Electronic Supplementary Material (ESI) for Green Chemistry.

Visible-Light-Induced Metal and Reagent-Free Oxidative Coupling of sp^2 C─H Bond with Organo-Dichalcogenides: Synthesis of 3-Organochalcogenyl Indoles

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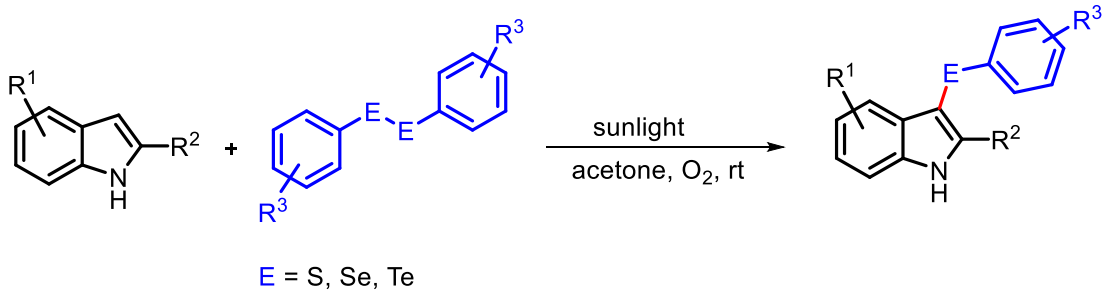
1. General experimental details

All reactions were carried out in oven-dried glassware with magnetic stirring. Indoles used in this study were obtained from commercial sources and used without further purification. Solvents screened in this report were used as purchased from suppliers. All the photo-induced reactions were performed using borosilicate glassware (5/10 mL RBF) under sunlight/CFL bulb (26 W). Substituted aryl disulfides, bis(4-methylphenyl) disulfide, bis(2-benzothiazolyl) disulfide, bis(4-fluorophenyl) disulfide, bis(2-bromophenyl) disulfide, bis(4-chlorophenyl) disulfide and bis(2,5-dichlorophenyl) disulfide, *n*-hexyl disulfide were prepared by the oxidation of respective thiols by using *tert*-butyl hydroperoxide. Diphenyl ditelluride[1], dinaphthyl ditelluride[1] and aryl diselenides: dinaphthyl diselenide,[1] bis-(4-methoxyphenyl) diselenide,[1] bis-(4-chlorophenyl) diselenide,[2] bis-(4- trifluoromethylphenyl) diselenide,[2] bis-(2-bromophenyl) diselenide,[2] bis-(2-chlorophenyl) diselenide,[2] bis-(4-bromophenyl) diselenide,[2] bis-(4-methylphenyl) diselenide,[1] bis-(2-methylphenyl) diselenide,[1] bis-(2-fluorophenyl) diselenide,[2] bis(3,4,5-trimethoxyphenyl)diselenide,[3] were prepared by following the reported procedure. Diphenyl diselenide used here were obtained from commercial sources. NMR experiments were carried out on Bruker 400/500MHz spectrometer in $CDCl_3$/DMSO-d_6 solvents and chemical shifts are reported in ppm. High resolution mass spectroscopic (HRMS) analysis is performed on quadrupole-time-of-flight Bruker MicroTOF-Q II mass spectrometer equipped with an ESI (+ve/−ve) or atmospheric pressure chemical ionization (APCI) source. GC-MS analysis is performed on Agilent 7200/Agilent Technologies MS-S975C inert XLEI/CIMSD with triple axis detector. UV-Vis study was performed on Agilent Technologies Cary (5000) series UV-Vis-NIR spectrophotometer. TLC plates (Merck silica gel (^{60}F254) plates) used for monitoring the reactions were purchased from Merck.

2. General experimental procedure for chalcogenylation of indoles

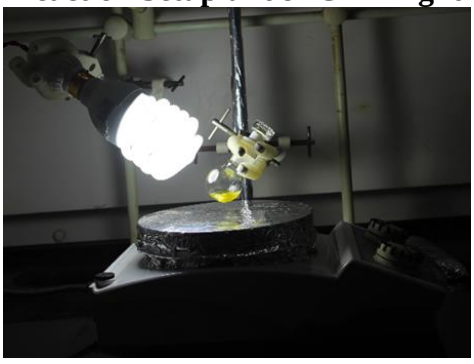


E = S, Se, Te

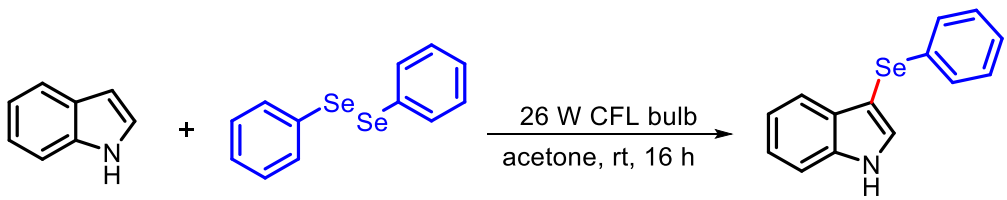
Indoles (0.2 mmol, 1.0 equiv.) aryl diselenide/disulfide/ditelluride (0.1 mmol, 0.5 equiv.) were added to a 5 mL round bottom flask with magnetic stir bar in acetone (O_2 purged, 2 mL). The reaction mixture was stirred for disulfide, diselenide and ditelluride substrates are 9-11 h, 6-7 h, and 5-6 h, respectively under sunlight. The progress of the reaction was monitored by TLC. After completion of the reaction, solvent was removed on rotary evaporator under vacuum. The residue was purified by flash column chromatography on silica gel to afford the desired product.

Reaction Setup in Day Light

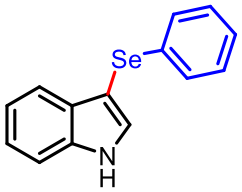
Reaction Setup under CFL Light



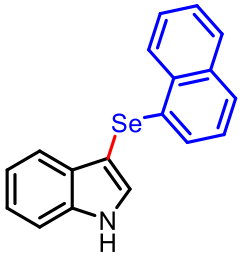
Experimental procedure under CFL light: The above reaction mixture was stirred up to 15-18 h under house hold CFL (26 W) bulb at room temperature. The progress of the reaction was monitored by TLC.



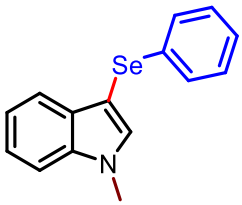
1a **1ab** 66%



3-(Phenylselanyl)-1H-indole (2a).[4] White solid, yield 42 mg (78%), ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.38 (bs, 1H), 7.64 (d, *J* = 7.9 Hz, 1H), 7.48 – 7.40 (m, 2H), 7.25 (dd, *J* = 8.1, 5.8 Hz, 3H), 7.18 (d, *J* = 7.6 Hz, 1H), 7.15 – 7.09 (m, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 136.4, 133.8, 131.2, 130.0, 128.9, 128.7, 125.6, 122.9, 120.9, 120.4, 111.4, 98.2; ^{77}Se NMR (95 MHz, $CDCl_3$) δ 210.5; GC-LRMS *m/z* calcd for $C_{14}H_{11}NSe$ $[M]^+$ 273.0, found 272.9.

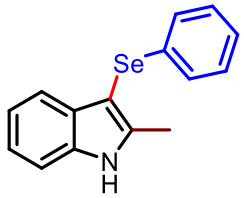


3-(Naphthalen-1-ylselanyl)-1H-indole (2b).[5] Pale brown liquid, yield 47 mg (74%), ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.46 (bs, 1H), 8.37 (d, *J* = 8.3 Hz, 1H), 7.87 (d, *J* = 8.1 Hz, 1H), 7.69 – 7.61 (m, 3H), 7.58 – 7.52 (m, 2H), 7.47 (d, *J* = 8.2 Hz, 1H), 7.33 – 7.29 (m 1H), 7.22 – 7.15 (m, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 136.5, 133.9, 132.6, 132.3, 131.5, 130.0, 128.6, 126.9, 126.24, 126.22, 126.08, 126.05, 125.7, 123.0, 120.9, 120.4, 111.4, 97.4; ^{77}Se NMR (95 MHz, $CDCl_3$) δ 174.6; GC-LRMS *m/z* calcd for $C_{18}H_{13}NSe$ $[M]^+$ 323.0, found 322.9.

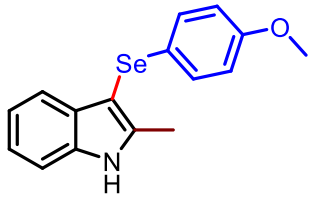


1-Methyl-3-(phenylselanyl)-1H-indole (2c).[4] White solid, yield 43 mg (76%), ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.67 (dt, *J* = 7.9, 1.0 Hz, 1H), 7.42 (dt, *J* = 8.2, 1.0 Hz, 1H), 7.37 (s, 1H), 7.35 – 7.32 (m, 1H), 7.29 – 7.26 (m, 2H), 7.23 – 7.20 (m, 1H), 7.18 – 7.10 (m, 3H), 3.88 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 137.5, 135.6, 134.2, 130.7, 128.9, 128.6, 125.5, 122.4, 120.5, 120.4, 109.5, 96.0, 33.1; ^{77}Se NMR (95 MHz, $CDCl_3$) δ 207.6 ppm; GC-LRMS *m/z* calcd for

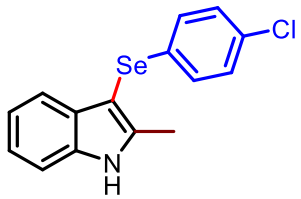
$C_{15}H_{13}NSe$ $[M]^+$ 287.0, found 286.9.



2-Methyl-3-(phenylselanyl)-1H-indole (2d).[4] White solid, yield 44 mg (77%), ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.25 (bs, 1H), 7.63 (dt, *J* = 7.7, 1.0 Hz, 1H), 7.36 (dt, *J* = 8.0, 1.0 Hz, 1H), 7.27 – 7.22 (m, 3H), 7.21 – 7.13 (m, 4H), 2.57 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 140.9, 135.8, 134.0, 131.3, 129.0, 128.4, 125.4, 122.1, 120.7, 119.8, 110.5, 96.2, 13.2; ^{77}Se NMR (76 MHz, $CDCl_3$) δ 184.6 ppm; GC-LRMS *m/z* calcd for $C_{15}H_{13}NSe$ $[M]^+$ 287.0, found 286.9.

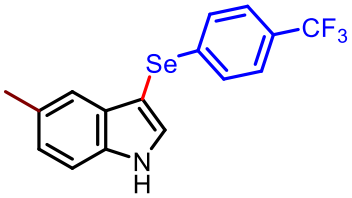


3-((4-Methoxyphenyl)selanyl)-2-methyl-1H-indole (2e). Brown liquid, yield 49 mg (78%), ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.21 (bs, 1H), 7.66 – 7.61 (m, 1H), 7.33 (dt, *J* = 8.0, 0.9 Hz, 1H), 7.24 – 7.16 (m, 4H), 6.76 – 6.72 (m, 2H), 3.75 (s, 3H), 2.58 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 158.2, 140.4, 135.7, 131.2, 130.7, 123.7, 122.0, 120.6, 119.8, 114.8, 110.5, 97.4, 55.3, 13.2; ^{77}Se NMR (95 MHz, $CDCl_3$) δ 174.0 ppm; HRMS (ESI) *m/z* calcd for $C_{16}H_{15}NOSe$ $[M-H]^+$ 316.0236, found 316.0256.

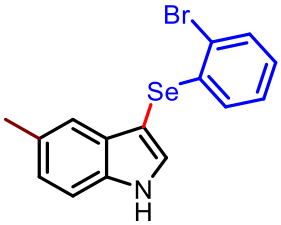


3-((4-Chlorophenyl)selanyl)-2-methyl-1H-indole (2f). Light brown solid, yield 51 mg (79%), ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.33 (bs, 1H), 7.56 (d, *J* = 7.8 Hz, 1H), 7.37 (d, *J* = 8.0 Hz, 1H), 7.26 – 7.21 (m, 1H), 7.20 – 7.16 (m, 1H), 7.11 (s, 4H), 2.57 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 140.9, 135.8, 132.2, 131.3, 131.0, 129.6, 129.0, 122.3, 120.8, 119.6, 110.6, 96.0, 13.2; ^{77}Se NMR (95 MHz, $CDCl_3$) δ 188.0 ppm; HRMS (ESI) *m/z* calcd for $C_{15}H_{12}ClNSe$

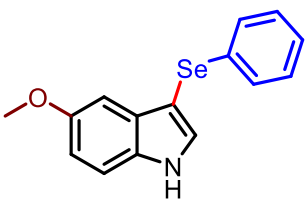
$[M-H]^+$ 319.9738, found 319.9765.



5-Methyl-3-((4-(trifluoromethyl)phenyl)selanyl)-1H-indole (2g). Cream solid, yield 57 mg (80%), ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.43 (bs, 1H), 7.49 (d, *J* = 2.6 Hz, 1H), 7.45 – 7.42 (m, 1H), 7.39 (d, *J* = 8.2 Hz, 3H), 7.31 (d, *J* = 8.2 Hz, 2H), 7.17 (dd, *J* = 8.4, 1.6 Hz, 1H), 2.48 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 139.8 (q, *J* = 1.3 Hz), 134.8, 131.7, 130.7, 129.9, 128.0, 127.6 (q, *J* = 32.6 Hz), 125.6 (q, *J* = 3.8 Hz), 124.9, 124.3 (q, *J* = 271.9 Hz), 119.6, 111.2, 96.3, 21.5; ^{77}Se NMR (95 MHz, $CDCl_3$) δ 222.8; HRMS (ESI) *m/z* calcd for $C_{16}H_{12}F_3NSe$ $[M-H]^+$ 354.0004, found 354.0024.

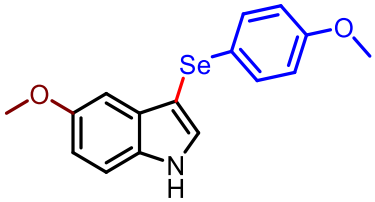


3-((2-Bromophenyl)selanyl)-5-methyl-1H-indole (2h). Buff solid, yield 62 mg (85%), ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.46 (bs, 1H), 7.50 (dd, *J* = 7.5, 1.8 Hz, 1H), 7.47 (d, *J* = 2.6 Hz, 1H), 7.45 (dd, *J* = 1.8, 0.9 Hz, 1H), 7.39 (d, *J* = 8.3 Hz, 1H), 7.16 (dd, *J* = 8.2, 1.6 Hz, 1H), 7.01 – 6.95 (m, 2H), 6.76 – 6.72 (m, 1H), 2.47 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 137.1, 134.9, 132.4, 132.2, 130.7, 130.1, 128.8, 127.7, 126.5, 124.9, 121.7, 119.8, 111.2, 97.3, 21.5; ^{77}Se NMR (95 MHz, $CDCl_3$) δ 235.3; HRMS (APCI) *m/z* calcd for $C_{15}H_{12}BrNSe$ $[M+H]^+$ 365.9388, found 365.9360.

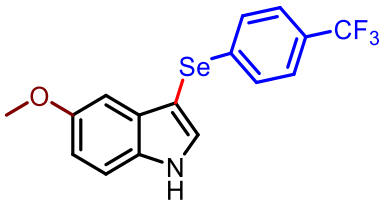


5-Methoxy-3-(phenylselanyl)-1H-indole (2i).[4] Brown solid, yield 46 mg (77%), ^{1}H NMR

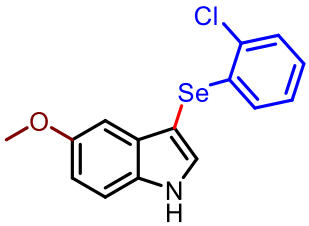
(400 MHz, $CDCl_3$) δ 8.43 (bs, 1H), 7.47 – 7.43 (m, 1H), 7.34 (d, *J* = 8.8 Hz, 1H), 7.26 (d, *J* = 1.8 Hz, 2H), 7.20 – 7.10 (m, 4H), 6.95 (dt, *J* = 8.8, 1.8 Hz, 1H), 3.84 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 155.1, 133.9, 131.9, 131.3, 130.8, 129.0, 128.5, 125.6, 113.5, 112.3, 101.6, 97.6, 55.8; ^{77}Se NMR (95 MHz, $CDCl_3$) δ 208.7; HRMS (ESI) *m/z* calcd for $C_{15}H_{13}NOSe$ $[M-H]^+$ 302.0079, found 302.0117.



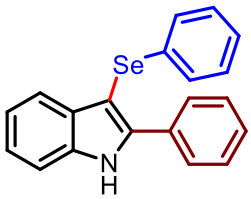
5-Methoxy-3-((4-methoxyphenyl)selanyl)-1H-indole (2j).[4] Colorless liquid, yield 50 mg (75%), ^1H NMR (500 MHz, $CDCl_3$) δ 8.34 (bs, 1H), 7.44 (d, *J* = 2.5 Hz, 1H), 7.32 (d, *J* = 8.8 Hz, 1H), 7.29 – 7.26 (m, 2H), 7.12 (d, *J* = 2.5 Hz, 1H), 6.92 (dd, *J* = 8.8, 2.5 Hz, 1H), 6.78 – 6.73 (m, 2H), 3.85 (s, 3H), 3.76 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 158.3, 155.0, 131.30, 131.27, 131.0, 130.7, 123.5, 114.8, 113.3, 112.1, 101.6, 99.0, 55.8, 55.3; ^{77}Se NMR (95 MHz, $CDCl_3$) δ 199.5; HRMS (ESI) *m/z* calcd for $C_{16}H_{15}NO_2Se$ $[M-H]^+$ 332.0185, found 332.0212.



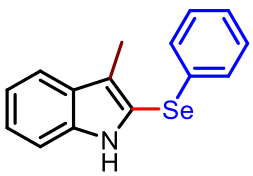
5-Methoxy-3-((4-(trifluoromethyl)phenyl)selanyl)-1H-indole (2k). Brown solid, yield 55 mg (74%), ^1H NMR (500 MHz, $CDCl_3$) δ 8.51 (bs, 1H), 7.50 (d, *J* = 2.6 Hz, 1H), 7.41 – 7.37 (m, 3H), 7.31 (dd, *J* = 8.7, 1.0 Hz, 2H), 7.05 (d, *J* = 2.5 Hz, 1H), 6.98 (dd, *J* = 8.9, 2.5 Hz, 1H), 3.84 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 155.3, 139.7 (q, *J* = 1.3 Hz), 132.2, 131.4, 130.5, 128.0, 127.6 (q, *J* = 32.5 Hz), 125.6 (q, *J* = 3.7 Hz), 124.4 (q, *J* = 273.2 Hz), 113.7, 112.4, 101.3, 96.5, 55.8; ^{77}Se NMR (95 MHz, $CDCl_3$) δ 223.1; HRMS (ESI) *m/z* calcd for $C_{16}H_{12}F_3NOSe$ $[M-H]^+$ 369.9953, found 369.9966.



3-((2-Chlorophenyl)selanyl)-5-methoxy-1H-indole (2l). Cream solid, yield 55 mg (82%), ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.53 (bs, 1H), 7.48 (d, *J* = 2.6 Hz, 1H), 7.38 (d, *J* = 8.7 Hz, 1H), 7.34 (dd, *J* = 7.9, 1.4 Hz 1H), 7.09– 7.05 (m, 2H), 6.98 (dd, *J* = 8.8, 2.5 Hz, 1H), 6.95 – 6.91 (m, 1H), 6.76 (dd, *J* = 7.9, 1.6 Hz, 1H), 3.84 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 155.3, 134.6, 132.7, 132.0, 131.5, 130.7, 129.2, 128.7, 127.2, 126.4, 113.8, 112.5, 101.4, 96.3, 56.0; ^{77}Se NMR (95 MHz, $CDCl_3$) δ 212.5; HRMS (ESI) *m/z* calcd for $C_{15}H_{12}ClNOSe$ $[M-H]^+$ 335.9687, found 335.9693.

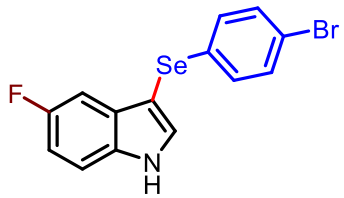


2-Phenyl-3-(phenylselanyl)-1H-indole (2m).[5] Yellow liquid, yield 55 mg (79%), ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.59 (bs, 1H), 7.79 – 7.74 (m, 2H), 7.71 (d, *J* = 7.9 Hz, 1H), 7.50 – 7.45 (m, 3H), 7.44 – 7.40 (m, 1H), 7.33 – 7.29 (m, 1H), 7.27 – 7.20 (m, 3H), 7.19 – 7.11 (m, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 142.1, 136.2, 134.1, 132.1, 132.0, 129.1, 128.6, 128.5, 128.3, 125.4, 123.3, 121.1, 120.9, 111.0, 95.9; ^{77}Se NMR (95 MHz, $CDCl_3$) δ 207.6; HRMS (ESI) *m/z* calcd for $C_{20}H_{15}NSe$ $[M+H]^+$ 349.0365, found 349.0364.

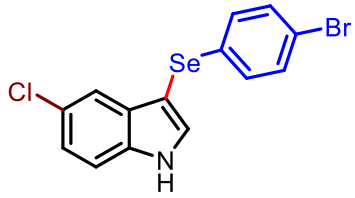


3-Methyl-2-(phenylselanyl)-1H-indole (2n).[6] Dark brown solid, yield 14 mg (25%), ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.98 (bs, 1H), 7.64 (d, *J* = 7.9 Hz, 1H), 7.32 – 7.16 (m, 8H), 2.46 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 137.6, 132.2, 129.4, 129.3, 128.4, 126.4, 123.2, 119.9, 119.6, 119.4, 118.2, 110.8, 10.4; ^{77}Se NMR (75 MHz, $CDCl_3$) δ 257.8; GC-LRMS *m/z* calcd for

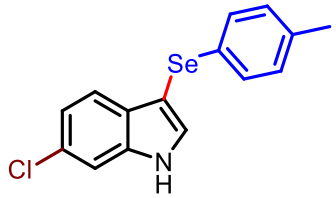
$C_{15}H_{13}NSe$ $[M]^+$ 287.0, found 287.0.



3-((4-Bromophenyl)selanyl)-5-fluoro-1H-indole (2o). White solid, yield 55 mg (75%), ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.58 (bs, 1H), 7.54 (d, *J* = 2.4 Hz, 1H), 7.39 (dd, *J* = 8.8, 4.2 Hz, 1H), 7.29 – 7.26 (m, 3H), 7.13 – 7.08 (m, 2H), 7.04 (td, *J* = 8.9, 2.5 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 158.8 (d, J = 236.9 Hz), 132.97, 132.87, 132.4, 132.0, 130.3, 129.5, 119.7, 112.3 (d, J = 9.5 Hz), 111.7 (d, J = 26.6 Hz), 105.3 (d, J = 24.2 Hz), 97.9 (d, J = 4.6 Hz); ^{77}Se NMR (95 MHz, $CDCl_3$) δ 215.7; HRMS (ESI) *m/z* calcd for $C_{14}H_9BrFNSe$ $[M-H]^+$ 367.8981, found 367.8989.

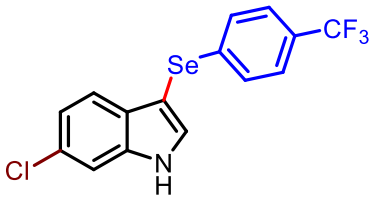


3-((4-Bromophenyl)selanyl)-5-chloro-1H-indole (2p). Olive green solid, yield 62 mg (81%), ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.53 (bs, 1H), 7.62 – 7.60 (m, 1H), 7.51 (d, *J* = 2.5 Hz, 1H), 7.39 – 7.37 (m, 1H), 7.29 – 7.27 (m, 2H), 7.25 (dd, *J* = 8.6, 2.0 Hz, 1H), 7.11 – 7.08 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 134.8, 132.6, 132.4, 132.0, 131.0, 130.3, 127.0, 123.6, 119.75, 119.71, 112.6, 97.6; ^{77}Se NMR (95 MHz, $CDCl_3$) δ 214.4; HRMS (APCI) *m/z* calcd for $C_{14}H_9BrClNSe$ $[M+H]^+$ 385.8840, found 385.8869.

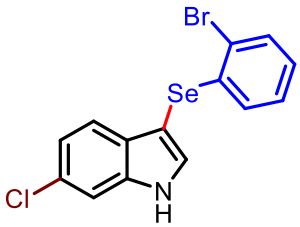


6-Chloro-3-(p-tolylselanyl)-1H-indole (2q). Colorless crystalline solid, yield 48 mg (75%), ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.42 (bs, 1H), 7.56 (d, *J* = 8.5 Hz, 1H), 7.45 (dd, *J* = 14.6, 2.1 Hz, 2H), 7.20 – 7.14 (m, 3H), 7.00 (d, *J* = 7.8 Hz, 2H), 2.28 (s, 3H); ^{13}C NMR (125 MHz,

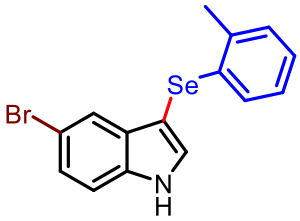
$CDCl_3$) δ 136.7, 135.8, 131.5, 129.9, 129.3, 129.2, 128.9, 128.6, 121.6, 121.4, 111.3, 99.1, 20.9; ^{77}Se NMR (95 MHz, $CDCl_3$) δ 207.0; HRMS (APCI) *m/z* calcd for $C_{15}H_{12}ClNSe$ $[M+H]^+$ 321.9894, found 321.9917.



6-Chloro-3-((4-(trifluoromethyl)phenyl)selanyl)-1H-indole (2r). Buff solid, yield 54 mg (72%), ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.61 (bs, 1H), 7.54– 7.49 (m, 3H), 7.39 (d, *J* = 8.3 Hz, 2H), 7.28 (d, *J* = 8.9 Hz, 2H), 7.19 (dd, *J* = 8.4, 1.8 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 139.1 (q, *J* = 1.4 Hz), 136.8, 132.2, 129.3, 128.3, 128.2, 127.9 (q, *J* = 32.4 Hz), 125.7 (q, *J* = 3.7 Hz), 124.2 (q, *J* = 271.9 Hz), 122.0, 121.1, 111.5, 97.3; ^{77}Se NMR (95 MHz, $CDCl_3$) δ 225.8; HRMS (APCI) *m/z* calcd for $C_{15}H_9ClF_3NSe$ $[M+H]^+$ 375.9611, found 375.9619.

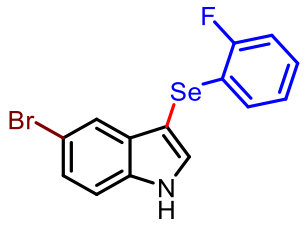


3-((2-Bromophenyl)selanyl)-6-chloro-1H-indole (2s). Light brown solid, yield 60 mg (78%), ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.59 (s, 1H), 7.55 – 7.48 (m, 4H), 7.18 (dd, *J* = 8.5, 1.8 Hz, 1H), 7.01 – 6.95 (m, 2H), 6.67 (dd, *J* = 7.4, 2.1 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 136.9, 136.5, 132.6, 132.5, 129.3, 128.7, 128.5, 127.7, 126.7, 121.9, 121.8, 121.3, 111.5, 98.4; ^{77}Se NMR (95 MHz, $CDCl_3$) δ 237.1; HRMS (APCI) *m/z* calcd for $C_{14}H_9BrClNSe$ $[M+H]^+$ 385.8840, found 385.8871.

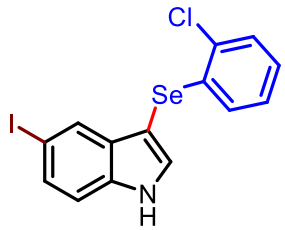


5-Bromo-3-(o-tolylselanyl)-1H-indole (2t).[7] Buff solid, yield 54 mg (74%), ^{1}H NMR (500

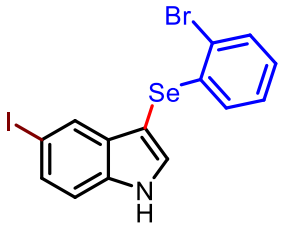
MHz, $CDCl_3$) δ 8.55 (bs, 1H), 7.78 (d, *J* = 1.8 Hz, 1H), 7.47 (d, *J* = 2.6 Hz, 1H), 7.38 (dd, *J* = 8.6, 1.9 Hz, 1H), 7.34 (d, *J* = 8.6 Hz, 1H), 7.17 (d, *J* = 7.2 Hz, 1H), 7.07 (td, *J* = 7.4, 1.3 Hz, 1H), 6.91 (td, *J* = 7.6, 1.4 Hz, 1H), 6.82 (d, *J* = 6.5 Hz, 1H), 2.51 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 136.2, 135.2, 134.1, 132.7, 132.0, 130.0, 127.8, 126.6, 126.0, 125.6, 123.0, 114.4, 112.9, 96.9, 21.3; ^{77}Se NMR (95 MHz, $CDCl_3$) δ 179.8; HRMS (ESI) *m/z* calcd for $C_{15}H_{12}BrNSe$ $[M-H]^+$ 363.9232, found 363.9262.



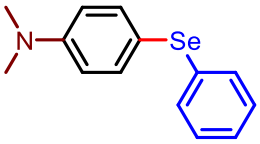
5-Bromo-3-((2-fluorophenyl)selanyl)-1H-indole (2u). Colorless crystalline solid, yield 54 mg (73%), ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.57 (s, 1H), 7.80 (d, *J* = 1.7 Hz, 1H), 7.52 (d, *J* = 2.5 Hz, 1H), 7.41 – 7.33 (m, 2H), 7.16 – 7.11 (m, 1H), 7.04 (td, *J* = 8.6, 8.1, 1.2 Hz, 1H), 6.89 – 6.82 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 159.9, 135.1, 133.1, 131.9, 130.2, 127.5, 126.1, 124.8, 122.9, 120.2, 115.1, 114.6, 113.0, 95.5; ^{77}Se NMR (95 MHz, $CDCl_3$) δ 159.9; HRMS (APCI) *m/z* calcd for $C_{14}H_9BrFNSe$ $[M+H]^+$ 369.9138, found 369.9168.



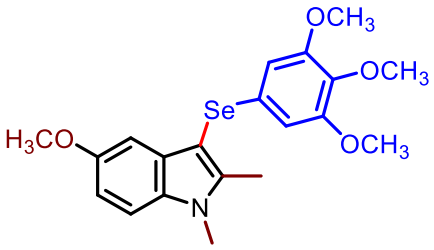
3-((2-Chlorophenyl)selanyl)-5-iodo-1H-indole (2v). Colorless crystalline solid, yield 72 mg (83%), ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.58 (bs, 1H), 8.00 – 7.97 (m, 1H), 7.57 (dd, *J* = 8.6, 1.7 Hz, 1H), 7.47 (d, *J* = 2.6 Hz, 1H), 7.34 (dd, *J* = 7.9, 1.4 Hz, 1H), 7.27 (d, *J* = 8.5 Hz, 1H), 7.08 (td, *J* = 7.6, 1.6 Hz, 1H), 6.96 – 6.92 (m, 1H), 6.69 (dd, *J* = 8.0, 1.6 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 135.7, 134.1, 132.8, 132.5, 132.0, 131.7, 129.3, 129.1, 128.6, 127.3, 126.6, 113.5, 96.2, 85.0; ^{77}Se NMR (95 MHz, $CDCl_3$) δ 212.8; HRMS (APCI) *m/z* calcd for $C_{14}H_9ClINSe$ $[M+H]^+$ 433.8704, found 433.8708.



3-((2-Bromophenyl)selanyl)-5-iodo-1H-indole (2w). Colorless crystalline solid, yield 81 mg (85%), ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.58 (bs, 1H), 8.00 – 7.97 (m, 1H), 7.57 (dd, *J* = 8.5, 1.7 Hz, 1H), 7.52 – 7.45 (m, 2H), 7.27 (d, *J* = 8.5 Hz, 1H), 7.02 – 6.96 (m, 2H), 6.69 – 6.64 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 136.5, 135.7, 132.8, 132.6, 132.4, 131.7, 129.1, 128.7, 127.8, 126.8, 121.8, 113.5, 97.3, 85.0; ^{77}Se NMR (95 MHz, $CDCl_3$) δ 235.6; HRMS (APCI) *m/z* calcd for $C_{14}H_9BrINSe$ $[M+H]^+$ 477.8198, found 477.8192.

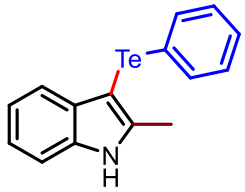


***N,N*-dimethyl-4-(phenylselanyl)aniline (2x)**. yellow liquid, yield 36 mg (65%), ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.56 – 7.52 (m, 2H), 7.35 – 7.31 (m, 2H), 7.25 – 7.21 (m, 2H), 7.20 – 7.16 (m, 1H), 6.73 – 6.70 (m, 2H), 3.02 (s, 6H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 150.5, 137.1, 134.6, 129.8, 129.0, 125.8, 113.7, 113.2, 40.3; ^{77}Se NMR (75 MHz, $CDCl_3$) δ 391.2; GC-LRMS *m/z* calcd for $C_{14}H_{15}NSe$ $[M]^+$ 277.0, found 277.1.

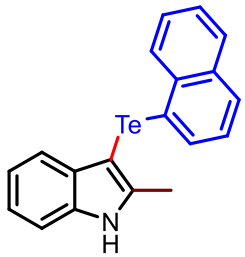


5-Methoxy-1,2-dimethyl-3-((3,4,5-trimethoxyphenyl)selanyl)-1H-indole (II). Light yellow solid, yield 26 mg (25%), ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.23 (d, *J* = 8.8 Hz, 1H), 7.10 (d, *J* = 2.4 Hz, 1H), 6.89 (dd, *J* = 8.8, 2.5 Hz, 1H), 6.46 (s, 2H), 3.85 (s, 3H), 3.79 (s, 3H), 3.77 (s, 3H), 3.69 (s, 6H), 2.58 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 154.9, 153.5, 142.8, 136.2, 132.4, 131.2, 128.7, 111.7, 109.8, 105.7, 101.5, 95.2, 60.8, 56.1, 55.9, 30.6, 12.2; HRMS (ESI)

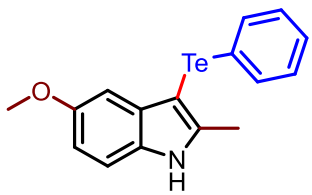
m/z calcd for $C_{20}H_{23}NO_4Se$ $[M+H]^+$ 422.0866, found 422.0854.



2-Methyl-3-(phenyltellanyl)-1H-indole (3a). Brown liquid, yield 41 mg (62%), ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.40 (s, 1H), 7.60 (d, *J* = 7.7 Hz, 1H), 7.40 – 7.34 (m, 3H), 7.24 – 7.18 (m, 2H), 7.15 – 7.12 (m, 1H), 7.11 – 7.07 (m, 2H), 2.67 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 143.3, 137.6, 136.6, 134.0, 129.1, 126.4, 122.2, 121.8, 120.7, 119.6, 110.3, 100.4, 15.5; HRMS (APCI) *m/z* calcd for $C_{15}H_{13}NTe$ $[M]^+$ 337.0105, found 337.0101.

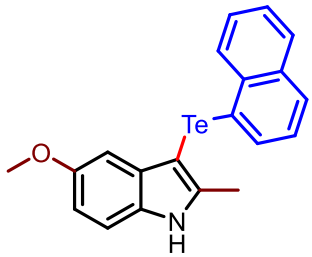


2-Methyl-3-(naphthalen-1-yltellanyl)-1H-indole (3b). Dark brown liquid, yield 46 mg (60%), ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.73 (s, 1H), 8.01 (d, *J* = 8.3 Hz, 1H), 7.81 (d, *J* = 8.1 Hz, 1H), 7.65 (d, *J* = 8.1 Hz, 1H), 7.57 (t, *J* = 7.5 Hz, 2H), 7.51 (t, *J* = 7.7 Hz, 1H), 7.37 (d, *J* = 8.0 Hz, 1H), 7.29 (s, 1H), 7.22 (t, *J* = 7.5 Hz, 1H), 7.15 (t, *J* = 7.5 Hz, 1H), 7.08 (t, *J* = 7.6 Hz, 1H), 2.67 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 143.8, 136.8, 135.2, 133.85, 133.81, 132.4, 129.1, 128.7, 127.0, 126.4, 126.3, 125.9, 122.2, 121.7, 120.7, 119.6, 118.6, 110.4, 15.5; HRMS (APCI) *m/z* calcd for $C_{19}H_{15}NTe$ $[M]^+$ 387.0262, found 387.0291.

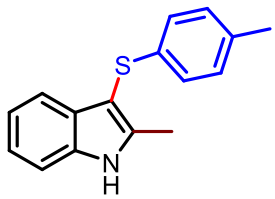


5-Methoxy-2-methyl-3-(phenyltellanyl)-1H-indole (3c). Mustard liquid, yield 42 mg (58%), ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.31 (s, 1H), 7.40 – 7.35 (m, 2H), 7.24 (d, *J* = 8.7 Hz, 1H), 7.17

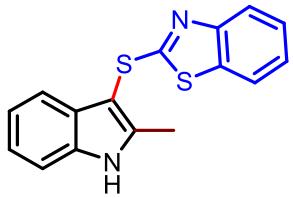
– 7.13 (m, 1H), 7.12 – 7.06 (m, 3H), 6.86 (dd, *J* = 8.7, 2.5 Hz, 1H), 3.86 (s, 3H), 2.64 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 155.0, 144.1, 137.6, 134.7, 133.9, 129.2, 126.4, 116.8, 112.1, 111.3, 103.7, 81.7, 55.9, 15.5; HRMS (ESI) *m/z* calcd for $C_{16}H_{15}NOTe$ $[M-H]^+$ 364.0115, found 364.0167.



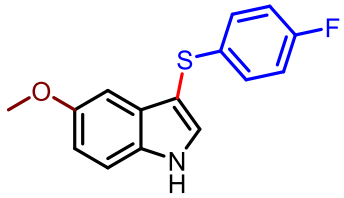
5-Methoxy-2-methyl-3-(naphthalen-1-yltellanyl)-1H-indole (3d). Dark brown liquid, yield 46 mg (55%),^{1}H NMR (500 MHz, $CDCl_3$) δ 8.37 (s, 1H), 8.02 (d, *J* = 8.3 Hz, 1H), 7.82 (d, *J* = 8.1 Hz, 1H), 7.67 (d, *J* = 8.1 Hz, 1H), 7.59 (ddd, *J* = 8.3, 6.9, 1.3 Hz, 1H), 7.53 (ddd, *J* = 8.2, 6.7, 1.2 Hz, 1H), 7.30 (dd, *J* = 7.3, 1.1 Hz, 1H), 7.26 (d, *J* = 8.7 Hz, 1H), 7.12 – 7.09 (m, 1H), 7.06 (d, *J* = 2.4 Hz, 1H), 6.87 (dd, *J* = 8.7, 2.5 Hz, 1H), 3.82 (s, 3H), 2.65 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 155.1, 144.4, 135.2, 134.7, 133.9, 132.2, 131.6, 129.0, 128.7, 127.0, 126.5, 126.3, 125.9, 118.6, 112.3, 111.2, 103.6, 80.8, 55.8, 15.6; HRMS (ESI) *m/z* calcd for $C_{20}H_{17}NOTe$ $[M]^+$ 417.0368, found 417.0384.



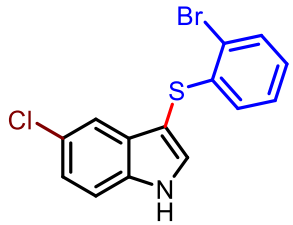
2-Methyl-3-(p-tolylthio)-1H-indole (4a).[8] Brown solid, yield 35 mg (69%), ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.22 (s, 1H), 7.60 (d, *J* = 7.8 Hz, 1H), 7.36 (d, *J* = 8.0 Hz, 1H), 7.23 (td, *J* = 7.5, 1.3 Hz, 1H), 7.17 (td, *J* = 7.6, 7.1, 1.1 Hz, 1H), 7.01 (s, 4H), 2.54 (s, 3H), 2.29 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 140.9, 135.7, 135.4, 134.3, 130.3, 129.5, 125.8, 122.1, 120.6, 119.0, 110.6, 99.9, 20.9, 12.2; GC-LRMS *m/z* calcd for $C_{16}H_{15}NS$ $[M]^+$ 253.1, found 253.1.



2-((2-Methyl-1H-indol-3-yl)thio)benzo[d]thiazole (4b).[9] Pale yellow solid, yield 43 mg (72%), ^{1}H NMR (500 MHz, DMSO-d_6) δ 12.01 (bs, 1H), 7.81 (d, J = 8.2 Hz, 1H), 7.74 (d, J = 8.0 Hz, 1H), 7.50 (dd, J = 7.9, 2.4 Hz, 2H), 7.37 (t, J = 7.7 Hz, 1H), 7.20 (dt, J = 17.7, 7.7 Hz, 2H), 7.10 (t, J = 7.5 Hz, 1H), 2.55 (s, 3H); ^{13}C NMR (125 MHz, DMSO-d_6) δ 174.1, 154.9, 144.0, 136.2, 135.4, 129.4, 126.5, 124.3, 122.4, 121.9, 121.5, 121.1, 117.8, 112.1, 95.5, 12.1; GC-LRMS *m/z* calcd for $C_{16}H_{12}N_2S_2$ $[M]^+$ 296.0, found 296.0.

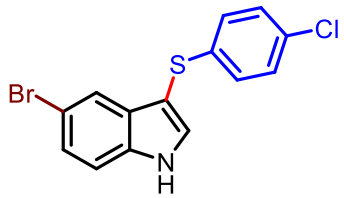


3-((4-Fluorophenyl)thio)-5-methoxy-1H-indole (4c).[10] Brown liquid, yield 34 mg (62%), ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.41 (s, 1H), 7.47 (d, J = 2.7 Hz, 1H), 7.35 (d, J = 8.8 Hz, 1H), 7.11 (dd, J = 8.6, 5.1 Hz, 2H), 7.06 (d, J = 2.4 Hz, 1H), 6.96 – 6.89 (m, 3H), 3.83 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 160.9 (d, J = 244.0 Hz), 155.2, 134.1 (d, J = 3.0 Hz), 131.4, 131.2, 129.7, 127.6 (d, J = 7.7 Hz), 115.7 (d, J = 21.9 Hz), 113.6, 112.5, 102.7, 100.7, 55.8; GC-LRMS *m/z* calcd for $C_{15}H_{12}FNOS$ $[M]^+$ 273.1, found 273.1.

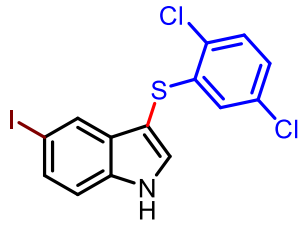


3-((2-Bromophenyl)thio)-5-chloro-1H-indole (4d). Olive solid, yield 47 mg (70%), ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.62 (s, 1H), 7.60 (d, J = 1.9 Hz, 1H), 7.56 (d, J = 2.6 Hz, 1H), 7.54 (dd, J = 7.8, 1.3 Hz, 1H), 7.42 (d, J = 8.6 Hz, 1H), 7.27 (dd, J = 8.7, 2.0 Hz, 1H), 7.03 (td, J = 7.7, 1.4 Hz, 1H), 6.96 (td, J = 7.6, 1.6 Hz, 1H), 6.62 (dd, J = 7.9, 1.5 Hz, 1H); ^{13}C NMR (125 MHz,

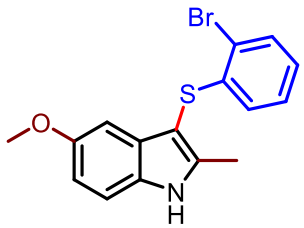
$CDCl_3$) δ 140.0, 134.9, 132.7, 132.6, 130.2, 127.6, 127.2, 126.2, 125.9, 123.8, 119.7, 119.1, 112.8, 101.9; HRMS (APCI) *m/z* calcd for $C_{14}H_9BrClNS$ $[M+H]^+$ 339.9379, found 339.9389.



5-Bromo-3-((4-chlorophenyl)thio)-1H-indole (4e).[11] Olive green solid, yield 44 mg (65%), ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.52 (bs, 1H), 7.74 (d, *J* = 1.7 Hz, 1H), 7.51 (d, *J* = 2.7 Hz, 1H), 7.39 (dd, *J* = 8.6, 1.9 Hz, 1H), 7.34 (d, *J* = 8.6 Hz, 1H), 7.18 – 7.15 (m, 2H), 7.04 – 7.01 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 137.3, 135.1, 131.9, 130.9, 130.7, 128.9, 127.1, 126.3, 122.1, 114.6, 113.2, 102.4; GC-LRMS *m/z* calcd for $C_{14}H_9BrClNS$ $[M]^+$ 336.9, found 338.9.



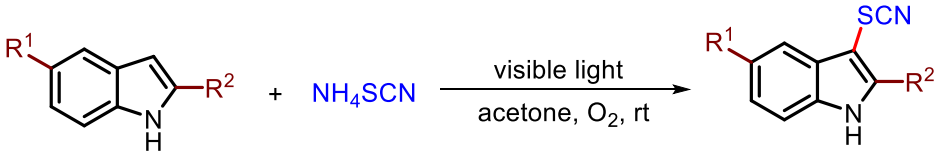
3-((2,5-Dichlorophenyl)thio)-5-iodo-1H-indole (4f). Buff solid, yield 65 mg (78%), ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.61 (bs, 1H), 7.93 (s, 1H), 7.58 (dd, *J* = 8.6, 1.7 Hz, 1H), 7.50 (d, *J* = 2.7 Hz, 1H), 7.38 (d, *J* = 2.2 Hz, 1H), 7.28 (d, *J* = 8.8 Hz, 1H),6.96 (dd, *J* = 8.6, 2.2 Hz, 1H), 6.54 (d, *J* = 8.6 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 136.9, 135.7, 132.1, 132.0, 131.2, 130.7, 130.6, 129.1, 128.2, 127.3, 127.0, 113.7, 100.3, 85.2; HRMS (APCI) *m/z* calcd for $C_{14}H_8Cl_2INS$ $[M]^+$ 418.8794, found 418.8823.



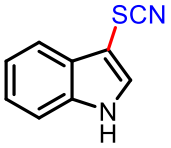
3-((2-Bromophenyl)thio)-5-methoxy-2-methyl-1H-indole (4g). Light brown solid, yield 47 mg (67%), ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.28 (s, 1H), 7.53 (dd, *J* = 7.8, 1.3 Hz, 1H), 7.27 (d, *J* = 8.7 Hz, 1H), 7.04 – 6.99 (m, 2H), 6.95 – 6.92 (m, 1H), 6.88 (dd, *J* = 8.7, 2.5 Hz, 1H), 6.56

(dd, *J* = 7.9, 1.6 Hz, 1H), 3.82 (s, 3H), 2.50 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 155.2, 142.3, 140.3, 132.6, 130.9, 130.3, 127.5, 125.9, 125.4, 119.6, 112.5, 111.5, 100.6, 98.1, 55.8, 12.3; HRMS (ESI) *m/z* calcd for $C_{16}H_{14}BrNOS$ $[M-H]^+$ 347.9875, found 347.9896.

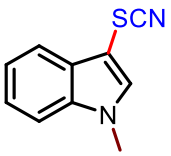
3. General experimental procedure for thiocyanation of indoles



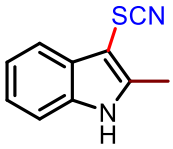
Indoles (0.2 mmol, 1.0 equiv.) ammonium thiocyanate (0.4 mmol, 2 equiv.) were added to a 5 mL round bottom flask with magnetic stir bar in acetone (O_2 purged, 2 mL). The reaction mixture was stirred for 8 h under sunlight. After completion of the reaction, solvent was removed on rotary evaporator under vacuum. The residue was purified by flash column chromatography on silica gel to afford the desired product.



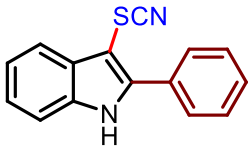
3-Thiocyanato-1H-indole (5a).[12] White solid, yield 26 mg (74%), ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.83 (s, 1H), 7.85 – 7.81 (m, 1H), 7.47 (d, *J* = 2.8 Hz, 1H), 7.45 – 7.42 (m, 1H), 7.36 – 7.32 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 136.1, 131.1, 127.7, 123.8, 121.9, 118.7, 112.23, 112.21, 91.9; GC-LRMS *m/z* calcd for $C_9H_6N_2S$ $[M]^+$ 174.0, found 174.1.



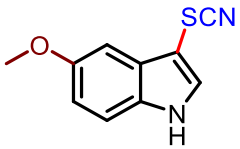
1-Methyl-3-thiocyanato-1H-indole (5b).[12] White solid, yield 25 mg (67%), ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.87 – 7.80 (m, 1H), 7.43 – 7.38 (m, 3H), 7.38 – 7.33 (m, 1H), 3.82 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 137.2, 135.1, 128.5, 123.4, 121.6, 118.9, 111.9, 110.2, 89.9, 33.4; GC-LRMS *m/z* calcd for $C_{10}H_8N_2S$ $[M]^+$ 188.0, found 188.1.



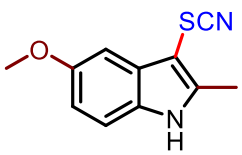
2-Methyl-3-thiocyanato-1H-indole (5c).[12] Colorless crystalline solid, yield 27 mg (71%), ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.59 (s, 1H), 7.73 – 7.70 (m, 1H), 7.34 – 7.30 (m, 1H), 7.29 – 7.24 (m, 2H), 2.51 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 142.1, 135.1, 128.7, 123.0, 121.6, 118.1, 112.2, 111.3, 88.8, 12.0; GC-LRMS *m/z* calcd for $C_{10}H_8N_2S$ $[M]^+$ 188.0, found 188.1.



2-Phenyl-3-thiocyanato-1H-indole (5d).[12] Pale orange solid, yield 32 mg (65%), ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.75 (s, 1H), 7.91 – 7.82 (m, 1H), 7.81 – 7.70 (m, 2H), 7.62 – 7.55 (m, 2H), 7.55 – 7.51 (m, 1H), 7.48 – 7.45 (m, 1H), 7.39 – 7.34 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 143.1, 135.4, 130.0, 129.7, 129.6, 129.1, 128.6, 124.1, 122.1, 119.1, 111.9, 111.6. 89.2; HRMS (ESI) *m/z* calcd for $C_{15}H_{10}N_2S$ $[M+Na]^+$ 273.0457, found 273.0456.

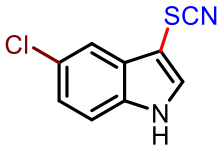


5-Methoxy-3-thiocyanato-1H-indole (5e).[12] Beige solid, yield 28 mg (69%), ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.74 (s, 1H), 7.46 (d, *J* = 2.9 Hz, 1H), 7.32 (d, *J* = 8.9 Hz, 1H), 7.21 (d, *J* = 2.4 Hz, 1H), 6.97 (dd, *J* = 8.9, 2.4 Hz, 1H), 3.94 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 155.7, 131.5, 130.9, 128.5, 114.5, 113.1, 112.1, 99.8, 91.4, 55.9; HRMS (ESI) *m/z* calcd for $C_{10}H_8N_2OS$ $[M+Na]^+$ 227.0250, found 227.0239.

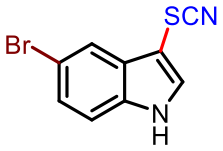


5-Methoxy-2-methyl-3-thiocyanato-1H-indole (5f). Beige solid, yield 27 mg (63%), ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.61 (s, 1H), 7.19 (dd, *J* = 8.8, 1.0 Hz, 1H), 7.15 – 7.12 (m, 1H),

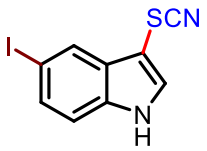
6.89 – 6.86 (m, 1H), 3.93 (d, *J* = 1.4 Hz, 3H), 2.51 (d, *J* = 1.2 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 155.5, 142.5, 129.9, 129.6, 112.9, 112.2, 112.1, 99.9, 88.3, 55.9 (d, *J* = 1.5 Hz), 12.1 (d, *J* = 1.0 Hz).



5-Chloro-3-thiocyanato-1H-indole (5g).[12] White solid, yield 25 mg (60%), ^{1}H NMR (500 MHz, DMSO-d_6) δ 7.98 (s, 1H), 7.64 (s, 1H), 7.55 (d, *J* = 8.7 Hz, 1H), 7.27 (d, *J* = 8.6 Hz, 1H); ^{13}C NMR (125 MHz, DMSO-d_6) δ 135.1, 134.9, 129.0, 126.4, 123.6, 117.4, 115.0, 112.8, 90.0; HRMS (ESI) *m/z* calcd for $C_9H_5ClN_2S$ $[M+H]^+$ 208.9935, found 208.9933.



5-Bromo-3-thiocyanato-1H-indole (5h).[12] White solid, yield 28 mg (56%), ^{1}H NMR (500 MHz, DMSO-d_6) δ 12.20 (s, 1H), 8.06 (d, *J* = 2.1 Hz, 1H), 7.82 (d, *J* = 1.8 Hz, 1H), 7.52 (d, *J* = 8.6 Hz, 1H), 7.41 (dd, *J* = 8.6, 1.9 Hz, 1H); ^{13}C NMR (125 MHz, DMSO-d_6) δ 135.6, 135.1, 129.7, 126.1, 120.4, 115.4, 114.2, 112.6, 89.9; HRMS (APCI) *m/z* calcd for $C_9H_5BrN_2S$ $[M]^+$ 253.9331 found 253.9329.



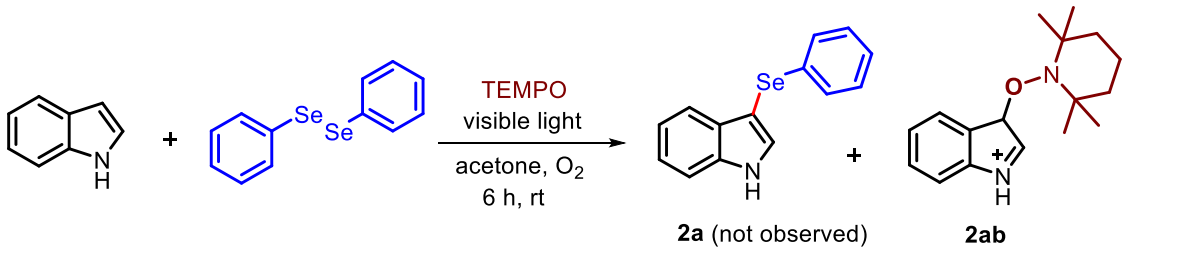
5-Iodo-3-thiocyanato-1H-indole (5i). Beige solid, yield 31 mg (51%), ^{1}H NMR (500 MHz, DMSO-d_6) δ 7.96 (d, *J* = 1.3 Hz, 1H), 7.91 (s, 1H), 7.53 (dd, *J* = 8.5, 1.6 Hz, 1H), 7.39 (d, *J* = 8.5 Hz, 1H); ^{13}C NMR (125 MHz, DMSO-d_6) δ 135.7, 134.2, 131.6, 130.2, 126.5, 115.7, 112.8, 89.5, 85.5; HRMS (ESI) *m/z* calcd for $C_9H_5IN_2S$ $[M+Na]^+$ 322.9110, found 322.9112.

Mechanistic investigation

Control Experiment with radical scavenger

Reaction was performed in the presence of a radical scavenger TEMPO (2 equiv. 0.2 mmol, 31.2 mg) under the standard reaction conditions. The reaction mixture was stirred for 6 h. The reaction was completely inhibited, showing the participation of radical species in this transformation. The mass analysis of the reaction mixture showed no desired product (**2a**) was observed; instead, TEMPO-indole adduct (**2ab**) was formed (Scheme S1 and Figure S1).

Scheme S1. Optimized reaction with TEMPO



Acquisition Parameter					
Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.2 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	7.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste

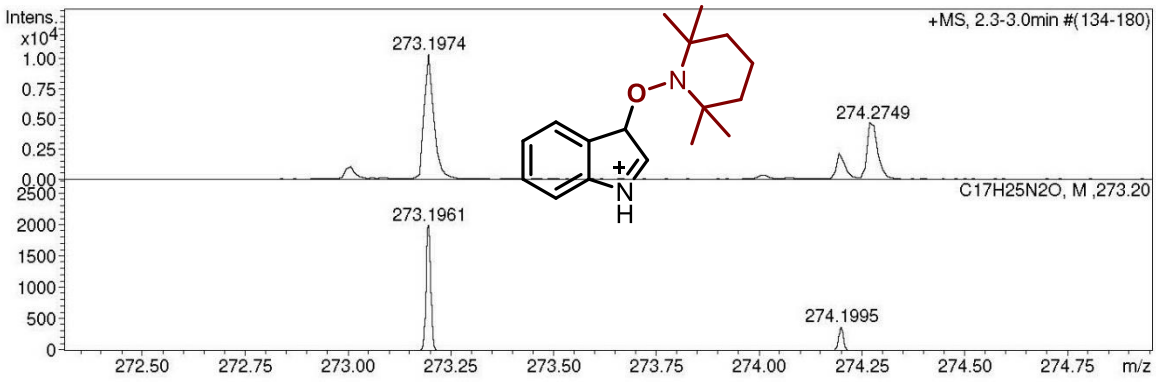
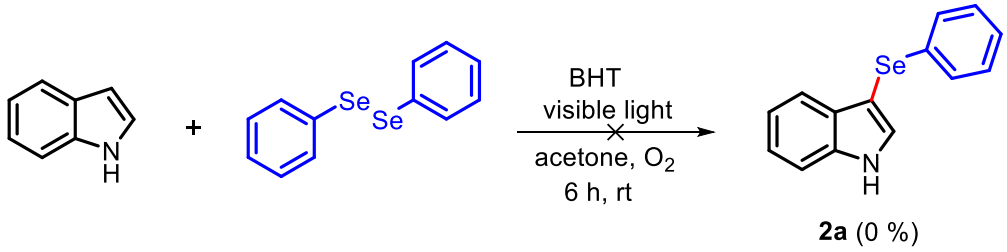


Figure S1. HRMS of the crude reaction mixture containing TEMPO

Next control experiment was carried out in the presence of another radical scavenger 2,6-Di-*tert*-butyl-4-methylphenol (BHT, 2 equiv. 0.2 mmol, 44 mg) . The reaction mixture was stirred

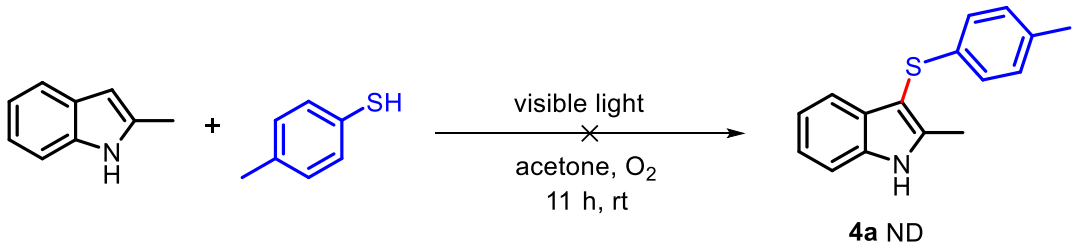
for 6 h under sunlight. No desired product (**2a**) was observed (Scheme S2).

Scheme S2. Optimized reaction with BHT



Afterward, the reaction of *para*-toluenethiol (0.5 equiv) with 2-methyl indole (1 equiv) under the optimized reaction conditions was studied (Scheme S3). Desired 3-sulfenyl indole **4a** was not realized, although, trace of oxidized *para*-tolyl disulfide was observed.

Scheme S3. Reaction of *para*-toluenethiol to 2-methyl indole



Electrochemical analysis of catalyst

Electrochemical cyclic voltammetry (CV) experiment were carried out by three electrode configurations with a glassy carbon (GC) working electrode, a platinum counter electrode, and standard calomel electrode (SCE).

Reaction condition: Indole **1a** (2 mM) in 5 mL CH_3CN containing 0.1 M tetrabutylammonium hexafluorophosphate ($TBAPF_6$) with scan rate 5 m Vs^{-1}. E_{ox} = 0.455 V, E_{red} = 0.540 V, $E_{1/2}$= 0.497 V.

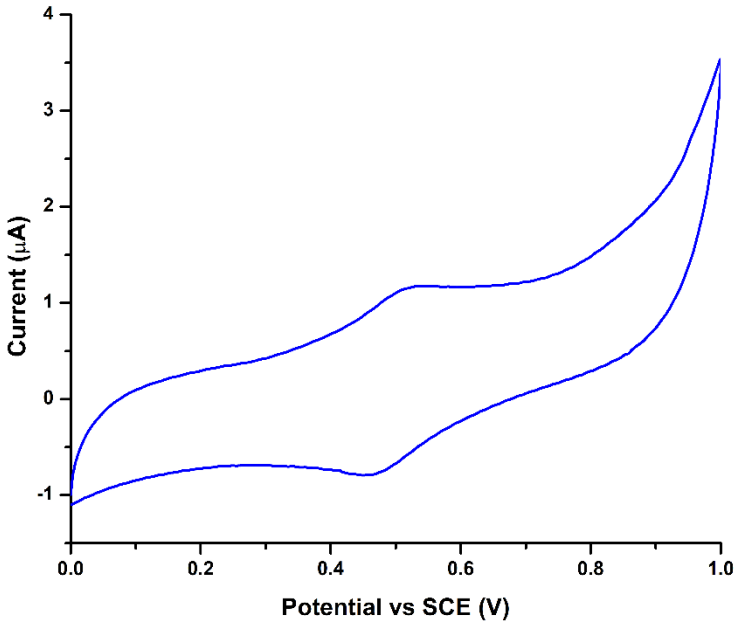
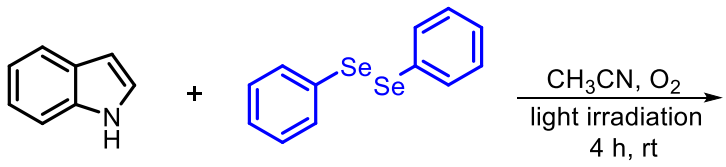


Figure S2. Cyclic Voltammogram of **1a** in CH_3CN

EPR experiment

To investigate the reaction pathways, EPR experiment was conducted on the reaction mixture (Scheme S4). The reaction was carried out using indole (0.1 mmol) and Diselenide (0.05 mmol) in 5 mL RB in CH_3CN. The reaction mixture was irradiated under sunlight for 5 h then transferred to EPR tube and EPR spectra was recorded. The presence of radical in EPR spectrum of the reaction mixture suggests that the radicals are involved in the reaction mixture.

Scheme S4. Reaction with diselenide in acetonitrile under O_2 atmosphere



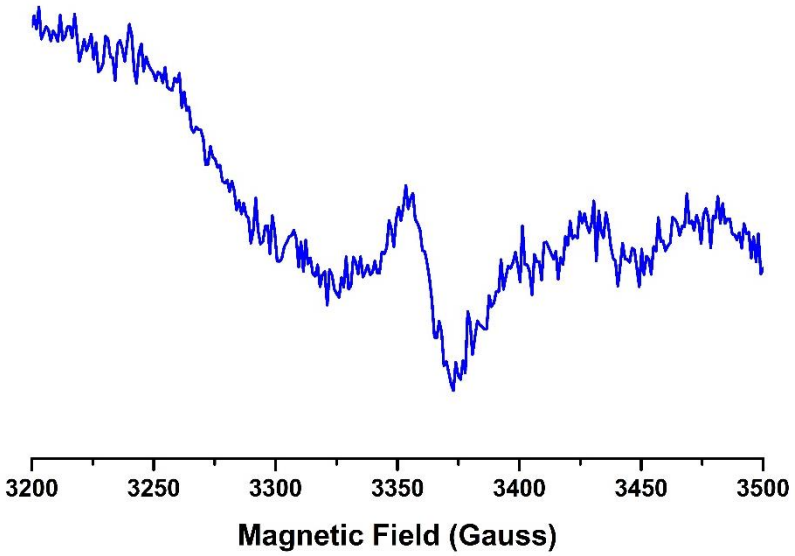
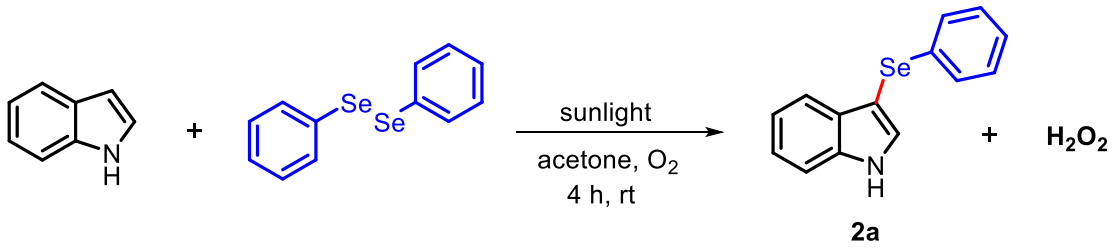


Figure S3. EPR spectrum of reaction mixture in CH_3CN

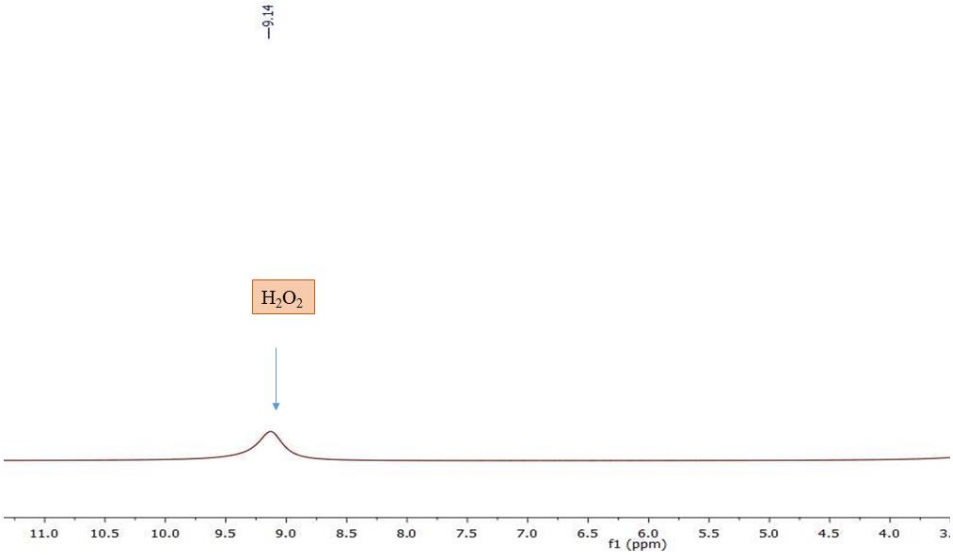
Hydrogen peroxide detection experiment

The reaction was carried out at 0.1 mmol of **1a** using 0.05 mmol of **1ab** in in 5 mL RB then 0.6 mL of CD_3CN was added to the mixture (Scheme S5). The reaction mixture was irradiated under visible light for 4 h then transferred to NMR tube and NMR spectra was recorded. NMR study suggests the formation of hydrogen peroxide in the reaction.

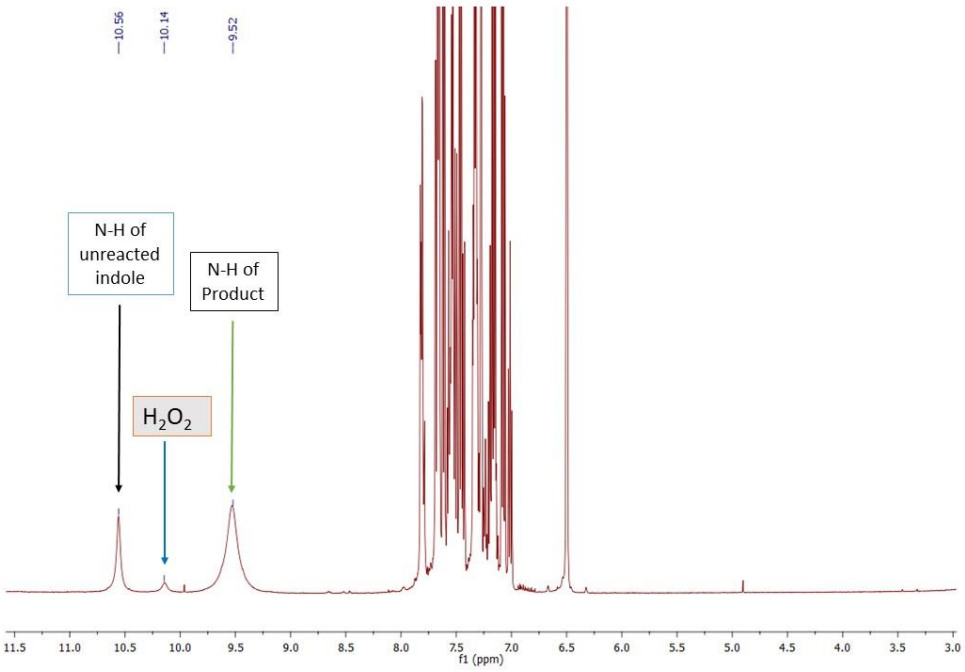
Scheme S5. Hydrogen peroxide evolution experiment



^{1}H NMR of commercially available hydrogen peroxide in CD_3CN



^{1}H NMR of the crude reaction mixture of 2a (peak at 10.14 ppm belongs to hydrogen peroxide)



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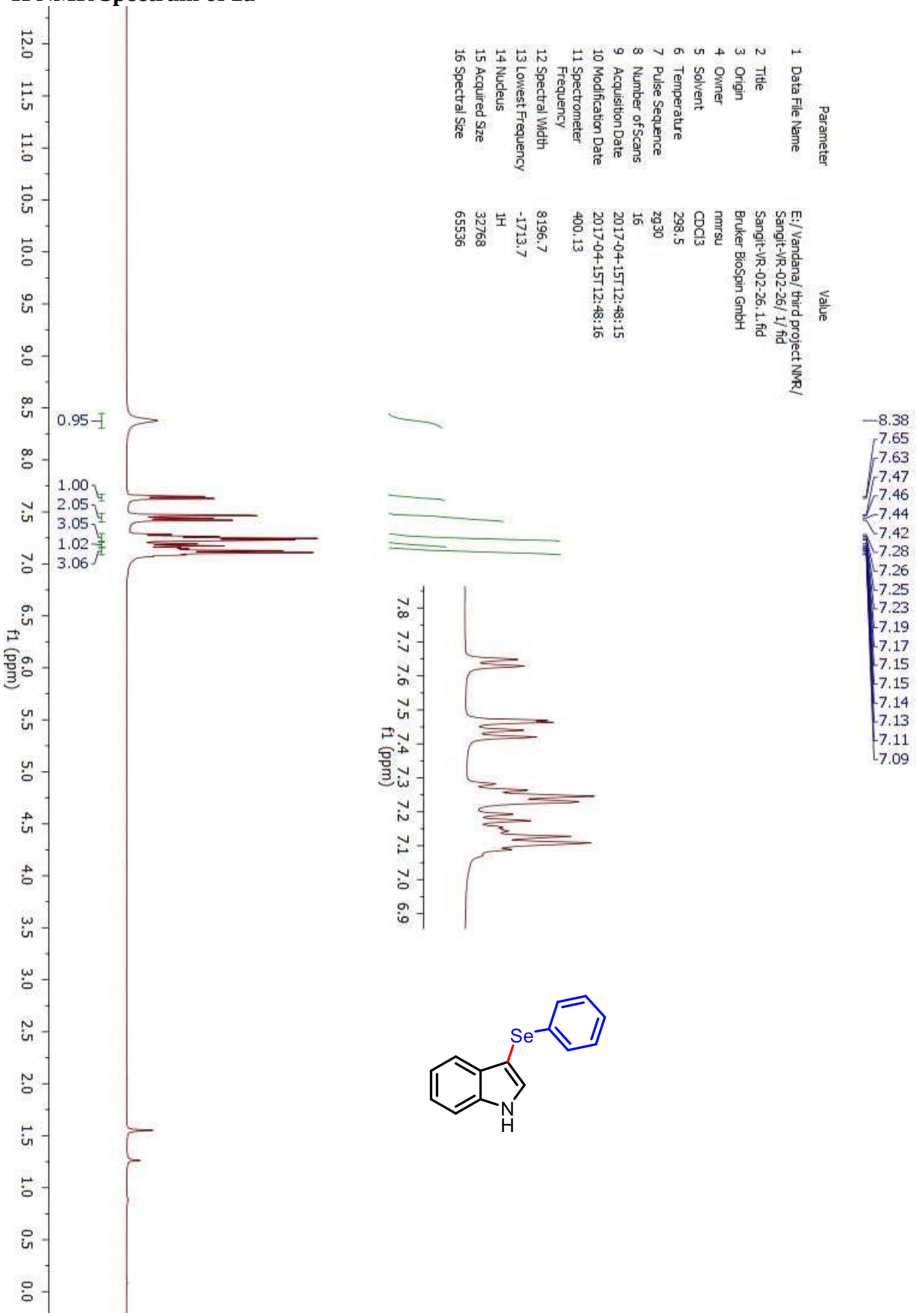
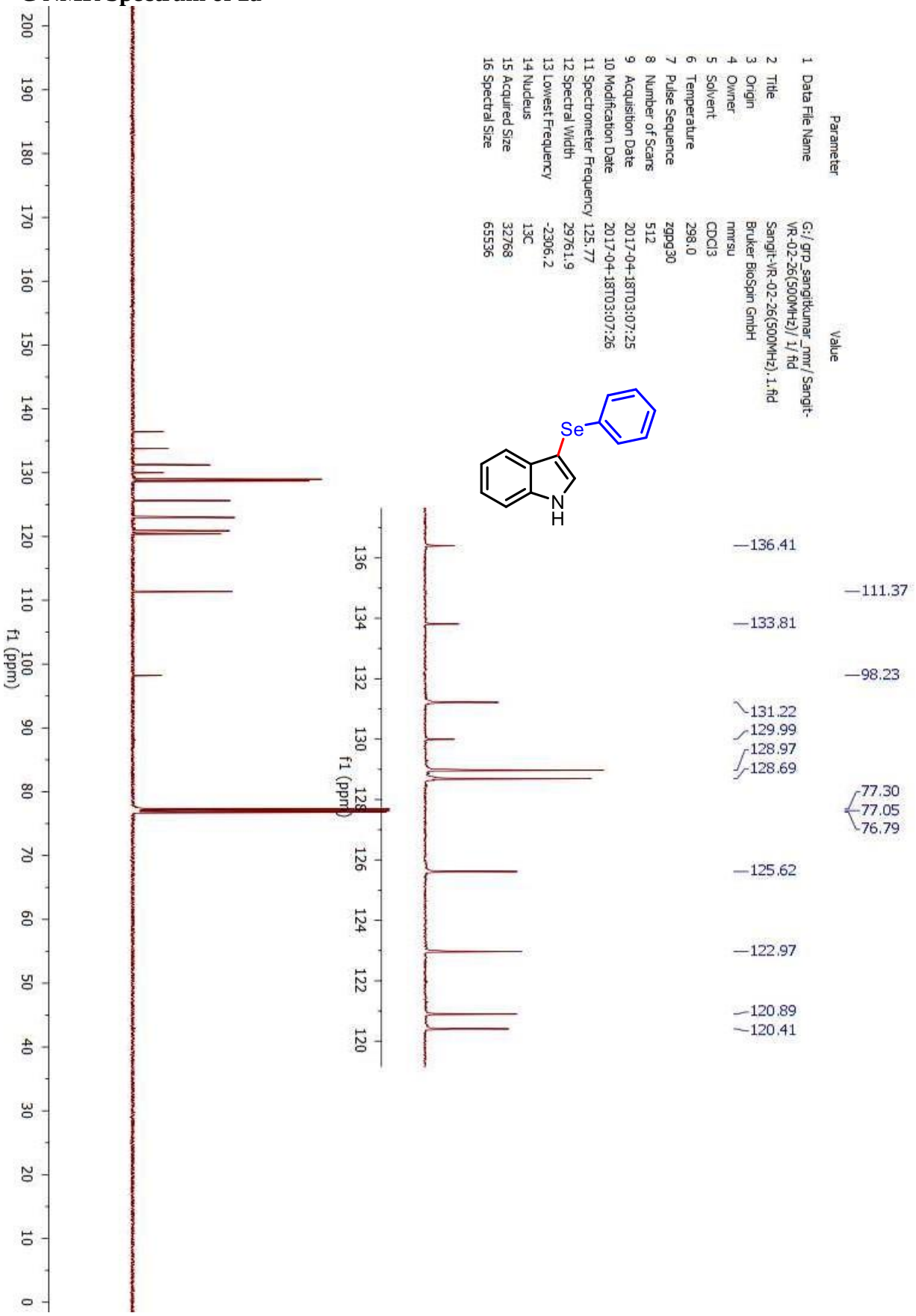
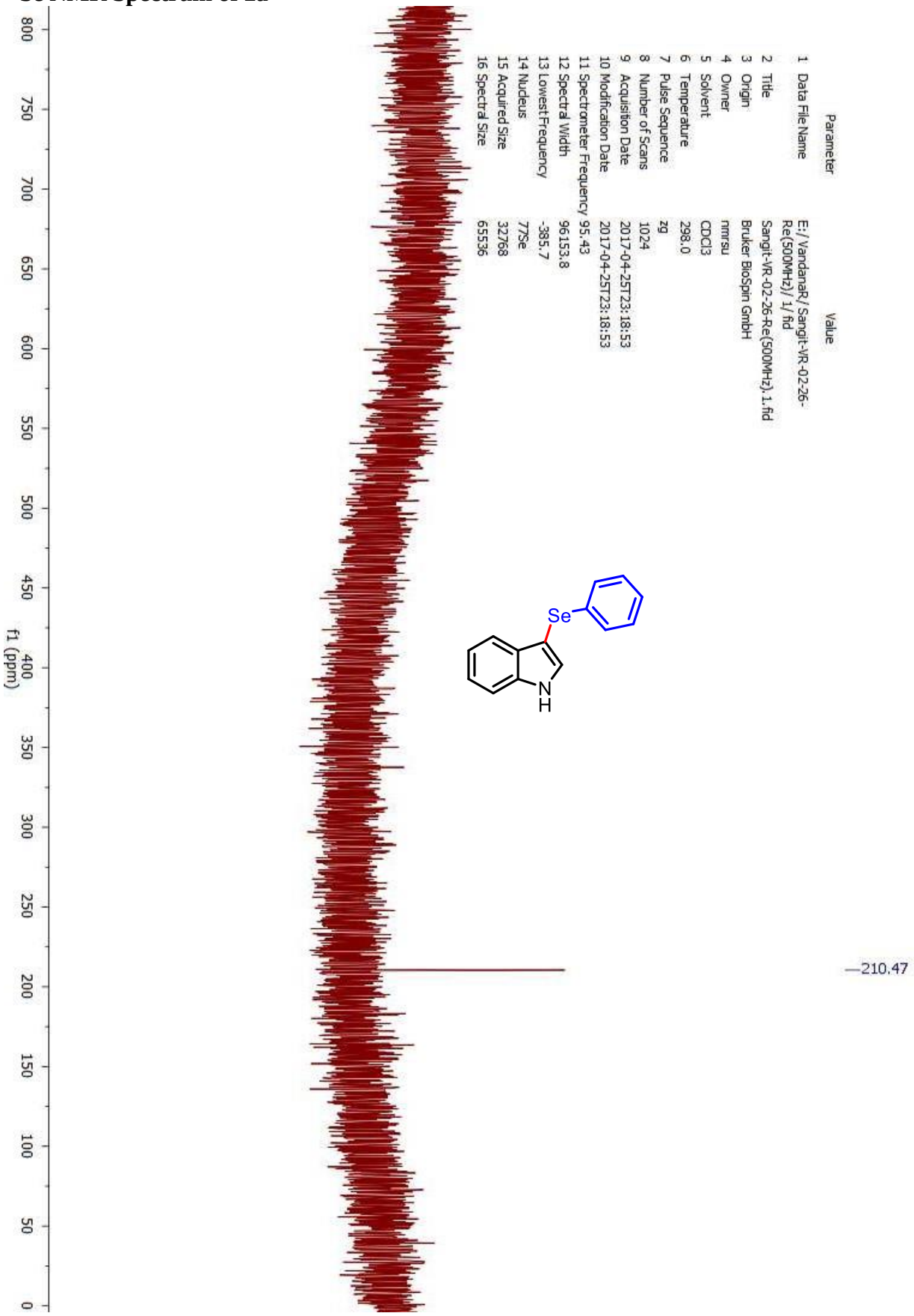
^{1}H NMR Spectrum of 2a



^{13}C NMR Spectrum of 2a



^{77}Se NMR Spectrum of 2a



Mass Spectrum of 2a

Acquisition date: 15/11/17

Sample ID: VR-02-26

Supervisor: Dr S Kumar

Operator: **IISERB-CIF-Mass Facility**

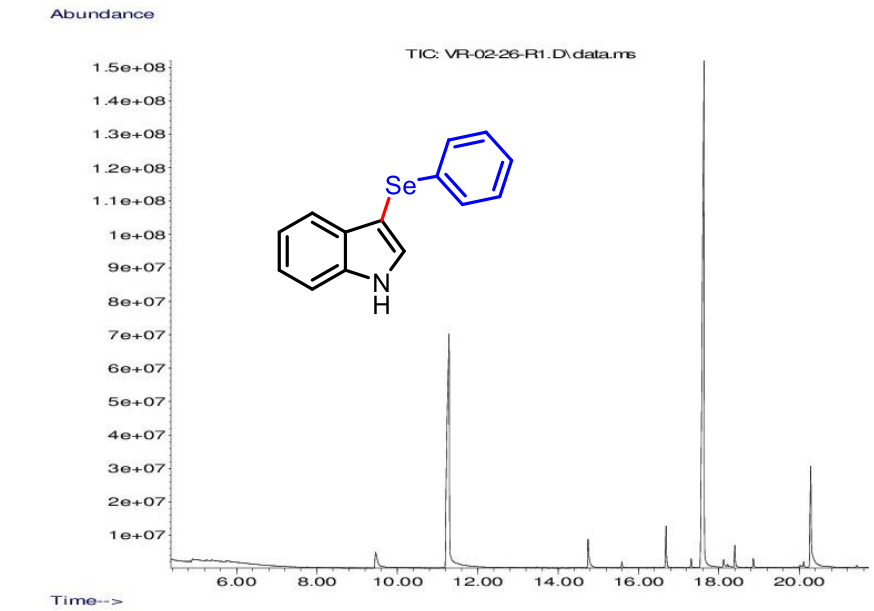
***Instrument*:** **Agilent 7890A GC with 5975C MS system**

