Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry.

Electronic Supplementary Information

Glutathione peroxidase mimics based on conformationally-restricted, *peri*-like 4,5-disubstituted fluorene dichalcogenides

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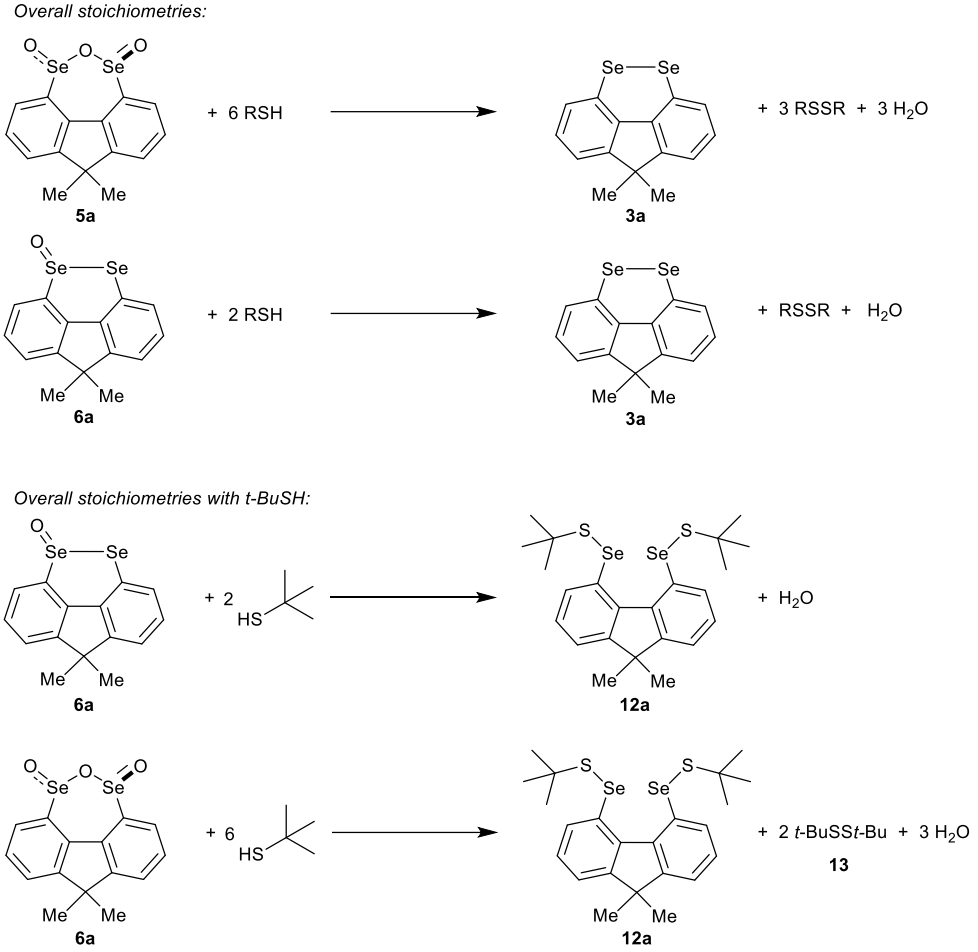
General experimental

All reagents were bought commercially from either Sigma-Aldrich (Merck), Alfa Aesar, Acros Organics, Fisher Scientific, VWR, or Fluorochem, and were used as sold unless stated. *n*BuLi was bought as a 2.5 M solution in hexanes and titrated with menthol and 'blue'.[1] *m*CPBA was purified by washing with a pH 7 phosphate buffer which was prepared from 0.1 M NaOH(aq) (154 mL) and 0.2 M $KH_2PO_{4(aq)}$ (94 mL), distilled water was added up to 376 mL. A solution of *m*CPBA (77% w/w, 10 g) in Et_2O (100 mL) was washed with the buffer solution; the combined organic layers were then dried over $MgSO_4$, evaporated under reduced pressure to yield pure *m*CPBA (7.3 g, 73%). All reactions were performed under an atmosphere of argon in oven or flame dried flasks. All solvents were bought from one of the above suppliers, and used without further drying or purification unless stated. *N*,*N*,*N*',*N*'-Tetramethylethylenediamine (TMEDA) was distilled from CaH_2 and stored over KOH. Silica gel on aluminium-backed TLC plates were used for reaction monitoring, supplied from Merck. The plates were visualised in UV (254 nm) and standard laboratory visualizing agents: $KMnO_4$, anisaldehyde, vanillin, curcumin, iodine powder. Purification by flash column chromatography was performed on Sigma-Aldrich or Fluorochem silica gel, pore size 60 Å, 230–400 mesh particle size, 40–63 μm particle size. Infra-red spectra were recorded neat (oil) or with the aid of an ATR-attachment (solid) on a Perkin Elmer Spectrum 100 FT-IR spectrometer, only selected absorbances (νmax, cm^{-1}) are reported. Melting points were recorded using open glass capillaries on a Gallenkamp melting point apparatus and are uncorrected. MS data are reported as m/z (%) (relative intensity except in cases where only the parent ion is observed). ^{1}H and ^{13}C NMR spectra were recorded on a Bruker AVIII300, Bruker AVIII400, Bruker NEO400 and Bruker AV4-500 in the solvents indicated. The solvent signals were used as references: ^{1}H NMR: residual $CHCl_3$ (7.26 ppm) and CD_3OD (3.31 ppm); ^{13}C NMR; $CDCl_3$ (77.16 ppm) and CD_3OD (49.00 ppm). ^{77}Se NMR data was recorded on a Bruker AVIII400 (76 MHz ^{77}Se, T = 293 K) with diphenyl diselenide (463 ppm) as the external standard.[2] Coupling constants (J) are reported in Hz, and are reported as observed, not averaged between the two environments that share them. The following abbreviations are used to describe multiplicity in ^{1}H-NMR: m (multiplet), s (singlet), d (doublet), t (triplet), hept (heptet), ap. (apparent). The distinction between multiplet and stack is as follows: a multiplet is a single environment that is too convoluted to establish its multiplicity correctly, a stack is where multiple environments overlap and their fidelity is lost. ^{1}H and ^{13}C assignments were made based on HMBC and HSQC NMR data.

Diselenide **2b** was prepared according to the literature procedure.[3]

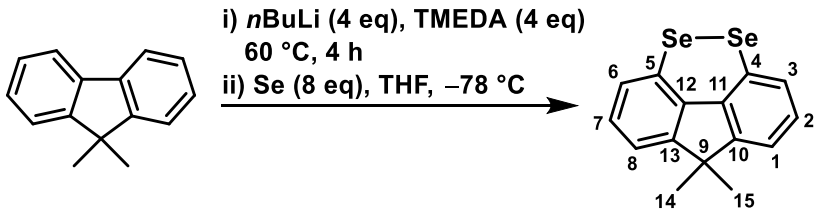
Overall stoichiometries in the reaction of 5a and 6a with thiols



Scheme S1 Overall stoichiometries in the reaction of **5a** and **6a** with thiols

Experimental procedures and analytical data

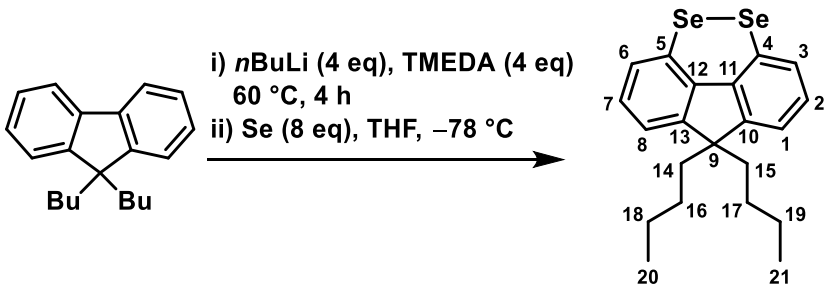
9,9-Dimethyl-9*H*-fluoreno[4,5-*cde*][1,2]diselenine (3a)



4a **3a**

Diselenide **3a** is a novel compound, prepared according to a modified literature procedure.[4] *n*BuLi (2.39 M in hexanes, 8.6 mL, 20.6 mmol) was added dropwise over 10 min at rt to a solution of 9,9-dimethyl-9*H*-fluorene (**4a**) (1.00 g, 5.14 mmol) in TMEDA (3.1 mL, 20.6 mmol). The reaction mixture was stirred at 60 °C for 4 h. The reaction mixture was cooled to –78 °C and THF (20 mL) was added followed by the addition of selenium (3.25 g, 41.13 mmol) as a single portion. The cooling bath was removed and the reaction mixture was allowed to stir for 16 h. H_2O (150 mL) was added and the mixture was extracted with Et_2O (3 × 50 mL). The combined organic layers were dried over $MgSO_4$, filtered and concentrated under reduced pressure. The crude material was purified by flash column chromatography (100% *n*-hexane). Diselenide **3a** was obtained as a dark burgundy crystalline solid (938 mg, 52%). R_f 0.54 (*n*-hexane); mp 135–136 °C; ν_{max}(solid neat, ATR)/cm^{-1} 3052, 2956, 2895, 2859, 1561, 1403, 1188, 937, 782, 727; δ_H (400 MHz, $CDCl_3$) 7.23–7.19 (6 H, stack, H-1, H-2, H-3, H-6, H-7 and H-8), 1.45 (6 H, s, H-14 and H-15); δ_C (101 MHz, $CDCl_3$) 154.1 (C, C-10 and C-13), 137.7 (C, C-11 and C-12), 129.5 (CH, C-1 and C-8 or C-2 and C-7 or C-3 and 6), 126.3 (CH, C-1 and C-8 or C-2 and C-7 or C-3 and C-6), 121.6 (CH, C-1 and C-8 or C-2 and C-7 or C-3 and C-6), 117.8 (C, C-4 and C-5), 47.2 (C, C-9), 26.5 (CH_3, C-14 and C-15); ^{77}Se NMR (76 MHz, $CDCl_3$) 236.2; *m/z* (ASAP$^+$) found 351.9285 ($M^{+\bullet}$, $C_{15}H_{12}{}^{80}Se_2$ requires 351.9272).

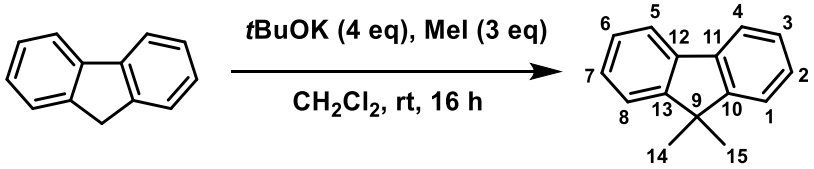
9,9-Dibutyl-9*H*-fluoreno[4,5-*cde*][1,2]diselenine (3b)



4b **3b**

Compound **3b** was prepared according a modified literature procedure.[4] *n*BuLi (2.00 M in hexanes, 7.19 mL, 14.38 mmol) was added dropwise over 10 min to a solution of 9,9-dibutyl-9*H*-fluorene (**4b**) (1.00 g, 3.59 mmol) in TMEDA (2.15 mL, 14.38 mmol). The reaction mixture was then stirred at 60 °C for 4 h. The reaction mixture was then cooled to –78 °C and THF (20 mL) was added followed by addition of selenium (2.21 g, 27.98 mmol) as a single portion. The cooling bath was removed and the reaction was allowed to stir for 16 h. H_2O (150 mL) was added and the mixture was extracted with Et_2O (3 × 50 mL). The combined organic layers were dried over $MgSO_4$, filtered and concentrated under reduced pressure. The crude material was purified by flash column chromatography (100% *n*-hexane). Diselenide **3b** was obtained as a dark burgundy crystalline solid (747 mg, 48%). R_f 0.54 (*n*-hexane); mp 129–133 °C; ν_{max}(solid neat, ATR)/cm^{-1} 3038, 2953, 2924, 2851, 1561, 1405, 1106, 999, 789, 733; δ_H (400 MHz, $CDCl_3$) 7.27–7.23 (2 H, m, H-2 and H-7), 7.18–7.16 (4 H, stack, H-1, H-3, H-6 and H-8), 1.98–1.94 (4 H, m, H-14 and H-15), 1.00 (4 H, sext, *J* = 8.0, H-16 and H-17), 0.75 (6 H, t, *J* = 8.0, H-20 and H-21), 0.71–0.66 (4 H, m, H-18 and H-19); δ_C (101 MHz, $CDCl_3$) 151.5 (C, C-10 and C-13), 139.6 (C, C-11 and C-12), 129.3 (CH, C-3 and C-6), 126.3 (CH, C-2 and C-7), 122.0 (CH, C-1 and C-8), 117.8 (C, C-4 and C-5), 55.5 (C, C-9), 39.7 (CH_2, C-14 and 15), 26.1 (CH_2, C-16 and 17), 23.1 (CH_2, C-18 and 19), 13.9 (CH_3, C-20 and 21); ^{77}Se NMR (76 MHz, $CDCl_3$) 235.2; *m/z* (EI$^+$) found 436.0208 ($M^{+\bullet}$, $C_{21}H_{24}{}^{80}Se_2$ requires 436.0208).

9,9-Dimethyl-9*H*-fluorene (4a)



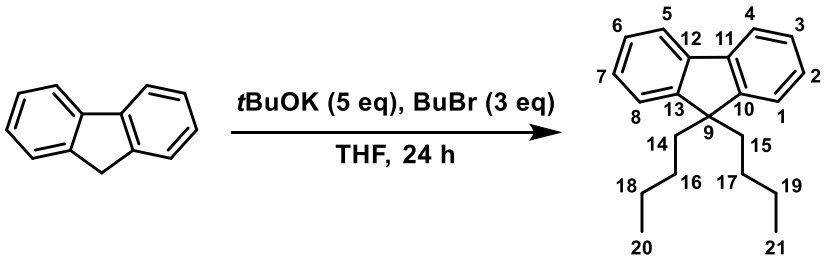
4a

Known compound **4a** was prepared according to the literature procedure.[5] *t*BuOK (11.80 g, 105.00 mmol) was added as a single portion at rt to a solution of fluorene (5.00 g, 30.00 mmol) in THF (50 mL). Iodomethane (10.00 g, 75.00 mmol) was added dropwise over a period of 10 min. The reaction mixture was allowed to stir at rt for 16 h. The mixture was filtered and the filtrate was treated with saturated NH_4Cl solution (250 mL) and extracted with CH_2Cl_2 (2 × 100 mL). The combined organic layers were dried over $MgSO_4$, filtered and concentrated under reduced pressure. The crude material was purified by flash column chromatography (*n*-hexane 100%) giving compound **4a** (1.08 g, 45%) as a white crystalline solid. R_f 0.42 (*n*-hexane); mp 96–98 °C; ν_{max}(solid neat, ATR)/cm^{-1} 2927, 2927, 2856, 1465, 1447, 1376, 1332, 1299, 1221, 1128; δ_H (400 MHz, $CDCl_3$) 7.86 – 7.83 (2 H, m, H-1 and H-8), 7.56 – 7.54 (2 H, m, H-4 and H-5), 7.48 – 7.40 (4 H, stack, H-2, H-3, H-6 and H-7), 1.65 (6 H, s, H-14 and H-

15); δ_C (101 MHz, $CDCl_3$) 153.7 (C, C-10 and C-13), 139.3 (C, C-11 and C-12), 127.4 (CH, C-2 and C-7), 127.1 (CH, C-3 and C-6), 122.7 (CH, C-4 and C-5), 120.1 (CH, C-1 and C-8), 46.9 (C, C-9), 27.3 (CH_3, C-14 and C-15); *m/z* (ASAP$^+$) found 195.1178 ([M+H]$^+$, $C_{15}H_{15}$ requires 195.1174).

Analytical data in agreement with literature values.[6]

9,9-Dibutyl-9*H*-fluorene (4b)

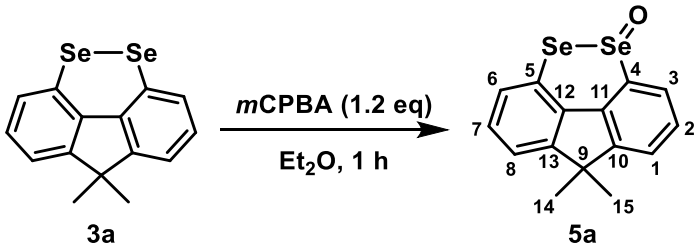


4b

Known compound **4b** was prepared according a modified literature procedure.[4] *t*BuOK (3.55 g, 29.1 mmol) was added in one portion at rt to a solution of fluorene (967 mg, 5.82 mmol) in THF (40 mL). 1-Bromobutane (1.9 mL, 17.5 mmol) was added dropwise to the solution over 1 min. The reaction mixture was then allowed to stir at rt for 16 h. NH_4Cl (50 mL) was added and the resulting mixture was extracted with CH_2Cl_2 (3 × 20 mL). The combined organic layers were dried over $MgSO_4$, filtered and concentrated under reduced pressure. The crude material was purified by flash column chromatography (*n*-hexane 100%). Compound **4b** (990 mg, 61%) was obtained as a white crystalline solid. R_f 0.60 (*n*-hexane); mp 49–50 °C; ν_{max}(solid neat, ATR)/cm^{-1} 2927, 2927, 2856, 1465, 1447, 1376, 1332, 1299, 1221, 1128; δ_H (400 MHz, $CDCl_3$) 7.73–7.17 (2 H, m, H-4 and H-5), 7.37–7.29 (6 H, stack, H-1, H-2, H-3, H-6, H-7 and H-8), 2.01–1.96 (4 H, m, H-14 and H-15), 1.07 (4 H, sext, *J* = 8.0, H-16 and H-17), 0.67 (6 H, t, *J* = 7.0, H-20 and H-21), 0.54–0.64 (4 H, m, H-18 and H-19); δ_C (101 MHz, $CDCl_3$) 150.8 (C, C-10 and C-13), 141.2 (C, C-11 and C-12), 127.1, 126.8, 123.0 and 119.8 (CH, C-1 – C-8), 55.1 (C, C-9), 40.3 (CH_2, C-14 and C-15), 26.1 (CH_2, C-16 and C-17), 23.2 (CH_2, C-18 and C-19), 13.9 (CH_3, C-20 and C-21); *m/z* (AP$^+$) 279 ([M+H]$^+$ 279.1, 11%), 221.2 (12), 179.1 (53), 167.1 (100).

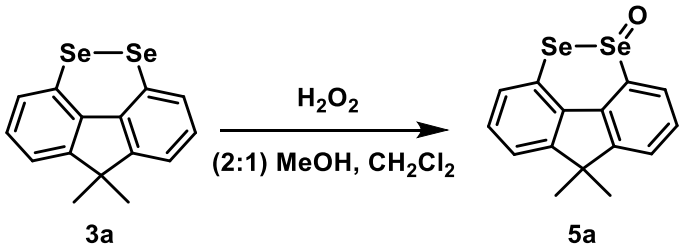
Analytical data in agreement with literature values.[7]

9,9-Dimethyl-9*H*-fluoreno[4,5-*cde*][1,2]diselenine 4-oxide (5a) via *m*CPBA oxidation



*m*CPBA (59 mg, 0.34 mmol) was added as a single portion at rt to a solution of 9,9-dimethyl-9*H*-fluoreno[4,5-*cde*][1,2]diselenine (**3a**) (98 mg, 0.28 mmol) in Et_2O (5 mL) and allowed to stir at rt for 15 min. Compound **5a** precipitated during this time. The solid was filtered under vacuum and washed with Et_2O (3 × 15 mL). Compound **5a** was obtained as a yellow powder (43 mg, 42%). Evaporation of the filtrate gave recovered diselenide **3a** (56 mg, 57%). R_f 0.15 (Et_2O); mp 191–192 °C; ν_{max}(solid neat, ATR)/cm^{-1} 2957, 2919, 2858, 1567, 1427, 1407, 830, 782; δ_H (400 MHz, $CDCl_3$) 7.73 (1 H, dd, *J* = 7.6, 1.0, H-3), 7.67 (1 H, dd, *J* = 7.5, 1.0, H-1), 7.60 (1 H, t, *J* = 7.6, H-2), 7.49 (1 H, dd, *J* = 7.6, 1.5, H-6), 7.45 (1 H, t, *J* = 7.6, H-7), 7.39 (1 H, dd, *J* = 7.1, 1.4, H-8), 1.57 (3 H, s, H-14 or H-15), 1.55 (3H, s, H-14 or H-15); δ_C (101 MHz, $CDCl_3$) 156.0 (C, C-10), 155.3 (C, C-13), 134.1 (C, C-11), 133.4 (C, C-12), 130.7 (C, C-4), 130.2 (CH, C-7), 129.5 (CH, C-2), 127.0 (CH, C-6), 126.2 (CH, C-1), 125.5 (CH, C-3), 121.3 (CH, C-8), 116.7 (C, C-5), 47.6 (C, C-9), 27.1 (CH_3, C-14 or C-15), 26.5 (CH_3, C-14 or C-15); ^{77}Se NMR (76 MHz, $CDCl_3$) 1047.3, 709.0; *m/z* (ASAP$^+$) found 368.9302 ($M^{+\bullet}$, $C_{15}H_{12}O^{80}Se_2$ requires 368.9300)

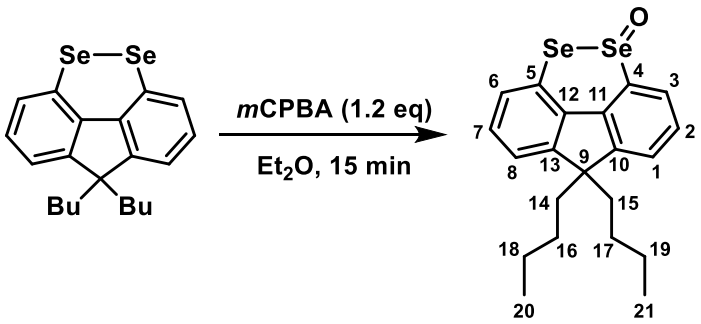
9,9-Dimethyl-9*H*-fluoreno[4,5-*cde*][1,2]diselenine 4-oxide (5a) *via* H_2O_2 oxidation



$H_2O_{2(aq)}$ (9.77 M in H_2O, 0.3 mL, 2.93 mmol) was added rapidly to a solution of 9,9-dimethyl-9*H*-fluoreno[4,5-*cde*][1,2]diselenine (**3a**) (100 mg, 0.29 mmol) in a 2:1 mixture of MeOH (13.5 mL) and CH_2Cl_2 (6.5 mL) and allowed to stir at rt for 24 h. The solvent was removed under reduced pressure. H_2O (15 mL) was added and the mixture was extracted with Et_2O (2 × 15 mL). The combined organic layers were dried over $MgSO_4$, filtered and concentrated under reduced pressure. The crude material was purified by flash column chromatography (4:1, *n*-

hexane: Et_2O). Diselenide **3a** (59 mg, 59%) eluted from the column first, followed by **5a** (36 mg, 34%).

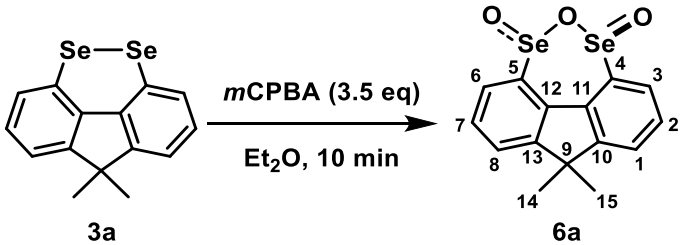
9,9-Dibutyl-9*H*-fluoreno[4,5-*cde*][1,2]diselenine 4-oxide (5b) via *m*CPBA oxidation



3b **5b**

*m*CPBA (47 mg, 0.27 mmol) was added as a single portion to a solution of 9,9-dibutyl-9*H*-fluoreno[4,5-*cde*][1,2]diselenine (**3b**) (100 mg, 0.23 mmol) in Et_2O (5 mL) and allowed to stir at rt for 15 min. Compound **5b** precipitated during this time. The solid was filtered under vacuum and washed with Et_2O (3 × 15 mL). Compound **5b** was obtained as a yellow powder (40 mg, 38%). Evaporation of the filtrate gave diselenide **3b** (53 mg, 53%). Data for **5b**: R_f 0.15 (Et_2O); mp 147–149 °C; ν_{max}(solid neat, ATR)/cm^{-1} 2955, 2916, 2274, 1368, 1246, 816, 732; δ_H (400 MHz, $CDCl_3$) 7.75–7.70 (1 H, m, H-3), 7.59–7.58 (2 H, stack, H-1 and H-2), 7.47–7.41 (2 H, stack, H-6 and H-7), 7.39 (1 H, *dd*, *J* = 7.8 and 1.4, H-8), 2.06–2.02 (4 H, m, H-14 and H-15), 1.12–1.05 (4 H, m, H-16 and H-17), 0.68 (8 H, ap. q, *J* = 7.8, H-20, H-21 and H-18 or H-19), 0.59–0.52 (2 H, m, H-18 or H-19); δ_C (101 MHz, $CDCl_3$) 153.5 (C, C-10), 152.7 (C, C-13), 135.6 (C, C-11), 134.9 (C, C-12), 130.7 (C, C-4), 130.1 (CH, C-7), 129.3 (CH, C-2), 126.8 (CH, C-6), 126.4 (CH, C-1), 125.5 (CH, C-3), 121.7 (CH, C-8), 116.7 (C, C-5), 55.8 (C, C-9), 39.84 (CH_2, C-14 or 15), 39.79 (CH_2, C-14 or 15), 26.1 (CH_2, C-16 and 17), 23.1 (CH_2, C-18 or 19), 23.0 (CH_2, C-18 or 19), 13.9 (CH_3, C-20 or 21), 13.8 (CH_3, C-20 or 21); ^{77}Se NMR (76 MHz, $CDCl_3$) 710.0, 1046.2; *m/z* (ASAP$^+$) 453.0239 ([M+H]$^+$, $C_{21}H_{25}{}^{80}Se_2O$ requires 453.0240)

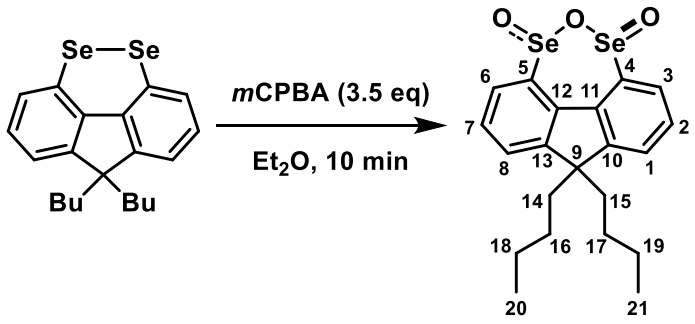
***trans*-Seleninic anhydride 6a**



3a **6a**

Compound **6a** is a novel compound, prepared according to a modified literature procedure.[3] *m*CPBA (603 mg, 3.49 mmol) was added as a single portion at rt to a solution of 9*H*-fluoreno[4,5-*cde*][1,2]diselenine (**3a**) (350 mg, 0.99 mmol) in Et_2O (5 mL) and allowed to stir at rt for 15 min. Seleninic anhydride **6a** precipitated during this time. The solid was filtered under vacuum and washed with Et_2O (3 × 15 mL). Seleninic anhydride **6a** was obtained as a white powder (389 mg, 98%) that required no further purification. mp 221–222 °C; ν_{max}(solid neat, ATR)/cm^{-1} 2951, 2924, 2852, 2397, 1453, 1411, 813, 795, 736, 673; δ_H (400 MHz, $CDCl_3$) 7.71–7.68 (4 H, stack, H-1, H-3, H-6 and H-8), 7.60 (2 H, t, *J* = 7.6, H-2 and H-7), 1.54 (6 H, s, H-14 and H-15); δ_C (101 MHz, $CDCl_3$) 157.3 (C, C-10 and C-13), 148.3 (C, C-11 and C-12), 135.5 (C, C-4 and C-5), 129.2 (CH, C-1 and C-8 or C-3 and C-6), 127.2 (CH, C-1 and C-8 or C-3 and C-6), 127.0 (CH, C-2 and C-7), 47.8 (C, C-9), 27.1 (CH_3, C-14 and C-15); ^{77}Se NMR (76 MHz, $CDCl_3$) 1278.3; *m/z* (ASAP$^+$) found 400.9204 ($M^{+\bullet}$, $C_{15}H_{12}{}^{80}Se_2O_3$ requires 400.9198).

