Supplementary Material

Hypervalent iodine-catalyzed conjugate addition with sulfonamides

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1. General Information

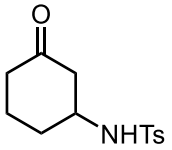
Commercial reagents and solvents were purchased from Sigma Aldrich, Oakwood Chemicals, Alfa Aesar, Matrix Scientific, Acros Organic and were used as received. Organic solutions were concentrated under reduced pressure on an IKA rotary evaporator using an acetone-dry ice bath. Chromatographic purification of products was accomplished using flash chromatography on 230-400 mesh silica gel. Thin-layer chromatography (TLC) was performed on Analtech 250 mm silica gel HLF UV-250 plates. Visualization of the developed plates was performed by fluorescent quenching and/or potassium permanganate. ^{1}H and ^{13}C NMR spectra were recorded on a Bruker instrument (600 and 150 MHz) or INOVA 600 (600 and 150 MHz) and are internally referenced to residual protio solvent signals (for $CDCl_3$, 7.27 and 77.0 ppm, respectively). Data for ^{1}H NMR are reported as follows: chemicals shift (ppm), multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, h = heptet, m = multiplet, br = broad), integration, coupling constant (Hz). ^{13}C spectra were reported as chemical shifts in ppm and multiplicity where appropriate.

2. Experimental Procedures

General Procedure for hypervalent iodine-catalyzed conjugate addition reaction

To an 8 mL vial equipped with a stir bar was added 4-iodotoluene (5 mg, 0.02 mmol), Selectfluor (14 mg, 0.04 mmol) and sulfonamide (0.2 mmol). Then solvent (MeCN, 0.4 mL) was added via a syringe, followed by enone (0.4 mmol). The reaction mixture was then stirred 16 h at room temperature. The reaction mixture was concentrated under reduced pressure to give the crude product, which was purified by column chromatography on silica gel to afford the pure product.

3. Spectral Characterization of Products



4-Methyl-N-(3-oxocyclohexyl)benzenesulfonamide (3)

This compound was prepared according to the General Procedure using, 4-methylbenzenesulfonamide (34 mg, 0.2 mmol) and cyclohex-2-en-1-one (38 µL, 0.4 mmol). After purification by column chromatography SiO_2 (30% to 50% EtOAc/hexanes), The title compound was isolated as a white solid (45 mg, 84% yield).

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.75 (d, *J* = 9.0 Hz, 2 H), 7.30 (d, *J* = 9.0 Hz, 2 H), 5.44 (d, *J* = 9.0 Hz, 1 H), 3.56-3.46 (m, 1 H), 2.49 (dd, *J* = 12.0, 4.5 Hz, 1 H), 2.43 (s, 3 H), 2.34-2.26 (m, 1 H), 2.25-2.20 (m, 2 H), 2.02-1.91 (m, 2 H), 1.69-1.52 (m, 2 H); ^{13}C NMR (150 MHz, $CDCl_3$) ??208.2, 143.7, 137.5, 129.8, 126.9, 52.4, 48.4, 40.5, 31.8, 21.6, 21.5; IR (Neat): 3225.2, 2953.4, 1700.7, 1451.6, 1326.3, 1150.1 1086.5 cm^{-1}.

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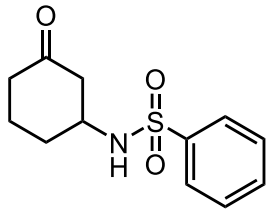
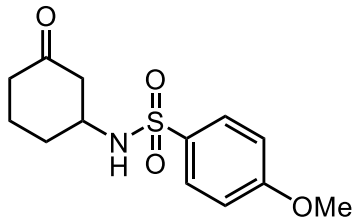


N-(3-oxocyclohexyl)benzenesulfonamide (4)

This compound was prepared according to the General Procedure using, benzenesulfonamide (31 mg, 0.2 mmol) and cyclohex-2-en-1-one (38 µL, 0.4 mmol). After purification by column chromatography SiO_2 (30% to 50% EtOAc/hexanes), The title compound was isolated as a white solid (39 mg, 77% yield).

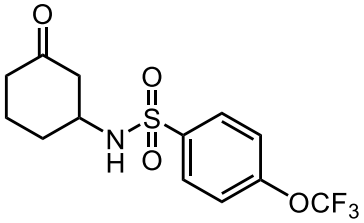
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.88 (d, *J* = 7.6 Hz, 2 H), 7.60 (t, *J* = 9.6 Hz, 1 H), 7.53 (t, *J* = 7.7 Hz, 2 H), 5.36 (d, *J* = 7.3 Hz, 1 H), 3.61-3.52 (m, 1 H), 2.50 (dd, *J* = 14.7, 4.8 Hz, 1 H), 2.34-2.28 (m, 1 H), 2.26-2.18 (m, 2 H), 2.03-1.93 (m, 2 H), 1.70-1.55 (m, 2 H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 208.1, 140.5, 132.9, 129.2, 126.8, 52.4, 48.4, 40.5, 31.8, 21.6; IR (Neat): 3256.2, 2946.4, 1709.7, 1452.7, 1321.5, 1152.6, 1064.7 cm^{-1}.



4-Methoxy-N-(3-oxocyclohexyl)benzenesulfonamide (5)

This compound was prepared according to the General Procedure using, 4-methoxybenzenesulfonamide (37 mg, 0.2 mmol) and cyclohex-2-en-1-one (38 µL, 0.4 mmol). After purification by column chromatography SiO_2 (30% to 50% EtOAc/hexanes), The title compound was isolated as a white solid (36 mg, 64% yield).

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.81 (d, *J* = 8.7 Hz, 2 H), 6.98 (m, *J* = 8.7 Hz, 2 H), 5.18 (d, *J* = 7.3 Hz, 1 H), 3.88 (s, 3 H), 3.56-3.47 (m, 1 H), 2.50 (dd, *J* = 14.4, 4.5 Hz, 1 H), 2.34-2.27 (m, 1 H), 2.26-2.17 (m, 2 H), 2.03-1.92 (m, 2 H), 1.70-1.55 (m, 2 H); ^{13}C NMR (150 MHz, $CDCl_3$) 208.2, 163.0, 132.0, 129.1, 114.4, 55.6, 52.4, 48.4, 40.5, 31.8, 21.7; IR (Neat): 3183.0, 2960.1, 1698.5, 1594.2, 1498.9, 1459.0, 1258.9, 1151.3, 1089.9 cm^{-1}.



N-(3-oxocyclohexyl)-4-(trifluoromethoxy)benzenesulfonamide (6)

This compound was prepared according to the General Procedure using, 4-(trifluoromethoxy)benzenesulfonamide (48 mg, 0.2 mmol) and cyclohex-2-en-1-one (38 µL, 0.4 mmol). After purification by column chromatography SiO_2 (30% to 50% EtOAc/hexanes), The title compound was isolated as a colorless oil (45 mg, 67% yield).

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.93 (d, *J* = 8.8 Hz, 2 H), 7.35 (d, *J* = 8.8 Hz, 2 H), 5.47 (d, *J* = 7.8 Hz, 1 H), 3.66-3.56 (m, 1 H), 2.55 (dd, *J* = 14.4, 4.8 Hz, 1 H), 2.37-2.30 (m, 1 H), 2.30-2.20 (m, 2 H), 2.05-1.94 (m, 2 H), 1.74-1.59 (m, 2 H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 208.2, 152.2, 138.9, 129.1, 121.0, 120.2 (q J_{C-F} = 255 Hz) , 52.6, 48.3, 40.5, 31.7, 21.5; IR (Neat): 3275.0, 2960.5, 1704.7, 1588.8, 1491.8, 1252.3, 1207.6, 1151.9, 1090.1 cm^{-1}.

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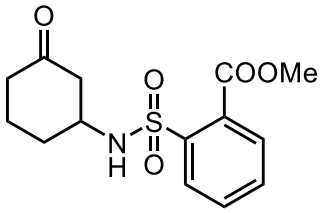
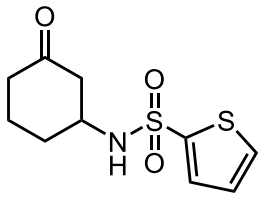


Methyl 2-(N-(3-oxocyclohexyl)sulfamoyl)benzoate (7)

This compound was prepared according to the General Procedure using, methyl 2-sulfamoylbenzoate (43 mg, 0.2 mmol) and cyclohex-2-en-1-one (38 µL, 0.4 mmol). After purification by column chromatography SiO_2 (30% to 50% EtOAc/hexanes), The title compound was isolated as a colorless oil (38 mg, 61% yield).

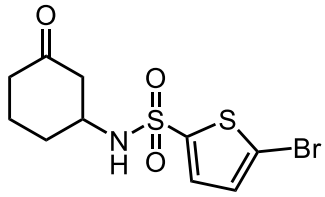
^{1}H NMR (600 MHz, $CDCl_3$) δ 8.13-8.08 (m, 1 H), 7.88-7.84 (m, 1 H), 7.69-7.62 (m, 2 H), 6.23 (d, *J* = 6.8 Hz, 1 H), 3.98 (s, 3 H), 3.68-3.60 (m, 1 H), 2.48 (dd, *J* = 14.4, 4.5 Hz, 1 H), 2.35-2.28 (m, 1 H), 2.28-2.18 (m, 2 H), 2.07-1.95 (m, 2 H), 1.73-1.56 (m, 2 H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 207.8, 168.1, 140.0, 132.5, 132.0, 131.0, 130.0, 129.4, 53.4, 52.9, 48.3, 40.5, 31.9, 21.7; IR (Neat): 3287.0, 2955.8, 1710.1, 1435.0, 1276.6, 1161.0, 1058.9 cm^{-1}.



N-(3-oxocyclohexyl)thiophene-2-sulfonamide (8)

This compound was prepared according to the General Procedure using, thiophene-2-sulfonamide (33 mg, 0.2 mmol) and cyclohex-2-en-1-one (38 µL, 0.4 mmol). After purification by column chromatography SiO_2 (30% to 50% EtOAc/hexanes), The title compound was isolated as a white solid (38 mg, 73% yield).

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.62 (dd, *J* = 12.9, 4.8 Hz, 2 H), 7.10 (t, *J* = 4.3 Hz, 1 H), 5.36 (d, *J* = 7.1 Hz, 1 H), 3.70-3.60 (m, 1 H), 2.57 (dd, *J* = 14.2, 4.4 Hz, 1 H), 2.37-2.31 (m, 1 H), 2.31-2.21 (m, 2 H), 2.09-1.96 (m, 2 H), 1.75-1.59 (m, 2 H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 208.1, 141.4, 132.3, 132.2, 127.5, 52.8, 48.3, 40.5, 31.7, 21.6; IR (Neat): 3239.5, 3095.3, 2955.4, 1708.0, 1455.2, 1321.5, 1143.0, 1068.1 cm^{-1}.



