Electronic Supplementary Material (ESI) for ChemComm.

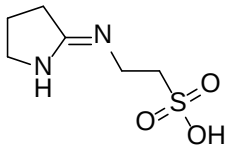
TABLE OF CONTENT

Experimental part

Solvents were purified and dried by standard methods. Melting points were determined with an electrothermal capillary melting point apparatus. ^{1}H NMR spectra were recorded with Agilent ProPulse 600 spectrometer (at 600 MHz for ^{1}H NMR, 151 MHz for ^{13}C NMR), Bruker 170 Avance 500 spectrometer (at 500 MHz for ^{1}H NMR, 126 MHz for ^{13}C NMR) and Varian Unity Plus 400 spectrometer (at 400 MHz for ^{1}H NMR, 101 MHz for ^{13}C NMR). Chemical shifts are reported in ppm relative to internal tetramethylsilane (TMS; for ^{1}H and ^{13}C). HRMS were recorded with Agilent 6224 TOF LC/MS.

General procedure for the synthesis of cyclic amidines 7

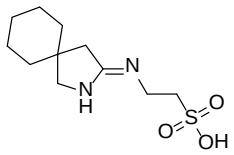
To a stirred solution of cyclic imidate **6a** (99.1 g, 1 mol, 1.1 eq.) in methanol (1 L) was added taurine (112.6 g, 0.9 mol, 1 eq.) and the resulting suspension was refluxed for two days. The reaction mixture was allowed cooling to room temperature and volatiles were removed under reduced pressure. The residue was triturated with MTBE (500 mL), filtered off, washed with MTBE (3×400 mL) and dried under reduced pressure to give amidine **7a** that was used in the next step without further purification.



2-(pyrrolidin-2-ylideneamino)ethanesulfonic acid 7a

M.p. 271-273°C, a white powder, 173.0 g (purity 90%, yield 92.3%)

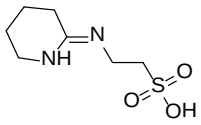
^{1}H NMR (400 MHz, D_2O) δ 3.73-3.68 (m, 4H), 3.20 (t, *J* =6.8 Hz, 2H), 2.87 (t, *J* =8.0 Hz, 2H), 2.26-2.16 (m, 2H). ^{13}C NMR (150 MHz, D_2O) δ 169.1 (C), 48.4 (CH_2), 47.7 (CH_2), 39.1 (CH_2), 30.6 (CH_2), 20.2 (CH_2). HRMS (ESI/TOF-Q) m/z: $[M]^+$ calcd for $C_6H_{12}N_2O_3S$, 192.0569; found 192.0565.



2-(2-azaspiro[4.5]decan-3-ylideneamino)ethanesulfonic acid 7b

M.p. 257-259°C, a white powder, 23.7g (yield 87.1%).

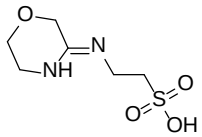
^{1}H NMR (400 MHz, CF_3COOD) δ 8.29-7.76 (m, 2H), 4.22-4.18 (m, 2H), 3.87-3.77 (m, 4H), 3.08-3.03 (m, 2H), 1.84-1.75 (m, 10H). ^{13}C NMR (126 MHz, CF_3COOD) δ 168.8, 57.8, 49.7, 48.8, 42.1, 40.5, 38.5, 35.3, 34.8, 24.1, 21.8. HRMS (ESI/TOF-Q) m/z: $[M]^+$ calcd for $C_{11}H_{20}N_2O_3S$, 260.1195; found 260.1191.



2-(piperidin-2-ylideneamino)ethanesulfonic acid 7c

M.p. 255-257°C, a white powder, 112.67 g (yield 94.1%)

^{1}H NMR (400 MHz, D_2O) δ 3.62 (br.s, 2H), 3.42 (br.s, 2H), 3.18 (br.s, 2H), 2.59 (br.s, 2H), 1.81 (br.s, 4H). ^{13}C NMR (150 MHz, D_2O) δ 163.6 (C), 48.2 (CH_2), 41.5 (CH_2), 36.9 (CH_2), 26.1 (CH_2), 20.3 (CH_2), 17.5 (CH_2). HRMS (ESI/TOF-Q) m/z: $[M]^+$ calcd for $C_7H_{14}N_2O_3S$, 206.0725; found 206.0723.

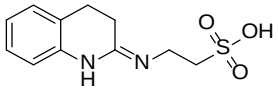


2-(morpholin-3-ylideneamino)ethanesulfonic acid 7d

M.p. 273-275°C, a white powder, 94.23 g (yield 87.6%)

^{1}H NMR (400 MHz, D_2O) δ 4.59 (s, 2H), 4.04 (t, J =5.2 Hz, 2H), 3.71 (t, J =6.4 Hz, 2H), 3.57 (t, J =5.2 Hz, 2H), 3.24 (t, J =6.4 Hz, 2H). ^{13}C NMR (100 MHz, D_2O) δ

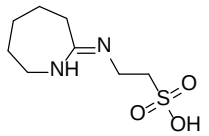
160.4 (C), 63.1 (CH_2), 63.0 (CH_2), 48.1 (CH_2), 39.6 (CH_2), 37.2 (CH_2). HRMS (ESI/TOF-Q) m/z: $[M]^+$ calcd for $C_6H_{12}N_2O_4S$, 208.0518; found 208.0507.



2-((3,4-dihydroquinolin-2(1H)-ylidene)amino)ethanesulfonic acid 7e

M.p. 325-327°C, a white powder, 112.7 g (yield 93.7%)

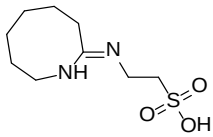
^{1}H NMR (400 MHz, CF_3COOD) δ 9.78-9.64 (m, 1H), 8.64-8.43 (m, 1H), 7.55-7.42 (m, 4H), 4.46-4.42 (m, 2H), 3.99-3.94 (m, 2H), 3.32-3.21 (m, 4H). ^{13}C NMR (126 MHz, CF_3COOD) δ 162.7 (C), 131.9 (C), 127.9 (CH), 127.7 (CH), 126.7 (CH), 123.5 (C), 117.2 (CH), 48.5 (CH_2), 37.5 (CH_2), 26.7 (CH_2), 21.5 (CH_2). HRMS (ESI/TOF-Q) m/z: $[M]^+$ calcd for $C_{11}H_{14}N_2O_3S$, 254.0725; found 254.0719.



2-(azepan-2-ylideneamino)ethanesulfonic acid 7f

M.p. 271-274°C, a white powder, 107.6 g (yield 95.3%)

^{1}H NMR (400 MHz, D_2O) δ 3.63 (t, *J* =6.4 Hz, 2H), 3.51-3.49 (m, 2H), 3.20 (t, *J* =6.8 Hz, 2H), 2.69-2.67 (m, 2H), 1.79-1.66 (m, 6H). ^{13}C NMR (125 MHz, D_2O) δ 169.2 (C), 47.6 (CH_2), 43.7 (CH_2), 37.1 (CH_2), 31.8 (CH_2), 28.5 (CH_2), 26.9 (CH_2), 22.5 (CH_2). HRMS (ESI/TOF-Q) m/z: $[M]^+$ calcd for $C_8H_{16}N_2O_3S$, 220.0882; found 220.0874.



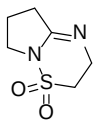
2-(azocan-2-ylideneamino)ethanesulfonic acid 7g

M.p. 286-289°C, a grey powder, 112.1 g (yield 93.9%)

^{1}H NMR (500 MHz, D_2O) δ 3.68 (t, *J* =6.50 Hz, 2H), 3.59 (t, *J* =5.5 Hz, 2H), 3.21 (t, *J* =6.5 Hz, 2H), 2.67 (t, *J* =6.0 Hz, 2H), 1.86-1.82 (m, 2H), 1.75-1.70 (m, 2H), 1.61-1.52 (m, 4H) ^{13}C NMR (125 MHz, D_2O) δ 167.7 (C), 47.6 (CH_2), 42.0 (CH_2), 36.9 (CH_2), 30.0 (CH_2), 28.3 (CH_2), 28.1 (CH_2), 24.1 (CH_2), 23.3 (CH_2). HRMS (ESI/TOF-Q) m/z: $[M]^+$ calcd for $C_9H_{18}N_2O_3S$, 234.1038; found 234.1028.

General procedure for the synthesis of thiadiazine derivatives 8

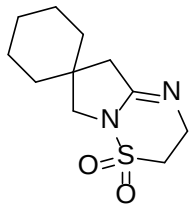
Sulfonic acid **7a** (125.0 g, 0.65 mol, 1 eq.) was dissolved in $POCl_3$ (400 mL), then PCl_5 (135.4 g, 0.65 mol, 1 eq.) was added at 20°C. The resulting white suspension was stirred for 24 hours at 35°C. Upon completion, $POCl_3$ was removed under reduced pressure at the same temperature. The residue was triturated with MTBE (500 mL), filtered off and washed with MTBE (5×400 mL) to afford a crude sulfonyl chloride that was used in the next step without further purification. To a stirred suspension of the crude sulfonyl chloride (0.65 mol, 1 eq.) in dry CH_3CN (1000 mL) was added in one portion DIPEA (180.6 g, 1.4 mol, 2.15 eq.) and the reaction mixture was stirred for 12 hours at room temperature (25°C). All volatiles were removed under reduced pressure leaving semi-oil residue. It was diluted with DCM (1 L), K_2CO_3 (269.5 g, 1.95 mol, 3 eq.) and water (25 mL) were added. The reaction mixture was stirred for 2 hours at room temperature. Upon completion, inorganics were filtered off, and was washed with DCM (4×500 mL). Organic washings were combined and concentrated under reduced pressure (after evaporation of DCM at 10 Torr /30°C, excess of DIPEA was removed at 10 Torr /70°C) to give pure annulated thiadiazine **8a**.



3,6,7,8-tetrahydro-2H-pyrrolo[1,2-b][1,2,4]thiadiazine 4,4-dioxide 8a

M.p. 58-60°C, a beige powder, 94.6 g (yield 83.5%)

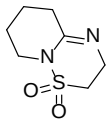
^{1}H NMR (400 MHz, DMSO-d_6) δ 3.84-3.81 (m, 2H), 3.63 (t, J =6.8 Hz, 2H), 3.33 (t, J =6.0 Hz, 2H), 2.54-2.5 (m, 2H), 1.95-1.88 (m, 2H). ^{13}C NMR (126 MHz, DMSO-d_6) δ 156.6 (C), 46.0 (CH_2), 44.7 (CH_2), 44.2 (CH_2), 31.2 (CH_2), 18.9 (CH_2). HRMS (ESI/TOF-Q) m/z: $[M]^+$ calcd for $C_6H_{10}N_2O_2S$, 174.0463; found 174.0459.



2',3',6',8'-tetrahydrospiro[cyclohexane-1,7'-pyrrolo[1,2-b][1,2,4]thiadiazine] 4',4'-dioxide 8b

M.p. 98-103°C, a white solid 15.1 g (yield 91.1%).

^{1}H NMR (400 MHz, DMSO-d_6) δ 3.82 (t, J =5.60 Hz, 2H), 3.41 (s, 2H), 3.35 (t, J =6.00 Hz, 2H), 2.39 (s, 2H), 1.44-1.36 (m, 10H). ^{13}C NMR (100 MHz, DMSO-d_6) δ 156.5 (C), 54.0 (CH_2), 46.4 (CH_2), 45.3 (CH_2), 43.5 (C), 37.8 (CH_2), 34.8 (CH_2), 25.8 (CH_2), 22.9 (CH_2). HRMS (ESI/TOF-Q) m/z: $[M]^+$ calcd for $C_{11}H_{18}N_2O_2S$, 242.1089; found 242.1084.

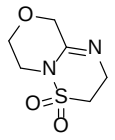


2,3,6,7,8,9-hexahydropyrido[1,2-b][1,2,4]thiadiazine 4,4-dioxide 8c

M.p. 68-70°C, a beige powder, 93.6 g (yield 93.4%)

^{1}H NMR (400 MHz, DMSO-d_6) δ 3.85-3.82 (m, 2H), 3.49-3.46 (m, 2H), 3.42-3.39 (m, 2H), 2.36-2.33 (m, 2H), 1.81-1.75 (m, 2H), 1.71-1.65 (m, 2H) ^{13}C NMR (126 MHz, DMSO-d_6) δ 151.7 (C), 44.9 (CH_2), 44.3 (CH_2), 42.4 (CH_2), 32.9 (CH_2), 23.0

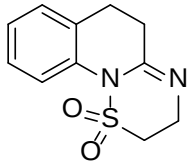
(CH_2), 20.9 (CH_2). HRMS (ESI/TOF-Q) m/z: $[M]^+$ calcd for $C_7H_{12}N_2O_2S$, 188.0619; found 188.0616.



3,6,7,9-tetrahydro-2H-[1,4]oxazino[4,3-b][1,2,4]thiadiazine 4,4-dioxide 8d

M.p. 91°C, a white crystal, 97.1 g (yield 91.2%)

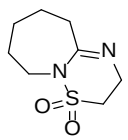
^{1}H NMR (400 MHz, DMSO-d_6) δ 4.12 (s. 2H), 3.92-3.86 (m, 4H), 3.56-3.49 (m, 4H). ^{13}C NMR (150 MHz, DMSO-d_6) δ 147.8 (C), 68.4 (CH_2), 64.9 (CH_2), 45.4 (CH_2), 45.0 (CH_2), 40.8 (CH_2). HRMS (ESI/TOF-Q) m/z: $[M]^+$ calcd for $C_6H_{10}N_2O_3S$, 190.0412; found 190.0404.



2,3,5,6-tetrahydro-[1,2,4]thiadiazino[2,3-a]quinoline 1,1-dioxide 8e

M.p. 126-128°C, yellow solid, 89.3 g (yield 79.1%).

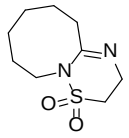
^{1}H NMR (400 MHz, DMSO-d_6) δ 7.54 (d, J =4.20 Hz, 1H), 7.29-7.25 (m, 2H), 7.16-7.12 (m, 1H), 3.96-3.93 (m, 2H), 3.77 (t, J =6.00 Hz, 2H), 2.81-2.78 (m, 2H), 2.60-2.57 (m, 2H). ^{13}C NMR (126 MHz, DMSO-d_6) δ 151.1 (C), 132.7 (C), 130.3 (C), 128.2 (CH), 126.6 (CH), 124.9 (CH), 120.3 (CH), 47.2 (CH_2), 43.2 (CH_2), 33.2 (CH_2), 25.0 (CH_2). HRMS (ESI/TOF-Q) m/z: $[M]^+$ calcd for $C_{11}H_{12}N_2O_2S$, 236.0619; found 236.0615.



3,6,7,8,9,10-hexahydro-2H-[1,2,4]thiadiazino[2,3-a]azepine 4,4-dioxide 8f

M.p. 54-56°C, a beige powder, 101.8 g (yield 94.3%)

^{1}H NMR (400 MHz, DMSO-d_6) δ 3.81 (t, J =6.0 Hz, 2H), 3.71-3.69 (m, 2H), 3.39 (t, J =6.4 Hz, 2H), 2.52-2.50 (m, 2H), 1.60 (br.s, 6H). ^{13}C NMR (126 MHz, DMSO-d_6) δ 156.5 (C), 45.4 (CH_2), 44.9 (CH_2), 41.8 (CH_2), 37.3 (CH_2), 29.9 (CH_2), 28.6 (CH_2), 25.5 (CH_2). HRMS (ESI/TOF-Q) m/z: $[M]^+$ calcd for $C_8H_{14}N_2O_2S$, 202.0776; found 202.0772.



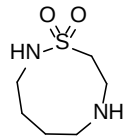
2,3,6,7,8,9,10,11-octahydro-[1,2,4]thiadiazino[2,3-a]azocine 4,4-dioxide 8g

M.p. 77-79°C, a brown oil, 87.7 g (yield 82.3%)

^{1}H NMR (500 MHz, DMSO-d_6) δ 3.87-3.81 (m, 4H), 3.37 (t, J =7.5 Hz, 2H), 2.50-2.47 (m, 2H), 1.68-1.58 (m, 4H), 1.55-1.46 (m, 4H) ^{13}C NMR (126 MHz, DMSO-d_6) δ 155.4 (C), 45.3 (CH_2), 45.1 (CH_2), 42.0 (CH_2), 34.1 (CH_2), 30.6 (CH_2), 29.8 (CH_2), 25.1 (CH_2), 23.8 (CH_2). HRMS (ESI/TOF-Q) m/z: $[M]^+$ calcd for $C_9H_{16}N_2O_2S$, 216.0932; found 216.0929.

General procedure for reductive cleavage of thiadiazine derivatives

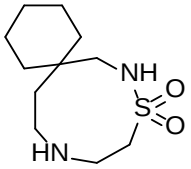
To a stirred solution of thiadiazine derivative **8a** (34.8 g, 200 mmol, 1 eq.) in glacial acetic acid (150 mL) was added portion wise $NaBH_3CN$ (25.1 g, 400 mmol, 2 eq.) for 0.5 h keeping temperature below 30°C. The reaction mixture was stirred for 1h at 25°C-30°C, 1 h at 50°C, 21 h at 25°C. The reaction mixture was diluted with water (75 mL) and was cooled to 15°C. It was quenched with 50% aqueous NaOH till pH = 14 at 20°C. A precipitated solid was filtered off and washed with $CHCl_3$ (5×150 mL). The filtrate was extracted with $CHCl_3$ (4×200 mL), the combined organics were dried over Na_2SO_4 and evaporated under reduced pressure till dryness to afford a crude product **11a** that was purified by trituration with MTBE.



1,2,7-thiadiazonane 1,1-dioxide 11a

M.p. 61°C, a white powder, 22.5 g (yield 63.1%)

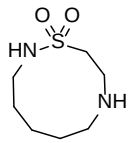
^{1}H NMR (400 MHz, DMSO-d_6) δ 6.74 (br.s, 1H), 3.33-3.13 (m, 5H), 2.94-2.92 (m, 2H), 2.68-2.67 (m, 2H), 1.62-1.54 (m, 4H). ^{13}C NMR (100 MHz, DMSO-d_6) δ 52.9 (CH_2), 49.5 (CH_2), 45.0 (CH_2), 43.4 (CH_2), 28.7 (CH_2), 28.6 (CH_2). HRMS (ESI/TOF-Q) m/z: $[M]^+$ calcd for $C_6H_{14}N_2O_2S$, 178.0776; found 178.0772.



9-thia-8,12-diazaspiro[5.8]tetradecane 9,9-dioxide 11b

M.p. 129-133°C, a white solid, 3.3 g (yield 89.7%).

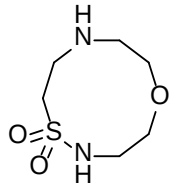
^{1}H NMR (500 MHz, DMSO-d_6) δ 6.94 (br.s, 1H), 3.32 (br.s, 2H), 3.27 (br.s, 2H), 2.88 (br.s, 2H), 2.70 (br.s, 2H), 1.49-1.21 (m, 10H), 1.12-1.08 (m, 2H). ^{13}C NMR (126 MHz, DMSO-d_6) δ 54.6 (CH_2), 51.3 (CH_2), 44.9 (CH_2), 44.6 (CH_2), 36.3 (CH_2), 33.9 (C), 33.0 (CH_2), 25.9 (CH_2), 21.0 (CH_2). HRMS (ESI/TOF-Q) m/z: $[M]^+$ calcd for $C_{11}H_{22}N_2O_2S$, 246.1402; found 246.1395.



1,2,8-thiadiazecane 1,1-dioxide 11c

M.p. 98°C, a white powder, 14.3 g (yield 72.3%). Crude compound was purified by column chromatography ($CHCl_3$/MeOH 90/10, SiO_2) followed by triturating with MTBE (3×80 mL).

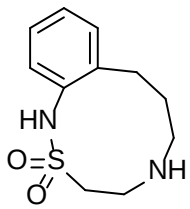
^{1}H NMR (400 MHz, DMSO-d_6) 6.74 (br.s, 1H), 3.18-3.15 (m, 2H), 3.09-3.08 (m, 2H), 2.95-2.92 (m, 2H), 2.59-2.57 (m, 2H), 2.27 (br.s, 1H), 1.58-1.43 (m, 6H). ^{13}C NMR (126 MHz, DMSO-d_6) δ 47.9 (CH_2), 44.7 (CH_2), 43.3 (CH_2), 41.8 (CH_2), 26.9 (CH_2), 25.6 (CH_2), 20.4 (CH_2). HRMS (ESI/TOF-Q) m/z: $[M]^+$ calcd for $C_7H_{16}N_2O_2S$, 192.0932; found 192.0924.



1,5,4,8-oxathiadiazecane 5,5-dioxide 11d

M.p. 79°C, a white powder, 6.4 g (yield 67.1%). Crude compound was purified by column chromatography (first eluent $CHCl_3$/CH_3CN, SiO_2, second eluent MTBE/MeOH 70/30, Al_2O_3).

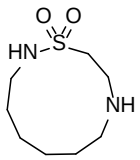
^{1}H NMR (400 MHz, DMSO-d_6) δ 7.02 (br.s, 1H), 3.52-3.48 (m, 4H), 3.26-3.23 (m, 2H), 3.15-3.12 (m, 2H), 3.01-2.97 (m, 2H), 2.75-2.72 (m, 2H). ^{13}C NMR (150 MHz, DMSO-d_6) δ 71.2 (CH_2), 68.6 (CH_2), 51.1 (CH_2), 47.4 (CH_2), 44.3 (CH_2), 43.6 (CH_2). HRMS (ESI/TOF-Q) m/z: $[M]^+$ calcd for $C_6H_{14}N_2O_3S$, 194.0725; found 194.0726.



3,4,5,6,7,8-hexahydro-1H-benzo[c][1,2,8]thiadiazecine 2,2-dioxide 11e

M.p. 154-157°C, a yellow powder, 4.8 g (yield 92.7%).

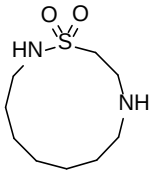
^{1}H NMR (400 MHz, DMSO-d_6) δ 7.60 (d, J =3.80 Hz, 1H), 7.19-7.15 (m, 2H), 7.04-7.01 (m, 1H), 3.36 (br.s, 1H), 3.09 (br.s, 2H), 2.99 (br.s, 2H), 2.69 (br.s, 2H), 2.19 (br.s, 2H), 1.69 (br.s, 2H). ^{13}C NMR (100 MHz, DMSO-d_6) δ 138.5 (C), 131.2 (CH), 130.8 (C), 127.5 (CH), 124.3 (CH), 118.6 (CH), 45.9 (CH_2), 43.5 (CH_2), 41.9 (CH_2), 30.0 (CH_2), 26.6 (CH_2). HRMS (ESI/TOF-Q) m/z: $[M]^+$ calcd for $C_{11}H_{16}N_2O_2S$, 240.0932; found 240.0927.



1-thia-2,9-diazacycloundecane 1,1-dioxide 11f

M.p. 61°C, a white powder, 19.3 g (yield 87.5%)

^{1}H NMR (400 MHz, DMSO-d_6) δ 6.66 (br.s, 1H), 3.13-3.07 (m, 4H), 2.93-2.91 (m, 2H), 2.60 (br.s, 2H), 2.00 (br.s, 1H), 1.43-1.41 (m, 8H). ^{13}C NMR (126 MHz, DMSO-d_6) δ 50.2 (CH_2), 46.7 (CH_2), 44.7 (CH_2), 41.6 (CH_2), 27.9 (CH_2), 27.4 (CH_2), 24.1 (CH_2), 22.9 (CH_2). HRMS (ESI/TOF-Q) m/z: $[M]^+$ calcd for $C_8H_{18}N_2O_2S$, 206.1089; found 206.1079.

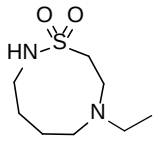


1-thia-2,10-diazacyclododecane 1,1-dioxide 11g

M.p. 68°C, a white powder, 27.1 g (yield 91.1%)

^{1}H NMR (400 MHz, DMSO-d_6) δ 6.93 (br.s, 1H), 3.09 (br.s, 2H), 2.98 (br.s, 2H), 2.86 (br.s, 2H), 2.53 (br.s, 2H), 1.99 (br.s, 1H), 1.51-1.37 (m, 10H). ^{13}C NMR (150 MHz, DMSO-d_6) δ 50.7 (CH_2), 46.4 (CH_2), 42.9 (CH_2), 40.1 (CH_2), 25.9 (CH_2), 25.0

(CH_2), 24.3 (CH_2), 23.5 (CH_2), 23.4 (CH_2). HRMS (ESI/TOF-Q) m/z: $[M]^+$ calcd for $C_9H_{20}N_2O_2S$, 220.1245; found 220.1240.



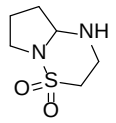
7-ethyl-1,2,7-thiadiazonane 1,1-dioxide 11h

Yellow oil, 13.2 g (yield 86.7%). The compound was prepared in a similar manner to that of **14a**.

^{1}H NMR (400 MHz, DMSO-d_6) δ 6.63 (br.s, 1H), 3.26-3.21 (m, 4H), 2.73 (t, J =6.0 Hz, 2H), 2.54-2.50 (m, 4H), 1.62-1.58 (m, 4H), 0.99 (t, J =7.2 Hz, 3H). ^{13}C NMR (100 MHz, DMSO-d_6) δ 54.5 (CH_2), 53.4 (CH_2), 51.6 (CH_2), 50.0 (CH_2), 42.9 (CH_2), 28.6 (CH_2), 25.9 (CH_2), 12.5 (CH_3). HRMS (ESI/TOF-Q) m/z: $[M]^+$ calcd for $C_8H_{18}N_2O_2S$, 206.1089; found 206.1084.

General procedure for reducing thiadiazine derivatives

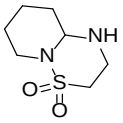
To a solution of annulated thiadiazine **8a** (34.8 g, 200 mmol) in MTBE (150 mL) was added dioxane/HCl till pH=1. The resulting white precipitate was filtered-off, washed with MTBE (3×100 mL) and dried under reduced pressure. The stirred solution of hydrochloride (200 mmol) in dry methanol (200 mL) was cooled to -10°C. $NaBH_4$ (15.1 g, 400 mmol, 2 eq.) was added portionwise to the reaction mixture and was stirred for 24 h. Upon completion, the reaction mixture was diluted with H_2O (30 mL), stirred for 10 min and volatiles were evaporated under reduced pressure. The residue was triturated with $CHCl_3$ (200 mL) and filtered off. The residue was washed with $CHCl_3$ (3×100 mL). The combined washings were dried over Na_2SO_4 and evaporated under reduced pressure to afford a crude **9a**.



Hexahydro-1H-pyrrolo[1,2-b][1,2,4]thiadiazine 4,4-dioxide 9a

M.p. 68°C, a white powder, 32.3 g (yield 91.6%)

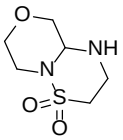
^{1}H NMR (400 MHz, DMSO-d_6) δ 4.42-4.38 (m, 1H), 3.31-3.28 (m, 1H), 3.23-3.19 (m, 2H), 3.07-2.99 (m, 1H), 2.91-2.88 (m, 1H), 2.84-2.77 (m, 1H), 2.68-2.62 (m, 1H), 2.09-2.04 (m, 1H), 2.02-1.90 (m, 1H), 1.85-1.74 (m, 1H), 1.67-1.59 (m, 1H) ^{13}C NMR (150 MHz, DMSO-d_6) δ 76.7 (CH), 45.2 (CH_2), 45.2 (CH_2), 44.0 (CH_2), 31.7 (CH_2), 21.3 (CH_2). HRMS (ESI/TOF-Q) m/z: $[M]^+$ calcd for $C_6H_{12}N_2O_2S$, 176.0619; found 176.0613.



Octahydropyrido[1,2-b][1,2,4]thiadiazine 4,4-dioxide 9c

M.p. 96°C, a white powder, 29.7 g (yield 86.7%)

^{1}H NMR (400 MHz, DMSO-d_6) δ 4.31 (s, 1H), 3.29-3.26 (m, 1H), 3.12-2.97 (m, 4H), 2.90-2.88 (m, 2H), 1.69-1.61 (m, 3H), 1.51-1.41 (m, 3H). ^{13}C NMR (126 MHz, DMSO-d_6) δ 71.3 (CH), 44.7 (CH_2), 43.4 (CH_2), 41.0 (CH_2), 30.7 (CH_2), 24.9 (CH_2), 19.0 (CH_2). HRMS (ESI/TOF-Q) m/z: $[M]^+$ calcd for $C_7H_{14}N_2O_2S$, 190.0776; found 190.0772.

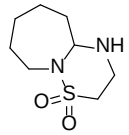


Hexahydro-1H-[1,4]oxazino[4,3-b][1,2,4]thiadiazine 4,4-dioxide 9d

M.p. 172°C, a white powder, 17.5 g (yield 93.1%)

^{1}H NMR (400 MHz, DMSO-d_6) δ 4.20 (d, J =11.6 Hz, 1H), 3.85-3.82 (m, 1H), 3.71-3.68 (m, 1H), 3.59-3.50 (m, 2H), 3.31-2.25 (m, 2H), 3.08-2.94 (m, 5H). ^{13}C NMR (150 MHz, DMSO-d_6) δ 70.0 (CH_2), 69.9 (CH), 66.3 (CH_2), 44.0 (CH_2), 43.4 (CH_2),

41.0 (CH_2). HRMS (ESI/TOF-Q) m/z: $[M]^+$ calcd for $C_6H_{12}N_2O_3S$, 192.0569; found 192.0563.



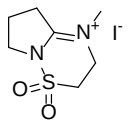
Octahydro-1H-[1,2,4]thiadiazino[2,3-a]azepine 4,4-dioxide 9f

M.p. 81°C, a white powder, 17.9 g (yield 93.2%)

^{1}H NMR (400 MHz, DMSO-d_6) δ 4.46-4.39 (m, 1H), 3.32-3.22 (m, 2H), 3.06-2.97 (m, 2H), 2.90-2.87 (m, 1H), 2.75-2.69 (m, 1H), 2.61-2.53 (m, 1H), 2.21-2.14 (m, 1H), 1.80-1.72 (m, 2H), 1.64-1.57 (m, 1H), 1.38-1.16 (m, 4H). ^{13}C NMR (100 MHz, DMSO-d_6) δ 74.4 (CH), 46.7 (CH_2), 43.8 (CH_2), 41.6 (CH_2), 34.8 (CH_2), 31.3 (CH_2), 30.0 (CH_2), 23.0 (CH_2). HRMS (ESI/TOF-Q) m/z: $[M]^+$ calcd for $C_8H_{16}N_2O_2S$, 204.0932; found 204.0927.

General procedure for the synthesis of tertiary salts 12

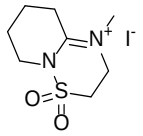
CH_3I (49.7 g, 350 mmol, 7 eq.) was added to a stirring solution of **8a** (8.7 g, 50 mmol) in acetone (100 mL). The reaction mixture was refluxed for 3 h. After cooling to rt the precipitated solid was filtered off, washed with ether (3×100 mL) and dried under argon atmosphere to afford a pure quaternary salt **12a**. Compounds **12d** and **12e** were used in the next step without any characterization.



1-methyl-3,6,7,8-tetrahydro-2H-pyrrolo[1,2-b][1,2,4]thiadiazin-1-ium 4,4-dioxide iodide 12a

M.p. 215-217°C, a white powder, 12.7 g (yield 80.4%)

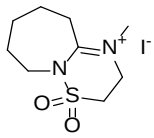
^{1}H NMR (400 MHz, DMSO-d_6) 4.25 (br.s, 2H), 4.19 (br.s, 2H), 4.01-3.97 (m, 2H), 3.36 (s, 3H), 3.26-3.22 (m, 2H), 2.17-2.15 (m, 2H). ^{13}C NMR (100 MHz, DMSO-d_6) δ 169.4 (C), 50.2 (CH_2), 50.0 (CH_2), 44.7 (CH_2), 42.8 (CH_3), 33.6 (CH_2), 18.6 (CH_2). Elemental analysis: Calcd. for $C_7H_{13}IN_2O_2S$ (316.16): C 26.59, H 4.14, I 40.14 N 8.86, O 10.12, S 10.14 Found: C 26.66, H 4.09, I 40.19 N 8.90, S 10.16.



1-methyl-2,3,6,7,8,9-hexahydropyrido[1,2-b][1,2,4]thiadiazin-1-ium 4,4-dioxide iodide 12b

M.p. 173°C, a white powder, 7.2 g (yield 72.9%)

^{1}H NMR (400 MHz, DMSO-d_6) δ 4.33-4.29 (m, 2H), 4.26-4.22 (m, 2H), 3.71-3.67 (m, 2H), 3.33 (s, 3H), 2.92-2.88 (m, 2H), 1.84-1.76 (m, 4H). ^{13}C NMR (100 MHz, DMSO-d_6) δ 165.6, 50.1, 43.9, 43.6, 42.5, 29.5, 20.6, 18.0**.** Elemental analysis: Calcd. for $C_8H_{15}IN_2O_2S$ (330.19): C 29.10, H 4.58, I 38.43, N 8.48, O 9.69, S 9.71 Found: C 29.16, H 4.63, I 38.49, N 8.49, S 9.78.



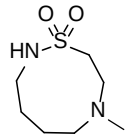
1-methyl-3,6,7,8,9,10-hexahydro-2H-[1,2,4]thiadiazino[2,3-a]azepin-1-ium 4,4-dioxide iodide 12c

M.p. 158-161°C, a white powder, 6.3 g (yield 67.0%)

^{1}H NMR (400 MHz, DMSO-d_6) δ 4.31-4.26 (m, 4H), 4.05-4.02 (m, 2H), 3.47 (s, 3H), 3.06-3.04 (m, 2H), 1.76-1.69 (m, 6H) ^{13}C NMR (126 MHz, DMSO-d_6) δ 169.6 (C), 49.8 (CH_2), 44.2 (CH_2), 43.9 (CH_3), 43.8 (CH_2), 30.4 (CH_2), 26.9 (CH_2), 26.9 (CH_2), 21.6 (CH_2). Elemental analysis: Calcd. for $C_9H_{17}IN_2O_2S$ (344.21): C 31.40, H 4.98, I 36.87, N 8.14, O 9.30, S 9.32 Found: C 31.37, H 5.04, I 36.92, N 8.17, S 9.28.

General procedure for reductive cleavage of tertiary salts 12

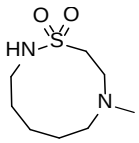
To a stirred solution of **12a** (15.8 g, 50 mmol, 1 eq.) in glacial acetic acid (80 mL) was added portionwise $NaBH_3CN$ (12.6 g, 200 mmol, 4 eq.). The reaction mixture was stirred for 24 h at 80°C. The reaction mixture was diluted with water (40 mL) and allowed cooling to 20°C. It was quenched with 50% aqueous NaOH till pH=14 at 20°C. The precipitated solid was filtered off and washed with $CHCl_3$ (5×75 mL). The filtrate was extracted with $CHCl_3$ (3×70 mL), combined organics were dried over Na_2SO_4 and evaporated under reduced pressure till dryness to afford a pure *N*-Me azasultam **14a.**



7-methyl-1,2,7-thiadiazonane 1,1-dioxide 14a

Yellow oil, 9.0 g (yield 94.2%).

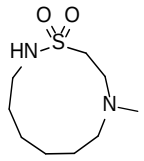
^{1}H NMR (400 MHz, DMSO-d_6) δ 6.65 (br.s, 1H), 3.26-3.23 (m, 4H), 2.64 (t, *J* =6.0 Hz, 2H), 2.44 (m, 2H), 2.25 (s, 3H), 1.58 (m, 4H). ^{13}C NMR (126 MHz, DMSO-d_6) δ 56.2 (CH_2), 53.0 (CH_2), 51.5 (CH_2), 45.4 (CH_3), 42.2 (CH_2), 28.2 (CH_2), 24.5 (CH_2). HRMS (ESI/TOF-Q) m/z: $[M]^+$ calcd for $C_7H_{16}N_2O_2S$, 192.0932; found 192.0929.



8-methyl-1,2,8-thiadiazecane 1,1-dioxide 14b

M.p. 49-52°C, a white powder, 8.7 g (yield 96.1%)

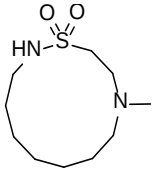
^{1}H NMR (400 MHz, DMSO-d_6) δ 6.45 (br.s, 1H), 3.25-3.22 (m, 2H), 3.09 (br.s, 2H), 2.66-2.64 (m, 2H), 2.34 (br.s, 2H), 2.15 (s, 3H), 1.49 (br.s, 6H). ^{13}C NMR (126 MHz, DMSO-d_6) δ 52.7 (CH_2), 52.5 (CH_2), 49.3 (CH_2), 42.9 (CH_3), 42.1 (CH_2), 26.4 (CH_2), 25.3 (CH_2), 20.7 (CH_2). HRMS (ESI/TOF-Q) m/z: $[M]^+$ calcd for $C_8H_{18}N_2O_2S$, 206.1089; found 206.1080.



9-methyl-1-thia-2,9-diazacycloundecane 1,1-dioxide 14c

M.p. 63-66°C, a white powder, 7.3 g (yield 92.7%)

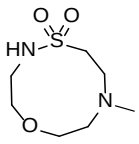
^{1}H NMR (400 MHz, DMSO-d_6) δ 6.50 (br.s, 1H), 3.19-3.17 (m, 2H), 3.06 (br.s, 2H), 2.65-2.63 (m, 2H), 2.38 (m, 2H), 2.09 (s, 3H), 1.40-1.34 (m, 8H). ^{13}C NMR (126 MHz, DMSO-d_6) δ 53.7 (CH_2), 53.3 (CH_2), 50.3 (CH_2), 41.4 (CH_3), 40.0 (CH_2), 26.7 (CH_2), 24.5 (CH_2), 22.3 (CH_2), 20.9 (CH_2). HRMS (ESI/TOF-Q) m/z: $[M]^+$ calcd for $C_9H_{20}N_2O_2S$, 220.1245; found 220.1235.



10-methyl-1-thia-2,10-diazacyclododecane 1,1-dioxide 14d

M.p. 68-71°C, a white powder, 7.8 g (yield 94.7%)

^{1}H NMR (400 MHz, DMSO-d_6) δ 7.12 (br.s, 1H), 3.14 (br.s, 2H), 2.99 (br.s, 2H), 2.58 (br.s, 2H), 2.32 (br.s, 2H), 2.13 (s, 3H), 1.44-1.35 (m, 10H). ^{13}C NMR (100 MHz, DMSO-d_6) δ 55.8 (CH_2), 50.5 (CH_2), 48.7 (CH_2), 43.6 (CH_3), 40.9 (CH_2), 26.2 (CH_2), 24.6 (CH_2), 23.1 (CH_2), 22.8 (CH_2), 22.4 (CH_2). HRMS (ESI/TOF-Q) m/z: $[M]^+$ calcd for $C_{10}H_{22}N_2O_2S$, 234.1402; found 234.1395.



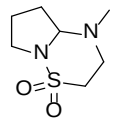
8-methyl-1,5,4,8-oxathiadiazecane 5,5-dioxide 14e (purity 92%)

M.p. 105-109°C, a white powder, 4.7 g (yield 94.0%)

^{1}H NMR (400 MHz, DMSO-d_6) δ 6.89 (br.s, 1H), 3.47-3.42 (m, 4H), 3.19 (br.s, 2H), 3.03 (br.s, 2H), 2.85-2.82 (br.s, 2H), 2.43 (br.s, 2H), 2.18 (s, 3H). ^{13}C NMR (126 MHz, DMSO-d_6) δ 68.9 (CH_2), 68.5 (CH_2), 53.4 (CH_2), 50.1 (CH_2), 47.7 (CH_2), 43.4 (CH_2), 43.0 (CH_3). HRMS (ESI/TOF-Q) m/z: $[M]^+$ calcd for $C_7H_{16}N_2O_3S$, 208.0882; found 208.0874.

General procedure of reduction reaction of tertiary salts 12

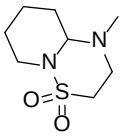
The stirred solution of tertiary salt **12a** (25.3 g, 80 mmol, 1 eq.) in dry methanol (200 mL) was cooled to -10°C. To the reaction mixture was added portionwise $NaBH_4$ (6.0 g, 160 mmol, 2 eq.) and was stirred for 24 h. The reaction mixture was diluted with H_2O (20 mL), stirred for 10 min and volatiles were evaporated under reduced pressure. The residue was triturated with $CHCl_3$ (150 mL) and filtered off. The residue was washed with $CHCl_3$ (3×100 mL). The washings were collected, dried over Na_2SO_4 and evaporated under reduced pressure to afford **13a.**



1-methylhexahydro-1H-pyrrolo[1,2-b][1,2,4]thiadiazine 4,4-dioxide 13a

M.p. 96-99°C, a white powder, 13.3 g (yield 87.8%)

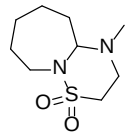
^{1}H NMR (400 MHz, DMSO-d_6) δ 3.92-3.90 (m, 1H), 3.34-3.20 (m, 3H), 3.17-3.13 (m, 1H), 3.10-3.04 (m, 1H), 2.85-2.79 (m, 1H), 2.19 (s, 3H), 2.02-1.76 (m, 4H) ^{13}C NMR (100 MHz, DMSO-d_6) δ 81.2 (CH), 53.5 (CH_2), 46.1 (CH_2), 45.7 (CH_2), 38.8 (CH), 30.3 (CH_2), 21.5 (CH_2). HRMS (ESI/TOF-Q) m/z: $[M]^+$ calcd for $C_7H_{14}N_2O_2S$, 190.0776; found 190.0772.



1-methyloctahydropyrido[1,2-b][1,2,4]thiadiazine 4,4-dioxide 13b

M.p. 74-76°C, a white powder, 5.1 g (yield 91.1%)

^{1}H NMR (400 MHz, DMSO-d_6) δ 3.92 (s, 1H), 3.36-3.29 (m, 1H), 3.23-3.08 (m, 4H), 2.70-2.63 (m, 1H), 2.18 (s, 3H), 2.01-1.99 (m, 1H), 1.72-1.69 (m, 1H), 1.53-1.43 (m, 4H). ^{13}C NMR (126 MHz, DMSO-d_6) δ 75.7 (CH), 53.3 (CH_2), 44.9 (CH_2), 42.8 (CH_2), 39.4 (CH_3), 28.2 (CH_2), 24.9 (CH_2), 18.2 (CH_2). HRMS (ESI/TOF-Q) m/z: $[M]^+$ calcd for $C_8H_{16}N_2O_2S$, 204.0932; found 204.0929.



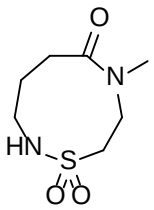
1-methyloctahydro-1H-[1,2,4]thiadiazino[2,3-a]azepine 4,4-dioxide 13c

Yellow oil, 6.7 g (yield 84.6%)

^{1}H NMR (400 MHz, DMSO-d_6) δ 4.08-4.04 (m, 1H), 3.52-4.48 (m, 1H), 3.11-3.05 (m, 4H), 2.90-2.79 (m, 1H), 2.20 (s, 3H), 2.15-2.08 (m, 1H), 1.76-1.69 (m, 3H), 1.57-1.48 (m, 1H), 1.40-1.22 (m, 3H). ^{13}C NMR (126 MHz, DMSO-d_6) δ 78.1 (CH), 52.9 (CH_2), 45.8 (CH_2), 43.3 (CH_2), 37.4 (CH_3), 32.4 (CH_2), 30.9 (CH_2), 29.1 (CH_2), 22.8 (CH_2). HRMS (ESI/TOF-Q) m/z: $[M]^+$ calcd for $C_9H_{18}N_2O_2S$, 218.1089; found 218.1083.

Procedure for hydrolytic cleavage of tertiary salt 12a

Solid K_2CO_3 (97 mg, 0.7 mmol, 2.2 eq) was added to the solution of tertiary salt **12a** (100 mg, 0.32 mmol, 1 eq.) in water (3 mL). The reaction mixture stirred at room temperature for 24h. It was extracted with $CHCl_3$ (3×15 mL), the combined extracts were dried under Na_2SO_4 and concentrated under reduced pressure to afford **15**.



7-methyl-1,2,7-thiadiazonan-6-one 1,1-dioxide 15

M.p. 191°C, a white powder, 51 mg (yield 78.5%).

^{1}H NMR (500 MHz, DMSO-d_6) δ 6.99 (t, J =5.50 Hz, 1H), 3.82-3.80 (m, 2H), 3.25-3.23 (m, 2H), 3.19-3.15 (m, 2H), 2.79 (s, 3H), 2.39-2.37 (m, 2H), 1.77-1.72 (m, 2H). ^{13}C NMR (126 MHz, DMSO-d_6) δ 173.6 (C), 51.0 (CH_2), 45.9 (CH_2), 41.8 (CH_2), 32.9 (CH_3), 29.4 (CH_2), 27.3 (CH_2). HRMS (ESI/TOF-Q) m/z: $[M]^+$ calcd for $C_7H_{14}N_2O_3S$, 206.0725; found 206.0716.

X-ray experimental part

X-ray diffraction studies of compounds **11g** ($C_9H_{20}N_2O_2S$, H_2O) and **15** ($C_7H_{14}N_2O_3S$) have been performed using the Bruker APEX II diffractometer (graphite monochromated MoK_α radiation, CCD detector, φ- and ω-scaning, $2\Theta_{max}$ = 50°). The structures were solved by direct method using SHELXTL package [1, 2]. Positions of the hydrogen atoms were located from electron density difference maps and refined using "riding" model with U_{iso} = nU_{eq} (n=1.5 for methyl group or n=1.2 for other hydrogen atoms) of the carrier atom.

Crystallographic data for **11g**:

The colourless crystals of **11g** are monoclinic. At 173 K a = 10.7318(3), b = 8.4291(3), c = 13.947(5) Å, β = 99.819(2)º, V = 1243.22(7) Å^3, M_r = 238.34, Z = 4, space group $P2_1/n$, d_{calc}= 1.273 g/cm^3, μ(MoK_α) = 0.253 mm^{-1}, F(000) = 520. Intensities of 15580 reflections (2190 independent, R_{int}=0.022) were measured. Full-matrix least-squares refinement against F^2 in anisotropic approximation for non-hydrogen atoms

using 2190 reflections was converged to wR_2 = 0.097 (R_1 = 0.038 for 1855 reflections with F>4σ(F), S = 1.046). The final atomic coordinates, and crystallographic data for molecule **11g** have been deposited to with the Cambridge Crystallographic Data Centre, 12 Union Road, CB2 1EZ, UK (fax: +44-1223-336033; e-mail: deposit@ccdc.cam.ac.uk) and are available on request quoting the deposition numbers CCDC 2266969).

Crystallographic data for **15**:

The colourless crystals of **15** are monoclinic. At 173 K *a* = 9.4126(2), *b* = 9.9316(3), *c* = 10.3407(3) Å, *β* = 103.903(2)º, V = 938.35(4) Å^3, M_r = 206.26, Z = 4, space group $P2_1/c$, d_{calc}= 1.460 g/cm^3, μ(MoK_α) = 0.323 mm^{-1}, F(000) = 440. Intensities of 9961 reflections (1647 independent, R_{int}=0.016) were measured. Full-matrix least-squares refinement against F^2 in anisotropic approximation for non-hydrogen atoms using 1647 reflections was converged to wR_2 = 0.086 (R_1 = 0.032 for 1507 reflections with F>4σ(F), S = 1.070). The final atomic coordinates, and crystallographic data for molecule **15** have been deposited to with the Cambridge Crystallographic Data Centre, 12 Union Road, CB2 1EZ, UK (fax: +44-1223-336033; e-mail: deposit@ccdc.cam.ac.uk) and are available on request quoting the deposition numbers CCDC 2266968).

Reference:

1. Sheldrick GM (2015) Acta Crystallographica Section A Foundations and Advances, 71, 3–8
2. Sheldrick, G. M. (2015). *Acta Crystallographica Section C*, **71**, 3–8

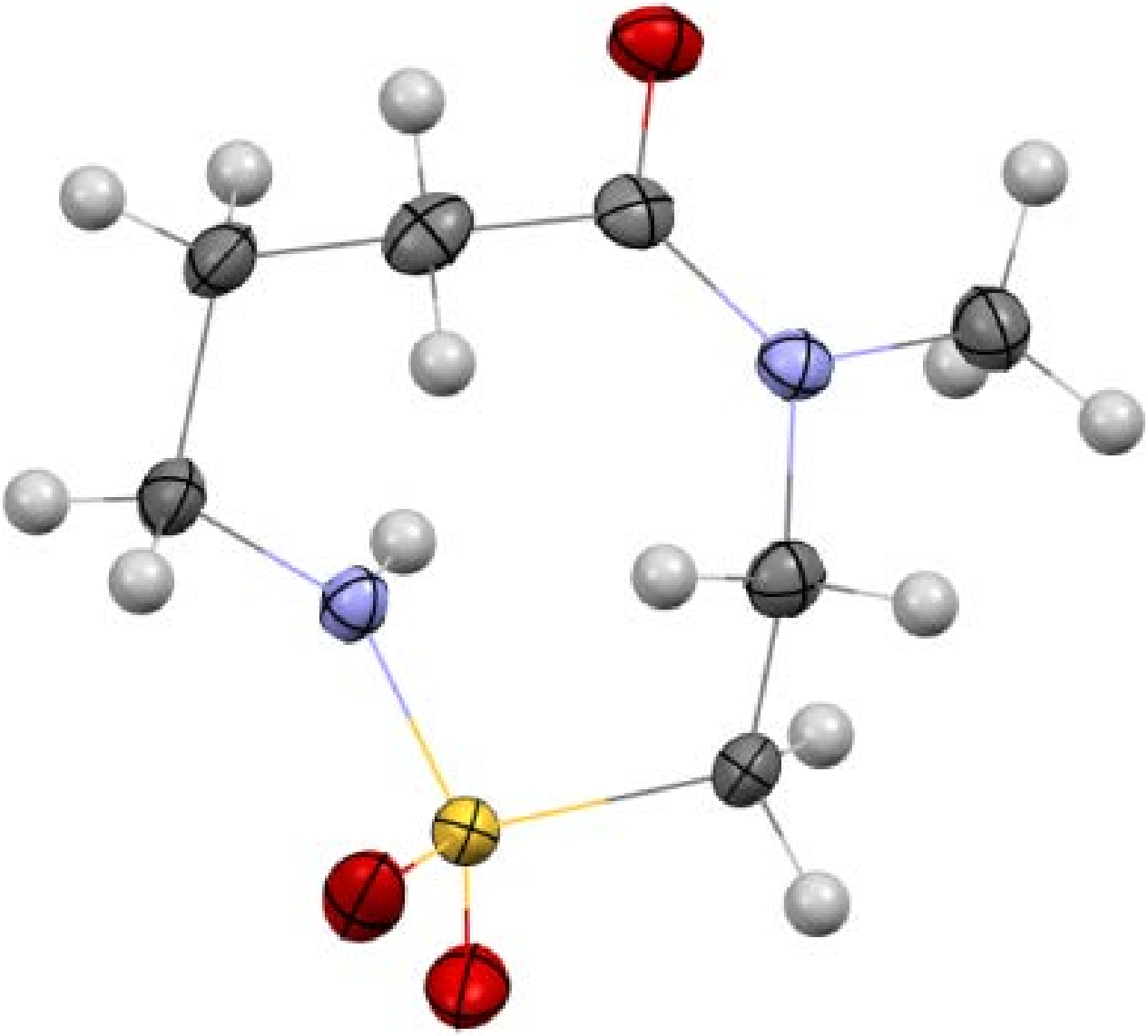


Figure S1. Molecular structure of compound **15** with the thermal displacements of non-hydrogen atoms at the 50% probability level.

