Supporting Information

Catalytic Generation of Vinylthionium Ions. (4+3)-Cycloadditions and Friedel-Crafts Alkylations

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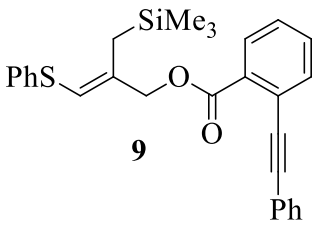
General information

All glassware and needles were dried in an oven at 120 ^{o}C and cooled in a desiccator prior to use. Nitroethane, dichloromethane and acetonitrile were distilled over calcium hydride. Diethyl ether and THF were distilled over sodium-benzophenone. *n*-Butyllithium was purchased from Sigma-Aldrich and titrated with diphenylacetic acid. 2-Alkyl and 2,5-dialkylfuran derivatives were purchased or prepared from the alkylation of furan with *n*-butyllithium and an alkyl halide. Alkylation and cycloaddition products were purified by flash chromatography using 230-400 mesh silica as the stationary phase. ^{1}H NMR spectra were recorded on a Bruker DRX-500 (500 MHz) spectrometer with chemical shifts reported in δ ppm with tetramethylsilane (0.00 ppm) as an internal reference ^{13}C NMR spectra were recorded on the same instrument at 125 MHz in $CDCl_3$ solution with $CDCl_3$ (77.0 ppm) as an internal reference. Infrared spectra were recorded on a Perkin Elmer 1600 series FT-IR spectrometer. High-resolution mass spectra were performed by College of Science Major Instrumentation Center, Old Dominion University, on a Bruker 12 Tesla APEX -Qe FTICR-MS with an Apollo II ion source.

Experimental Procedures

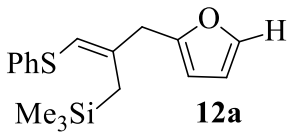


9

(Z)-3-(phenylthio)-2-((trimethylsilyl)methyl)allyl-2-(phenylethynyl)benzoate: (Z)-3-(phenylthio)-2-((trimethylsilyl)methyl)prop-2-en-1-ol (0.87 g, 3.4 mmol) and 2-(phenylethynyl)benzoic acid (0.84 g, 37.7 mmol) were dissolved in 80 mL of a CH_2Cl_2/THF mixture (1:1) and cooled to 0 ^oC. 4-Dimethylamino pyridine (0.042 g, 0.34 mmol) and then DCC (0.78 g, 3.4 mmol) was added to the reaction flask. After stirring for 15 minutes, the mixture was warmed to room temperature and stirred for 4 hours. The mixture was filtered over Celite and purified by flash chromatography (2% EtOAc: Hexanes) to afford **9** as a clear, yellow oil (1.1g, 76%). ^1H NMR (500 MHz, $CDCl_3$) δ 8.05 (d, *J*=7.5Hz, 1H), 7.70 (d, *J*=7.5 Hz, 1H), 7.58 (m, 2H), 7.55 (t, *J*= 8.0 Hz, 1H), 7.42 (t, *J*=7.5 Hz, 1H), 7.34 (m, 5H), 7.27 (t, *J*=7.5 Hz, 2H), 7.19 (t, *J*= 7.5 Hz, 1H), 6.33 (s, 1H), 4.89 (s, 2H), 1.94 (s, 2H), 0.014 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 165.8, 136.8, 136.3, 134.1, 131.7, 131.6, 131.5, 130.5, 128.8, 128.4, 128.3, 128.2, 127.8, 125.9, 123.7, 123.1, 118.2, 94.4, 88.1, 68.8, 21.6, -0.6; IR (cm^{-1}) 3055, 2950, 2891, 1729, 1709, 1492, 1436, 1287, 1248, 1123, 1064, 850, 754, 685; HRMS calcd for $(C_{29}H_{30}O_2SSi)Na^+$ 493.1627, not observed.

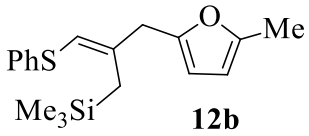
General Procedure for the preparation of compounds 12a-i, 16a-f

Compound **9** (0.025 g, 0.05 mmol) was dissolved in 5 mL of dichloromethane and degassed for 10 minutes. The alkylfuran derivative (0.05 mmol), triphenylphosphine gold chloride (0.0013g, 0.0027 mmol) and then silver triflate (0.0007g, 0.0027 mmol) was added to the reaction vessel. After 10 minutes, the reaction was quenched with sat. $NaHCO_3$ and extracted with CH_2Cl_2 (2X 5 mL). The combined extracts were washed with water, dried with Na_2SO_4, concentrated *in vacuo* and purified using flash chromatography (Hexanes) to afford clear and colorless oils.



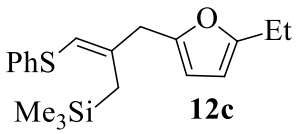
12a

(Z)-(2-(furan-2-ylmethyl)-3-(phenylthio)allyl)trimethylsilane. Isolated as a colorless oil in 93% yield (0.015 g). ^{1}H NMR (500 MHz, $CDCl_3$) δ7.38 (m, 1H), 7.30-7.28 (m, 4H), 7.17-7.16 (m, 1H), 6.34 (t, *J*=2.5 Hz, 1H), 6.13 (d, *J*=3.0 Hz, 1H), 5.88 (s, 1H), 3.48 (s, 2H), 1.87 (s, 2H), 0.13 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 152.8, 141.9, 141.4, 137.4, 128.7, 127.5, 125.3, 114.3, 110.2, 106.8, 37.5, 23.5, -0.71; IR (cm^{-1}) 3053, 2950, 2917, 1581, 1478, 1248, 1148, 1009, 850, 737; HRMS calcd for $(C_{18}H_{24}OSSi)Na^{+}$ 339.1209, found 339.1208.



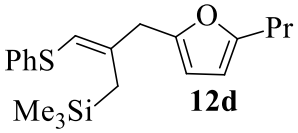
12b

(Z)-trimethyl(2-((5-methylfuran-2-yl)methyl)-3-(phenylthio)allyl)silane. Isolated as a colorless oil in 93% yield (0.017 g). ^{1}H NMR (500 MHz, $CDCl_3$) δ7.31 (m, 4H), 7.17-7.16 (m, 1H), 5.99 (d, *J*= 3.0 Hz, 1H), 5.91 (d, *J*=2.0 Hz, 1H), 5.89 (s, 1H), 3.42 (s, 2H), 2.30 (s, 3H), 1.88 (s, 2H), 0.12 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 150.9, 150.8, 142.6, 137.6, 128.6, 127.4, 125.2, 113.8, 107.4, 105.9, 37.6, 23.6, 13.4, -0.71; IR (cm^{-1}) 3072, 3050, 2953, 2917, 2888,1581, 1478, 1436, 1416, 1248, 1015, 853, 779, 737; HRMS calcd for $(C_{18}H_{24}OSSi)Na^{+}$ 338.1264, found 338.1266.



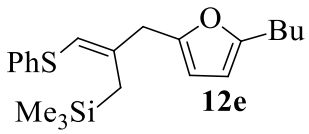
12c

(Z)-(2-((5-ethylfuran-2-yl)methyl)-3-(phenylthio)allyl)trimethylsilane. Isolated as a colorless oil in 95% yield (0.017 g). ^{1}H NMR (500 MHz, $CDCl_3$) δ7.30 (m, 4H), 7.17-7.16 (m, 1H), 6.00 (d, *J*=2.5 Hz, 1H), 5.91 (d, *J*=2.5 Hz, 1H), 5.87 (s, 1H), 3.65 (q, *J*=7.5 Hz, 2H), 3.43 (s, 2H), 1.88 (s, 2H), 1.25 (t, *J*=7.0 Hz, 3H), 0.12 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 156.7, 150.6, 142.8, 137.6, 128.6, 127.3, 125.2, 113.7, 107.2, 104.3, 37.6, 23.6, 21.3, 12.1, -0.7; IR (cm^{-1}) 3107, 3072, 3056, 2955, 2914, 2892, 1951, 1875, 1776, 1576, 1560, 1478, 1442, 1477, 1237, 1021; HRMS calcd for $(C_{19}H_{26}OSSi)Na^{+}$ 353.1365, found 353.1366.



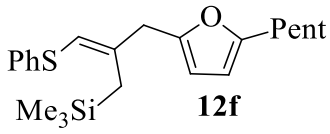
12d

(Z)-trimethyl(3-(phenylthio)-2-((5-propylfuran-2-yl)methyl)allyl)silane. Isolated as a colorless oil in 91% yield (0.017 g). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.29 (m, 4H), 7.17-7.16 (m, 1H), 6.00 (d, *J*=3.0 Hz, 1H), 5.92 (d, *J*=3.0 Hz, 1H), 5.87 (s, 1H), 3.43 (s, 2H), 2.61 (t, *J*=7.0 Hz, 2H), 1.88 (s, 2H), 1.72-1.65 (sextet, *J*=7.5 Hz, 2H), 0.97 (t, *J*=7.5Hz, 3H), 0.13 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 155.3, 150.6, 142.9, 137.6, 128.6, 127.3, 125.2, 113.7, 107.2, 105.2, 37.6, 30.0, 23.6, 21.3, 13.6, -0.7; IR (cm^{-1}) 3064, 2958, 2929, 1940, 1634, 1585, 1478, 1442, 1254, 1147, 1086, 1025; HRMS calcd for $(C_{20}H_{28}OSSi)Na^{+}$ 367.1522, found 367.1525.



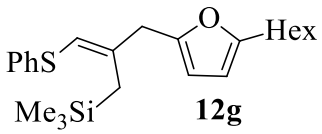
12e

(Z)-(2-((5-butylfuran-2-yl)methyl)-3-(phenylthio)allyl)trimethylsilane. Isolated as a colorless oil in 75% yield (0.015 g). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.29 (m, 4H), 7.17-7.16 (m, 1H), 5.99 (d, *J*=2.5 Hz, 1H), 5.91 (d, *J*=3.0 Hz, 1H), 5.86 (s, 1H), 3.42 (s, 1H), 2.61 (t, *J*=7.5 Hz, 2H), 1.87 (s, 2H), 1.67-1.61 (pentet, *J*=7.5 Hz, 2H), 1.43-1.36 (sextet, *J*=8.0 Hz, 2H), 0.94 (t, *J*=7.0 Hz, 3H), 0.12 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 155.5, 150.6, 142.8, 137.6, 128.6, 127.3, 125.2, 113.7, 107.2, 105.0, 37.6, 30.1, 27.6, 23.6, 22.1, 13.7, -0.7; IR (cm^{-1}) 3060, 2953, 2933, 2872, 1944, 1801, 1589, 1482, 1437, 1380, 1339, 1082, 1025, 898; HRMS calcd for $(C_{21}H_{30}OSSi)Na^{+}$ 381.1678, found 381.1681.



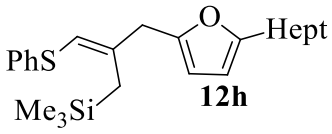
12f

(Z)-trimethyl(2-((5-pentylfuran-2-yl)methyl)-3-(phenylthio)allyl)silane. Isolated as a colorless oil in 70% yield (0.014 g). ^{1}H NMR (500 MHz, $CDCl_3$) 7.28 (m, 4H), 7.17-7.16 (m, 1H), 5.99 (d, *J*=3.0 Hz, 1H), 5.91 (d, *J*=2.5 Hz, 1H), 5.87 (s, 1H), 3.42 (s, 2H), 2.61 (t, *J*=7.5 Hz, 2H), 1.88 (s, 2H), 1.68-1.62 (pentet, *J*=7.0 Hz, 2H), 1.35 (m, 4H), 0.91 (t, *J*=7.0 Hz, 3H), 0.12 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 155.5, 150.6, 142.8, 137.6, 128.6, 127.3, 125.2, 113.7, 107.2, 105.0, 37.6, 31.3, 27.9, 27.6, 23.6, 22.3, 13.9, -0.72; IR (cm^{-1}) 3056, 2962, 2929, 2868, 1952, 1801, 1638, 1580, 1478, 1437, 1384, 1331, 1249, 1090, 1029; HRMS calcd for $(C_{22}H_{32}OSSi)Na^{+}$ 395.1835, found 395.1740.



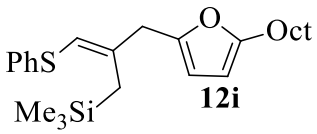
12g

(Z)-trimethyl(2-((5-hexylfuran-2-yl)methyl)-3-(phenylthio)allyl)silane. Isolated as a colorless oil in 68% yield (0.014 g). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.28 (m, 4H), 7.17-7.16 (m, 1H), 5.97 (d, *J*=3.0 Hz, 1H), 5.90 (d, *J*=2.5 Hz, 1H), 5.87 (s, 1H), 3.42 (s, 2H), 2.61 (t, *J*=7.5 Hz, 2H), 1.88 (s, 2H), 1.68-1.62 (pentet, *J*=7.0 Hz, 2H), 1.36 (m, 6H), 0.93 (t, *J*=7.0 Hz, 3H), 0.12 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 155.5, 150.6, 142.8, 137.6, 128.6, 127.3, 125.2, 113.7, 107.2, 105.0, 37.6, 31.4, 28.7, 28.0, 27.9, 23.6, 22.4, 13.9, -0.7; IR (cm^{-1}) 3080, 2953, 2925, 2868, 1940, 1858, 1638, 1638, 1589, 1482, 1437, 1249, 1082, 1021; HRMS cald for $(C_{23}H_{34}OSSi)Na^{+}$ 409.1991, found 409.1996.



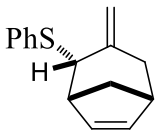
12h

(Z)-trimethyl(2-((5-heptylfuran-2-yl)methyl)-3-(phenylthio)allyl)silane. Isolated as a colorless oil in 66% yield (0.013 g). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.28 (m, 4H), 7.17-7.16 (m, 1H), 5.99 (d, *J*=3.0 Hz, 1H), 5.90 (d, *J*=3.0 Hz, 1H), 5.87 (s, 1H), 3.42 (s, 2H), 2.60 (t, *J*=7.5 Hz, 2H), 1.88 (s, 2H), 1.65 (pentet, *J*=7.0 Hz, 2H), 1.36-1.29 (m, 8H), 0.9 (t, *J*=7.0 Hz, 3H), 0.12 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 155.5, 150.6, 142.8, 137.6, 128.6, 127.3, 125.2, 113.7, 107.2, 105.0, 37.6, 31.6, 29.0, 28.9, 28.0, 23.6, 22.5, 13.9, -0.7; IR (cm^{-1}) 3072, 3051, 2958, 2925, 2872, 1932, 1793, 1634, 1585, 1478, 1437, 1380, 1348, 1094, 1025, 898; HRMS calcd for $(C_{24}H_{36}OSSi)Na^{+}$ 423.2148, found 423.2152.



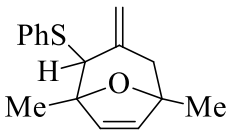
12i

(Z)-trimethyl(2-((5-octylfuran-2-yl)methyl)-3-(phenylthio)allyl)silane. Isolated as a colorless oil in 60% yield (0.011 g). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.28 (m, 4H), 7.17-7.16 (m, 1H), 6.00 (d, *J*=3 Hz, 1H), 5.91 (d, *J*=3.0 Hz, 1H), 5.87 (s, 1H), 3.43 (s, 2H), 2.61 (t, *J*=8.0 Hz, 2H), 1.88 (s, 2H), 1.68-1.62 (pentet, *J*=7.5 Hz, 2H), 1.36-1.28 (m, 10H), 0.90 (t, *J*=7.0 Hz, 3H), 0.12 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 155.5, 150.6, 142.8, 137.6, 128.6, 127.3, 125.2, 113.7, 107.2, 105.0, 37.6, 31.7, 29.2, 29.1, 28.0, 23.6, 22.5, 14.0, -0.7; IR (cm^{-1}) 3072, 3060, 2962, 2929, 2872, 1928, 1801, 1642, 1589, 1474, 1442, 1339, 1249, 1094; HRMS calcd for $(C_{25}H_{38}OSSi)Na^{+}$ 437.2304, found 437.2308.



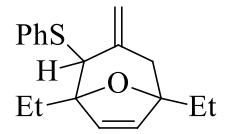
10a/b

((1R,2S,5R)-3-methylenebicyclo[3.2.1]oct-6-en-2-yl)(phenyl)sulfane. Isolated as a colorless oil in 72% yield (0.022 g). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.46-7.44 (m, 1H, minor isomer), 7.41-7.40 (m, 2H, major isomer), 7.30-7.23 (m, 2H, major isomer, 3H, minor isomer), 7.19 (t, 1H, major isomer, *J*=7.5 Hz), 5.99 major isomer (s, 2H), 5.94 (dd, 1H, minor isomer, *J*=5.5, 2.6 Hz), 5.90 (dd, 1H, minor isomer, *J*=5.5, 2.6 Hz), 5.32 (s, 1H, major isomer), 4.89 (s, 1H, major isomer), 4.60 (s, 1H, minor isomer), 4.38 (s, 1H, minor isomer), 3.94 (s, 1H, major isomer), 3.69 (s, 1H, minor isomer), 2.83-2.82 (m, 1H, major isomer), 2.79 (dt, 1H, minor isomer, *J*=5.0, 2.8 Hz), 2.66-2.61 (m, 1H, major isomer; 1H, minor isomer), 2.39 (d, 1H, major isomer, *J*=14.5 Hz), 2.28 (dt, 1H, major isomer, *J*=14.5, 2.5 Hz), 2.17 (d, 1H, minor isomer, *J*=11.0 Hz), 2.07-2.02 (m, 1H, major isomer), 1.80-1.71 minor isomer (m, 1H, minor isomer), 1.57 major isomer (d, 1H, major isomer, *J*=10.5 Hz); ^{13}C NMR (125 MHz, $CDCl_3$) δ 144.4 (C), 144.1 (C), 136.4 (C), 136.1 (CH), 136.0 (CH), 135.5 (C), 134.0 (CH), 133.5 (CH), 131.6 (CH), 130.6 (CH), 128.9 (CH), 128.7 (CH), 127.5 (CH), 126.2 (CH), 115.7 (CH), 115.3 (CH), 54.3 (2xCH), 45.2 (CH), 44.4 (CH_2), 44.0 (CH), 39.44 (CH), 39.37 (CH), 38.0 (CH_2), 37.6 (CH_2), 34.2 (CH_2).



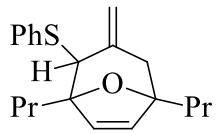
16a

1,5-dimethyl-3-methylene-2-(phenylthio)-8-oxabicylco[3.2.1]oct-6-ene. Isolated as a colorless oil in 85% yield (0.012 g). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.40 (d, *J*=8.0 Hz, 2H, 7.29 (t, *J*=7.5 Hz, 2H), 7.19 (t, *J*=7.5 Hz, 1H), 5.9 (d, *J*=6.0 Hz, 1H), 5.95 (d, *J*=6.0 Hz, 1H), 5.61 (s, 1H), 5.04 (s, 1H), 3.83 (s, 1H), 2.55 (d, *J*=14.0 Hz, 1H), 2.34 (d, *J*=14.0 Hz, 1H), 1.57 (s, 3H), 1.42 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 143.0, 137.6, 136.4, 133.0, 129.1, 128.8, 125.8, 116.0, 88.4, 84.8, 59.1, 43.3, 23.1, 22.5; IR (cm^{-1}) 3072, 2974, 2925, 1944, 1612, 1585, 1474, 1437, 1380, 1339, 1086; HRMS calcd for $(C_{16}H_{18}OS)Na^{+}$ 281.0970, found 281.0974.



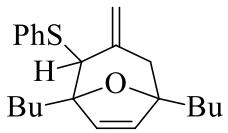
16b

1,5-diethyl-3-methylene-2-(phenylthio)-8-oxabicylco[3.2.1]oct-6-ene. Isolated as a colorless oil in 82% yield (0.020 g). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.38 (d, *J*=8.5Hz, 2H), 7.28 (t, *J*=7.5 Hz, 2H), 7.18 (t, *J*=7.5Hz, 1H), 5.93 (d, *J*=2.5 Hz, 2H), 5.61 (d, *J*=2.0 Hz, 1H), 5.04 (d, *J*= 2.0 Hz, 1H), 3.85 (s, 1H), 2.50 (d, *J*=14.5 Hz, 1H), 2.34 (d, *J*=14.5 Hz, 1H), 2.28-2.24 (m, 1H), 1.79-1.74 (m, 1H), 1.73-1.70 (m, 2H), 0.96 (t, *J*=7.5 Hz, 3H), 0.88 (t, *J*=7.5 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 143.4, 137.7, 135.3, 132.2, 128.9, 128.8, 125.7, 115.8, 91.1, 88.0, 58.2, 42.1, 29.2, 27.4, 8.1, 7.9; IR (cm^{-1}) 3076, 2953, 2925, 2868, 1944, 1797, 1634, 1580, 1482, 1437, 1380, 1335, 1090, 1025; HRMS calcd for $(C_{18}H_{22}OS)Na^{+}$ 309.1283, found 309.1286.



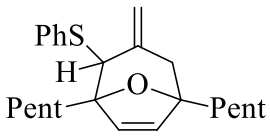
16c

1,5-dipropyl-3-methylene-2-(phenylthio)-8-oxabicylco[3.2.1]oct-6-ene. Isolated as a colorless oil in 79% yield (0.024 g). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.38 (d, *J*=7.5 Hz, 2H), 7.29 (t, *J*=7.5 Hz, 2H), 7.18 (t, *J*=7.5 Hz, 1H), 5.92 (d, *J*=6.5 Hz, 2H), 5.91 (d, *J*=1.5 Hz, 1H), (d, *J*=1.5 Hz, 1H), 3.84 (s, 1H), 2.53 (d, *J*=14.0 Hz, 1H), 2.28 (d, *J*=14.0 Hz, 1H), 2.21 (m, 1H), 1.73-1.68 (m, 1H), 1.67-1.61 (m, 2H), 1.52-1.40 (m, 1H), 1.39-1.25 (m, 4H), 0.98 (t, *J*=7.5 Hz, 3H), 0.88 (d, *J*=7.5 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 143.3, 137.7, 135.2, 132.2, 128.8, 128.7, 125.7, 115.9, 90.6, 87.6, 58.3, 42.4, 38.8, 36.9, 17.2, 17.0, 14.4, 14.3; IR (cm^{-1}) ; HRMS calcd for $(C_{18}H_{22}OS)Na^{+}$ 337.1596, found 337.1599.



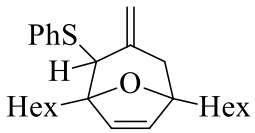
16d

1,5-dibutyl-3-methylene-2-(phenylthio)-8-oxabicylco[3.2.1]oct-6-ene. Isolated as a colorless oil in 76% yield (0.015 g). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.38 (d, *J*=8.0 Hz, 2H), 7.29 (t, *J*=7.5 Hz, 2H), 7.18 (t, *J*=7.5 Hz, 1H), 5.92 (d, *J*=6.5 Hz, 2H), 5.60 (d, *J*=1.5 Hz, 1H), 5.03 (d, *J*=1.5 Hz, 1H), 3.85 (s, 1H), 2.52 (d, *J*=14.5 Hz, 1H), 2.33 (d, *J*=14.0 Hz, 1H), 2.25-2.18 (m, 1H), 1.74-1.70 (m, 1H), 1.69-1.64 (m, 1H), 1.40-1.35 (m, 4H), 1.33-1.17 (m, 4H), 0.93 (t, *J*=7.0 Hz, 3H), 0.85 (t, *J*= 7.0 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 143.4, 137.7, 135.5, 132.3, 129.0, 128.7, 125.7, 115.9, 90.7, 87.6, 58.3, 42.4, 36.2, 34.4, 26.0, 25.8, 23.0, 22.8, 13.9, 13.8; IR (cm^{-1}) 3064, 2962, 2929, 2876, 1928, 1580, 1474, 1437, 1249, 1139, 1090, 1021; HRMS calcd for $(C_{22}H_{30}OS)Na^{+}$ 365.1909, found 365.1912.



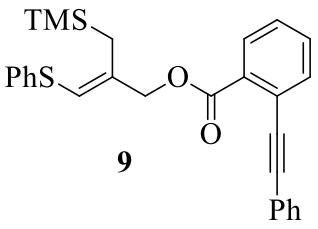
16e

1,5-dipentyl-3-methylene-2-(phenylthio)-8-oxabicylco[3.2.1]oct-6-ene. Isolated as a colorless oil in 71% yield (0.016 g). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.38 (d, *J*=8.0 Hz, 2H), (t, *J*=7.5 Hz, 2H), 7.18 (t, *J*=7.5 Hz, 1H), 5.92 (d, *J*=6.5 Hz, 2H), 5.59 (d, *J*=1.5 Hz, 1H), 5.03 (d, *J*=1.5 Hz, 1H), 3.85 (s, 1H), 2.51 (d, *J*=14.0 Hz, 1H), 2.33 (d, *J*=14.0 Hz, 1H), 2.23-2.17 Hz (m, 1H), 1.74-1.71 (m, 1H), 1.70-1.68 (m, 1H), 1.58-1.47 (m, 1H), 1.47-1.32 (m, 6H), 1.20-1.31 (m, 6H), 0.91 (t, *J*=6.5 Hz, 3H), 0.85 (t, *J*=6.5 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 143.4, 137.7, 135.5, 132.3, 129.0, 128.7, 125.7, 115.9, 90.7, 87.6, 58.3, 42.4, 36.5, 34.6, 32.1, 32.0, 23.5, 23.3, 22.4, 13.9; IR (cm^{-1}) 3068, 2966, 2933, 2872, 1940,1883, 1797, 1634, 1585, 1478, 1433,1380, 1339, 1245, 1017; HRMS calcd for $(C_{24}H_{34}OS)Na^{+}$ 393.2222, found 393.2225.

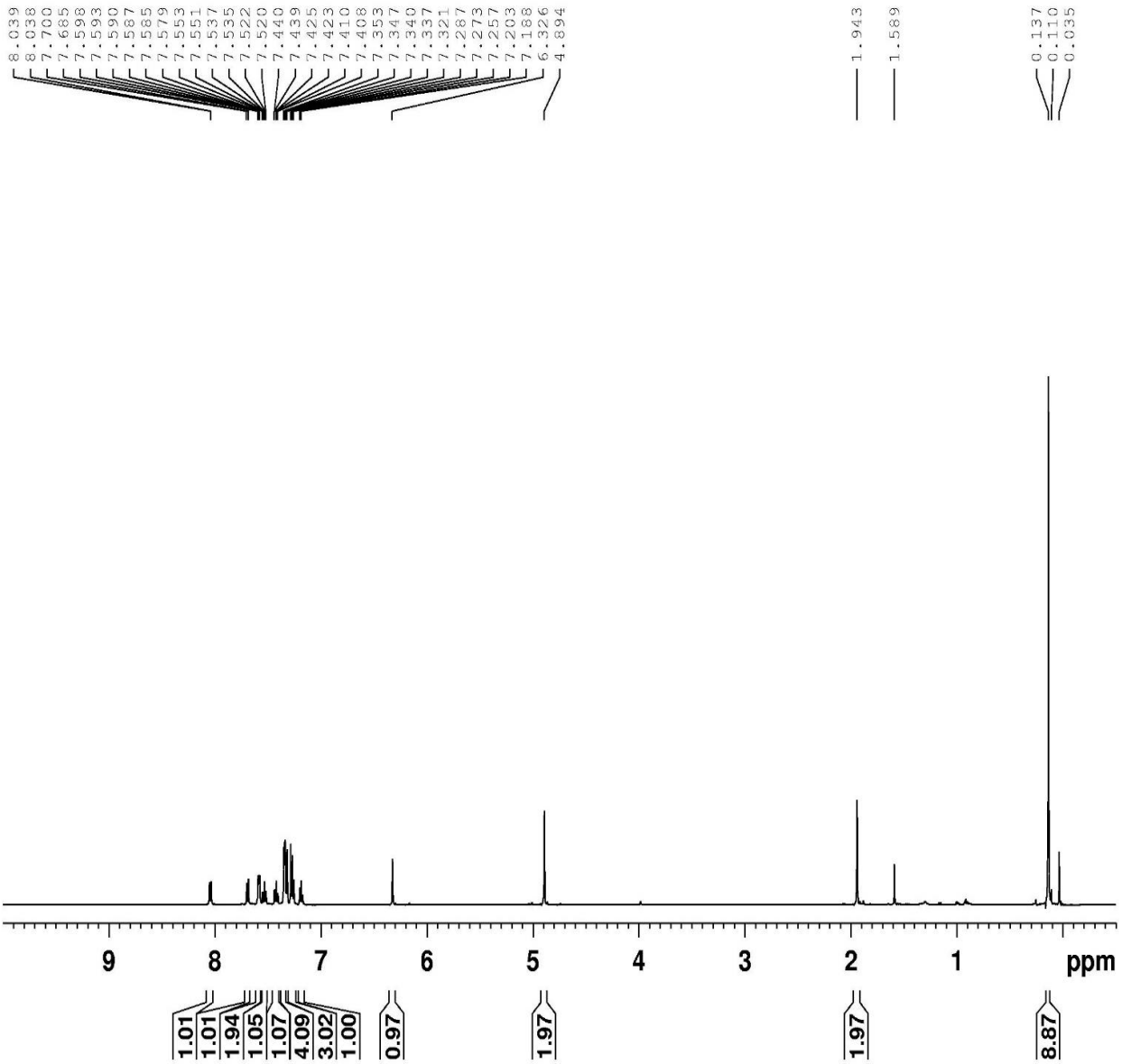


16f

1,5-dihexyl-3-methylene-2-(phenylthio)-8-oxabicylco[3.2.1]oct-6-ene. Isolated as a colorless oil in 65% yield (0.013 g). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.38 (d, *J*=8.0 Hz, 2H), 7.28 (t, *J*=8.0 Hz, 2H), 7.18 (t, *J*=7.5 Hz, 1H), 5.91 (d, *J*=6.0 Hz, 2H), 5.59 (d, *J*=1.0 Hz, 1H), 5.03 (d, *J*=1.0 Hz, 1H), 3.85 (s, 1H), 1.74-1.69 (m, 1H), 1.68-1.64 (m, 1H), 1.48-1.42 (m, 1H), 1.36-1.22 (m, 16H), 0.90 (t, *J*=6.0 Hz, 3H), 0.85 (t, *J*=6.0 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 143.4, 137.7, 135.2, 132.3, 129.0, 128.7, 125.7, 115.9, 90.7, 87.6, 58.3, 42.4, 36.5, 34.6, 31.6, 29.5, 29.4, 23.8, 23.6, 22.5, 22.4, 13.9; IR (cm^{-1}) 3072, 3051, 2962, 2925, 2872, 1936, 1862, 1785, 1634, 1580, 1478, 1433, 1376, 1339, 1254, 1147, 1086; HRMS calcd for $(C_{26}H_{38}OS)Na^{+}$ 421.2535, found 421.2538.