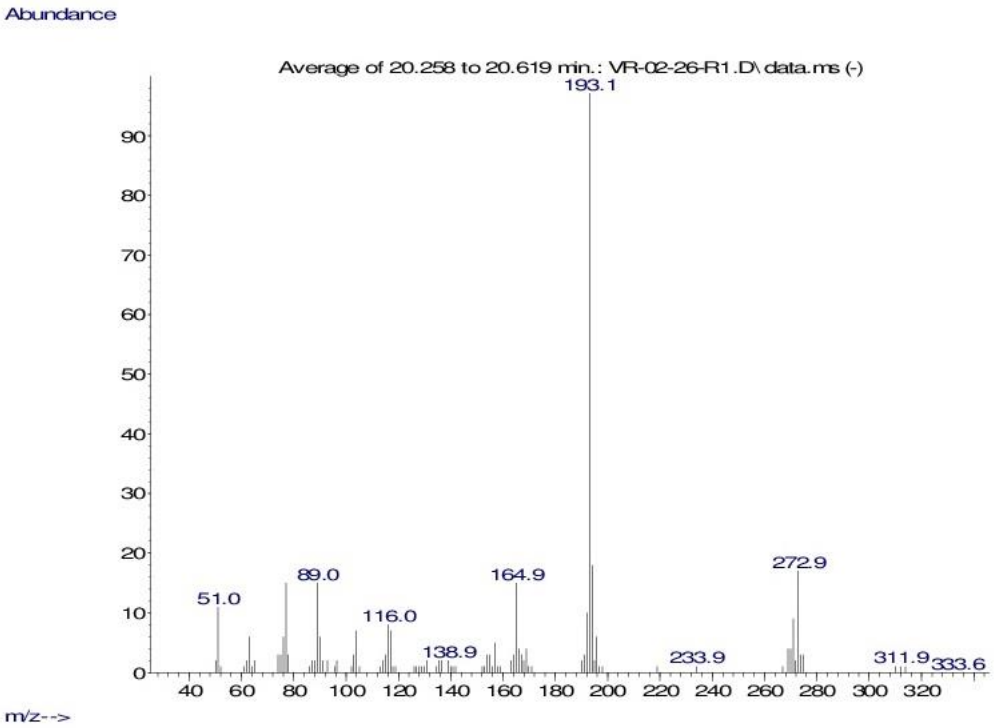
***Column*: HP-5**

Ionization: **EI (70 eV)**

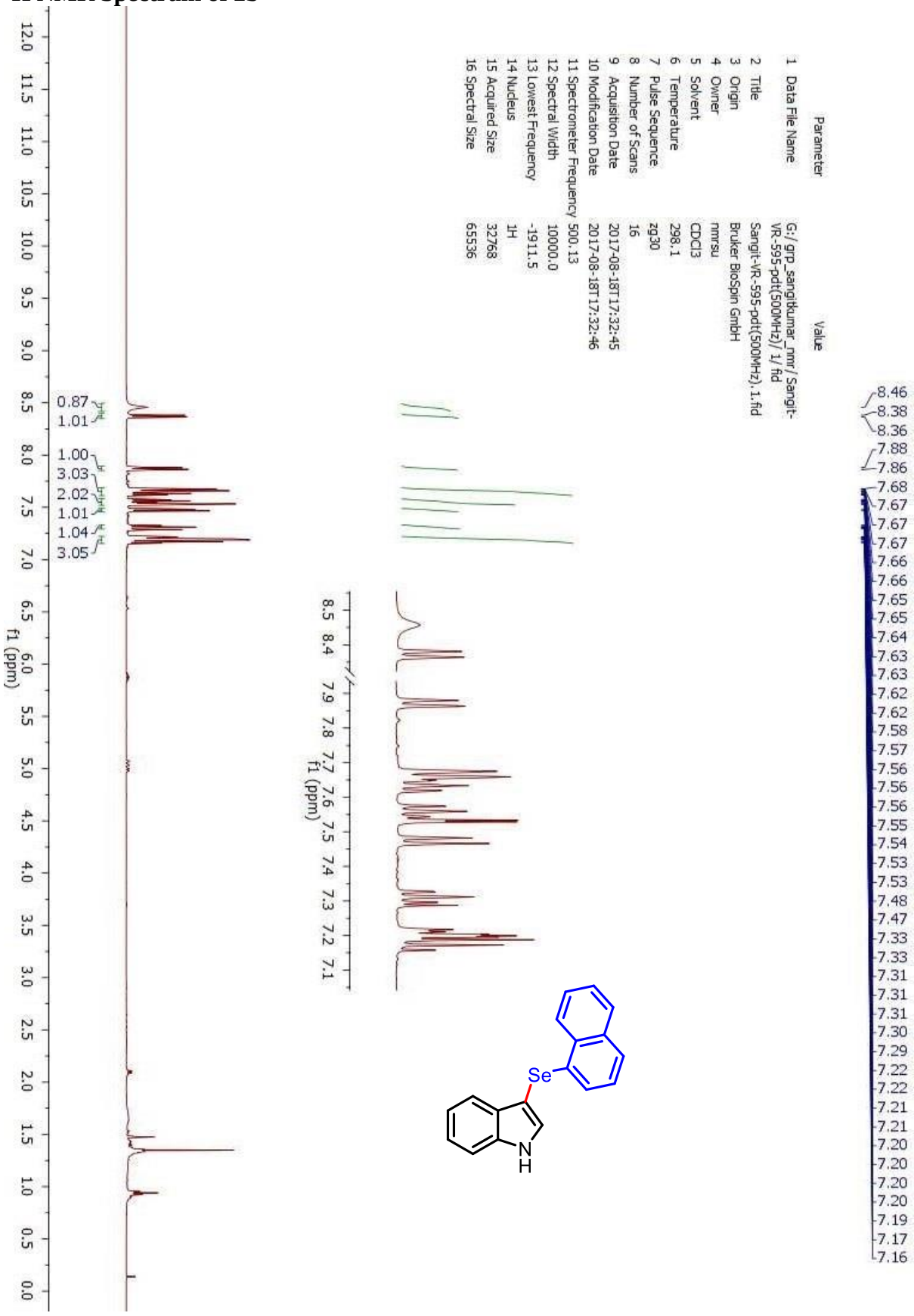
Method: **General_1_HP5_80_DEG.M**

***MSD*: Single Quad.**

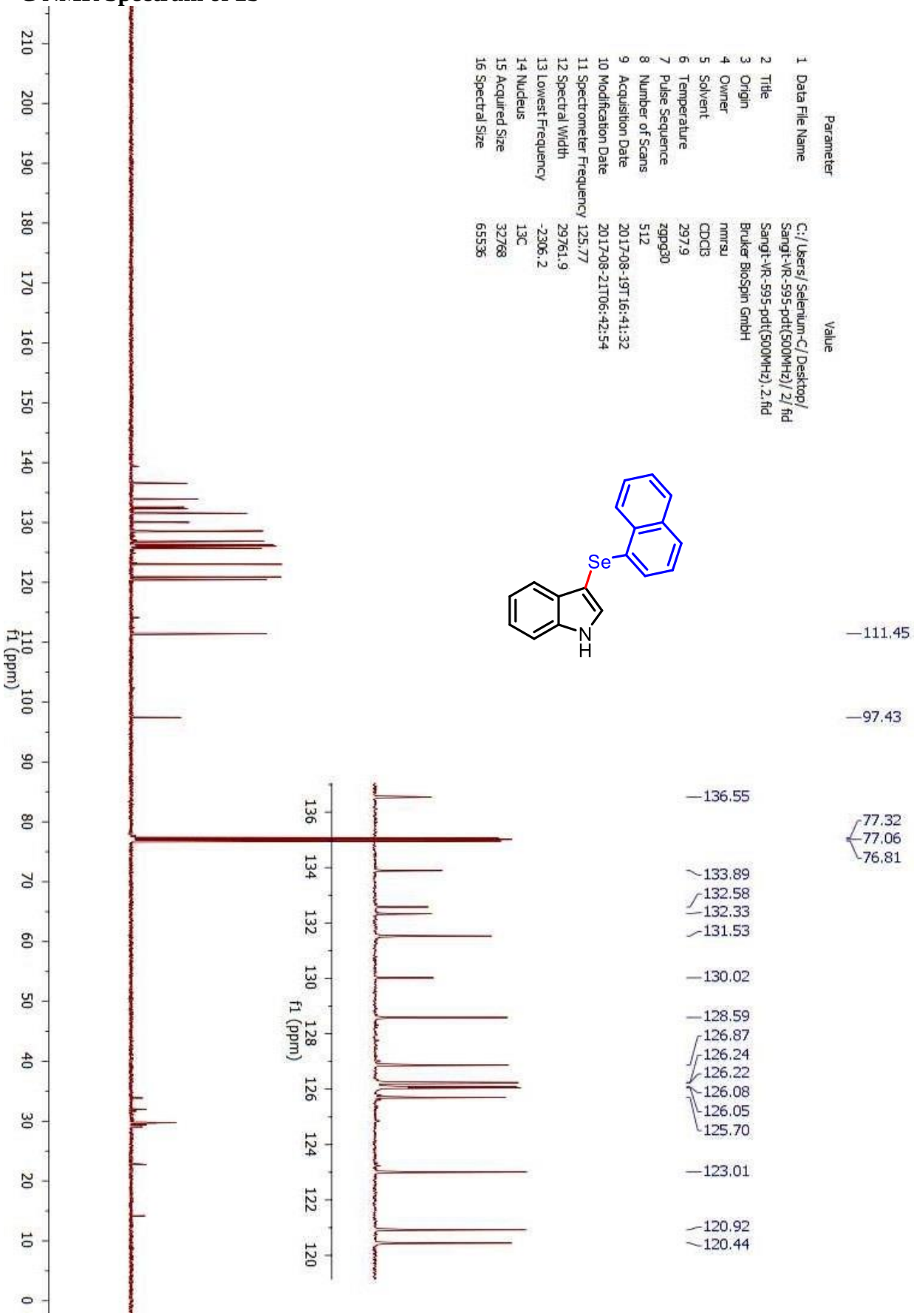




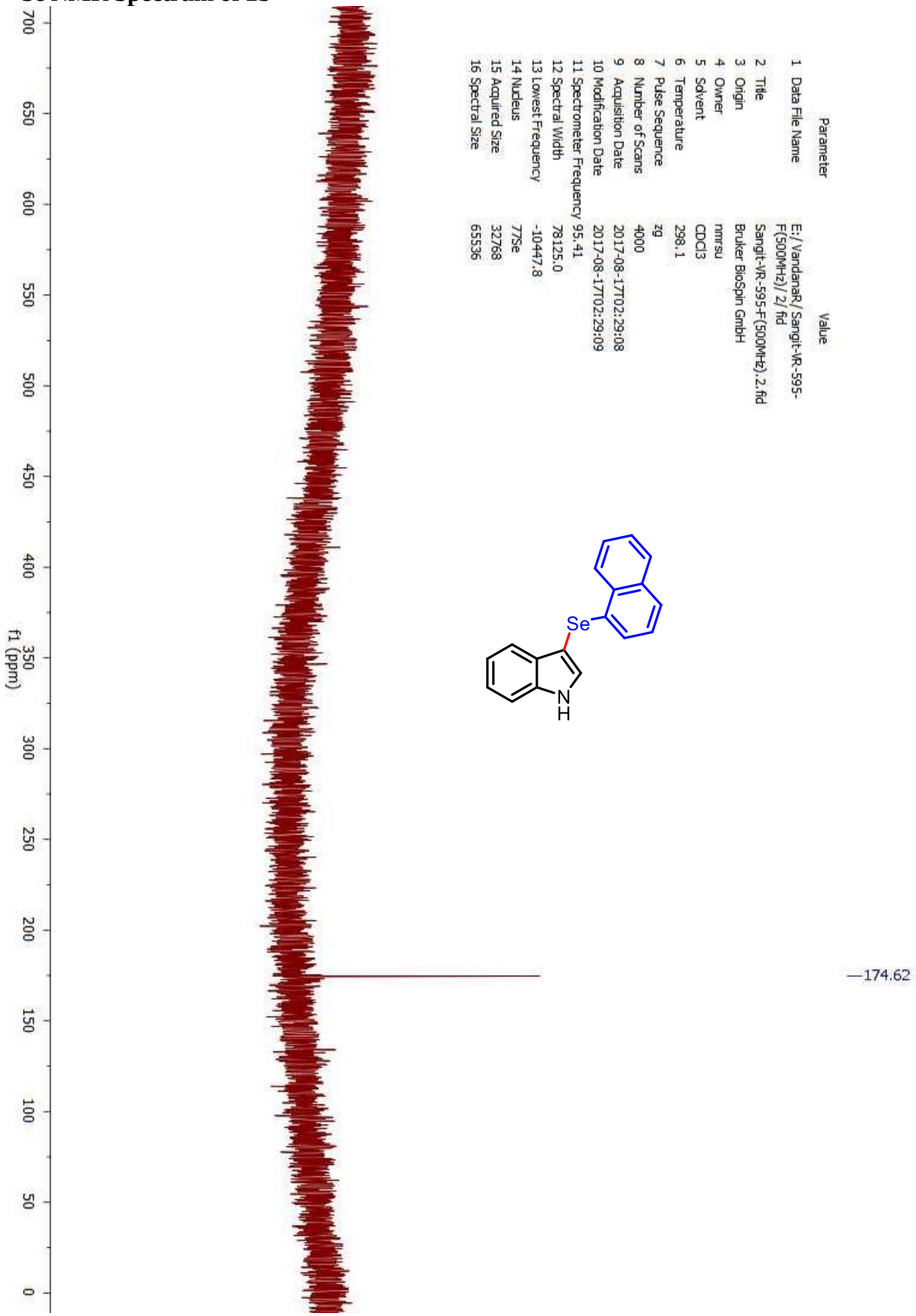
^{1}H NMR Spectrum of 2b



^{13}C NMR Spectrum of 2b



^{77}Se NMR Spectrum of 2b



Mass Spectrum of 2b

Acquisition date: 11/12/17

Sample ID: VR-595

Supervisor: Dr. S Kumar

Operator: **IISERB-CIF-Mass Facility**

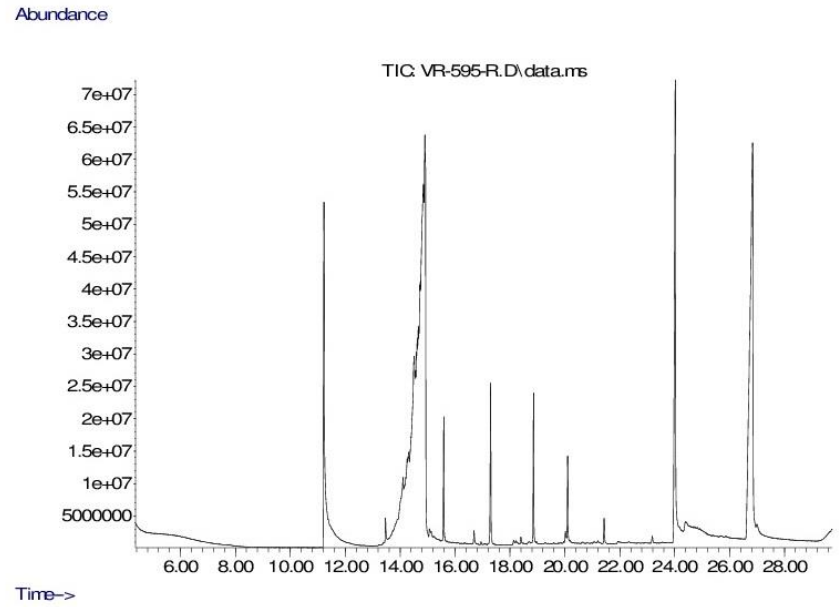
***Instrument*: Agilent 7890A GC with 5975C MS system**

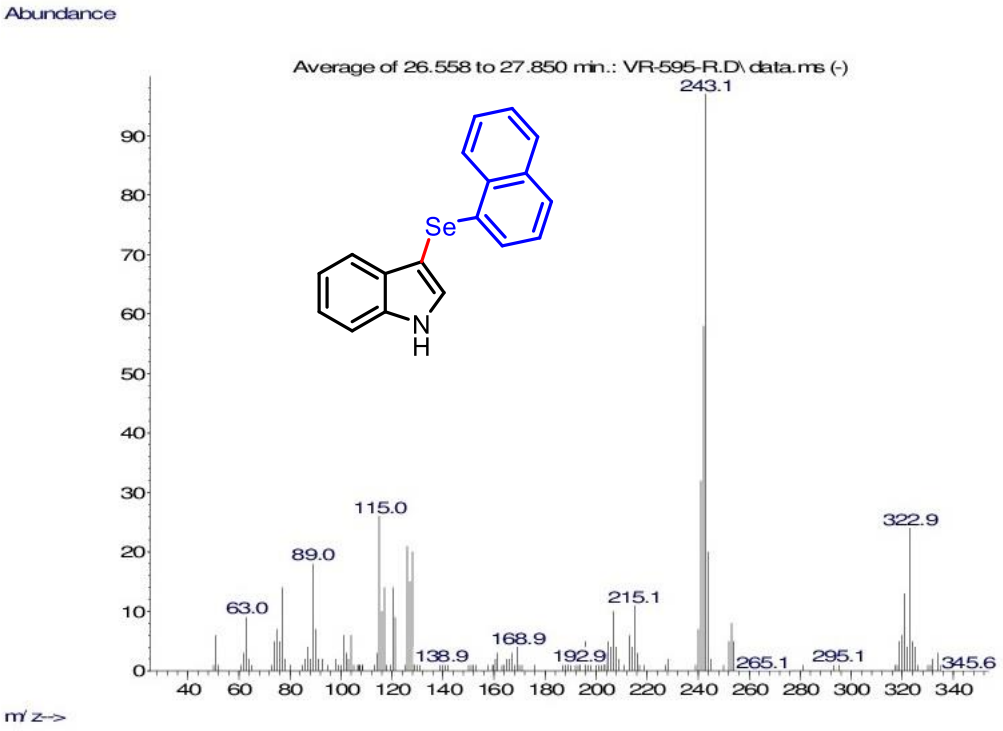
***Column*: HP-5**

Ionization: **EI (70 eV)**

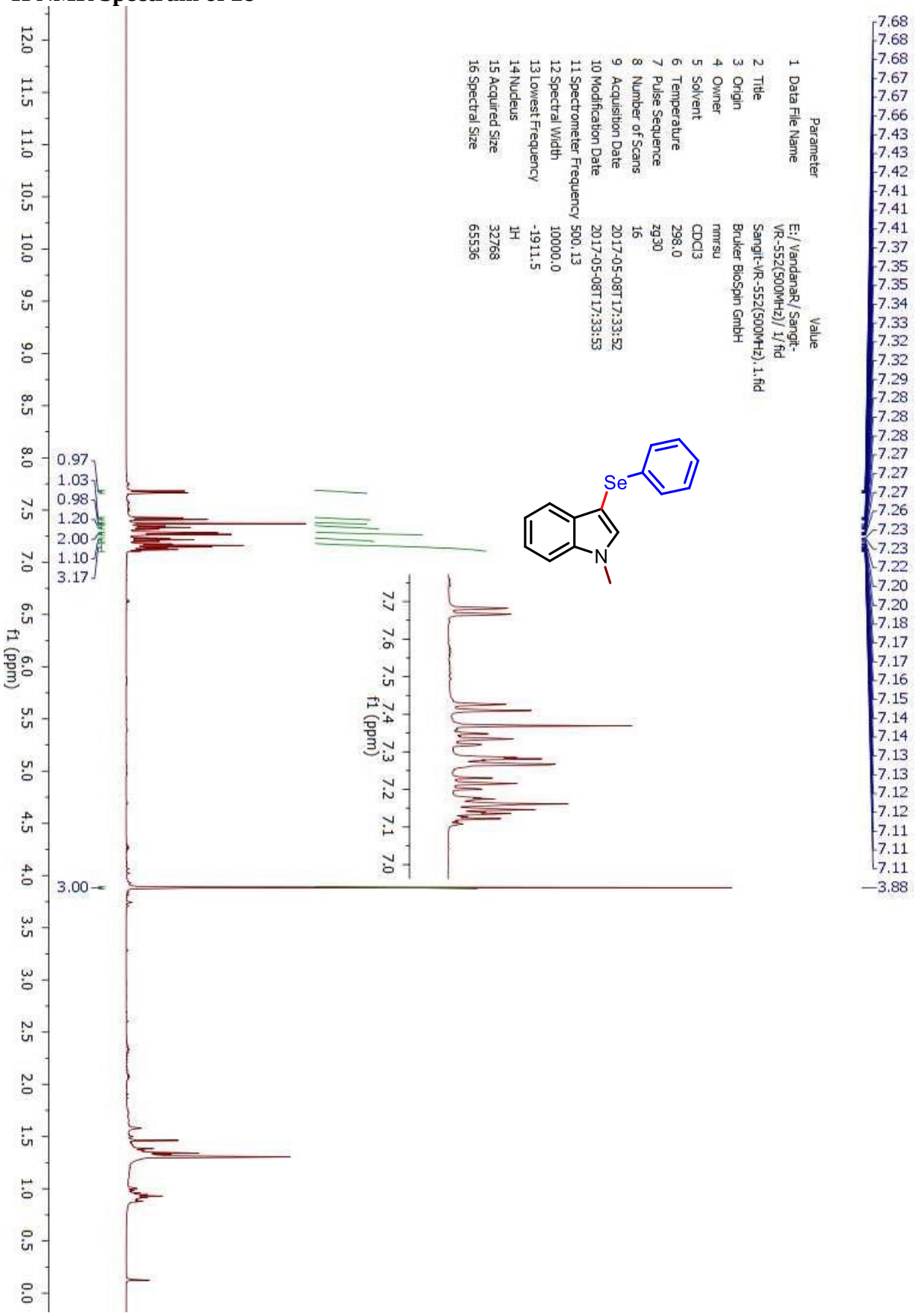
Method: **General_1_HP5_80_DEG.M**

***MSD*: Single Quad.**

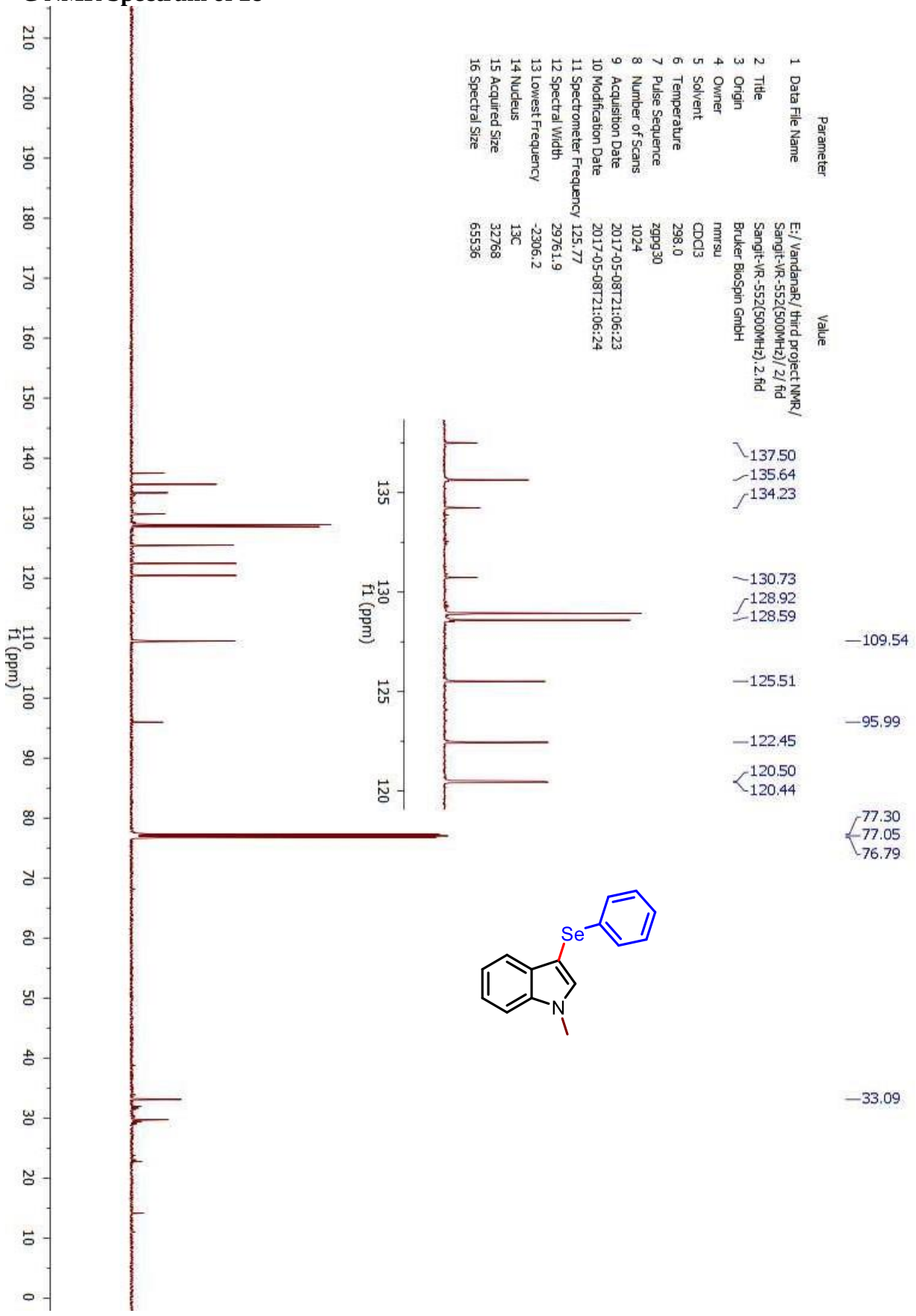




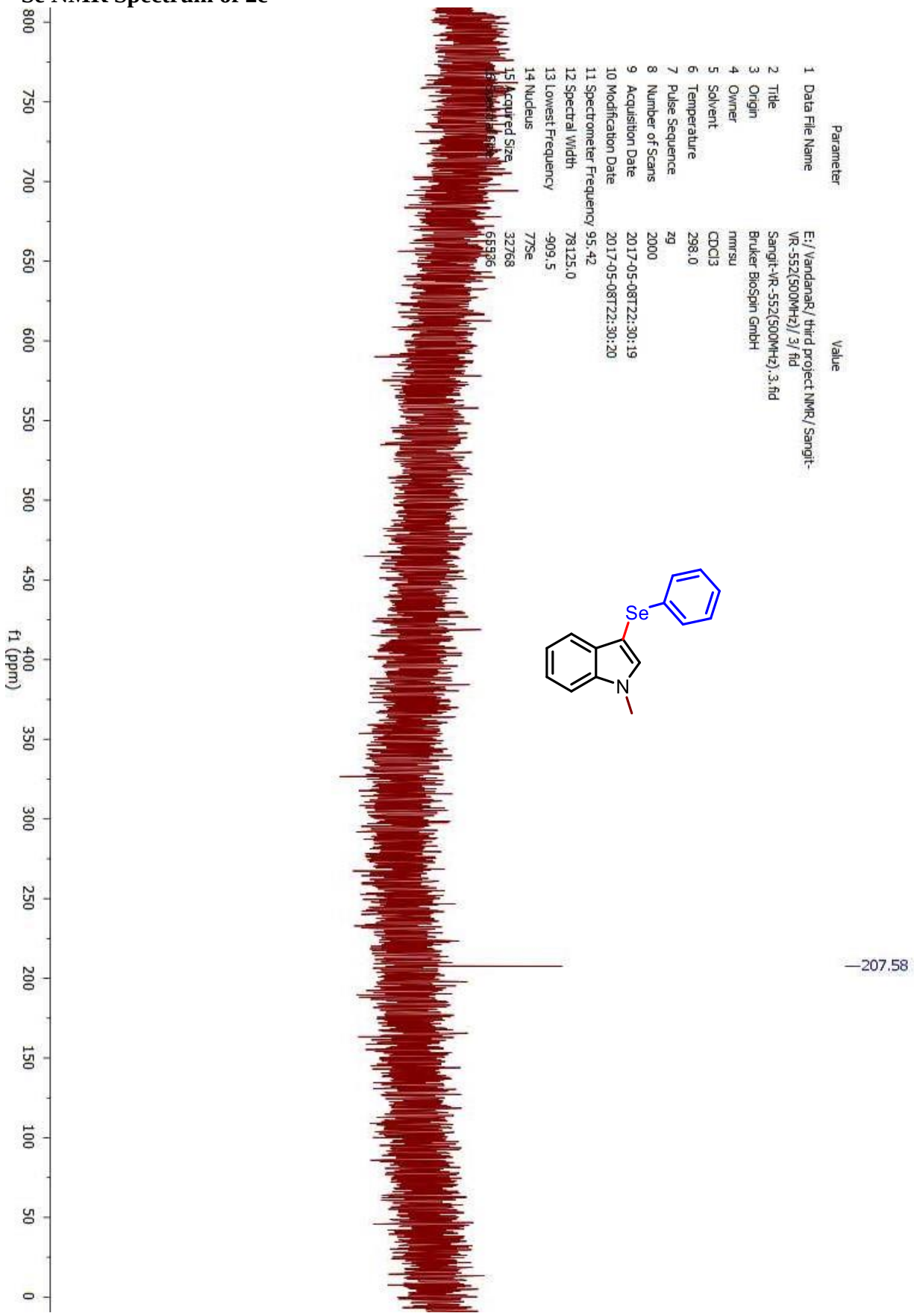
^{1}H NMR Spectrum of 2c



^{13}C NMR Spectrum of 2c



^{77}Se NMR Spectrum of 2c



Mass Spectrum of 2c

Acquisition date: 09/12/17

Sample ID: VR-02-25

Supervisor: Dr. S Kumar

Operator: **IISERB-CIF-Mass Facility**

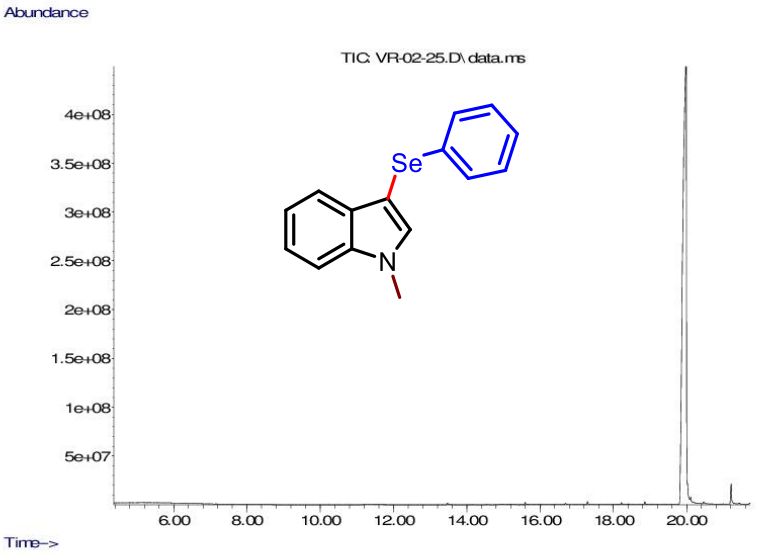
***Instrument*: Agilent 7890A GC with 5975C MS system**

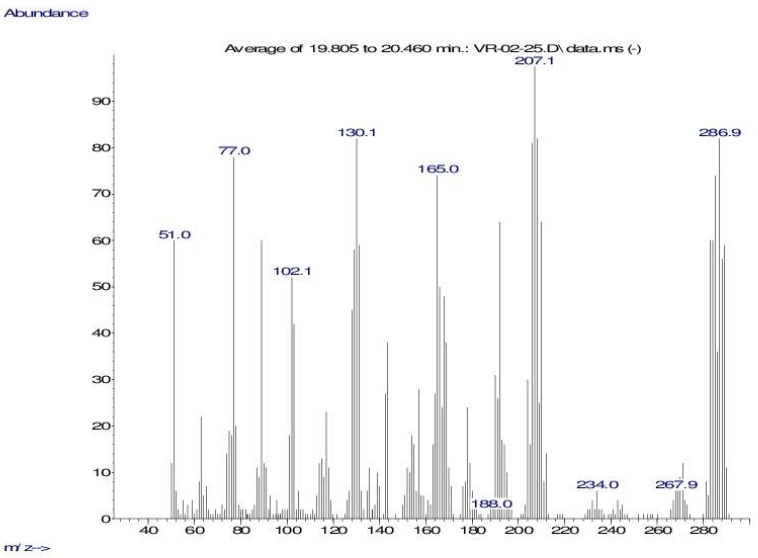
***Column*: HP-5**

Ionization: **EI (70 eV)**

Method: **General_1_HP5_80_DEG.M**

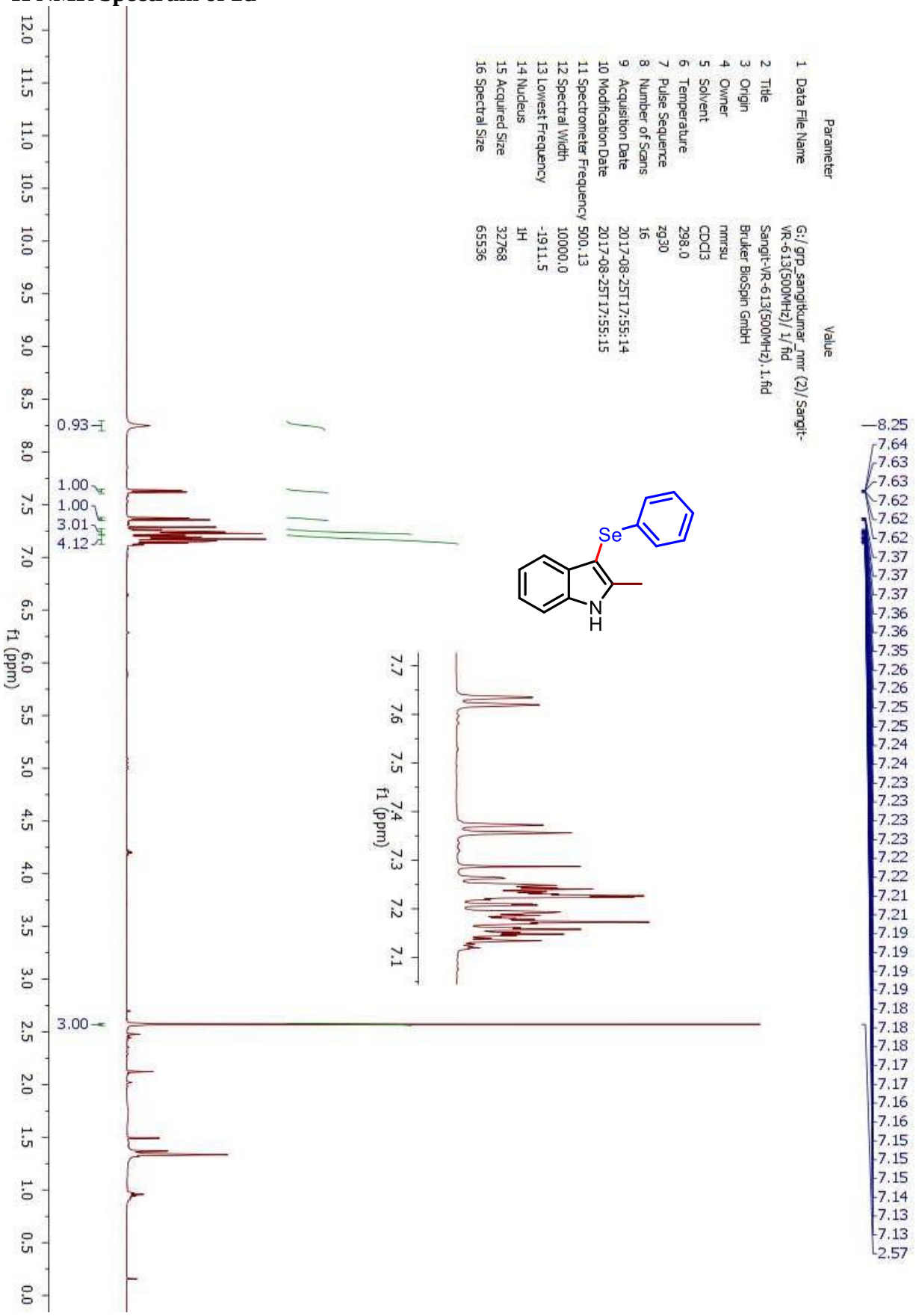
***MSD*: Single Quad.**



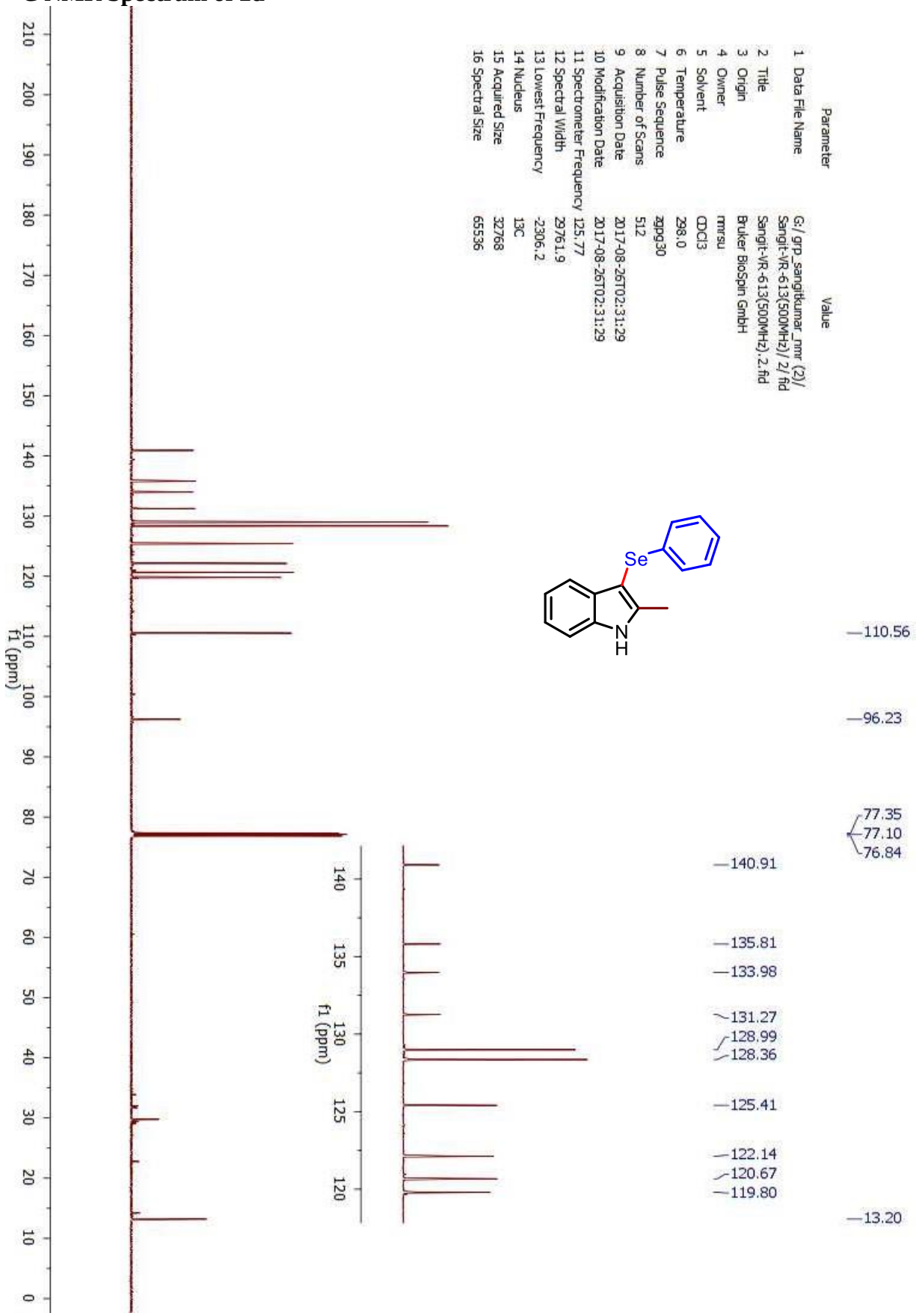


Agilent Technologies

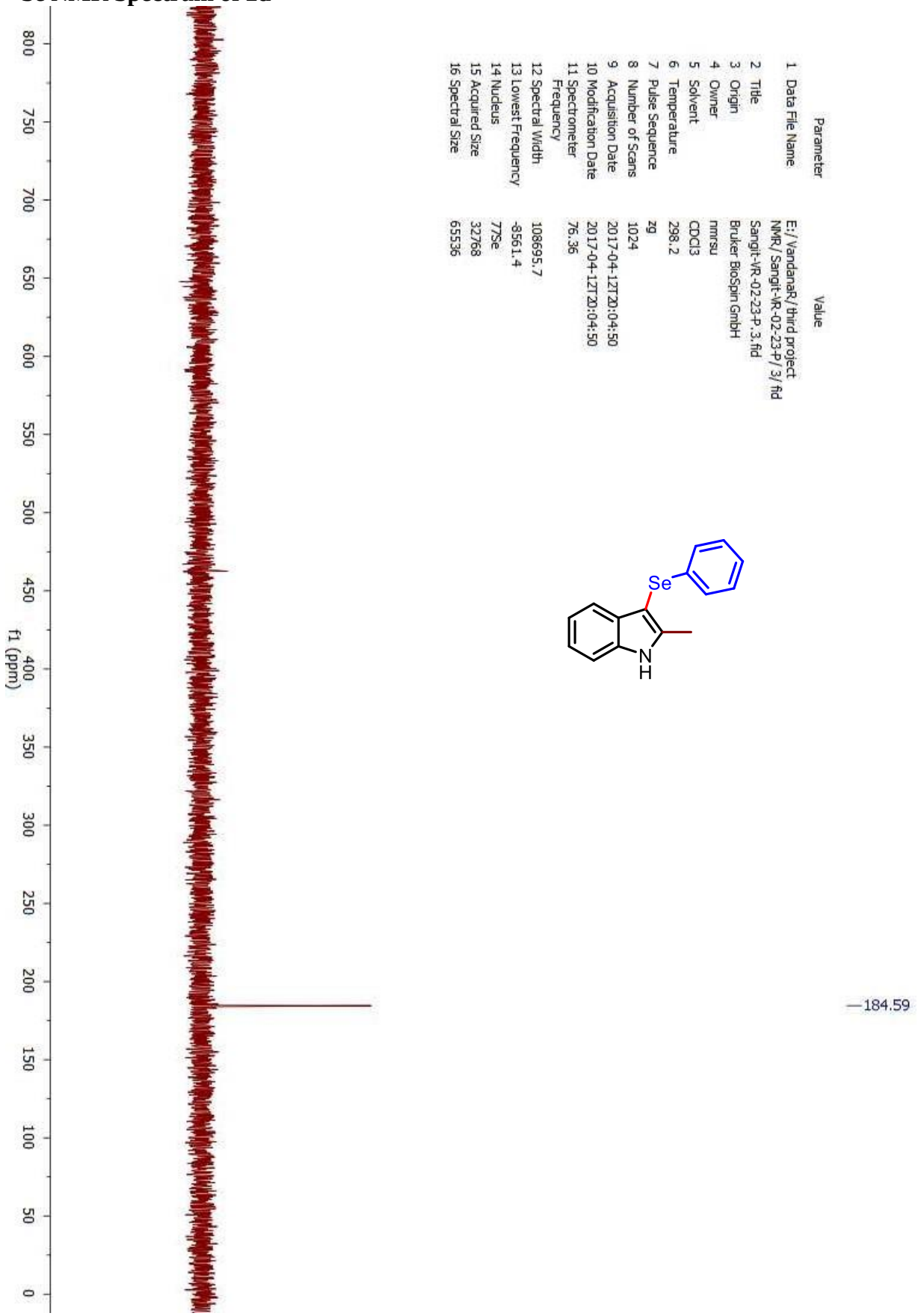
^{1}H NMR Spectrum of 2d



^{13}C NMR Spectrum of 2d



^{77}Se NMR Spectrum of 2d



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1	Data File Name	E:/VandanaR/third project NMR/Sangit-VR-02-23-P/3/fid
2	Title	Sangit-VR-02-23-P, 3, fid
3	Origin	Bruker BioSpin GmbH
4	Owner	nmrsu
5	Solvent	CDCl3
6	Temperature	298.2
7	Pulse Sequence	zg
8	Number of Scans	1024
9	Acquisition Date	2017-04-12T20:04:50
10	Modification Date	2017-04-12T20:04:50
11	Spectrometer Frequency	76.36
12	Spectral Width	108695.7
13	Lowest Frequency	-8561.4
14	Nucleus	77Se
15	Acquired Size	32768
16	Spectral Size	65536

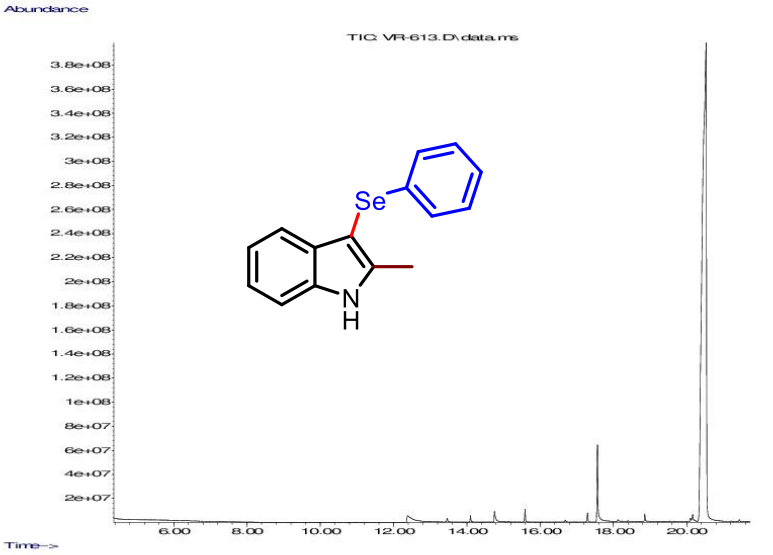
Mass Spectrum of 2d

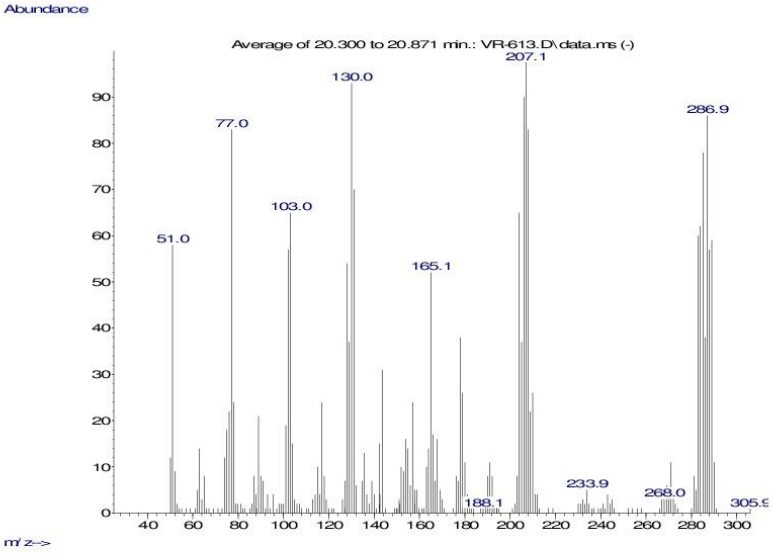
Acquisition date: 09/12/17

Sample ID: VR-613 | ***Supervisor***: Dr. S Kumar | ***Operator:*** **IISERB-CIF-Mass Facility**

Instrument*: Agilent 7890A GC with 5975C MS system** | ***Column*: HP-5** | ***Ionization: **EI (70 eV)**

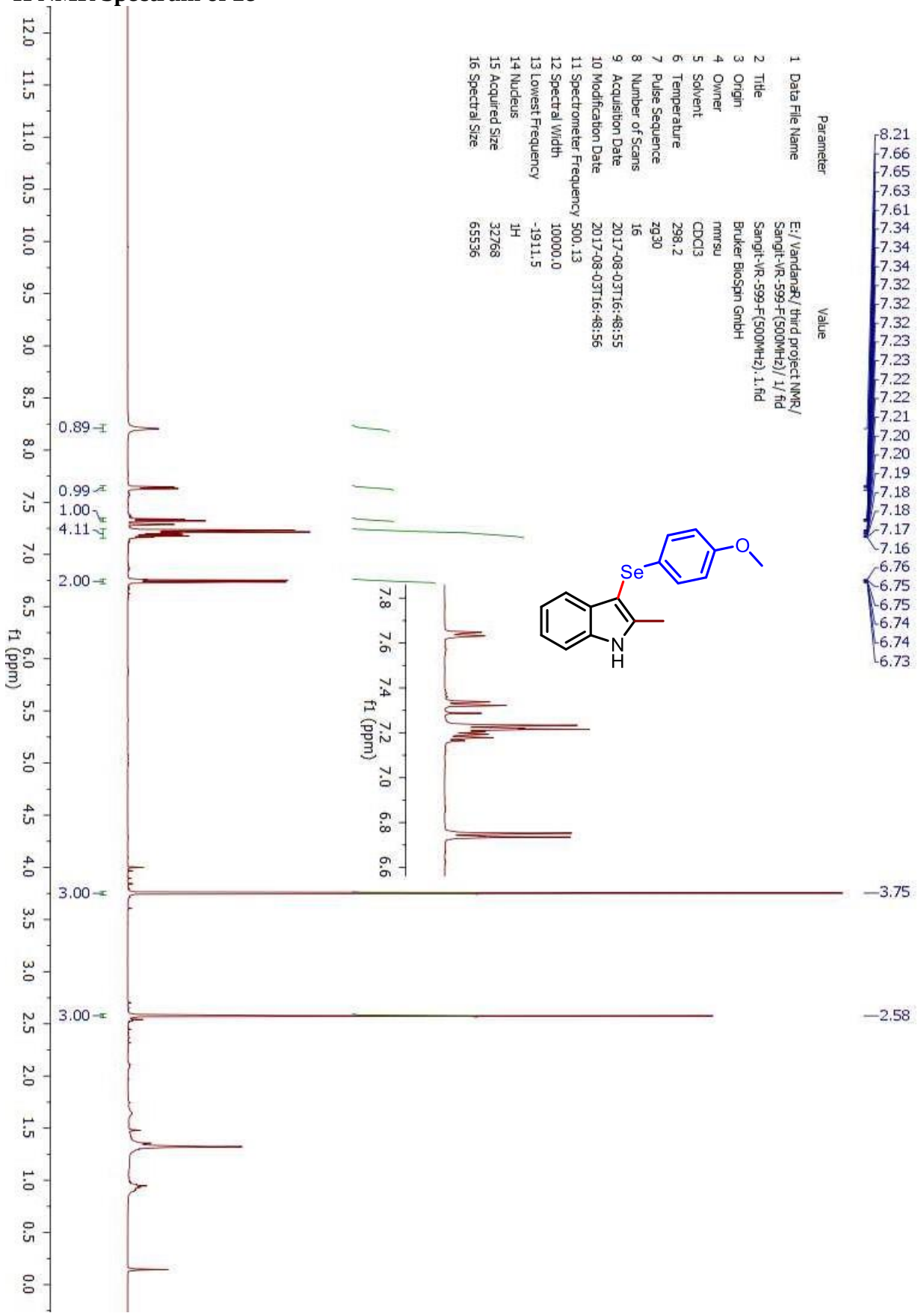
Method: **General_1_HP5_80_DEG.M** | ***MSD*: Single Quad.**



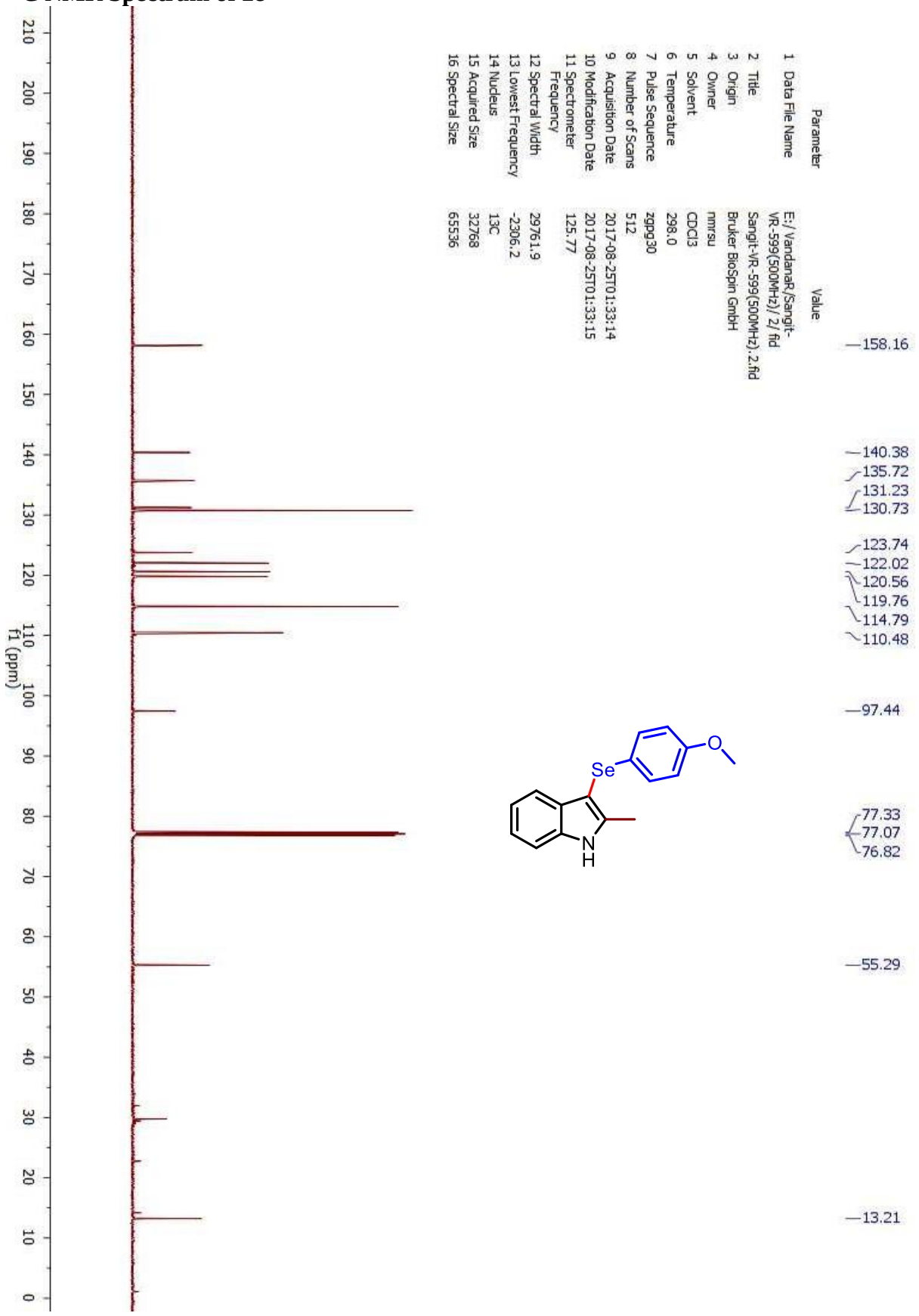


Agilent Technologies

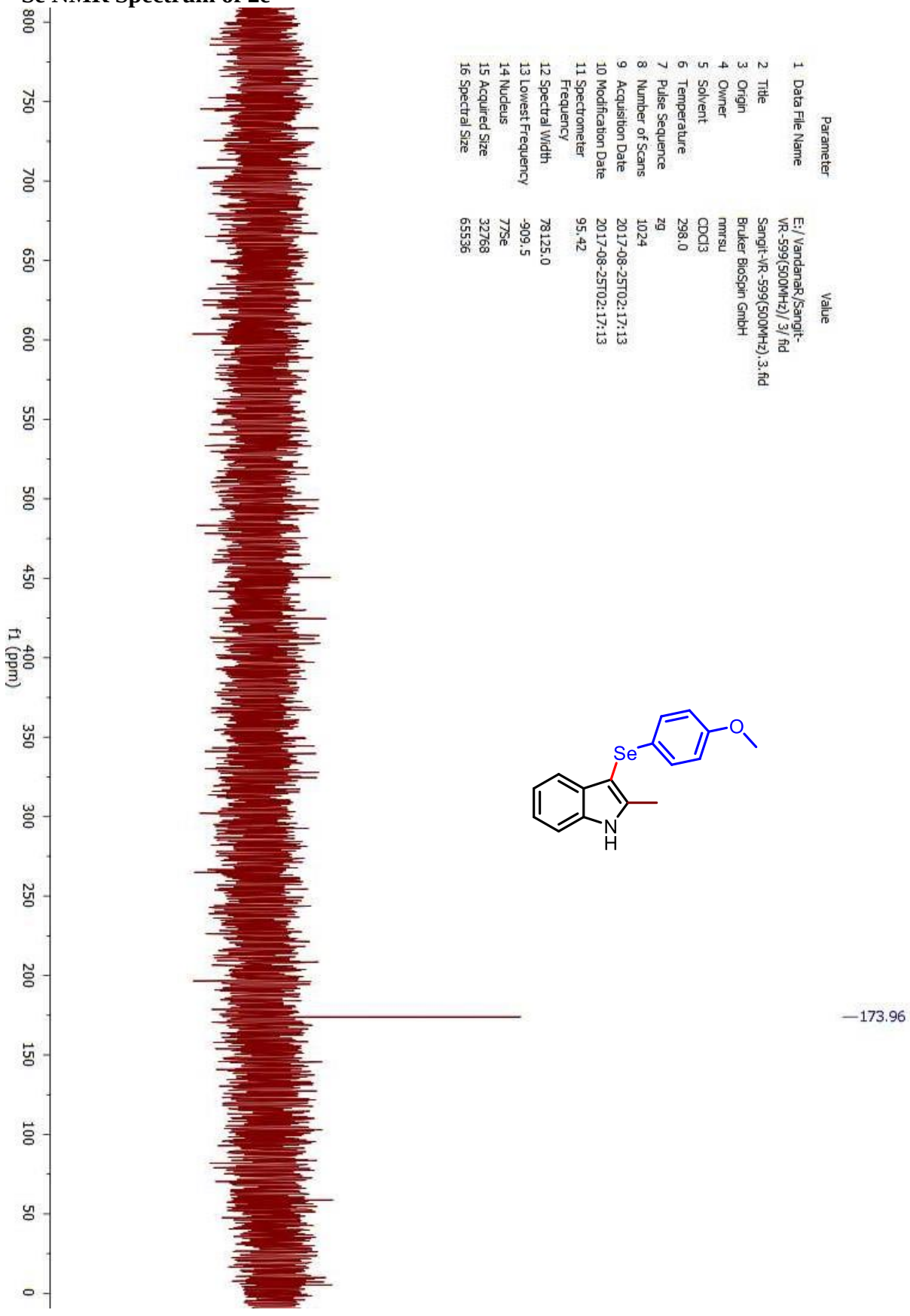
^{1}H NMR Spectrum of 2e



^{13}C NMR Spectrum of 2e



^{77}Se NMR Spectrum of 2e



Mass Spectrum of 2e

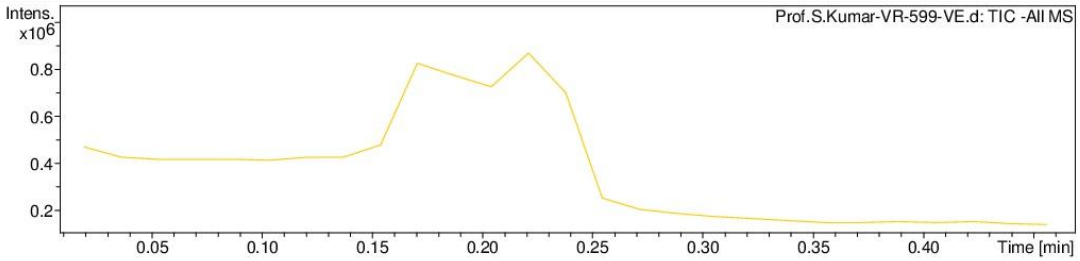
Display Report

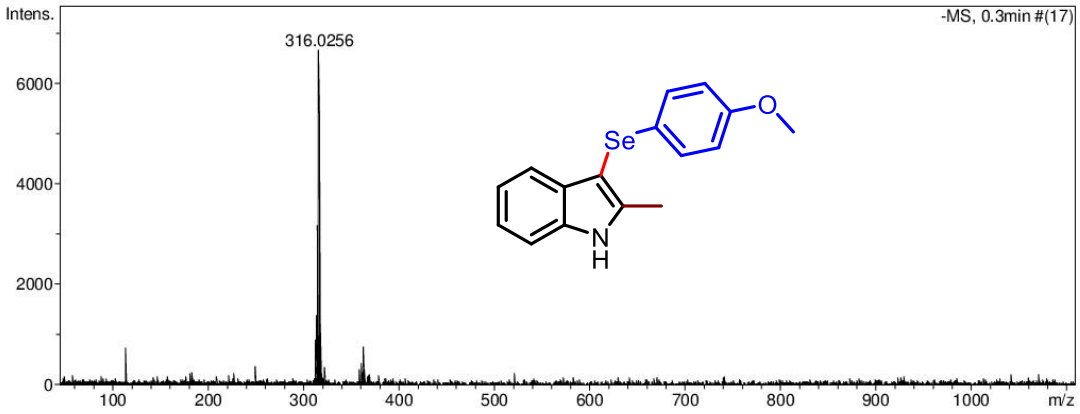
Analysis Info

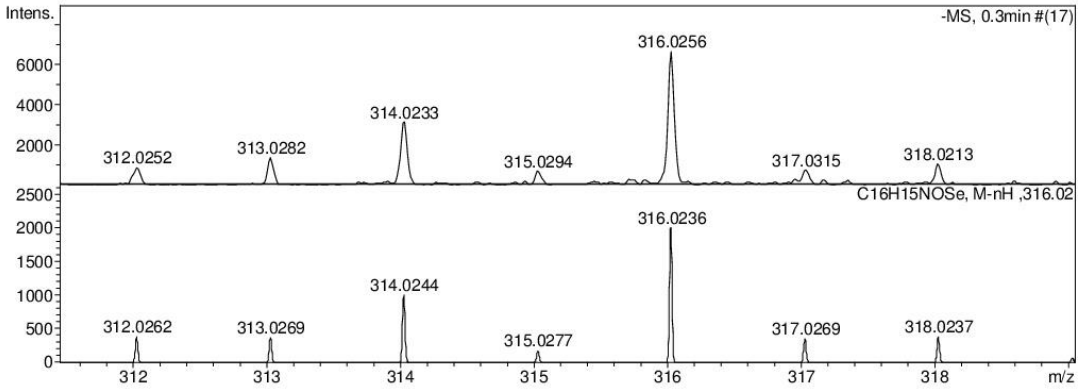
Analysis Name	D:\Data\NEW USER DATA 2017\2018\JUNE 2018\11 JUNE\Prof.S.Kumar-VR-599-VE.d	Acquisition Date	6/11/2018 4:32:23 PM
Method	tune_low.m	Operator	RUCHI
Sample Name	VR-599-VE	Instrument	micrOTOF-Q II 10330
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Negative	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	2500 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	100.0 Vpp	Set Divert Valve	Waste







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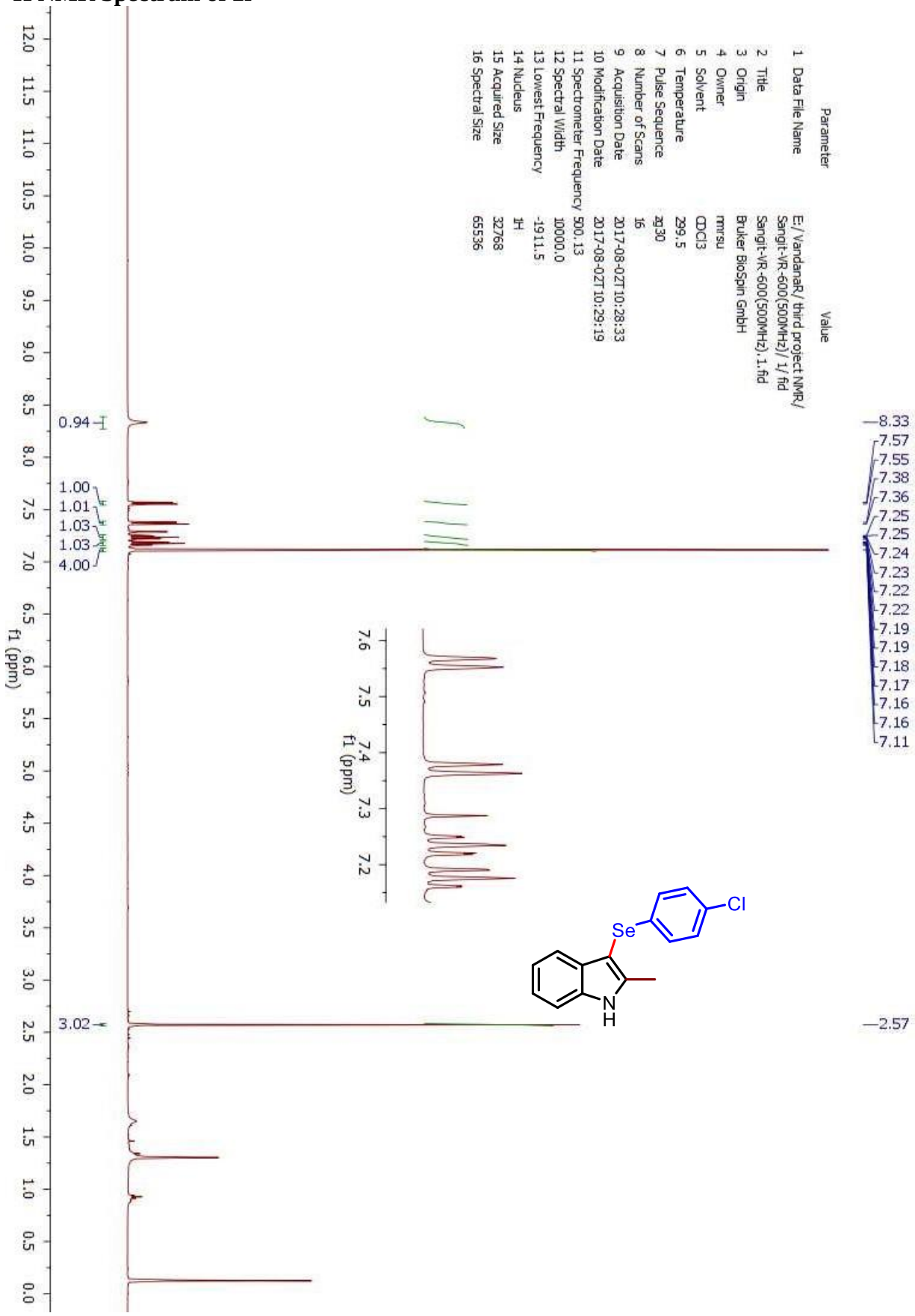
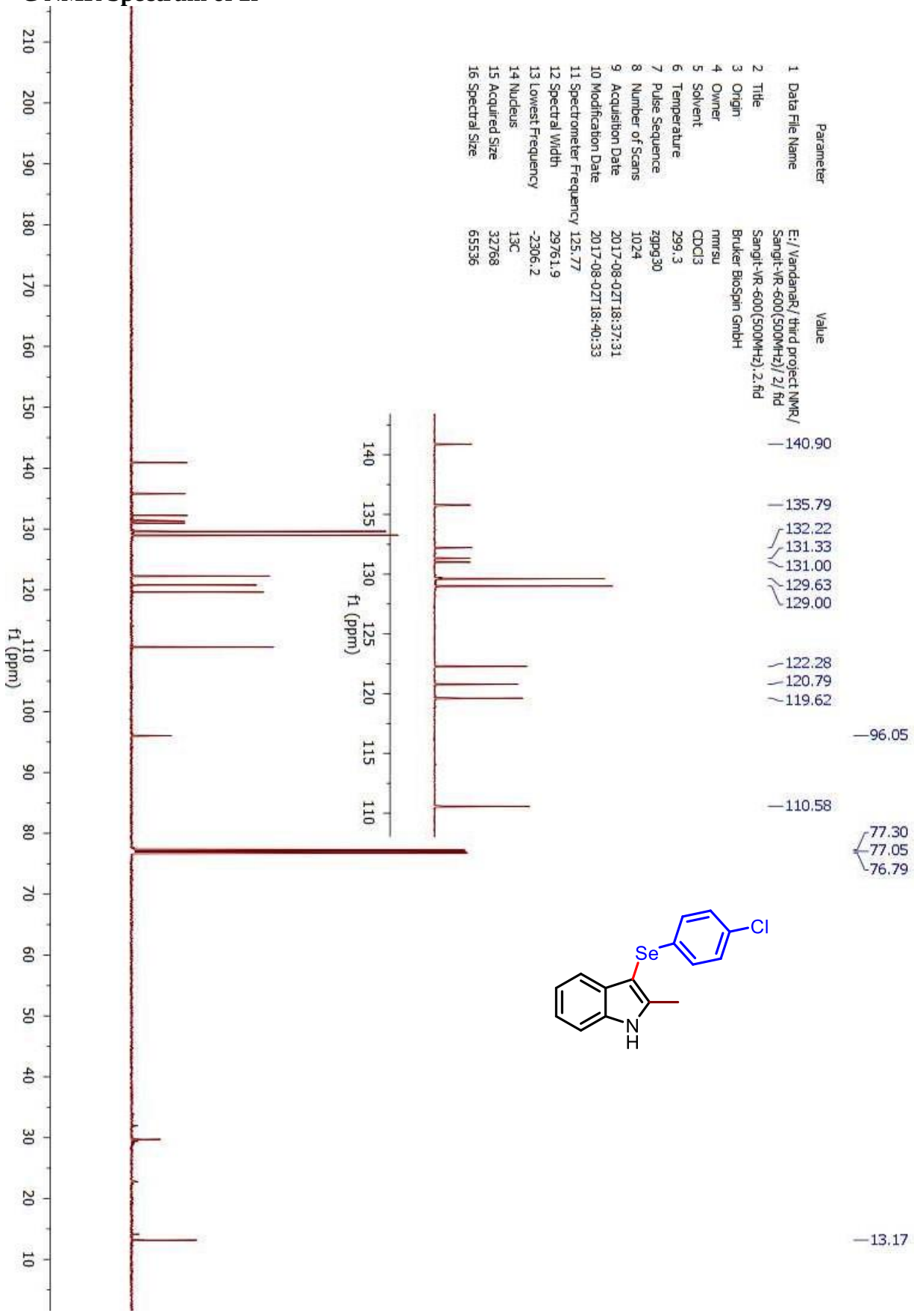
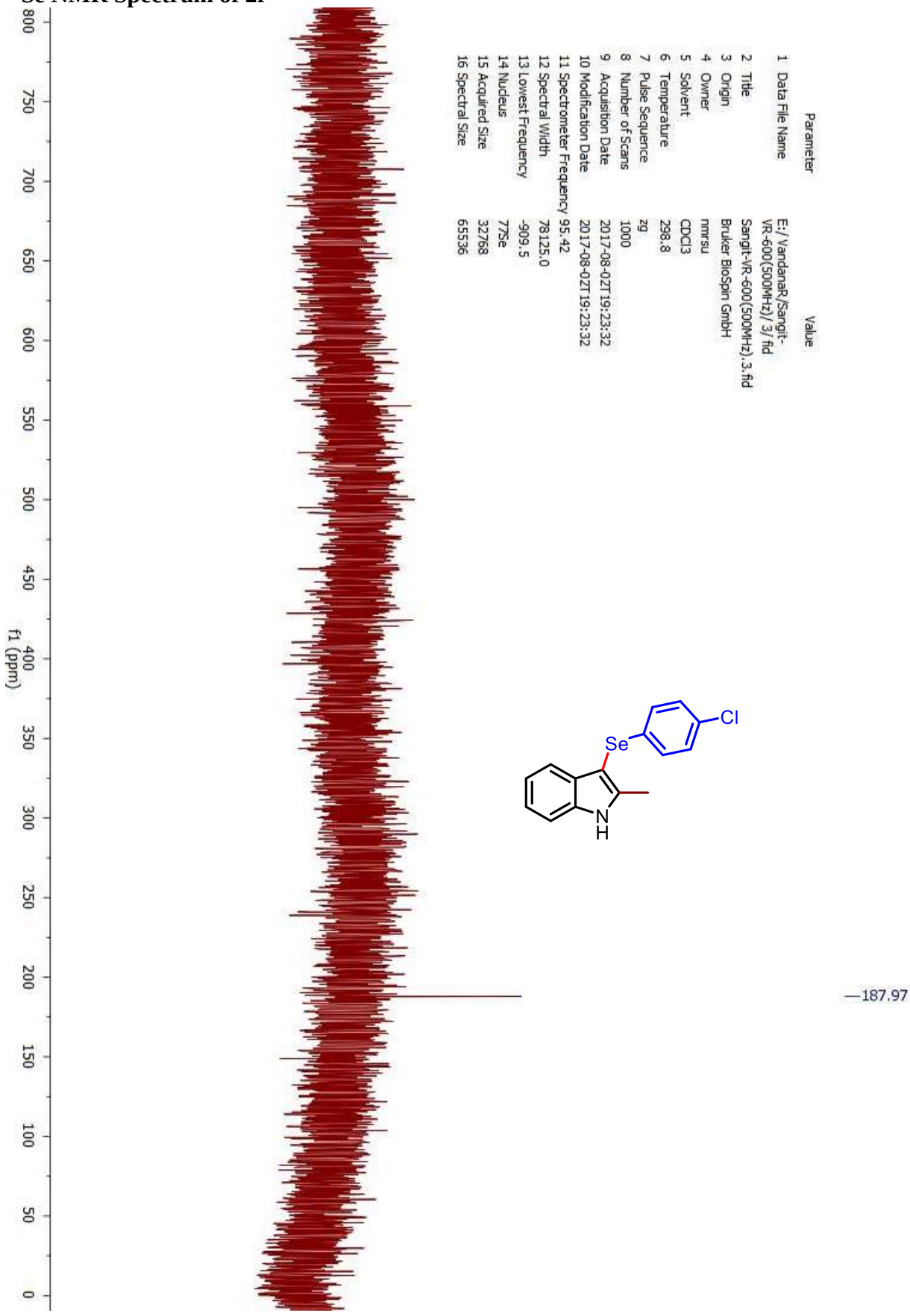
^{1}H NMR Spectrum of 2f



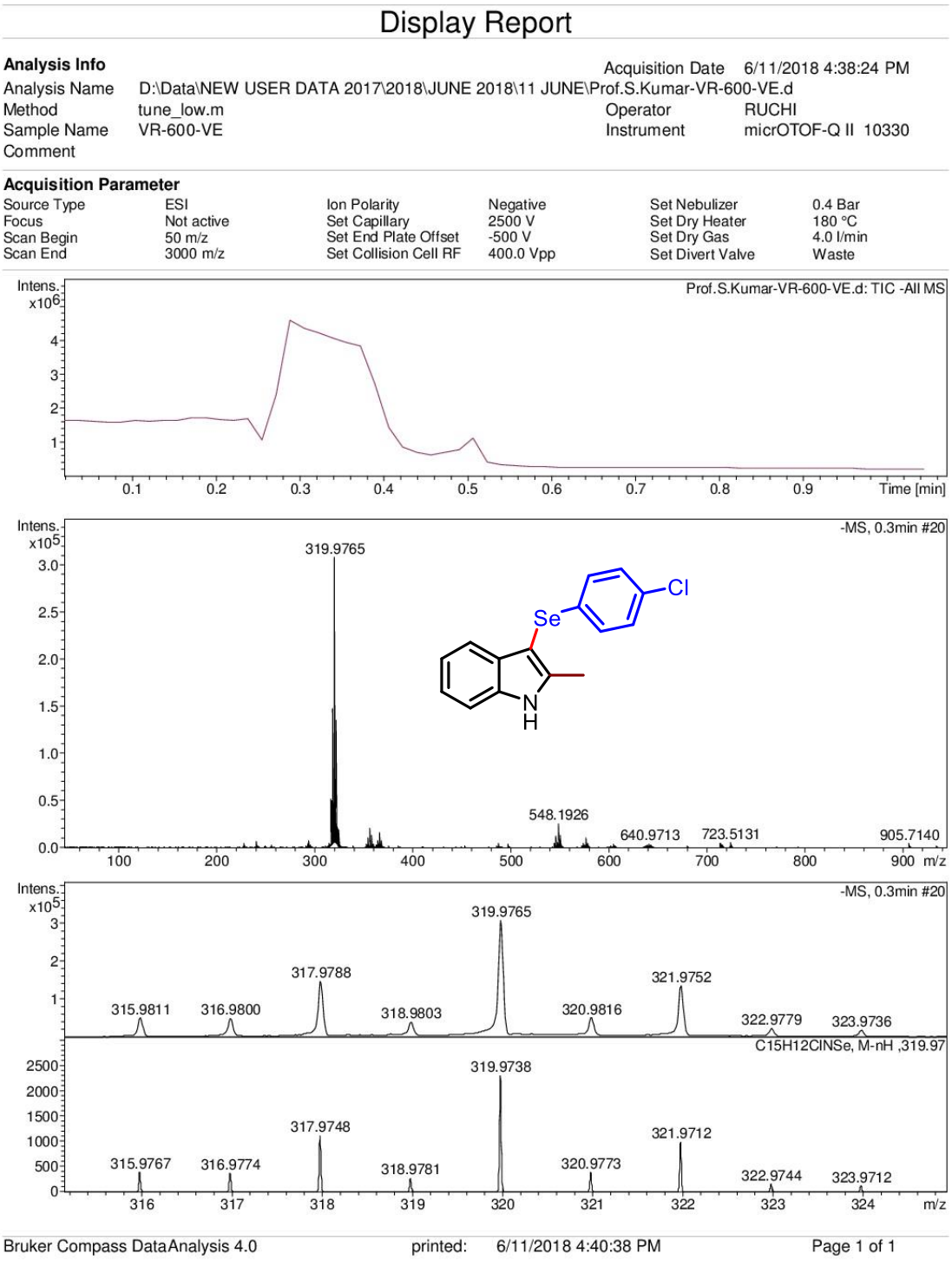
^{13}C NMR Spectrum of 2f



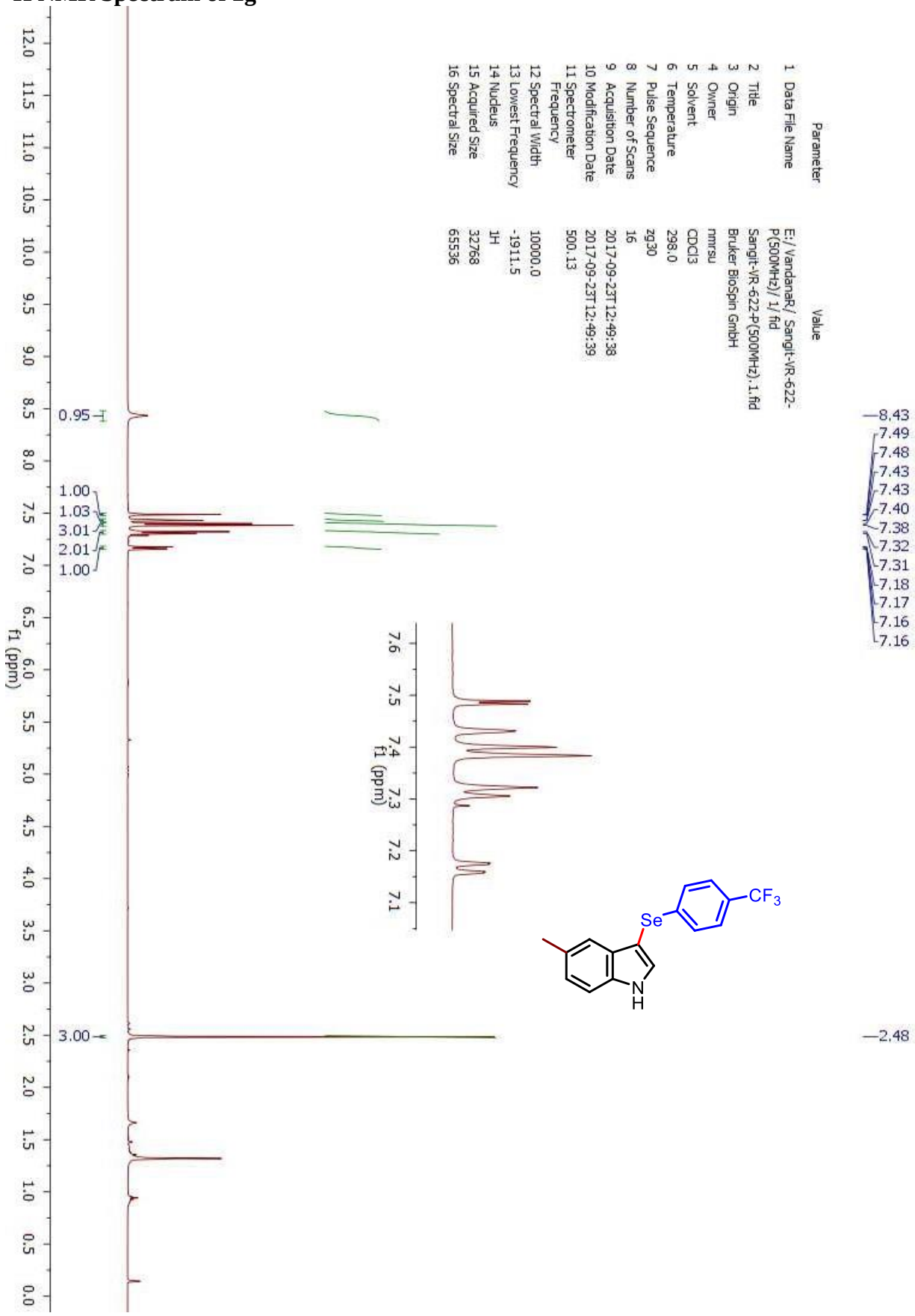
^{77}Se NMR Spectrum of 2f



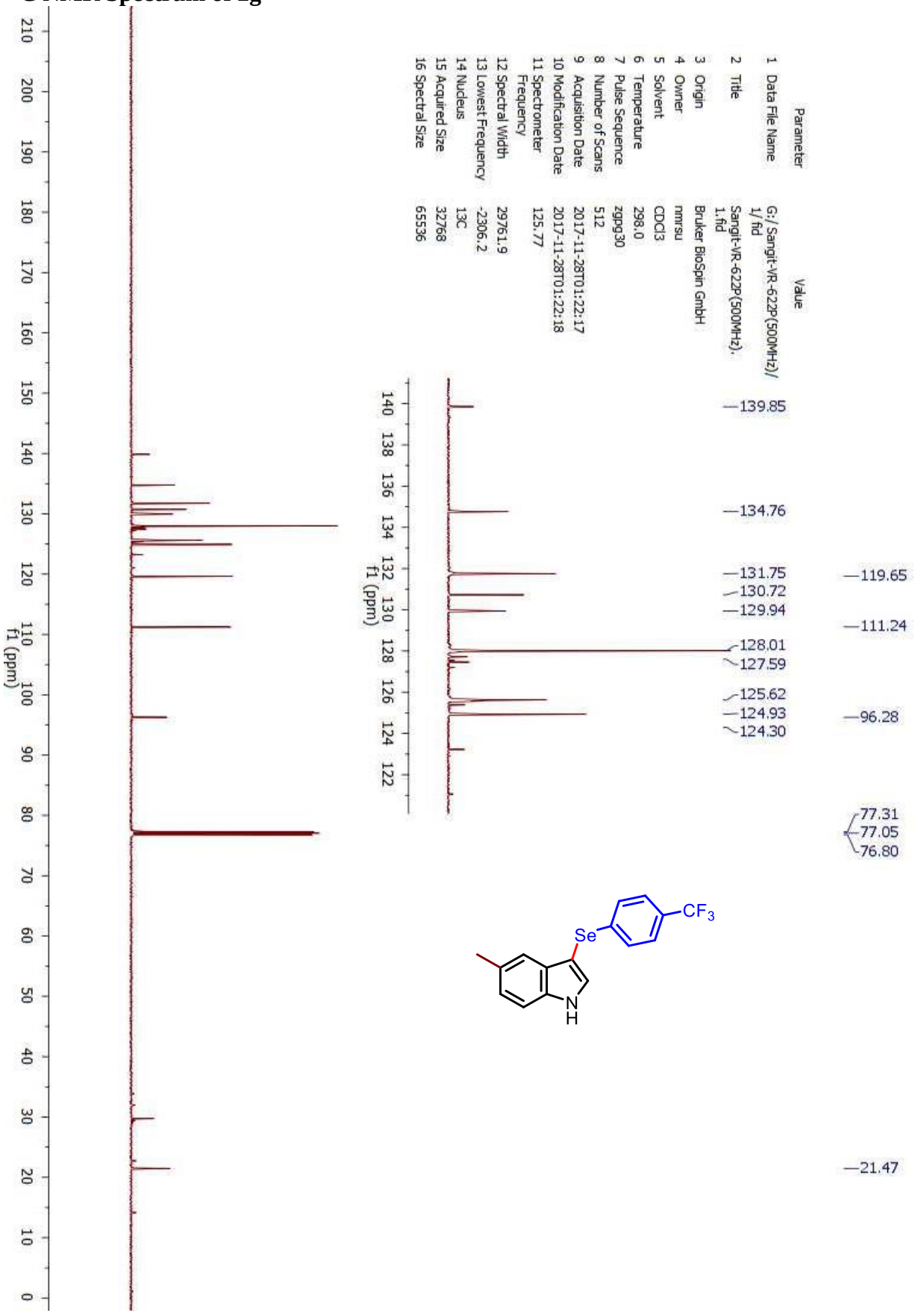
Mass Spectrum of 2f



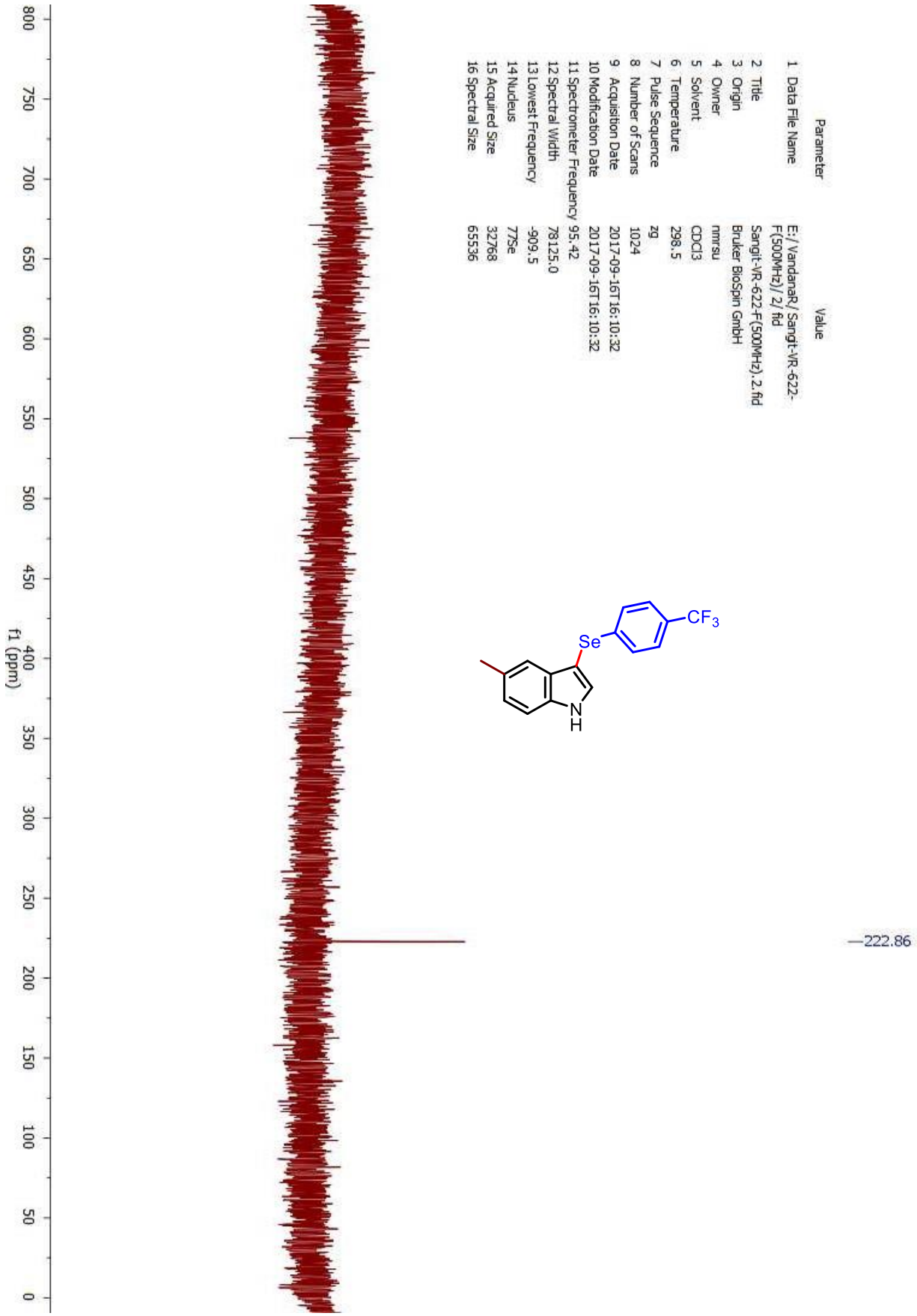
^{1}H NMR Spectrum of 2g



^{13}C NMR Spectrum of 2g



^{77}Se NMR Spectrum of 2g



Mass Spectrum of 2g

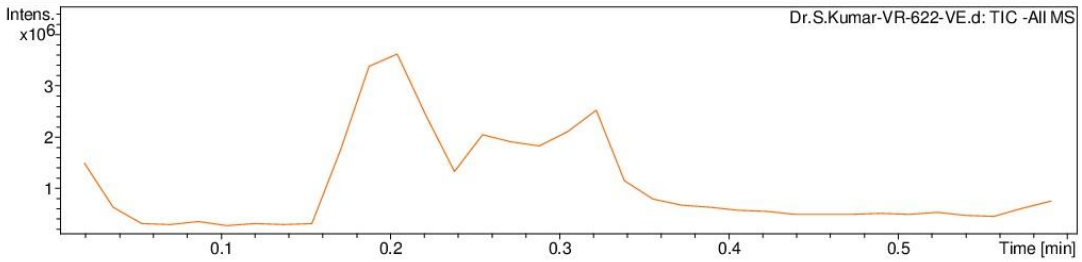
Display Report

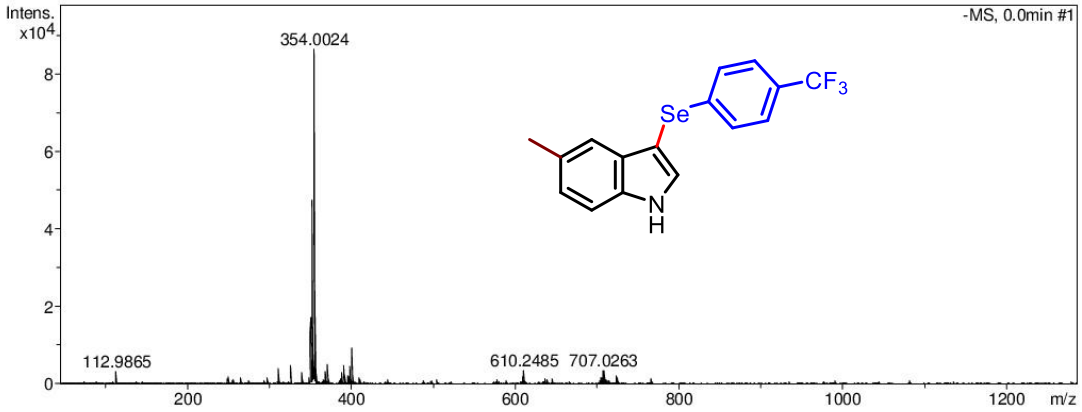
Analysis Info

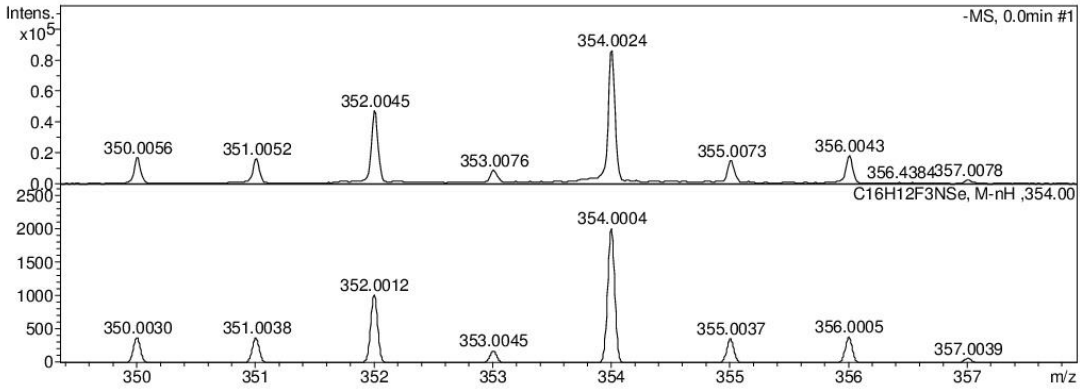
Analysis Name	D:\Data\NEW USER DATA 2017\2018\May-2018\21 -05-2108\Dr.S.Kumar-VR-622-VE.d	Acquisition Date	5/21/2018 5:02:21 PM
Method	tune_low.m	Operator	RUCHI
Sample Name	VR-622-VE	Instrument	micrOTOF-Q II 10330
Comment			