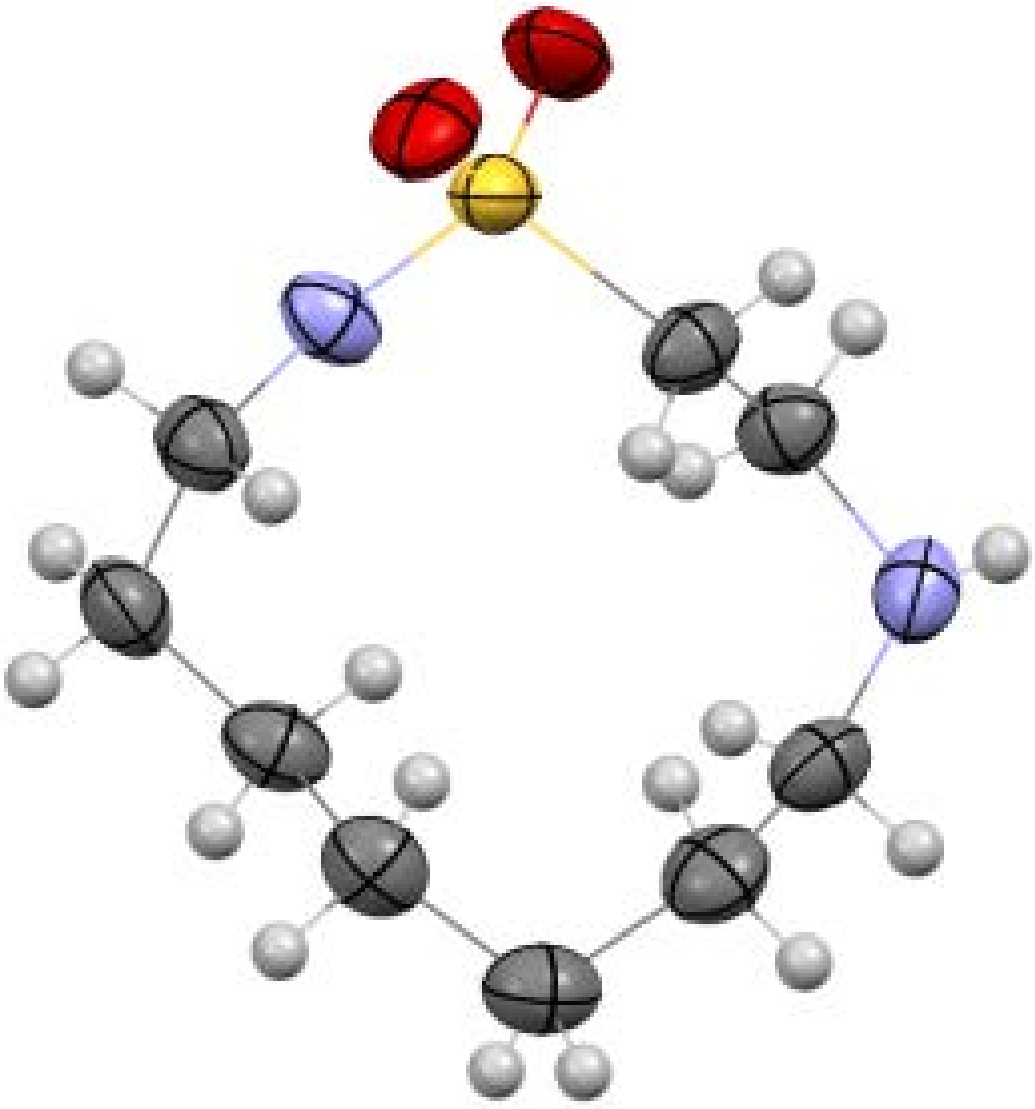
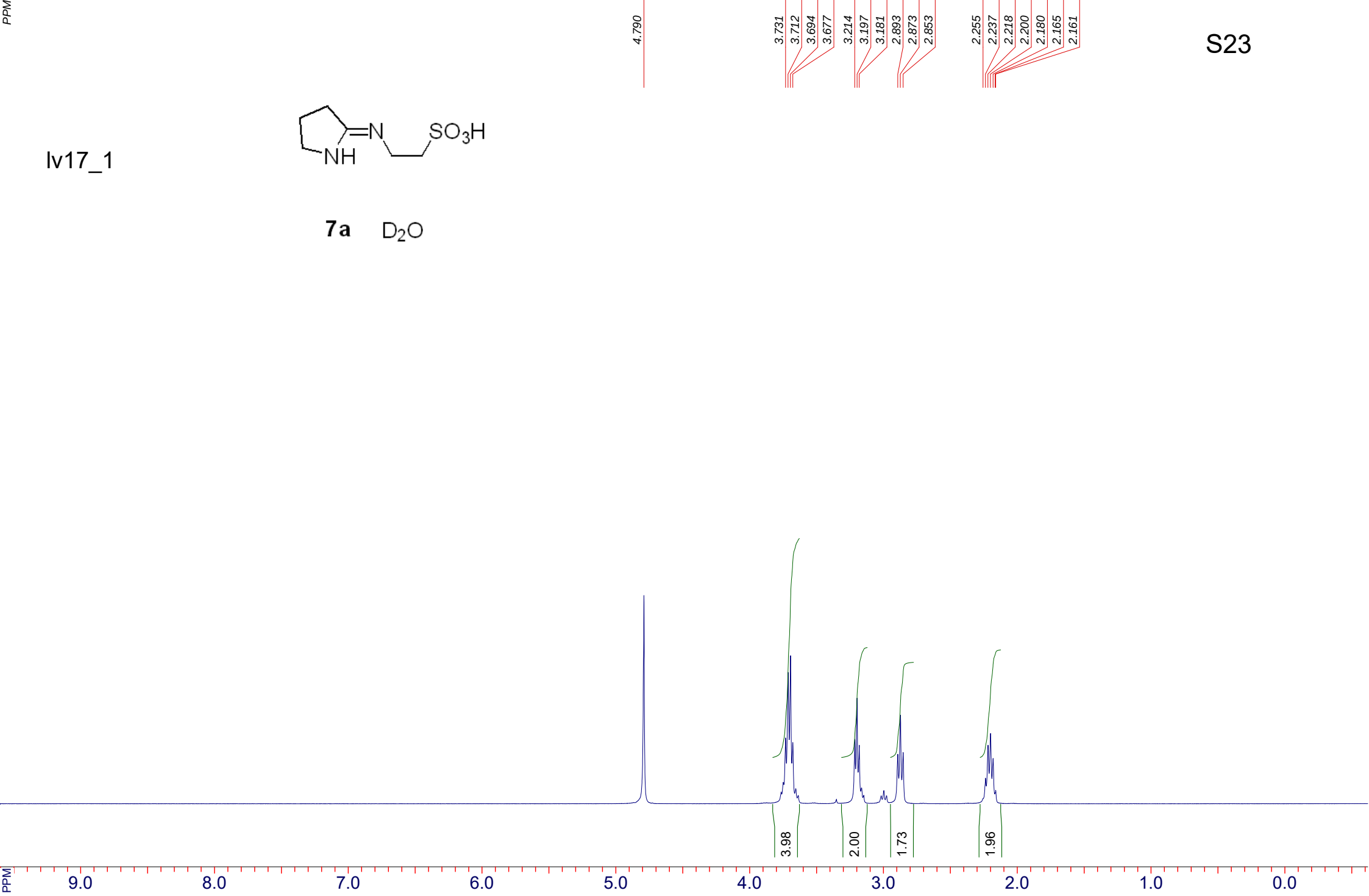


Figure S2. Molecular structure of compound **11g** with the thermal displacements of non-hydrogen atoms at the 50% probability level.

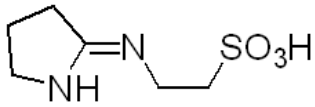
lv17_1

7a D_2O

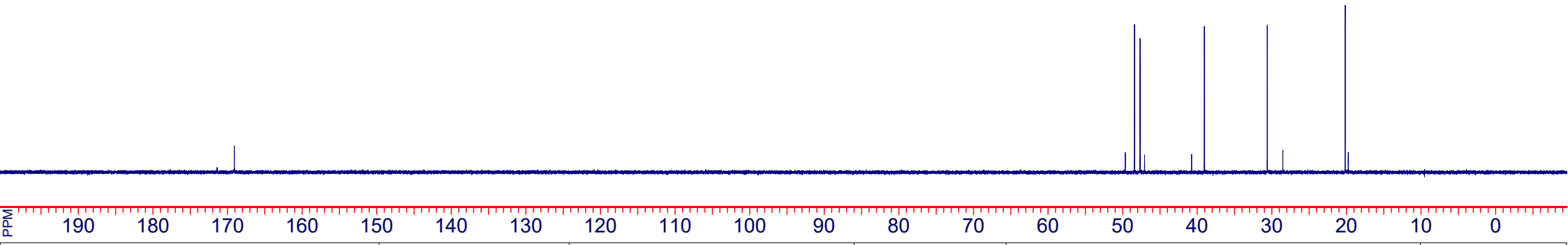


File name: lv17_1	Operator: nmr	SF: 400.1300 MHz	NSC: 1	PW: 0.00 usec, RG: 25	SI: 32768
Date: 30-May-2022	Solvent: DMSO	SW: 8224 Hz	TE: 300 K	AQ: 1.99 sec, RD: 0.00 sec	

lv-17_C13.fid



7a D_2O



File name: lv-17_C13.fid	Operator:	SF: 150.8301 MHz	NSC: 0	PW: 3.09 usec, RG: 60	SI: 65536
Date: 02-Jun-2022	Solvent: d2o	SW: 37879 Hz	TE: 298 K	AQ: 0.87 sec, RD: 0.00 sec	

PPM

169.10

48.43

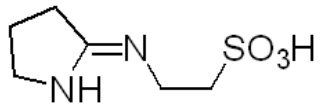
47.68

39.07

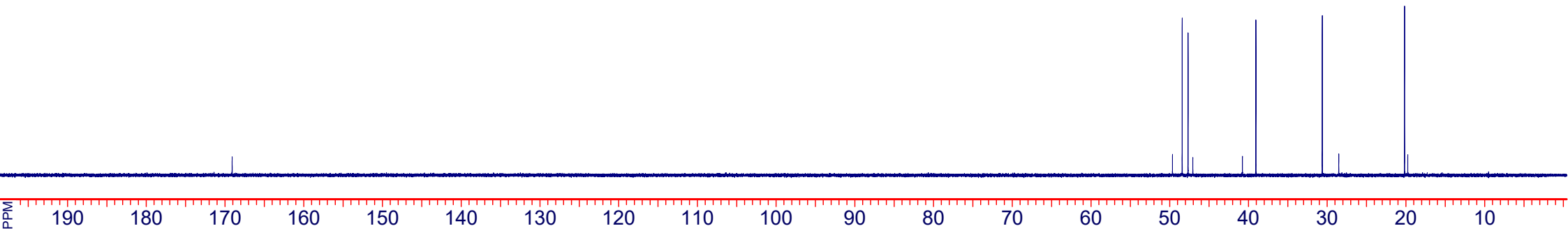
30.63

30.18

lv-17_C13APT.fid

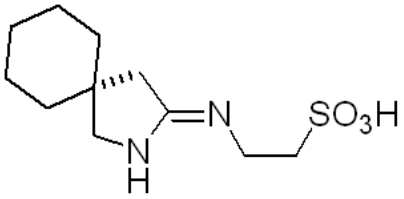


7a D_2O

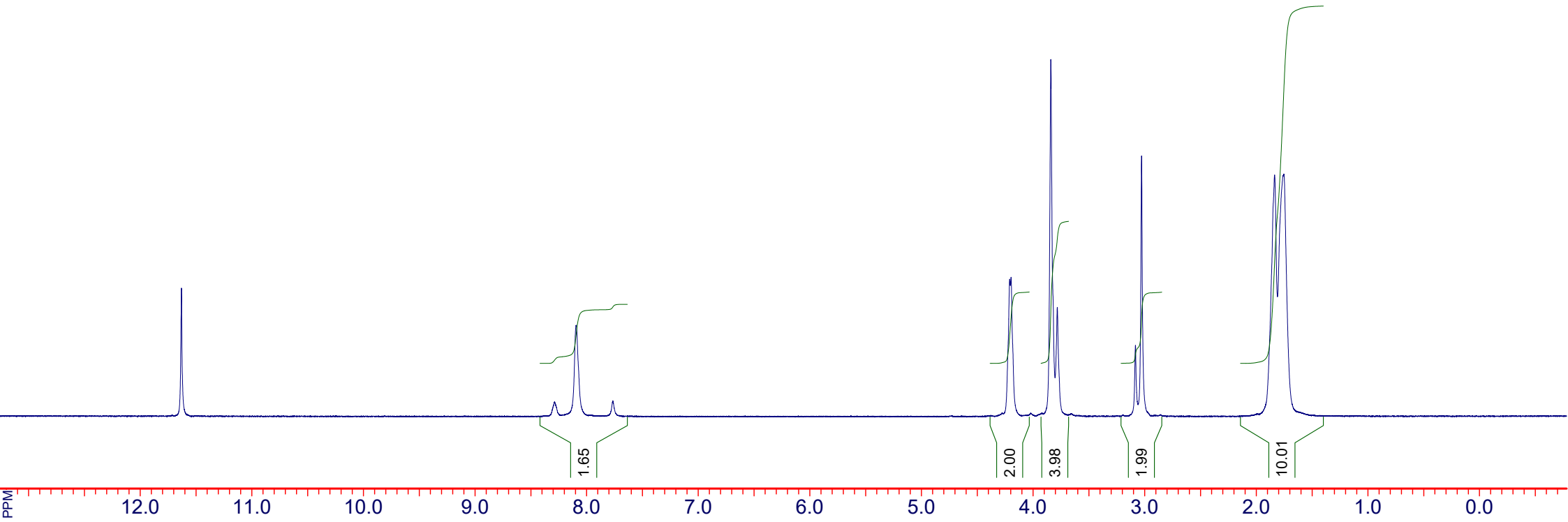


File name: lv-17_C13APT.fid	Operator:	SF: 150.8329 MHz	NSC: 0	PW: 4.63 usec, RG: 60	SI: 131072
Date: 02-Jun-2022	Solvent: d2o	SW: 39063 Hz	TE: 298 K	AQ: 0.87 sec, RD: 0.00 sec	

lv-615.fid



7b CF_3COOD



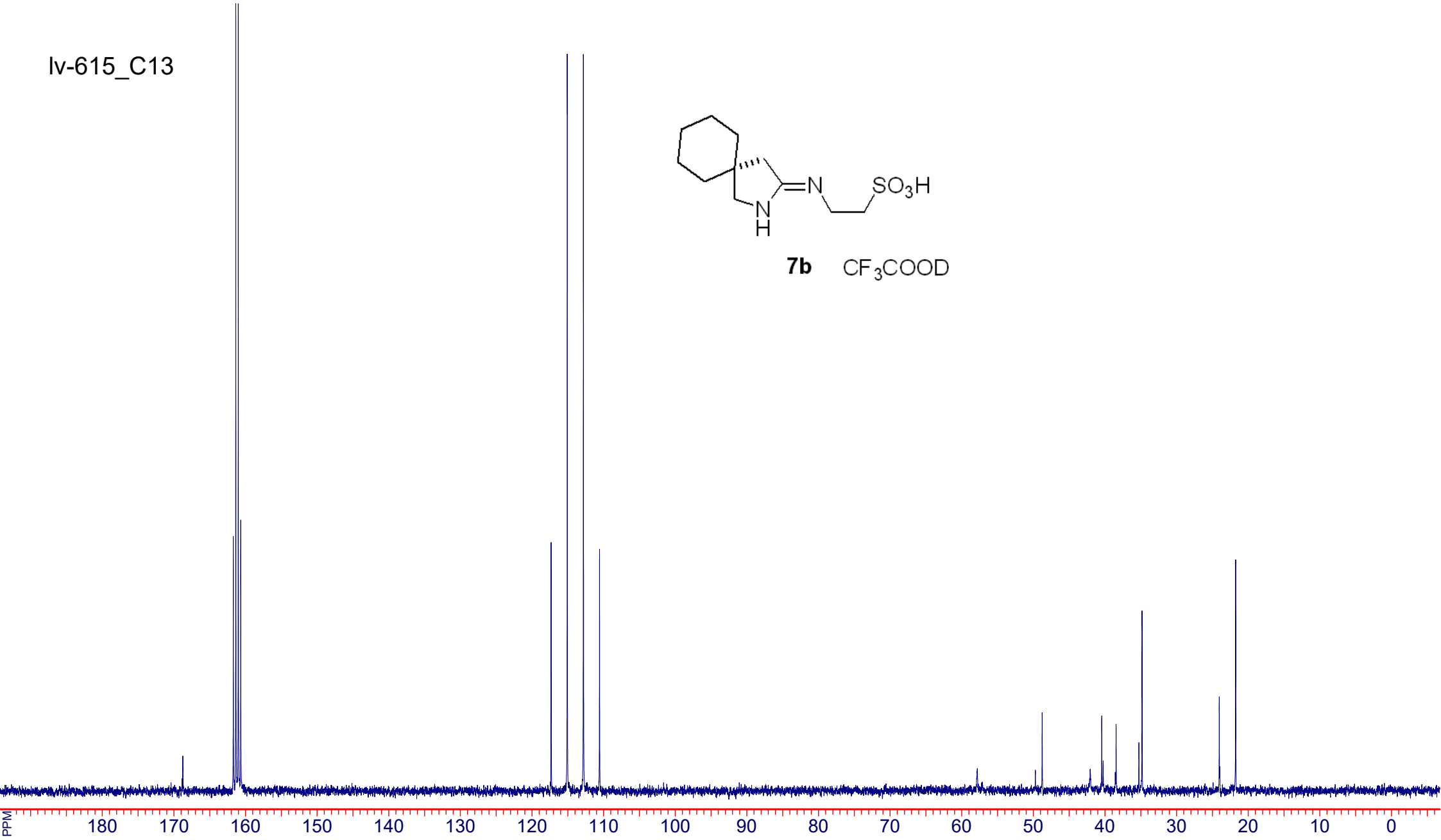
File name: lv-615.fid	Operator:	SF: 399.9697 MHz	NSC: 0	PW: 10.90 usec, RG: 10	SI: 32768
Date: 27-Dec-2022	Solvent: TFA	SW: 8000 Hz	TE: 298 K	AQ: 2.00 sec, RD: 0.00 sec	

PPM

168.75 161.70 161.36 161.01 160.66 117.34 115.08 112.83 110.57 57.83 49.73 48.78 42.07 40.46 38.46 35.27 34.83 24.05 21.76

lv-615_C13

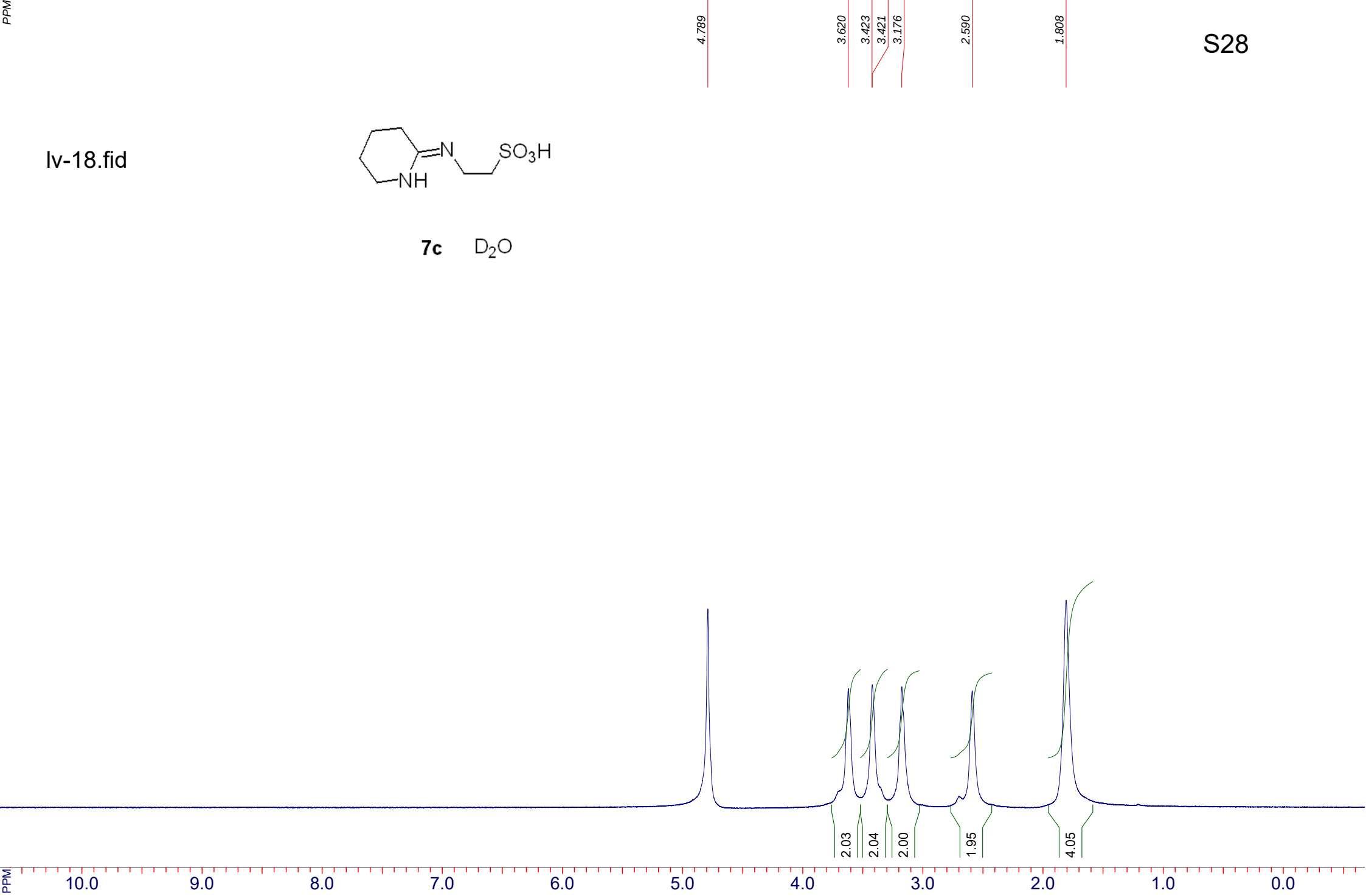
7b CF_3COOD



File name: lv-615_C13	Operator: root	SF: 125.6429 MHz	NSC: 97	PW: 0.00 usec, RG: 51200	SI: 131072
Date: 28-Dec-2022	Solvent: TFA	SW: 32680 Hz	TE: 683 K	AQ: 1.57 sec, RD: 0.00 sec	

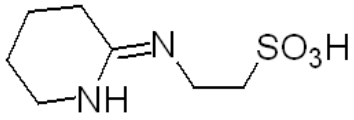
lv-18.fid

7c D_2O

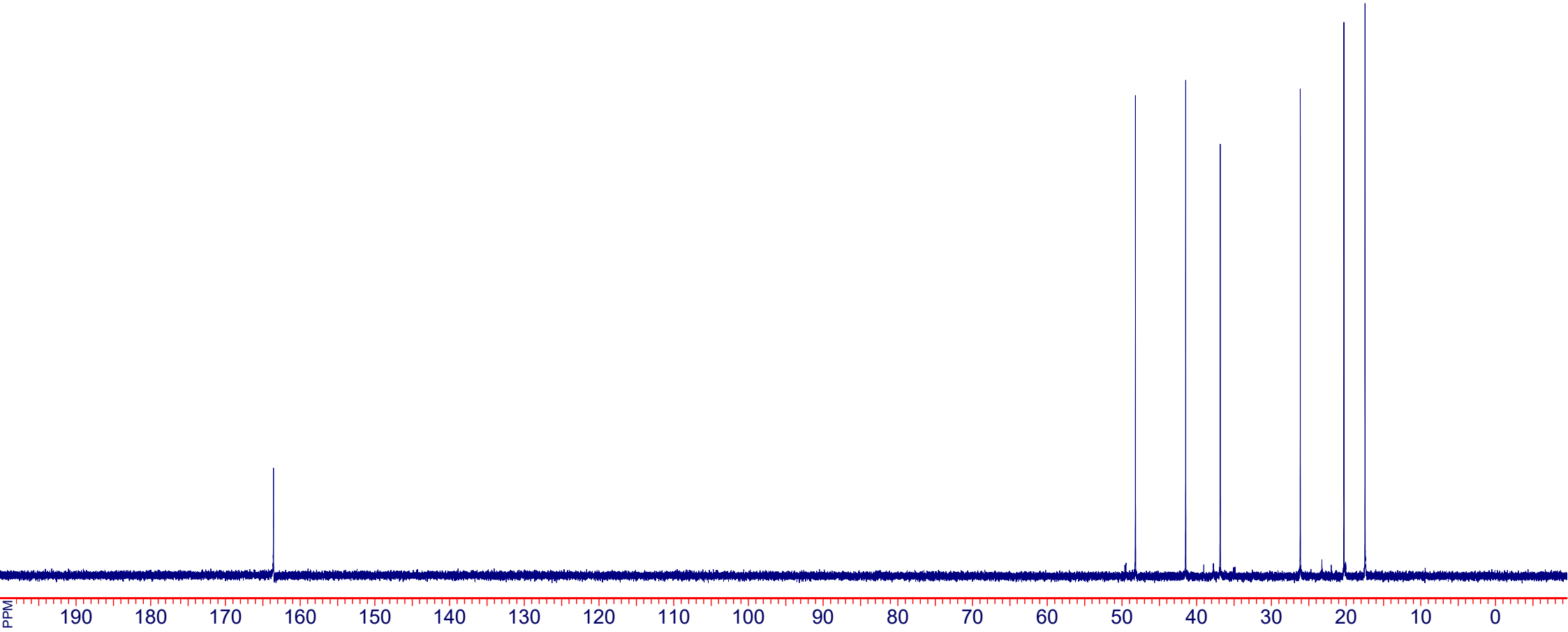


File name: lv-18.fid	Operator:	SF: 399.9724 MHz	NSC: 0	PW: 10.90 usec, RG: 10	SI: 32768
Date: 30-May-2022	Solvent: d2o	SW: 8000 Hz	TE: 298 K	AQ: 2.00 sec, RD: 0.00 sec	

lv-18_C13.fid

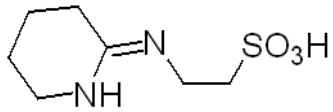


7c D_2O

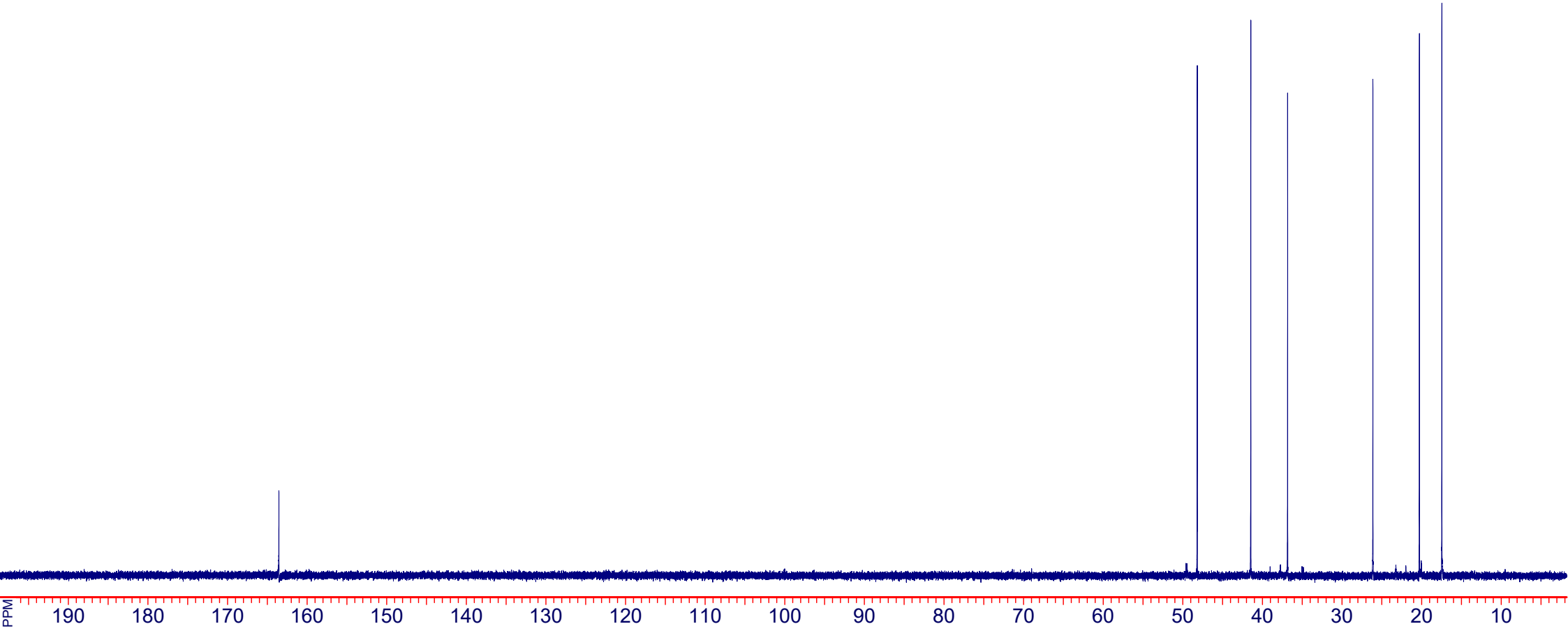


File name: lv-18_C13.fid	Operator:	SF: 150.8301 MHz	NSC: 0	PW: 3.09 usec, RG: 60	SI: 65536
Date: 02-Jun-2022	Solvent: d2o	SW: 37879 Hz	TE: 298 K	AQ: 0.87 sec, RD: 0.00 sec	

lv-18_C13APT.fid



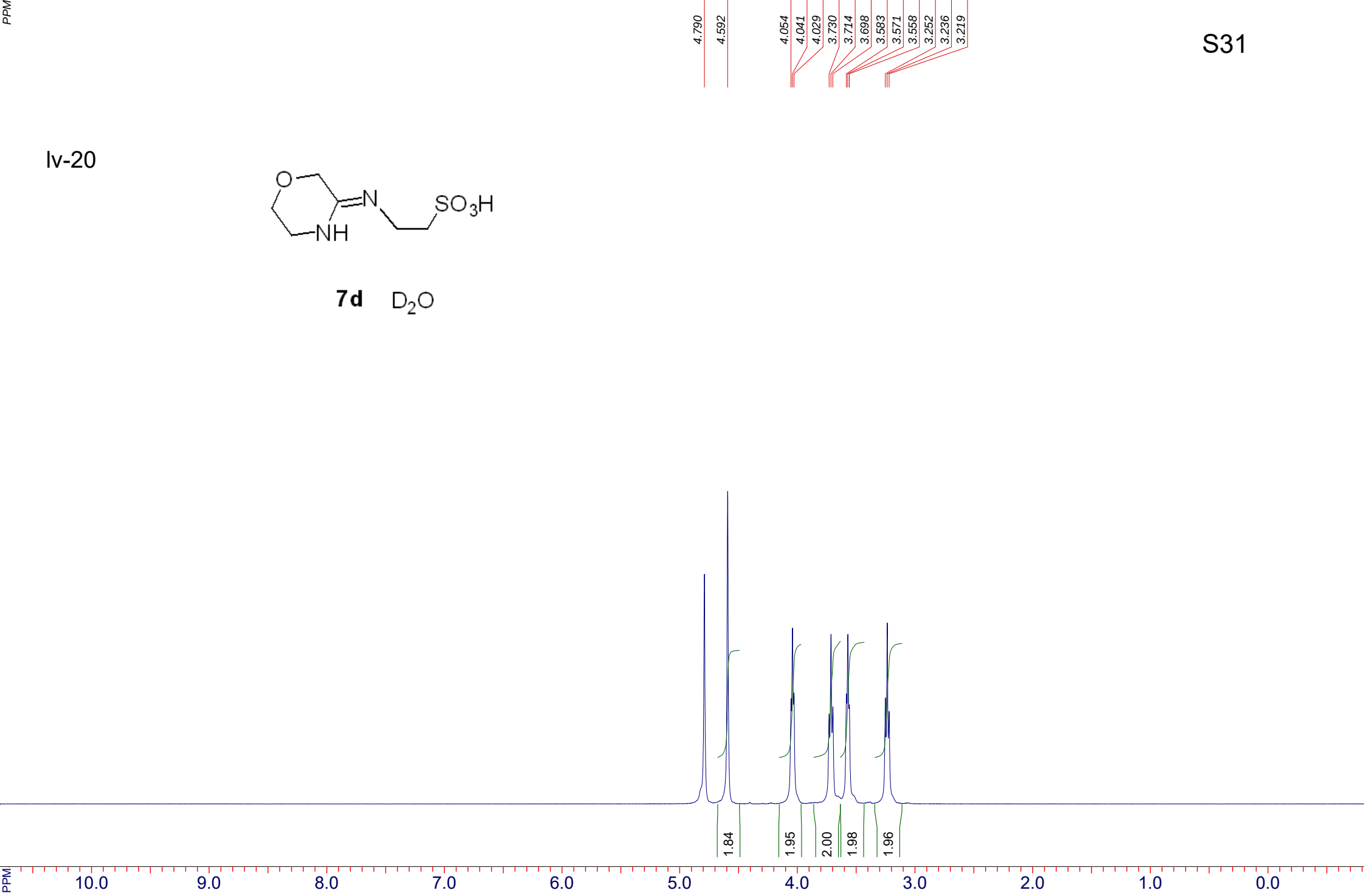
7c D_2O



File name: lv-18_C13APT.fid	Operator:	SF: 150.8329 MHz	NSC: 0	PW: 4.63 usec, RG: 60	SI: 131072
Date: 02-Jun-2022	Solvent: d2o	SW: 39063 Hz	TE: 298 K	AQ: 0.87 sec, RD: 0.00 sec	

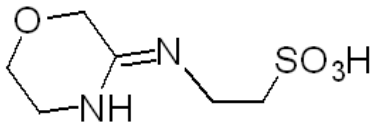
lv-20

7d D_2O

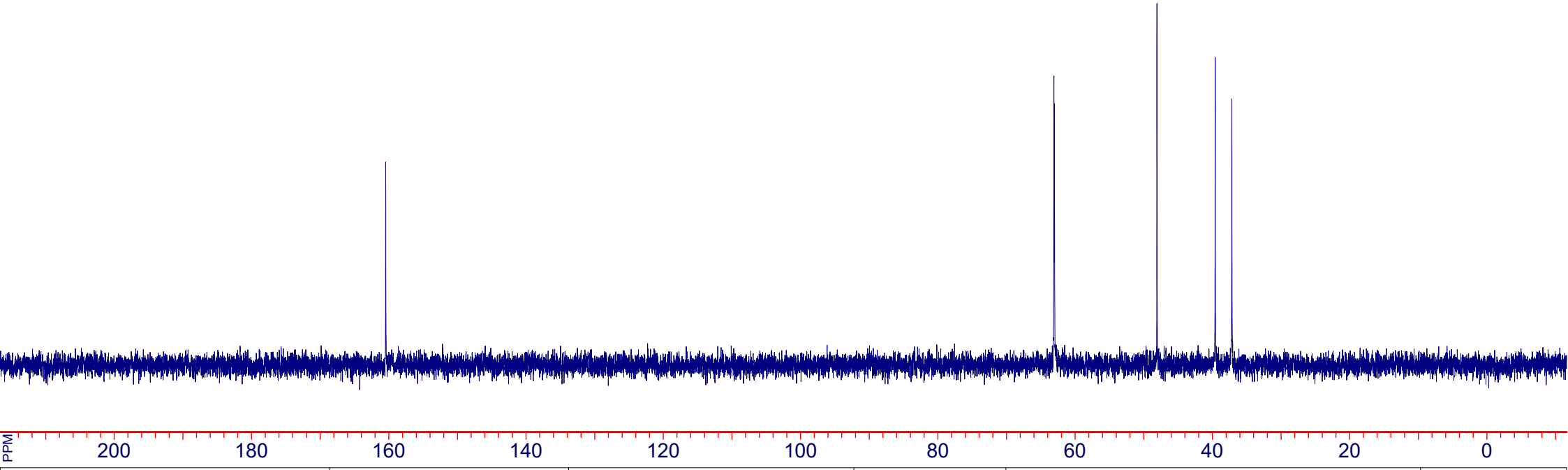


File name: lv-20	Operator: nmr	SF: 400.1300 MHz	NSC: 1	PW: 0.00 usec, RG: 22	SI: 32768
Date: 08-Jun-2022	Solvent: D2O	SW: 8224 Hz	TE: 300 K	AQ: 1.99 sec, RD: 0.00 sec	

lv-20_C13



7d D_2O



File name: lv-20_C13	Operator: nmr	SF: 100.6128 MHz	NSC: 175	PW: 0.00 usec, RG: 2050	SI: 32768
Date: 08-Jun-2022	Solvent: D2O	SW: 26042 Hz	TE: 300 K	AQ: 1.26 sec, RD: 0.00 sec	

PPM

160.42

63.09

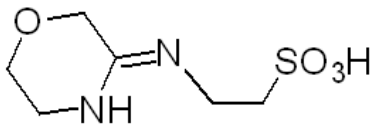
63.00

48.07

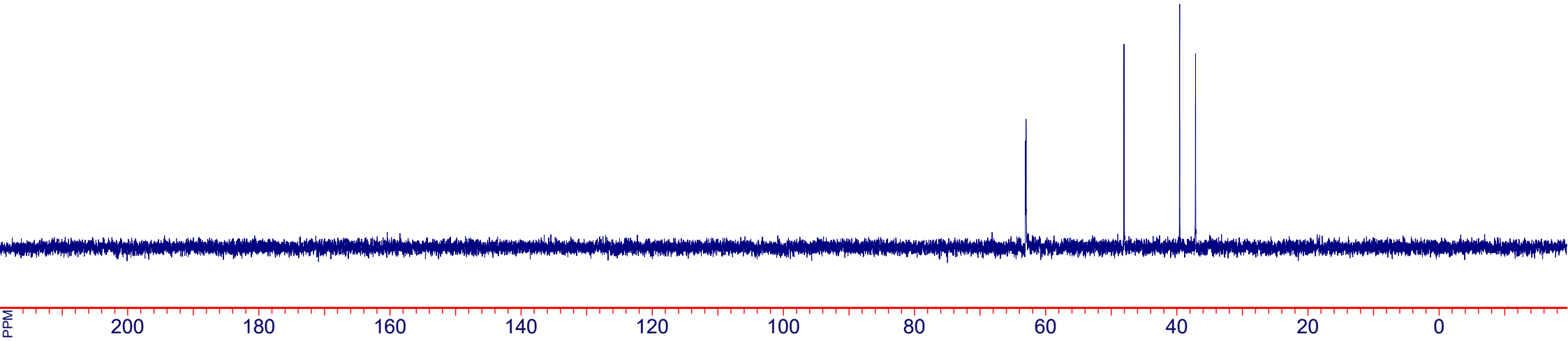
39.58

37.16

lv-20_C13APT

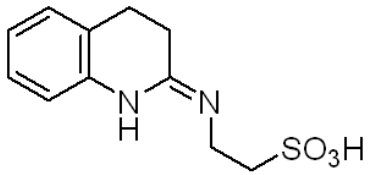


7d D_2O

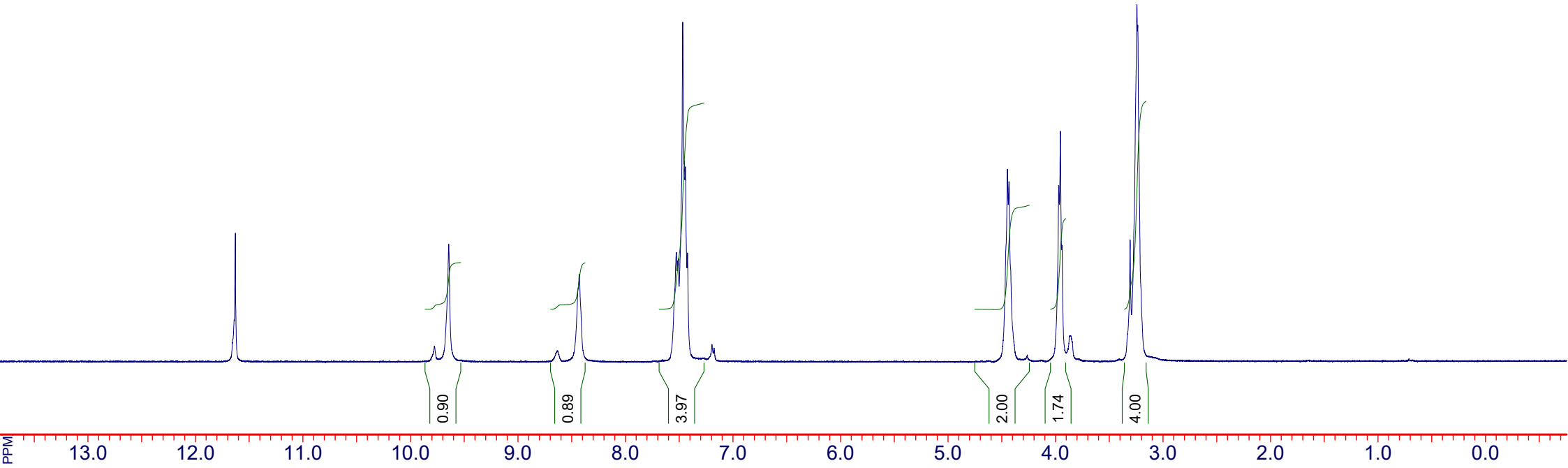


File name: lv-20_C13APT	Operator: nmr	SF: 100.6128 MHz	NSC: 1326	PW: 0.00 usec, RG: 2050	SI: 65536
Date: 09-Jun-2022	Solvent: D2O	SW: 24038 Hz	TE: 300 K	AQ: 1.06 sec, RD: 0.00 sec	

lv-488.fid



7e CF_3COOD



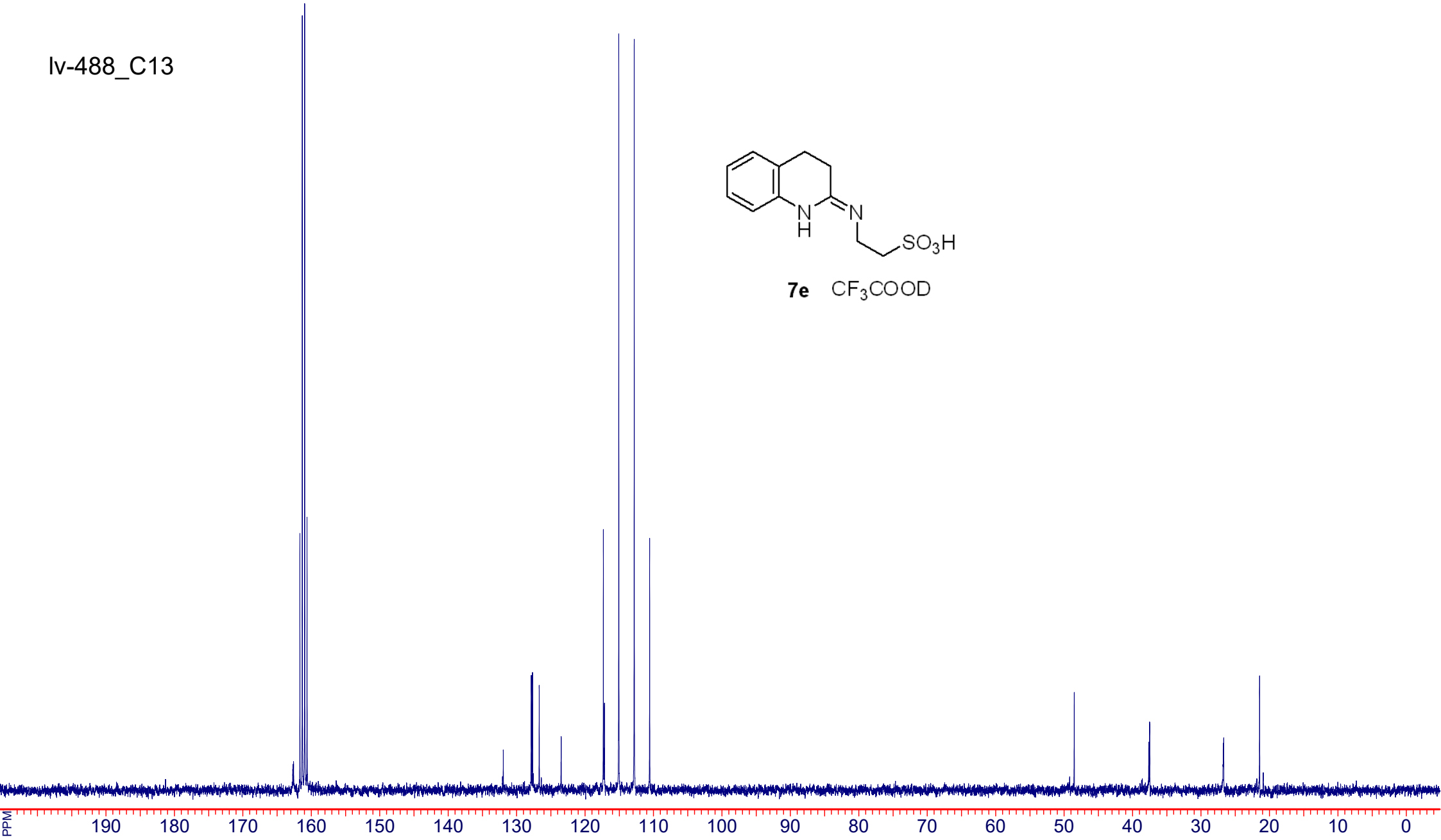
File name: lv-488.fid	Operator:	SF: 399.9697 MHz	NSC: 0	PW: 10.90 usec, RG: 10	SI: 32768
Date: 27-Dec-2022	Solvent: TFA	SW: 8000 Hz	TE: 298 K	AQ: 2.00 sec, RD: 0.00 sec	

PPM

162.67 161.66 161.32 160.97 160.62 131.95 127.86 127.68 126.70 123.49 117.33 117.17 115.07 112.82 110.57 48.53 37.52 26.70 21.45

lv-488_C13

7e CF_3COOD



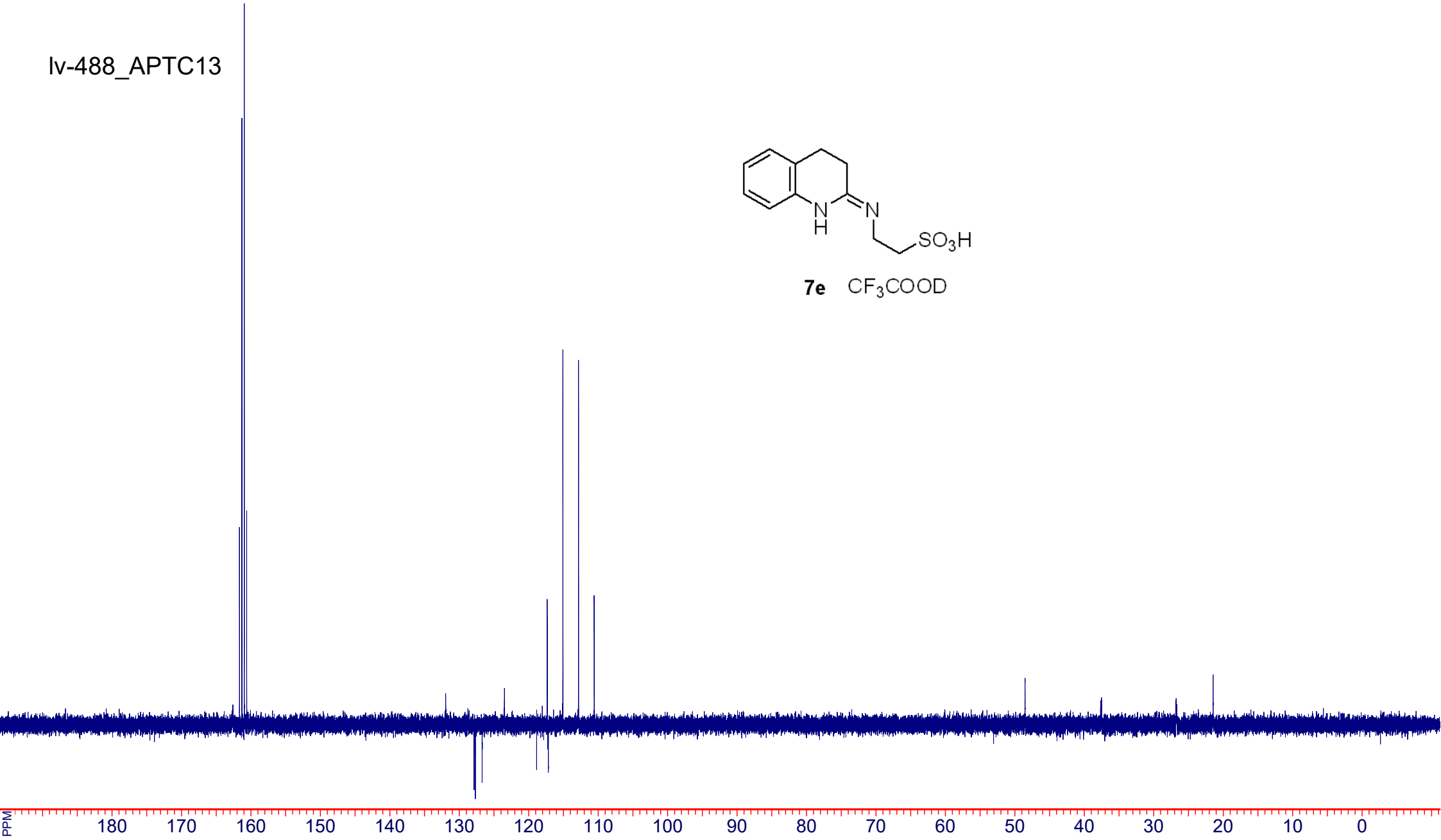
File name: lv-488_C13	Operator: root	SF: 125.6429 MHz	NSC: 60	PW: 0.00 usec, RG: 51200	SI: 131072
Date: 28-Dec-2022	Solvent: TFA	SW: 32680 Hz	TE: 683 K	AQ: 1.57 sec, RD: 0.00 sec	

