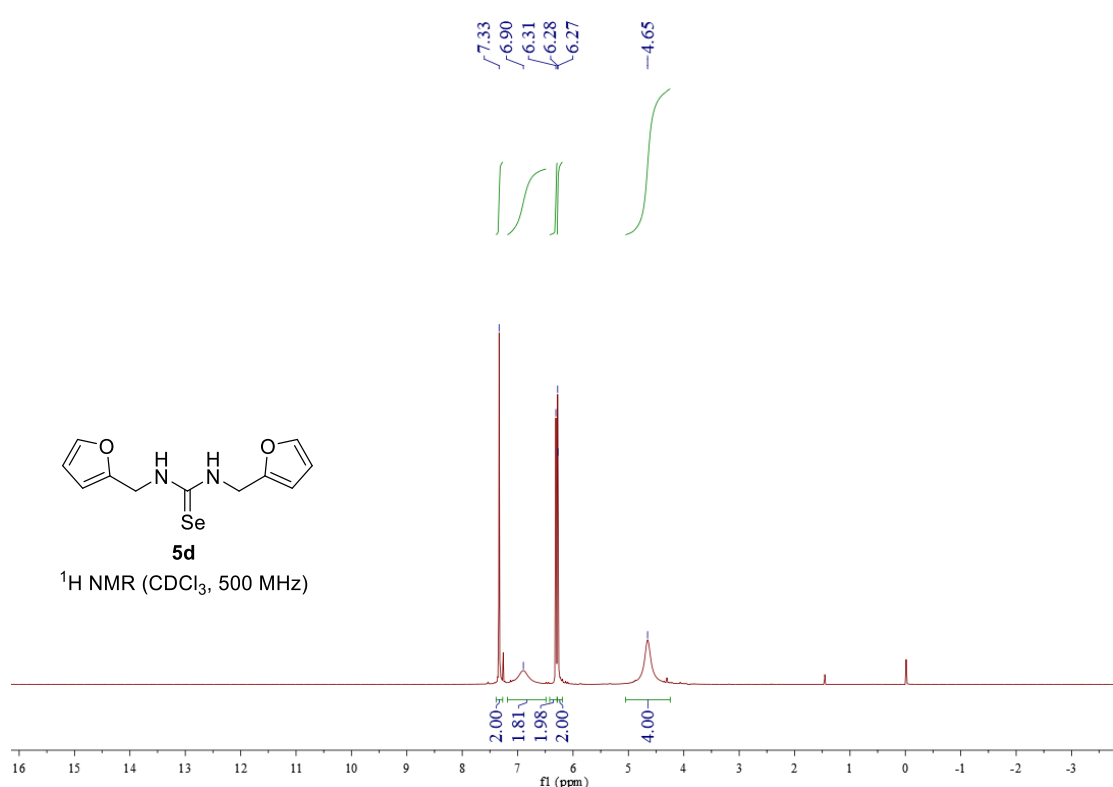
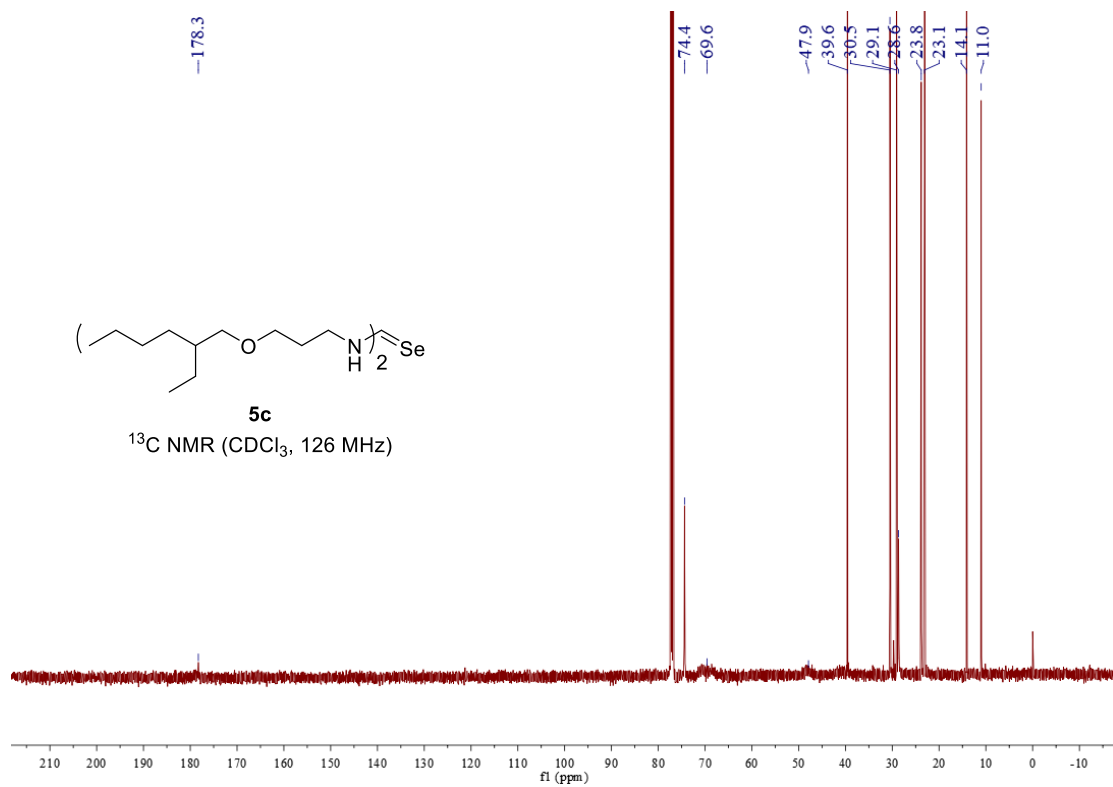


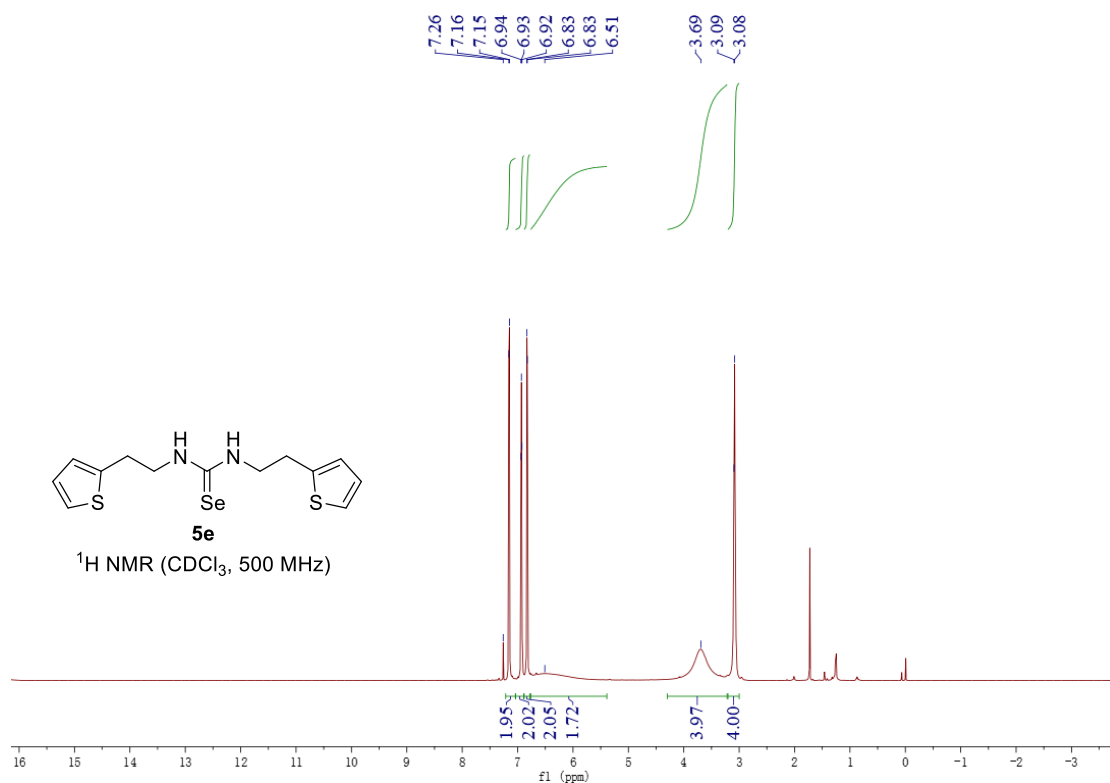
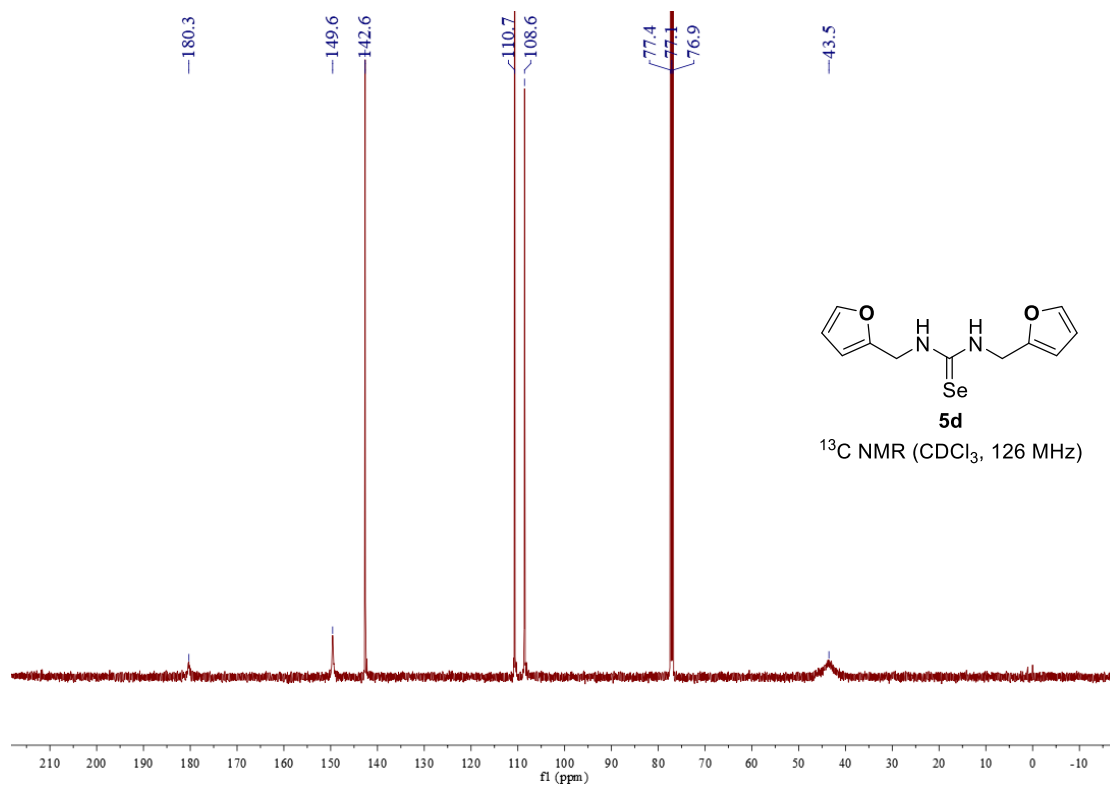


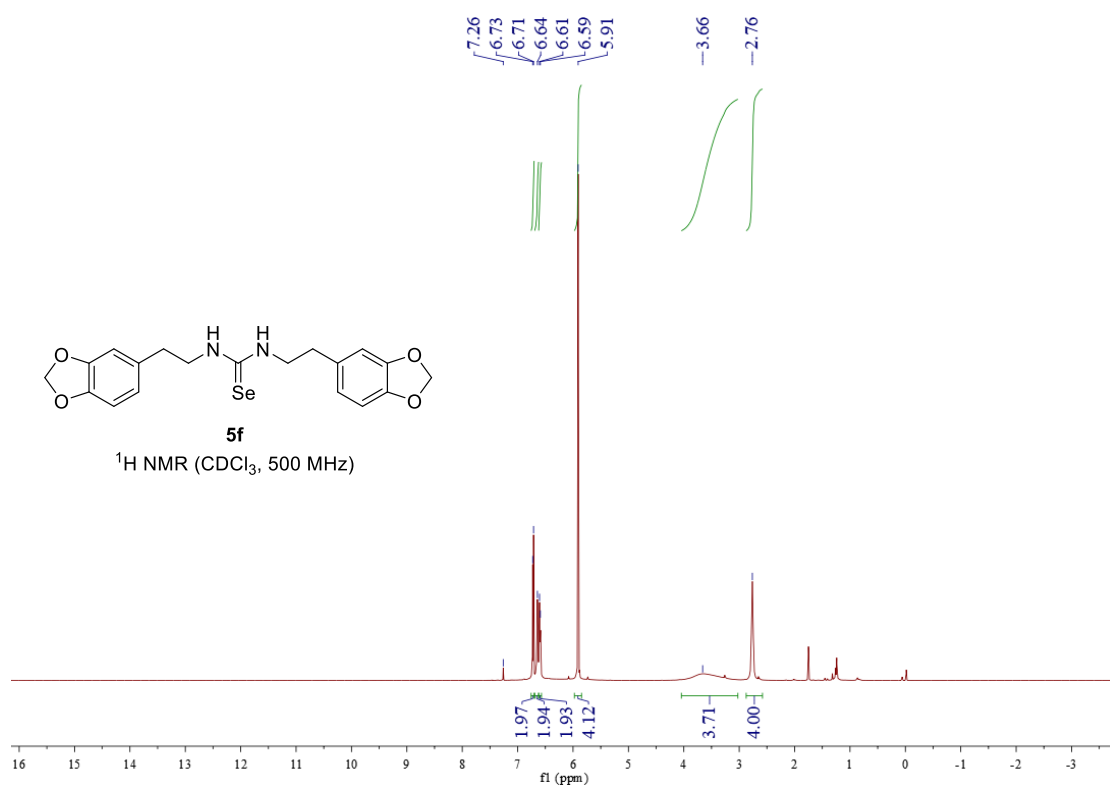
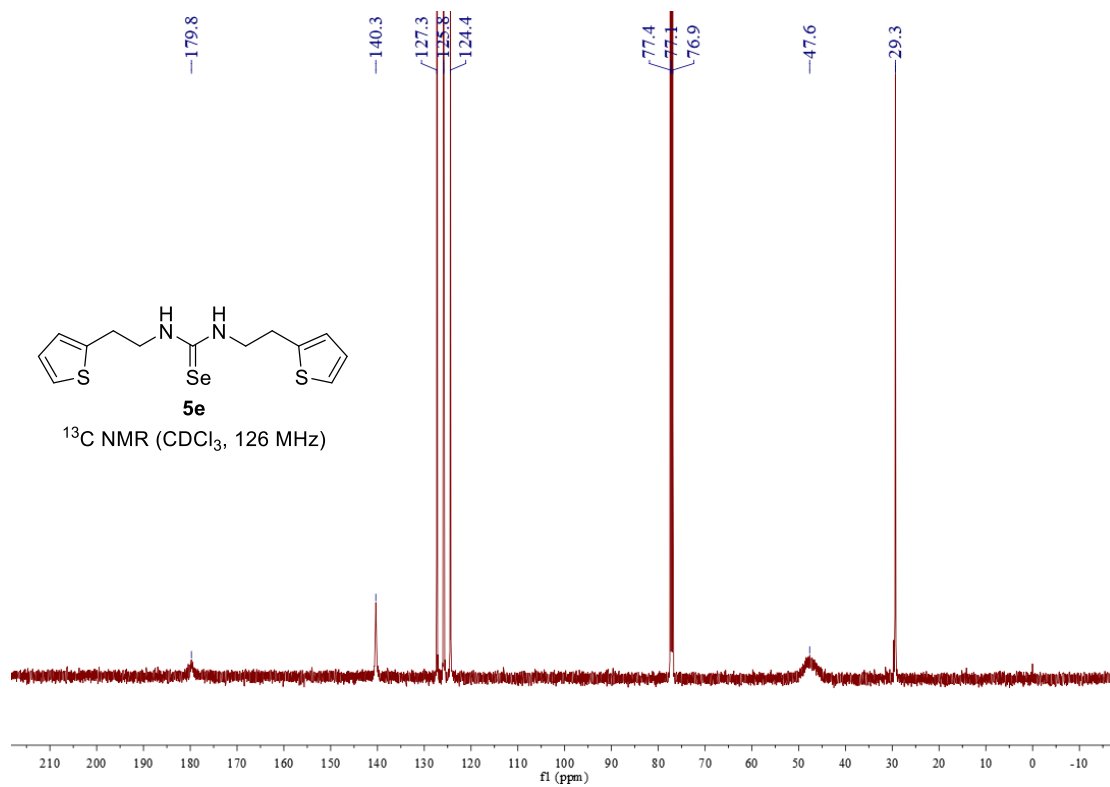