TMS
PhS
O
O
Ph

9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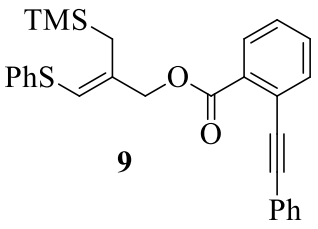
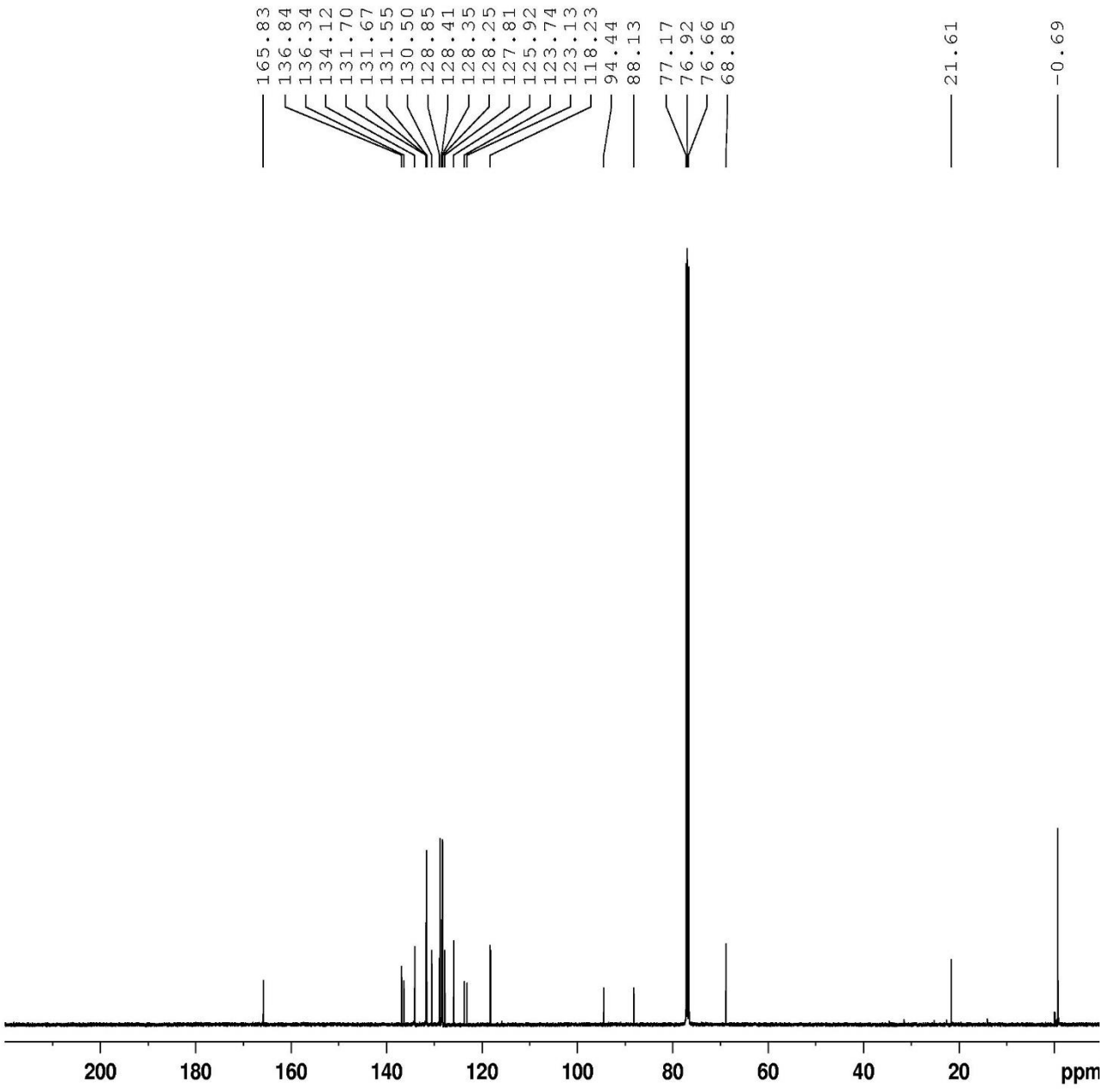
8.038
7.700
7.685
7.598
7.593
7.590
7.587
7.585
7.579
7.553
7.551
7.537
7.535
7.522
7.520
7.440
7.439
7.425
7.423
7.410
7.408
7.353
7.347
7.340
7.337
7.321
7.287
7.273
7.257
7.203
7.188
6.326
4.894
1.943
1.589
0.137
0.110
0.035
9
8
7
6
5
4
3
2
1
ppm
1.01
1.01
1.94
1.05
1.07
4.09
3.02
1.00
0.97
1.97
1.97
8.87


TMS
PhS
O
O
Ph

9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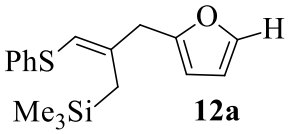
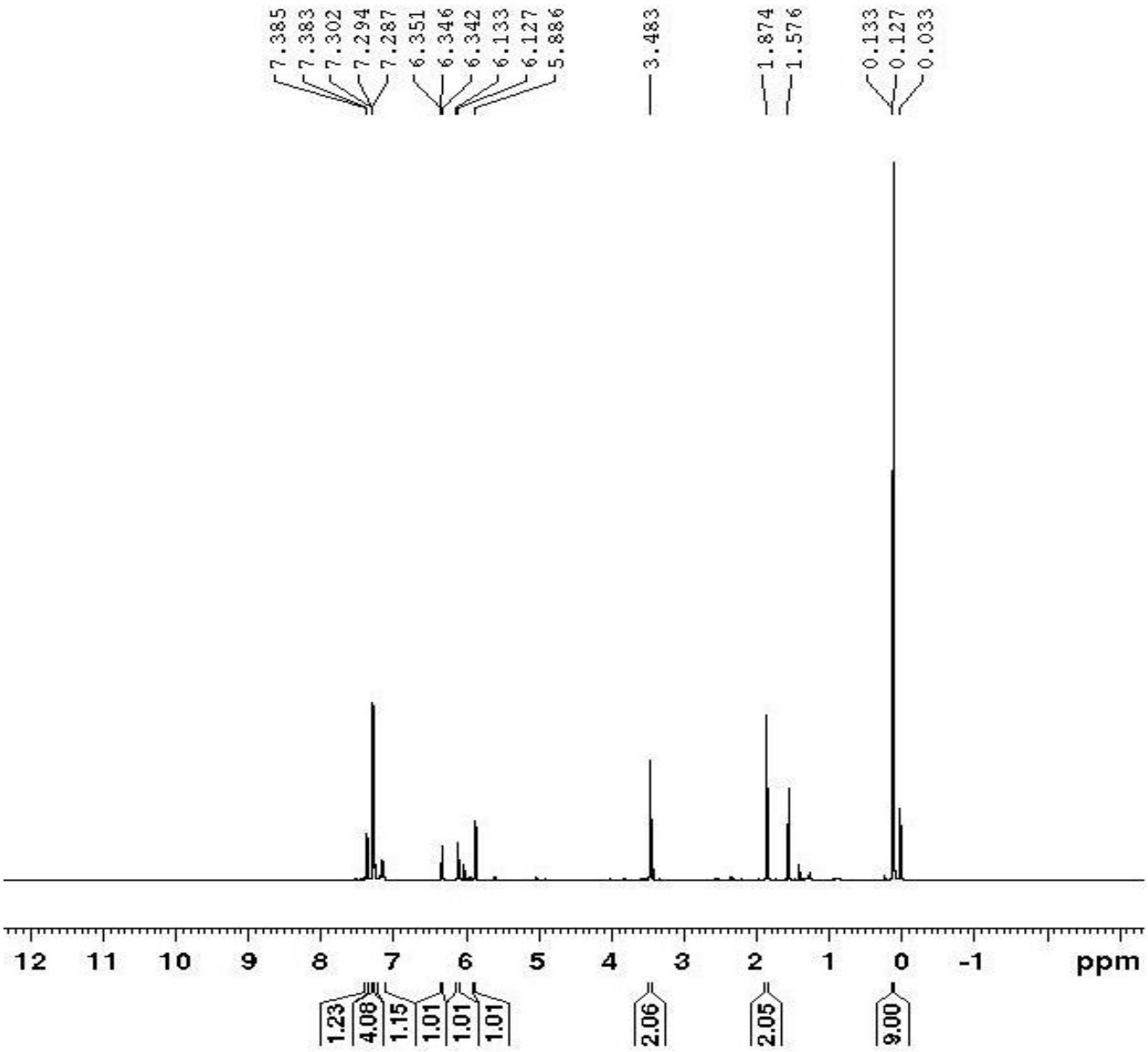
136.84
136.34
134.12
131.70
131.67
131.55
130.50
128.85
128.41
128.35
128.25
127.81
125.92
123.74
123.13
118.23
94.44
88.13
77.17
76.92
76.66
68.85
21.61
-0.69
200
180
160
140
120
100
80
60
40
20
ppm


PhS
Me3Si
O
H

12a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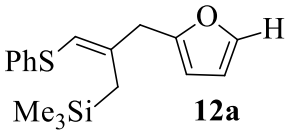
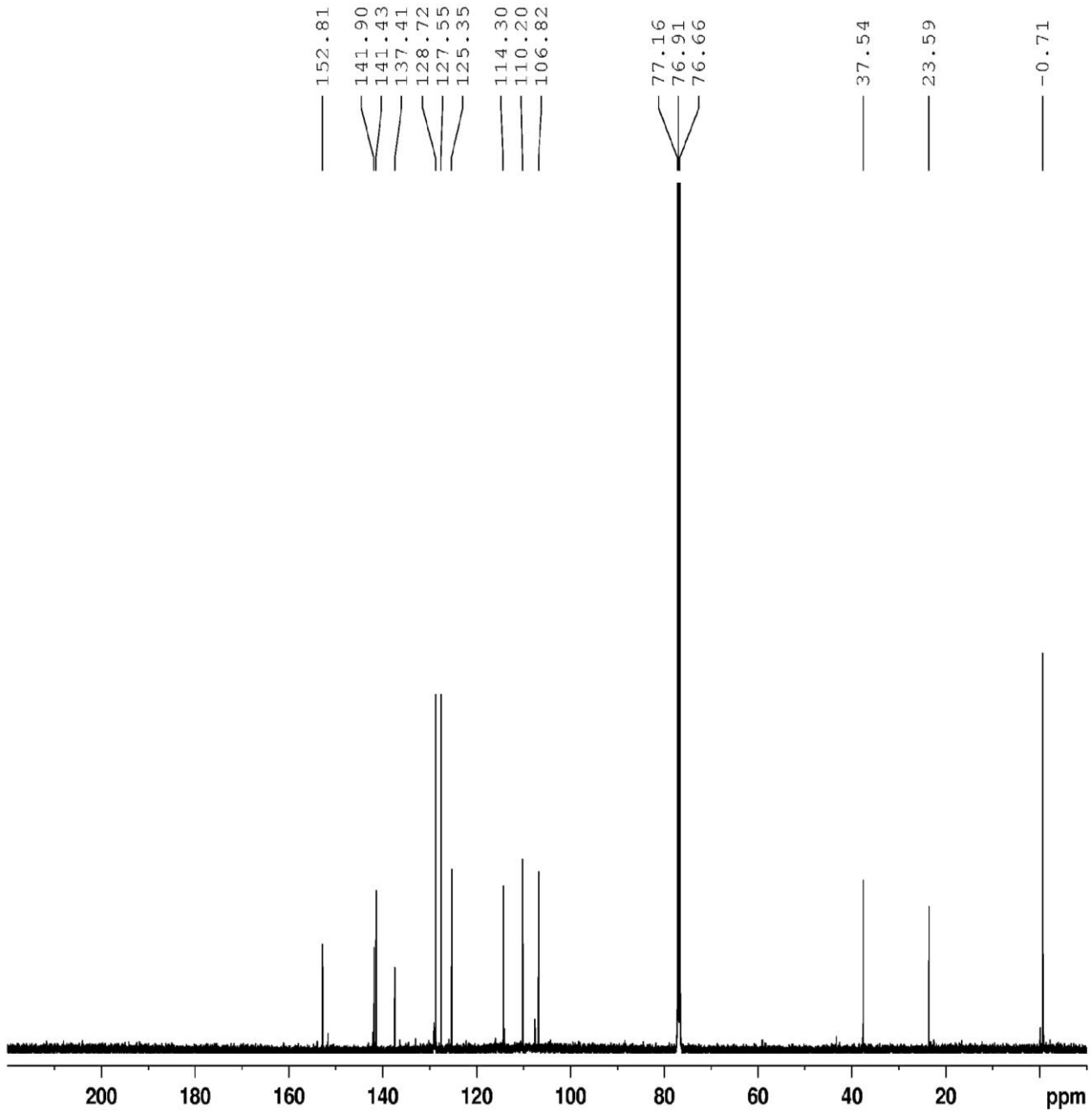
7.302
7.294
7.287
6.351
6.346
6.342
6.133
6.127
5.886
3.483
1.874
1.576
0.133
0.127
0.033
12 11 10 9 8 7 6 5 4 3 2 1 0 -1 ppm
1.23
4.08
1.15
1.01
1.01
1.01
2.06
2.05
9.00


PhS
Me3Si
O
H

12a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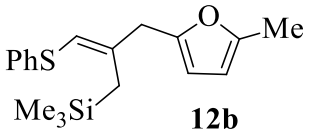
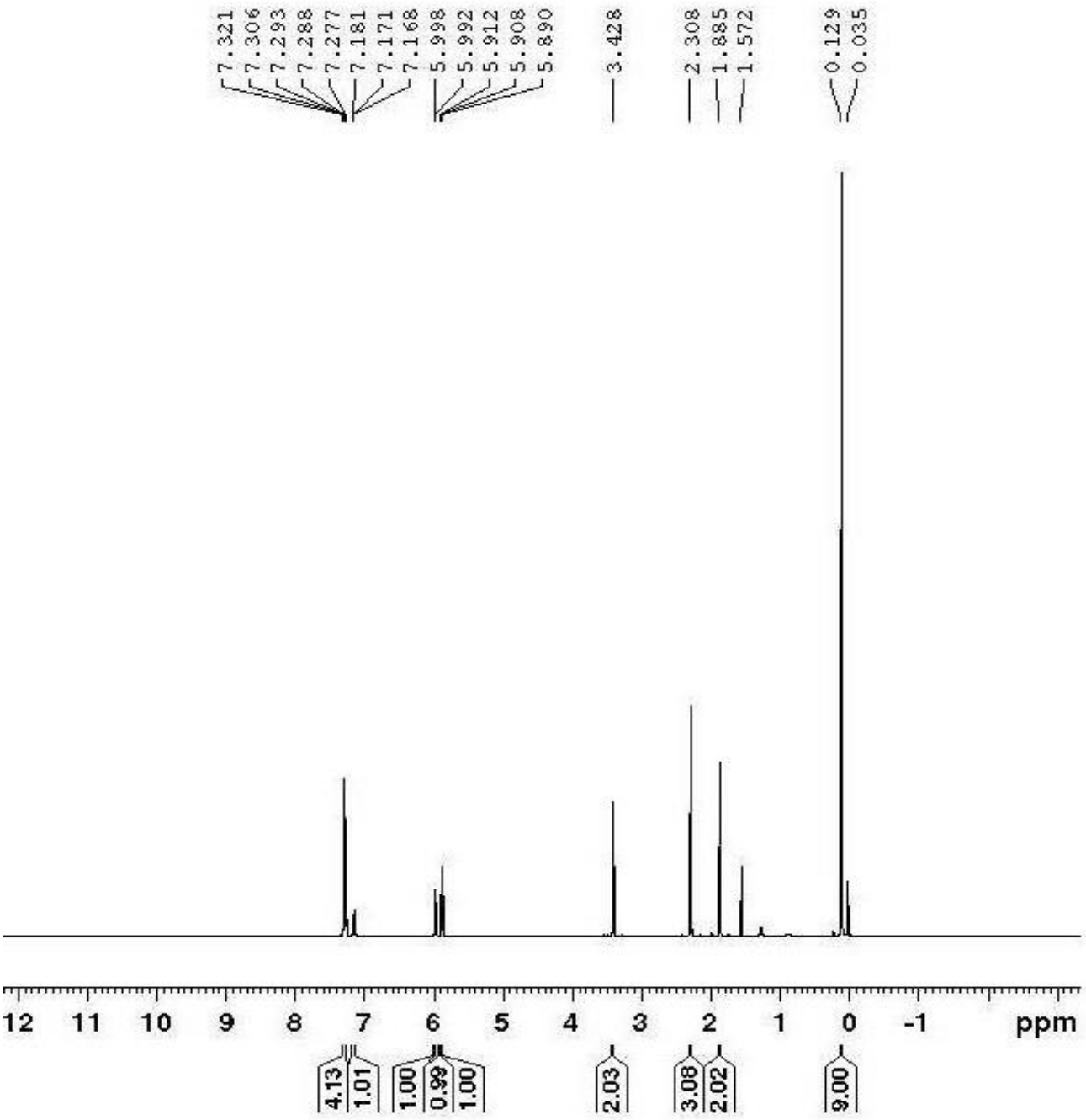
141.90
141.43
137.41
128.72
127.55
125.35
114.30
110.20
106.82
77.16
76.91
76.66
37.54
23.59
-0.71
200
180
160
140
120
100
80
60
40
20
ppm


PhS
Me$_3$Si
O
Me

12b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7.306
7.293
7.288
7.277
7.181
7.171
7.168
5.998
5.992
5.912
5.908
5.890
3.428
2.308
1.885
1.572
0.129
0.035
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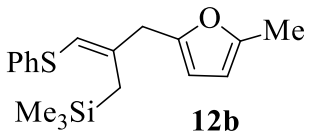
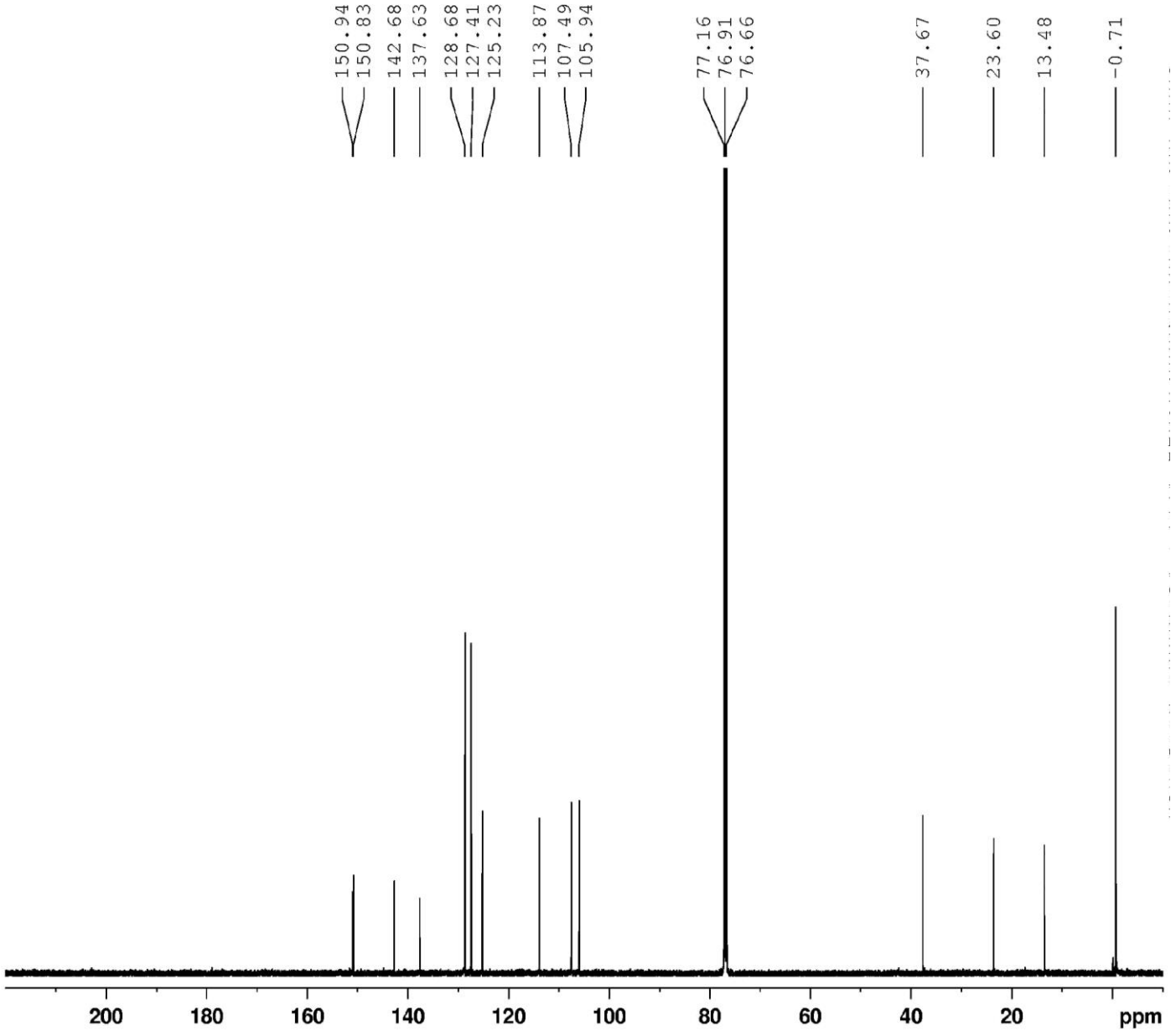
4.13
1.01
1.00
0.99
1.00
2.03
3.08
2.02
9.00


PhS
Me3Si
O
Me

12b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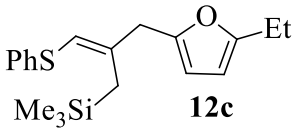
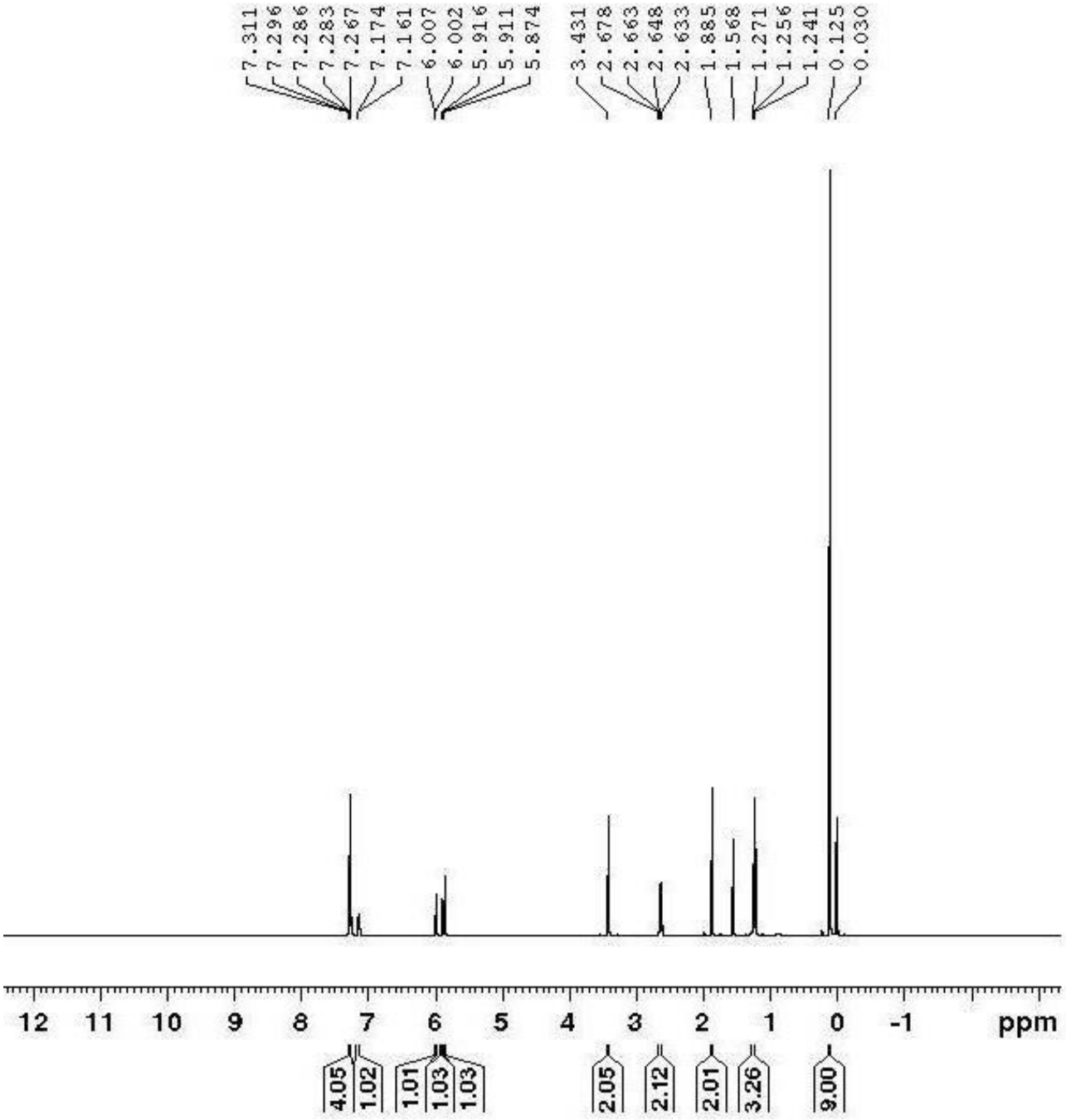
150.83
142.68
137.63
128.68
127.41
125.23
113.87
107.49
105.94
77.16
76.91
76.66
37.67
23.60
13.48
-0.71
200
180
160
140
120
100
80
60
40
20
ppm


PhS
Me3Si
O
Et

12c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7.267
7.174
7.161
6.007
6.002
5.916
5.911
5.874
3.431
2.678
2.663
2.648
2.633
1.885
1.568
1.271
1.256
1.241
0.125
0.030
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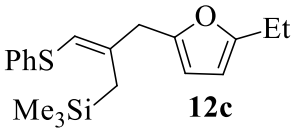
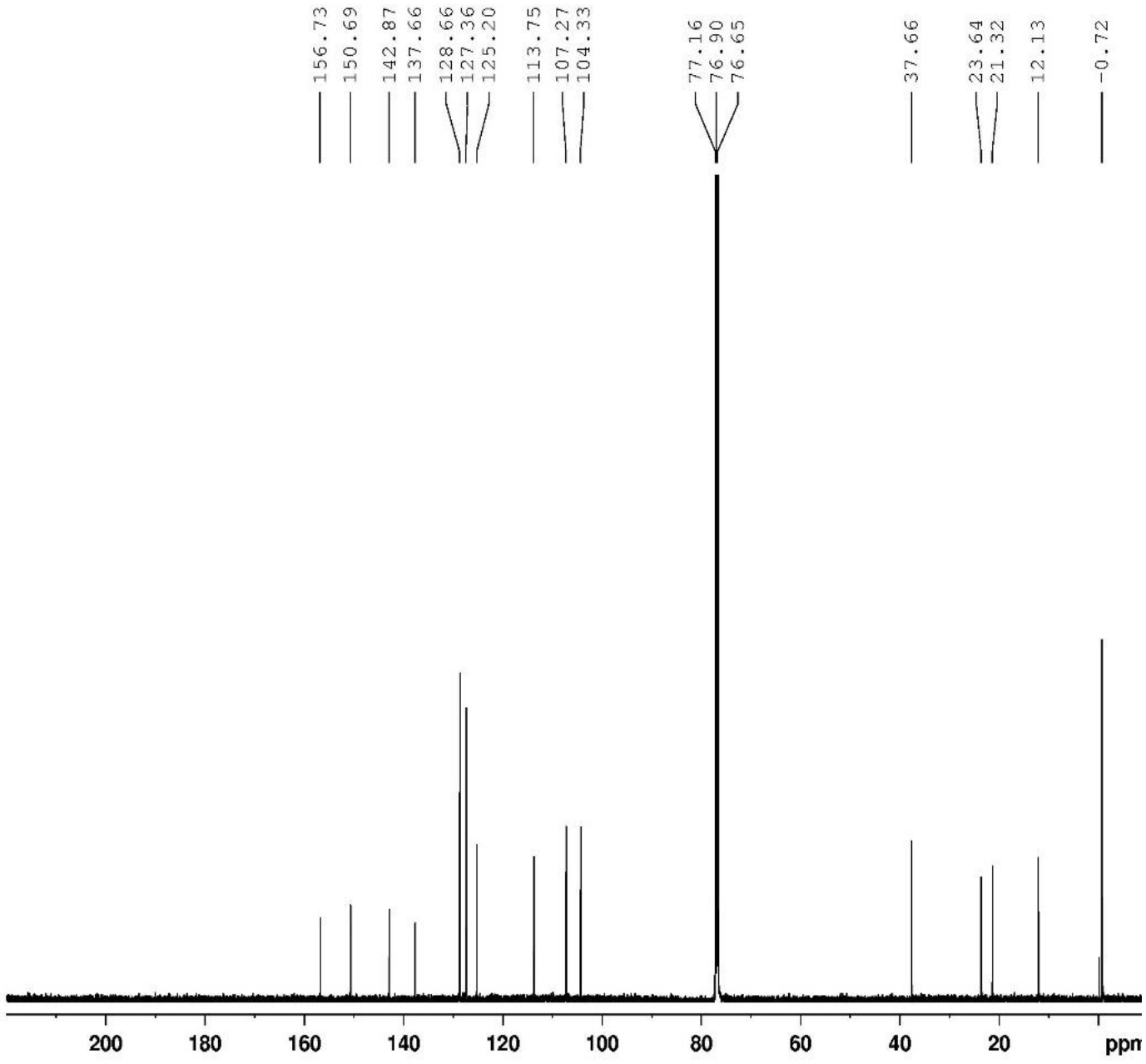
1.02
1.01
1.03
1.03
2.05
2.12
2.01
3.26
9.00


PhS
Me3Si
O
Et

12c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77.16
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76.65
37.66
23.64
21.32
12.13
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180
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140
120
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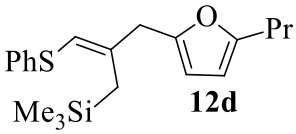
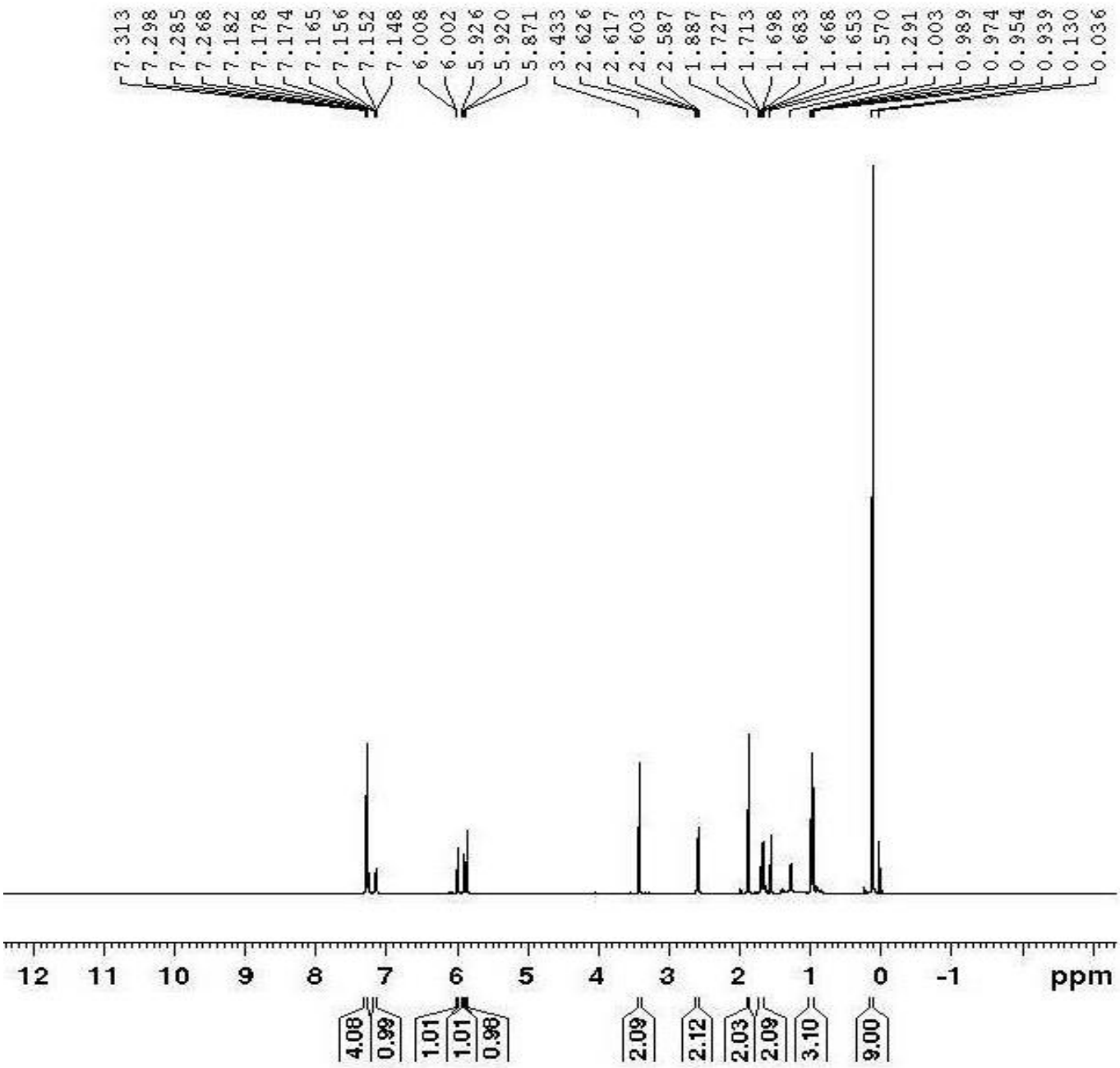
80
60
40
20
ppm


PhS
Me3Si
O
Pr

12d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7.182
7.178
7.174
7.165
7.156
7.152
7.148
6.008
6.002
5.926
5.920
5.871
3.433
2.626
2.617
2.603
2.587
1.887
1.727
1.713
1.698
1.683
1.668
1.653
1.570
1.291
1.003
0.989
0.974
0.954
0.939
0.130
0.036
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
ppm
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1.01
1.01
0.98
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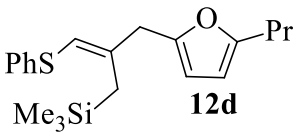
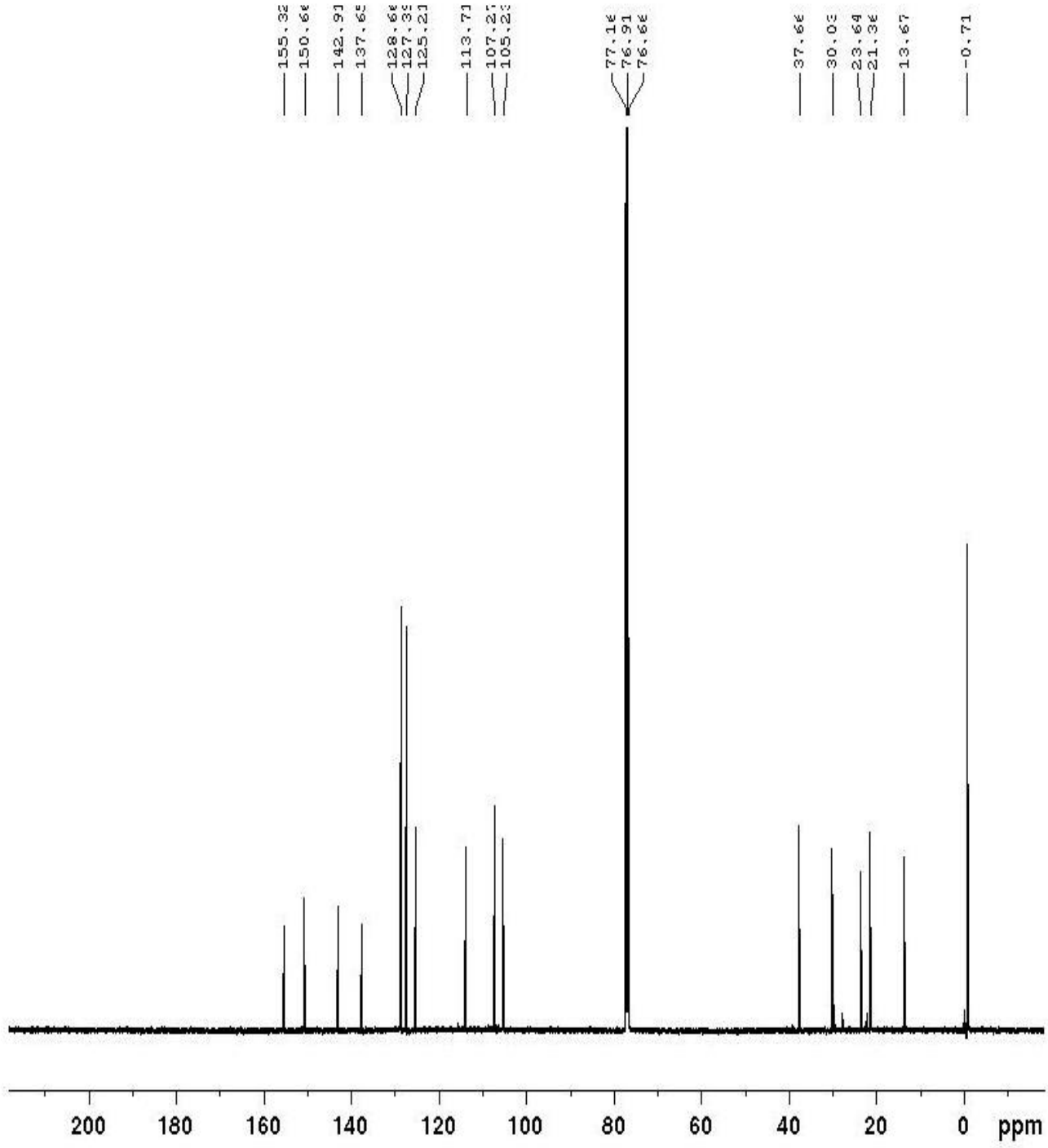
2.12
2.03
2.09
3.10
9.00