***trans*-Seleninic anhydride 6b**

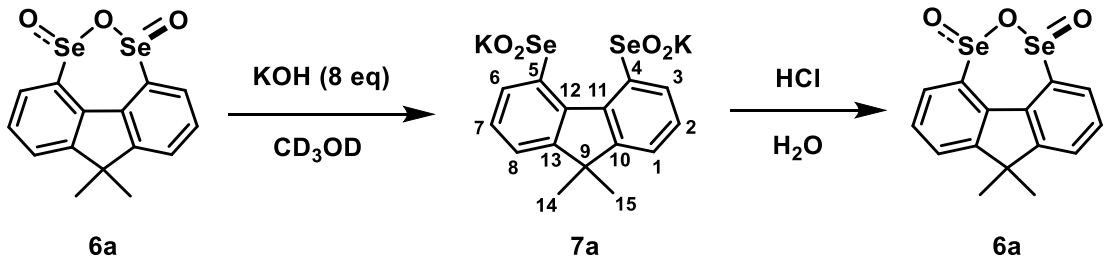


3b **6b**

Compound **6b** is a novel compound, prepared according to a modified literature procedure.[8] *m*CPBA (395 mg, 2.29 mmol) was added as a single portion at rt to a solution of 9,9-dibutyl-9*H*-fluoreno[4,5-*cde*][1,2]diselenine (**3b**) (284 mg, 0.65 mmol) in Et_2O (5 mL). The reaction mixture was allowed to stir at rt for 15 min. Compound **6b** precipitated during this time. The solid was filtered under vacuum and washed with Et_2O (3 × 15 mL). Seleninic anhydride **6b** was obtained as a white powder (300 mg, 95%) that required no further purification. mp 175–178 °C; ν_{max}(solid neat, ATR)/cm^{-1} 2956, 2927, 2854, 2394, 1457, 1405, 818, 797, 737, 671; δ_H (400 MHz, CD_3OD) 7.69 (2 H, dd, *J* = 7.1 and 1.4, H-3 and H-6 or H-1 and H-8), 7.67 (2 H, dd, *J* = 7.8 and 1.4, H-3 and H-6 or H-1 and H-8), 7.58 (2 H, t, *J* = 7.8, H-2 and H-7), 2.13–1.95 (4 H, m, H-14 and H-15), 0.99–0.84 (4 H, m, H-16 and H-17), 0.59 (6 H, t, *J* = 7.4, H-20 and H-21), 0.25–0.14 (4 H, m, H-18 and H-19); δ_C (101 MHz, CD_3OD) 155.5 (C, C-10 and C-13), 146.6 (C, C-11 and C-12), 139.8 (C, C-4 and C-5), 129.2 (CH, C-1 and C-8), 127.7 (CH, C-3 and C-6), 127.1 (CH, C-2 and C-7), 56.4 (C, C-9), 41.3

(CH_2, C-14 and C-15), 27.0 (CH_2, C-16 and C-17), 23.8 (CH_2, C-18 and C-19), 14.0 (CH_3, C-20 and C-21); ^{77}Se NMR (76 MHz, CD_3OD) 1175.9; *m/z* (ASAP$^+$) 485.0147 found ($[M+H]^+$, $C_{21}H_{25}{}^{80}Se_2O_3$ requires 485.0138).

Potassium 9,9-dimethyl-9*H*-fluorene-4,5-diseleninate (7a) and reformation of *trans*-seleninic anhydride (6a)

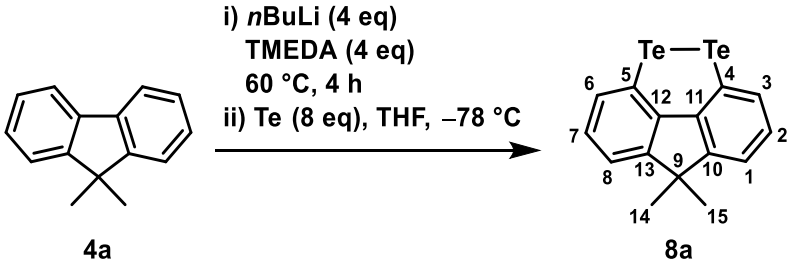


6a **7a** **6a**

Seleninic anhydride **6a** (50 mg, 0.125 mmol) was added as a single portion to a solution of KOH (56 mg, 1.00 mmol) in CD_3OD (4.0 mL). The reaction mixture was allowed to stir at rt for 2 h. NMR spectroscopy revealed the presence of **7a**. $HCl_{(aq)}$ (1 M, 5 mL) was added to the reaction mixture to afford a precipitate. The solid was filtered under vacuum and washed with Et_2O (3 × 10 mL). Seleninic anhydride **6a** was obtained as a white powder (43 mg, 86%) that required no further purification.

Data for **7a**: δ_H (400 MHz, CD_3OD) 8.04 – 7.84 (2 H, broad, *J* = 7.8, H-1 and H-8), 7.51 – 7.27 (4 H, broad, *J* = 7.6, H-2 and H-7), 1.38 – 1.08 (6 H, broad, H-14 and H-15); δ_C (101 MHz, CD_3OD) 154.9 (C, C-10 and C-13), 149.5 (C, C-11 and C-12), 134.9 (C, C-4 and C-5), 128.8 (CH, C-2 and C-7), 124.6 (CH, C-1 and C-8 or C-3 and C-6), 124.5 (CH, C-1 and C-8 or C-3 and C-6), 46.0 (C, C-9), 26.5 (CH_3, C-14 and C-15); ^{77}Se NMR (76 MHz, CD_3OD) 1136.0.

9,9-Dimethyl-9*H*-fluoreno[4,5-*cde*][1,2]ditellurine (8a)

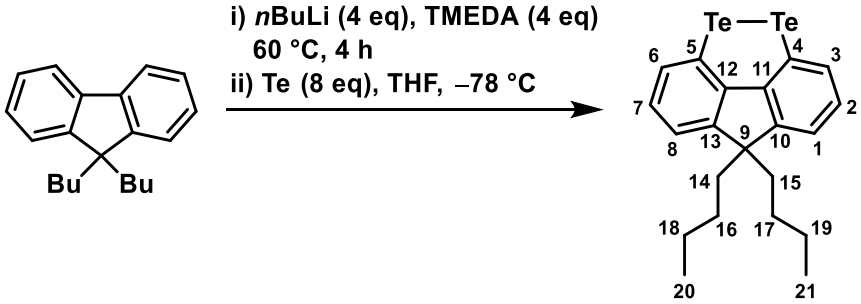


4a **8a**

Compound **8a** is a novel compound, prepared according to a modified literature procedure.[4] *n*BuLi (2.13 M in hexanes, 4.8 mL, 10.22 mmol) was added dropwise over 10 min to a solution of 9,9-dimethyl-9*H*-fluorene (**4a**) (500 mg, 2.57 mmol) in TMEDA (1.5 mL, 10.28 mmol). The reaction mixture was stirred at 60 °C for 4 h. The reaction mixture was cooled to –78 °C and THF (15 mL) was added followed by addition of tellurium (2.62 g, 20.5 mmol) as a single

portion. The cooling bath was removed and the reaction mixture was allowed to warm to rt and stirred for 16 h. H_2O (150 mL) was added and the mixture was extracted with Et_2O (3 × 50 mL). The combined organic layers were dried over $MgSO_4$, filtered and concentrated under reduced pressure. The crude material was purified by flash column chromatography (100% *n*-hexane). Ditelluride **8a** was obtained as a dark purple crystalline solid (284 mg, 25%). R_f 0.10 (*n*-hexane); mp degradation above 250 °C; ν_{max}(solid neat, ATR)/cm^{-1} 2953, 1556, 1451, 1417, 1396, 1183, 782, 727; δ_H (400 MHz, $CDCl_3$) 7.44 (2 H, dd, *J* = 7.4 and 1.1, H-1 and H-8), 7.20 (2 H, dd, *J* = 7.5 and 1.1, H-2 and H-7), 7.13 (2 H, t, *J* = 7.5, H-3 and H-6), 1.43 (6 H, s, H-14 and H-15); δ_C (101 MHz, $CDCl_3$) 155.0 (C, C-10 and C-13), 143.9 (C, C-11 and C-12), 132.6 (CH, C-3 and C-6), 129.5 (CH, C-2 and C-7), 122.7 (CH, C-1 and C-8), 94.2 (C, C-4 and C-5), 46.0 (C, C-9), 27.0 (CH_3, C-14 and C-15); *m/z* (ASAP$^+$) found 452.9141 (M$^{+\bullet}$, $C_{15}H_{12}{}^{130}Te_2$ requires 452.9143).

9,9-Dibutyl-9*H*-fluoreno[4,5-*cde*][1,2]ditellurine (8b)

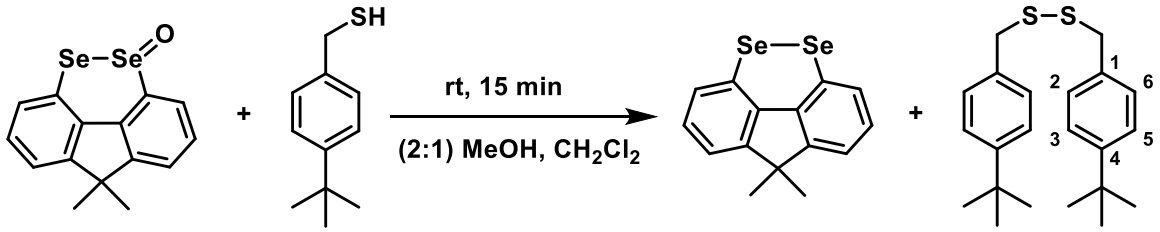


4b **8b**

Compound **8b** is a novel compound, prepared according to a modified literature procedure.[4] *n*BuLi (2.13 M in hexanes, 3.4 mL, 7.24 mmol) was added dropwise over 10 min at rt to a solution of 9,9-dibutyl-9*H*-fluorene (**4b**) (500 mg, 1.79 mmol) in TMEDA (1.0 mL, 7.16 mmol). The reaction mixture was allowed to stir at 60 °C for 4 h. The reaction mixture was cooled to –78 °C and THF (15 mL) was added followed by addition of tellurium (1.83 g, 14.32 mmol) as a single portion. The cooling bath was removed and the reaction mixture was allowed to warm to rt and left to stir for 16 h. H_2O (150 mL) was added and the mixture was extracted with Et_2O (3 × 50 mL). The combined organic layers were dried over $MgSO_4$, filtered and concentrated under reduced pressure. The crude material was purified by flash column chromatography (100% *n*-hexane) yielding ditelluride **8b** as a dark purple crystalline solid (104 mg, 11%). R_f 0.10 (*n*-hexane); mp degradation above 250 °C; ν_{max}(solid neat, ATR)/cm^{-1} 2953, 1556, 1451, 1417, 1396, 1183, 782, 727; δ_H (400 MHz, $CDCl_3$) 7.44 (2 H, dd, *J* = 7.4, 1.1, H-1 and 8), 7.14–7.10 (4 H, stack, H-2, H-3, H-6 and H-7), 1.92–1.88 (4 H, m, H-14 and H-15), 1.06 (4 H, sext, *J* = 8.0, H-16 and H-17), 0.67 (6 H, t, *J* = 7.3, H-20 and H-21), 0.59–0.51 (4 H, m, H-18

and H-19); δ_C (101 MHz, $CDCl_3$) 152.2 (C, C-10 and C-13), 145.9 (C, C-11 and C-12), 132.5 (CH, C-3 and C-6), 129.2 (CH, C-2 and C-7), 122.9 (CH, C-1 and C-8), 90.0 (C, C-4 and C-5), 54.1 (C, C-9), 40.2 (CH_2, C-14 and C-15), 25.9 (CH_2, C-16 and C-17), 23.1 (CH_2, C-18 and C-19), 13.9 (CH_3, C-20 and C-21); *m/z* (ASAP$^+$) found 536.0015 (M^+, $C_{21}H_{24}{}^{130}Te_2$ requires 536.0005).

Reaction of 9,9-dimethyl-9*H*-fluoreno[4,5-*cde*][1,2]diselenine 4-oxide (5a) with (4-(*tert*-butyl)phenyl)methanethiol (10)



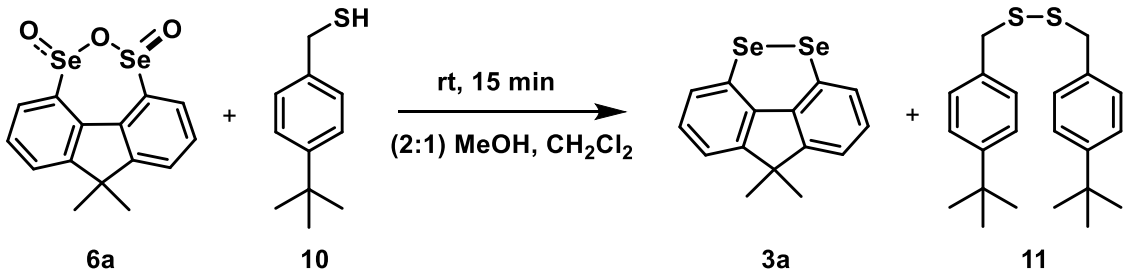
5a **10** **3a** **11**

(4-(*tert*-Butyl)phenyl)methanethiol (**10**) (720 mg, 3.99 mmol) was added to a solution of 9,9-dimethyl-9*H*-fluoreno[4,5-*cde*][1,2]diselenine 4-oxide (**5a**) (150 mg, 0.41 mmol) in a 2:1 mixture of MeOH (18.6) and CH_2Cl_2 (9.3 mL) and stirred at rt for 15 min. The solvent was removed under reduced pressure. The residue was purified by flash column chromatography (9:1, hexane: Et_2O) to firstly afford 9*H*-fluoreno[4,5-*cde*][1,2]diselenine (**3a**) (122 mg, 85%), followed by 1,2-bis(4-(*tert*-butyl)benzyl)disulfane **11** (133 mg, 91%), followed by thiol **10** (540 mg, 75% recovered thiol, 94% based on theoretical consumption of 2 equivalents of thiol, scheme S1).

Data for disulfide **11**: R_f 0.30 (*n*-hexane); mp 63–64 °C; δ_H (400 MHz, $CDCl_3$) 7.25 (4 H, d, *J* = 8.3, H-3 and H-5), 7.09 (4 H, d, *J* = 8.4, H-2 and H-6), 3.51 (4H, s, CH_2), 1.22 (18 H, s, CH_3); δ_C (101 MHz, $CDCl_3$) 150.5 (C, C-4), 134.3 (C, C-1), 129.2 (CH, C-2 and C-6), 125.5 (CH, C-3 and C-5), 43.0 (CH_2), 34.6 (C), 31.5 (CH_3); *m/z* (ASAP$^+$) found 376.2133 ([M + NH_4]$^+$, $C_{22}H_{34}NS_2$ requires 376.2133).

Analytical data are in agreement with literature values.[9]

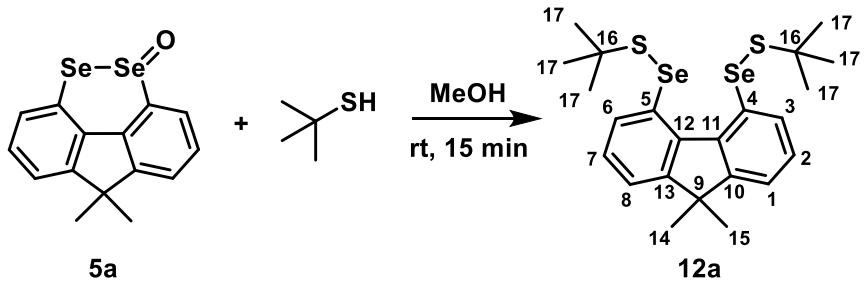
Reaction of *trans*-seleninic anhydride 6a with (4-(*tert*-butyl)phenyl)methanethiol (10)



6a 10 3a 11

(4-(*tert*-Butyl)phenyl)methanethiol (**10**) (676 mg, 3.75 mmol) was added to a solution of *trans*-seleninic anhydride **6a** (150 mg, 0.38 mmol) in a 2:1 mixture of MeOH and CH_2Cl_2 (26 mL) and stirred at rt for 15 min. The solvent was removed under reduced pressure. The residue was purified by flash column chromatography (9:1, hexane: Et_2O) to firstly afford 9*H*-fluoreno[4,5-*cde*][1,2]diselenine (**3a**) (126 mg, 96%), followed by 1,2-bis(4-(*tert*-butyl)benzyl)disulfane **11** (357 mg, 88%), followed by thiol **10** (269 mg, 40% recovered thiol, 100% based on theoretical consumption of 6 equivalents of thiol, scheme S1).

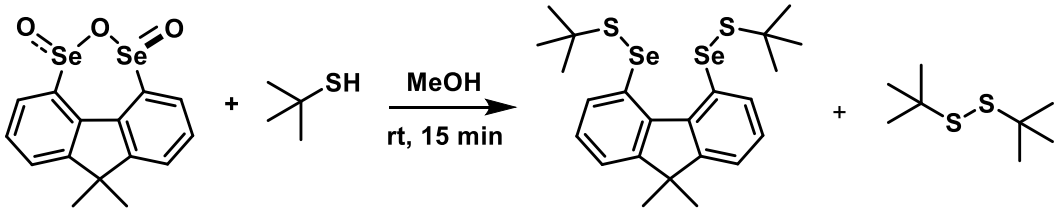
Reaction of 9,9-dimethyl-9*H*-fluoreno[4,5-*cde*][1,2]diselenine 4-oxide 5a with *tert*-butylthiol



5a 12a

tert-Butylthiol (414 mg, 4.59 mmol) was added to a solution of 9,9-dimethyl-9*H*-fluoreno[4,5-*cde*][1,2]diselenine 4-oxide (**5a**) (169 mg, 0.46 mmol) in MeOH (23.0 mL) at 0 °C and stirred at 0 °C for 15 min. The solvent was removed under reduced pressure. The residue was purified by flash column chromatography (hexane 100%) to firstly afford *tert*-butylthiol (300 mg, 72% recovered thiol, 91% based on theoretical consumption of 2 equivalents of thiol, Scheme S2) followed by **12a** as a beige gel (230 mg, 94%). R_f 0.32 (*n*-hexane); δ_H (400 MHz, $CDCl_3$) 8.00 (2 H, dd, *J* = 6.4, 2.5, H-3 and H-6), 7.39–7.34 (4H, stack, H-1, H-2, H-7 and H-8), 1.41 (6 H, s, H-14 and H-15), 0.97 (18 H, s, H-17); δ_C (101 MHz, $CDCl_3$) 153.9 (C, C-10 and C-13), 147.3 (C, C-11 and C-12), 135.6 (CH, C-2 and C-7), 129.2 (CH, C-1 and C-8 or C-3 and C-6), 128.2 (C, C-4 and C-5), 121.9 (CH, C-1 and C-8 or C-3 and C-6), 47.0 (C, C-16), 46.8 (C, C-9), 30.5 (CH_3, C-17), 27.9 (2 x CH_3, C-14 and C-15); ^{77}Se NMR (76 MHz, $CDCl_3$) 398.6; *m/z* (AP^+) found 530.0121 ($M^{+\bullet}$, $C_{23}H_{30}S_2{}^{80}Se_2$ requires 530.0119).

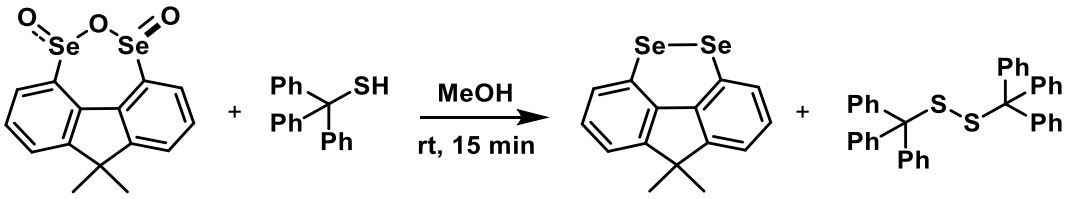
Reaction of *trans*-seleninic anhydride 6a with *tert*-butylthiol



6a 12a 13

tert-Butylthiol (252 mg, 2.79 mmol) was added to a solution of *trans*-seleninic anhydride **6a** (112 mg, 0.28 mmol) in MeOH (14.0 mL) at 0 °C and allowed to stir at 0 °C for 15 min. The solvent was removed under reduced pressure. The residue was purified by flash column chromatography (*n*-hexane 100%) to firstly afford *tert*-butyl thiol (100 mg, 40% recovered thiol, 100% based of theoretical consumption of 6 equivalents of thiol, Scheme S2) followed by **12a** as a beige gel (140 mg, 94%) and *tert*-butyl disulfide (99 mg, 99% based on theoretical formation of 2 equivalents of disulfide, Scheme S2).

Reaction of *trans*-seleninic anhydride 6a with triphenylmethanethiol (15)



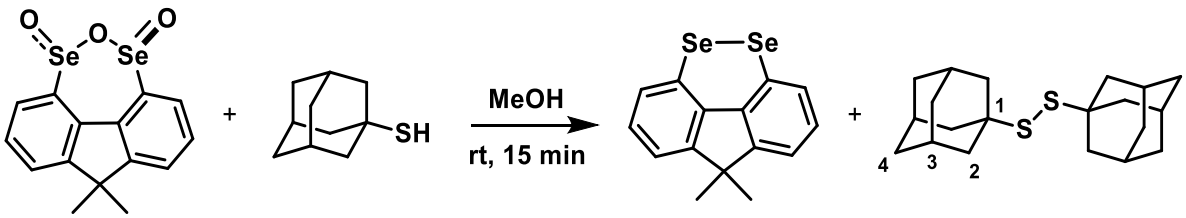
6a 15 3a 16

Triphenylmethanethiol (**15**) (692 mg, 2.50 mmol) was added to a solution of *trans*-seleninic anhydride **6a** (100 mg, 0.25 mmol) in MeOH (12.0 mL) and allowed to stir at rt for 15 min. The solvent was removed under reduced pressure. The residue was purified by flash column chromatography (hexane 100%) to firstly afford 9*H*-fluoreno[4,5-*cde*][1,2]diselenine (**3a**) (86 mg, 98%), followed by trityl disulfide **16** (397 mg, 0.72 mmol, 96%), followed by triphenylmethanethiol (**15**) (242 mg, 35% recovered thiol, 88% based on theoretical consumption of 6 equivalents of thiol, scheme S1).

Data for disulfide **16**: R_f 0.30 (*n*-hexane); mp 150–152 °C; δ_H (400 MHz, $CDCl_3$) 7.35–7.32 (30 H, m); δ_C (101 MHz, $CDCl_3$) 143.8 (C), 129.4 (CH), 127.8 (CH), 126.9 (CH), 73.5 (C).

Analytical data are in agreement with literature values.[10]

Reaction of *trans*-seleninic anhydride 6a with 1-adamantanethiol (17)



6a **17** **3a** **18**

1-Adamantanethiol (**17**) (210 mg, 1.25 mmol) was added to a solution of *trans*-seleninic anhydride **6a** (50 mg, 0.12 mmol) in MeOH (6.0 mL) and allowed to stir at rt for 15 min. The solvent was removed under reduced pressure. The residue was purified by flash column chromatography (hexane 100%) to firstly afford 9*H*-fluoreno[4,5-*cde*][1,2]diselenine (**3a**) (39 mg, 89%), followed by Di-*tert*-adamantyl disulfide **18** (97 mg, 0.29 mmol, 77%), followed by 1-adamantanethiol **17** (74 mg, 35% recovered thiol, 89% based on theoretical consumption of 6 equivalents of thiol, scheme S1).

Disulfide **18**; R_f 0.36 (*n*-hexane); mp 223–224 °C; δ_H (400 MHz, $CDCl_3$) 2.06 (3 H, s, H-3), 1.82 (6 H, s, H-2), 1.70–1.63 (6 H, m, H-4); δ_C (101 MHz, $CDCl_3$) 47.4 (C, C-1), 43.2 (CH_2, C-2), 36.2 (CH_2, C-4), 30.1 (CH, C-3)

Analytical data are in agreement with literature values.[11]

X-ray crystallography

Crystal Data for 9,9-dimethyl-9*H*-fluoreno[4,5-*cde*][1,2]diselenine (3a): $C_{15}H_{12}Se_2$ (*M* =350.17 g/mol): tetragonal, space group I-4 (no. 82), *a* = 17.1924(2) Å, *c* = 8.8177(2) Å, *V* = 2606.32(8) Å^3, *Z* = 8, *T* = 100.00(10) K, μ(CuKα) = 6.862 mm^{-1}, *Dcalc* = 1.785 g/cm^3, 12547 reflections measured (7.272° ≤ 2Θ ≤ 149.15°), 2627 unique (R_{int} = 0.0198, R_{sigma} = 0.0134) which were used in all calculations. The final R_1 was 0.0146 (I > 2σ(I)) and wR_2 was 0.0369 (all data).

Crystals of **3a** were obtained by slow evaporation from dichloromethane solution.

Crystal Data for 9,9-dibutyl-9H-fluoreno[4,5-cde][1,2]diselenine (3b): $C_{21}H_{24}Se_2$ (*M* =434.32 g/mol): triclinic, space group P1 (no. 1), *a* = 9.1936(2) Å, *b* = 12.7735(3) Å, *c* = 17.6722(4) Å, *α* = 73.363(2)°, *β* = 79.2638(18)°, *γ* = 71.888(2)°, *V* = 1879.11(8) Å^3, *Z* = 4, *T* = 100.01(10) K, μ(Cu Kα) = 4.875 mm^{-1}, *Dcalc* = 1.535 g/cm^3, 32449 reflections measured (5.25° ≤ 2Θ ≤ 147.836°), 14219 unique (R_{int} = 0.0347, R_{sigma} = 0.0397) which were used in all calculations. The final R_1 was 0.0292 (I > 2σ(I)) and wR_2 was 0.0748 (all data).

Crystals of **3b** were obtained by slow evaporation from hexane solution.

The datasets were measured on an Agilent SuperNova diffractometer using an Atlas detector. The data collections were driven and processed and numerical absorption corrections based on gaussian integration over a multifaceted crystal model were applied using CrysAlisPro.[S1] The structure of **9,9-dimethyl-9*H*-fluoreno[4,5-*cde*][1,2]diselenine (3a)** was solved using ShelXT,[S2] that of **9,9-dibutyl-9*H*-fluoreno[4,5-*cde*][1,2]diselenine (3b)** was solved using ShelXS, [S3] and both structures were refined by a full-matrix least-squares procedure on F^2 in ShelXL.[S4] All non-hydrogen atoms were refined with anisotropic displacement parameters. All hydrogen atoms were added at calculated positions and refined by use of a riding model with isotropic displacement parameters based on the equivalent isotropic displacement parameter (U_{eq}) of the parent atom.

The structure of **9,9-dibutyl-9*H*-fluoreno[4,5-*cde*][1,2]diselenine** (**3b**) has been refined as an inversion twin with the refined percentage ratio of enantiomers being 69.6 (15): 30.4 (15). The structure contains four crystallography-independent molecules. In Molecule 4 the selenium atoms Se(31)-Se(32)/Se(1')-Se(2') are disordered over two positions are a refined percentage occupancy ratio of 62.7 (2): 37.3 (2).