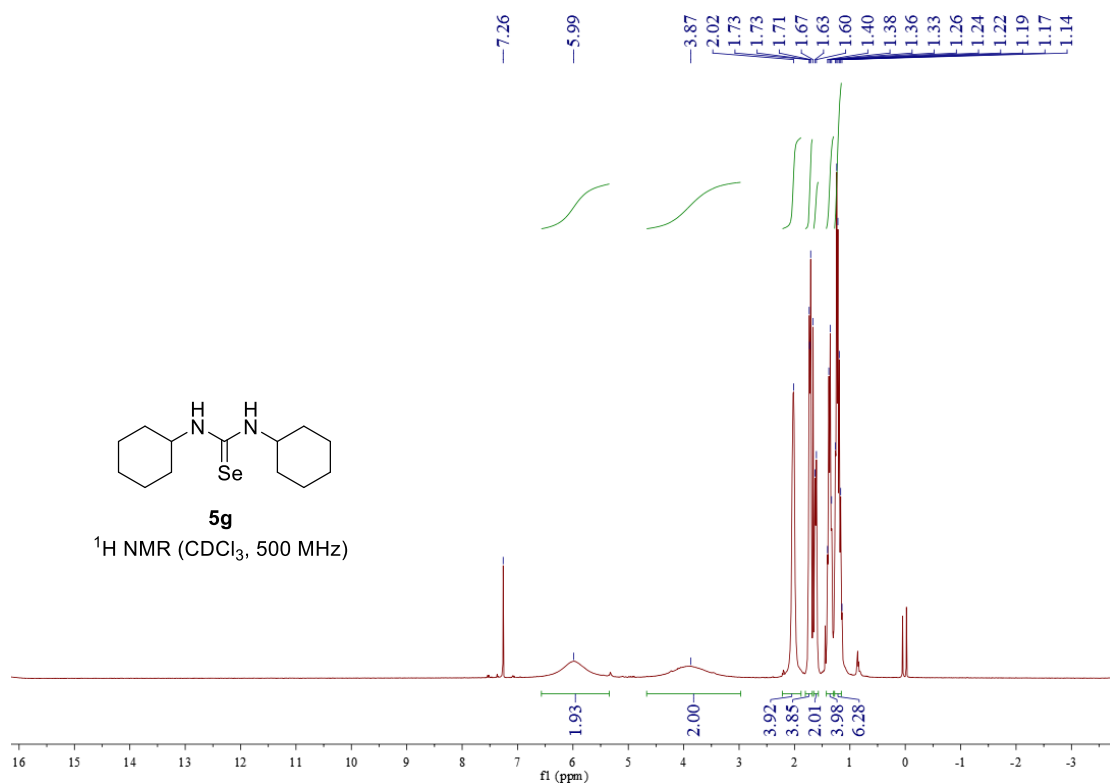
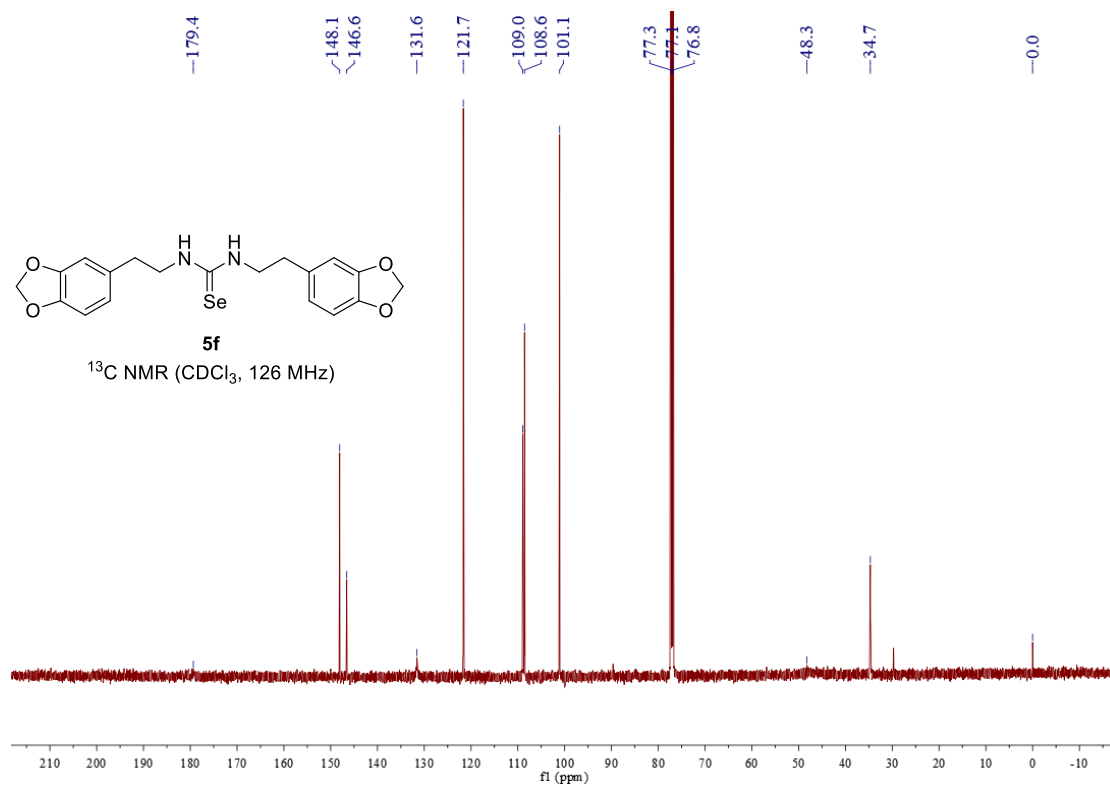


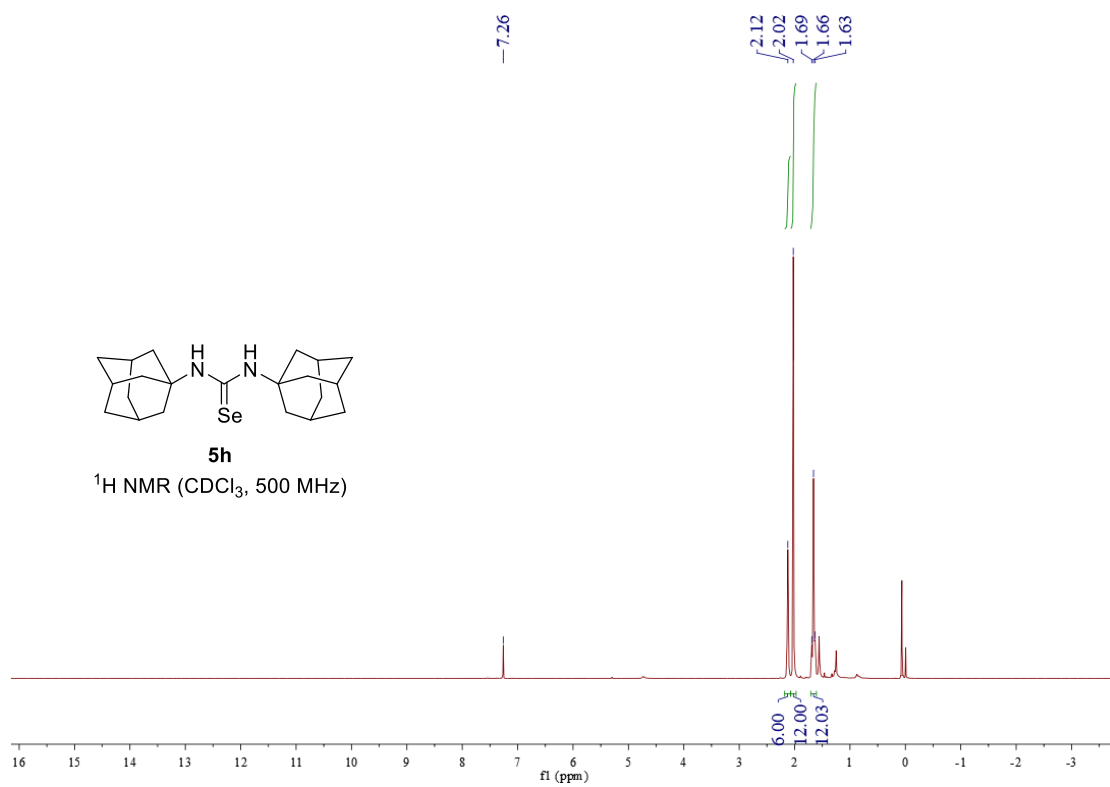
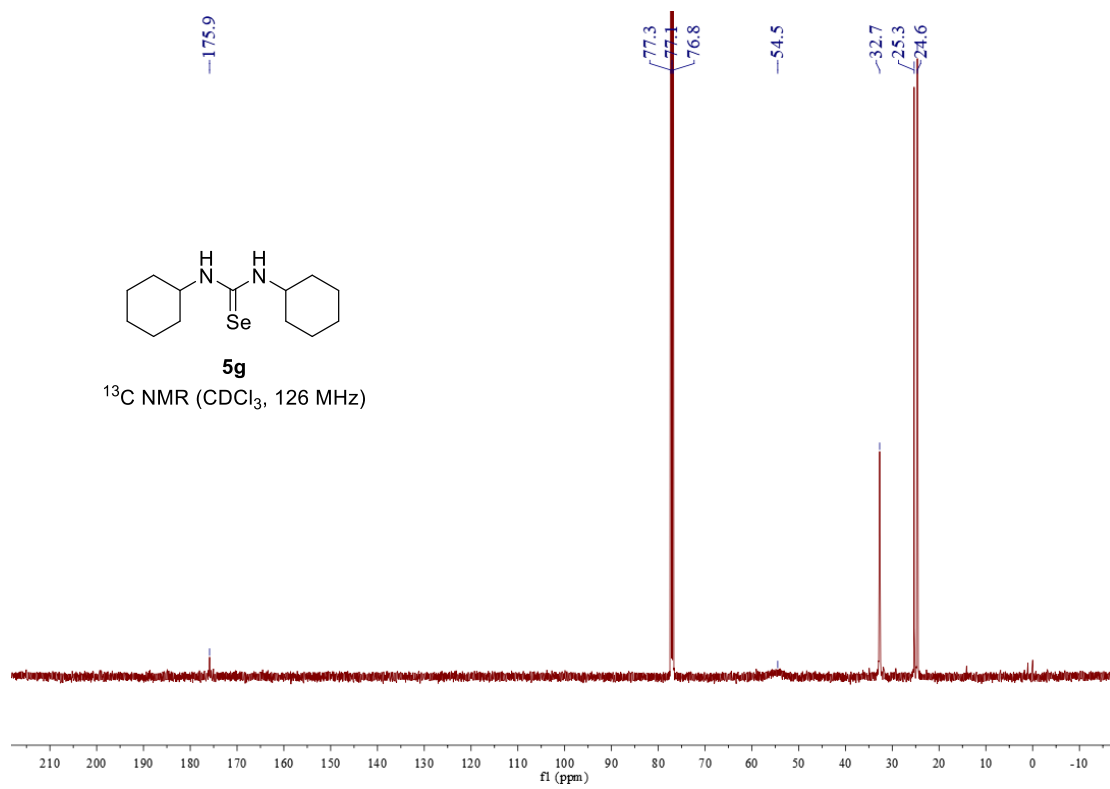