PPM

162.66 161.65 161.30 160.96 160.61 131.96 127.87 127.69 126.72 123.50 117.35 117.19 115.09 112.84 110.58 48.55 37.54 26.79 21.46

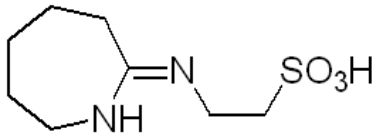
lv-488_APTC13

7e CF_3COOD

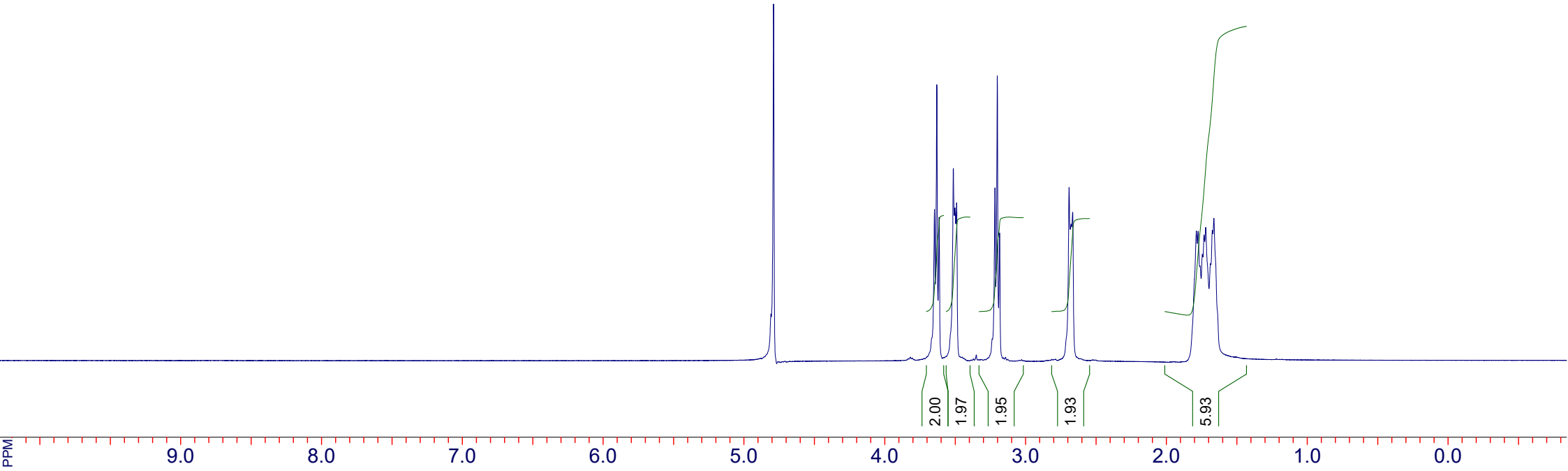


File name: lv-488_APTC13	Operator: root	SF: 125.6429 MHz	NSC: 74	PW: 0.00 usec, RG: 51200	SI: 65536
Date: 28-Dec-2022	Solvent: DMSO	SW: 32680 Hz	TE: 683 K	AQ: 1.57 sec, RD: 0.00 sec	

lv-19.fid

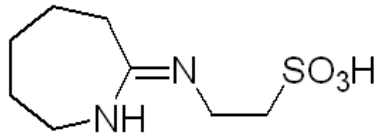


7f D_2O

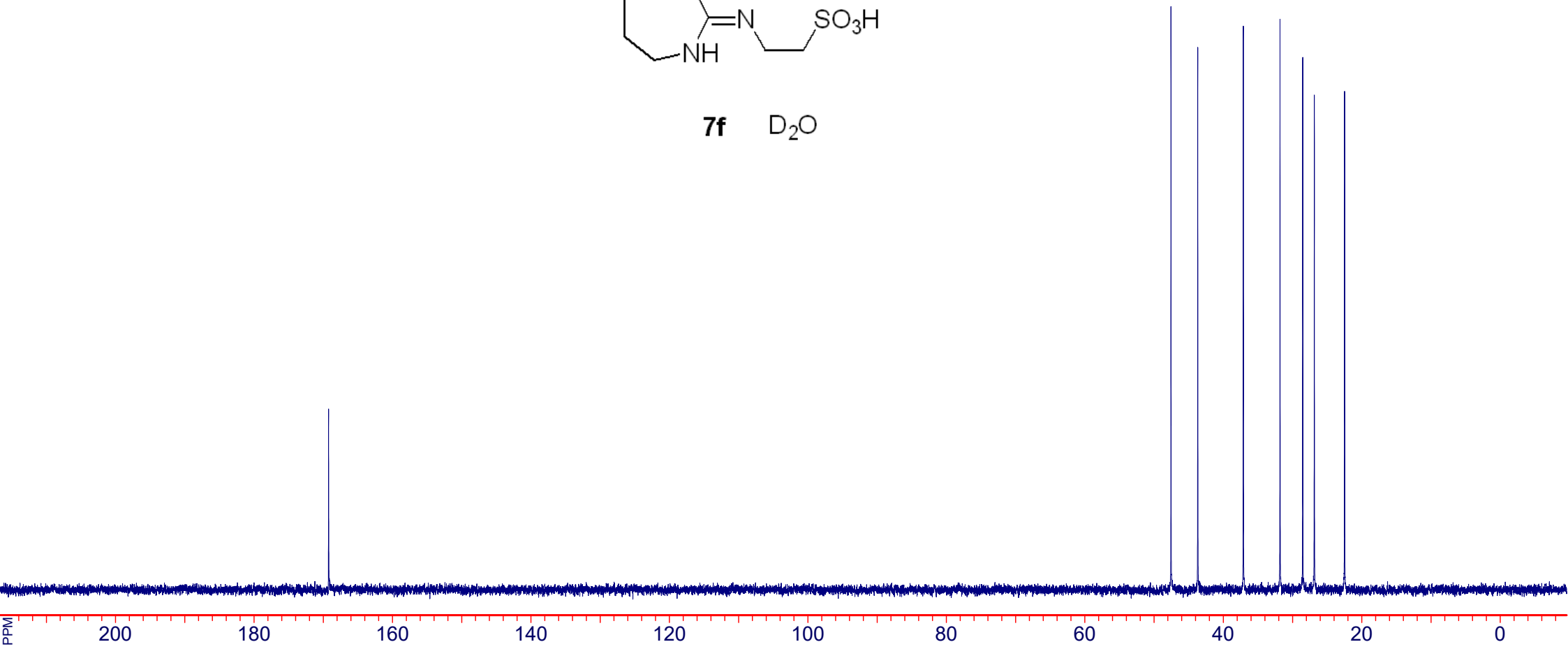


File name: lv-19.fid	Operator:	SF: 399.9733 MHz	NSC: 0	PW: 10.90 usec, RG: 24	SI: 32768
Date: 03-Jun-2022	Solvent: dmso	SW: 8000 Hz	TE: 298 K	AQ: 2.00 sec, RD: 0.00 sec	

lv-19_C13

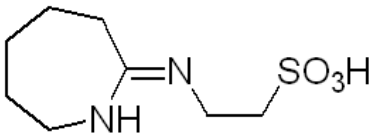


7f D_2O

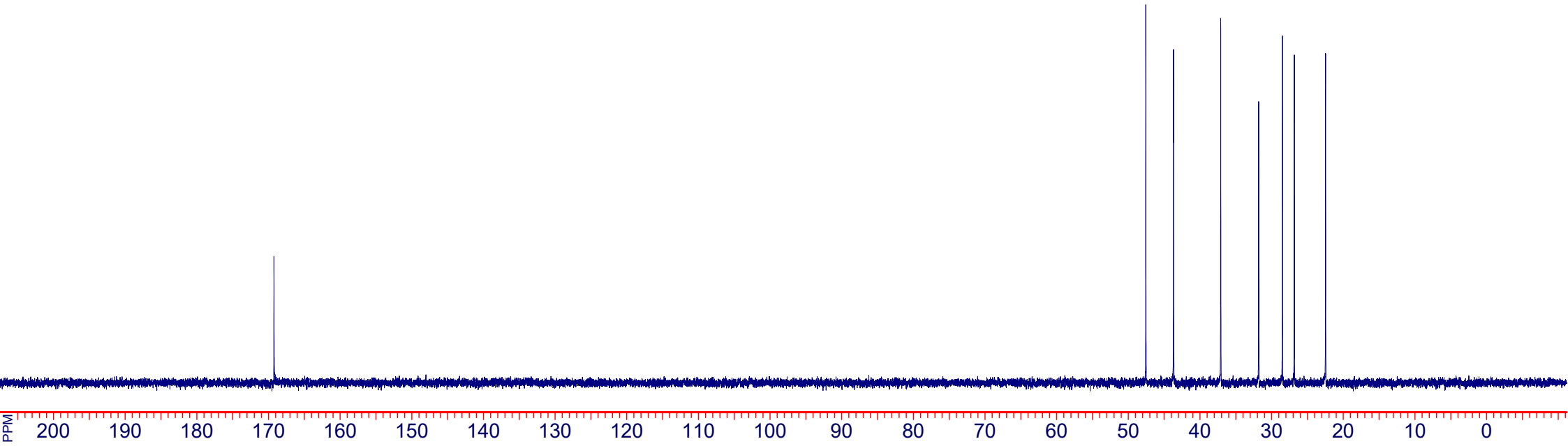


File name: lv-19_C13	Operator: root	SF: 125.6681 MHz	NSC: 316	PW: 0.00 usec, RG: 51200	SI: 131072
Date: 05-Jun-2022	Solvent: D2O	SW: 32680 Hz	TE: 683 K	AQ: 0.78 sec, RD: 0.00 sec	

lv-19_C13APT

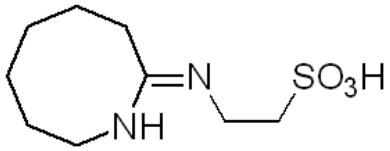


7f D_2O

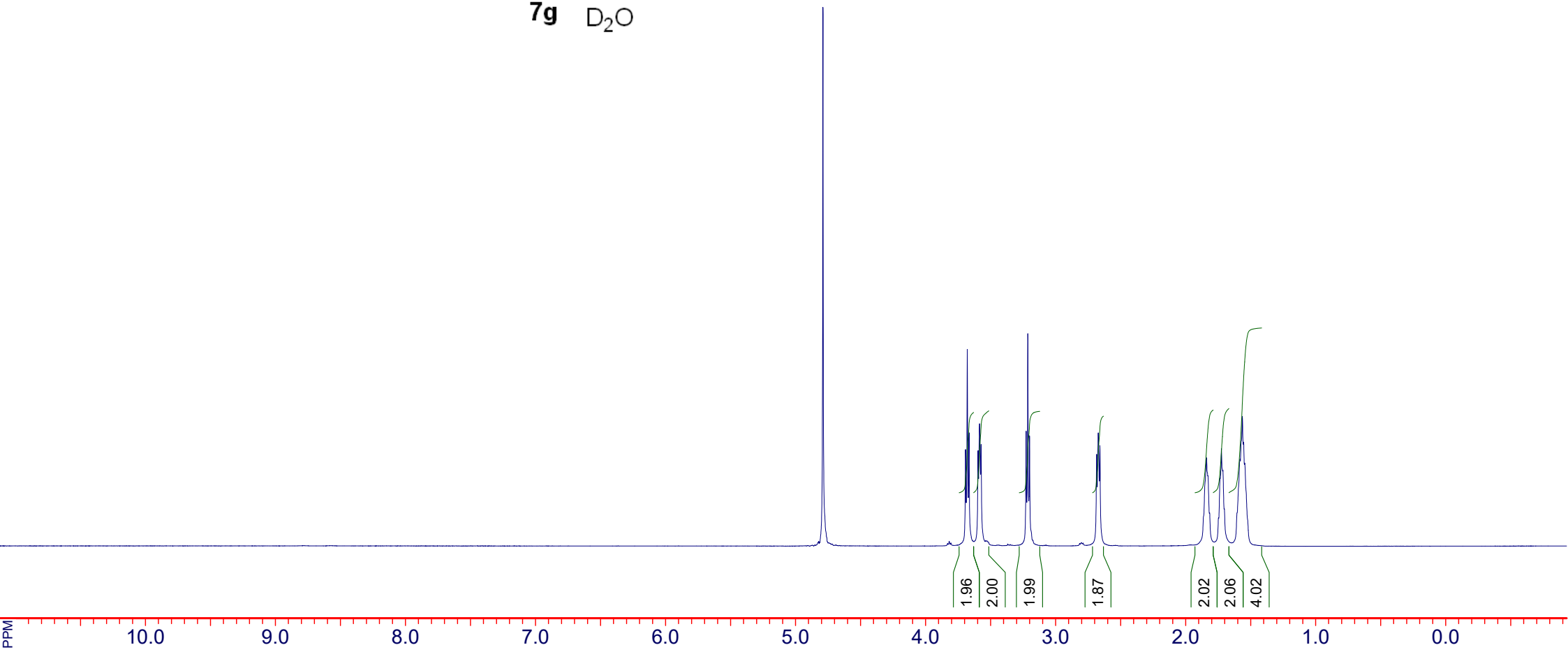


File name: lv-19_C13APT	Operator: root	SF: 125.6681 MHz	NSC: 330	PW: 0.00 usec, RG: 51200	SI: 65536
Date: 05-Jun-2022	Solvent: D2O	SW: 32680 Hz	TE: 683 K	AQ: 1.57 sec, RD: 0.00 sec	

lv-21

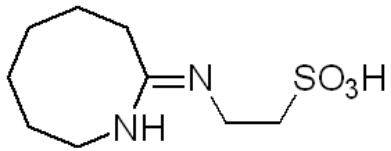


7g D_2O

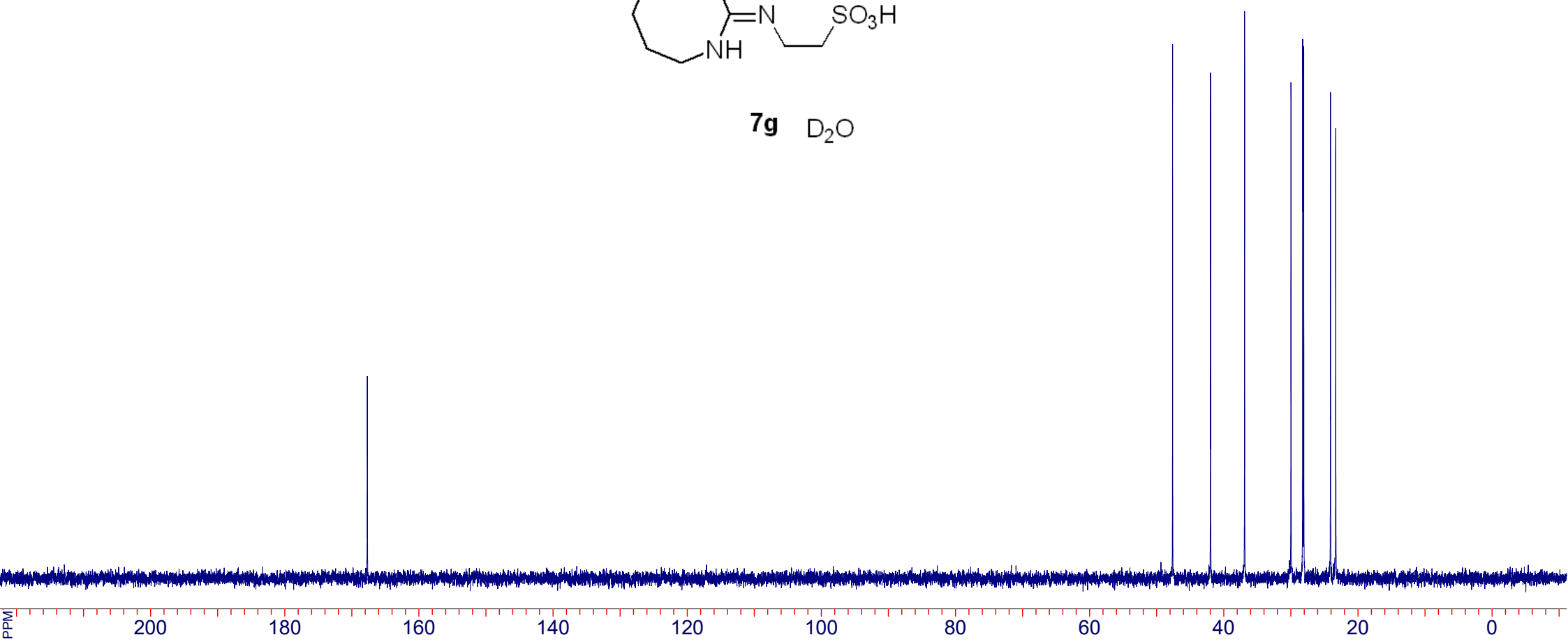


File name: lv-21	Operator: root	SF: 499.7730 MHz	NSC: 1	PW: 0.00 usec, RG: 22	SI: 32768
Date: 01-Jun-2022	Solvent: D2O	SW: 10776 Hz	TE: 683 K	AQ: 1.52 sec, RD: 0.00 sec	

lv-21_C13

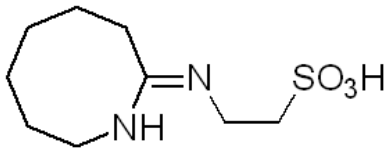


7g D_2O

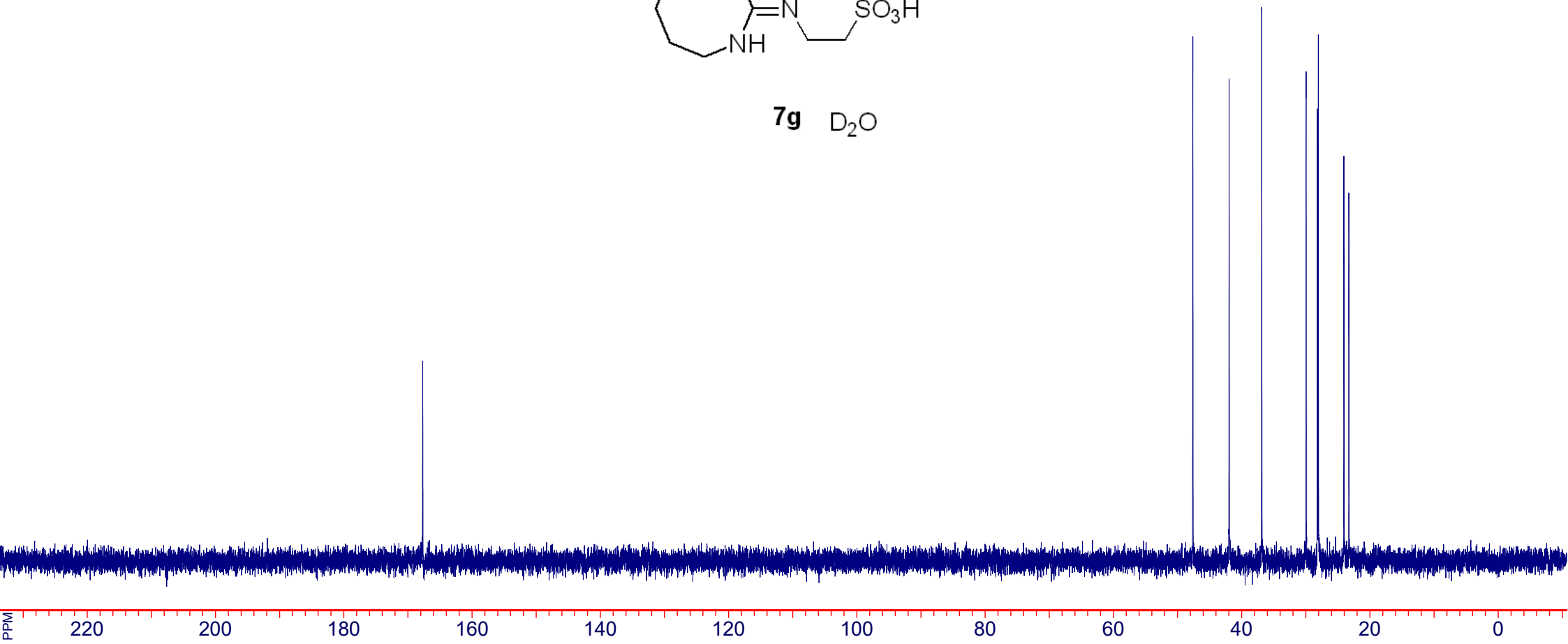


File name: lv-21_C13	Operator: root	SF: 125.6681 MHz	NSC: 76	PW: 0.00 usec, RG: 51200	SI: 131072
Date: 01-Jun-2022	Solvent: D2O	SW: 32680 Hz	TE: 683 K	AQ: 0.78 sec, RD: 0.00 sec	

lv-21_C13APT



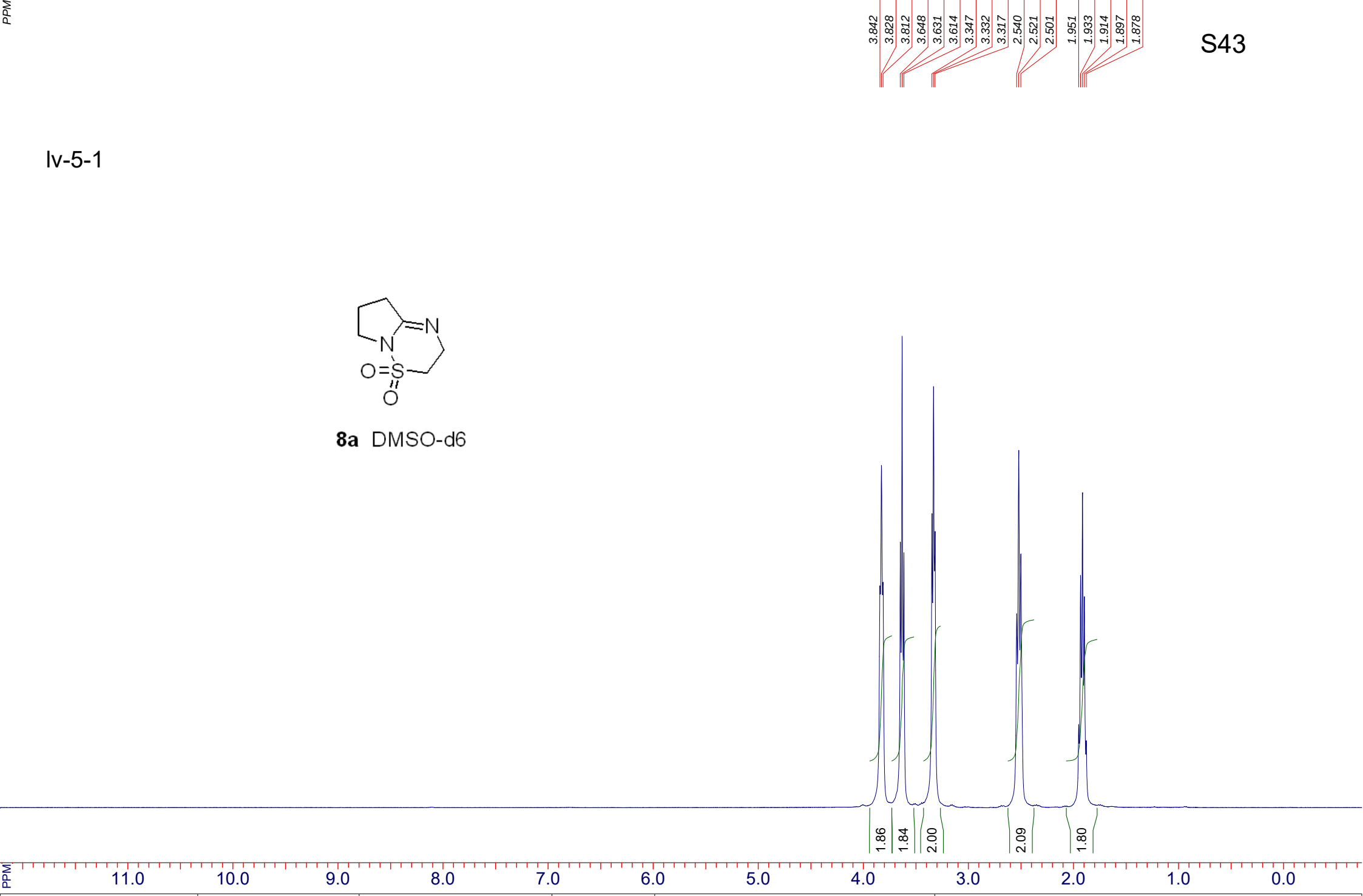
7g D_2O



File name: lv-21_C13APT	Operator: root	SF: 125.6681 MHz	NSC: 60	PW: 0.00 usec, RG: 51200	SI: 65536
Date: 01-Jun-2022	Solvent: D2O	SW: 32680 Hz	TE: 683 K	AQ: 1.57 sec, RD: 0.00 sec	

lv-5-1

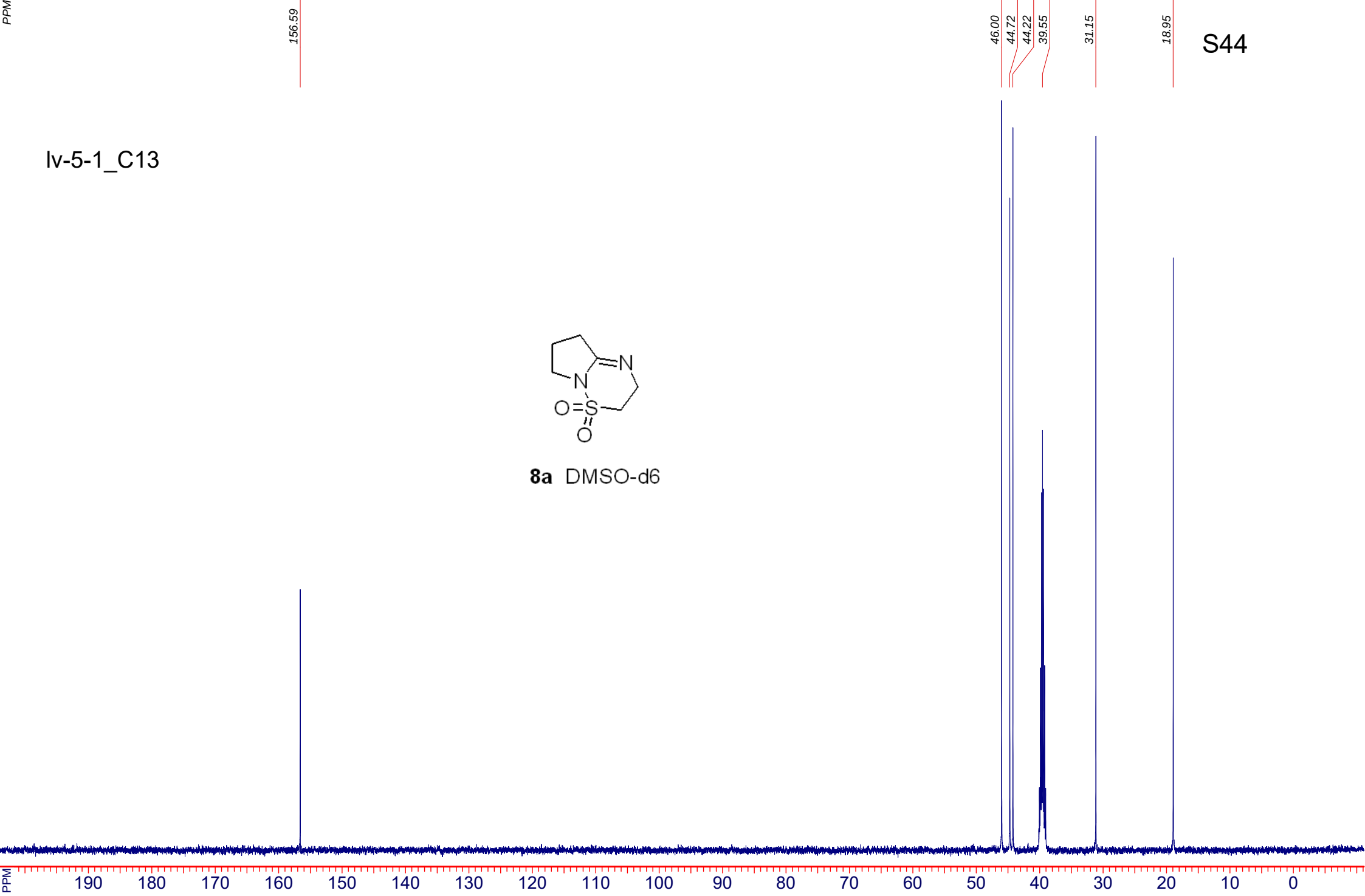
8a DMSO-d6



File name: lv-5-1	Operator: nmr	SF: 400.1300 MHz	NSC: 1	PW: 0.00 usec, RG: 25	SI: 32768
Date: 26-May-2022	Solvent: DMSO	SW: 8224 Hz	TE: 300 K	AQ: 1.99 sec, RD: 0.00 sec	Parameter file, TOPSPINVersion 2.1

lv-5-1_C13

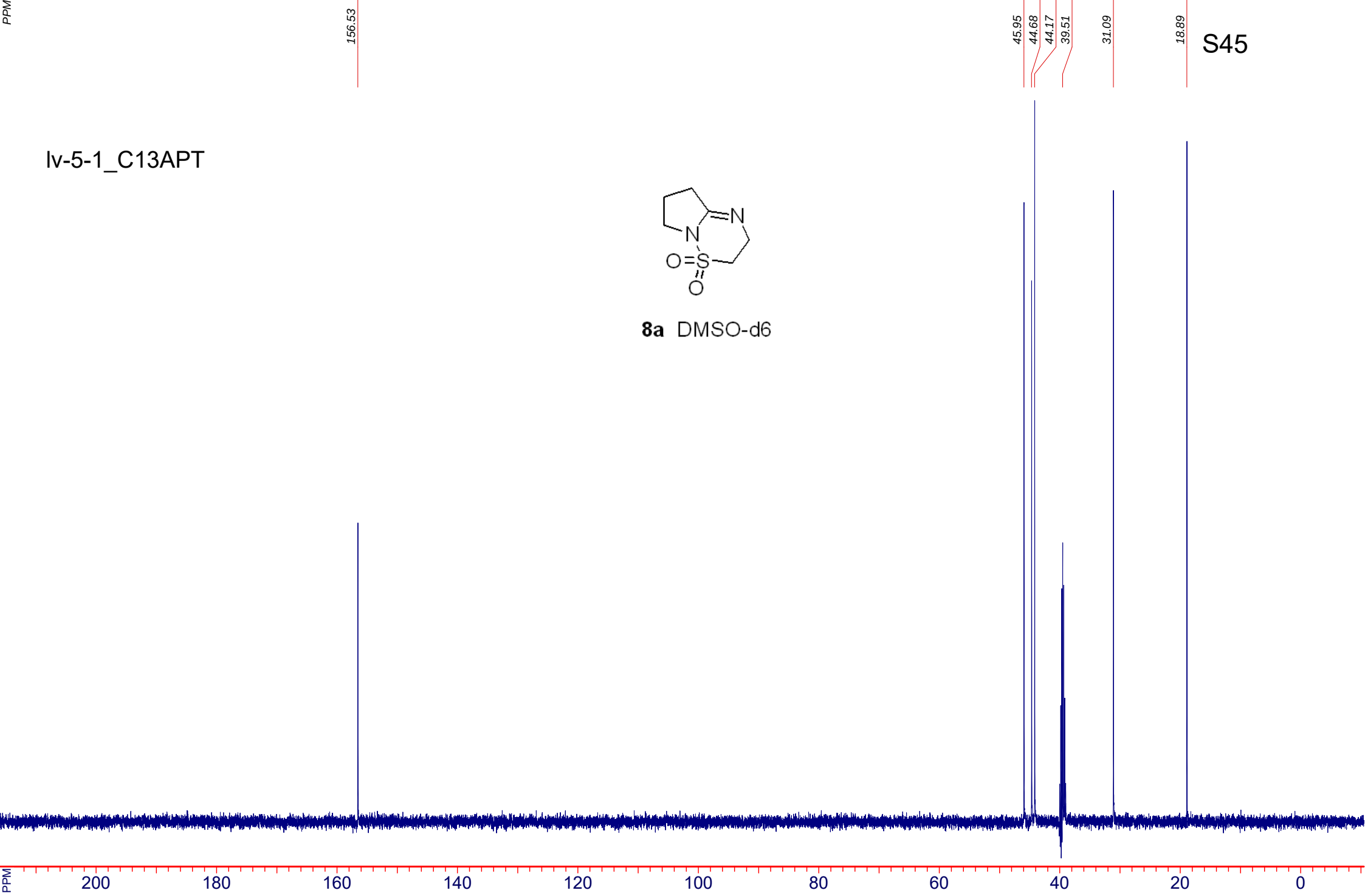
8a DMSO-d6



File name: lv-5-1_C13	Operator: root	SF: 125.6681 MHz	NSC: 250	PW: 0.00 usec, RG: 51200	SI: 131072
Date: 26-May-2022	Solvent: DMSO	SW: 32680 Hz	TE: 683 K	AQ: 0.78 sec, RD: 0.00 sec	

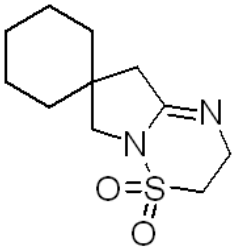
lv-5-1_C13APT

8a DMSO-d6



File name: lv-5-1_C13APT	Operator: root	SF: 125.6681 MHz	NSC: 179	PW: 0.00 usec, RG: 51200	SI: 65536
Date: 26-May-2022	Solvent: DMSO	SW: 32680 Hz	TE: 683 K	AQ: 1.57 sec, RD: 0.00 sec	

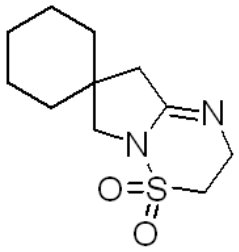
lv623



8b DMSO-d6

File name: lv623	Operator: nmr	SF: 400.1300 MHz	NSC: 1	PW: 0.00 usec, RG: 25	SI: 32768
Date: 18-Nov-2022	Solvent: DMSO	SW: 8224 Hz	TE: 300 K	AQ: 1.99 sec, RD: 0.00 sec	

lv-623_C13

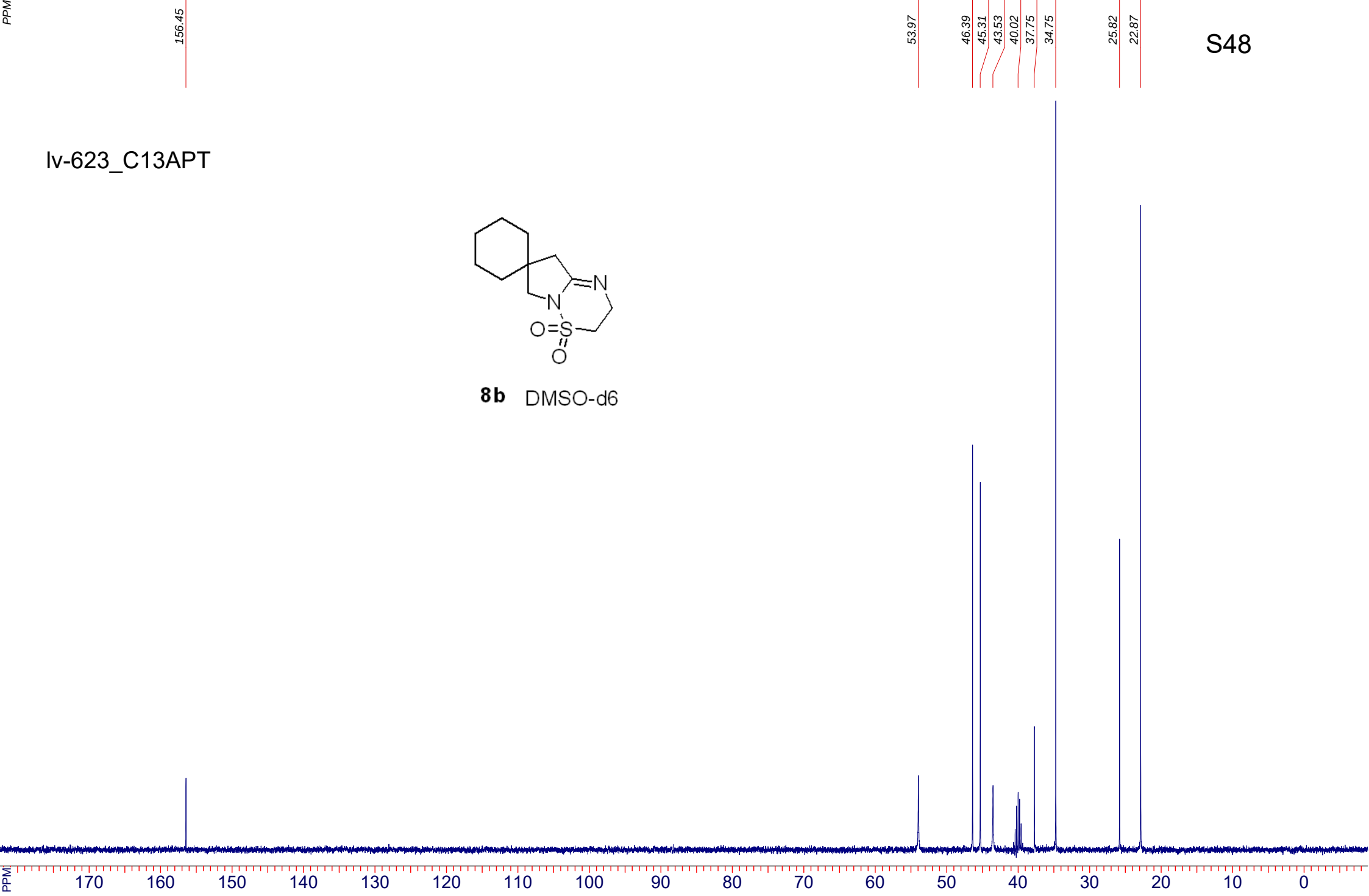


8b DMSO-d6

File name: lv-623_C13	Operator: nmr	SF: 100.6128 MHz	NSC: 89	PW: 0.00 usec, RG: 2050	SI: 32768
Date: 09-Nov-2022	Solvent: DMSO	SW: 26042 Hz	TE: 300 K	AQ: 0.98 sec, RD: 0.00 sec	

lv-623_C13APT

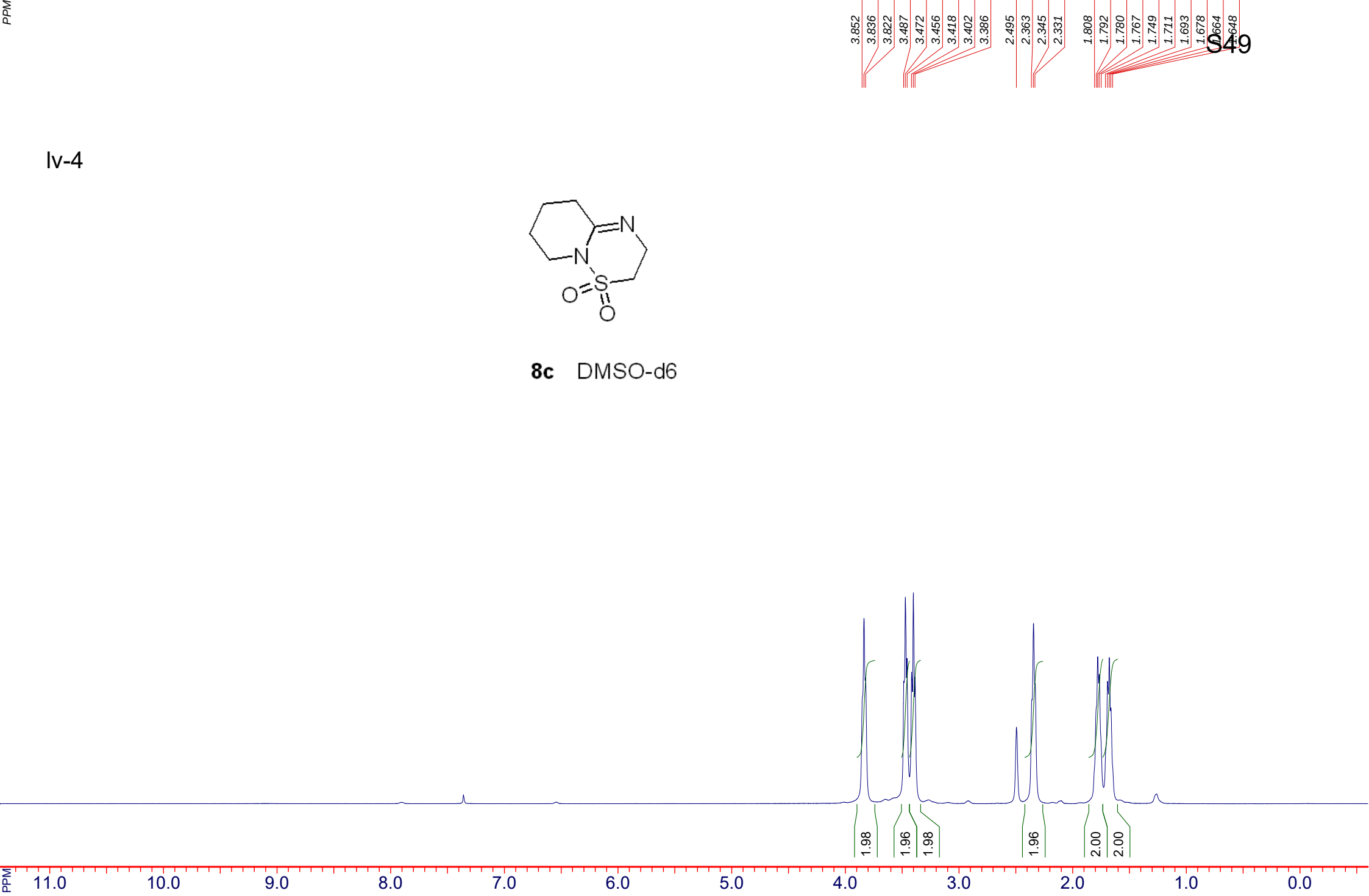
8b DMSO-d6



File name: lv-623_C13APT	Operator: nmr	SF: 100.6128 MHz	NSC: 162	PW: 0.00 usec, RG: 2050	SI: 65536
Date: 09-Nov-2022	Solvent: DMSO	SW: 24038 Hz	TE: 300 K	AQ: 1.06 sec, RD: 0.00 sec	

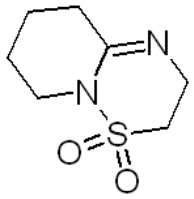
lv-4

8c DMSO-d6

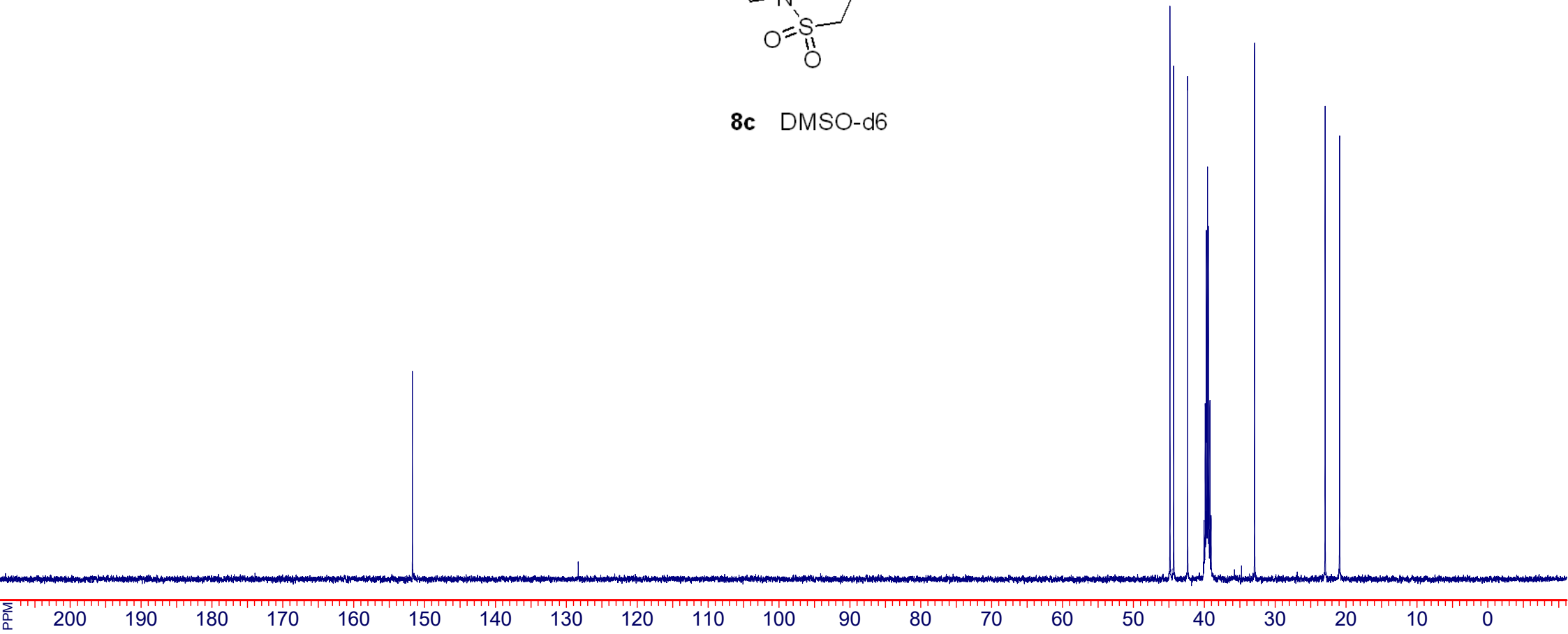


File name: lv-4	Operator: nmr	SF: 400.1300 MHz	NSC: 1	PW: 0.00 usec, RG: 25	SI: 32768
Date: 26-May-2022	Solvent: DMSO	SW: 8224 Hz	TE: 300 K	AQ: 1.99 sec, RD: 0.00 sec	

lv-4_C13



8c DMSO-d6



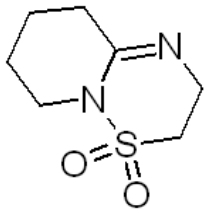
File name: lv-4_C13	Operator: root	SF: 125.6681 MHz	NSC: 250	PW: 0.00 usec, RG: 51200	SI: 131072
Date: 26-May-2022	Solvent: DMSO	SW: 32680 Hz	TE: 683 K	AQ: 0.78 sec, RD: 0.00 sec	