5-Bromo-N-(3-oxocyclohexyl)thiophene-2-sulfonamide (9)

This compound was prepared according to the General Procedure using, 5-bromothiophene-2-sulfonamide (48 mg, 0.2 mmol) and cyclohex-2-en-1-one (38 µL, 0.4 mmol). After purification by column chromatography SiO_2 (30% to 50% EtOAc/hexanes), The title compound was isolated as a white solid (55 mg, 81% yield).

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.38 (d, *J* = 3.9 Hz, 1 H), 7.07 (d, *J* = 3.7 Hz, 1 H), 5.28 (d, *J* = 7.1 Hz, 1 H), 3.70-3.61 (m, 1 H), 2.62 (dd, *J* = 14.4, 4.8 Hz, 1 H), 2.42-2.33 (m, 1 H), 2.32-2.23 (m, 2 H), 2.11-1.98 (m, 2 H), 1.77-1.64 (m, 2 H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 208.0, 142.2, 132.5, 130.5, 120.2, 52.8, 48.3, 40.5, 31.8, 21.6; IR (Neat): 3156.2, 2958.2, 1690.8, 1465.8, 1405.6, 1326.3, 1152.8, 1075.0 cm^{-1}.

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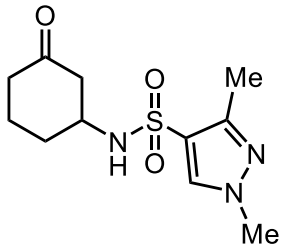
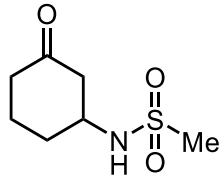


1,3-Dimethyl-N-(3-oxocyclohexyl)-1H-pyrazole-4-sulfonamide (10)

This compound was prepared according to the General Procedure using, 1,3-dimethyl-1H-pyrazole-4-sulfonamide (35 mg, 0.2 mmol) and cyclohex-2-en-1-one (38 µL, 0.4 mmol). After purification by column chromatography SiO_2 (70% to 100% EtOAc/hexanes), The title compound was isolated as a white solid (37 mg, 68% yield).

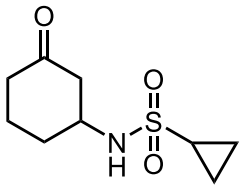
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.75 (s, 1 H), 4.67 (d, *J* = 7.3 Hz, 1 H), 3.87 (s, 3 H), 3.62-3.52 (m, 1 H), 2.61 (dd, *J* = 14.0, 4.5 Hz, 1 H), 2.39 (s, 3 H), 2.37-2.32 (m, 1 H), 2.30-2.21 (m, 2 H), 2.15-2.06 (m, 1 H), 2.05-1.97 (m, 1 H), 1.75-1.62 (m, 2 H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 207.9, 147.4, 134.0, 119.5, 52.3, 48.6, 40.6, 39.3, 32.1, 21.8, 12.3; IR (Neat): 3258.5, 3124.8, 2939.3, 1707.0, 1529.8, 1454.0, 1309.9, 1157.8, 1099.25 cm^{-1}.



N-(3-oxocyclohexyl)methanesulfonamide (11)

This compound was prepared according to the General Procedure using, methanesulfonamide (19 mg, 0.2 mmol) and cyclohex-2-en-1-one (38 µL, 0.4 mmol). After purification by column chromatography SiO_2 (50% to 70% EtOAc/hexanes), The title compound was isolated as a white solid (24 mg, 63% yield).

^{1}H NMR (600 MHz, $CDCl_3$) δ 4.77 (d, *J* = 7.1 Hz, 1 H), 3.85-3.77 (m, 1 H), 3.00 (s, 3 H), 2.75 (dd, *J* = 14.4, 4.8 Hz, 1 H), 2.43-2.35 (m, 2 H), 2.33-2.26 (m, 1 H), 2.22-2.15 (m, 1 H), 2.10-2.01 (m, 1 H), 1.80-1.71 (m, 2 H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 208.0, 52.7, 48.8, 42.1, 40.6, 32.2, 21.7; IR (Neat): 3259.5, 3007.8, 2955.8, 1703.4, 1451.0, 1306.4, 1143.9, 1082.6 cm^{-1}.



N-(3-oxocyclohexyl)cyclopropanesulfonamide (12)

This compound was prepared according to the General Procedure using, cyclopropanesulfonamide (24 mg, 0.2 mmol) and cyclohex-2-en-1-one (38 µL, 0.4 mmol). After purification by column chromatography SiO_2 (50% to 70% EtOAc/hexanes), The title compound was isolated as a white solid (27 mg, 62% yield).

^{1}H NMR (600 MHz, $CDCl_3$) δ 4.80 (d, *J* = 7.8 Hz, 1 H), 3.81-3.74 (m, 1 H), 2.77 (dd, *J* = 14.4, 4.8 Hz 1 H), 2.47-2.42 (m, 1 H), 2.41-2.32 (m, 2 H), 2.33-2.25 (m, 1 H), 2.24-2.17 (m, 1 H), 2.08-2.01 (m, 1 H), 1.80-1.67 (m, 2 H), 1.21-1.12 (m, 2 H), 1.08-0.98 (m, 2 H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 208.2, 52.6, 49.2, 40.6, 32.5, 31.4, 21.7, 5.9, 5.6; IR (Neat): 3243.6, 2958.9, 2883.2, 1706.2, 1455.7, 1317.6, 1138.8, 1149.8, 1071.1 cm^{-1}.

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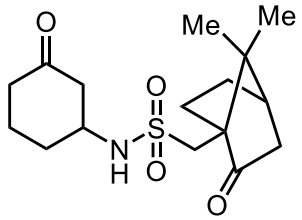
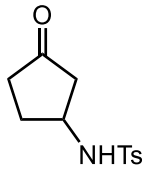


1-(7,7-dimethyl-2-oxobicyclo[2.2.1]heptan-1-yl)-N-(3-oxocyclohexyl)methanesulfonamide (13)

This compound was prepared according to the General Procedure using, (1S)-10-Camphorsulfonamide (46 mg, 0.2 mmol) and cyclohex-2-en-1-one (38 µL, 0.4 mmol). After purification by column chromatography SiO_2 (50% to 70% EtOAc/hexanes), The title compound was isolated as a colorless oil and inseparable mixture of diastereomers (31 mg, 47% yield).

^{1}H NMR (600 MHz, $CDCl_3$) δ 5.73 (d, *J* = 6.6 Hz, 1 H_{iso1}), 5.55 (d, *J* = 6.6 Hz, 1 H_{iso2}), 3.89-3.77 (m, 2 $H_{iso1+iso2}$), 3.40 (d, *J* = 15.1 Hz, 1 H_{iso1}), 3.37-3.33 (m, 1 H_{iso2}), 3.01 (d, *J* = 10.5 Hz, 1 H $_{iso1}$), 2.98 (d, *J* = 10.3 Hz, 1 H $_{iso2}$), 2.82 (dd, *J* = 14.4, 4.8, Hz, 1 H $_{iso1}$), 2.71 (dd, *J* = 14.4, 4.4 Hz, 1 H $_{iso2}$), 2.47-2.35 (m, 6 H $_{iso1+iso2}$), 2.34-2.26 (m, 2 H $_{iso1+iso2}$), 2.26-2.11 (m, 6 H $_{iso1+iso2}$), 2.11-1.98 (m, 5 H $_{iso1+iso2}$), 1.98-1.90 (m, 3 H $_{iso1+iso2}$), 1.86-1.68 (m, 4 H $_{iso1+iso2}$), 1.50-1.42 (m, 2 H $_{iso1+iso2}$), 1.03 (s, 3 H $_{iso1}$), 1.01 (s, 3 H $_{iso2}$), 0.94 (s, 3 H $_{iso2}$), 0.92 (s, 3 H $_{iso1}$); ^{13}C NMR (150 MHz, $CDCl_3$) δ 217.4, 217.1, 208.2, 208.1, 59.5, 59.3, 53.0, 52.7, 52.2, 51.6, 49.3, 49.0, 48.8, 48.3, 43.0, 42.9, 42.8, 42.7, 40.7, 40.6, 32.7, 31.8, 27.1, 27.0, 27.0, 26.6, 21.8, 21.7, 20.0, 19.9, 19.5, 19.4; IR (Neat): 3279.8, 2956.2, 2892.0, 1737.1, 1709.0, 1450.0, 1418.5, 1320.4, 1143.7, 1052.4 cm^{-1}.



4-methyl-N-(3-oxocyclopentyl)benzenesulfonamide (14)

This compound was prepared according to the General Procedure with slight modifications, using, 4-iodotoluene (10 mg, 0.04 mmol), Selectfluor (28 mg, 0.08 mmol) 4-methylbenzenesulfonamide (34 mg, 0.2 mmol) and cyclopent-2-en-1-one (33 µL, 0.4 mmol). After purification by column chromatography SiO_2 (30% to 50% EtOAc/hexanes), The title compound was isolated as a white solid (20 mg, 39% yield).

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.78 (d, *J* = 8.3 Hz, 2 H), 7.33 (d, *J* = 8.3 Hz, 2 H), 5.05 (d, *J* = 6.1 Hz, 1 H), 3.91-3.83 (m, 1 H), 2.45 (s, 3 H), 2.41 (dd, *J* = 18.7, 7.0 Hz, 1 H), 2.38-2.30 (m, 1 H), 2.28-2.19 (m, 1 H), 2.19-2.03 (m, 2 H), 1.91-1.83 (m, 1 H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 214.8, 144.0, 137.0, 129.9, 127.1, 51.3, 45.3, 36.7, 30.2, 21.6; IR (neat): 3240.0, 3183.0, 2919.8, 1733.8, 1596.0, 1463.8, 1323.4, 1149.4, 1091.3 cm^{-1}.

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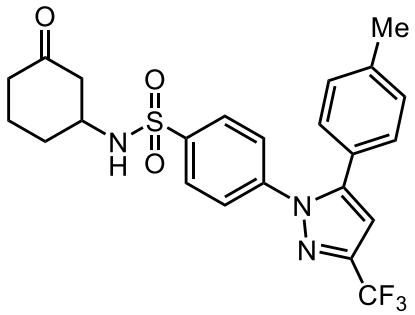
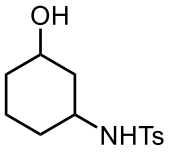


N-(3-oxocyclohexyl)-4-(3-(trifluoromethyl)-5-(4-(trifluoromethyl)phenyl)-1H-pyrazol-1-yl)benzenesulfonamide (15)

This compound was prepared according to the General Procedure using, Celecoxib (76 mg, 0.2 mmol) and cyclohex-2-en-1-one (38 µL, 0.4 mmol). After purification by column chromatography SiO_2 (50% to 70% EtOAc/hexanes), The title compound was isolated as a white solid (65 mg, 61% yield).