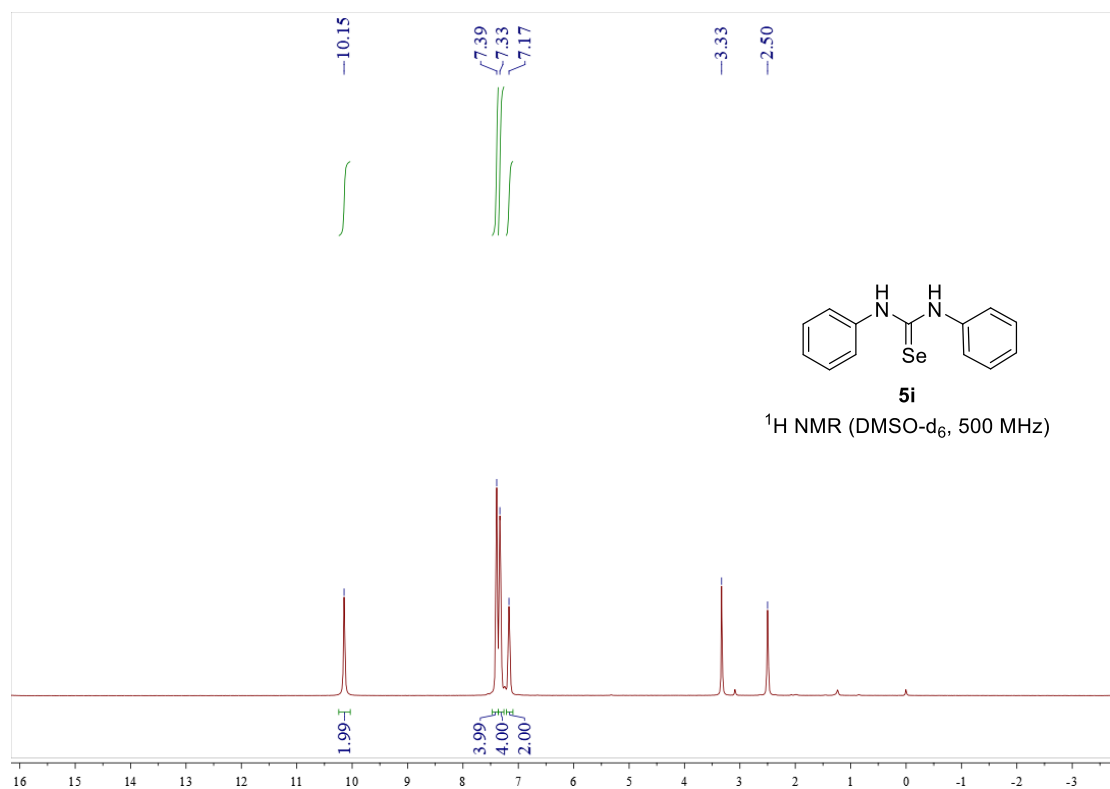
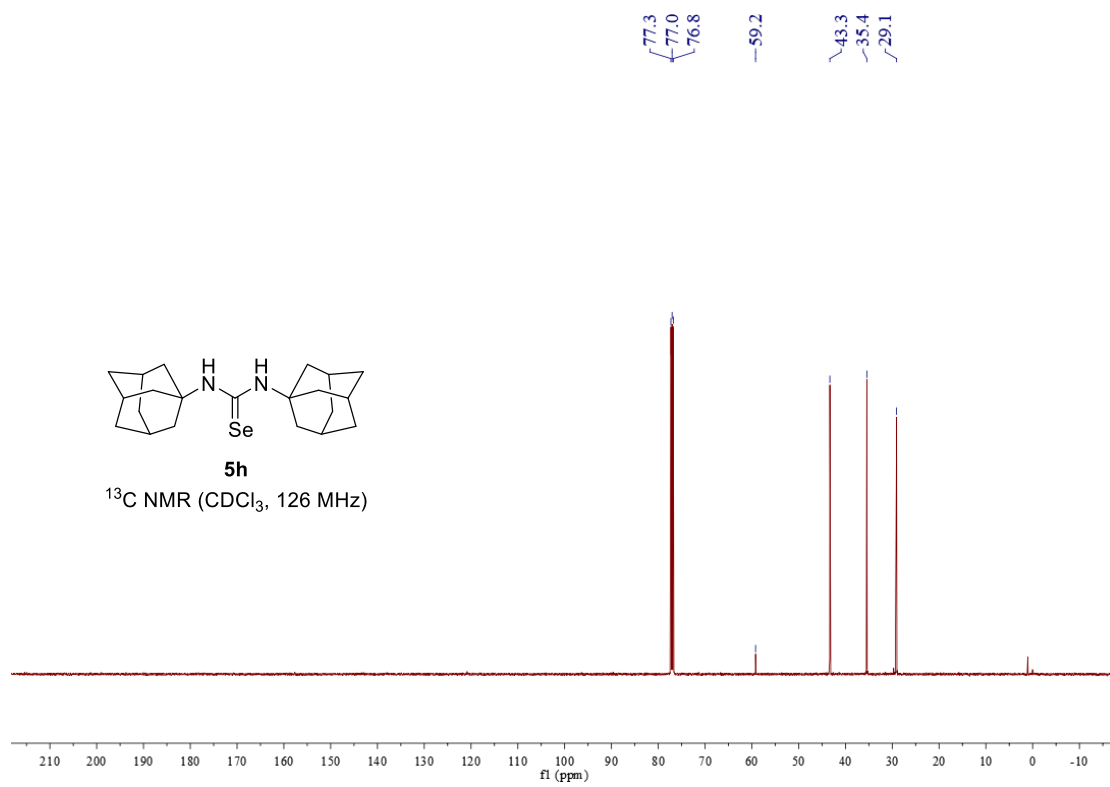


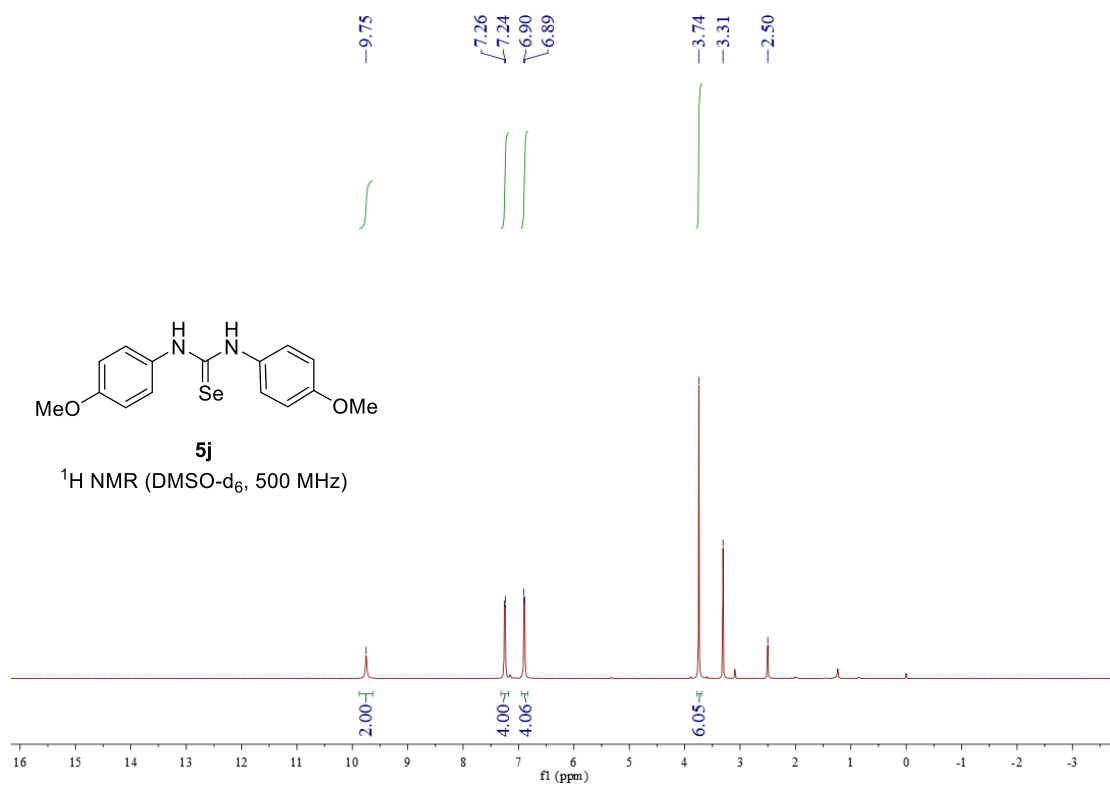
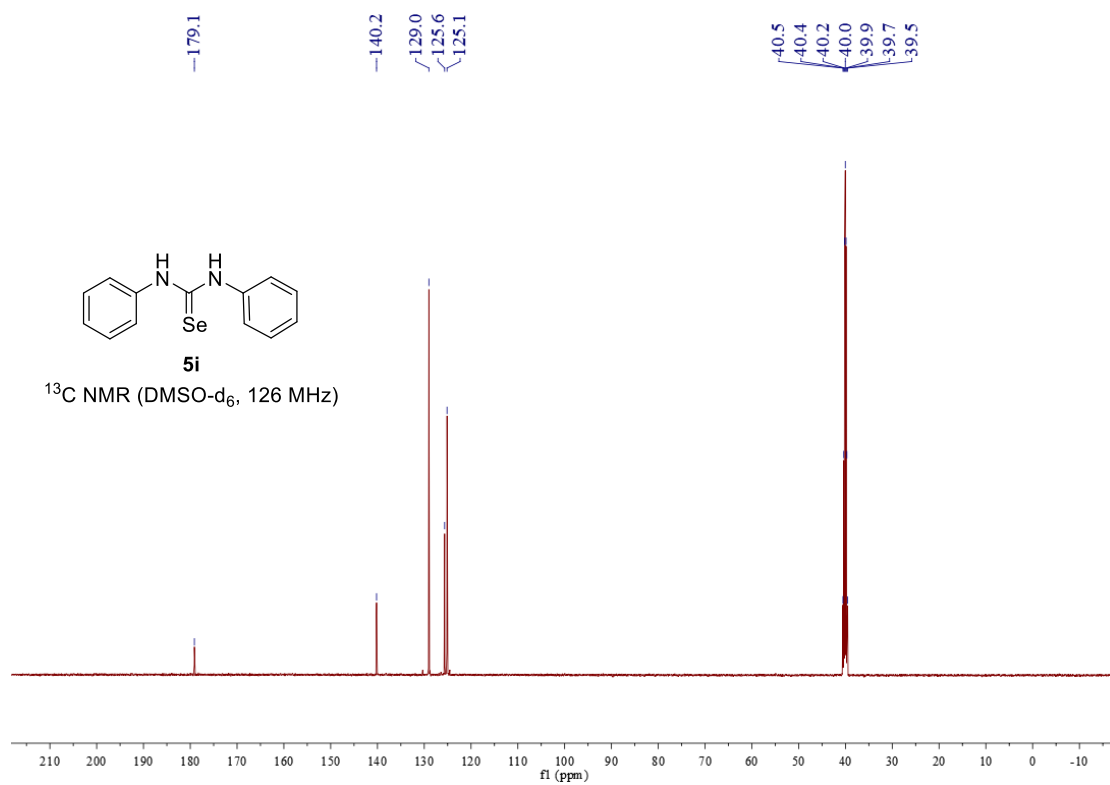


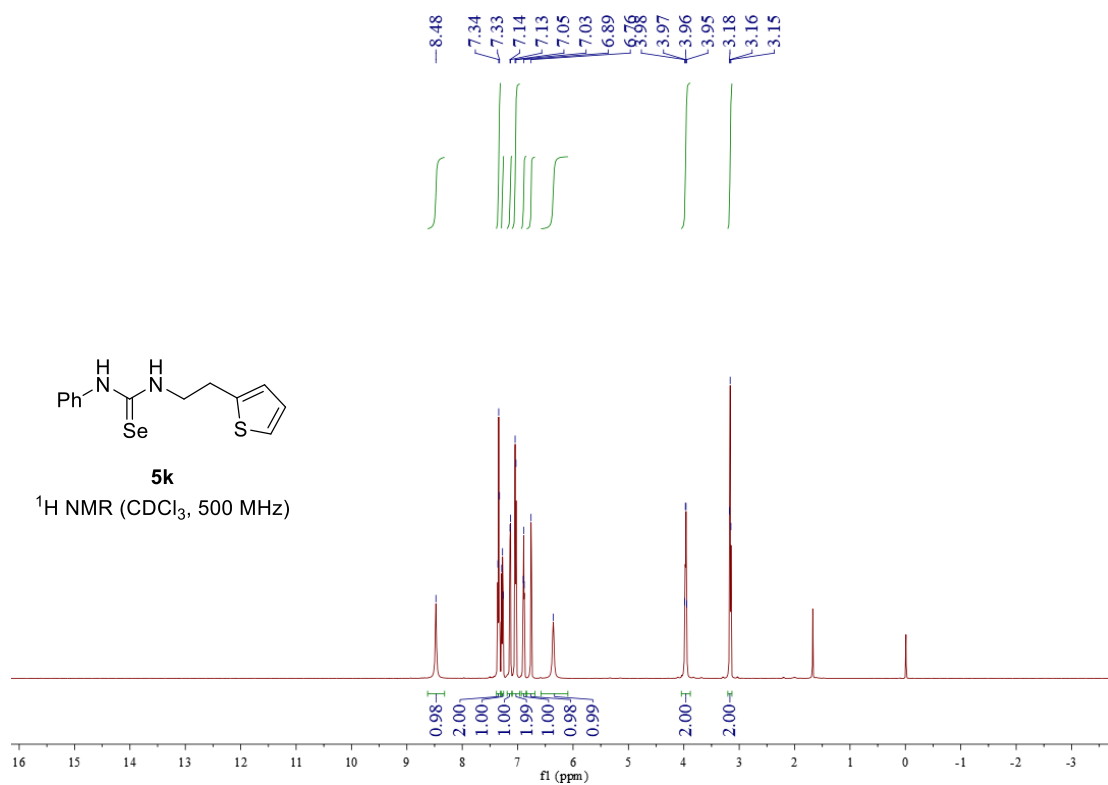
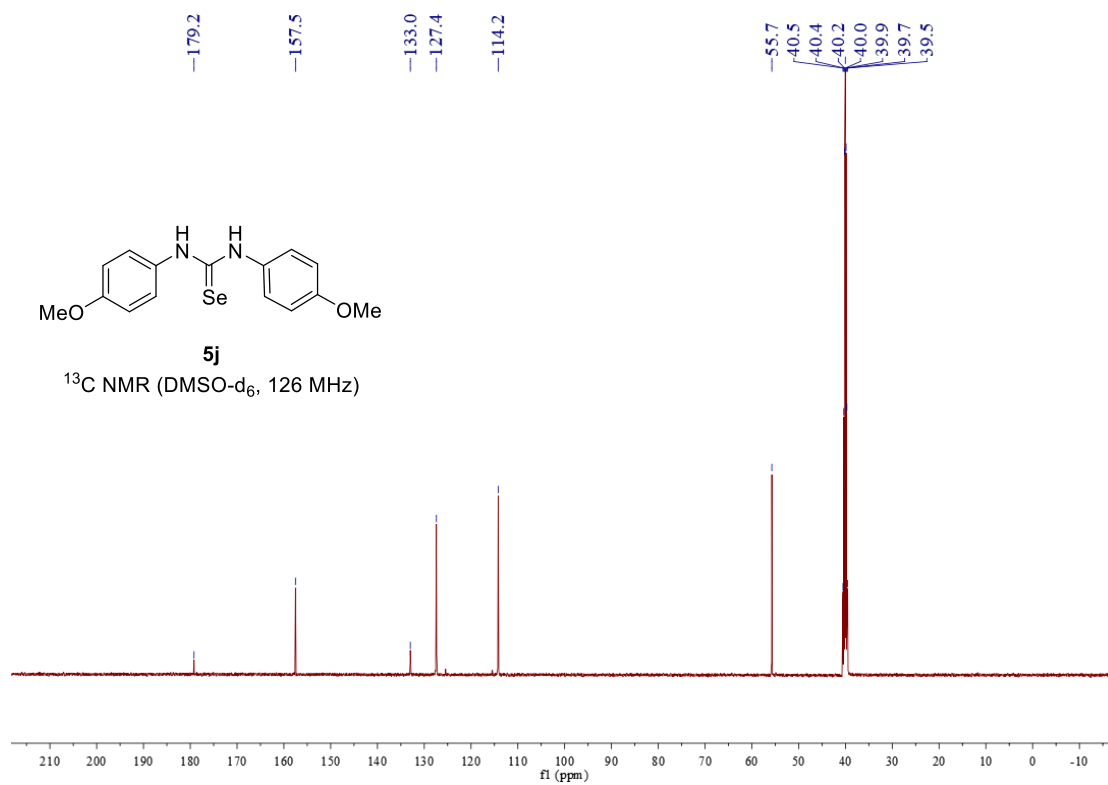