PPM

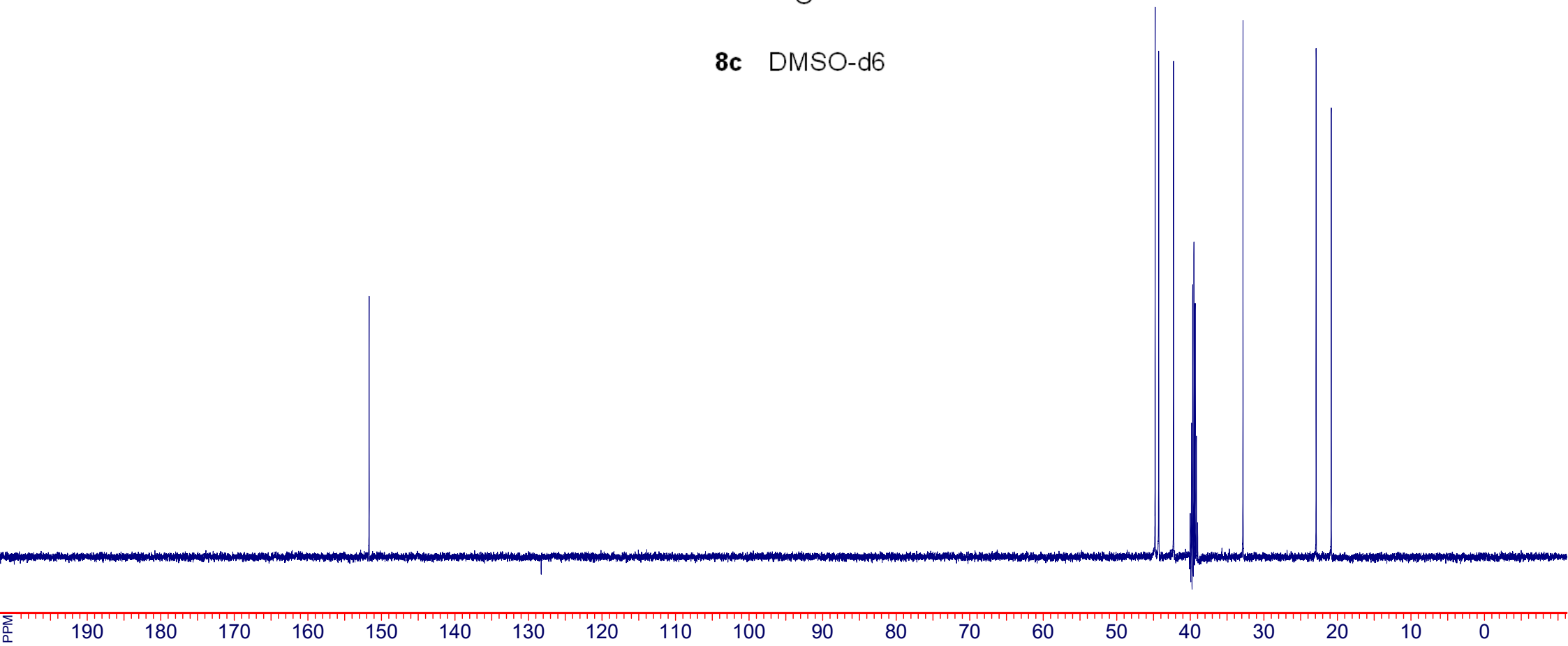
151.67

44.79 44.31 42.28 39.52 32.85 22.90 20.85

lv-4_C13APT



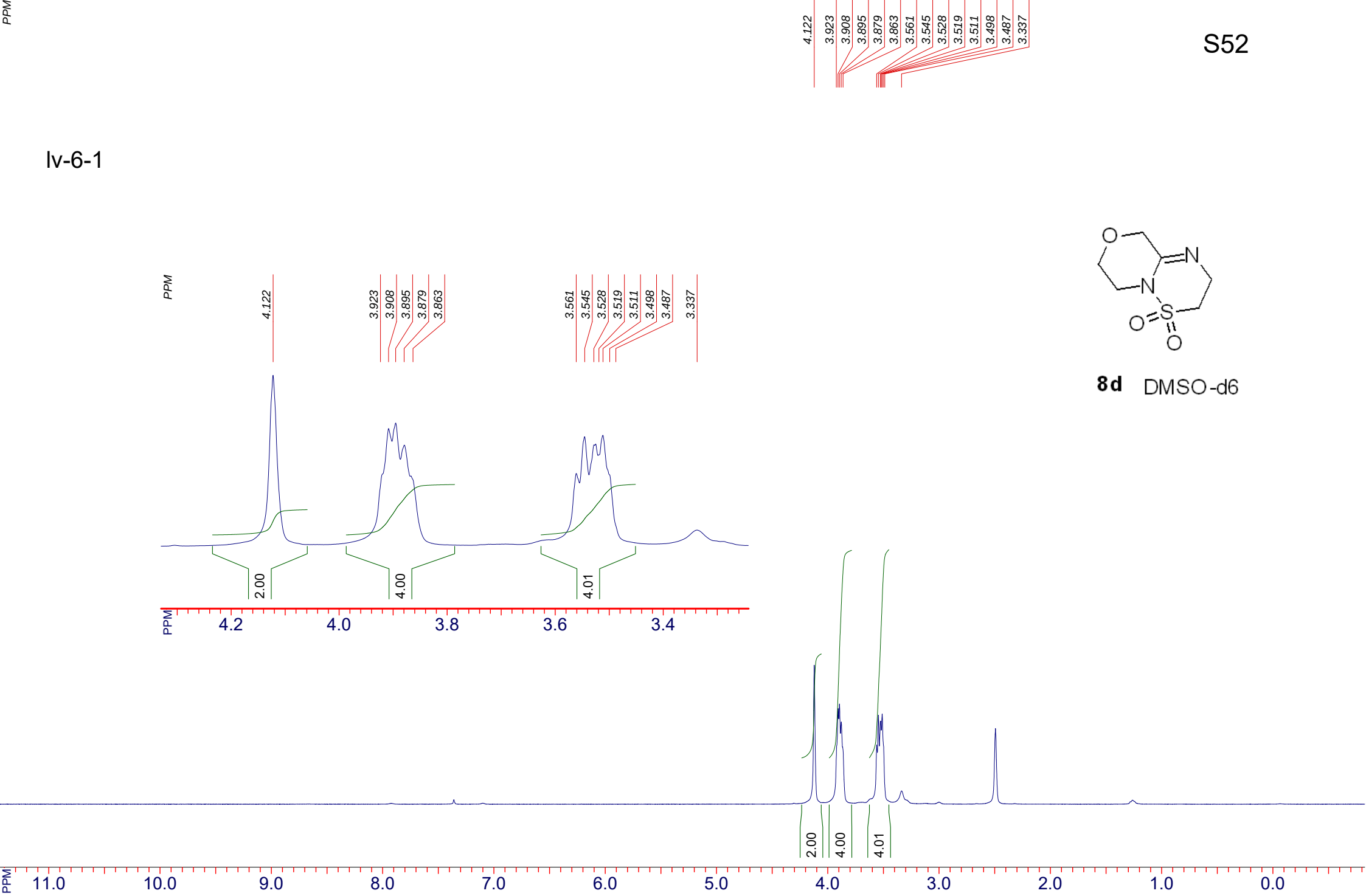
8c DMSO-d6



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

File name: lv-4_C13APT	Operator: root	SF: 125.6681 MHz	NSC: 250	PW: 0.00 usec, RG: 51200	SI: 65536
Date: 26-May-2022	Solvent: DMSO	SW: 32680 Hz	TE: 683 K	AQ: 1.57 sec, RD: 0.00 sec	

lv-6-1



8d DMSO-d6

File name: lv-6-1	Operator: nmr	SF: 400.1300 MHz	NSC: 1	PW: 0.00 usec, RG: 25	SI: 32768
Date: 27-May-2022	Solvent: DMSO	SW: 8224 Hz	TE: 300 K	AQ: 1.99 sec, RD: 0.00 sec	

PPM

147.83

68.42

64.87

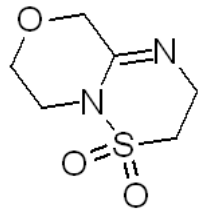
45.43

44.96

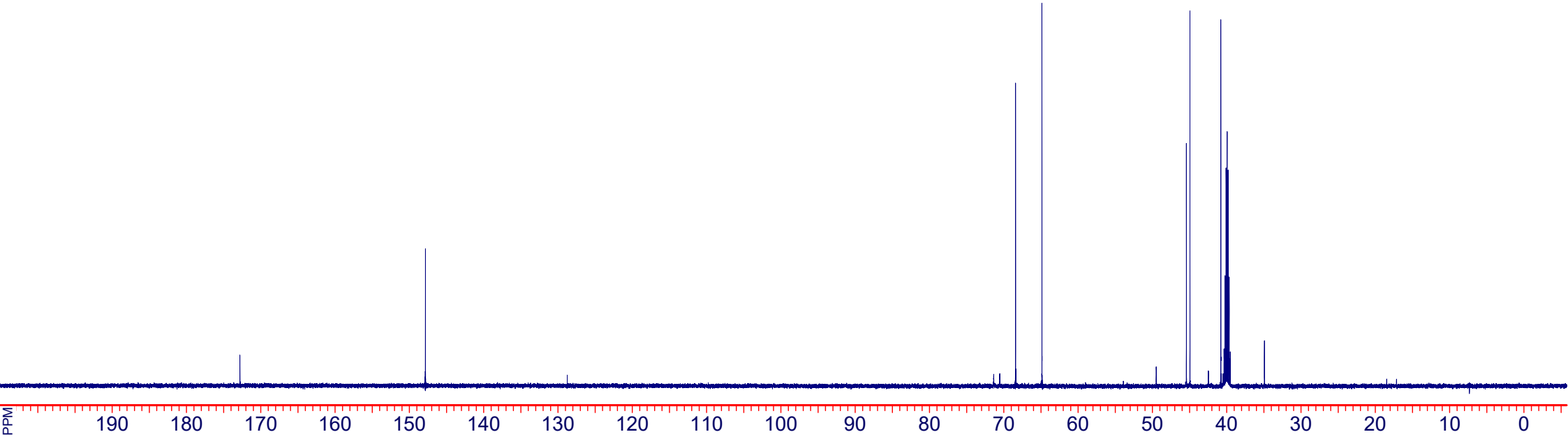
40.80

39.95

lv-6_C13.fid



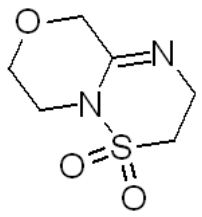
8d DMSO-d6



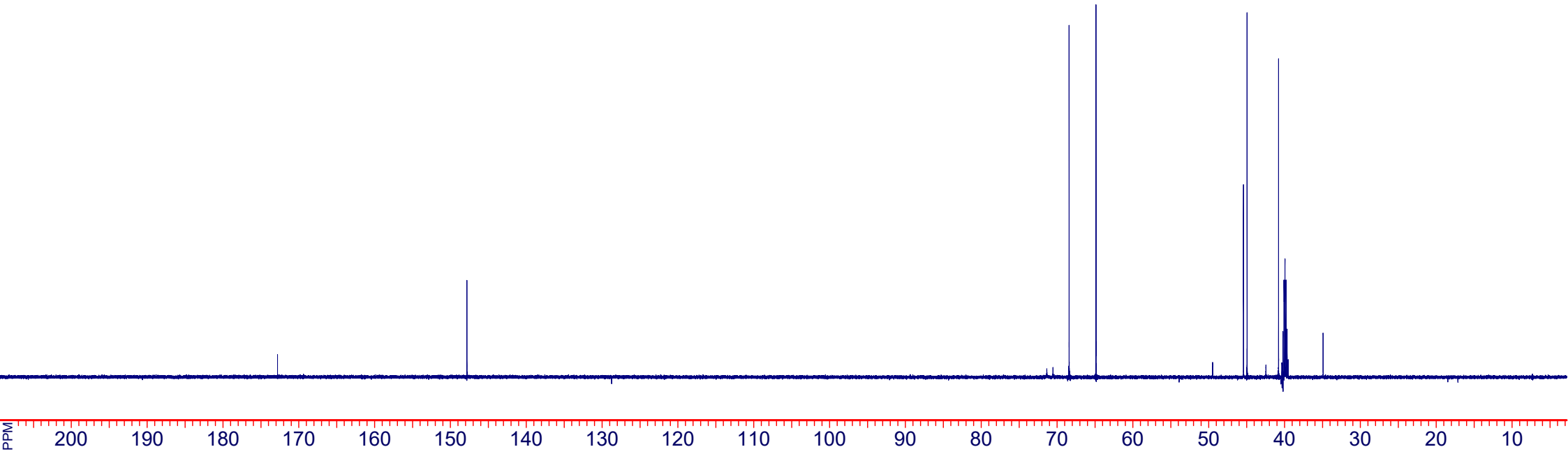
File name: lv-6_C13.fid	Operator:	SF: 150.8304 MHz	NSC: 0	PW: 3.09 usec, RG: 60	SI: 65536
Date: 31-May-2022	Solvent: dmso	SW: 37879 Hz	TE: 298 K	AQ: 0.87 sec, RD: 0.00 sec	

PPM 147.83 68.42 64.87 45.43 44.95 40.80 39.95

lv-6_C13APT.fid



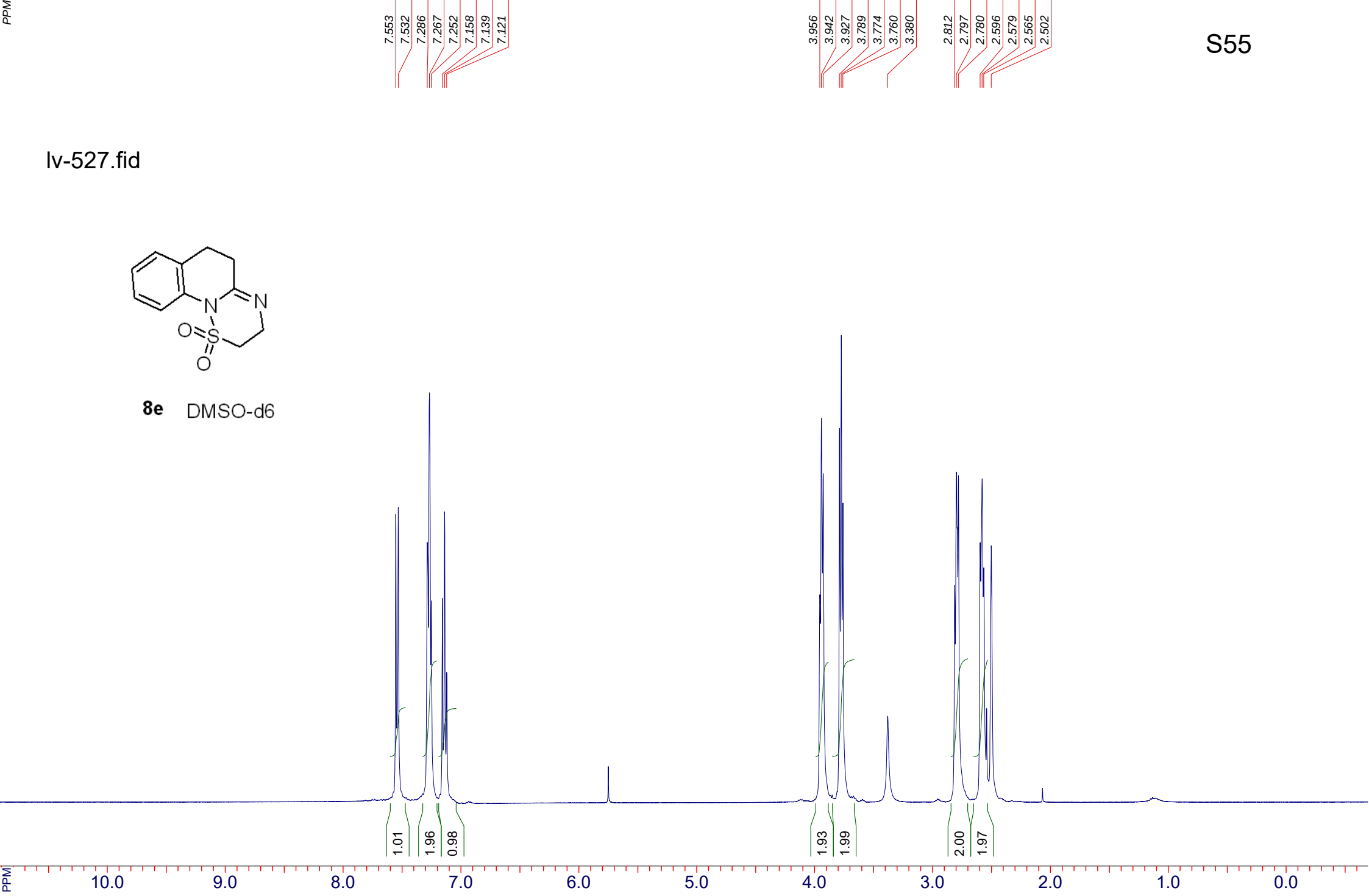
8d DMSO-d6



File name: lv-6_C13APT.fid	Operator:	SF: 150.8333 MHz	NSC: 0	PW: 4.63 usec, RG: 60	SI: 131072
Date: 31-May-2022	Solvent: dmso	SW: 39063 Hz	TE: 298 K	AQ: 0.87 sec, RD: 0.00 sec	

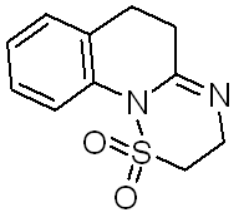
lv-527.fid

8e DMSO-d6



File name: lv-527.fid	Operator:	SF: 399.9733 MHz	NSC: 0	PW: 10.90 usec, RG: 24	SI: 32768
Date: 03-Oct-2022	Solvent: dmso	SW: 8000 Hz	TE: 298 K	AQ: 2.00 sec, RD: 0.00 sec	

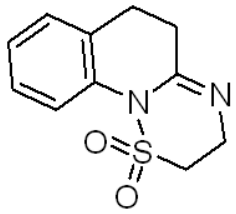
lv-527_C13



8e DMSO-d6

File name: lv-527_C13	Operator: root	SF: 125.6681 MHz	NSC: 96	PW: 0.00 usec, RG: 51200	SI: 131072
Date: 03-Oct-2022	Solvent: DMSO	SW: 32680 Hz	TE: 683 K	AQ: 0.78 sec, RD: 0.00 sec	

lv-527_C13APT



8e DMSO-d6

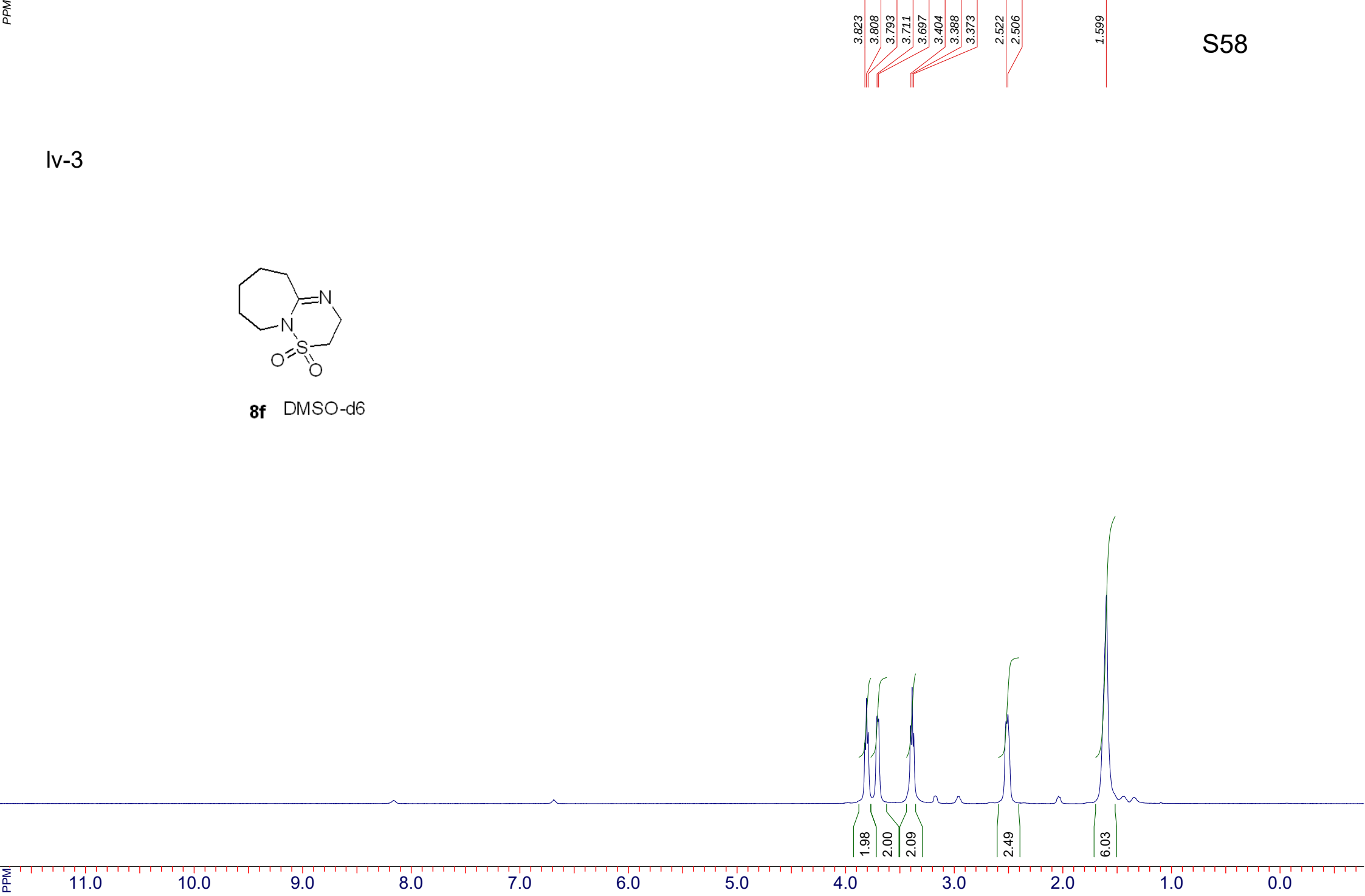
PPM 151.01 132.70 130.22 128.13 126.55 124.82 120.26 47.18 43.18 39.51 33.15 24.96

PPM 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0

File name: lv-527_C13APT	Operator: root	SF: 125.6681 MHz	NSC: 77	PW: 0.00 usec, RG: 51200	SI: 65536
Date: 03-Oct-2022	Solvent: DMSO	SW: 32680 Hz	TE: 683 K	AQ: 1.57 sec, RD: 0.00 sec	

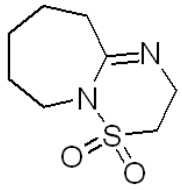
lv-3

8f DMSO-d6

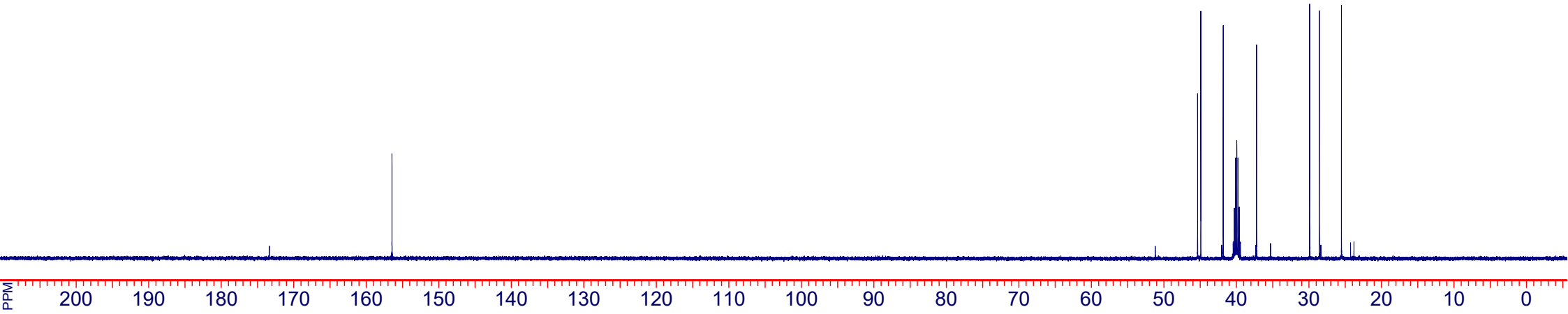


File name: lv-3	Operator: nmr	SF: 400.1300 MHz	NSC: 1	PW: 0.00 usec, RG: 25	SI: 32768
Date: 26-May-2022	Solvent: DMSO	SW: 8224 Hz	TE: 300 K	AQ: 1.99 sec, RD: 0.00 sec	

lv-3_C13.fid

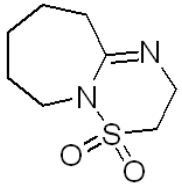


8f DMSO-d6



File name: lv-3_C13.fid	Operator:	SF: 125.6926 MHz	NSC: 0	PW: 3.27 usec, RG: 60	SI: 65536
Date: 26-May-2022	Solvent: dmso	SW: 31250 Hz	TE: 298 K	AQ: 1.05 sec, RD: 0.00 sec	

lv-3_C13APT.fid

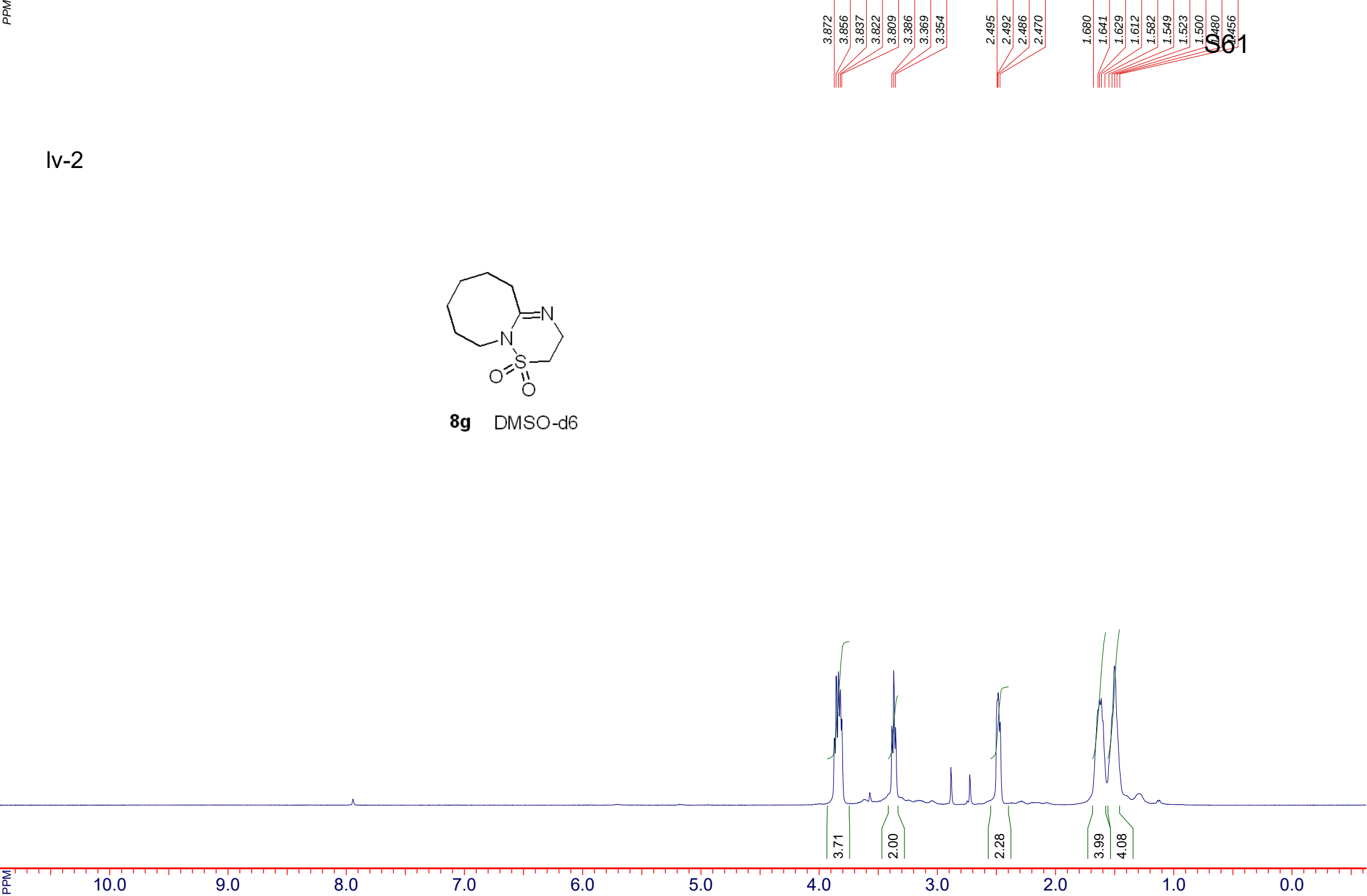


8f DMSO-d6

File name: lv-3_C13APT.fid	Operator:	SF: 125.6925 MHz	NSC: 0	PW: 5.00 usec, RG: 60	SI: 131072
Date: 26-May-2022	Solvent: dmso	SW: 32895 Hz	TE: 298 K	AQ: 1.95 sec, RD: 0.00 sec	Automated Probe tuning parameter

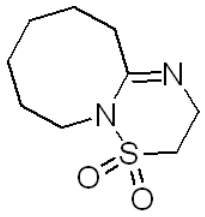
lv-2

8g DMSO-d6



File name: lv-2	Operator: nmr	SF: 400.1300 MHz	NSC: 1	PW: 0.00 usec, RG: 25	SI: 32768
Date: 27-May-2022	Solvent: DMSO	SW: 8224 Hz	TE: 300 K	AQ: 1.99 sec, RD: 0.00 sec	

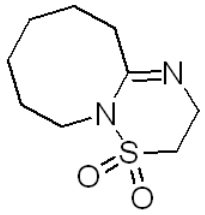
lv-2_C13



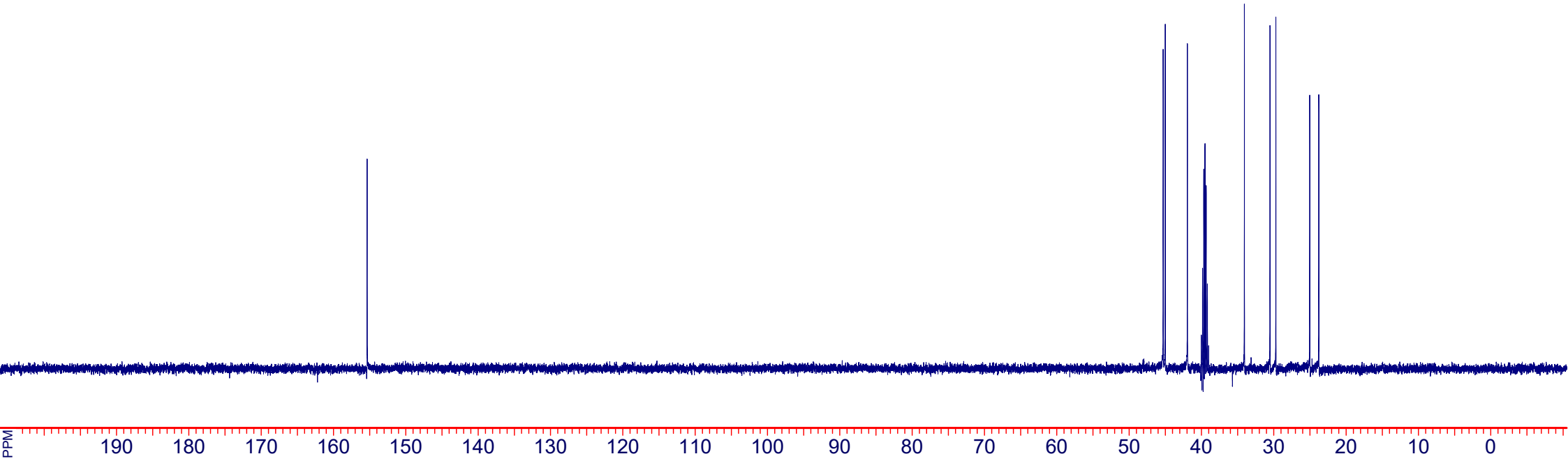
8g DMSO-d6

File name: lv-2_C13	Operator: root	SF: 125.6681 MHz	NSC: 356	PW: 0.00 usec, RG: 51200	SI: 131072
Date: 28-May-2022	Solvent: DMSO	SW: 32680 Hz	TE: 683 K	AQ: 0.78 sec, RD: 0.00 sec	

lv-2_C13APT



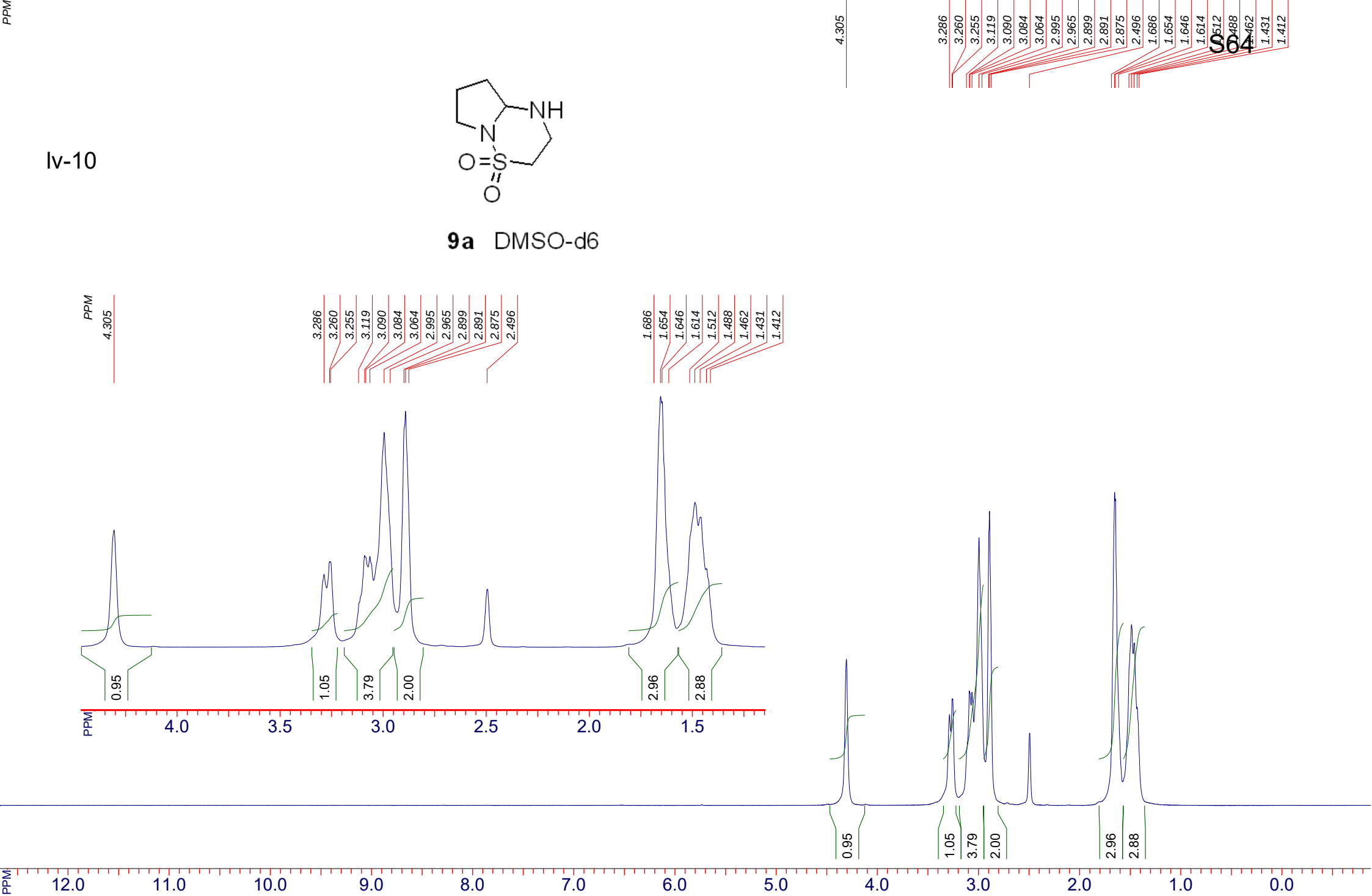
8g DMSO-d6



File name: lv-2_C13APT	Operator: root	SF: 125.6681 MHz	NSC: 200	PW: 0.00 usec, RG: 51200	SI: 65536
Date: 28-May-2022	Solvent: DMSO	SW: 32680 Hz	TE: 683 K	AQ: 1.00 sec, RD: 0.00 sec	

lv-10

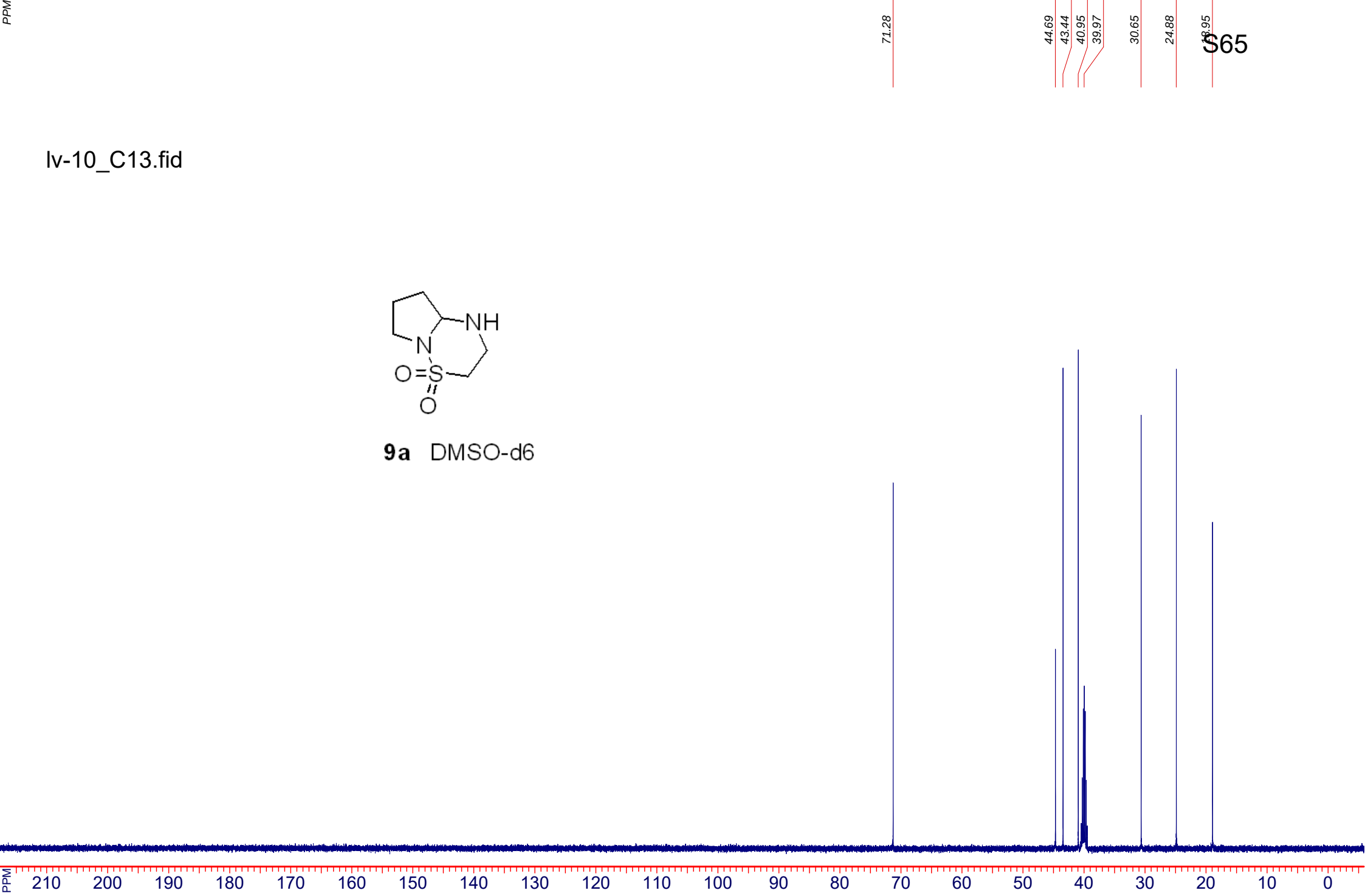
9a DMSO-d6



File name: lv-10	Operator: nmr	SF: 400.1300 MHz	NSC: 1	PW: 0.00 usec, RG: 25	SI: 32768
Date: 26-May-2022	Solvent: DMSO	SW: 8224 Hz	TE: 300 K	AQ: 1.99 sec, RD: 0.00 sec	

lv-10_C13.fid

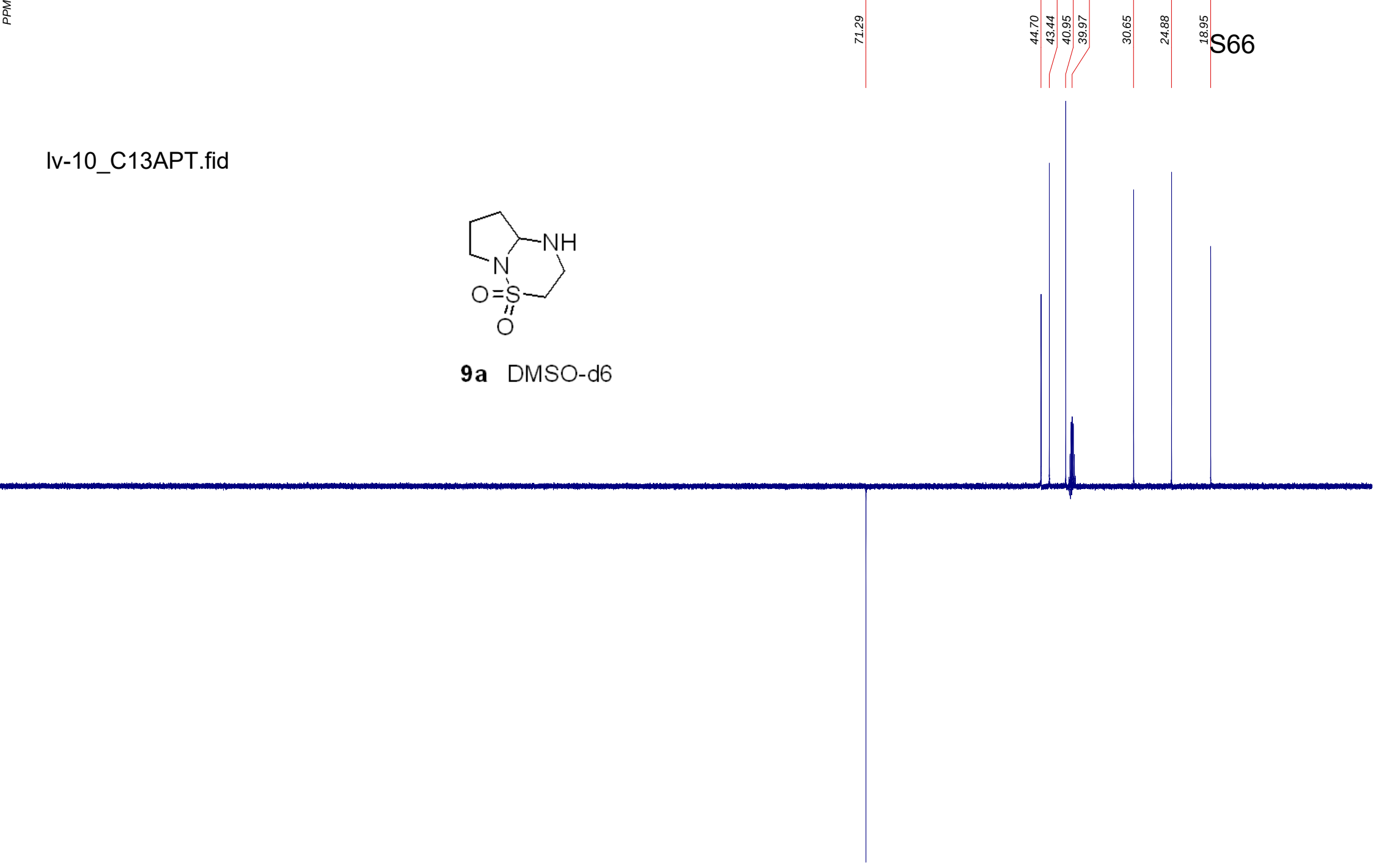
9a DMSO-d6



File name: lv-10_C13.fid	Operator:	SF: 125.6926 MHz	NSC: 0	PW: 3.27 usec, RG: 60	SI: 65536
Date: 26-May-2022	Solvent: dmso	SW: 31250 Hz	TE: 298 K	AQ: 1.05 sec, RD: 0.00 sec	

lv-10_C13APT.fid

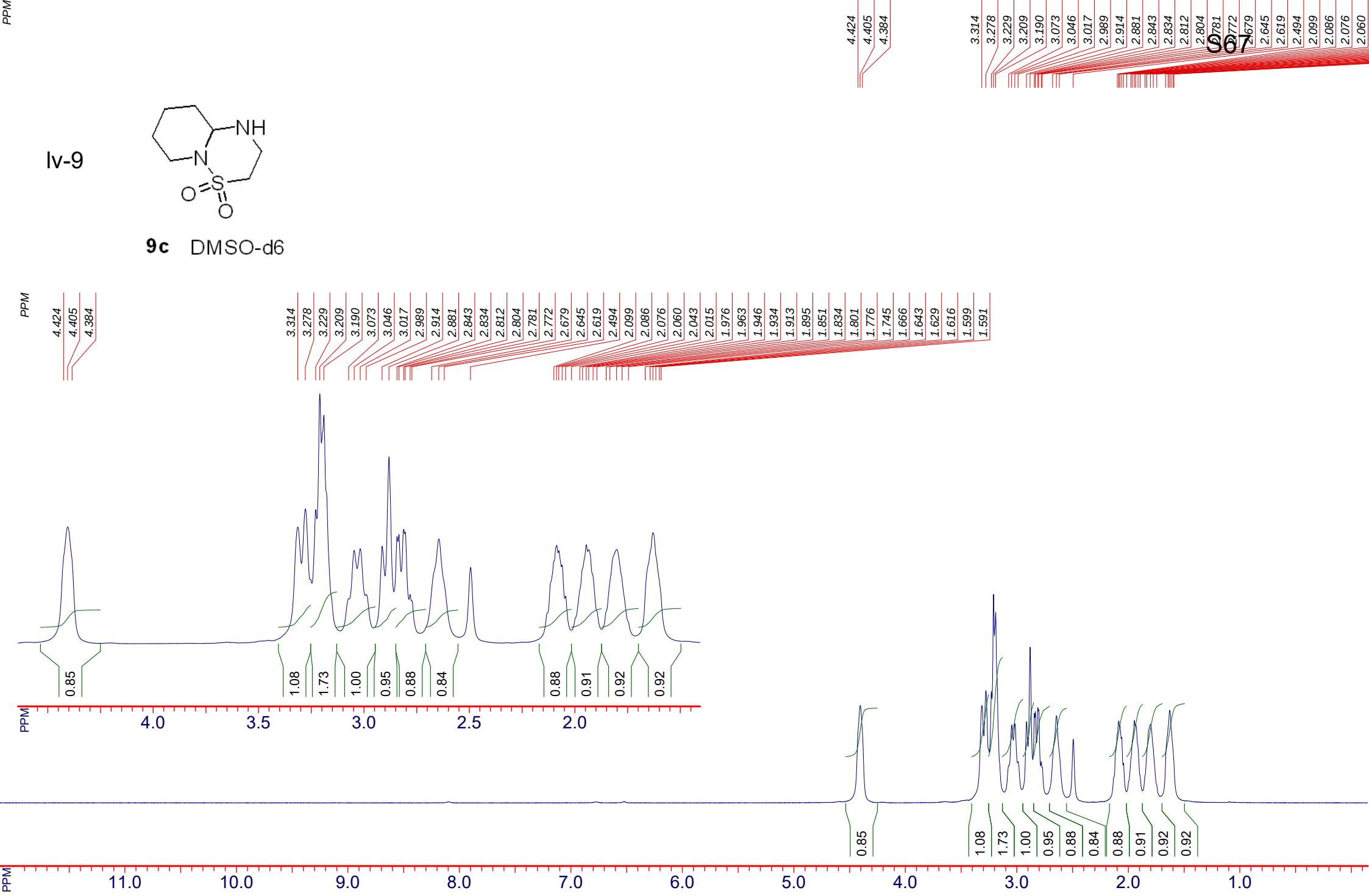
9a DMSO-d6



File name: lv-10_C13APT.fid	Operator:	SF: 125.6925 MHz	NSC: 0	PW: 5.00 usec, RG: 60	SI: 131072
Date: 26-May-2022	Solvent: dmso	SW: 32895 Hz	TE: 298 K	AQ: 1.95 sec, RD: 0.00 sec	Automated Probe tuning parameter

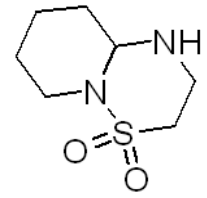
lv-9

9c DMSO-d6

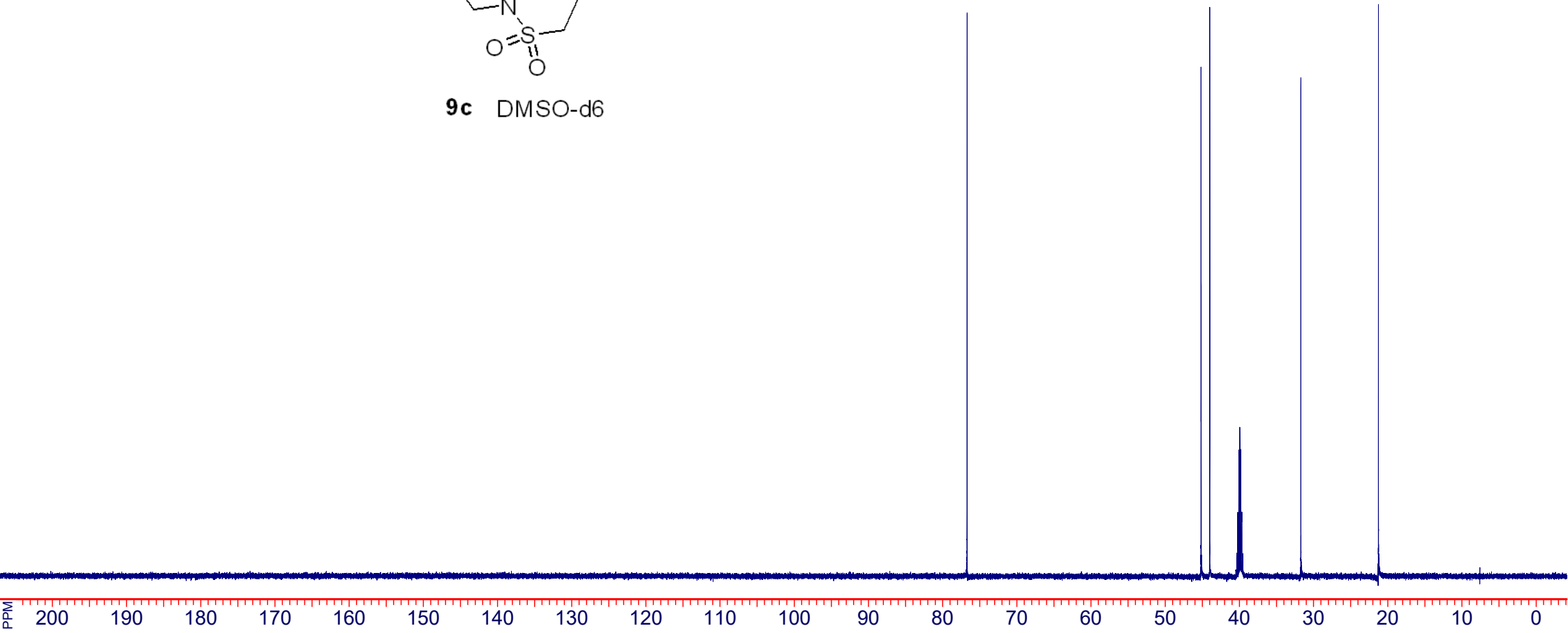


File name: lv-9	Operator: nmr	SF: 400.1300 MHz	NSC: 1	PW: 0.00 usec, RG: 25	SI: 32768
Date: 26-May-2022	Solvent: DMSO	SW: 8224 Hz	TE: 300 K	AQ: 1.99 sec, RD: 0.00 sec	

lv-9_C13.fid



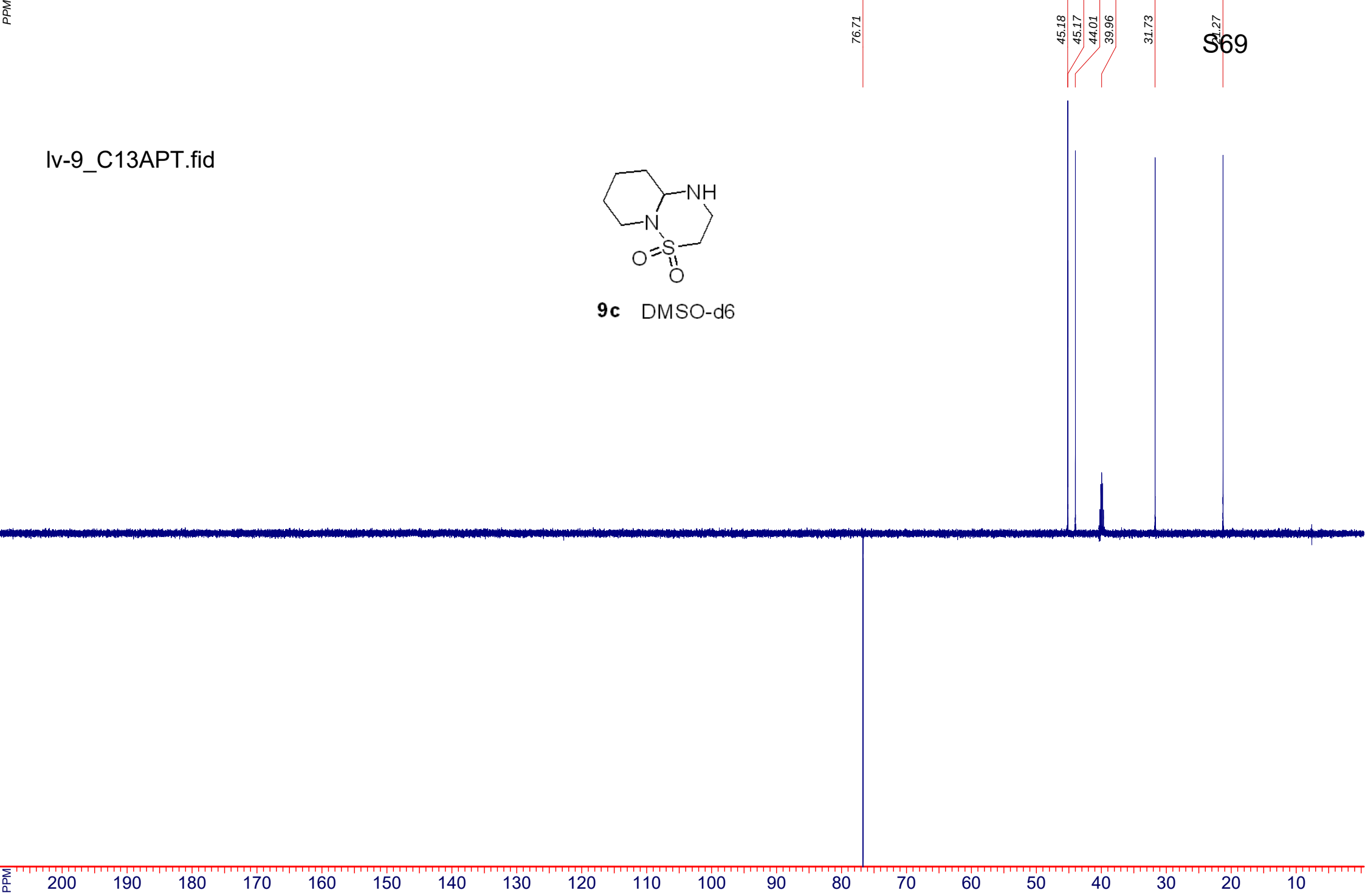
9c DMSO-d6



File name: lv-9_C13.fid	Operator:	SF: 150.8304 MHz	NSC: 0	PW: 3.09 usec, RG: 60	SI: 65536
Date: 26-May-2022	Solvent: dmso	SW: 37879 Hz	TE: 298 K	AQ: 0.87 sec, RD: 0.00 sec	