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.86 (d, *J* = 8.5 Hz, 2 H), 7.49 (d, *J* = 8.5 Hz, 2 H), 7.19 (d, *J* = 7.8 Hz, 2 H), 7.11 (d, *J* = 8.1 Hz, 2 H), 6.76 (s, 1 H), 4.99 (d, *J* = 7.3 Hz, 1 H), 3.65-3.57 (m, 1 H), 2.55 (dd, *J* = 14.4, 4.8 Hz, 1 H), 2.39 (s, 3 H), 2.37-2.31 (m, 1 H), 2.29-2.18 (m, 2 H), 2.02-1.93 (m, 2 H), 1.71-1.60 (m, 2 H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 207.7, 145.3, 144.11 (q, J_{C-F} = 39.0 Hz), 142.6, 140.0, 139.8, 129.8, 128.7, 127.9, 125.7, 125.6, 121.0 (q, J_{C-F} = 265.5 Hz) 106.3, 52.6, 48.4, 40.5, 31.7, 21.6, 21.3; IR (Neat); 3266.0, 2955.4, 1708.0, 1596.6, 1471.4, 1235.6, 1158.1, 1131.1, 1094.4 cm^{-1}.



N-(3-hydroxycyclohexyl)-4-methylbenzenesulfonamide (16)

To an 8 ml vial equipped with magnetic stir bar was added 4-methyl-N-(3-oxocyclohexyl)benzene sulfonamide (53 mg, 0.2 mmol) and anhydrous THF (1 mL). Then $AlCl_3$ (40 mg, 0.3 mmol) was added, followed by $LiAlH_4$ (12 mg, 0.3 mmol). The reaction mixture was stirred at room temperature for 16 h. Then the reaction was quenched with aqueous NaOH solution (0.5 mL, 1M), and the organic layer was separated. The aqueous layer was extracted with EtOAc (2×2 mL), and the combined organic layers were dried over anhydrous Na_2SO_4. The resulting solution was concentrated under reduced pressure. The crude product was isolated by column chromatography SiO_2 (30% to 50% EtOAc/hexanes), as a colorless oil (37 mg, 69% yield). Only the major diastereomer is presented in the ^{1}H and ^{13}C NMR spectra.

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.76 (d, *J* = 8.3 Hz, 2 H), 7.30 (d, *J* = 8.1 Hz, 2 H), 5.22 (br. s., 1 H), 3.72 (br. s., 1 H), 3.36-3.23 (m, 1 H), 2.43 (s, 3 H), 1.88 (d, *J* = 12.2 Hz, 1 H), 1.81-1.72 (m, 2 H), 1.72-1.60 (m, 2 H), 1.36-1.18 (m, 4 H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 143.2, 138.3, 129.7, 126.9, 68.4, 50.4, 40.3 33.7, 32.5, 21.5, 19.2; IR; 3489.5, 3273.2, 2936.4, 2860.5, 1452.0, 1319.1, 1153.1, 1091.2, 1038.2 cm^{-1}.

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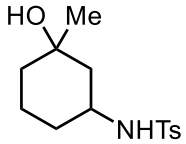

N-(3-hydroxy-3-methylcyclohexyl)-4-methylbenzenesulfonamide (17)

To an 8 ml vial equipped with magnetic stir bar was added 4-methyl-N-(3-oxocyclohexyl) benzenesulfonamide (53 mg, 0.2 mmol) and anhydrous THF (1 mL). After cooling to 0 ˚C, MeMgBr in THF (1.7 mL, 0.5 mmol) was added. The reaction mixture was stirred at room temperature for 16 h. Then the reaction was quenched with sat. NH_4Cl solution (1 mL), and the organic layer was separated. The aqueous layer was extracted with EtOAc (2×2 mL), and the combined organic layers were dried over anhydrous Na_2SO_4. The resulting solution was concentrated under reduced pressure. The crude product isolated by column chromatography SiO_2 (30% to 50% EtOAc/hexanes), as a colorless oil and inseparable mixture of diastereomers (35 mg, 62% yield).

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.78 (d, *J* = 6 Hz, 2 H_{iso1}), 7.76 (d, *J* = 6 Hz, 2 H_{iso2}), 7.31 (d, *J* = 6.0 Hz, 2 H_{iso1}), 7.28 (d, *J* = 6.0 Hz, 2 H_{iso2}), 6.23 (br. s., 1 $H_{iso1/iso2}$), 4.50 (d, *J* = 9.0 Hz, 1 $H_{iso1/iso2}$), 3.62-3.52 (m, 1 H $_{iso1}$), 3.5-3.41 (m, 1 H $_{iso2}$), 2.43 (s, 6 $H_{iso1+iso2}$), 1.90-1.75 (m, 3 H $_{iso1+iso2}$), 1.68-1.43 (m, 6 H $_{iso1+iso2}$), 1.44-1.32 (m, 3 H $_{iso1+iso2}$), 1.24-1.17 (m+s, 4 H $_{iso2}$), 1.14 (s, 3 H H $_{iso1}$), 1.06-0.94 (m, 1 H H $_{iso1}$); ^{13}C NMR (150 MHz, $CDCl_3$) δ 143.2, 142.9, 138.6, 138.1, 129.8, 129.7, 129.5, 126.9, 126.9, 71.2, 70.5, 49.6, 49.4, 46.4, 41.5, 38.6, 37.3, 33.3, 31.5, 31.3, 21.5, 21.5, 20.4, 17.0; IR; 3500.8, 3275.5, 2933.1, 2865.3, 1447.2, 1320.9, 1152.2, 1093.7 cm^{-1}.

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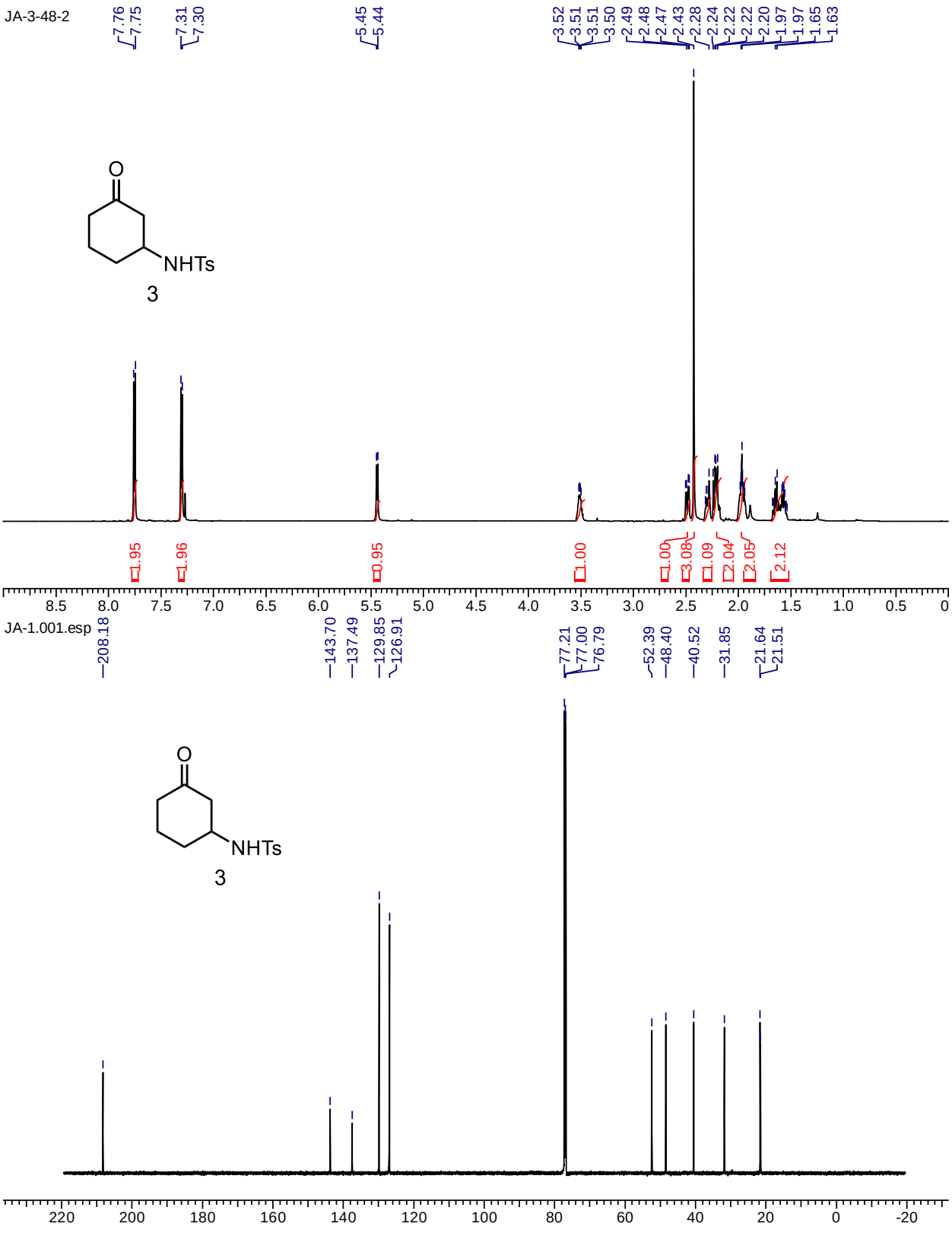
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2.22
2.20
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1.65
1.63
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1.00
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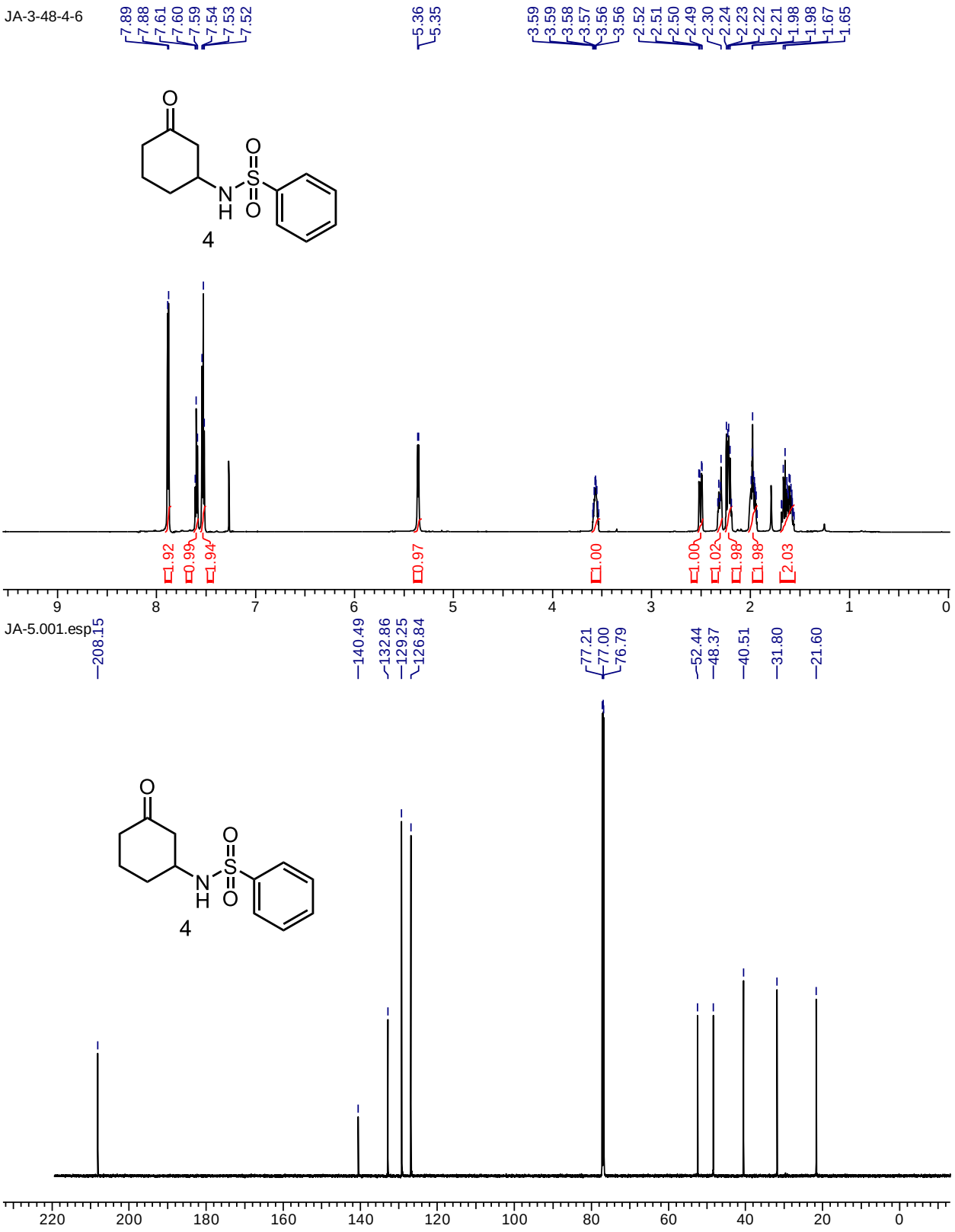
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4