Acquisition Parameter

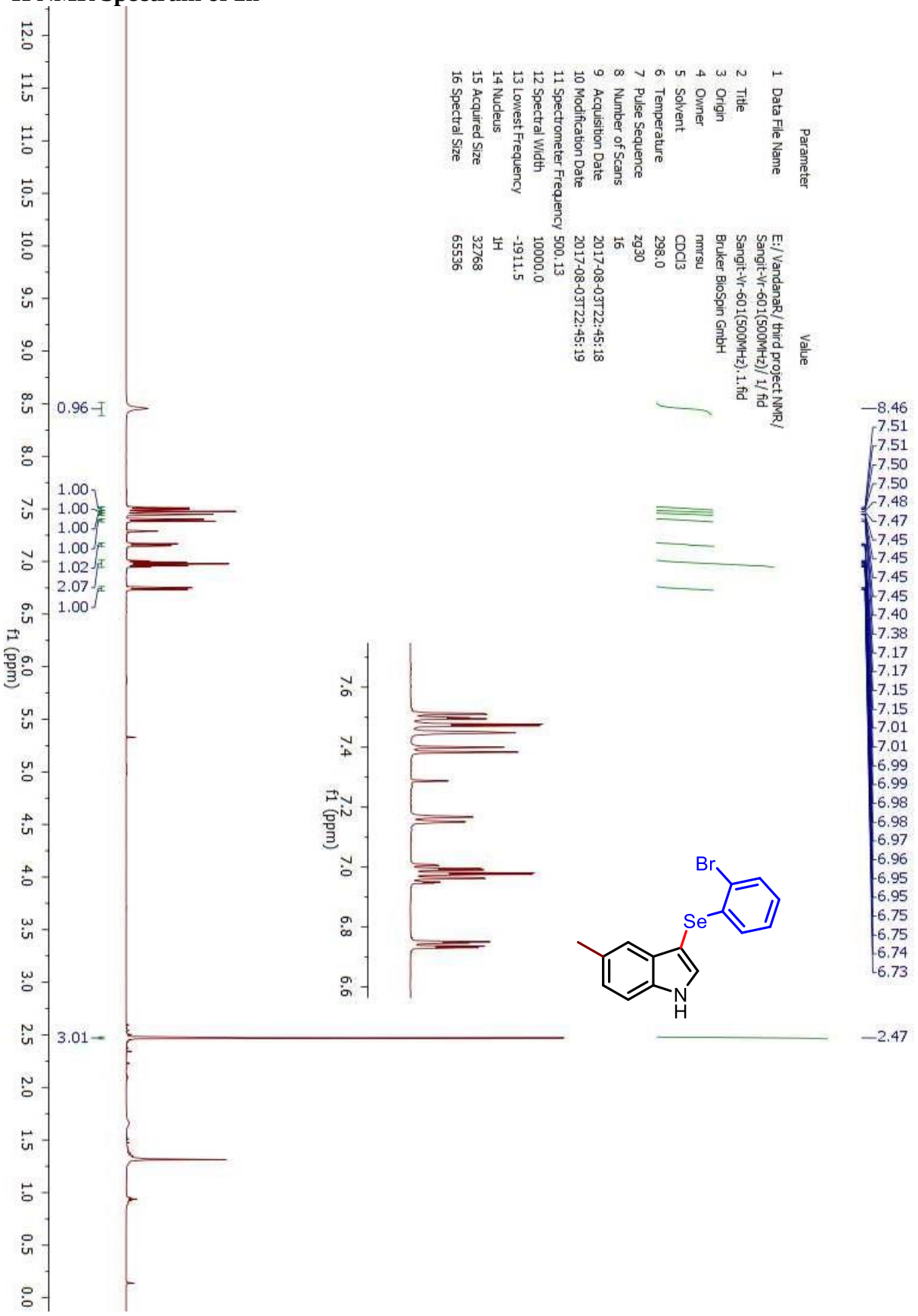
Source Type	ESI	Ion Polarity	Negative	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	2500 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	100.0 Vpp	Set Divert Valve	Waste



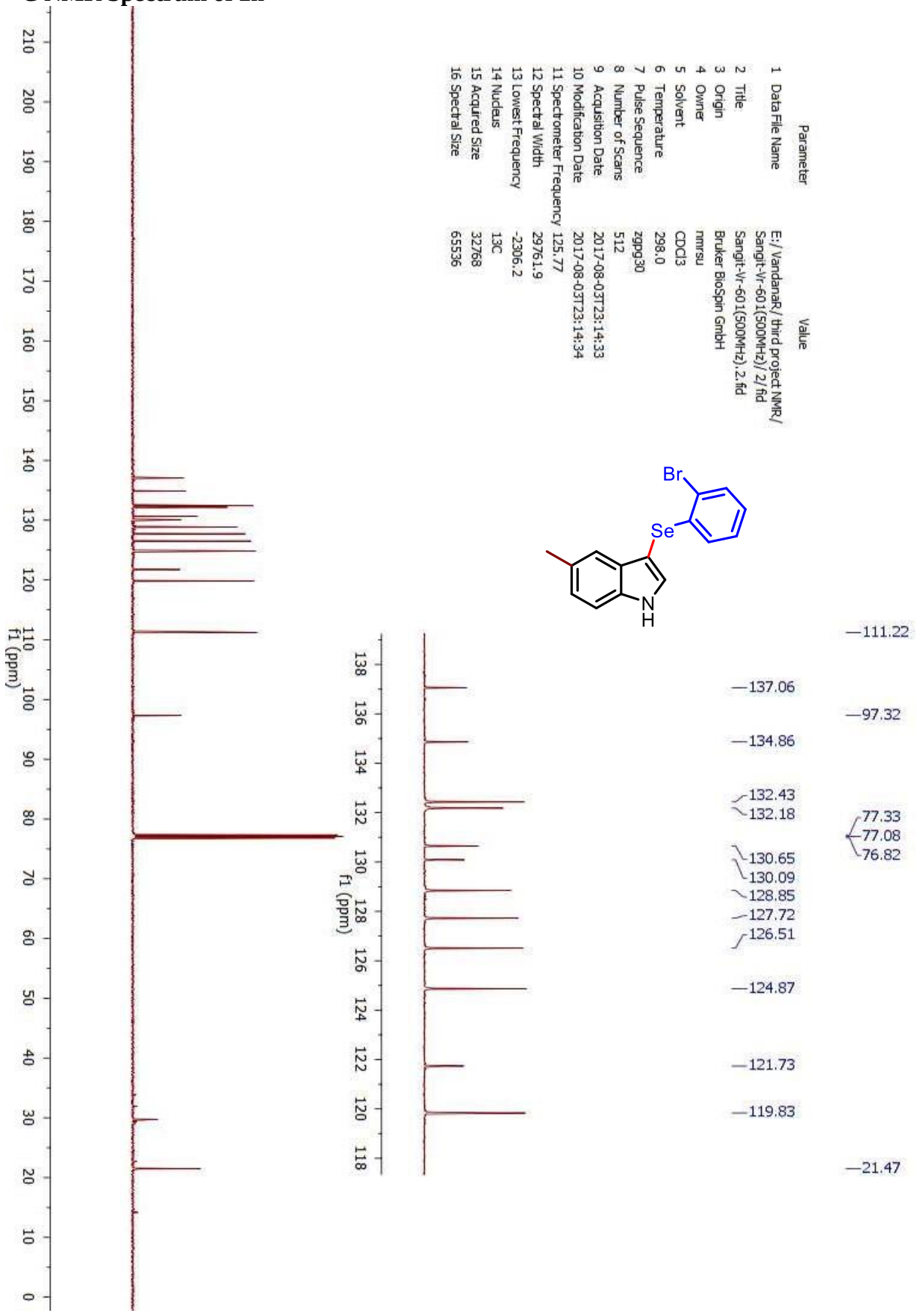




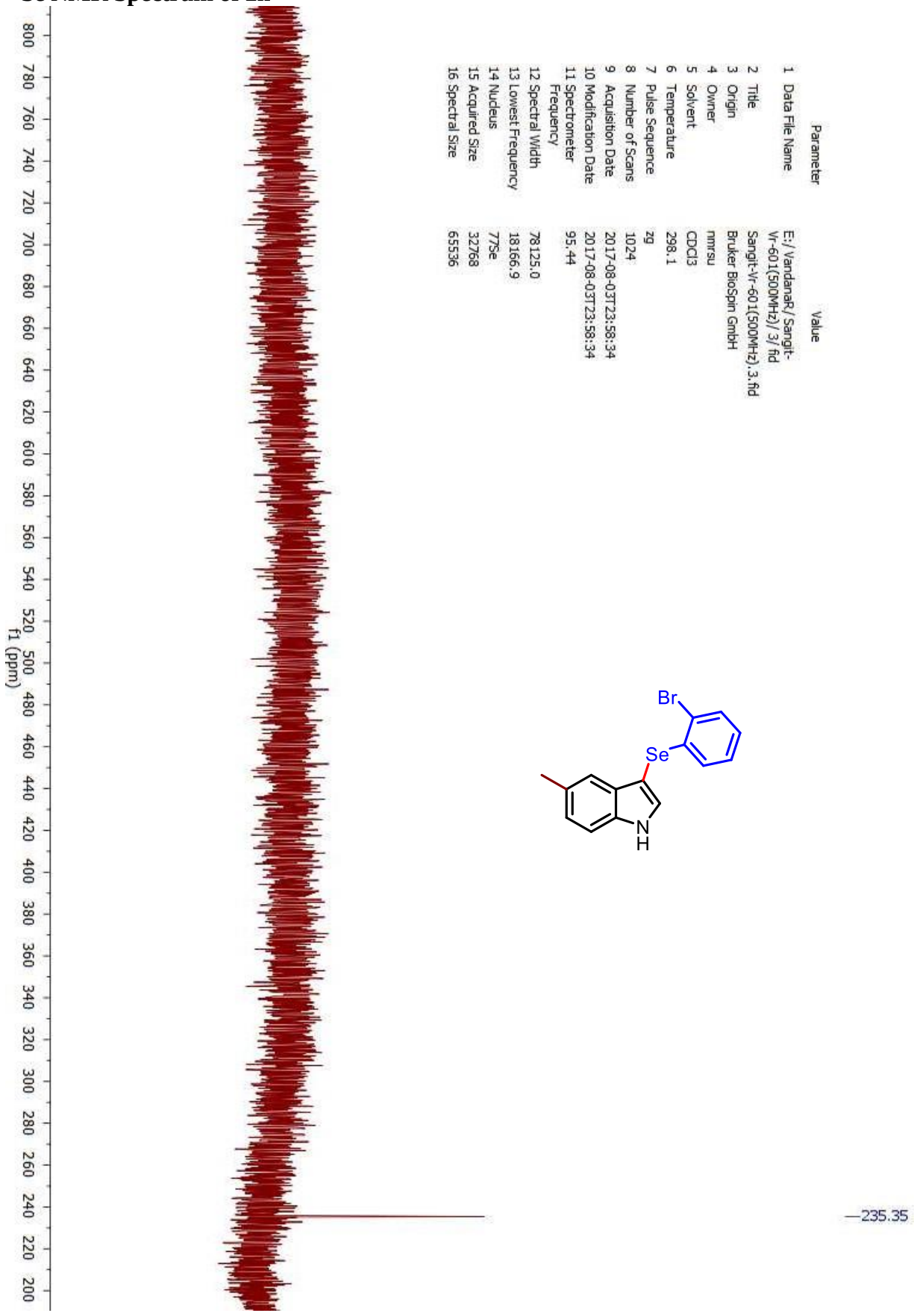
^{1}H NMR Spectrum of 2h



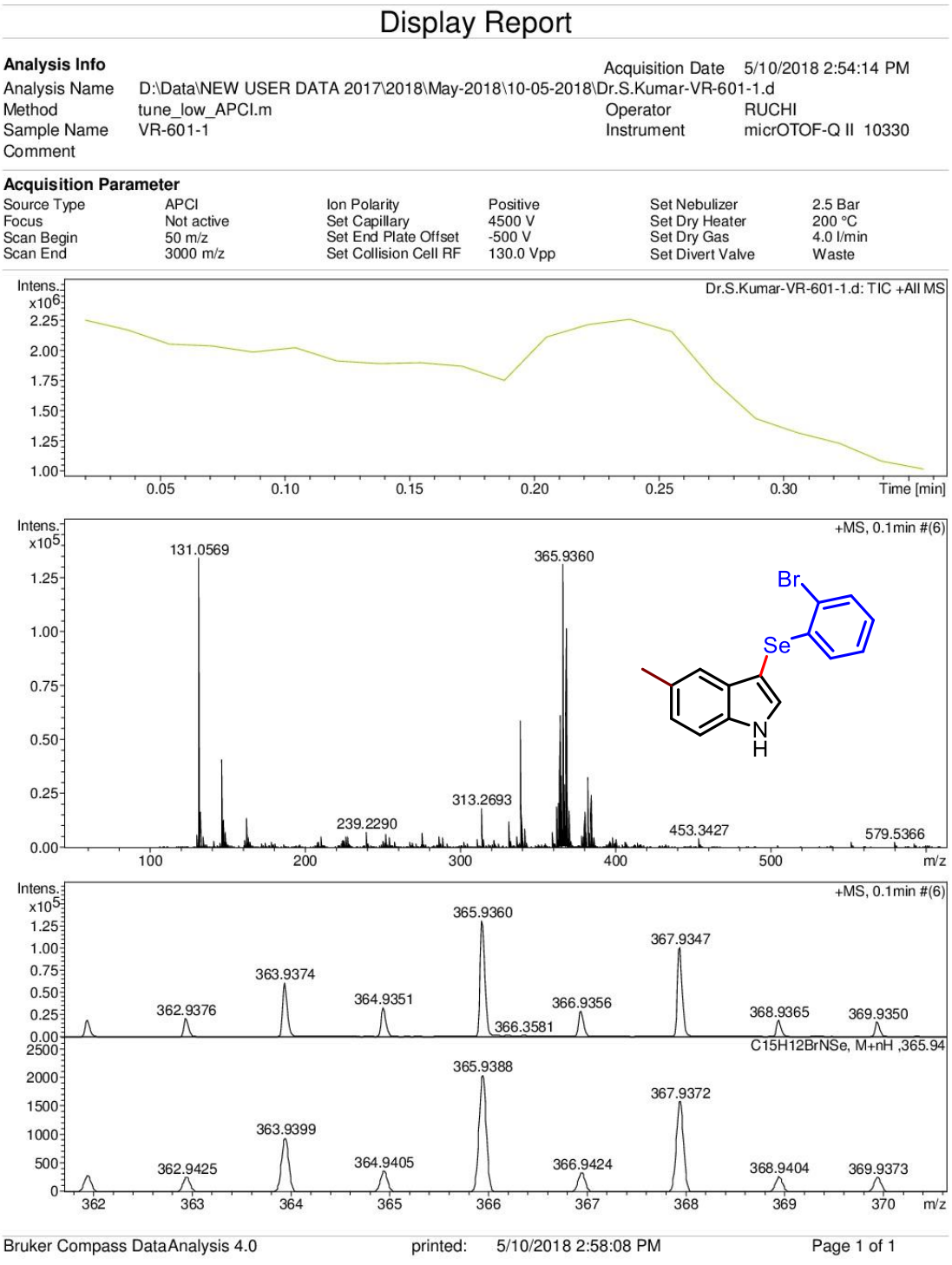
^{13}C NMR Spectrum of 2h



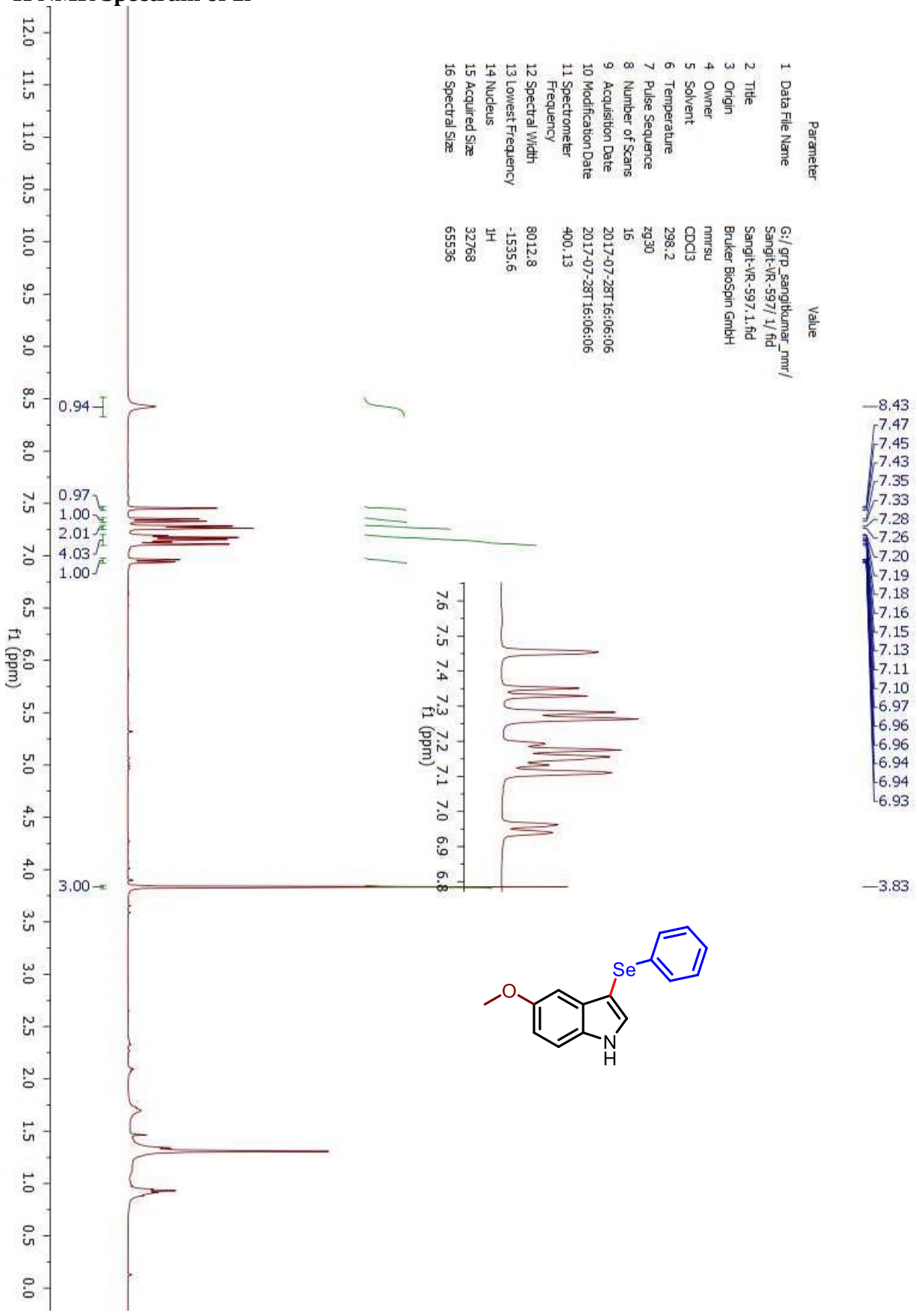
^{77}Se NMR Spectrum of 2h



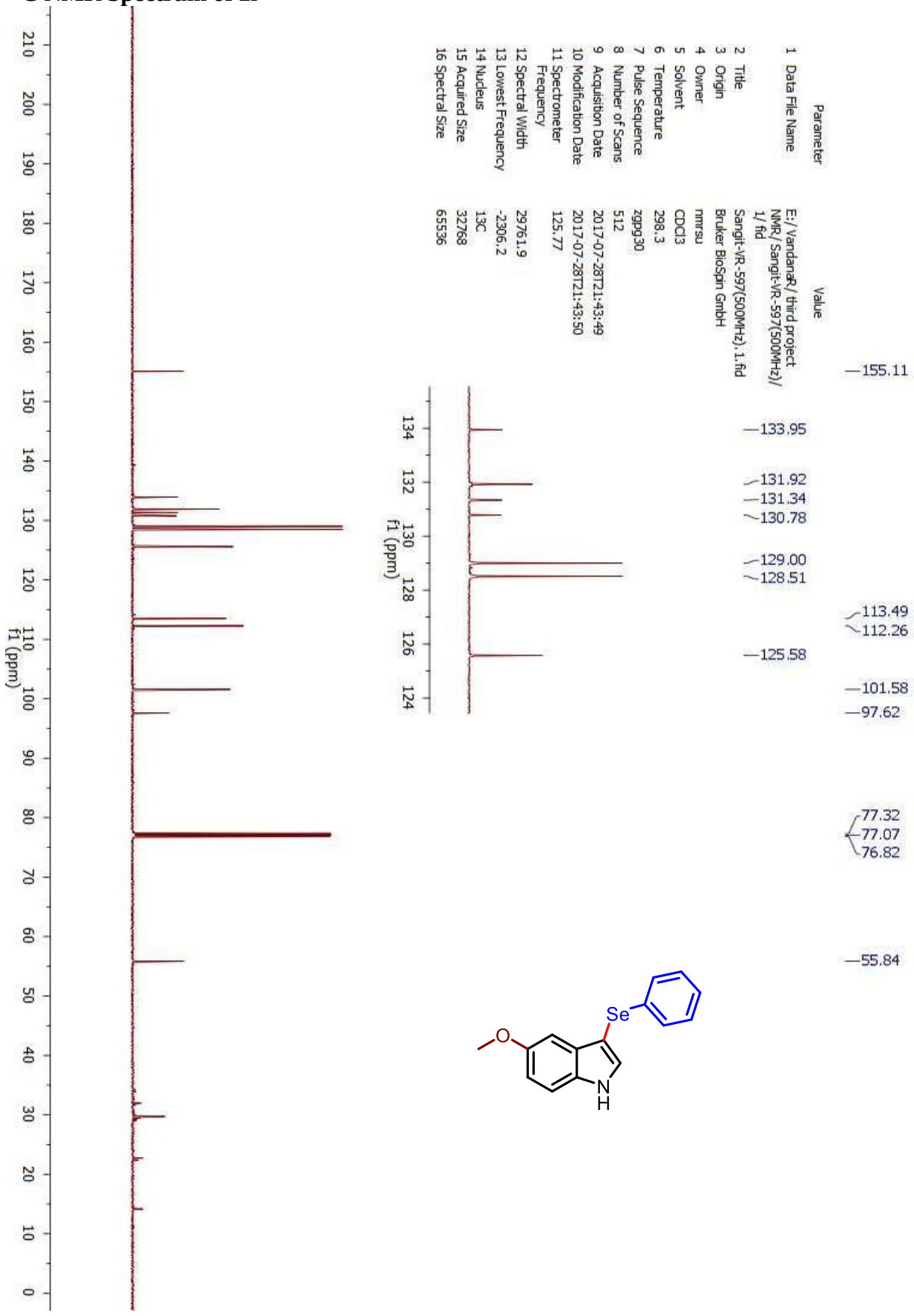
Mass Spectrum of 2h



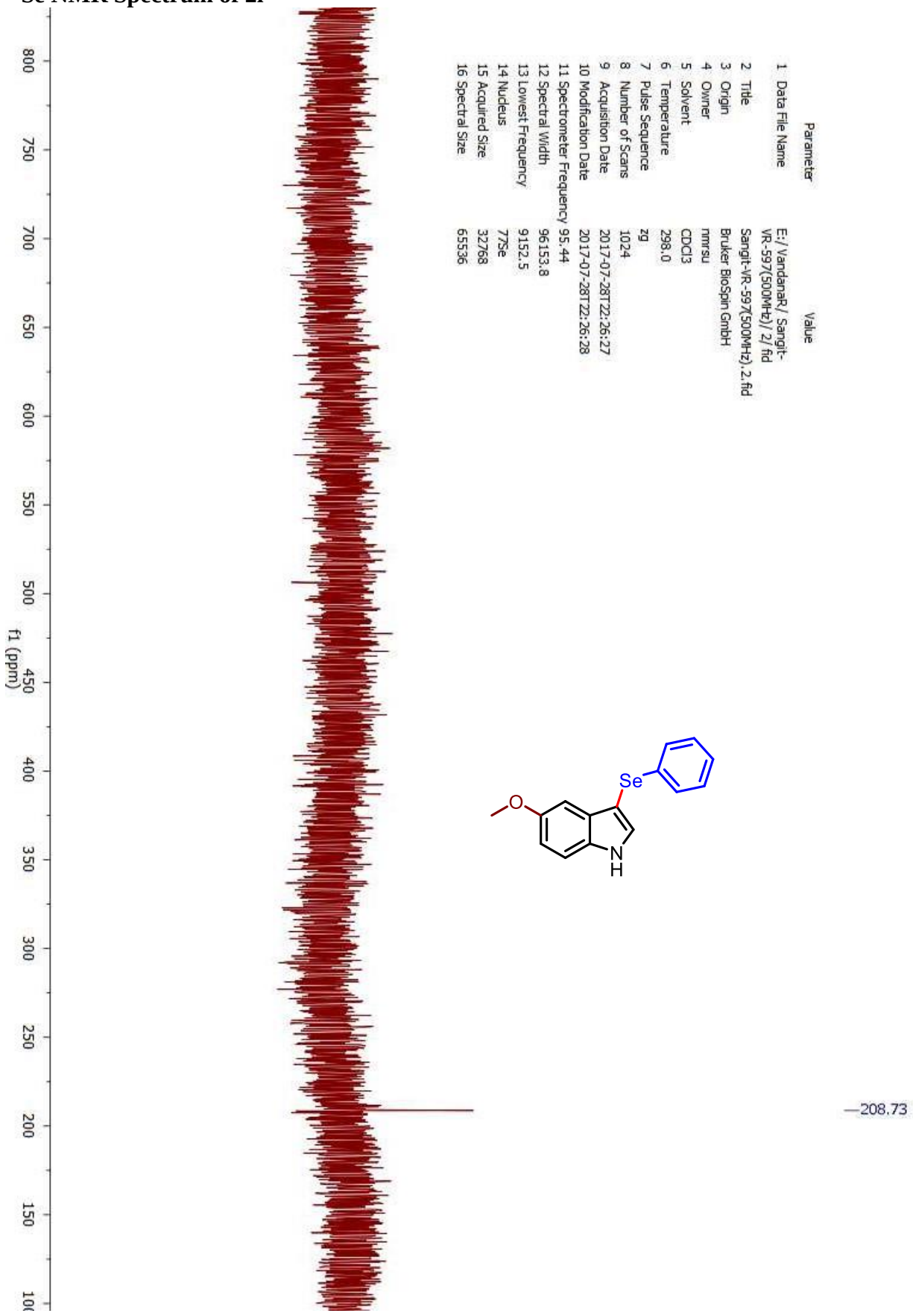
^{1}H NMR Spectrum of 2i



^{13}C NMR Spectrum of 2i



^{77}Se NMR Spectrum of 2i



Mass Spectrum of 2i

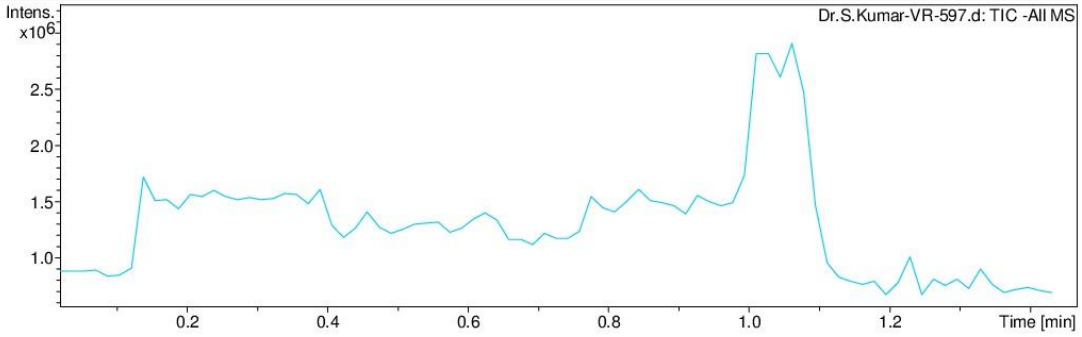
Display Report

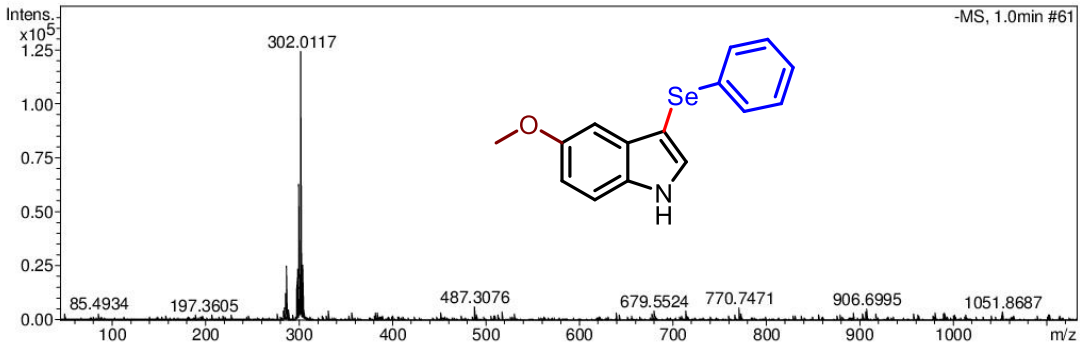
Analysis Info

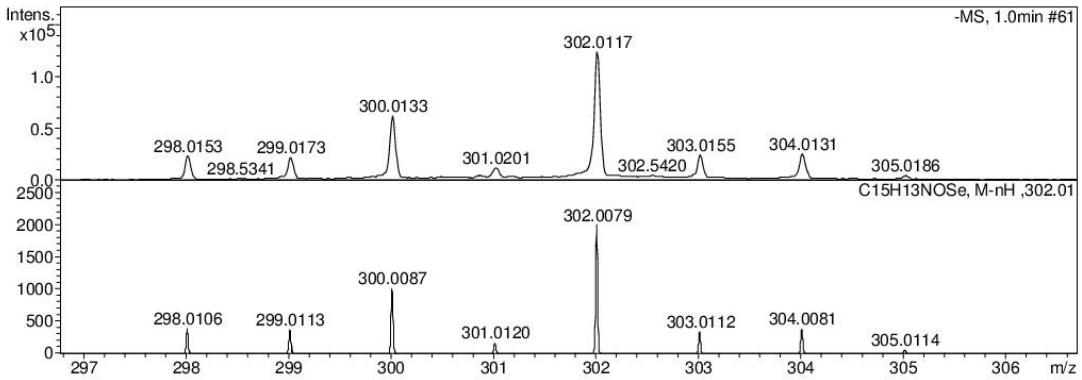
Analysis Name	D:\Data\NEW USER DATA 2017\DEC-2017\15 dec\Dr.S.Kumar-VR-597.d	Acquisition Date	12/15/2017 5:16:19 PM
Method	tune_low.m	Operator	RUCHI
Sample Name	VR-597	Instrument	micrOTOF-Q II 10330
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Negative	Set Nebulizer	5.8 psi
Focus	Not active	Set Capillary	2500 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	100.0 Vpp	Set Divert Valve	Waste







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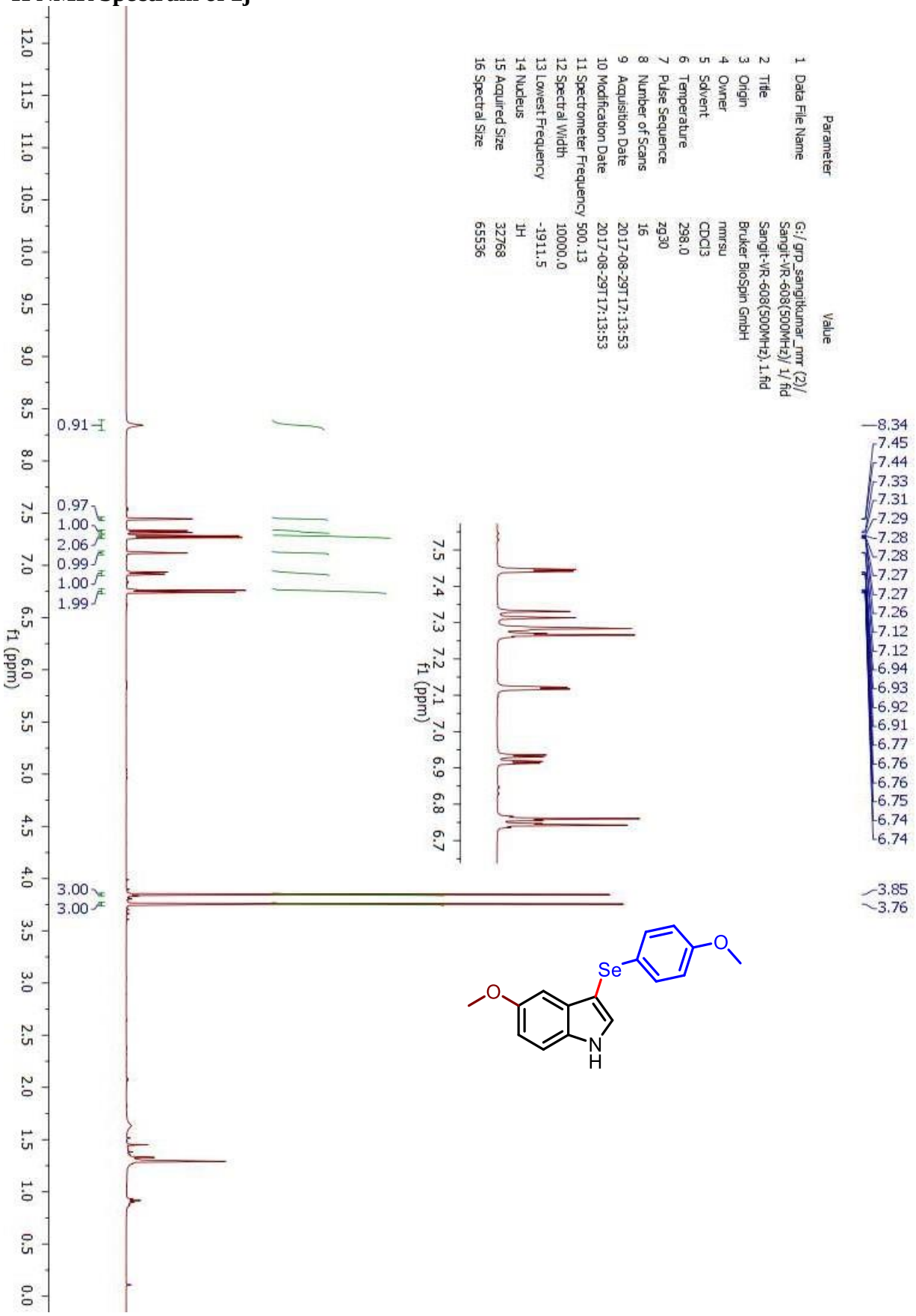
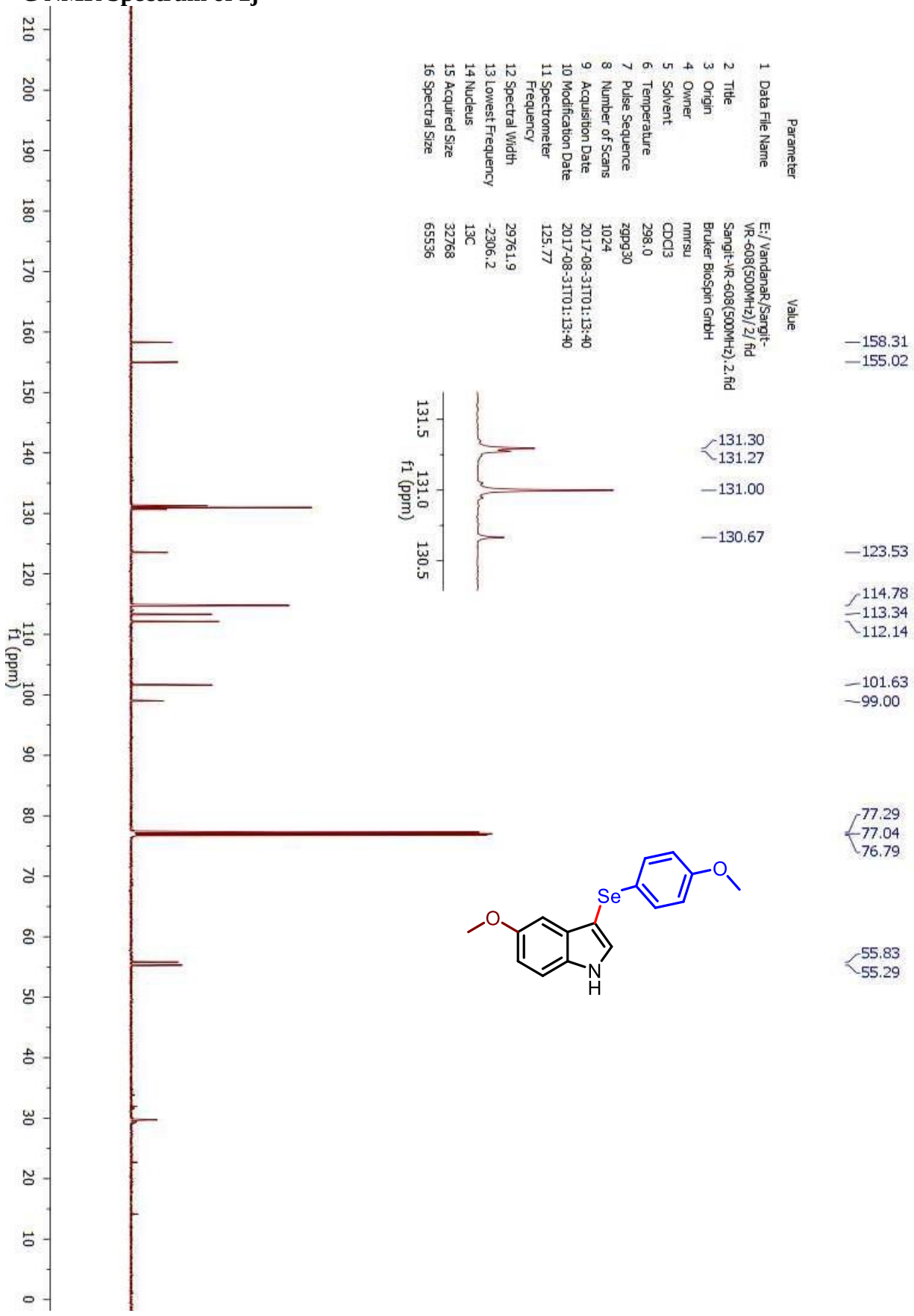
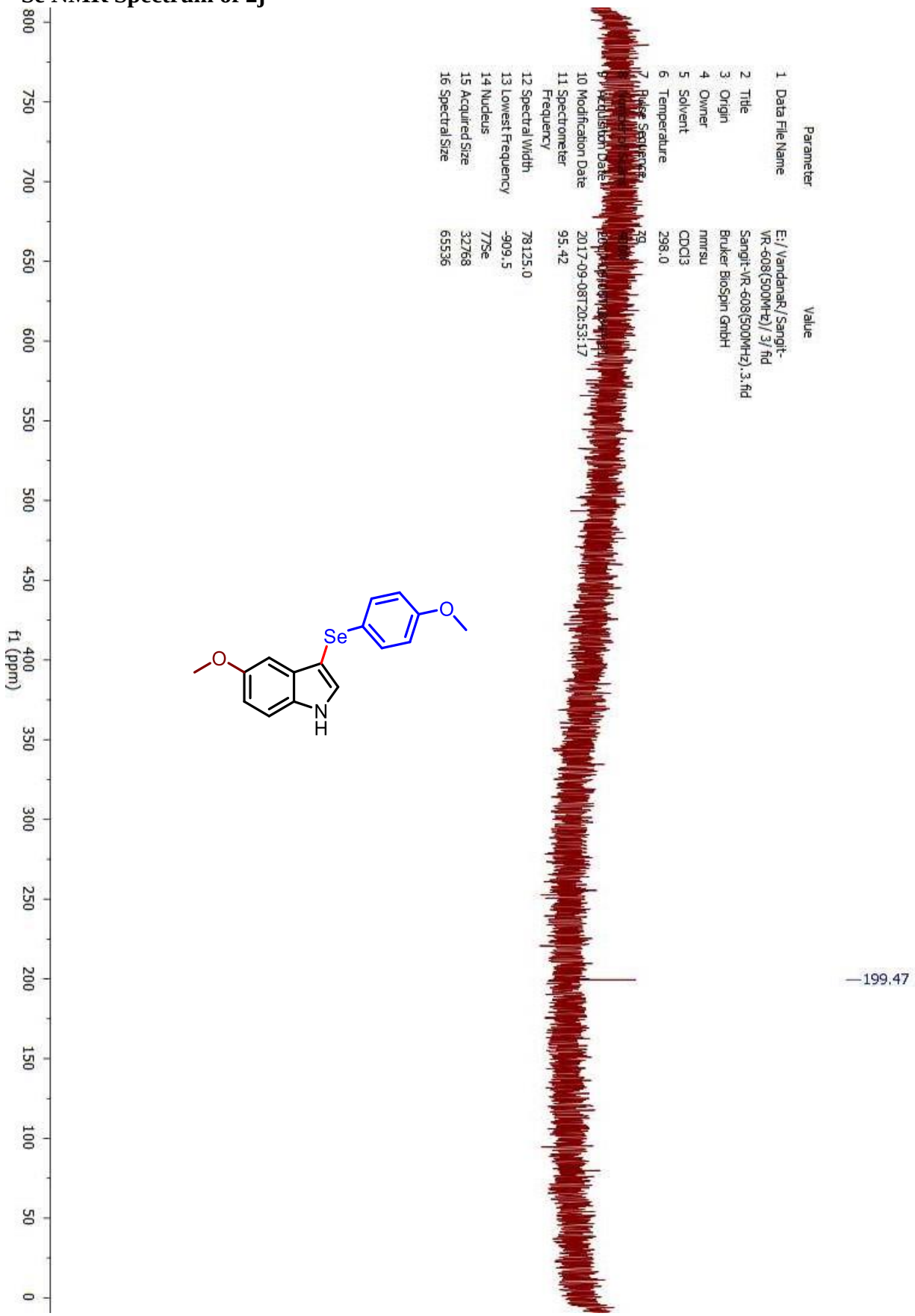
^{1}H NMR Spectrum of 2j



^{13}C NMR Spectrum of 2j



^{77}Se NMR Spectrum of 2j



Mass Spectrum of 2j

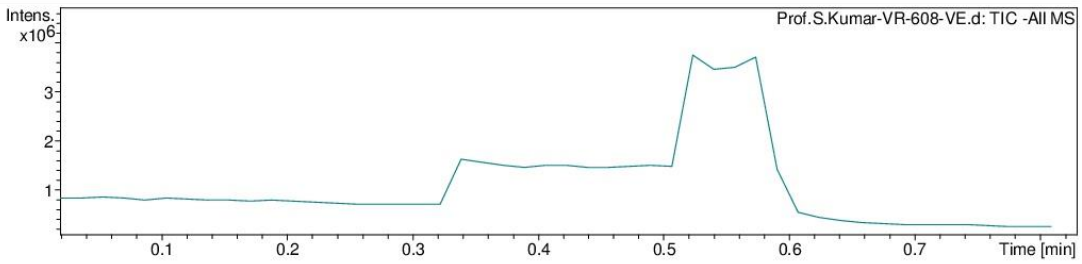
Display Report

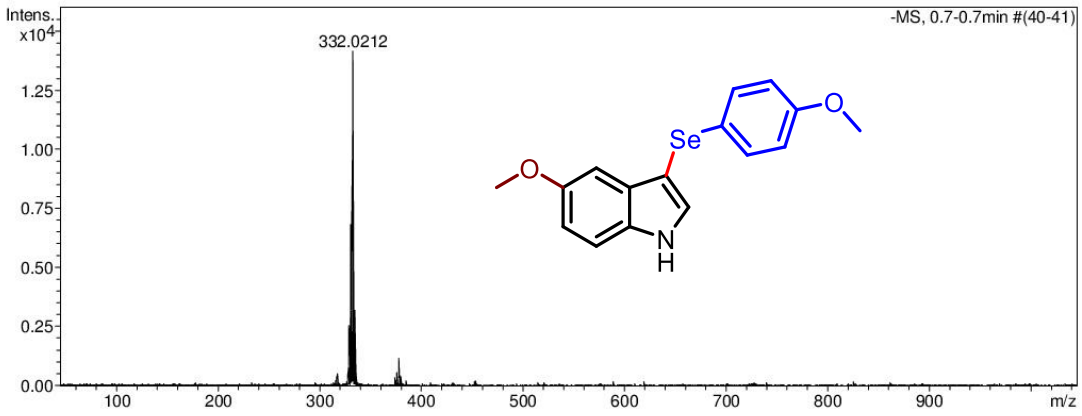
Analysis Info

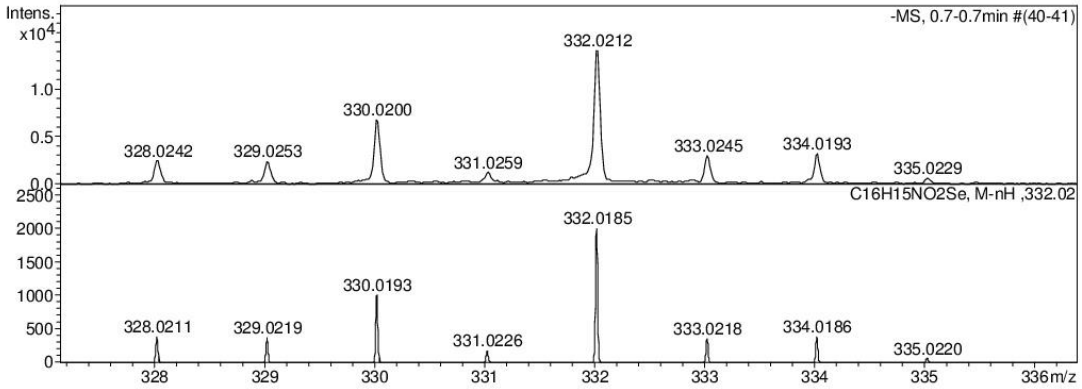
Analysis Name	D:\Data\NEW USER DATA 2017\2018\JUNE 2018\11 JUNE\Prof.S.Kumar-VR-608-VE.d	Acquisition Date	6/11/2018 4:34:43 PM
Method	tune_low.m	Operator	RUCHI
Sample Name	VR-608-VE	Instrument	micrOTOF-Q II 10330
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Negative	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	2500 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	100.0 Vpp	Set Divert Valve	Waste







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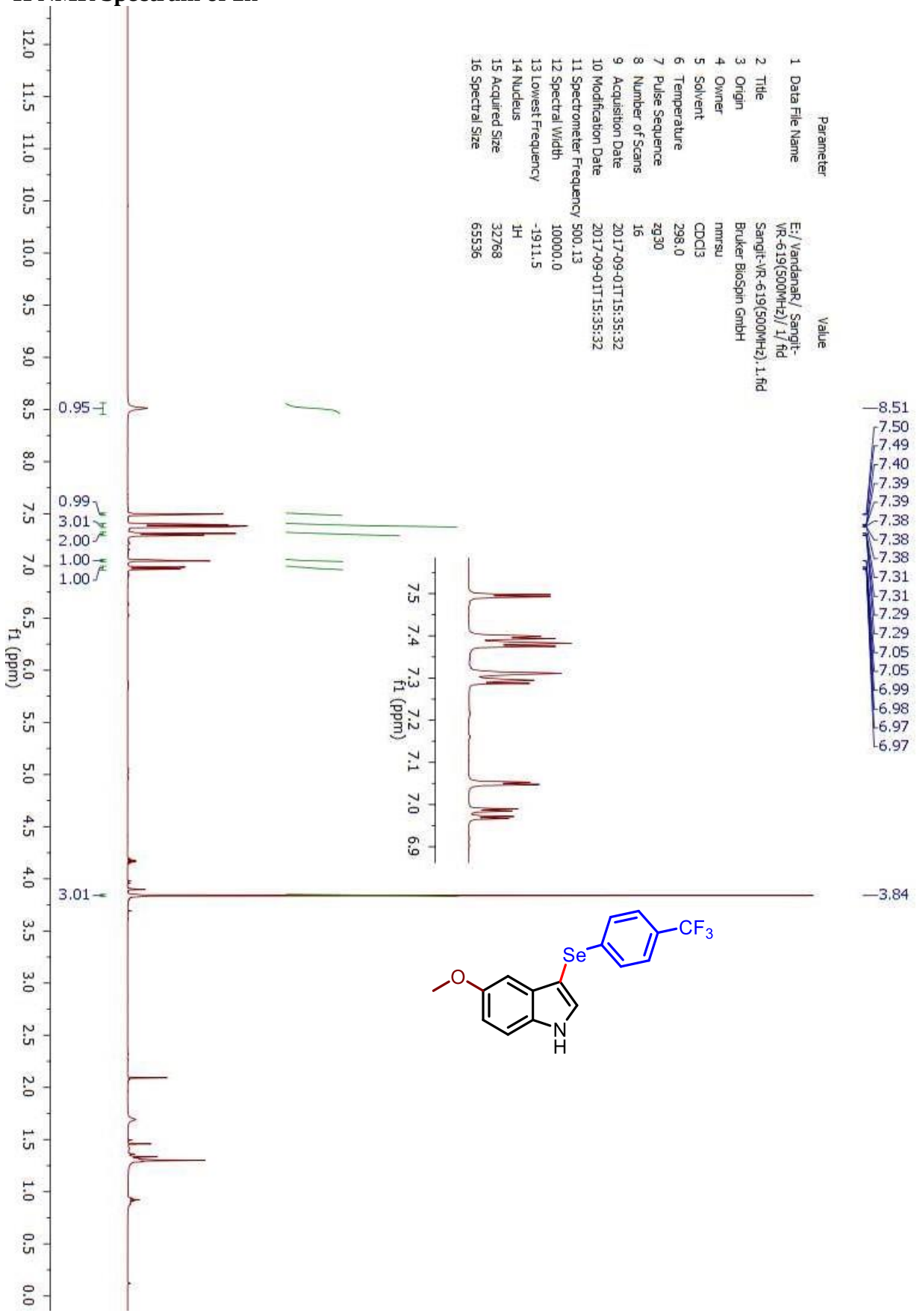
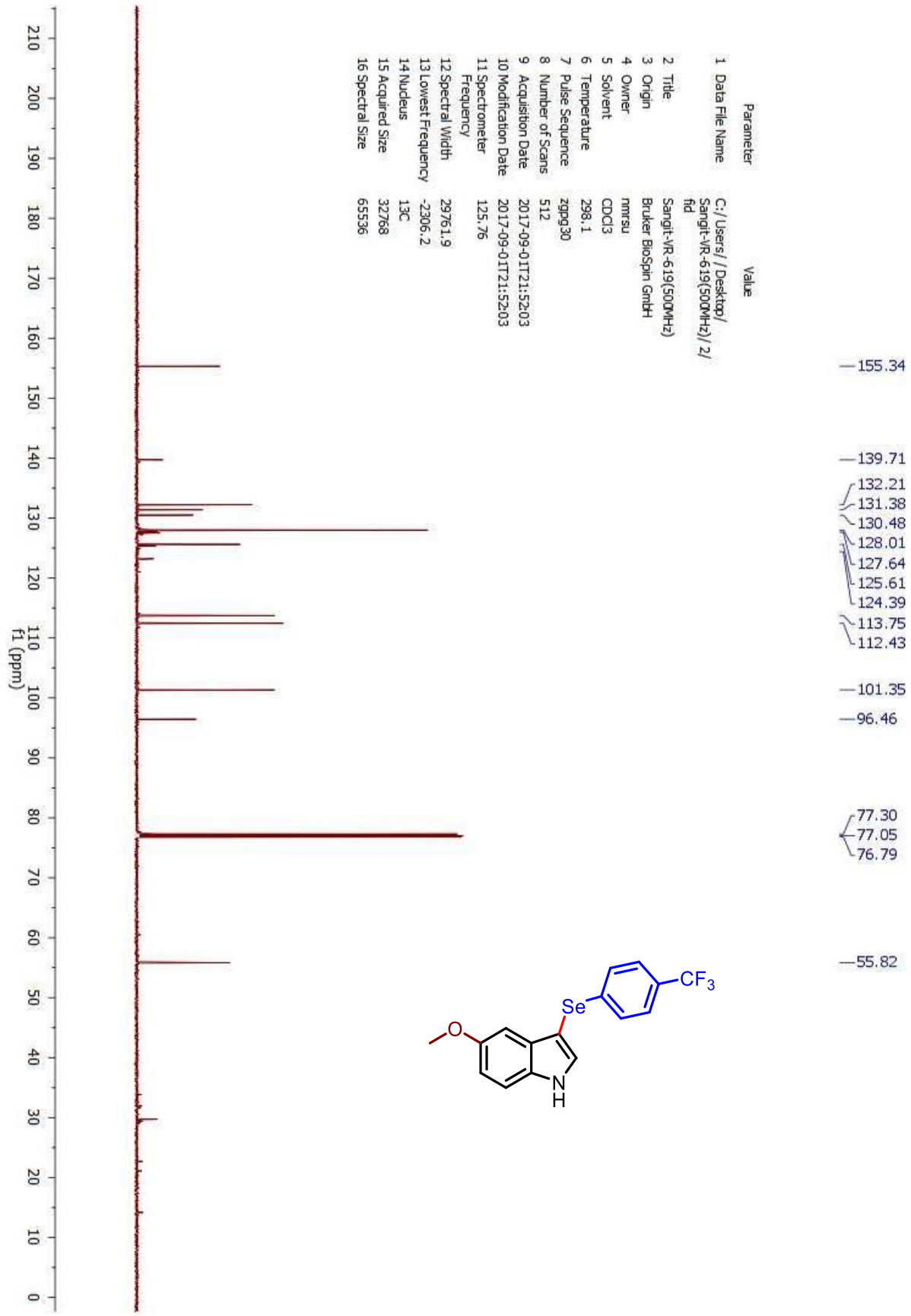
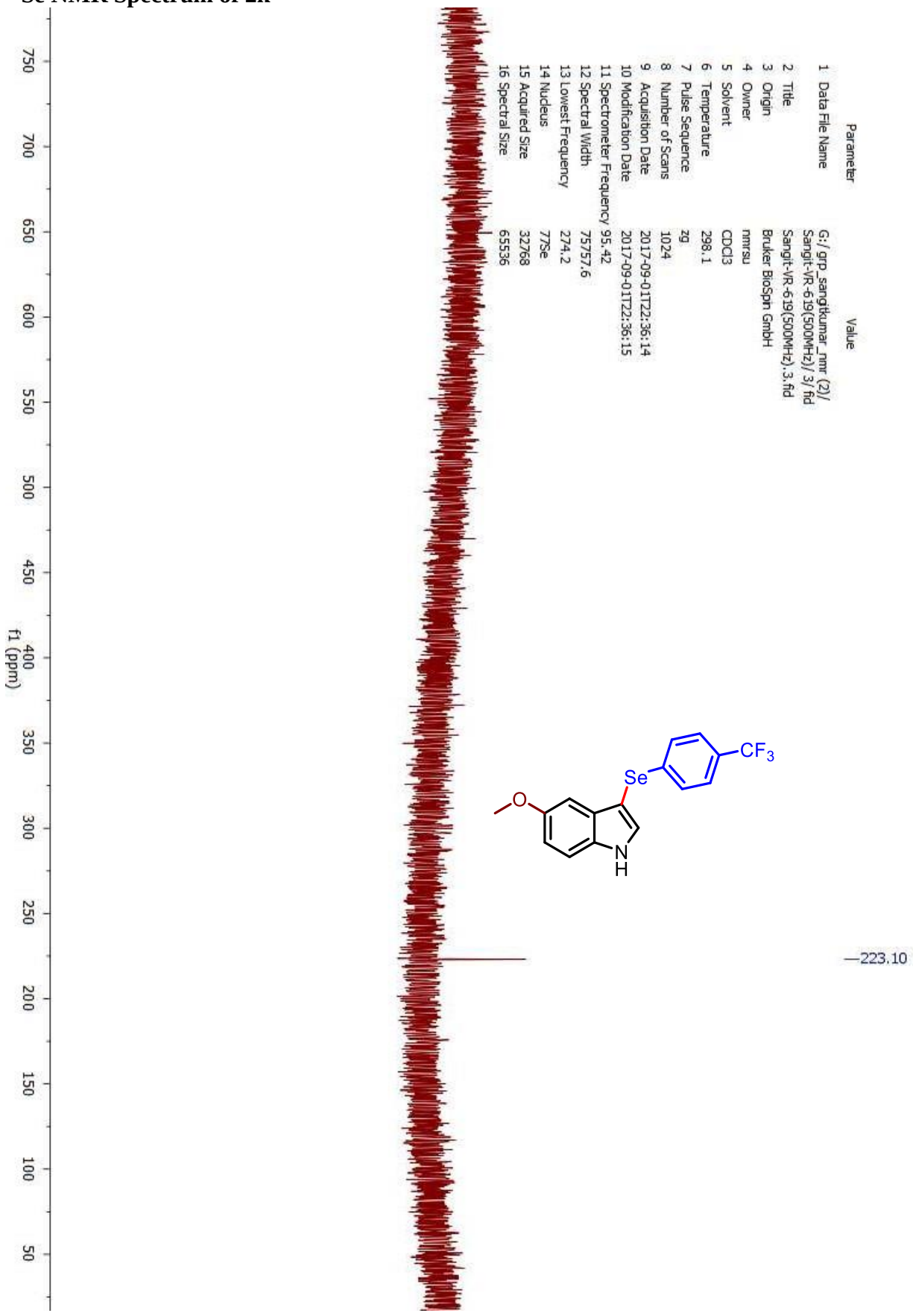
^{1}H NMR Spectrum of 2k



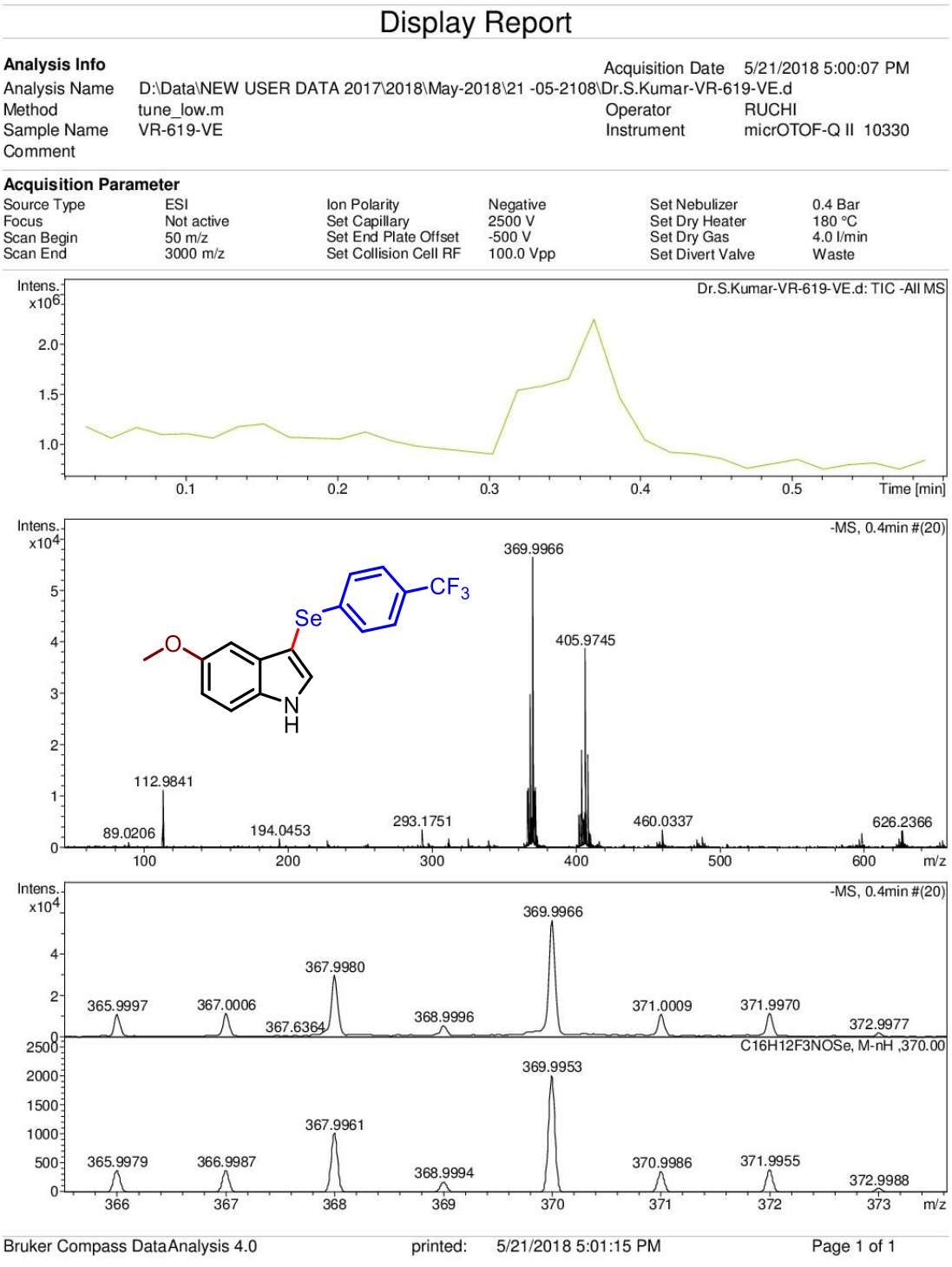
^{13}C NMR Spectrum of 2k



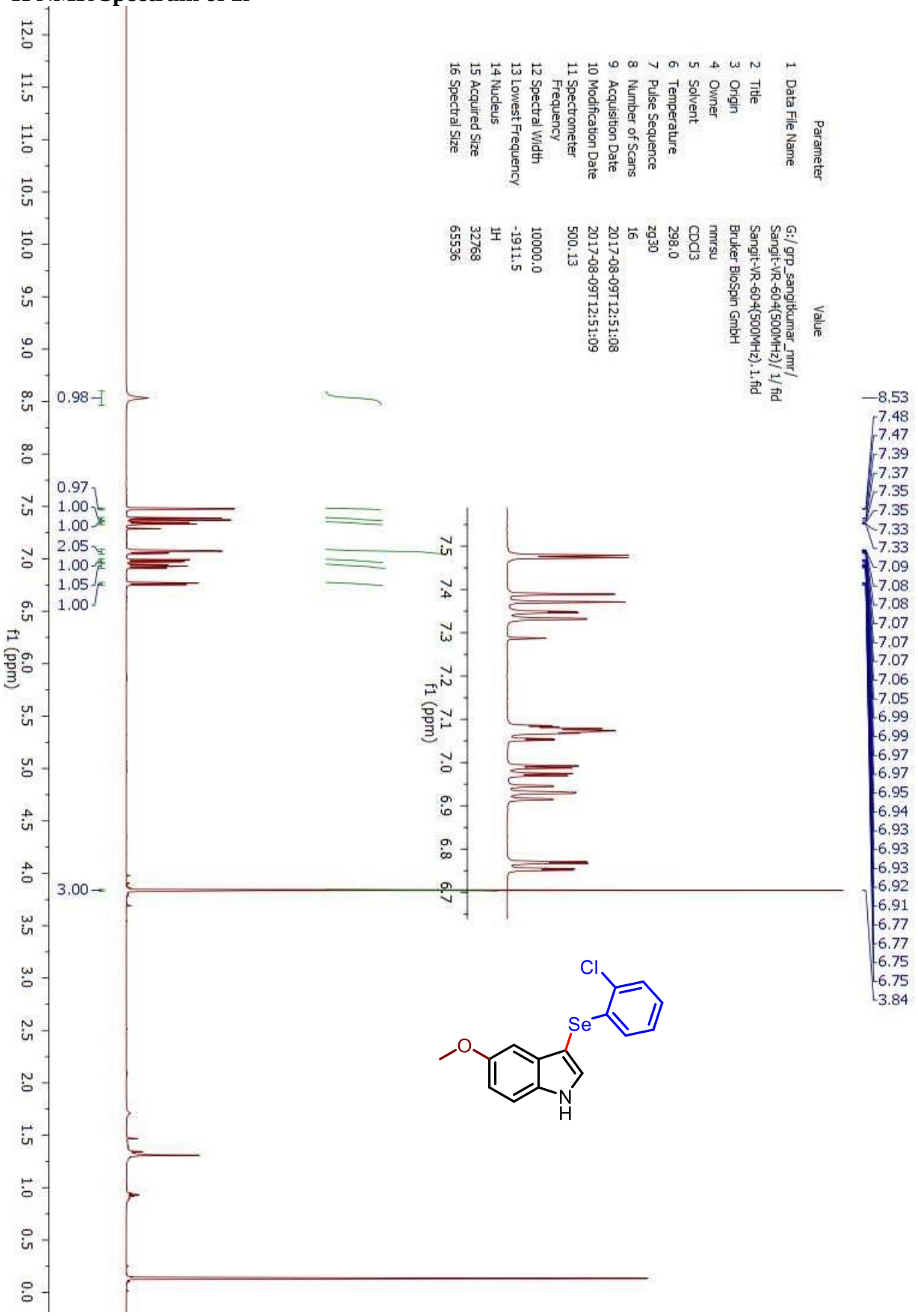
^{77}Se NMR Spectrum of 2k



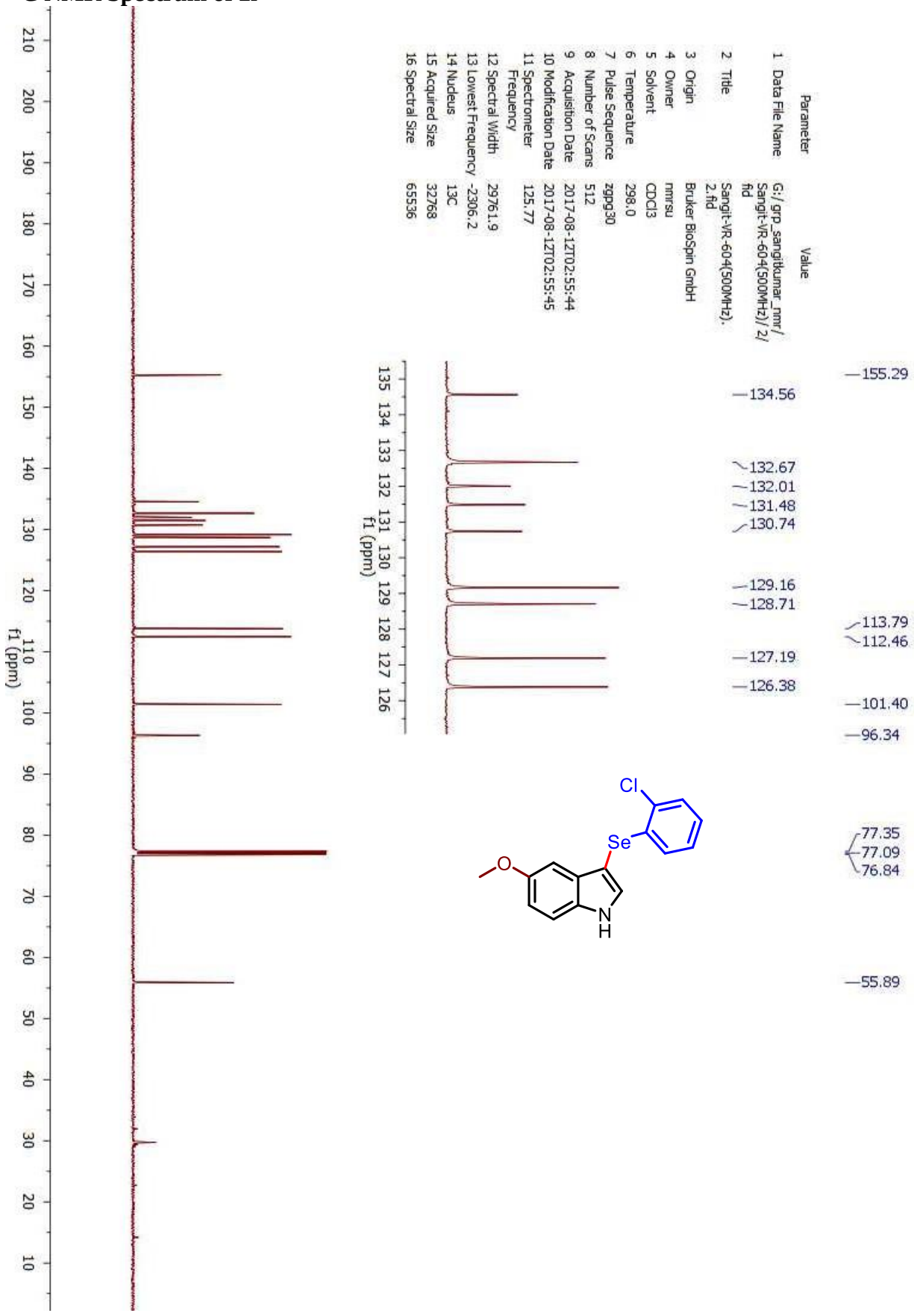
Mass Spectrum of 2k



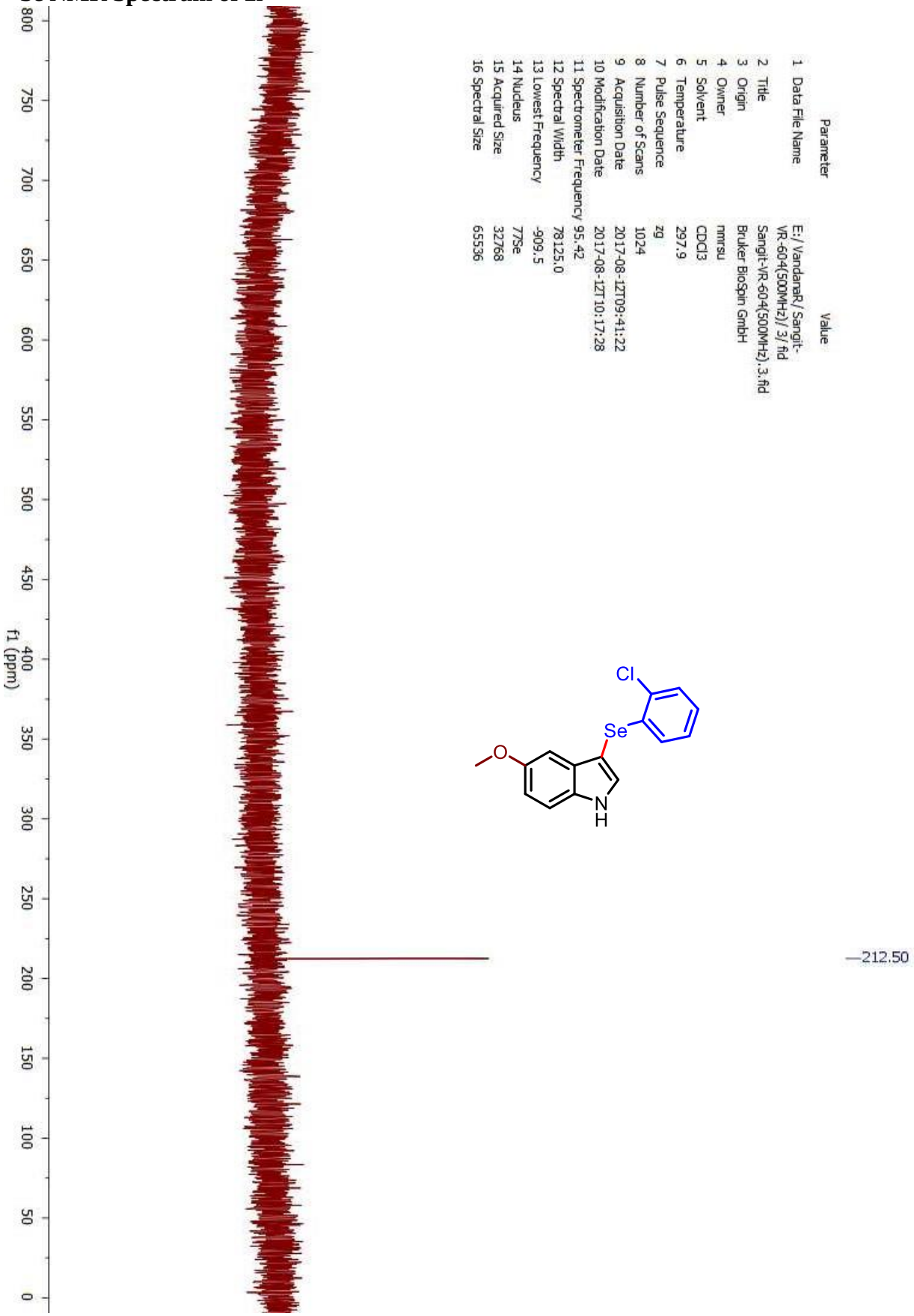
^{1}H NMR Spectrum of 2l



^{13}C NMR Spectrum of 2l



^{77}Se NMR Spectrum of 2l



	Parameter	Value
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2	Title	Sangit-VR-604(500MHz).3.fid
3	Origin	Bruker BioSpin GmbH
4	Owner	nmrsu
5	Solvent	CDCl3
6	Temperature	297.9
7	Pulse Sequence	zg
8	Number of Scans	1024
9	Acquisition Date	2017-08-12T09:41:22
10	Modification Date	2017-08-12T10:17:28
11	Spectrometer Frequency	95.42
12	Spectral Width	78125.0
13	Lowest Frequency	-909.5
14	Nucleus	77Se
15	Acquired Size	32768
16	Spectral Size	65536

Mass Spectrum of 2l

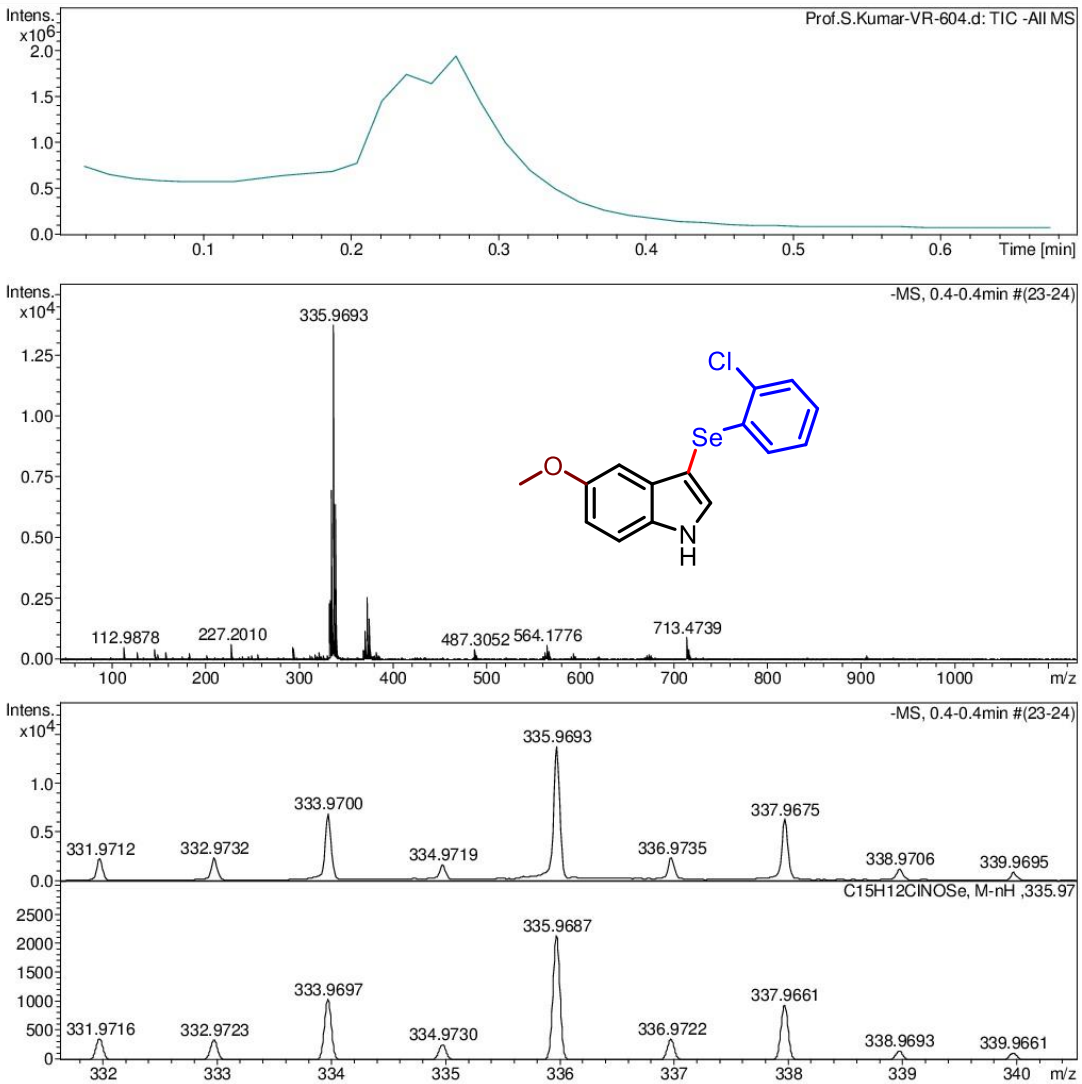
Display Report

Analysis Info

Analysis Name	D:\Data\NEW USER DATA 2017\2018\JUNE 2018\12 JUNE\Prof.S.Kumar-VR-604.d	Acquisition Date	6/12/2018 3:52:02 PM
Method	tune_low_neg.m	Operator	RUCHI
Sample Name	VR-604	Instrument	micrOTOF-Q II 10330
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Negative	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	2500 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste



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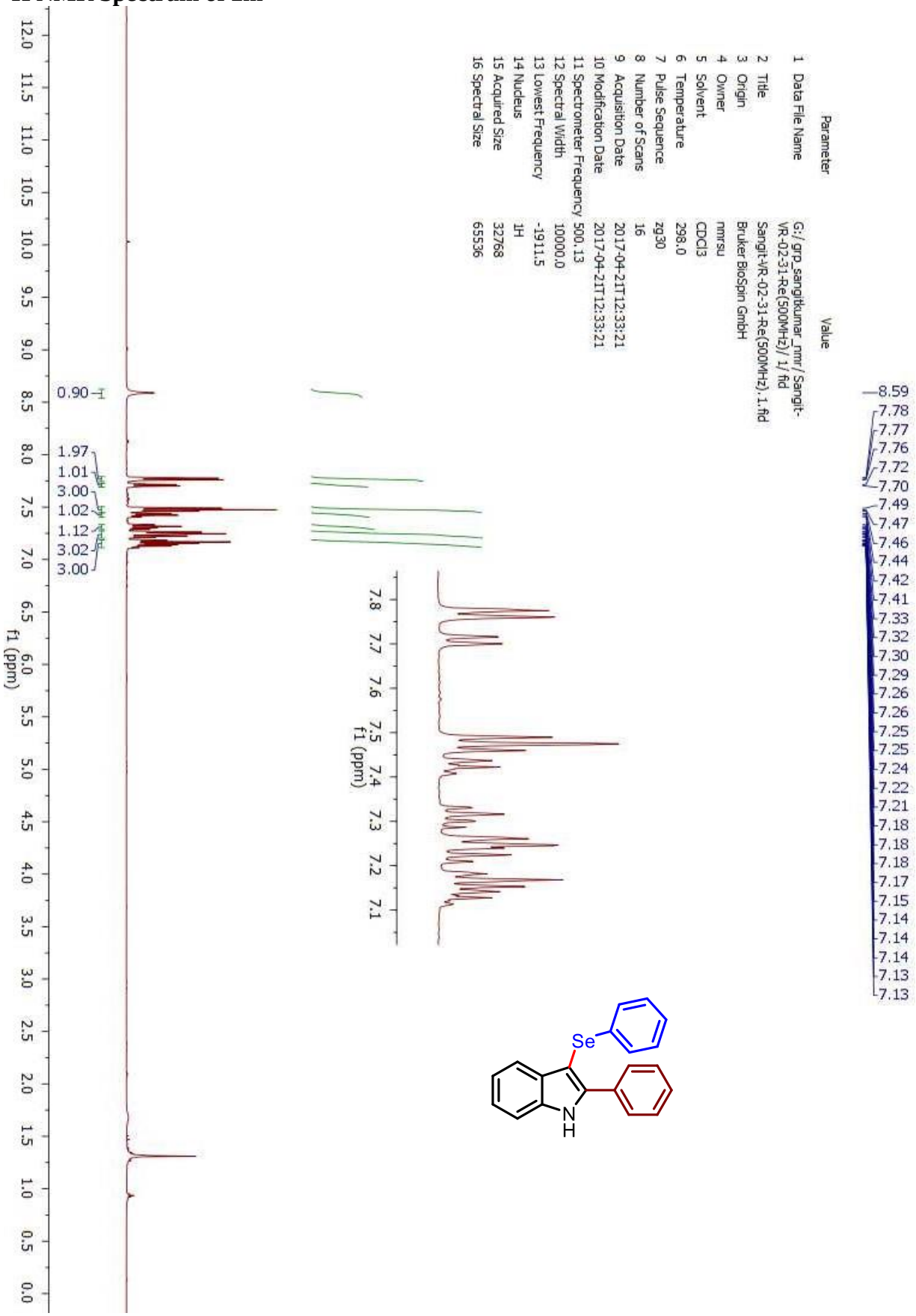
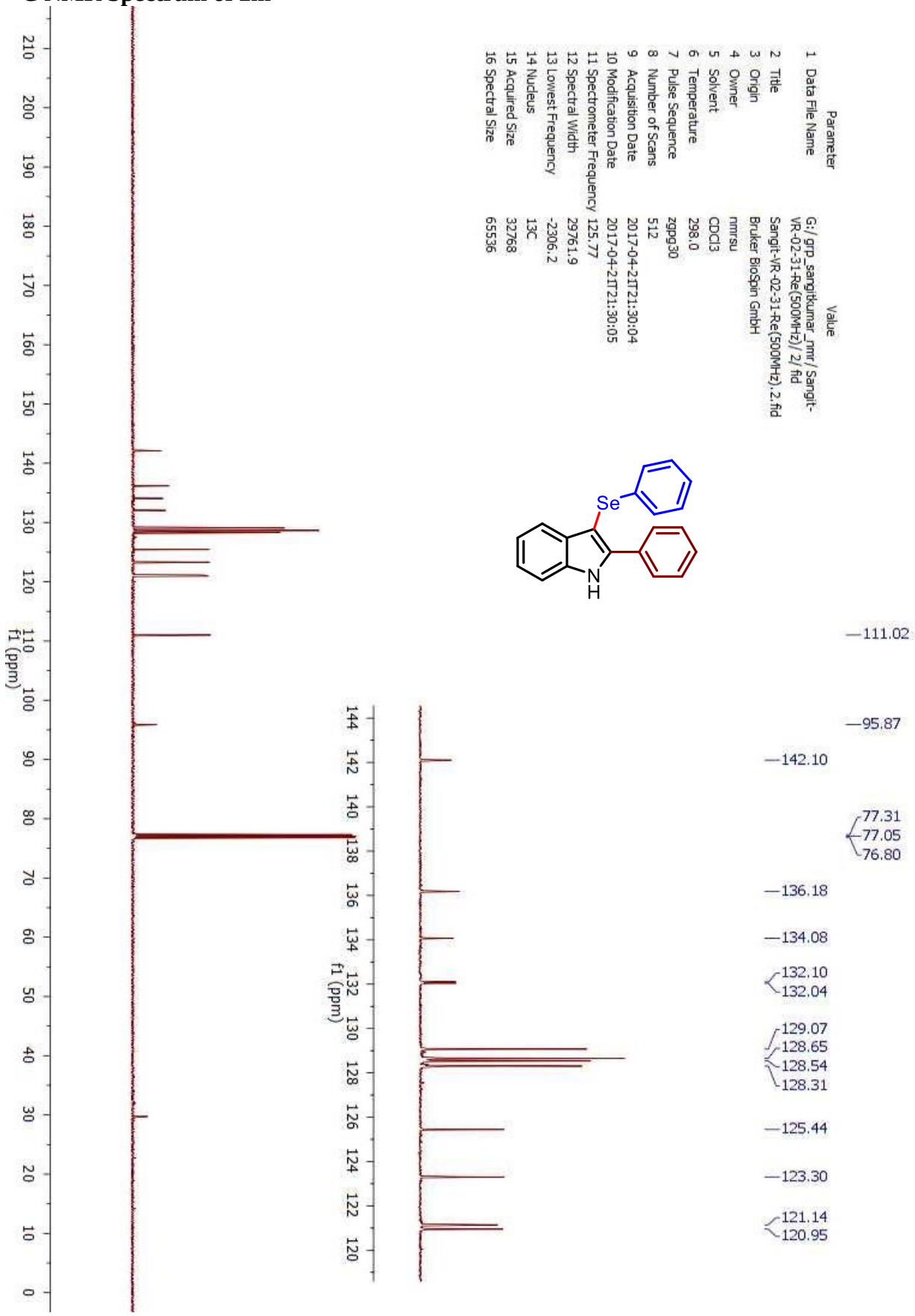
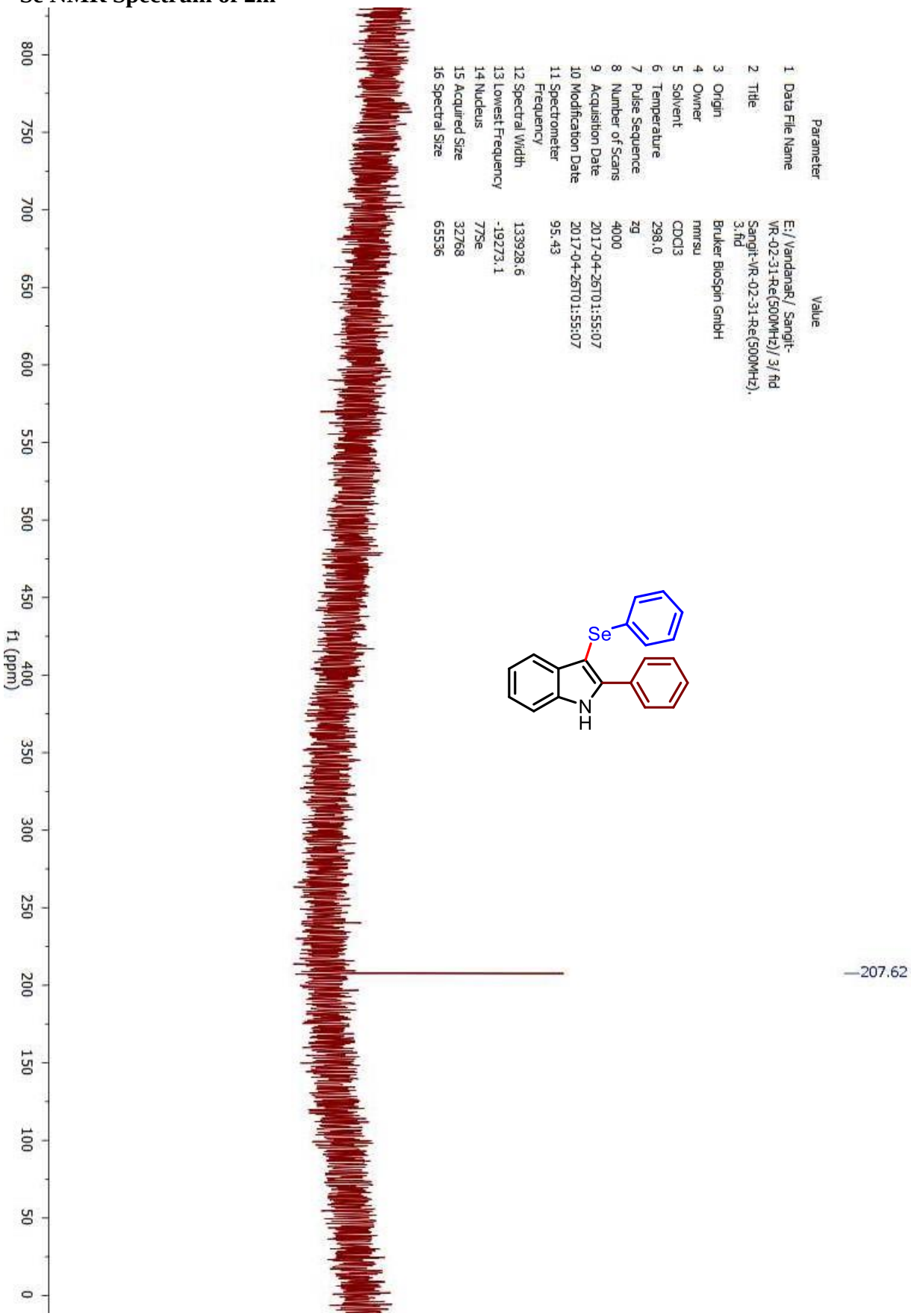
^{1}H NMR Spectrum of 2m