Figures and reports were produced using OLEX2.[S5] The CIFs for the crystal structures of **9,9-dimethyl-9*H*-fluoreno[4,5-*cde*][1,2]diselenine (3a)** and **9,9-dibutyl-9*H*-fluoreno[4,5-*cde*][1,2]diselenine (3b)** and have been deposited with the CCDC and have been given the deposition numbers CCDC 2099301 and CCDC 1470801. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

Crystallography references

[S1] CrysAlisPro, Agilent Technologies, Version 1.171.37.35, **2014** and Version 1.171.39.46, **2018**.
[S2] G. M. Sheldrick, *Acta Cryst.* **2008**, *A64*, 112-122.
[S3] G. M. Sheldrick, *Acta Cryst.* **2015**, *A71*, 3-8.
[S4] G. M. Sheldrick, *Acta Cryst.* **2015**, *C71*, 3-8.
[S5] O. V. Dolomanov, L. J. Bourhis, R. J. Gildea, J. A. K. Howard, H. Puschmann, *J. Appl. Crystallogr.* **2009**, *42*, 339-341.

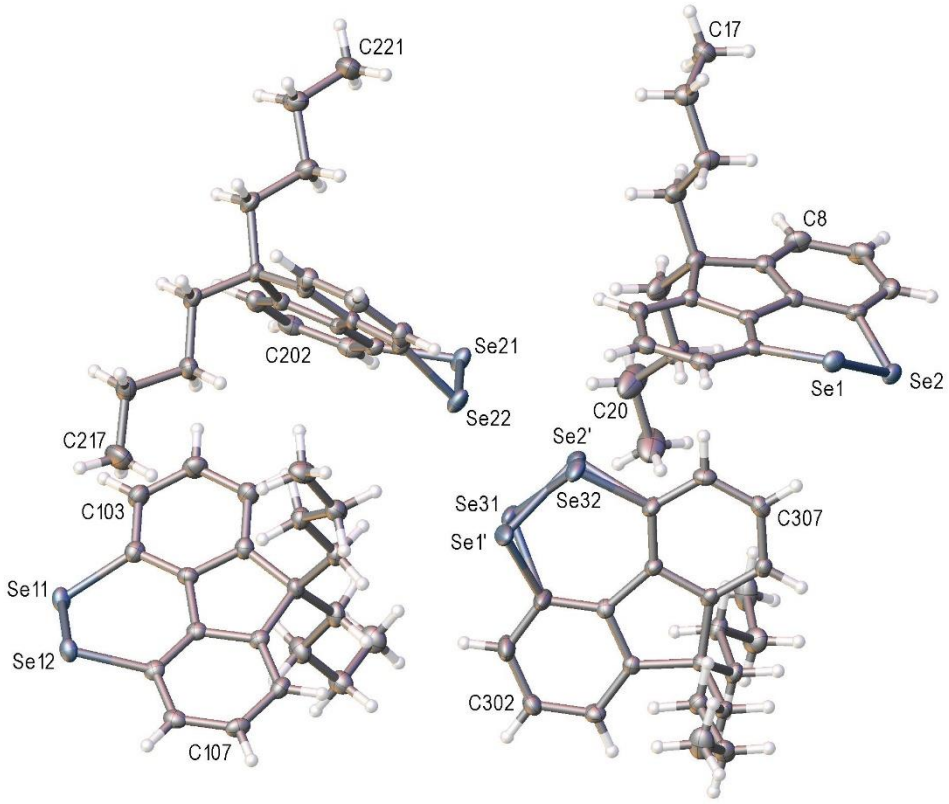


Figure S1 Crystal structure of **9,9-dibutyl-9*H*-fluoreno[4,5-*cde*][1,2]diselenine (3b)** with ellipsoids drawn at the 50 % probability level. The structure contains four crystallography-independent molecules. In molecule 4 the selenium atoms Se(31)-Se(32)/Se(1')-Se(2') are disordered over two positions are a refined percentage occupancy ratio of 62.7 (2): 37.3 (2).

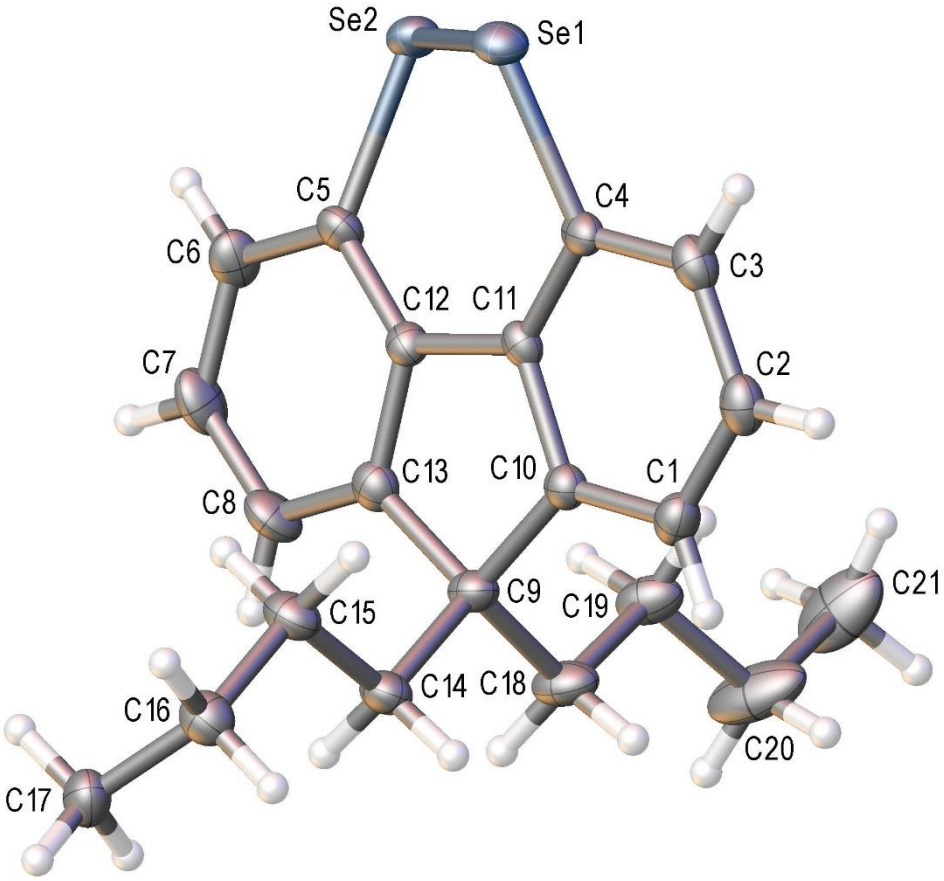


Figure S2 Crystal structure of molecule 1 of **9,9-dibutyl-9*H*-fluoreno[4,5-*cde*][1,2]diselenine (3b)** with ellipsoids drawn at the 50 % probability level. The structure contains four crystallography-independent molecules of which only one is shown for clarity.

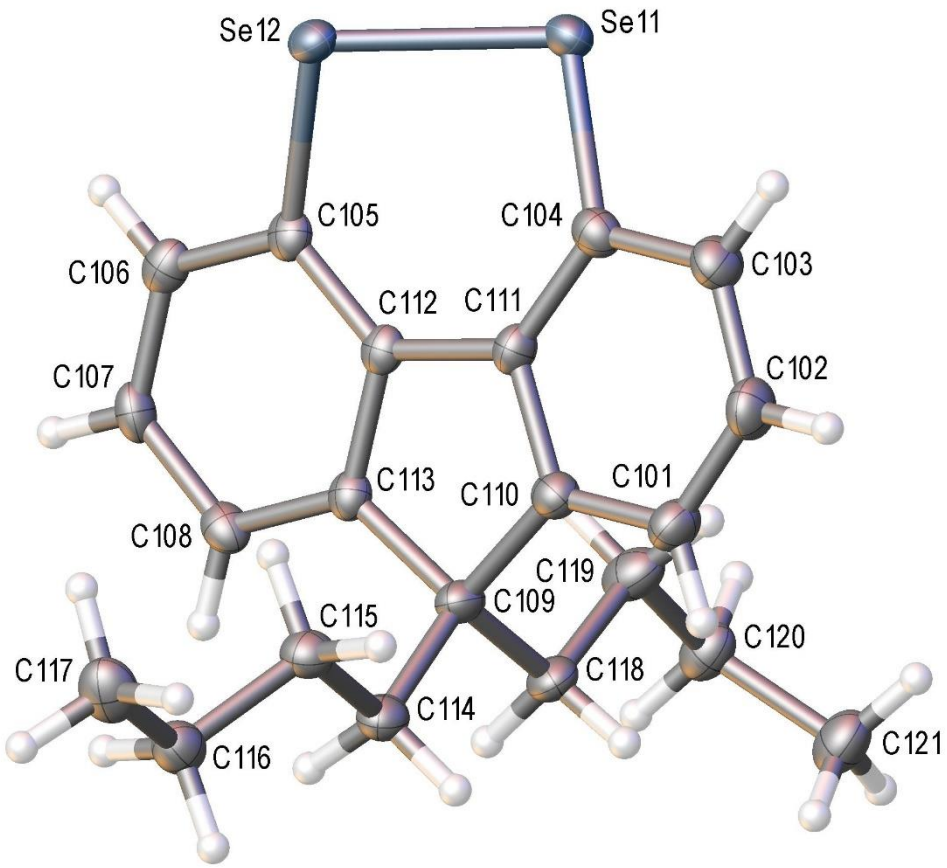


Figure S3 Crystal structure of molecule 2 of **9,9-dibutyl-9*H*-fluoreno[4,5-*cde*][1,2]diselenine (3b)** with ellipsoids drawn at the 50 % probability level. The structure contains four crystallography-independent molecules of which only one is shown for clarity.

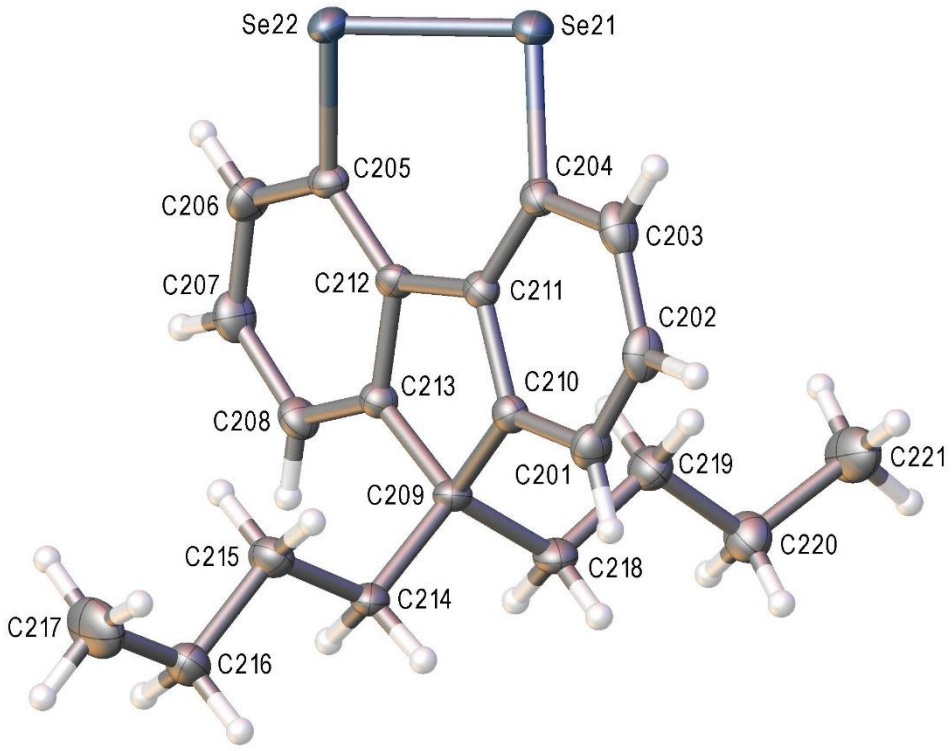


Figure S4 Crystal structure of molecule 3 of **9,9-dibutyl-9*H*-fluoreno[4,5-*cde*][1,2]diselenine (3b)** with ellipsoids drawn at the 50 % probability level. The structure contains four crystallography-independent molecules of which only one is shown for clarity.

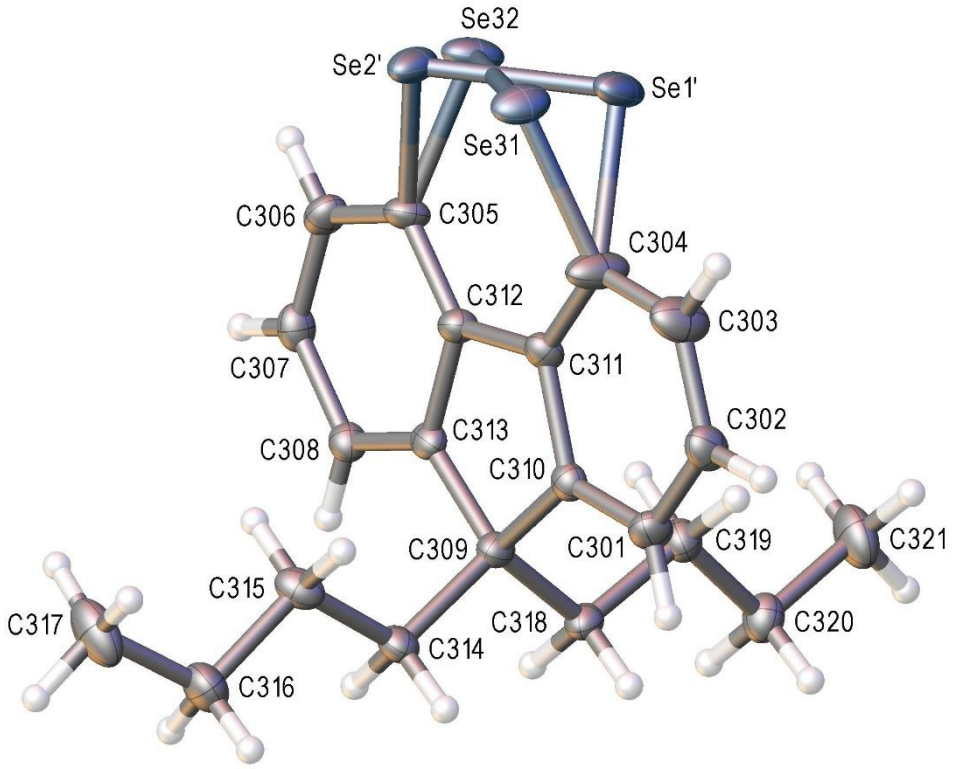


Figure S5 Crystal structure of molecule 4 of **9,9-dibutyl-9*H*-fluoreno[4,5-cde][1,2]diselenine (3b)** with ellipsoids drawn at the 50 % probability level. The structure contains four crystallography-independent molecules of which only one is shown for clarity. The selenium atoms Se(31)-Se(32)/Se(1')-Se(2') are disordered over two positions are a refined percentage occupancy ratio of 62.7 (2): 37.3 (2).

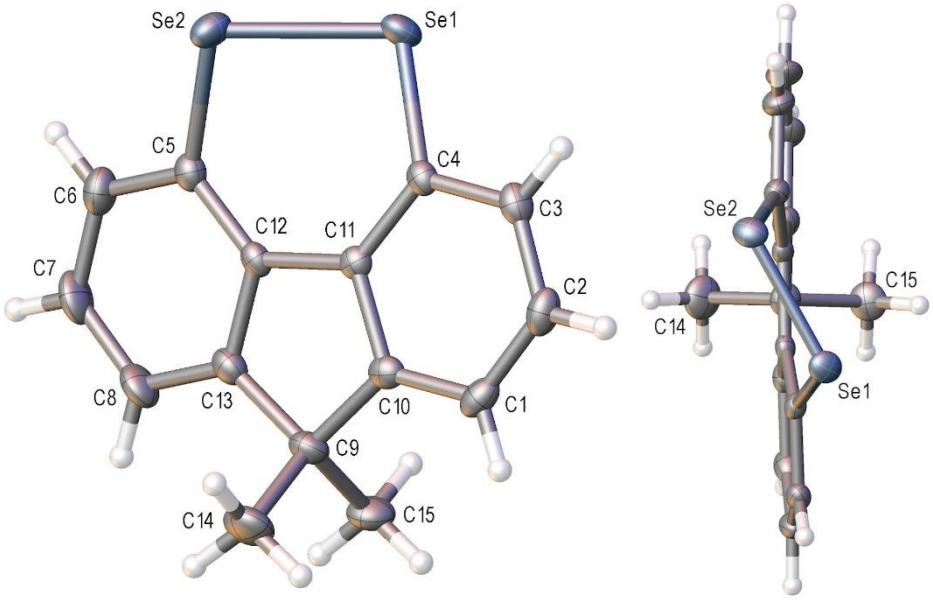


Figure S6 Two views of the crystal structure of **9,9-dimethyl-9*H*-fluoreno[4,5-*cde*][1,2]diselenine (3a)** with ellipsoids drawn at the 50 % probability level.

3b	**Molecule 1**	**Molecule 2**	**Molecule 3**	**Molecule 4**	**Average** All Molecules	**Average** Molecules 1-3
C(4)-Se(1)-Se(2)-C(5)	39.9(3)	–42.2(2)	–44.7(3)	43.2(3) –50.2(2)	44.0(35)*	42.3(20)*
C4-C11-C12-C5	10.6(9)	–9.8(9)	–13.4(8)	2.2(9)	9(4)	11.3(15)

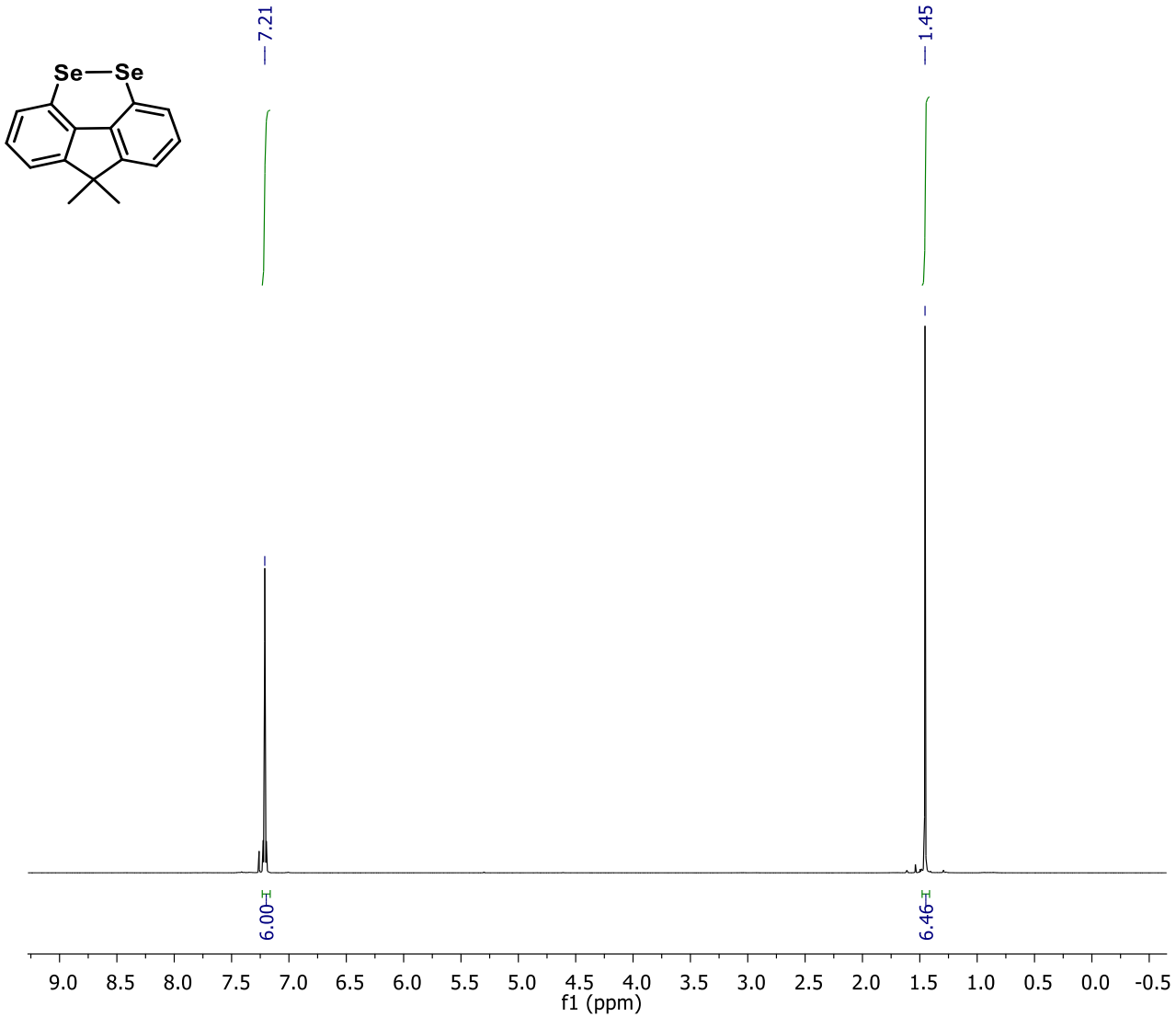
Table S1: Dihedral angles (°) in **3b**. The structure contains four crystallography-independent molecules, with equivalent parameters given and labelled as in Molecule 1. In Molecule 4 the selenium atoms Se(31)-Se(32)/Se(1')-Se(2') are disordered over two positions. Average values from all four molecules and also for Molecules 1-3 are given. *In the case of torsion angles and differences the modulus of the parameters are used to calculate the average values.

Compound	**2a**	**2b**	**3a**	**3b**[a]	**9**[b]
CCDC Numbers **CCDC Refcodes**	236815 ZZZBKS01	2011ncs0489	2099301	1470801	207/49 TAKFAM01
C-Se-Se-C	–2.28(13)	–1.50(7)	–41.35(12)	42.3(20)*	59.0(3) –59.0(4) –57.0(4)
C4-C11-C12-C5				11.3(15)	

Table S2: Dihedral angles (°) in diselenides **2a**, **2b**, **3a, 3b** and **9**. *In the case of torsion angles and differences the modulus of the parameters are used to calculate average values. [a]For **3b** the average value calculated from Molecules 1-3 for each parameter is given (see Table S1). [b]**9** contains three crystallographically-independent molecules, two of which lie on a 2-fold rotation axis.

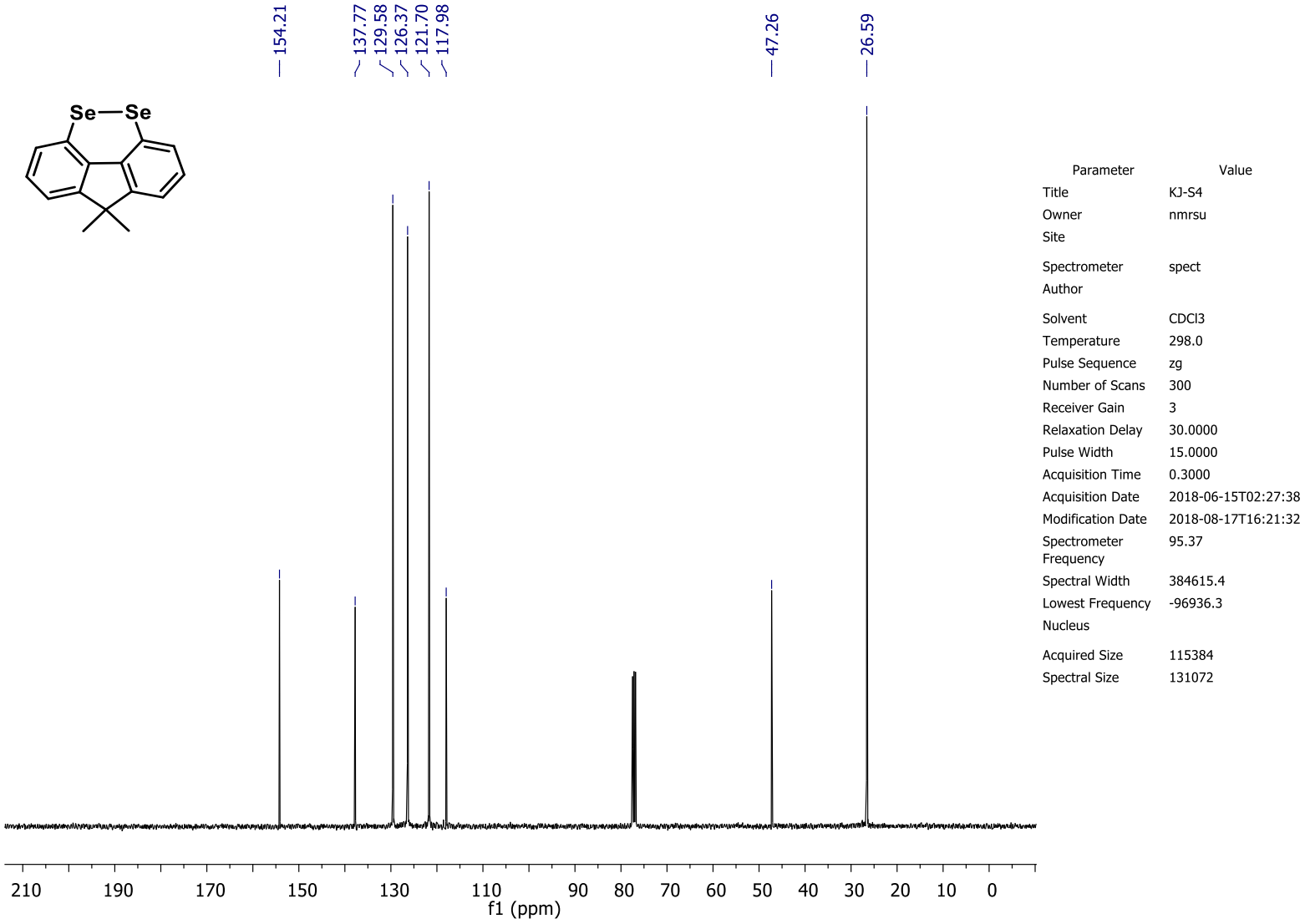
Copies of NMR spectra

9,9-Dimethyl-9*H*-fluoreno[4,5-*cde*][1,2]diselenine (**3a**): ^{1}H NMR spectrum in $CDCl_3$ (400 MHz, 296 K)