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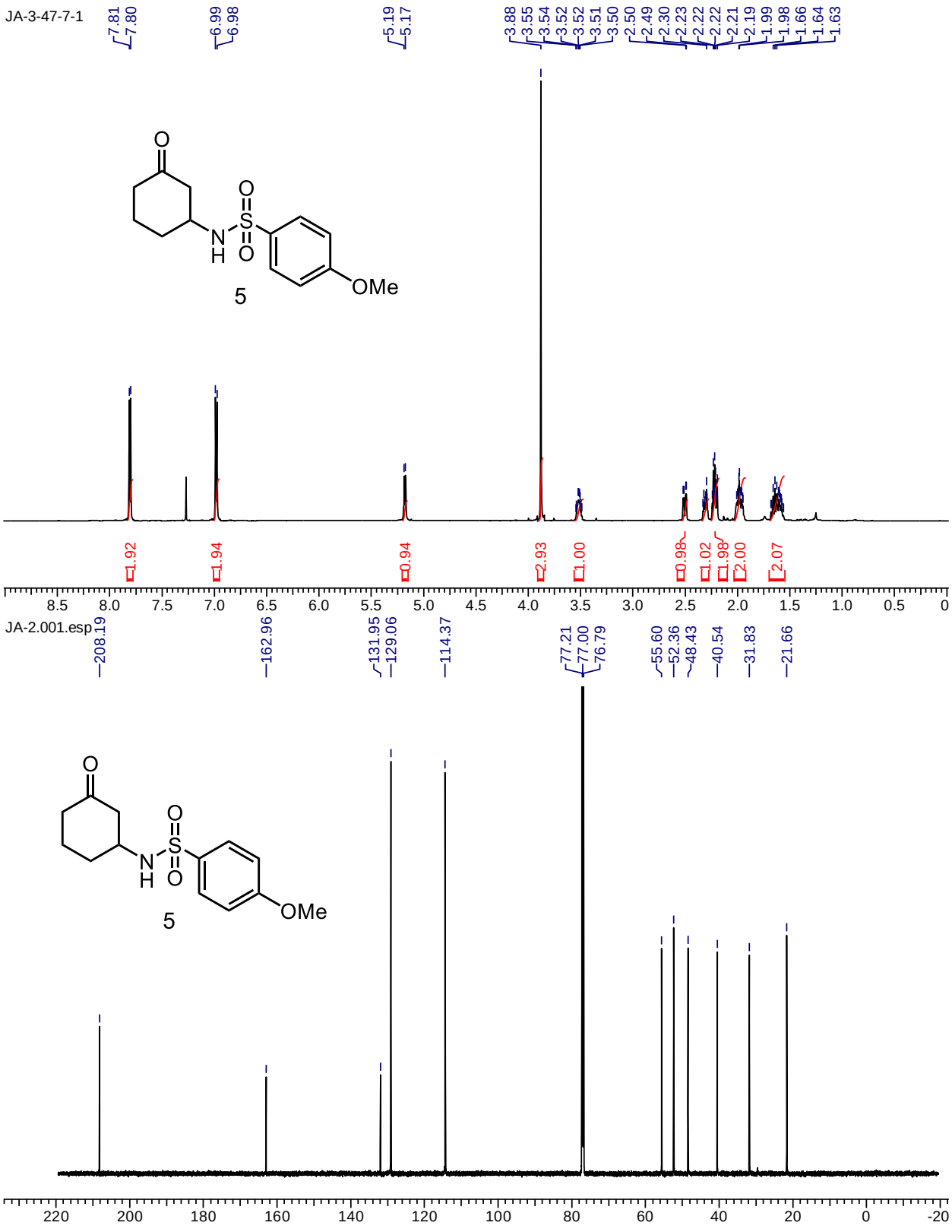
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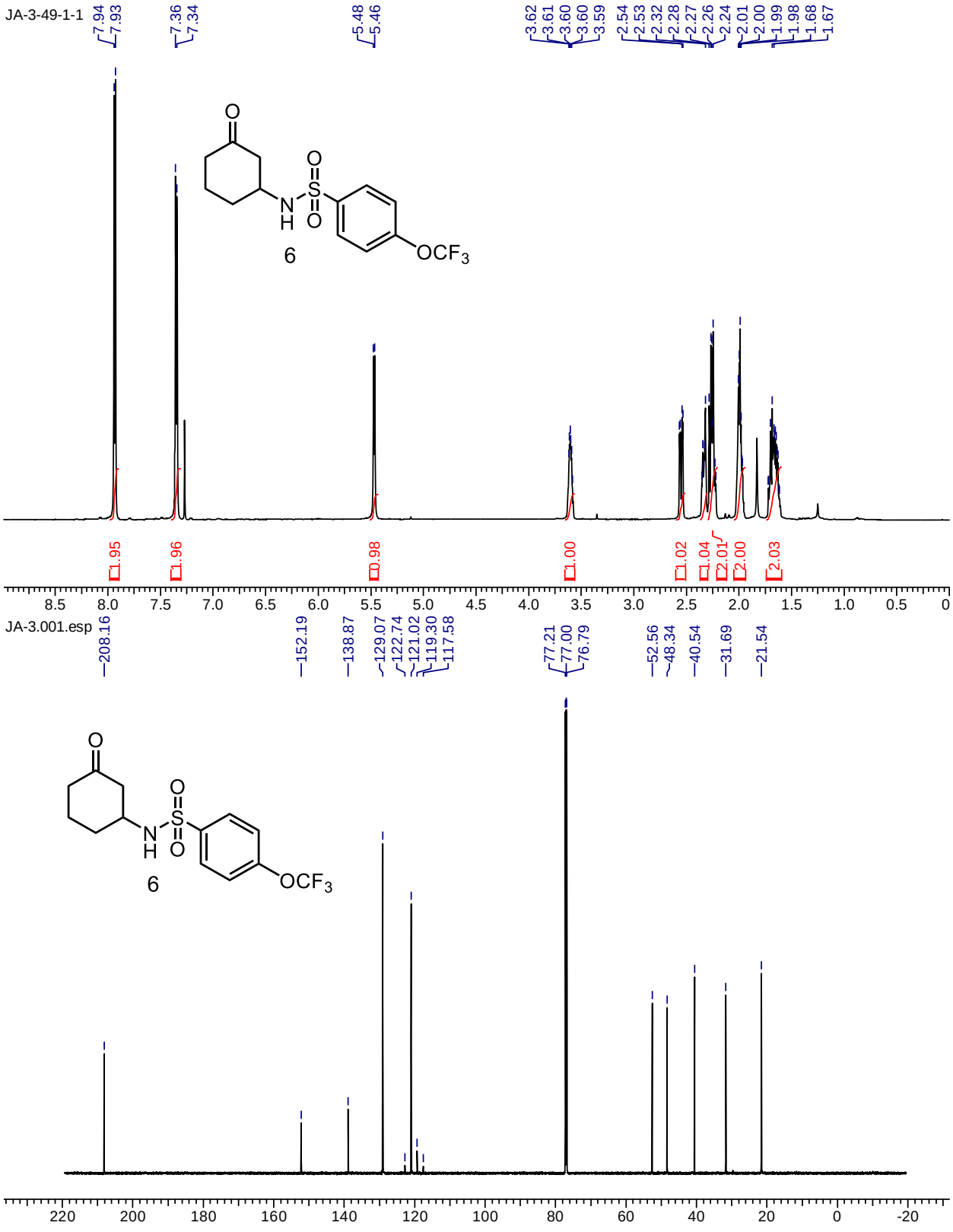
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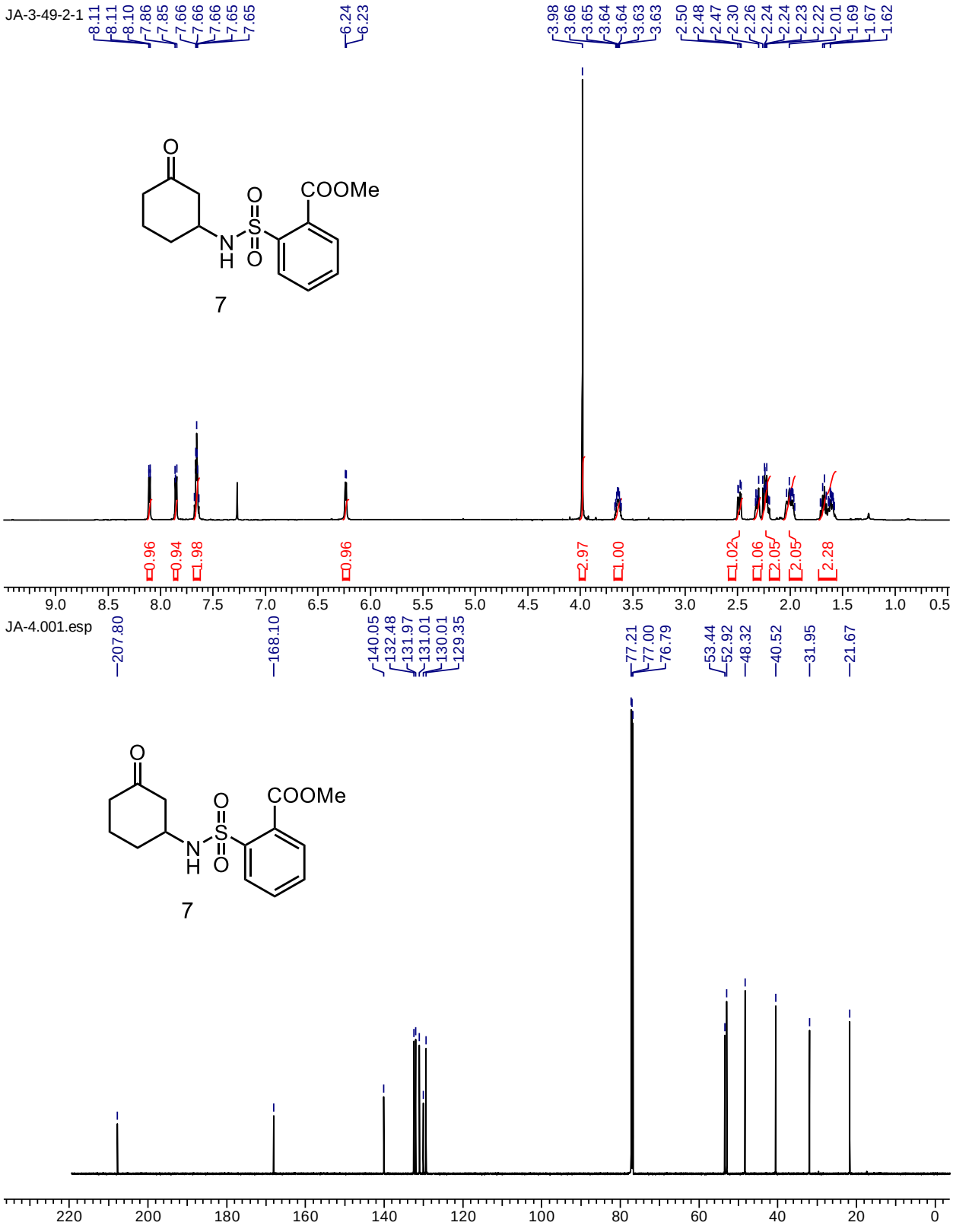
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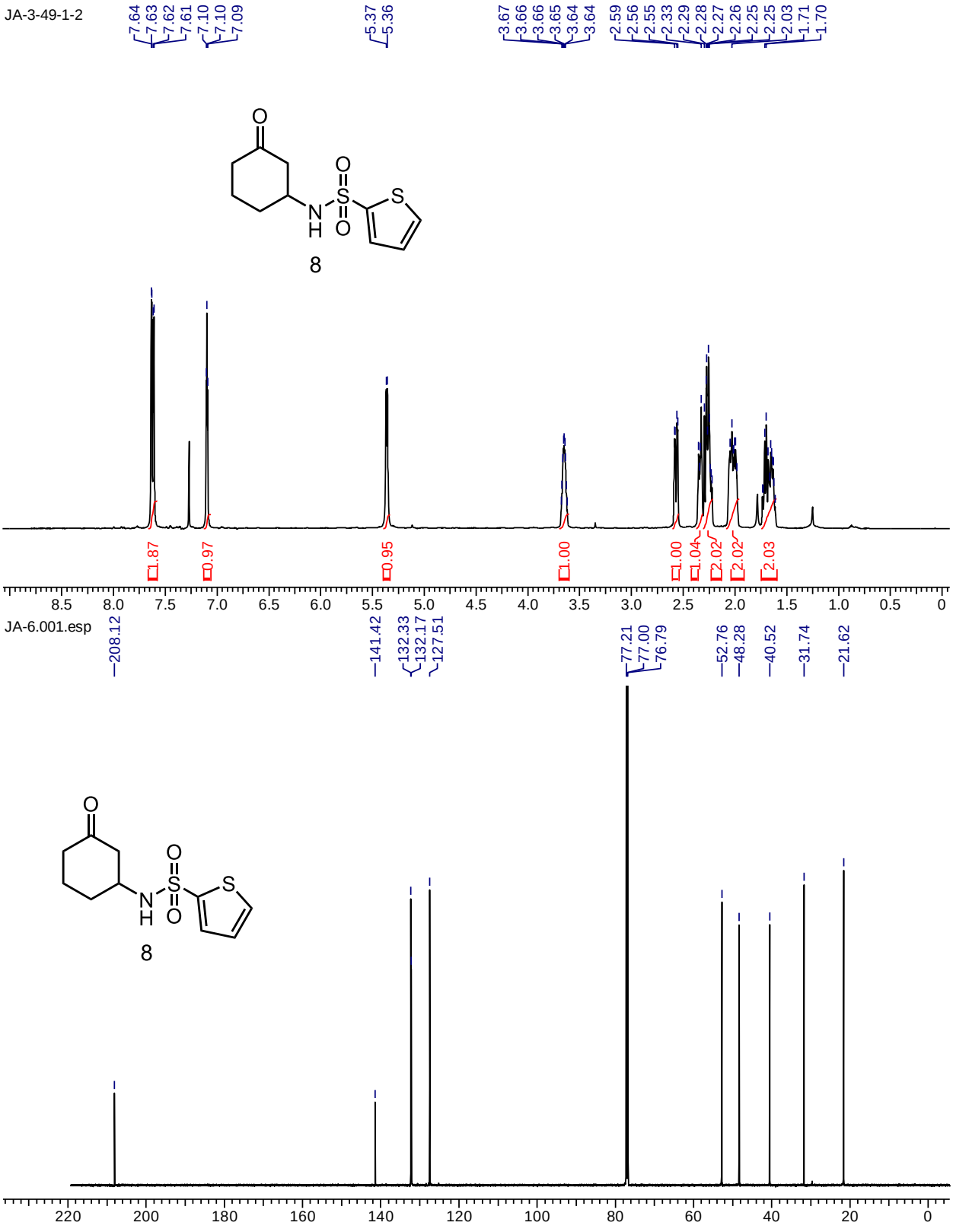