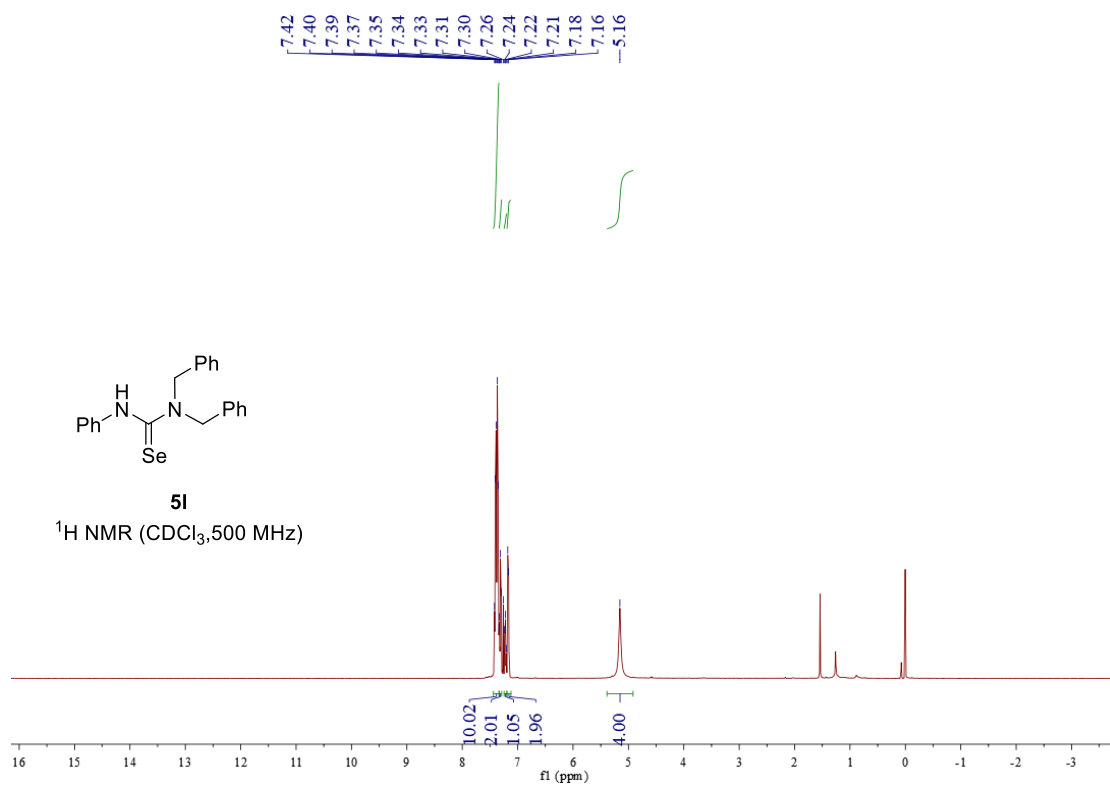
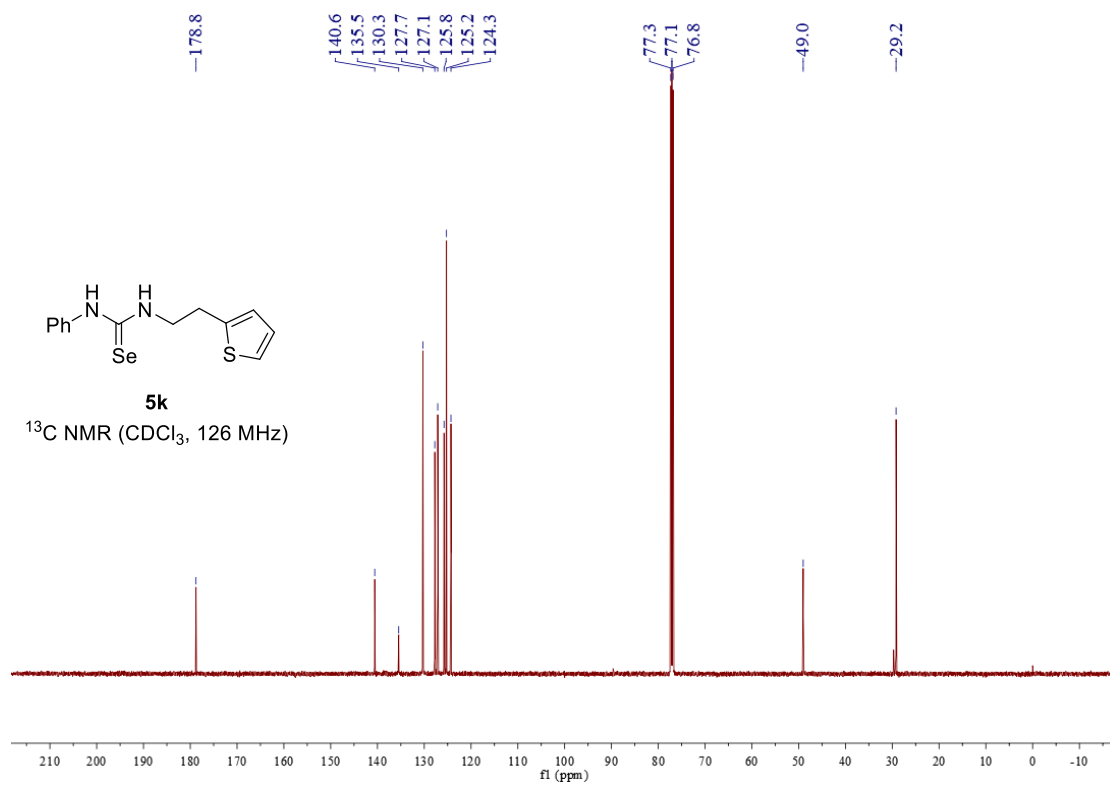


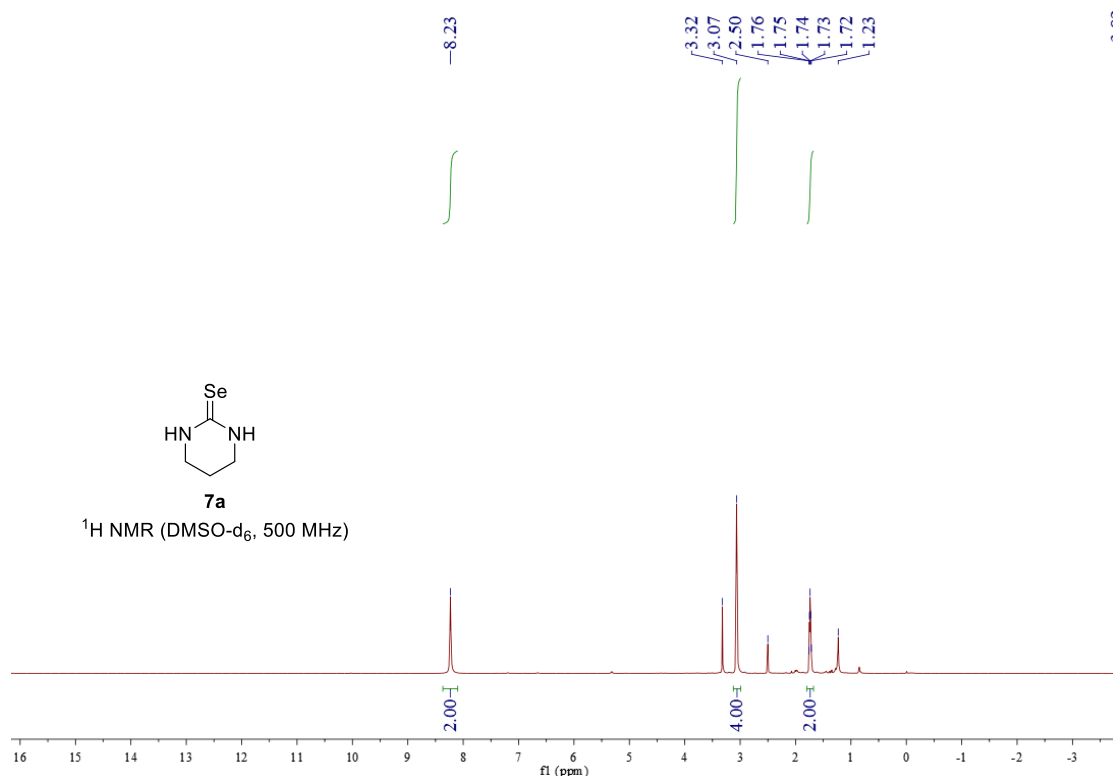
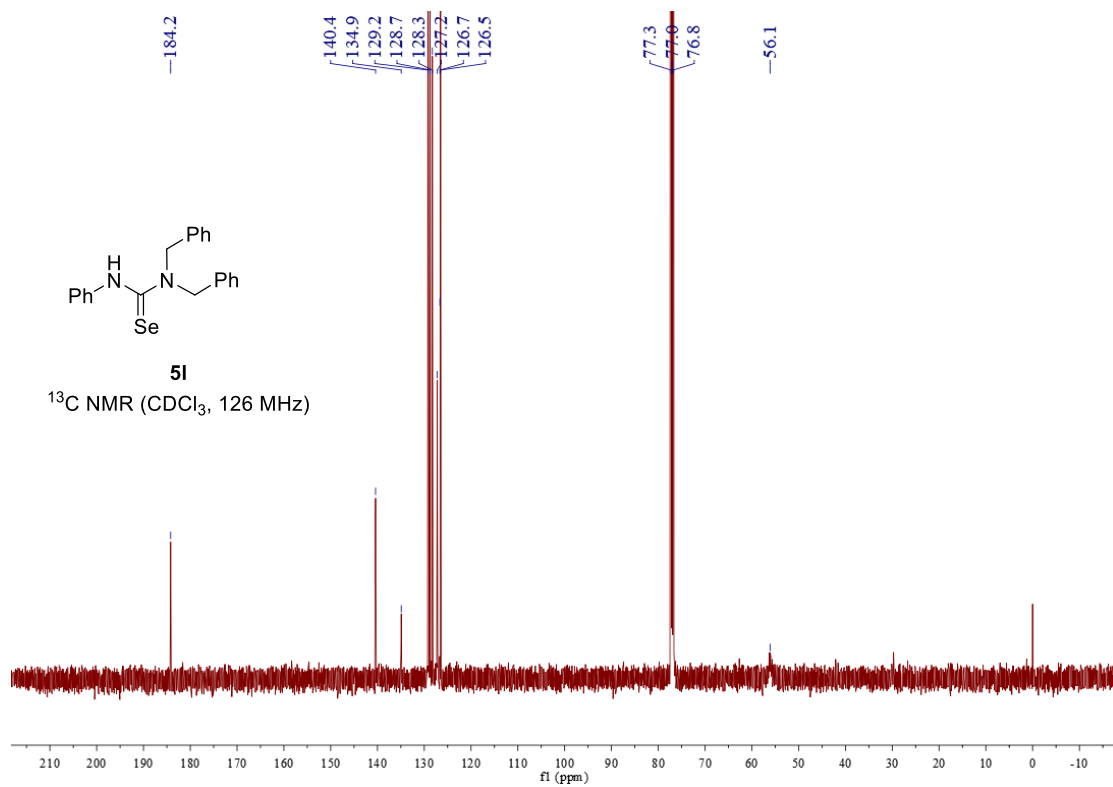