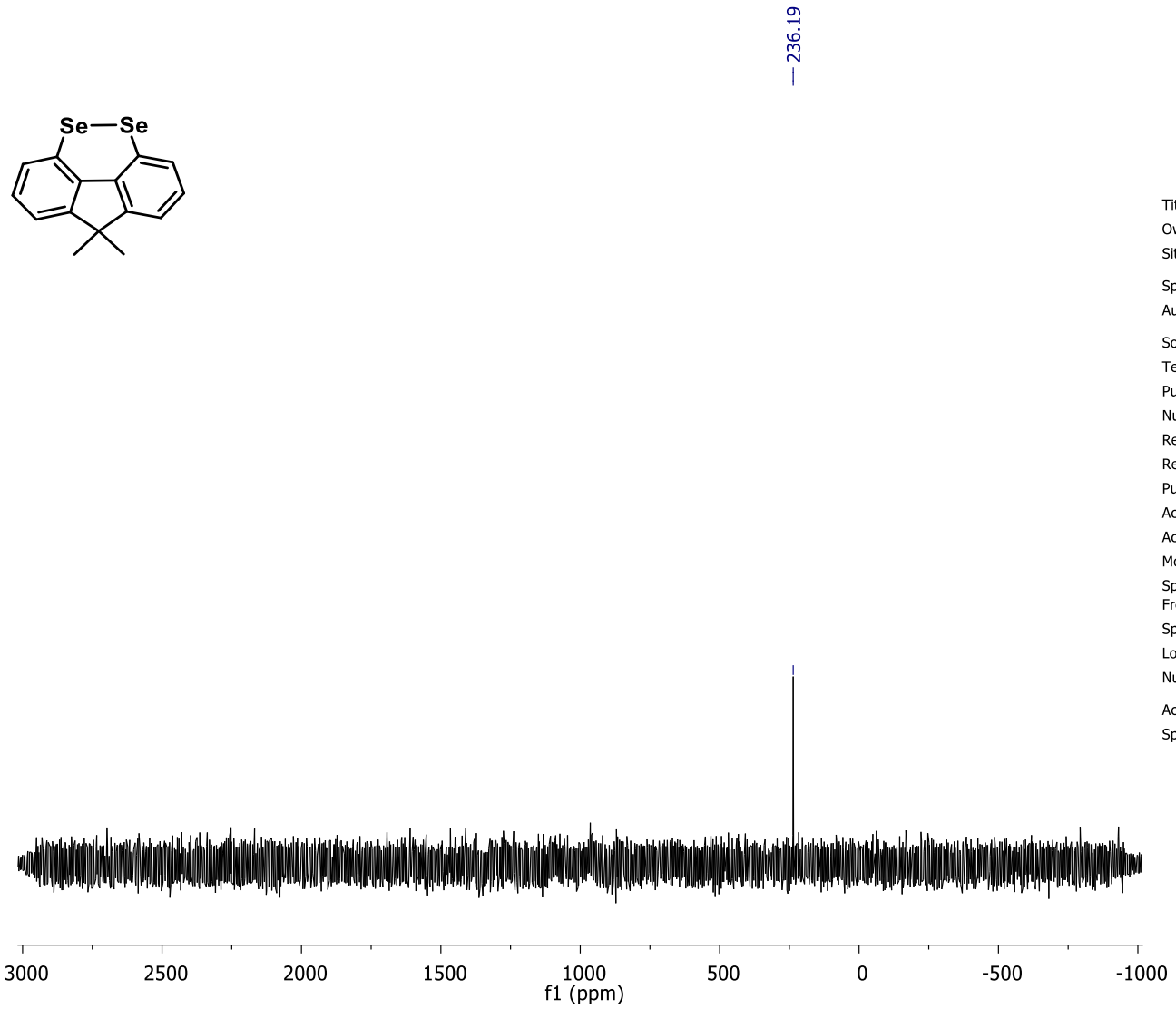
Parameter	Value
Title	KJ-5-SE ME
Comment	
Origin	Bruker BioSpin GmbH
Owner	Facility
Site	
Spectrometer	spect
Author	
Solvent	CDCl3
Temperature	295.8
Pulse Sequence	zg30
Number of Scans	32
Receiver Gain	203
Relaxation Delay	1.5000
Pulse Width	11.0600
Acquisition Time	1.9923
Acquisition Date	2018-09-20T05:17:00
Modification Date	2018-09-21T11:26:06
Spectrometer Frequency	400.13
Spectral Width	8223.7
Lowest Frequency	-1720.7
Nucleus	1H
Acquired Size	16384
Spectral Size	32768

9,9-Dimethyl-9H-fluoreno[4,5-*cde*][1,2]diselenine (**3a**): ^{13}C NMR spectrum in $CDCl_3$ (101 MHz, 296 K)



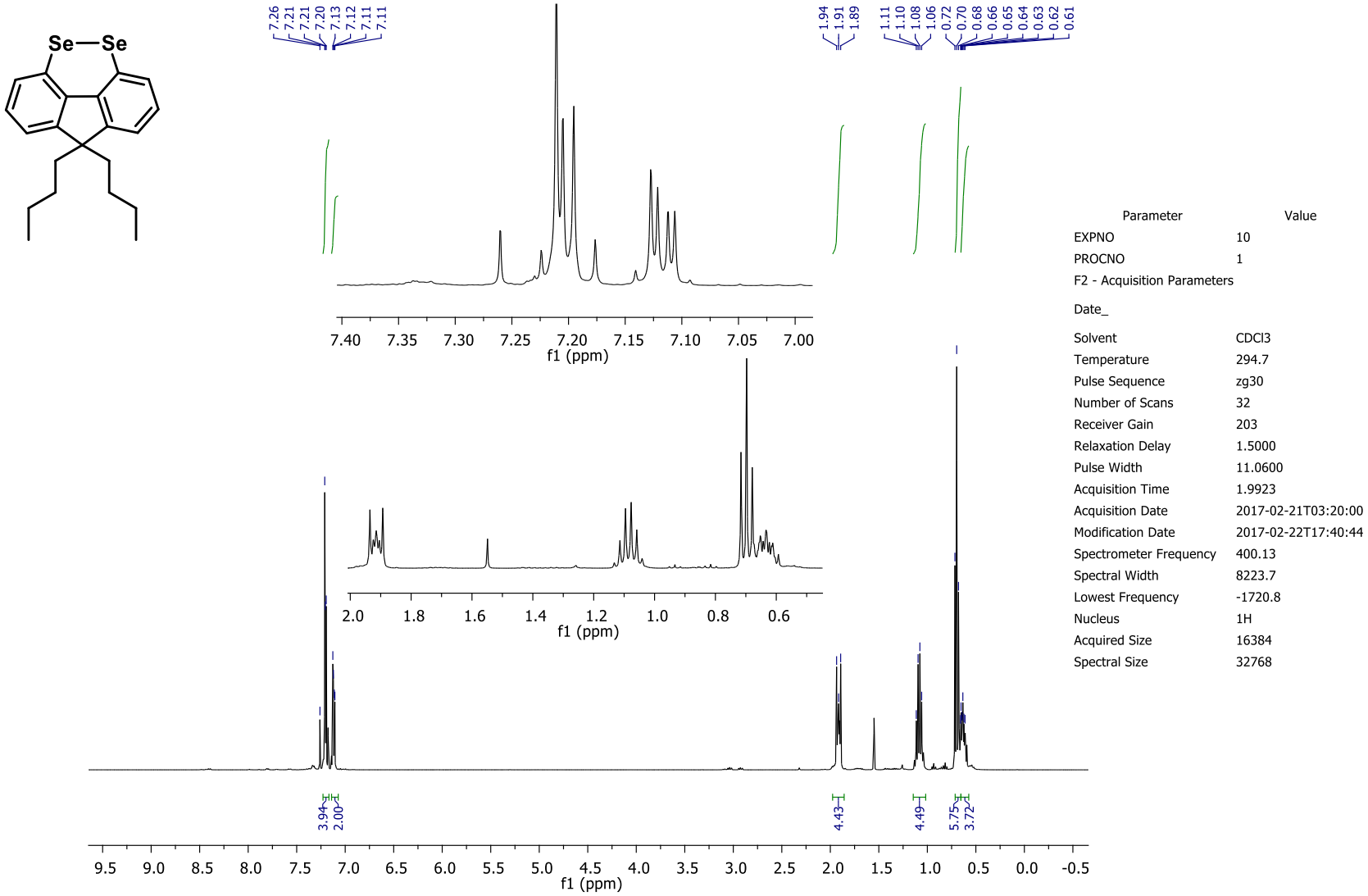
Parameter	Value
Title	KJ-S4
Owner	nmrsu
Site	
Spectrometer	spect
Author	
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zg
Number of Scans	300
Receiver Gain	3
Relaxation Delay	30.0000
Pulse Width	15.0000
Acquisition Time	0.3000
Acquisition Date	2018-06-15T02:27:38
Modification Date	2018-08-17T16:21:32
Spectrometer Frequency	95.37
Spectral Width	384615.4
Lowest Frequency	-96936.3
Nucleus	
Acquired Size	115384
Spectral Size	131072

9,9-Dimethyl-9H-fluoreno[4,5-*cde*][1,2]diselenine (**3a**): ^{77}Se NMR spectrum in $CDCl_3$ (95 MHz, 296 K)



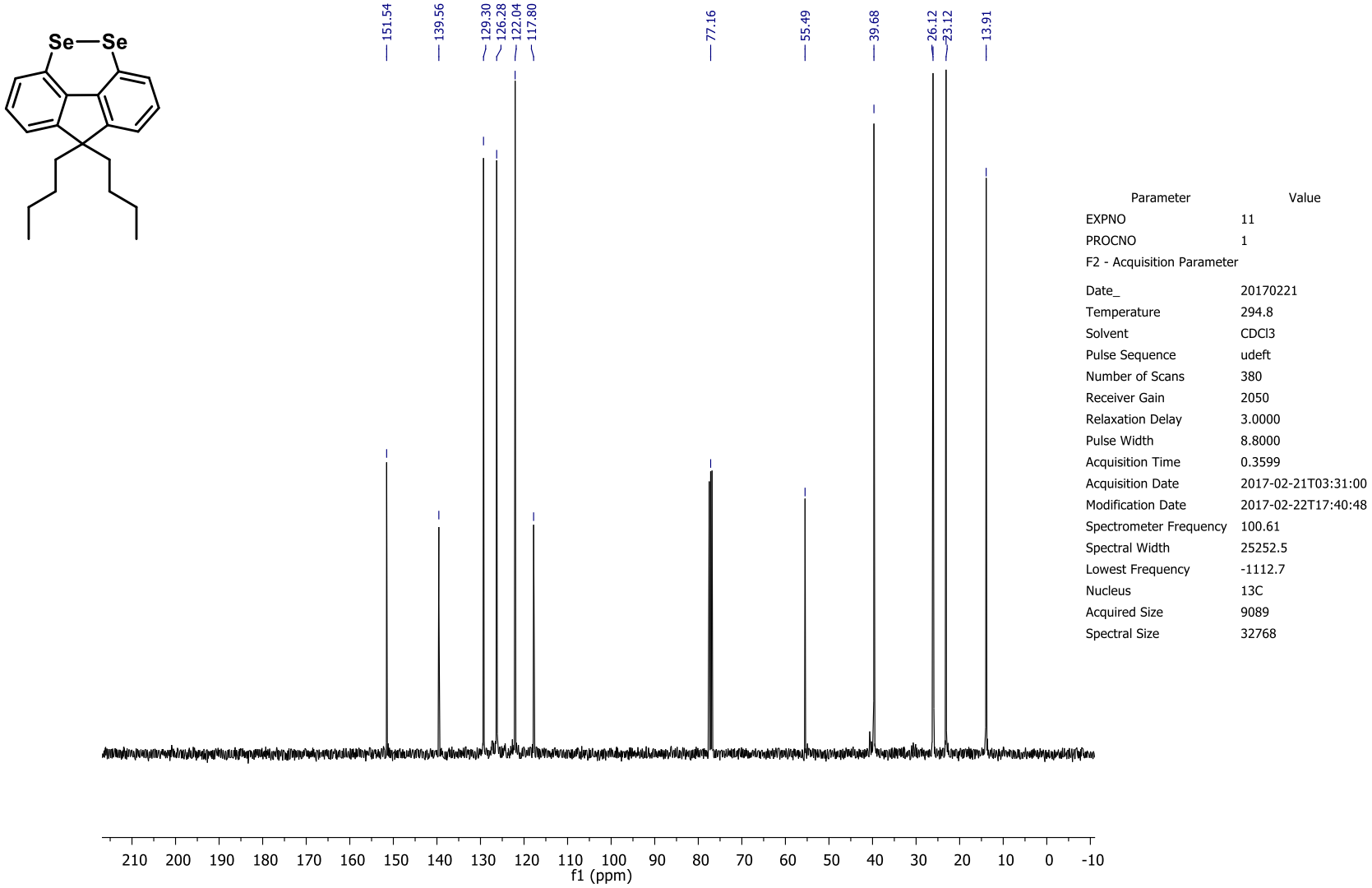
Parameter	Value
Title	KJ-S4
Owner	nmrsu
Site	
Spectrometer	spect
Author	
Solvent	CDCl3
Temperature	298.0
Pulse Sequence	zg
Number of Scans	300
Receiver Gain	3
Relaxation Delay	30.0000
Pulse Width	15.0000
Acquisition Time	0.3000
Acquisition Date	2018-06-15T02:27:38
Modification Date	2018-08-17T16:21:32
Spectrometer Frequency	95.37
Spectral Width	384615.4
Lowest Frequency	-96936.3
Nucleus	
Acquired Size	115384
Spectral Size	131072

9,9-Dibutyl-9*H*-fluoreno[4,5-*cde*][1,2]diselenine (**3b**): ^{1}H NMR spectrum in $CDCl_3$ (400 MHz, 295 K)



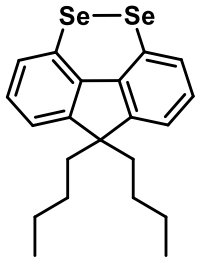
Parameter	Value
EXPNO	10
PROCNO	1
F2 - Acquisition Parameters	
Date_	
Solvent	CDCl3
Temperature	294.7
Pulse Sequence	zg30
Number of Scans	32
Receiver Gain	203
Relaxation Delay	1.5000
Pulse Width	11.0600
Acquisition Time	1.9923
Acquisition Date	2017-02-21T03:20:00
Modification Date	2017-02-22T17:40:44
Spectrometer Frequency	400.13
Spectral Width	8223.7
Lowest Frequency	-1720.8
Nucleus	1H
Acquired Size	16384
Spectral Size	32768

9,9-Dibutyl-9*H*-fluoreno[4,5-*cde*][1,2]diselenine (**3b**): ^{13}C NMR spectrum in $CDCl_3$ (101 MHz, 295 K)



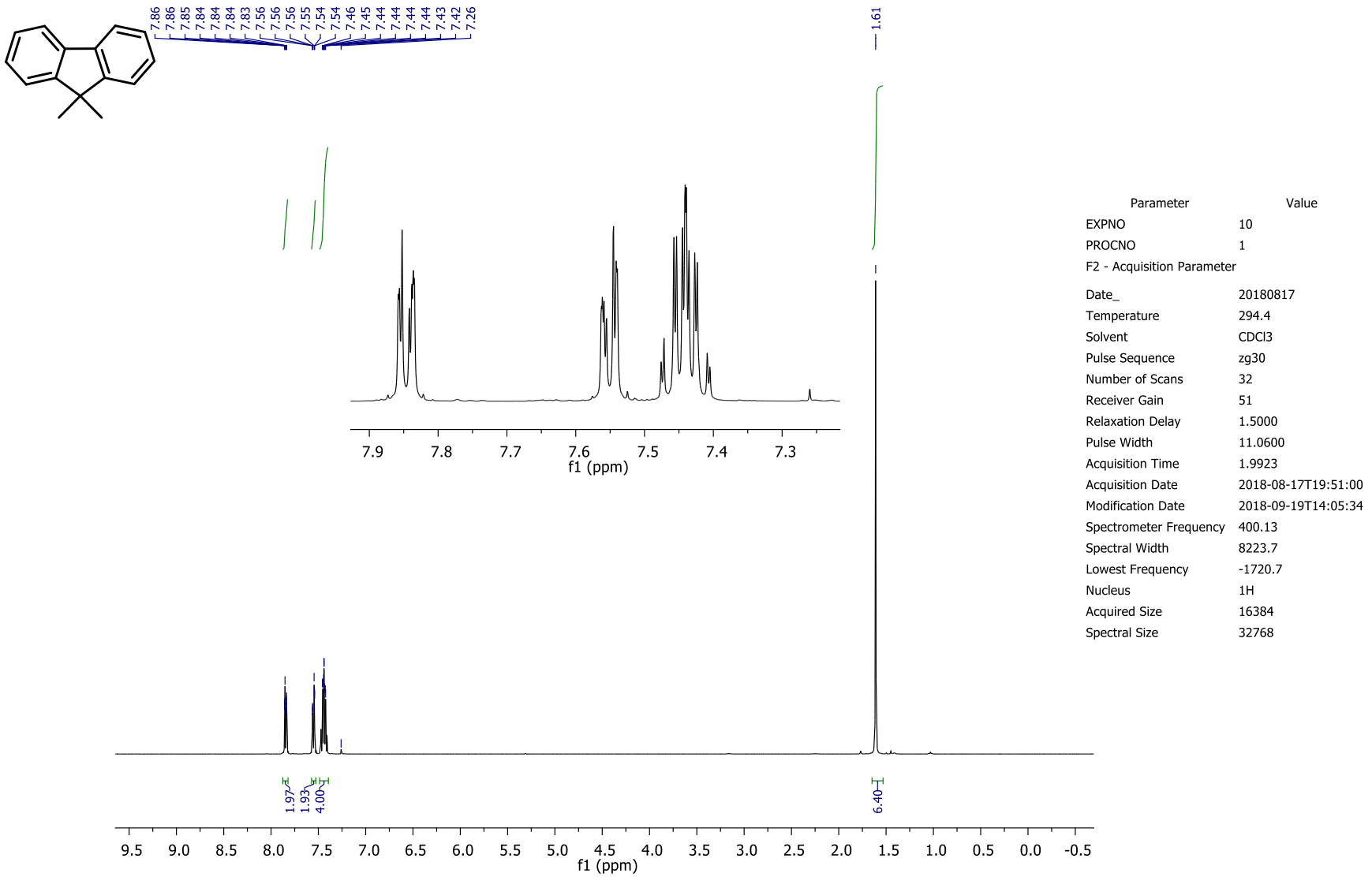
Parameter	Value
EXPNO	11
PROCNO	1
F2 - Acquisition Parameter	
Date_	20170221
Temperature	294.8
Solvent	CDCl3
Pulse Sequence	udeft
Number of Scans	380
Receiver Gain	2050
Relaxation Delay	3.0000
Pulse Width	8.8000
Acquisition Time	0.3599
Acquisition Date	2017-02-21T03:31:00
Modification Date	2017-02-22T17:40:48
Spectrometer Frequency	100.61
Spectral Width	25252.5
Lowest Frequency	-1112.7
Nucleus	13C
Acquired Size	9089
Spectral Size	32768

9,9-Dibutyl-9*H*-fluoreno[4,5-*cde*][1,2]diselenine (**3b**): ^{77}Se NMR spectrum in $CDCl_3$ (95 MHz, 298 K)



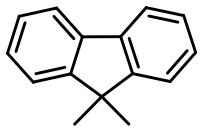
Parameter	Value
EXPNO	2
PROCNO	1
F2 - Acquisition Parameter	
Date_	20180604
Temperature	298.0
Solvent	CDCl3
Pulse Sequence	zg
Number of Scans	300
Receiver Gain	3
Relaxation Delay	30.0000
Pulse Width	15.0000
Acquisition Time	0.3000
Acquisition Date	2018-06-04T15:45:53
Modification Date	2018-08-17T16:21:54
Spectrometer Frequency	95.37
Spectral Width	384615.4
Lowest Frequency	-96936.3
Nucleus	
Acquired Size	115384
Spectral Size	131072

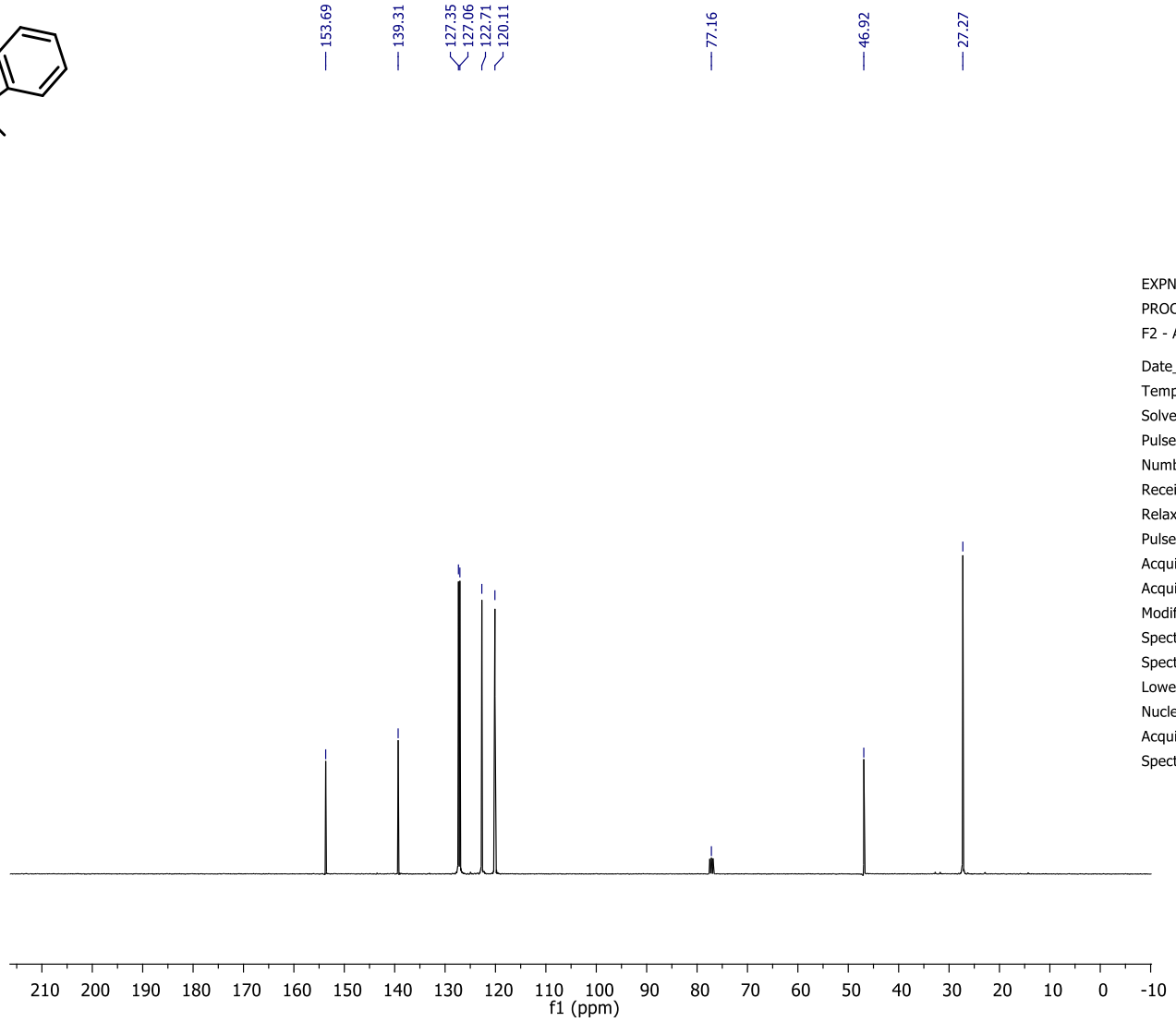
9,9-Dimethyl-9*H*-fluorene (**4a**): ^{1}H NMR spectrum in $CDCl_3$ (400 MHz, 294 K)



Parameter	Value
EXPNO	10
PROCNO	1
F2 - Acquisition Parameter	
Date_	20180817
Temperature	294.4
Solvent	CDCl3
Pulse Sequence	zg30
Number of Scans	32
Receiver Gain	51
Relaxation Delay	1.5000
Pulse Width	11.0600
Acquisition Time	1.9923
Acquisition Date	2018-08-17T19:51:00
Modification Date	2018-09-19T14:05:34
Spectrometer Frequency	400.13
Spectral Width	8223.7
Lowest Frequency	-1720.7
Nucleus	1H
Acquired Size	16384
Spectral Size	32768

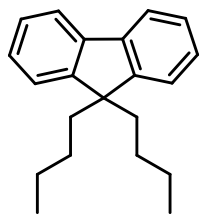
9,9-Dimethyl-9*H*-fluorene (**4a**): ^{13}C NMR spectrum in $CDCl_3$ (101 MHz, 295 K)

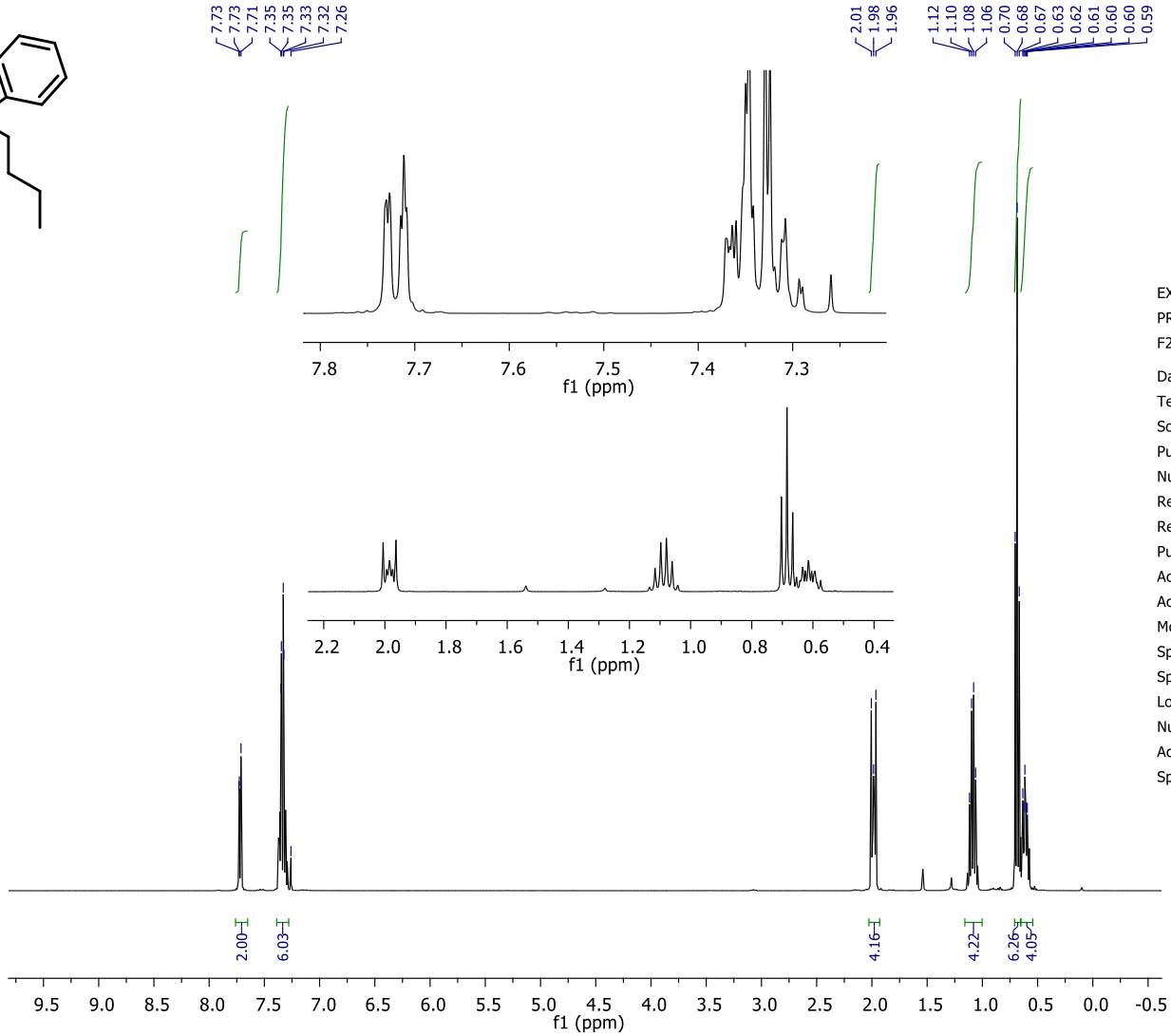




Parameter	Value
EXPNO	13
PROCNO	1
F2 - Acquisition Parameter	
Date_	20180817
Temperature	294.7
Solvent	CDCl3
Pulse Sequence	udeft
Number of Scans	380
Receiver Gain	2050
Relaxation Delay	3.0000
Pulse Width	8.8000
Acquisition Time	0.3599
Acquisition Date	2018-08-17T20:27:00
Modification Date	2018-09-19T14:05:38
Spectrometer Frequency	100.61
Spectral Width	25252.5
Lowest Frequency	-1128.8
Nucleus	13C
Acquired Size	9089
Spectral Size	32768