PhS
Me3Si
O
Pr

12d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127.35
125.21
113.71
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105.23
77.16
76.91
76.66
37.66
30.03
23.64
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13.67
-0.71
200
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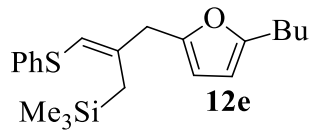
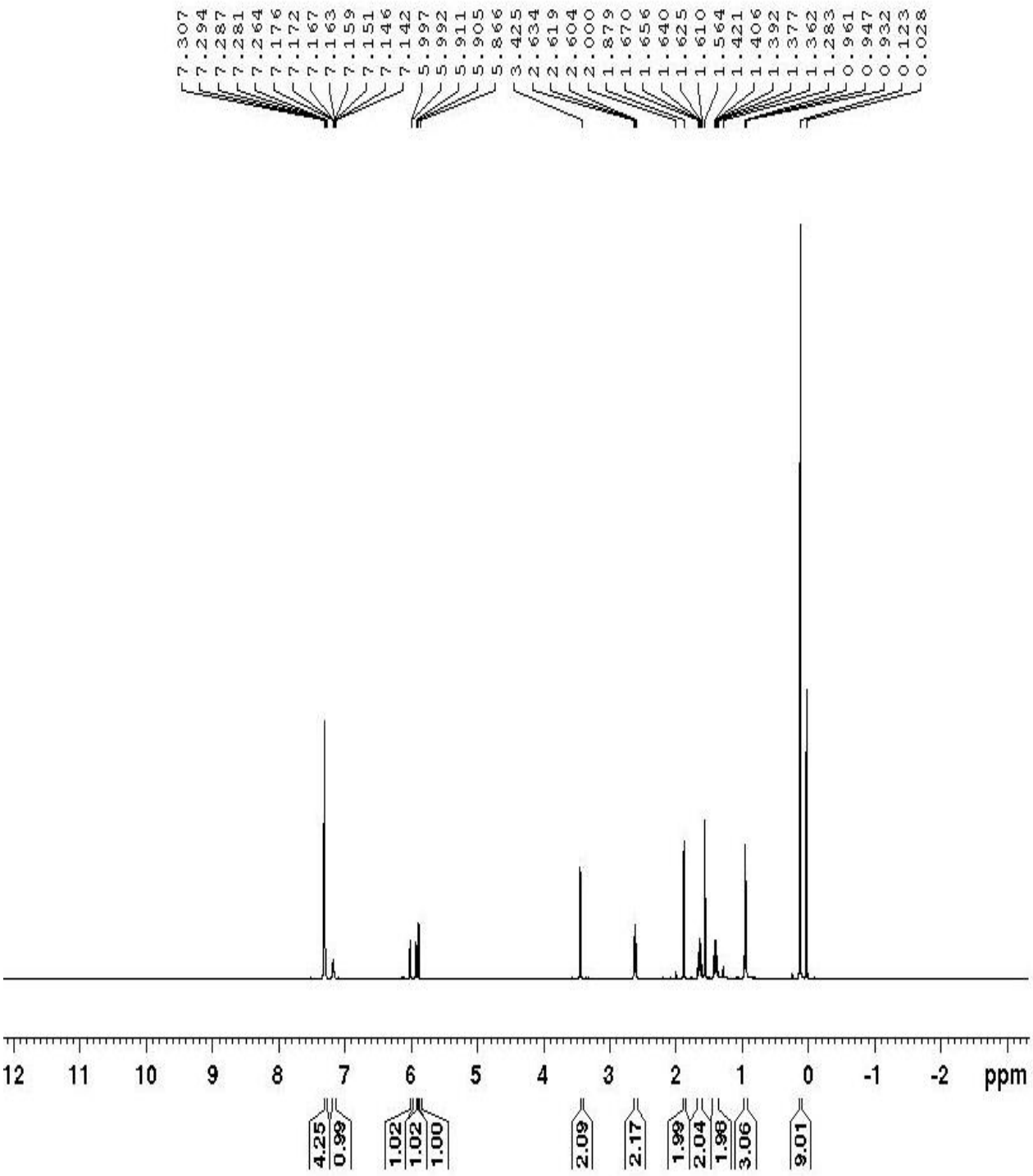
160
140
120
100
80
60
40
20
0
ppm


PhS
Me3Si
O
Bu

12e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7.264
7.176
7.172
7.167
7.163
7.159
7.151
7.146
7.142
5.997
5.992
5.911
5.905
5.866
3.425
2.634
2.619
2.604
2.000
1.879
1.670
1.656
1.640
1.625
1.610
1.564
1.421
1.406
1.392
1.377
1.362
1.283
0.961
0.947
0.932
0.123
0.028
12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 ppm
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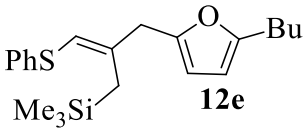
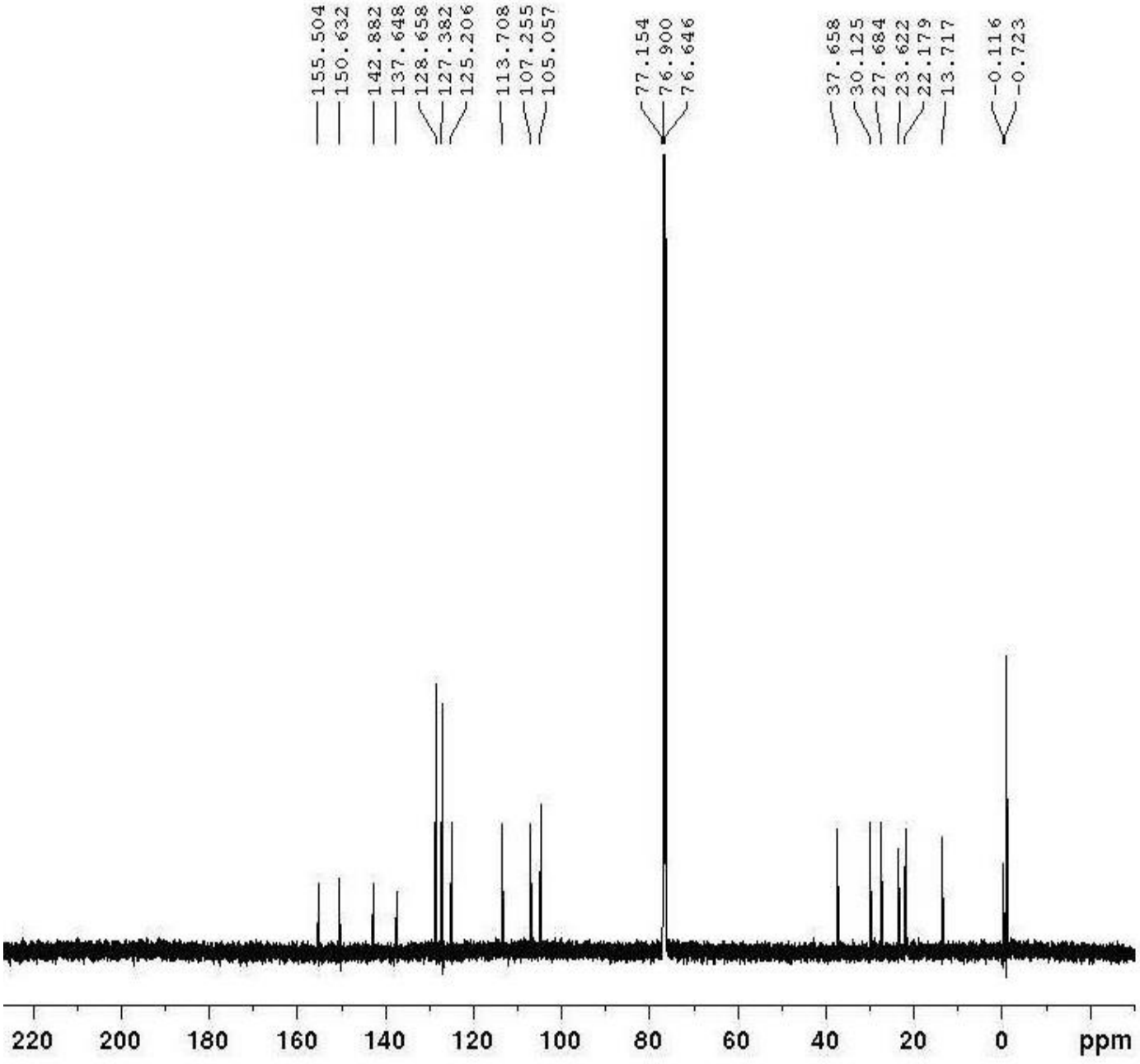
1.02
1.02
1.00
2.09
2.17
1.99
2.04
1.98
3.06
9.01


PhS
Me3Si
O
Bu

12e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113.708
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105.057
77.154
76.900
76.646
37.658
30.125
27.684
23.622
22.179
13.717
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-0.723
220
200
180
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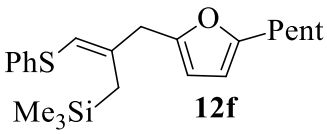
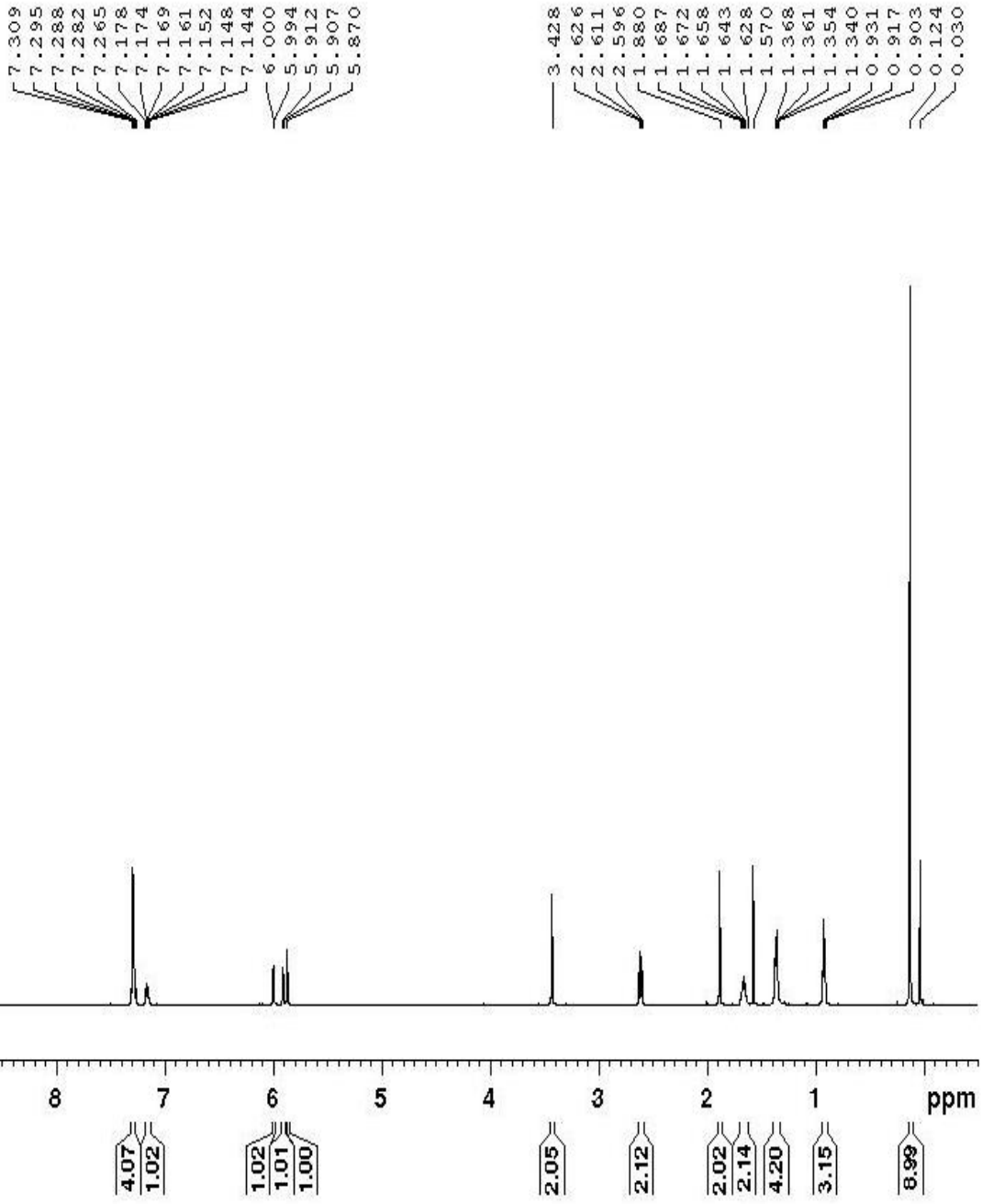
140
120
100
80
60
40
20
0
ppm


PhS
Me3Si
O
Pent

12f


7.309
7.295
7.288
7.282
7.265
7.178
7.174
7.169
7.161
7.152
7.148
7.144
6.000
5.994
5.912
5.907
5.870
3.428
2.626
2.611
2.596
1.880
1.687
1.672
1.658
1.643
1.628
1.570
1.368
1.361
1.354
1.340
0.931
0.917
0.903
0.124
0.030
9
8
7
6
5
4
3
2
1
ppm
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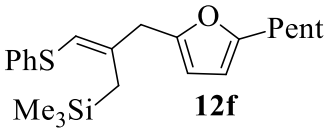
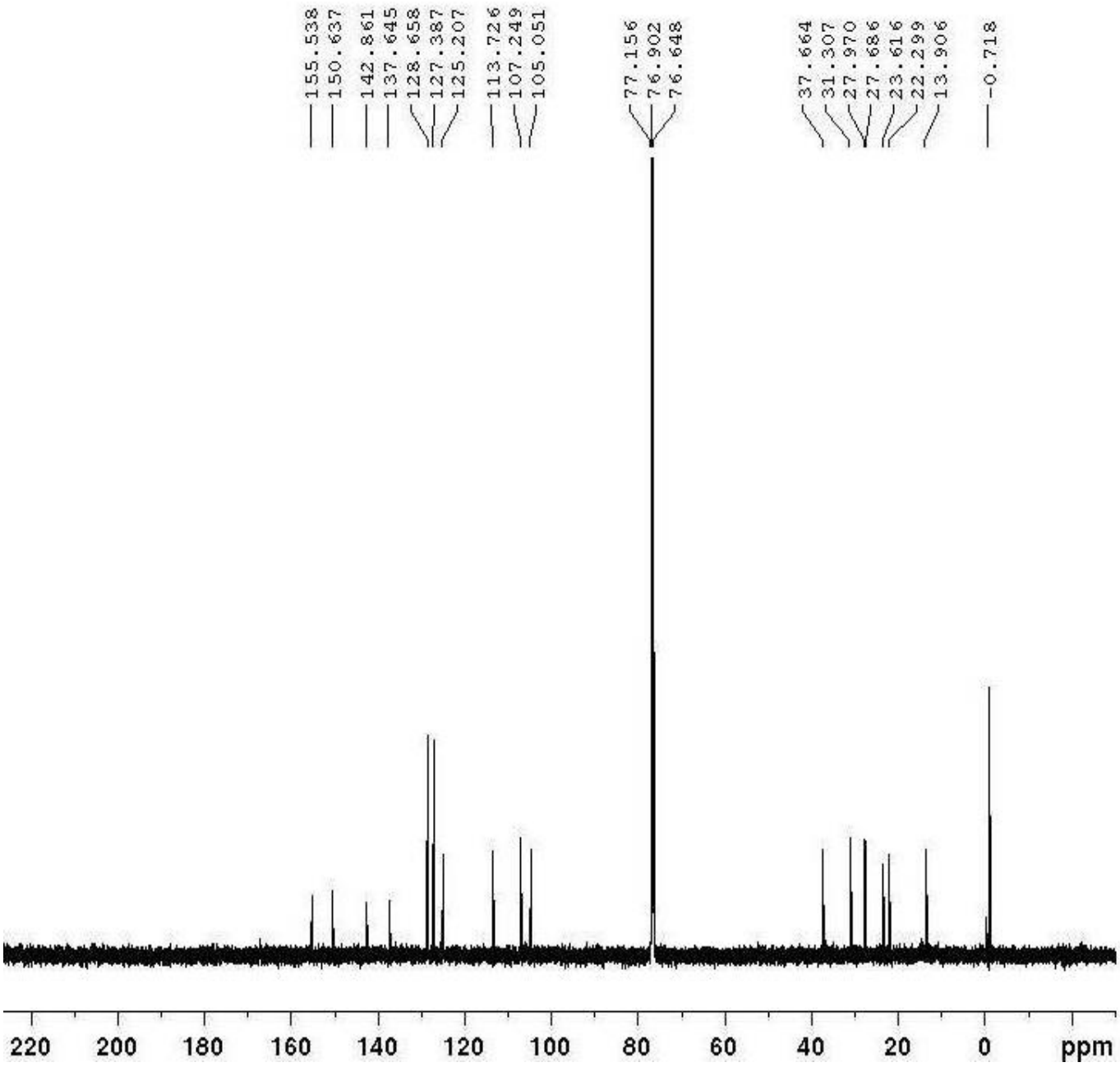
1.02
1.01
1.00
2.05
2.12
2.02
2.14
4.20
3.15
8.99


PhS
Me3Si
O
Pent

12f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76.902
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31.307
27.970
27.686
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22.299
13.906
-0.718
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200
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120
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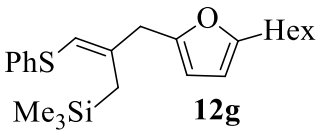
40
20
0
ppm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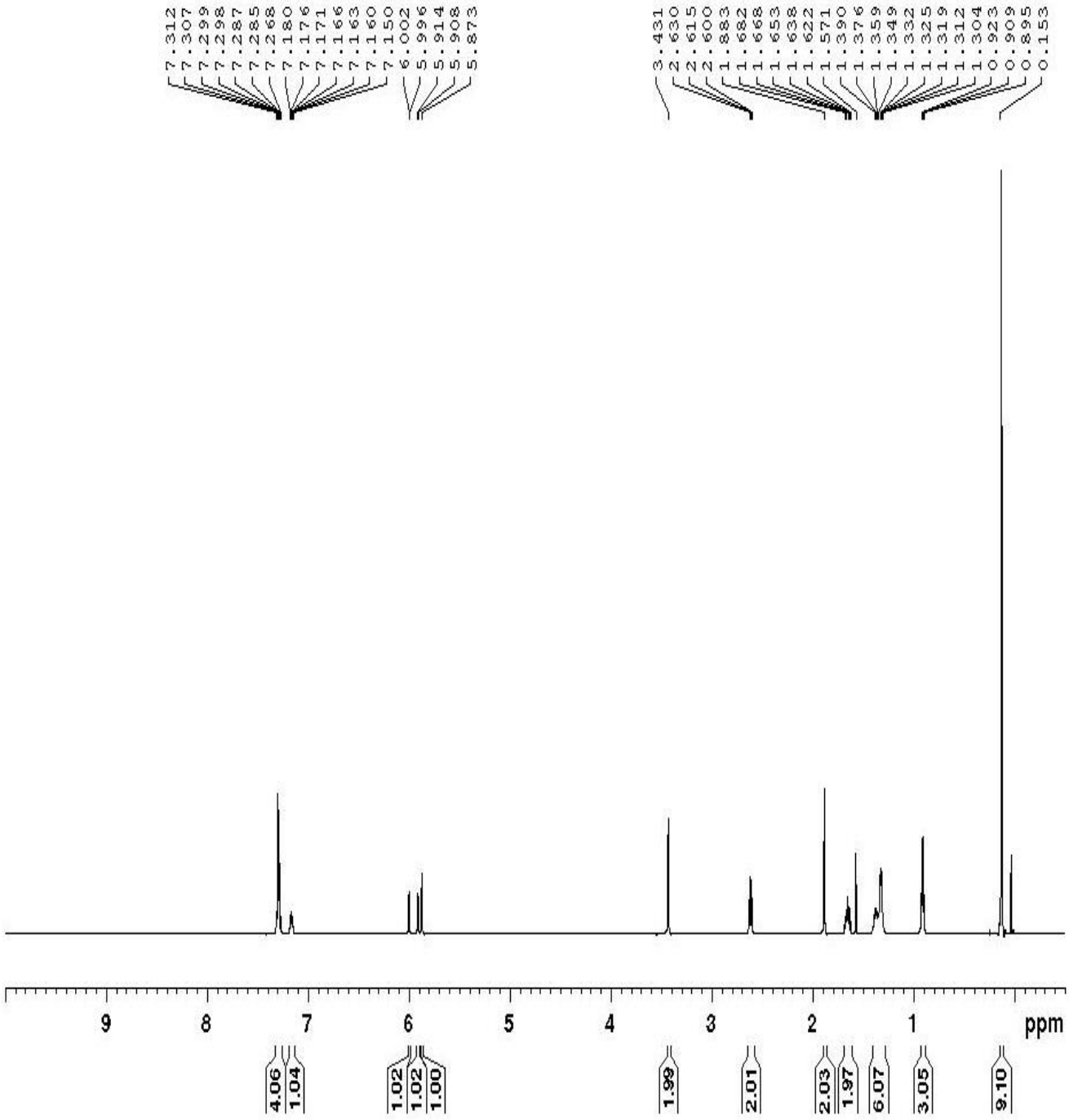
Me3Si
O
Hex

12g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7.180
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7.171
7.166
7.163
7.160
7.150
6.002
5.996
5.914
5.908
5.873
3.431
2.630
2.615
2.600
1.883
1.682
1.668
1.653
1.638
1.622
1.571
1.390
1.376
1.359
1.349
1.332
1.325
1.319
1.312
1.304
0.923
0.909
0.895
0.153
9
8
7
6
5
4
3
2
1
ppm
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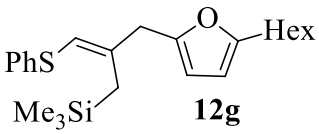
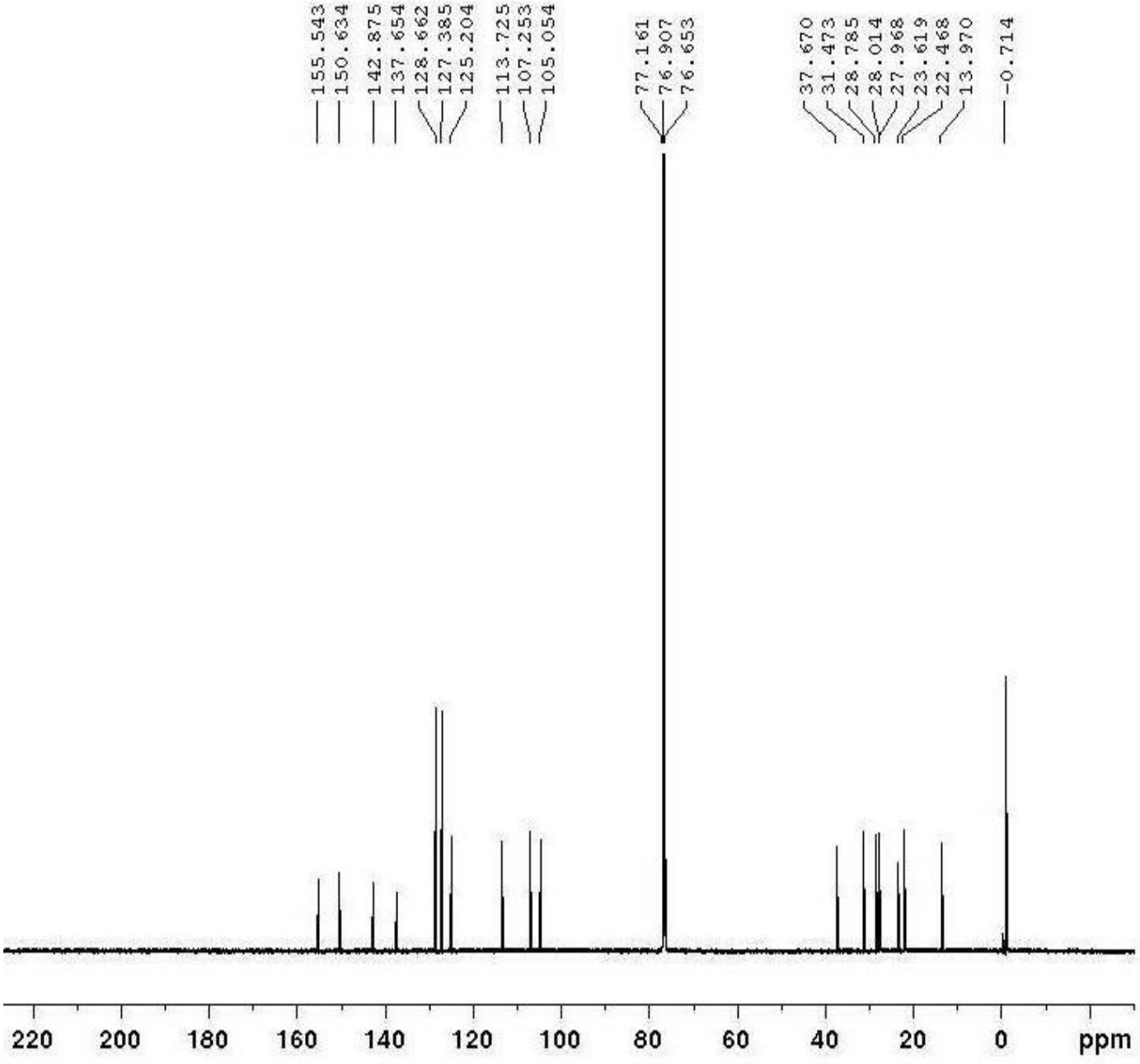
1.04
1.02
1.02
1.00
1.99
2.01
2.03
1.97
6.07
3.05
9.10


PhS
Me3Si
O
Hex

12g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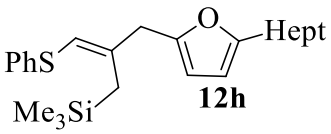
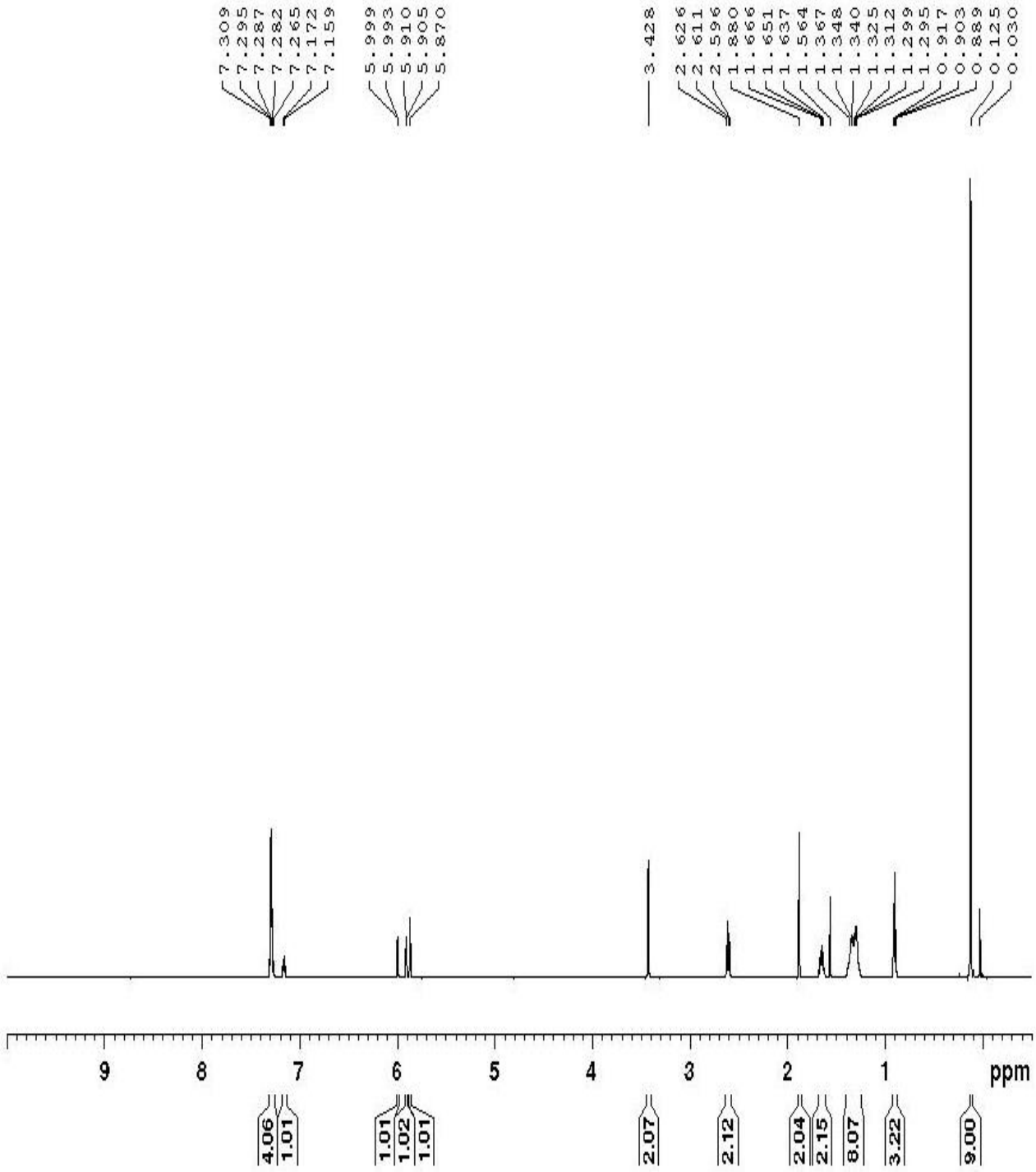
113.725
107.253
105.054
77.161
76.907
76.653
37.670
31.473
28.785
28.014
27.968
23.619
22.468
13.970
-0.714
220
200
180
160
140
120
100
80
60
40
20
0
ppm


PhS
Me3Si
O
Hept

12h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7.282
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7.172
7.159
5.999
5.993
5.910
5.905
5.870
3.428
2.626
2.611
2.596
1.880
1.666
1.651
1.637
1.564
1.367
1.348
1.340
1.325
1.312
1.299
1.295
0.917
0.903
0.889
0.125
0.030
9
8
7
6
5
4
3
2
1
ppm
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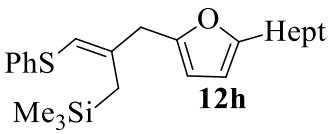
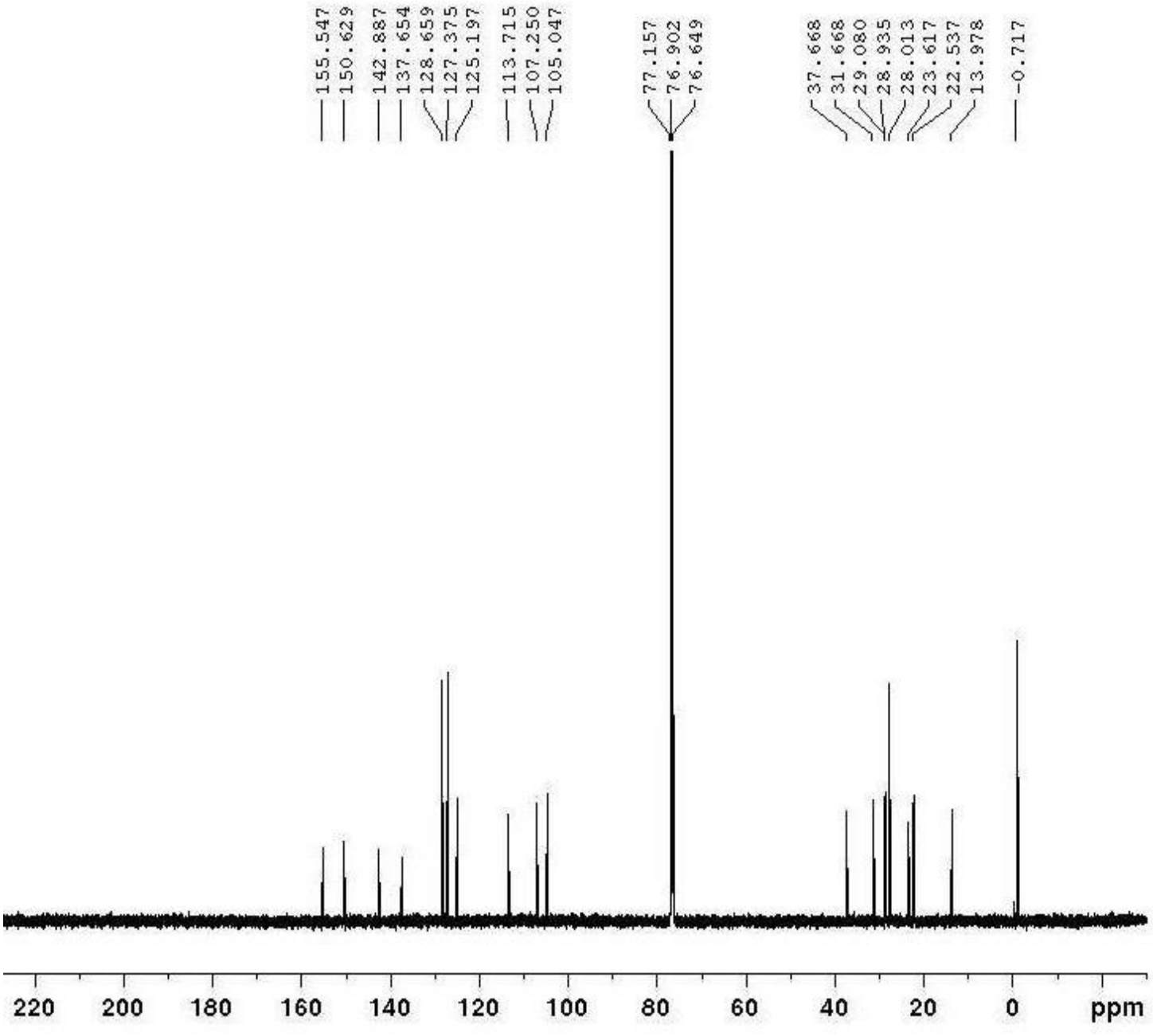
1.01
1.01
1.02
1.01
2.07
2.12
2.04
2.15
8.07
3.22
9.00