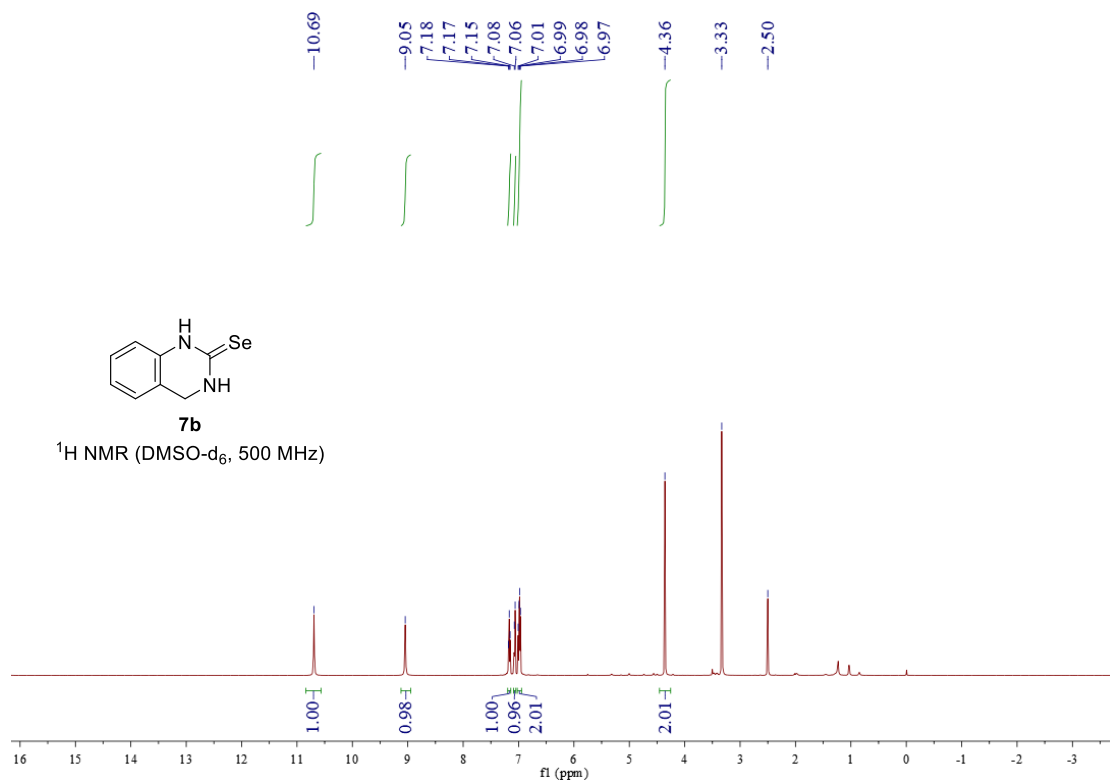
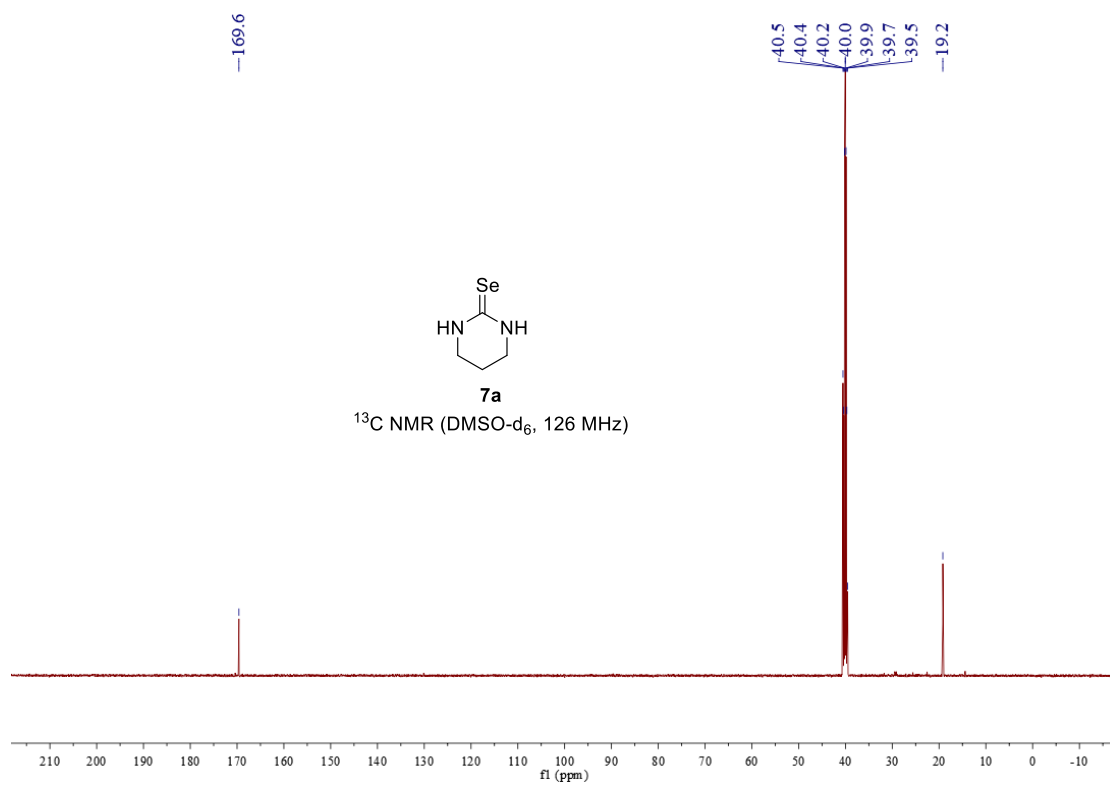


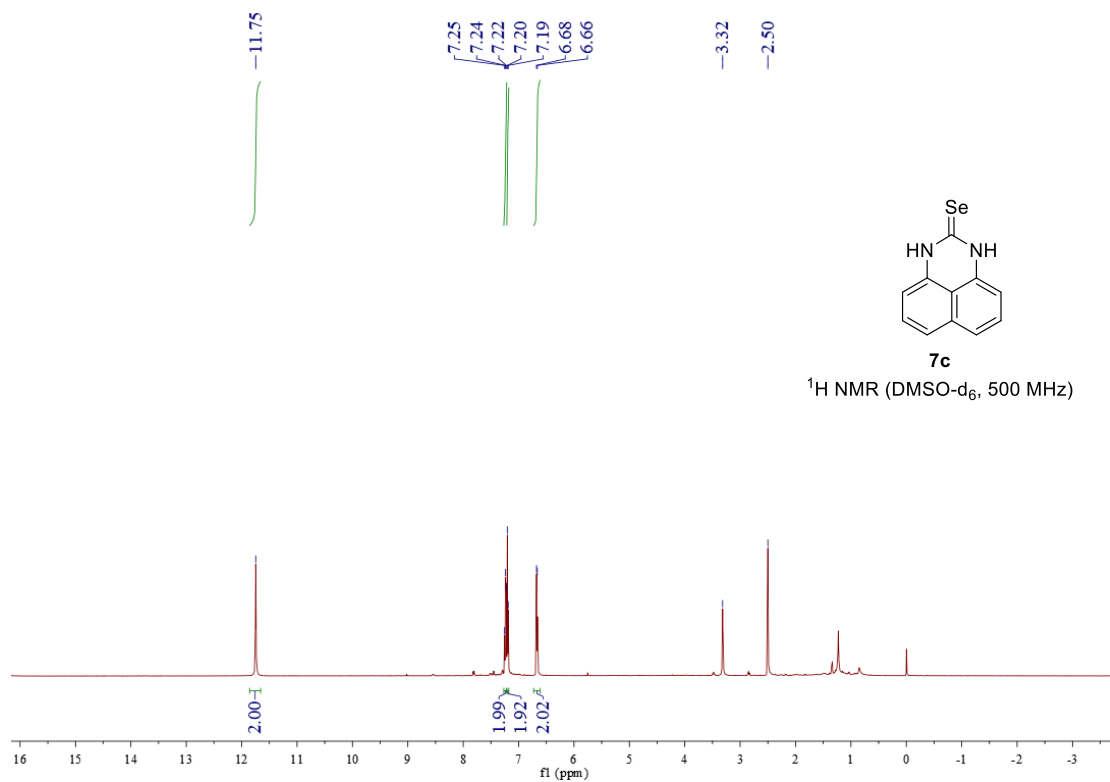
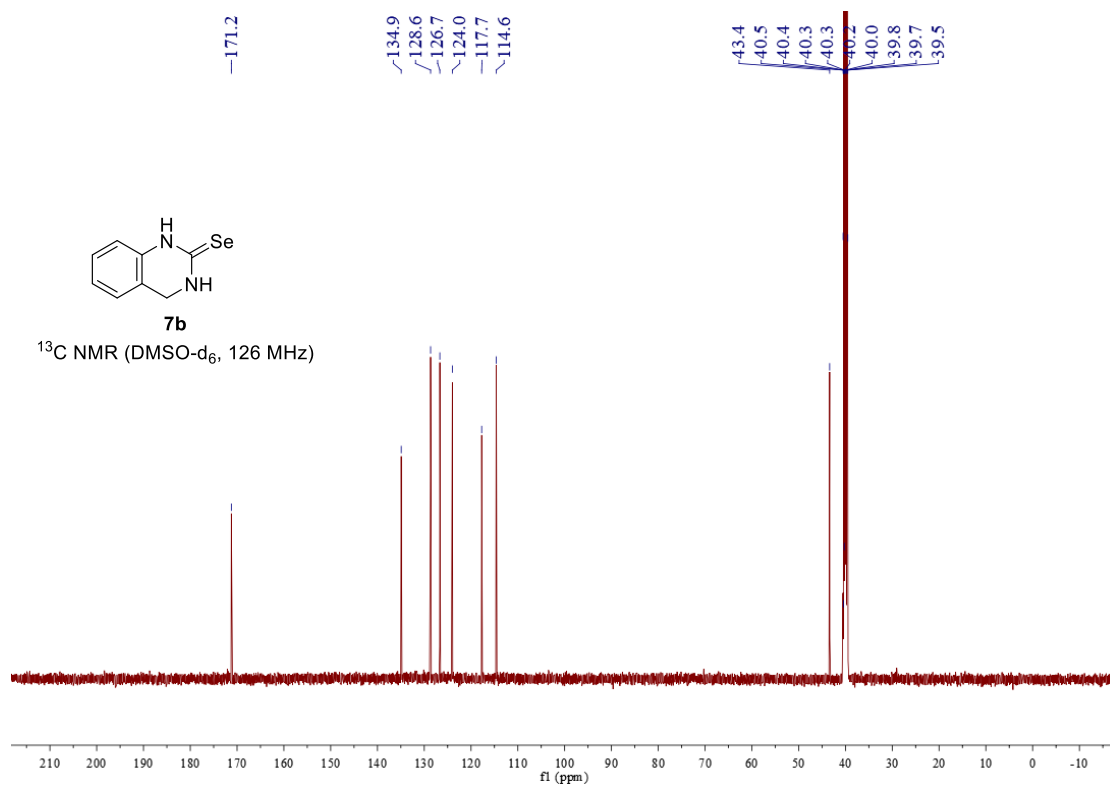


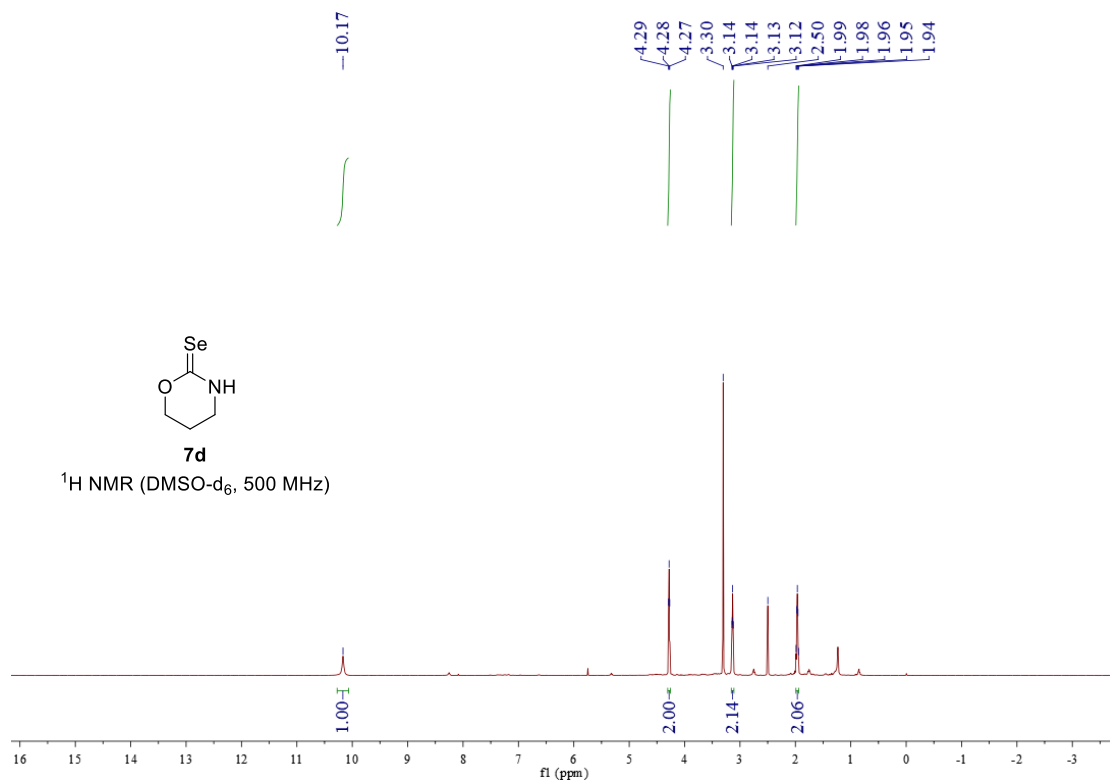
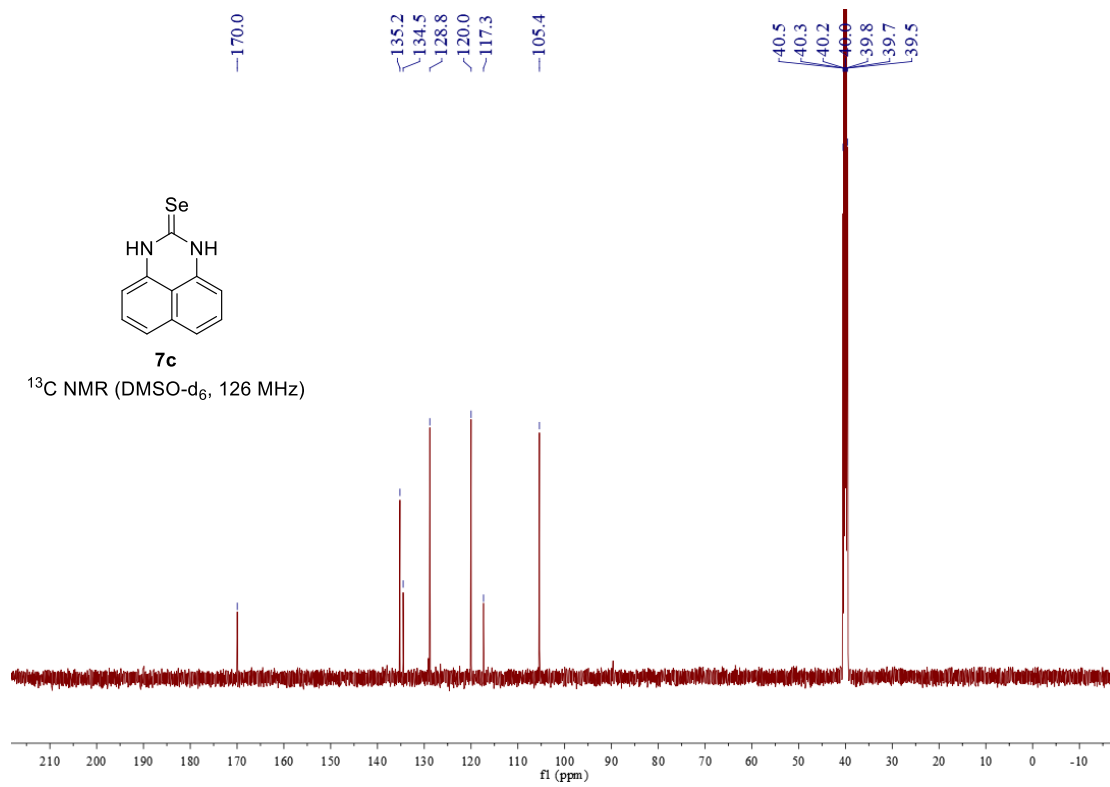