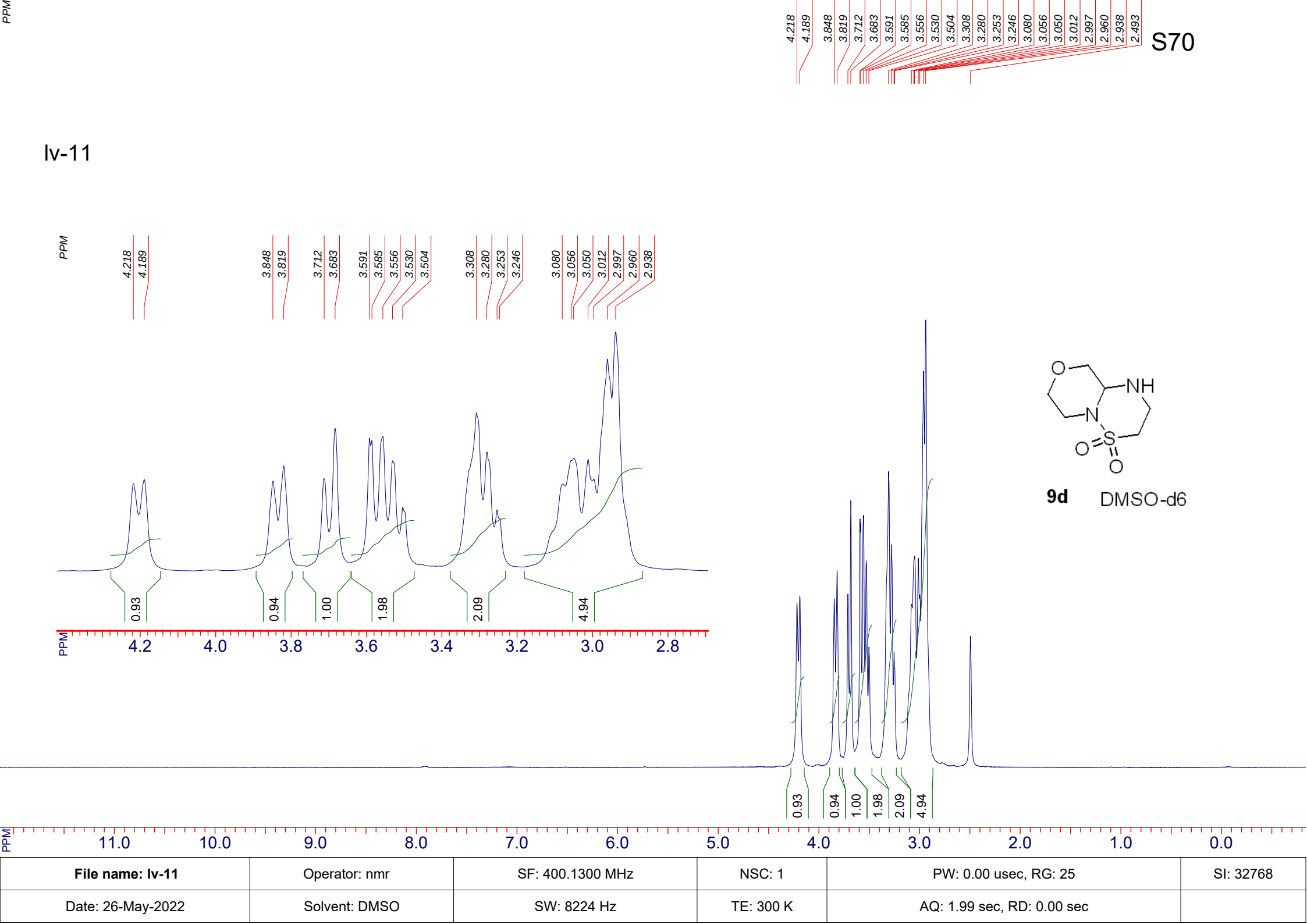
lv-9_C13APT.fid

9c DMSO-d6



File name: lv-9_C13APT.fid	Operator:	SF: 150.8333 MHz	NSC: 0	PW: 4.63 usec, RG: 60	SI: 131072
Date: 26-May-2022	Solvent: dmso	SW: 39063 Hz	TE: 298 K	AQ: 0.87 sec, RD: 0.00 sec	

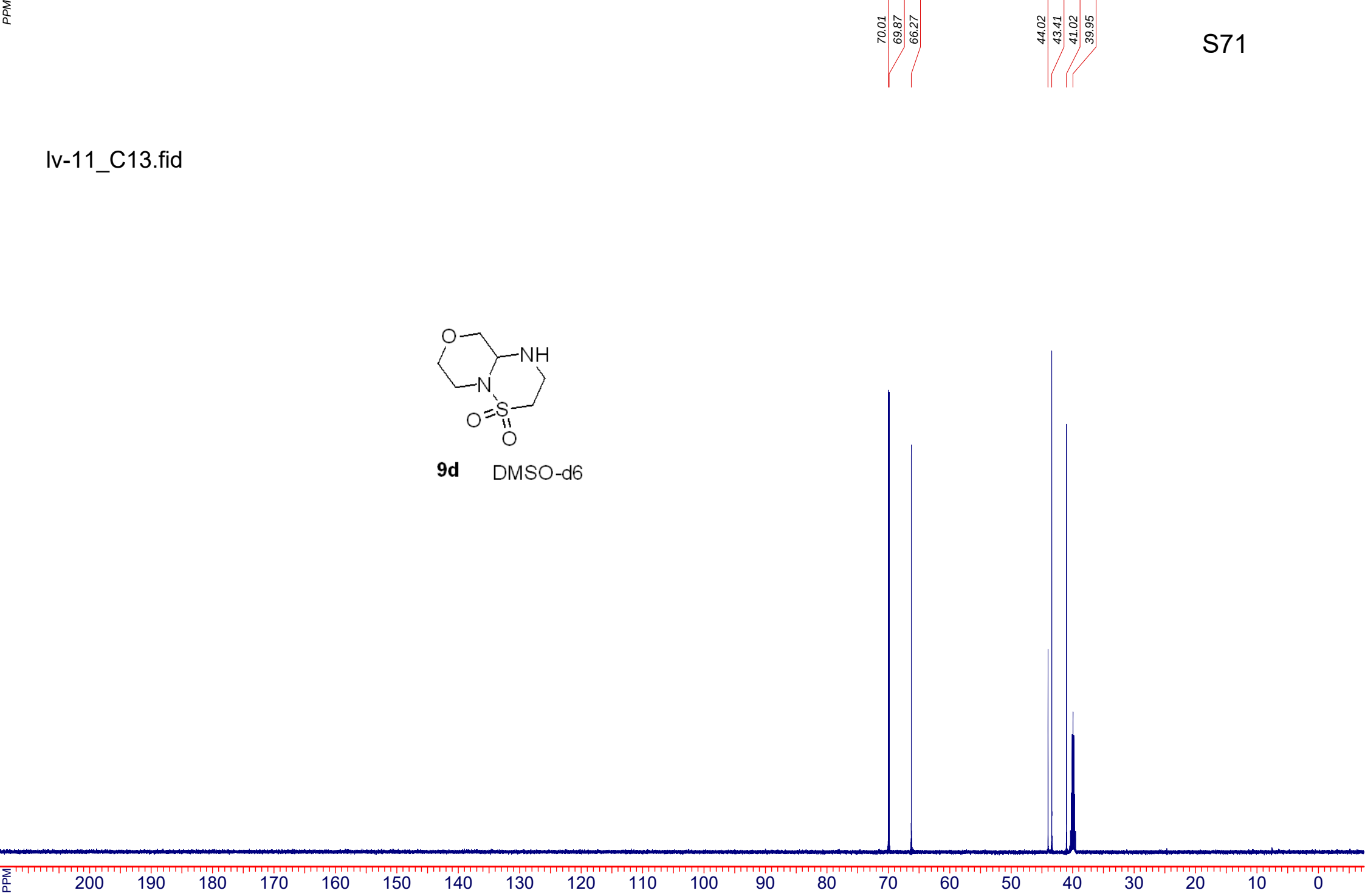
lv-11



File name: lv-11	Operator: nmr	SF: 400.1300 MHz	NSC: 1	PW: 0.00 usec, RG: 25	SI: 32768
Date: 26-May-2022	Solvent: DMSO	SW: 8224 Hz	TE: 300 K	AQ: 1.99 sec, RD: 0.00 sec	

lv-11_C13.fid

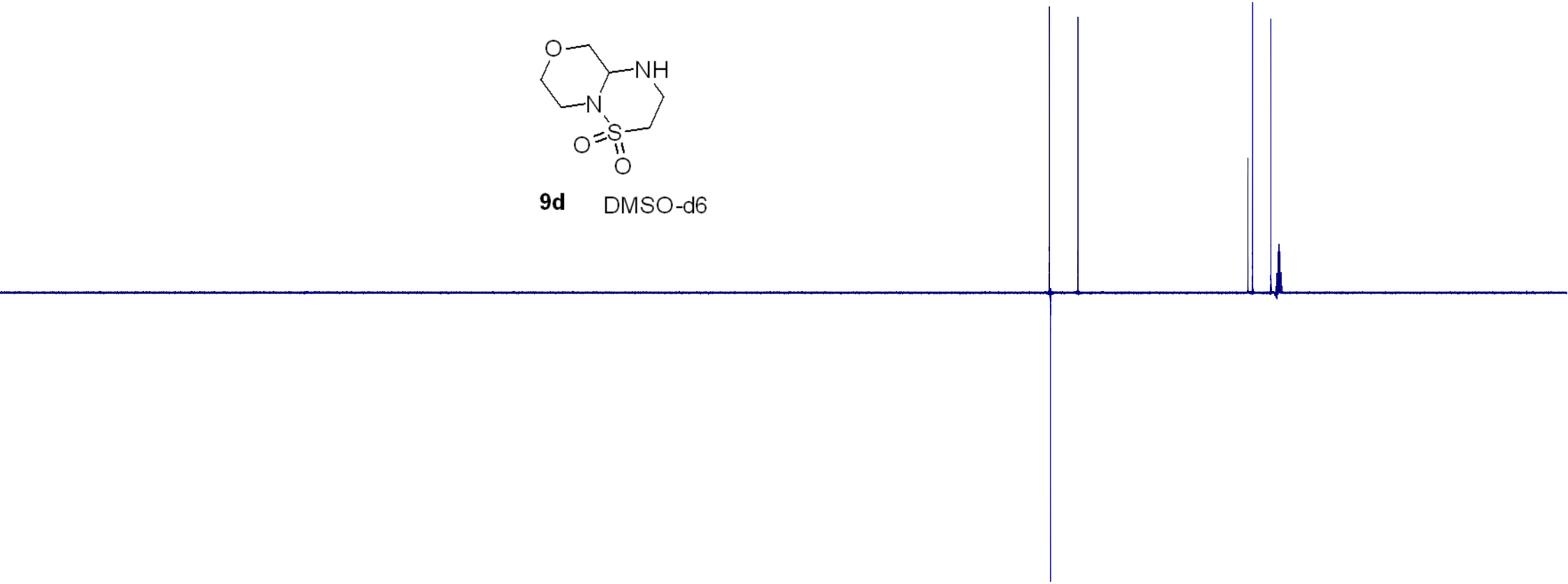
9d DMSO-d6



File name: lv-11_C13.fid	Operator:	SF: 150.8304 MHz	NSC: 0	PW: 3.09 usec, RG: 60	SI: 65536
Date: 26-May-2022	Solvent: dmso	SW: 37879 Hz	TE: 298 K	AQ: 0.87 sec, RD: 0.00 sec	

lv-11_C13APT.fid

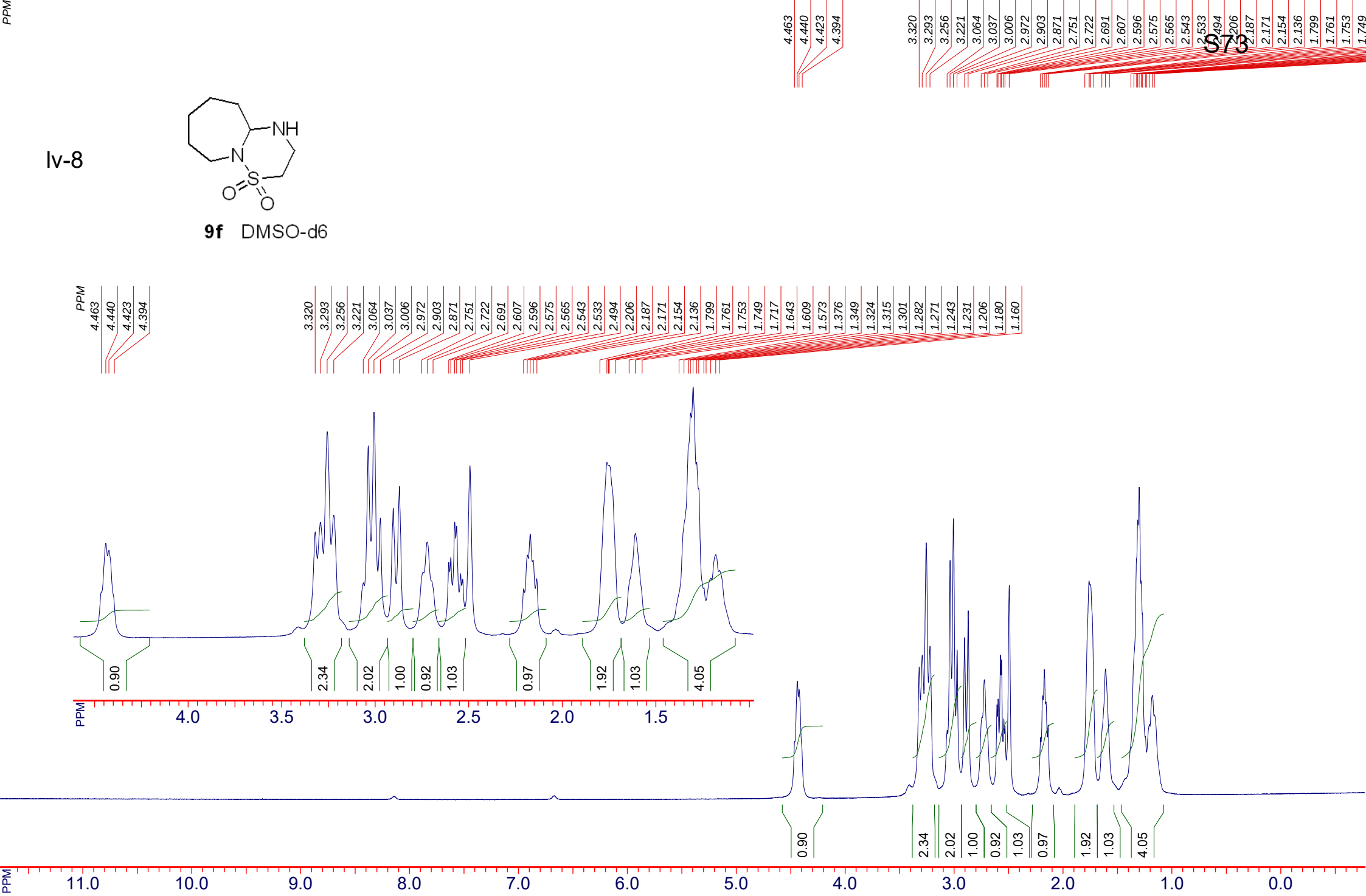
9d DMSO-d6



File name: lv-11_C13APT.fid	Operator:	SF: 150.8333 MHz	NSC: 0	PW: 4.63 usec, RG: 60	SI: 131072
Date: 26-May-2022	Solvent: dmso	SW: 39063 Hz	TE: 298 K	AQ: 0.87 sec, RD: 0.00 sec	

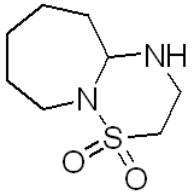
lv-8

9f DMSO-d6



File name: lv-8	Operator: nmr	SF: 400.1300 MHz	NSC: 1	PW: 0.00 usec, RG: 25	SI: 32768
Date: 26-May-2022	Solvent: DMSO	SW: 8224 Hz	TE: 300 K	AQ: 1.99 sec, RD: 0.00 sec	Parameter file, TOPSPINVersion 2.1

lv8_C13



9f DMSO-d6

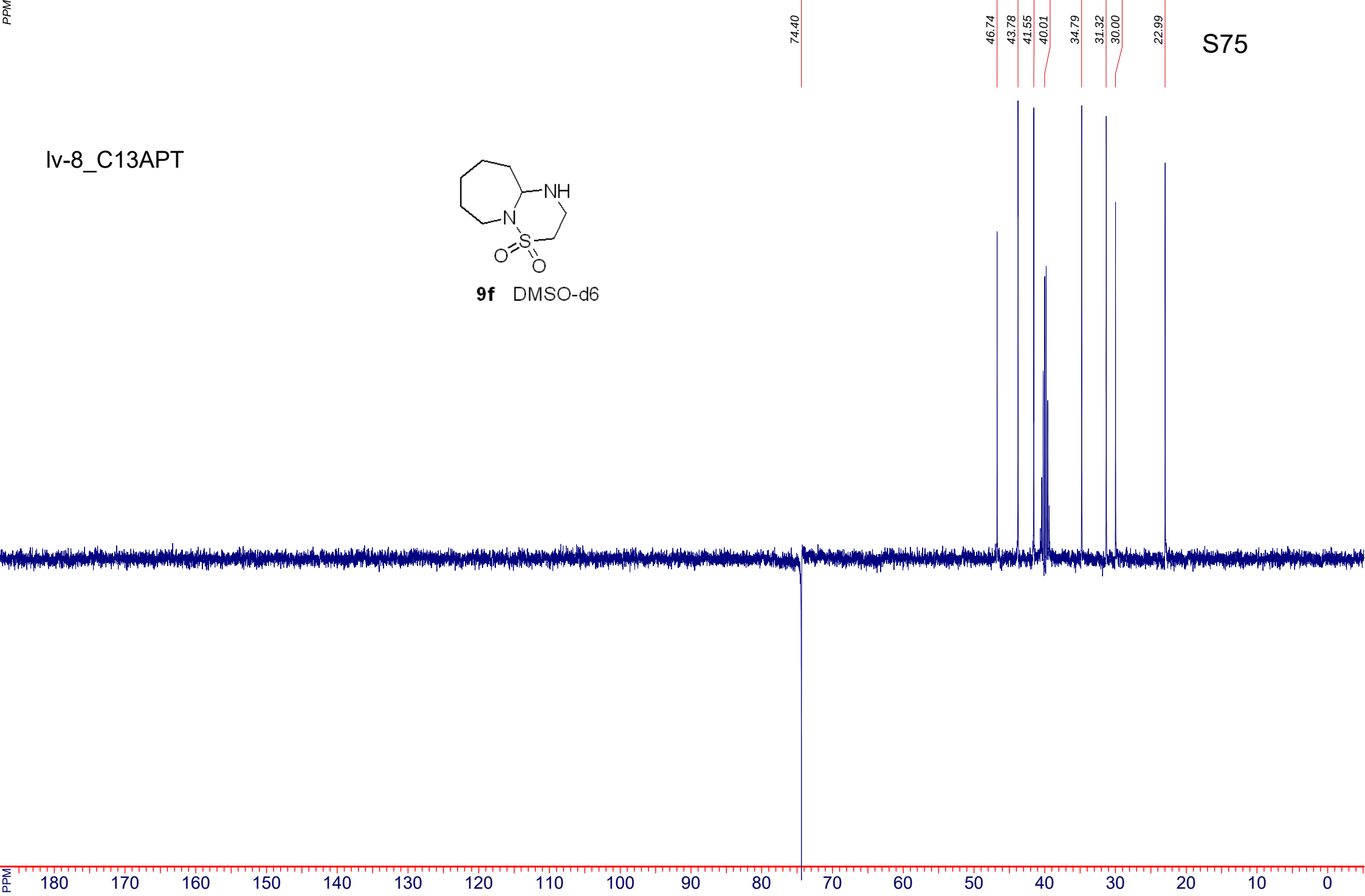
74.40 46.74 43.79 41.55 40.01 34.79 31.32 30.01 22.99

PPM 130 120 110 100 90 80 70 60 50 40 30 20 10 0

File name: lv8_C13	Operator: nmr	SF: 100.6128 MHz	NSC: 367	PW: 0.00 usec, RG: 2050	SI: 32768
Date: 30-May-2022	Solvent: DMSO	SW: 26042 Hz	TE: 300 K	AQ: 0.98 sec, RD: 0.00 sec	

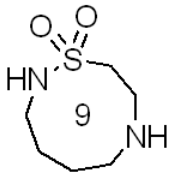
lv-8_C13APT

9f DMSO-d6

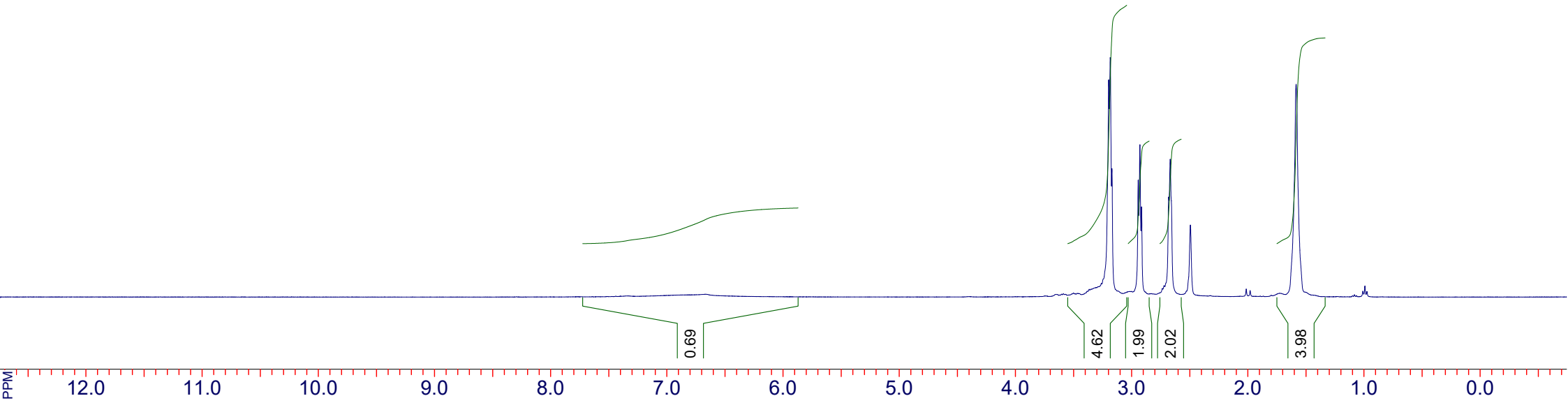


File name: lv-8_C13APT	Operator: nmr	SF: 100.6128 MHz	NSC: 10240	PW: 0.00 usec, RG: 2050	SI: 65536
Date: 31-May-2022	Solvent: DMSO	SW: 24038 Hz	TE: 300 K	AQ: 1.36 sec, RD: 0.00 sec	

LV-16



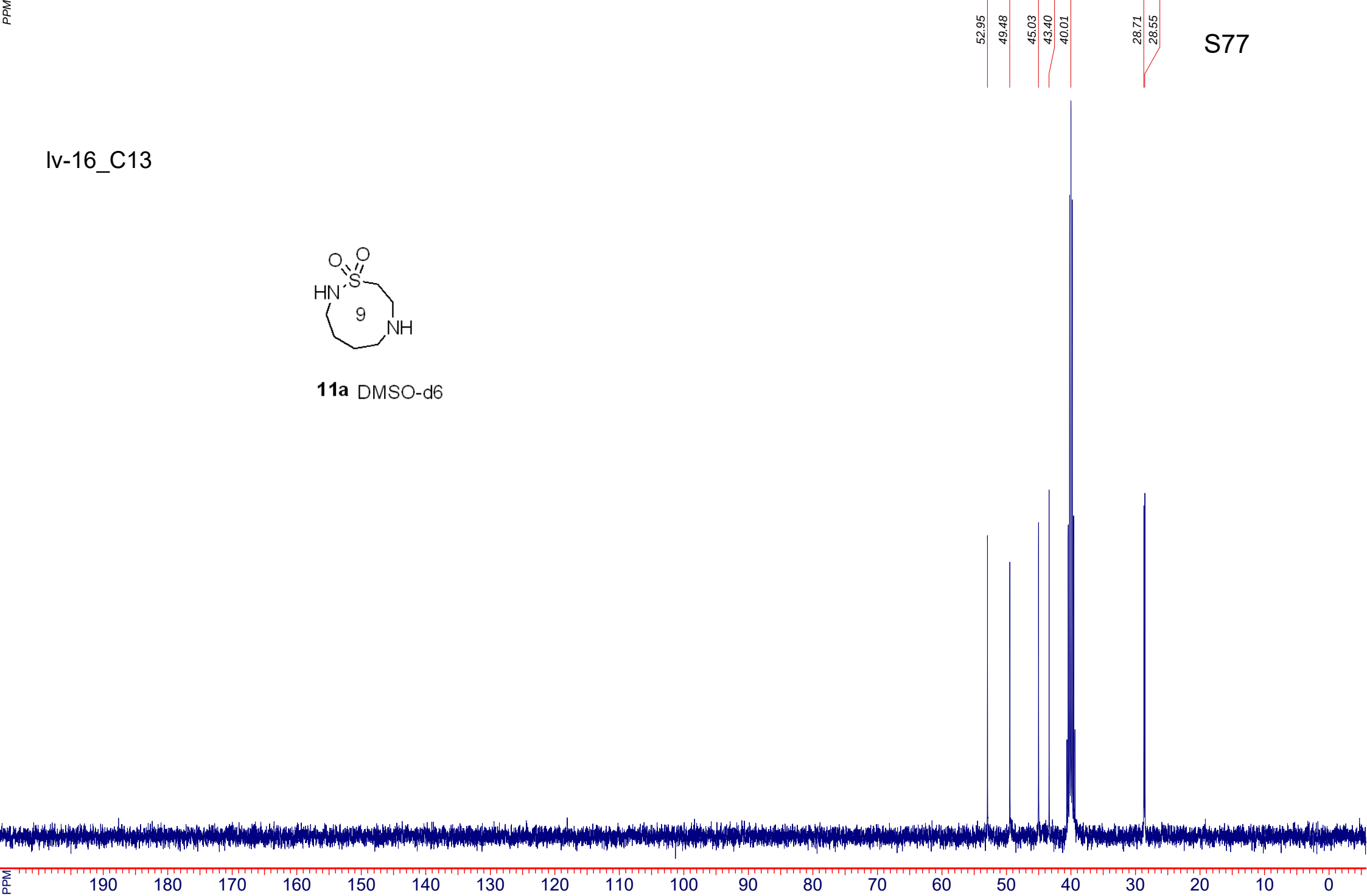
11a DMSO-d6



File name: LV-16	Operator: nmr	SF: 400.1300 MHz	NSC: 1	PW: 0.00 usec, RG: 25	SI: 32768
Date: 03-Jun-2022	Solvent: DMSO	SW: 8224 Hz	TE: 300 K	AQ: 1.99 sec, RD: 0.00 sec	Parameter file, TOPSPINVersion 2.1

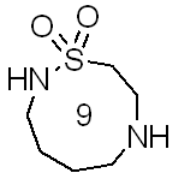
lv-16_C13

11a DMSO-d6

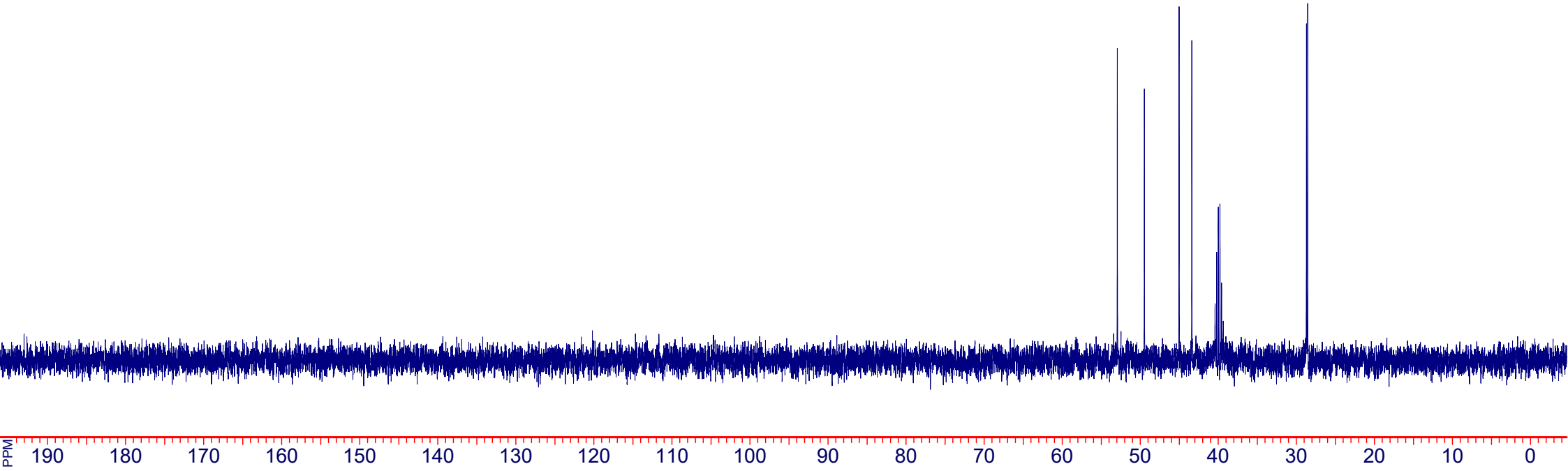


File name: lv-16_C13	Operator: nmr	SF: 100.6128 MHz	NSC: 122	PW: 0.00 usec, RG: 2050	SI: 32768
Date: 03-Jun-2022	Solvent: DMSO	SW: 26042 Hz	TE: 300 K	AQ: 0.98 sec, RD: 0.00 sec	Parameter file, TOPSPINVersion 2.1

lv-16_C13APT

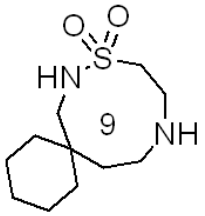


11a DMSO-d6

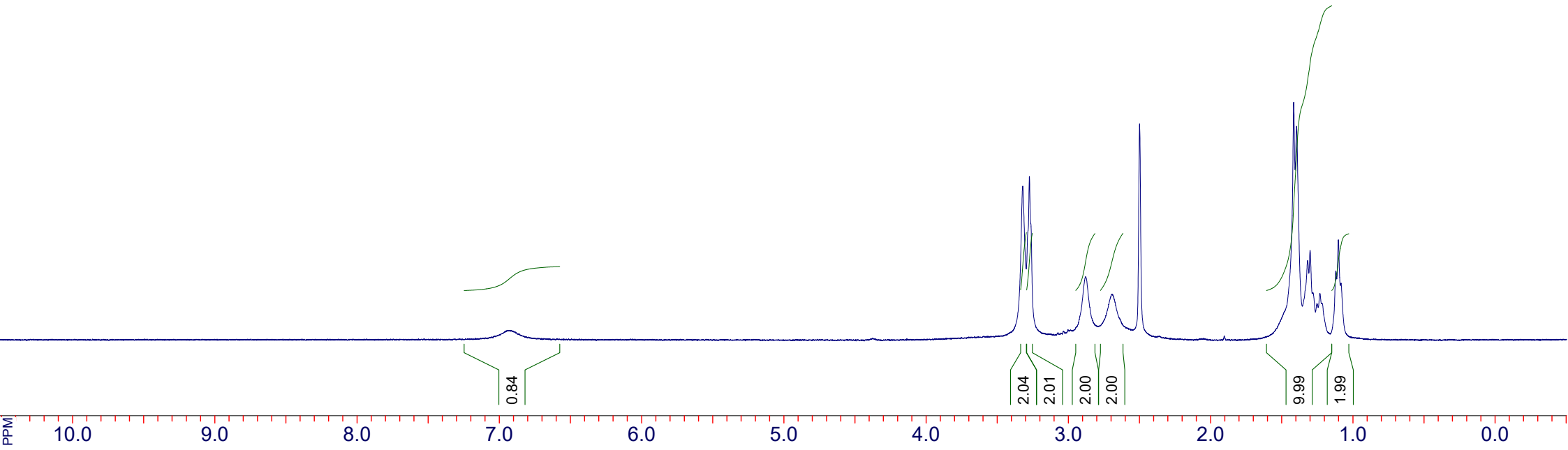


File name: lv-16_C13APT	Operator: nmr	SF: 100.6128 MHz	NSC: 357	PW: 0.00 usec, RG: 2050	SI: 65536
Date: 03-Jun-2022	Solvent: DMSO	SW: 24038 Hz	TE: 300 K	AQ: 1.36 sec, RD: 0.00 sec	Parameter file, TOPSPINVersion 2.1

lv633-1



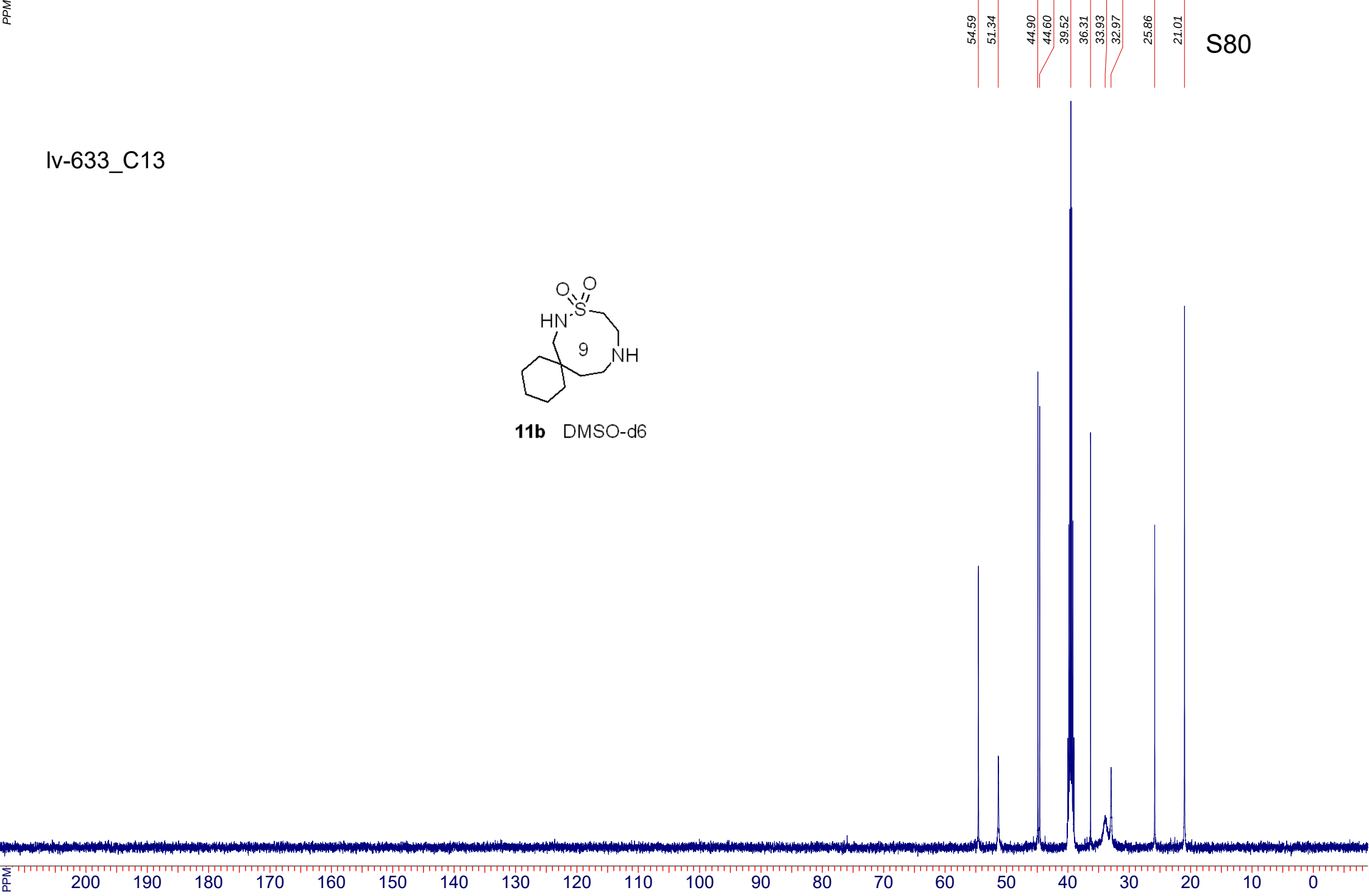
11b DMSO-d6



File name: lv633-1	Operator: root	SF: 499.6730 MHz	NSC: 1	PW: 0.00 usec, RG: 32	SI: 32768
Date: 10-Nov-2022	Solvent: DMSO	SW: 8993 Hz	TE: 683 K	AQ: 1.82 sec, RD: 0.00 sec	

lv-633_C13

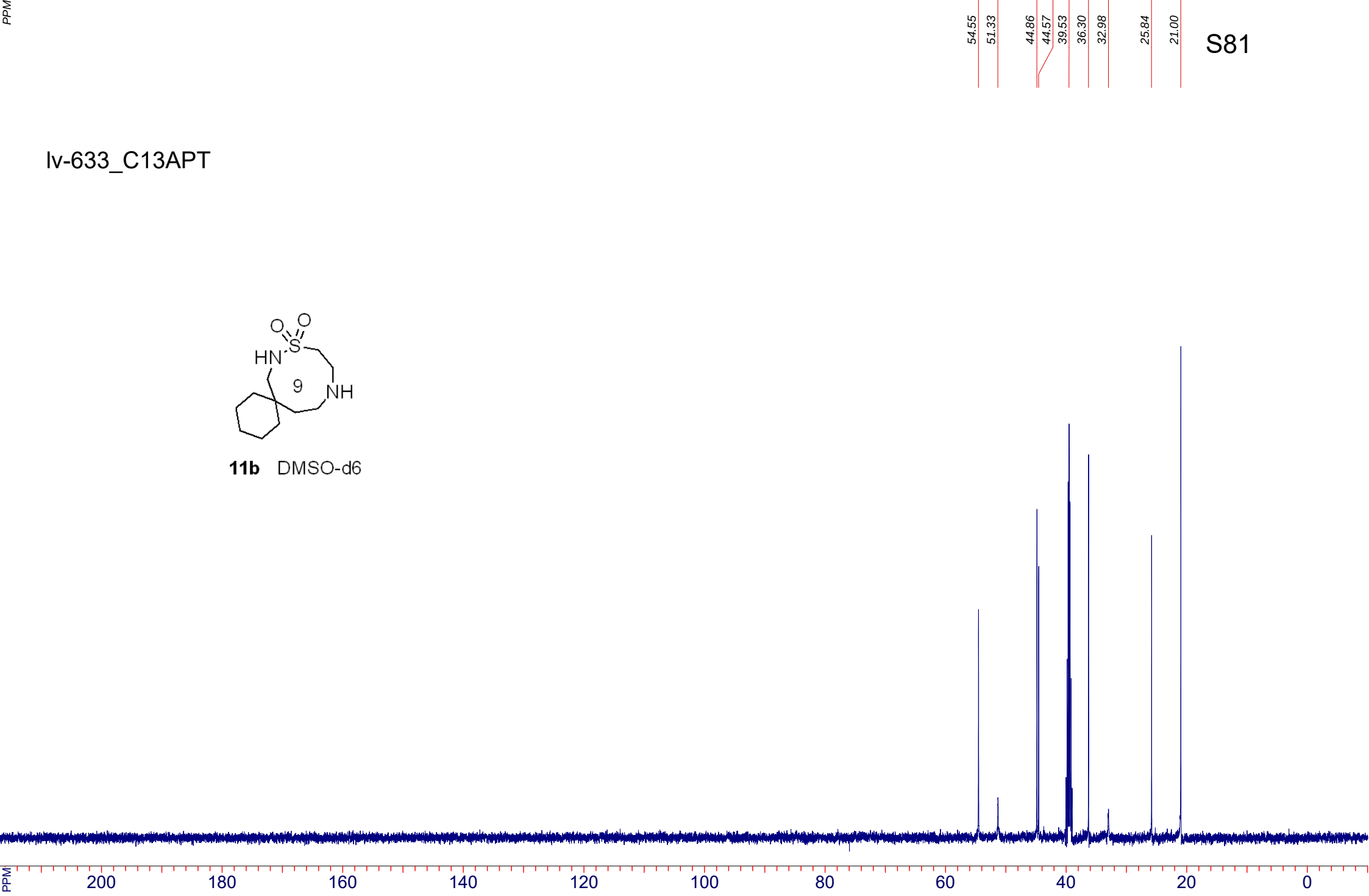
11b DMSO-d6



File name: lv-633_C13	Operator: root	SF: 125.6429 MHz	NSC: 500	PW: 0.00 usec, RG: 51200	SI: 131072
Date: 12-Nov-2022	Solvent: DMSO	SW: 32680 Hz	TE: 683 K	AQ: 0.78 sec, RD: 0.00 sec	

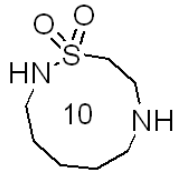
lv-633_C13APT

11b DMSO-d6

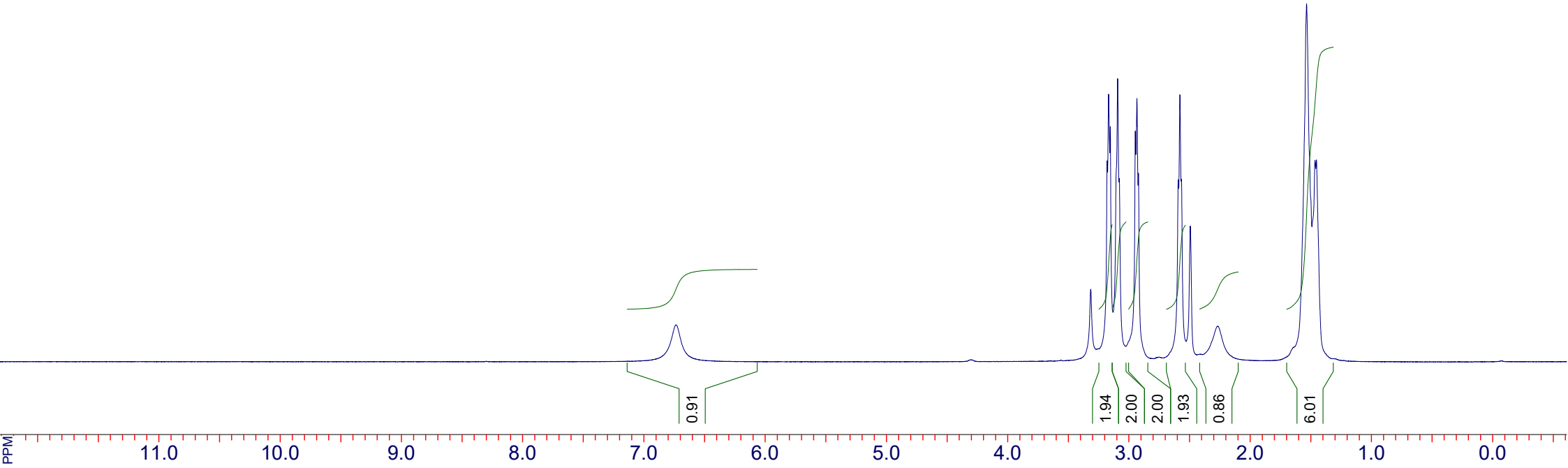


File name: lv-633_C13APT	Operator: root	SF: 125.6429 MHz	NSC: 600	PW: 0.00 usec, RG: 51200	SI: 65536
Date: 12-Nov-2022	Solvent: DMSO	SW: 32680 Hz	TE: 683 K	AQ: 1.00 sec, RD: 0.00 sec	

lv-13



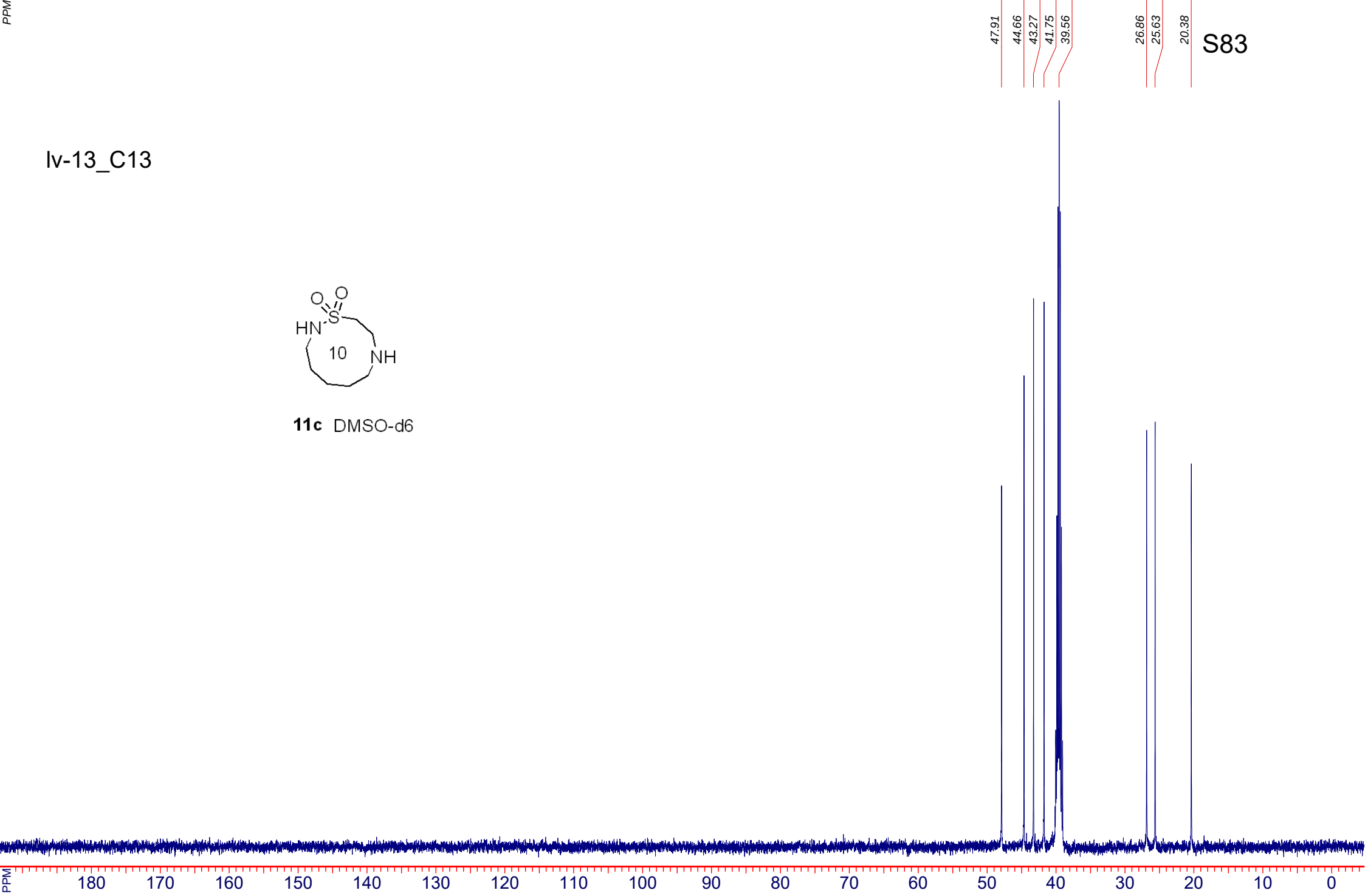
11c DMSO-d6



File name: lv-13	Operator: nmr	SF: 400.1300 MHz	NSC: 1	PW: 0.00 usec, RG: 25	SI: 32768
Date: 26-May-2022	Solvent: DMSO	SW: 8224 Hz	TE: 300 K	AQ: 1.99 sec, RD: 0.00 sec	Parameter file, TOPSPINVersion 2.1

lv-13_C13

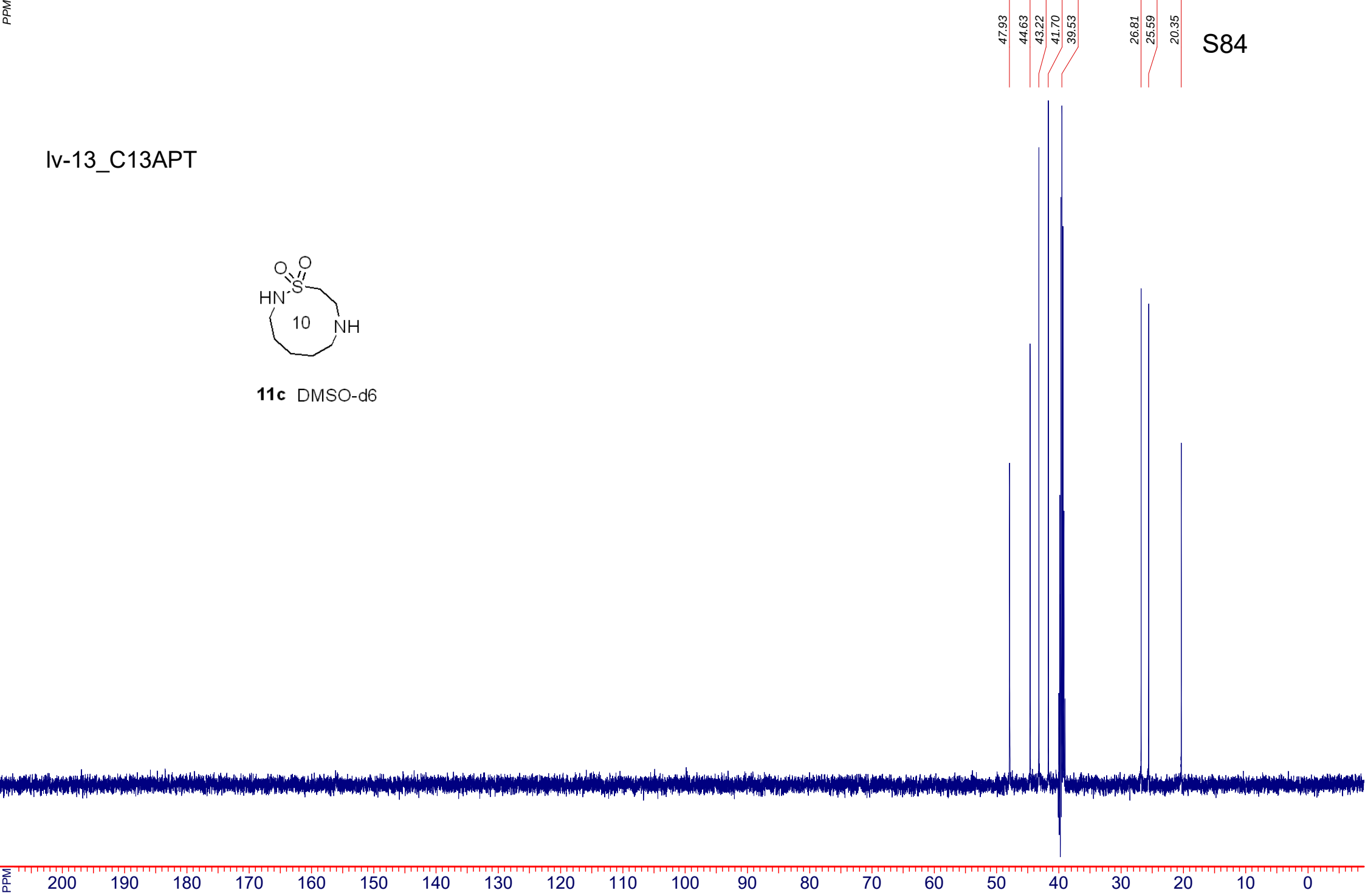
11c DMSO-d6



File name: lv-13_C13	Operator: root	SF: 125.6681 MHz	NSC: 237	PW: 0.00 usec, RG: 51200	SI: 131072
Date: 26-May-2022	Solvent: DMSO	SW: 32680 Hz	TE: 683 K	AQ: 0.78 sec, RD: 0.00 sec	