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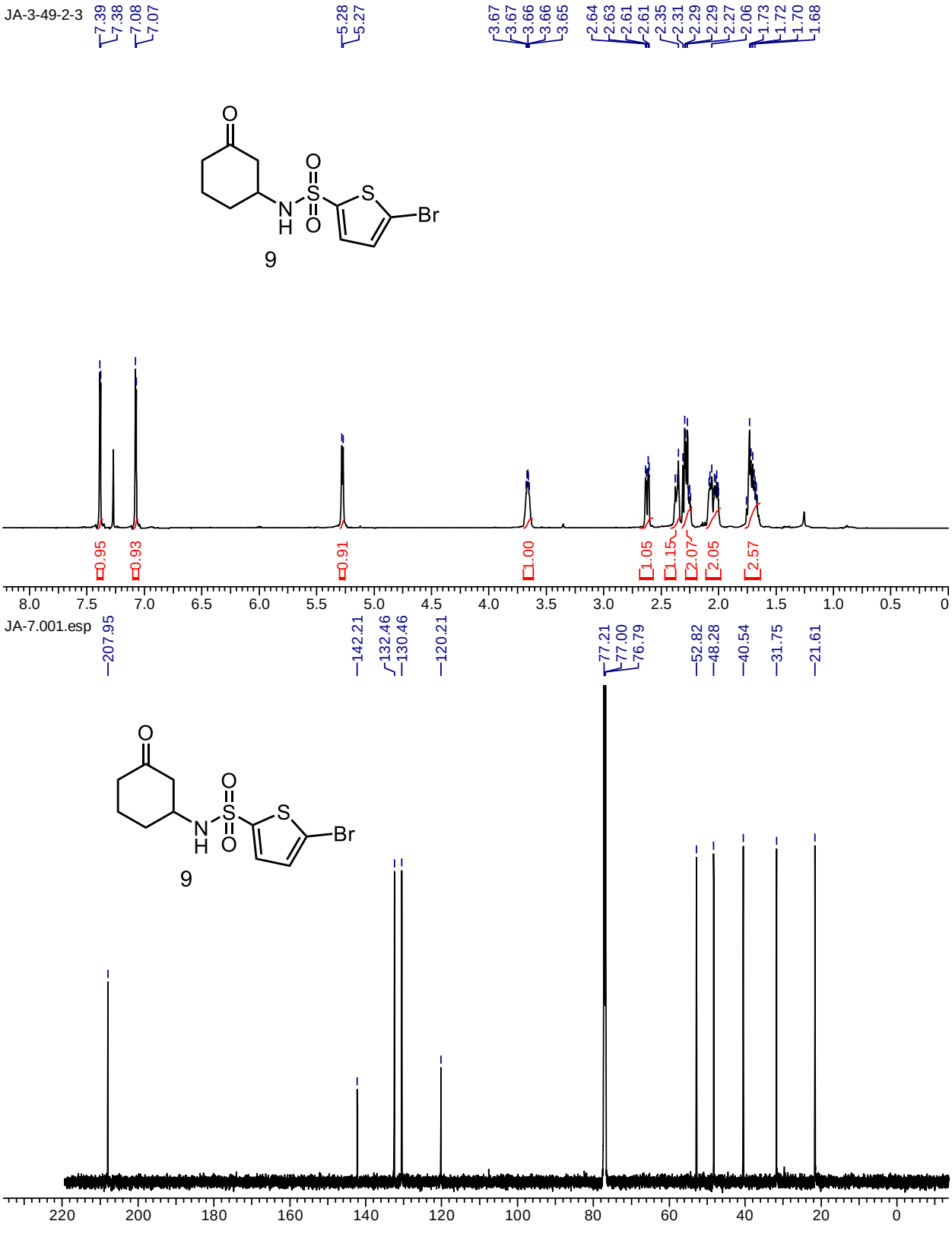
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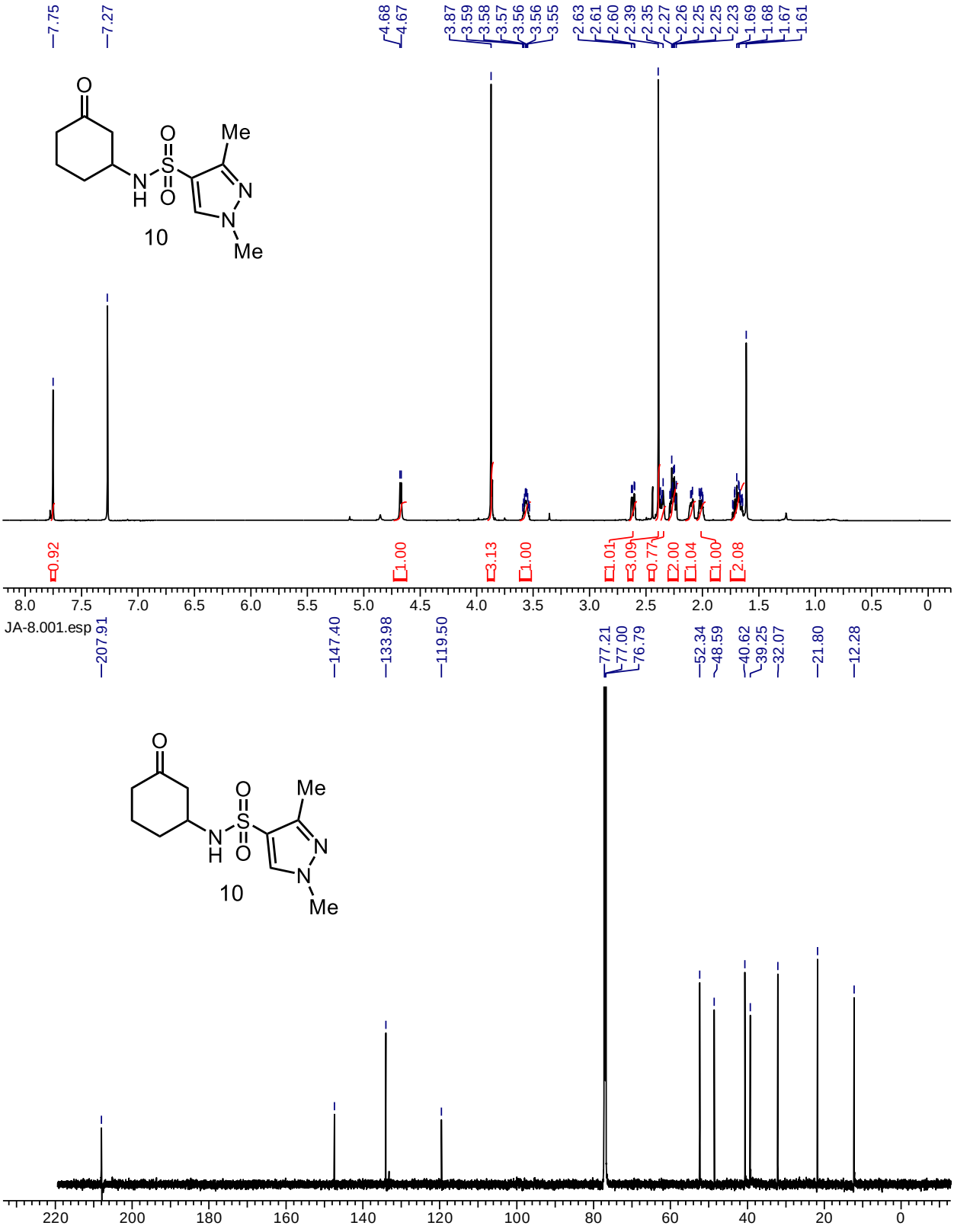
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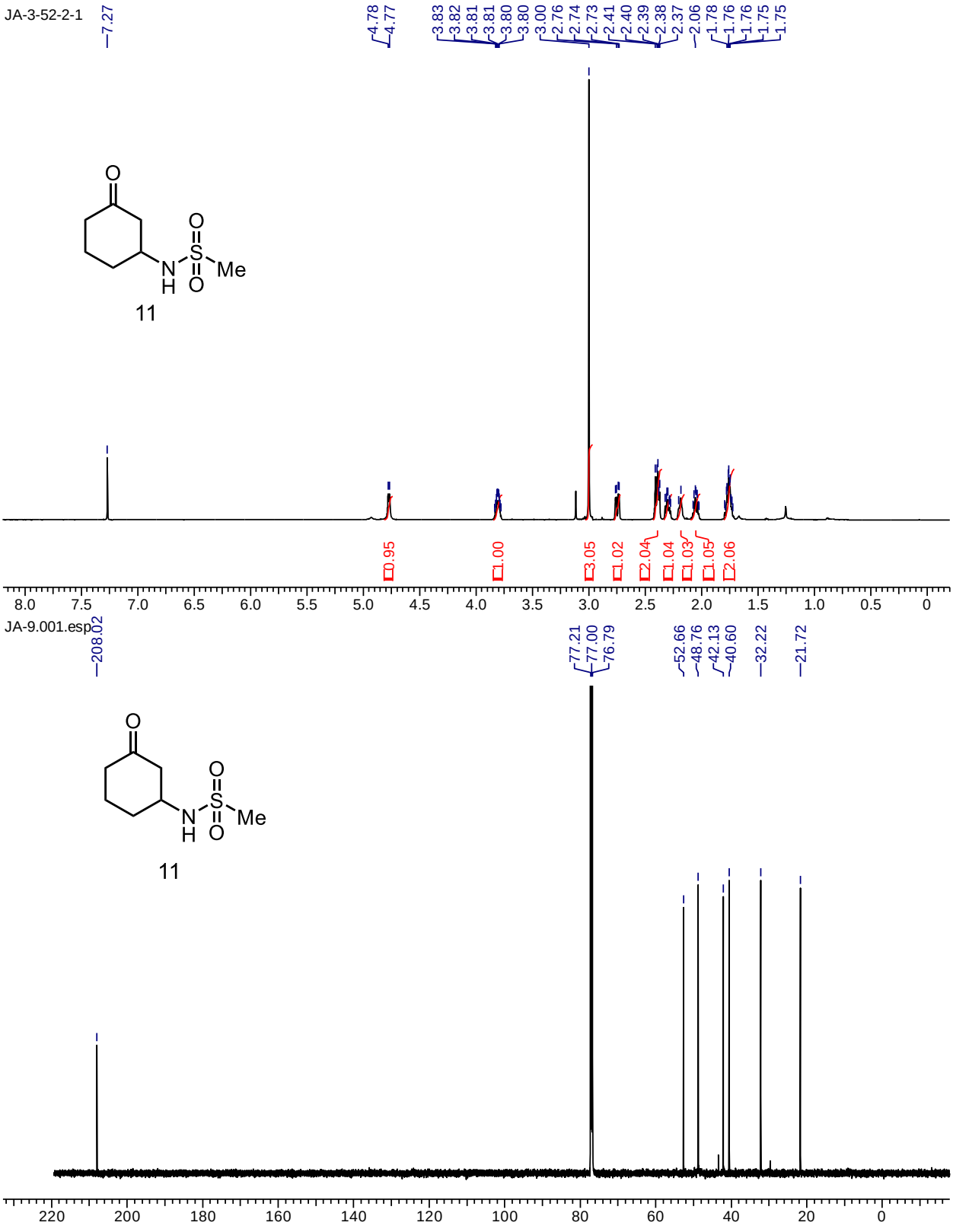
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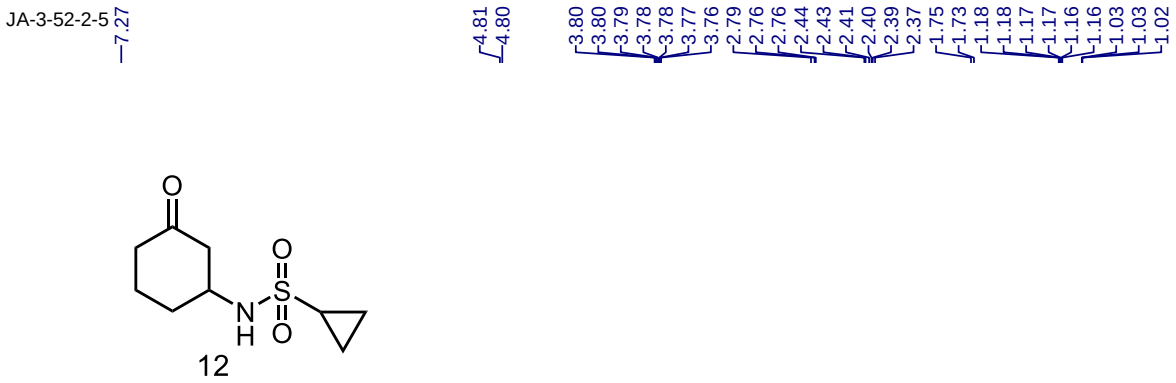