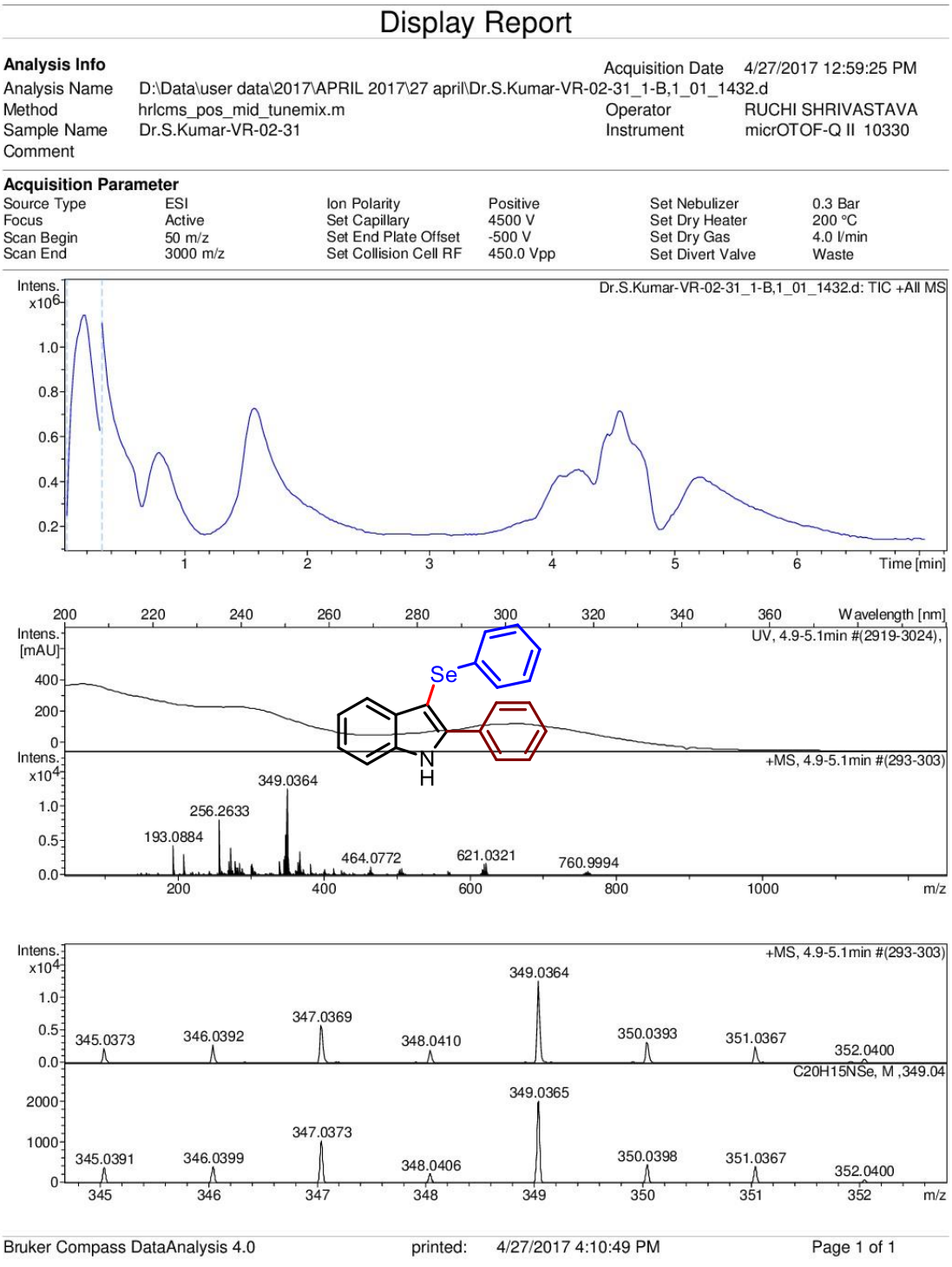
^{13}C NMR Spectrum of 2m



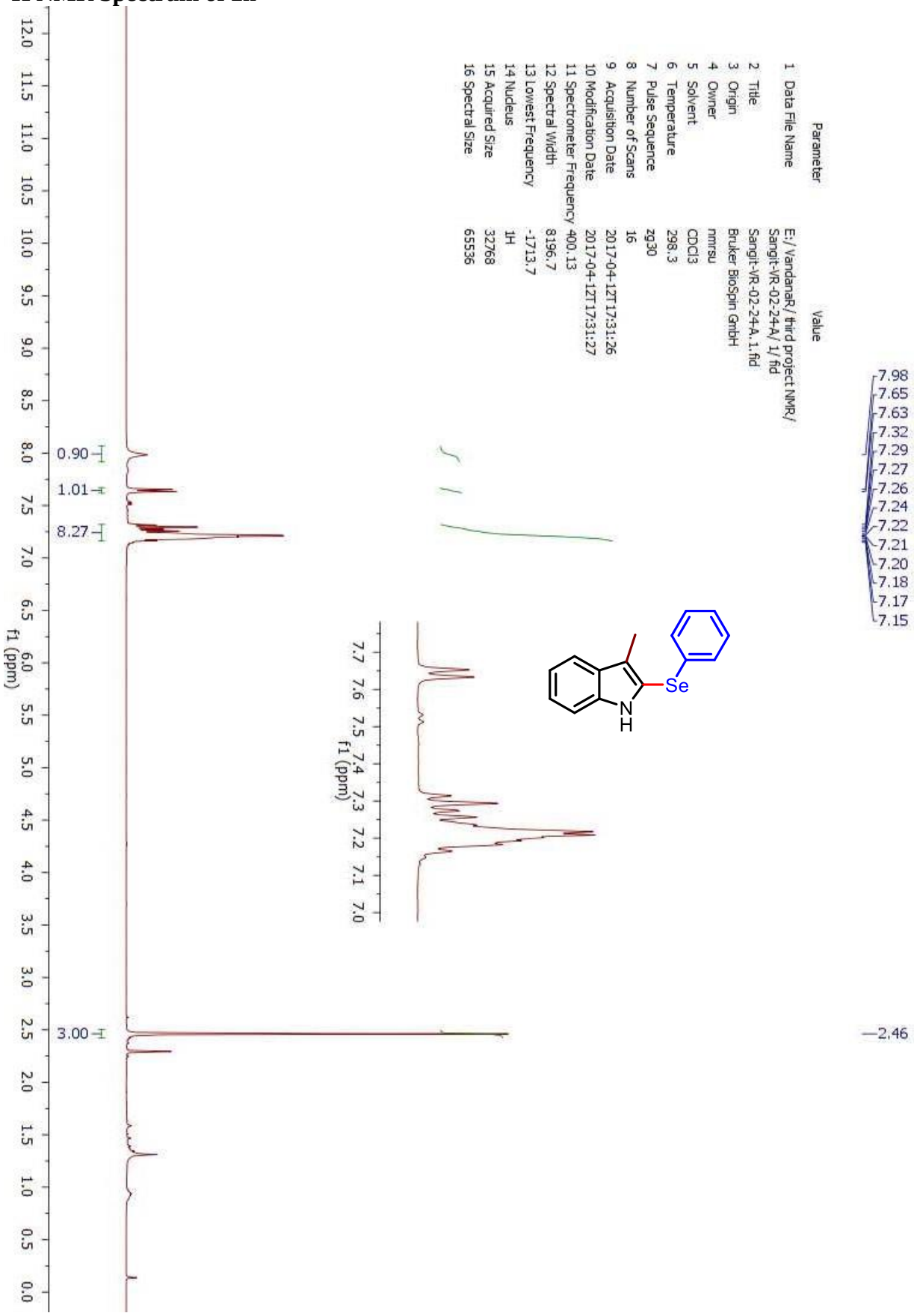
^{77}Se NMR Spectrum of 2m



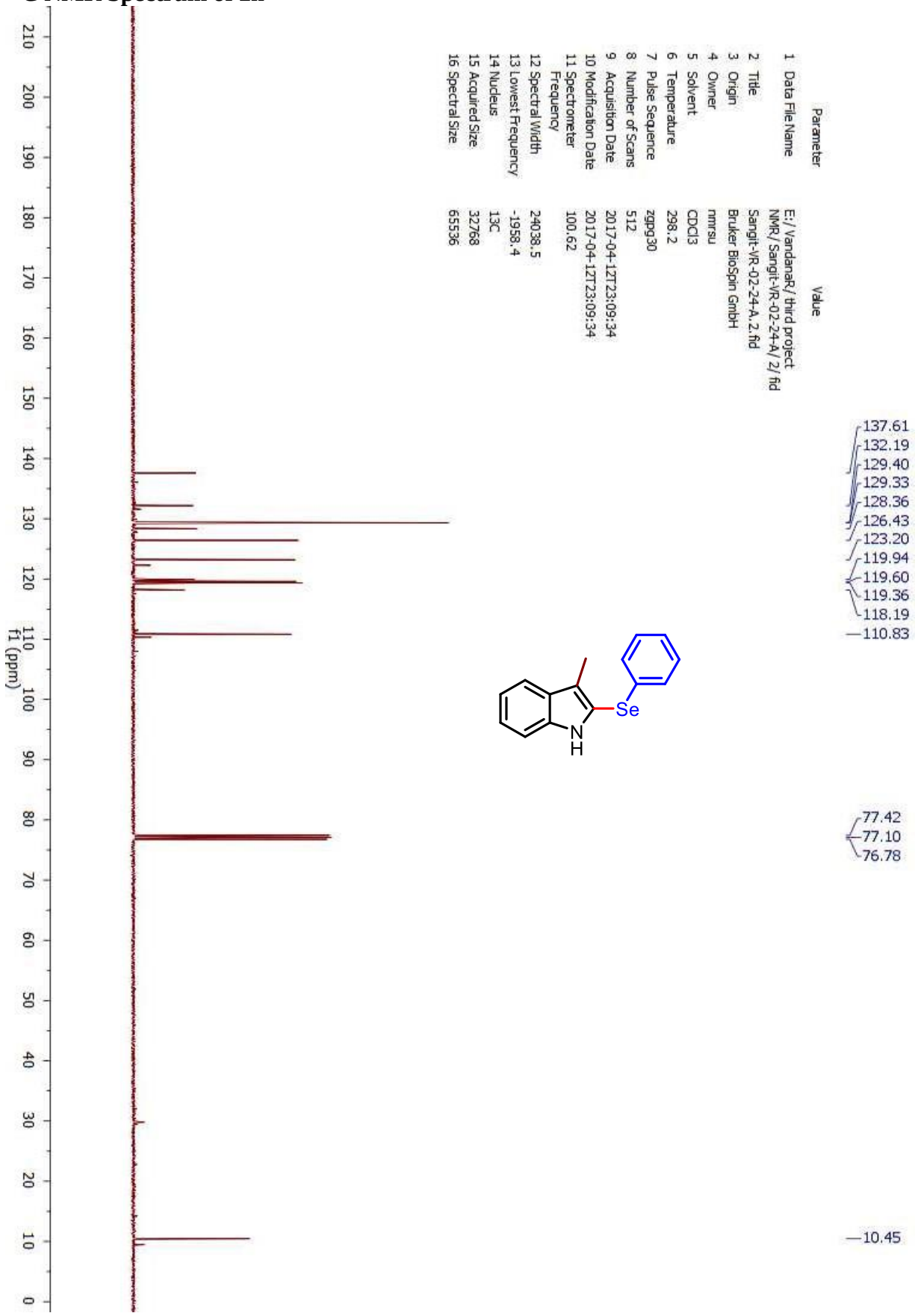
Mass Spectrum of 2m



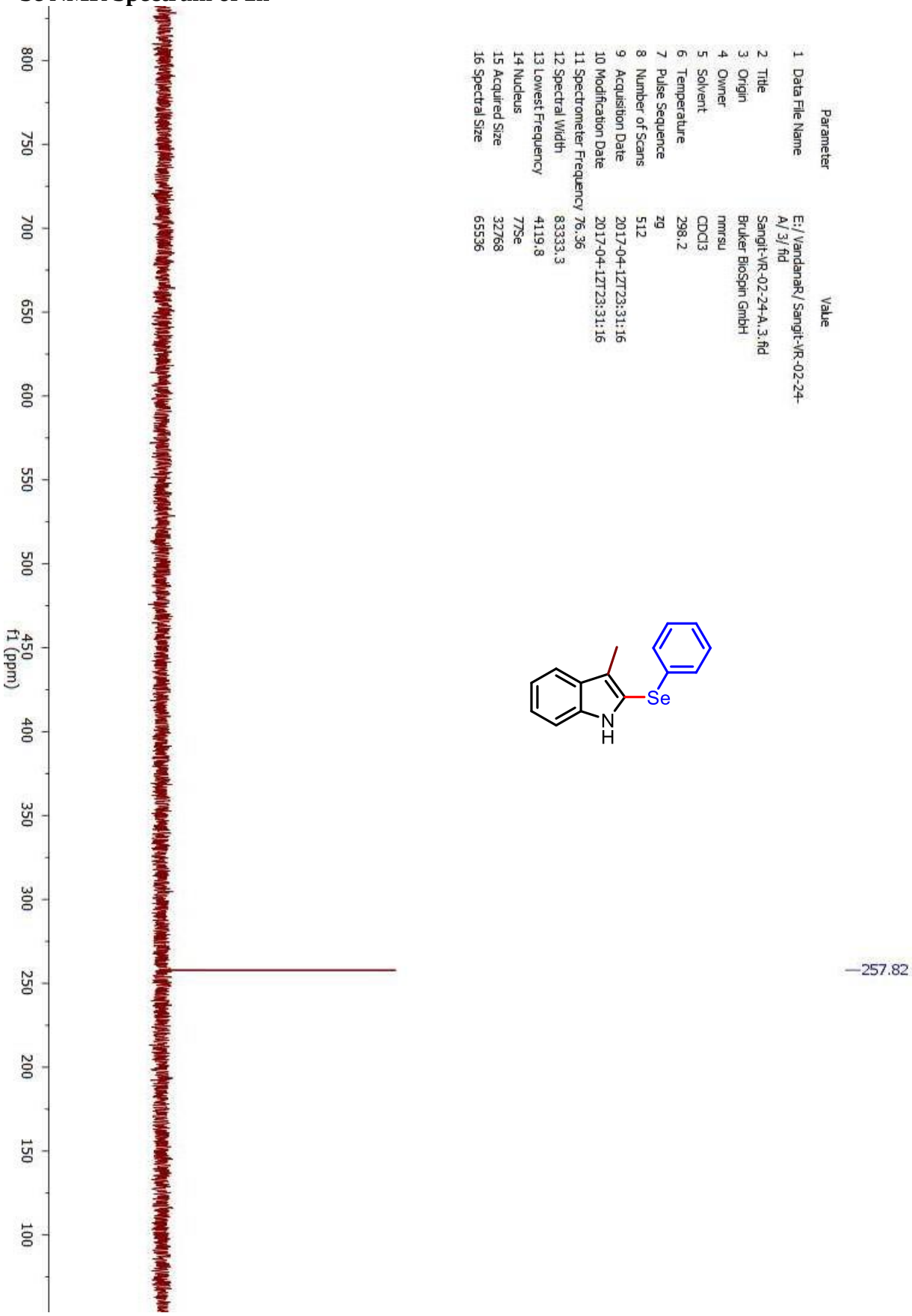
^{1}H NMR Spectrum of 2n



^{13}C NMR Spectrum of 2n



^{77}Se NMR Spectrum of 2n



Mass Spectrum of 3n

Acquisition date: 10/04/17

Sample ID: VR-02-24

Supervisor: Dr. S Kumar

Operator: **IISERB-CIF-Mass Facility**

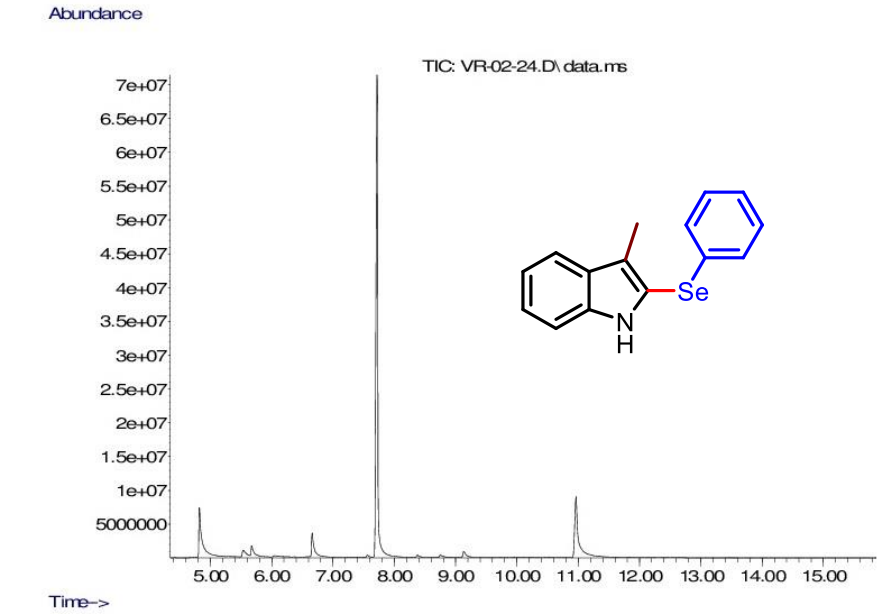
***Instrument*: Agilent 7890A GC with 5975C MS system**

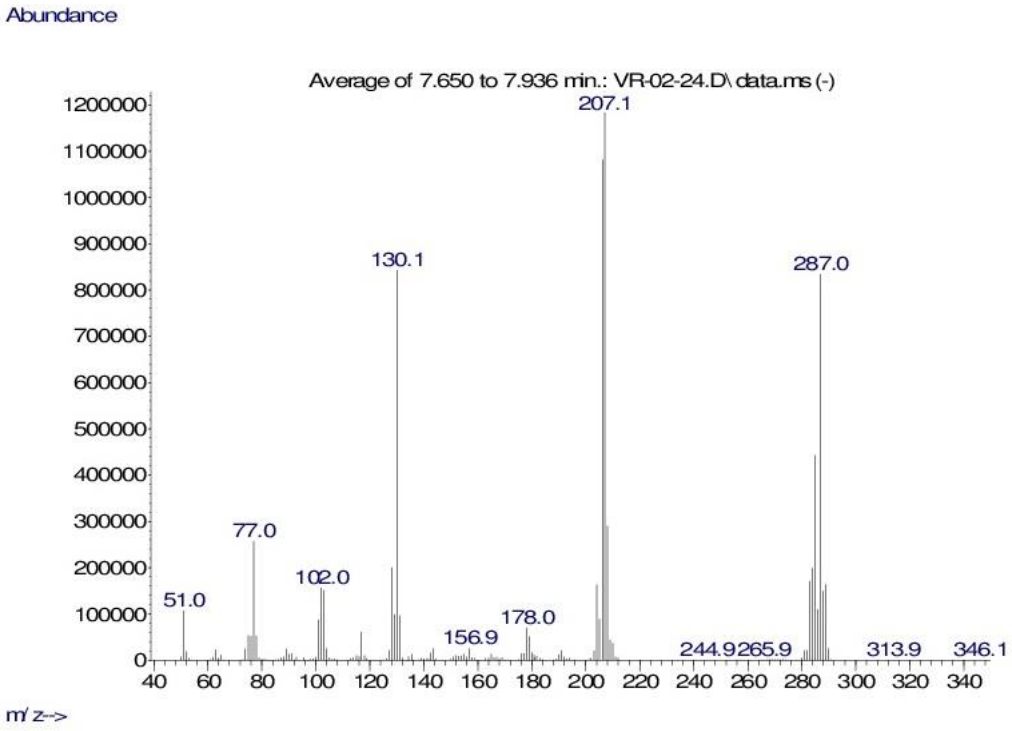
***Column*: HP-5**

Ionization: **EI (70 eV)**

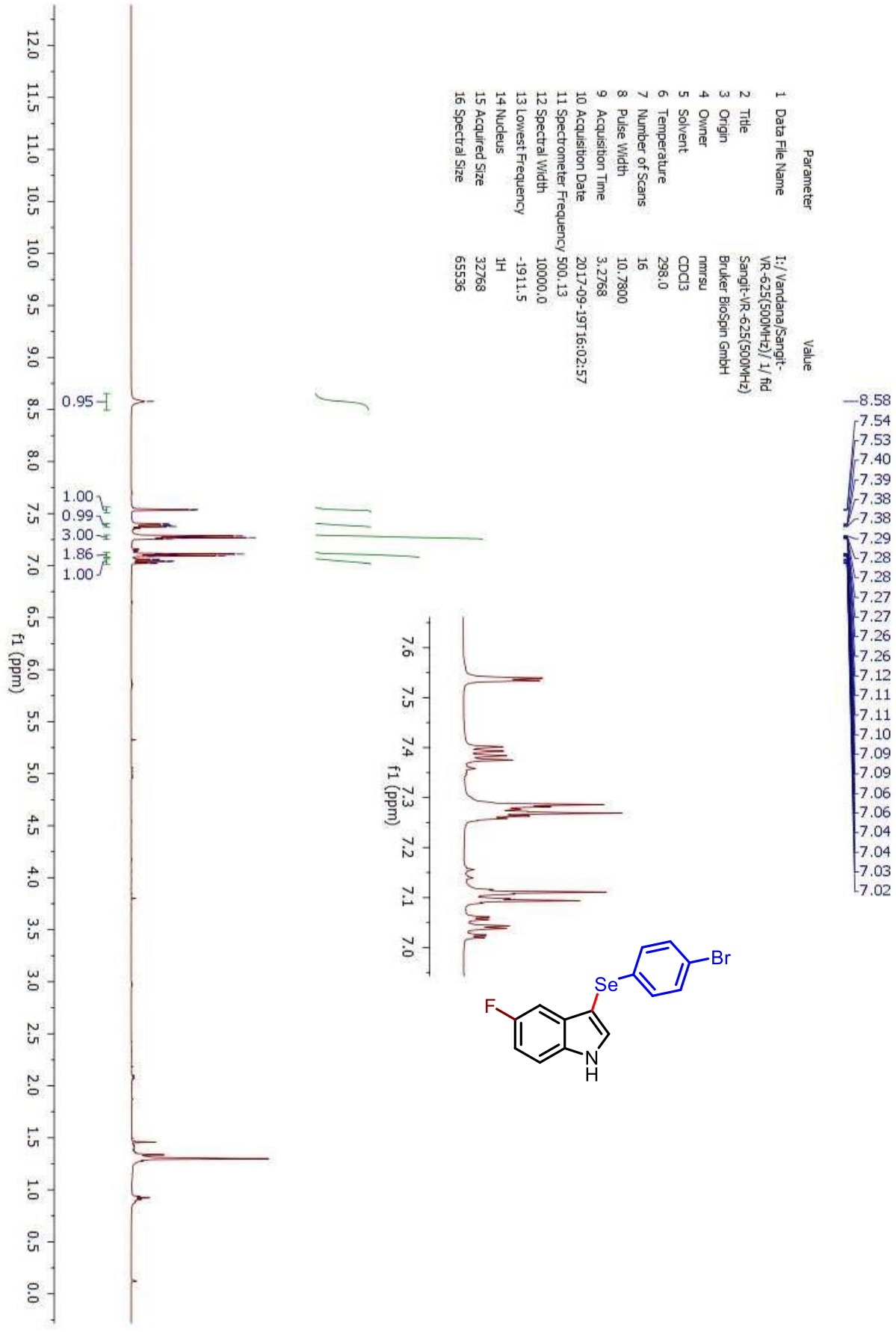
Method: **General_1_HP5_80_DEG.M**

***MSD*: Single Quad.**

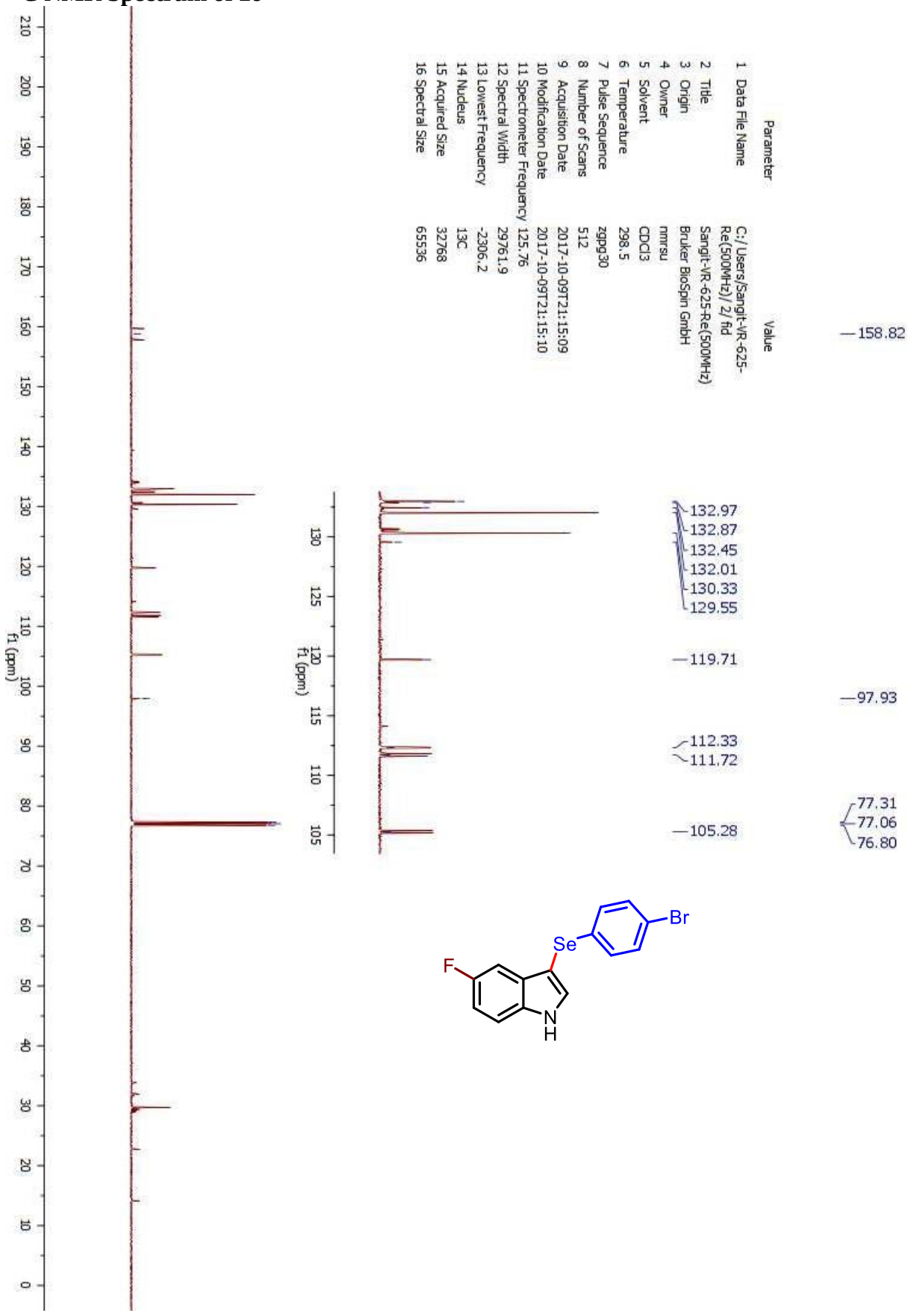




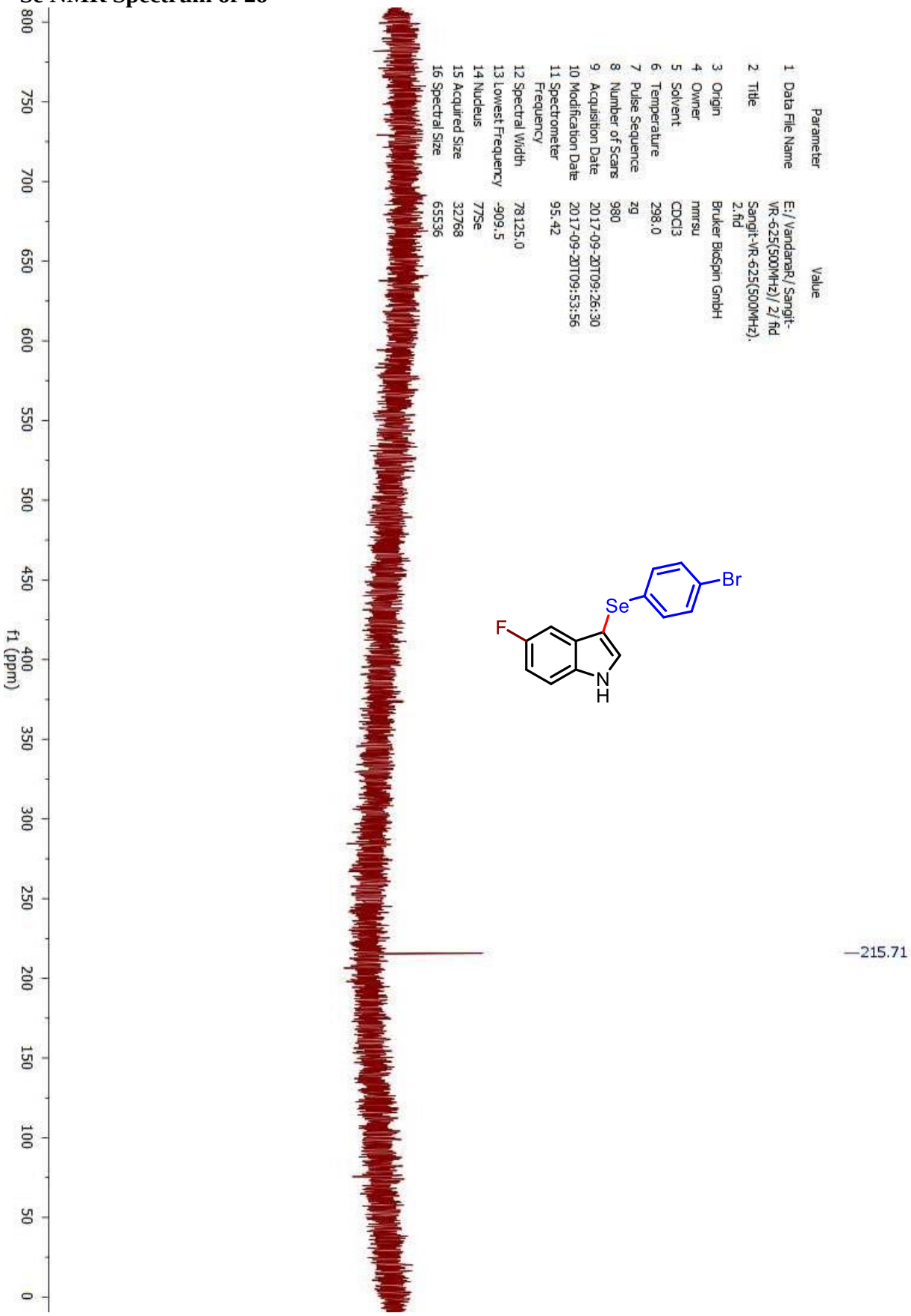
^{1}H NMR Spectrum of 2o



^{13}C NMR Spectrum of 2o



^{77}Se NMR Spectrum of 2o



Mass Spectrum of 2o

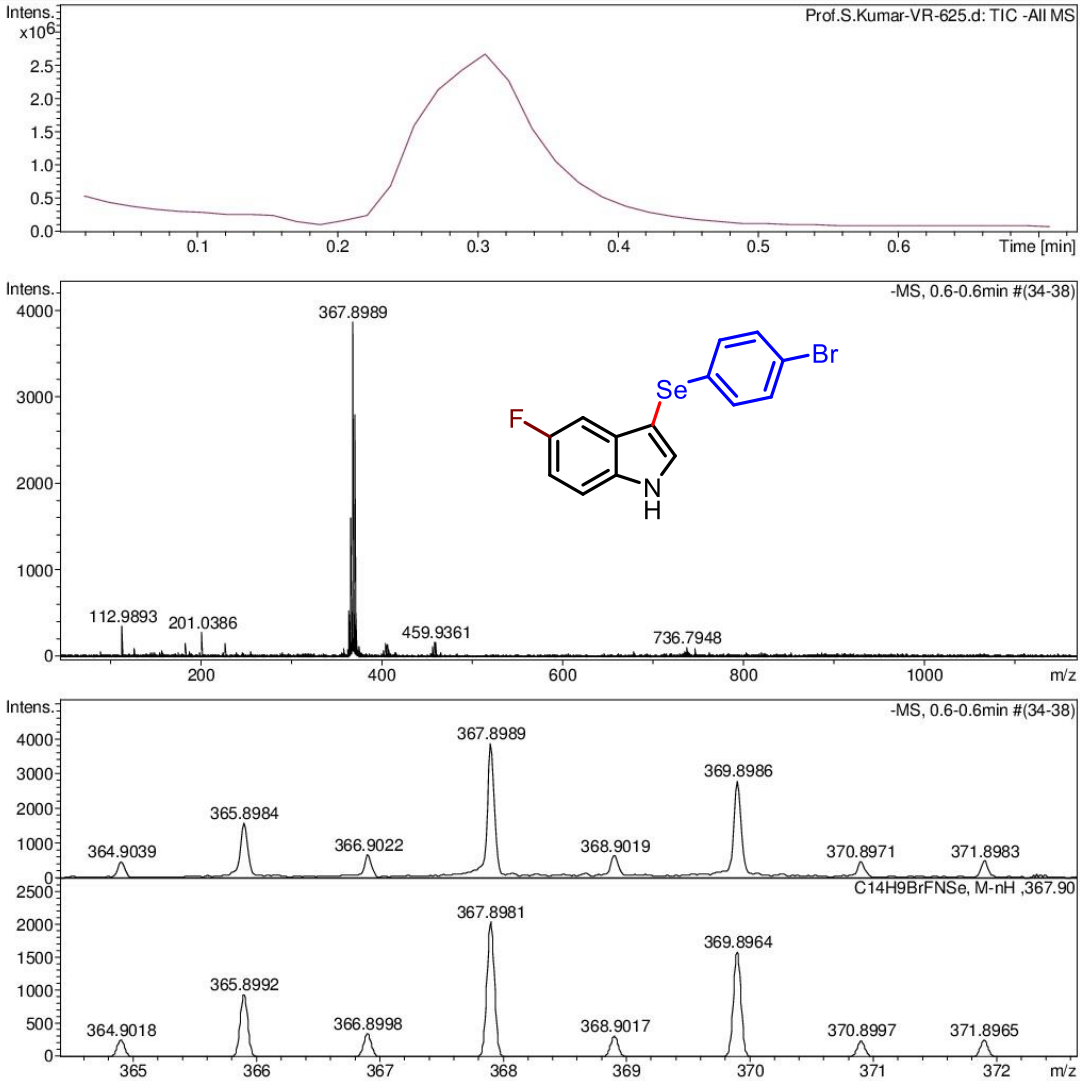
Display Report

Analysis Info

Analysis Name	D:\Data\NEW USER DATA 2017\2018\JUNE 2018\12 JUNE\Prof.S.Kumar-VR-625.d	Acquisition Date	6/12/2018 3:54:52 PM
Method	tune_low_neg.m	Operator	RUCHI
Sample Name	VR-625	Instrument	micrOTOF-Q II 10330
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Negative	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	2500 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste



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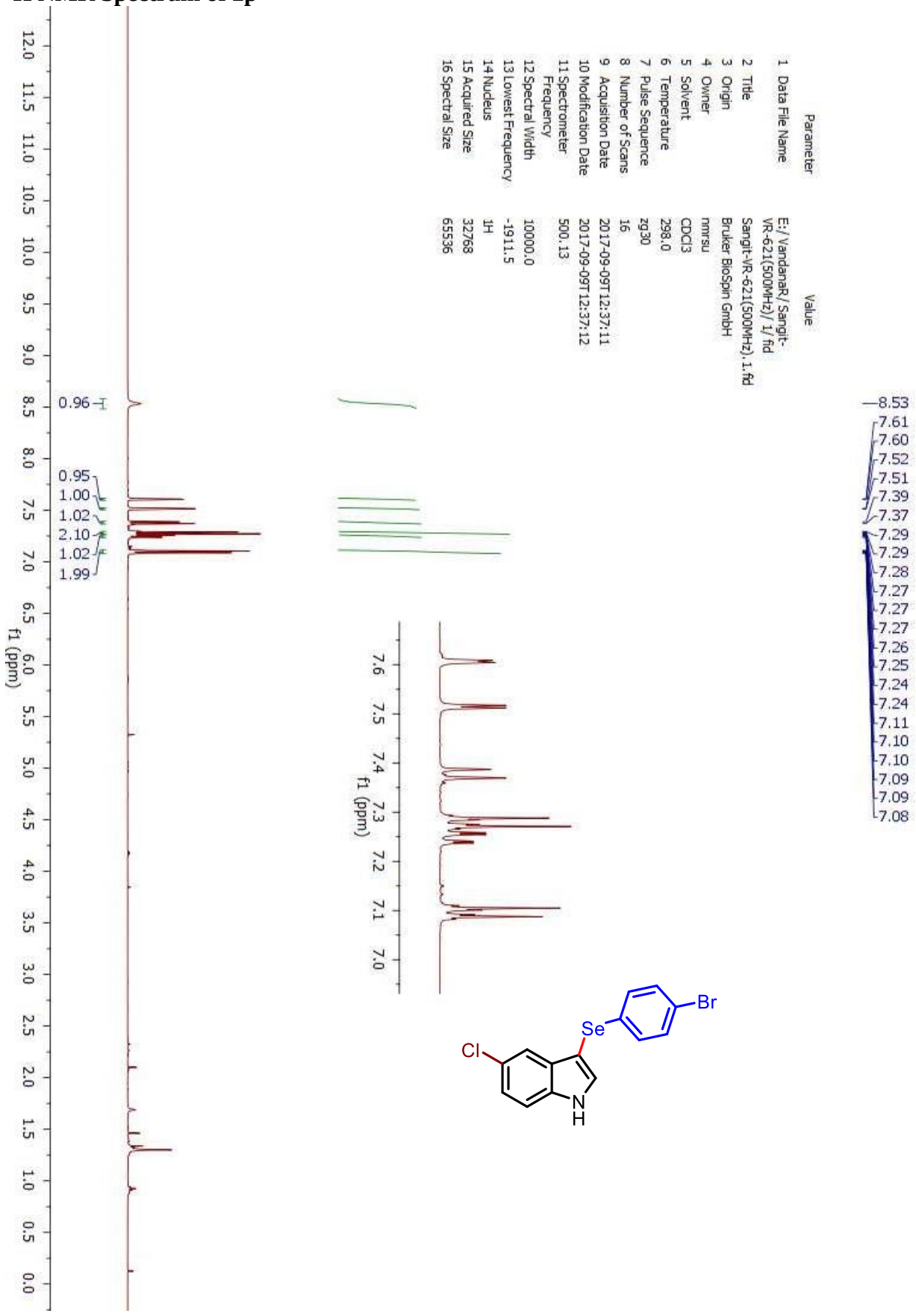
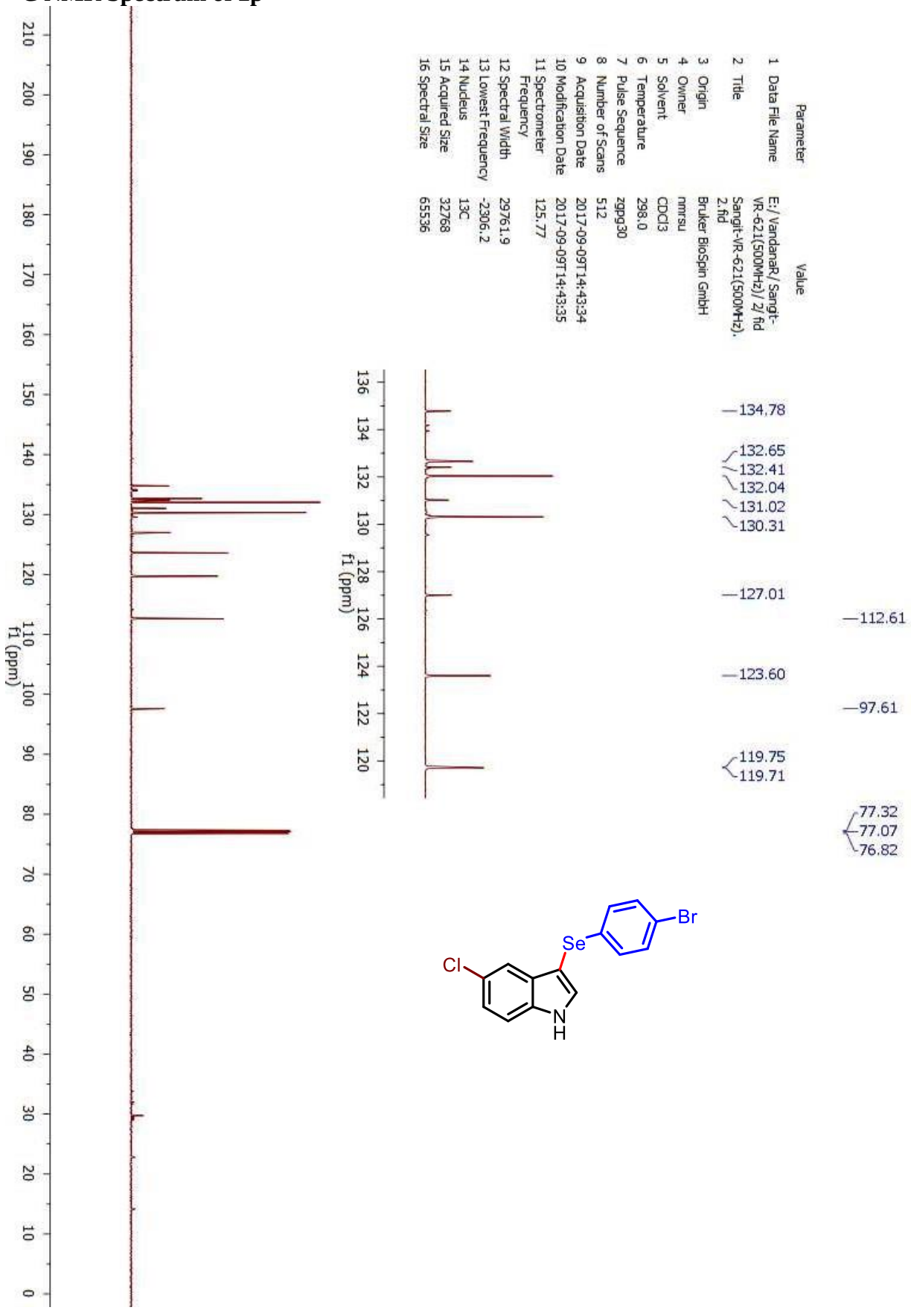
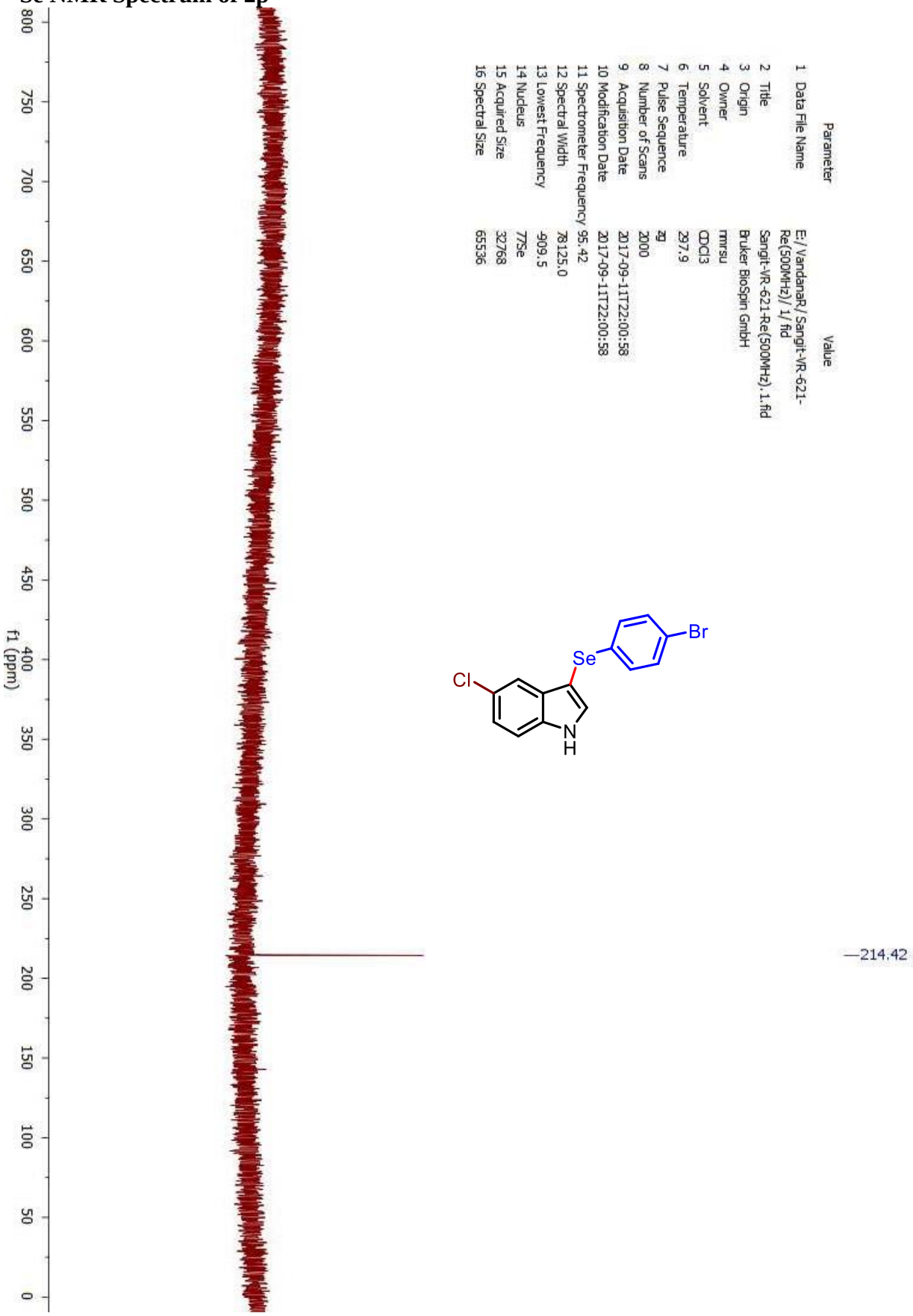
^{1}H NMR Spectrum of 2p