O
Hept
PhS
Me3Si

12h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113.715
107.250
105.047
77.157
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76.649
37.668
31.668
29.080
28.935
28.013
23.617
22.537
13.978
-0.717
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200
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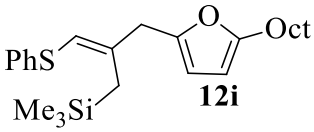
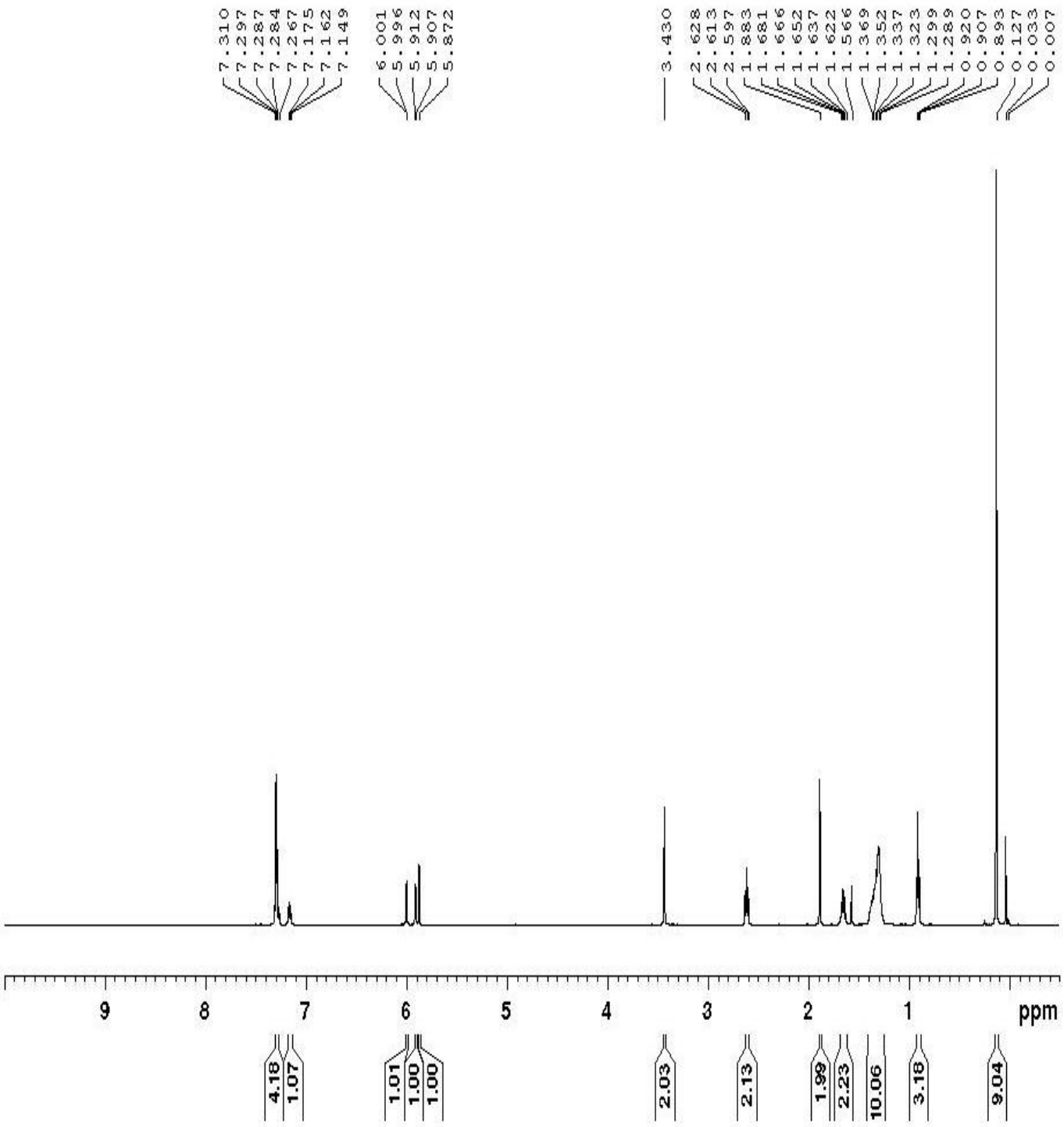
160
140
120
100
80
60
40
20
0
ppm


PhS
Me3Si
O
Oct

12i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7.284
7.267
7.175
7.162
7.149
6.001
5.996
5.912
5.907
5.872
3.430
2.628
2.613
2.597
1.883
1.681
1.666
1.652
1.637
1.622
1.566
1.369
1.352
1.337
1.323
1.299
1.289
0.920
0.907
0.893
0.127
0.033
0.007
9
8
7
6
5
4
3
2
1
ppm
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1.07
1.01
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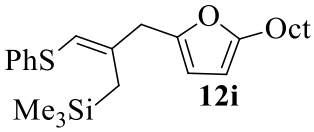
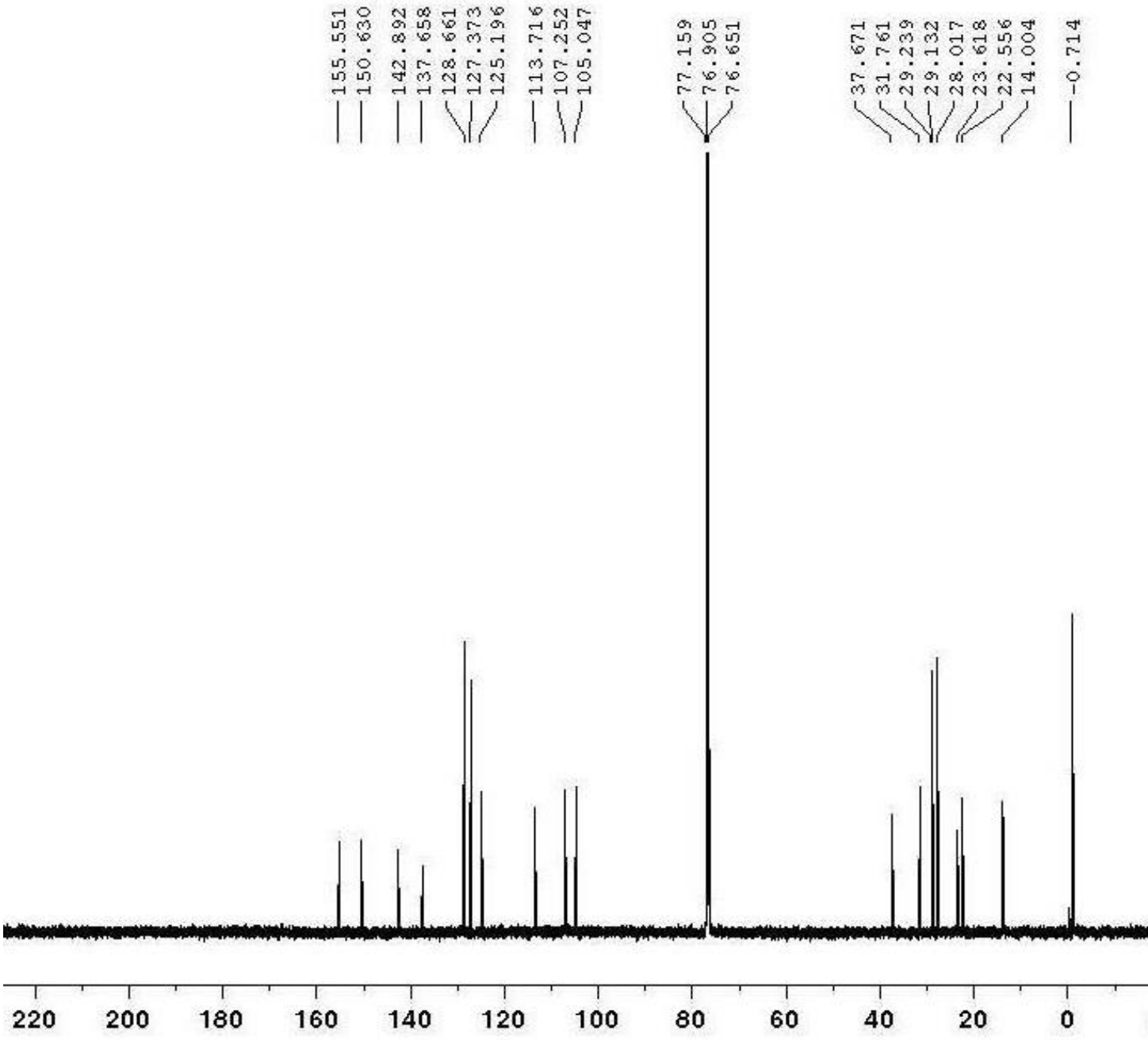
2.13
1.99
2.23
10.06
3.18
9.04


PhS
Me3Si
O
Oct

12i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113.716
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105.047
77.159
76.905
76.651
37.671
31.761
29.239
29.132
28.017
23.618
22.556
14.004
-0.714
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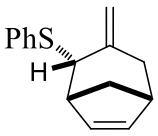
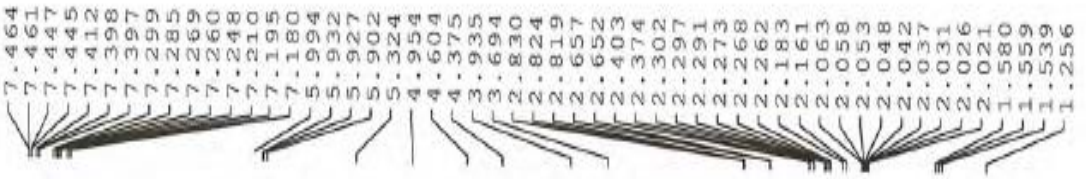
200
180
160
140
120
100
80
60
40
20
0


PhS
H

10a/b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7.299
7.285
7.269
7.260
7.248
7.210
7.195
7.180
5.994
5.932
5.927
5.902
5.324
4.954
4.604
4.375
4.935
3.694
2.830
2.824
2.819
2.657
2.652
2.403
2.374
2.302
2.297
2.291
2.273
2.268
2.262
2.183
2.161
2.063
2.058
2.053
2.048
2.042
2.037
2.031
2.026
2.021
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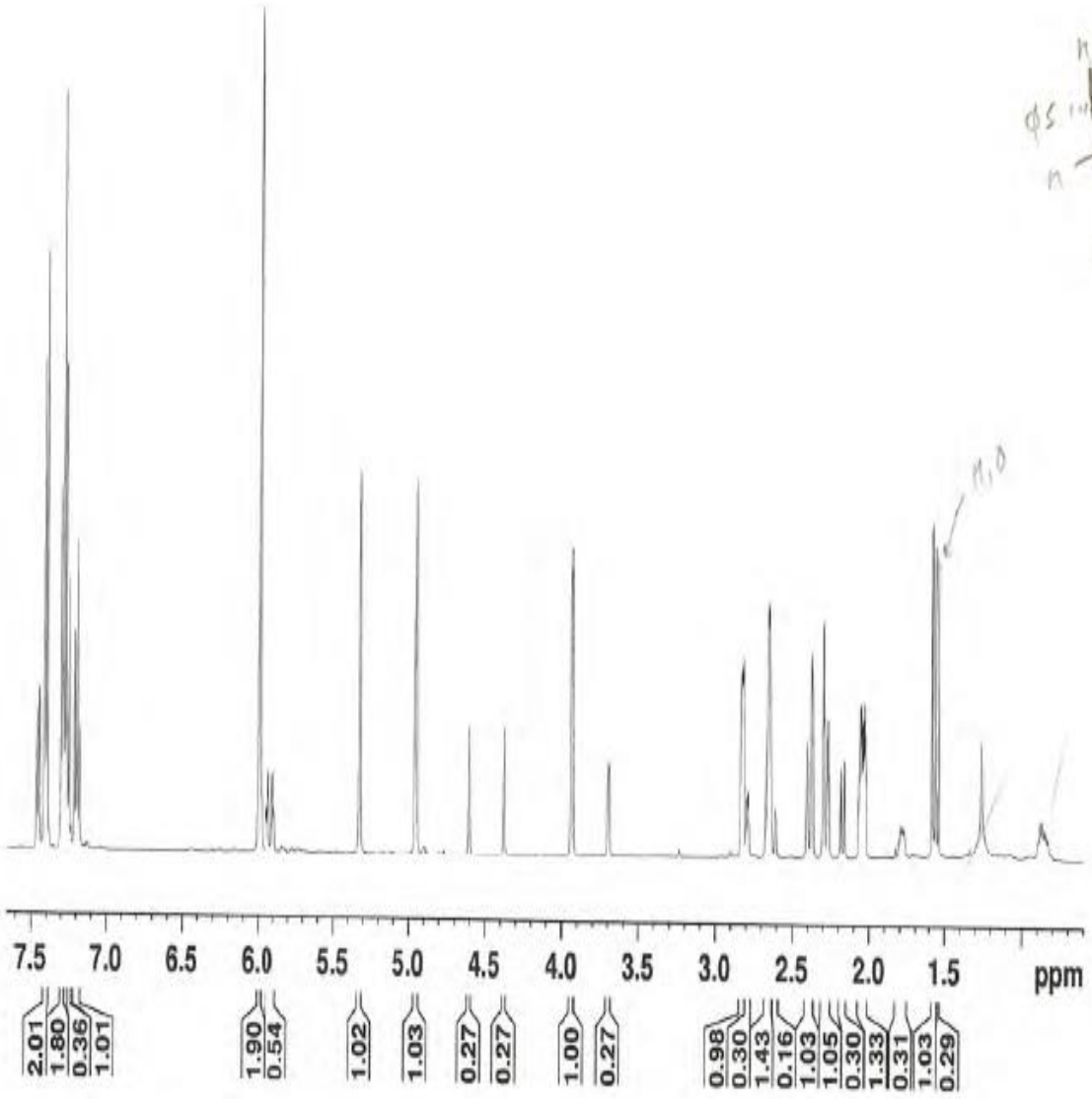
1.559
1.539
1.256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6.0
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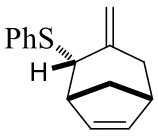
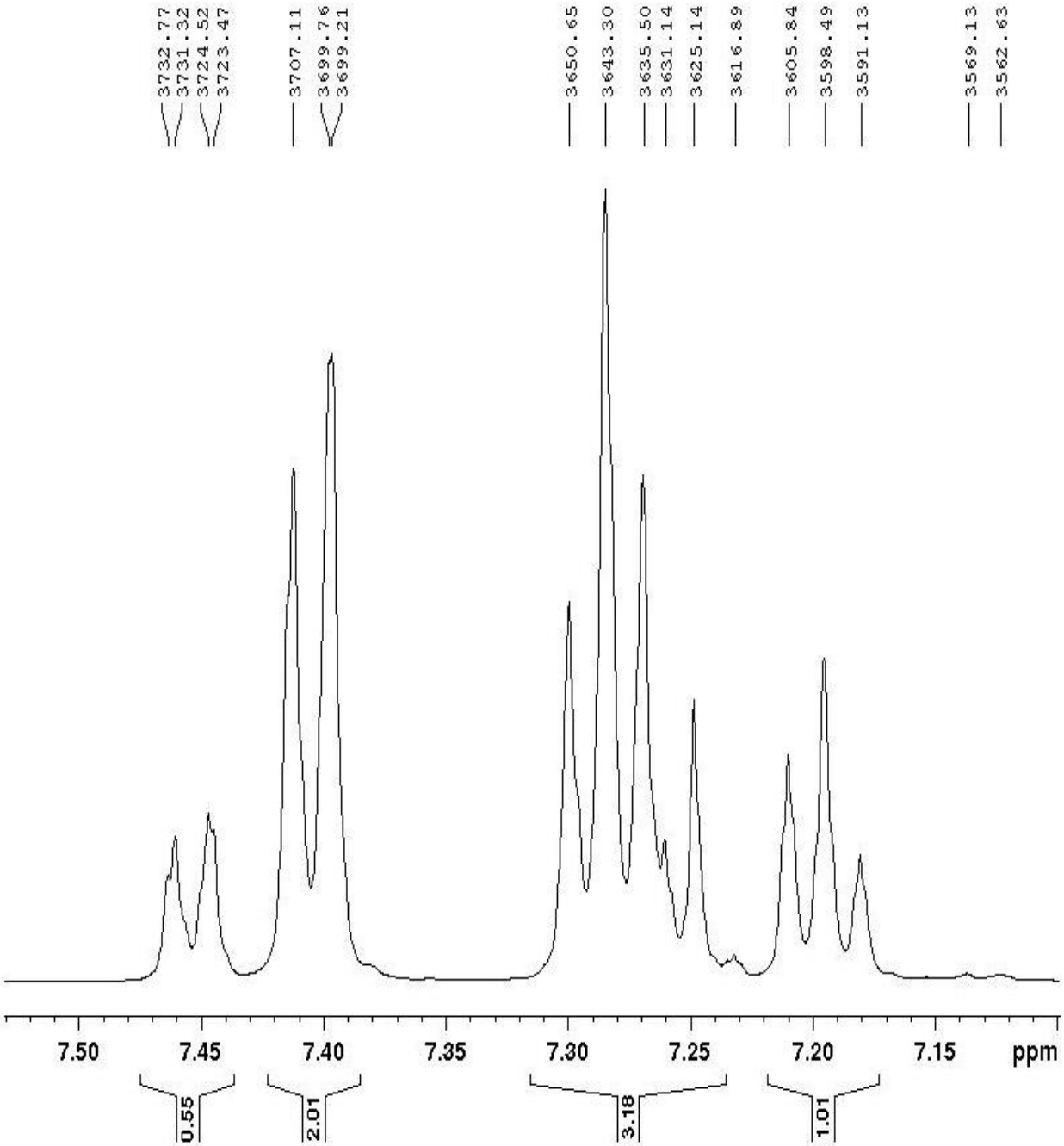
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
ppm
2.01
1.80
0.36
1.01
1.90
0.54
1.02
1.03
0.27
0.27
1.00
0.27
0.98
0.30
1.43
0.16
1.03
1.05
0.30
1.33
0.31
1.03
0.29


PhS
H

10a/b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3724.52
3723.47
3707.11
3699.76
3699.21
3650.65
3643.30
3635.50
3631.14
3625.14
3616.89
3605.84
3598.49
3591.13
3569.13
3562.63
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7.45
7.40
7.35
7.30
7.25
7.20
7.15
ppm
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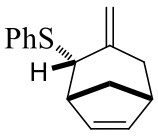
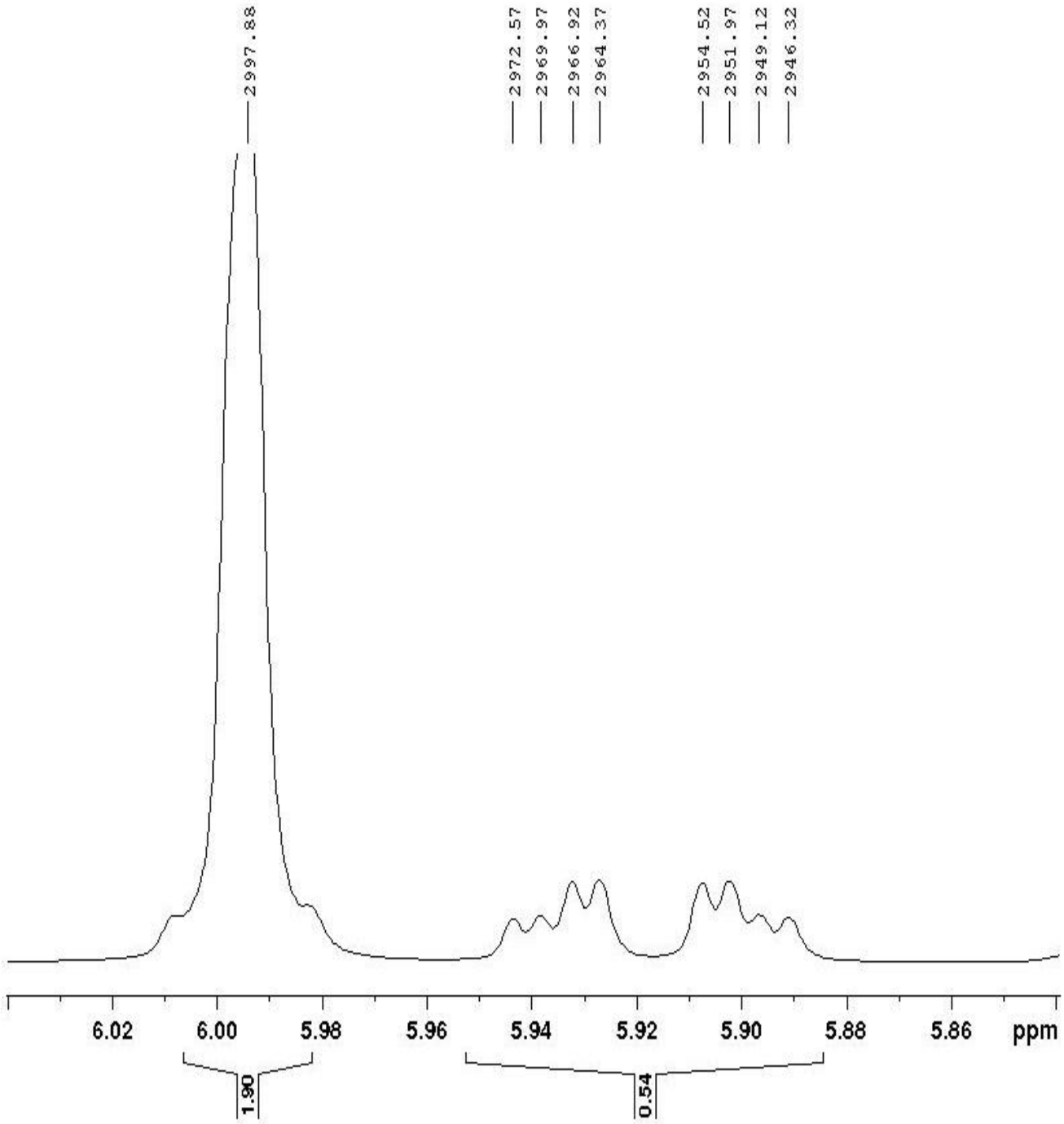
2.01
3.18
1.01


PhS
H

10a/b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2972.57
2969.97
2966.92
2964.37
2954.52
2951.97
2949.12
2946.32
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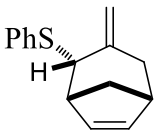
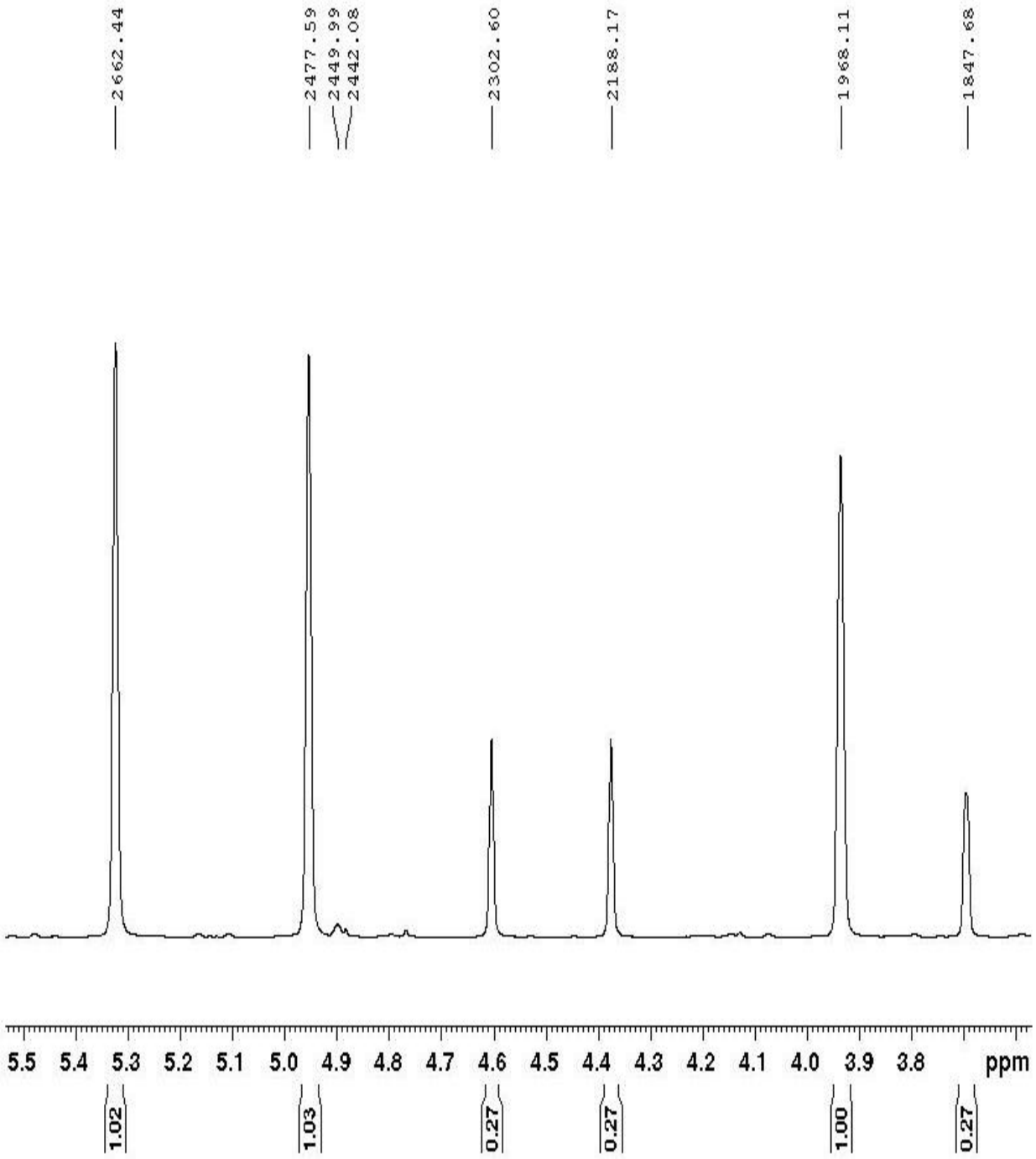
6.00
5.98
5.96
5.94
5.92
5.90
5.88
5.86
ppm
1.90
0.54


PhS
H

10a/b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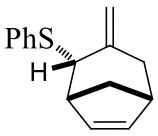
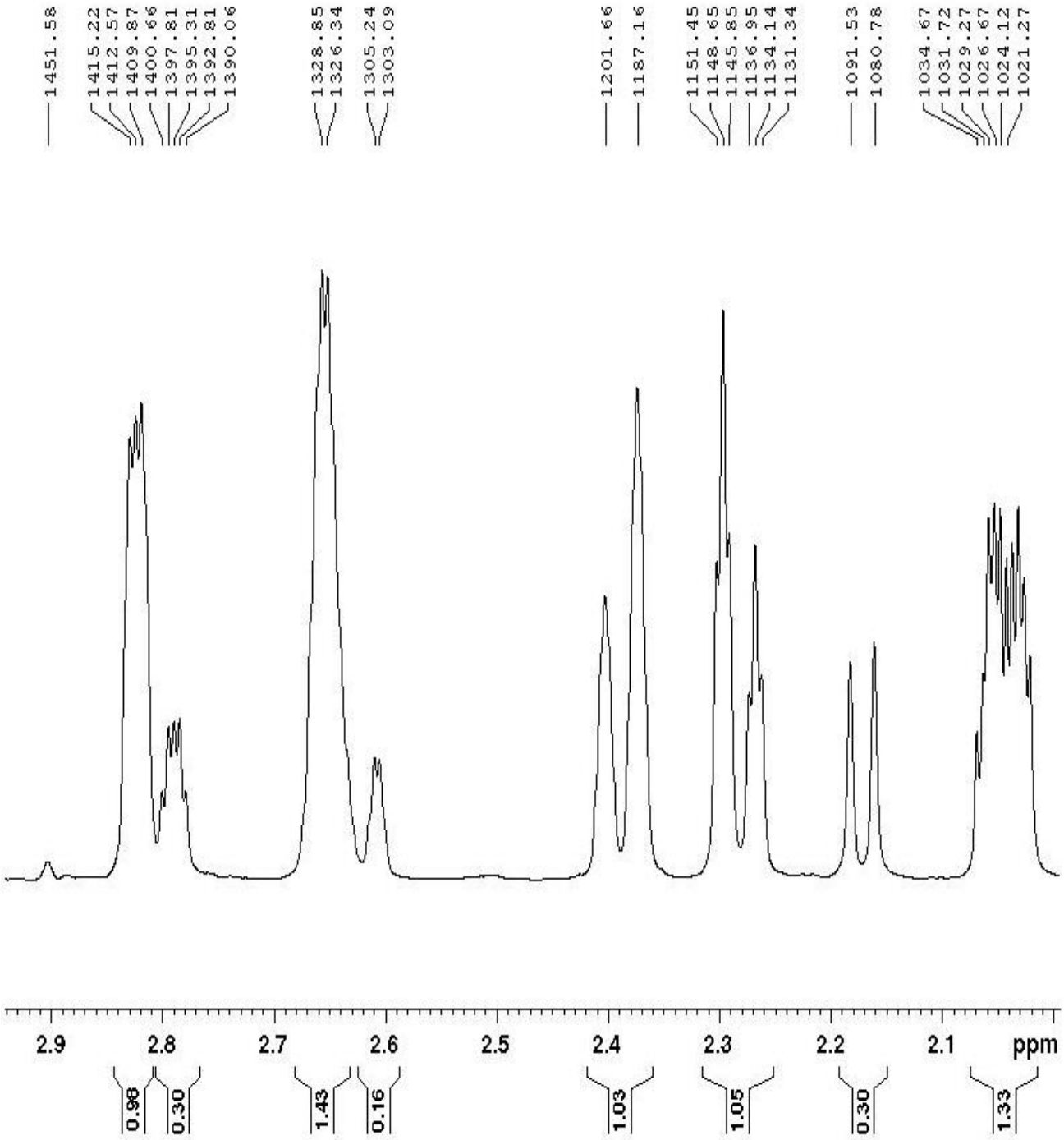
2477.59
2449.99
2442.08
2302.60
2188.17
1968.11
1847.68
5.5 5.4 5.3 5.2 5.1 5.0 4.9 4.8 4.7 4.6 4.5 4.4 4.3 4.2 4.1 4.0 3.9 3.8 ppm
1.02
1.03
0.27
0.27
1.00
0.27


PhS
H

10a/b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1412.57
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1400.66
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1395.31
1392.81
1390.06
1328.85
1326.34
1305.24
1303.09
1201.66
1187.16
1151.45
1148.65
1145.85
1136.95
1134.14
1131.34
1091.53
1080.78
1034.67
1031.72
1029.27
1026.67
1024.12
1021.27
2.9
2.8
2.7
2.6
2.5
2.4
2.3
2.2
2.1
ppm
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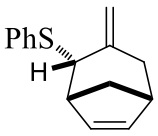
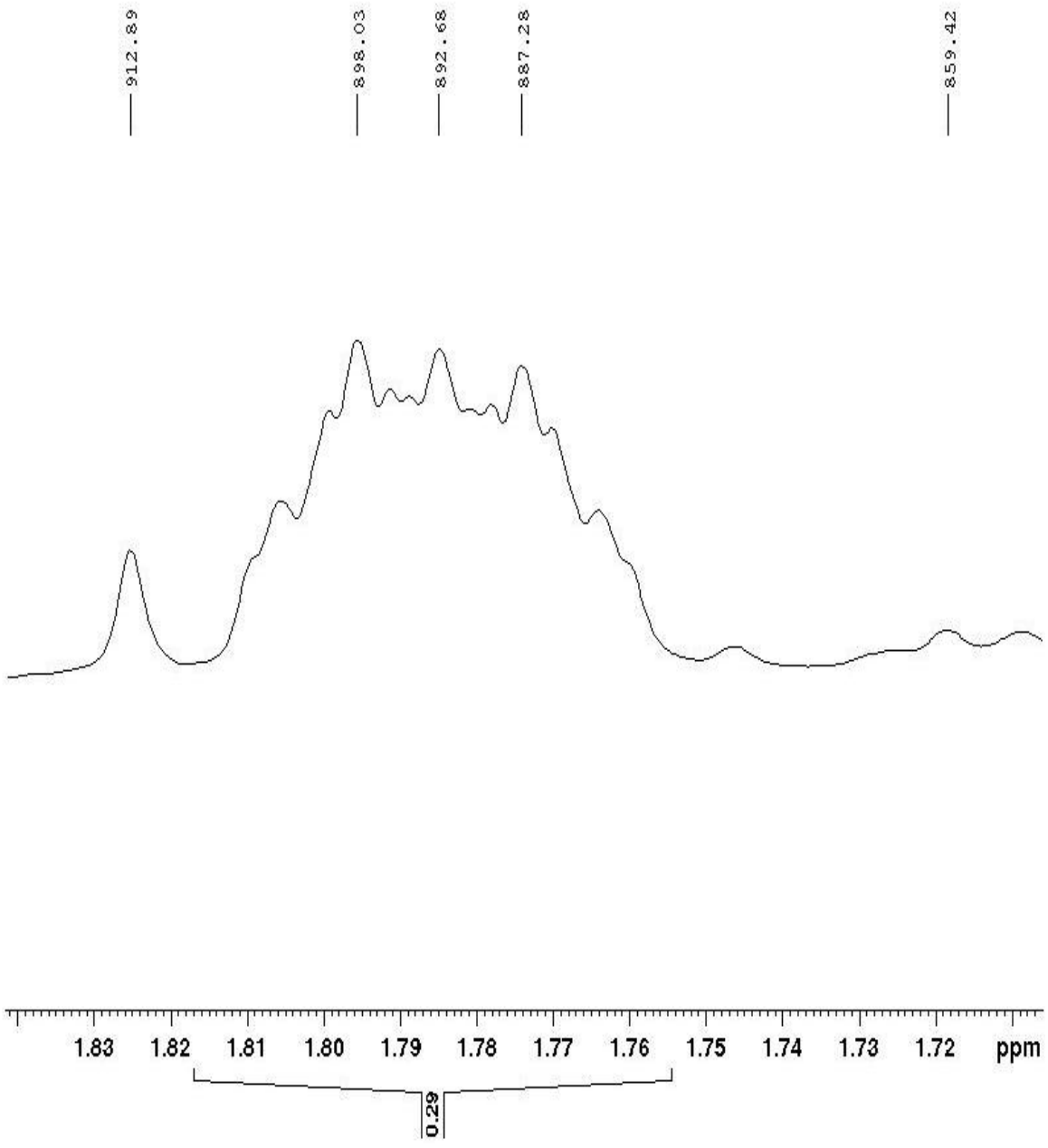
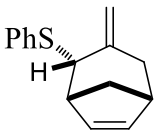
0.16
1.03
1.05
0.30
1.33