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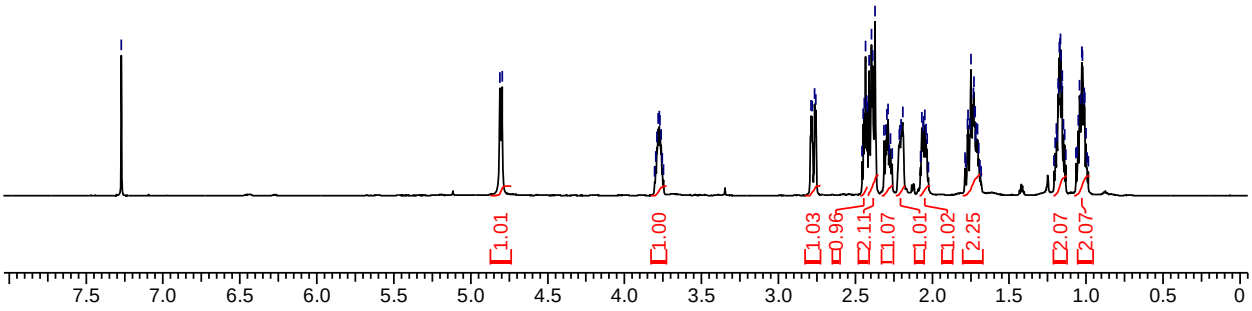