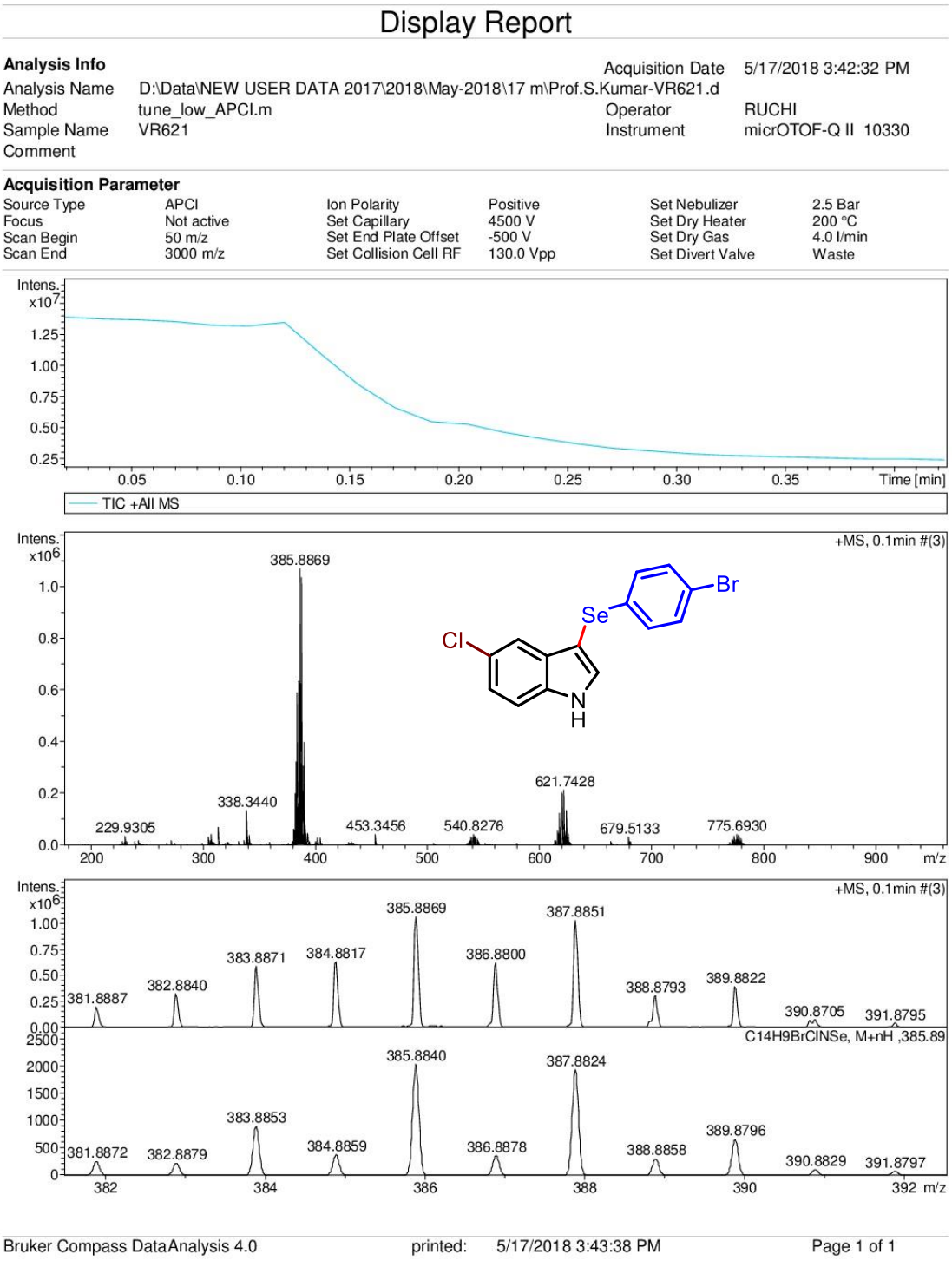
^{13}C NMR Spectrum of 2p



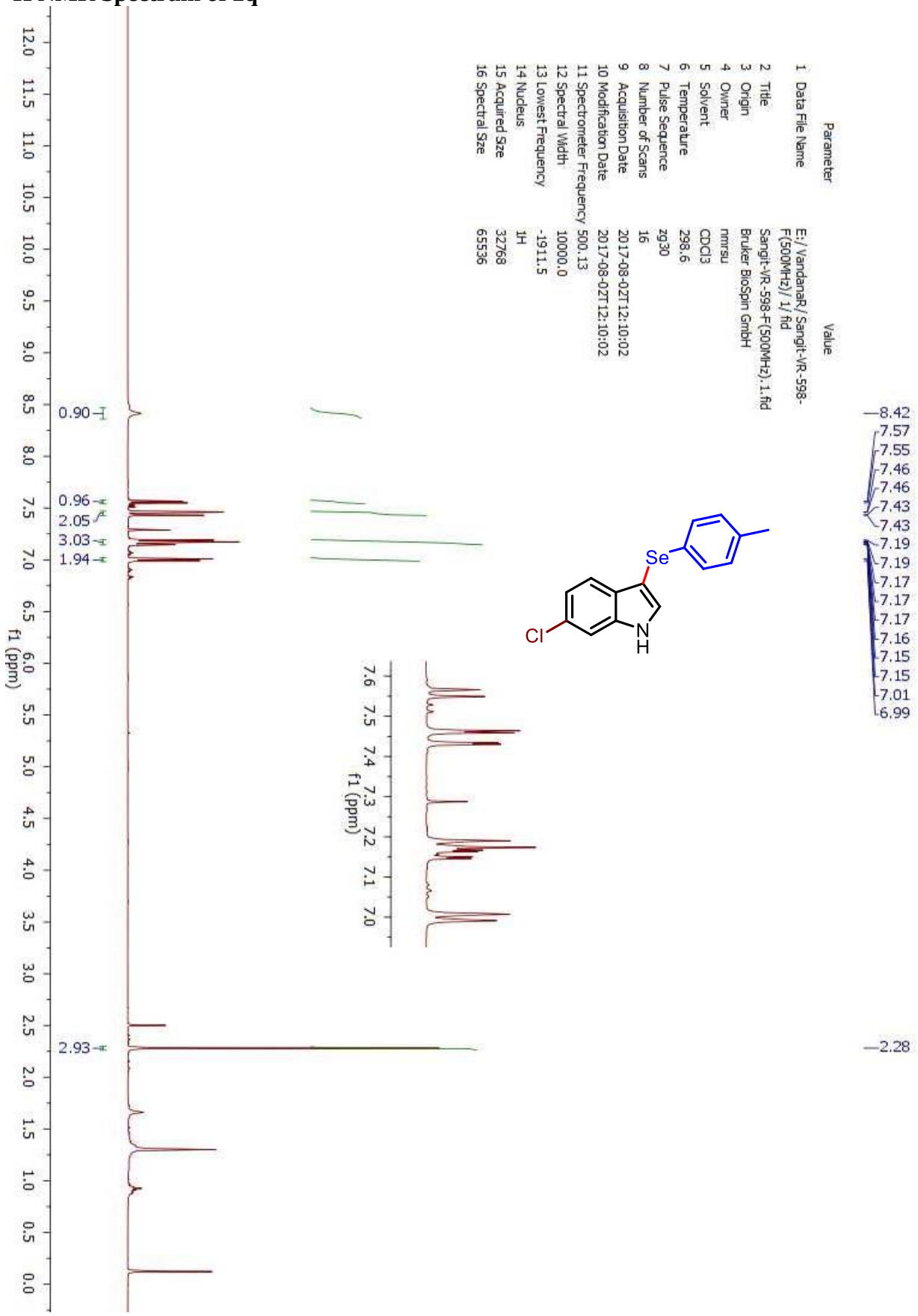
^{77}Se NMR Spectrum of 2p



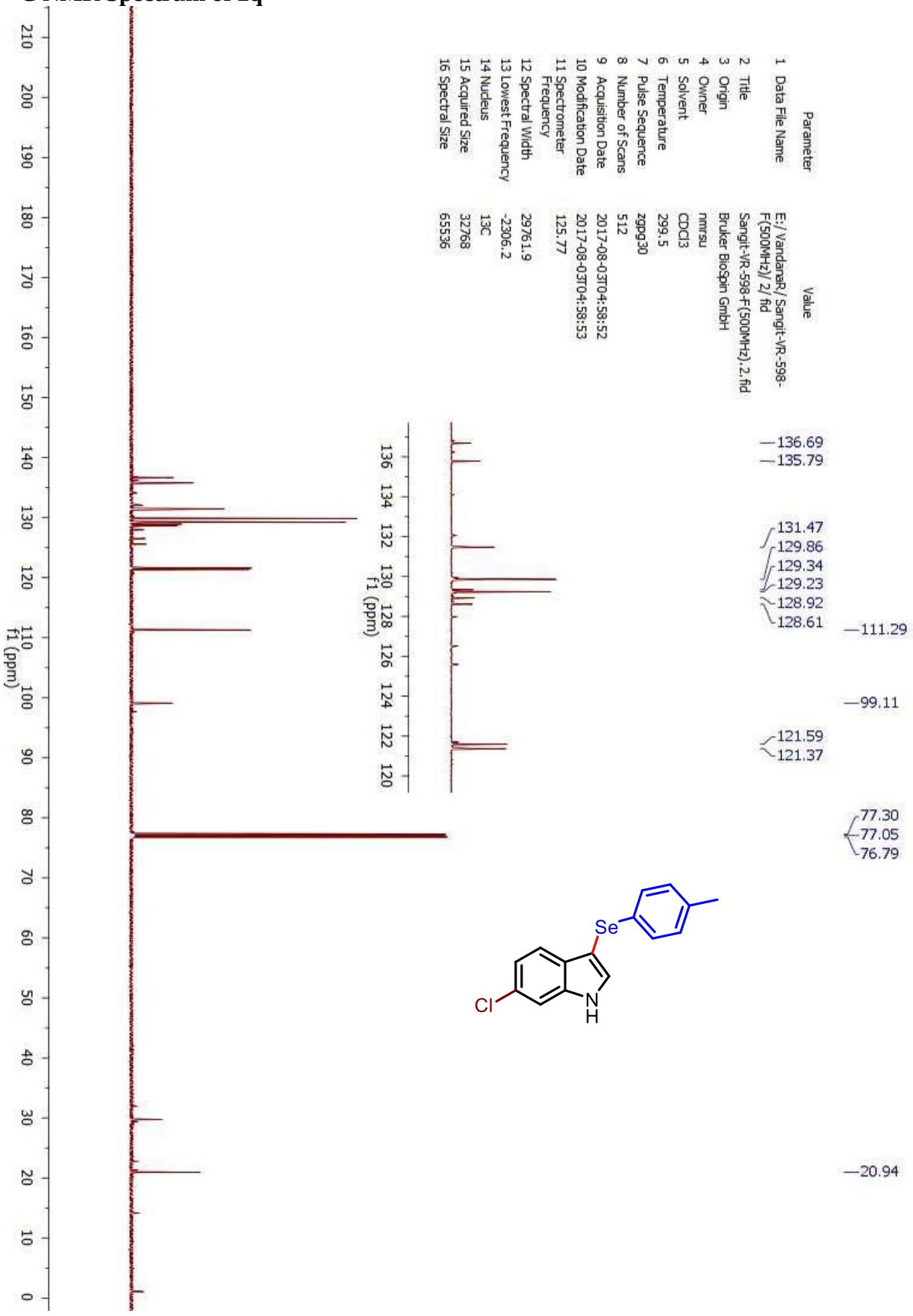
Mass Spectrum of 2p



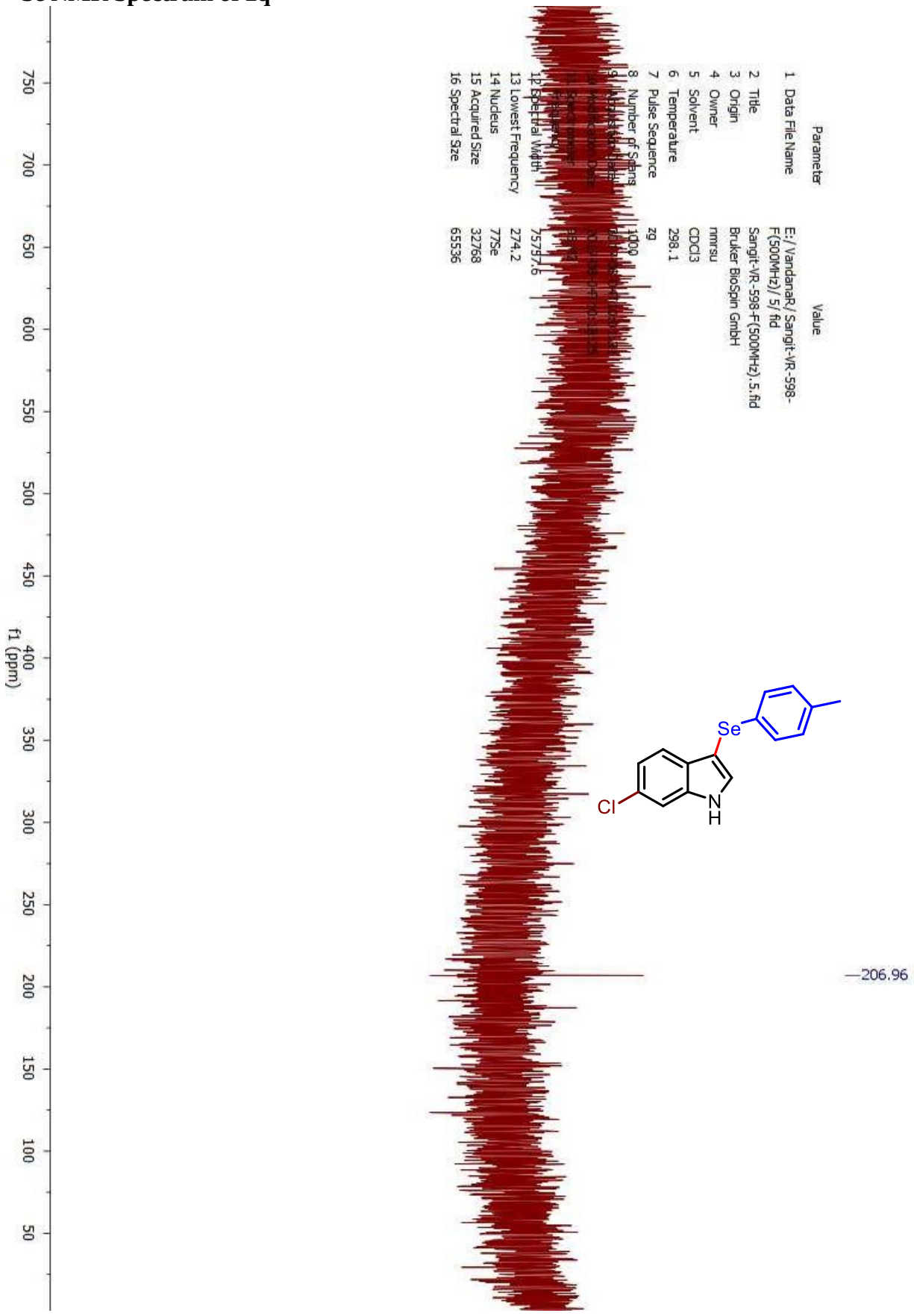
^{1}H NMR Spectrum of 2q



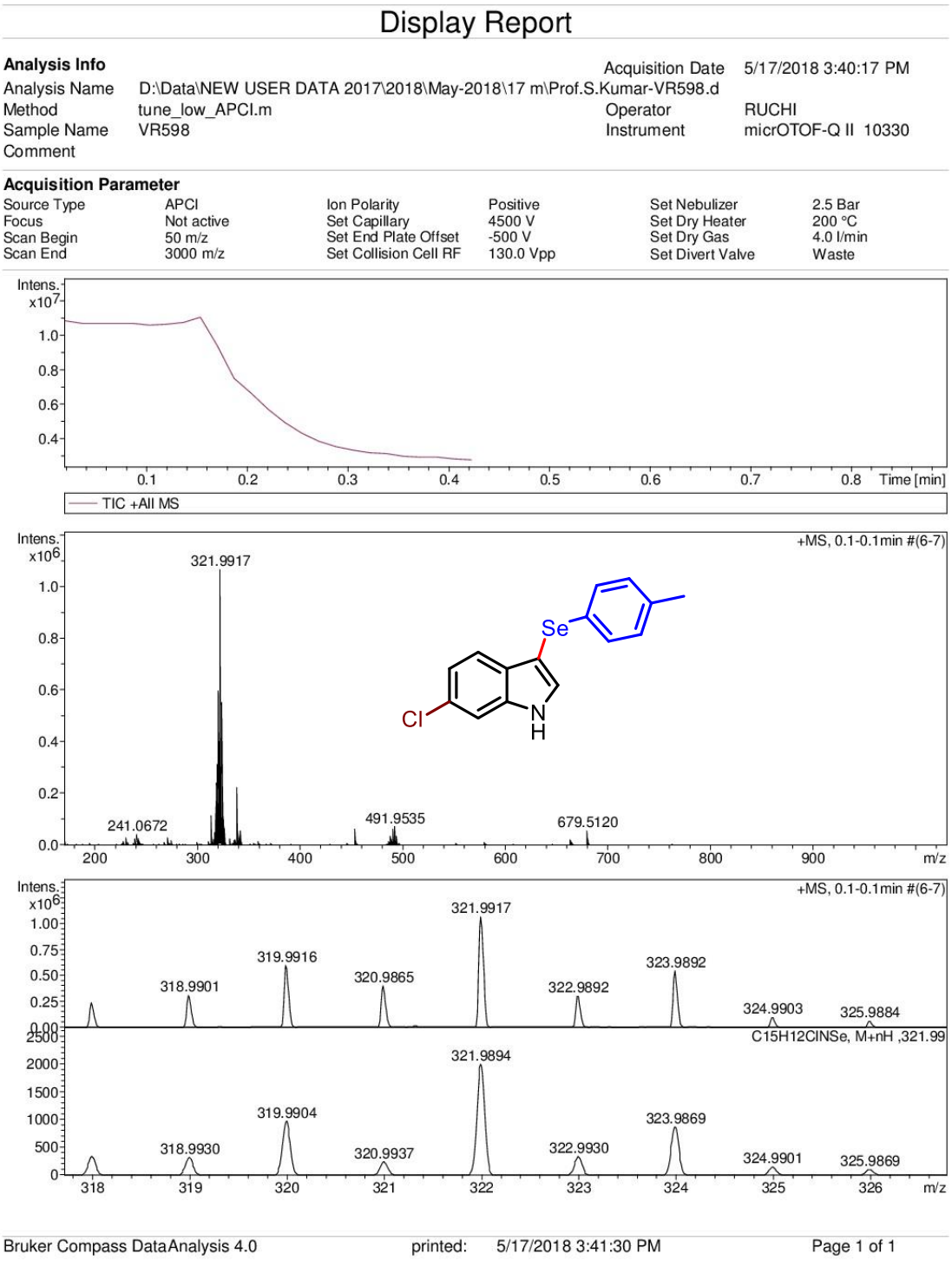
^{13}C NMR Spectrum of 2q



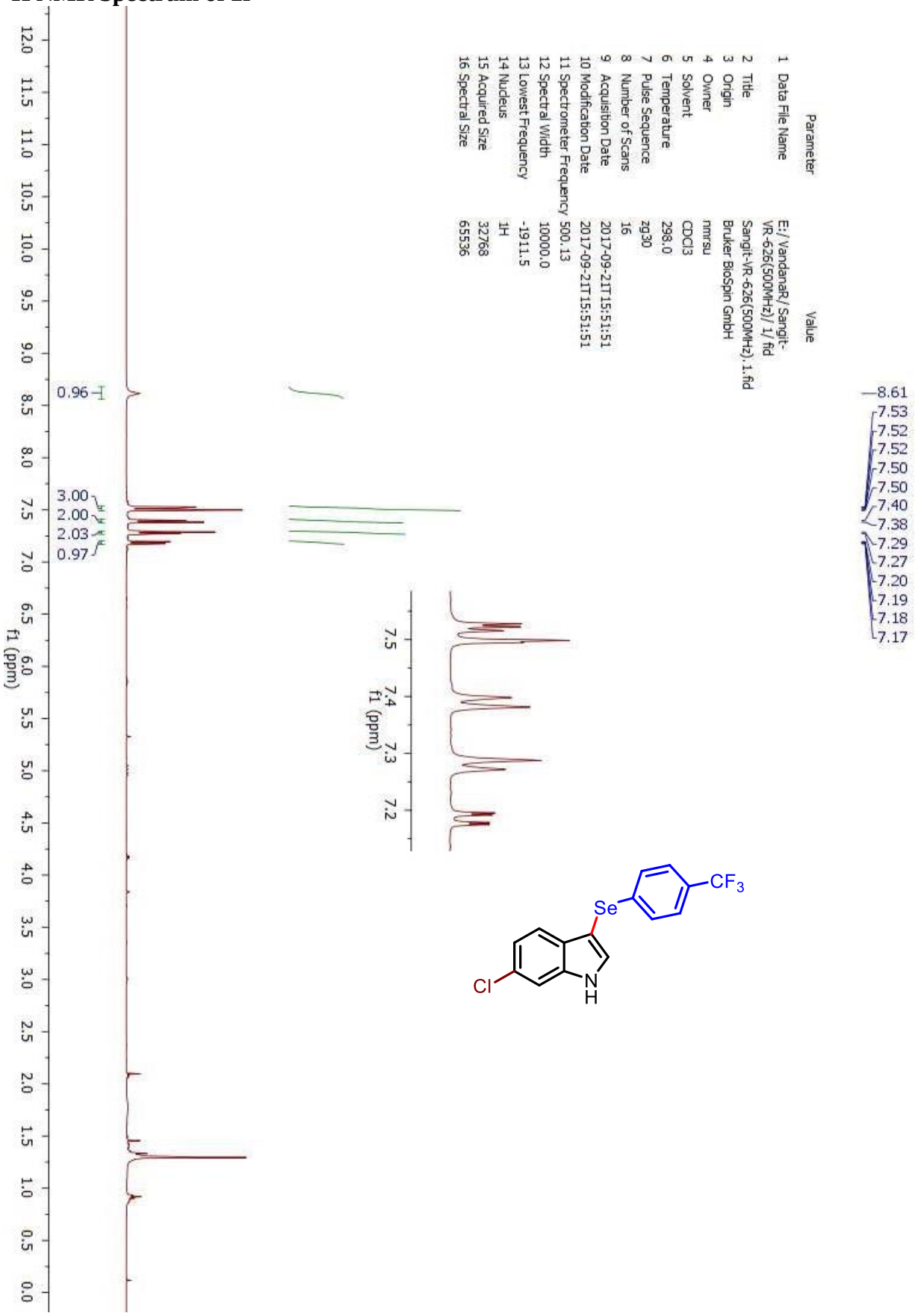
^{77}Se NMR Spectrum of 2q



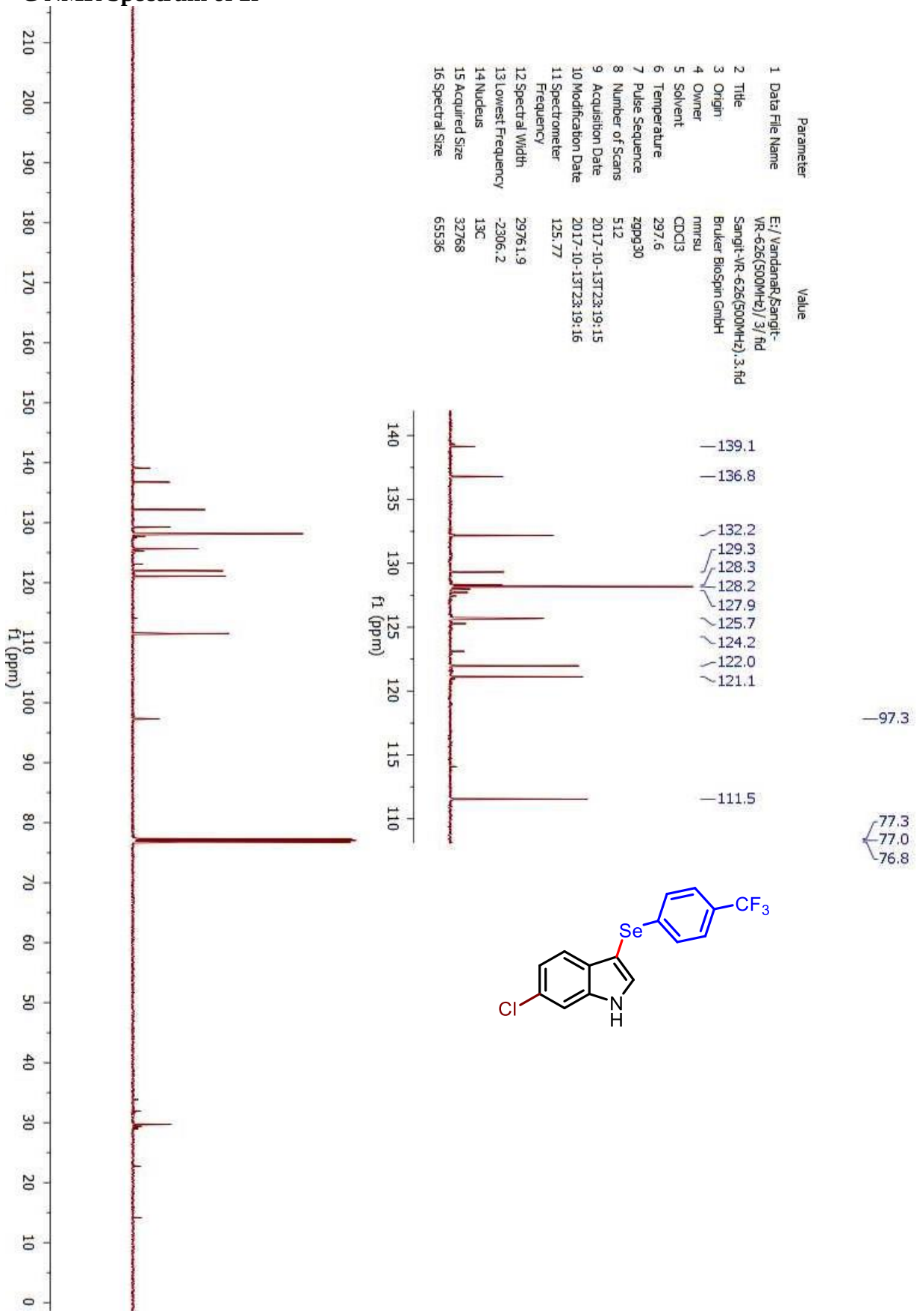
Mass Spectrum of 2q



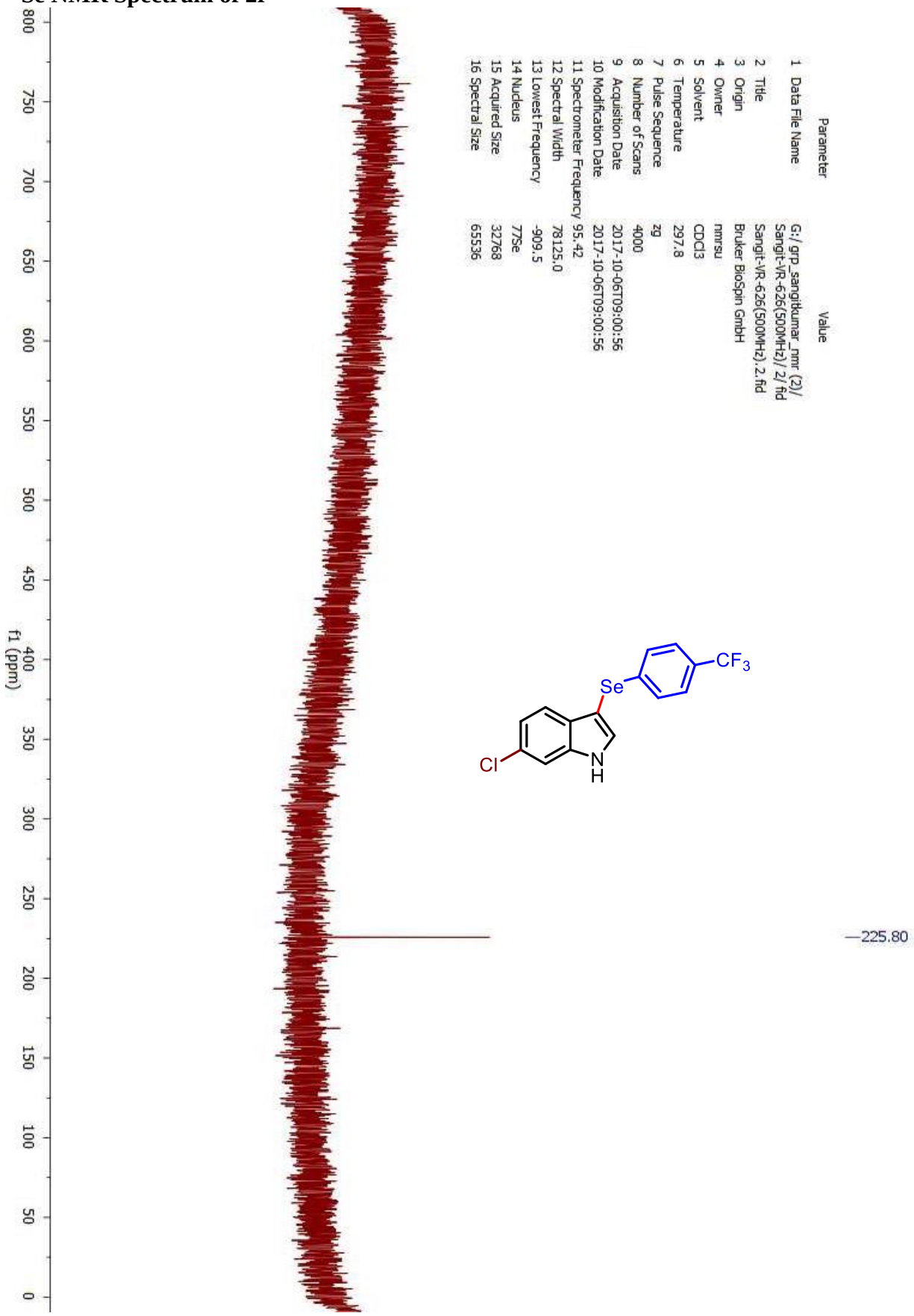
^{1}H NMR Spectrum of 2r



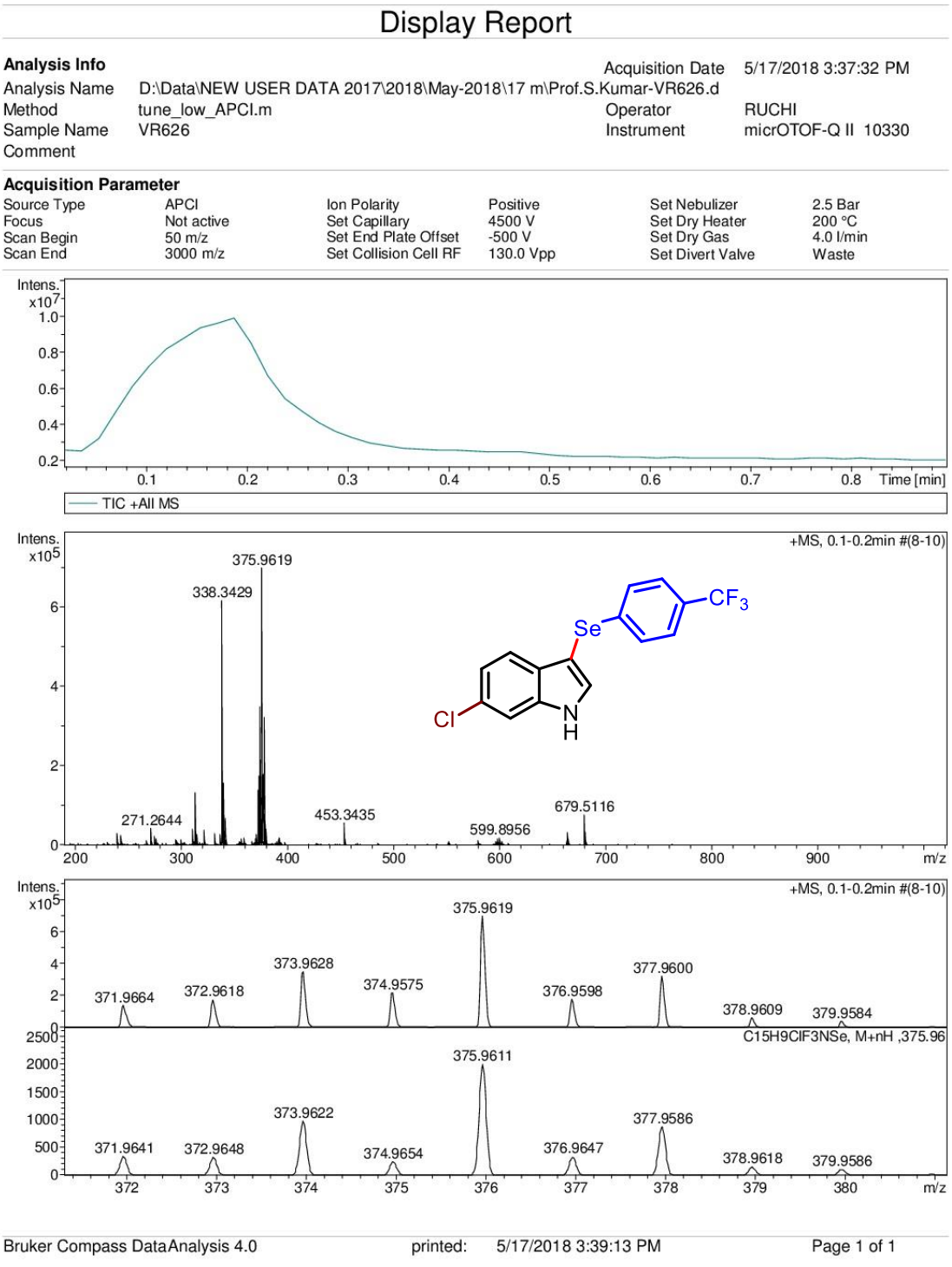
^{13}C NMR Spectrum of 2r



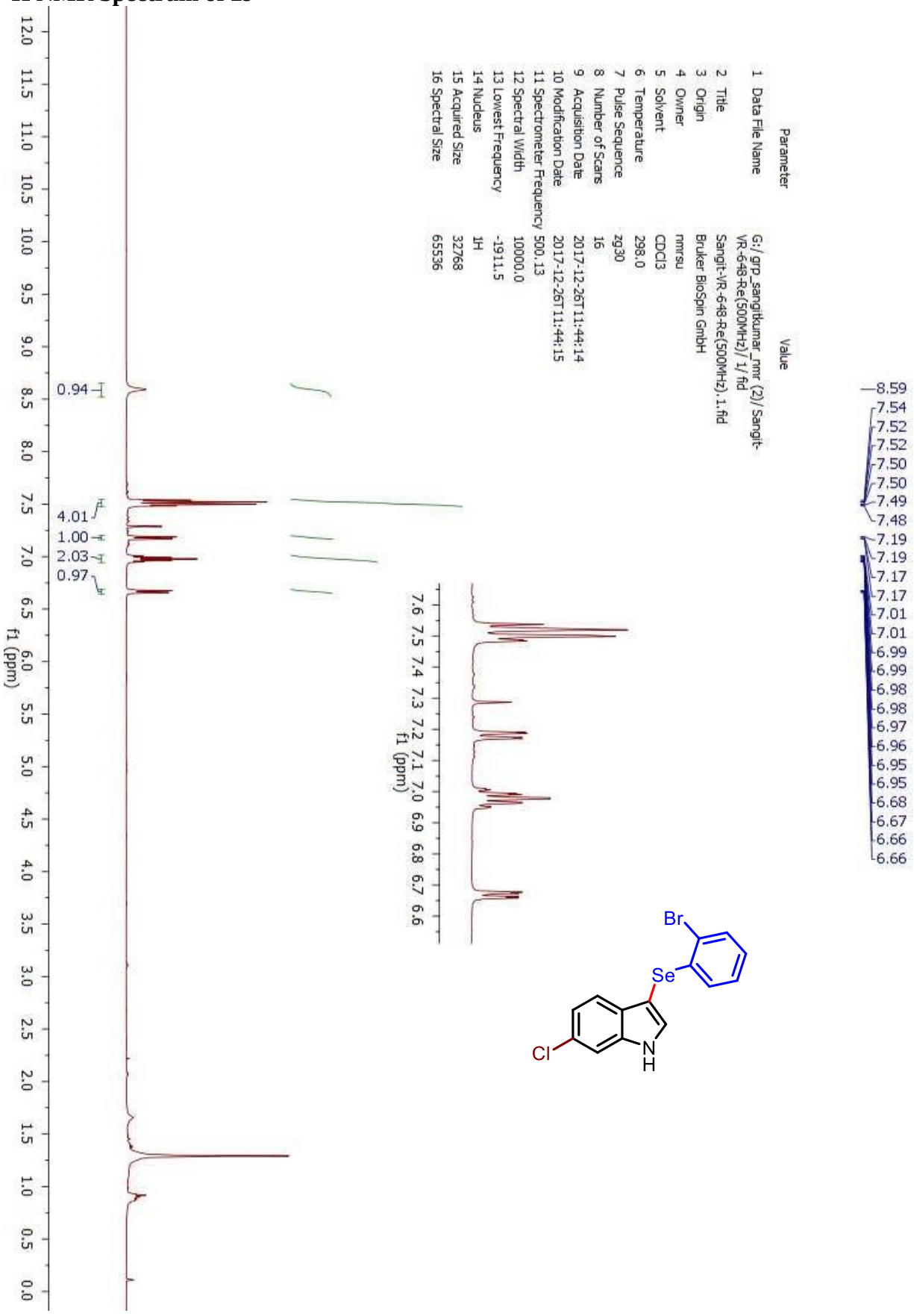
^{77}Se NMR Spectrum of 2r



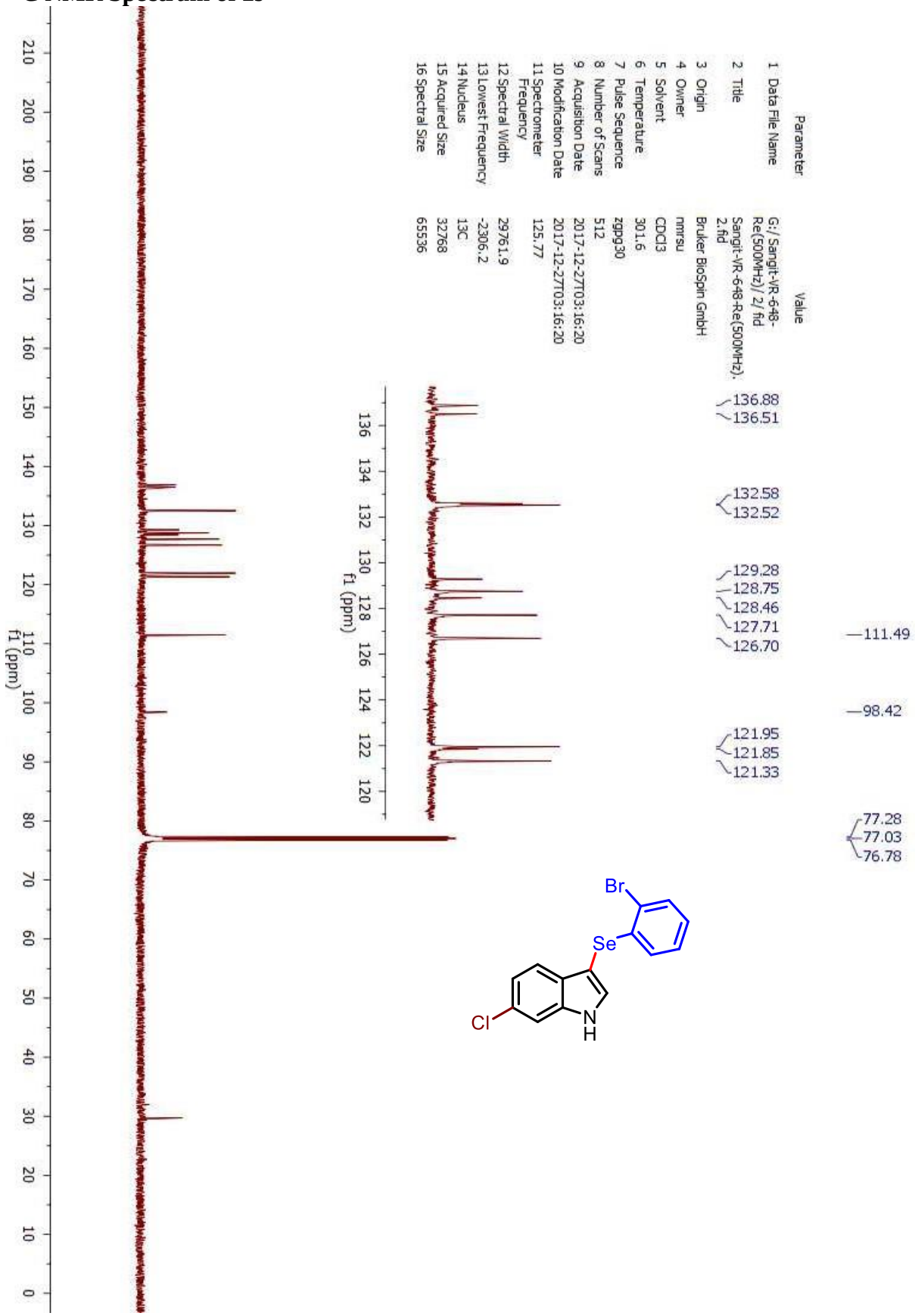
Mass Spectrum of 2r



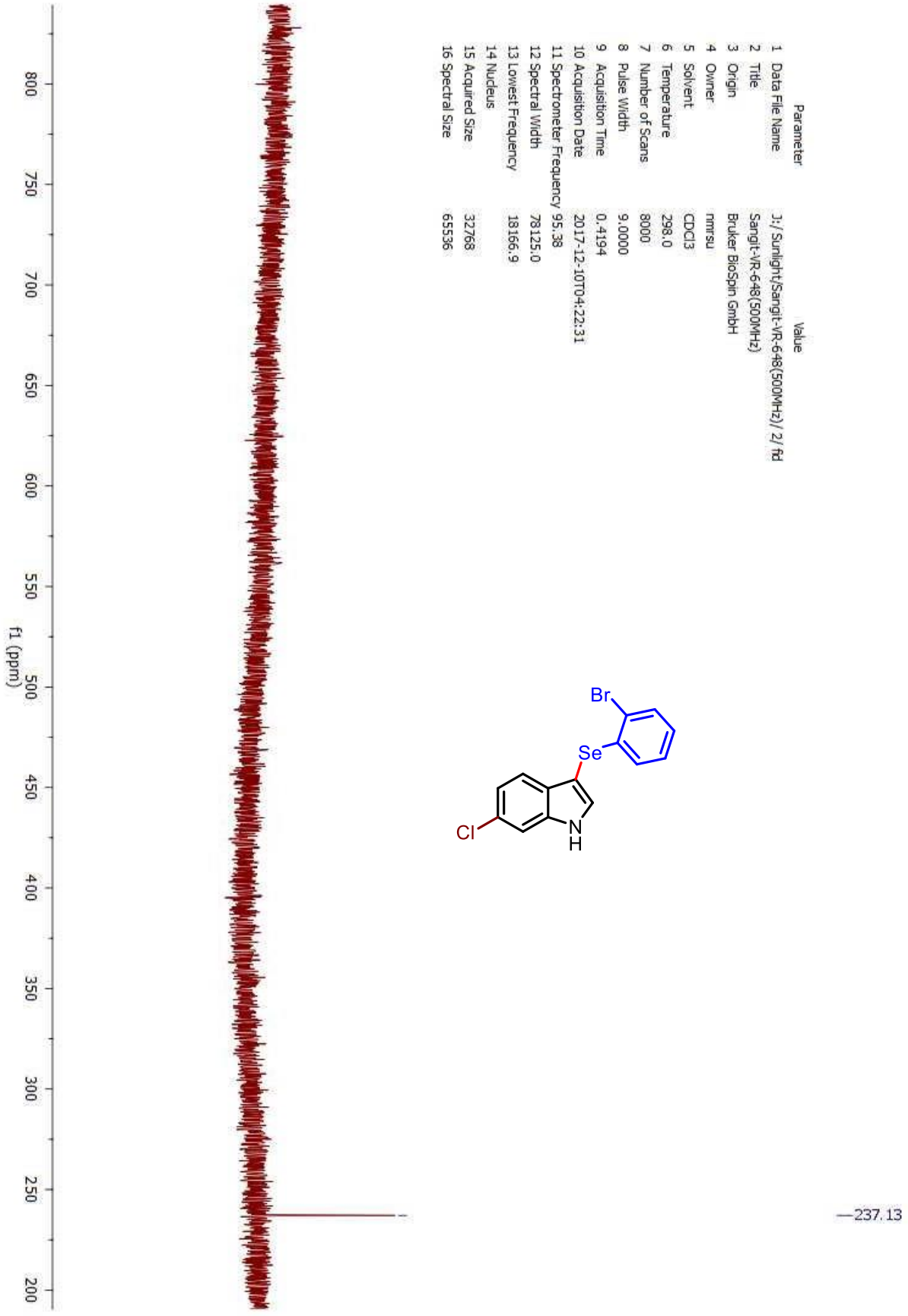
^{1}H NMR Spectrum of 2s



^{13}C NMR Spectrum of 2s



^{77}Se NMR Spectrum of 2s



	Parameter	Value
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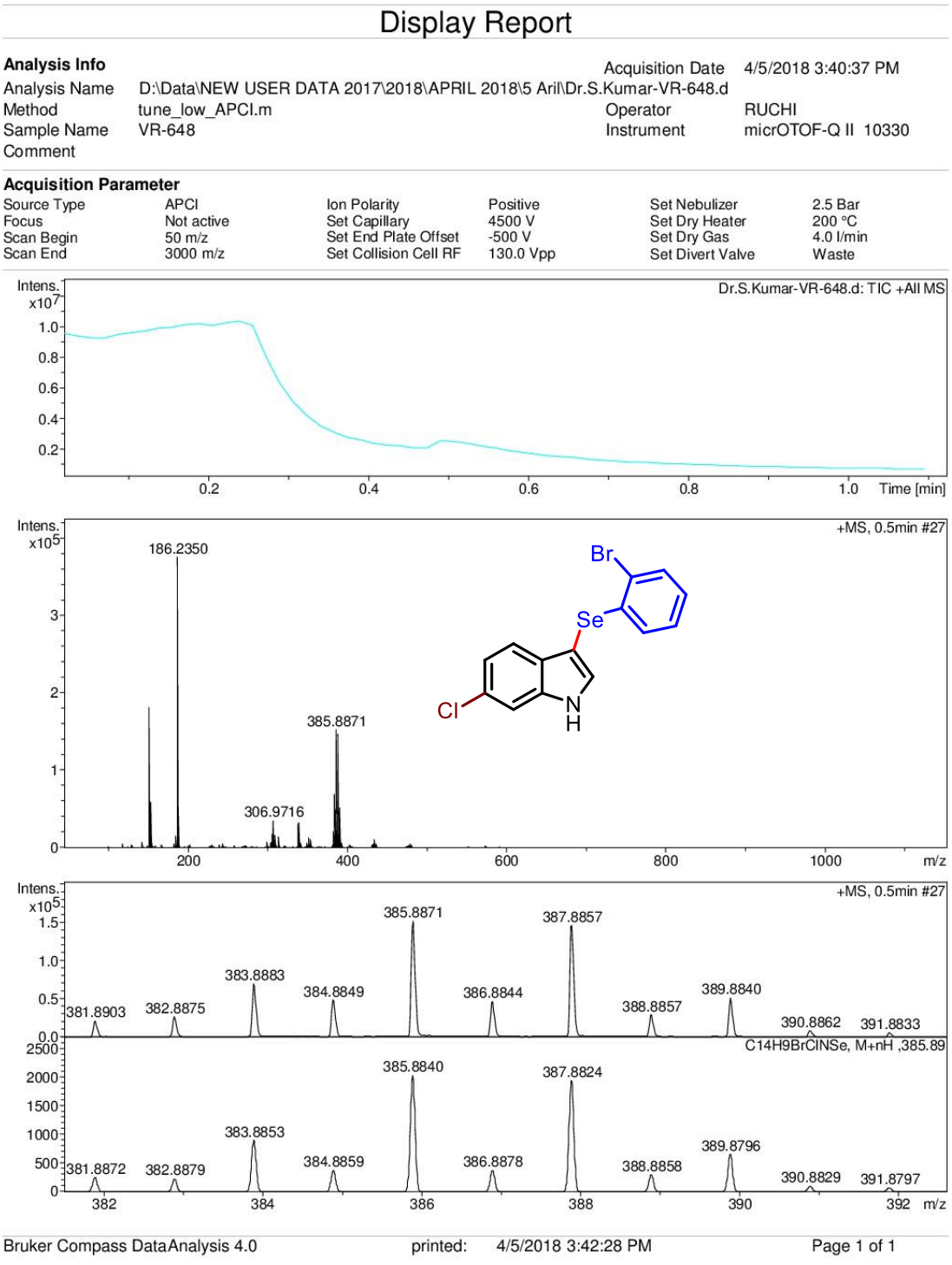
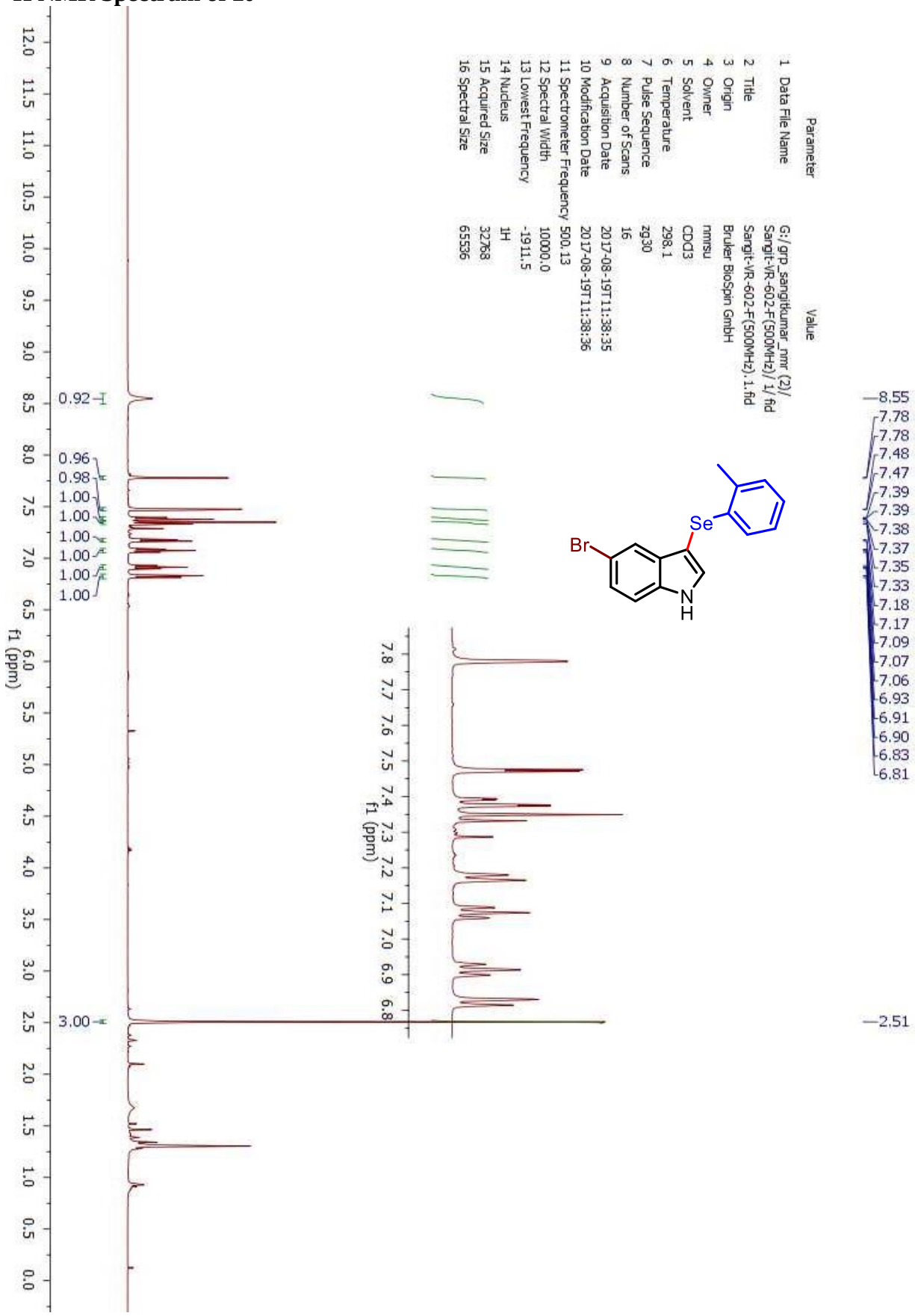
4	Owner	nmrsu
5	Solvent	CDCl3
6	Temperature	298.0
7	Number of Scans	8000
8	Pulse Width	9.0000
9	Acquisition Time	0.4194
10	Acquisition Date	2017-12-10T04:22:31
11	Spectrometer Frequency	95.38
12	Spectral Width	78125.0
13	Lowest Frequency	18166.9
14	Nucleus	
15	Acquired Size	32768
16	Spectral Size	65536

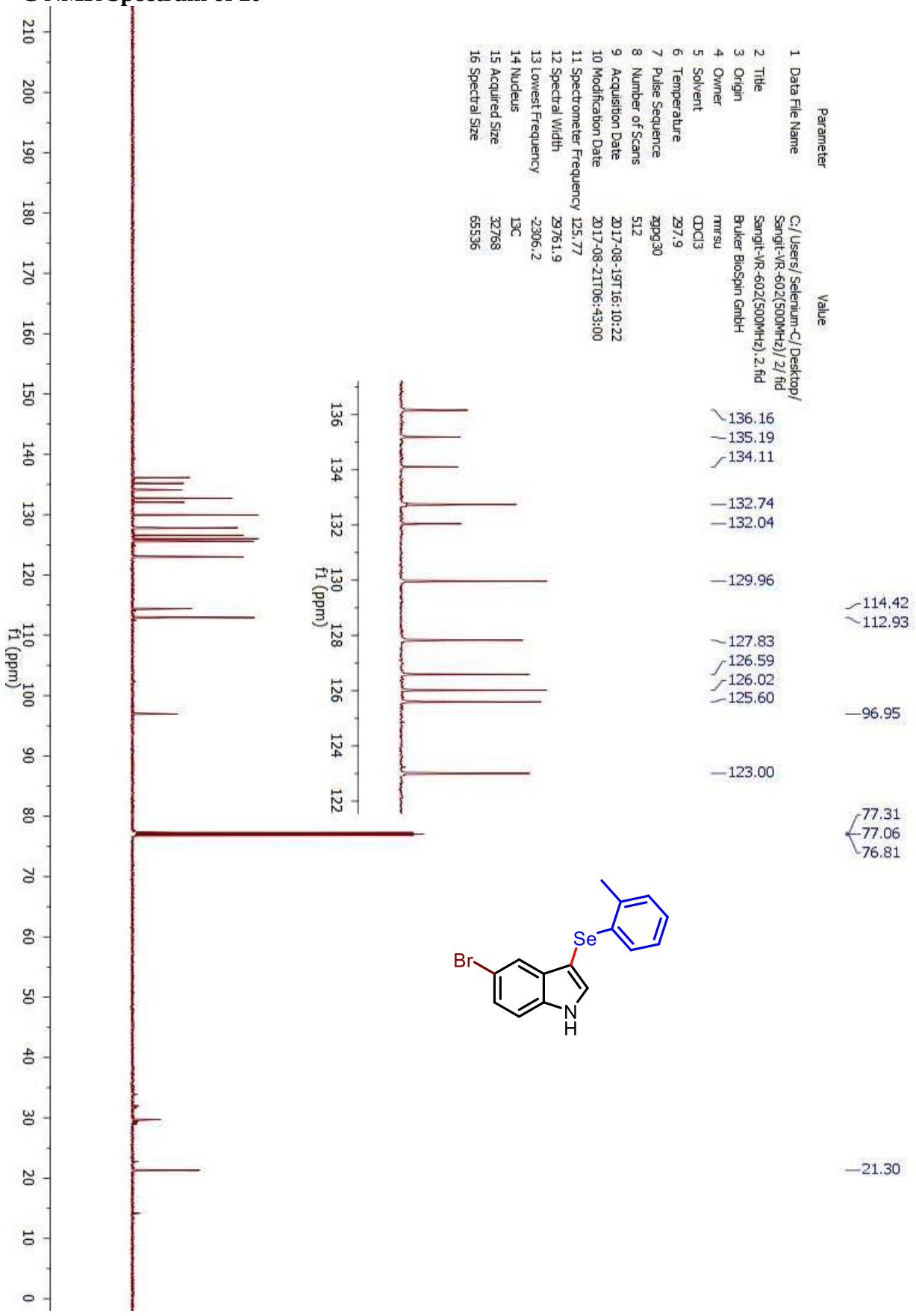
Mass Spectrum of 2s



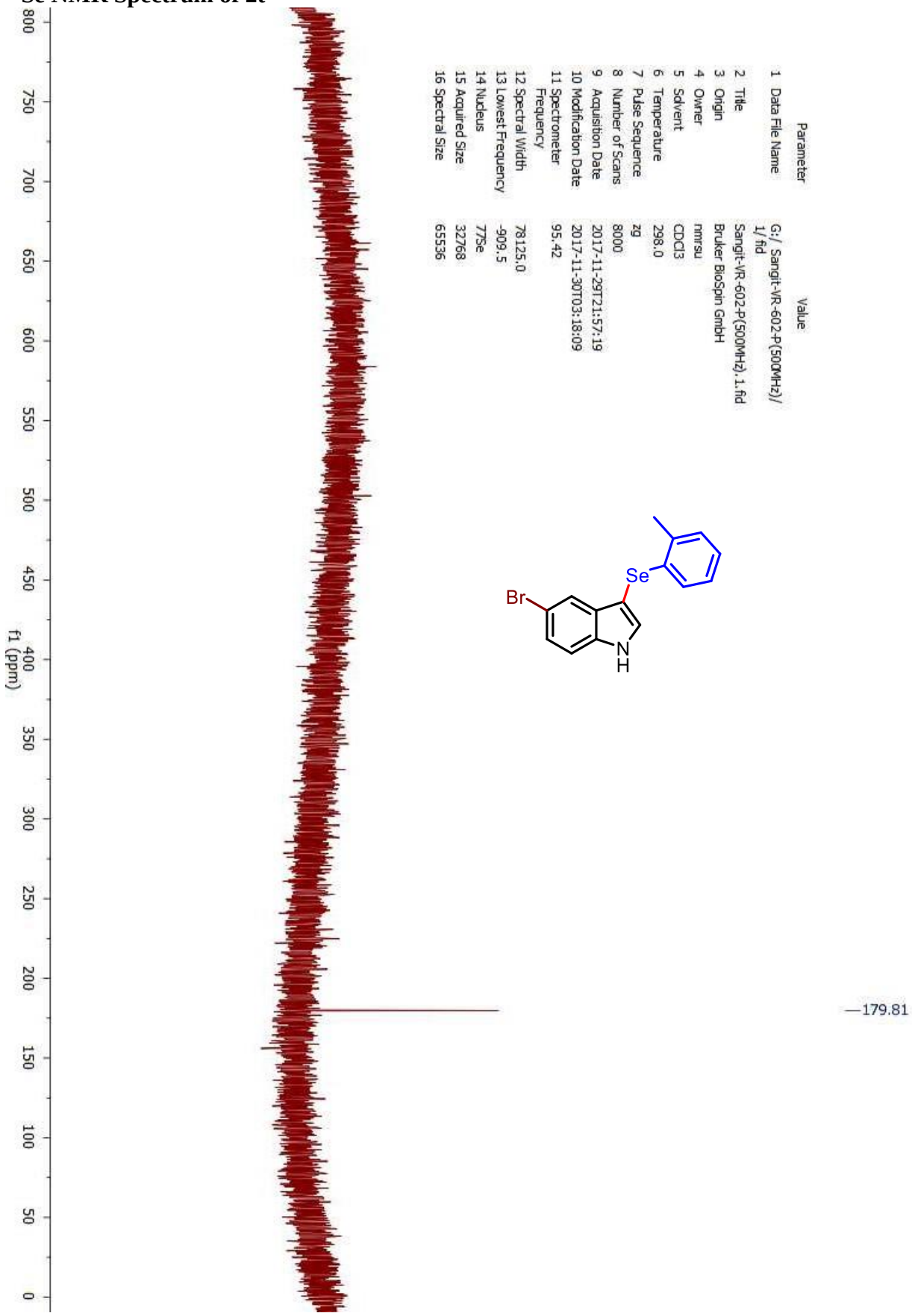
^{1}H NMR Spectrum of 2t



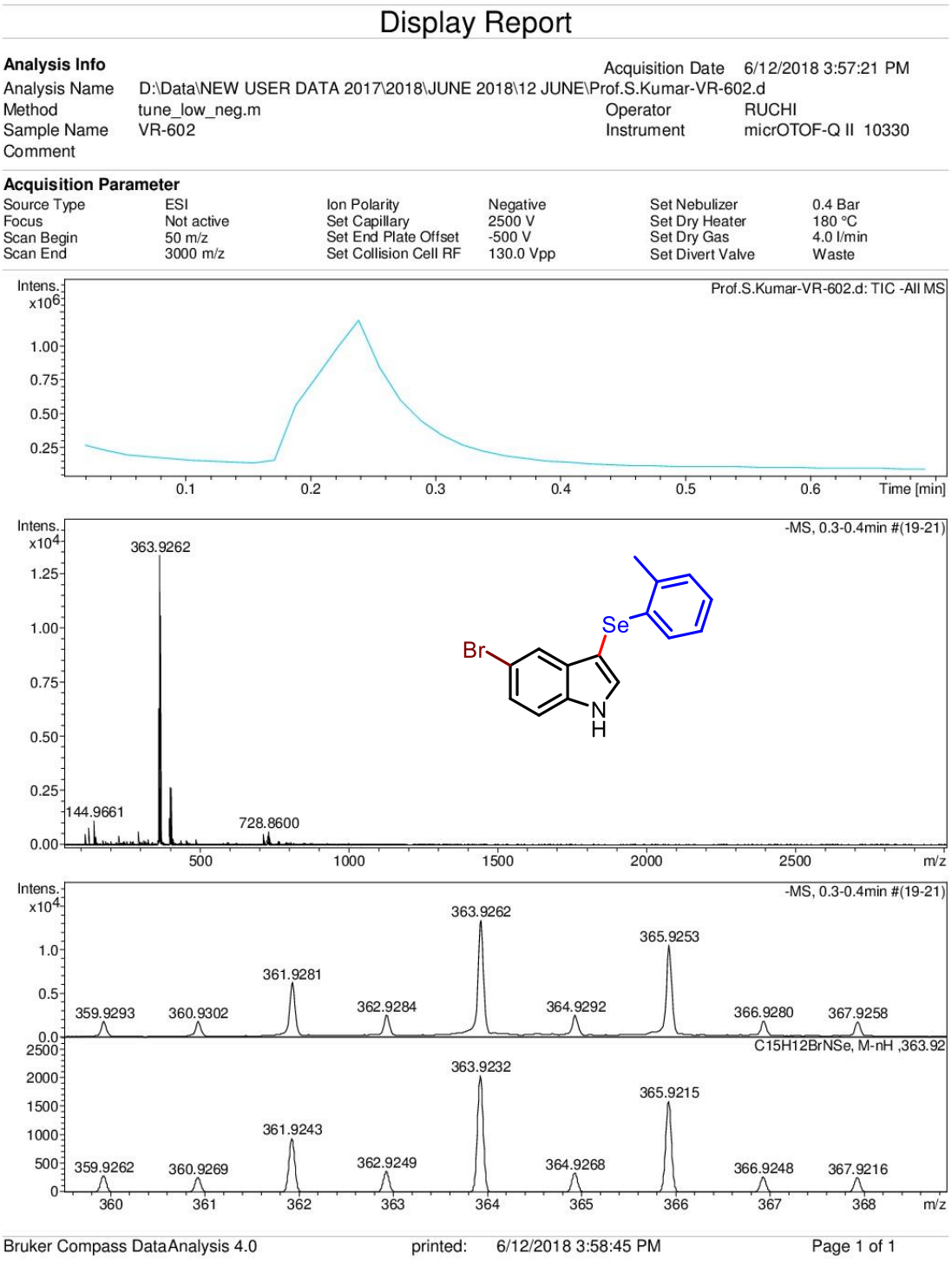
^{13}C NMR Spectrum of 2t



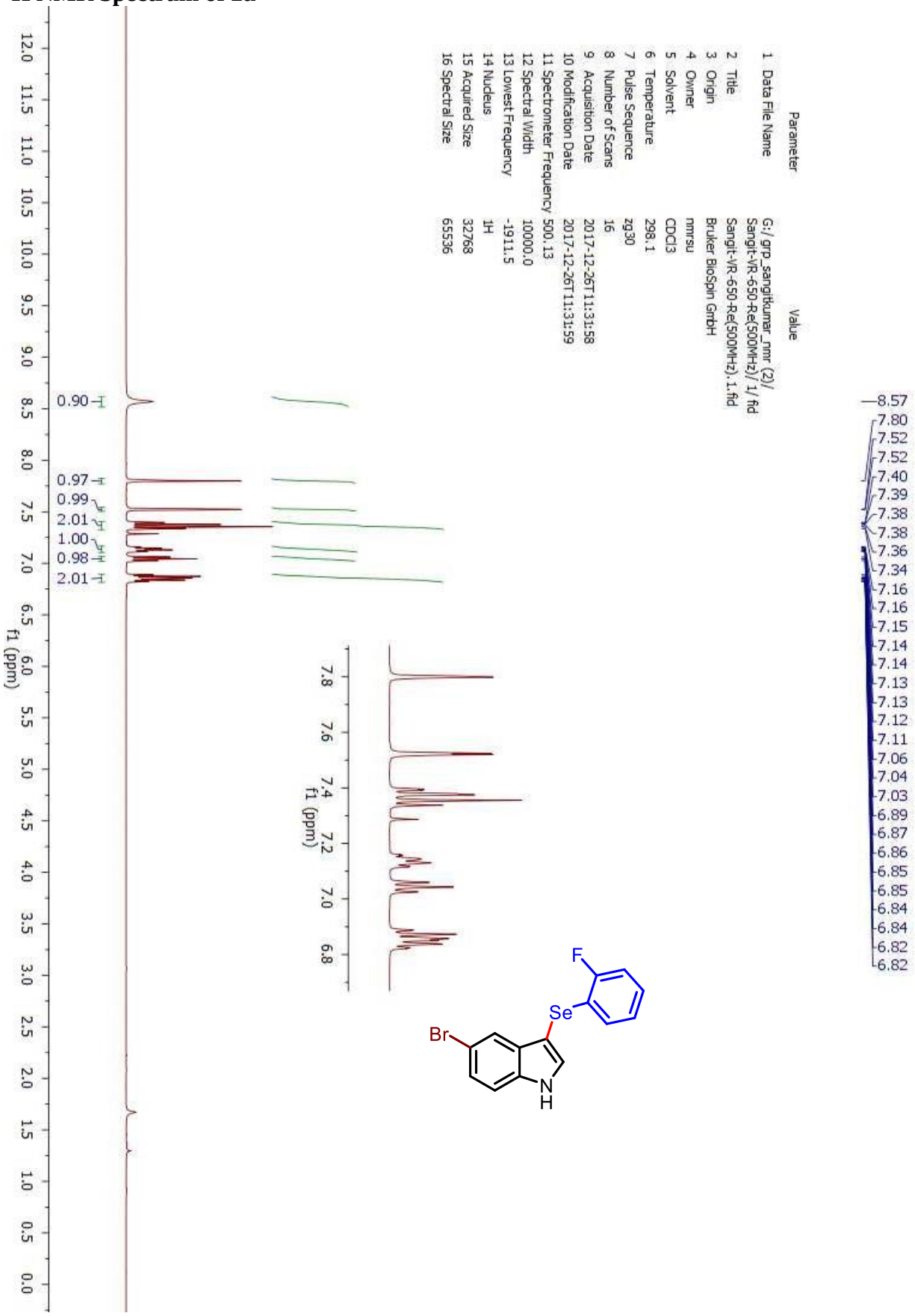
^{77}Se NMR Spectrum of 2t



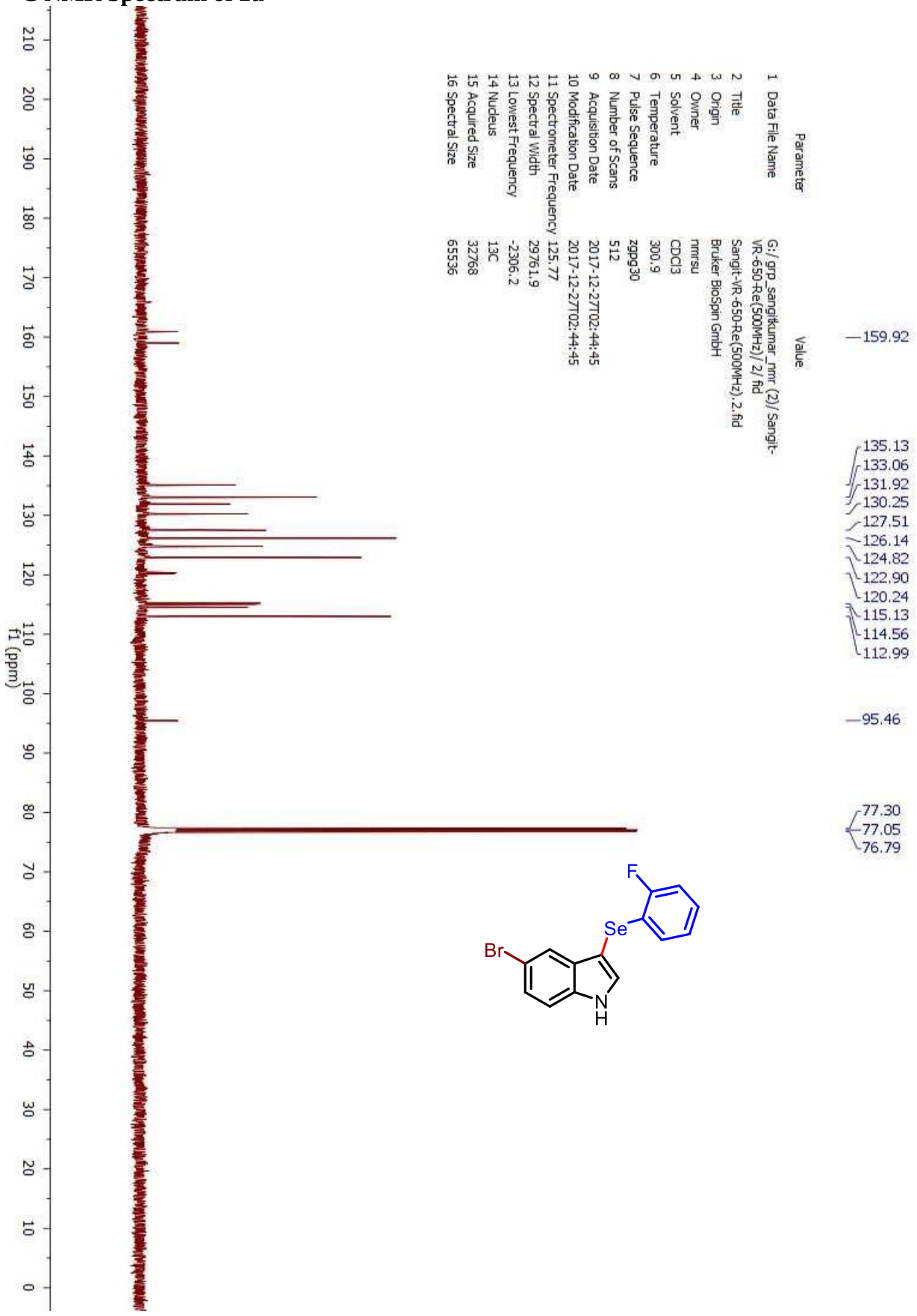
Mass Spectrum of 2t



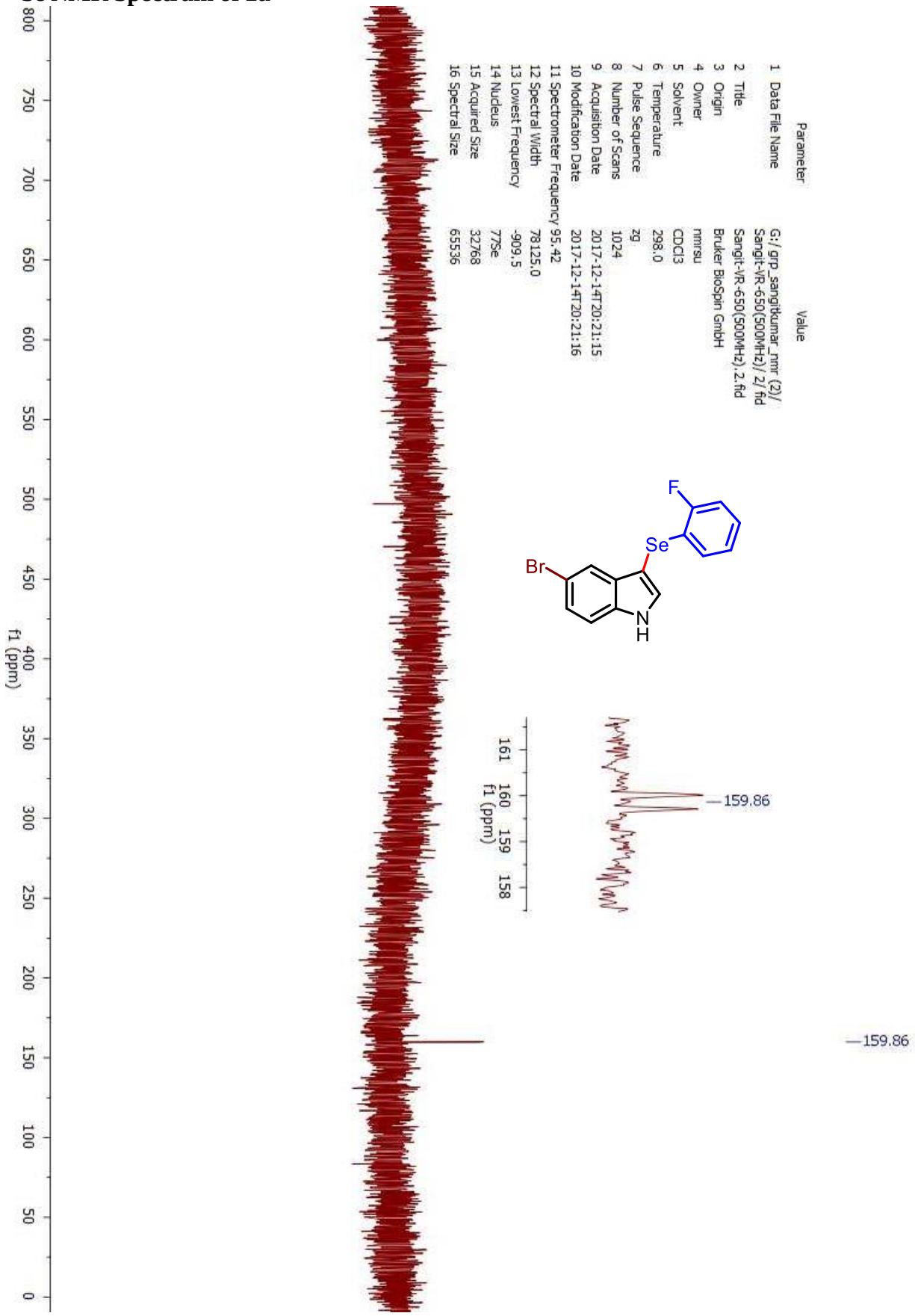
^{1}H NMR Spectrum of 2u



^{13}C NMR Spectrum of 2u



^{77}Se NMR Spectrum of 2u



Mass Spectrum of 2u

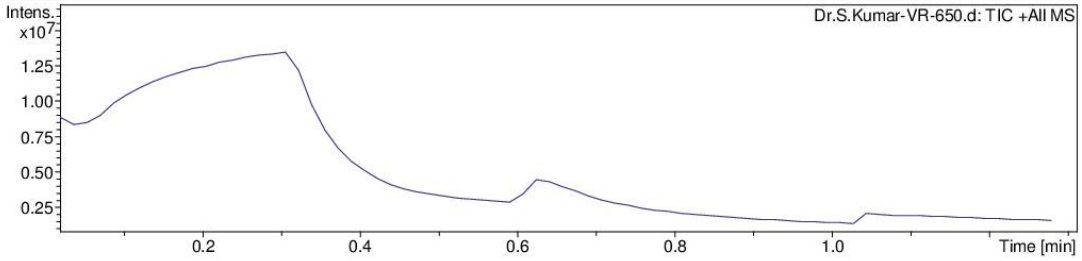
Display Report

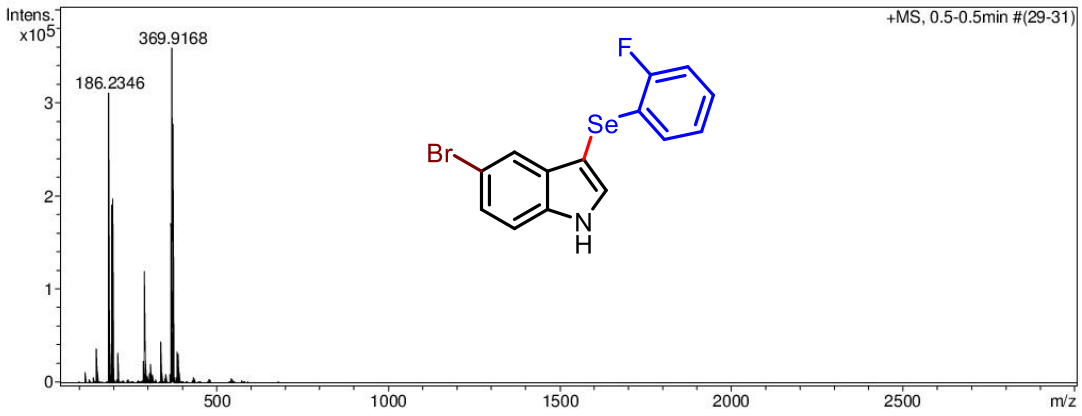
Analysis Info

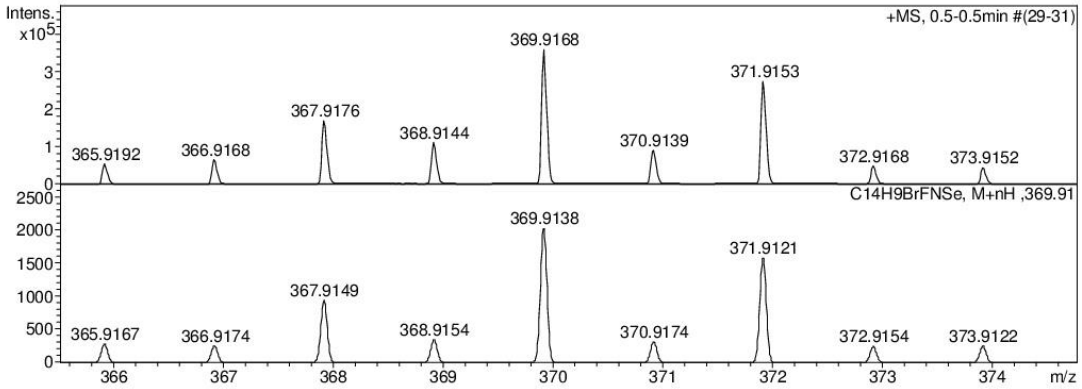
Analysis Name	D:\Data\NEW USER DATA 2017\2018\APRIL 2018\5 Aril\Dr.S.Kumar-VR-650.d	Acquisition Date	4/5/2018 3:43:27 PM
Method	tune_low_APCI.m	Operator	RUCHI
Sample Name	VR-650	Instrument	micrOTOF-Q II 10330
Comment			

Acquisition Parameter

Source Type	APCI	Ion Polarity	Positive	Set Nebulizer	2.5 Bar
Focus	Not active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste







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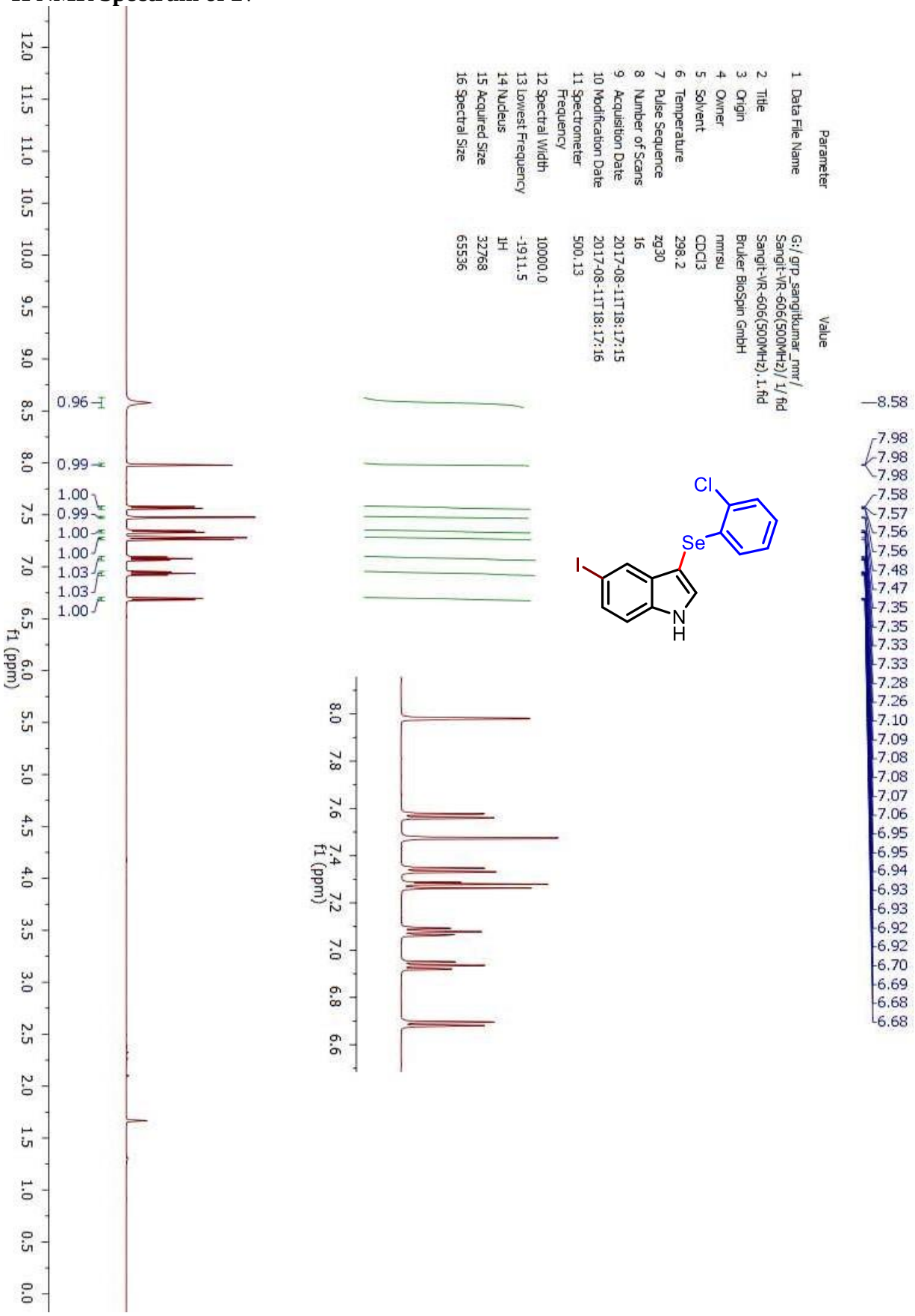
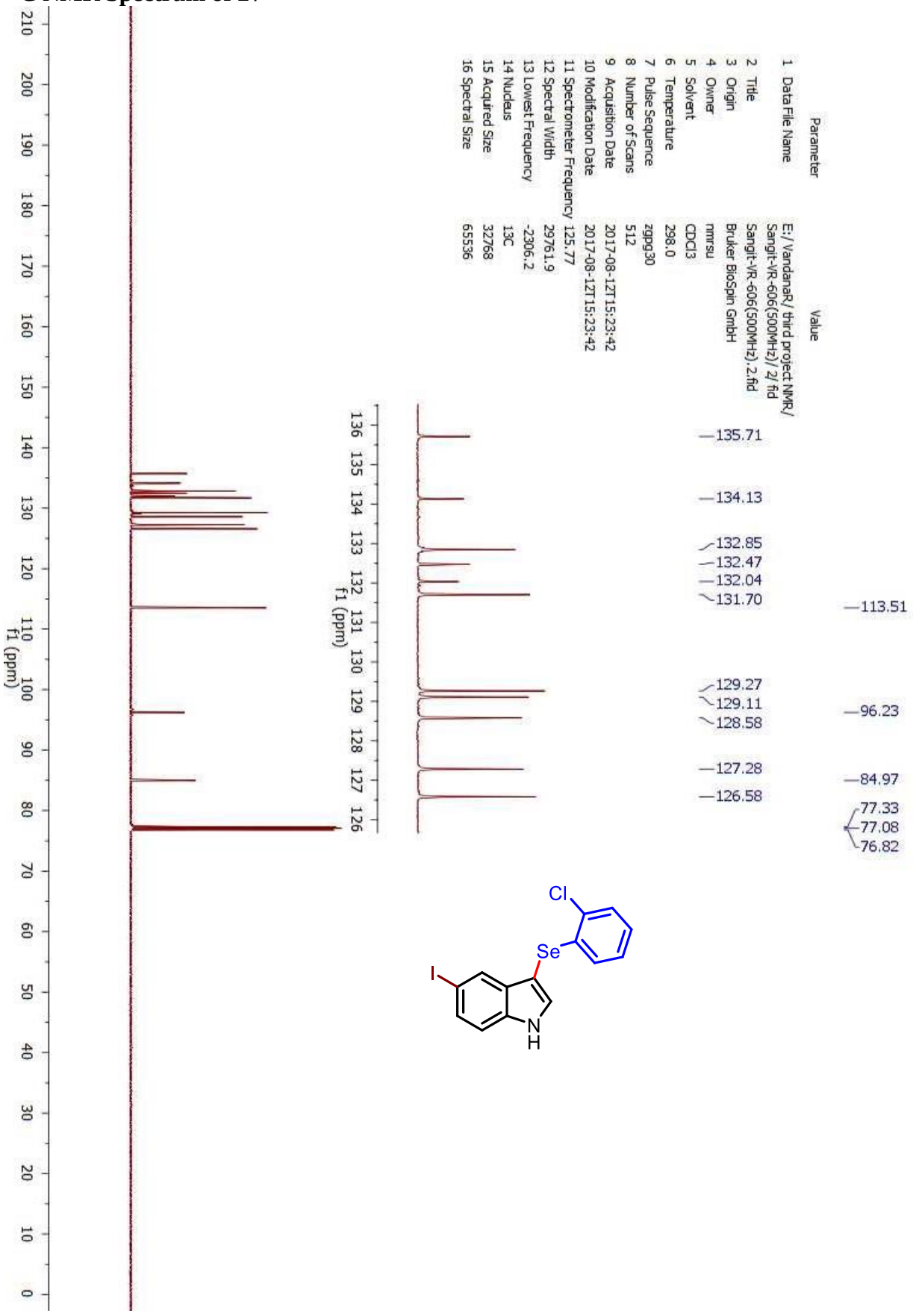
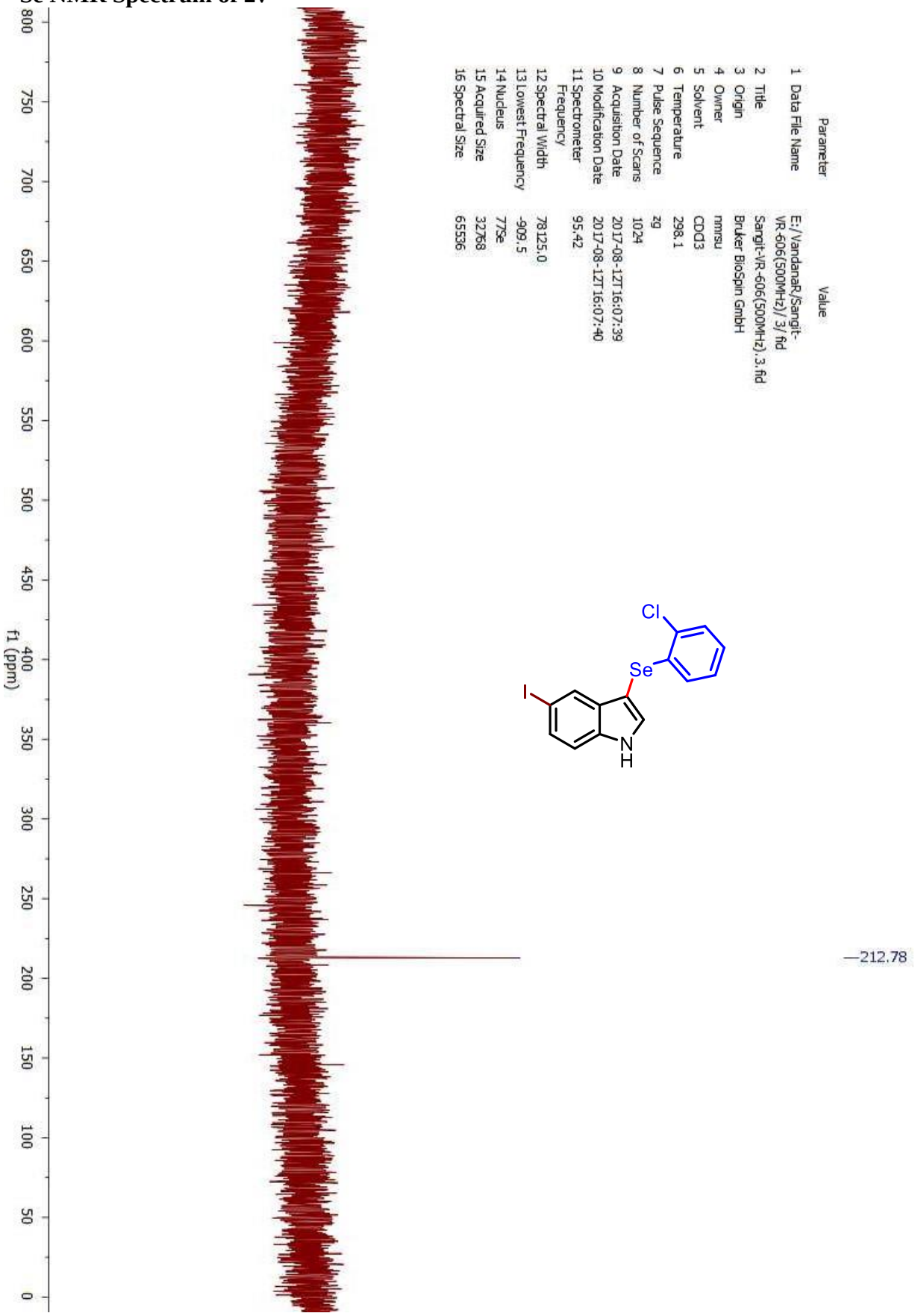
^{1}H NMR Spectrum of 2v



^{13}C NMR Spectrum of 2v



^{77}Se NMR Spectrum of 2v



Mass Spectrum of 2v

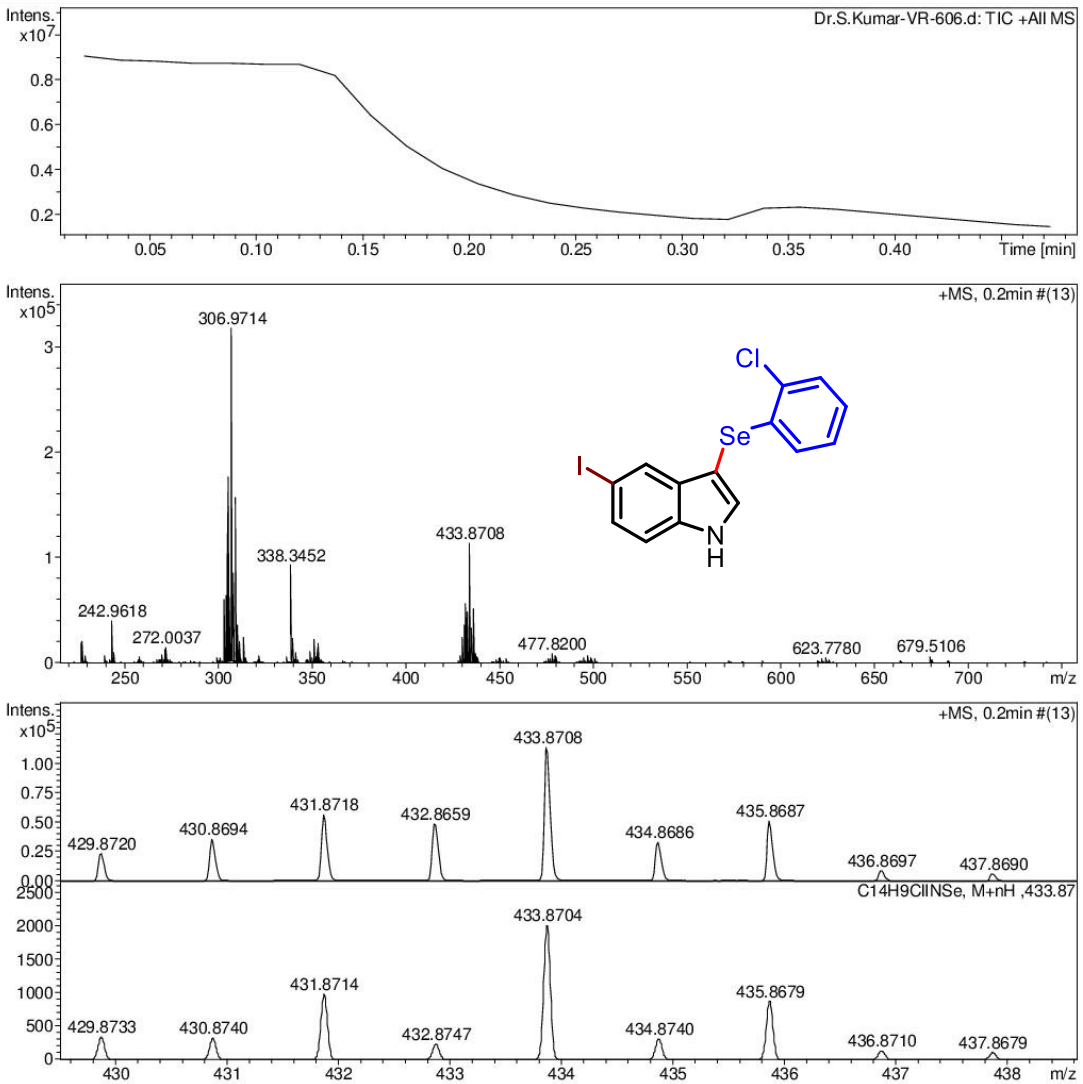
Display Report

Analysis Info

Analysis Name	D:\Data\NEW USER DATA 2017\2018\APRIL 2018\5 Aril\Dr.S.Kumar-VR-606.d	Acquisition Date	4/5/2018 3:38:30 PM
Method	tune_low_APCI.m	Operator	RUCHI
Sample Name	VR-606	Instrument	micrOTOF-Q II 10330
Comment			

Acquisition Parameter

Source Type	APCI	Ion Polarity	Positive	Set Nebulizer	2.5 Bar
Focus	Not active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste



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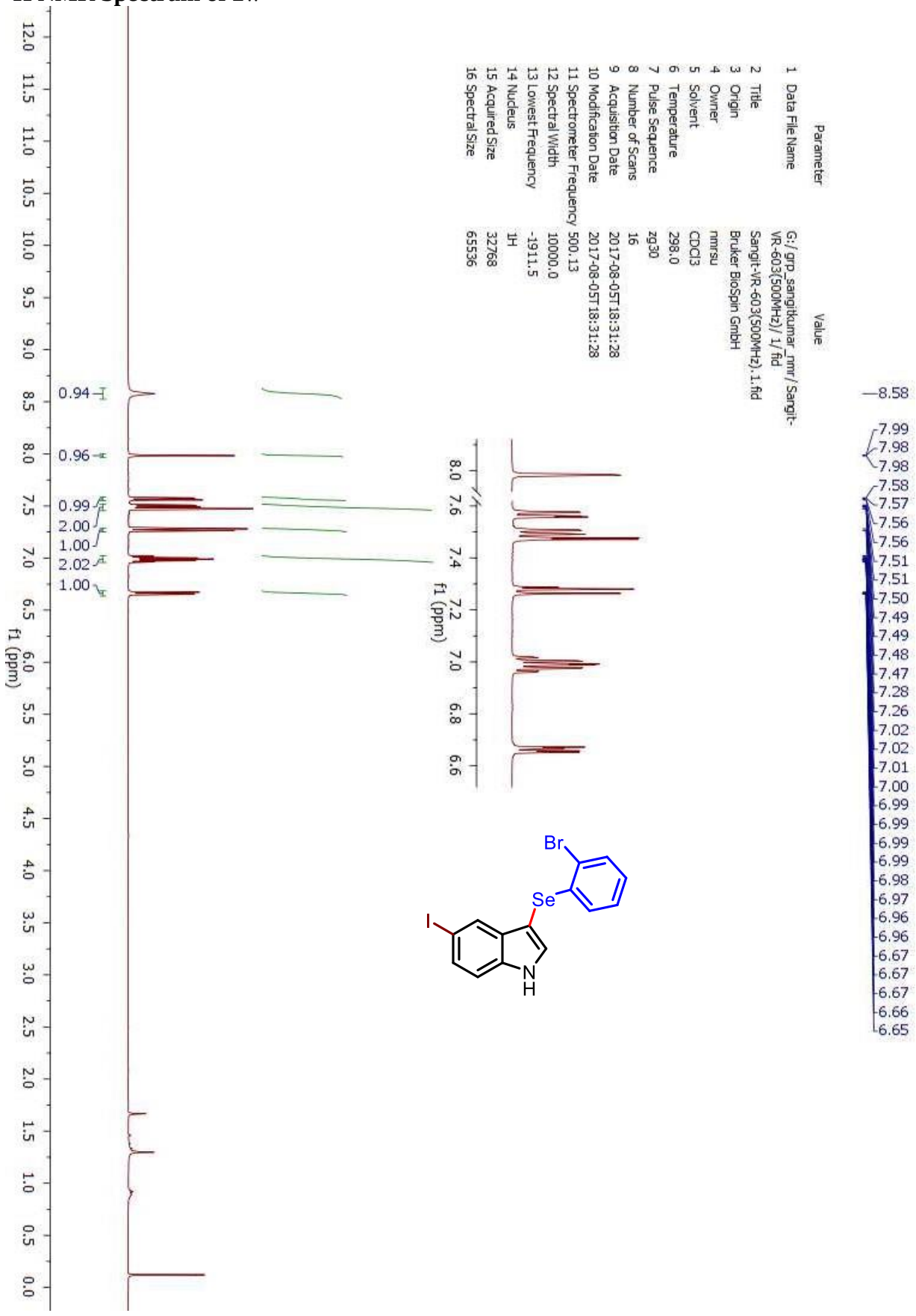
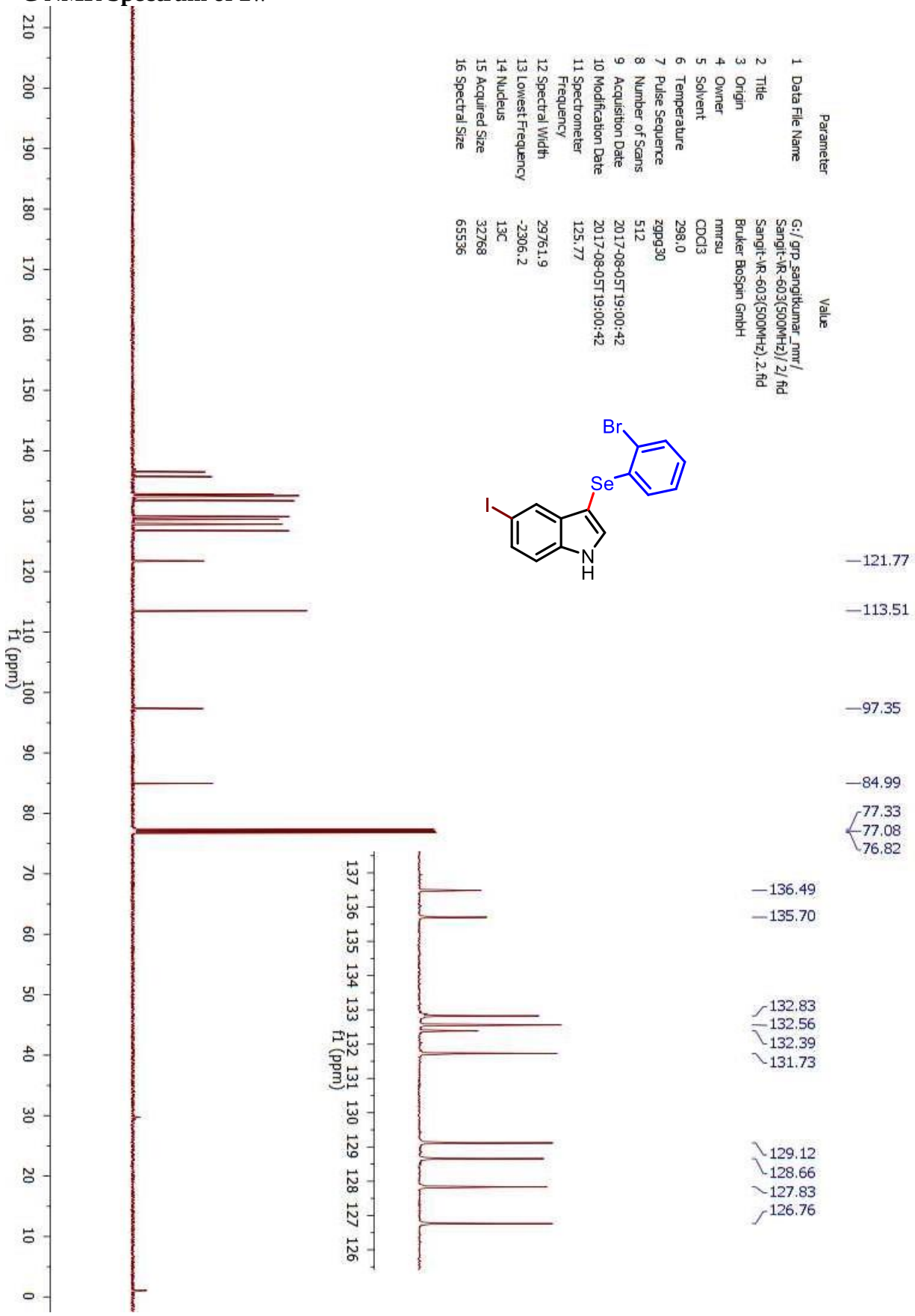
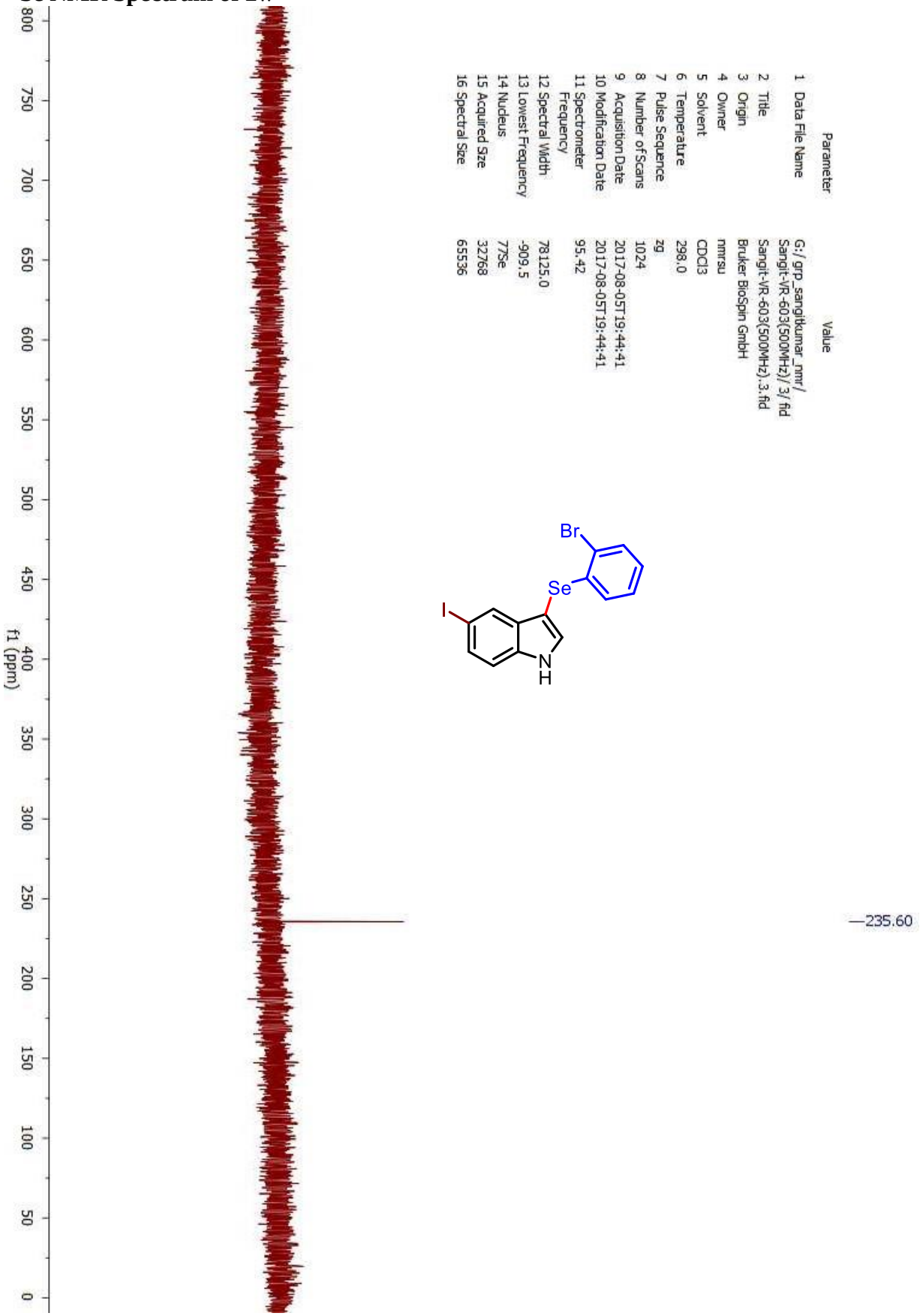
^{1}H NMR Spectrum of 2w



^{13}C NMR Spectrum of 2w



^{77}Se NMR Spectrum of 2w



Mass Spectrum of 2w

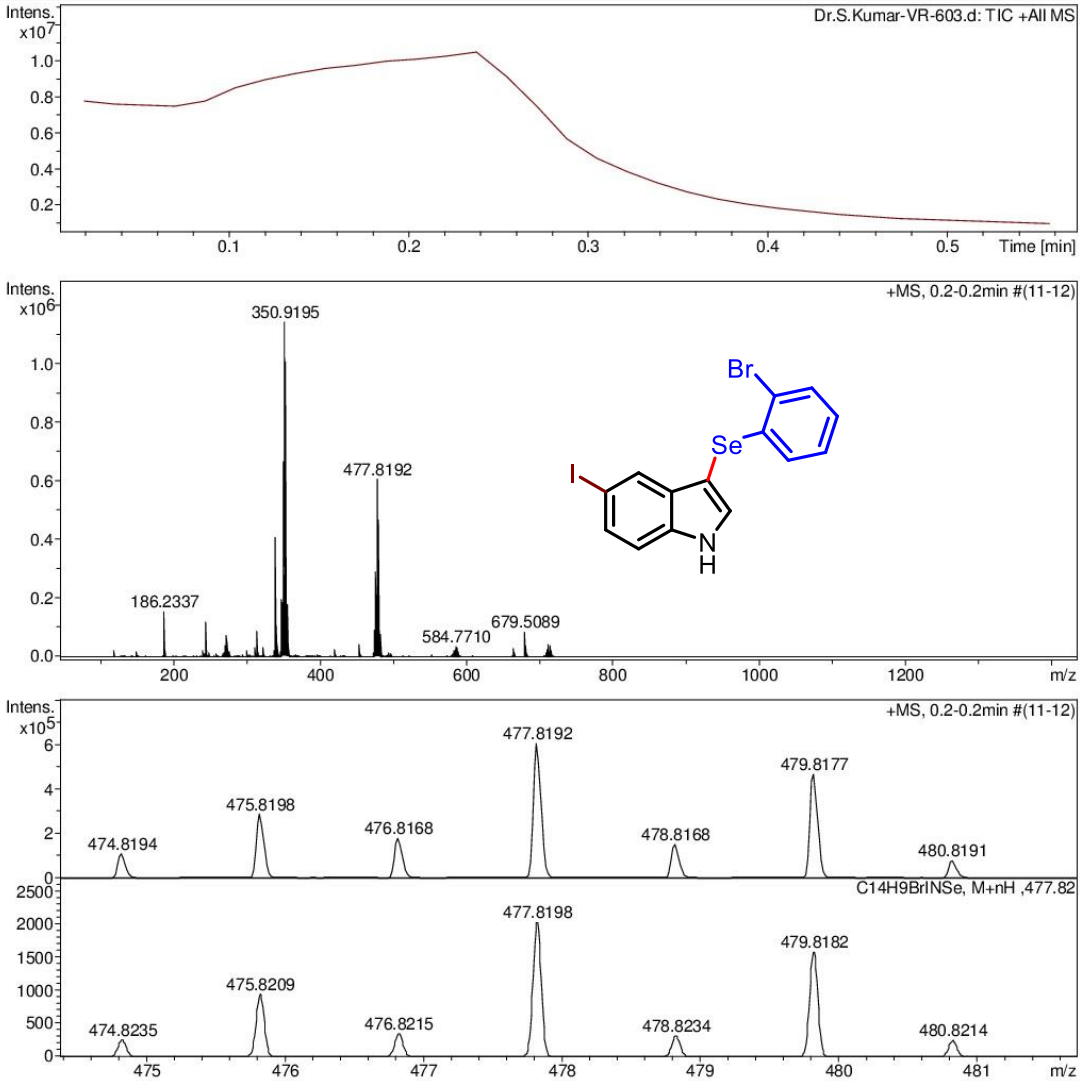
Display Report

Analysis Info

Analysis Name	D:\Data\NEW USER DATA 2017\2018\APRIL 2018\5 Aril\Dr.S.Kumar-VR-603.d	Acquisition Date	4/5/2018 3:36:12 PM
Method	tune_low_APCI.m	Operator	RUCHI
Sample Name	VR-603	Instrument	micrOTOF-Q II 10330
Comment			

Acquisition Parameter

Source Type	APCI	Ion Polarity	Positive	Set Nebulizer	2.5 Bar
Focus	Not active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste



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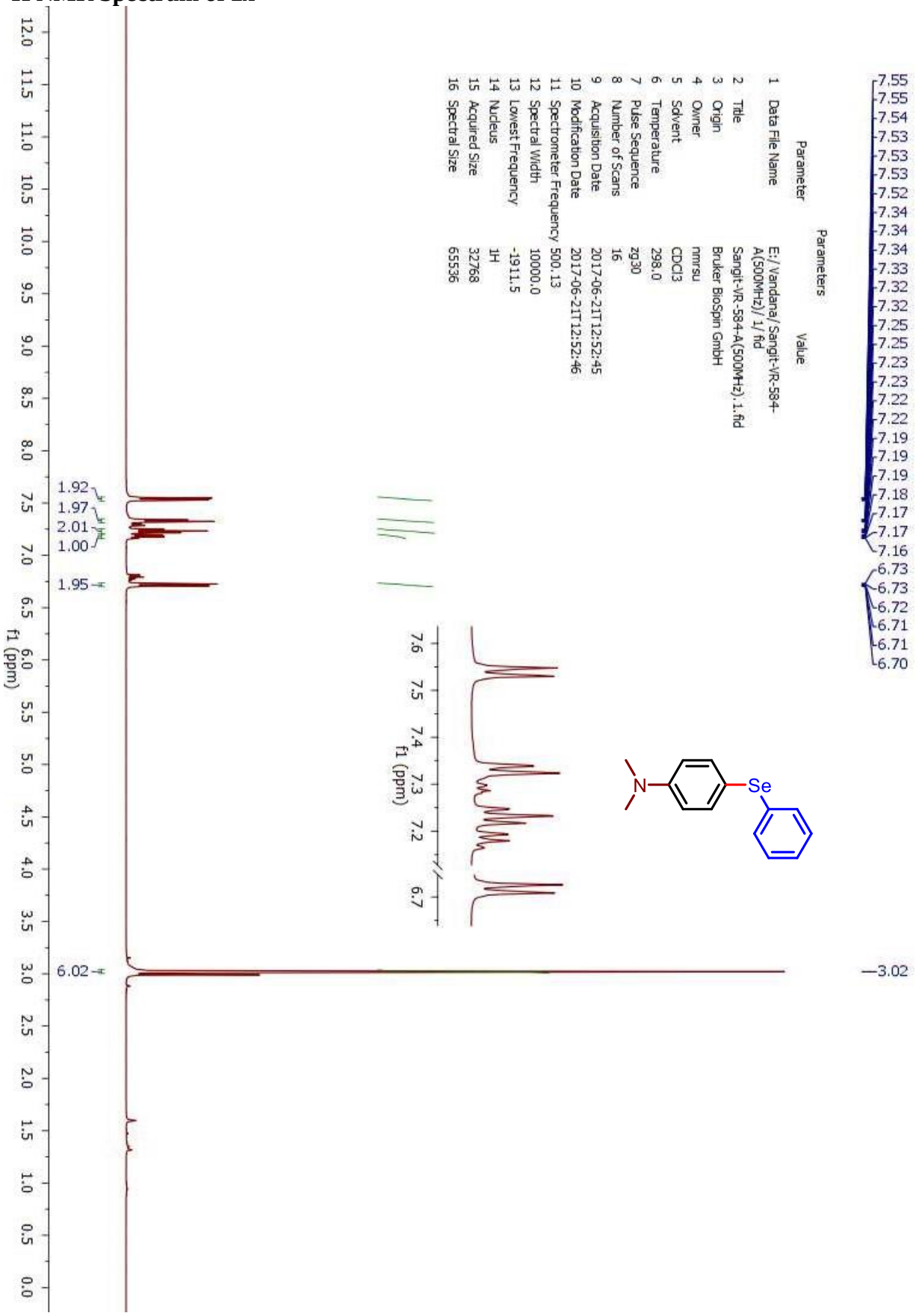
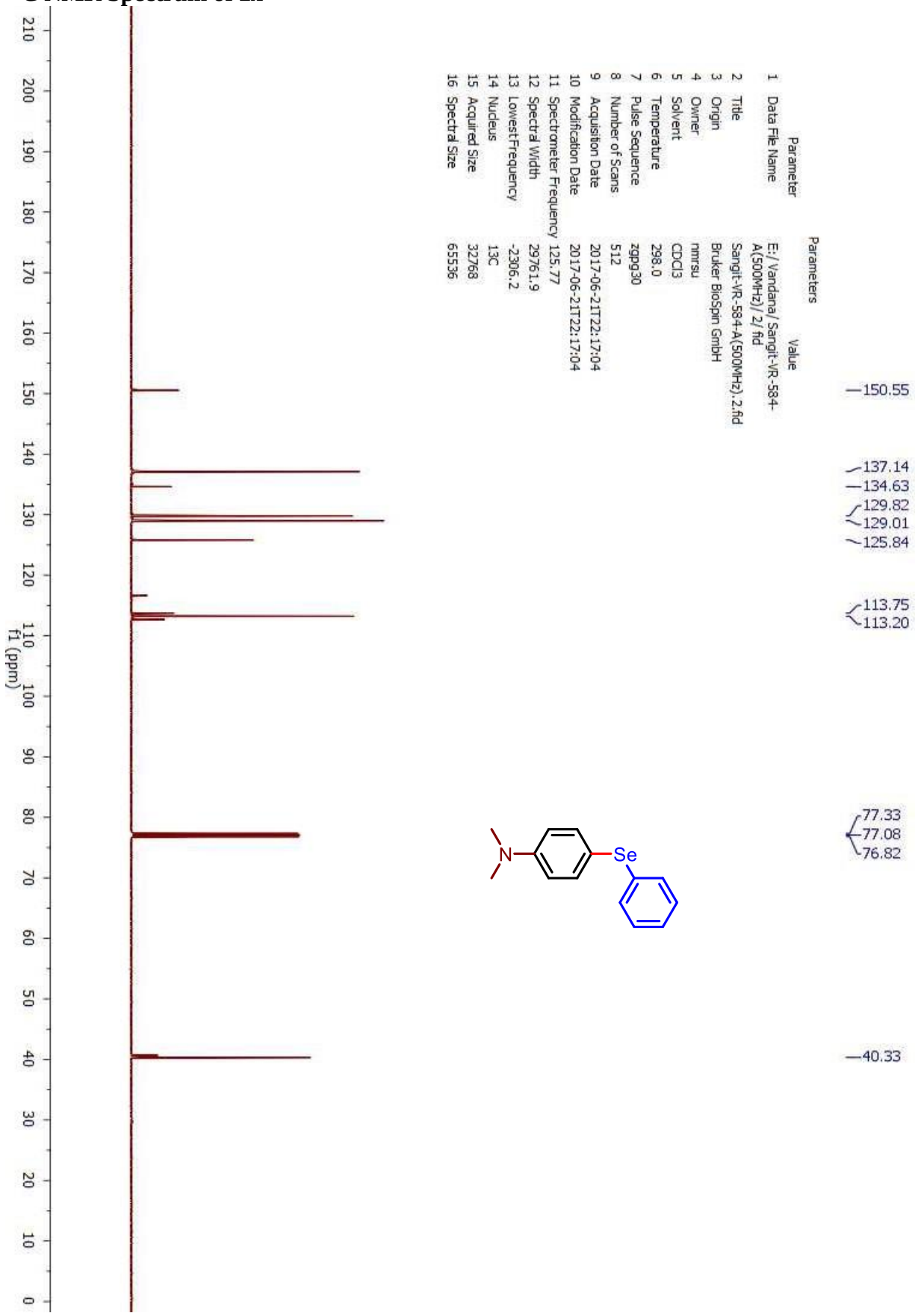
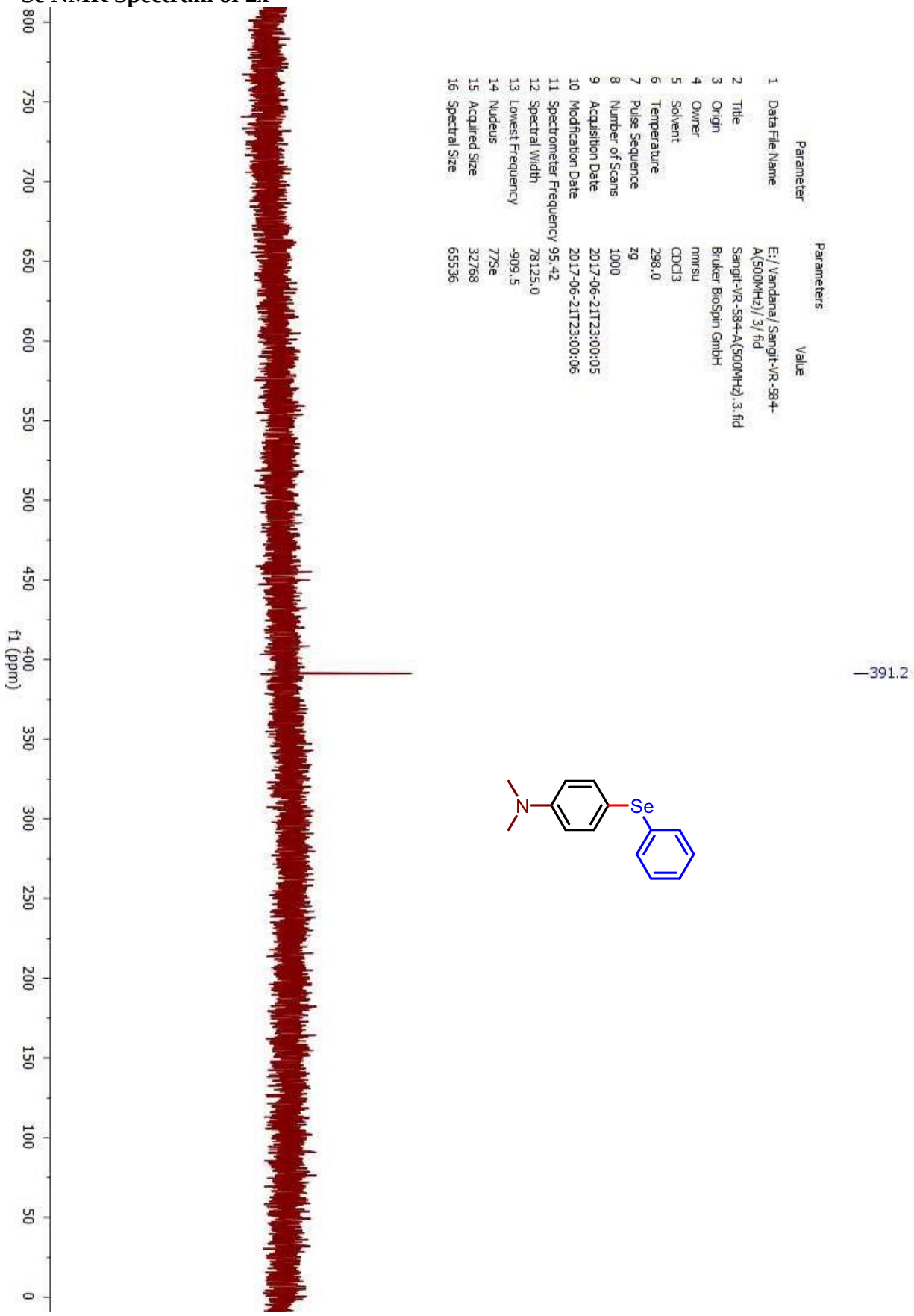
^{1}H NMR Spectrum of 2x