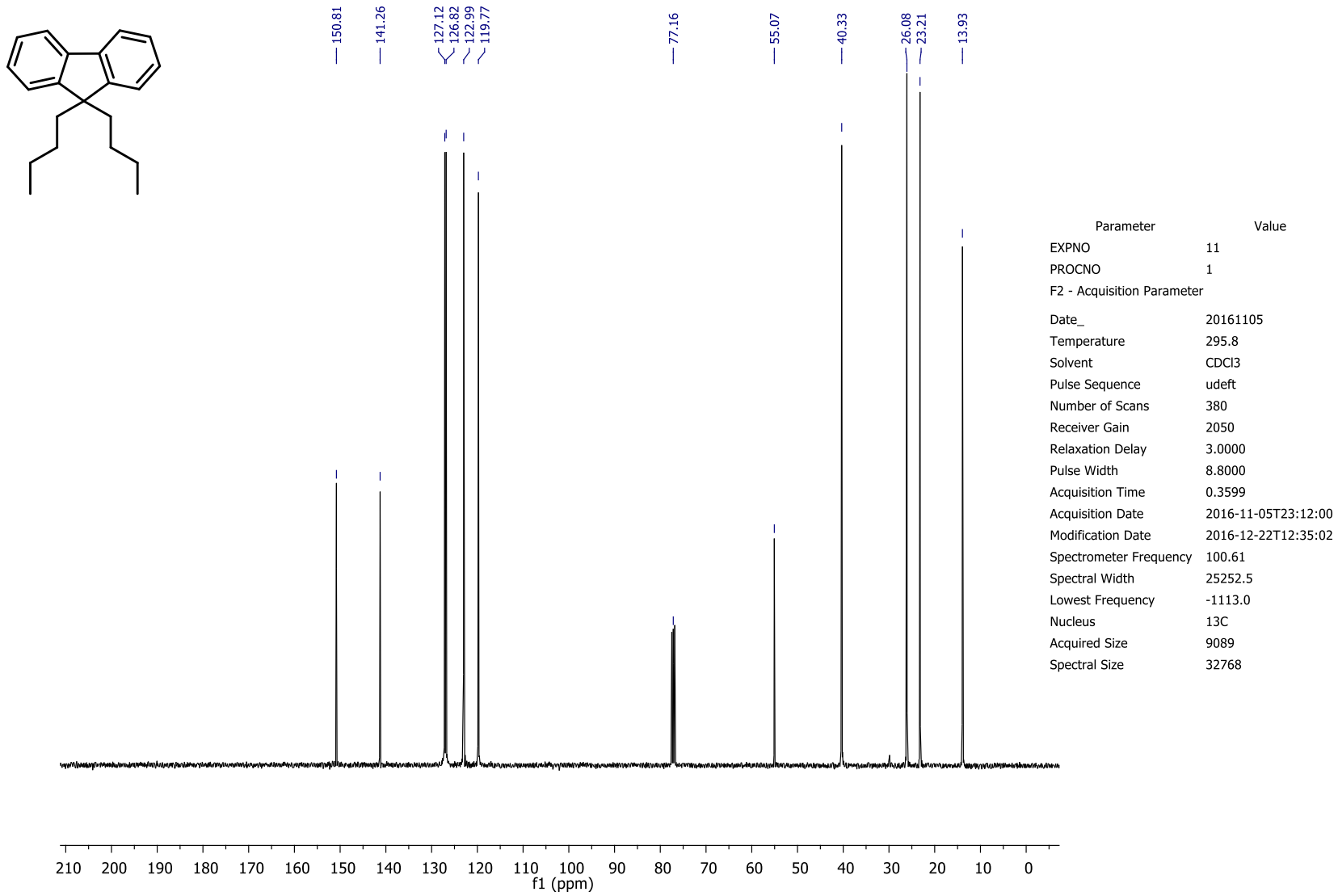
9,9-Dibutyl-9*H*-fluorene (**4b**): ^{1}H NMR spectrum in $CDCl_3$ (400 MHz, 296 K)





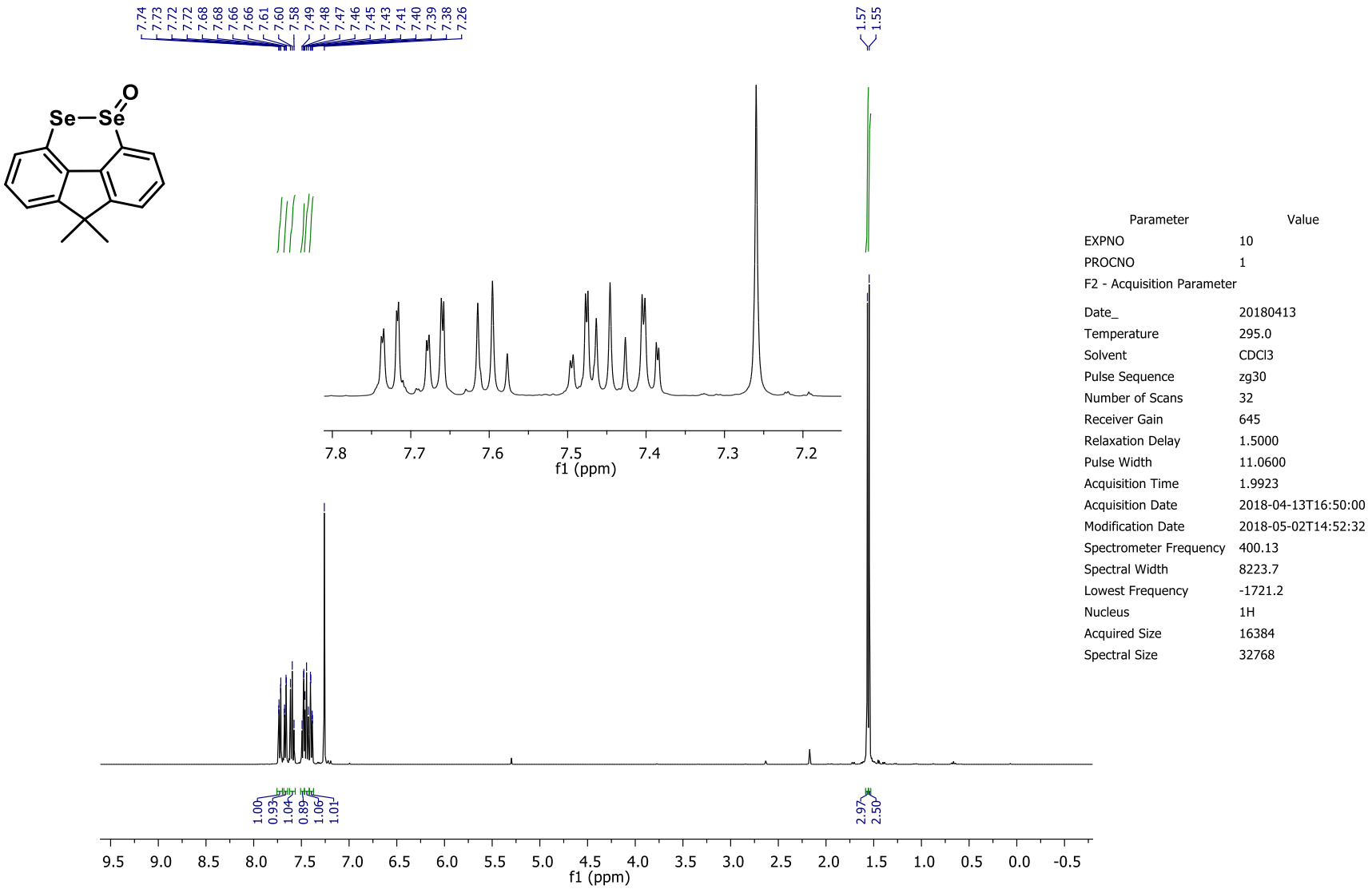
Parameter	Value
EXPNO	10
PROCNO	1
F2 - Acquisition Parameter	
Date_	20161105
Temperature	295.7
Solvent	CDCl3
Pulse Sequence	zg30
Number of Scans	32
Receiver Gain	114
Relaxation Delay	1.5000
Pulse Width	11.0600
Acquisition Time	1.9923
Acquisition Date	2016-11-05T23:02:00
Modification Date	2016-12-22T12:35:00
Spectrometer Frequency	400.13
Spectral Width	8223.7
Lowest Frequency	-1721.0
Nucleus	1H
Acquired Size	16384
Spectral Size	32768

9,9-Dibutyl-9*H*-fluorene (**4b**): ^{13}C NMR spectrum in $CDCl_3$ (101 MHz, 296 K)



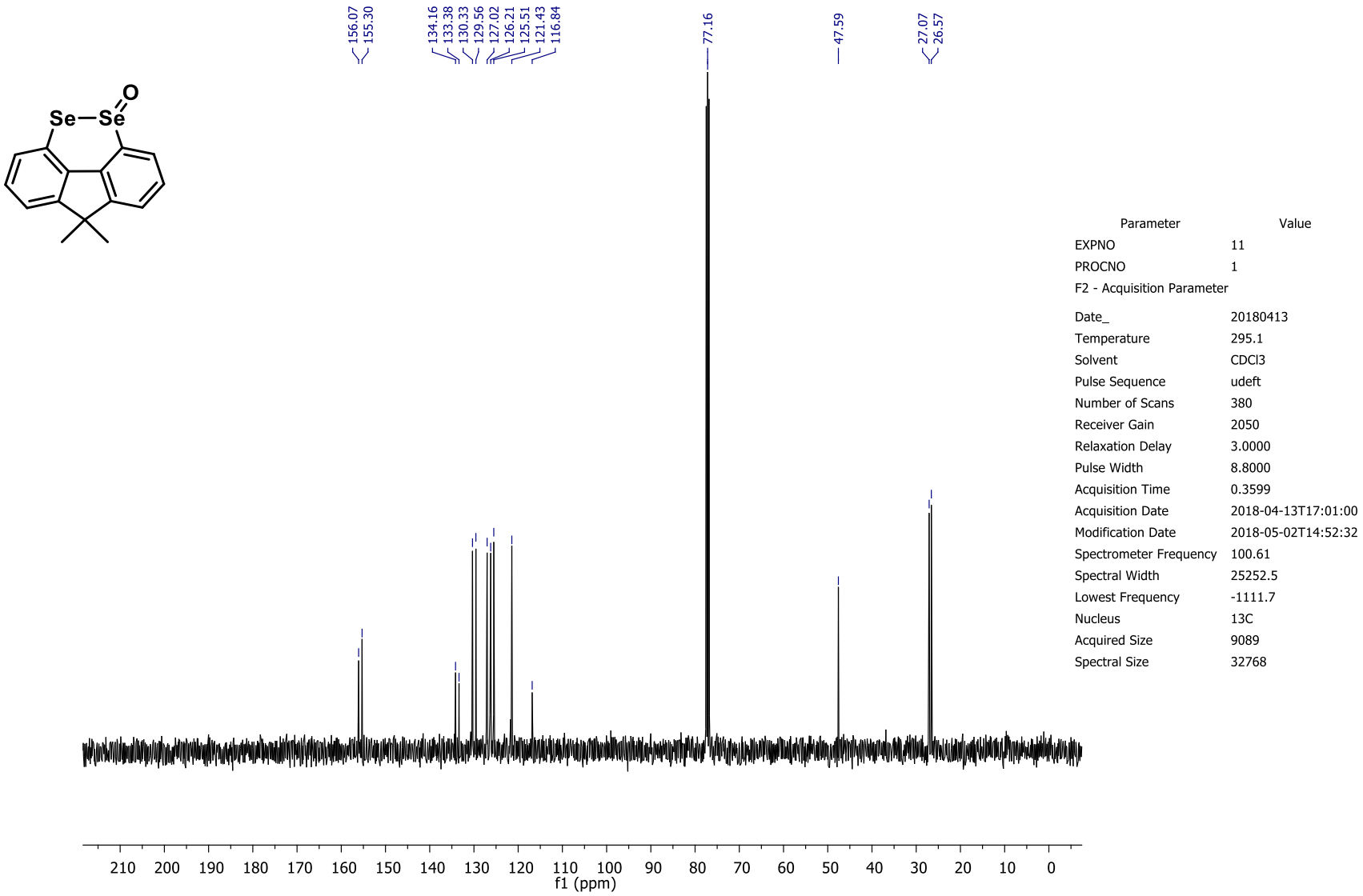
Parameter	Value
EXPNO	11
PROCNO	1
F2 - Acquisition Parameter	
Date_	20161105
Temperature	295.8
Solvent	CDCl3
Pulse Sequence	udeft
Number of Scans	380
Receiver Gain	2050
Relaxation Delay	3.0000
Pulse Width	8.8000
Acquisition Time	0.3599
Acquisition Date	2016-11-05T23:12:00
Modification Date	2016-12-22T12:35:02
Spectrometer Frequency	100.61
Spectral Width	25252.5
Lowest Frequency	-1113.0
Nucleus	13C
Acquired Size	9089
Spectral Size	32768

9,9-Dimethyl-9*H*-fluoreno[4,5-*cde*][1,2]diselenine 4-oxide (**5a**): ^{1}H NMR spectrum in $CDCl_3$ (400 MHz, 295 K)



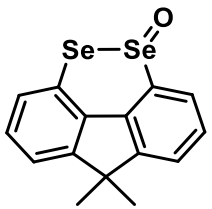
Parameter	Value
EXPNO	10
PROCNO	1
F2 - Acquisition Parameter	
Date_	20180413
Temperature	295.0
Solvent	CDCl3
Pulse Sequence	zg30
Number of Scans	32
Receiver Gain	645
Relaxation Delay	1.5000
Pulse Width	11.0600
Acquisition Time	1.9923
Acquisition Date	2018-04-13T16:50:00
Modification Date	2018-05-02T14:52:32
Spectrometer Frequency	400.13
Spectral Width	8223.7
Lowest Frequency	-1721.2
Nucleus	1H
Acquired Size	16384
Spectral Size	32768

9,9-Dimethyl-9*H*-fluoreno[4,5-*cde*][1,2]diselenine 4-oxide (**5a**): ^{13}C NMR spectrum in $CDCl_3$ (101 MHz, 295 K)



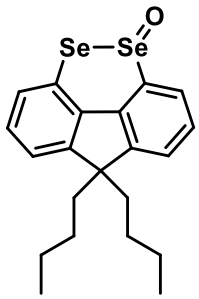
Parameter	Value
EXPNO	11
PROCNO	1
F2 - Acquisition Parameter	
Date_	20180413
Temperature	295.1
Solvent	CDCl3
Pulse Sequence	udeft
Number of Scans	380
Receiver Gain	2050
Relaxation Delay	3.0000
Pulse Width	8.8000
Acquisition Time	0.3599
Acquisition Date	2018-04-13T17:01:00
Modification Date	2018-05-02T14:52:32
Spectrometer Frequency	100.61
Spectral Width	25252.5
Lowest Frequency	-1111.7
Nucleus	13C
Acquired Size	9089
Spectral Size	32768

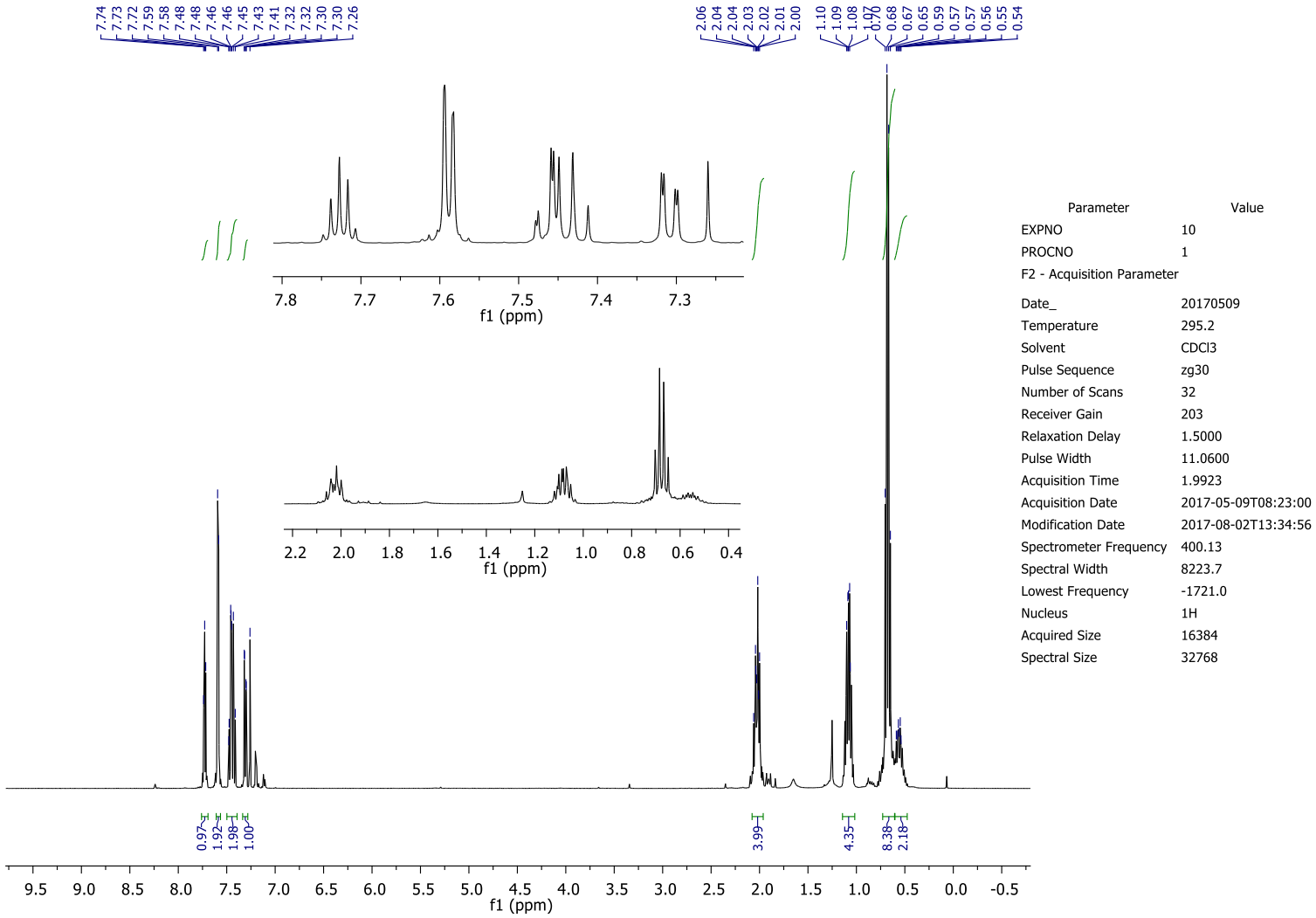
9,9-Dimethyl-9*H*-fluoreno[4,5-*cde*][1,2]diselenine 4-oxide (**5a**): ^{77}Se NMR spectrum in $CDCl_3$ (76 MHz, 298 K)



Parameter	Value
EXPNO	16
PROCNO	1
F2 - Acquisition Parameter	
Date_	20190716
Temperature	298.0
Solvent	CDCl3
Pulse Sequence	zg
Number of Scans	1500
Receiver Gain	5
Relaxation Delay	0.5000
Pulse Width	11.0000
Acquisition Time	0.2621
Acquisition Date	2019-07-16T23:42:22
Modification Date	2019-07-19T12:09:14
Spectrometer Frequency	76.31
Spectral Width	62500.0
Lowest Frequency	45060.8
Nucleus	
Acquired Size	16384
Spectral Size	65536

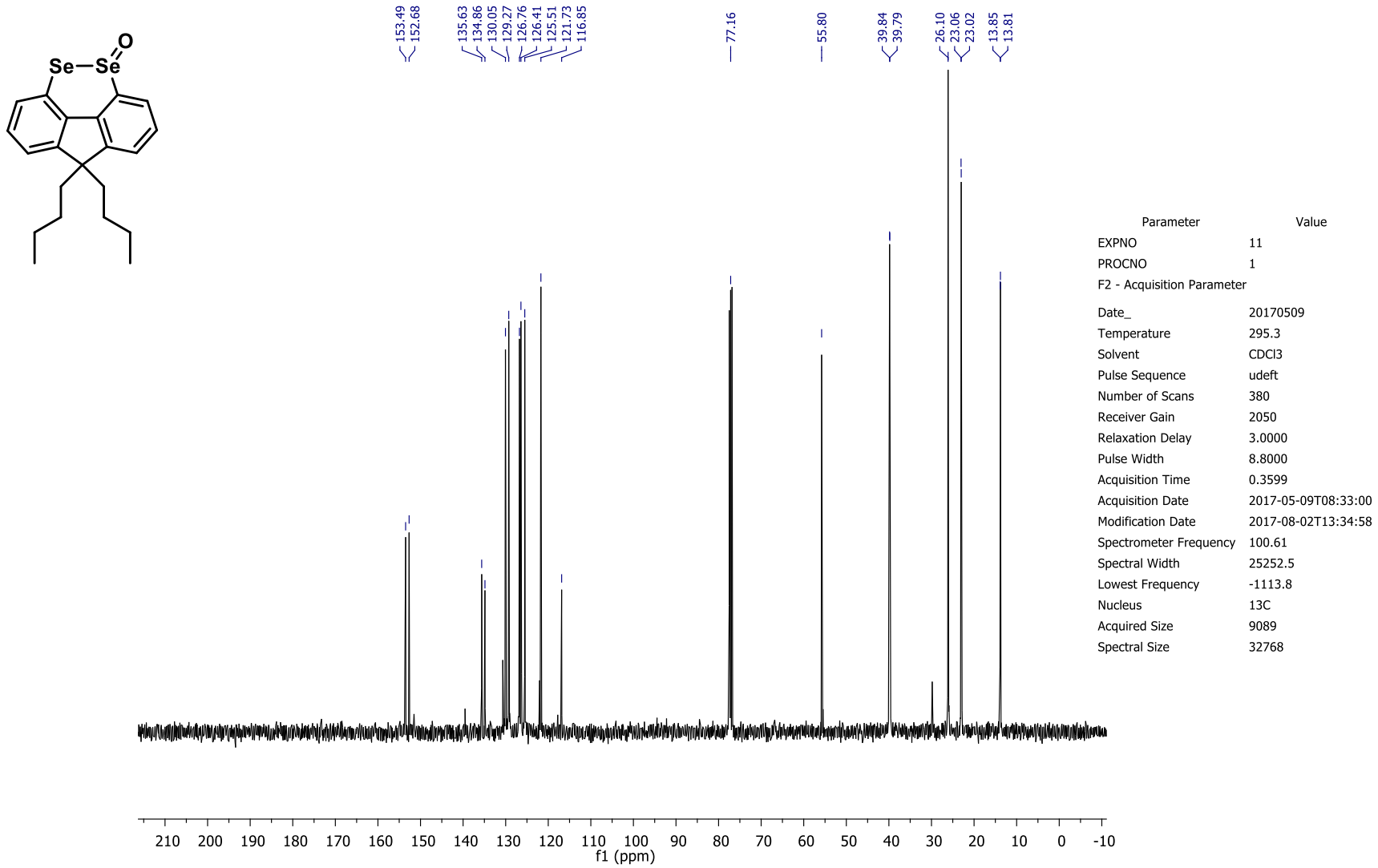
9,9-Dibutyl-9*H*-fluoreno[4,5-*cde*][1,2]diselenine 4-oxide (**5b**): ^{1}H NMR spectrum in $CDCl_3$ (400 MHz, 295 K)





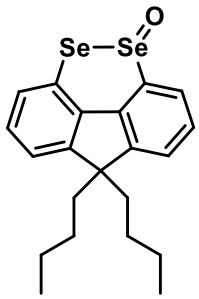
Parameter	Value
EXPNO	10
PROCNO	1
F2 - Acquisition Parameter	
Date_	20170509
Temperature	295.2
Solvent	CDCl3
Pulse Sequence	zg30
Number of Scans	32
Receiver Gain	203
Relaxation Delay	1.5000
Pulse Width	11.0600
Acquisition Time	1.9923
Acquisition Date	2017-05-09T08:23:00
Modification Date	2017-08-02T13:34:56
Spectrometer Frequency	400.13
Spectral Width	8223.7
Lowest Frequency	-1721.0
Nucleus	1H
Acquired Size	16384
Spectral Size	32768

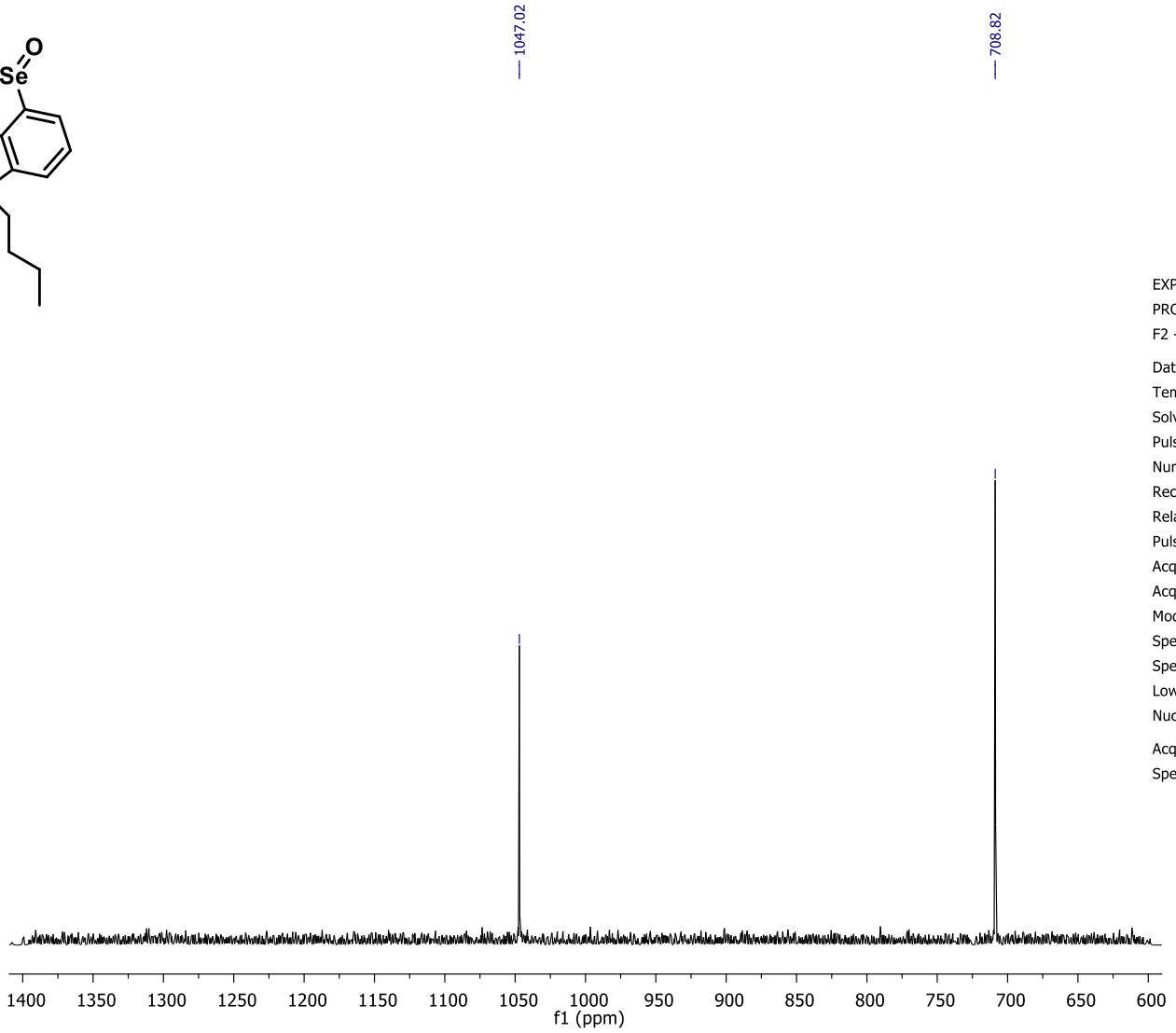
9,9-Dibutyl-9*H*-fluoreno[4,5-*cde*][1,2]diselenine 4-oxide (**5b**): ^{13}C NMR spectrum in $CDCl_3$ (101 MHz, 295 K)