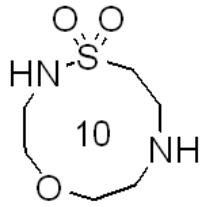
lv-13_C13APT

11c DMSO-d6

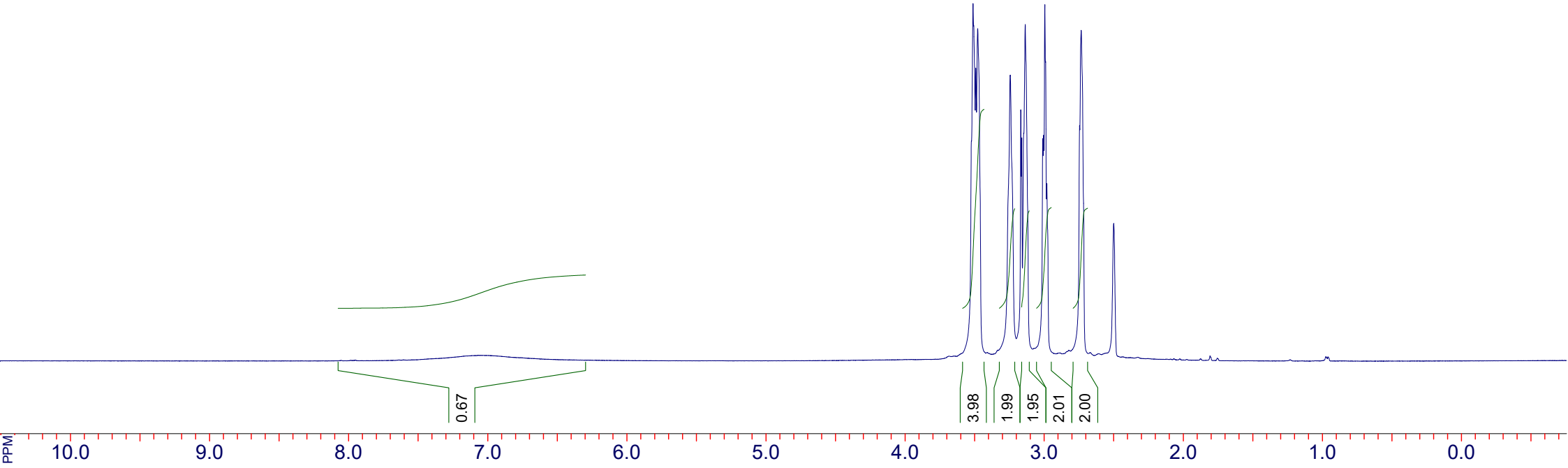


File name: lv-13_C13APT	Operator: root	SF: 125.6681 MHz	NSC: 237	PW: 0.00 usec, RG: 51200	SI: 65536
Date: 26-May-2022	Solvent: DMSO	SW: 32680 Hz	TE: 683 K	AQ: 1.57 sec, RD: 0.00 sec	

lv-693

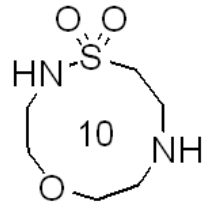


11d DMSO-d6

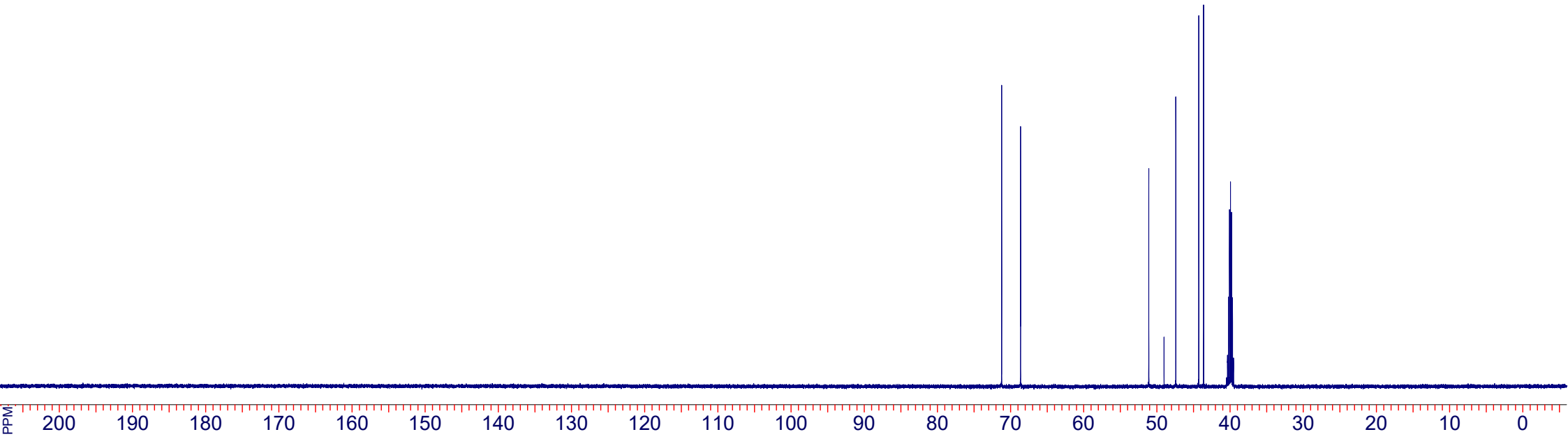


File name: lv-693	Operator: nmr	SF: 400.1300 MHz	NSC: 1	PW: 0.00 usec, RG: 25	SI: 32768
Date: 09-Dec-2022	Solvent: DMSO	SW: 8224 Hz	TE: 300 K	AQ: 1.99 sec, RD: 0.00 sec	

lv-693_C13.fid



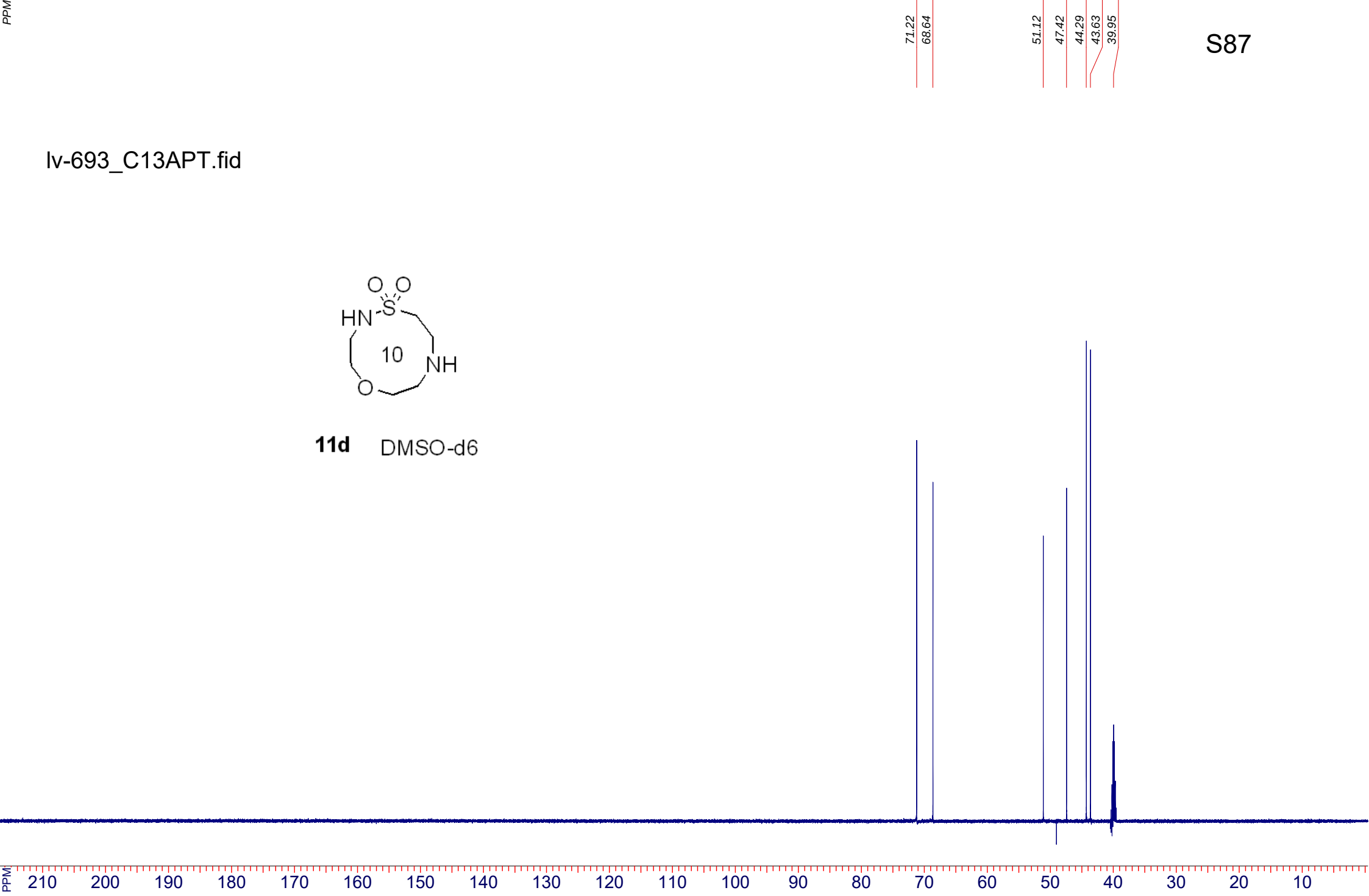
11d DMSO-d6



File name: lv-693_C13.fid	Operator:	SF: 150.8304 MHz	NSC: 0	PW: 3.09 usec, RG: 60	SI: 65536
Date: 09-Dec-2022	Solvent: dmso	SW: 37879 Hz	TE: 298 K	AQ: 0.87 sec, RD: 0.00 sec	

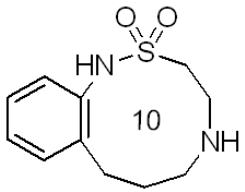
lv-693_C13APT.fid

11d DMSO-d6

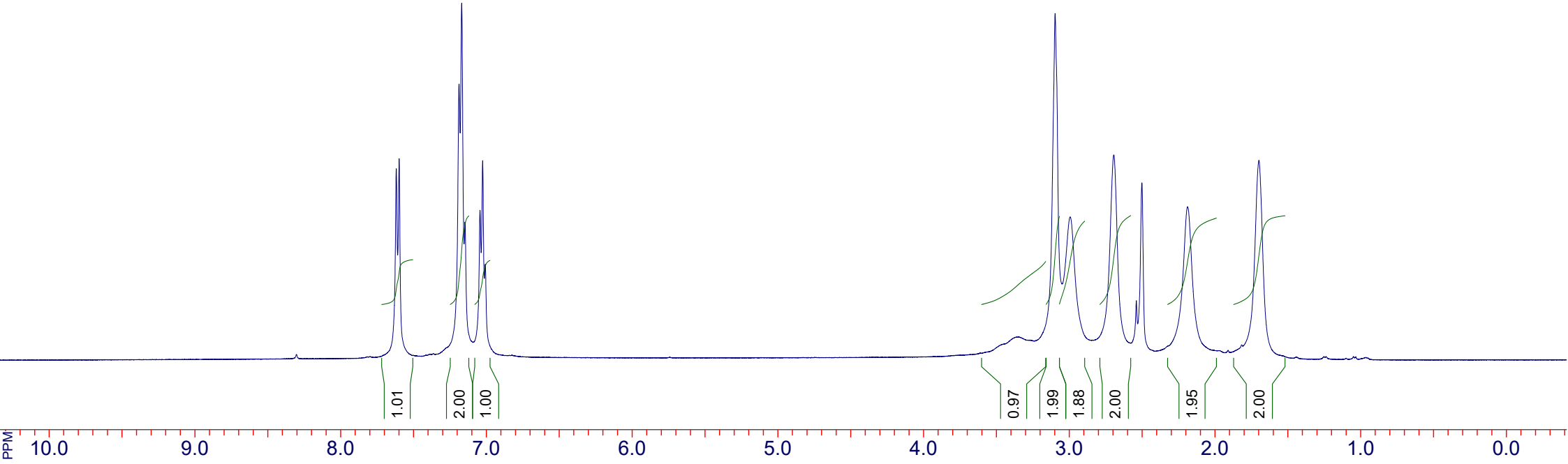


File name: lv-693_C13APT.fid	Operator:	SF: 150.8333 MHz	NSC: 0	PW: 4.63 usec, RG: 60	SI: 131072
Date: 09-Dec-2022	Solvent: dmso	SW: 39063 Hz	TE: 298 K	AQ: 0.87 sec, RD: 0.00 sec	

lv-538.fid



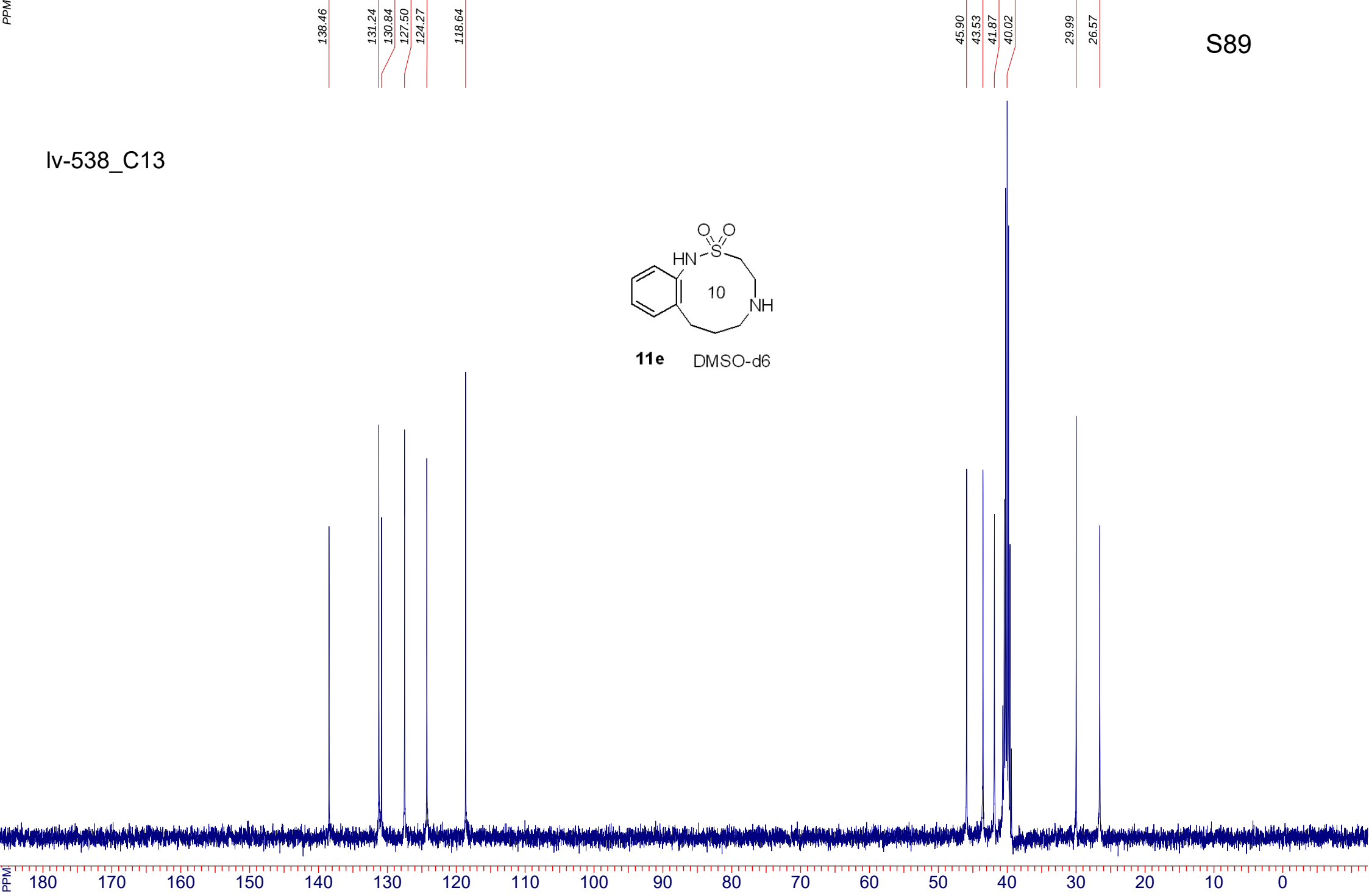
11e DMSO-d6



File name: lv-538.fid	Operator:	SF: 399.9733 MHz	NSC: 0	PW: 10.90 usec, RG: 24	SI: 32768
Date: 03-Oct-2022	Solvent: dmso	SW: 8000 Hz	TE: 298 K	AQ: 2.00 sec, RD: 0.00 sec	

lv-538_C13

11e DMSO-d6

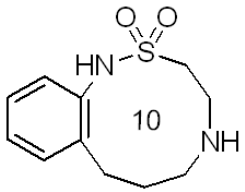


File name: lv-538_C13	Operator: nmr	SF: 100.6128 MHz	NSC: 204	PW: 0.00 usec, RG: 2050	SI: 32768
Date: 03-Oct-2022	Solvent: DMSO	SW: 26042 Hz	TE: 300 K	AQ: 0.98 sec, RD: 0.00 sec	

PPM

138.46 131.24 130.84 127.50 124.27 118.63 45.89 43.52 41.87 40.01 29.99 26.57

lv-538_C13APT

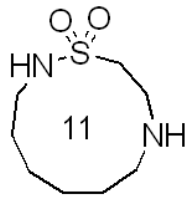


11e DMSO-d6

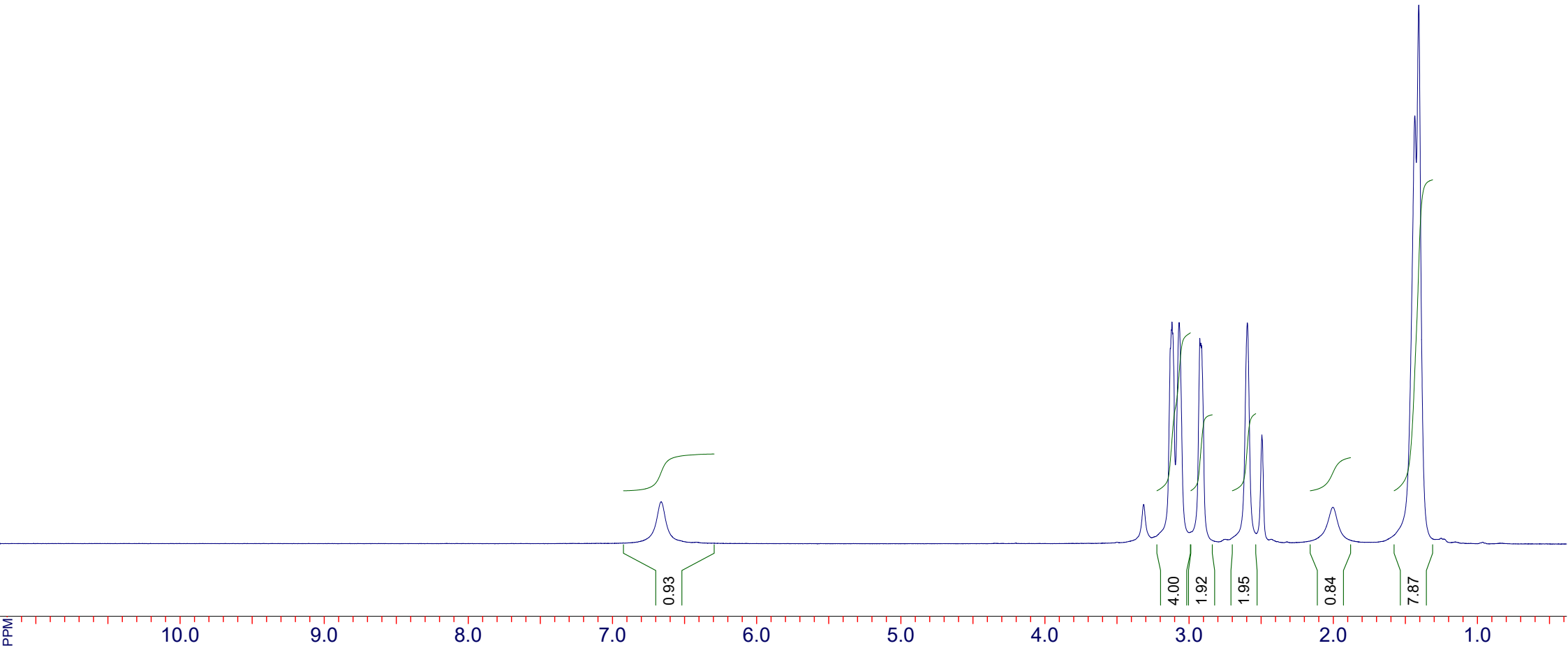
PPM 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0

File name: lv-538_C13APT	Operator: nmr	SF: 100.6128 MHz	NSC: 46	PW: 0.00 usec, RG: 2050	SI: 65536
Date: 03-Oct-2022	Solvent: DMSO	SW: 24038 Hz	TE: 300 K	AQ: 1.36 sec, RD: 0.00 sec	

lv-12



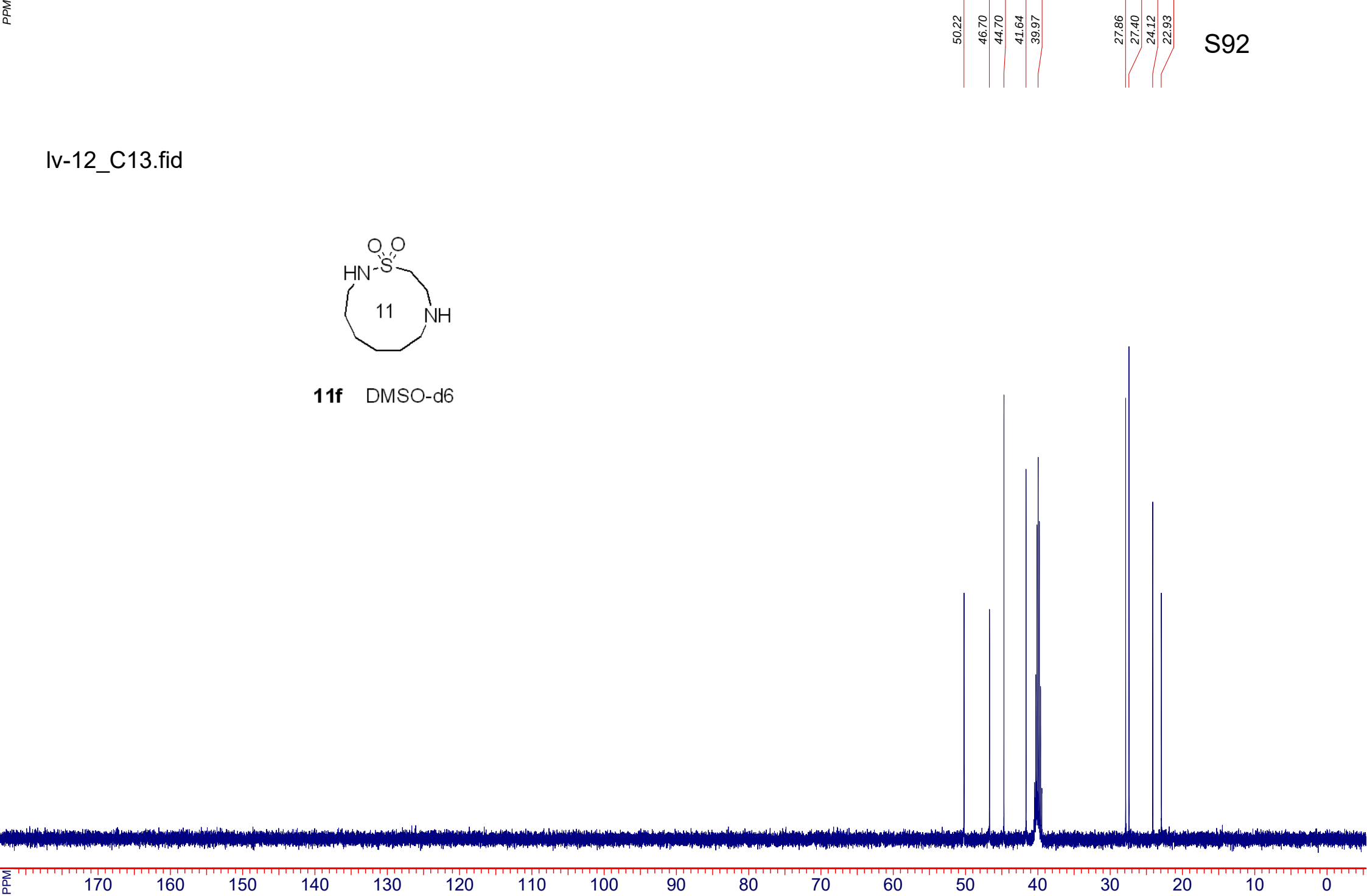
11f DMSO-d6



File name: lv-12	Operator: nmr	SF: 400.1300 MHz	NSC: 1	PW: 0.00 usec, RG: 25	SI: 32768
Date: 26-May-2022	Solvent: DMSO	SW: 8224 Hz	TE: 300 K	AQ: 1.99 sec, RD: 0.00 sec	

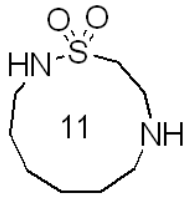
lv-12_C13.fid

11f DMSO-d6

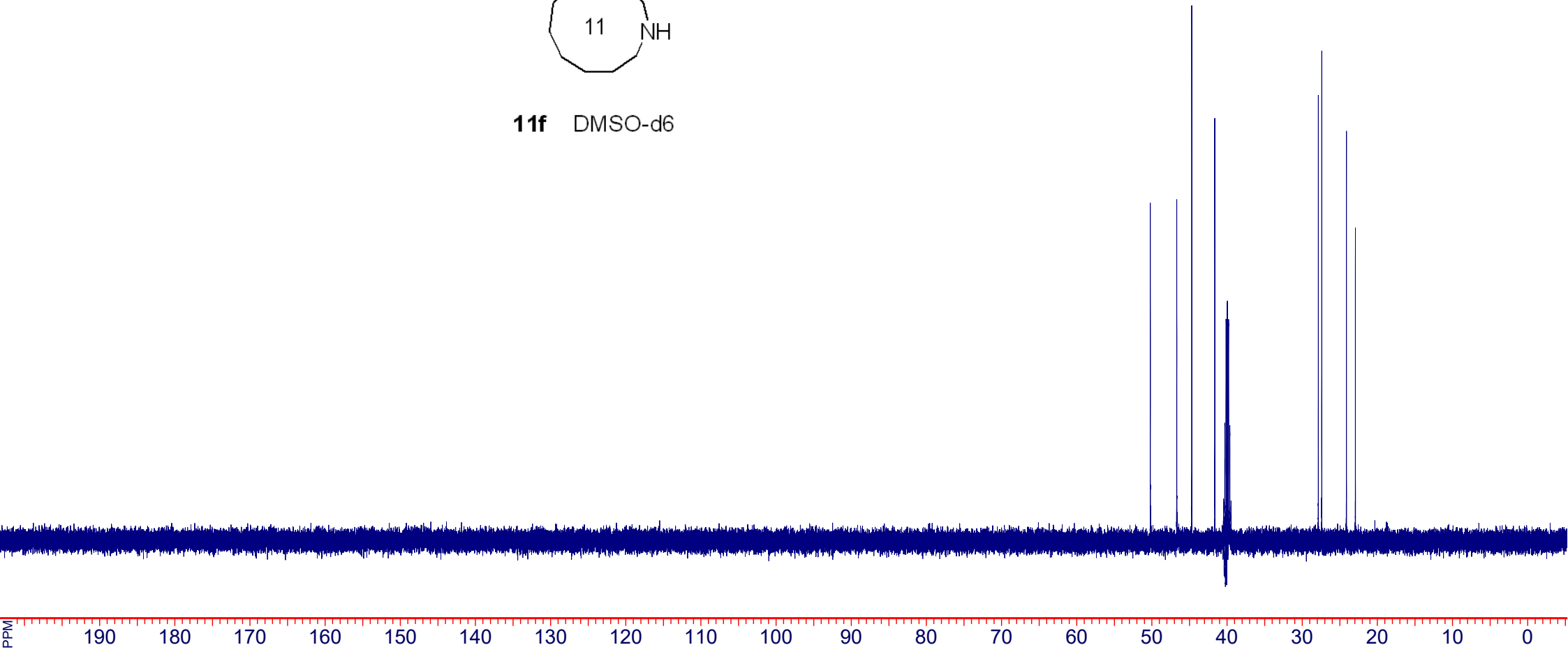


File name: lv-12_C13.fid	Operator:	SF: 125.6926 MHz	NSC: 0	PW: 3.27 usec, RG: 60	SI: 65536
Date: 26-May-2022	Solvent: dmso	SW: 31250 Hz	TE: 298 K	AQ: 1.05 sec, RD: 0.00 sec	

lv-12_C13APT.fid



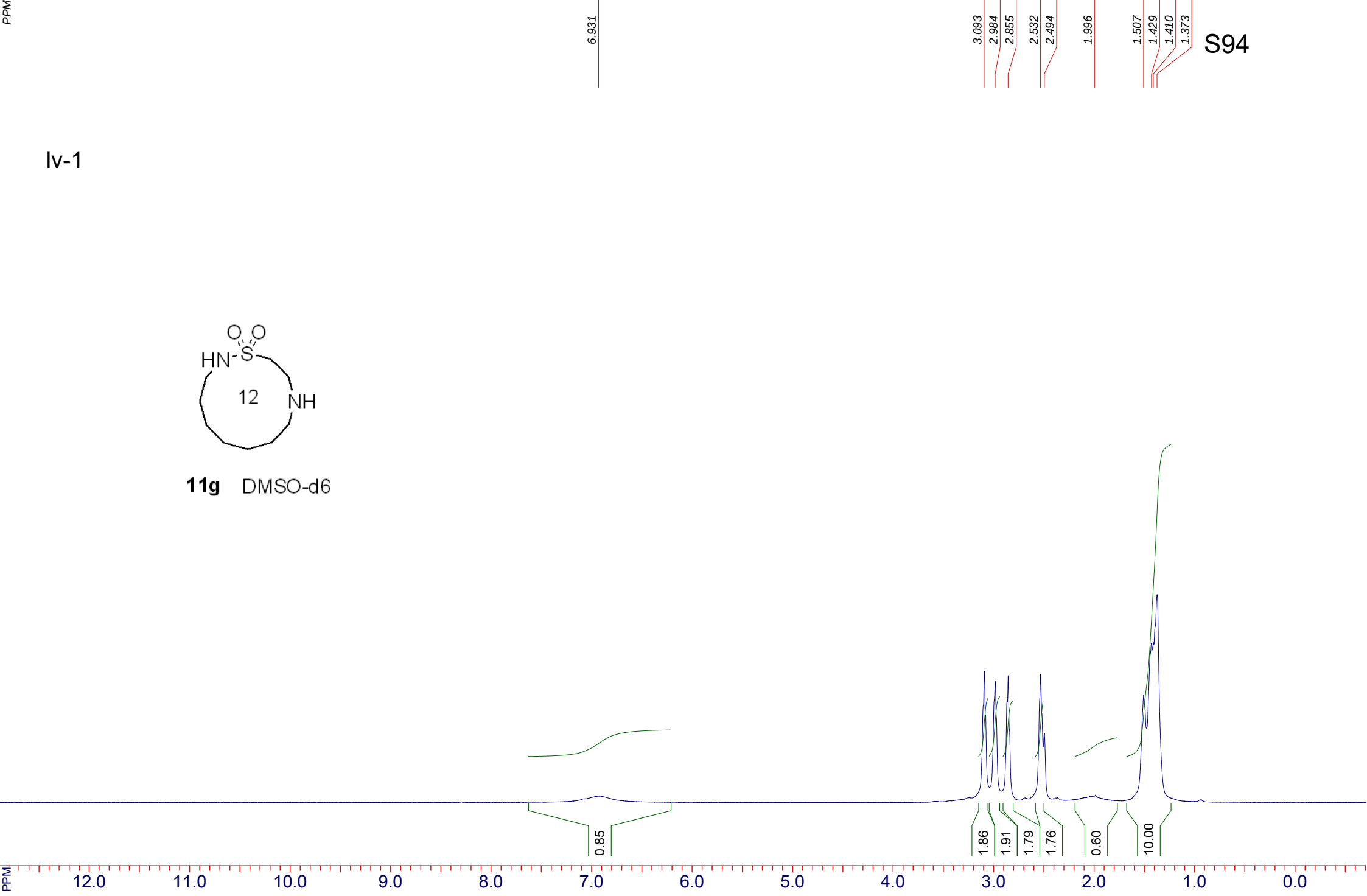
11f DMSO-d6



File name: lv-12_C13APT.fid	Operator:	SF: 125.6925 MHz	NSC: 0	PW: 5.00 usec, RG: 60	SI: 131072
Date: 26-May-2022	Solvent: dmso	SW: 32895 Hz	TE: 298 K	AQ: 1.95 sec, RD: 0.00 sec	Automated Probe tuning parameter

lv-1

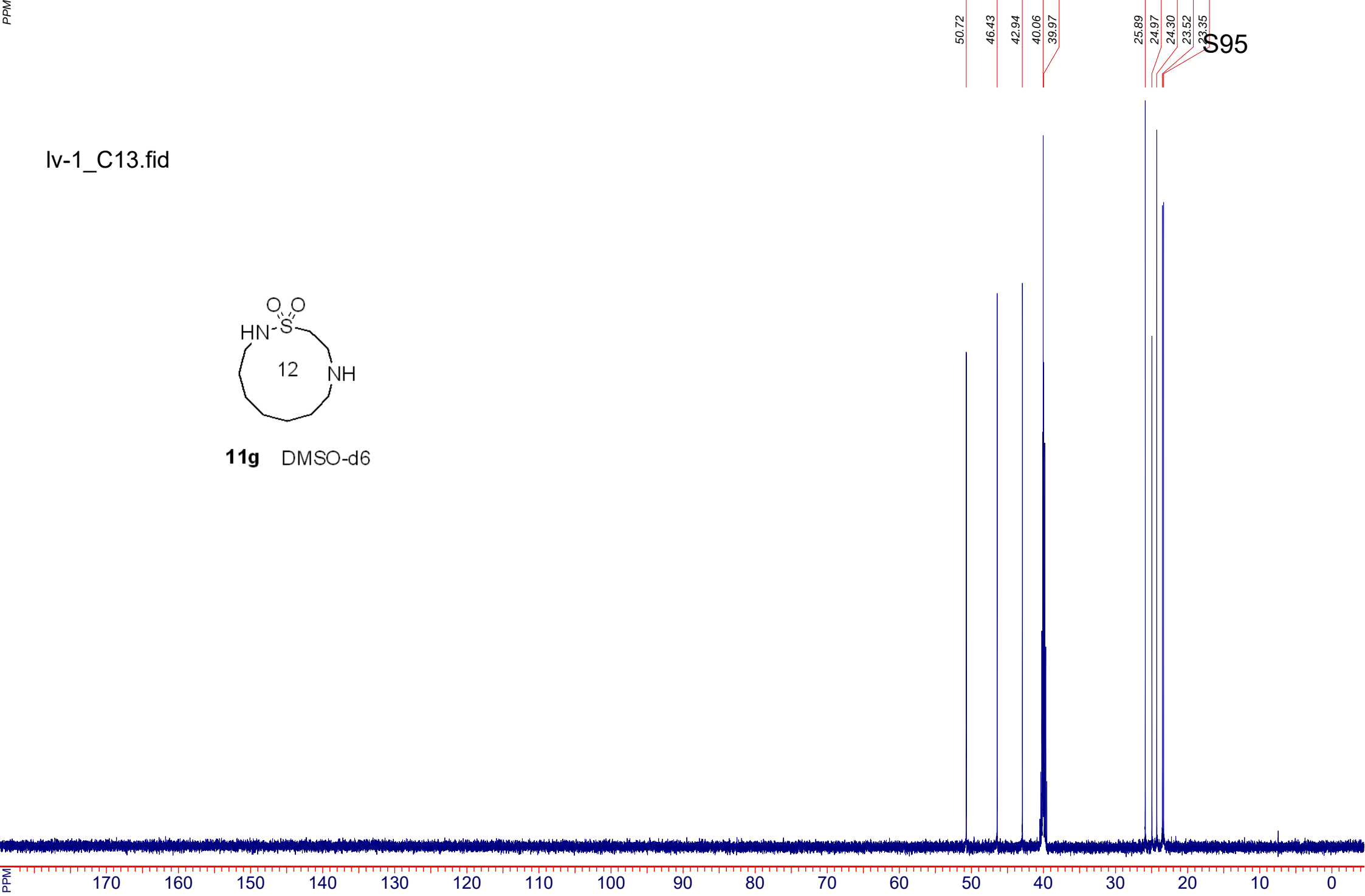
11g DMSO-d6



File name: lv-1	Operator: nmr	SF: 400.1300 MHz	NSC: 1	PW: 0.00 usec, RG: 25	SI: 32768
Date: 26-May-2022	Solvent: DMSO	SW: 8224 Hz	TE: 300 K	AQ: 1.99 sec, RD: 0.00 sec	

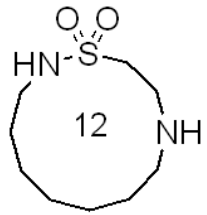
lv-1_C13.fid

11g DMSO-d6

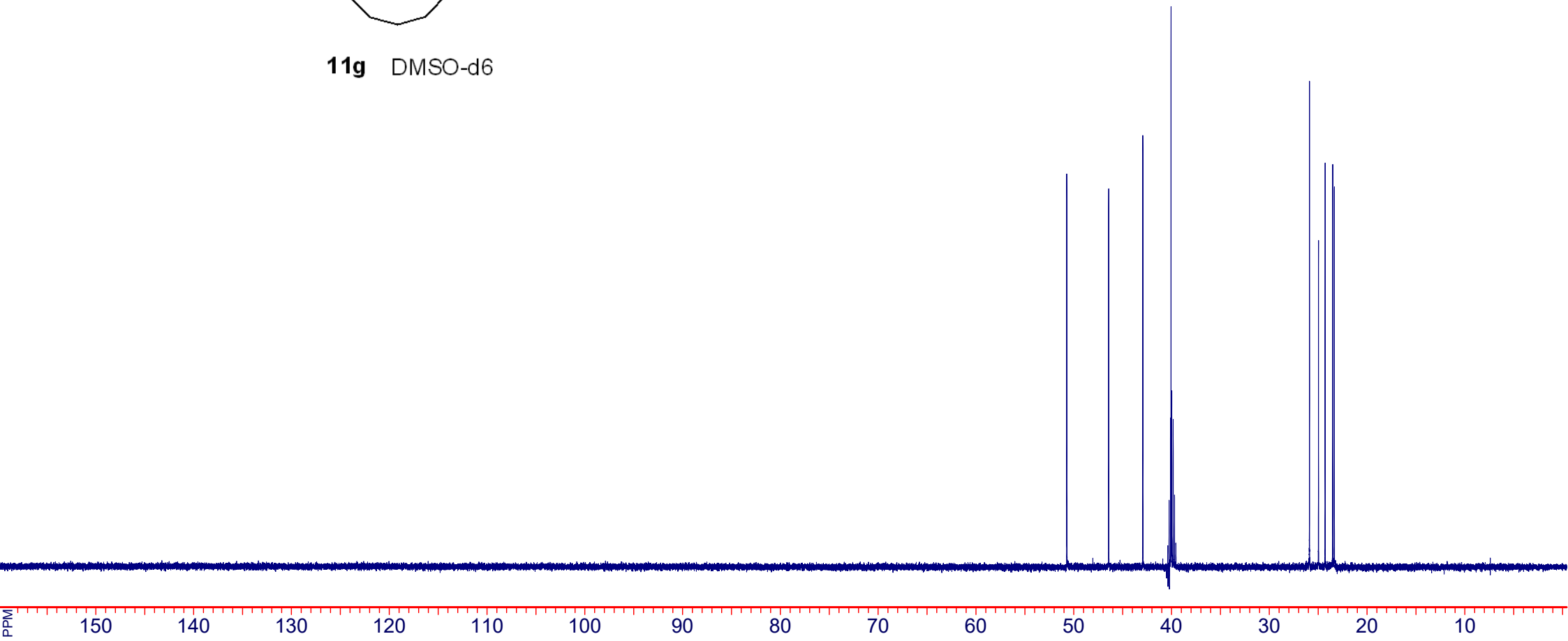


File name: lv-1_C13.fid	Operator:	SF: 150.8304 MHz	NSC: 0	PW: 3.09 usec, RG: 60	SI: 65536
Date: 26-May-2022	Solvent: dmso	SW: 37879 Hz	TE: 298 K	AQ: 0.87 sec, RD: 0.00 sec	

lv-1_C13APT.fid

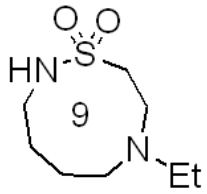


11g DMSO-d6

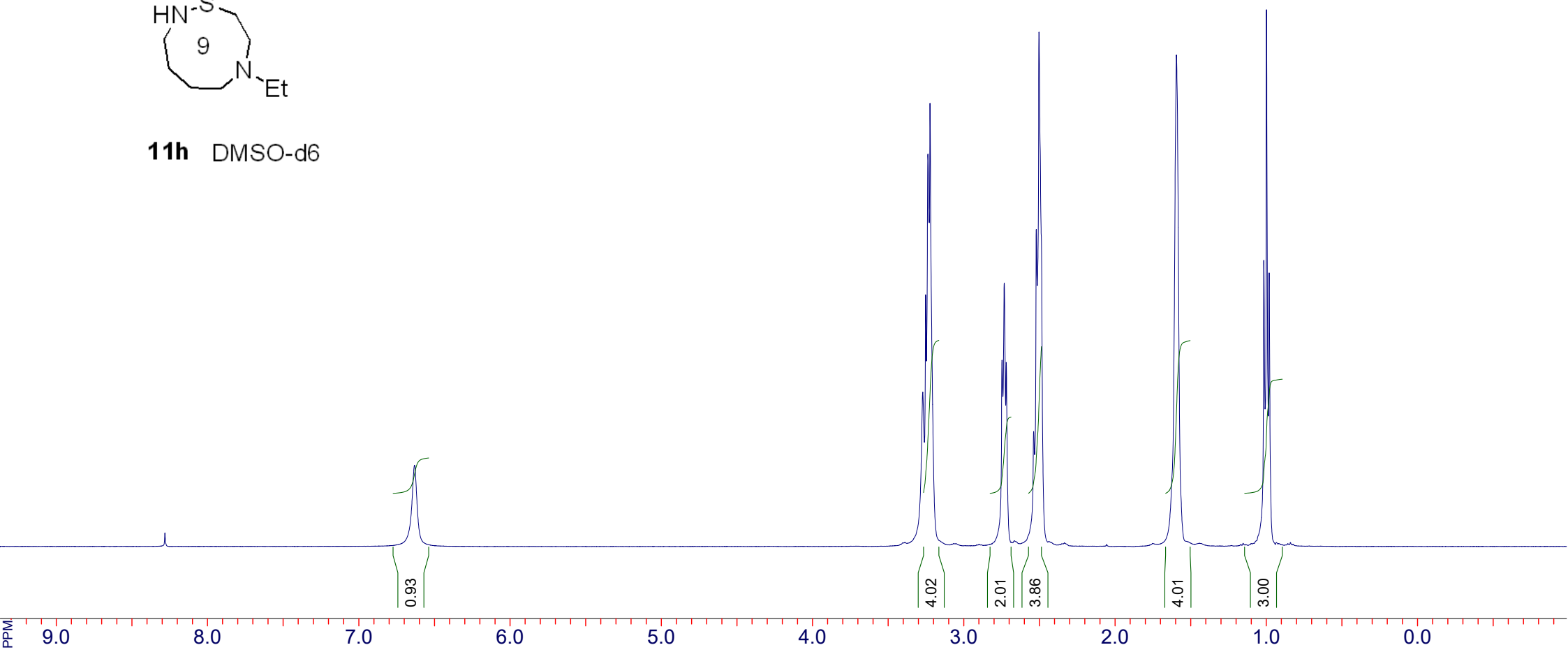


File name: lv-1_C13APT.fid	Operator:	SF: 150.8333 MHz	NSC: 0	PW: 4.63 usec, RG: 60	SI: 131072
Date: 26-May-2022	Solvent: dmso	SW: 39063 Hz	TE: 298 K	AQ: 0.87 sec, RD: 0.00 sec	

lv-887



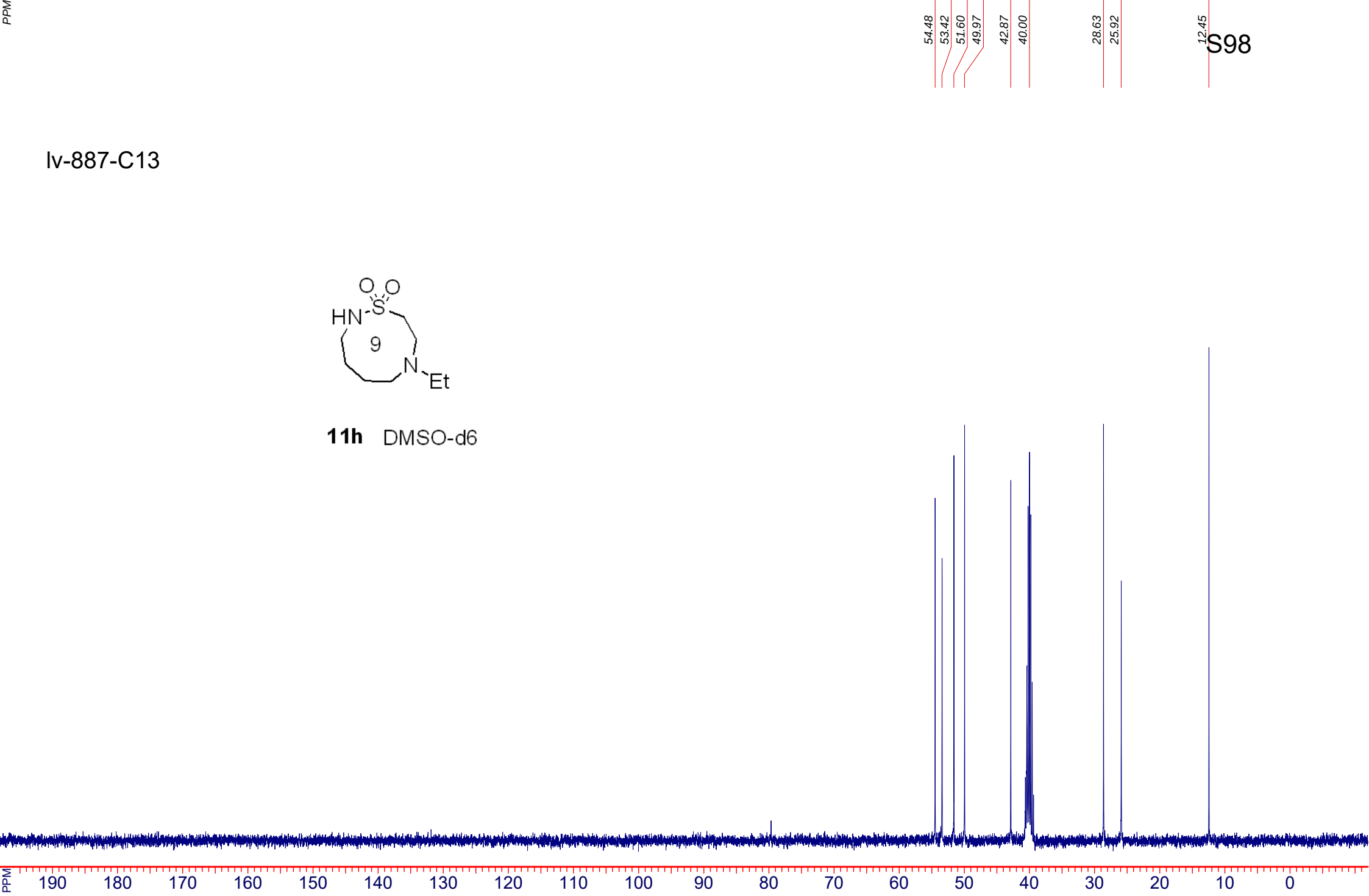
11h DMSO-d6



File name: lv-887	Operator: nmr	SF: 400.1300 MHz	NSC: 1	PW: 0.00 usec, RG: 25	SI: 32768
Date: 27-Feb-2023	Solvent: DMSO	SW: 8224 Hz	TE: 300 K	AQ: 1.99 sec, RD: 0.00 sec	

lv-887-C13

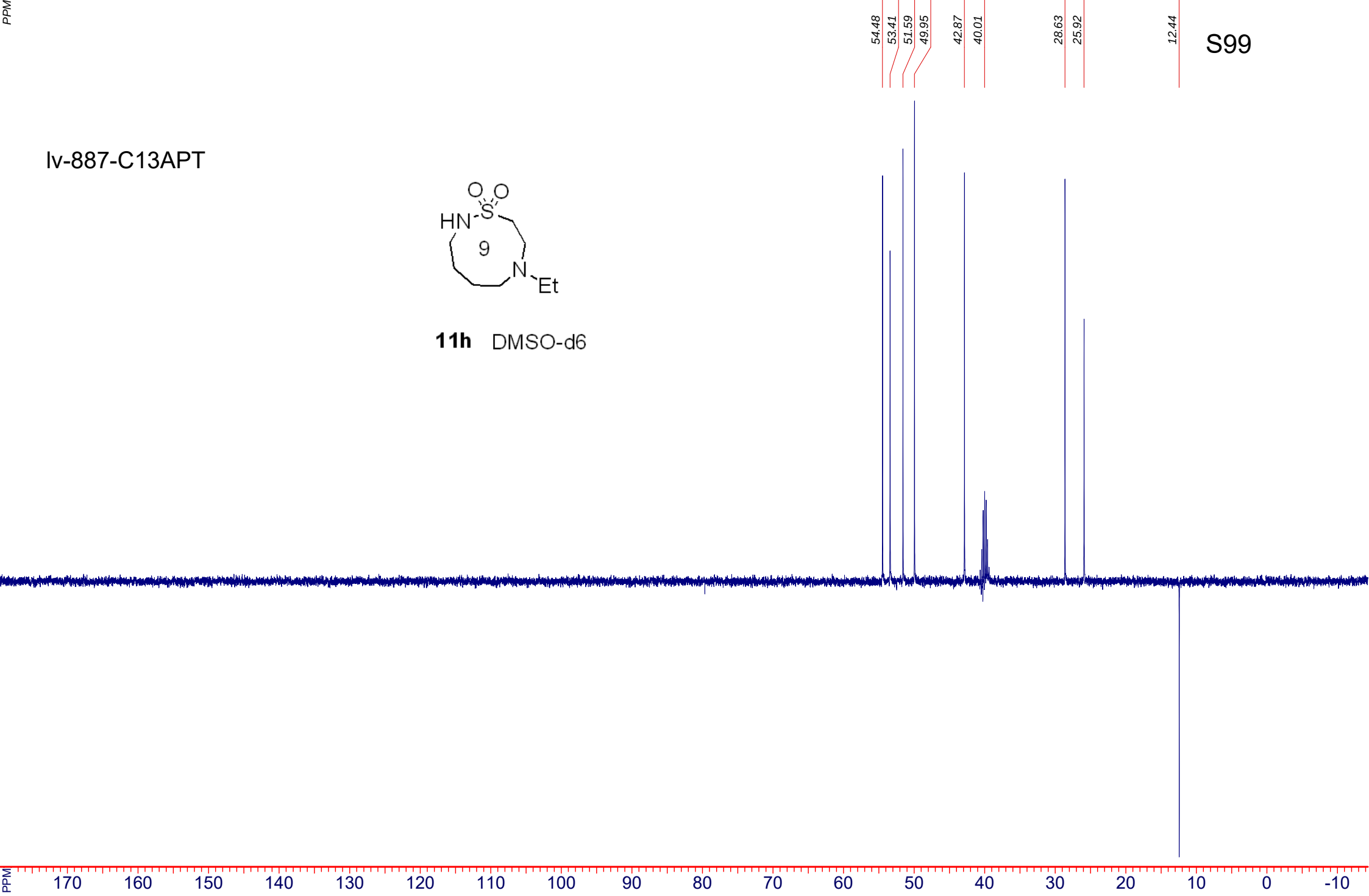
11h DMSO-d6



File name: lv-887-C13	Operator: nmr	SF: 100.6128 MHz	NSC: 114	PW: 0.00 usec, RG: 2050	SI: 32768
Date: 27-Feb-2023	Solvent: DMSO	SW: 26042 Hz	TE: 300 K	AQ: 0.98 sec, RD: 0.00 sec	