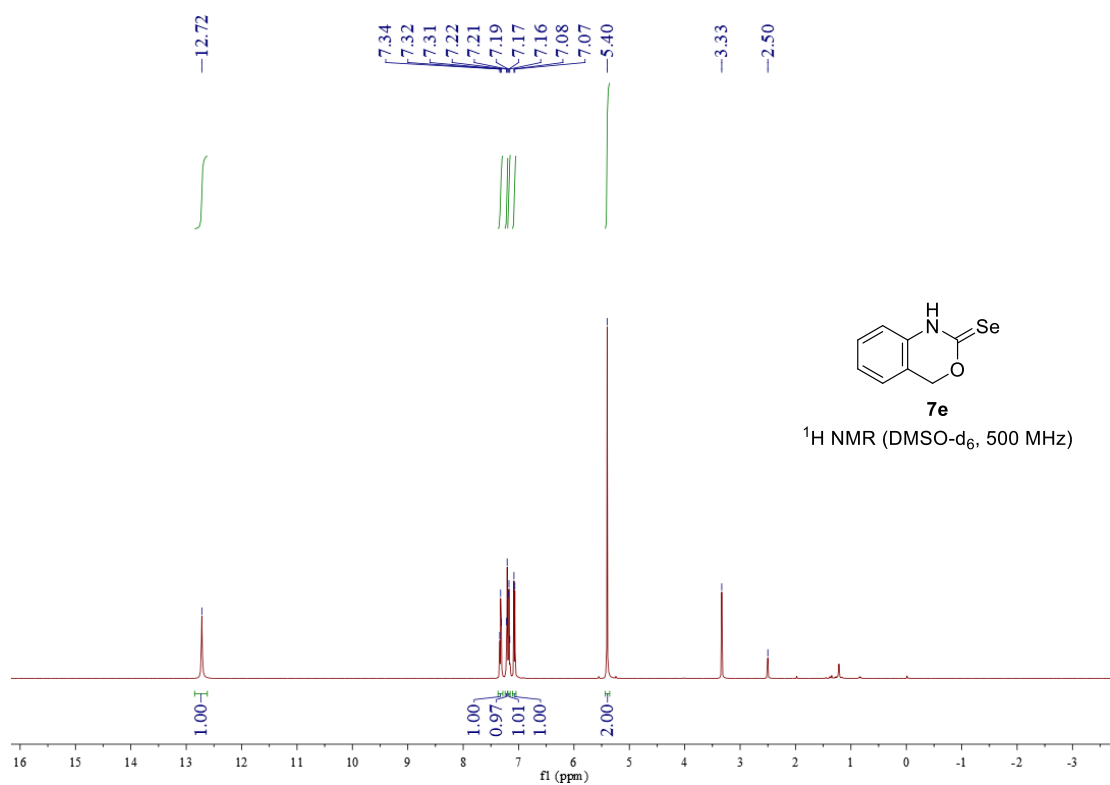
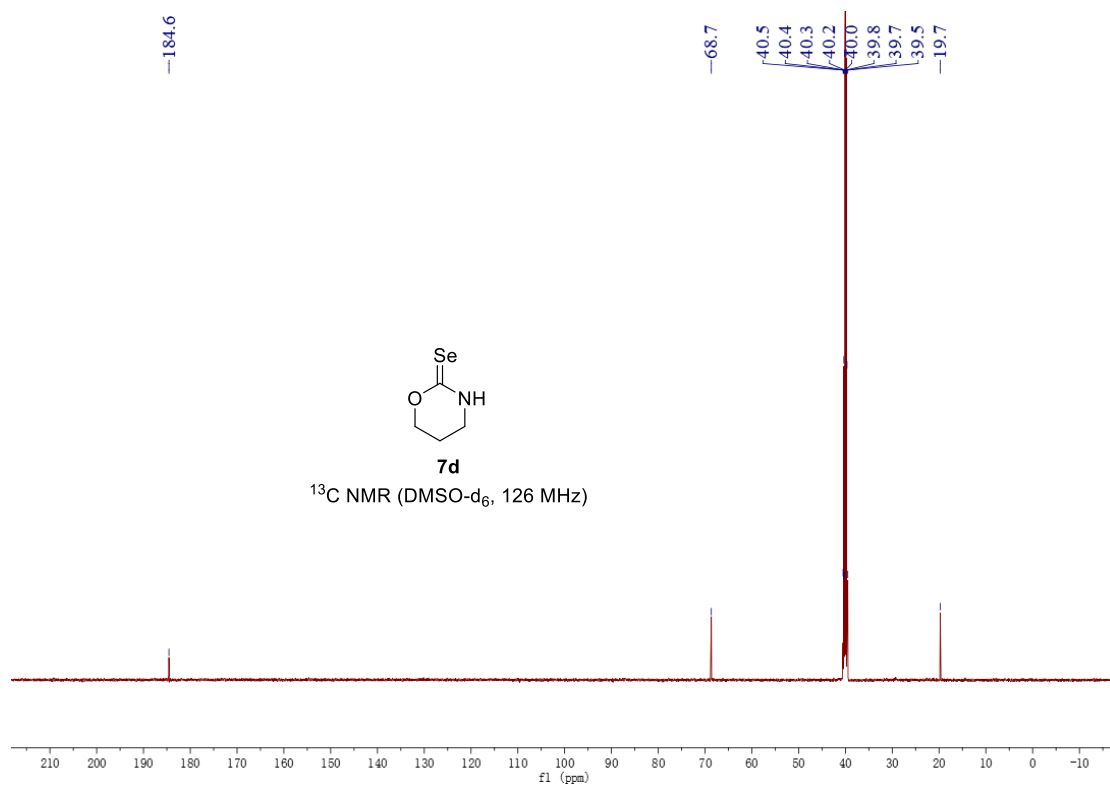


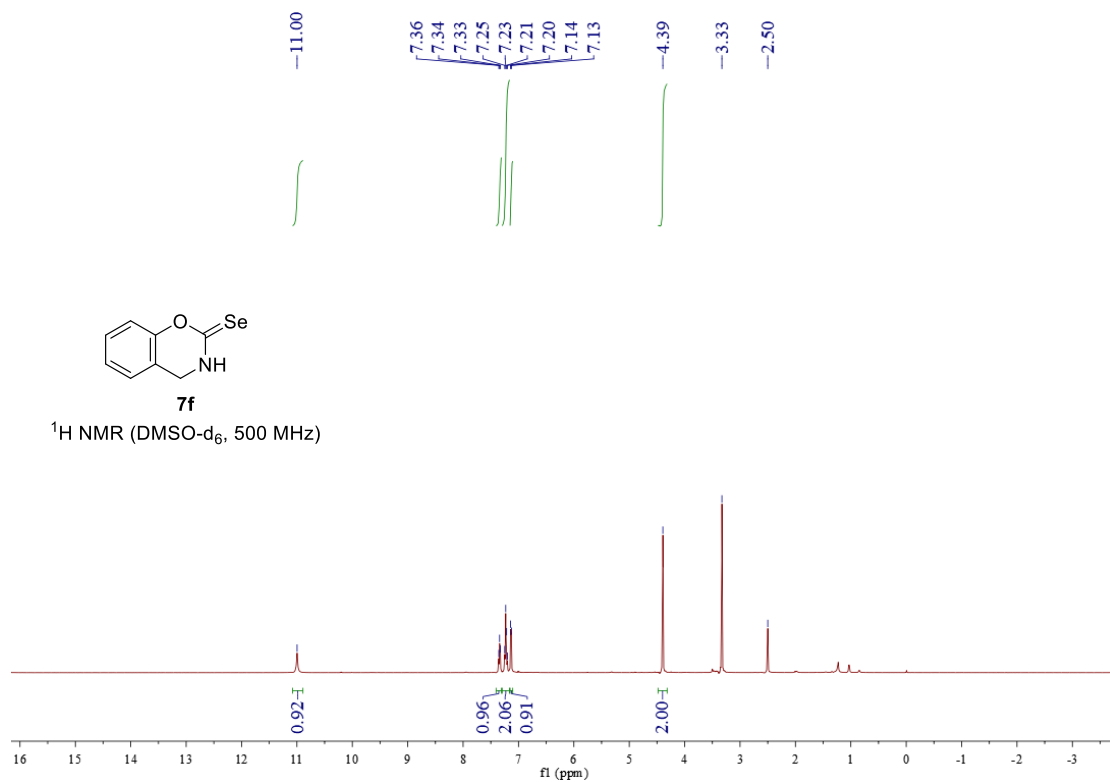
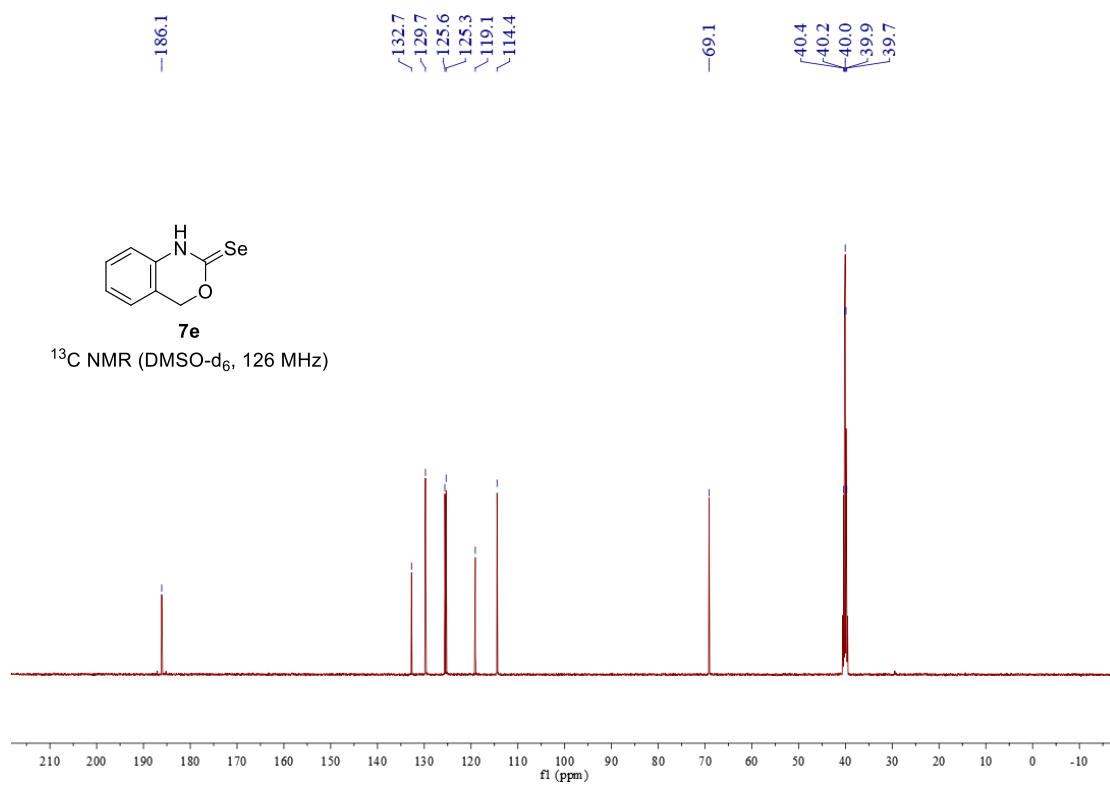