Parameter	Value
EXPNO	11
PROCNO	1
F2 - Acquisition Parameter	
Date_	20170509
Temperature	295.3
Solvent	CDCl3
Pulse Sequence	udeft
Number of Scans	380
Receiver Gain	2050
Relaxation Delay	3.0000
Pulse Width	8.8000
Acquisition Time	0.3599
Acquisition Date	2017-05-09T08:33:00
Modification Date	2017-08-02T13:34:58
Spectrometer Frequency	100.61
Spectral Width	25252.5
Lowest Frequency	-1113.8
Nucleus	13C
Acquired Size	9089
Spectral Size	32768

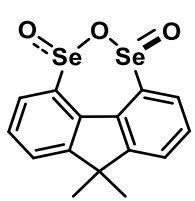
9,9-Dibutyl-9H-fluoreno[4,5-*cde*][1,2]diselenine 4-oxide (**5b**): ^{77}Se NMR spectrum in $CDCl_3$ (76 MHz, 298 K)

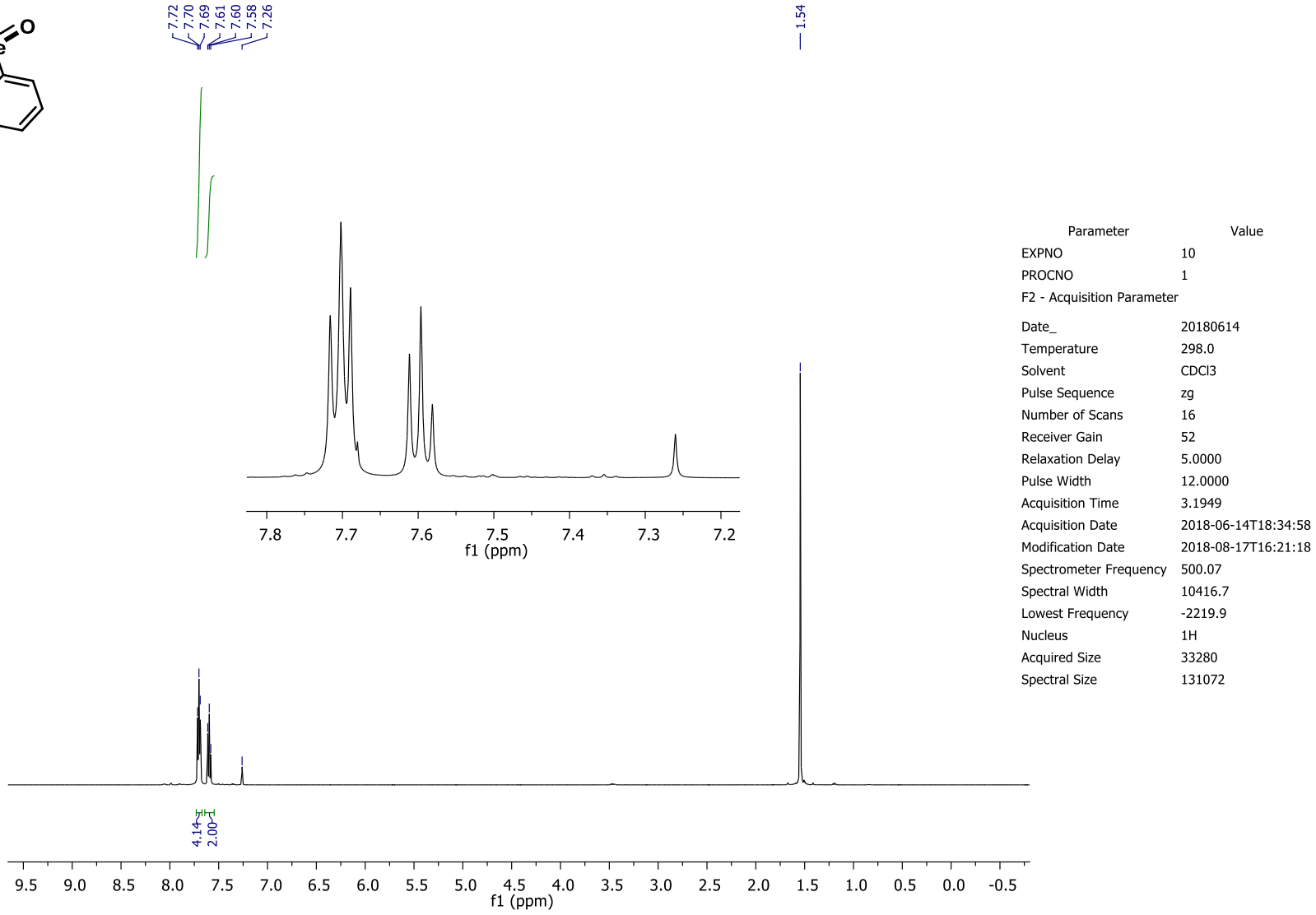




Parameter	Value
EXPNO	16
PROCNO	1
F2 - Acquisition Parameter	
Date_	20190716
Temperature	298.0
Solvent	CDCl3
Pulse Sequence	zg
Number of Scans	1500
Receiver Gain	5
Relaxation Delay	0.5000
Pulse Width	11.0000
Acquisition Time	0.2621
Acquisition Date	2019-07-16T23:42:22
Modification Date	2019-07-19T12:09:14
Spectrometer Frequency	76.31
Spectral Width	62500.0
Lowest Frequency	45060.8
Nucleus	
Acquired Size	16384
Spectral Size	65536

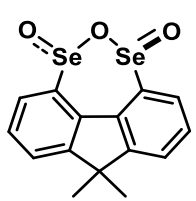
trans-Seleninic anhydride **6a**: ^{1}H NMR spectrum in $CDCl_3$ (500 MHz, 298 K)

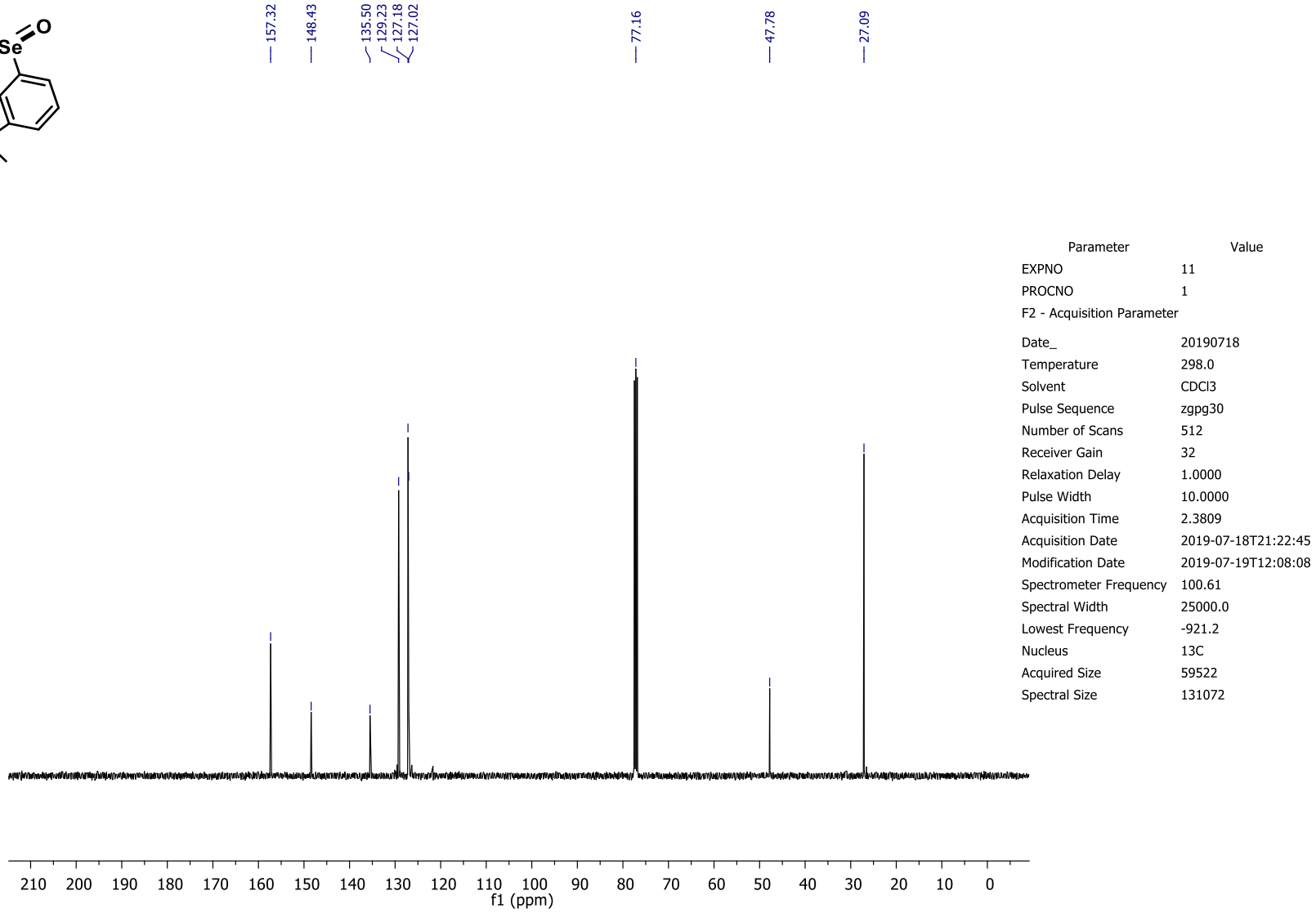




Parameter	Value
EXPNO	10
PROCNO	1
F2 - Acquisition Parameter	
Date_	20180614
Temperature	298.0
Solvent	CDCl3
Pulse Sequence	zg
Number of Scans	16
Receiver Gain	52
Relaxation Delay	5.0000
Pulse Width	12.0000
Acquisition Time	3.1949
Acquisition Date	2018-06-14T18:34:58
Modification Date	2018-08-17T16:21:18
Spectrometer Frequency	500.07
Spectral Width	10416.7
Lowest Frequency	-2219.9
Nucleus	1H
Acquired Size	33280
Spectral Size	131072

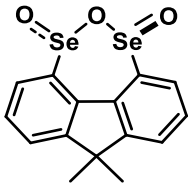
trans-Seleninic anhydride **6a**: ^{13}C NMR spectrum in $CDCl_3$ (101 MHz, 298 K)

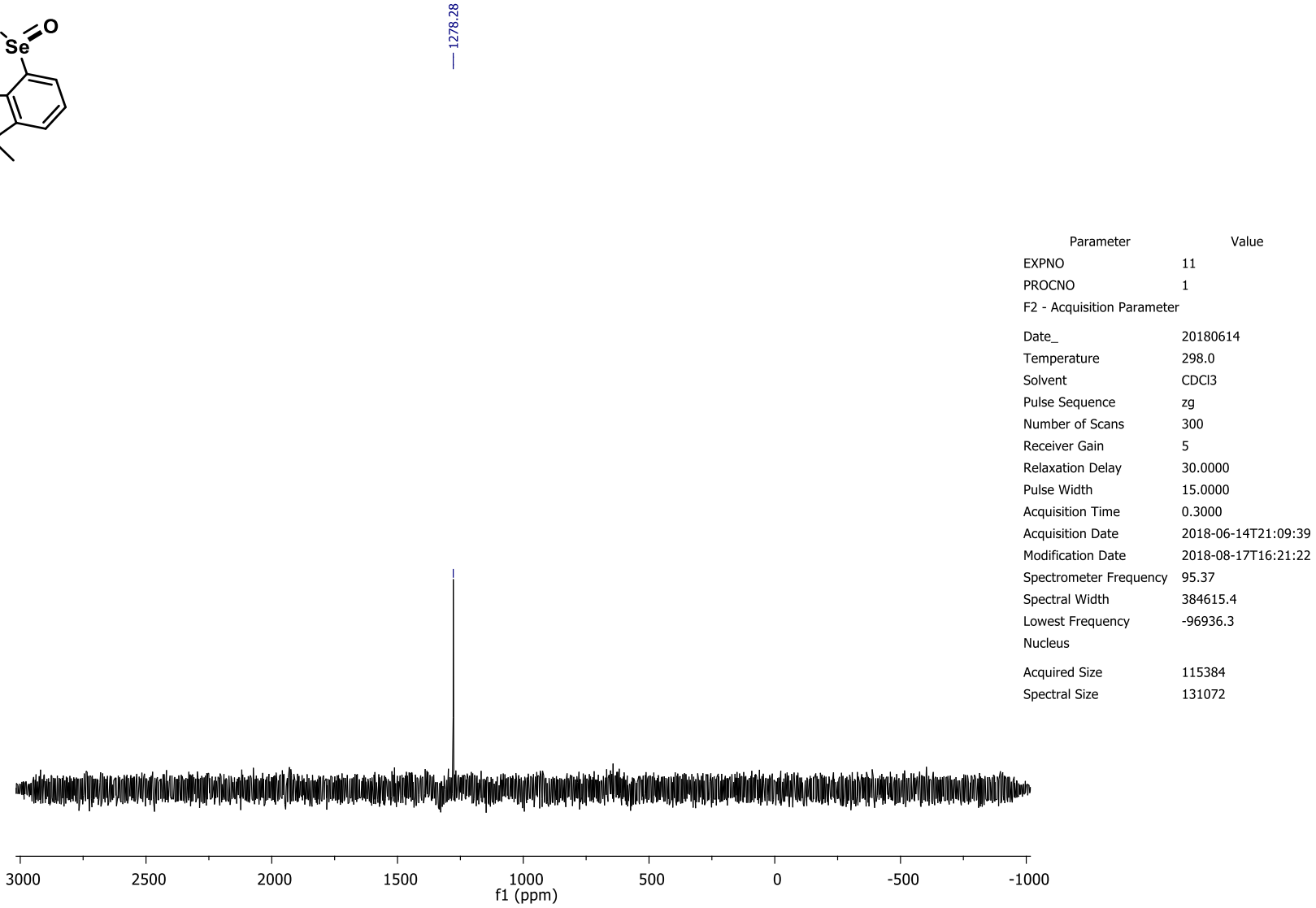




Parameter	Value
EXPNO	11
PROCNO	1
F2 - Acquisition Parameter	
Date_	20190718
Temperature	298.0
Solvent	CDCl3
Pulse Sequence	zgpg30
Number of Scans	512
Receiver Gain	32
Relaxation Delay	1.0000
Pulse Width	10.0000
Acquisition Time	2.3809
Acquisition Date	2019-07-18T21:22:45
Modification Date	2019-07-19T12:08:08
Spectrometer Frequency	100.61
Spectral Width	25000.0
Lowest Frequency	-921.2
Nucleus	13C
Acquired Size	59522
Spectral Size	131072

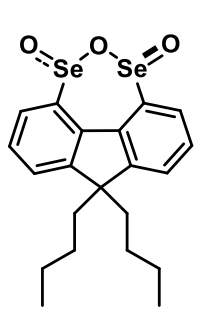
trans-Seleninic anhydride **6a**: ^{77}Se NMR spectrum in $CDCl_3$ (95 MHz, 298 K)

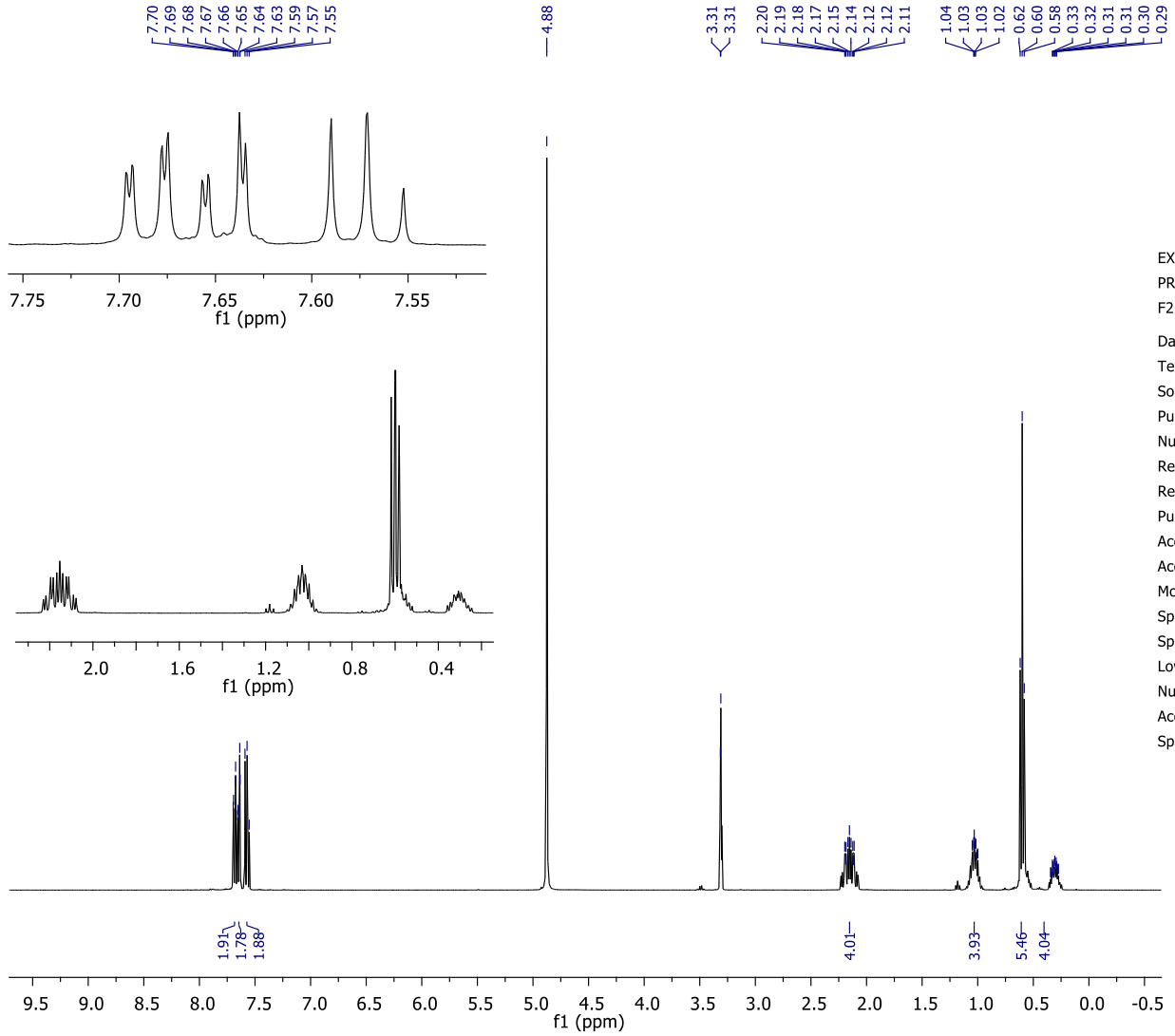




Parameter	Value
EXPNO	11
PROCNO	1
F2 - Acquisition Parameter	
Date_	20180614
Temperature	298.0
Solvent	CDCl3
Pulse Sequence	zg
Number of Scans	300
Receiver Gain	5
Relaxation Delay	30.0000
Pulse Width	15.0000
Acquisition Time	0.3000
Acquisition Date	2018-06-14T21:09:39
Modification Date	2018-08-17T16:21:22
Spectrometer Frequency	95.37
Spectral Width	384615.4
Lowest Frequency	-96936.3
Nucleus	
Acquired Size	115384
Spectral Size	131072

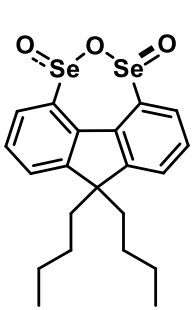
trans-Seleninic anhydride **6b**: ^{1}H NMR spectrum in MeOD (400 MHz, 295 K)

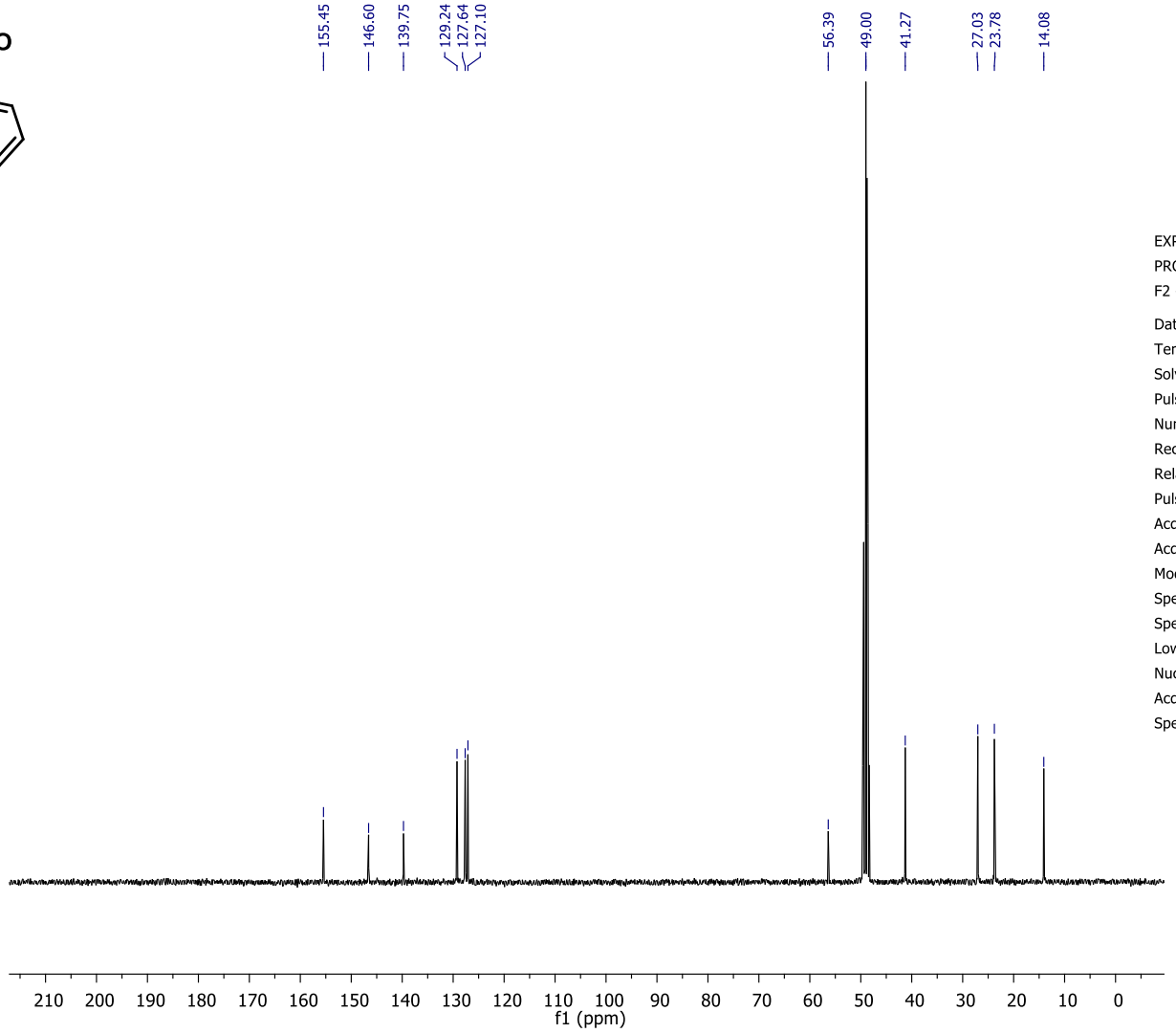




Parameter	Value
EXPNO	10
PROCNO	1
F2 - Acquisition Parameter	
Date_	20170806
Temperature	295.4
Solvent	MeOD
Pulse Sequence	zg30
Number of Scans	32
Receiver Gain	287
Relaxation Delay	1.5000
Pulse Width	11.0600
Acquisition Time	1.9923
Acquisition Date	2017-08-06T03:27:00
Modification Date	2017-08-08T20:09:00
Spectrometer Frequency	400.13
Spectral Width	8223.7
Lowest Frequency	-1718.5
Nucleus	1H
Acquired Size	16384
Spectral Size	32768

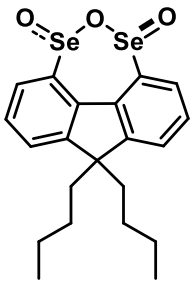
trans-Seleninic anhydride **6b**: ^{13}C NMR spectrum in MeOD (101 MHz, 295 K)

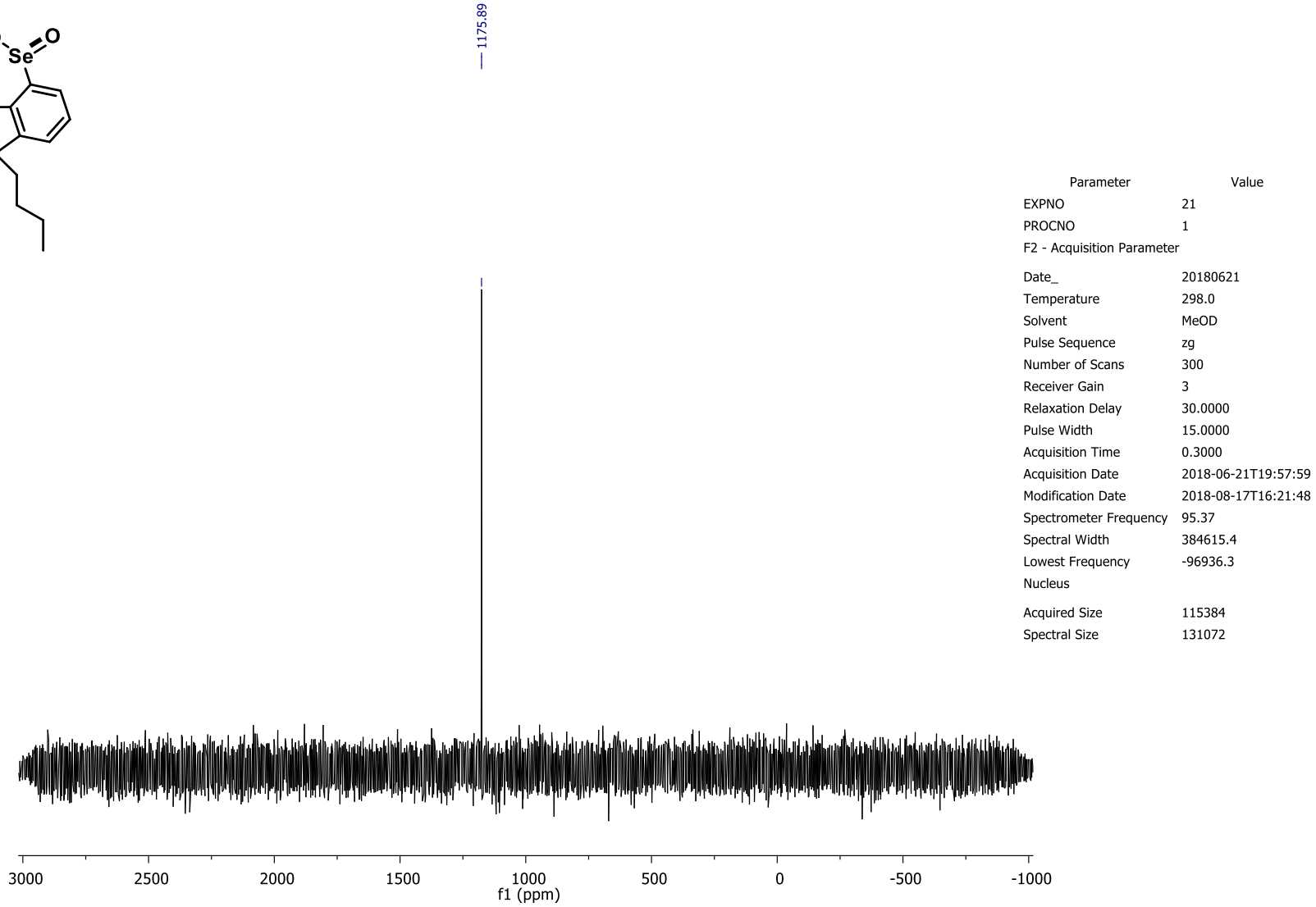




Parameter	Value
EXPNO	14
PROCNO	1
F2 - Acquisition Parameter	
Date_	20170806
Temperature	295.3
Solvent	MeOD
Pulse Sequence	udeft
Number of Scans	380
Receiver Gain	2050
Relaxation Delay	3.0000
Pulse Width	8.8000
Acquisition Time	0.3599
Acquisition Date	2017-08-06T05:04:00
Modification Date	2017-08-08T20:09:10
Spectrometer Frequency	100.61
Spectral Width	25252.5
Lowest Frequency	-985.4
Nucleus	13C
Acquired Size	9089
Spectral Size	32768