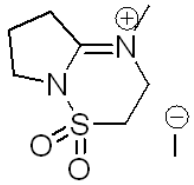
lv-887-C13APT

11h DMSO-d6



File name: lv-887-C13APT	Operator: nmr	SF: 100.6128 MHz	NSC: 105	PW: 0.00 usec, RG: 2050	SI: 65536
Date: 28-Feb-2023	Solvent: DMSO	SW: 24038 Hz	TE: 300 K	AQ: 1.36 sec, RD: 0.00 sec	

lv-22

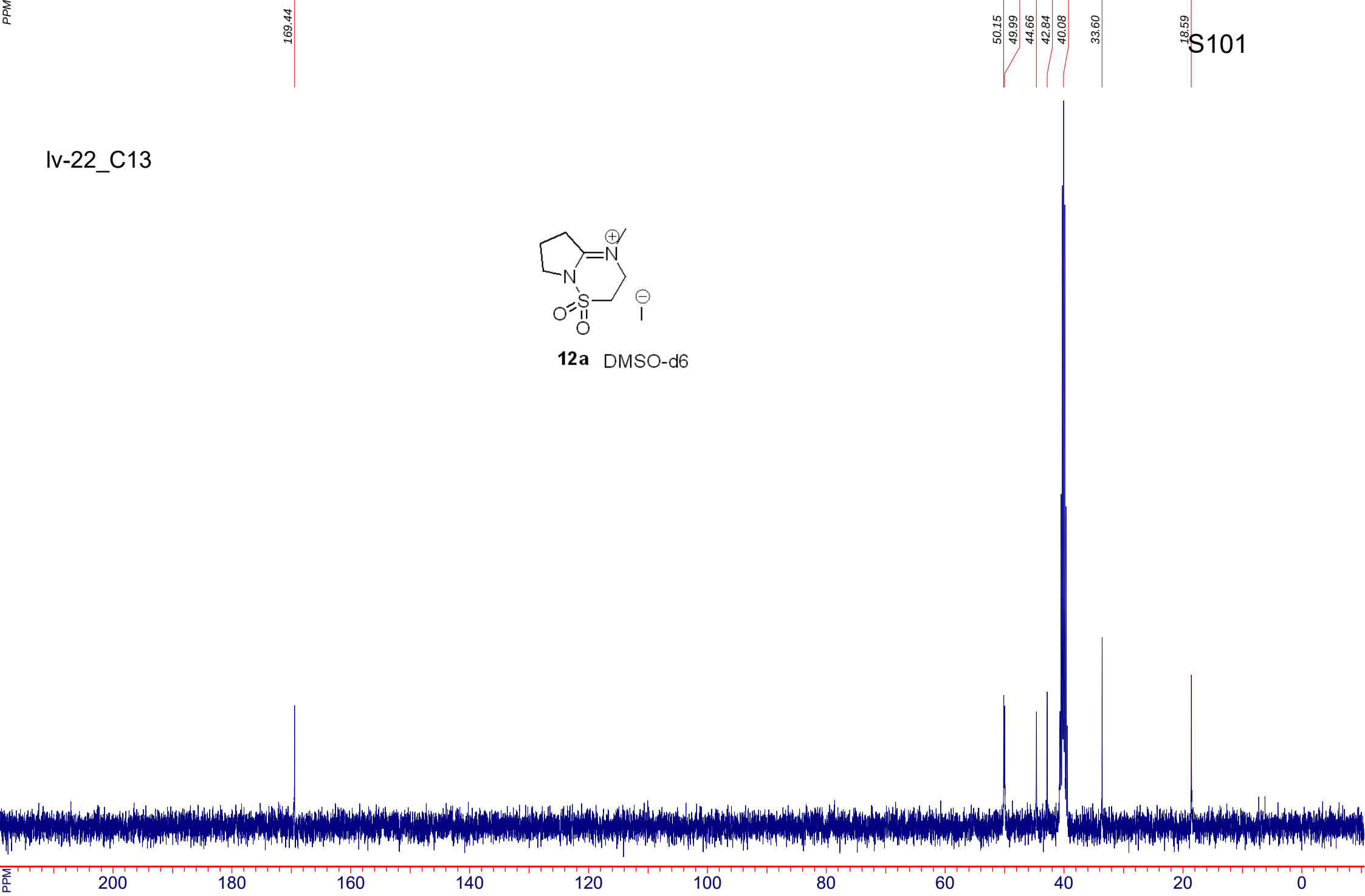


12a DMSO-d6

File name: lv-22	Operator: nmr	SF: 400.1300 MHz	NSC: 1	PW: 0.00 usec, RG: 25	SI: 32768
Date: 27-May-2022	Solvent: DMSO	SW: 8224 Hz	TE: 300 K	AQ: 1.99 sec, RD: 0.00 sec	

lv-22_C13

12a DMSO-d6



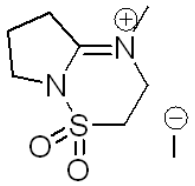
File name: lv-22_C13	Operator: nmr	SF: 100.6128 MHz	NSC: 91	PW: 0.00 usec, RG: 2050	SI: 32768
Date: 27-May-2022	Solvent: DMSO	SW: 26042 Hz	TE: 300 K	AQ: 0.98 sec, RD: 0.00 sec	

PPM

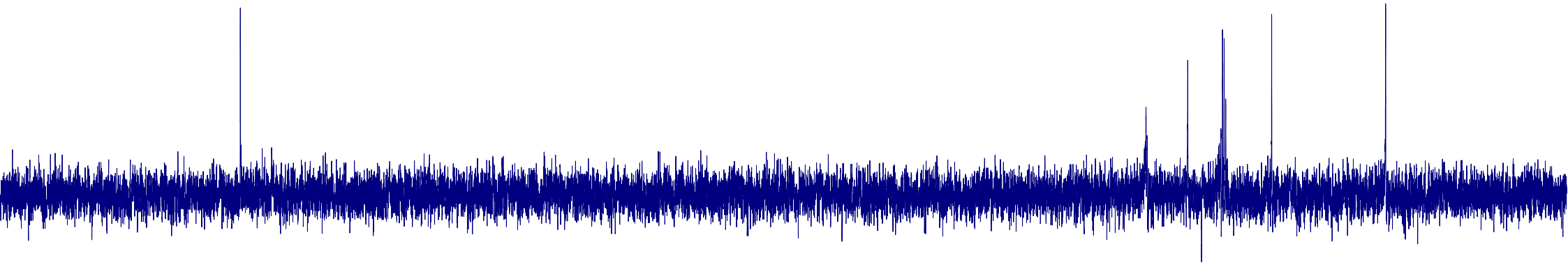
169.43

50.16 49.99 44.66 42.83 40.08 33.60 28.59

lv-22_C13APT



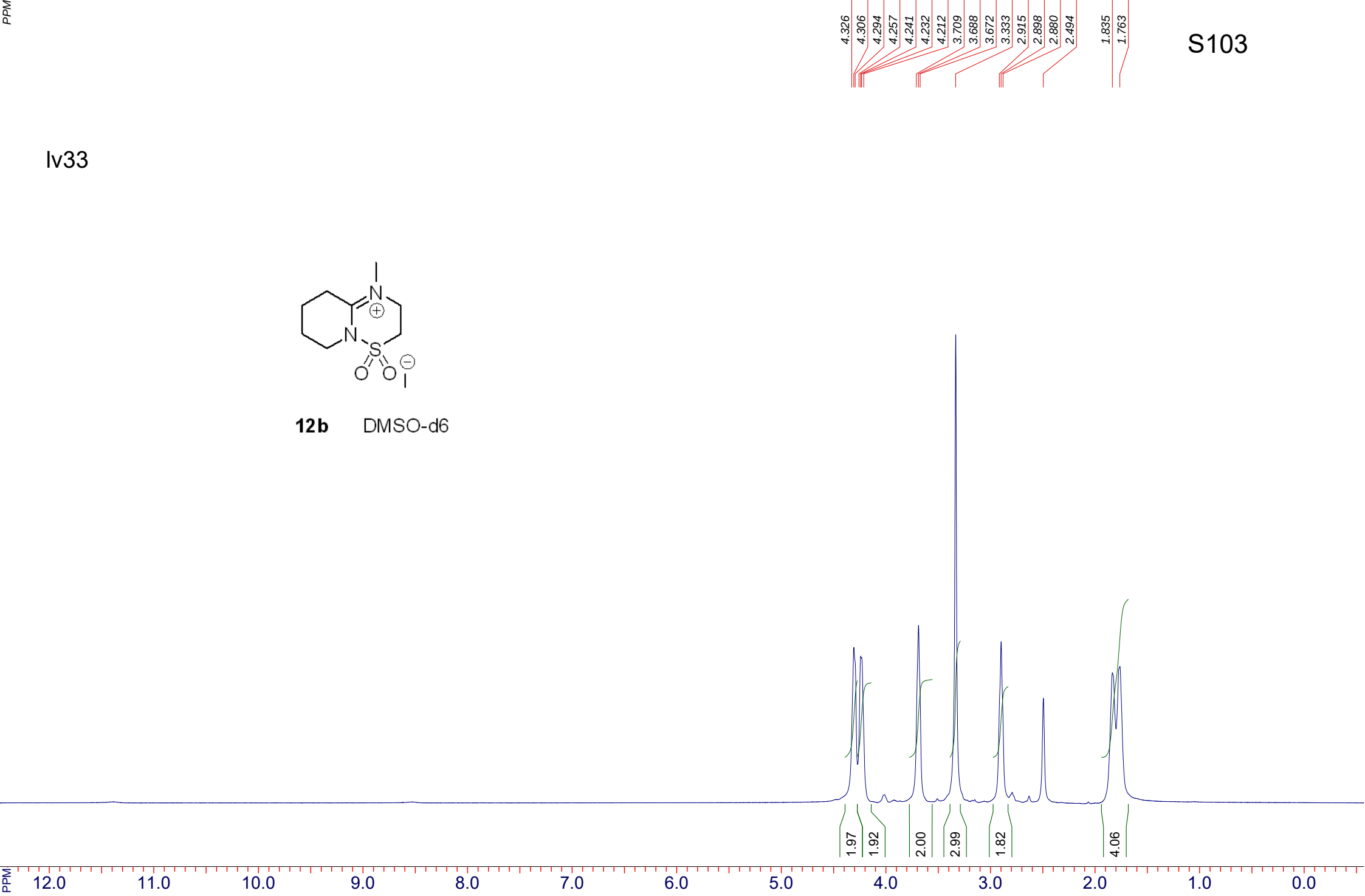
12a DMSO-d6



File name: lv-22_C13APT	Operator: nmr	SF: 100.6128 MHz	NSC: 455	PW: 0.00 usec, RG: 2050	SI: 65536
Date: 27-May-2022	Solvent: DMSO	SW: 24038 Hz	TE: 300 K	AQ: 1.36 sec, RD: 0.00 sec	

lv33

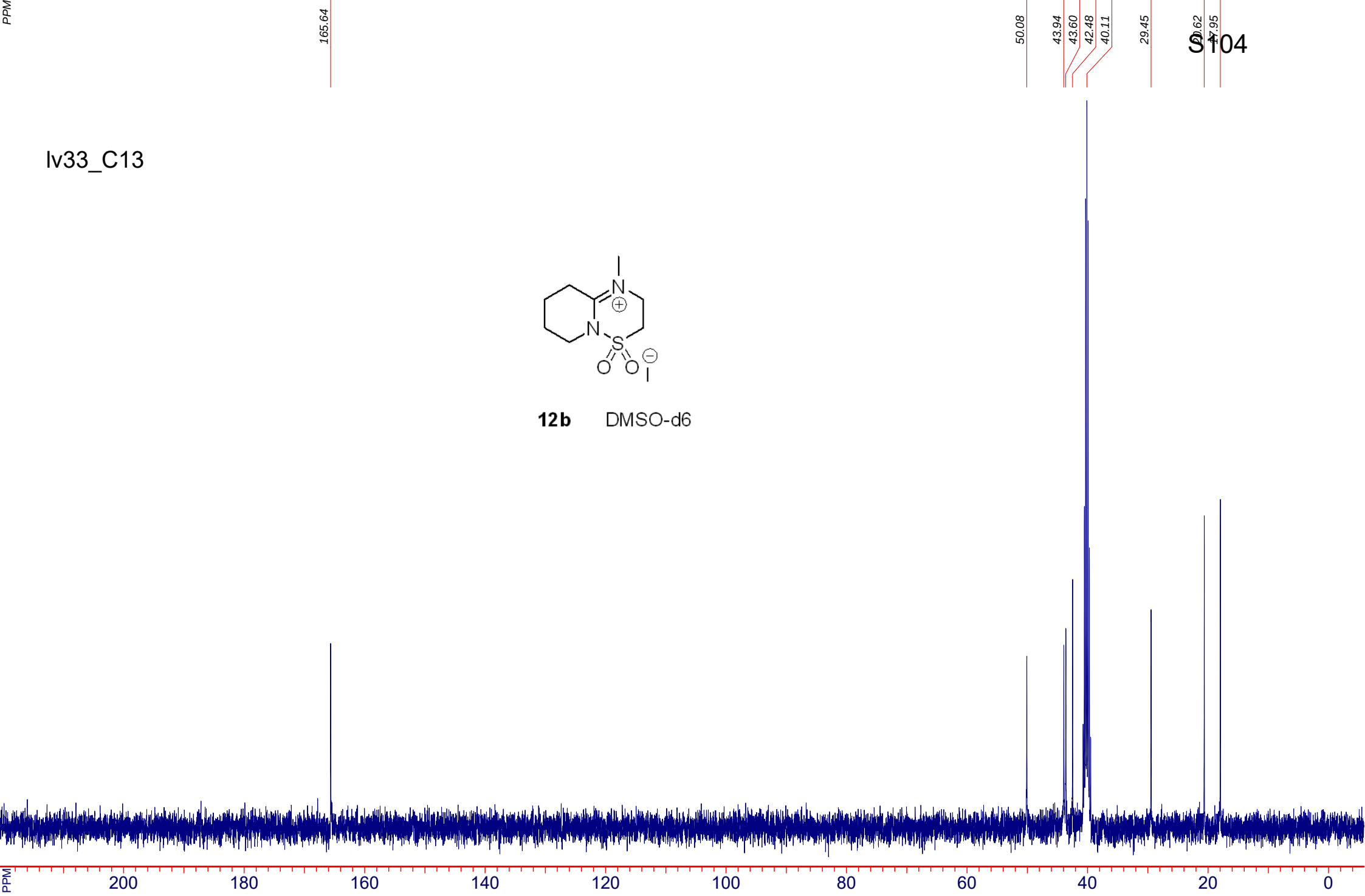
12b DMSO-d6



File name: lv33	Operator: nmr	SF: 400.1300 MHz	NSC: 1	PW: 0.00 usec, RG: 25	SI: 32768
Date: 30-May-2022	Solvent: DMSO	SW: 8224 Hz	TE: 300 K	AQ: 1.99 sec, RD: 0.00 sec	

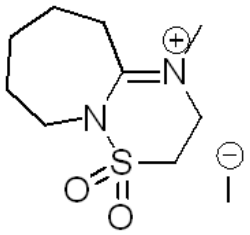
lv33_C13

12b DMSO-d6

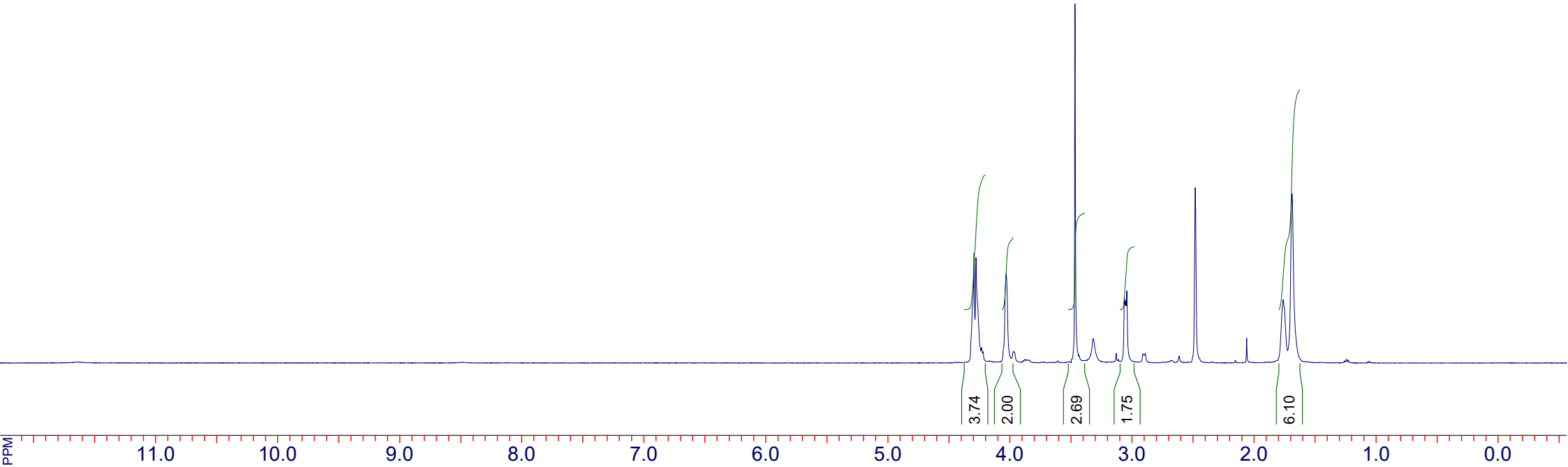


File name: lv33_C13	Operator: nmr	SF: 100.6128 MHz	NSC: 80	PW: 0.00 usec, RG: 2050	SI: 32768
Date: 30-May-2022	Solvent: DMSO	SW: 26042 Hz	TE: 300 K	AQ: 0.98 sec, RD: 0.00 sec	

lv-23

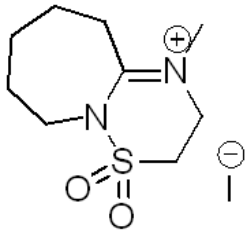


12c DMSO-d6



File name: lv-23	Operator: root	SF: 499.7730 MHz	NSC: 1	PW: 0.00 usec, RG: 22	SI: 32768
Date: 27-May-2022	Solvent: DMSO	SW: 10776 Hz	TE: 683 K	AQ: 1.52 sec, RD: 0.00 sec	

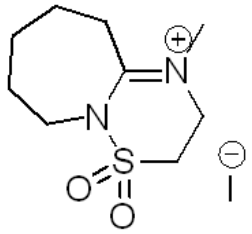
lv-23_C13



12c DMSO-d6

File name: lv-23_C13_APT	Operator: root	SF: 125.6681 MHz	NSC: 250	PW: 0.00 usec, RG: 51200	SI: 131072
Date: 27-May-2022	Solvent: DMSO	SW: 32680 Hz	TE: 683 K	AQ: 0.78 sec, RD: 0.00 sec	

lv-23_C13_APT

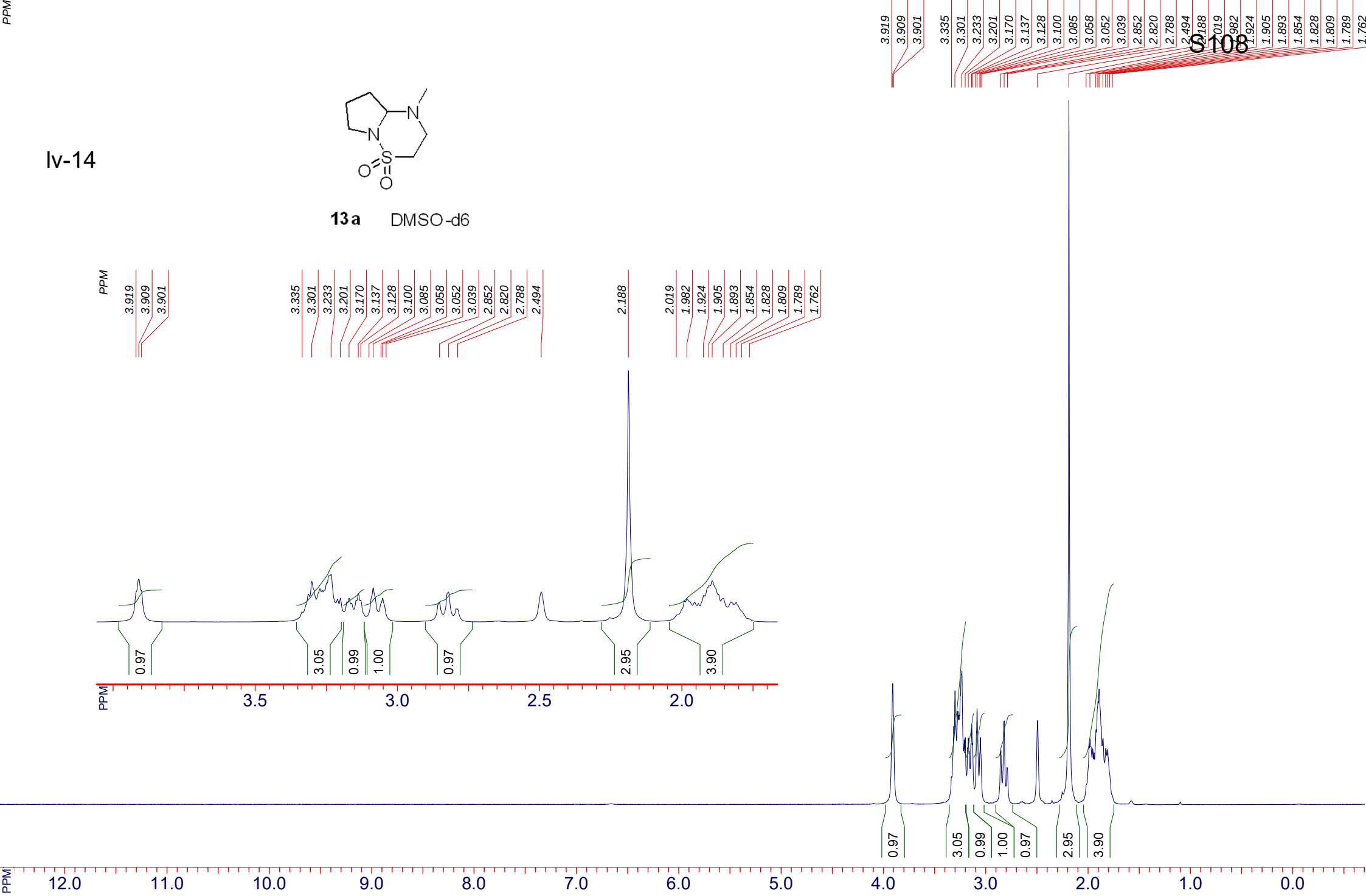


12c DMSO-d6

File name: lv-23_C13_APT	Operator: root	SF: 125.6681 MHz	NSC: 338	PW: 0.00 usec, RG: 51200	SI: 65536
Date: 27-May-2022	Solvent: DMSO	SW: 32680 Hz	TE: 683 K	AQ: 1.57 sec, RD: 0.00 sec	

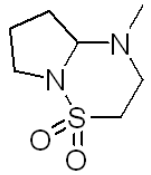
lv-14

13a DMSO-d6



File name: lv-14	Operator: nmr	SF: 400.1300 MHz	NSC: 1	PW: 0.00 usec, RG: 25	SI: 32768
Date: 26-May-2022	Solvent: DMSO	SW: 8224 Hz	TE: 300 K	AQ: 1.99 sec, RD: 0.00 sec	

lv-14_C13



13a DMSO-d6

81.16 53.49 46.12 45.69 40.03 38.84 30.33 21.50

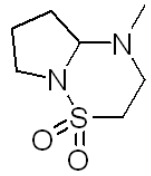
PPM

170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10

File name: lv-14_C13	Operator: nmr	SF: 100.6128 MHz	NSC: 214	PW: 0.00 usec, RG: 2050	SI: 32768
Date: 26-May-2022	Solvent: DMSO	SW: 26042 Hz	TE: 300 K	AQ: 0.98 sec, RD: 0.00 sec	

81.15
53.48
46.12
45.69
39.82
38.82
30.33
21.50

lv-14_C13APT



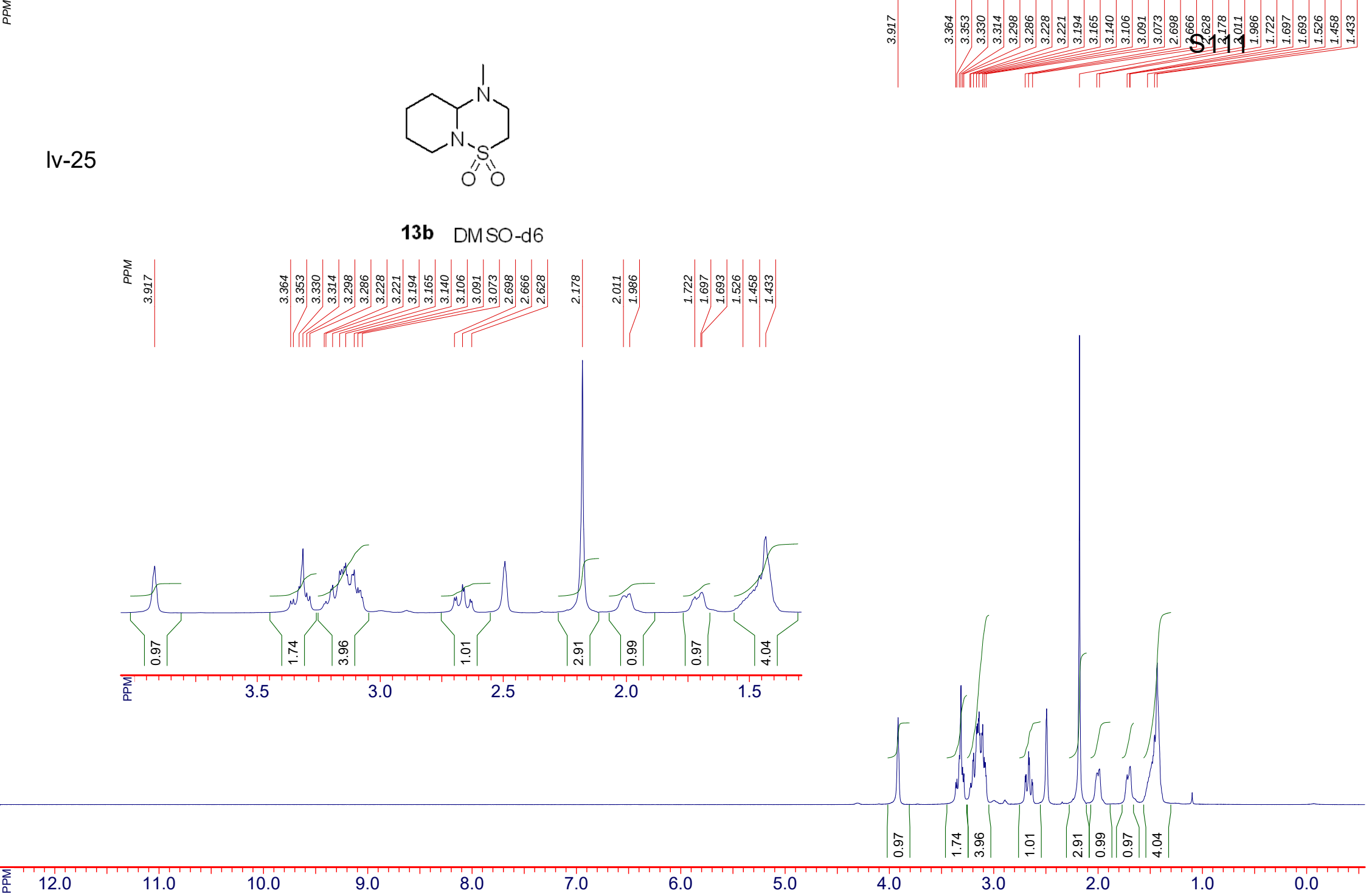
13a DMSO-d6

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

File name: lv-14_C13APT	Operator: nmr	SF: 100.6128 MHz	NSC: 280	PW: 0.00 usec, RG: 2050	SI: 65536
Date: 26-May-2022	Solvent: DMSO	SW: 24038 Hz	TE: 300 K	AQ: 1.06 sec, RD: 0.00 sec	

lv-25

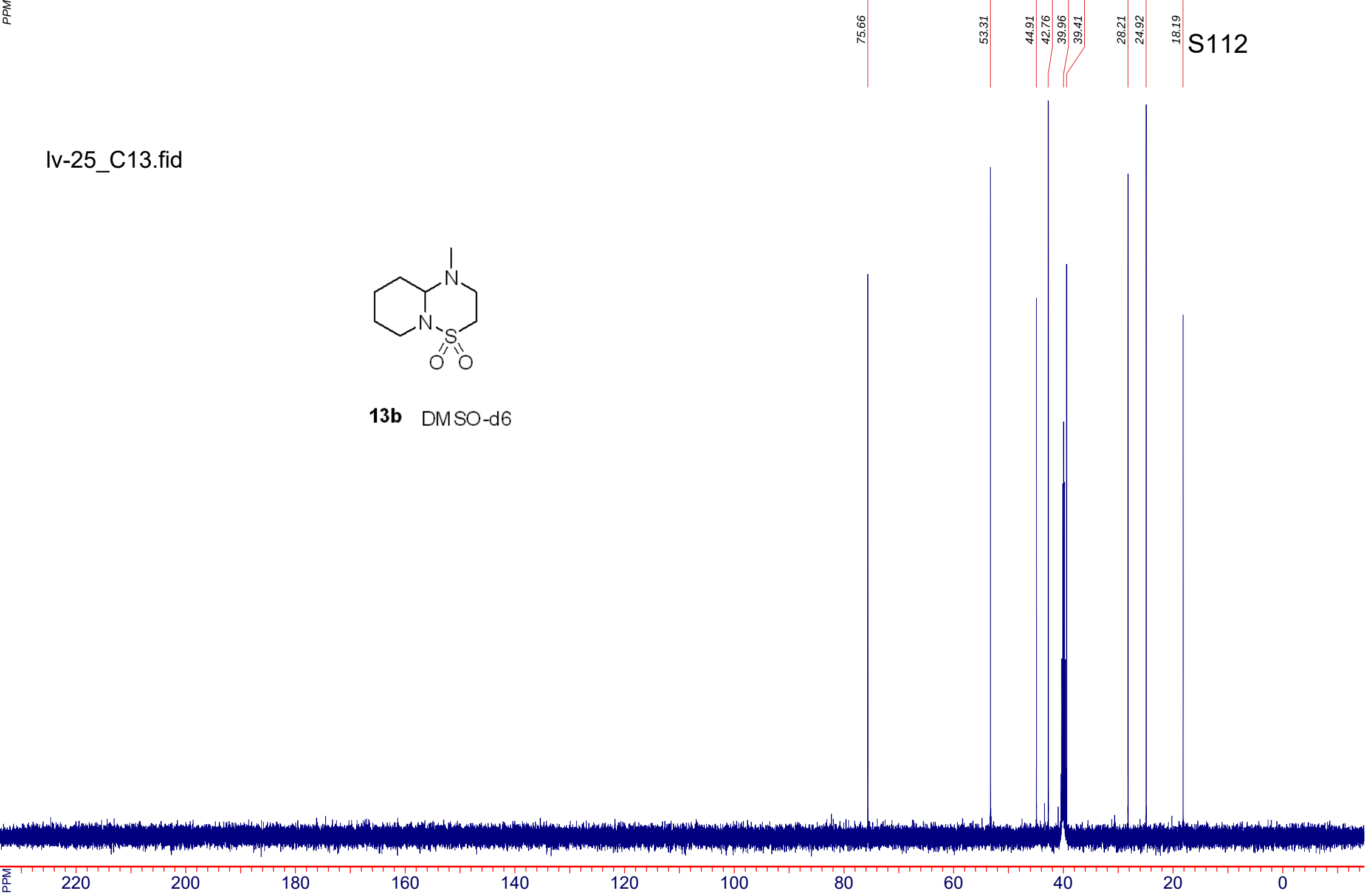
13b DMSO-d6



File name: lv-25	Operator: nmr	SF: 400.1300 MHz	NSC: 1	PW: 0.00 usec, RG: 25	SI: 32768
Date: 30-May-2022	Solvent: DMSO	SW: 8224 Hz	TE: 300 K	AQ: 1.99 sec, RD: 0.00 sec	

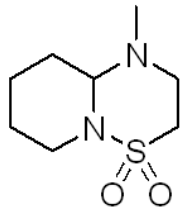
lv-25_C13.fid

13b DMSO-d6



File name: lv-25_C13.fid	Operator:	SF: 125.6926 MHz	NSC: 0	PW: 3.27 usec, RG: 60	SI: 65536
Date: 31-May-2022	Solvent: dmso	SW: 31250 Hz	TE: 298 K	AQ: 1.05 sec, RD: 0.00 sec	

lv-25_APTC13.fid



13b DMSO-d6

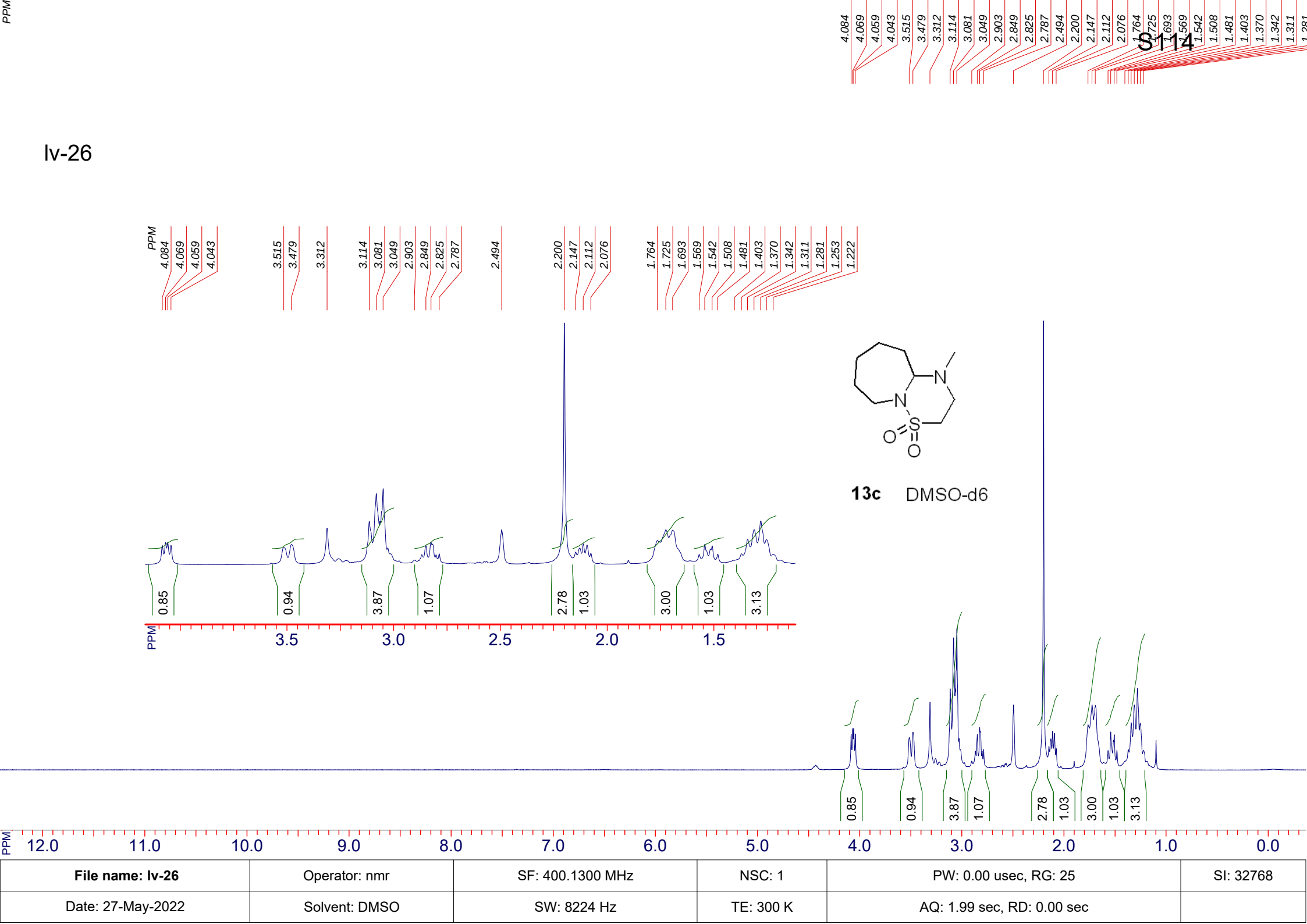
PPM

75.66 53.31 44.91 42.76 39.97 39.40 28.21 24.92 18.19

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

File name: lv-25_APTC13.fid	Operator:	SF: 125.6925 MHz	NSC: 0	PW: 5.00 usec, RG: 60	SI: 131072
Date: 31-May-2022	Solvent: dmso	SW: 32895 Hz	TE: 298 K	AQ: 1.95 sec, RD: 0.00 sec	Automated Probe tuning parameter

lv-26

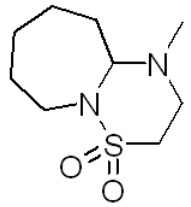


File name: lv-26	Operator: nmr	SF: 400.1300 MHz	NSC: 1	PW: 0.00 usec, RG: 25	SI: 32768
Date: 27-May-2022	Solvent: DMSO	SW: 8224 Hz	TE: 300 K	AQ: 1.99 sec, RD: 0.00 sec	

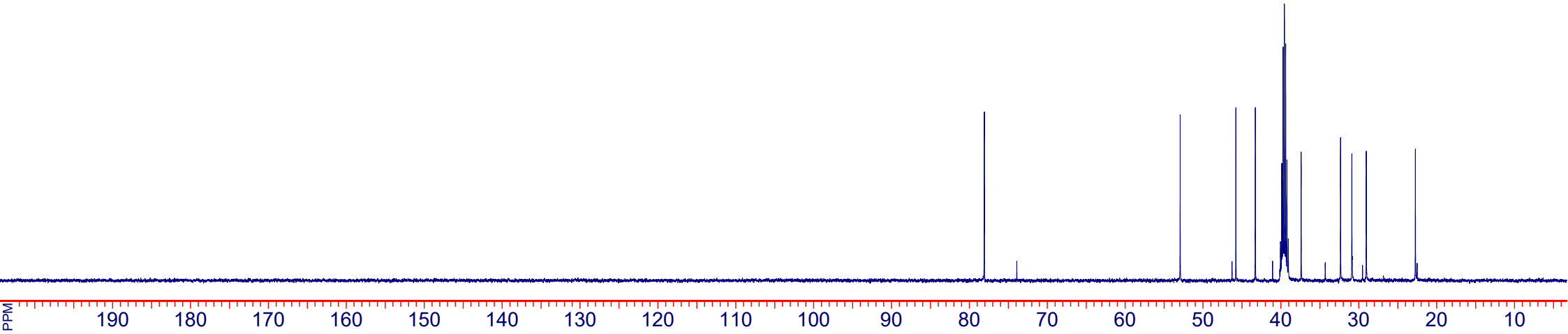
PPM

78.09 52.95 45.79 43.29 39.56 37.41 32.35 30.90 30.05 22.75

lv-26_C13



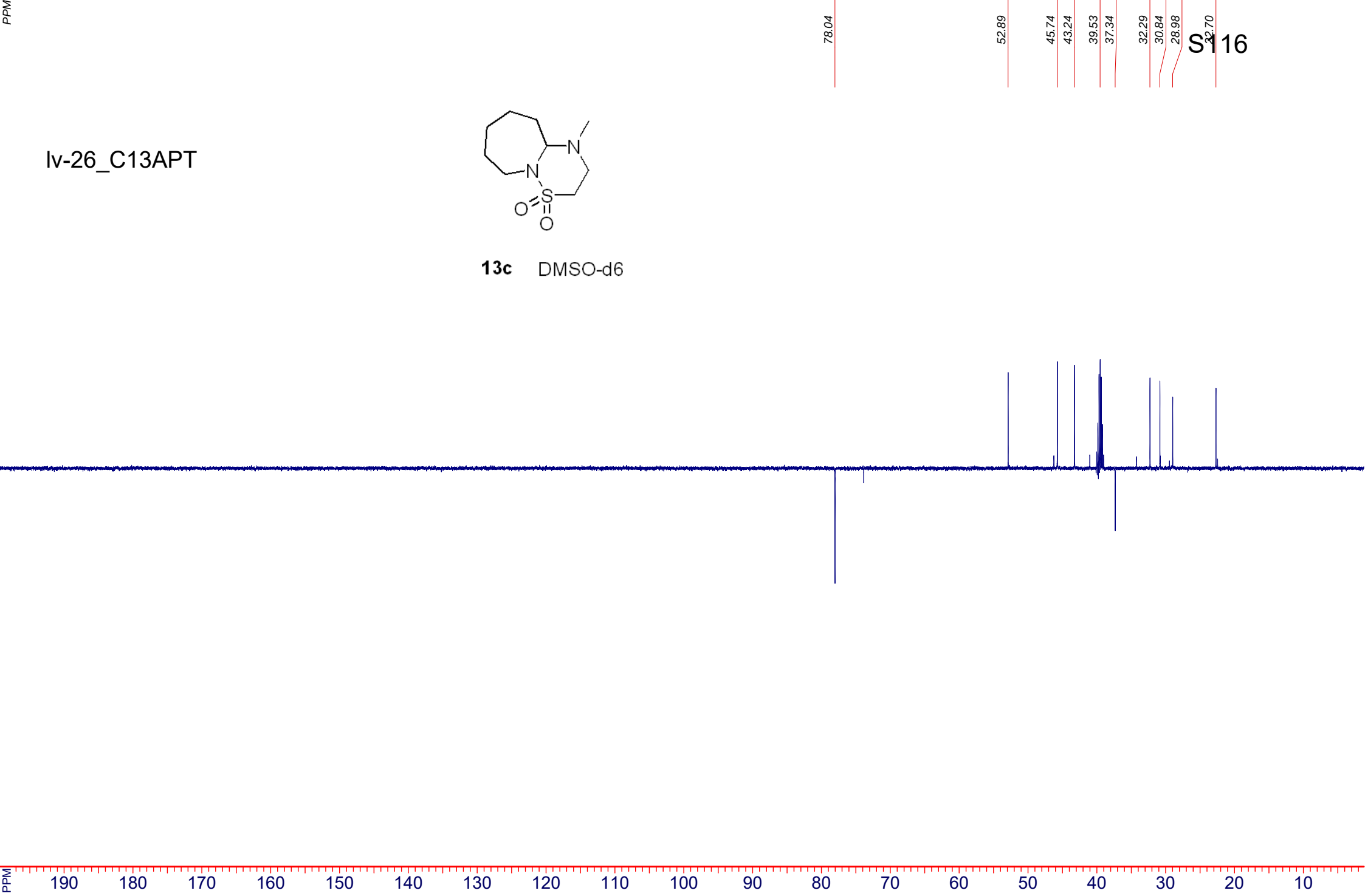
13c DMSO-d6



File name: lv-26_C13	Operator: root	SF: 125.6681 MHz	NSC: 400	PW: 0.00 usec, RG: 51200	SI: 131072
Date: 28-May-2022	Solvent: DMSO	SW: 32680 Hz	TE: 683 K	AQ: 0.78 sec, RD: 0.00 sec	

lv-26_C13APT

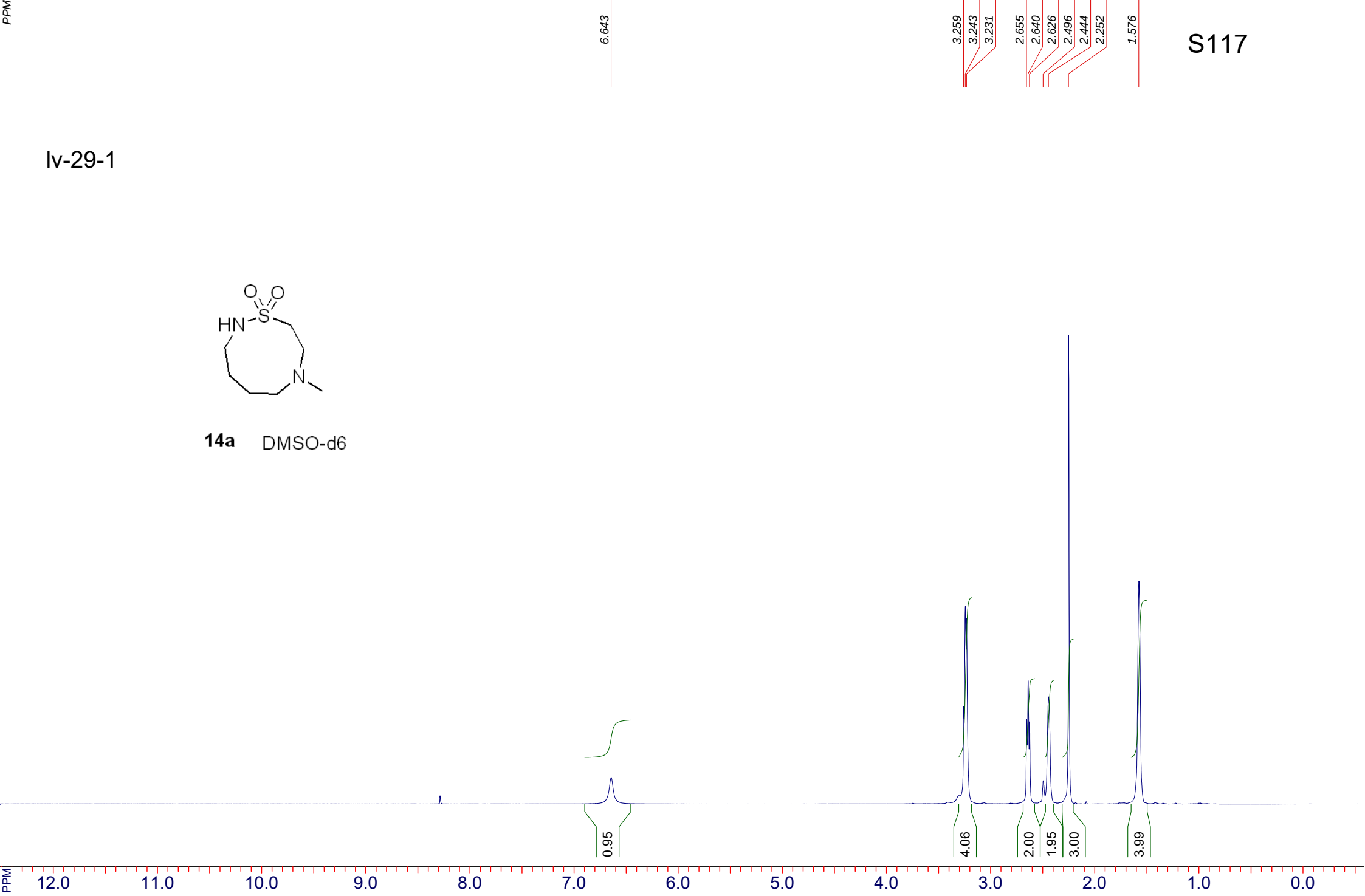
13c DMSO-d6



File name: lv-26_C13APT	Operator: root	SF: 125.6681 MHz	NSC: 250	PW: 0.00 usec, RG: 51200	SI: 65536
Date: 28-May-2022	Solvent: DMSO	SW: 32680 Hz	TE: 683 K	AQ: 1.00 sec, RD: 0.00 sec	