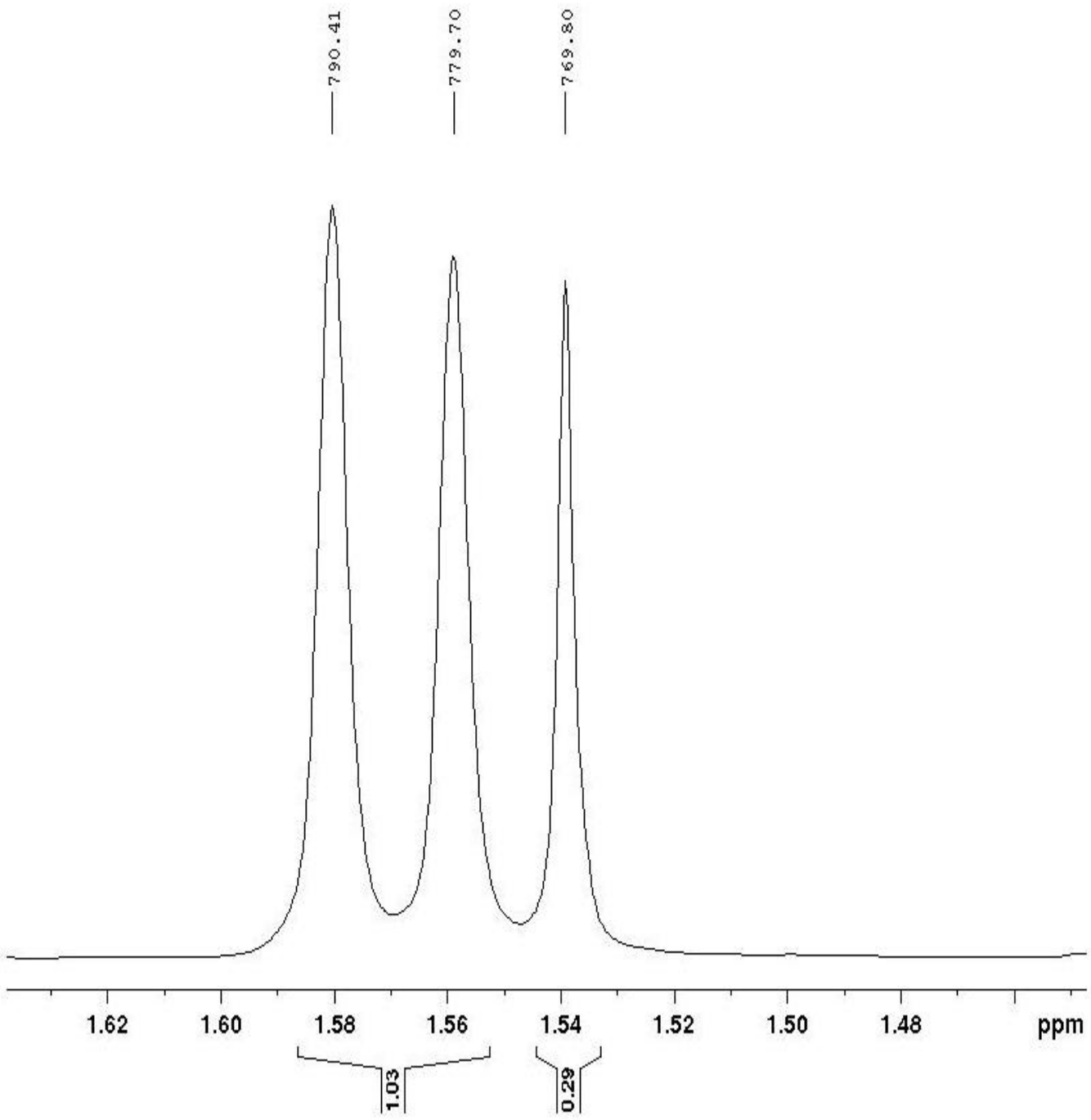

PhS
H

10a/b


912.89
898.03
892.68
887.28
859.42
1.83
1.82
1.81
1.80
1.79
1.78
1.77
1.76
1.75
1.74
1.73
1.72
ppm
0.29


PhS
H

10a/b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779.70
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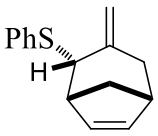
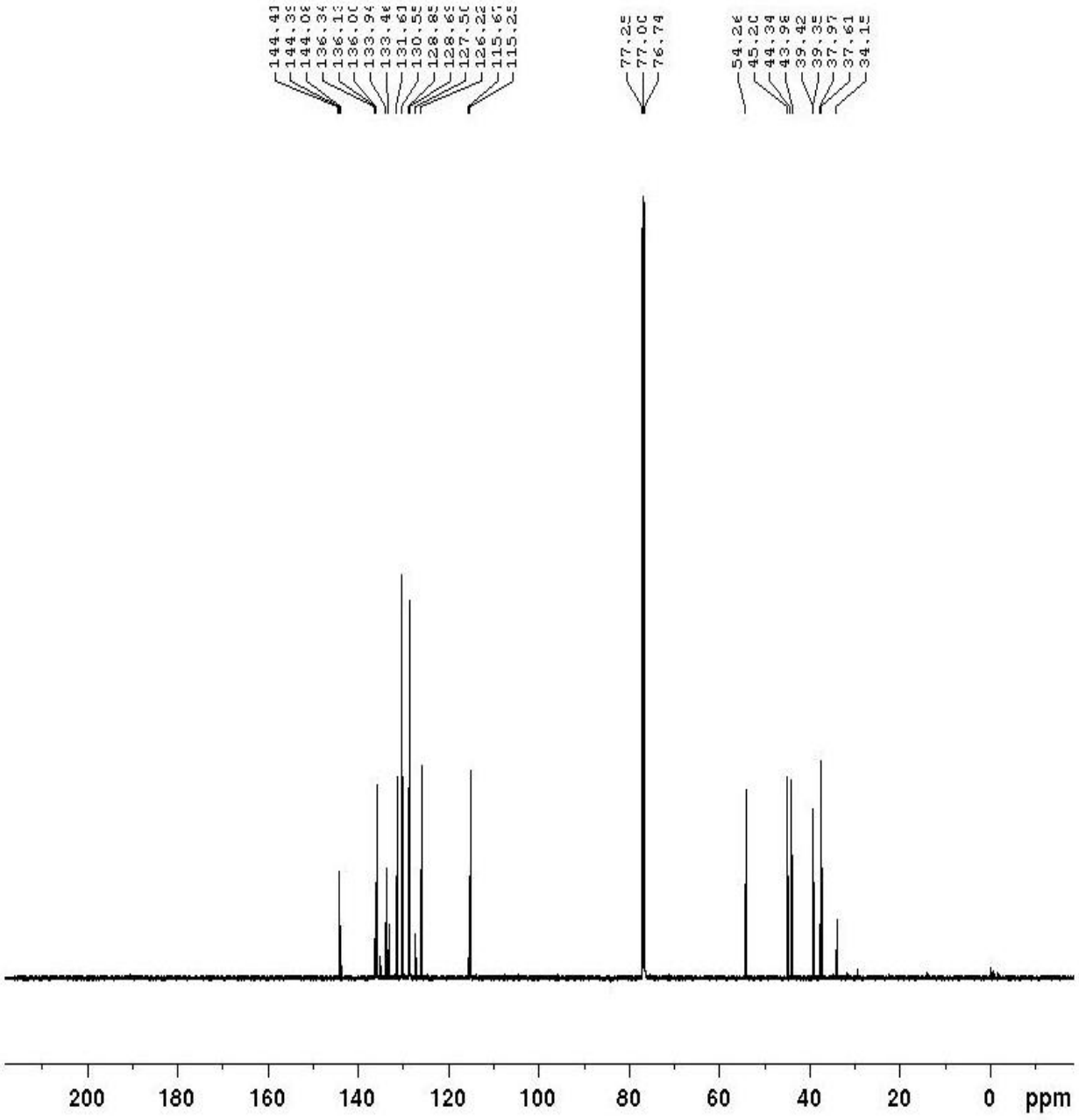
1.60
1.58
1.56
1.54
1.52
1.50
1.48
ppm
1.03
0.29


PhS
H

10a/b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144.3
144.0
136.3
136.1
136.0
133.9
133.4
131.6
130.5
128.8
128.6
127.5
126.2
115.6
115.2
77.2
77.0
76.74
54.2
45.2
44.34
43.9
39.42
39.3
37.97
37.61
34.1
200
180
160
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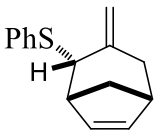
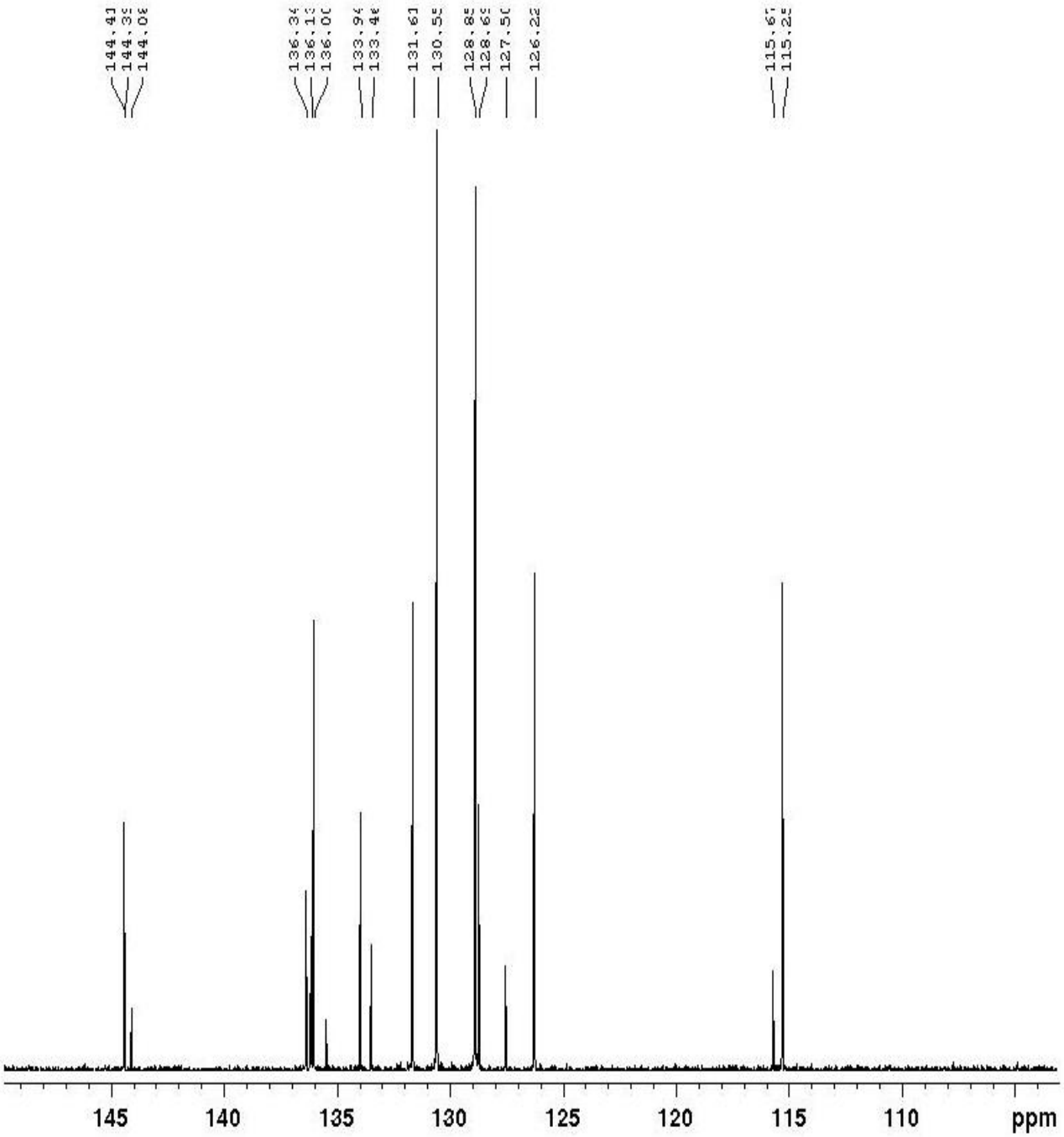
120
100
80
60
40
20
0
ppm


PhS
H

10a/b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136.13
136.00
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133.46
131.61
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127.50
126.22
115.67
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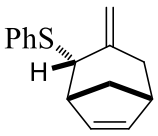
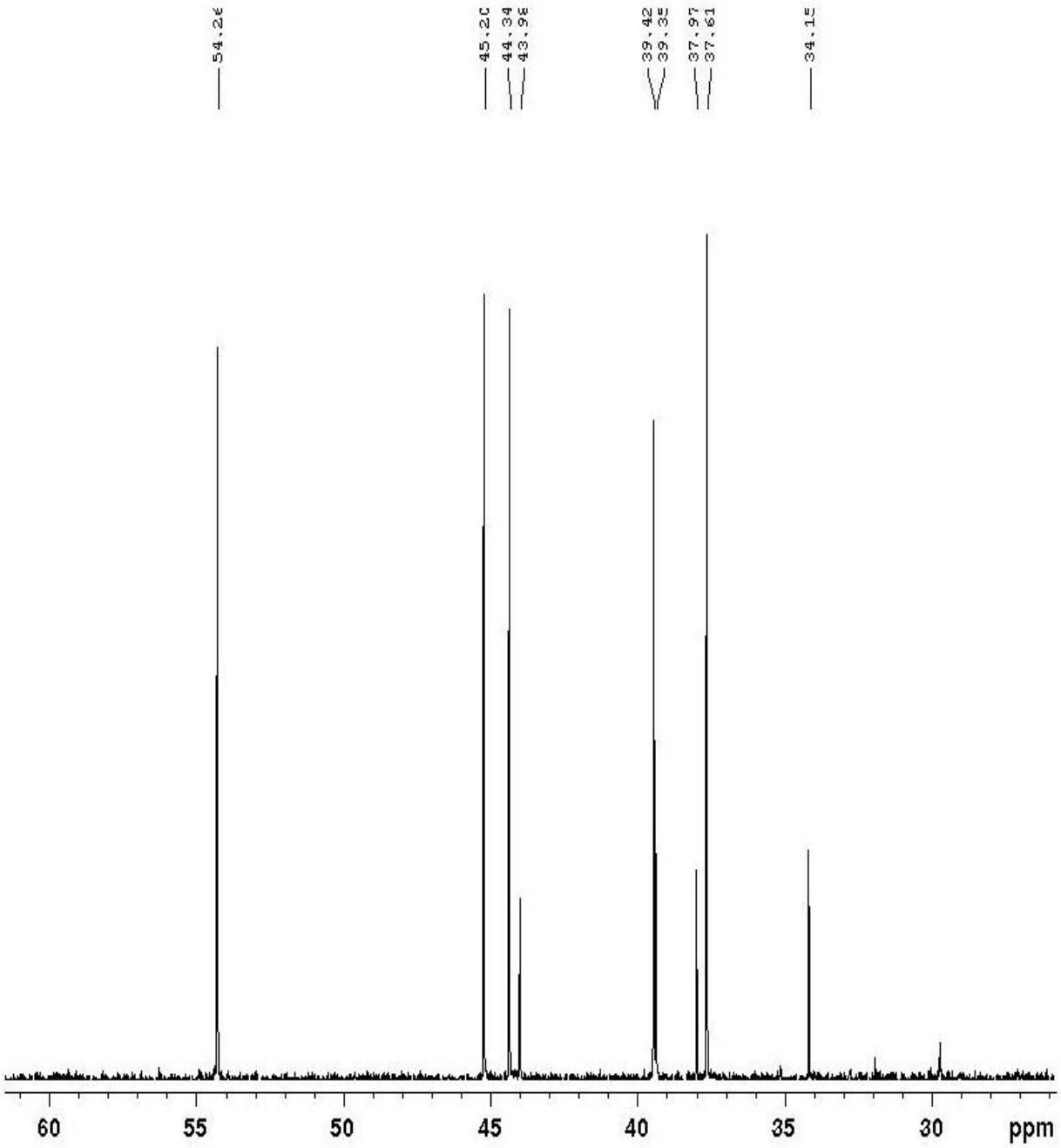
145
140
135
130
125
120
115
110
ppm


PhS
H

10a/b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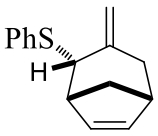
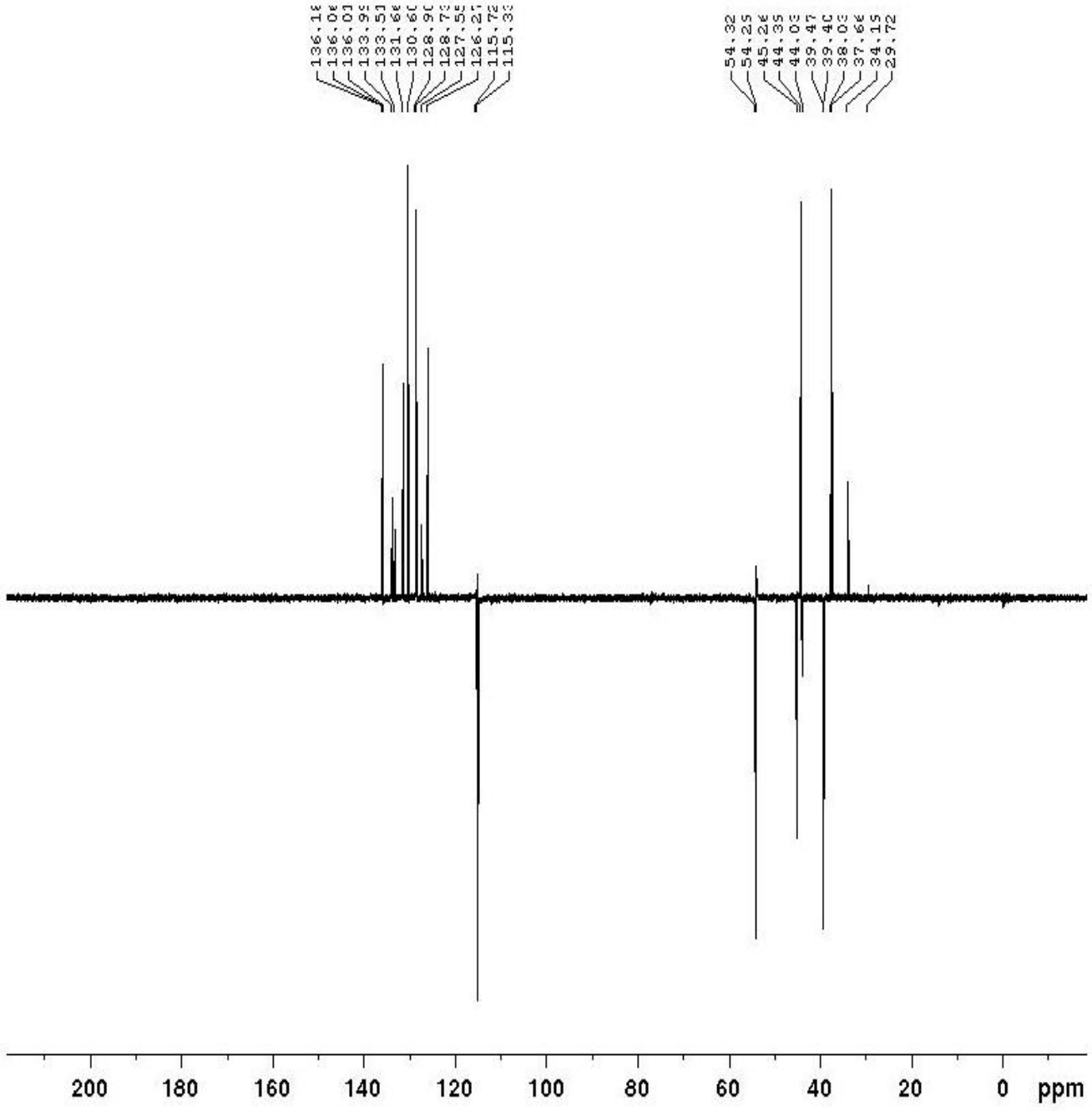
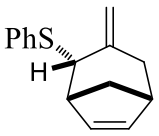
39.35
37.97
37.61
34.15
60
55
50
45
40
35
30
ppm

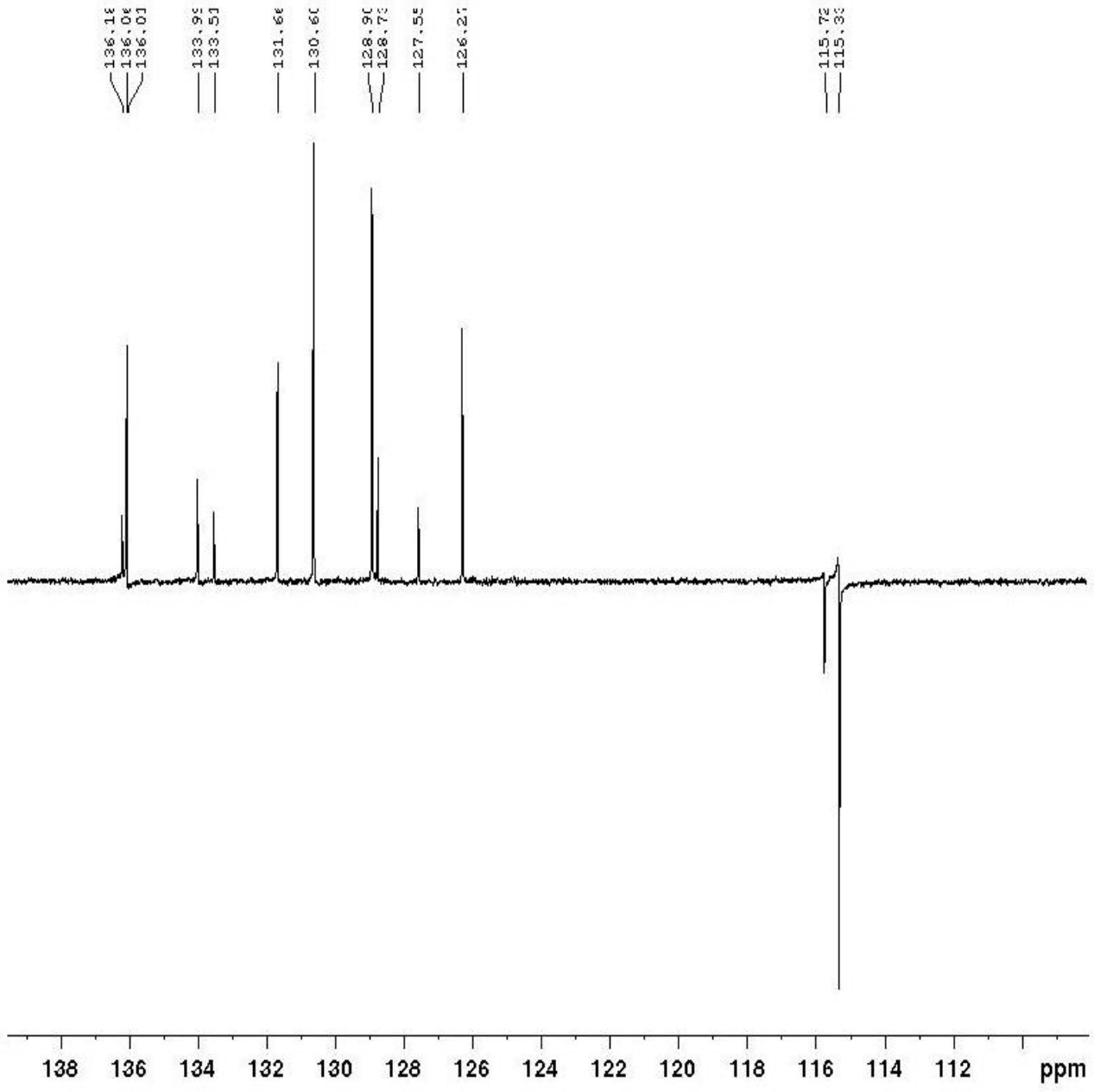

PhS
H

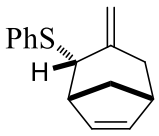
10a/b


136.18
136.06
136.01
133.95
133.51
131.66
130.60
128.90
128.73
127.55
126.27
115.72
115.33
54.32
54.25
45.26
44.35
44.03
39.47
39.40
38.03
37.66
34.15
29.72
200
180
160
140
120
100
80
60
40
20
0
ppm

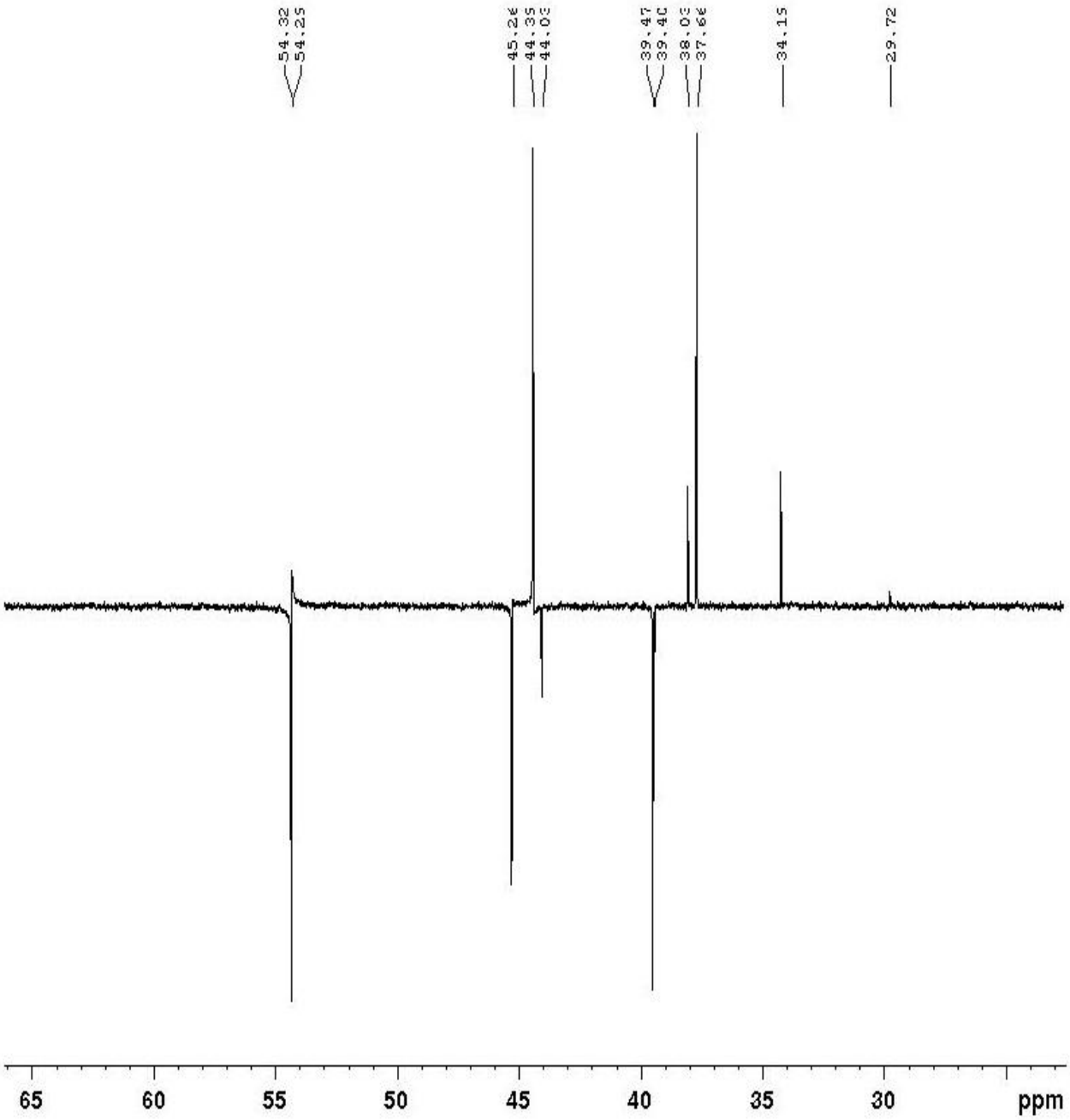

PhS
H

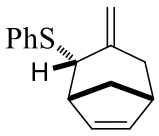
10a/b


136.18
136.04
136.01
133.95
133.51
131.68
130.60
128.90
128.73
127.55
126.27
115.72
115.38
138
136
134
132
130
128
126
124
122
120
118
116
114
112
ppm


PhS
H

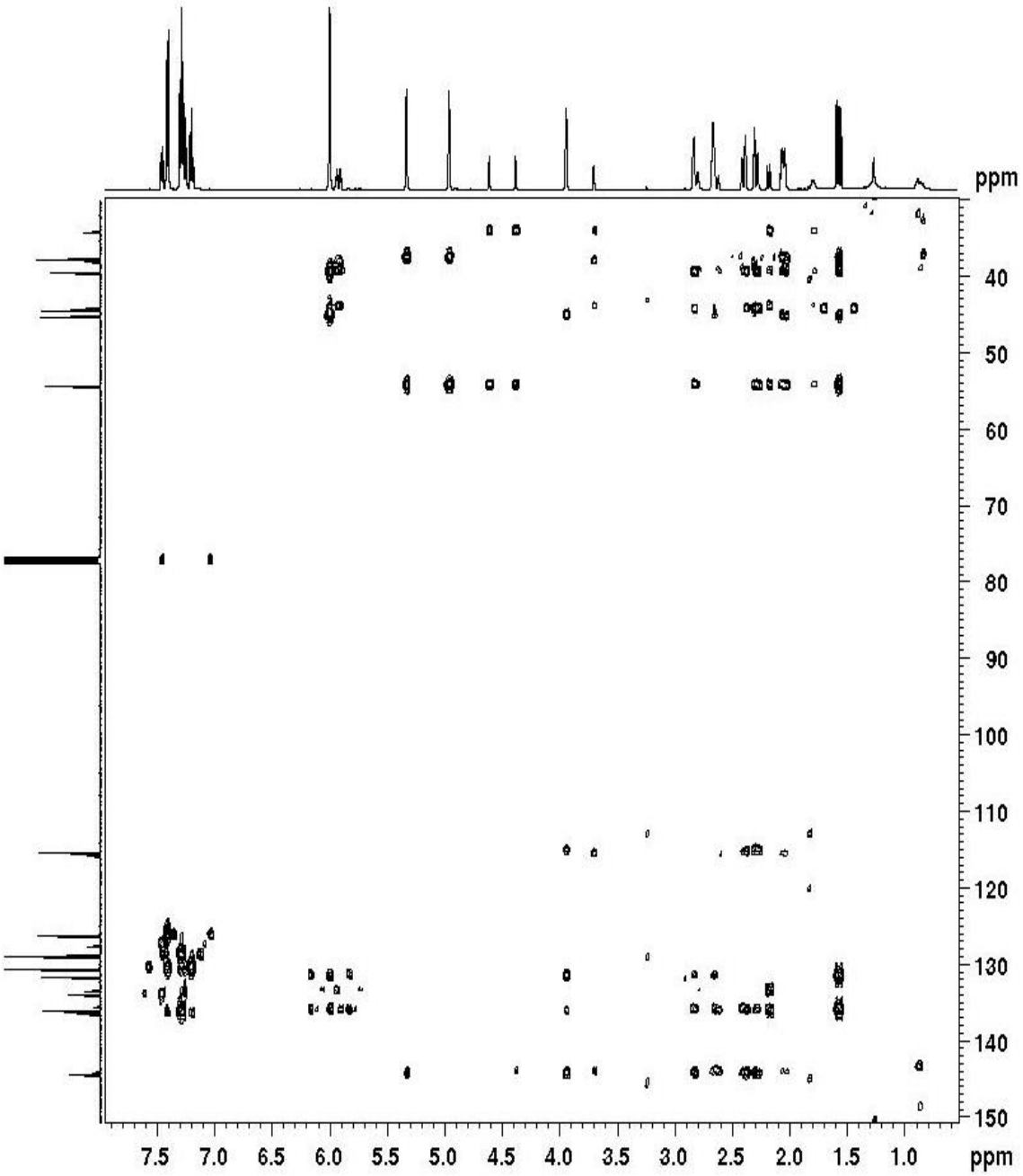
10a/b

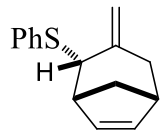

54.32
54.25
45.26
44.35
44.03
39.47
39.40
38.03
37.66
34.15
29.72
65
60
55
50
45
40
35
30
ppm


PhS
H

10a/b

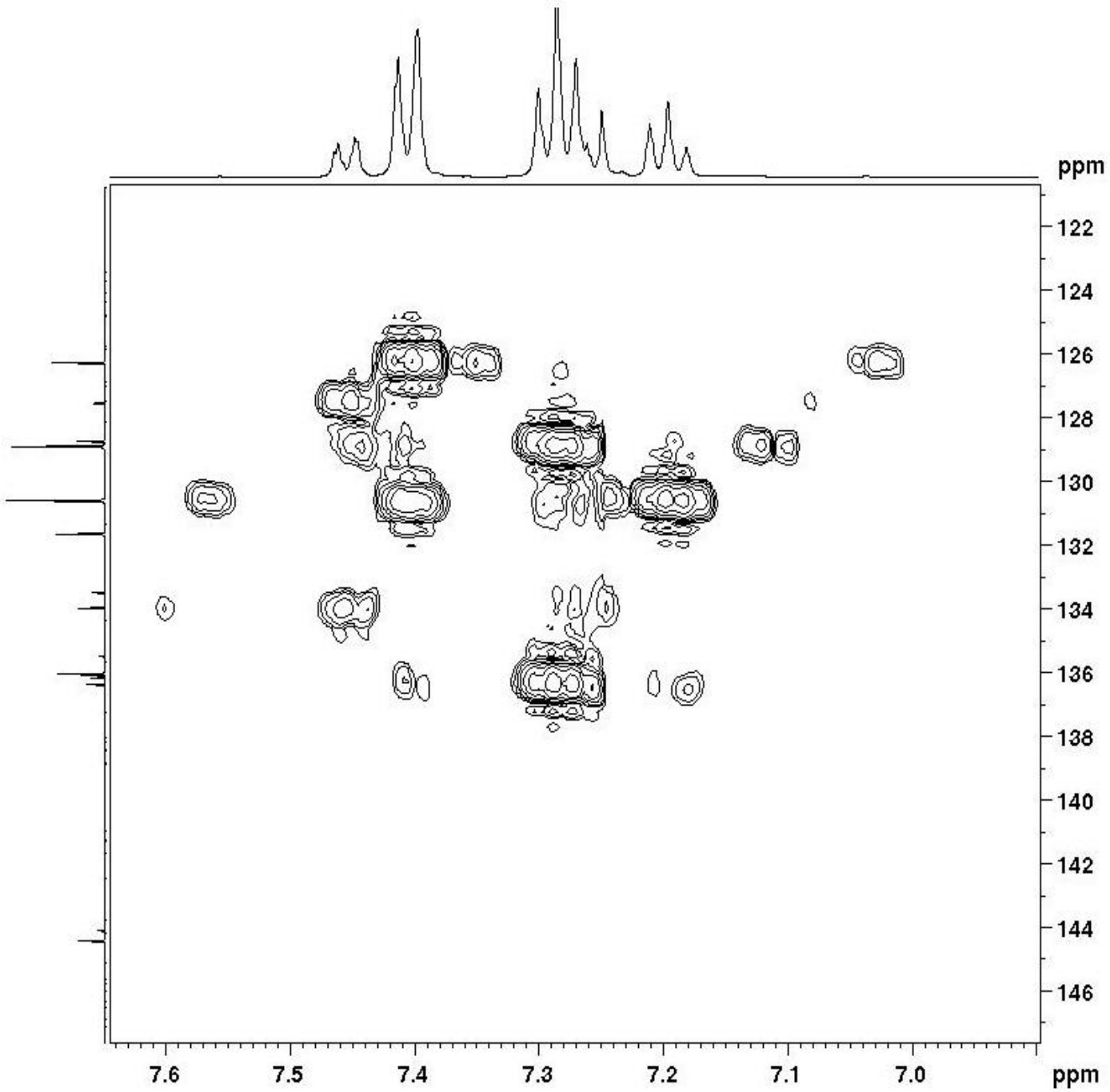
HMBC

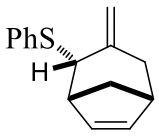

ppm
40
50
60
70
80
90
100
110
120
130
140
150
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm