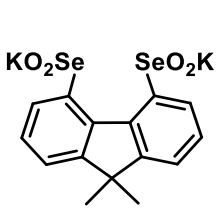
trans-Seleninic anhydride **6b**: ^{77}Se NMR spectrum in MeOD (95 MHz, 298 K)





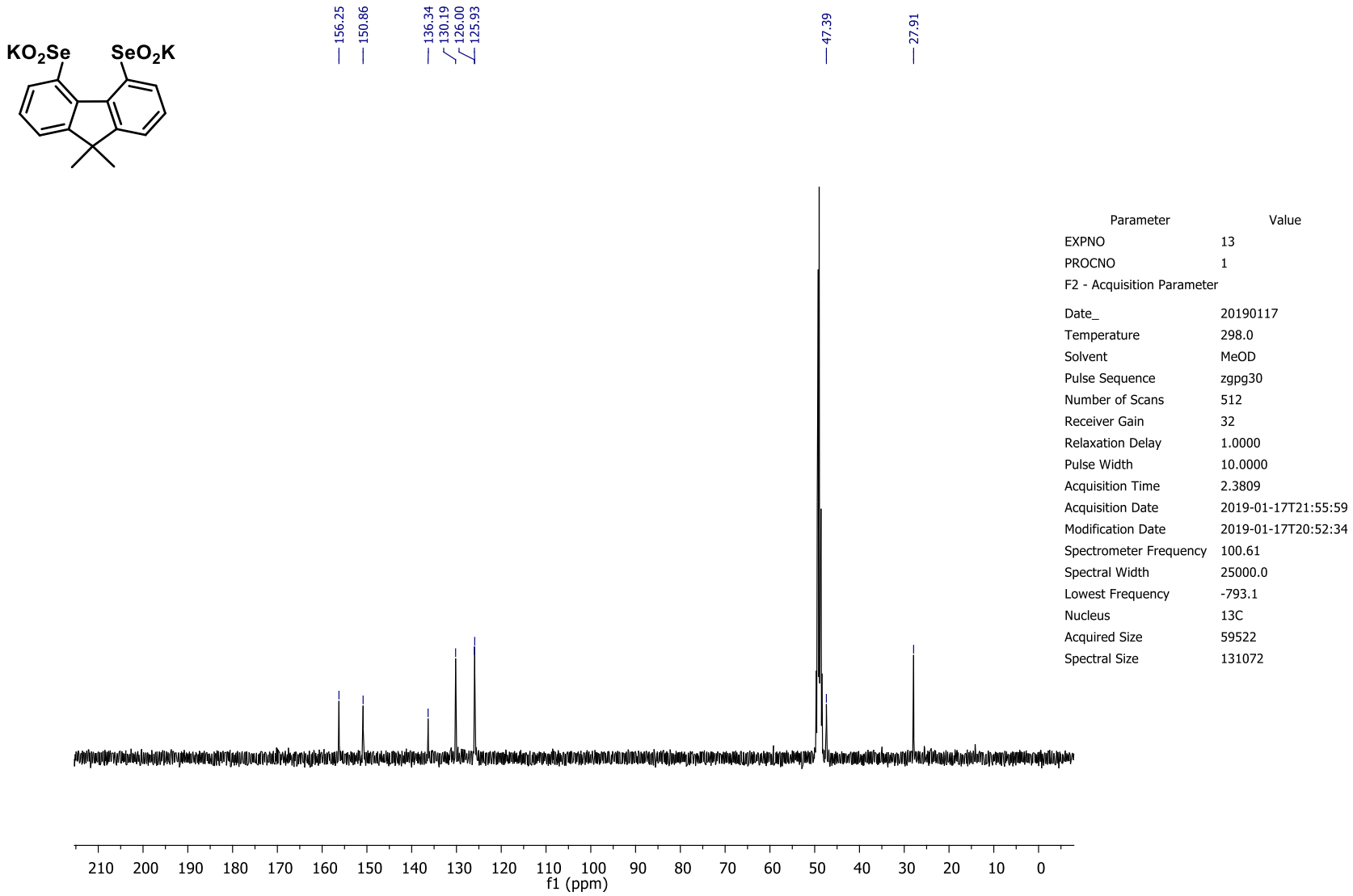
Parameter	Value
EXPNO	21
PROCNO	1
F2 - Acquisition Parameter	
Date_	20180621
Temperature	298.0
Solvent	MeOD
Pulse Sequence	zg
Number of Scans	300
Receiver Gain	3
Relaxation Delay	30.0000
Pulse Width	15.0000
Acquisition Time	0.3000
Acquisition Date	2018-06-21T19:57:59
Modification Date	2018-08-17T16:21:48
Spectrometer Frequency	95.37
Spectral Width	384615.4
Lowest Frequency	-96936.3
Nucleus	
Acquired Size	115384
Spectral Size	131072

Potassium 9,9-dimethyl-9*H*-fluorene-4,5-diseleninate (**7a**): ^{1}H NMR spectrum in MeOD (400 MHz, 295 K)



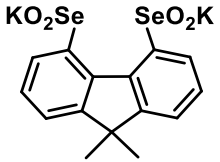
Parameter	Value
EXPNO	10
PROCNO	1
F2 - Acquisition Parameter	
Date_	20190117
Temperature	298.0
Solvent	MeOD
Pulse Sequence	zg30
Number of Scans	2
Receiver Gain	92
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	4.5875
Acquisition Date	2019-01-17T16:14:15
Modification Date	2019-01-17T15:10:52
Spectrometer Frequency	400.13
Spectral Width	7142.9
Lowest Frequency	-1270.4
Nucleus	1H
Acquired Size	32768
Spectral Size	131072

Potassium 9,9-dimethyl-9*H*-fluorene-4,5-diseleninate (**7a**): ^{13}C NMR spectrum in MeOD (101 MHz, 298 K)



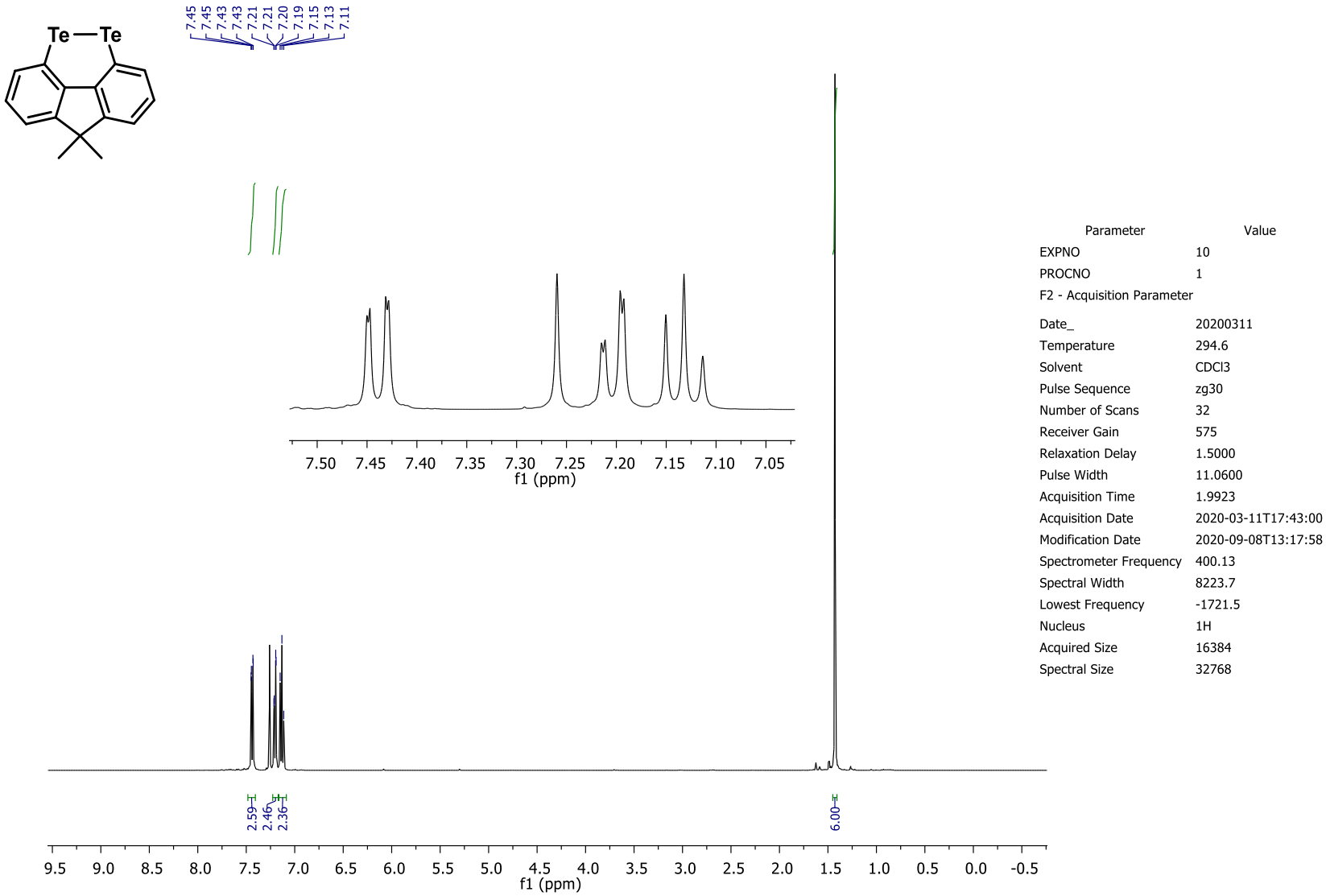
Parameter	Value
EXPNO	13
PROCNO	1
F2 - Acquisition Parameter	
Date_	20190117
Temperature	298.0
Solvent	MeOD
Pulse Sequence	zgpg30
Number of Scans	512
Receiver Gain	32
Relaxation Delay	1.0000
Pulse Width	10.0000
Acquisition Time	2.3809
Acquisition Date	2019-01-17T21:55:59
Modification Date	2019-01-17T20:52:34
Spectrometer Frequency	100.61
Spectral Width	25000.0
Lowest Frequency	-793.1
Nucleus	13C
Acquired Size	59522
Spectral Size	131072

Potassium 9,9-dimethyl-9*H*-fluorene-4,5-diseleninate (**7a**): ^{77}Se NMR spectrum in MeOD (76 MHz, 298 K)



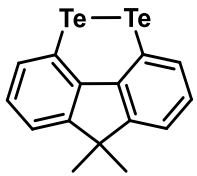
Parameter	Value
EXPNO	12
PROCNO	1
F2 - Acquisition Parameter	
Date_	20190117
Temperature	298.0
Solvent	MeOD
Pulse Sequence	zg
Number of Scans	1500
Receiver Gain	8
Relaxation Delay	0.5000
Pulse Width	11.0000
Acquisition Time	0.2621
Acquisition Date	2019-01-17T21:25:19
Modification Date	2019-01-17T20:21:56
Spectrometer Frequency	76.31
Spectral Width	62500.0
Lowest Frequency	44926.7
Nucleus	
Acquired Size	16384
Spectral Size	65536

9,9-Dimethyl-9*H*-fluoreno[4,5-*cde*][1,2]ditellurine (**8a**): ^{1}H NMR spectrum in $CDCl_3$ (400 MHz, 295 K)



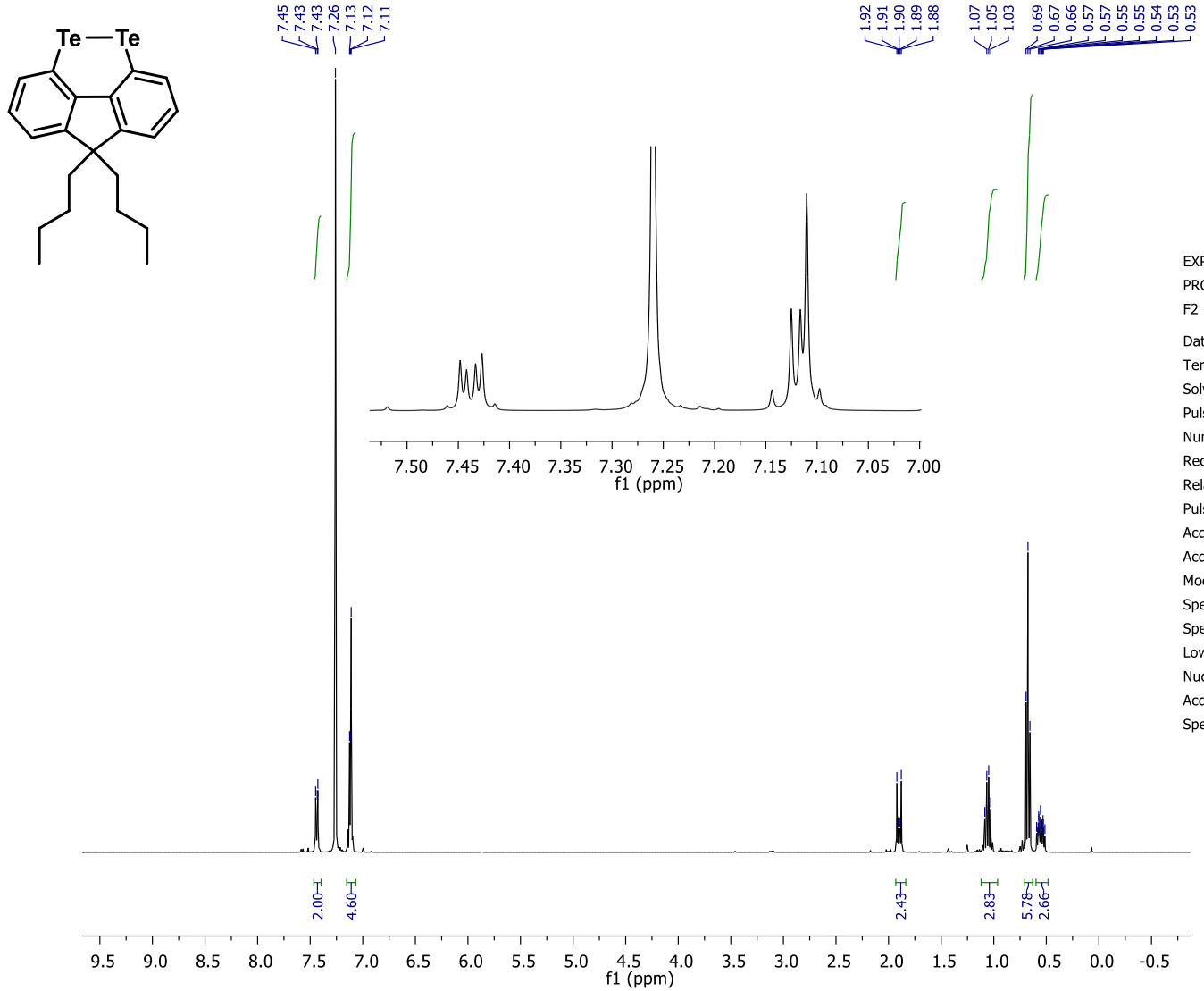
Parameter	Value
EXPNO	10
PROCNO	1
F2 - Acquisition Parameter	
Date_	20200311
Temperature	294.6
Solvent	CDCl3
Pulse Sequence	zg30
Number of Scans	32
Receiver Gain	575
Relaxation Delay	1.5000
Pulse Width	11.0600
Acquisition Time	1.9923
Acquisition Date	2020-03-11T17:43:00
Modification Date	2020-09-08T13:17:58
Spectrometer Frequency	400.13
Spectral Width	8223.7
Lowest Frequency	-1721.5
Nucleus	1H
Acquired Size	16384
Spectral Size	32768

9,9-Dimethyl-9*H*-fluoreno[4,5-*cde*][1,2]ditellurine (**8a**): ^{13}C NMR spectrum in $CDCl_3$ (101 MHz, 295 K)



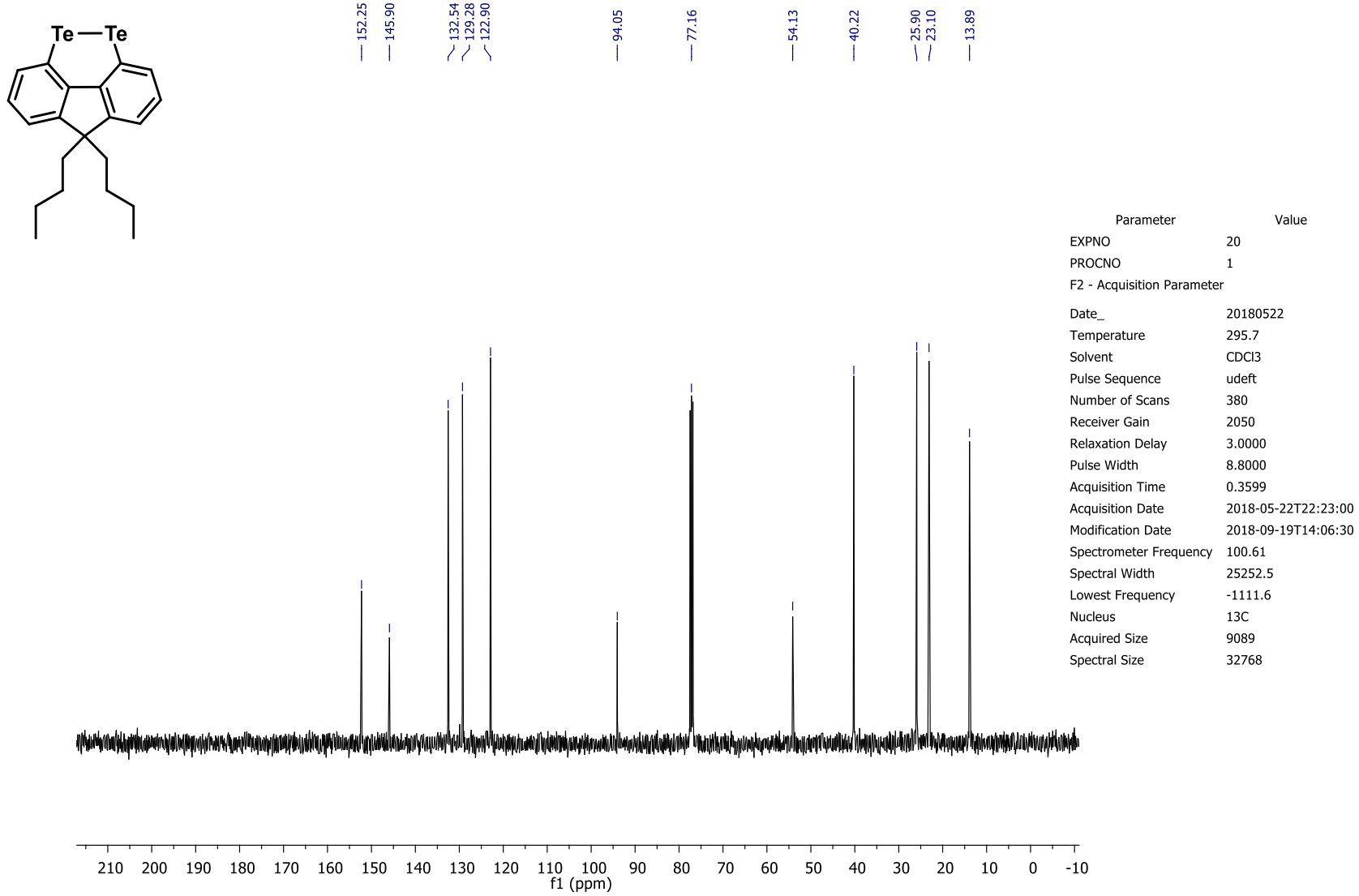
Parameter	Value
EXPNO	11
PROCNO	1
F2 - Acquisition Parameter	
Date_	20200311
Temperature	294.5
Solvent	CDCl3
Pulse Sequence	udeft
Number of Scans	380
Receiver Gain	2050
Relaxation Delay	3.0000
Pulse Width	8.8000
Acquisition Time	0.3599
Acquisition Date	2020-03-11T17:53:00
Modification Date	2020-09-08T13:17:58
Spectrometer Frequency	100.61
Spectral Width	25252.5
Lowest Frequency	-1113.3
Nucleus	13C
Acquired Size	9089
Spectral Size	32768

9,9-Dibutyl-9*H*-fluoreno[4,5-*cde*][1,2]ditellurine (**8b**): ^{1}H NMR spectrum in $CDCl_3$ (400 MHz, 295 K)



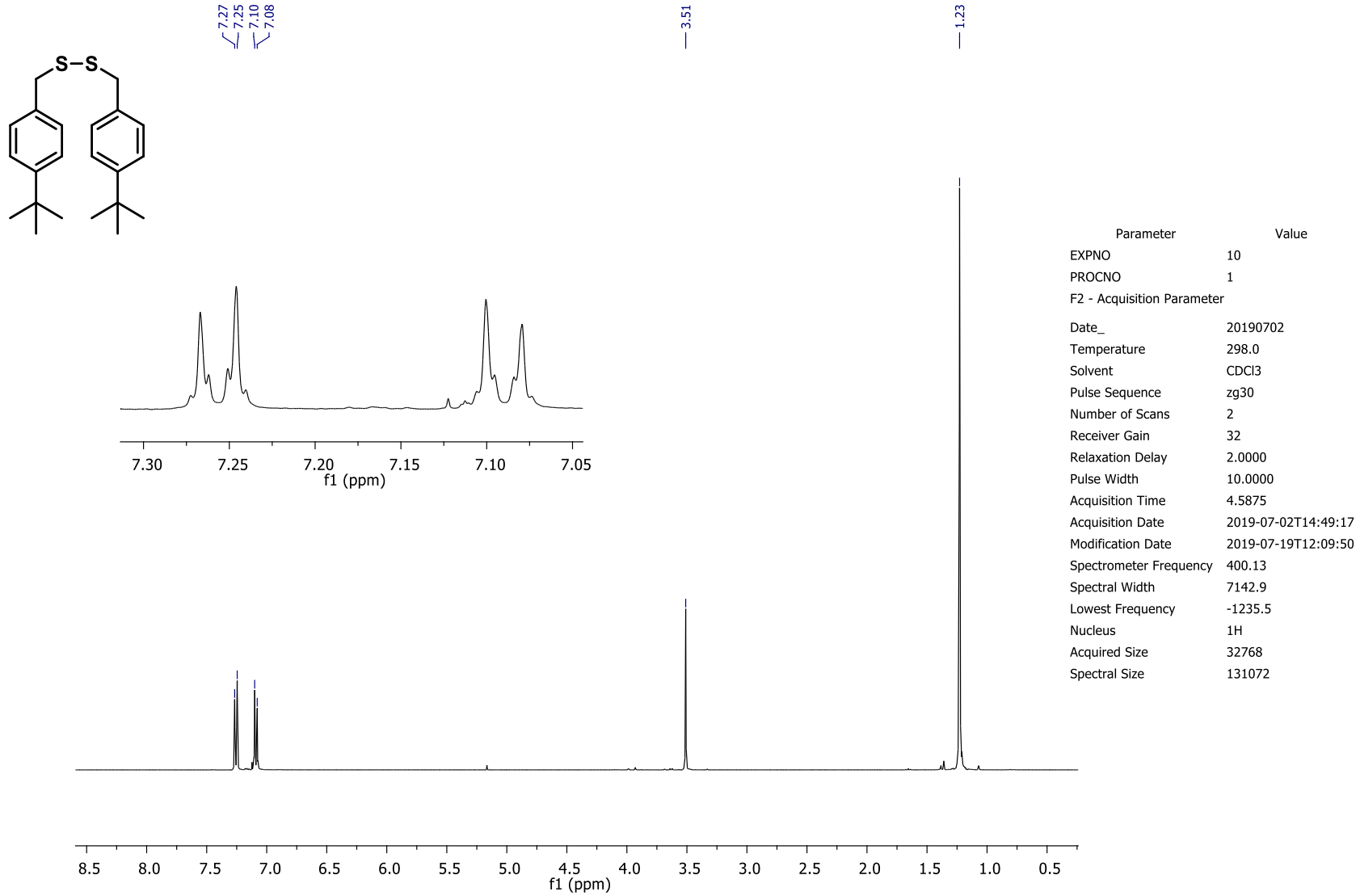
Parameter	Value
EXPNO	10
PROCNO	1
F2 - Acquisition Parameter	
Date_	20180522
Temperature	295.2
Solvent	CDCl3
Pulse Sequence	zg30
Number of Scans	32
Receiver Gain	812
Relaxation Delay	1.5000
Pulse Width	11.0600
Acquisition Time	1.9923
Acquisition Date	2018-05-22T15:45:00
Modification Date	2018-09-19T14:06:28
Spectrometer Frequency	400.13
Spectral Width	8223.7
Lowest Frequency	-1721.0
Nucleus	1H
Acquired Size	16384
Spectral Size	32768

9,9-Dibutyl-9H-fluoreno[4,5-*cde*][1,2]ditellurine (**8b**): ^{13}C NMR spectrum in $CDCl_3$ (101 MHz, 296 K)