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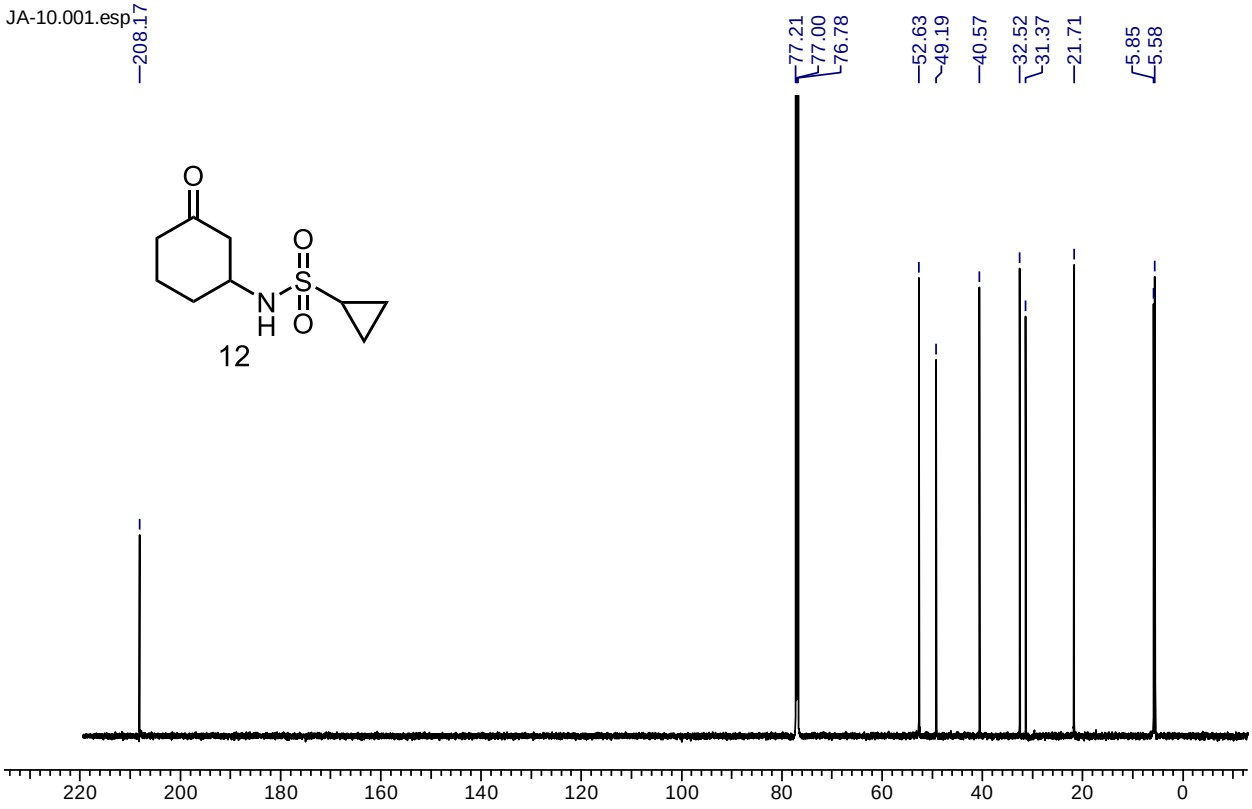

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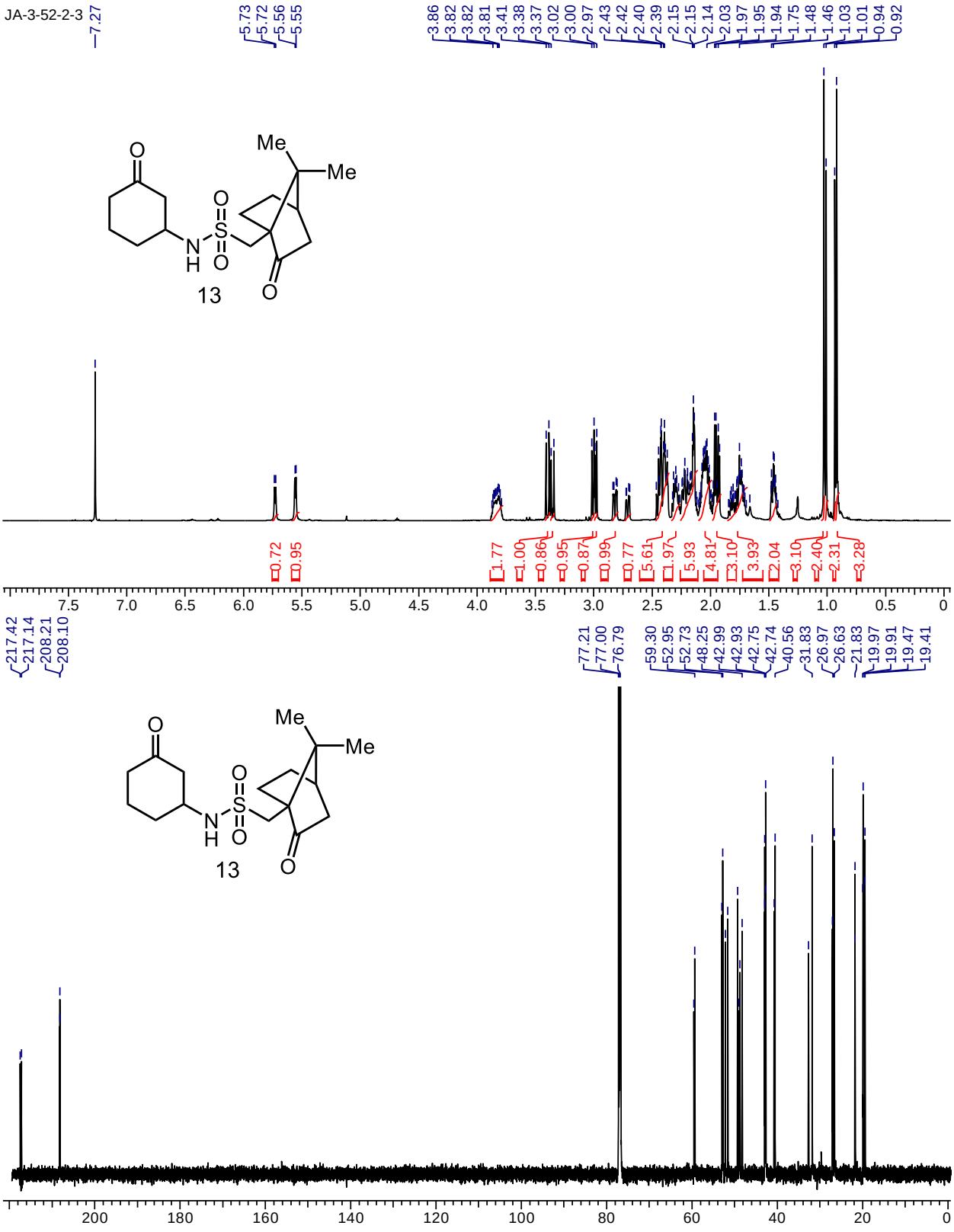
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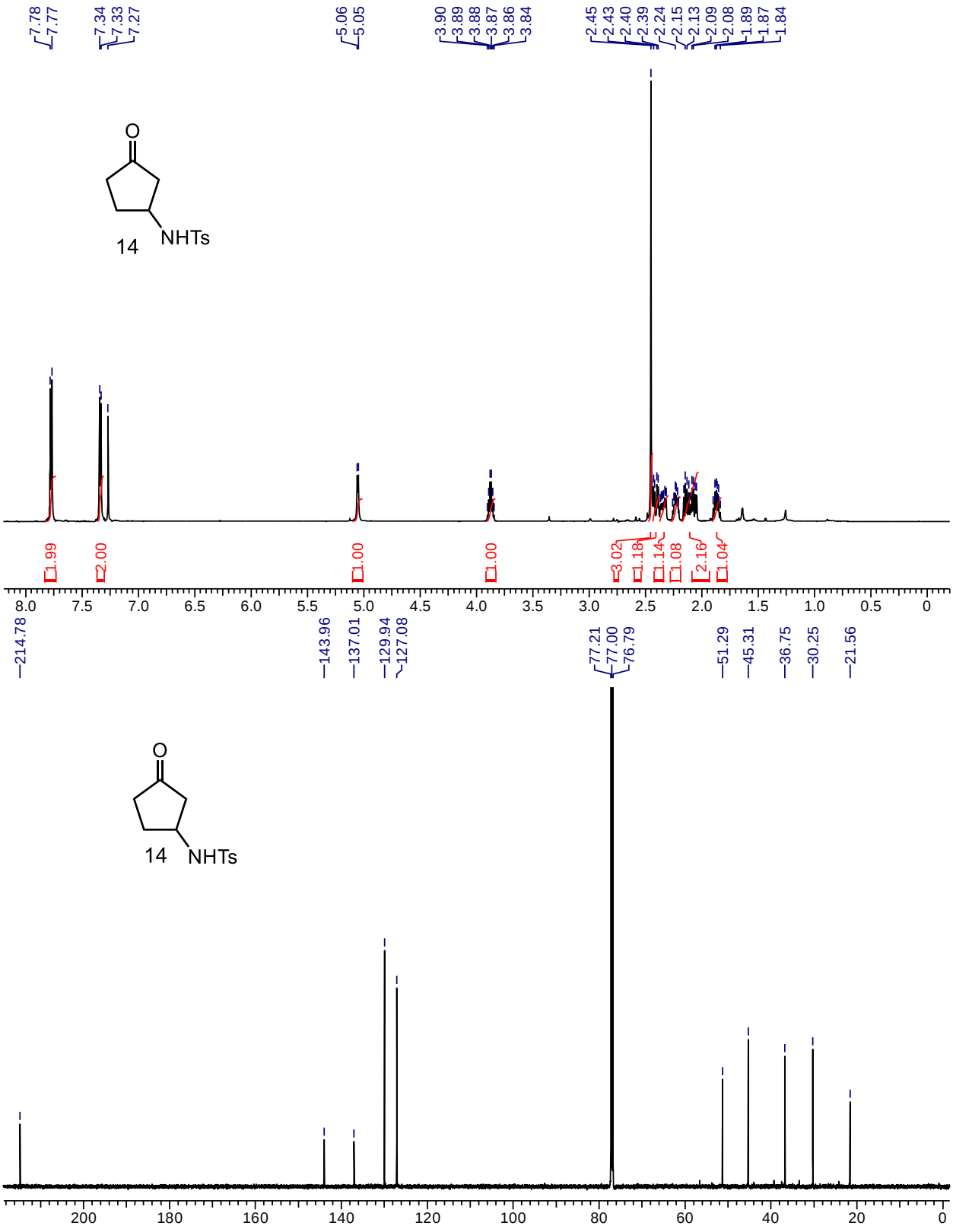
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NHTs