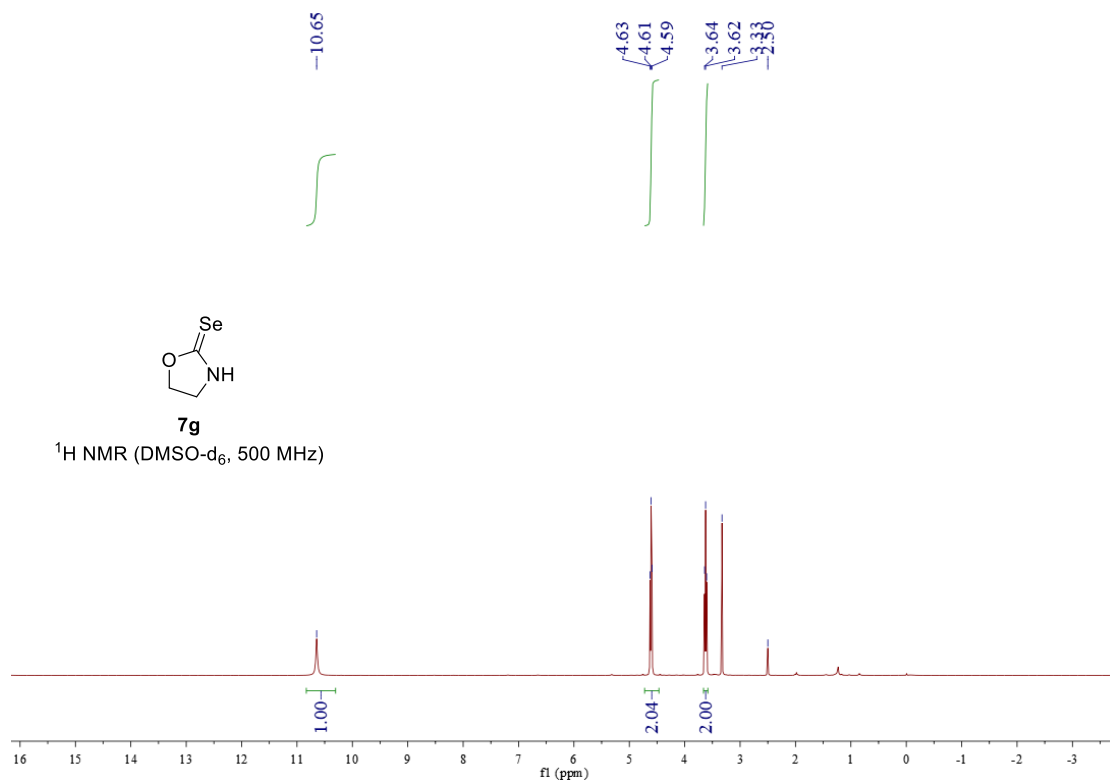
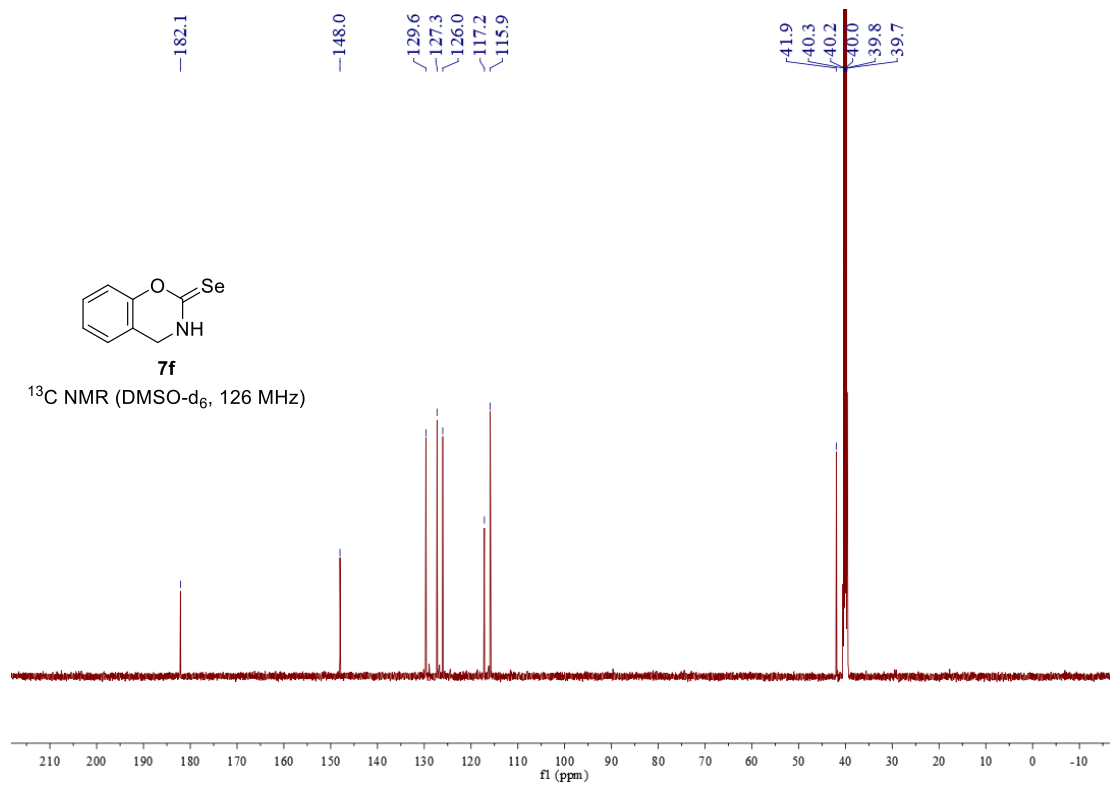


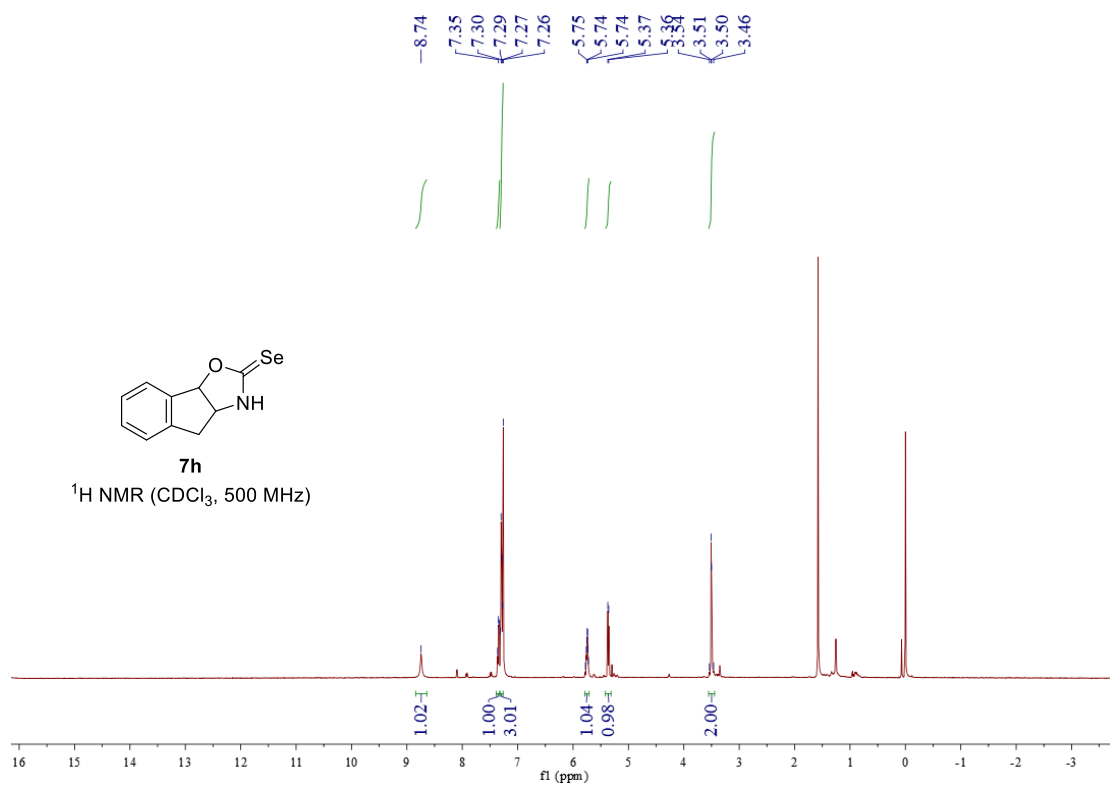
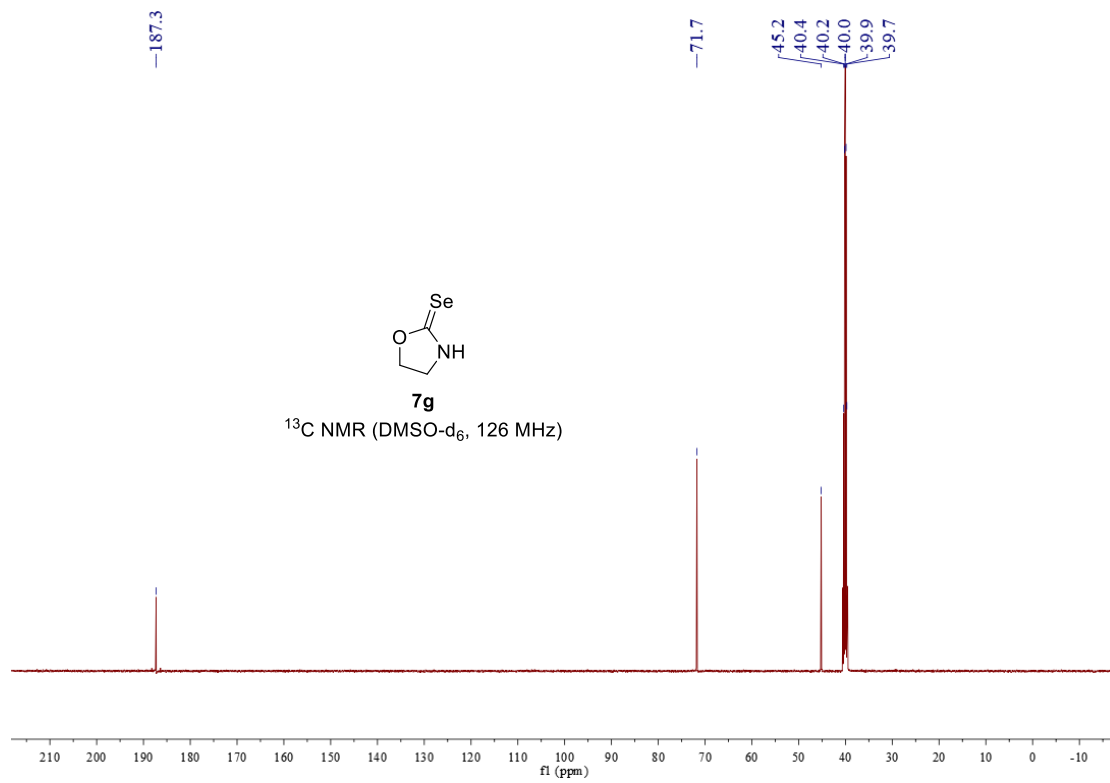


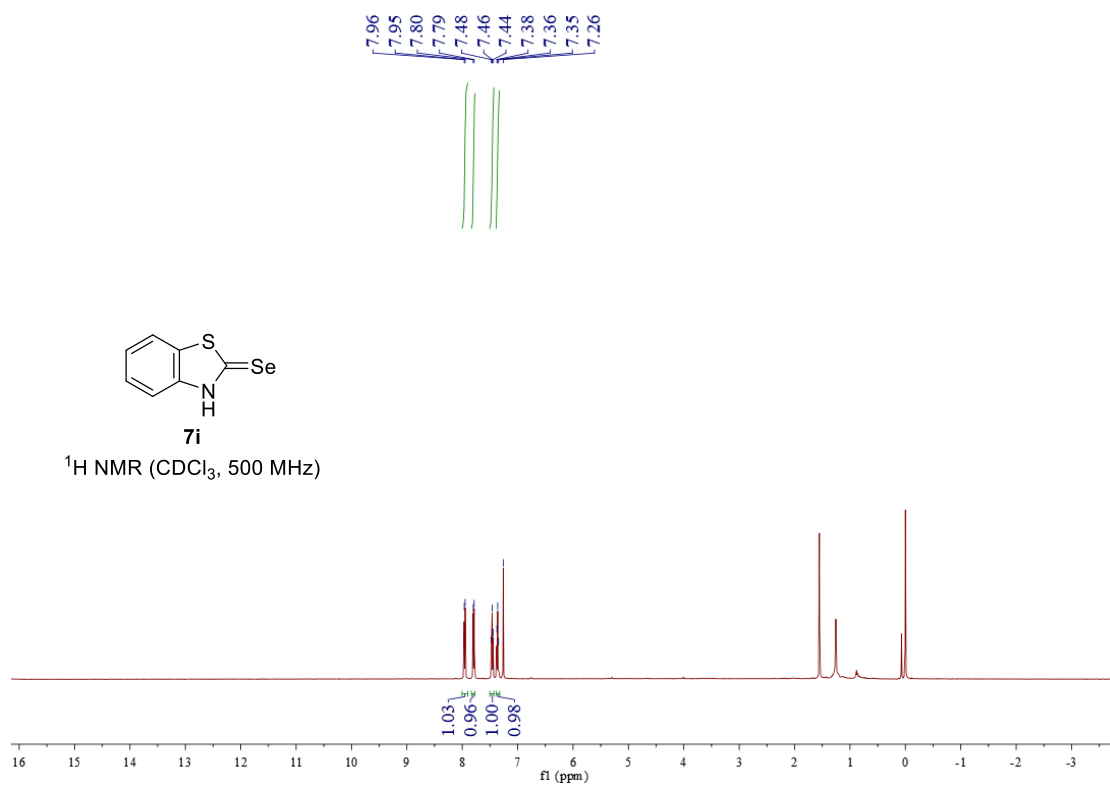
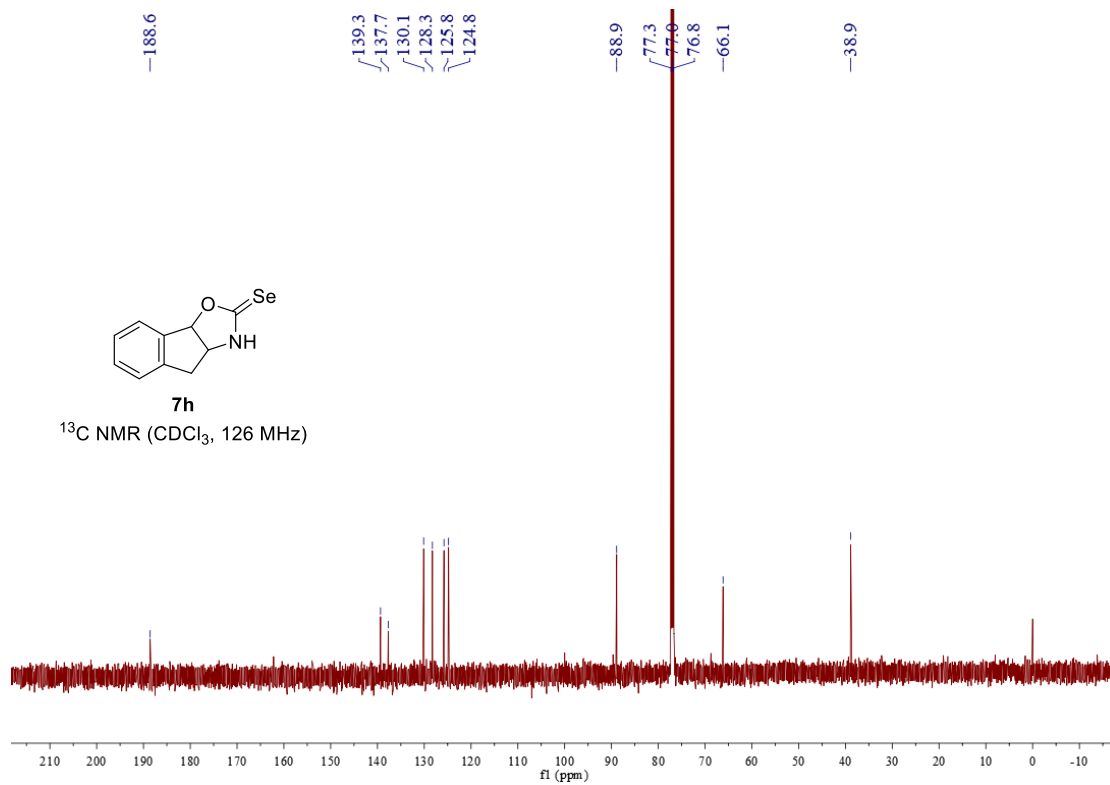