^{13}C NMR Spectrum of 2x



^{77}Se NMR Spectrum of 2x



Mass Spectrum of 2x

Acquisition date: **31/12/18**

Sample ID: **VR-551**

Supervisor: **Dr. S Kumar**

Operator: **IISERB-CIF-Mass Facility**

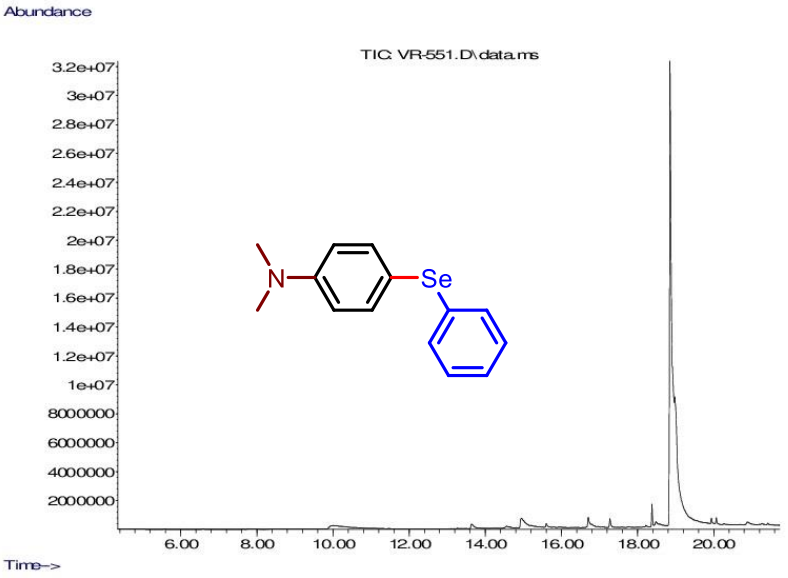
Instrument: Agilent 7890A GC with 5975C MS system

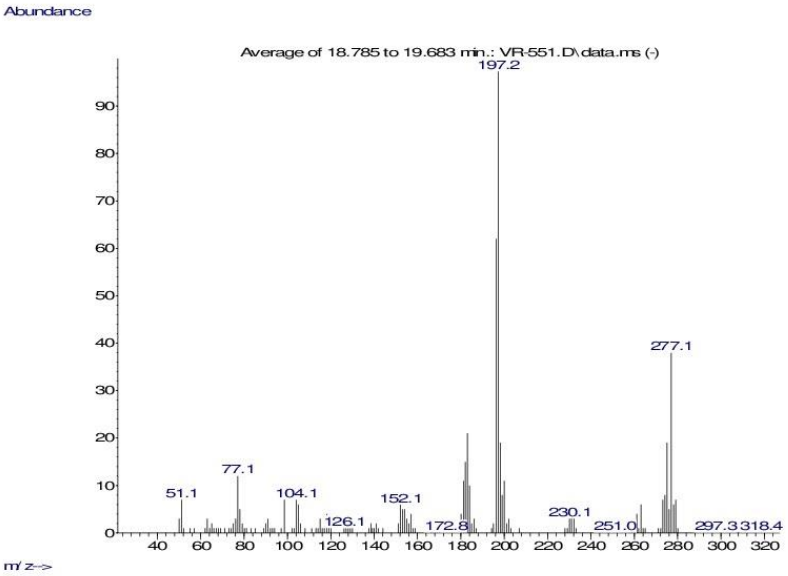
***Column*: HP-5**

Ionization: **EI (70 eV)**

Method: **General_1_HP5_80_DEG.M**

***MSD*: Single Quad.**





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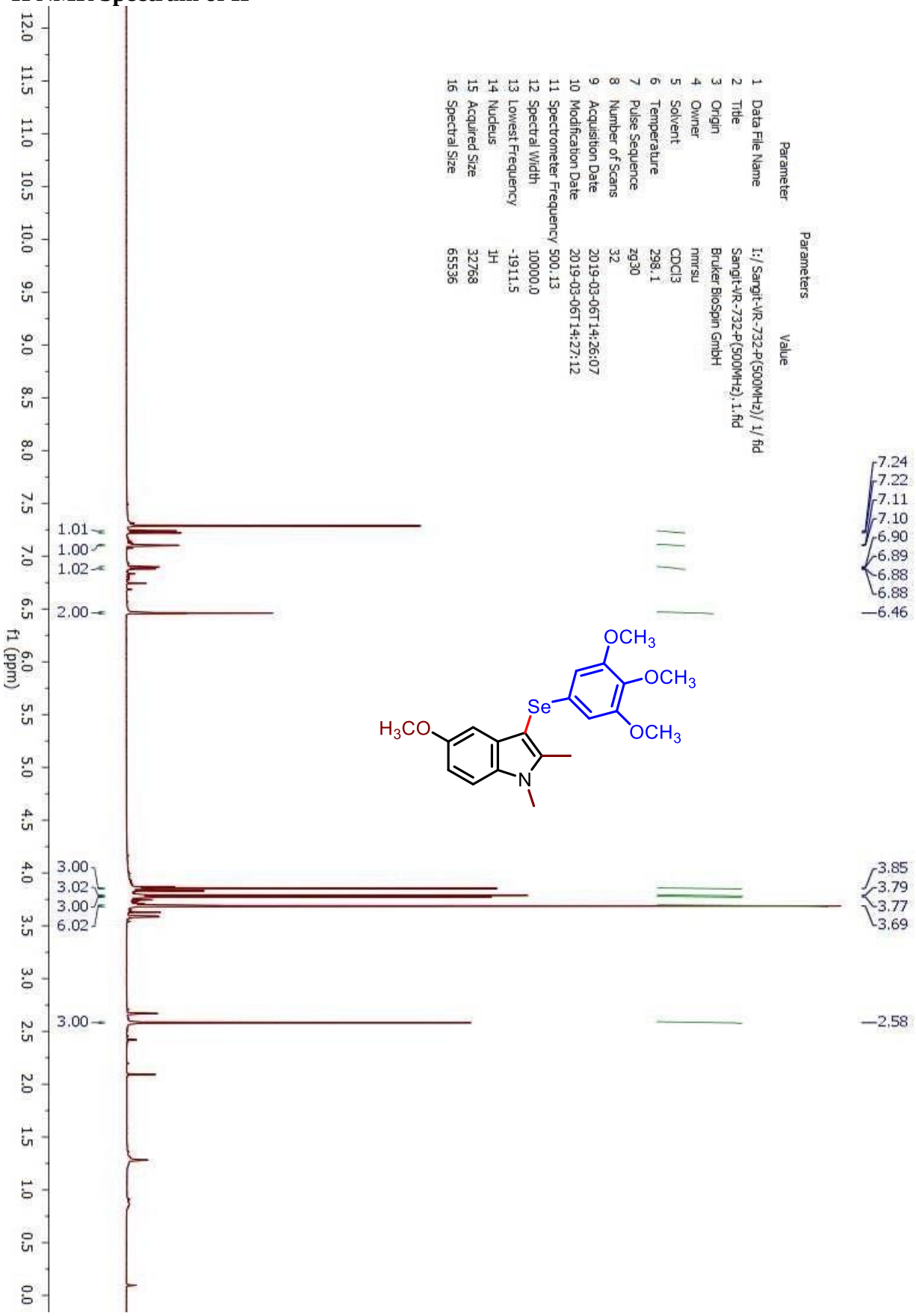
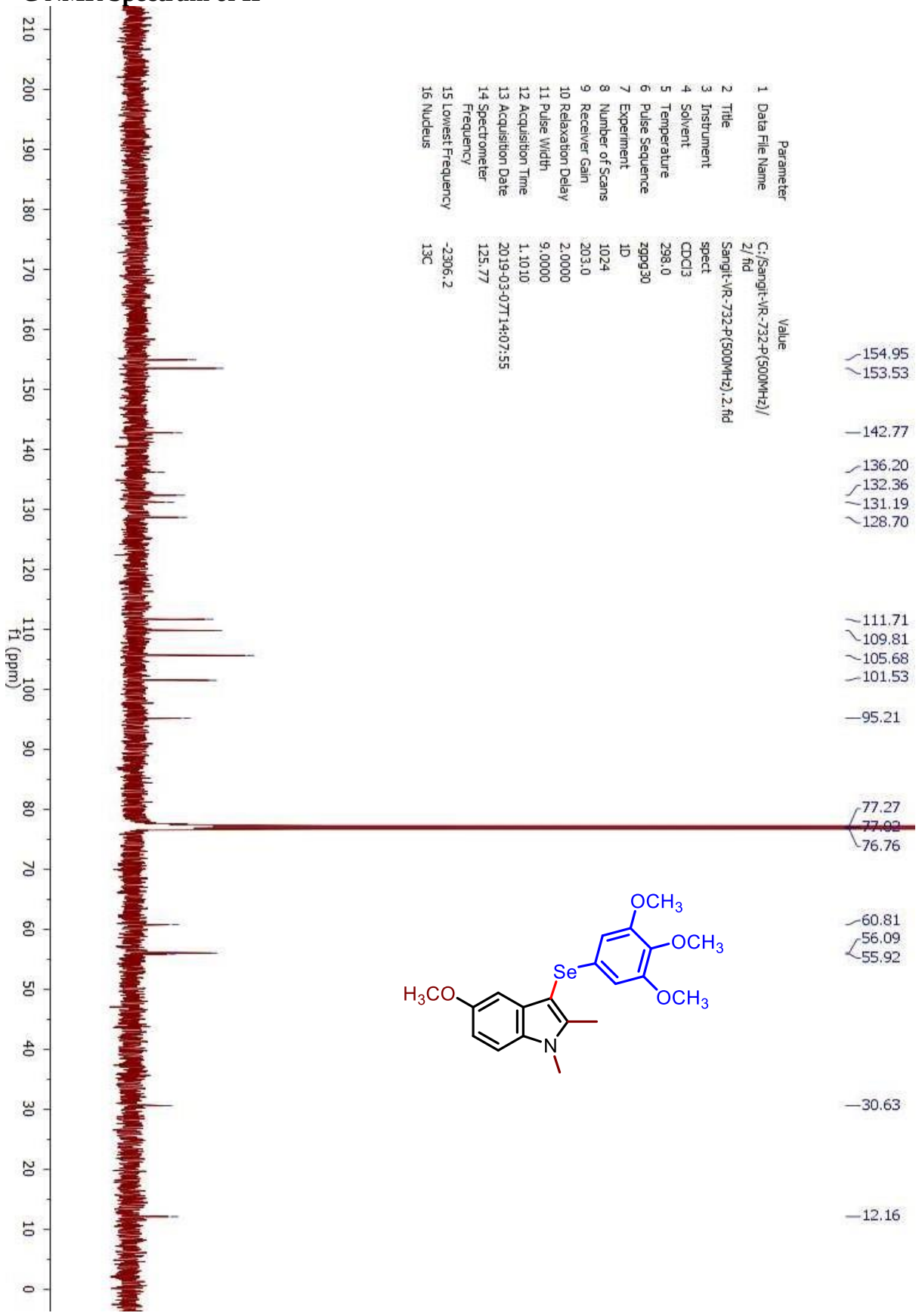
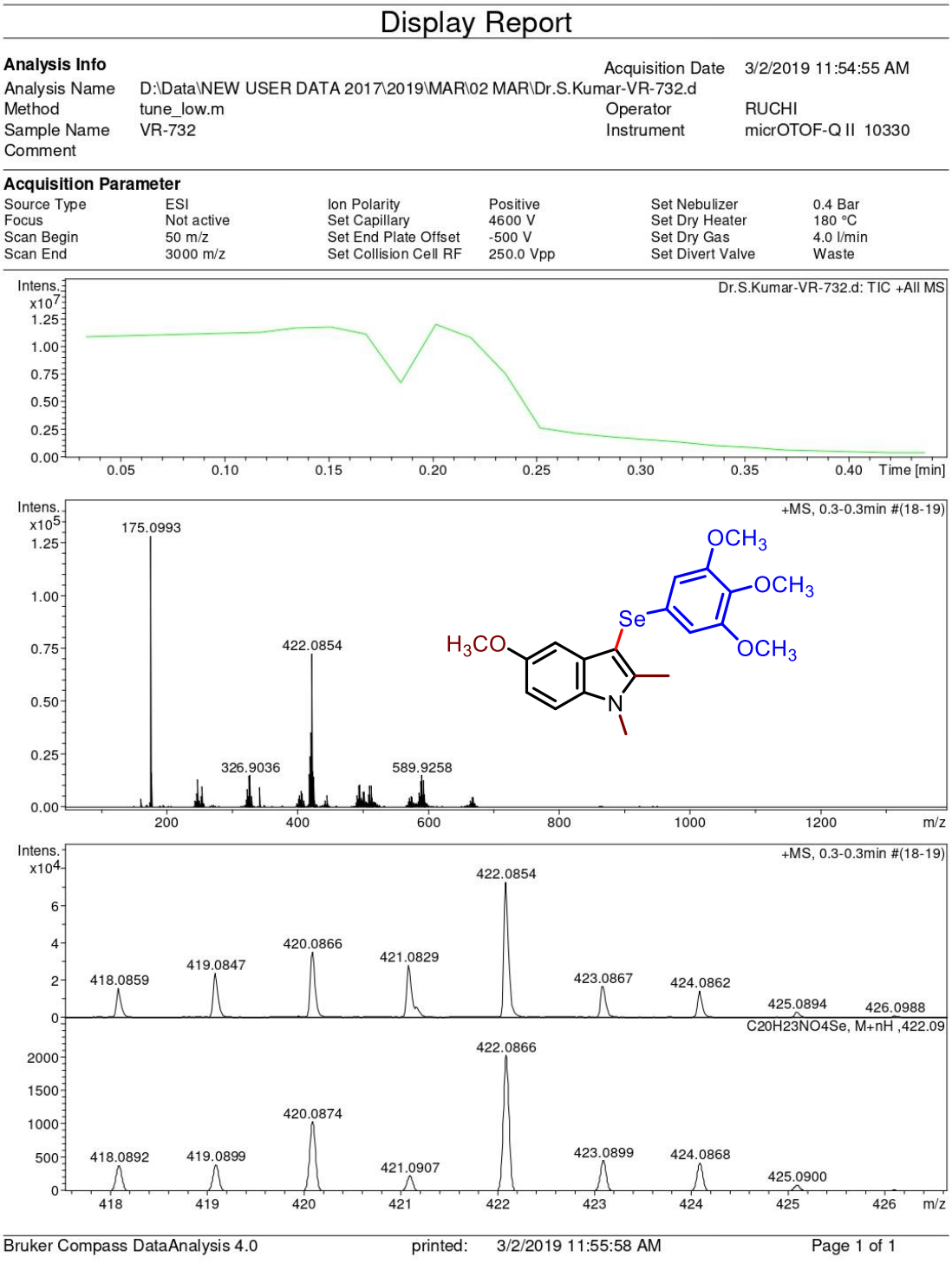
^{1}H NMR Spectrum of II



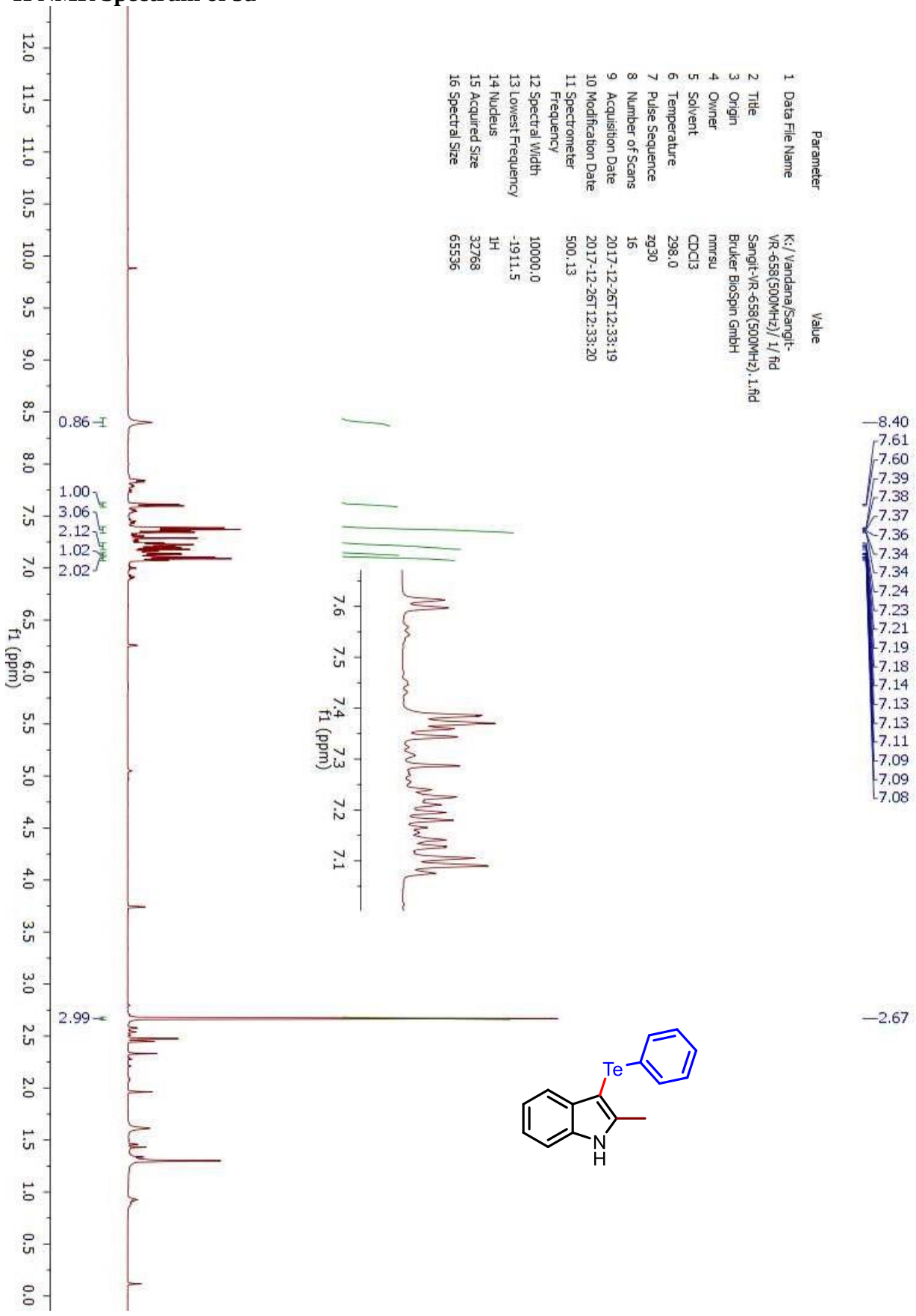
^{13}C NMR Spectrum of II



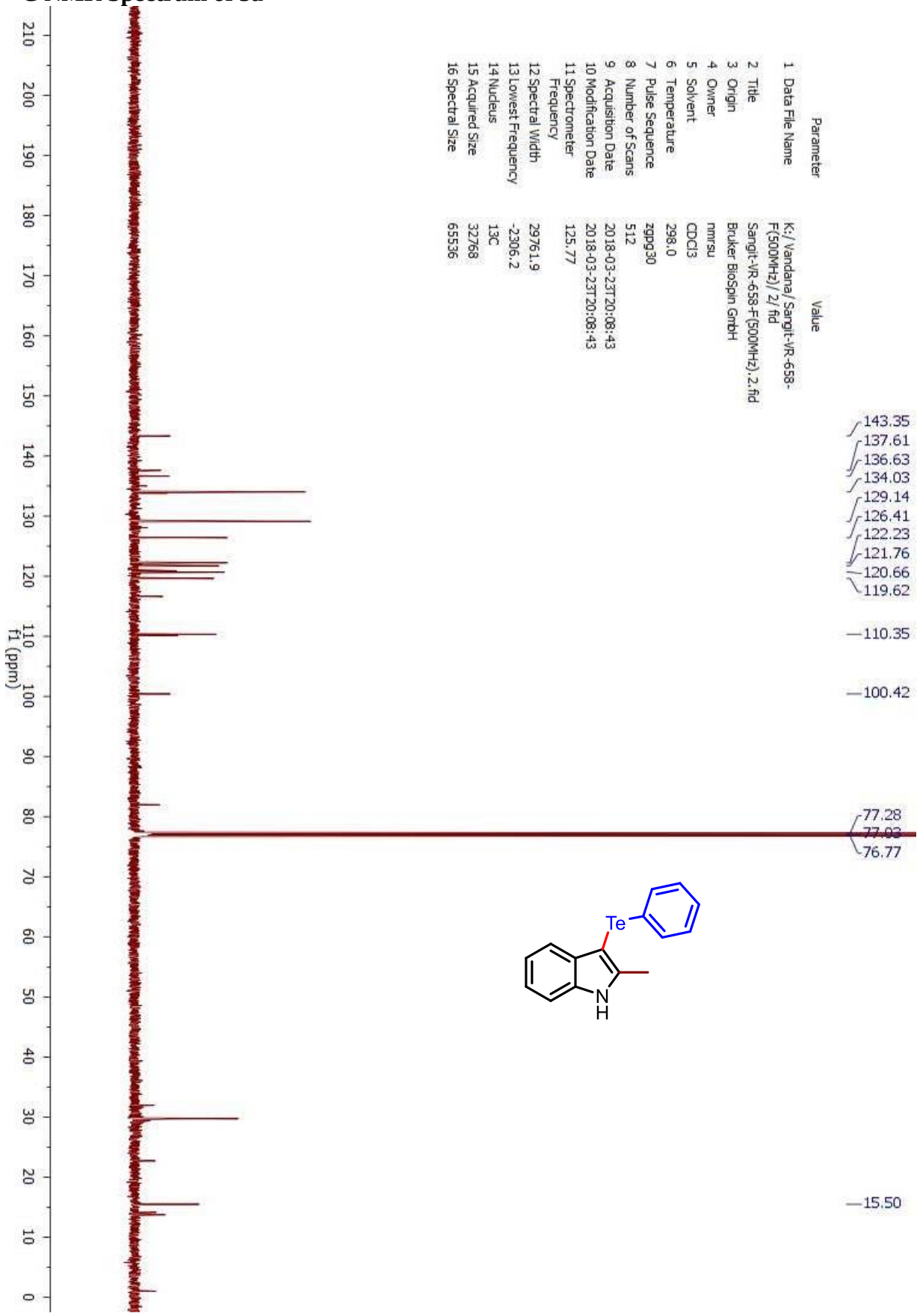
Mass Spectrum of II



^{1}H NMR Spectrum of 3a



^{13}C NMR Spectrum of 3a



Mass Spectrum of 3a

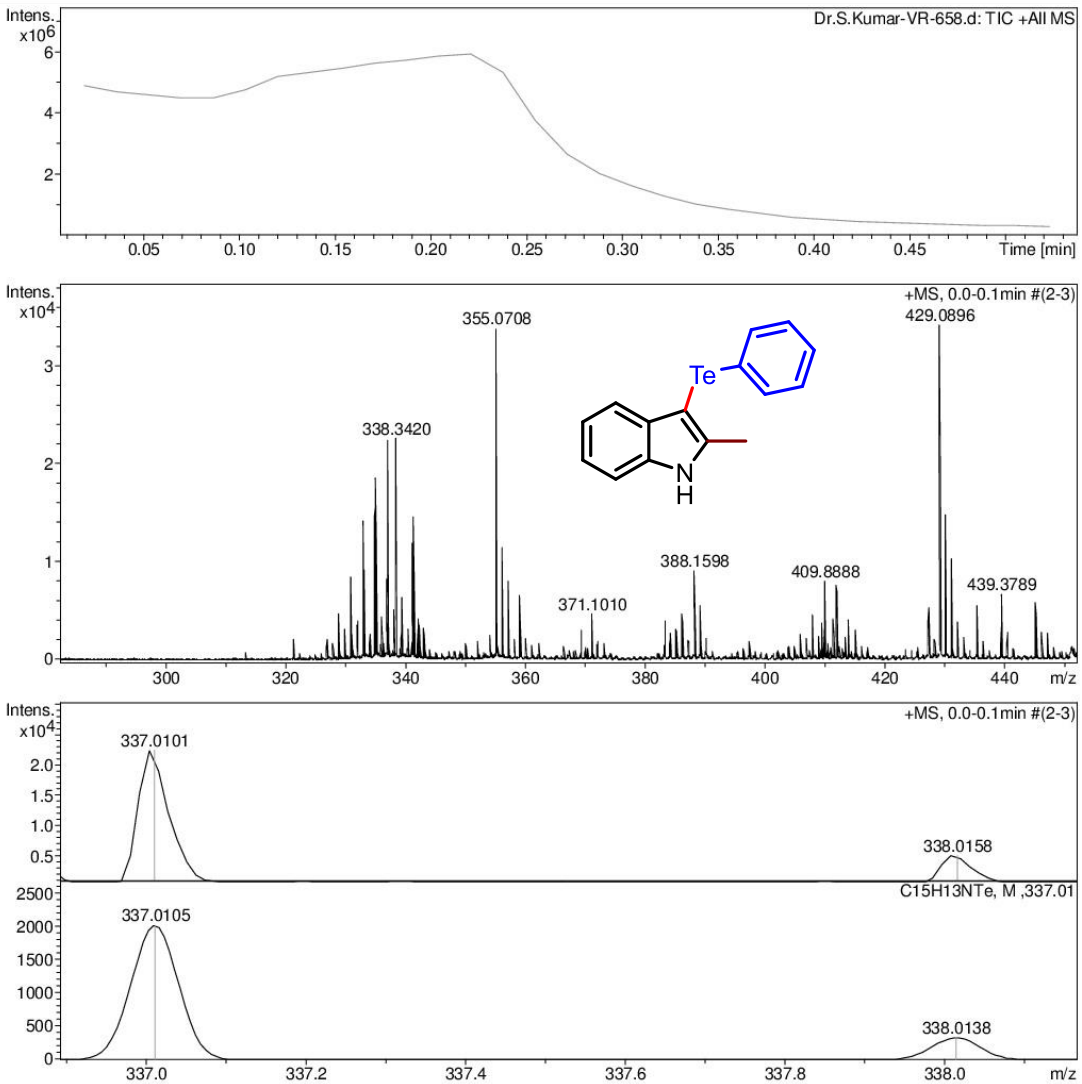
Display Report

Analysis Info

Analysis Name	D:\Data\NEW USER DATA 2017\2018\APRIL 2018\5 Aril\Dr.S.Kumar-VR-658.d	Acquisition Date	4/5/2018 3:30:42 PM
Method	tune_wide _APCI_23.06.m	Operator	RUCHI
Sample Name	VR-658	Instrument	micrOTOF-Q II 10330
Comment			

Acquisition Parameter

Source Type	APCI	Ion Polarity	Positive	Set Nebulizer	2.5 Bar
Focus	Not active	Set Capillary	4000 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	600.0 Vpp	Set Divert Valve	Waste



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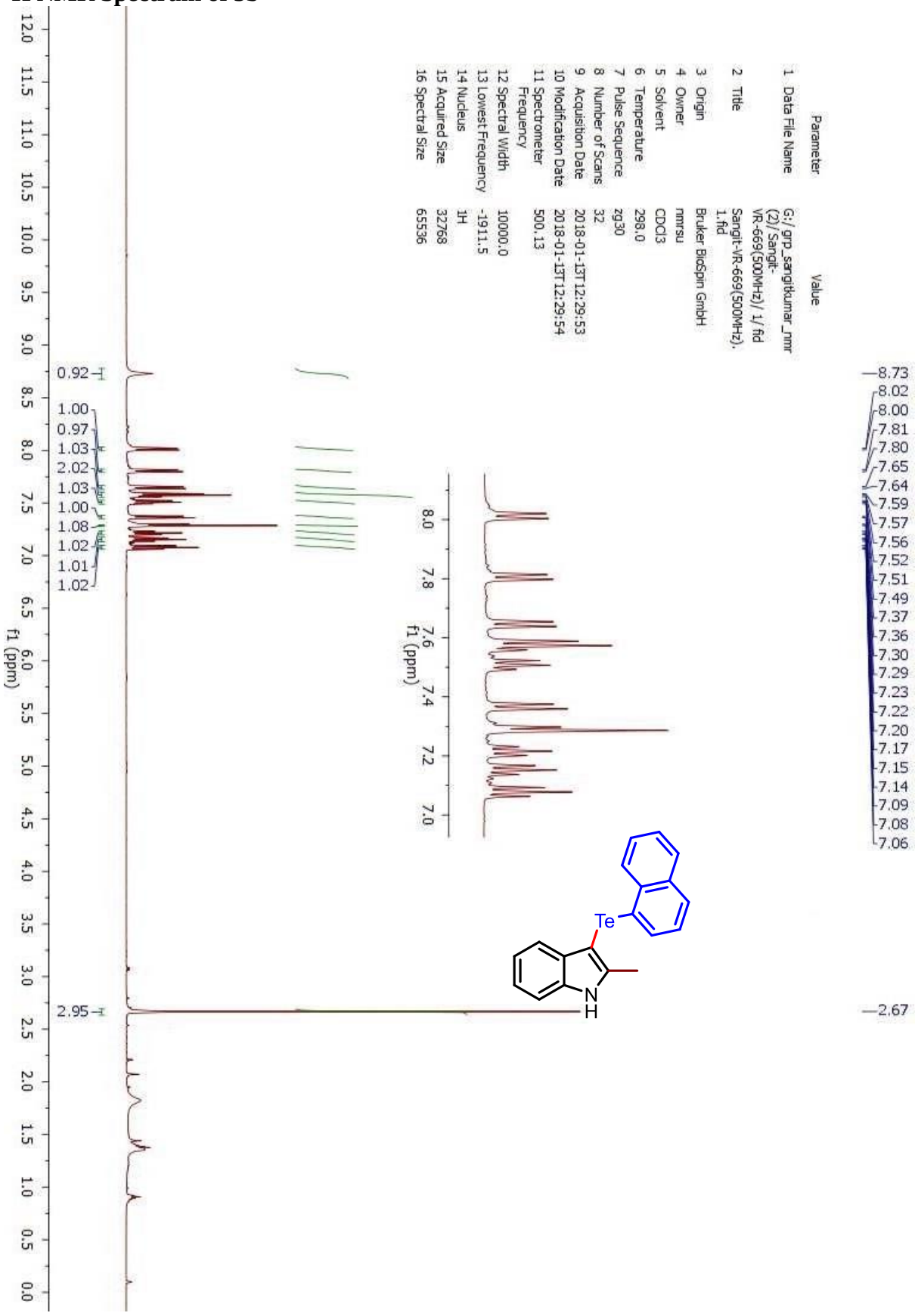
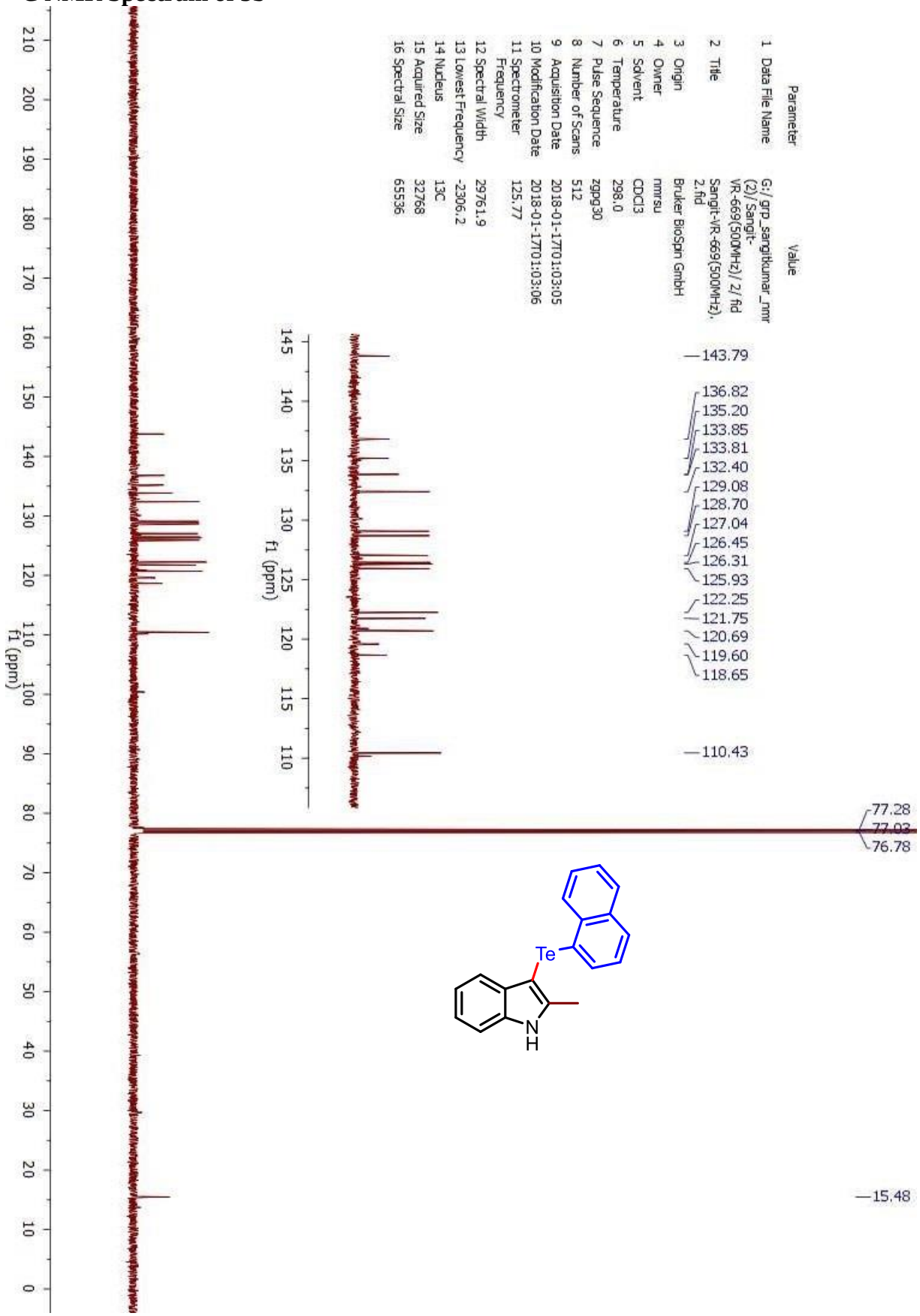
^{1}H NMR Spectrum of 3b



^{13}C NMR Spectrum of 3b



Mass Spectrum of 3b

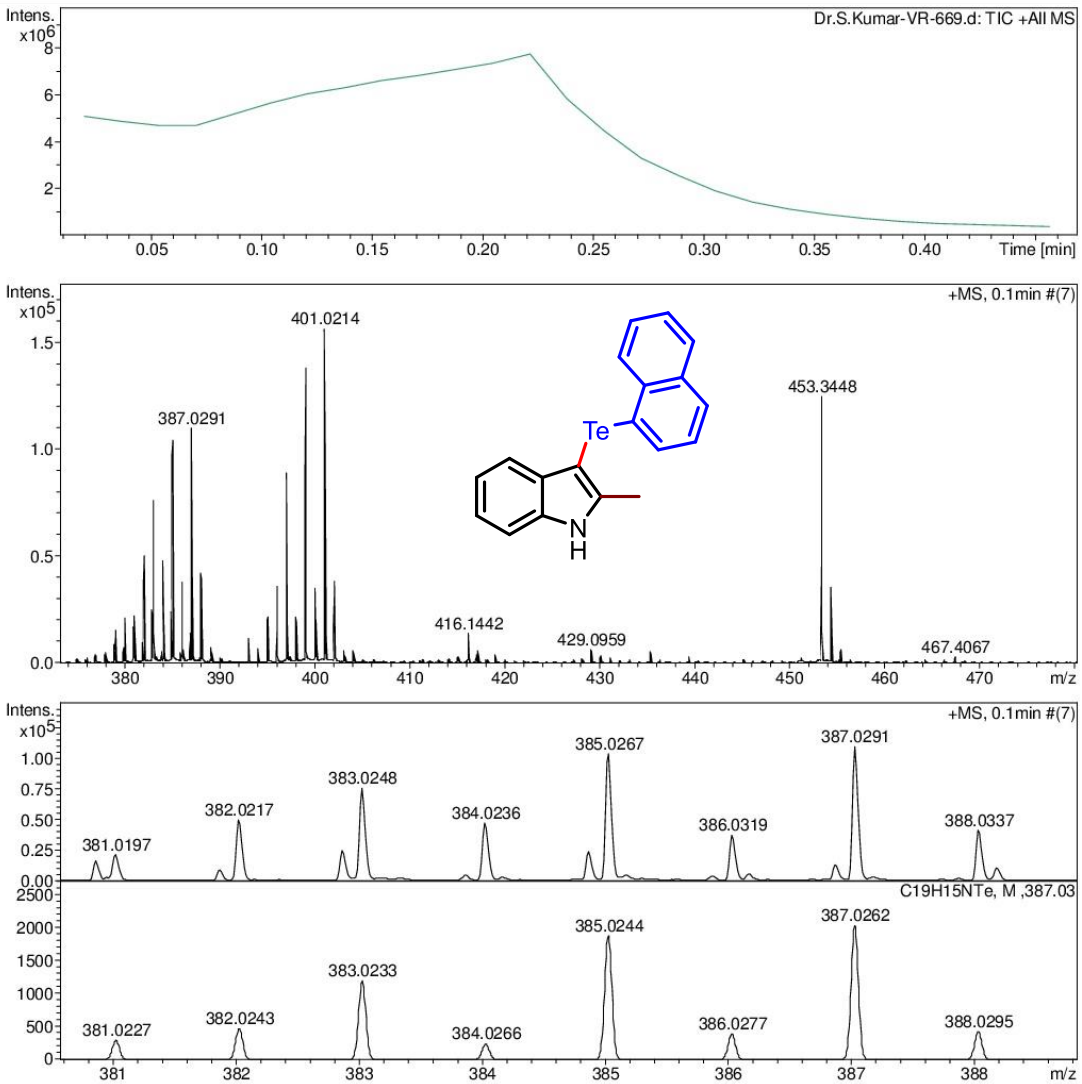
Display Report

Analysis Info

Analysis Name	D:\Data\NEW USER DATA 2017\2018\APRIL 2018\5 Aril\Dr.S.Kumar-VR-669.d	Acquisition Date	4/5/2018 3:33:16 PM
Method	tune_wide _APCI_23.06.m	Operator	RUCHI
Sample Name	VR-669	Instrument	micrOTOF-Q II 10330
Comment			

Acquisition Parameter

Source Type	APCI	Ion Polarity	Positive	Set Nebulizer	2.5 Bar
Focus	Not active	Set Capillary	4000 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	600.0 Vpp	Set Divert Valve	Waste



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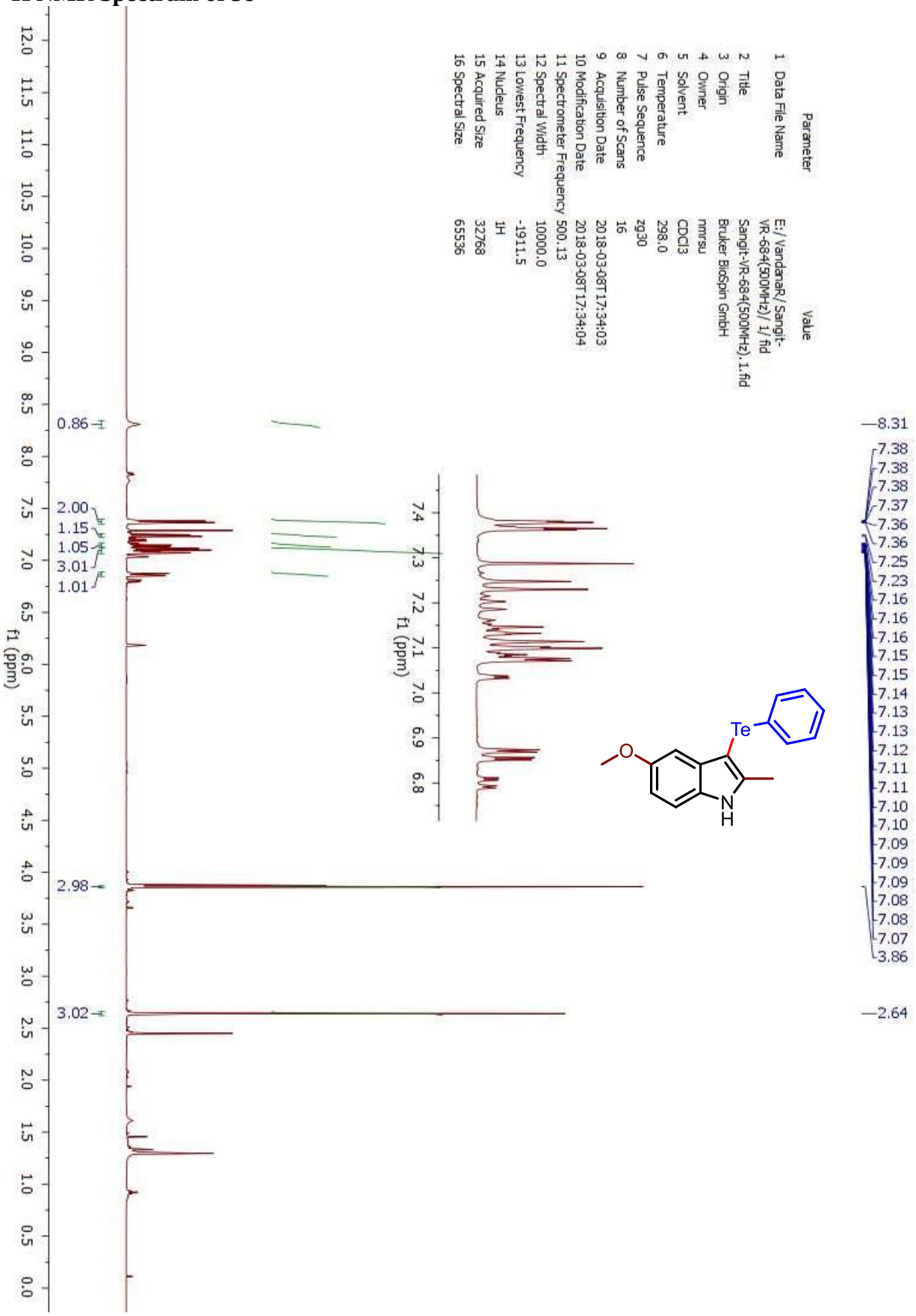
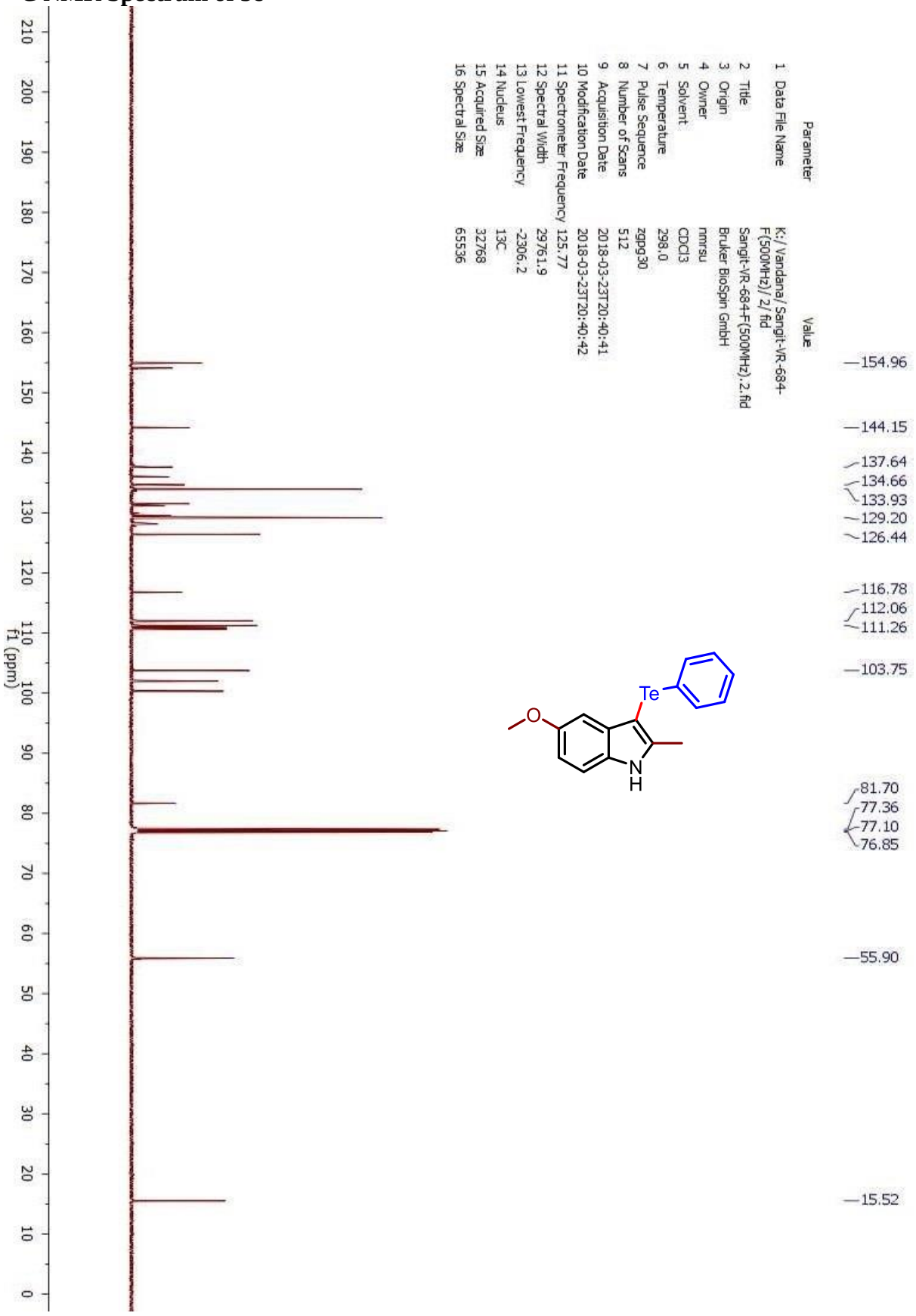
^{1}H NMR Spectrum of 3c



^{13}C NMR Spectrum of 3c



Mass Spectrum of 3c

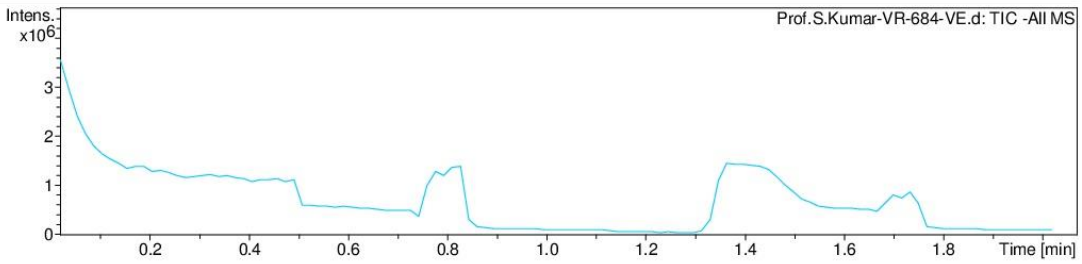
Display Report

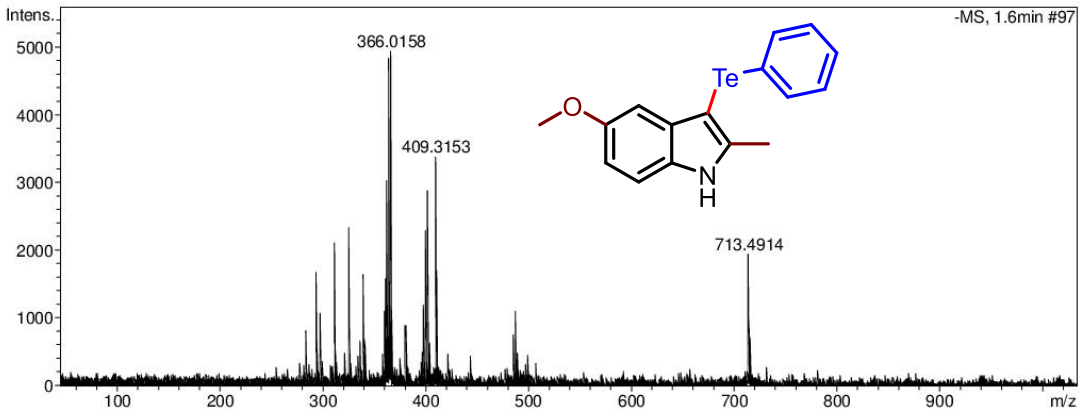
Analysis Info

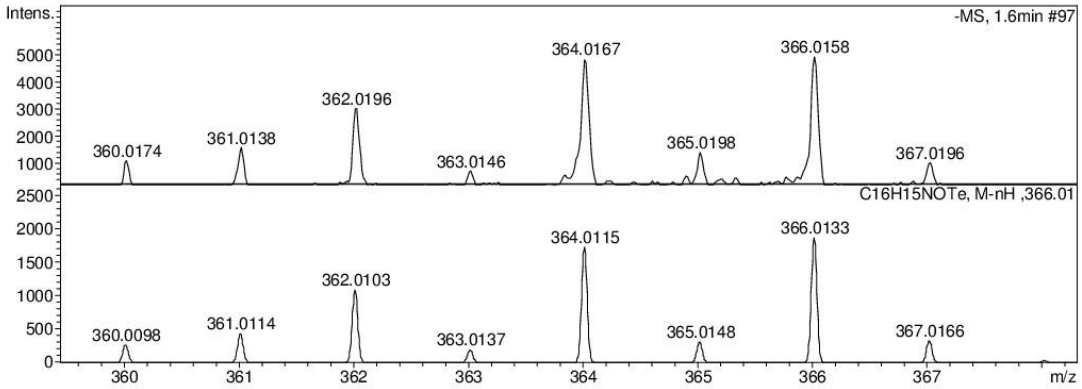
Analysis Name	D:\Data\NEW USER DATA 2017\2018\JUNE 2018\11 JUNE\Prof.S.Kumar-VR-684-VE.d	Acquisition Date	6/11/2018 4:43:04 PM
Method	tune_low.m	Operator	RUCHI
Sample Name	VR-684-VE	Instrument	micrOTOF-Q II 10330
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Negative	Set Nebulizer	0.4 Bar
Focus	Not active	Set Capillary	2500 V	Set Dry Heater	180 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	400.0 Vpp	Set Divert Valve	Waste







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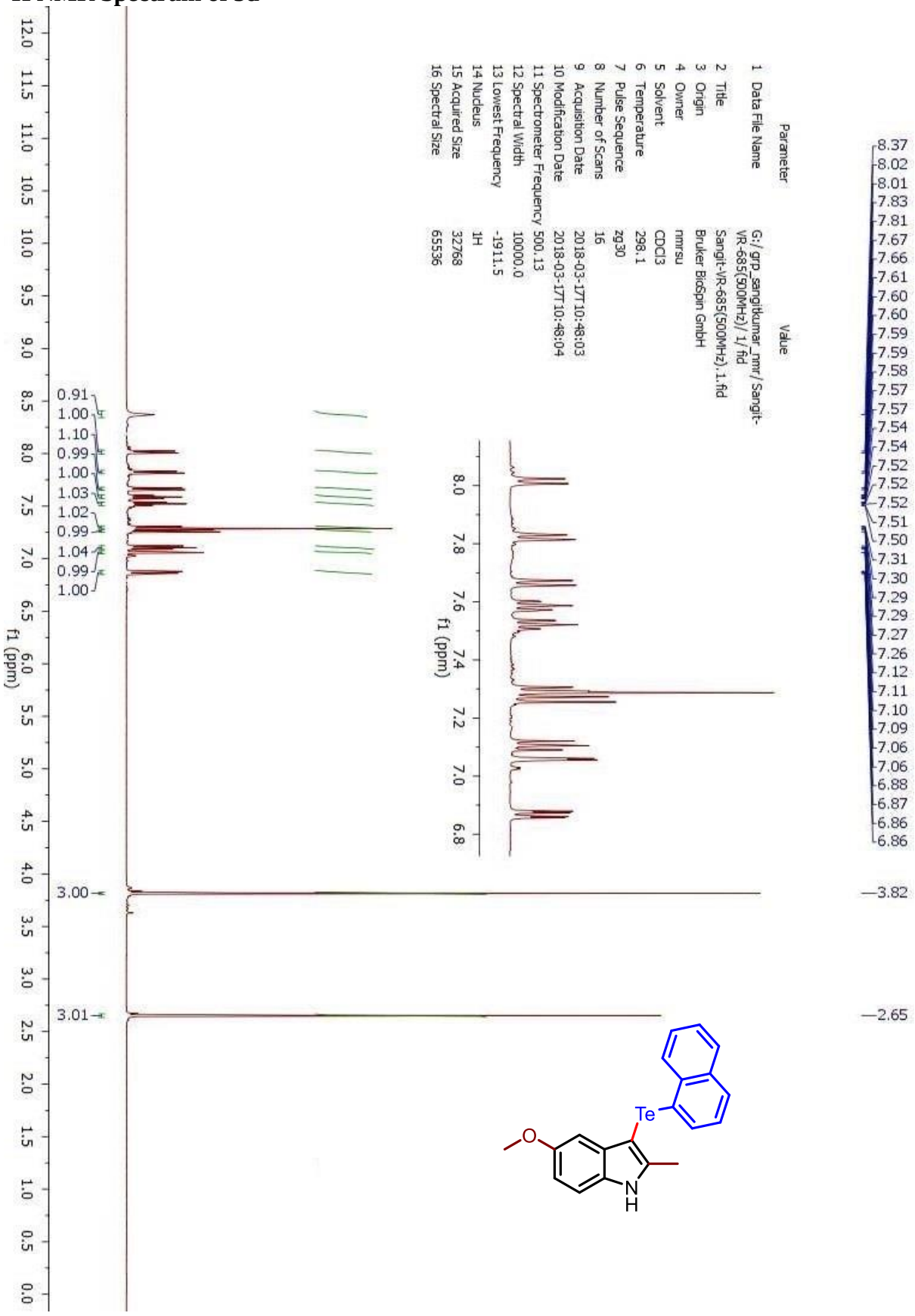
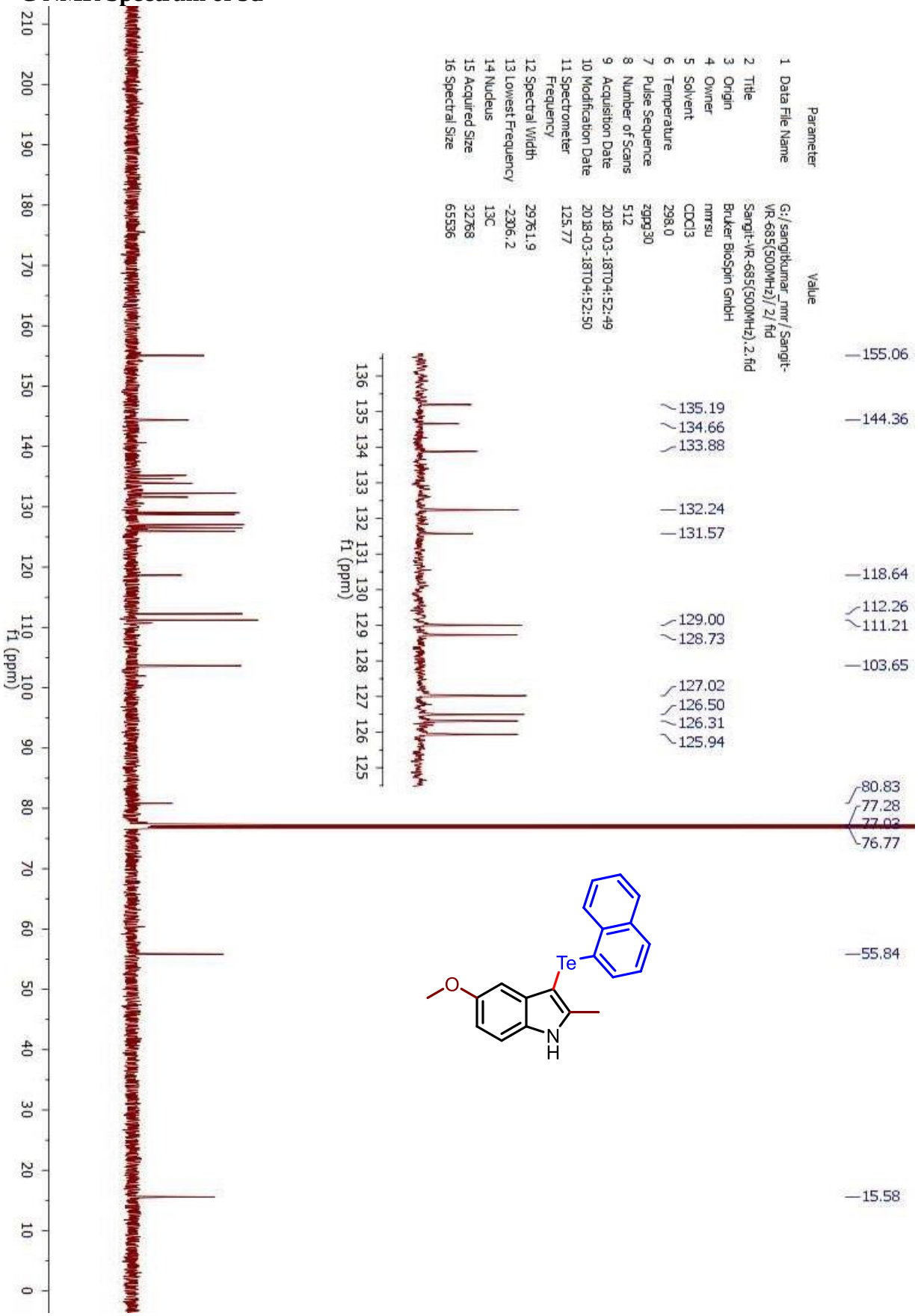
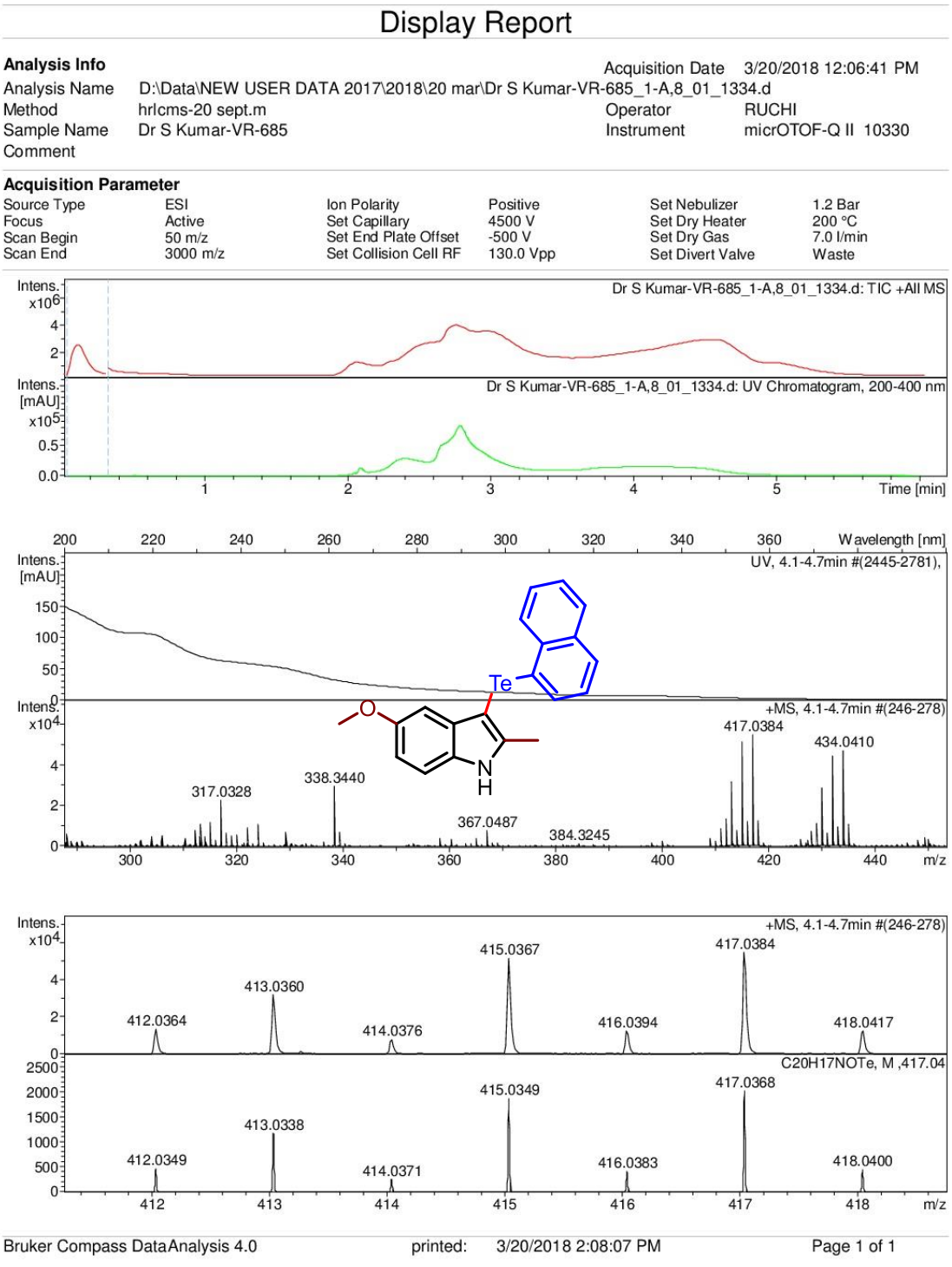
^{1}H NMR Spectrum of 3d



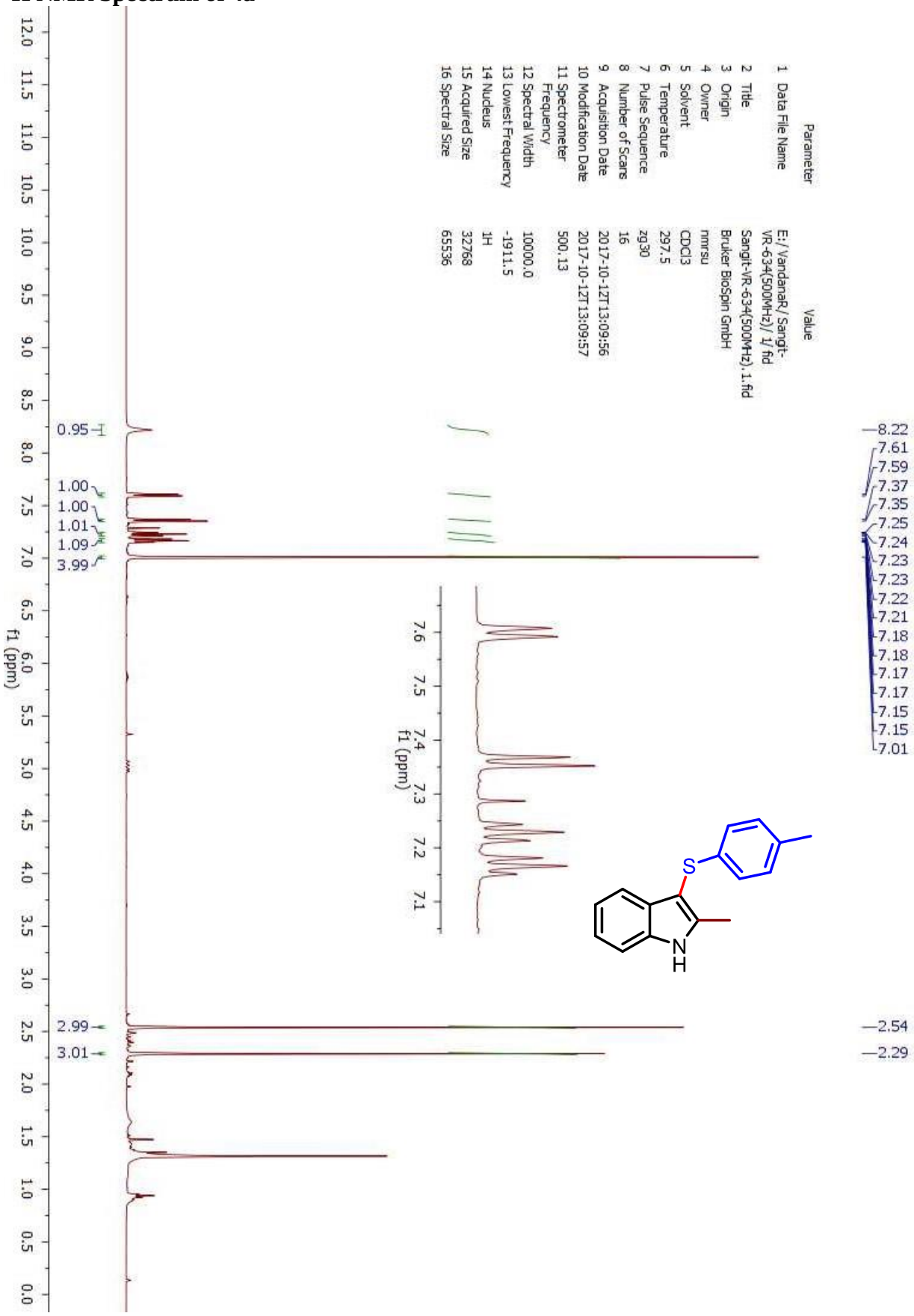
^{13}C NMR Spectrum of 3d



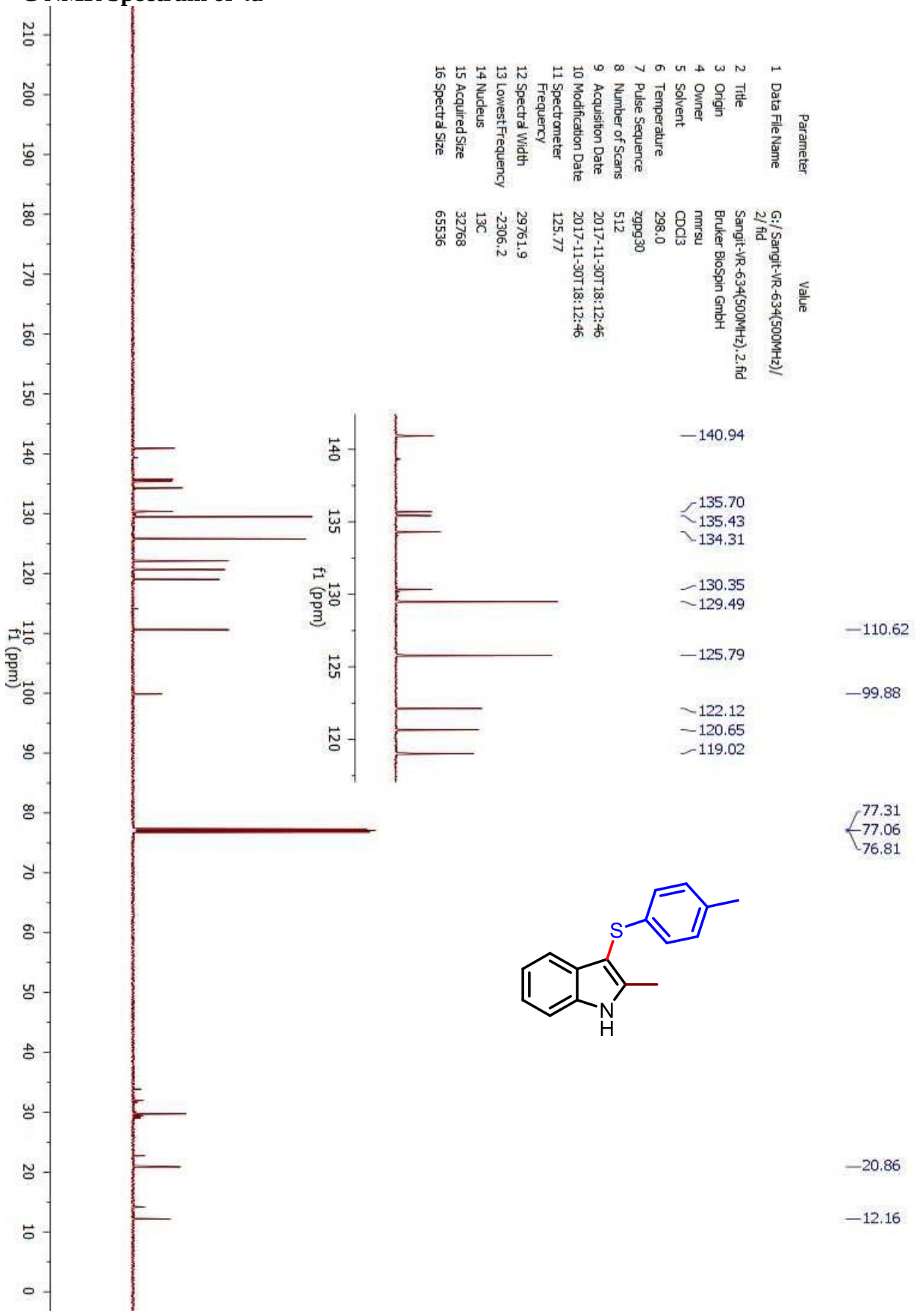
Mass Spectrum of 3d



^{1}H NMR Spectrum of 4a



^{13}C NMR Spectrum of 4a



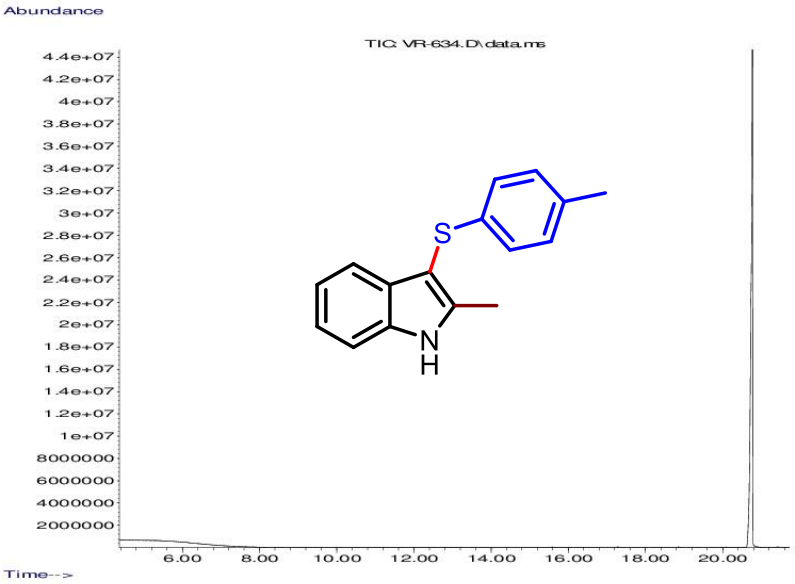
Mass Spectrum of 4a

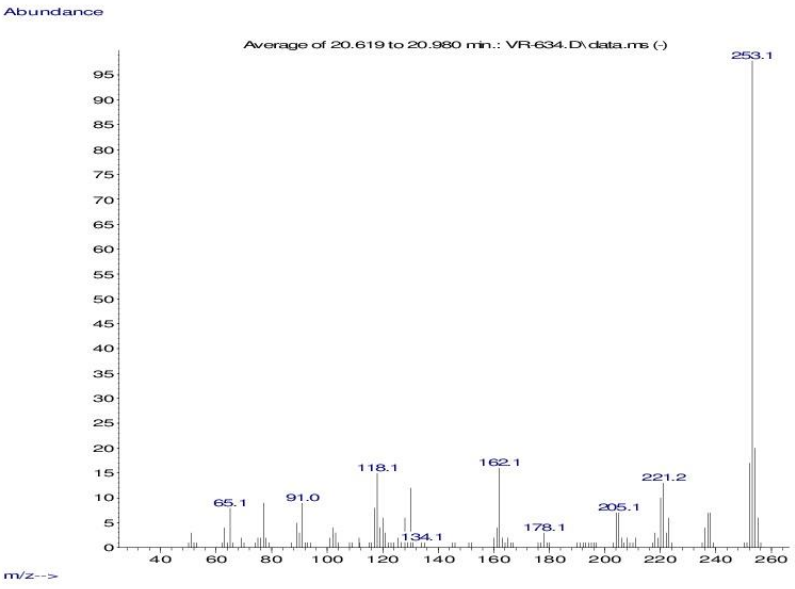
Acquisition date: 03/04/18

Sample ID: VR-634 | ***Supervisor***: Dr. S Kumar | ***Operator:*** **IISERB-CIF-Mass Facility**

Instrument: Agilent 7890A GC with 5975C MS system | ***Column*: HP-5** | ***Ionization:*** **EI (70 eV)**

Method: **General_1_HP5_80_DEG.M** | ***MSD*: Single Quad.**





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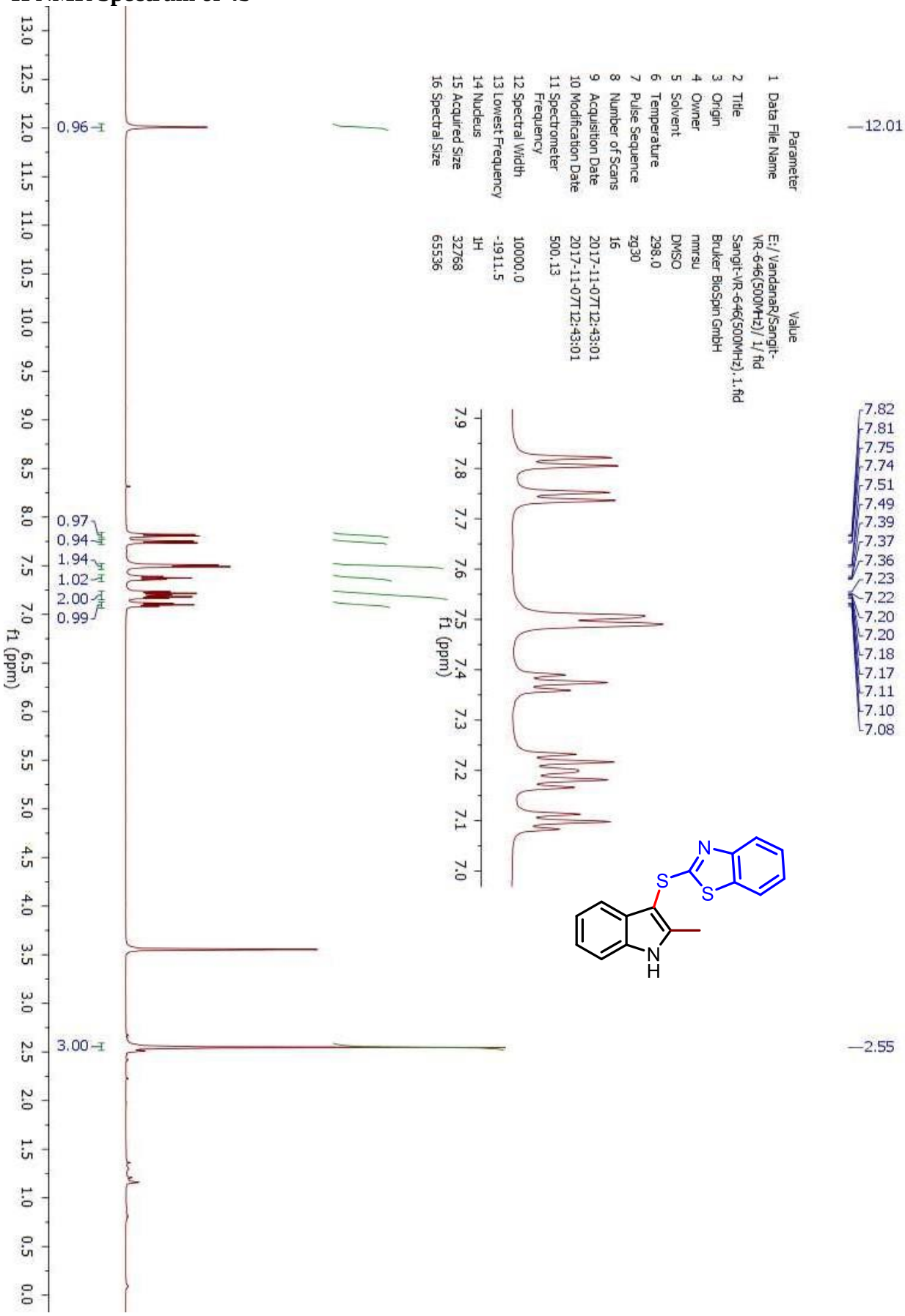
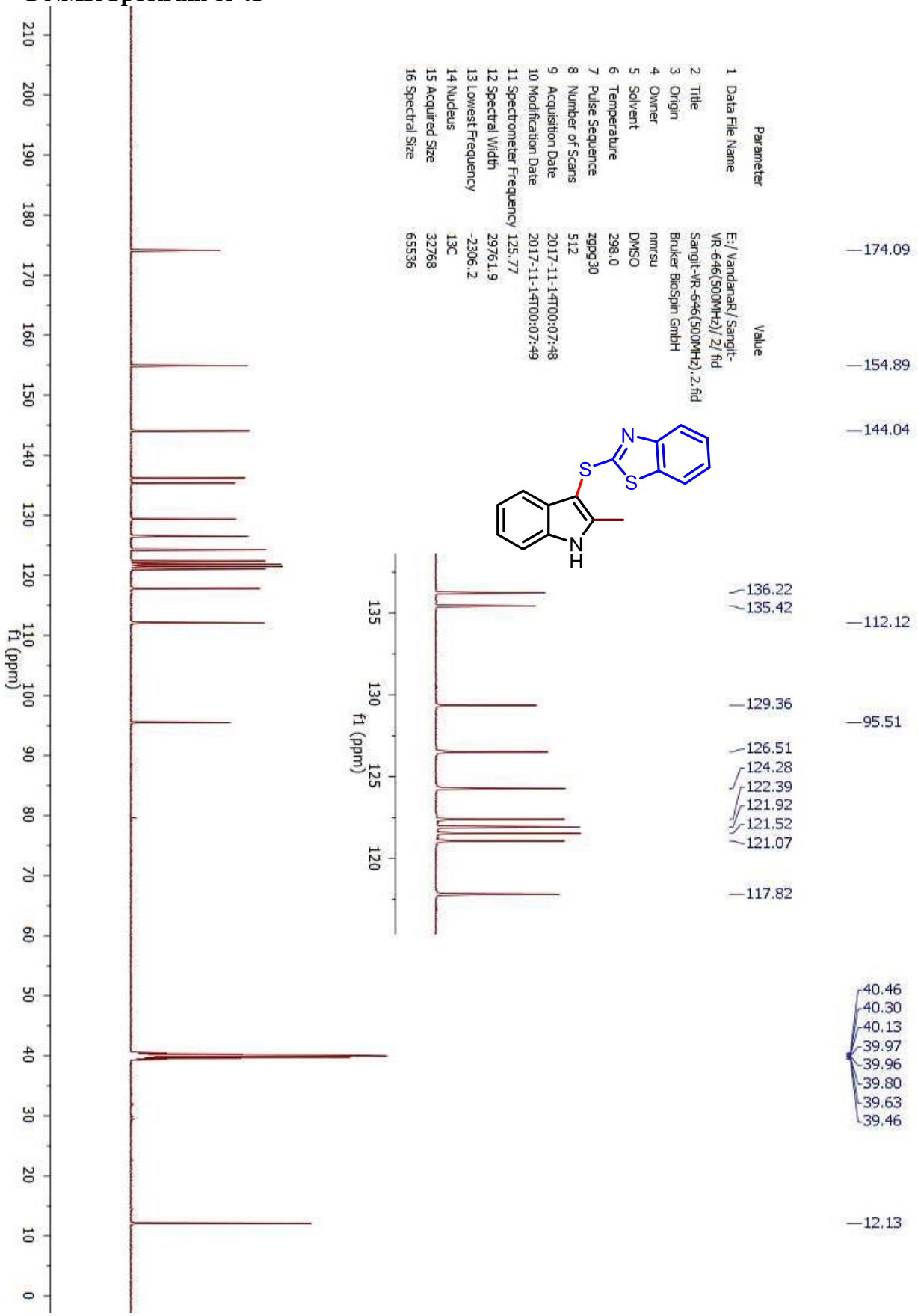
^{1}H NMR Spectrum of 4b



^{13}C NMR Spectrum of 4b



Mass Spectrum of 4b

Acquisition date: 03/04/18

Sample ID: VR-646

Supervisor: Dr. S Kumar

Operator: **IISERB-CIF-Mass Facility**

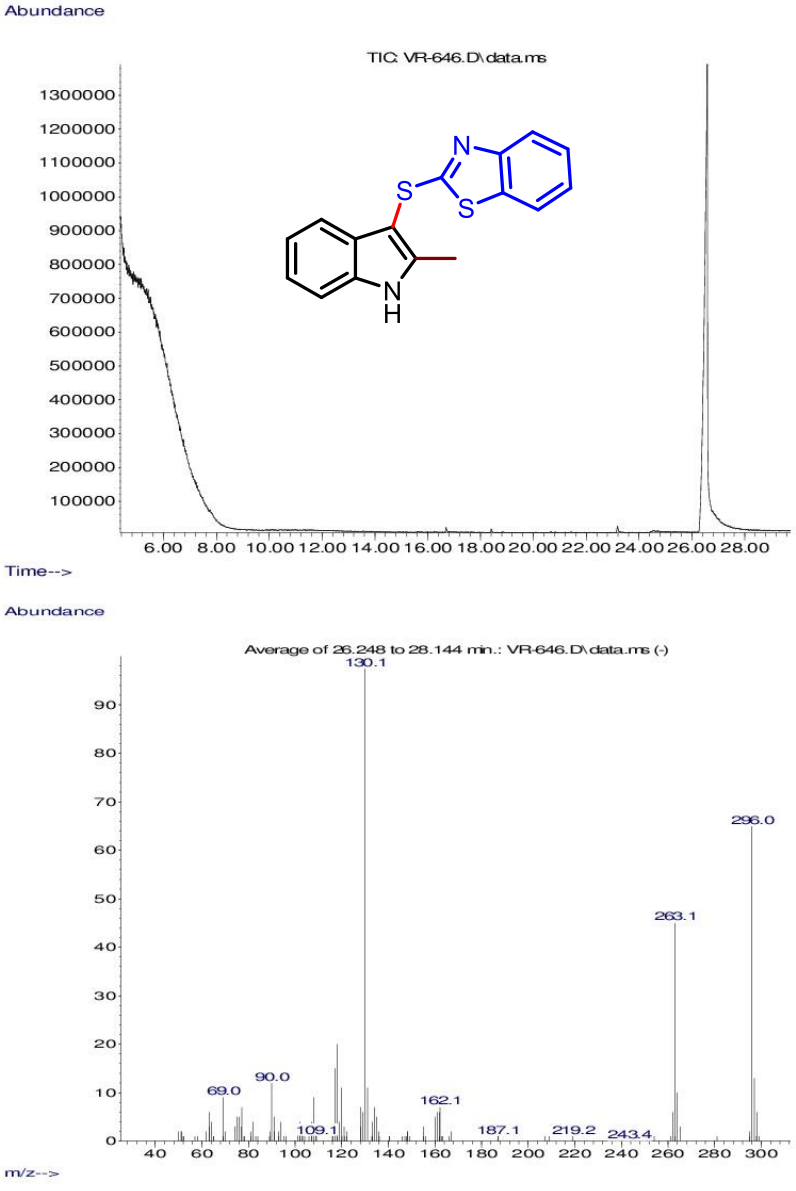
Instrument: Agilent 7890A GC with 5975C MS system