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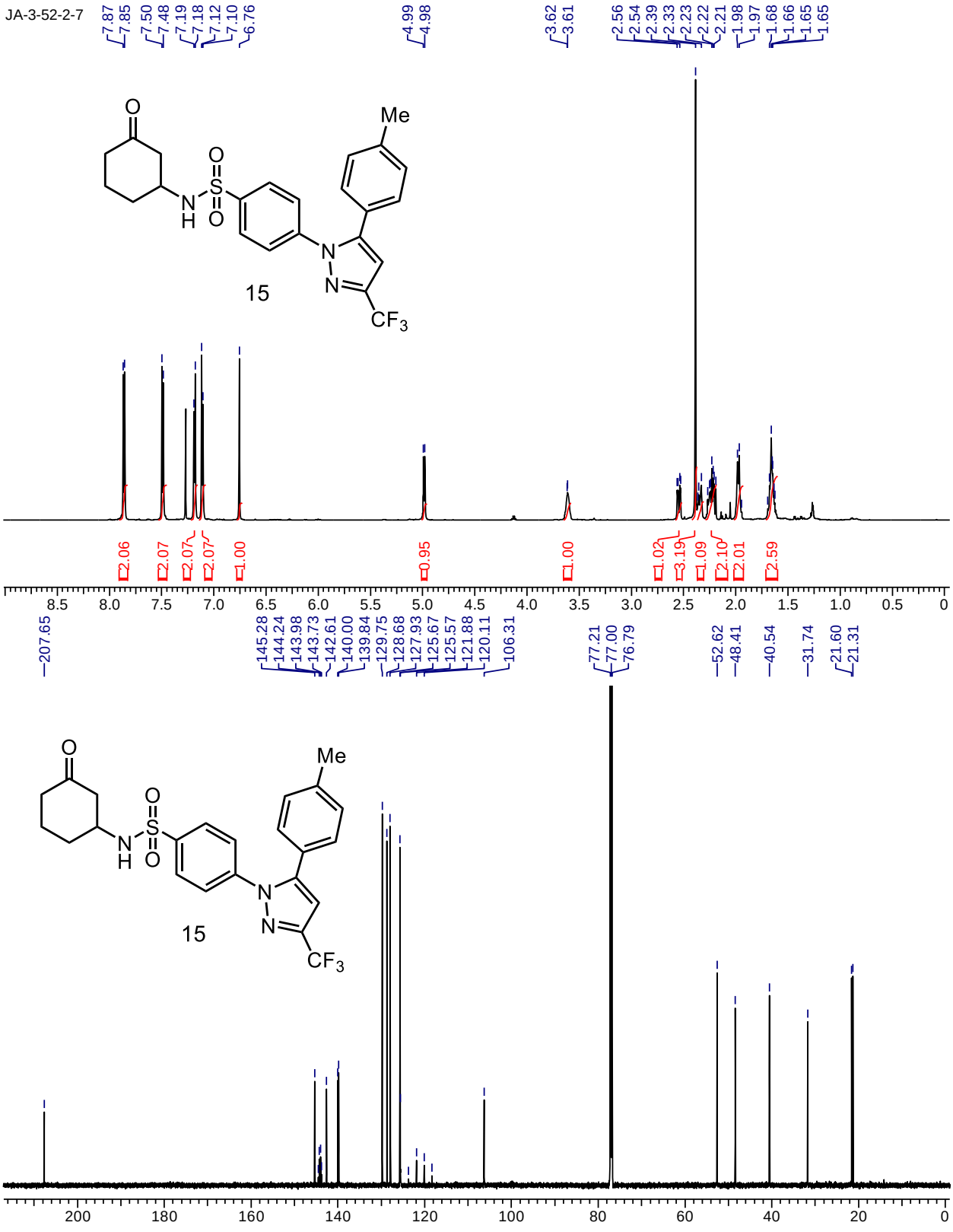
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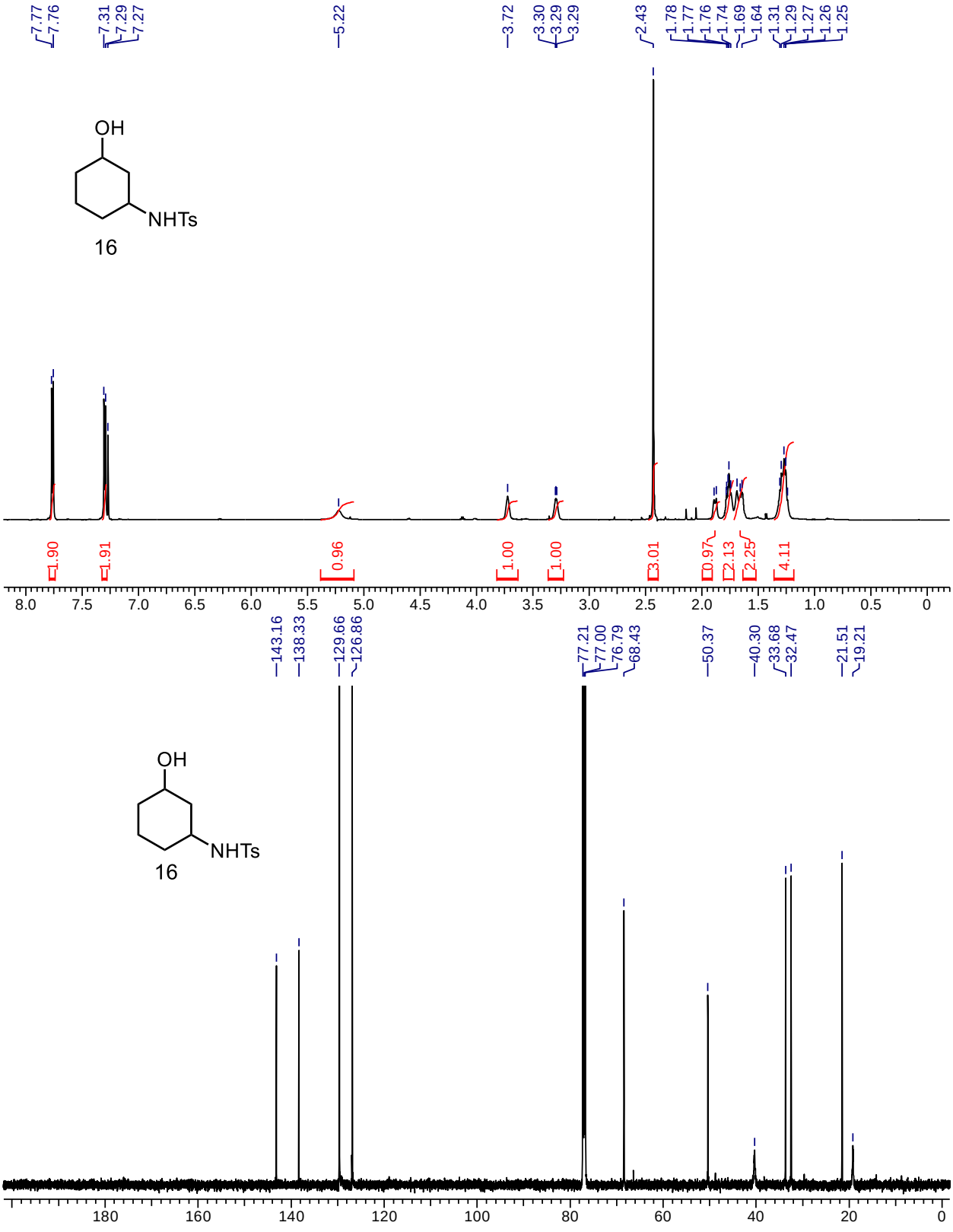
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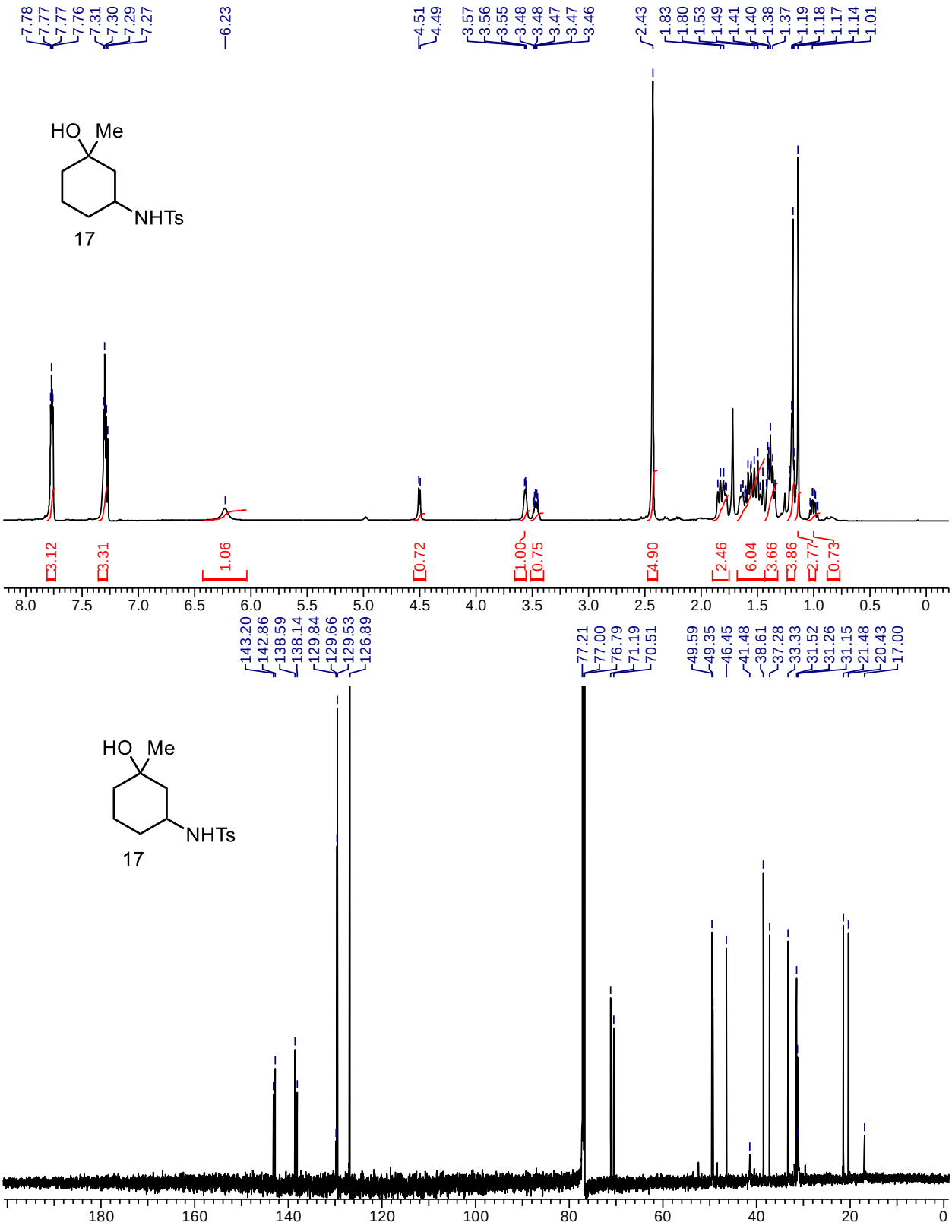
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HO Me
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