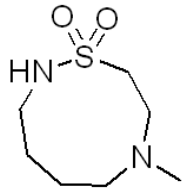
lv-29-1

14a DMSO-d6

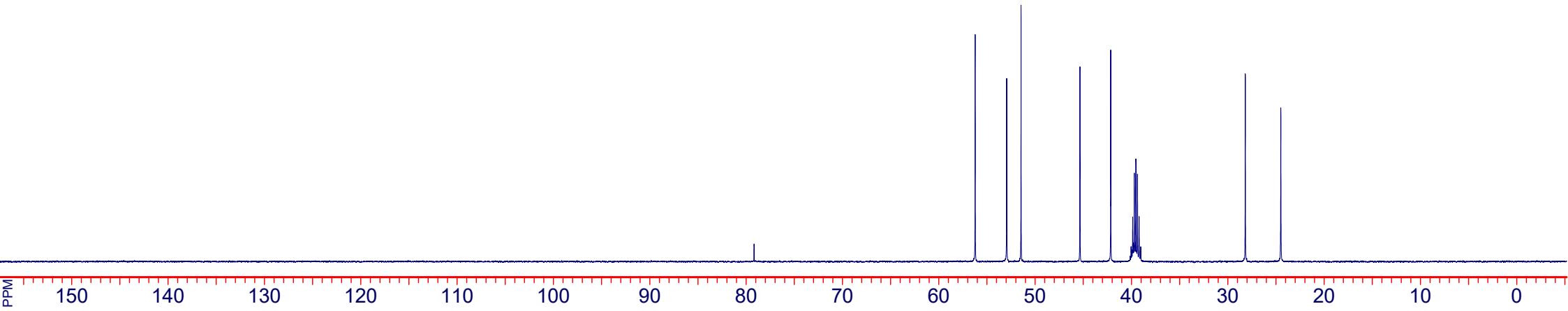


File name: lv-29-1	Operator: nmr	SF: 400.1300 MHz	NSC: 1	PW: 0.00 usec, RG: 25	SI: 32768
Date: 27-May-2022	Solvent: DMSO	SW: 8224 Hz	TE: 300 K	AQ: 1.99 sec, RD: 0.00 sec	

lv-29-1_C13



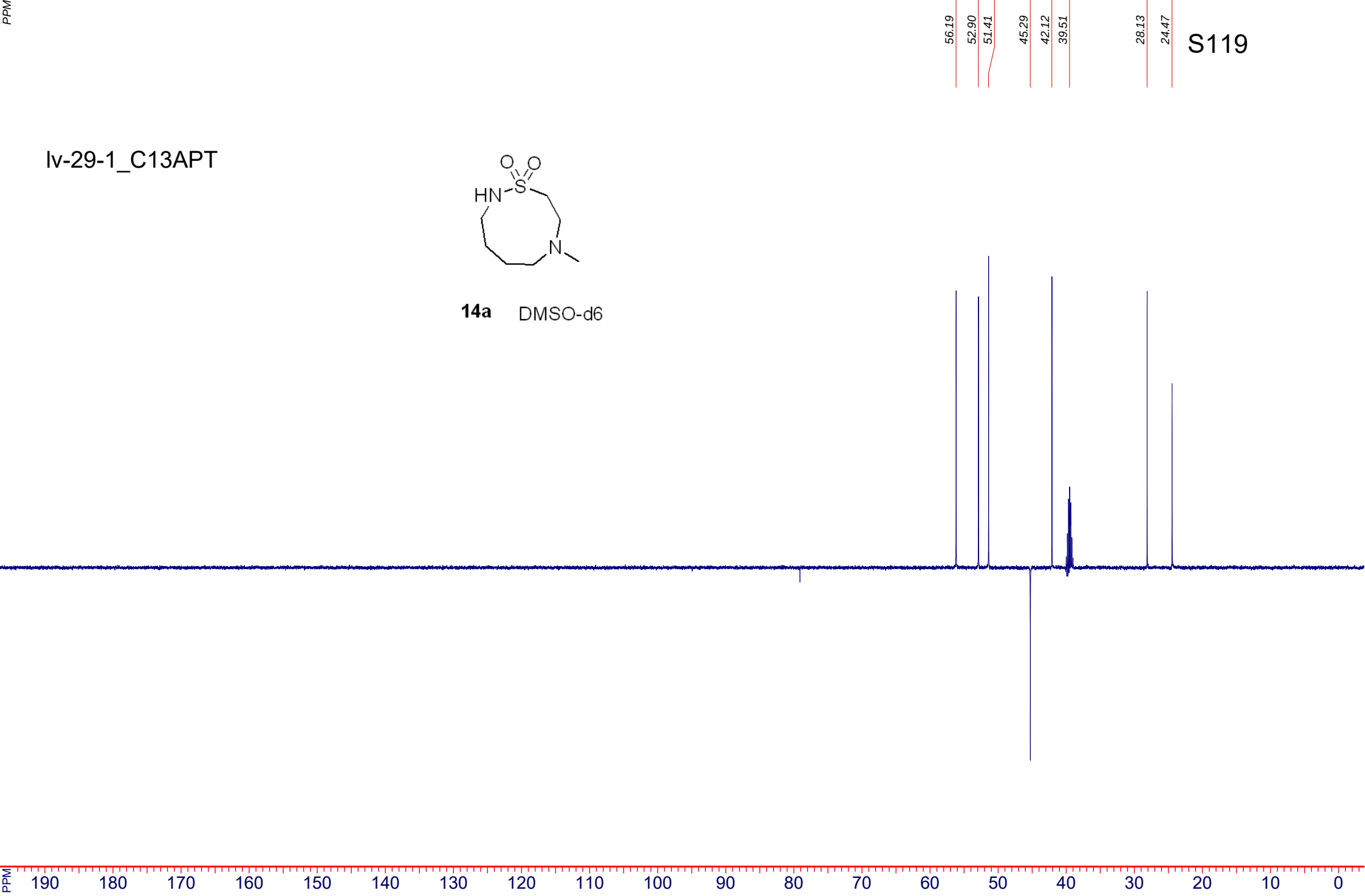
14a DMSO-d6



File name: lv-29-1_C13	Operator: root	SF: 125.6681 MHz	NSC: 400	PW: 0.00 usec, RG: 51200	SI: 131072
Date: 28-May-2022	Solvent: DMSO	SW: 32680 Hz	TE: 683 K	AQ: 0.78 sec, RD: 0.00 sec	

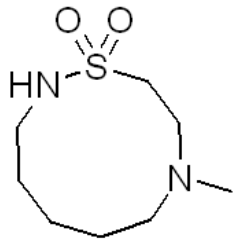
lv-29-1_C13APT

14a DMSO-d6

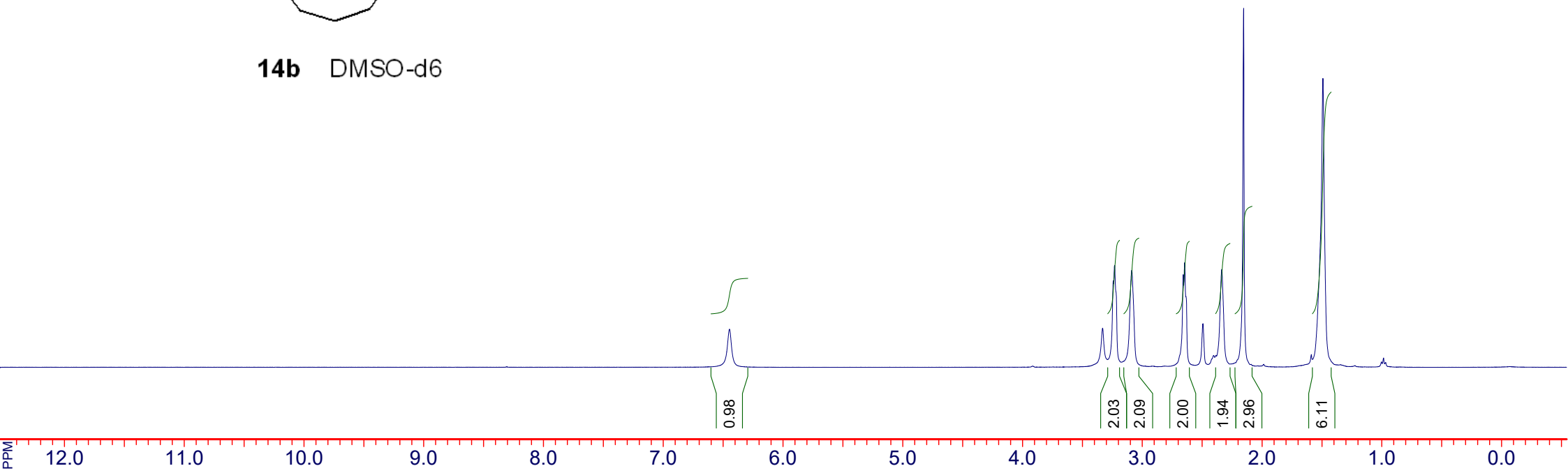


File name: lv-29-1_C13APT	Operator: root	SF: 125.6681 MHz	NSC: 300	PW: 0.00 usec, RG: 51200	SI: 65536
Date: 28-May-2022	Solvent: DMSO	SW: 32680 Hz	TE: 683 K	AQ: 1.00 sec, RD: 0.00 sec	

lv-30

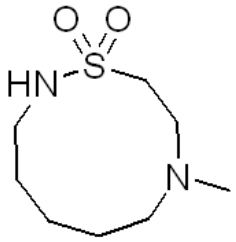


14b DMSO-d6

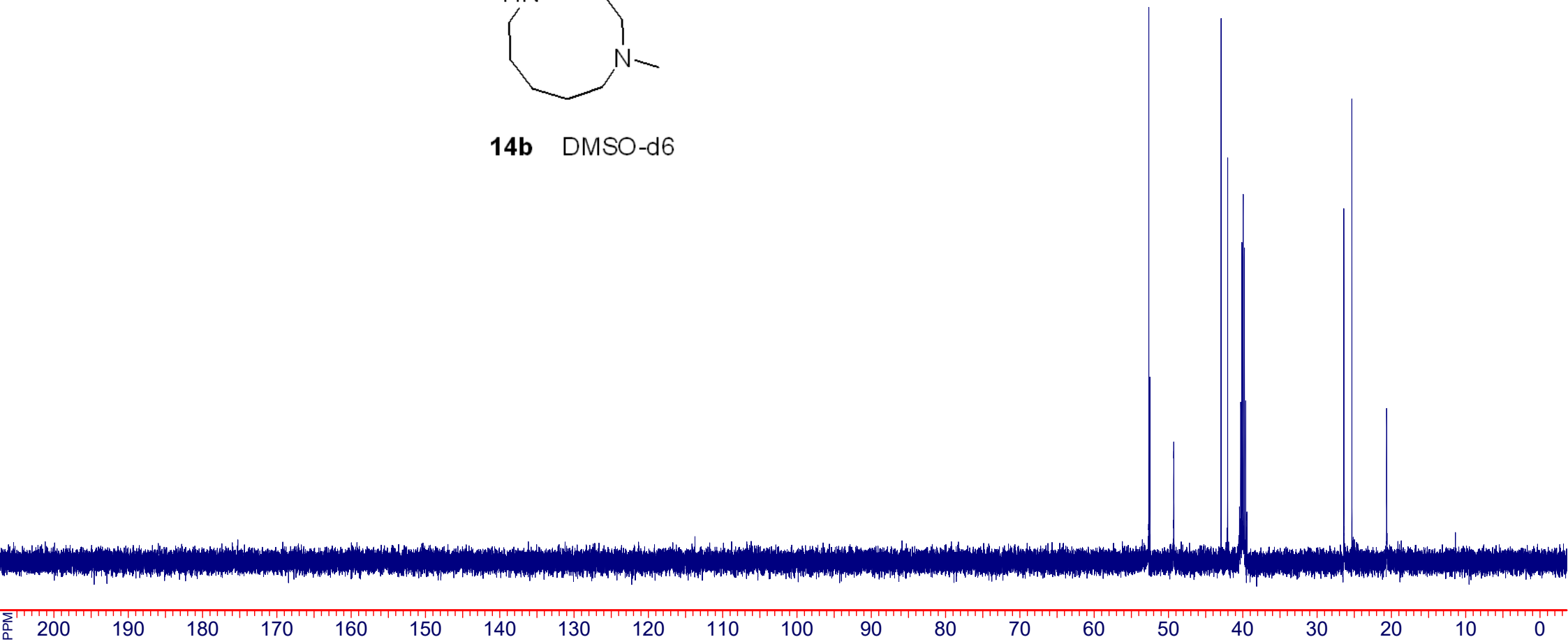


File name: lv-30	Operator: nmr	SF: 400.1300 MHz	NSC: 1	PW: 0.00 usec, RG: 25	SI: 32768
Date: 30-May-2022	Solvent: DMSO	SW: 8224 Hz	TE: 300 K	AQ: 1.99 sec, RD: 0.00 sec	

lv-30_C13.fid

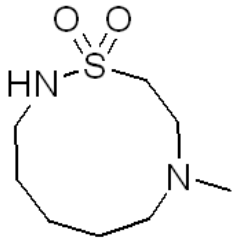


14b DMSO-d6



File name: lv-30_C13.fid	Operator:	SF: 125.6926 MHz	NSC: 0	PW: 3.27 usec, RG: 60	SI: 65536
Date: 31-May-2022	Solvent: dmso	SW: 31250 Hz	TE: 298 K	AQ: 1.05 sec, RD: 0.00 sec	

lv-30_APTC13.fid



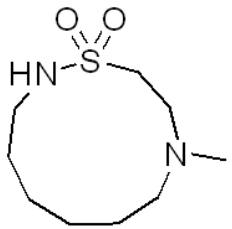
14b DMSO-d6

52.65 52.53 49.32 42.93 42.05 39.97 26.42 25.33 20.68

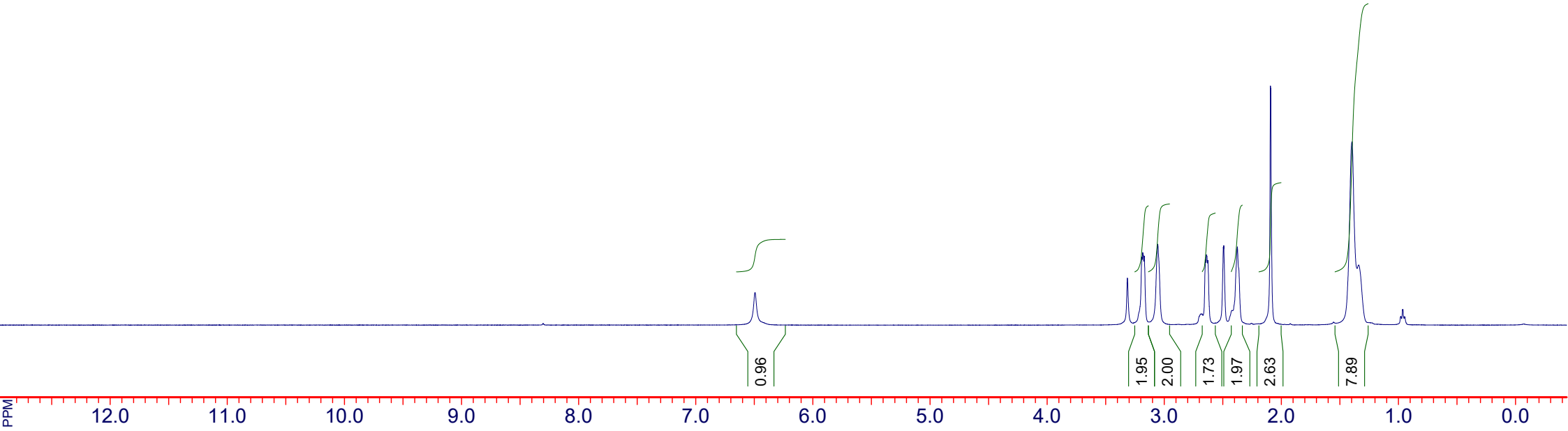
PPM 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10

File name: lv-30_APTC13.fid	Operator:	SF: 125.6925 MHz	NSC: 0	PW: 5.00 usec, RG: 60	SI: 131072
Date: 31-May-2022	Solvent: dmso	SW: 32895 Hz	TE: 298 K	AQ: 1.95 sec, RD: 0.00 sec	Automated Probe tuning parameter

lv-31

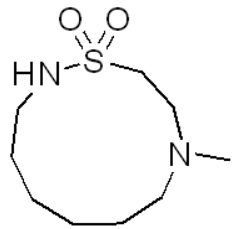


14c DMSO-d6

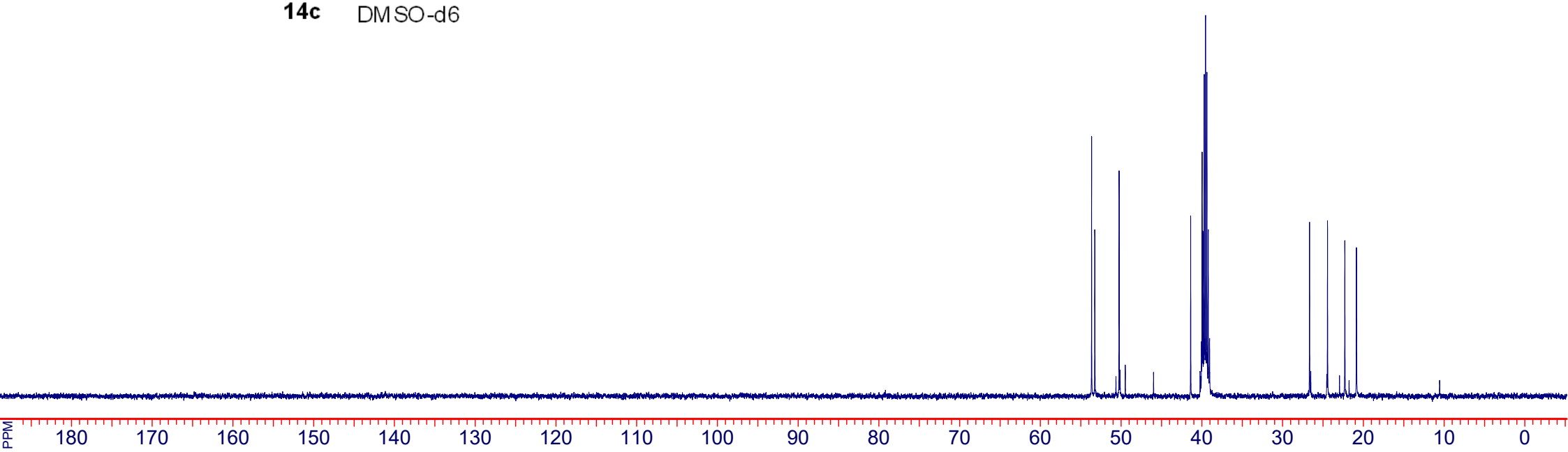


File name: lv-31	Operator: nmr	SF: 400.1300 MHz	NSC: 1	PW: 0.00 usec, RG: 25	SI: 32768
Date: 27-May-2022	Solvent: DMSO	SW: 8224 Hz	TE: 300 K	AQ: 1.99 sec, RD: 0.00 sec	

lv-31_C13

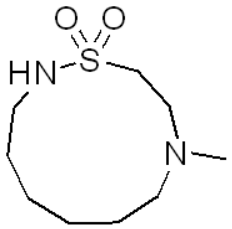


14c DMSO-d6

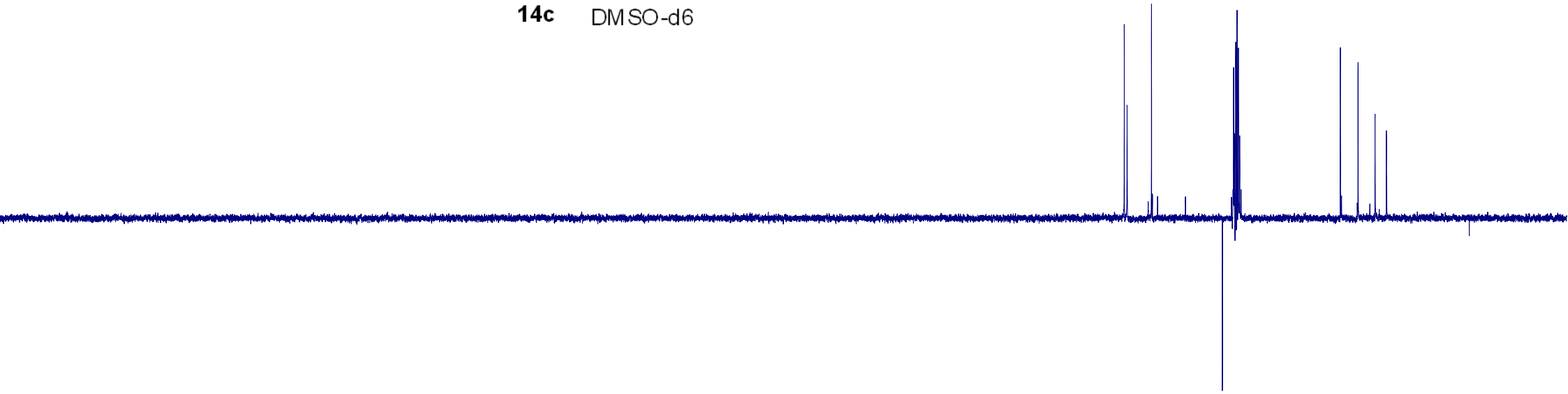


File name: lv-31_C13	Operator: root	SF: 125.6681 MHz	NSC: 291	PW: 0.00 usec, RG: 51200	SI: 131072
Date: 28-May-2022	Solvent: DMSO	SW: 32680 Hz	TE: 683 K	AQ: 0.78 sec, RD: 0.00 sec	

lv-31_C13APT

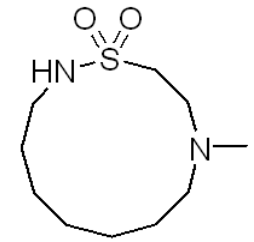


14c DMSO-d6

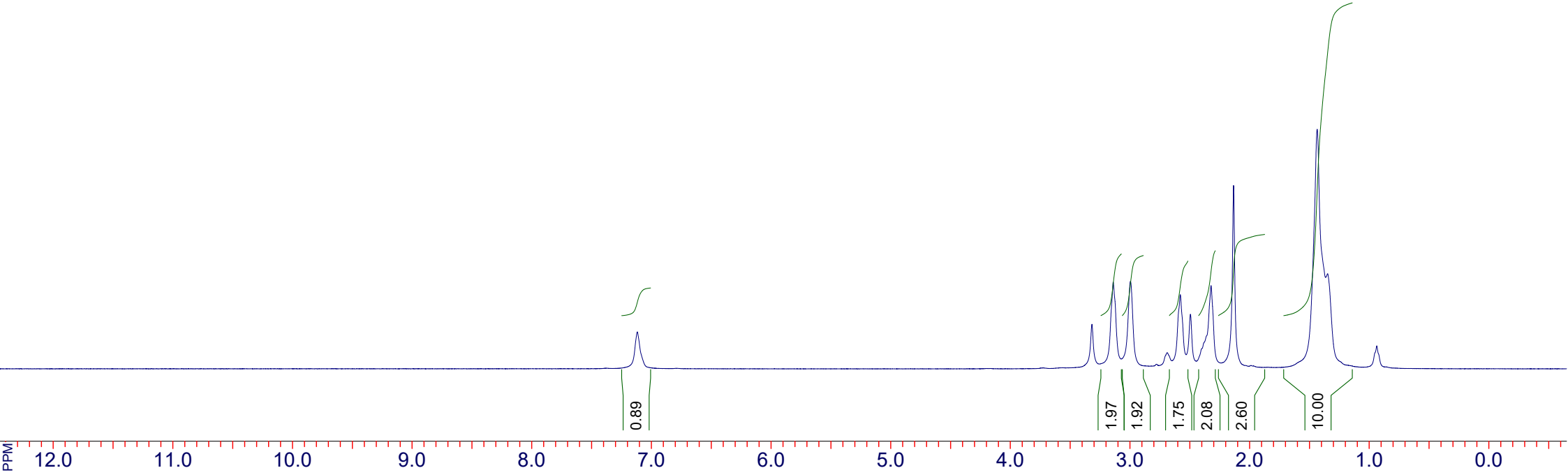


File name: lv-31_C13APT	Operator: root	SF: 125.6681 MHz	NSC: 267	PW: 0.00 usec, RG: 51200	SI: 65536
Date: 28-May-2022	Solvent: DMSO	SW: 32680 Hz	TE: 683 K	AQ: 1.00 sec, RD: 0.00 sec	

lv-35



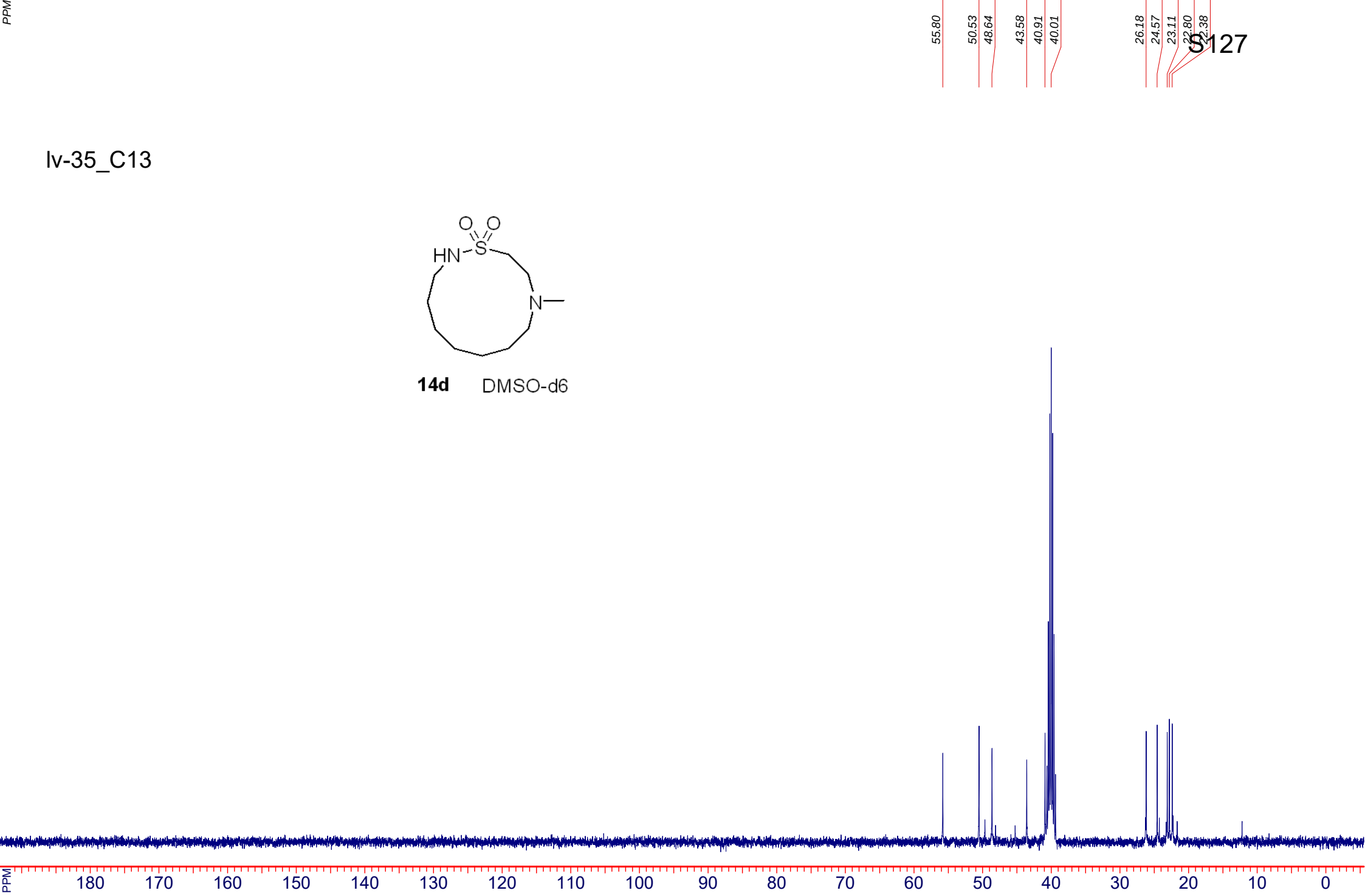
14d DMSO-d6



File name: lv-35_hexane	Operator: nmr	SF: 400.1300 MHz	NSC: 1	PW: 0.00 usec, RG: 25	SI: 32768
Date: 03-Jun-2022	Solvent: DMSO	SW: 8224 Hz	TE: 300 K	AQ: 1.99 sec, RD: 0.00 sec	Parameter file, TOPSPINVersion 2.1

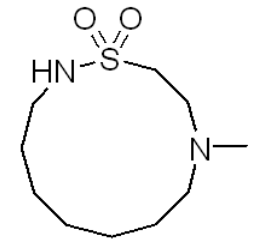
lv-35_C13

14d DMSO-d6

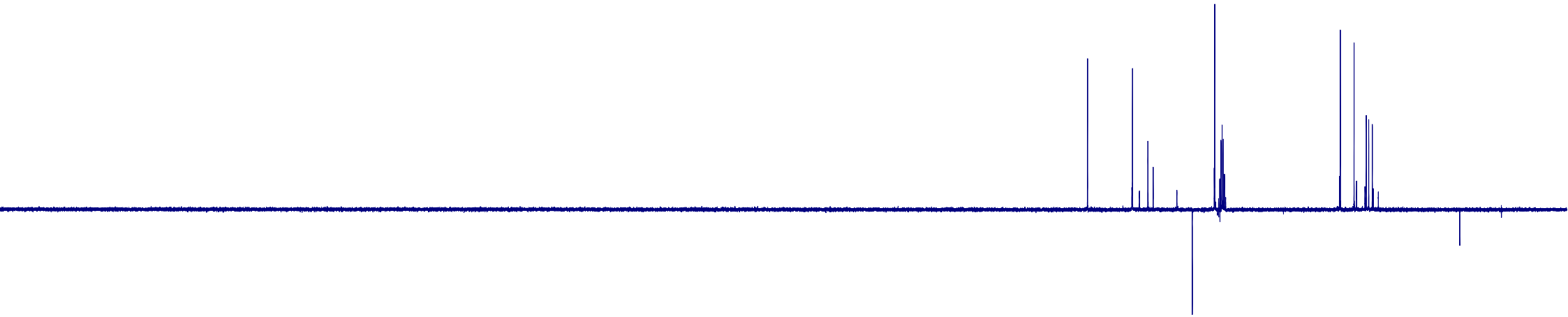


File name: lv-35_C13_hexane	Operator: nmr	SF: 100.6128 MHz	NSC: 297	PW: 0.00 usec, RG: 2050	SI: 32768
Date: 03-Jun-2022	Solvent: DMSO	SW: 26042 Hz	TE: 300 K	AQ: 0.98 sec, RD: 0.00 sec	Parameter file, TOPSPINVersion 2.1

lv-35_C13APT.fid

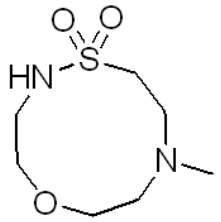


14d DMSO-d6

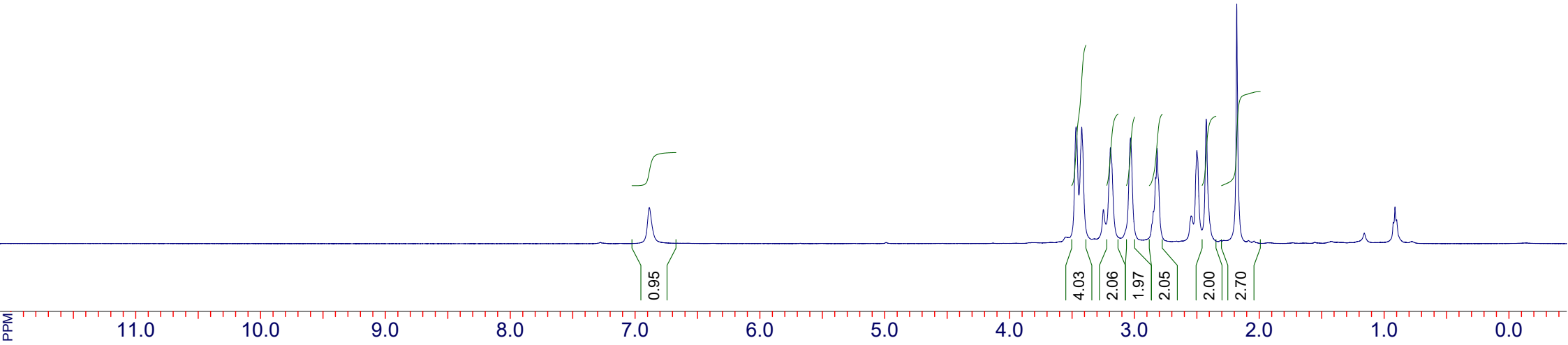


File name: lv-35_C13APT.fid	Operator:	SF: 150.8333 MHz	NSC: 0	PW: 4.63 usec, RG: 60	SI: 131072
Date: 02-Jun-2022	Solvent: dmso	SW: 39063 Hz	TE: 298 K	AQ: 0.87 sec, RD: 0.00 sec	

lv-32.fid

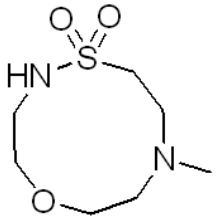


14e DMSO-d6

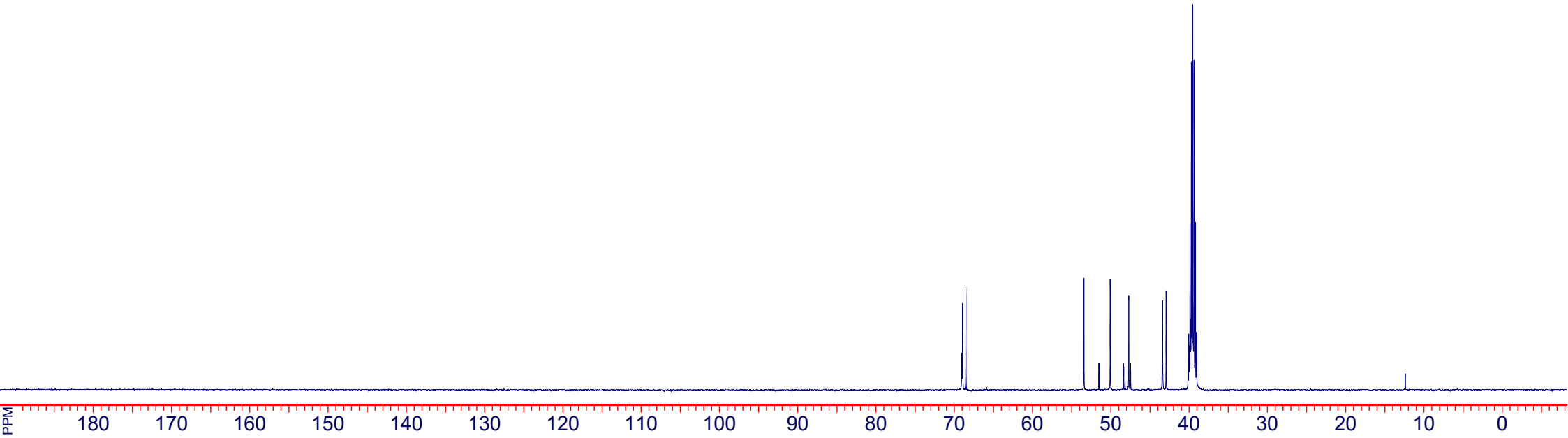


File name: lv-32.fid	Operator:	SF: 499.8203 MHz	NSC: 0	PW: 12.40 usec, RG: 12	SI: 32768
Date: 31-May-2022	Solvent: dmso	SW: 9328 Hz	TE: 298 K	AQ: 1.72 sec, RD: 0.00 sec	

lv-32_C13



14e DMSO-d6

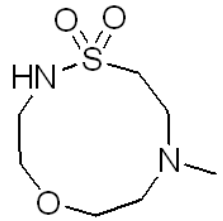


File name: lv-32_C13_C13APT	Operator: root	SF: 125.6681 MHz	NSC: 5120	PW: 0.00 usec, RG: 51200	SI: 131072
Date: 31-May-2022	Solvent: DMSO	SW: 32680 Hz	TE: 683 K	AQ: 0.78 sec, RD: 0.00 sec	

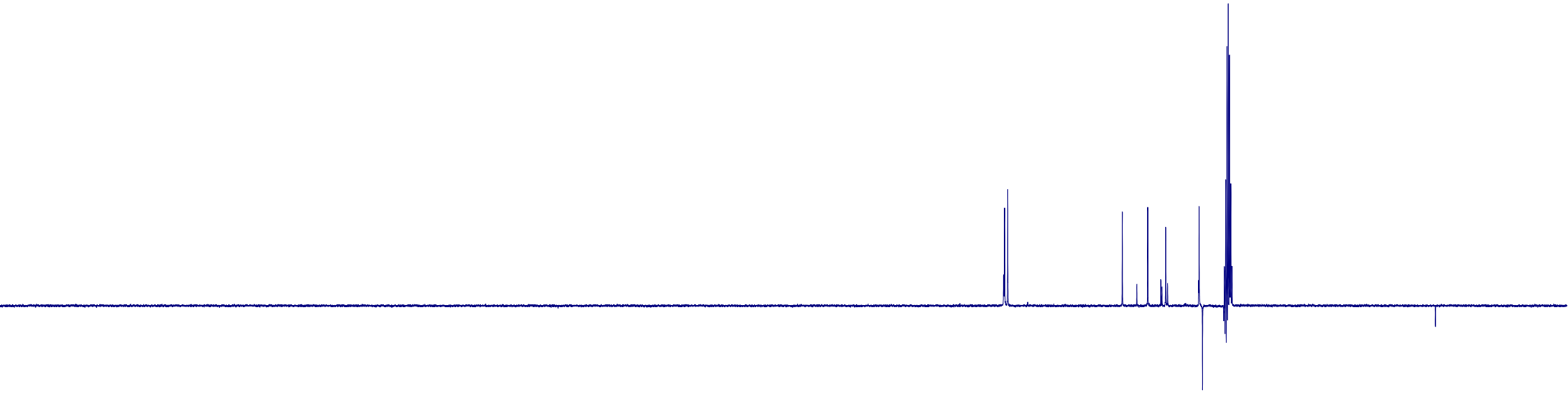
PPM

68.87
68.46
53.42
50.08
47.73
43.34
42.90
39.53

lv-32_C13_C13APT



14e DMSO-d6

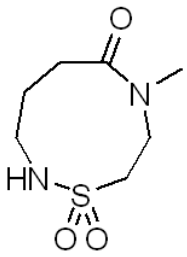


PPM

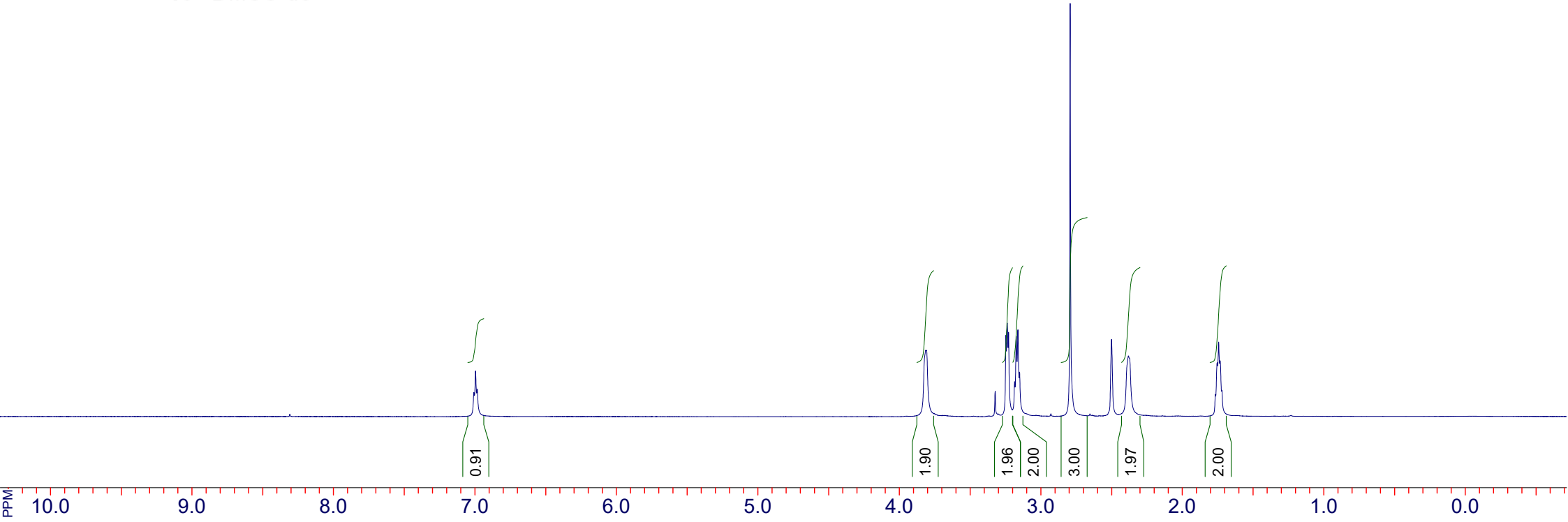
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0

File name: lv-32_C13_C13APT	Operator: root	SF: 125.6681 MHz	NSC: 5120	PW: 0.00 usec, RG: 51200	SI: 65536
Date: 01-Jun-2022	Solvent: DMSO	SW: 32680 Hz	TE: 683 K	AQ: 1.57 sec, RD: 0.00 sec	

lv-41.fid



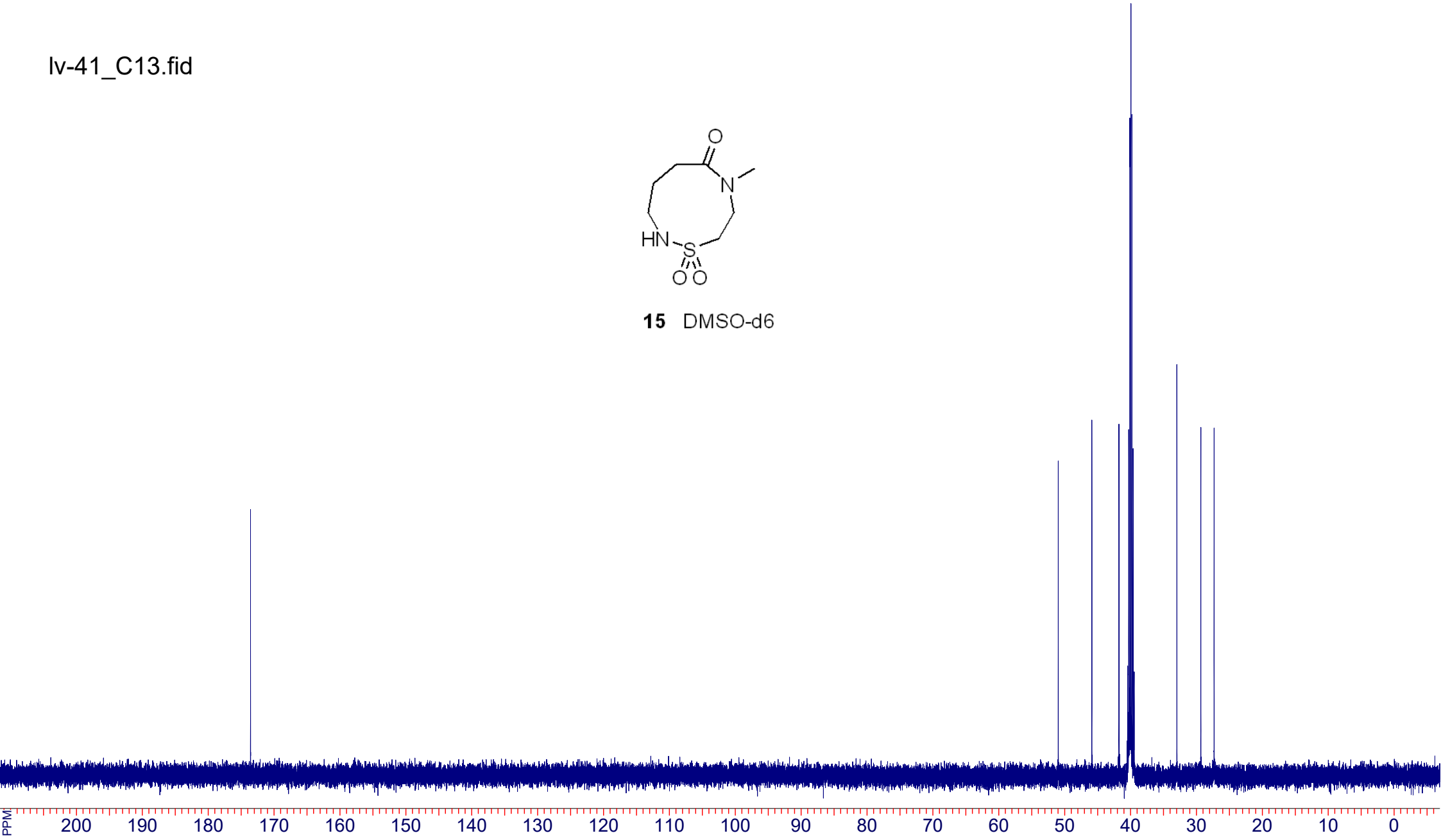
15 DMSO-d6



File name: lv-41.fid	Operator:	SF: 499.8203 MHz	NSC: 0	PW: 13.60 usec, RG: 12	SI: 32768
Date: 08-Jun-2022	Solvent: dmso	SW: 9328 Hz	TE: 298 K	AQ: 1.72 sec, RD: 0.00 sec	

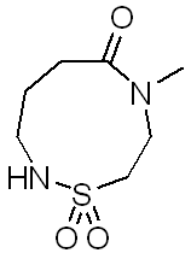
lv-41_C13.fid

15 DMSO-d6



File name: lv-41_C13.fid	Operator:	SF: 125.6926 MHz	NSC: 0	PW: 3.27 usec, RG: 60	SI: 65536
Date: 08-Jun-2022	Solvent: dmso	SW: 31250 Hz	TE: 298 K	AQ: 1.05 sec, RD: 0.00 sec	

lv-41_APT.fid



15 DMSO-d6

PPM 173.57 50.99 45.89 41.77 39.96 32.98 29.35 27.33

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

File name: lv-41_APT.fid	Operator:	SF: 125.6925 MHz	NSC: 0	PW: 5.00 usec, RG: 60	SI: 131072
Date: 08-Jun-2022	Solvent: dmso	SW: 32895 Hz	TE: 298 K	AQ: 1.95 sec, RD: 0.00 sec	Automated Probe tuning parameter