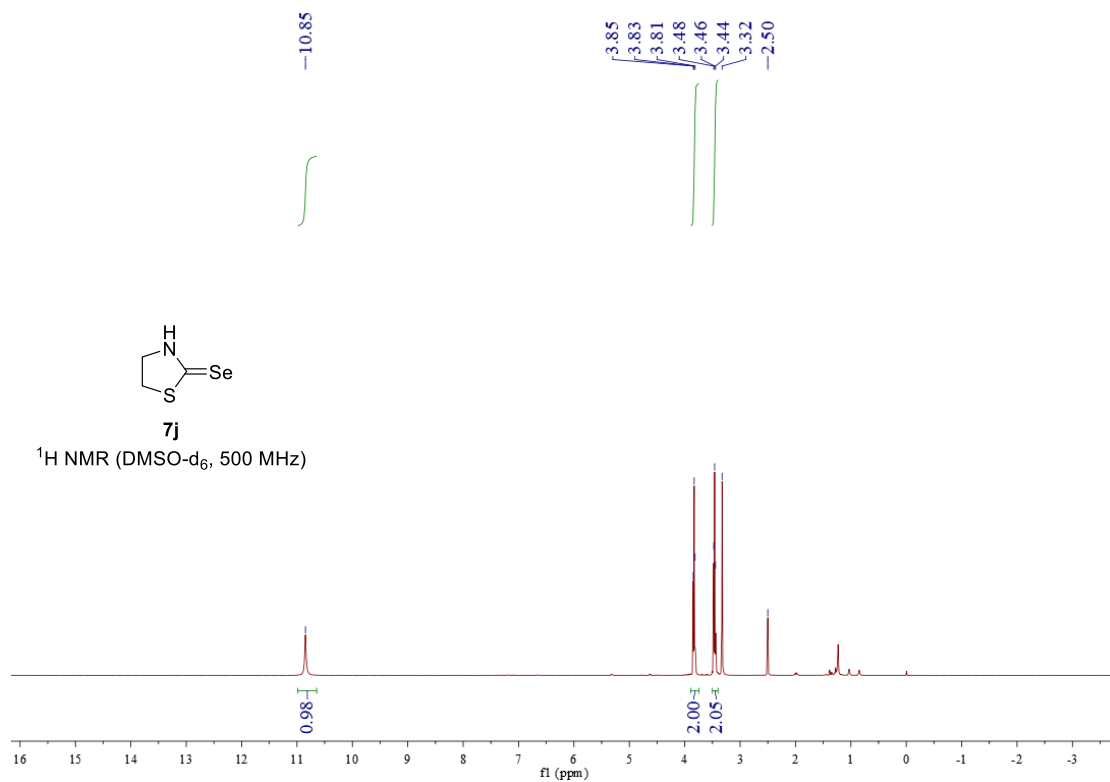
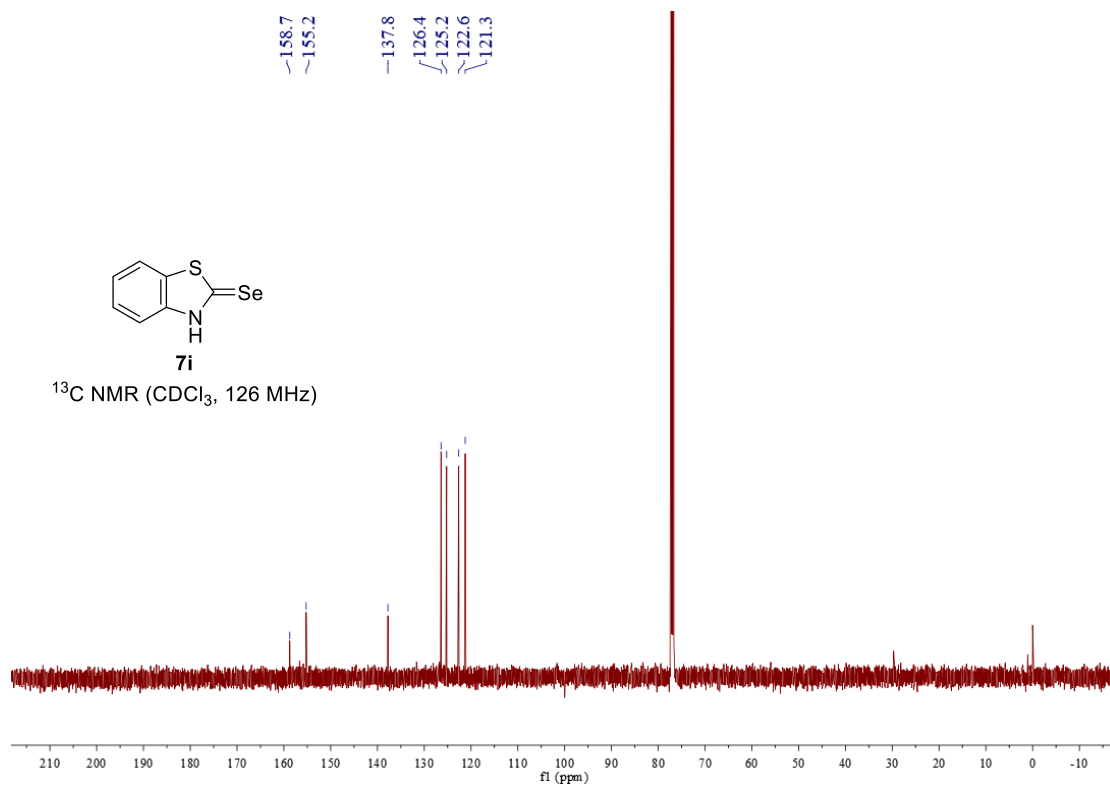


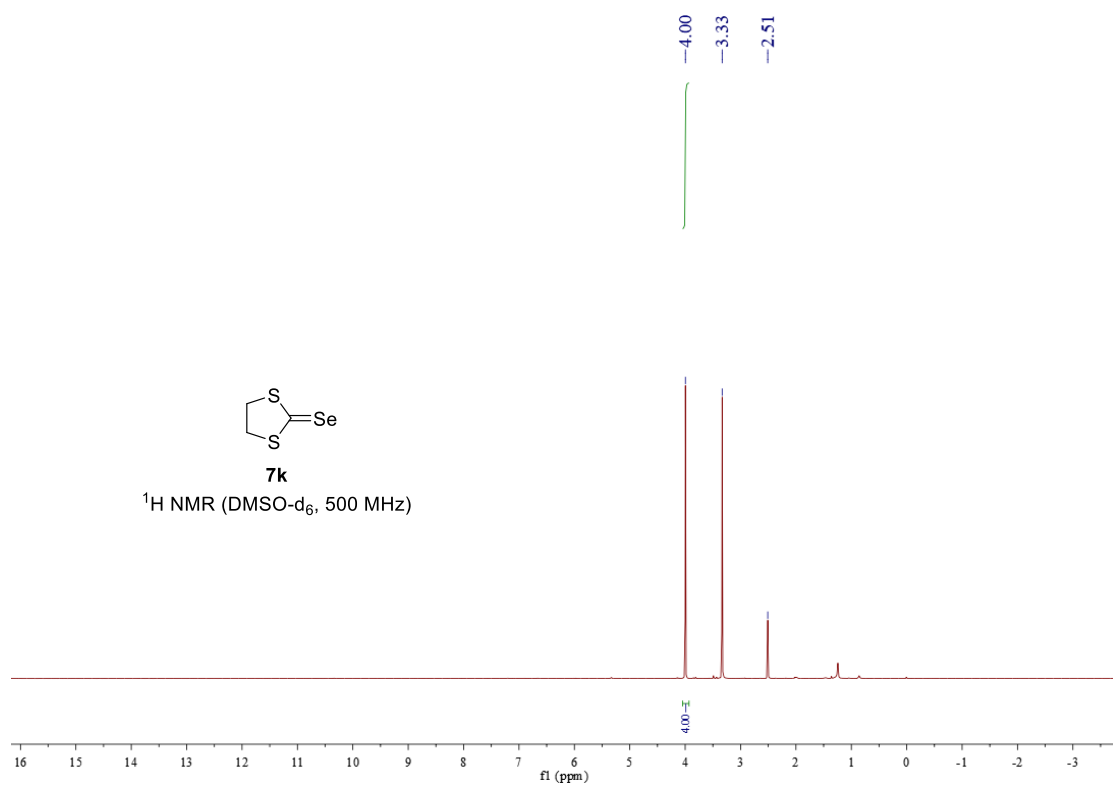
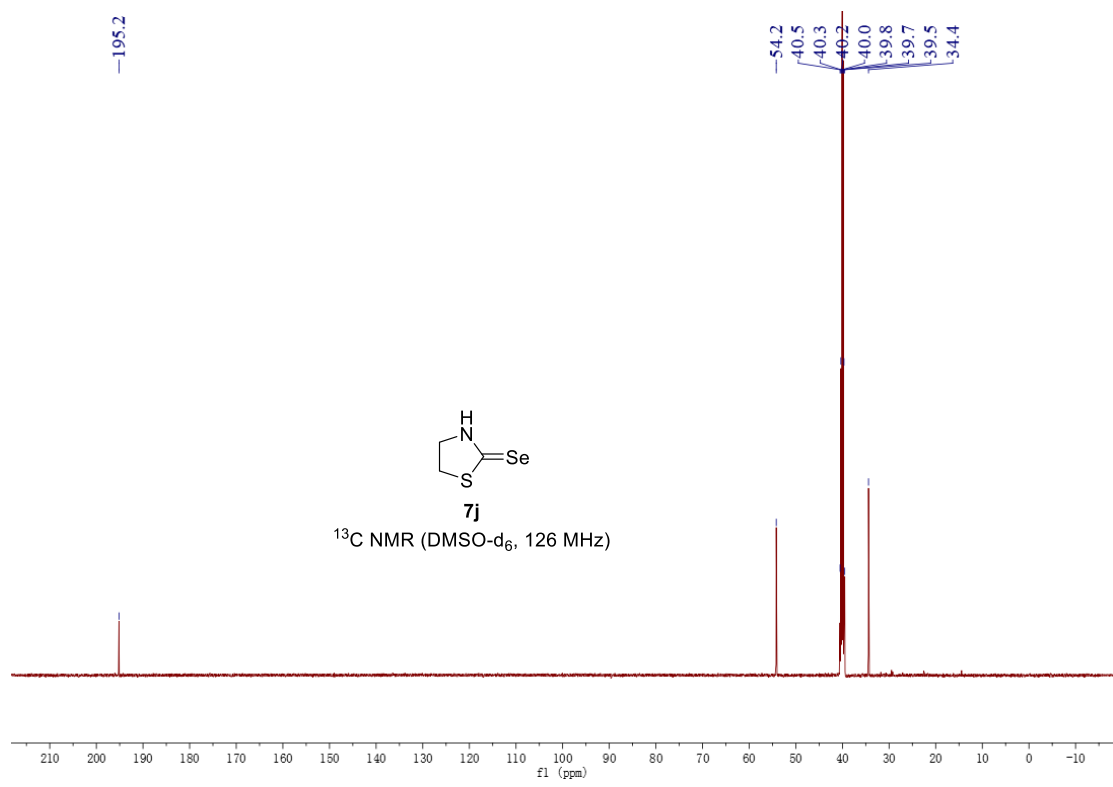






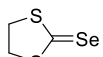






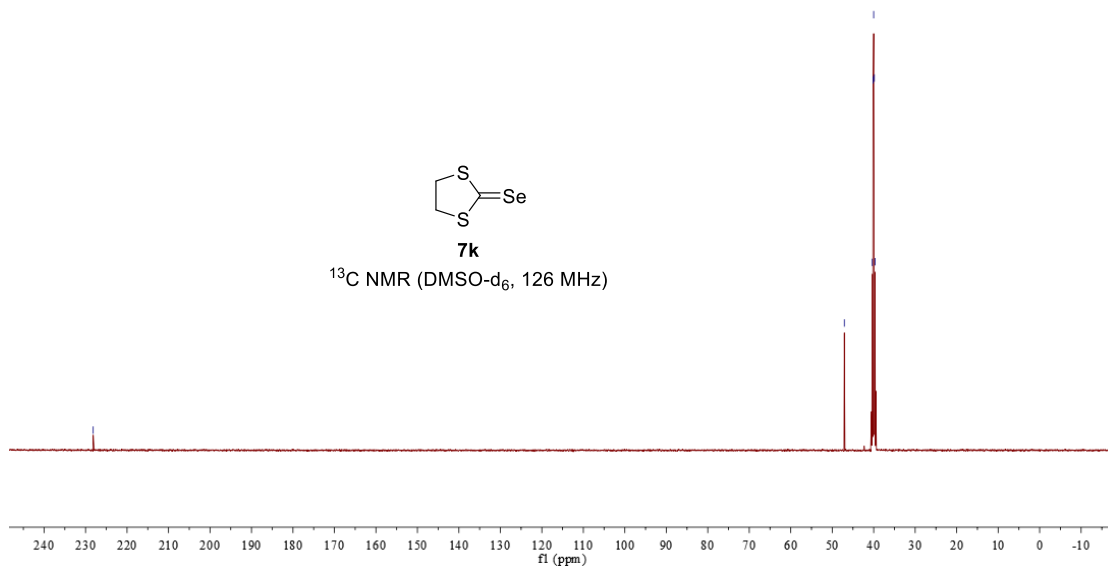
-228.2

47.1
40.3
40.2
40.0
39.8
39.7

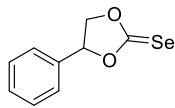


7k

^{13}C NMR (DMSO- d_6 , 126 MHz)



7.44
7.43
7.40
7.38
7.37
7.35
7.34
7.32
7.26
4.66
4.64
4.64
4.63
4.48
4.46
4.44



7l

^1H NMR (CDCl $_3$, 500 MHz)