***Column*: HP-5**

Method: **General_1_HP5_80_DEG.M**

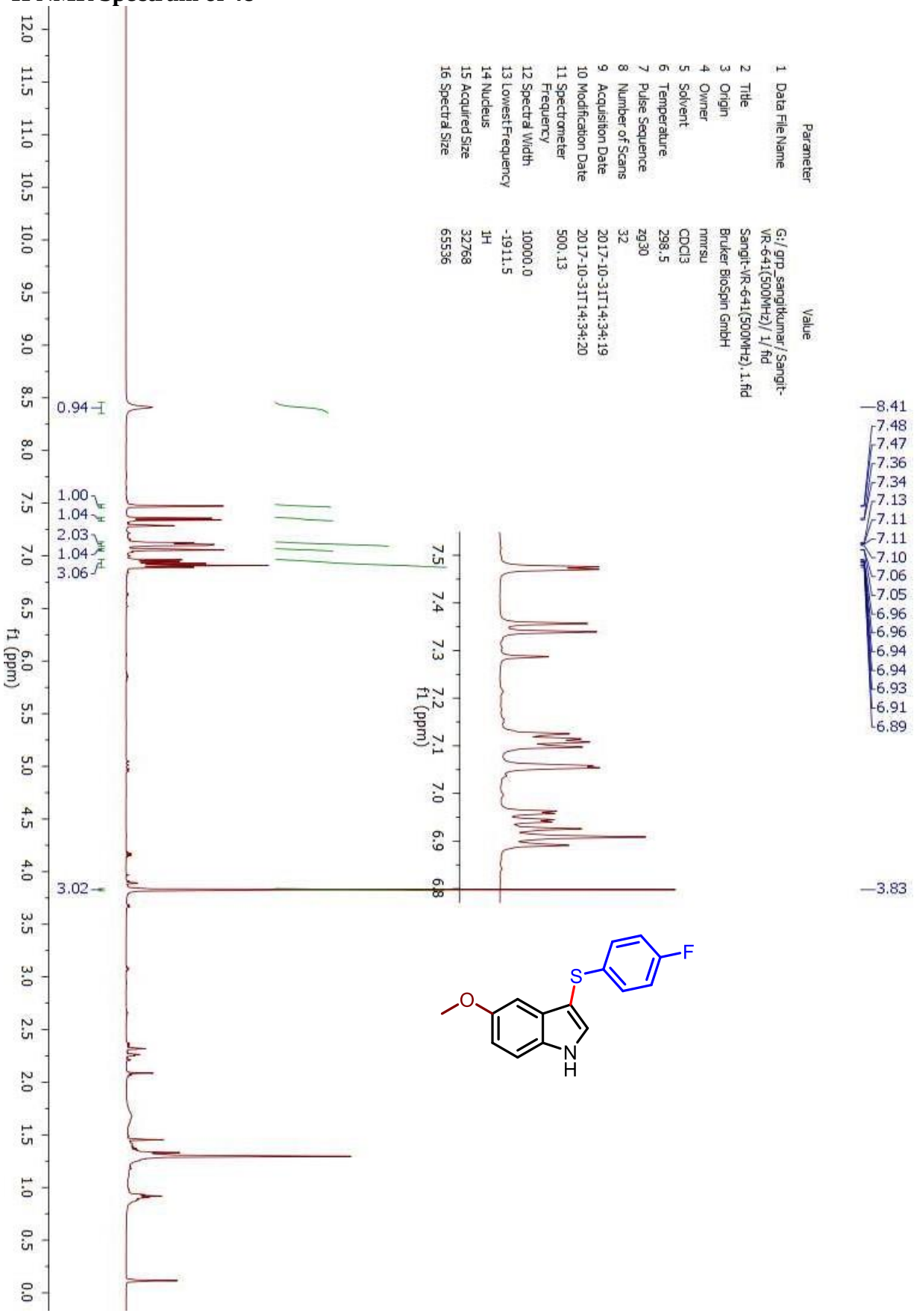
Ionization: **EI (70 eV)**

***MSD*: Single Quad.**

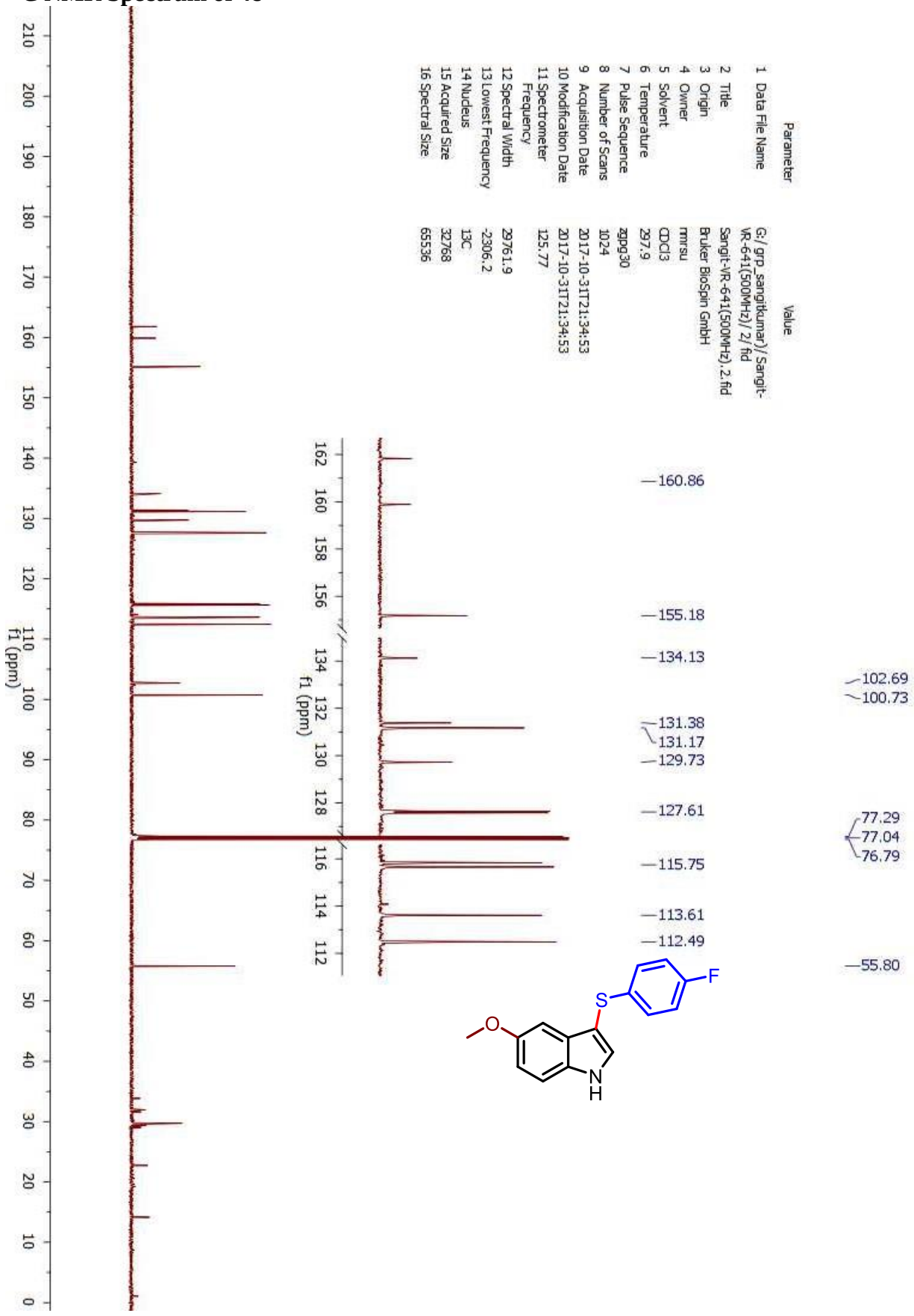


Agilent Technologies

^{1}H NMR Spectrum of 4c



^{13}C NMR Spectrum of 4c



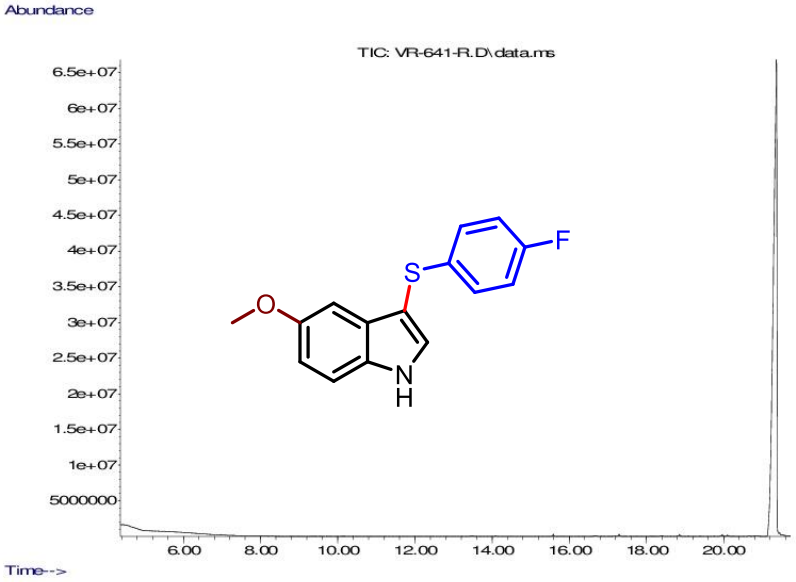
Mass Spectrum of 4c

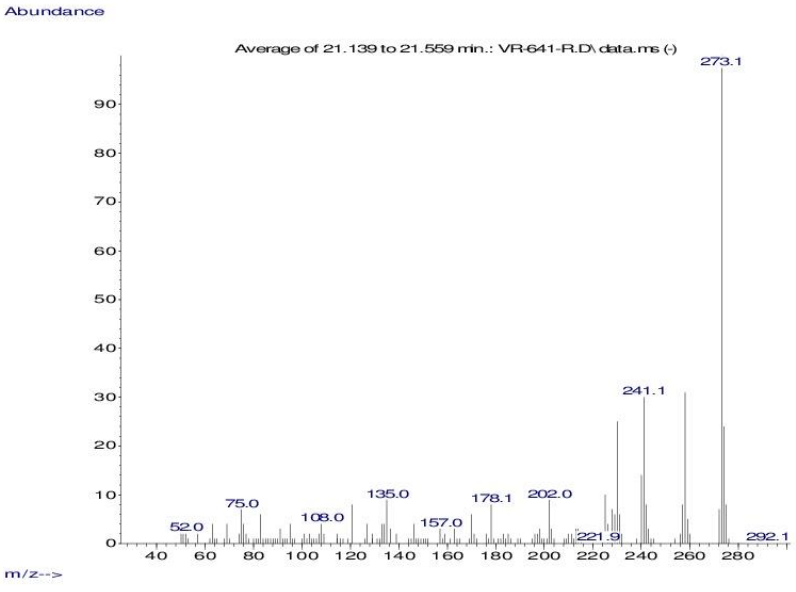
Acquisition date: 03/04/18

Sample ID: VR-641 | ***Supervisor***: Dr. S Kumar | ***Operator:*** **IISERB-CIF-Mass Facility**

Instrument: Agilent 7890A GC with 5975C MS system | ***Column*: HP-5** | ***Ionization:*** **EI (70 eV)**

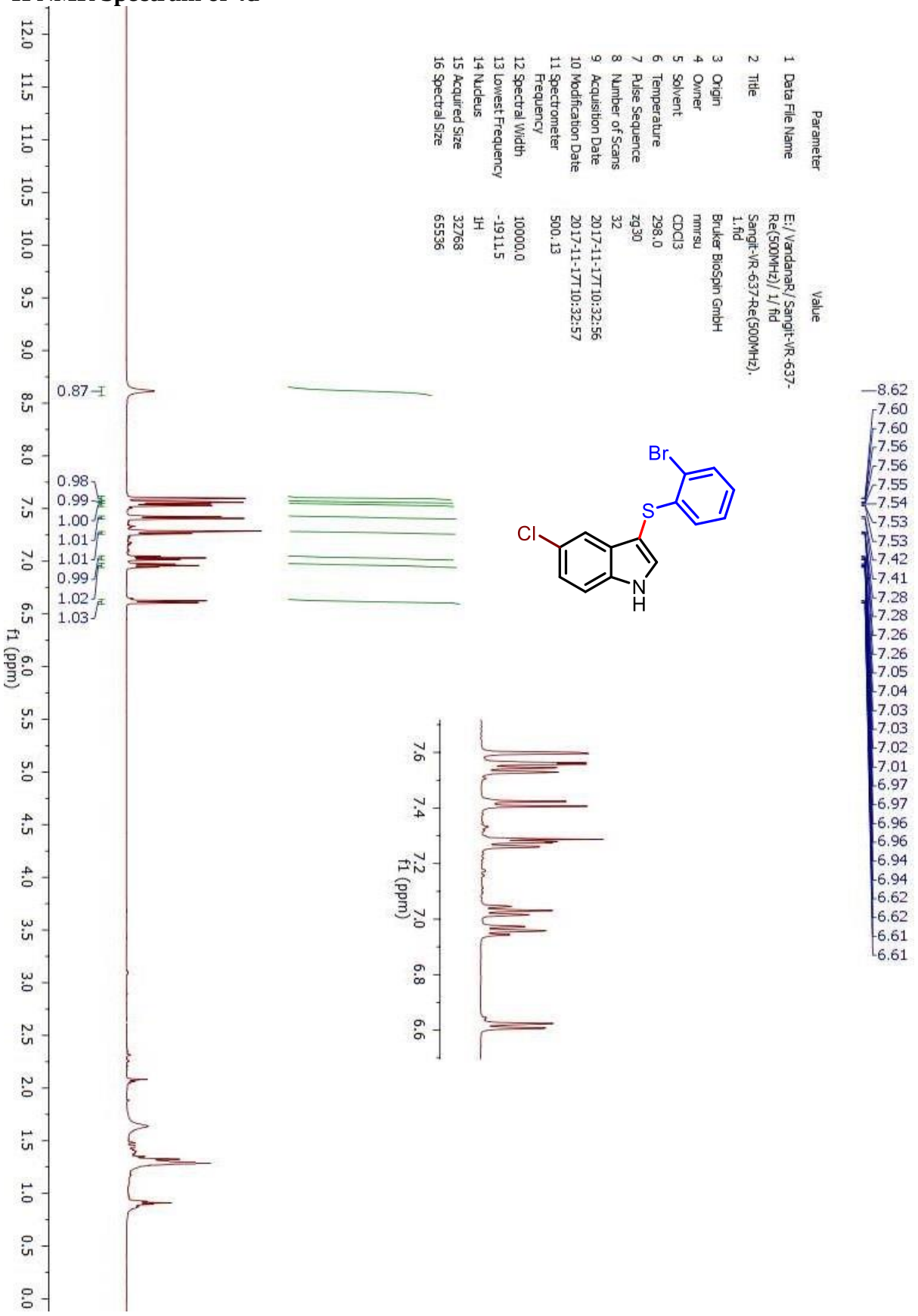
Method: **General_1_HP5_80_DEG.M** | ***MSD*: Single Quad.**



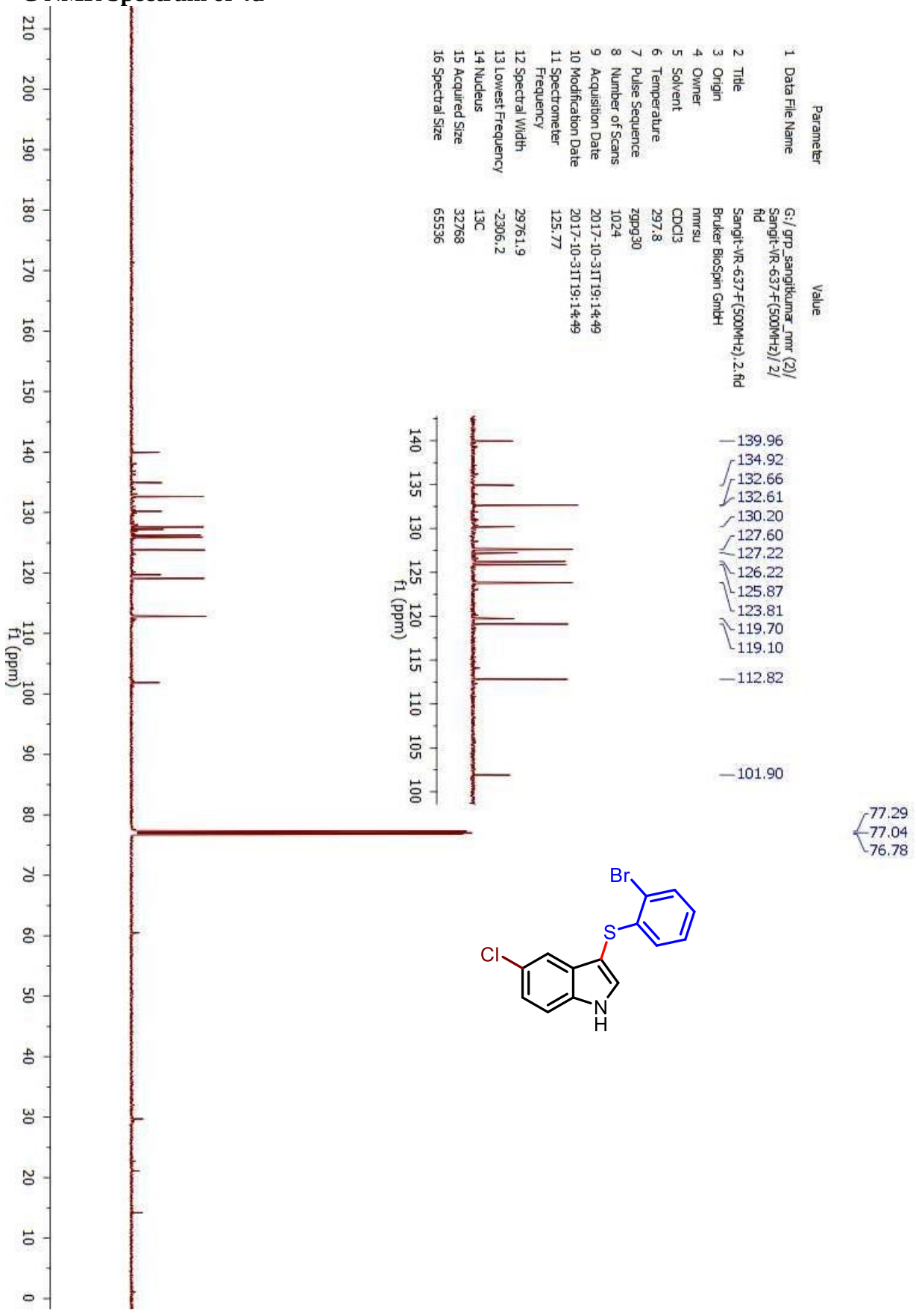


Agilent Technologies

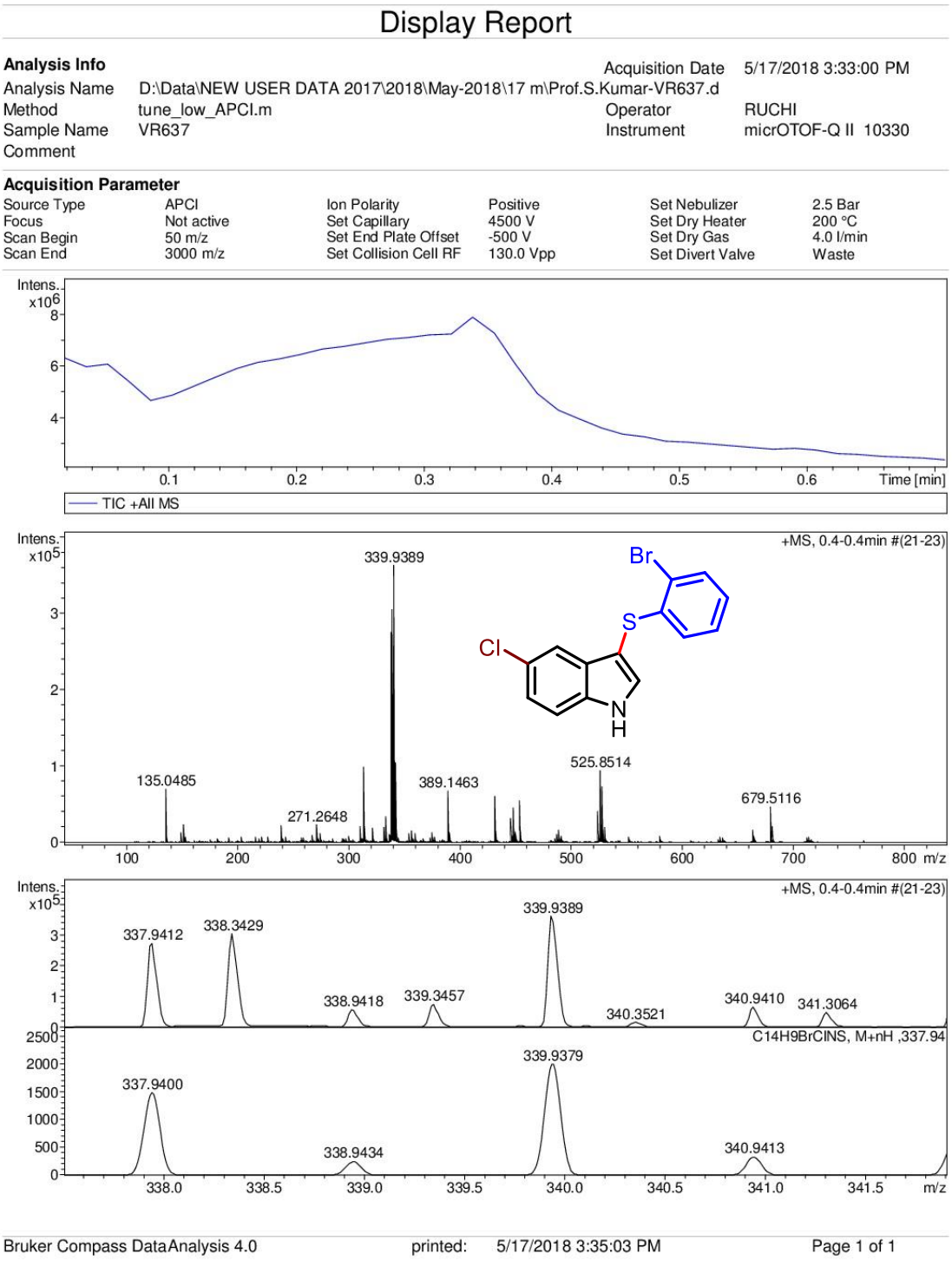
^{1}H NMR Spectrum of 4d



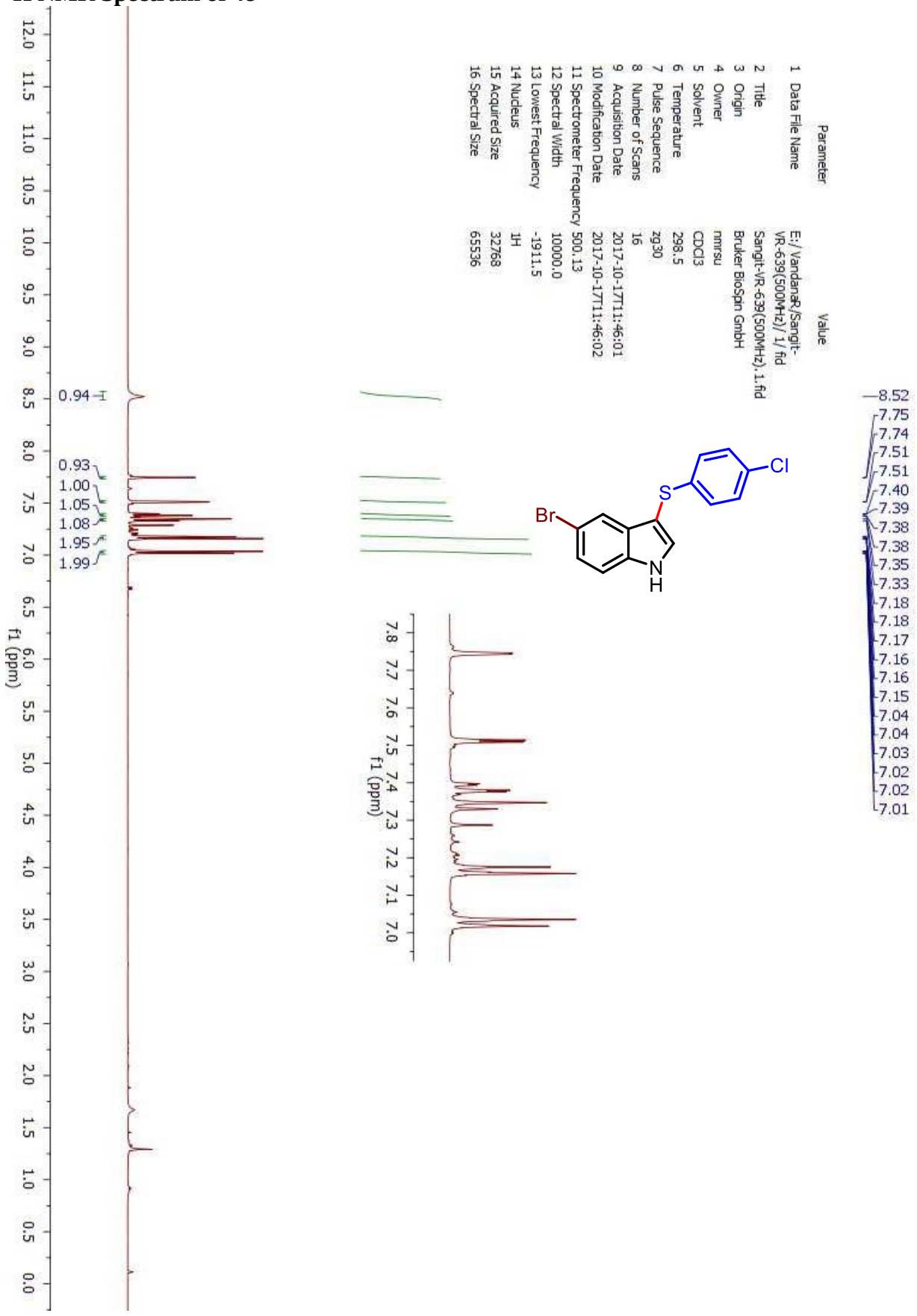
^{13}C NMR Spectrum of 4d



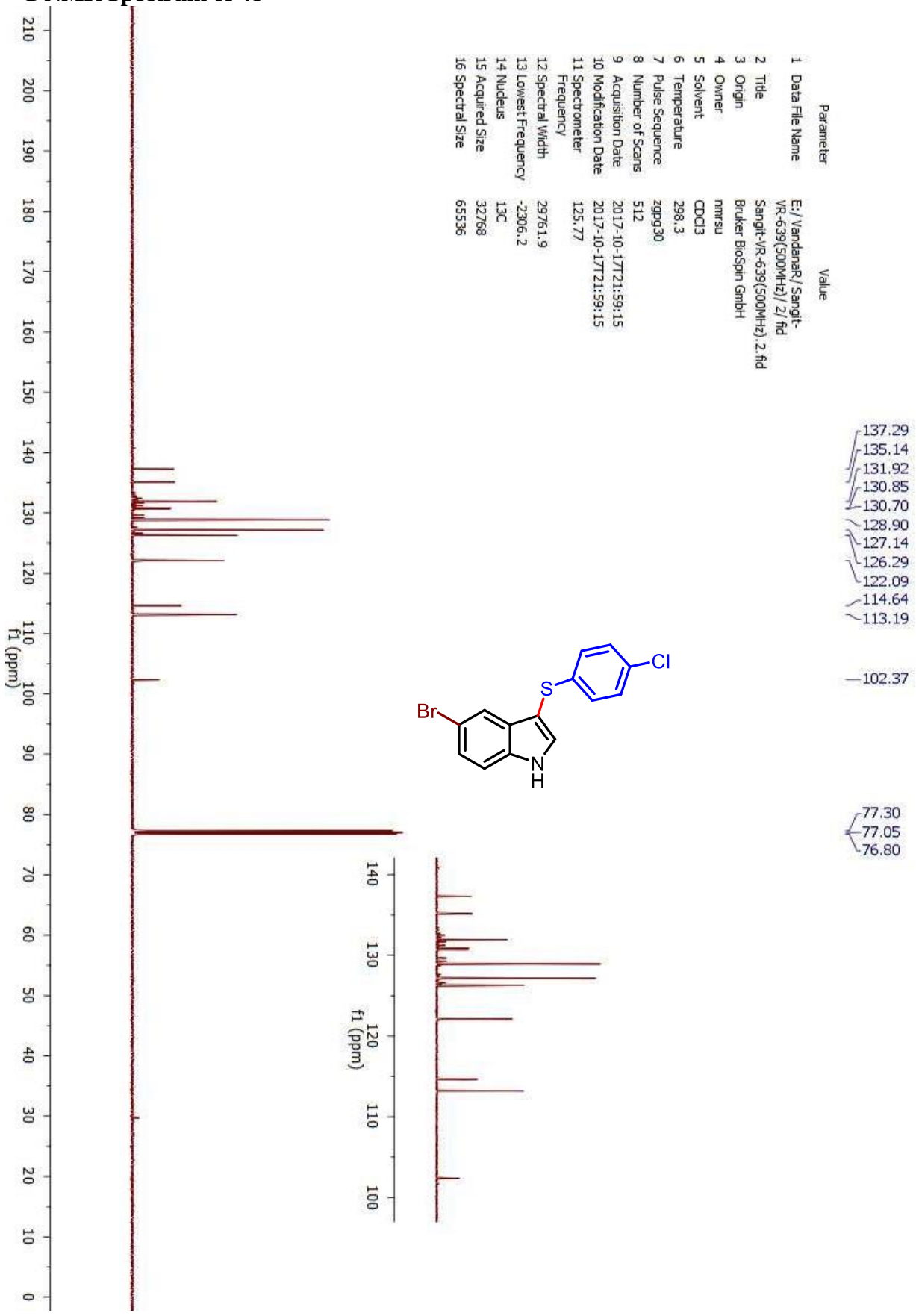
Mass Spectrum of 4d



^{1}H NMR Spectrum of 4e



^{13}C NMR Spectrum of 4e



Mass Spectrum of 4e

Acquisition date: 03/04/18

Sample ID: VR-639

Supervisor: Dr. S Kumar

Operator: **IISERB-CIF-Mass Facility**

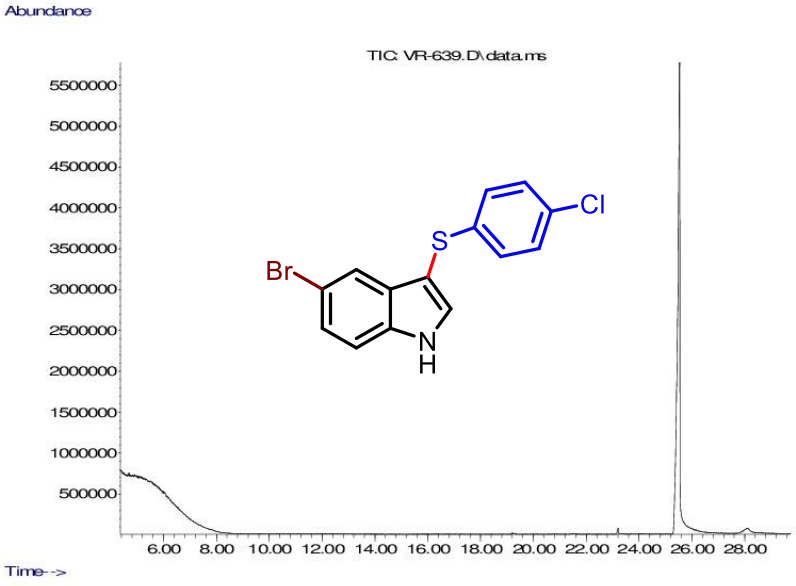
Instrument: Agilent 7890A GC with 5975C MS system

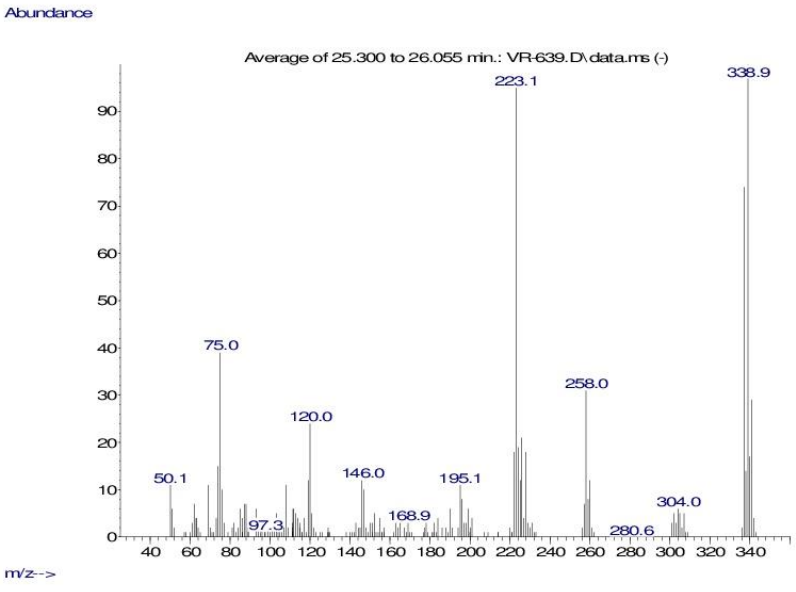
***Column*: HP-5**

Ionization: **EI (70 eV)**

Method: **General_1_HP5_80_DEG.M**

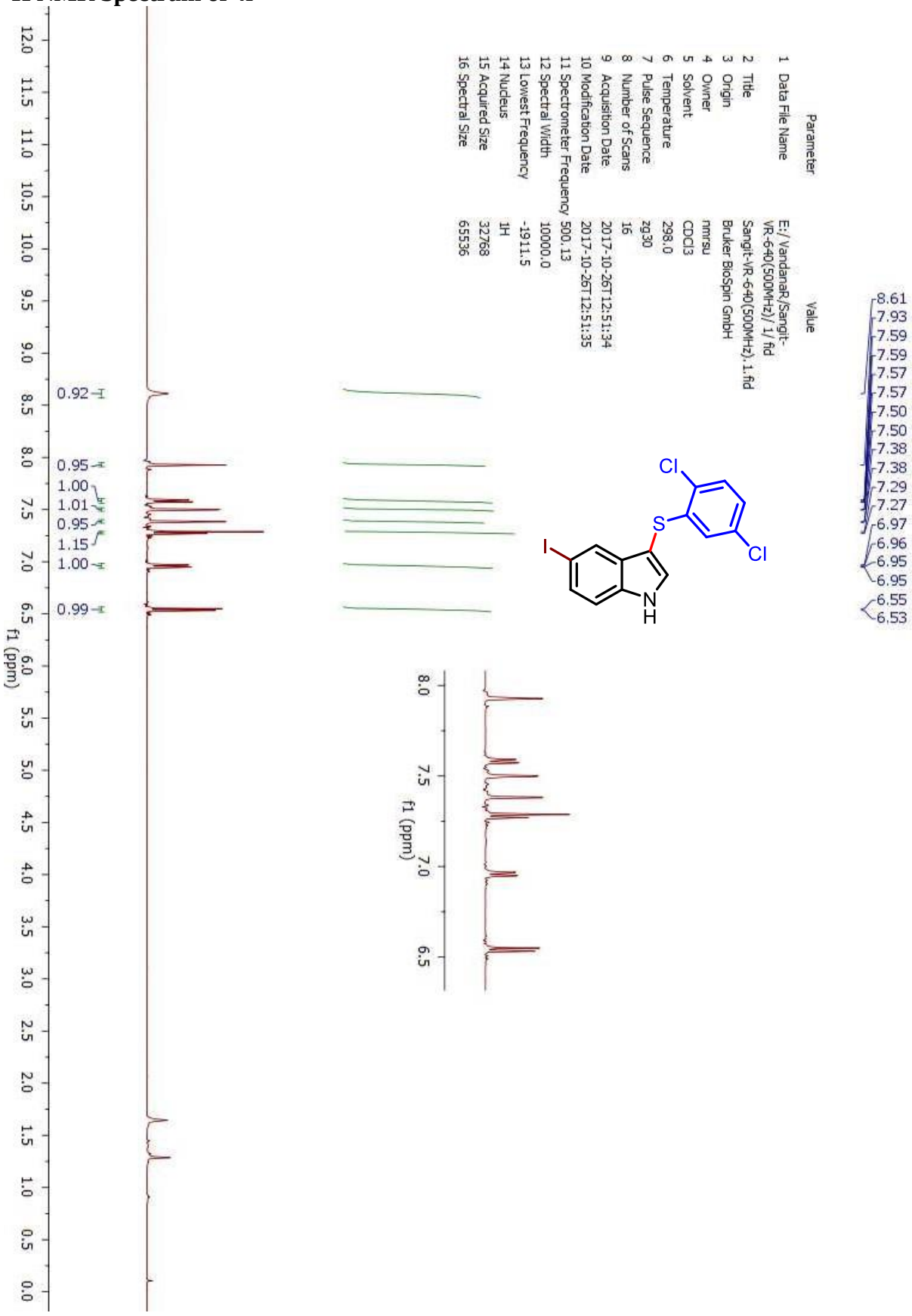
***MSD*: Single Quad.**



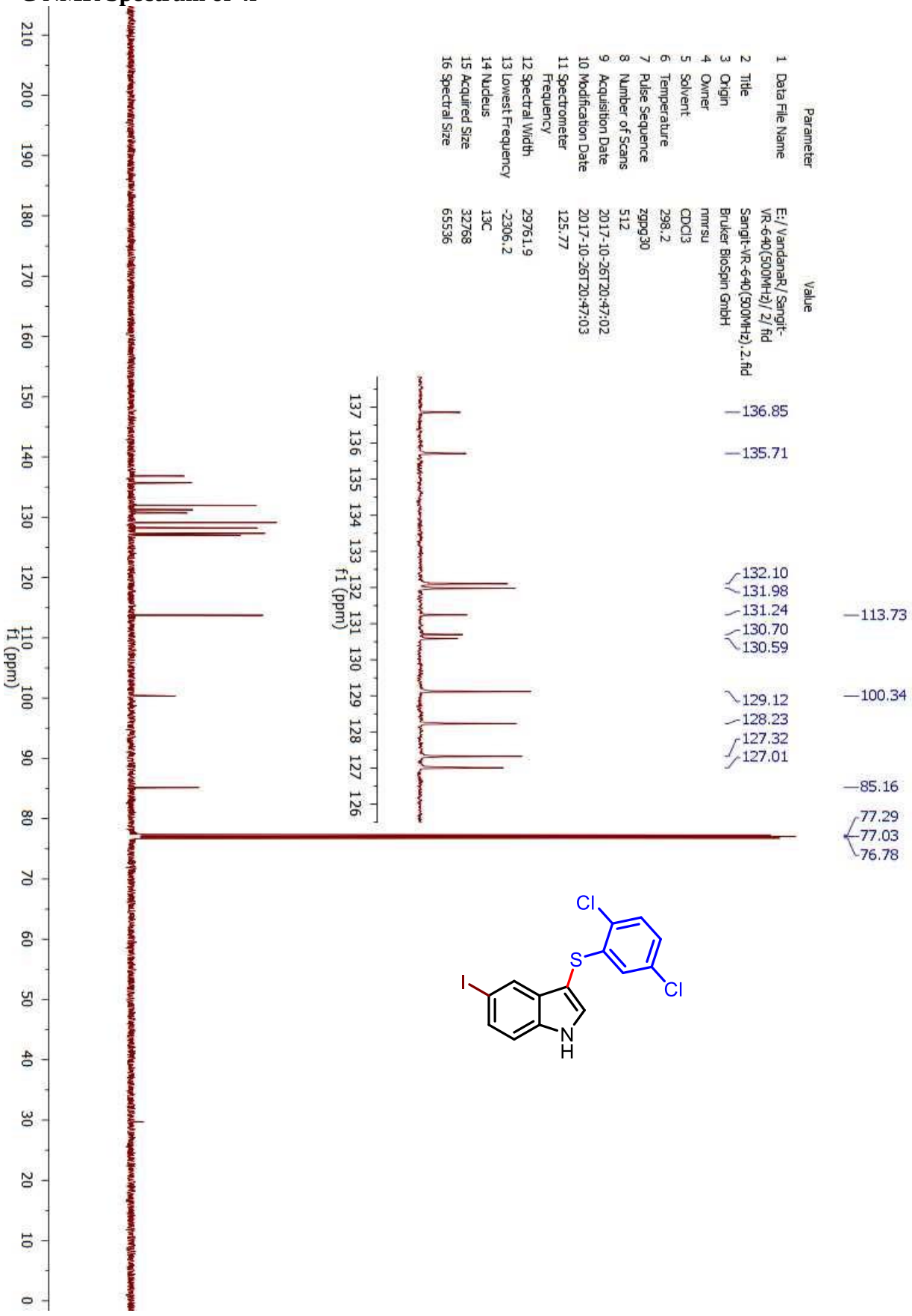


Agilent Technologies

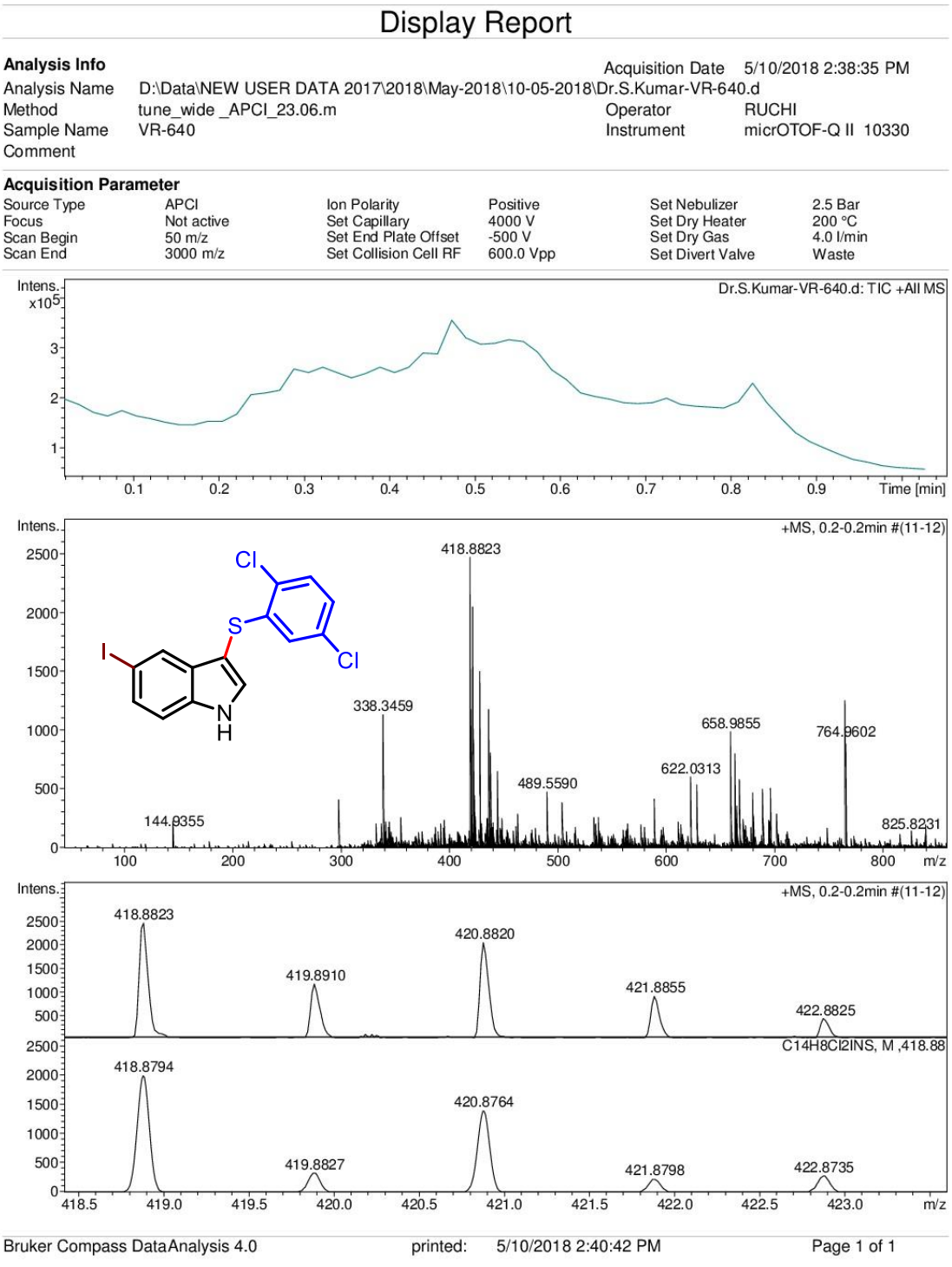
^{1}H NMR Spectrum of 4f



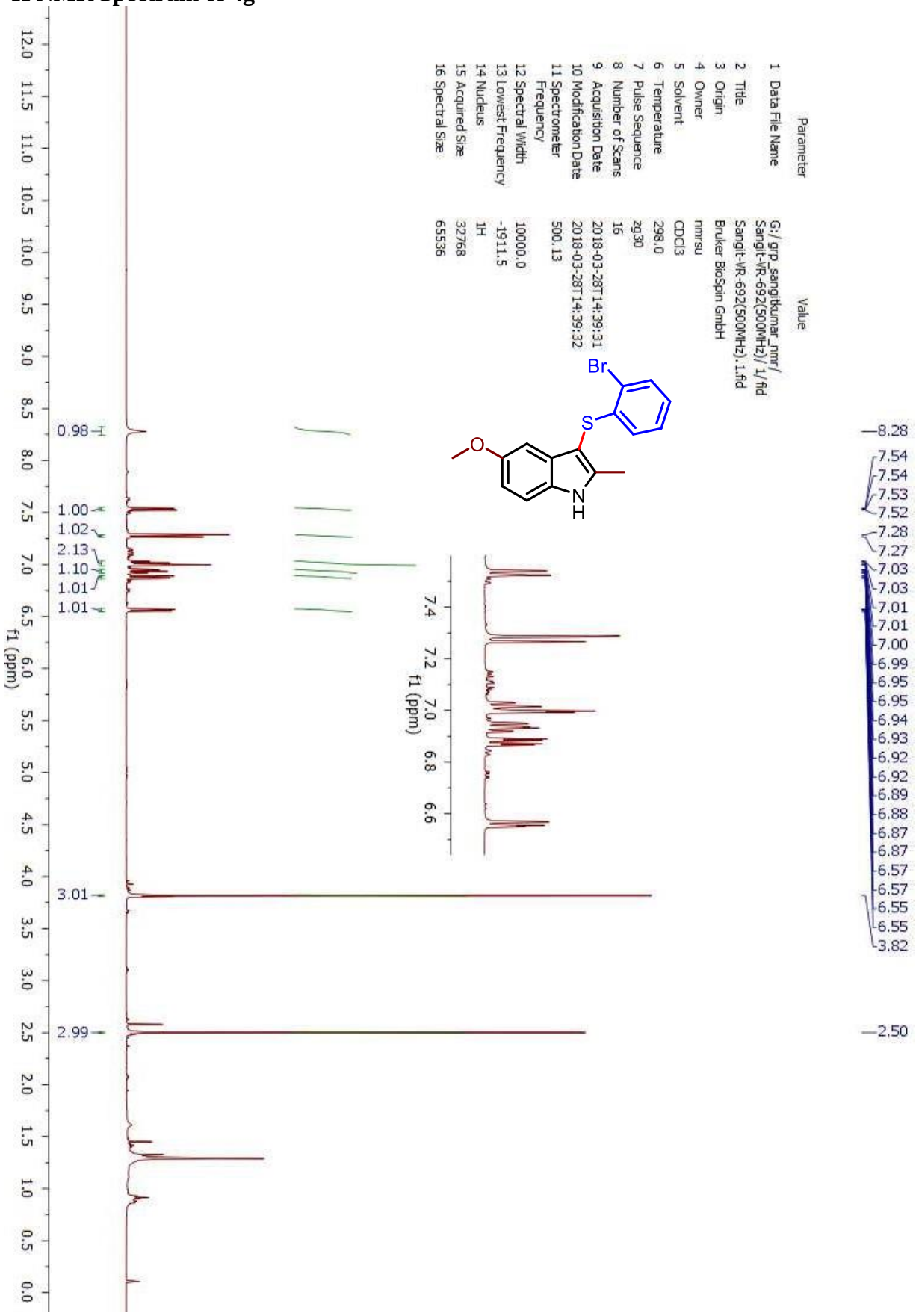
^{13}C NMR Spectrum of 4f



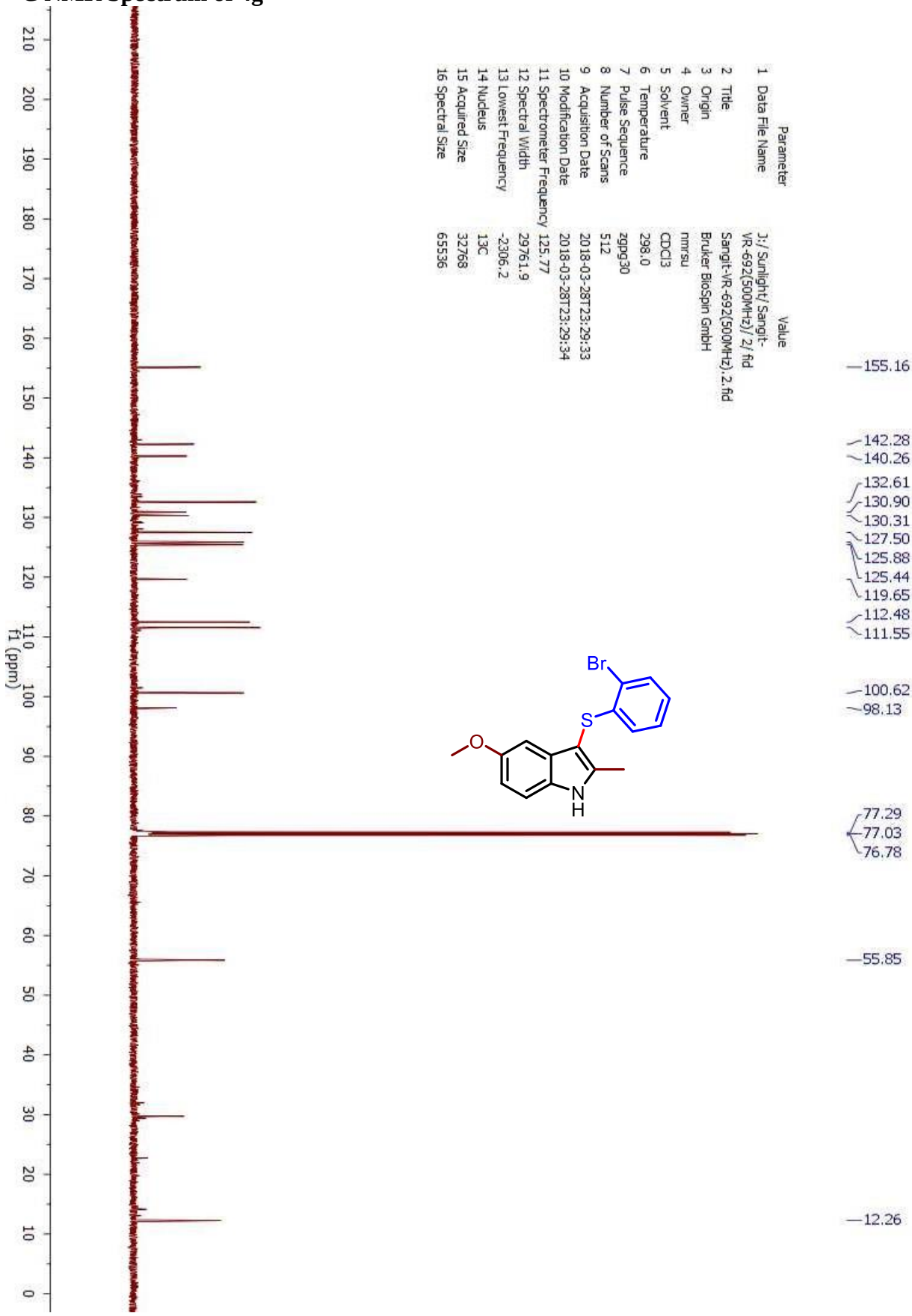
Mass Spectrum of 4f



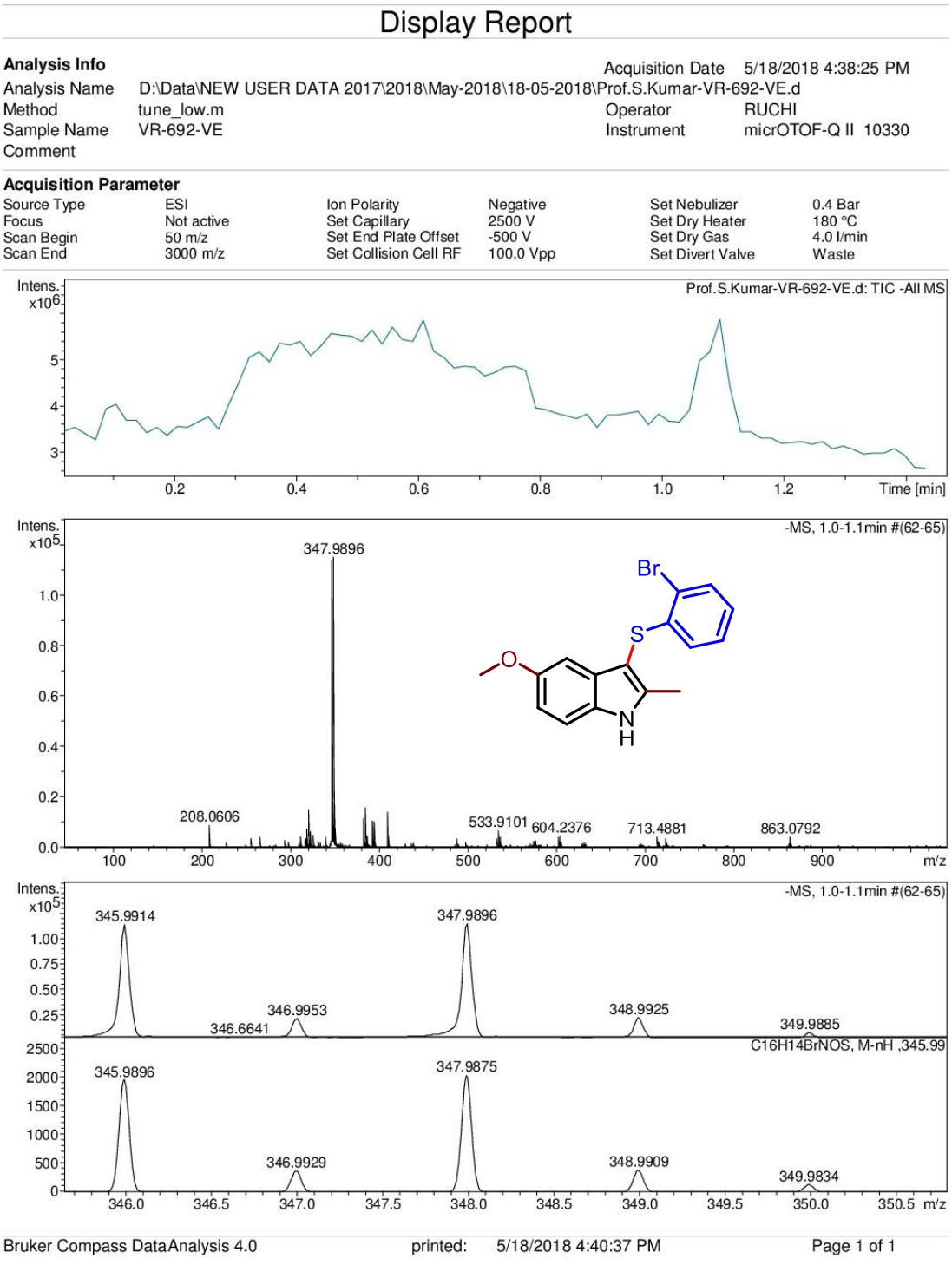
^{1}H NMR Spectrum of 4g



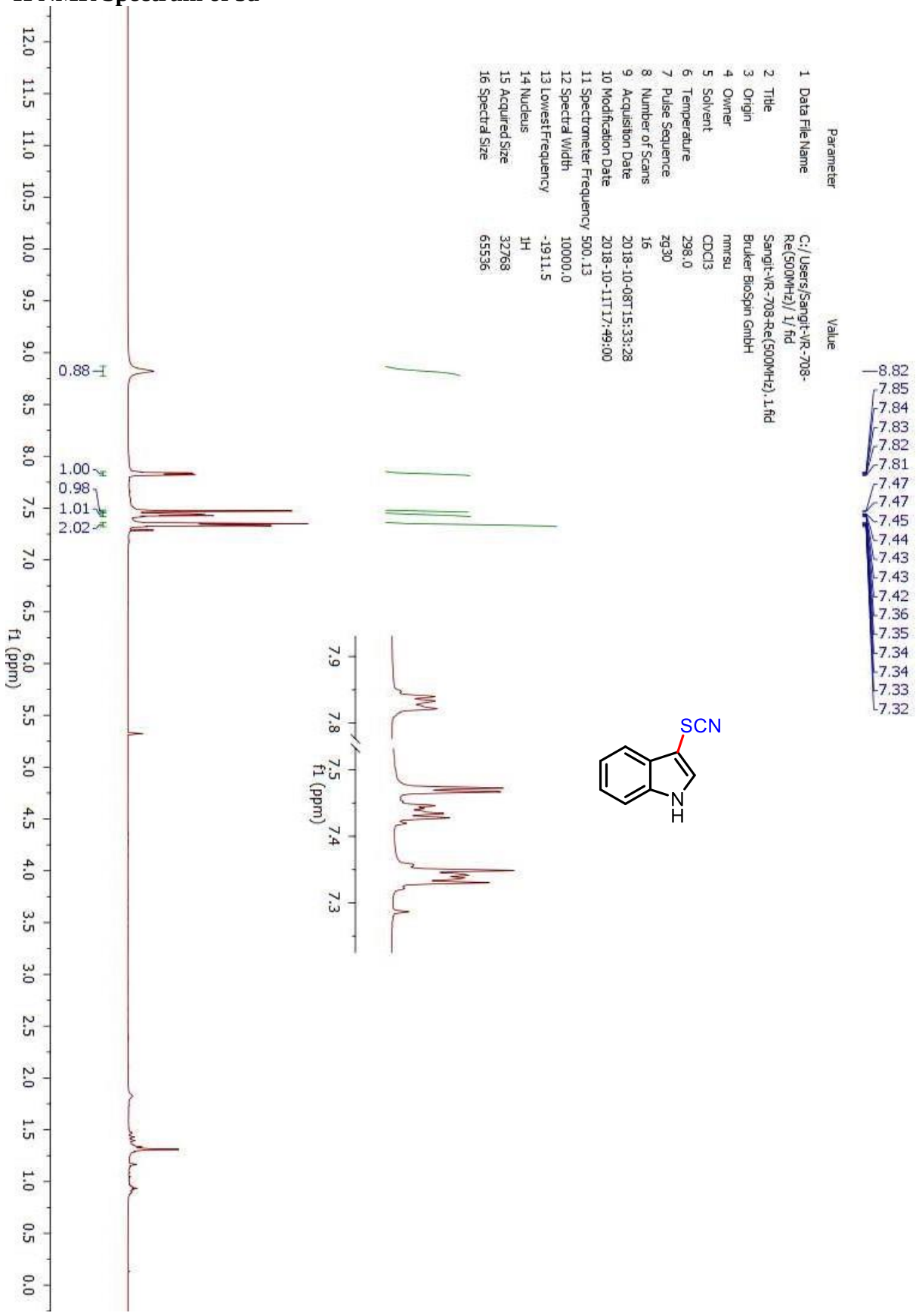
^{13}C NMR Spectrum of 4g



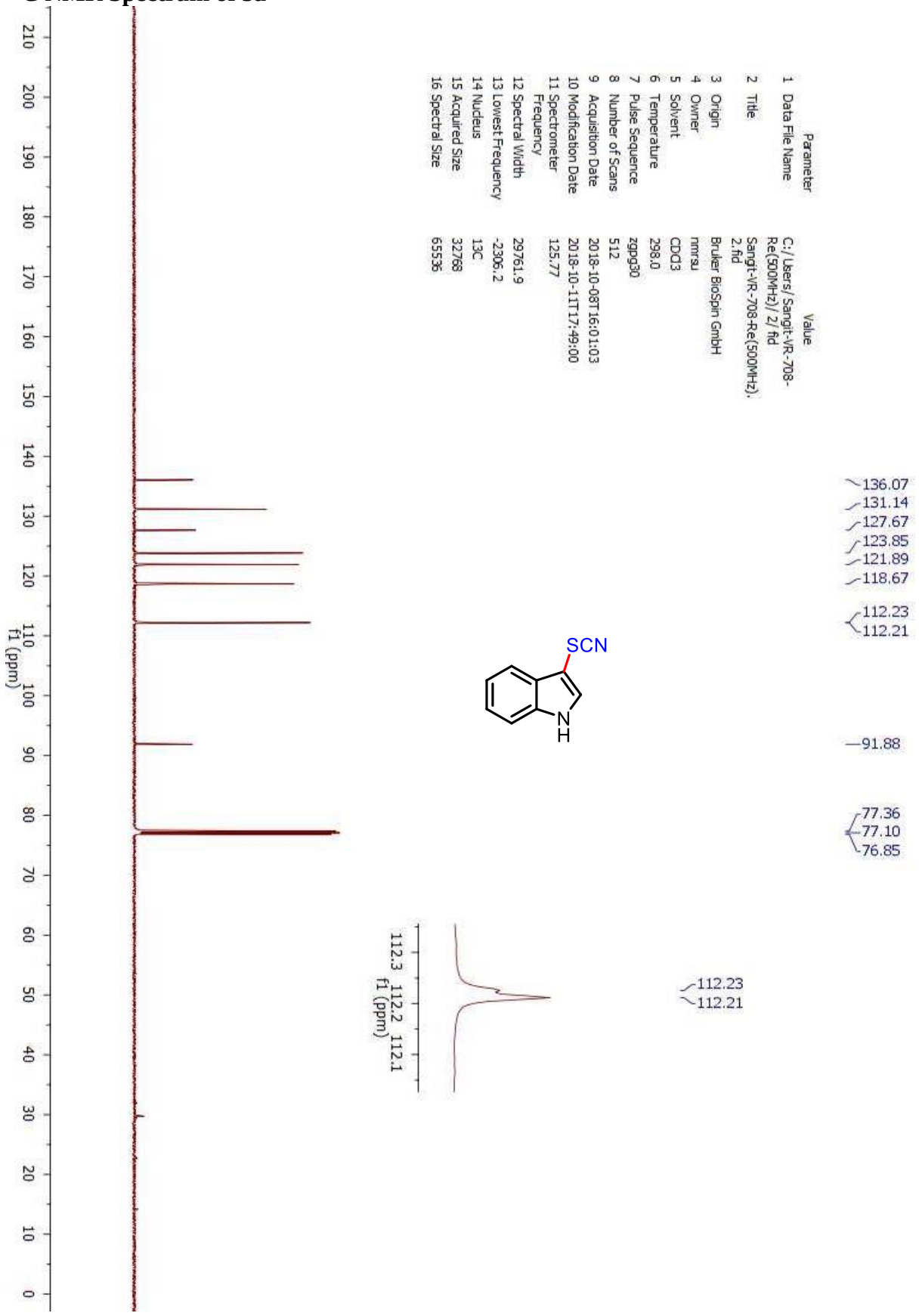
Mass Spectrum of 4g



^{1}H NMR Spectrum of 5a



^{13}C NMR Spectrum of 5a



Mass Spectrum of 5a

Acquisition date: 08/10/18

Sample ID: VR-708

Supervisor: Dr. S Kumar

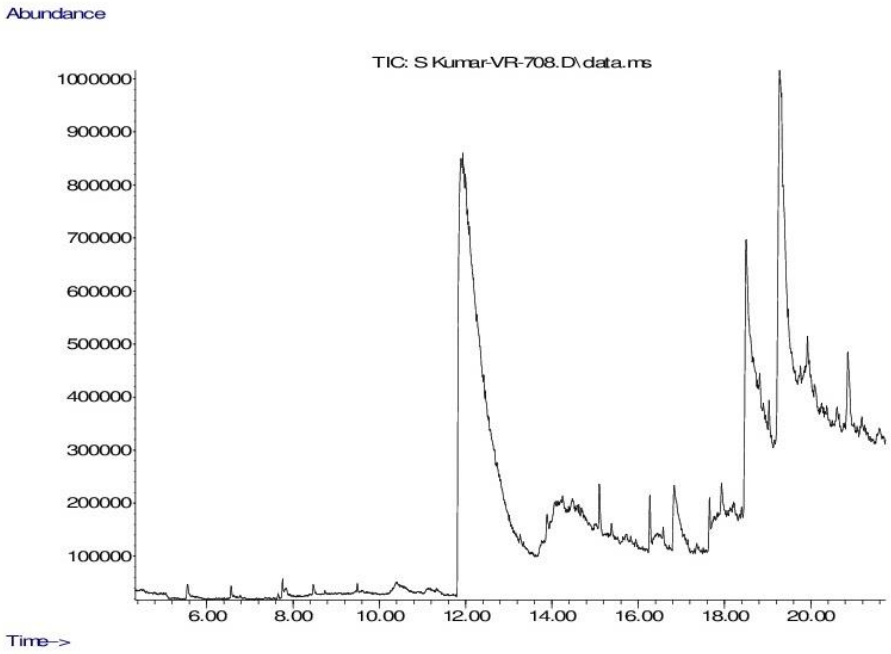
Operator: **IISERB-CIF-Mass Facility**

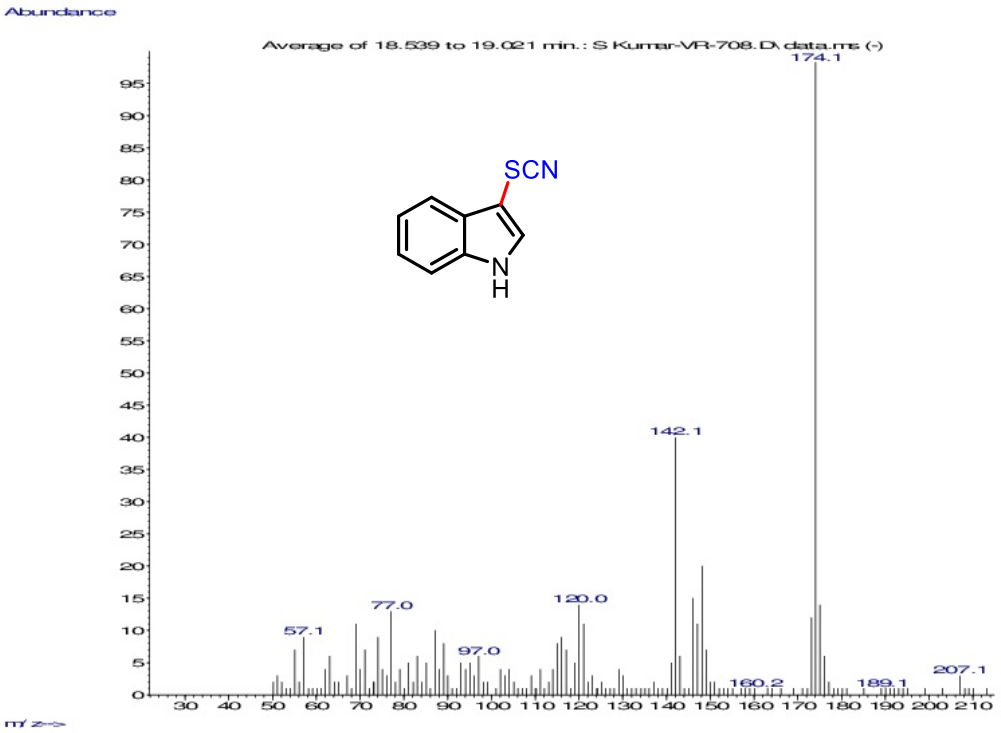
Instrument: Agilent 7890A GC with 5975C MS system

***Column*: HP-5**

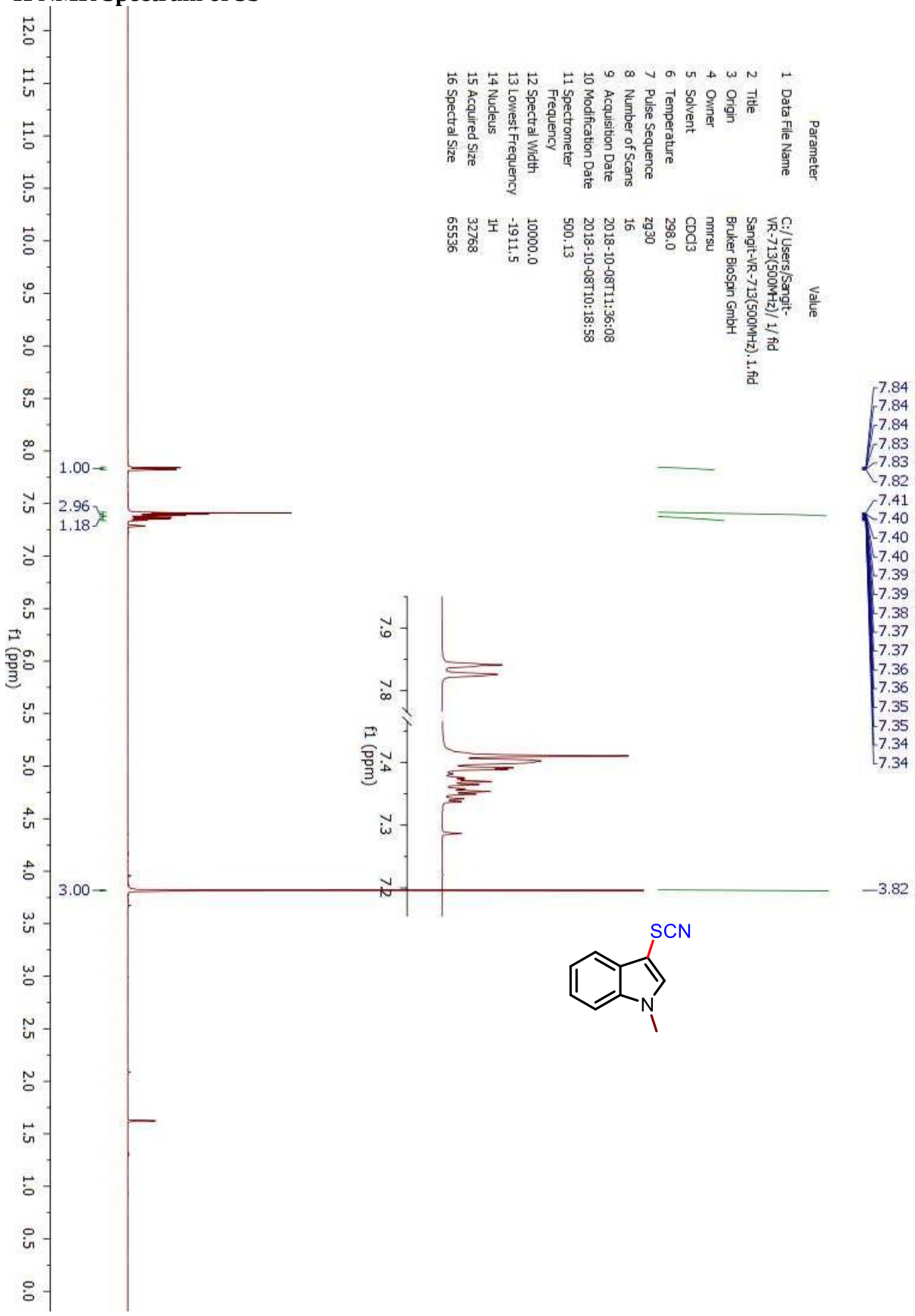
Method: **General_1_HP5_80_DEG.M**

***MSD*: Single Quad.**

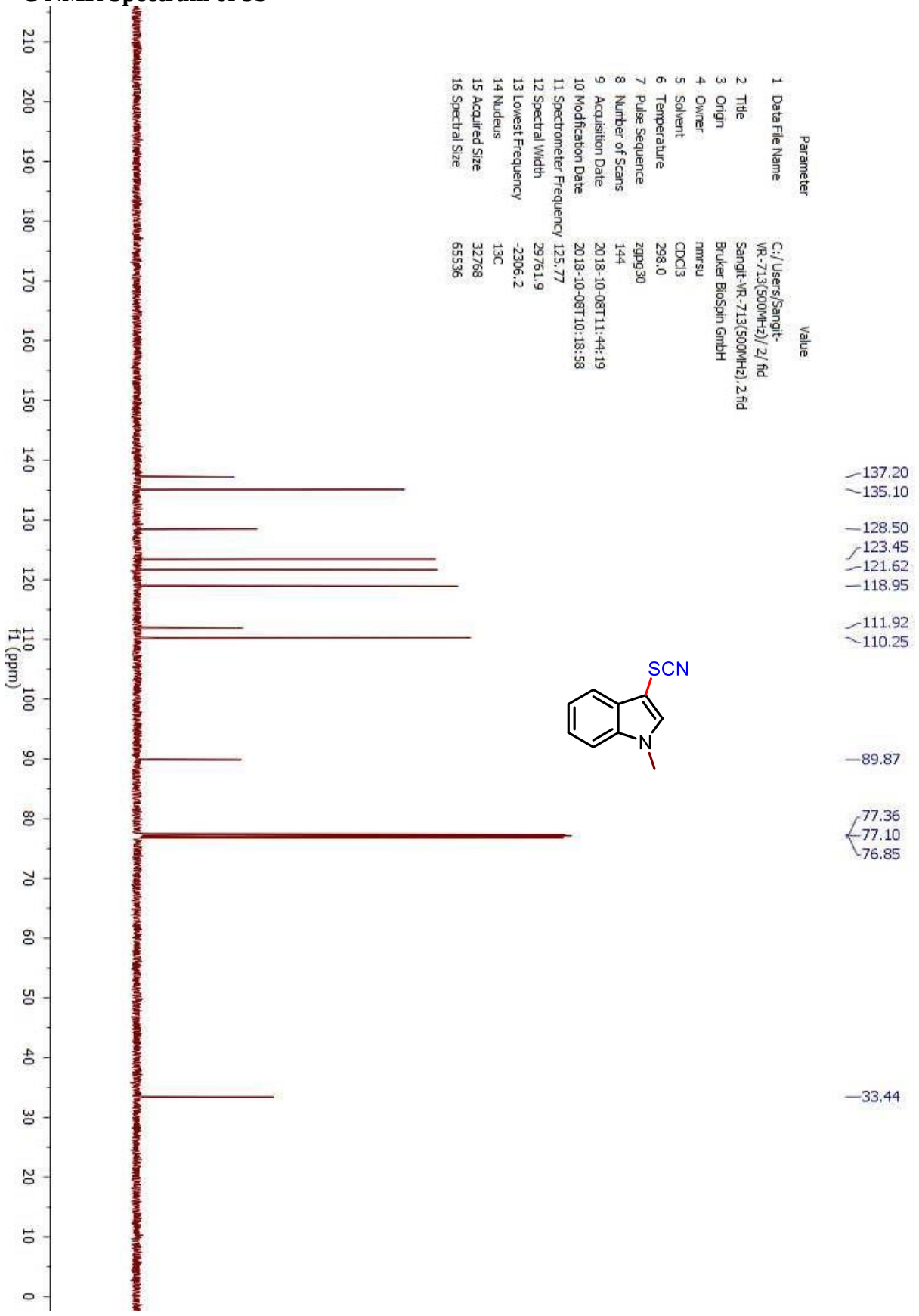




^{1}H NMR Spectrum of 5b



^{13}C NMR Spectrum of 5b



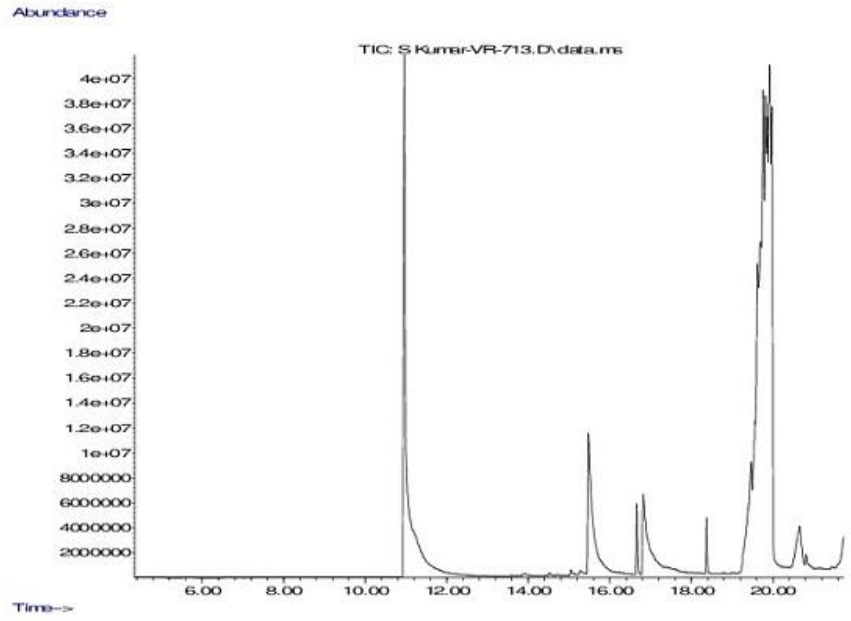
Mass Spectrum of 5b

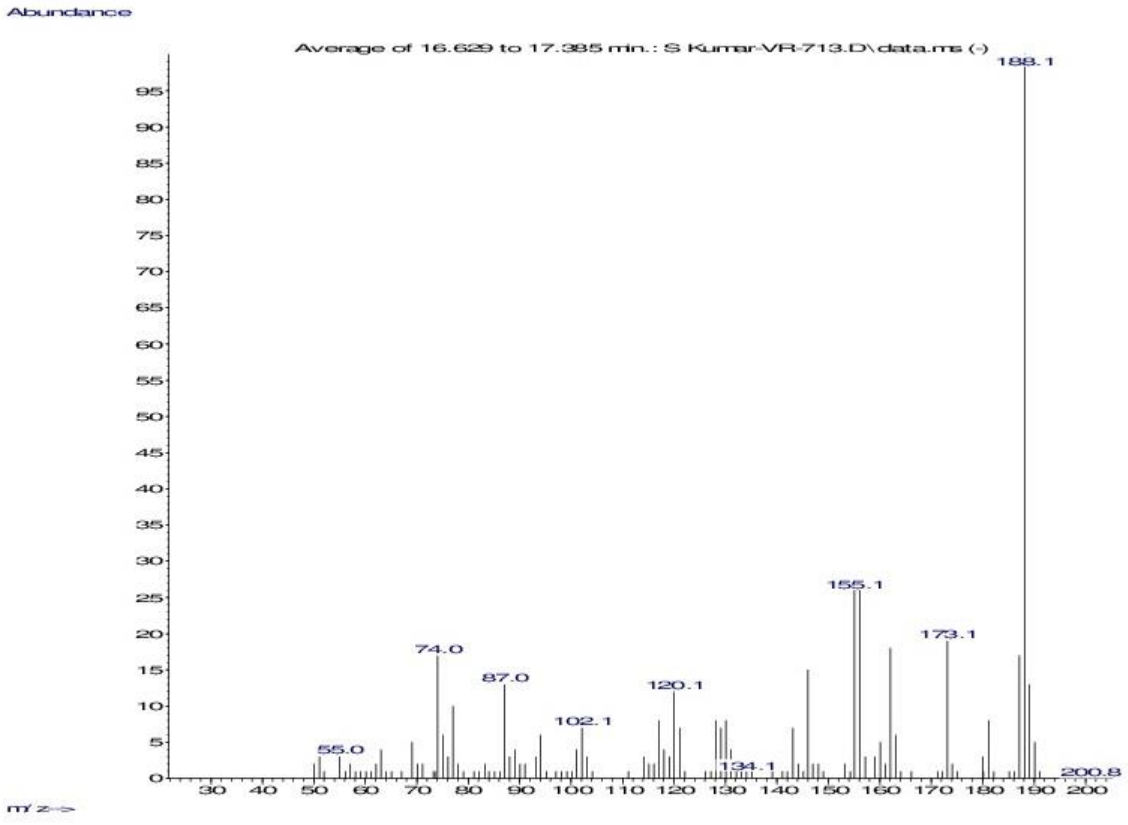
Acquisition date: 11/10/18

Sample ID: VR-713 | *Supervisor*: Dr. S Kumar | *Operator:* IISERB-CIF-Mass Facility

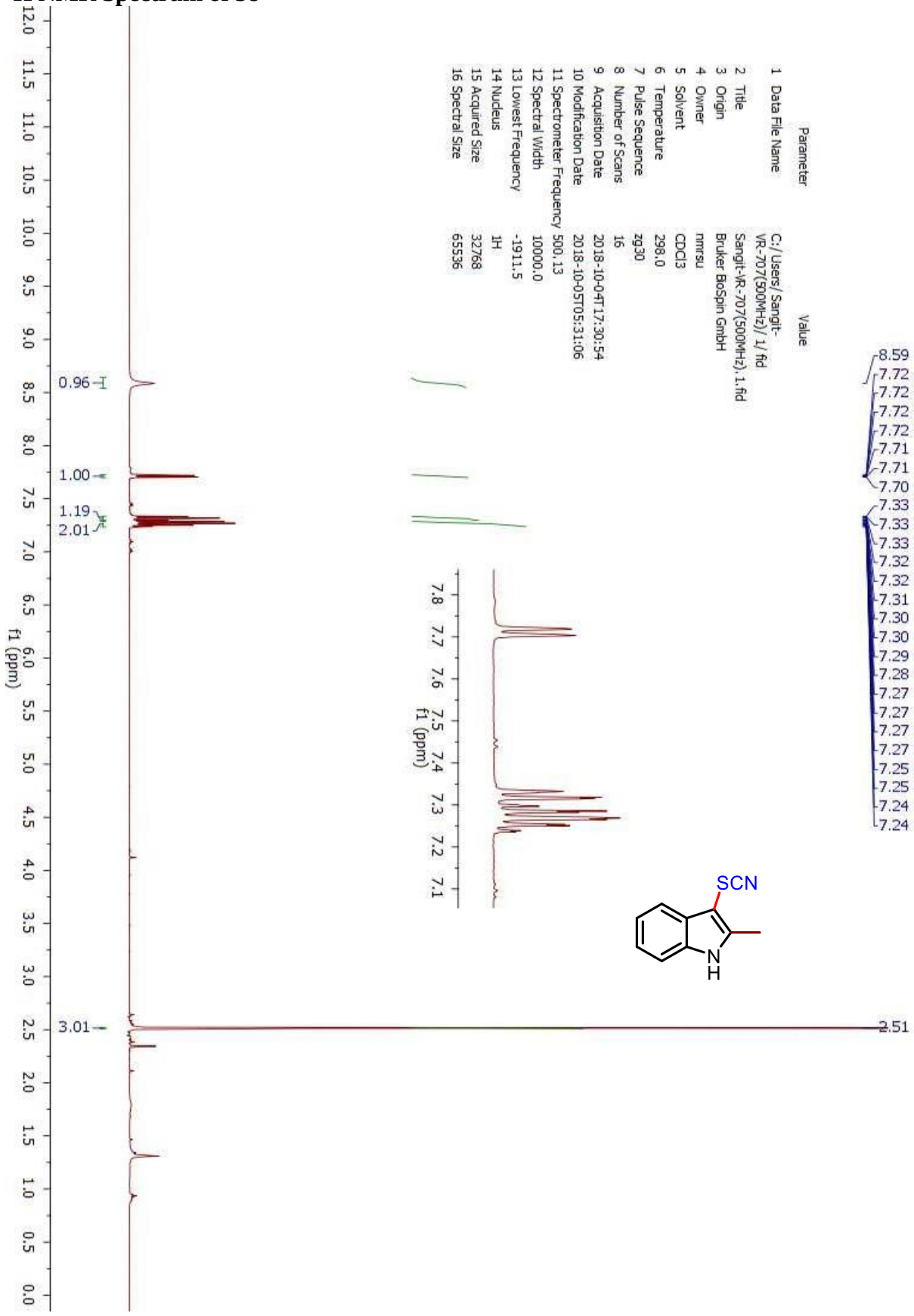
Instrument: Agilent 7890A GC with 5975C MS system | *Column*: HP-5 | *Ionization:* EI (70 eV)

Method: General_1_HP5_80_DEG.M | *MSD*: Single Quad.