PhS
H

10a/b

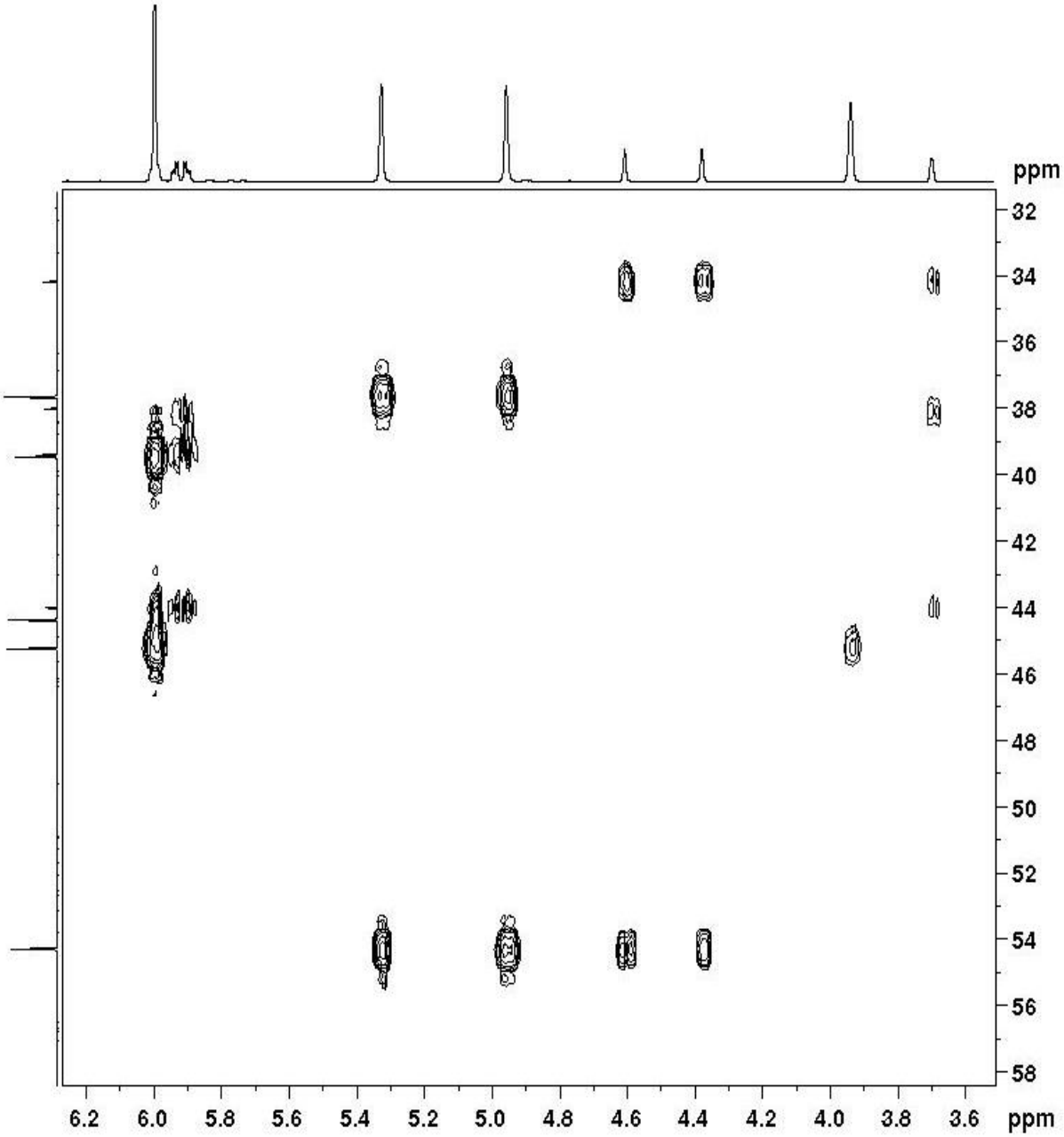
HMBC

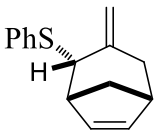

ppm
122
124
126
128
130
132
134
136
138
140
142
144
146
7.6
7.5
7.4
7.3
7.2
7.1
7.0
ppm


PhS
H

10a/b

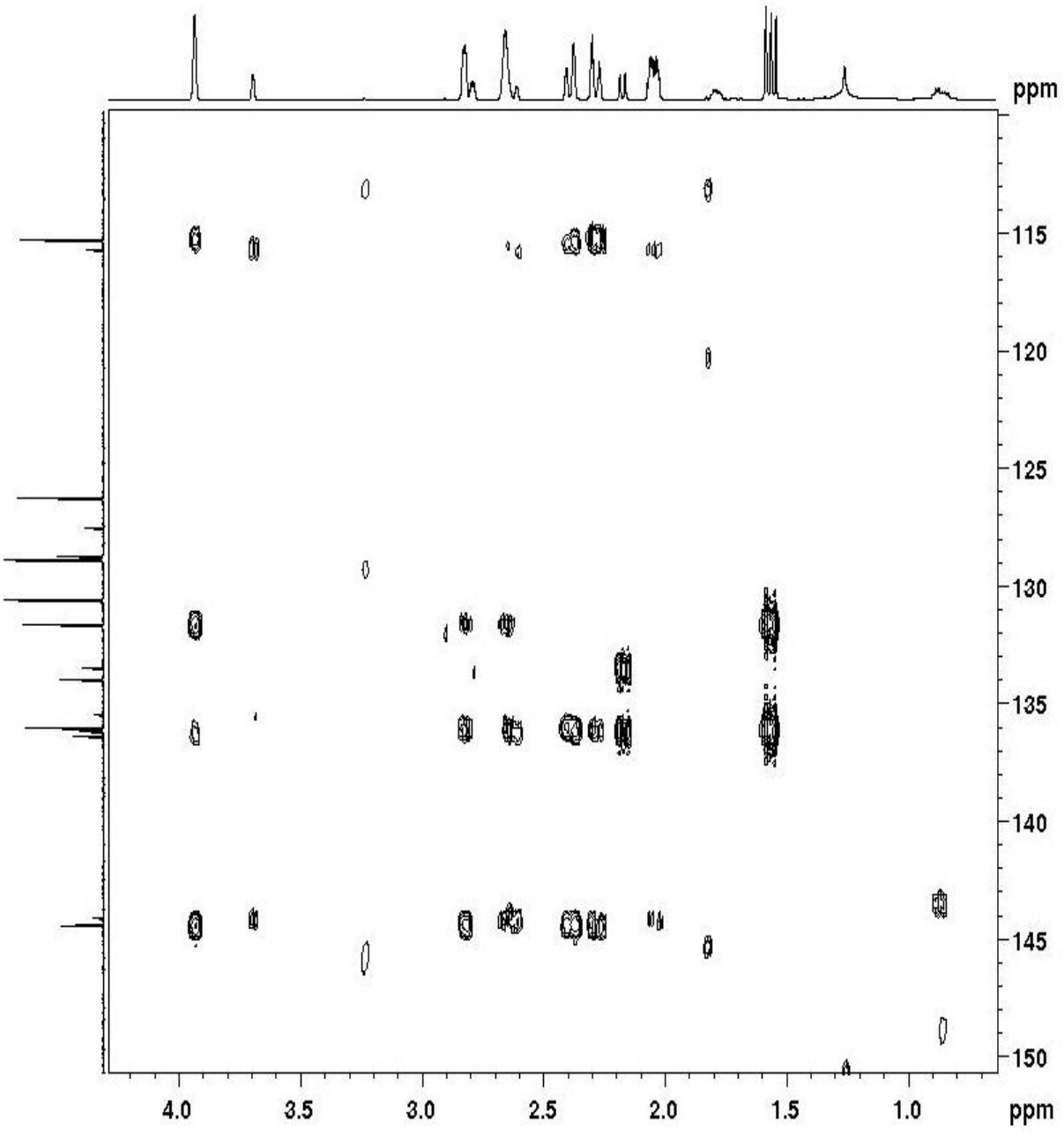
HMBC

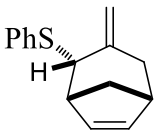

ppm
32
34
36
38
40
42
44
46
48
50
52
54
56
58
6.2
6.0
5.8
5.6
5.4
5.2
5.0
4.8
4.6
4.4
4.2
4.0
3.8
3.6
ppm



10a/b

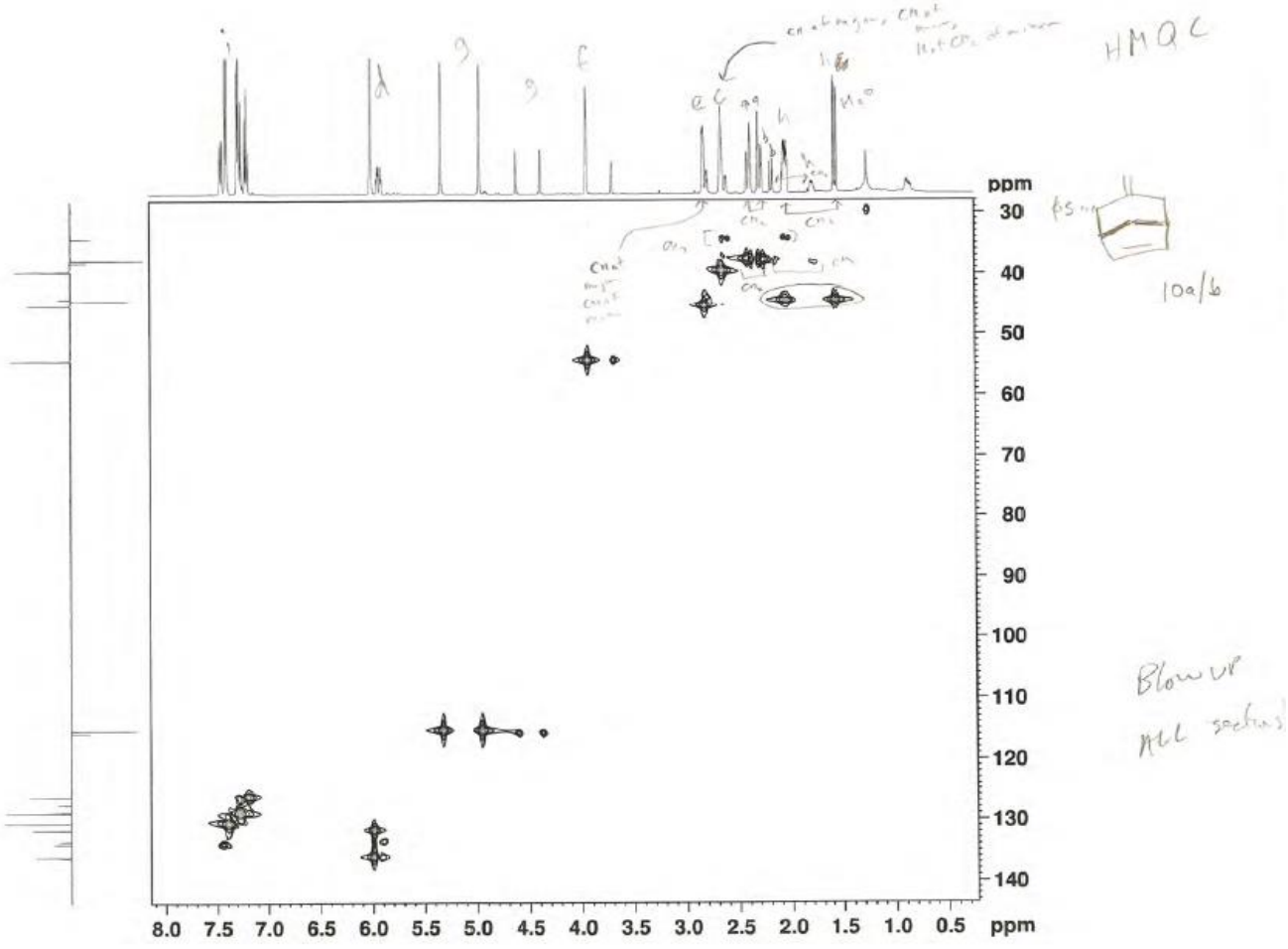
HMBC

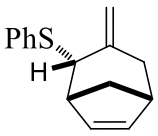



PhS
H

10a/b

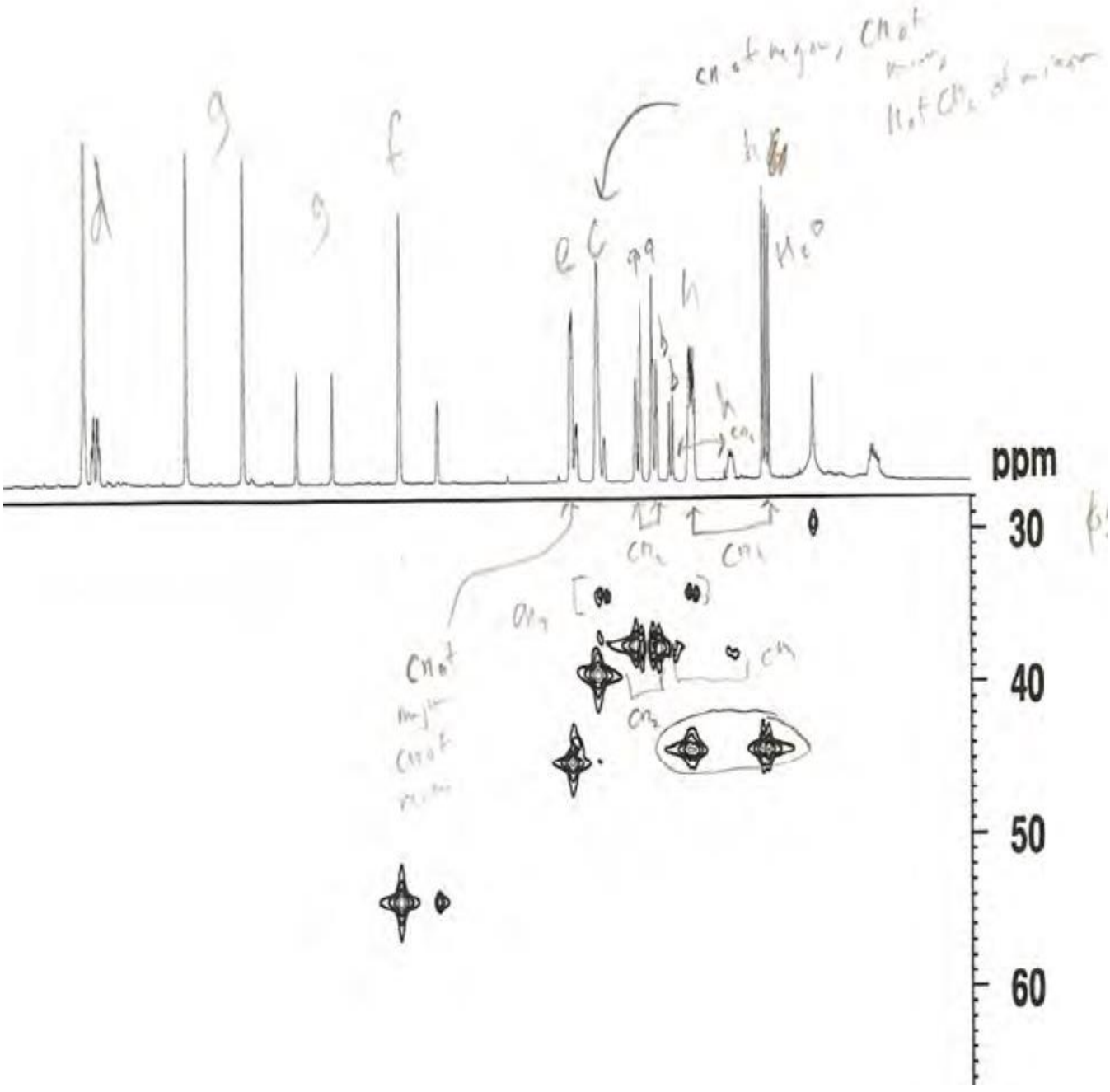
HMQC

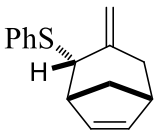

HMQC
10a/b
Blow up
ppm
30
40
50
60
70
80
90
100
110
120
130
140
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm



10a/b

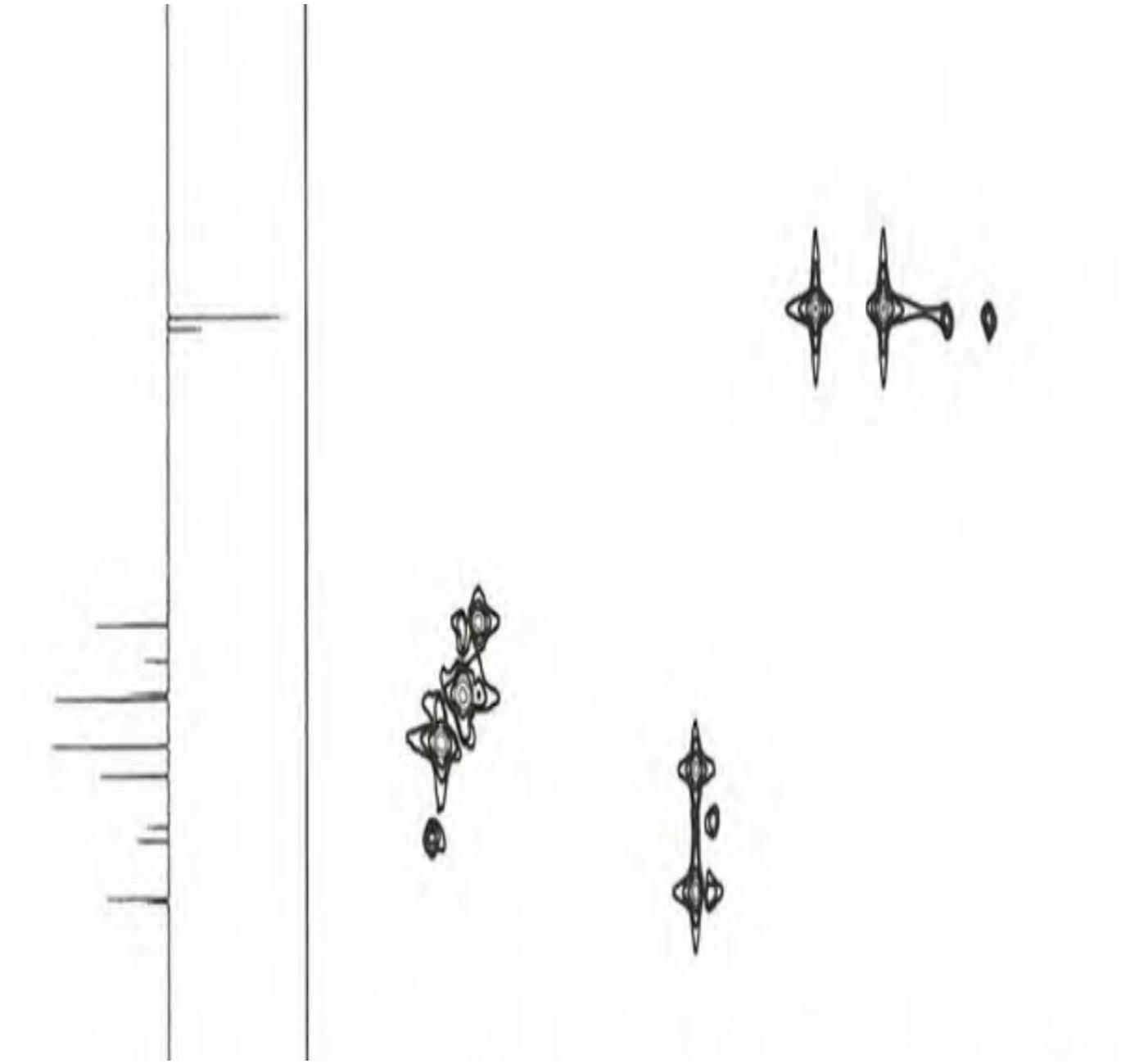
HMQC

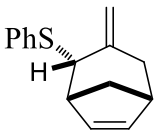




10a/b

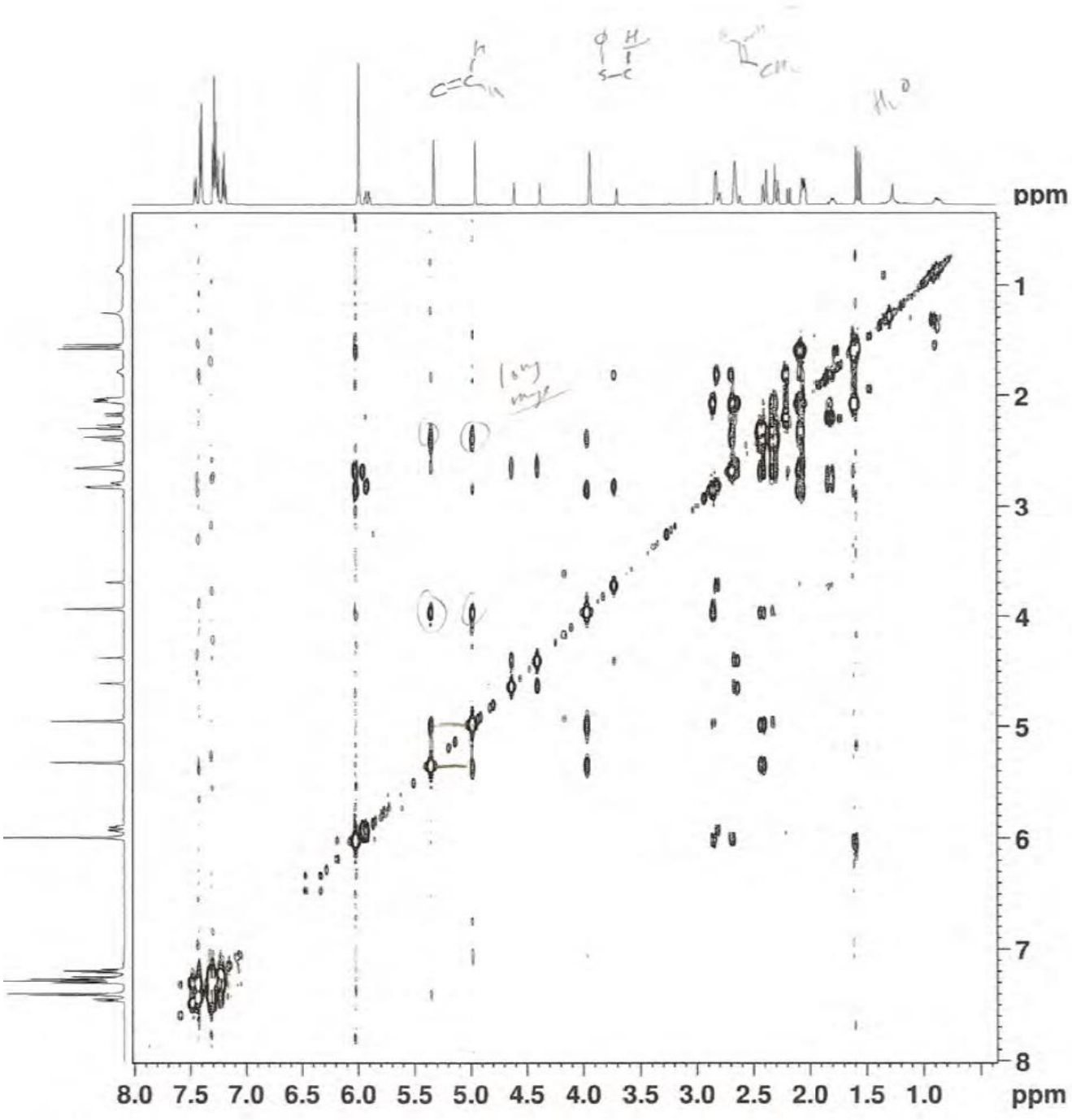
HMQC

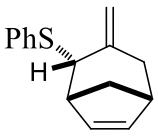




10a/b

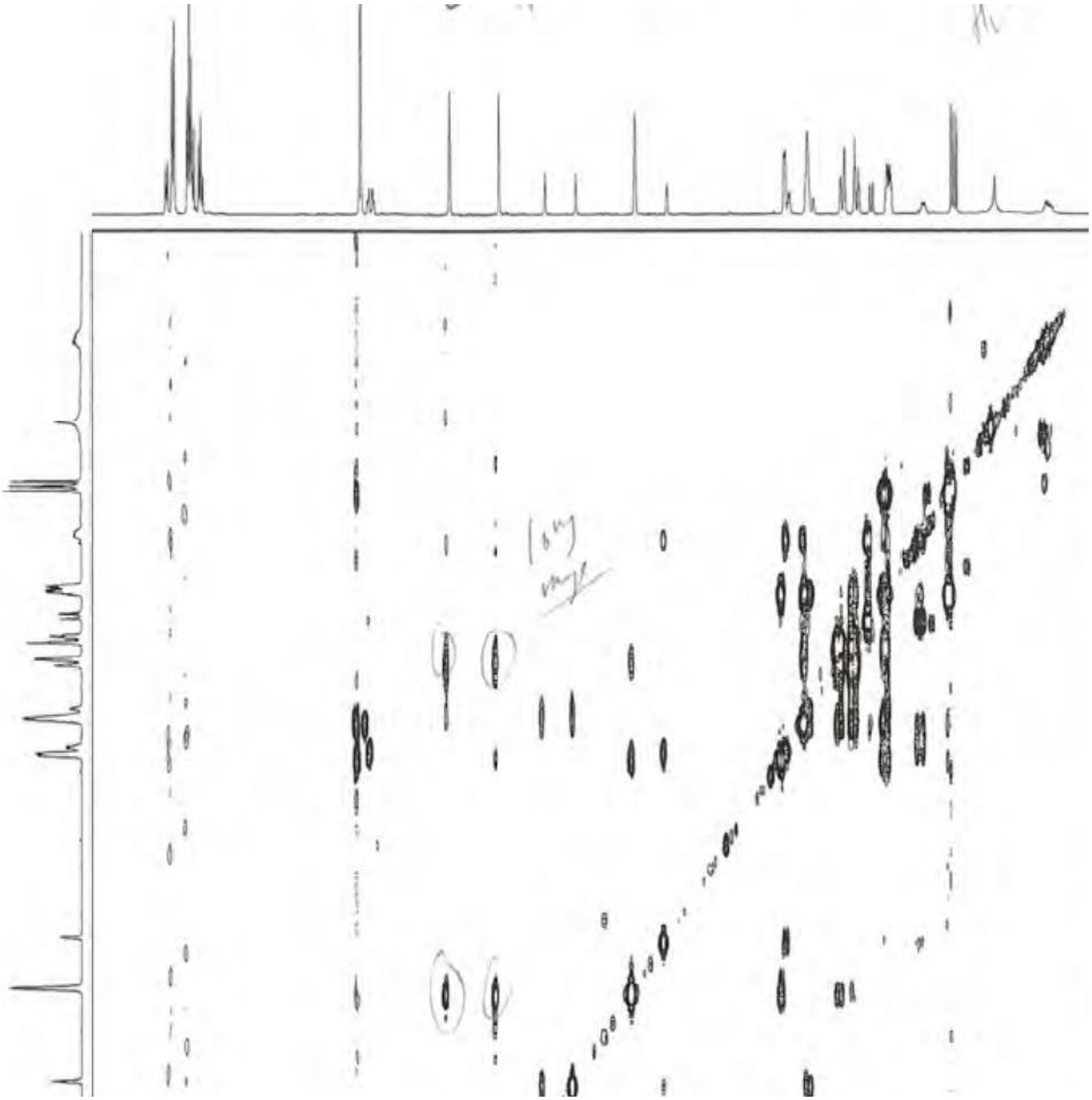
COSY

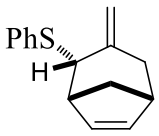




10a/b

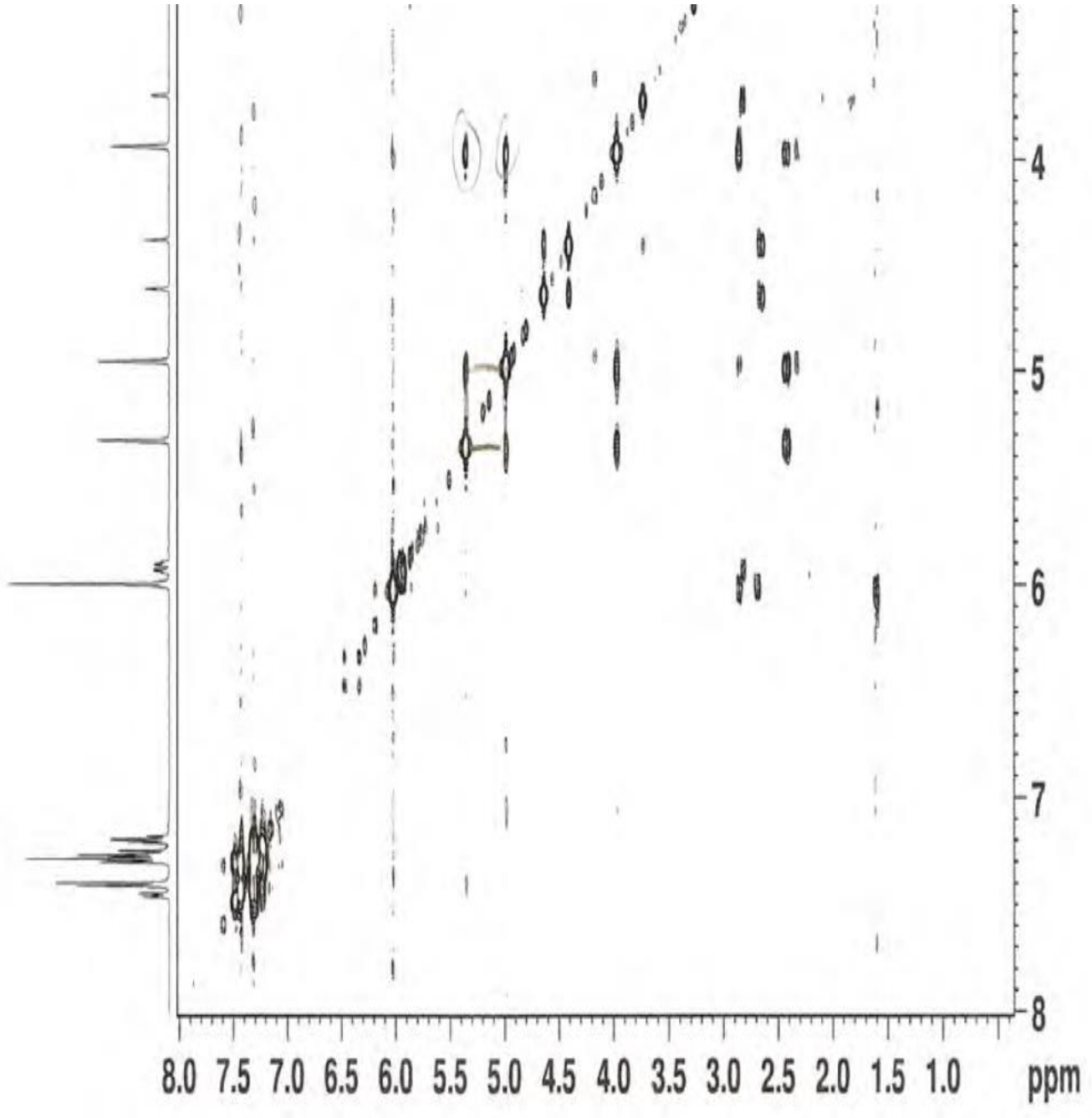
COSY

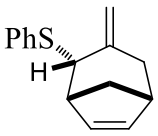




10a/b

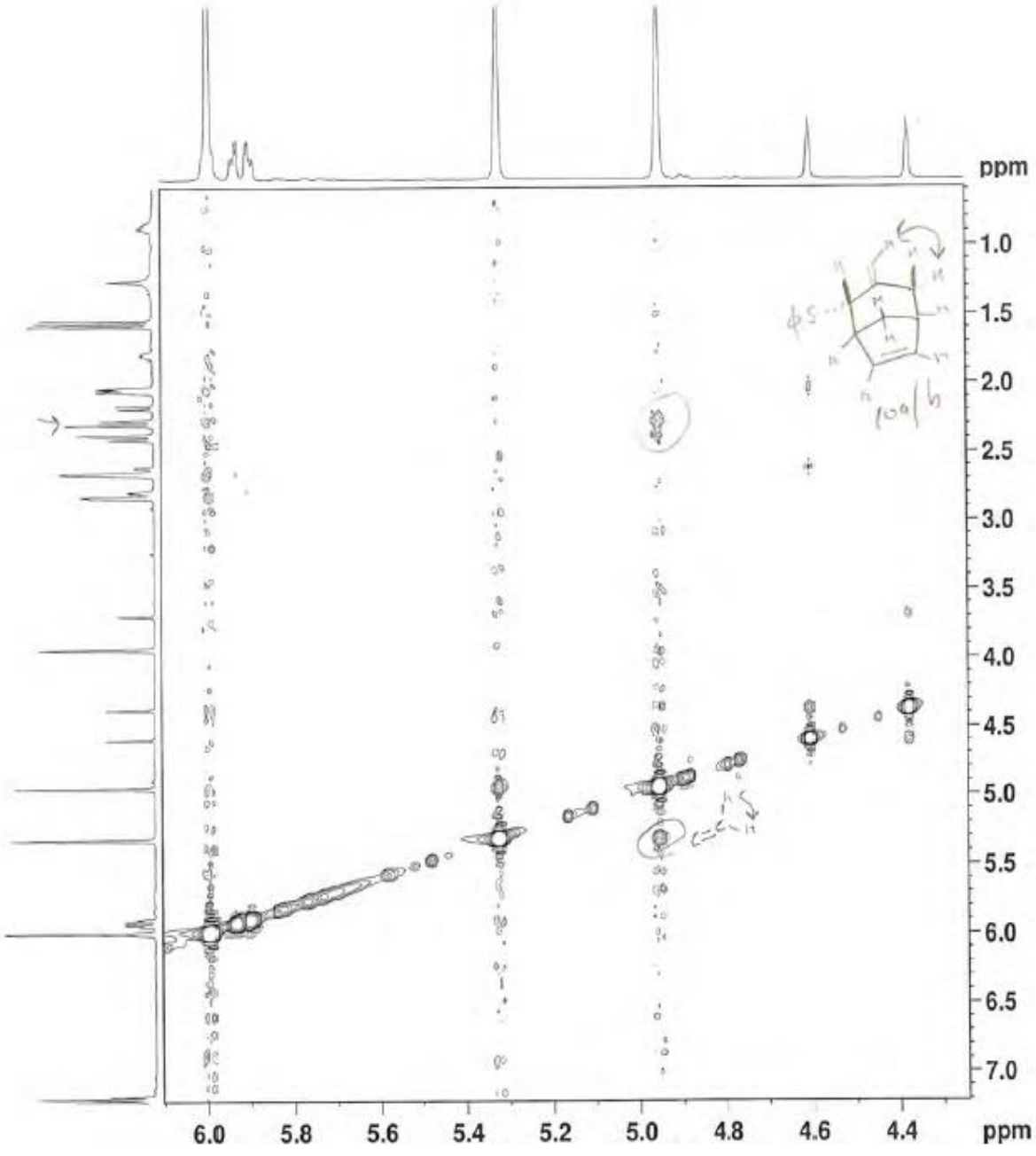
COSY

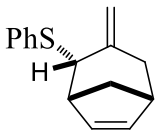



PhS
H

10a/b

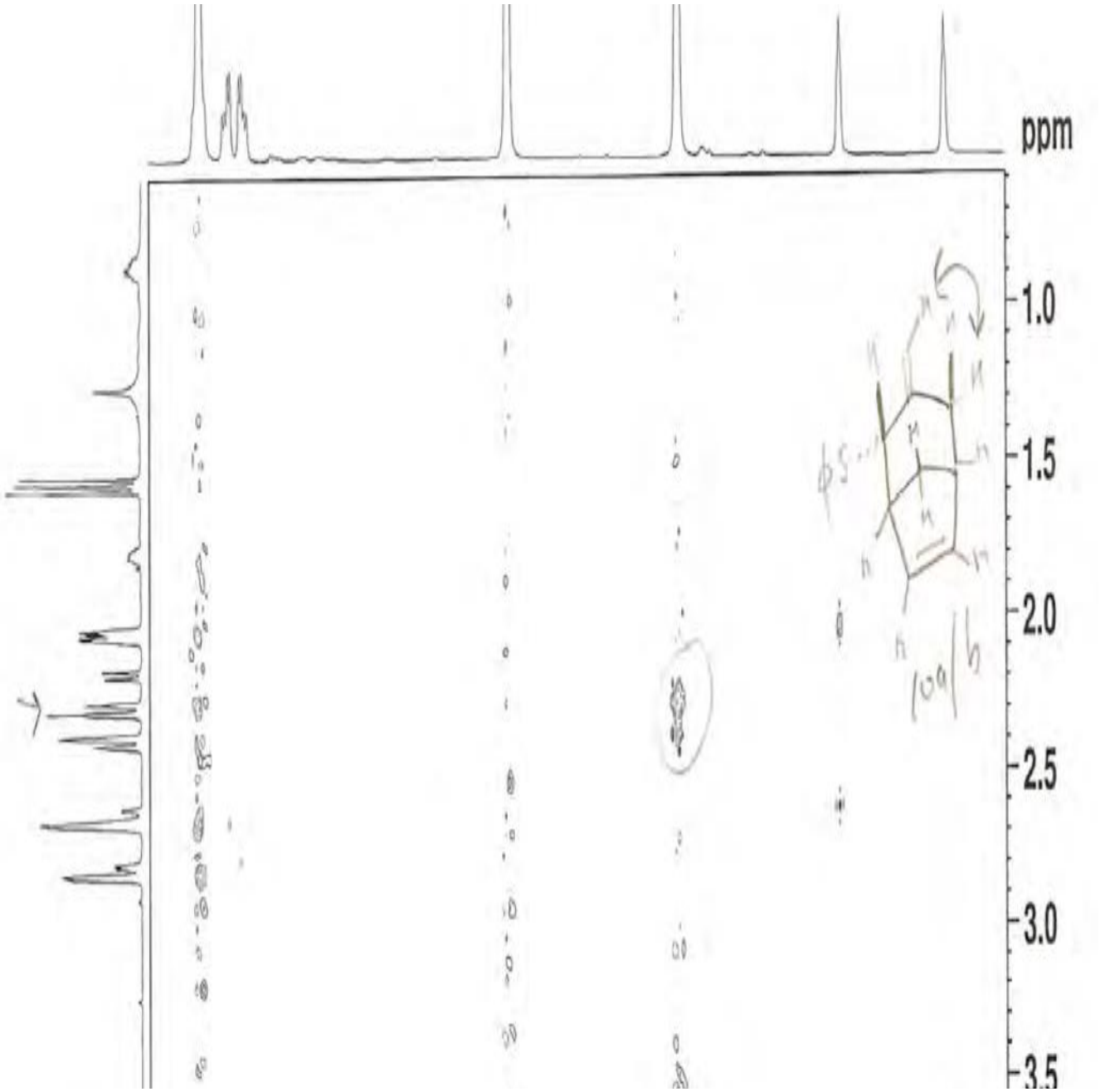
NOESY

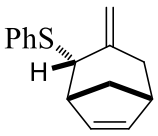

ppm
1.0
1.5
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.5
6.0
6.5
7.0
6.0
5.8
5.6
5.4
5.2
5.0
4.8
4.6
4.4
ppm