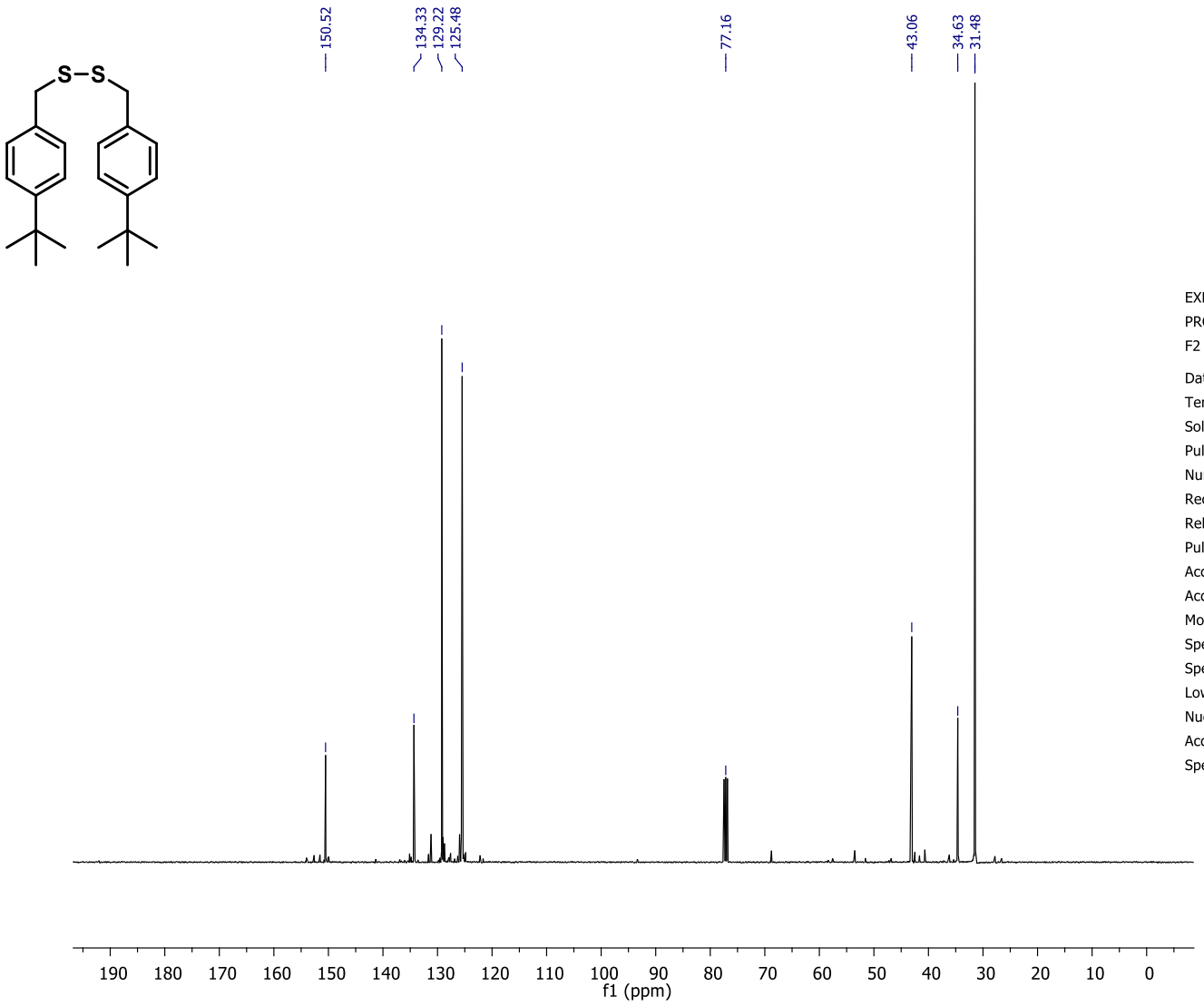
Parameter	Value
EXPNO	20
PROCNO	1
F2 - Acquisition Parameter	
Date_	20180522
Temperature	295.7
Solvent	CDCl3
Pulse Sequence	udeft
Number of Scans	380
Receiver Gain	2050
Relaxation Delay	3.0000
Pulse Width	8.8000
Acquisition Time	0.3599
Acquisition Date	2018-05-22T22:23:00
Modification Date	2018-09-19T14:06:30
Spectrometer Frequency	100.61
Spectral Width	25252.5
Lowest Frequency	-1111.6
Nucleus	13C
Acquired Size	9089
Spectral Size	32768

1,2-Bis(4-(*tert*-butyl)benzyl)disulfane (**11**): ^{1}H NMR spectrum in $CDCl_3$ (400 MHz, 298 K)



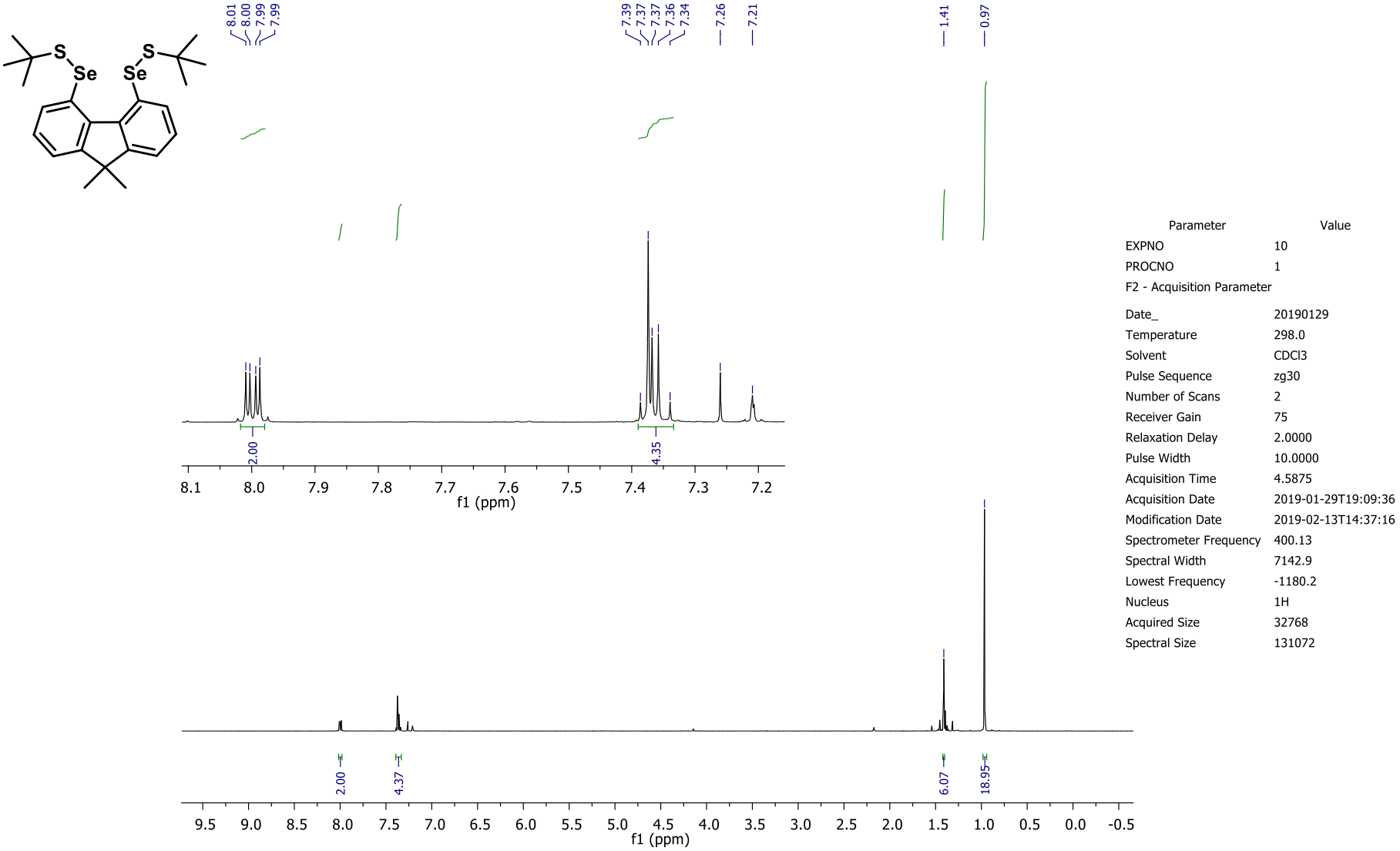
Parameter	Value
EXPNO	10
PROCNO	1
F2 - Acquisition Parameter	
Date_	20190702
Temperature	298.0
Solvent	CDCl3
Pulse Sequence	zg30
Number of Scans	2
Receiver Gain	32
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	4.5875
Acquisition Date	2019-07-02T14:49:17
Modification Date	2019-07-19T12:09:50
Spectrometer Frequency	400.13
Spectral Width	7142.9
Lowest Frequency	-1235.5
Nucleus	1H
Acquired Size	32768
Spectral Size	131072

1,2-Bis(4-(*tert*-butyl)benzyl)disulfane (**11**): ^{13}C NMR spectrum in $CDCl_3$ (101 MHz, 298 K)



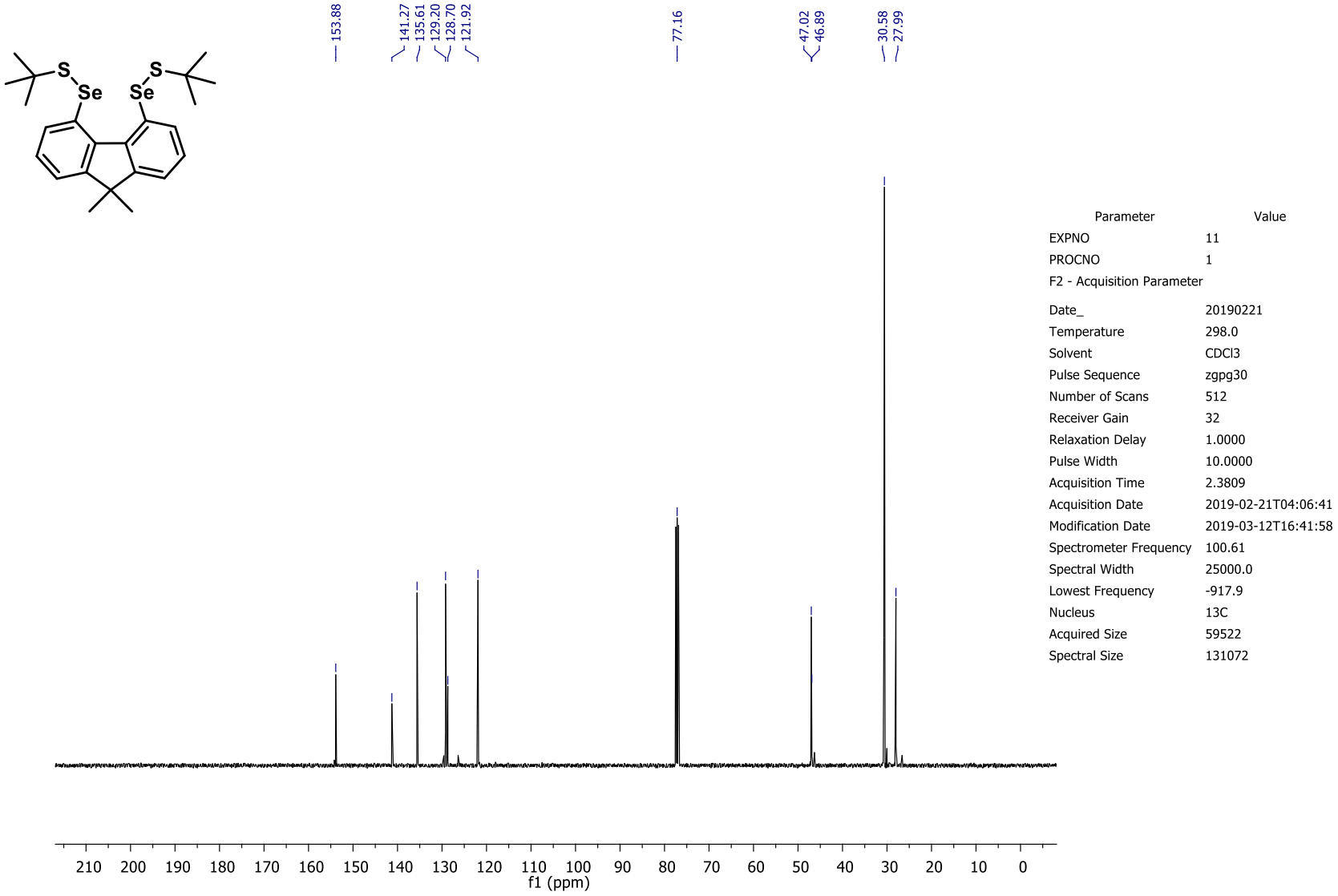
Parameter	Value
EXPNO	12
PROCNO	1
F2 - Acquisition Parameter	
Date_	20190704
Temperature	298.0
Solvent	CDCl3
Pulse Sequence	zgpg30
Number of Scans	512
Receiver Gain	29
Relaxation Delay	1.0000
Pulse Width	10.0000
Acquisition Time	2.3809
Acquisition Date	2019-07-04T08:02:52
Modification Date	2019-07-19T12:09:52
Spectrometer Frequency	100.61
Spectral Width	25000.0
Lowest Frequency	-930.3
Nucleus	13C
Acquired Size	59522
Spectral Size	131072

((9,9-Dimethyl-9*H*-fluorene-4,5-diyl)bis(selanediyl))bis(*tert*-butylsulfane) (**12a**): ^{1}H NMR spectrum in $CDCl_3$ (400 MHz, 298 K)

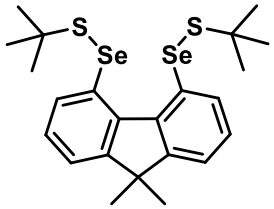


Parameter	Value
EXPNO	10
PROCNO	1
F2 - Acquisition Parameter	
Date_	20190129
Temperature	298.0
Solvent	CDCl3
Pulse Sequence	zg30
Number of Scans	2
Receiver Gain	75
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	4.5875
Acquisition Date	2019-01-29T19:09:36
Modification Date	2019-02-13T14:37:16
Spectrometer Frequency	400.13
Spectral Width	7142.9
Lowest Frequency	-1180.2
Nucleus	1H
Acquired Size	32768
Spectral Size	131072

((9,9-Dimethyl-9*H*-fluorene-4,5-diyl)bis(selanediyl))bis(*tert*-butylsulfane) (**12a**): ^{13}C NMR spectrum in $CDCl_3$ (101 MHz, 298 K)

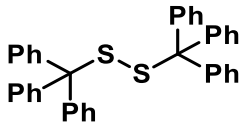


((9,9-Dimethyl-9*H*-fluorene-4,5-diyl)bis(selanediyl))bis(*tert*-butylsulfane) (**12a**): ^{77}Se NMR spectrum in $CDCl_3$ (76 MHz, 298 K)

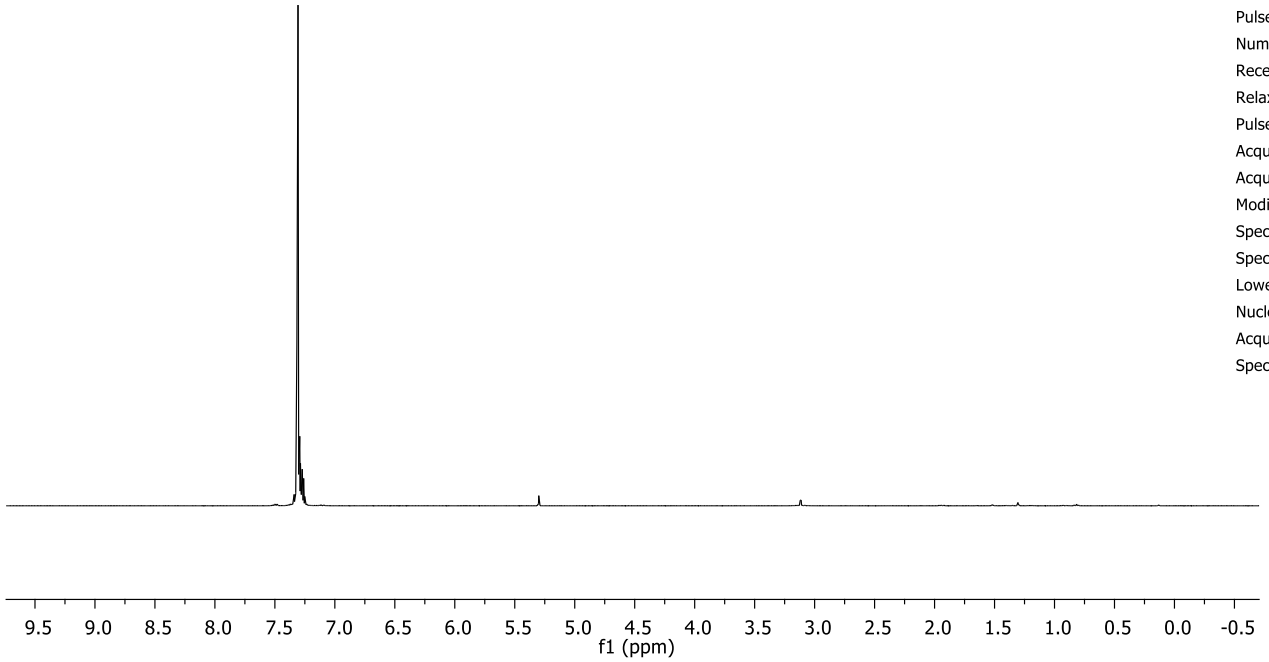


Parameter	Value
EXPNO	13
PROCNO	1
F2 - Acquisition Parameter	
Date_	20190221
Temperature	298.0
Solvent	CDCl3
Pulse Sequence	zg
Number of Scans	1500
Receiver Gain	3
Relaxation Delay	0.5000
Pulse Width	11.0000
Acquisition Time	0.2621
Acquisition Date	2019-02-21T04:46:28
Modification Date	2019-03-12T16:42:00
Spectrometer Frequency	76.31
Spectral Width	62500.0
Lowest Frequency	6905.4
Nucleus	
Acquired Size	16384
Spectral Size	65536

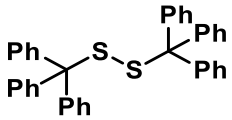
1,2-Dirityldisulfane (**16**): ^{1}H NMR spectrum in $CDCl_3$ (400 MHz, 298 K)



Parameter	Value
EXPNO	10
PROCNO	1
F2 - Acquisition Parameter	
Date_	20190717
Temperature	298.0
Solvent	CDCl3
Pulse Sequence	zg30
Number of Scans	2
Receiver Gain	52
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	4.5875
Acquisition Date	2019-07-17T16:57:47
Modification Date	2019-07-19T12:08:20
Spectrometer Frequency	400.13
Spectral Width	7142.9
Lowest Frequency	-1180.2
Nucleus	1H
Acquired Size	32768
Spectral Size	131072

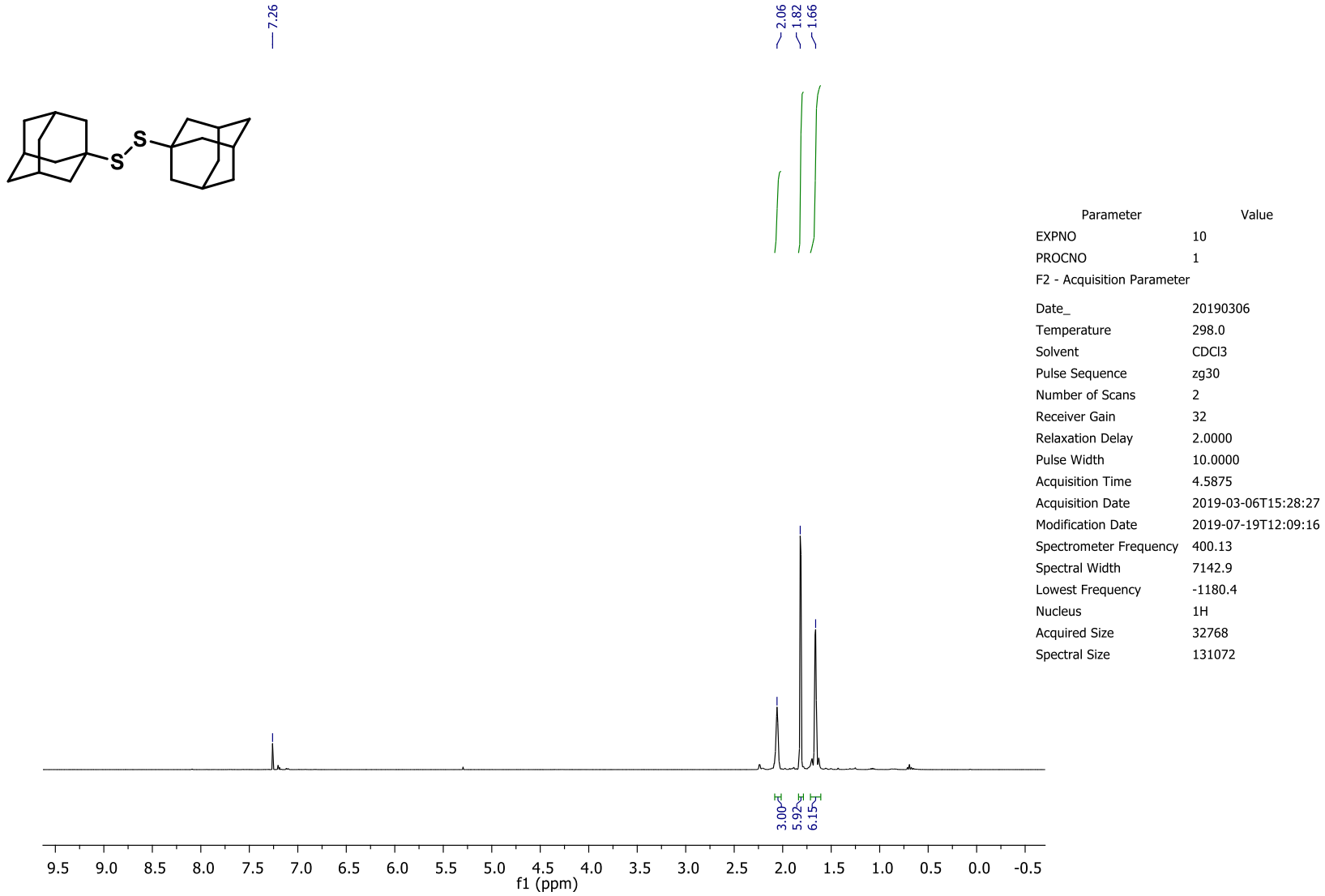


1,2-Dirityldisulfane (**16**): ^{13}C NMR spectrum in $CDCl_3$ (101 MHz, 298 K)



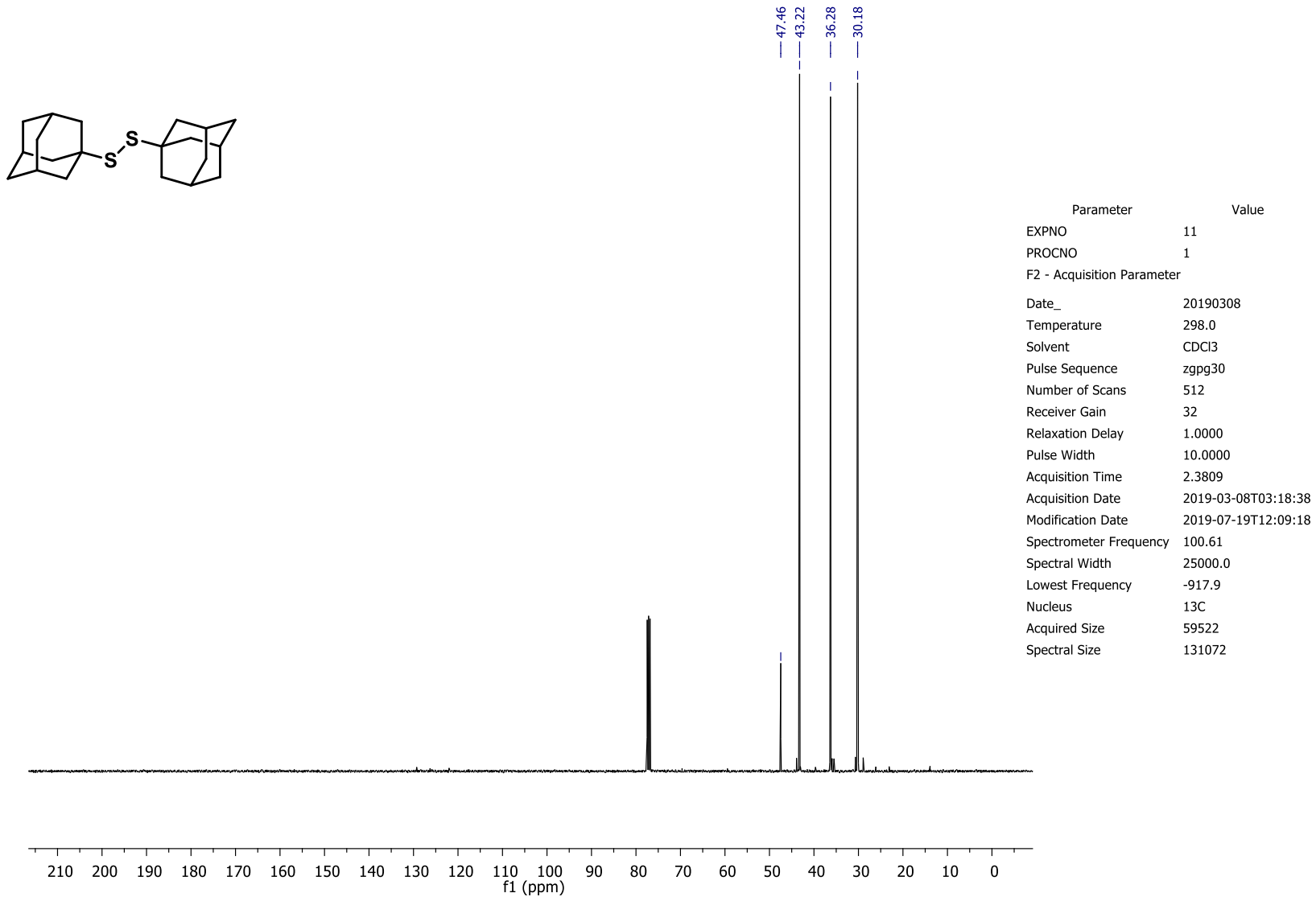
Parameter	Value
EXPNO	11
PROCNO	1
F2 - Acquisition Parameter	
Date_	20190717
Temperature	298.0
Solvent	CDCl3
Pulse Sequence	zgpg30
Number of Scans	512
Receiver Gain	32
Relaxation Delay	1.0000
Pulse Width	10.0000
Acquisition Time	2.3809
Acquisition Date	2019-07-17T18:16:45
Modification Date	2019-07-19T12:08:22
Spectrometer Frequency	100.61
Spectral Width	25000.0
Lowest Frequency	-929.5
Nucleus	13C
Acquired Size	59522
Spectral Size	131072

Di-*tert*-adamantyl disulfide (**18**): ^{1}H NMR spectrum in $CDCl_3$ (400 MHz, 298 K)



Parameter	Value
EXPNO	10
PROCNO	1
F2 - Acquisition Parameter	
Date_	20190306
Temperature	298.0
Solvent	CDCl3
Pulse Sequence	zg30
Number of Scans	2
Receiver Gain	32
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	4.5875
Acquisition Date	2019-03-06T15:28:27
Modification Date	2019-07-19T12:09:16
Spectrometer Frequency	400.13
Spectral Width	7142.9
Lowest Frequency	-1180.4
Nucleus	1H
Acquired Size	32768
Spectral Size	131072

Di-*tert*-adamantyl disulfide (**18**): ^{13}C NMR spectrum in $CDCl_3$ (101 MHz, 298 K)



Parameter	Value
EXPNO	11
PROCNO	1
F2 - Acquisition Parameter	
Date_	20190308
Temperature	298.0
Solvent	CDCl3
Pulse Sequence	zgpg30
Number of Scans	512
Receiver Gain	32
Relaxation Delay	1.0000
Pulse Width	10.0000
Acquisition Time	2.3809
Acquisition Date	2019-03-08T03:18:38
Modification Date	2019-07-19T12:09:18
Spectrometer Frequency	100.61
Spectral Width	25000.0
Lowest Frequency	-917.9
Nucleus	13C
Acquired Size	59522
Spectral Size	131072

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