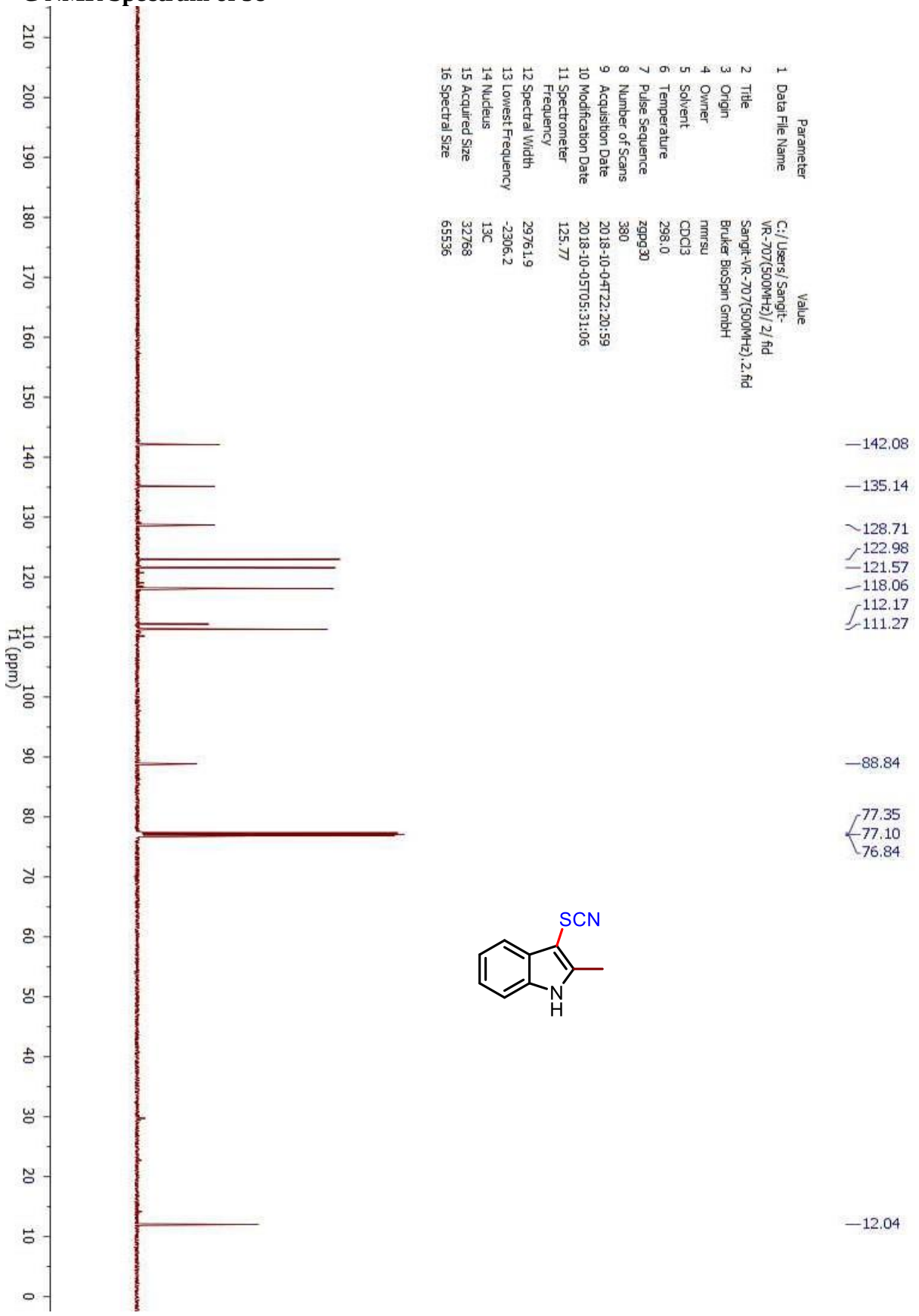




^{1}H NMR Spectrum of 5c



^{13}C NMR Spectrum of 5c



Mass Spectrum of 5c

Acquisition date: 11/10/18

Sample ID: VR-707

Supervisor: Dr. S Kumar

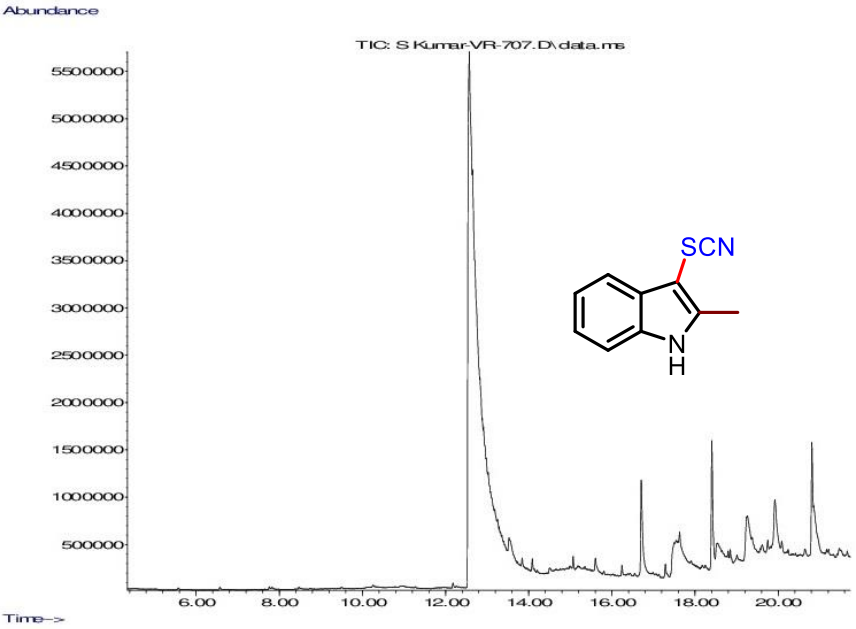
Operator: **IISERB-CIF-Mass Facility**

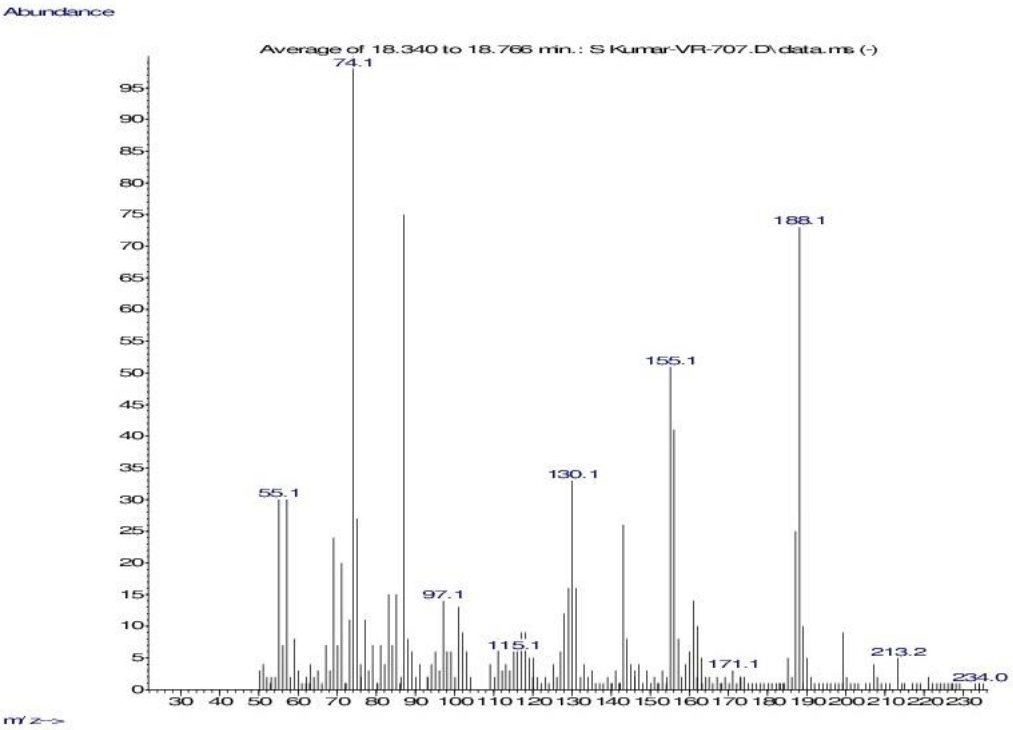
Instrument: Agilent 7890A GC with 5975C MS system

***Column*: HP-5**

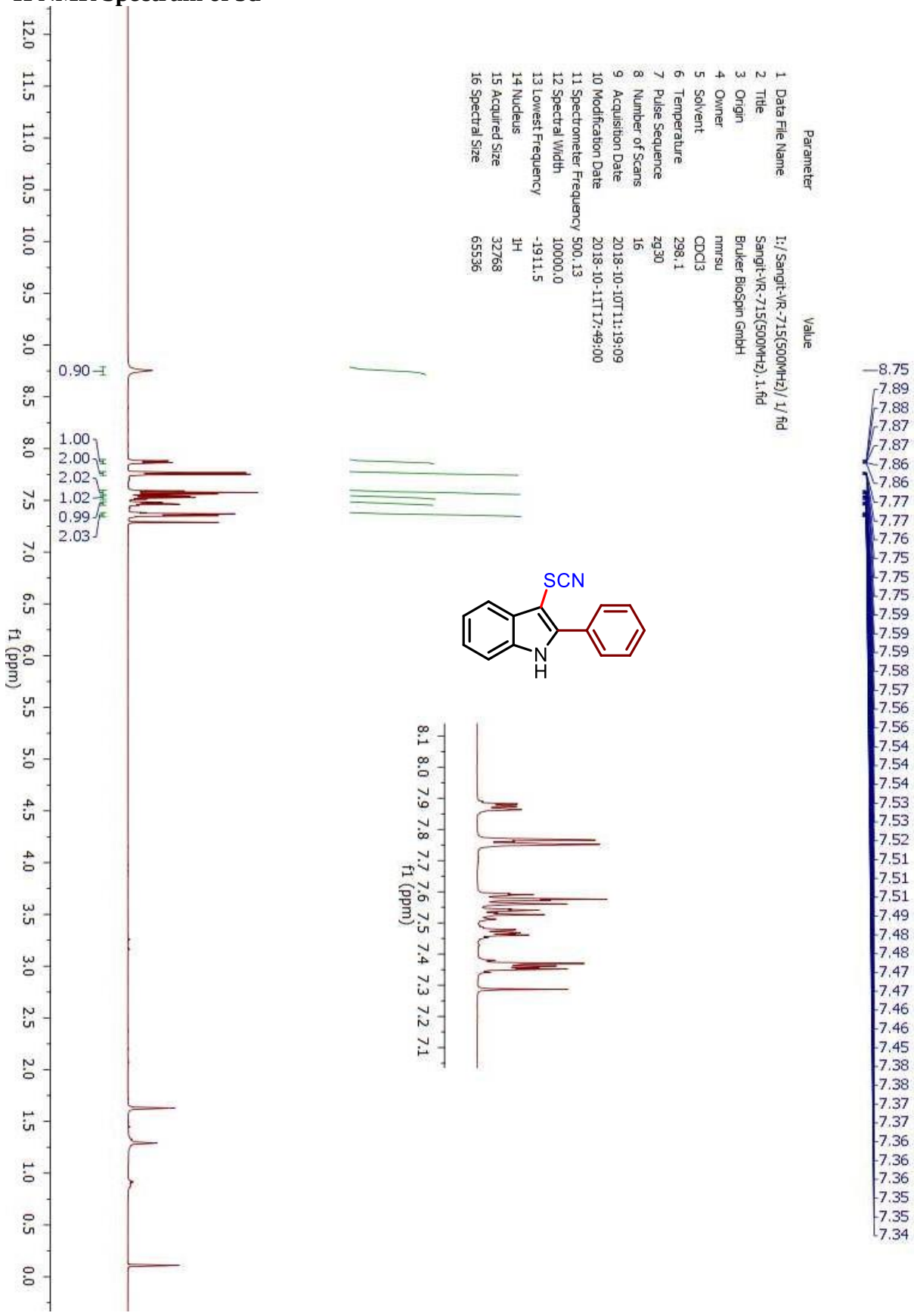
Method: **General_1_HP5_80_DEG.M**

***MSD*: Single Quad.**

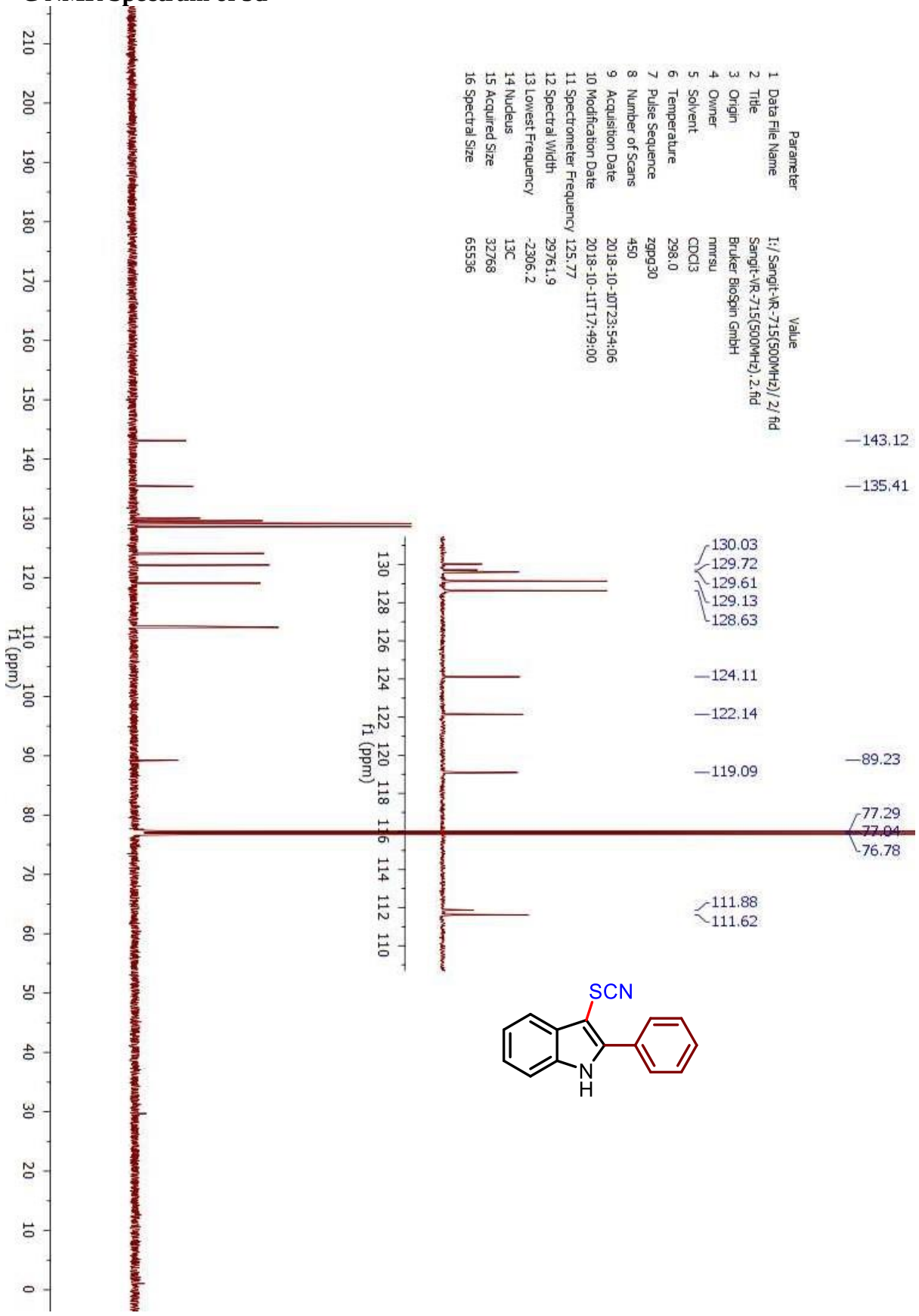




^{1}H NMR Spectrum of 5d



^{13}C NMR Spectrum of 5d



Mass Spectrum of 5d

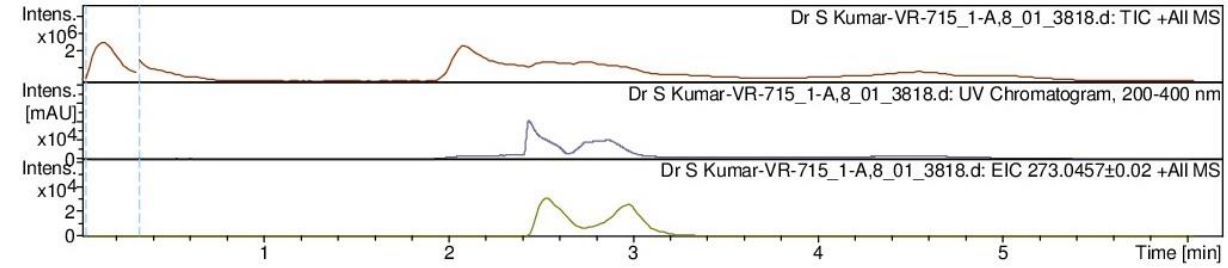
Display Report

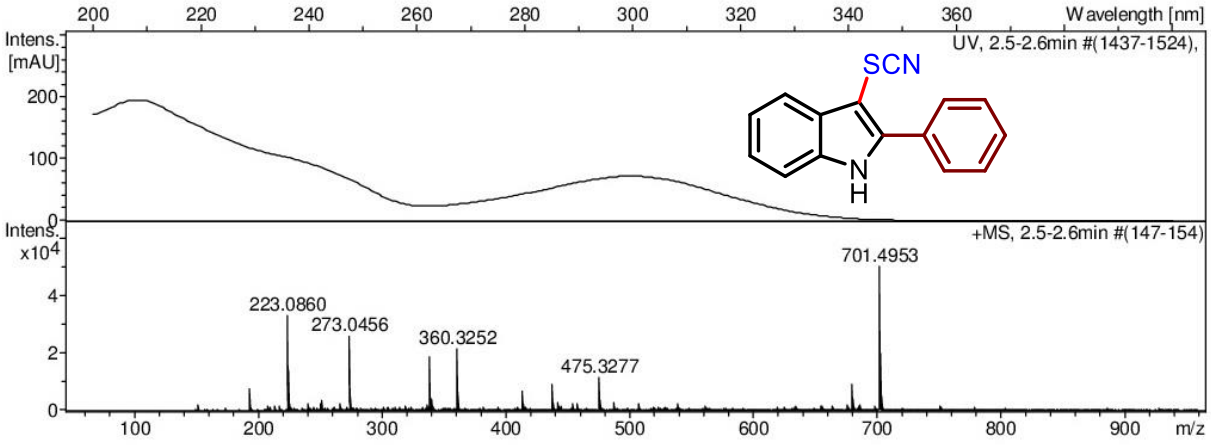
Analysis Info

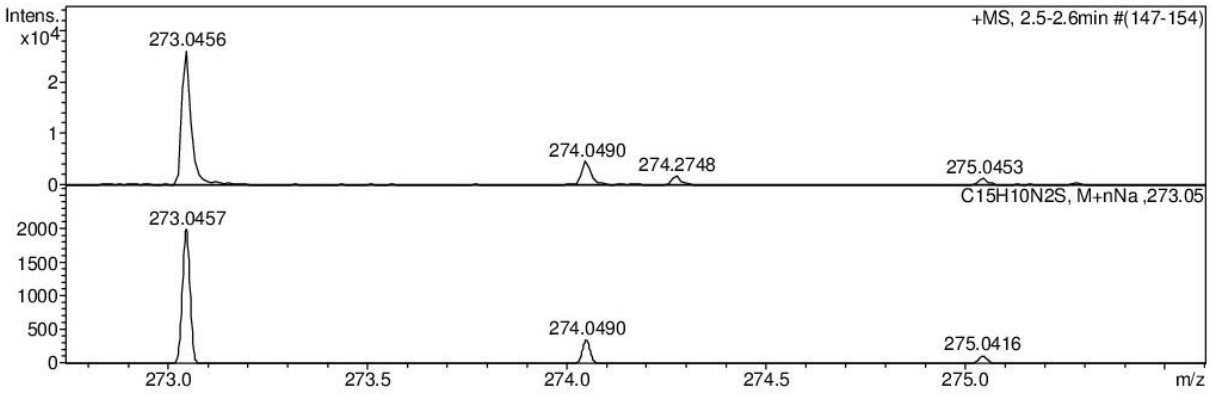
Analysis Name	D:\Data\NEW USER DATA 2017\2018\OCTOBER 2018\15 0ct\Dr S Kumar-VR-715_1-A,8_01_3818.d	Acquisition Date	10/15/2018 2:04:11 PM
Method	hrlcms-20 sept.m	Operator	RUCHI
Sample Name	Dr S Kumar-VR-715	Instrument	micrOTOF-Q II 10330
Comment			

Acquisition Parameter

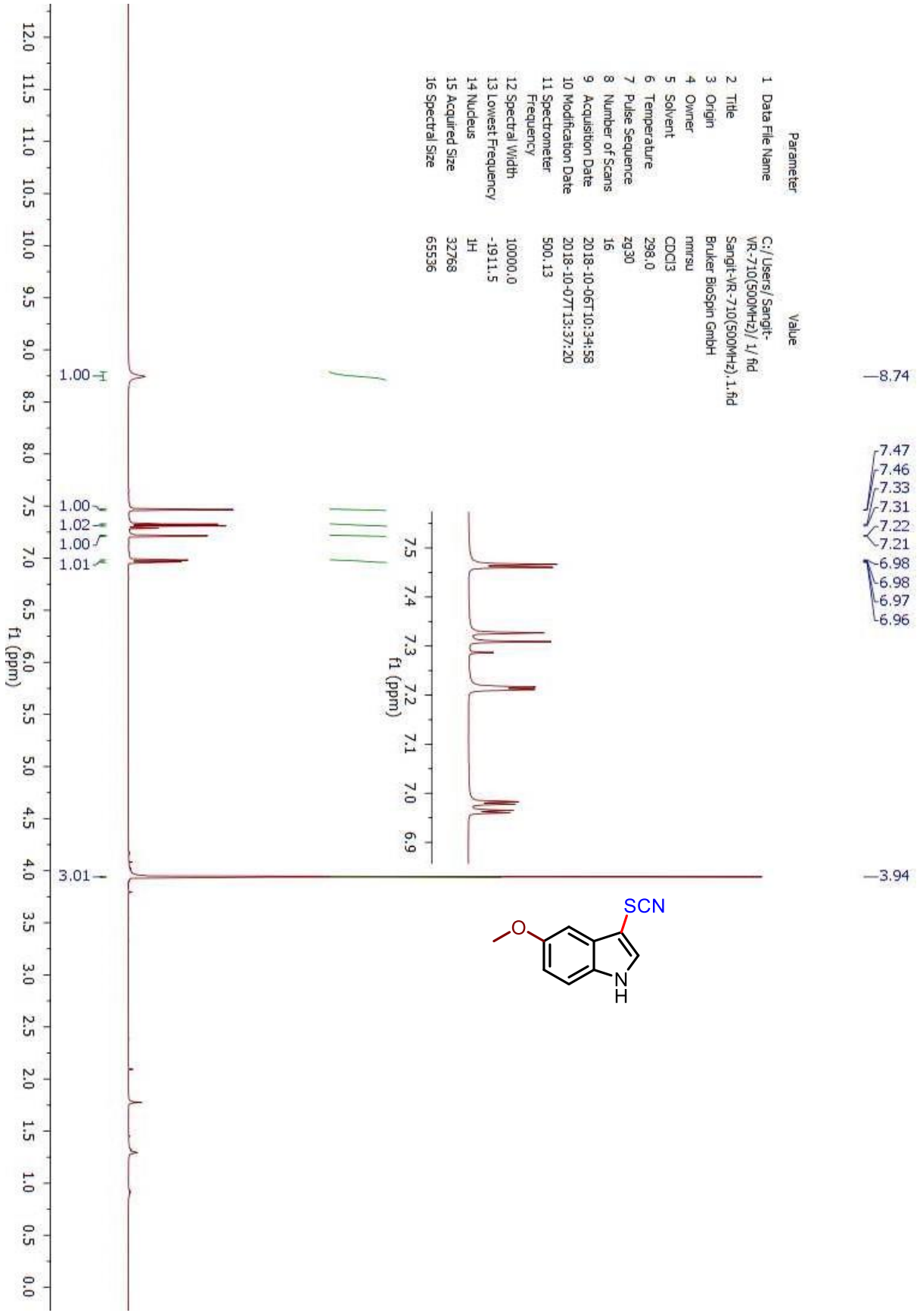
Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.2 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	7.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste



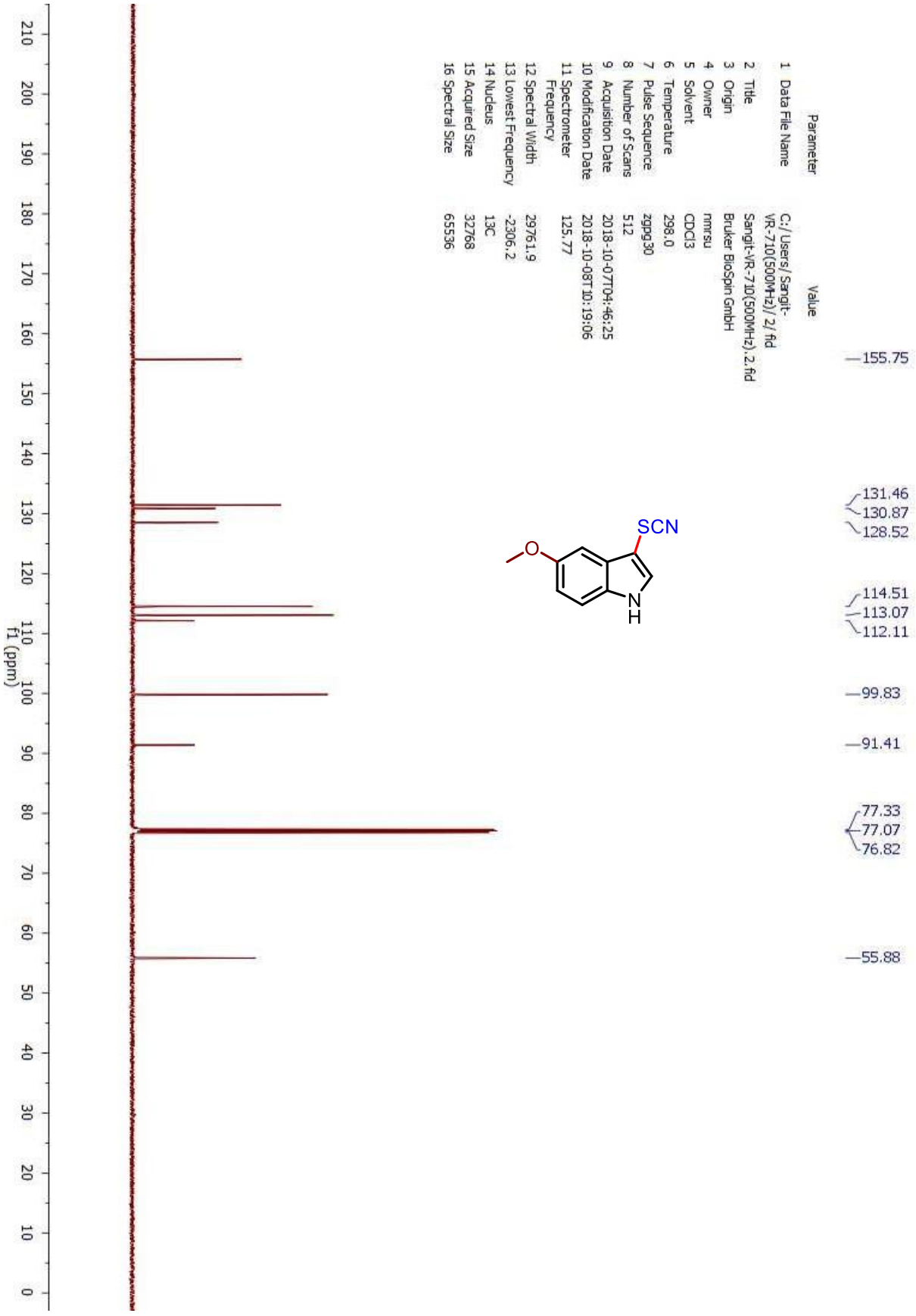




^{1}H NMR Spectrum of 5e



^{13}C NMR Spectrum of 5e



Mass Spectrum of 5e

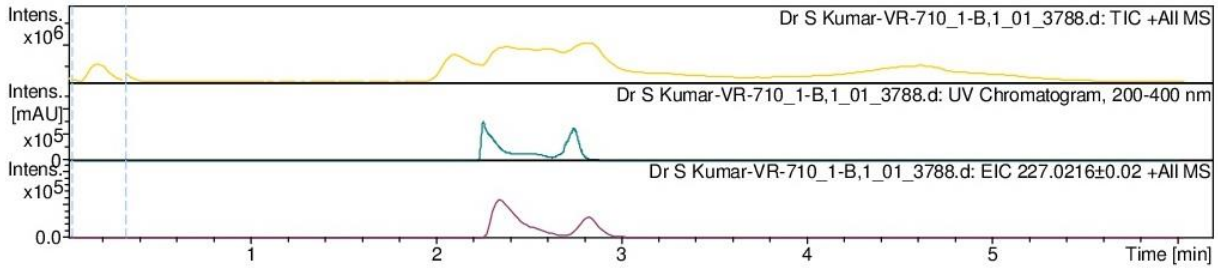
Display Report

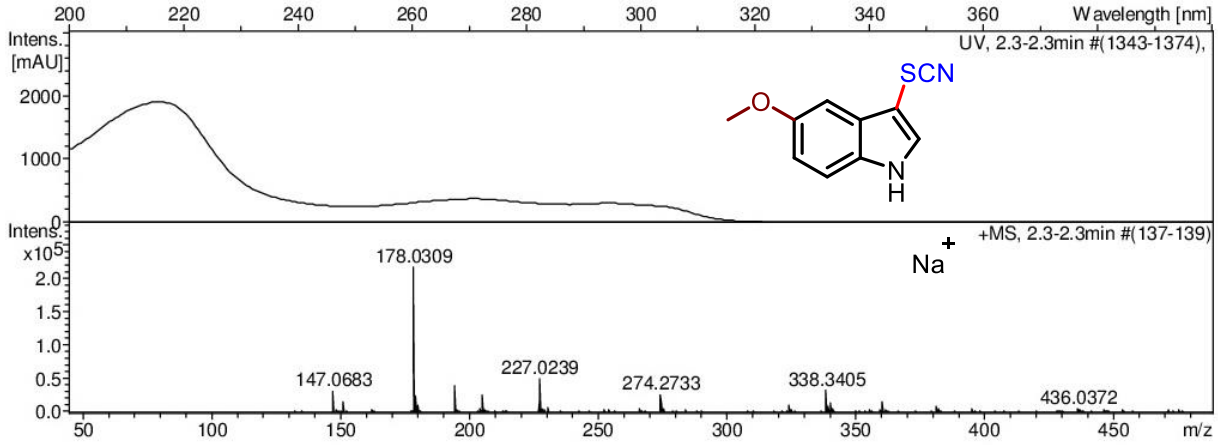
Analysis Info

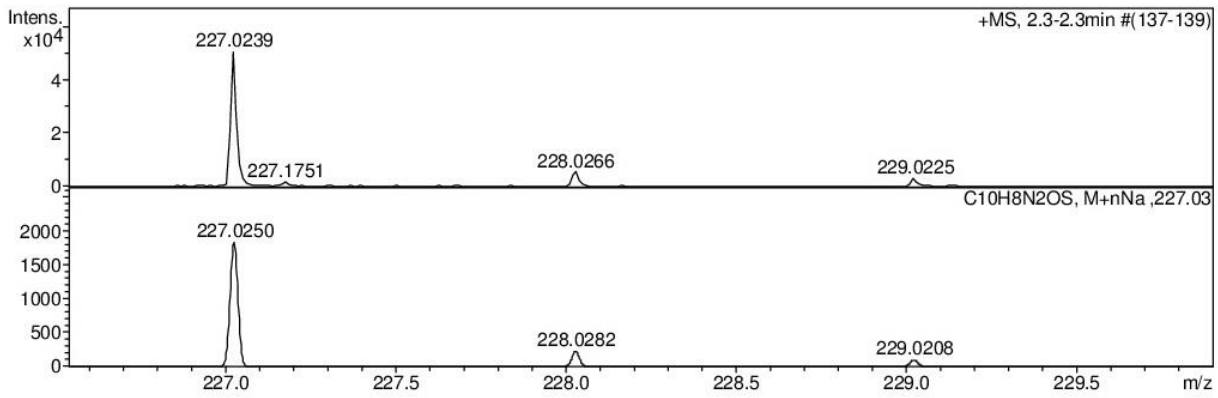
Analysis Name	D:\Data\NEW USER DATA 2017\2018\OCTOBER 2018\12 OCT\Dr S Kumar-VR-710_1-B,1_01_3788.d	Acquisition Date	10/12/2018 2:14:01 PM
Method	hrlcms-20 sept.m	Operator	RUCHI
Sample Name	Dr S Kumar-VR-710	Instrument	micrOTOF-Q II 10330
Comment			

Acquisition Parameter

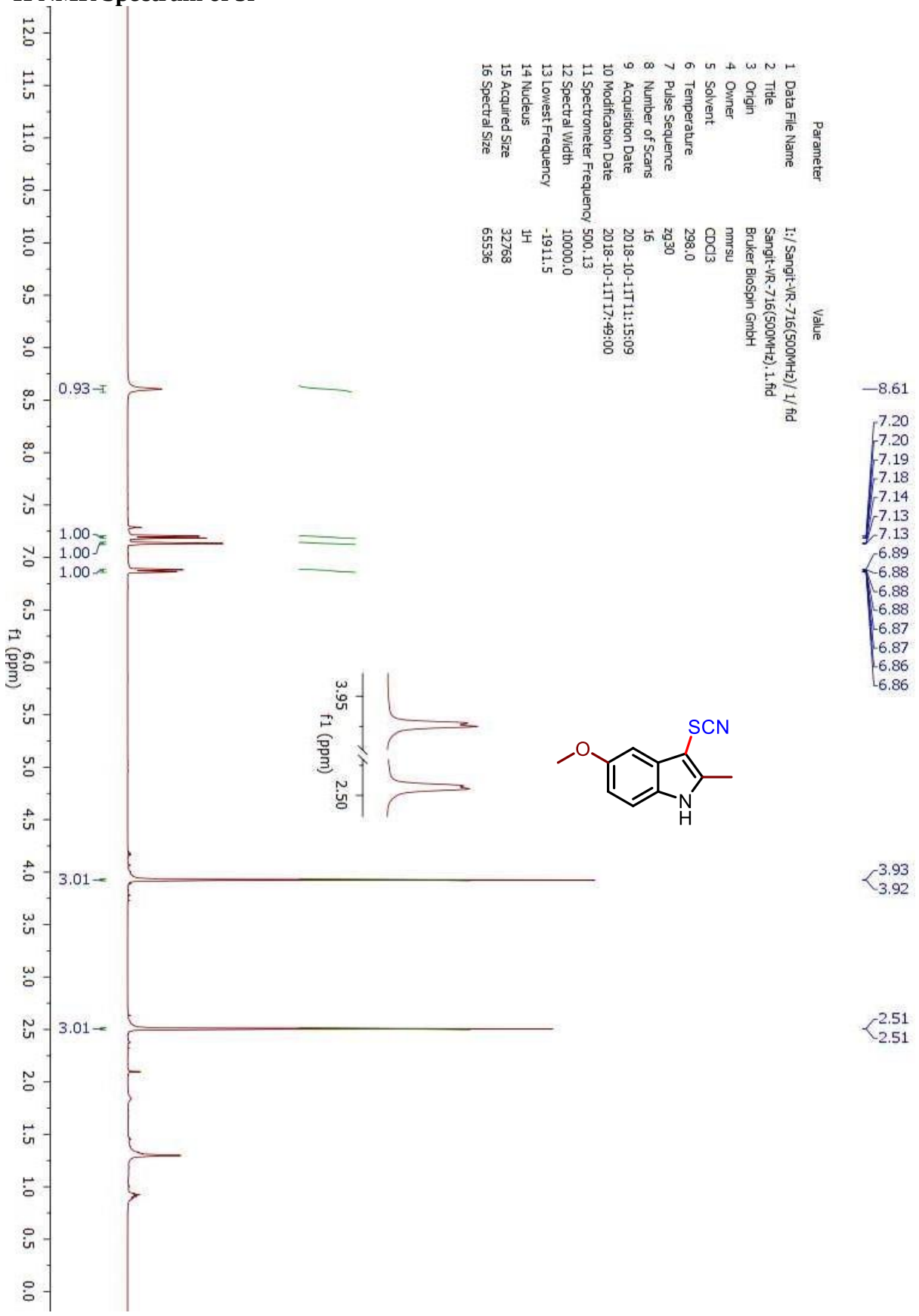
Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.2 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	7.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste



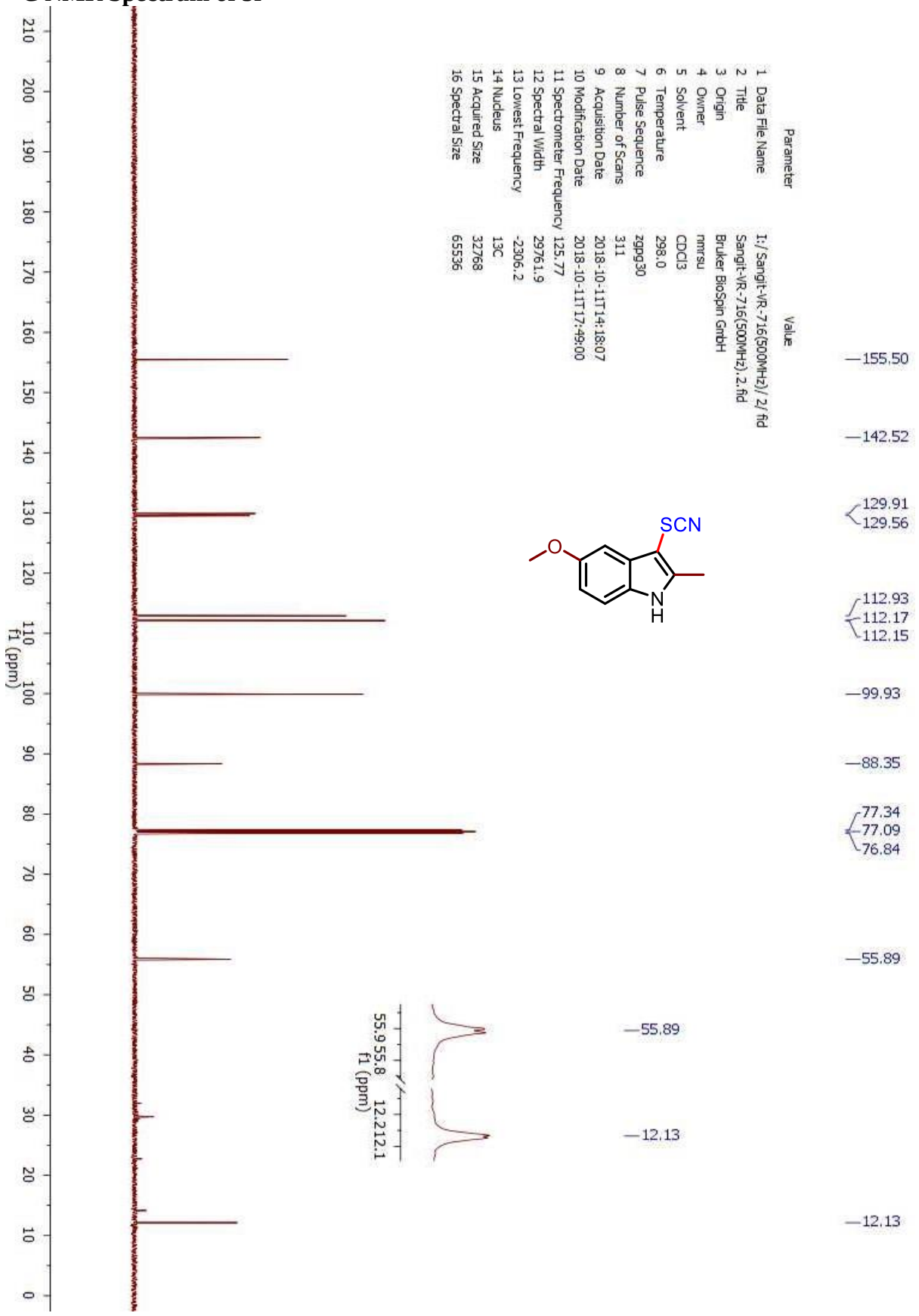




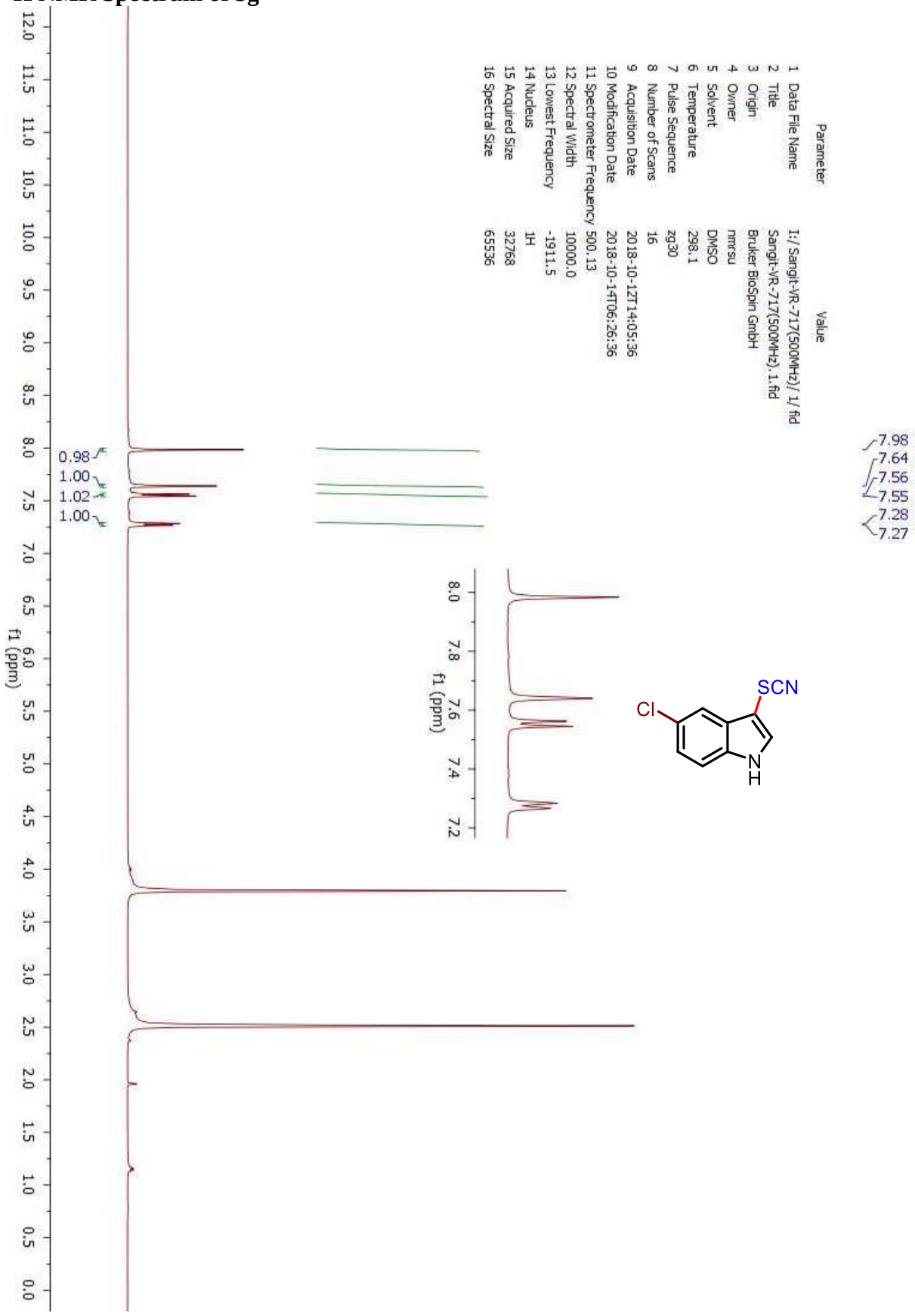
^{1}H NMR Spectrum of 5f



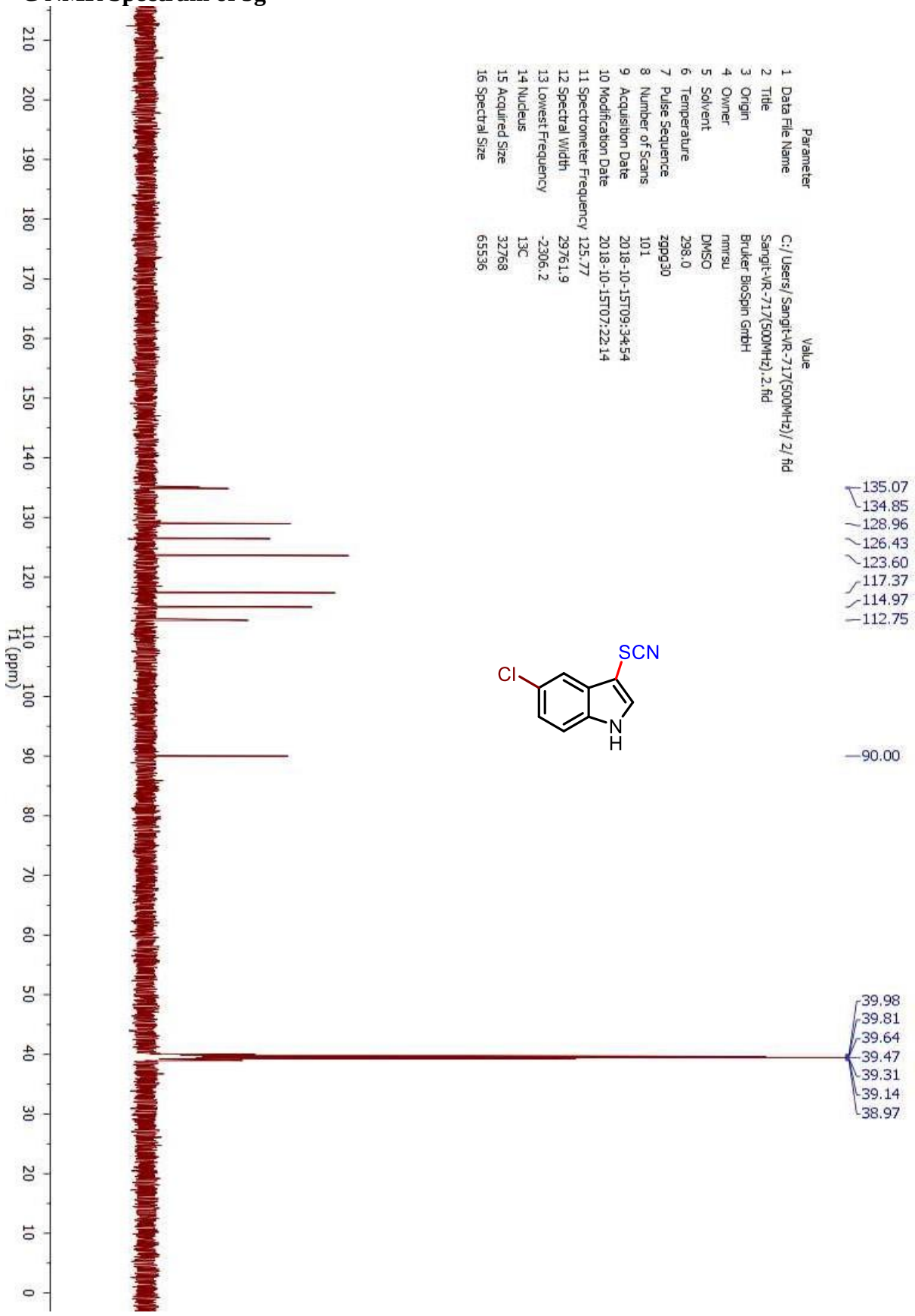
^{13}C NMR Spectrum of 5f



^{1}H NMR Spectrum of 5g



^{13}C NMR Spectrum of 5g



Mass Spectrum of 5g

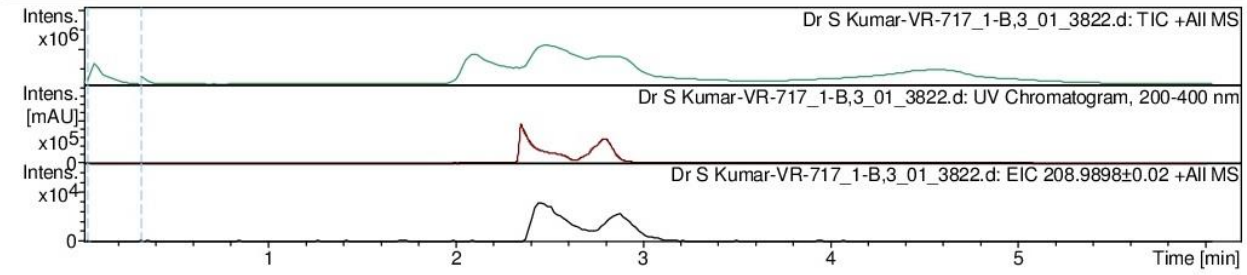
Display Report

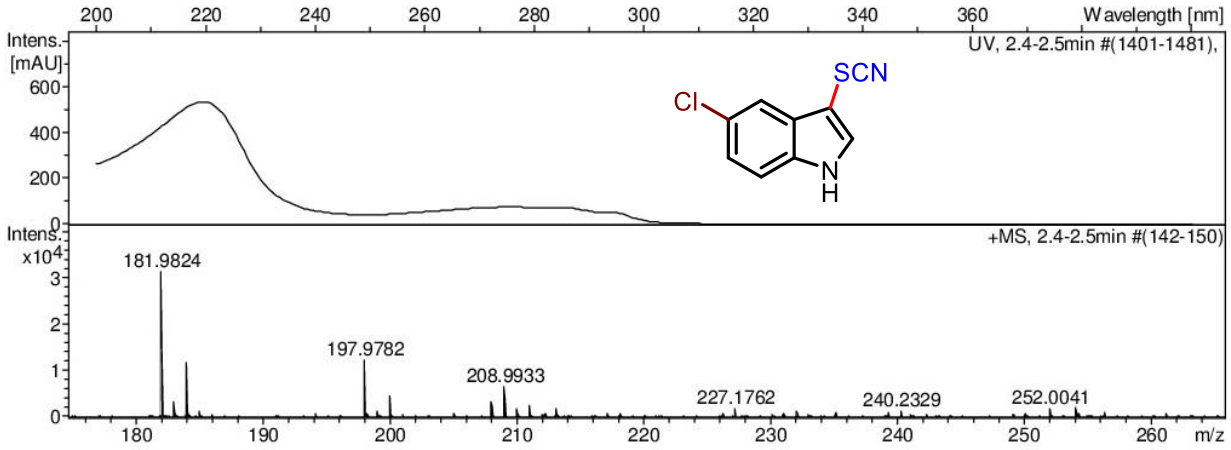
Analysis Info

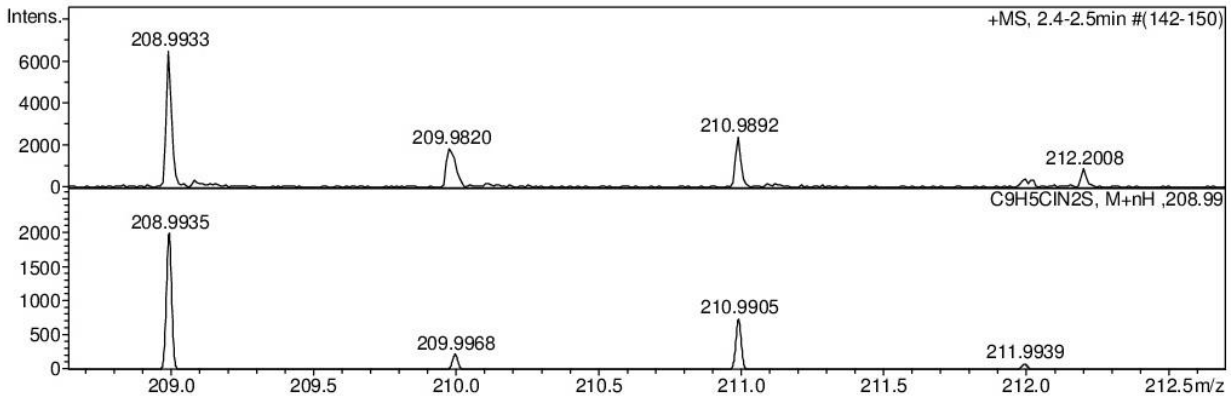
Analysis Name	D:\Data\NEW USER DATA 2017\2018\OCTOBER 2018\15 0ct\Dr S Kumar-VR-717_1-B,3_01_3822.d	Acquisition Date	10/15/2018 2:32:54 PM
Method	hrlcms-20 sept.m	Operator	RUCHI
Sample Name	Dr S Kumar-VR-717	Instrument	micrOTOF-Q II 10330
Comment			

Acquisition Parameter

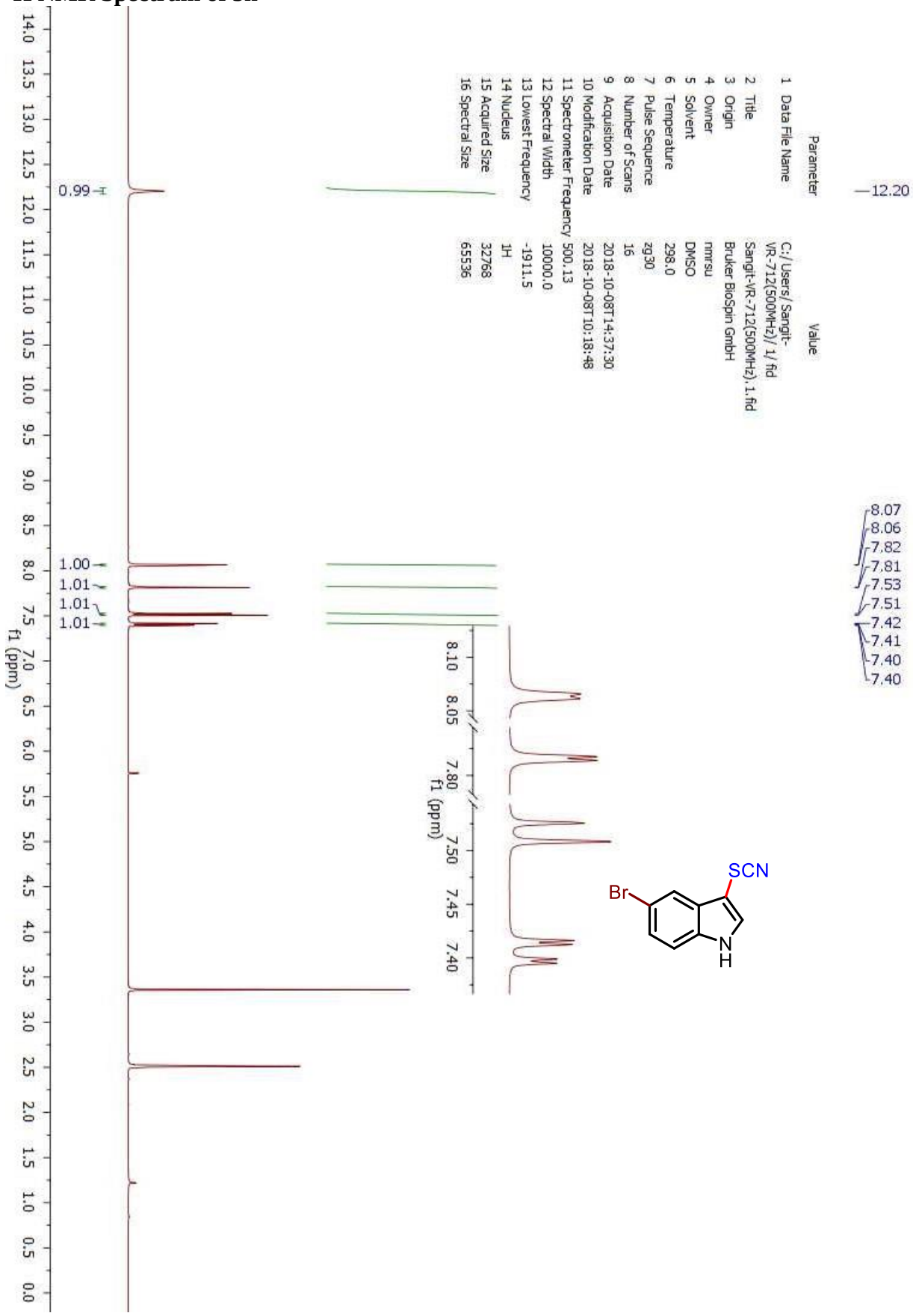
Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.2 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	7.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste



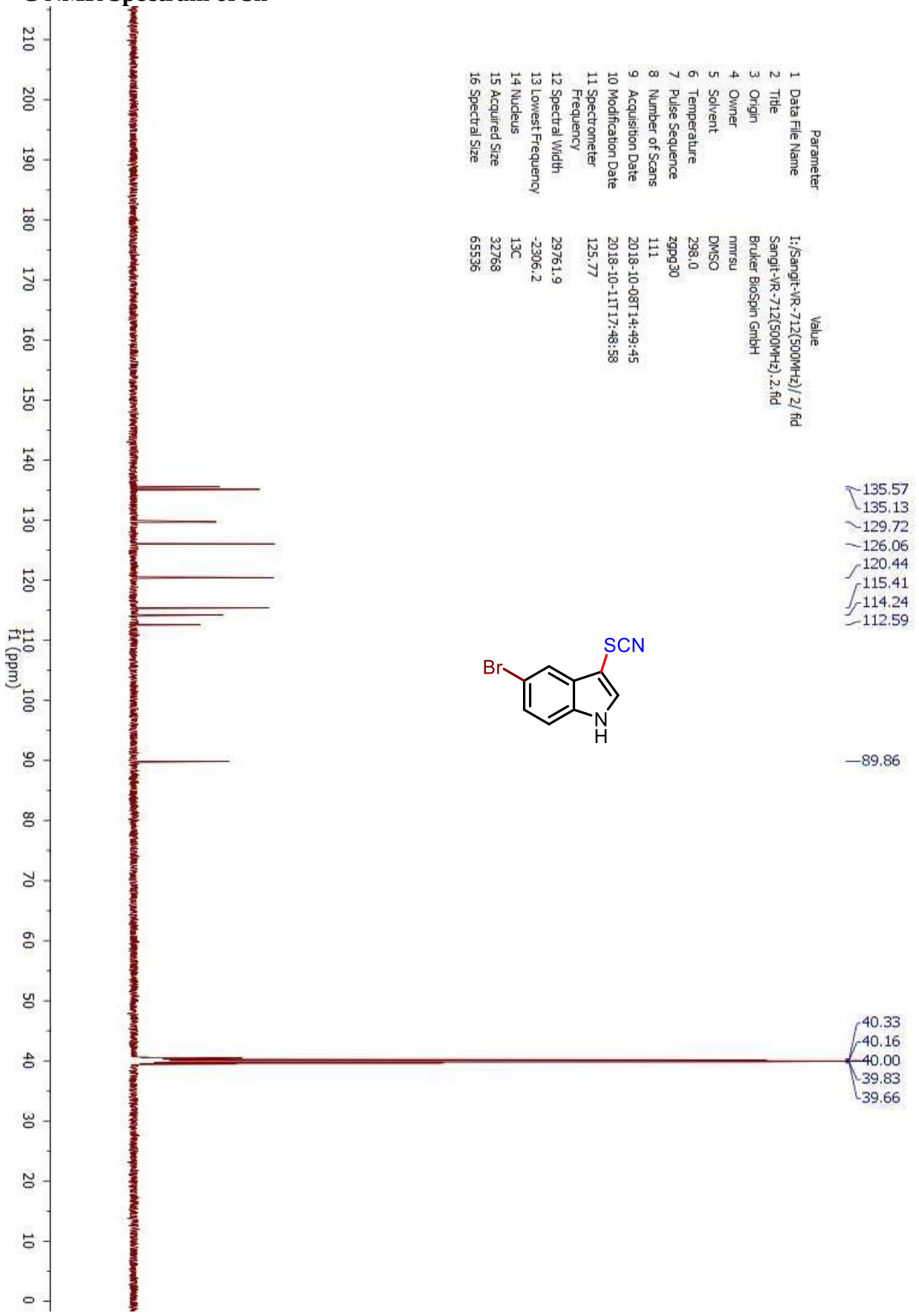




^{1}H NMR Spectrum of 5h



^{13}C NMR Spectrum of 5h



Mass Spectrum of 5h

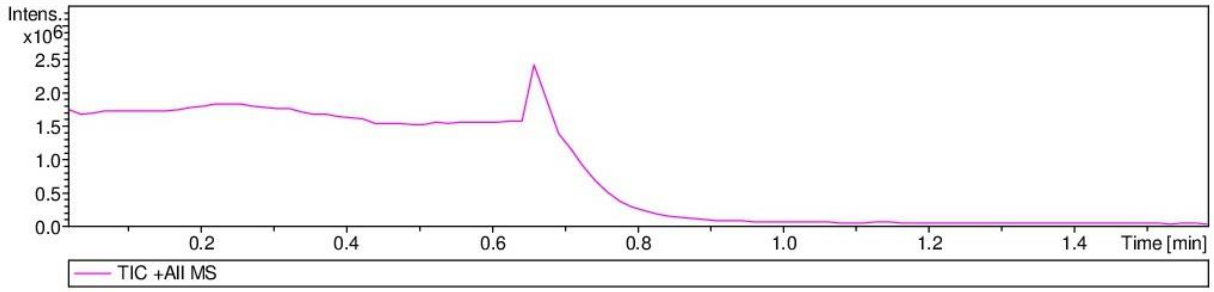
Display Report

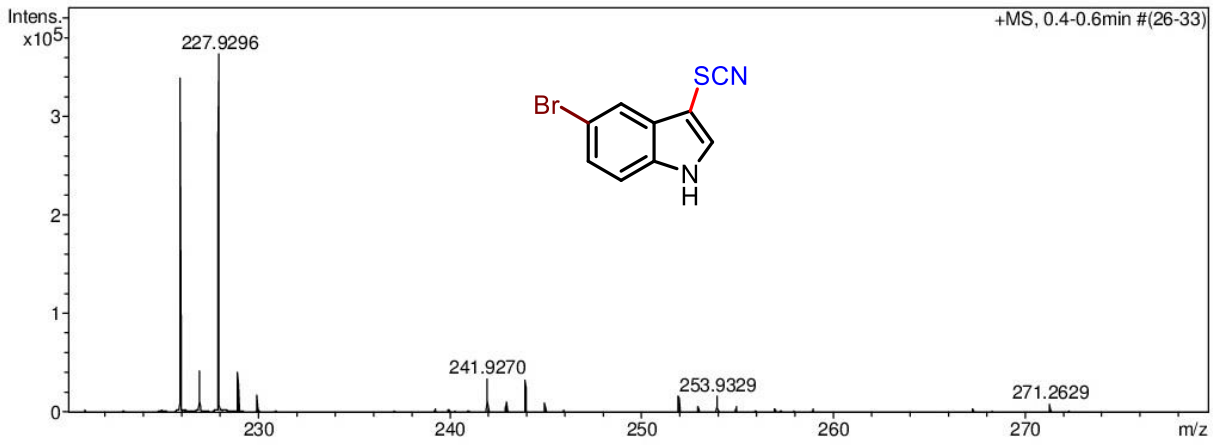
Analysis Info

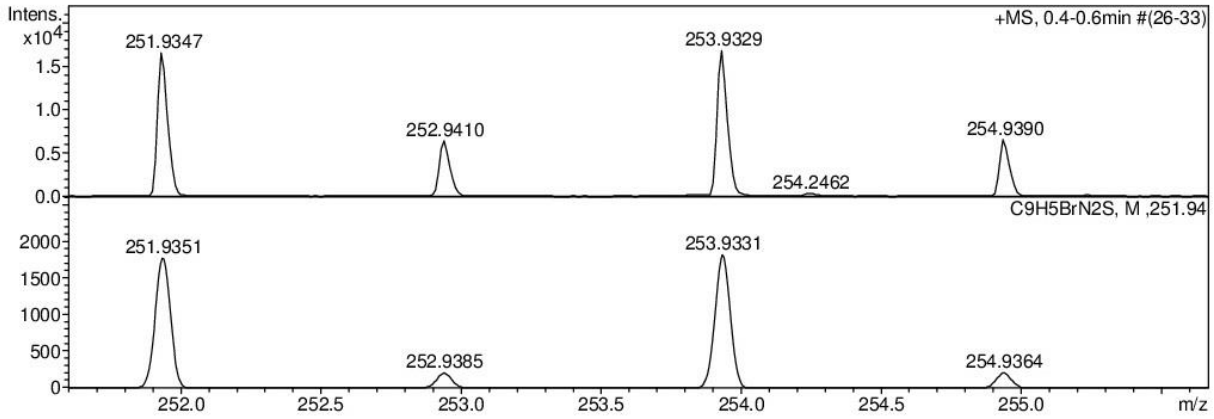
Analysis Name	D:\Data\NEW USER DATA 2017\2018\OCTOBER 2018\24 oct\Dr.S.Kumar-VR-712-A.d	Acquisition Date	10/25/2018 3:39:59 PM
Method	tune_low_APCI.m	Operator	RUCHI
Sample Name	VR-712-A	Instrument	micrOTOF-Q II 10330
Comment			

Acquisition Parameter

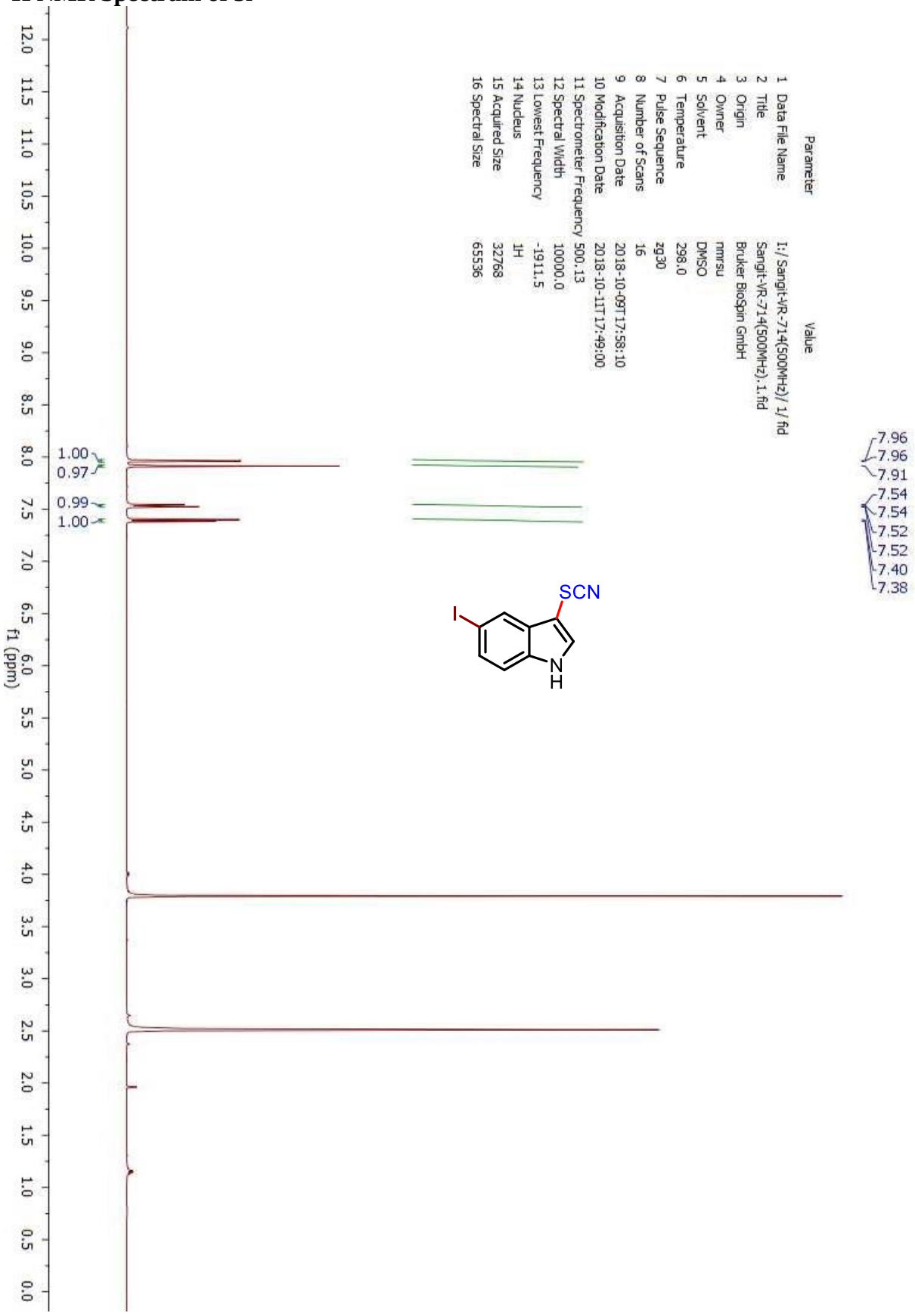
Source Type	APCI	Ion Polarity	Positive	Set Nebulizer	2.5 Bar
Focus	Not active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	100.0 Vpp	Set Divert Valve	Waste



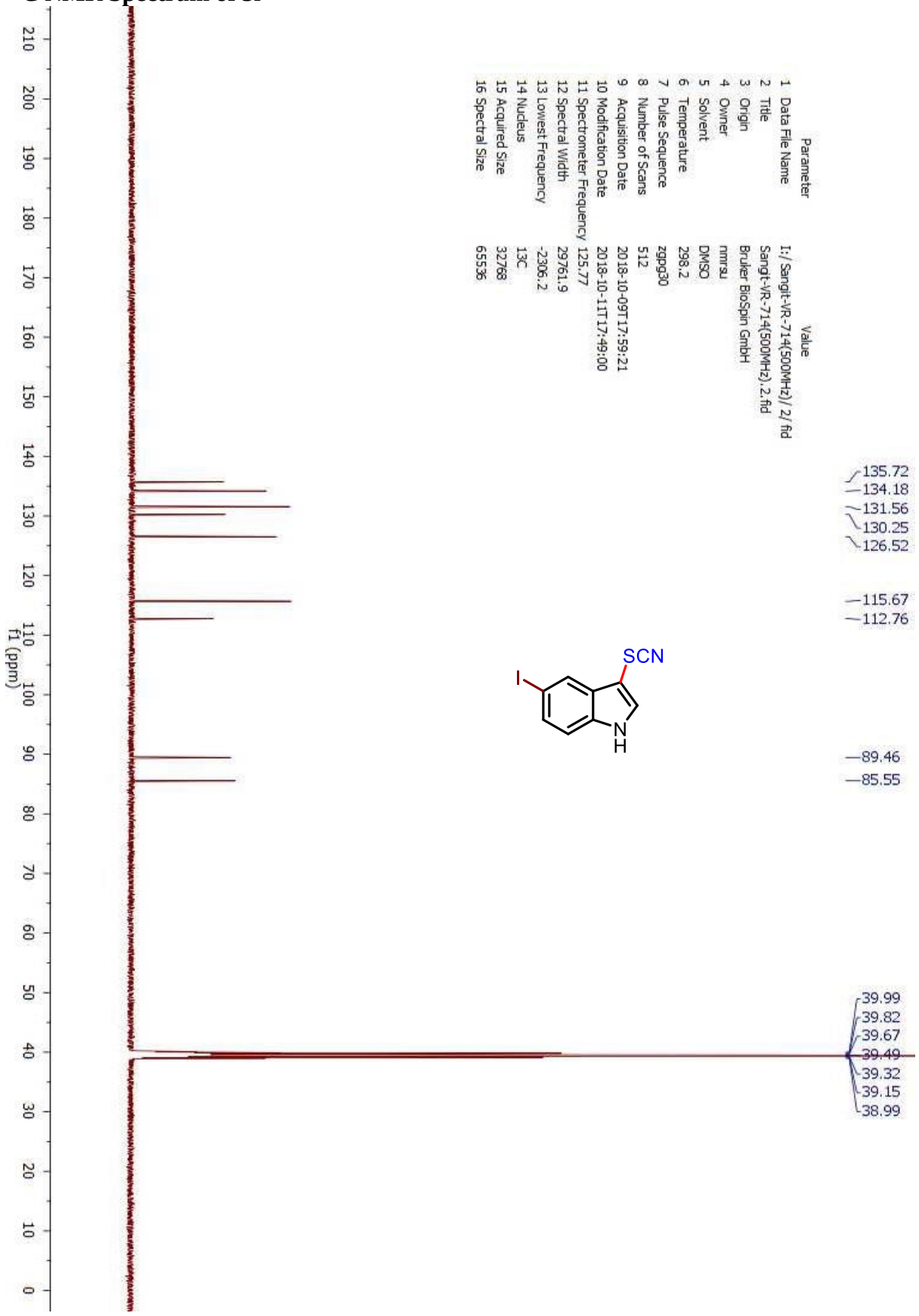




^{1}H NMR Spectrum of 5i



^{13}C NMR Spectrum of 5i



Mass Spectrum of 5i

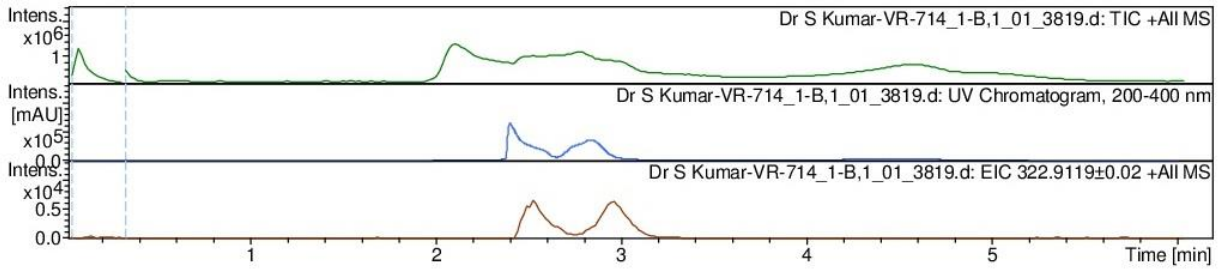
Display Report

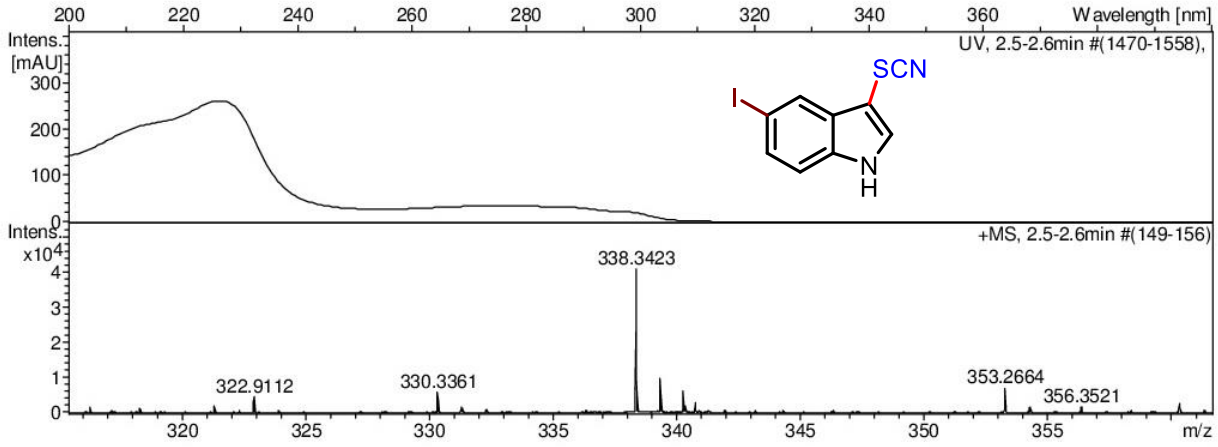
Analysis Info

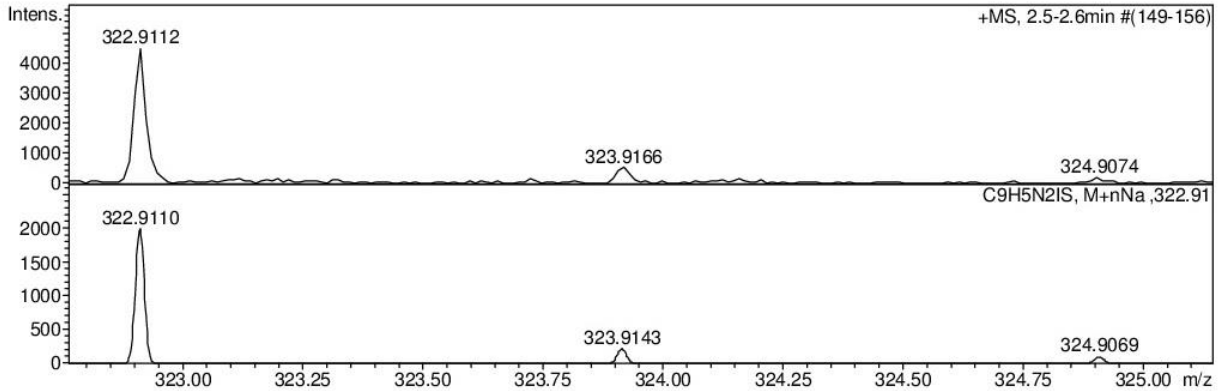
Acquisition Date	10/15/2018 2:11:20 PM		
Analysis Name	D:\Data\NEW USER DATA 2017\2018\OCTOBER 2018\15 0ct\Dr S Kumar-VR-714_1-B,1_01_3819.d		
Method	hrlcms-20 sept.m	Operator	RUCHI
Sample Name	Dr S Kumar-VR-714	Instrument	micrOTOF-Q II 10330
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.2 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	7.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste







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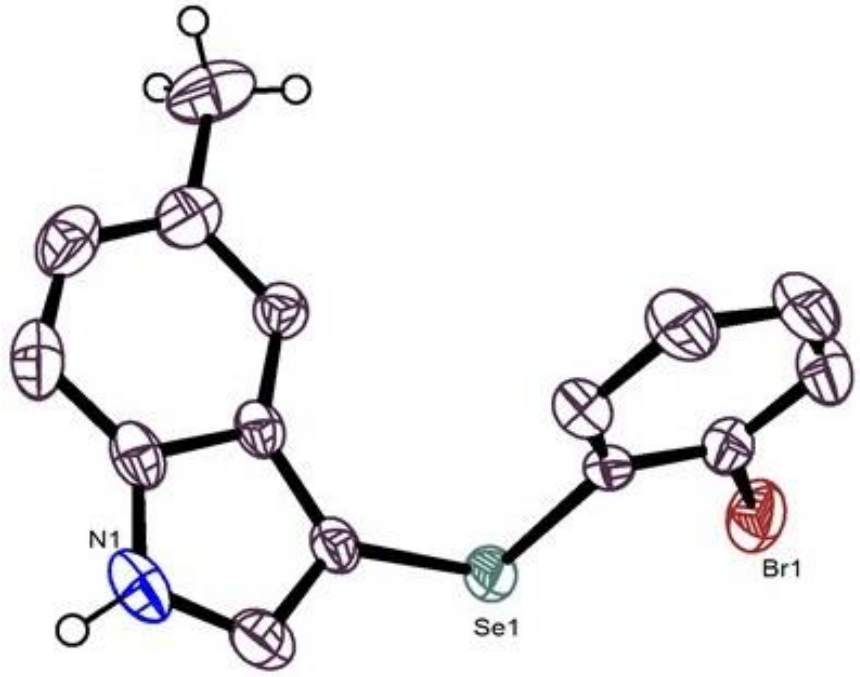
***ORTEP* view of 2h at 50% ellipsoidal probability (hydrogens are omitted for clarity)**



Packing diagram of 2h

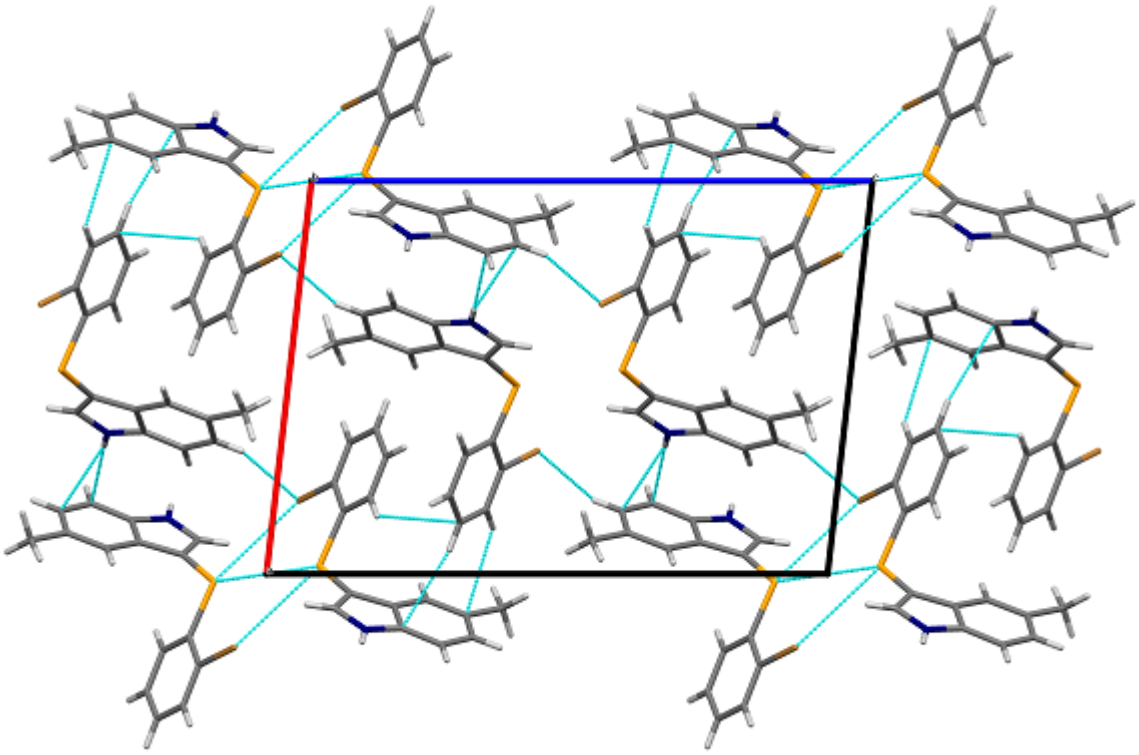


Table 1. Crystal data and structure refinement for 2h

Identification code	VR-601 (CCDC No. 1887962)	
Empirical formula	C_{15} H_{12} Br N Se	
Formula weight	365.13	
Temperature	298(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P 2_1/n	
Unit cell dimensions	a = 12.469 (4) Å	α= 90°.
	b = 6.265 (2) Å	β= 96.553(18)°.
	c = 17.738 (6) Å	γ = 90°.
Volume	1376.6(8) $Å^3$	
Z	4	
Density (calculated)	1.762 g/cm^3	
Absorption coefficient	5.611 mm^{-1}	
F(000)	712	
Theta range for data collection	2.311 to 28.832°.	
Index ranges	-16<=h<=16, -8<=k<=8, -24<=l<=24	
Reflections collected	23179	
Independent reflections	3581 [R(int) = 0.0883]	
Completeness to theta = 25.242°	100.0 %	
Absorption correction	None	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	3581 / 0 / 164	
Goodness-of-fit on F^2	0.991	
Final R indices [I>2sigma(I)]	R1 = 0.0487, wR2 = 0.1036	
R indices (all data)	R1 = 0.0825, wR2 = 0.1188	
Extinction coefficient	n/a	
Largest diff. peak and hole	1.433 and -1.278 $e.Å^{-3}$	

Table 2. Atomic coordinates and equivalent isotropic displacement parameters ($Å^2$) for VR-601. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor

	x	y	z	U(eq)
Se1	0.48060(3)	0.64594(8)	0.59231(2)	0.04270(15)
Br1	0.31322(5)	0.26095(9)	0.54261(3)	0.06261(19)
N1	0.6409(3)	1.1566(6)	0.6874(3)	0.0524(10)
C1	0.2680(3)	0.4890(8)	0.6019(2)	0.0453(11)
C2	0.1648(4)	0.4884(10)	0.6220(3)	0.0633(15)
C3	0.1317(4)	0.6510(11)	0.6654(3)	0.0678(16)
C4	0.2010(4)	0.8141(10)	0.6894(3)	0.0610(14)
C5	0.3052(4)	0.8141(8)	0.6687(3)	0.0463(11)
C6	0.3394(3)	0.6528(7)	0.6248(2)	0.0365(9)
C7	0.5490(3)	0.8620(7)	0.6537(2)	0.0361(9)
C8	0.5872(3)	1.0487(8)	0.6283(3)	0.0471(11)
C9	0.6379(3)	1.0423(7)	0.7526(3)	0.0417(10)
C10	0.5809(3)	0.8515(7)	0.7336(2)	0.0338(8)
C11	0.5655(3)	0.7058(7)	0.7899(2)	0.0383(9)
C12	0.6049(4)	0.7490(8)	0.8643(3)	0.0491(11)
C13	0.6611(4)	0.9411(9)	0.8802(3)	0.0555(13)
C14	0.6797(4)	1.0866(8)	0.8265(3)	0.0506(12)
C15	0.5868(5)	0.5953(11)	0.9272(3)	0.0740(18)

Table 3. Selected bond lengths [Å] for VR-601

Se1-C6	1.914(4)	C8-H8	0.9300
Se1-C7	1.880(4)	C5-C4	1.388(7)
Br1-C1	1.898(5)	C5-H5	0.9300
N1-C8	1.358(6)	C14-C13	1.358(8)
N1-C9	1.365(6)	C14-H14	0.9300
N1-H1	0.8600	C12-C13	1.405(8)
C10-C11	1.383(6)	C12-C15	1.510(8)
C10-C9	1.412(6)	C13-H13	0.9300
C10-C7	1.429(6)	C4-C3	1.374(8)

C6-C5	1.374(7)	C4-H4	0.9300
C6-C1	1.389(6)	C2-C3	1.368(9)
C7-C8	1.359(6)	C2-H2	0.9300
C11-C12	1.380(6)	C3-H3	0.9300
C11-H11	0.9300	C15-H15A	0.9600
C9-C14	1.382(7)	C15-H15B	0.9600
C1-C2	1.374(7)	C15-H15C	0.9600

Table 4. Selected bond angles [°] for VR-601

C7-Se1-C6	100.45(18)	C6-C5-H5	119.7
C8-N1-C9	109.5(4)	C4-C5-H5	119.7
C8-N1-H1	125.2	C13-C14-C9	117.0(4)
C9-N1-H1	125.2	C13-C14-H14	121.5
C11-C10-C9	119.6(4)	C9-C14-H14	121.5
C11-C10-C7	134.5(4)	C11-C12-C13	118.4(5)
C9-C10-C7	105.9(4)	C11-C12-C15	120.9(5)
C5-C6-C1	118.6(4)	C13-C12-C15	120.7(5)
C5-C6-Se1	122.9(3)	C14-C13-C12	123.7(5)
C1-C6-Se1	118.5(4)	C14-C13-H13	118.1
C8-C7-C10	107.5(4)	C12-C13-H13	118.1
C8-C7-Se1	125.5(3)	C3-C4-C5	119.5(5)
C10-C7-Se1	126.7(3)	C3-C4-H4	120.2
C12-C11-C10	119.8(4)	C5-C4-H4	120.2
C12-C11-H11	120.1	C3-C2-C1	119.7(5)
C10-C11-H11	120.1	C3-C2-H2	120.1
N1-C9-C14	130.9(4)	C1-C2-H2	120.1
N1-C9-C10	107.7(4)	C2-C3-C4	120.5(5)
C2-C1-C6	121.0(5)	C4-C3-H3	119.7
C2-C1-Br1	119.2(4)	C12-C15-H15A	109.5
C6-C1-Br1	119.8(3)	C12-C15-H15B	109.5

N1-C8-C7	109.5(4)	H15A-C15-H15B	109.5
N1-C8-H8	125.3	C12-C15-H15C	109.5
C7-C8-H8	125.3	H15A-C15-H15C	109.5
C6-C5-C4	120.7(5)	H15B-C15-H15C	109.5

Table 5. Anisotropic displacement parameters ($Å^2$) for VR-601. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Se1	0.0355(2)	0.0556(3)	0.0378(2)	-0.0061(2)	0.00765(16)	-0.0061(2)
Br1	0.0657(3)	0.0540(3)	0.0640(4)	-0.0044(3)	-0.0107(3)	-0.0114(3)
N1	0.047(2)	0.039(2)	0.073(3)	0.000(2)	0.016(2)	-0.0148(18)
C1	0.040(2)	0.054(3)	0.040(2)	0.009(2)	-0.0046(18)	-0.009(2)
C2	0.043(3)	0.080(4)	0.065(3)	0.010(3)	-0.001(2)	-0.023(3)
C3	0.034(2)	0.102(5)	0.069(4)	0.015(4)	0.014(2)	-0.009(3)
C4	0.040(3)	0.083(4)	0.062(3)	0.003(3)	0.016(2)	0.002(3)
C5	0.038(2)	0.057(3)	0.044(3)	0.002(2)	0.0086(19)	-0.009(2)
C6	0.0308(18)	0.051(2)	0.0277(19)	0.0108(19)	0.0015(15)	-0.0059(18)
C7	0.0255(17)	0.041(2)	0.042(2)	0.0011(19)	0.0057(15)	-0.0047(17)
C8	0.039(2)	0.051(3)	0.053(3)	0.011(2)	0.0111(19)	-0.003(2)
C9	0.0251(18)	0.043(2)	0.059(3)	-0.005(2)	0.0110(18)	-0.0020(17)
C10	0.0218(16)	0.037(2)	0.043(2)	-0.0040(18)	0.0076(15)	0.0011(16)
C11	0.035(2)	0.040(2)	0.040(2)	-0.0006(18)	0.0041(17)	0.0006(17)
C12	0.039(2)	0.062(3)	0.046(3)	-0.002(2)	0.004(2)	0.013(2)
C13	0.043(2)	0.075(4)	0.047(3)	-0.019(3)	0.000(2)	0.009(2)
C14	0.033(2)	0.051(3)	0.067(3)	-0.025(2)	0.005(2)	-0.0043(19)
C15	0.094(5)	0.085(5)	0.042(3)	0.013(3)	0.005(3)	0.023(4)

Table 6. Hydrogen coordinates and isotropic displacement parameters ($Å^2$) for VR-601

	x	y	z	U(eq)
H1	0.6721	1.2782	0.6842	0.063
H2	0.1177	0.3779	0.6063	0.076
H3	0.0617	0.6511	0.6788	0.081
H4	0.1784	0.9237	0.7192	0.073
H5	0.3522	0.9245	0.6848	0.056
H8	0.5779	1.0954	0.5782	0.057
H11	0.5287	0.5790	0.7778	0.046
H13	0.6871	0.9702	0.9304	0.067
H14	0.7188	1.2106	0.8388	0.061
H15A	0.5562	0.6704	0.9668	0.111
H15B	0.6545	0.5332	0.9473	0.111
H15C	0.5383	0.4845	0.9075	0.111