10a/b

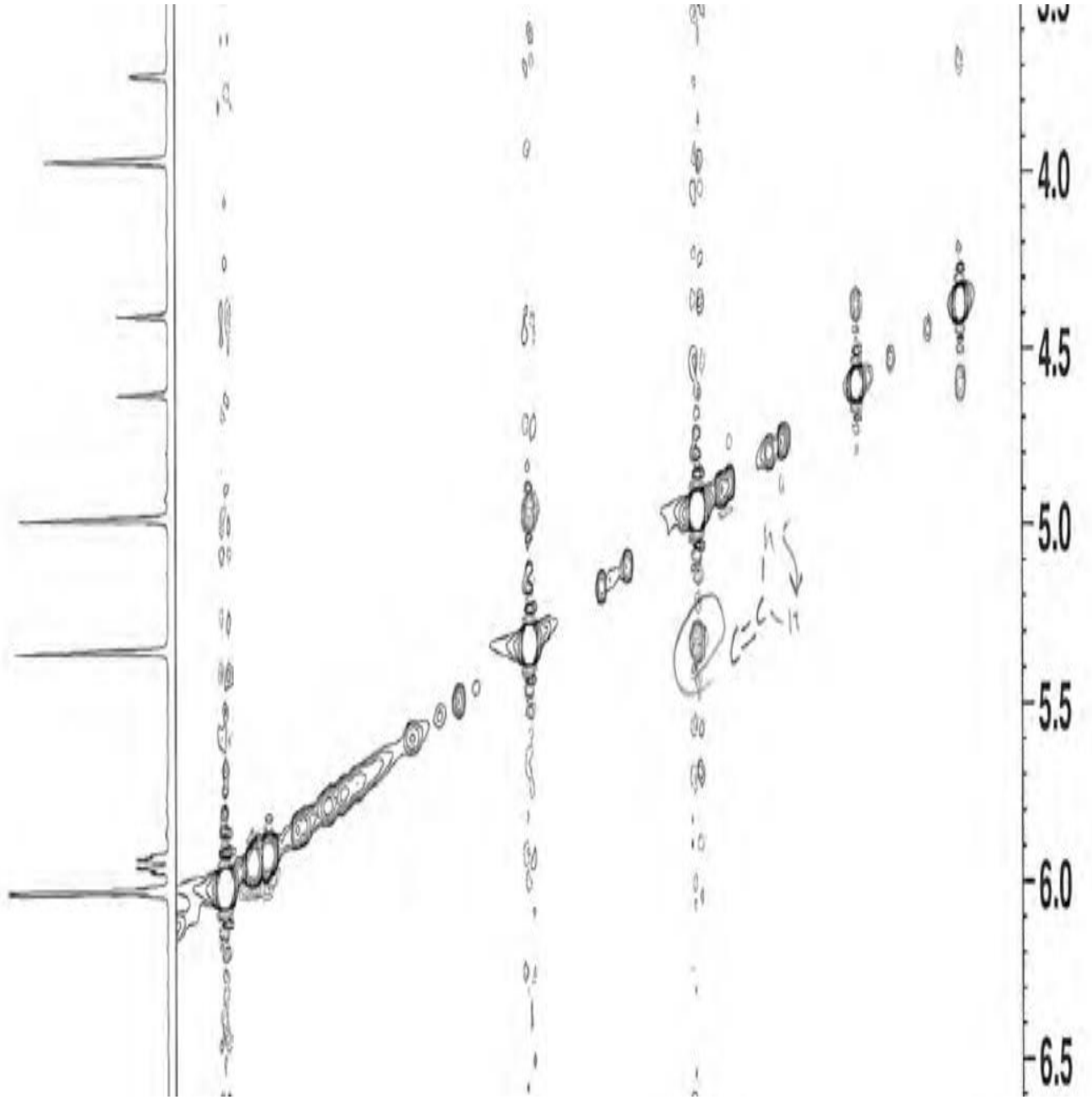
NOESY

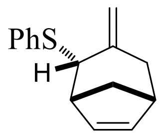




10a/b

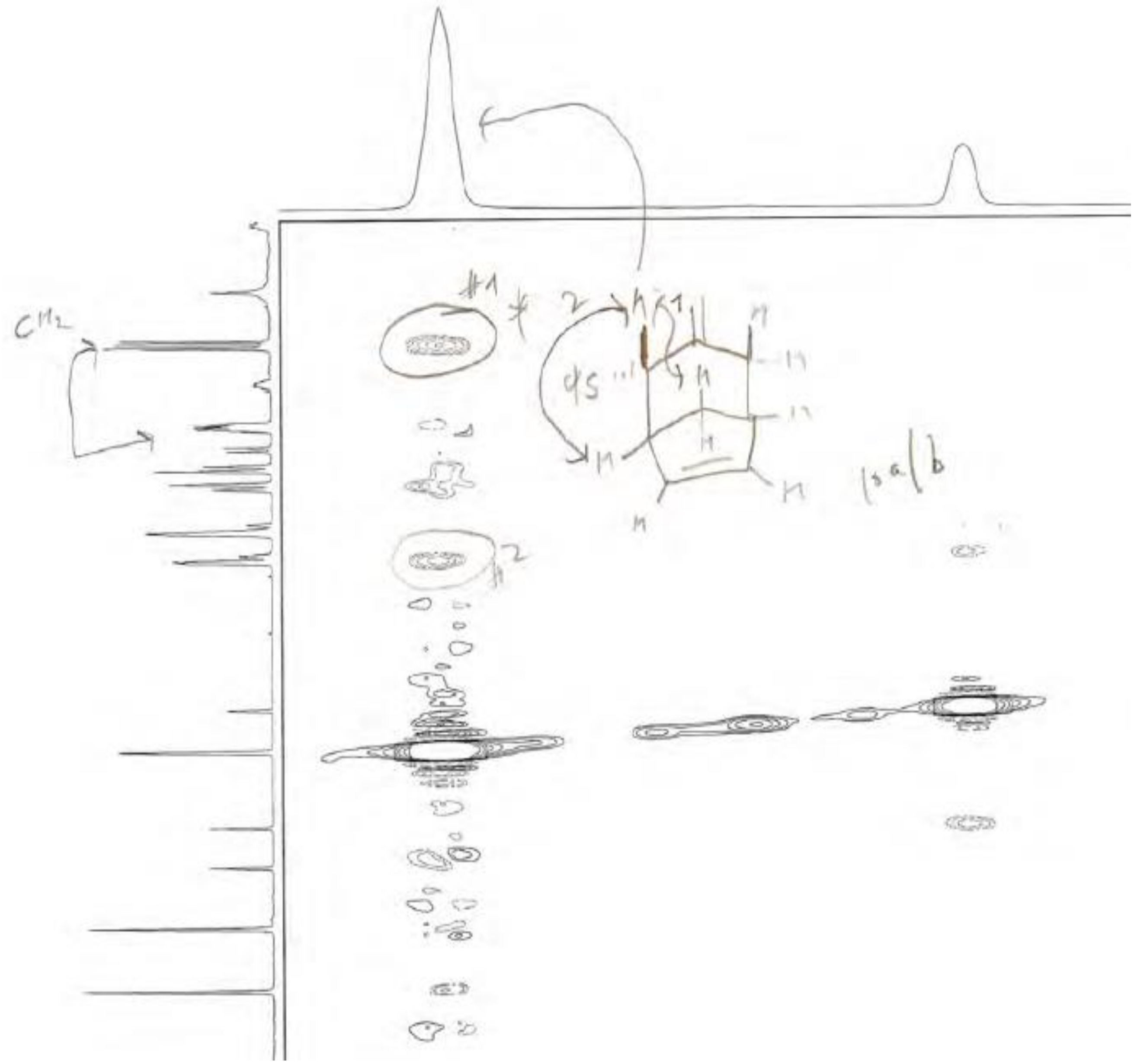
NOESY

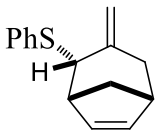




10a/b

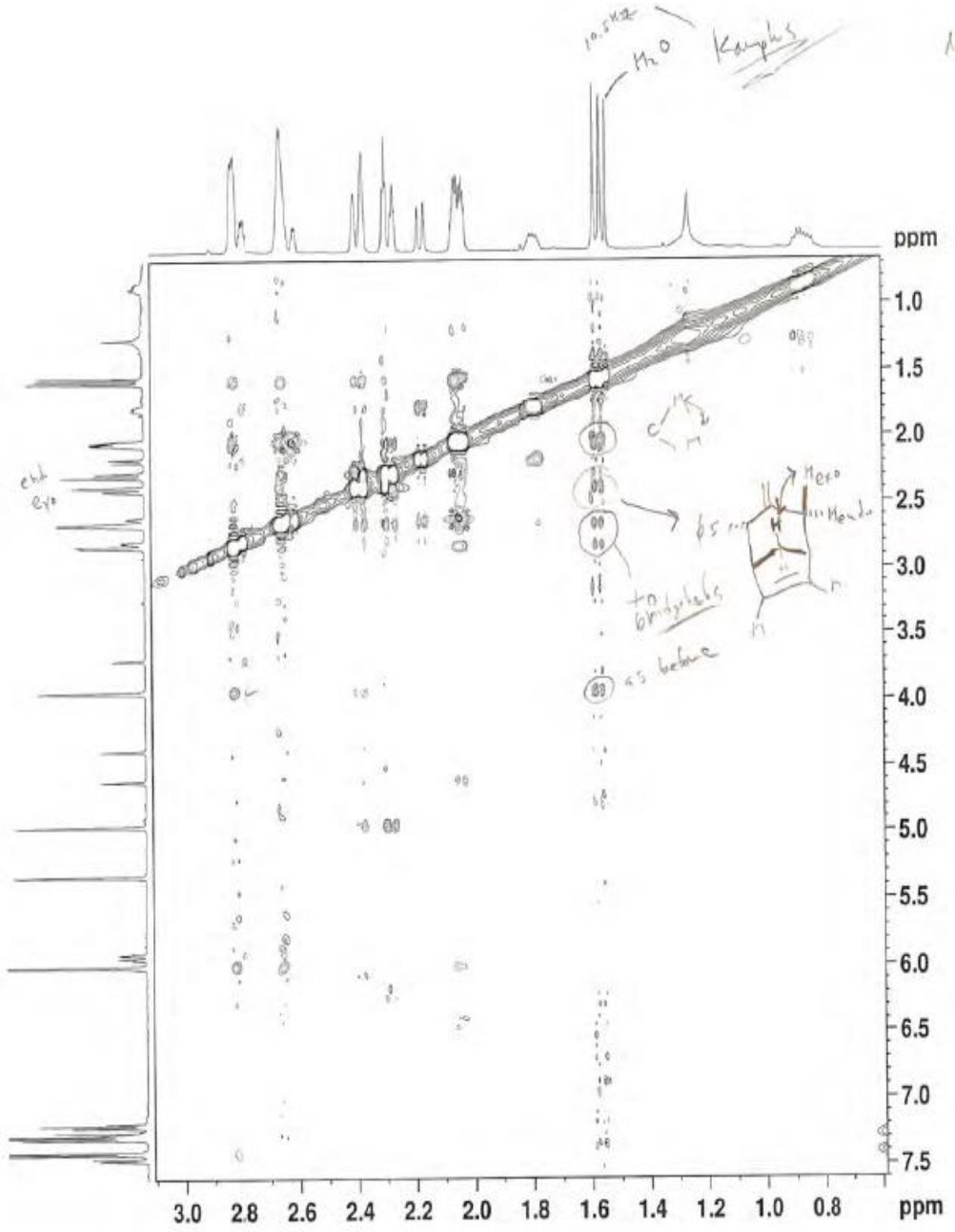
NOESY

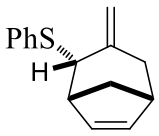



PhS
H

10a/b

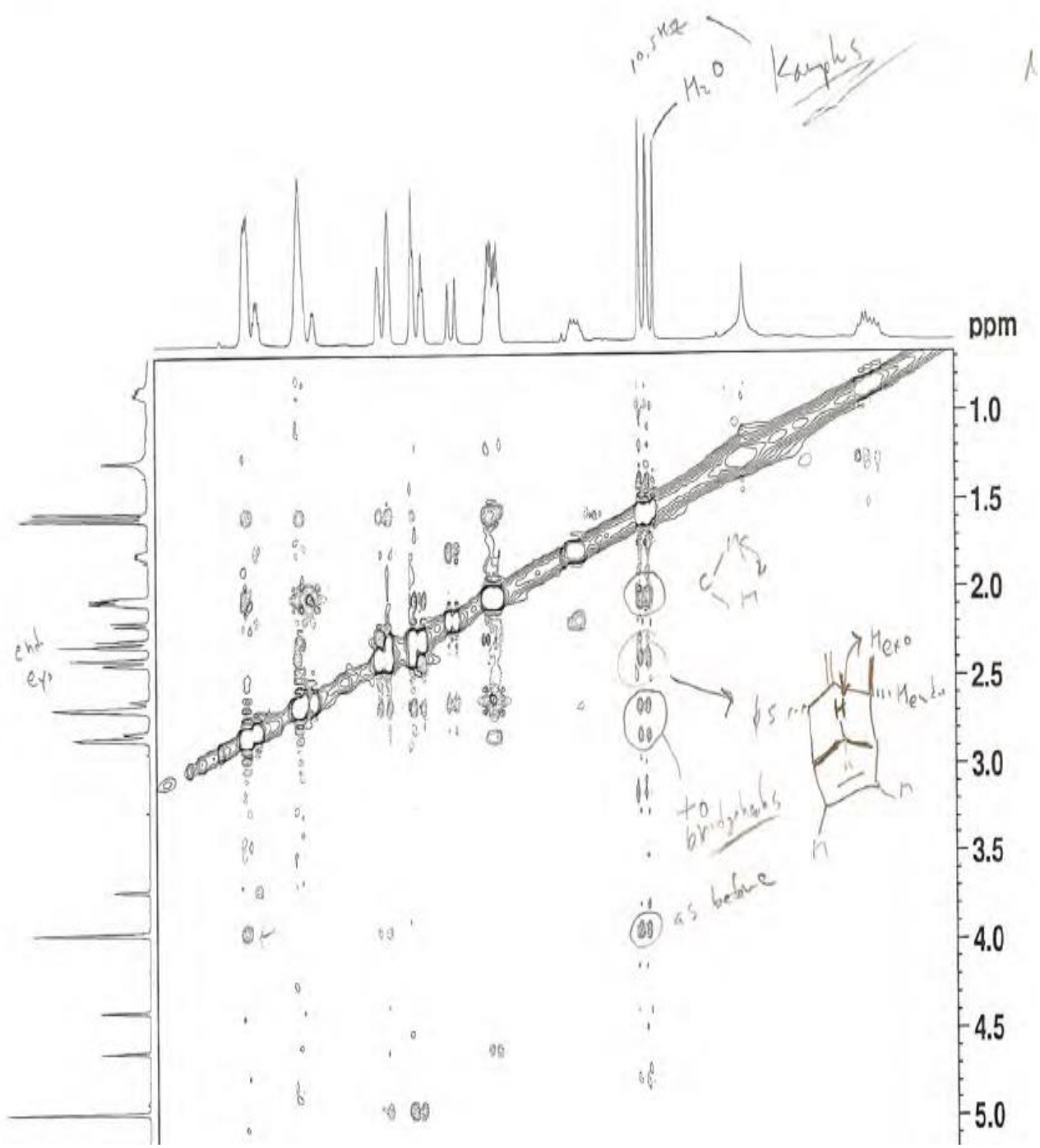
NOESY

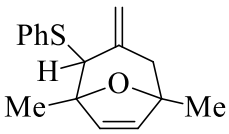

ppm
1.0
1.5
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.5
6.0
6.5
7.0
7.5
3.0
2.8
2.6
2.4
2.2
2.0
1.8
1.6
1.4
1.2
1.0
0.8
ppm



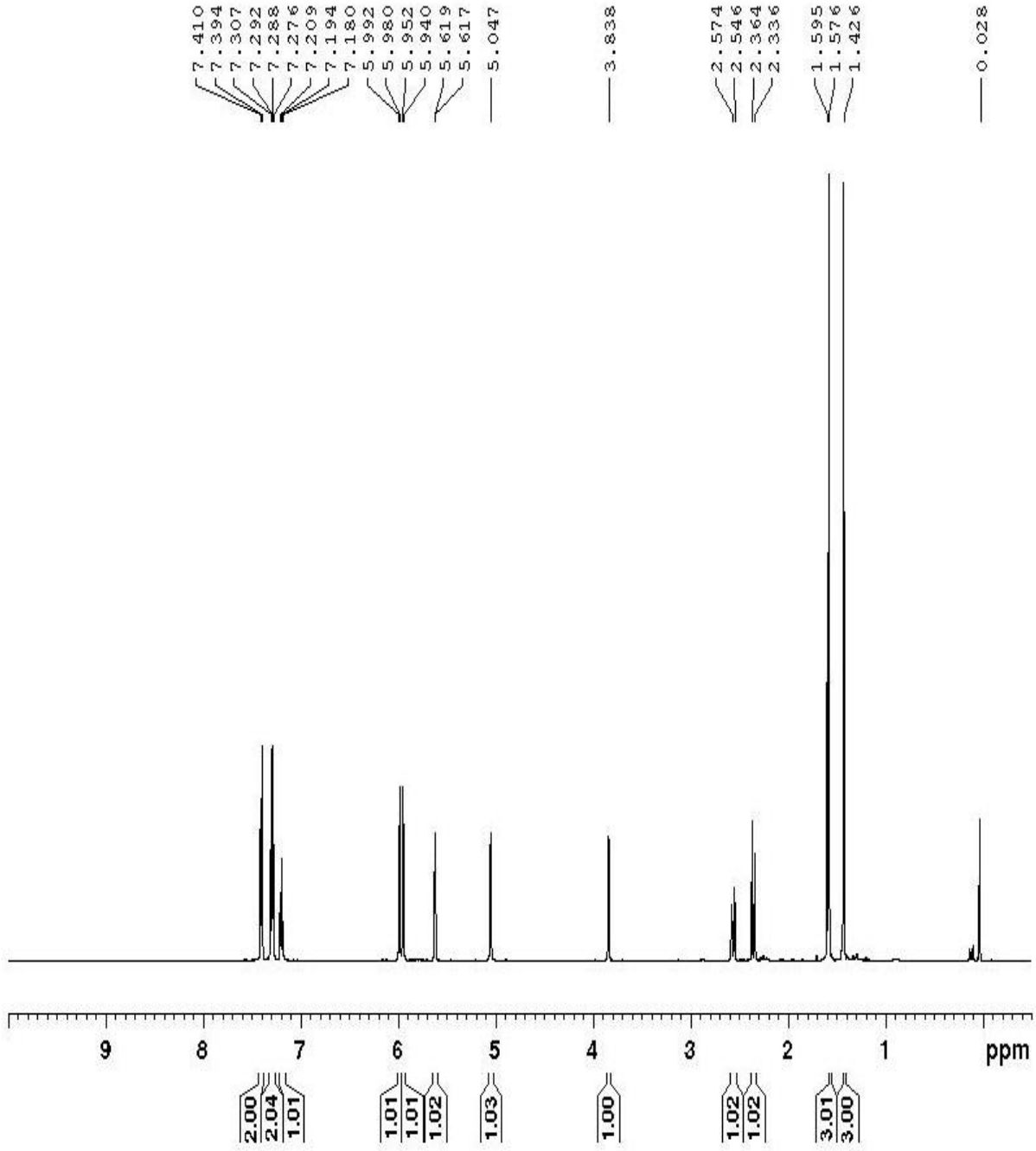
10a/b

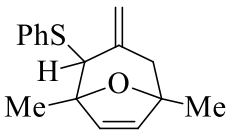
NOESY



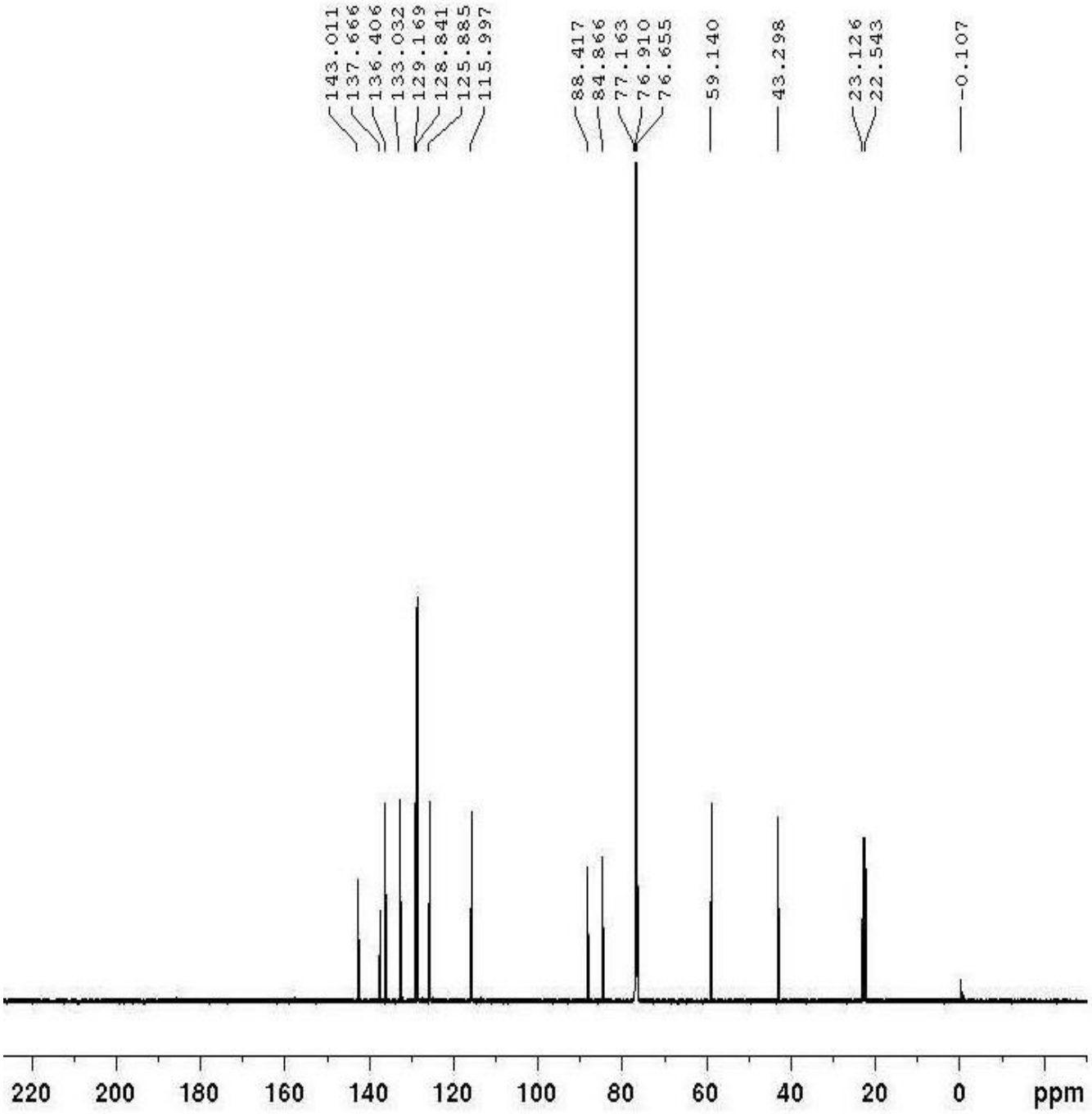

PhS
H
O
Me
Me

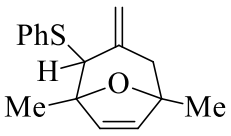
16a


7.410
7.394
7.307
7.292
7.288
7.276
7.209
7.194
7.180
5.992
5.980
5.952
5.940
5.619
5.617
5.047
3.838
2.574
2.546
2.364
2.336
1.595
1.576
1.426
0.028
9
8
7
6
5
4
3
2
1
ppm
2.00
2.04
1.01
1.01
1.01
1.02
1.03
1.00
1.02
1.02
3.01
3.00

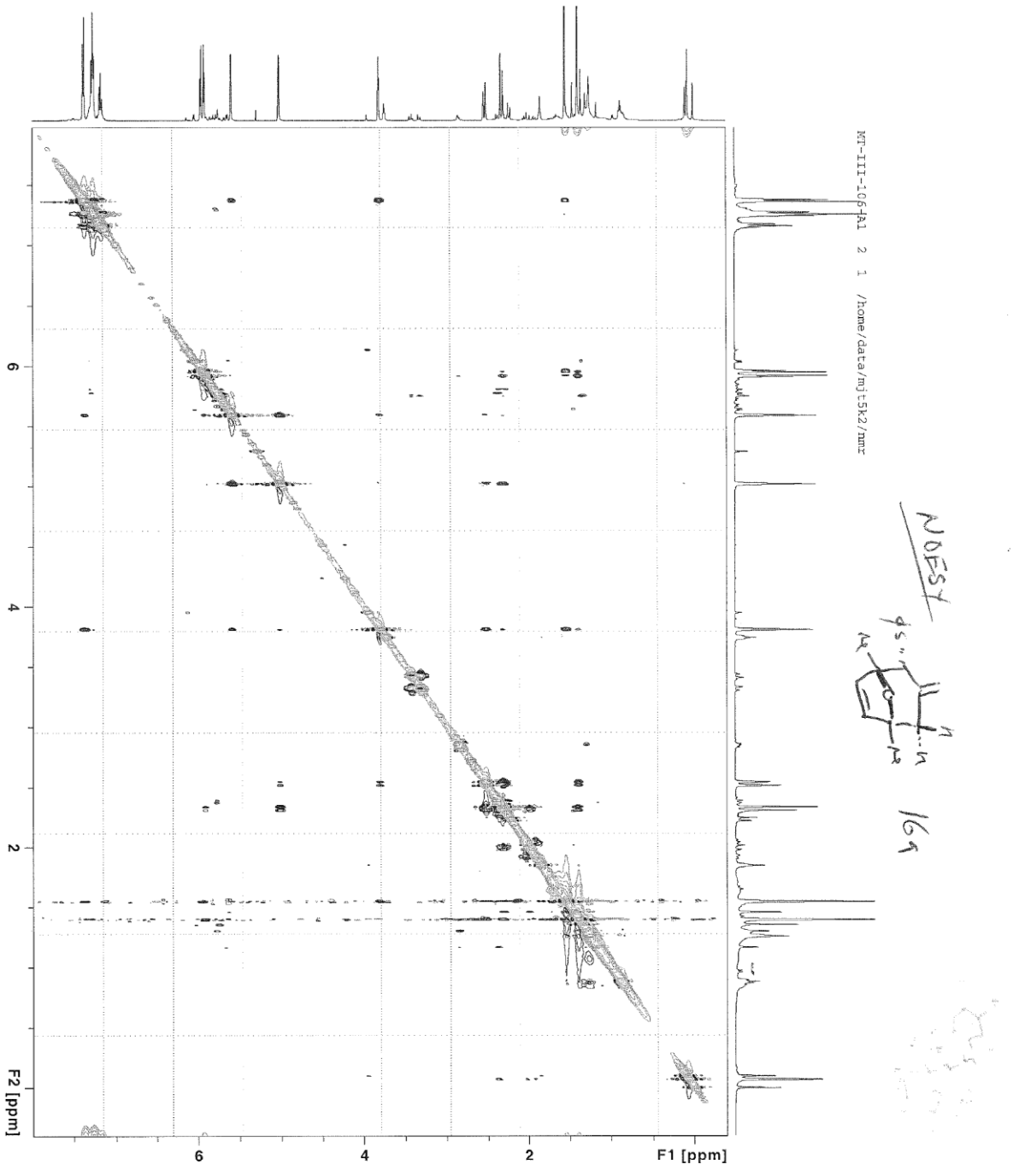

PhS
H
O
Me
Me

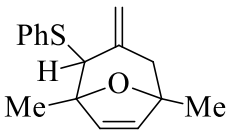
16a


143.011
137.666
136.406
133.032
129.169
128.841
125.885
115.997
88.417
84.866
77.163
76.910
76.655
59.140
43.298
23.126
22.543
-0.107
220
200
180
160
140
120
100
80
60
40
20
0
ppm

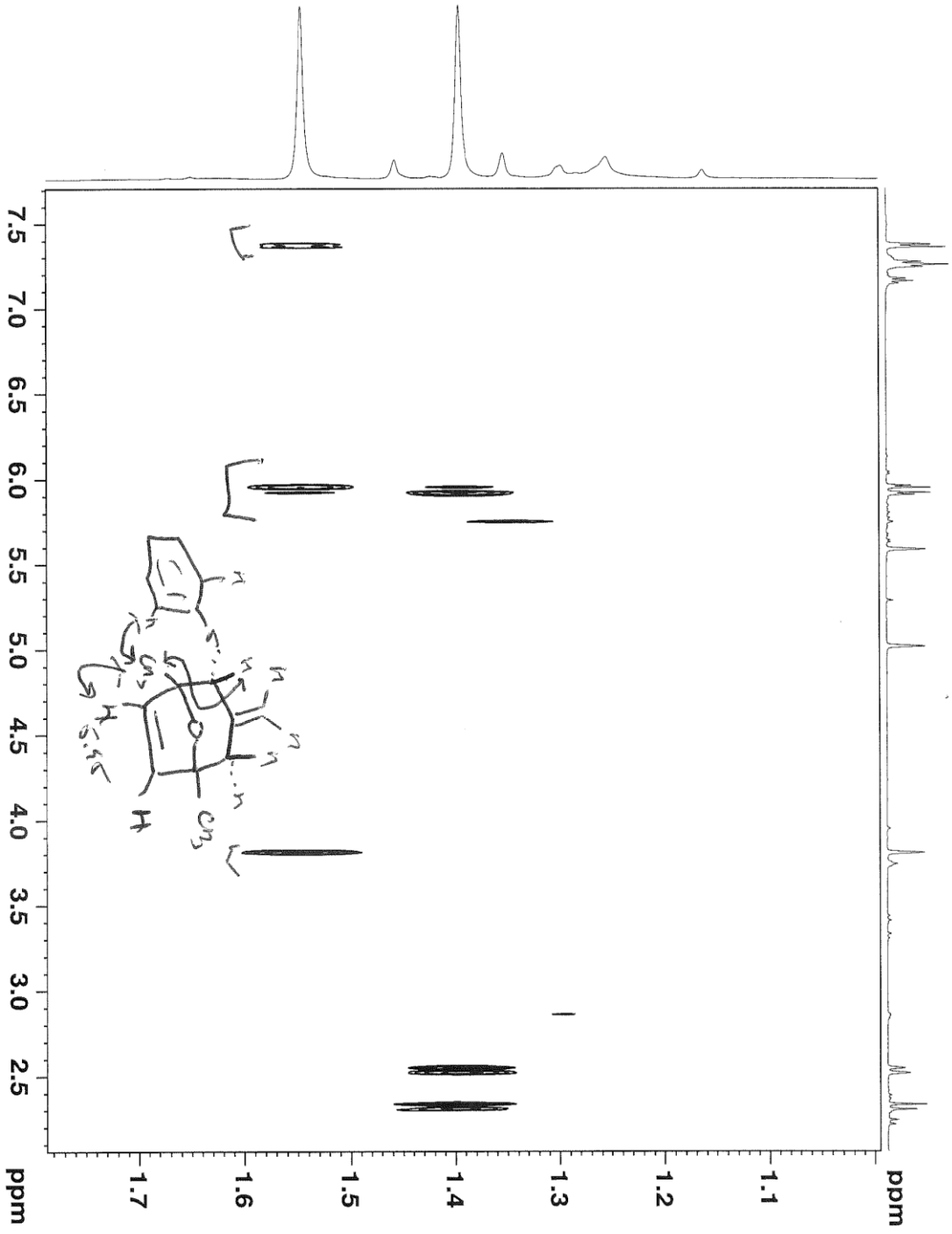

PhS
H
O
Me
Me

16a

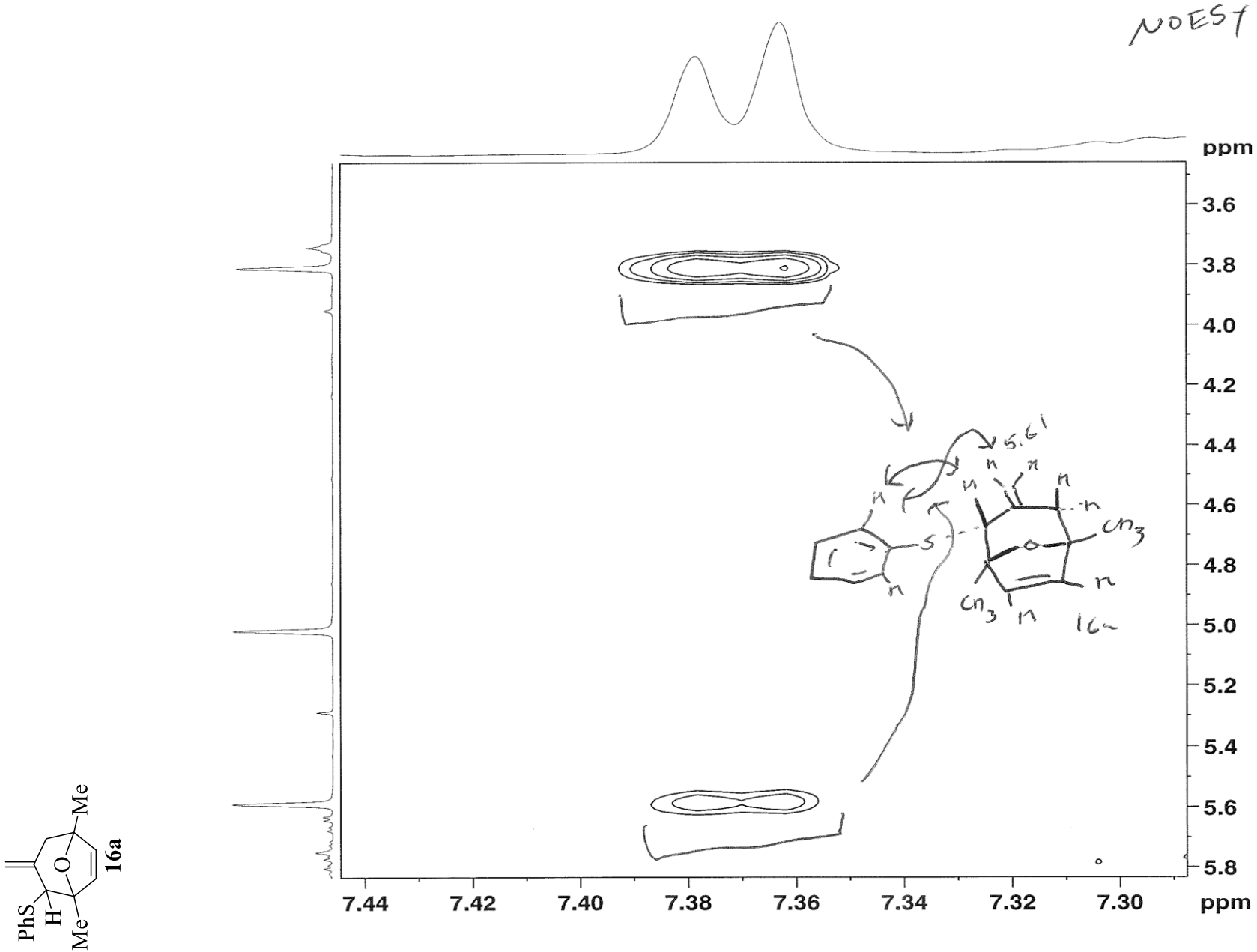

MT-III-106-A1 2 1 /home/data/mj5k2/nmr
F2 [ppm]
F1 [ppm]
6
4
2
NOESY
169

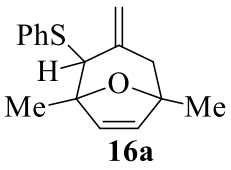

PhS
H
Me
O
Me

16a

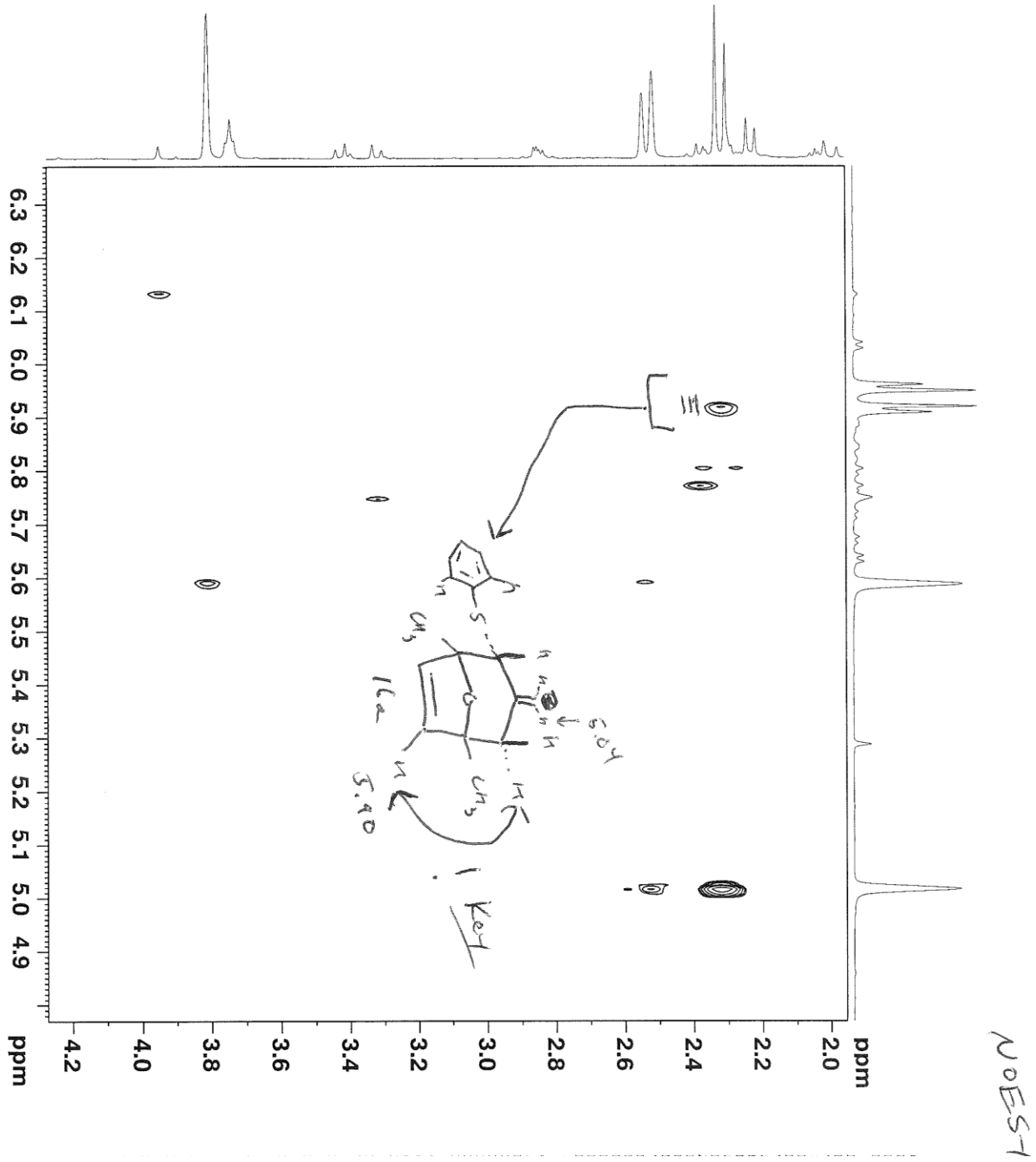

7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
ppm
1.7
1.6
1.5
1.4
1.3
1.2
1.1
ppm
H
CH3

NOESY


NOESY
ppm
3.6
3.8
4.0
4.2
4.4
4.6
4.8
5.0
5.2
5.4
5.6
5.8
7.44
7.42
7.40
7.38
7.36
7.34
7.32
7.30
ppm
5.61
S
O
16a
PhS
H
Me
Me
O
16a


PhS
H
Me
O
Me

16a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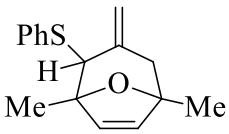
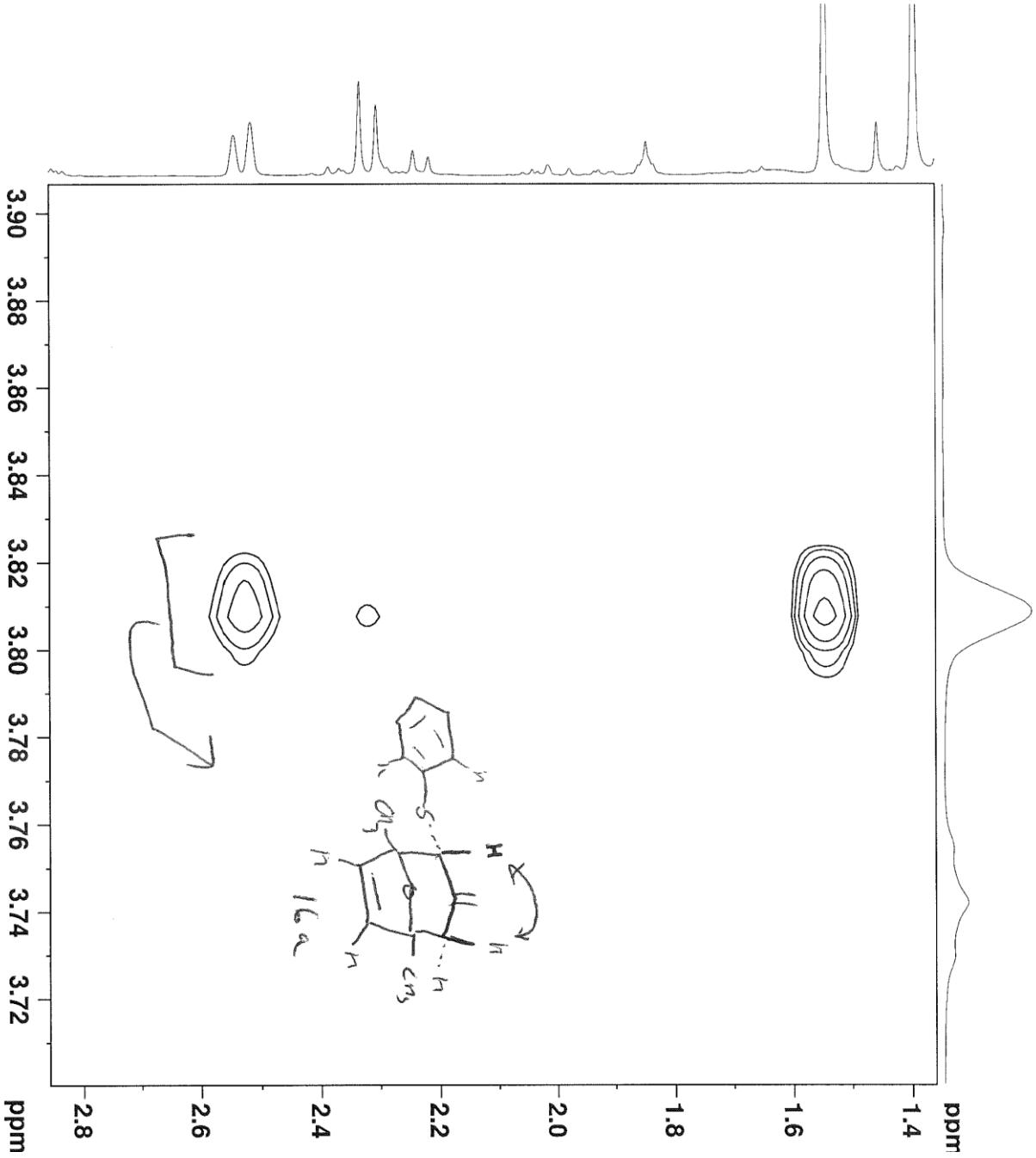
ppm
4.2 4.0 3.8 3.6 3.4 3.2 3.0 2.8 2.6 2.4 2.2 2.0
ppm
5.04
3.90
16a
key

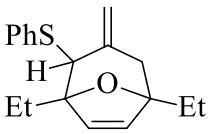
NOESY


PhS
H
O
Me
Me

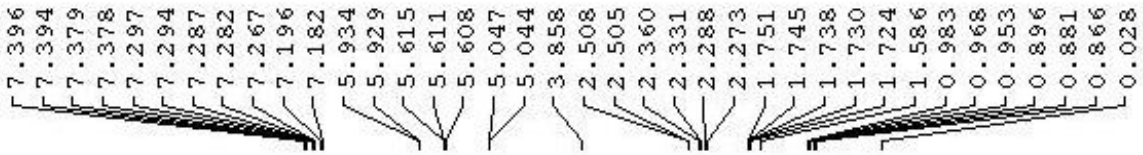
16a

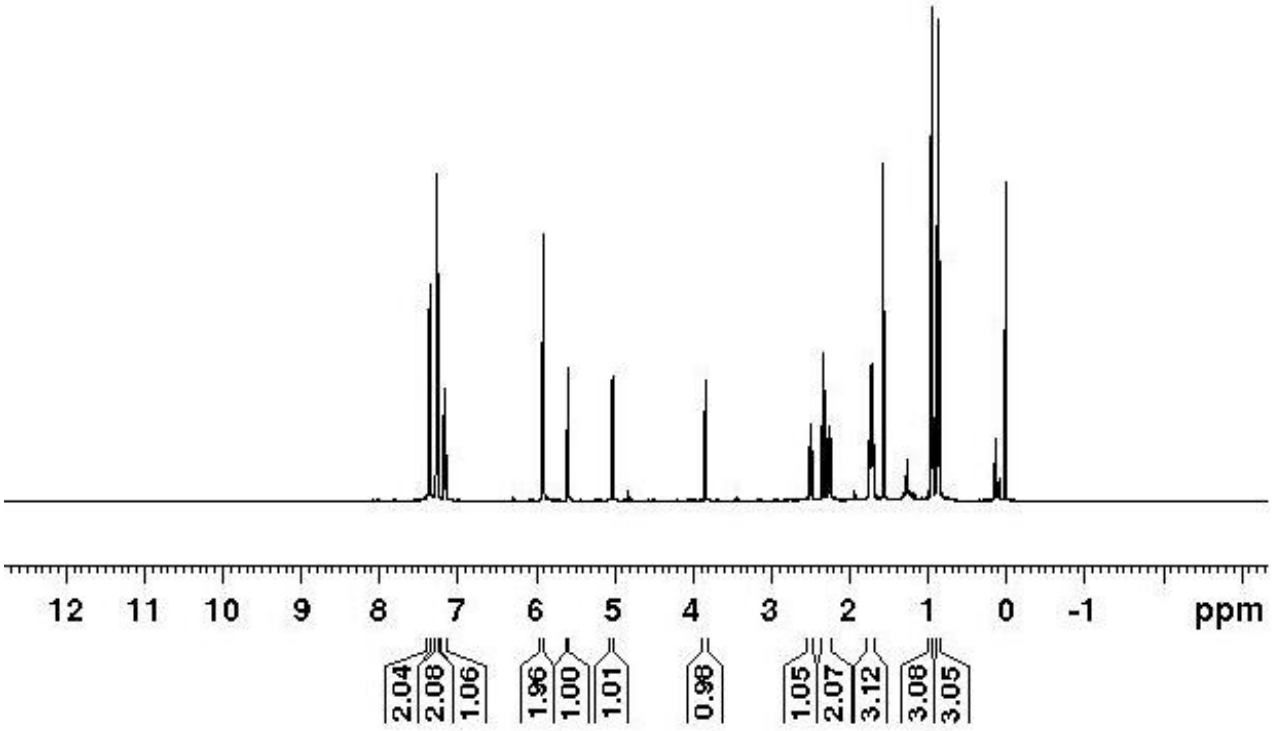
NOESY

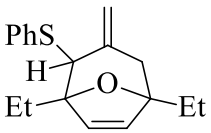

3.90
3.88
3.86
3.84
3.82
3.80
3.78
3.76
3.74
3.72
ppm
2.8
2.6
2.4
2.2
2.0
1.8
1.6
1.4
ppm
H
16a


PhS
H
O
Et
Et

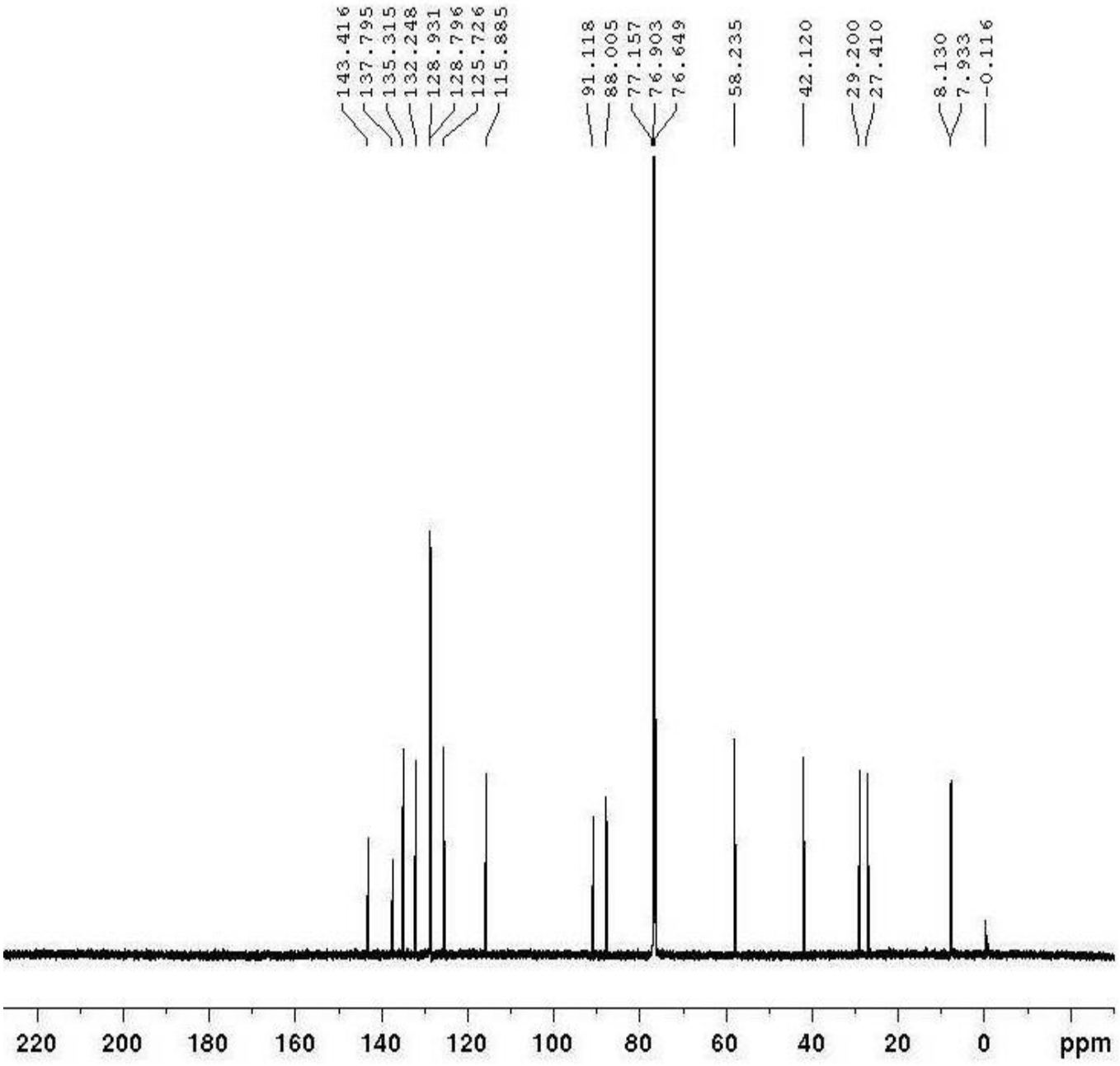
16b


7.396
7.394
7.379
7.378
7.297
7.294
7.287
7.282
7.267
7.196
7.182
5.934
5.929
5.615
5.611
5.608
5.047
5.044
3.858
2.508
2.505
2.360
2.331
2.288
2.273
1.751
1.745
1.738
1.730
1.724
1.586
0.983
0.968
0.953
0.896
0.881
0.866
0.028


12
11
10
9
8
7
6
5
4
3
2
1
0
-1
ppm
2.04
2.08
1.06
1.96
1.00
1.01
0.98
1.05
2.07
3.12
3.08
3.05


PhS
H
O
Et
Et

16b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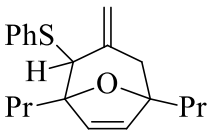
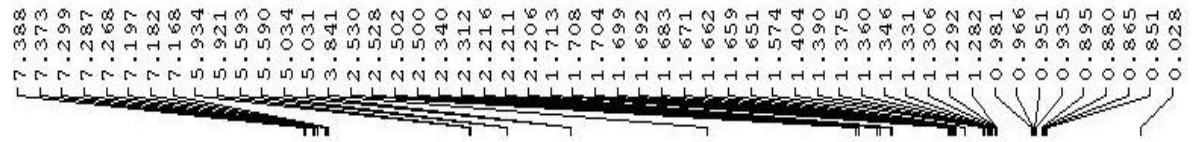
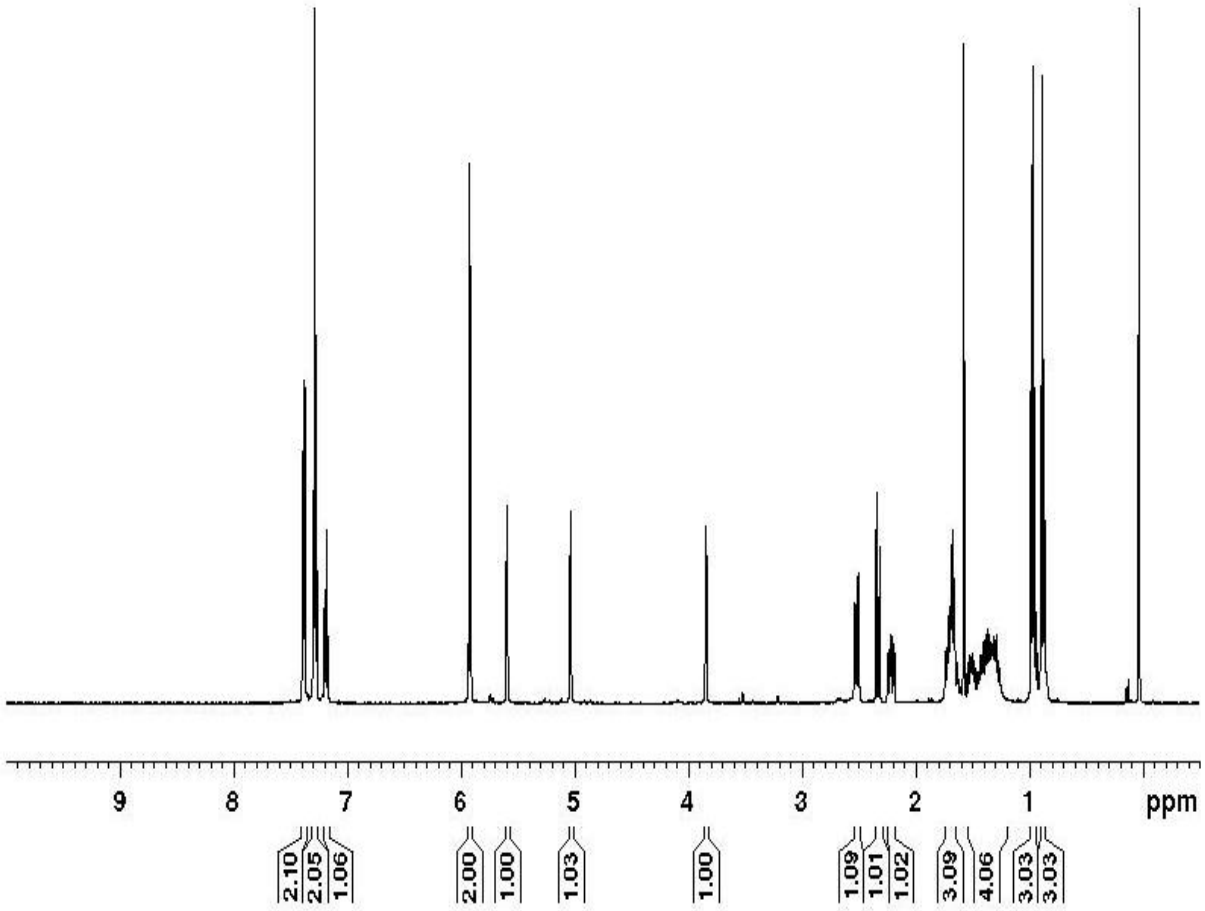
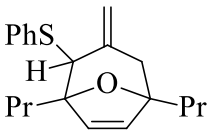
137.795
135.315
132.248
128.931
128.796
125.726
115.885
91.118
88.005
77.157
76.903
76.649
58.235
42.120
29.200
27.410
8.130
7.933
-0.116
220
200
180
160
140
120
100
80
60
40
20
0
ppm


PhS
H
O
Pr
Pr

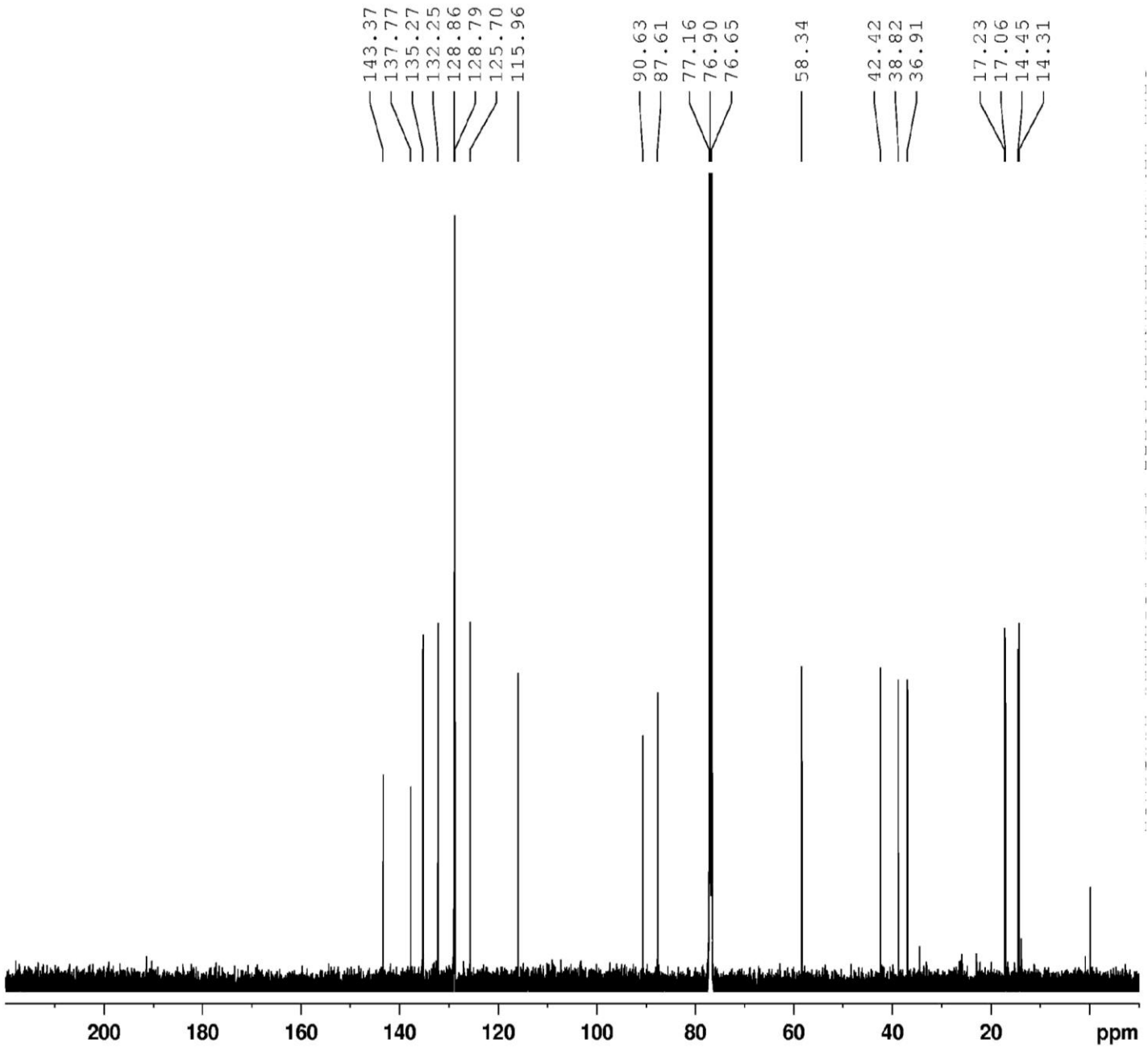
16c


7.388
7.373
7.299
7.287
7.268
7.197
7.182
7.168
5.934
5.921
5.593
5.590
5.034
5.031
3.841
2.530
2.528
2.502
2.500
2.340
2.312
2.216
2.211
2.206
1.713
1.708
1.704
1.699
1.692
1.683
1.671
1.662
1.659
1.651
1.574
1.404
1.390
1.375
1.360
1.346
1.331
1.306
1.292
1.282
0.981
0.966
0.951
0.935
0.895
0.880
0.865
0.851
0.028


9
8
7
6
5
4
3
2
1
ppm
2.10
2.05
1.06
2.00
1.00
1.03
1.00
1.09
1.01
1.02
3.09
4.06
3.03
3.03


PhS
H
O
Pr
Pr

16c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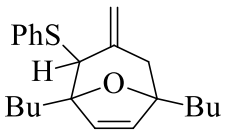
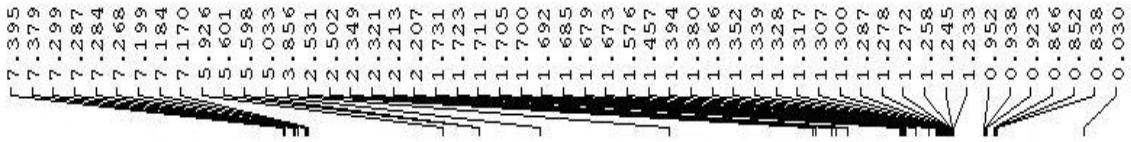
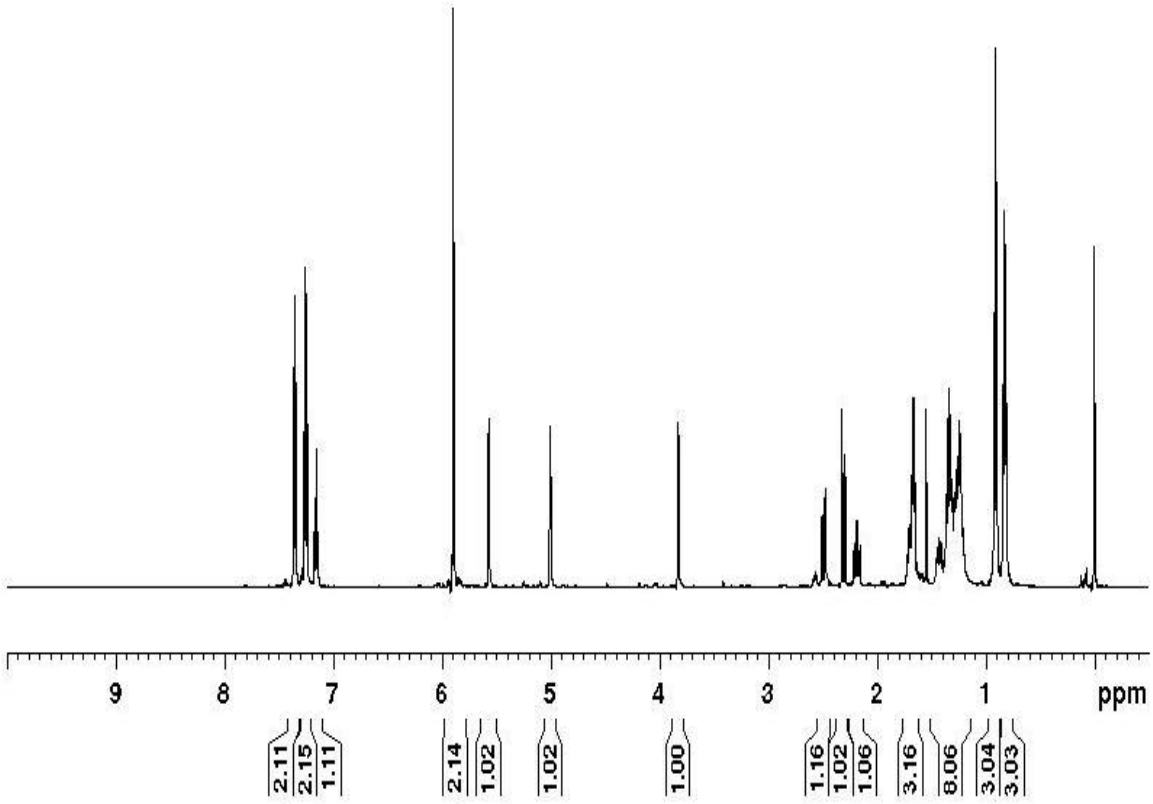
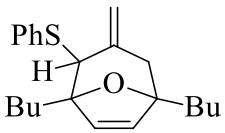
135.27
132.25
128.86
128.79
125.70
115.96
90.63
87.61
77.16
76.90
76.65
58.34
42.42
38.82
36.91
17.23
17.06
14.45
14.31
200
180
160
140
120
100
80
60
40
20
ppm


PhS
H
O
Bu
Bu

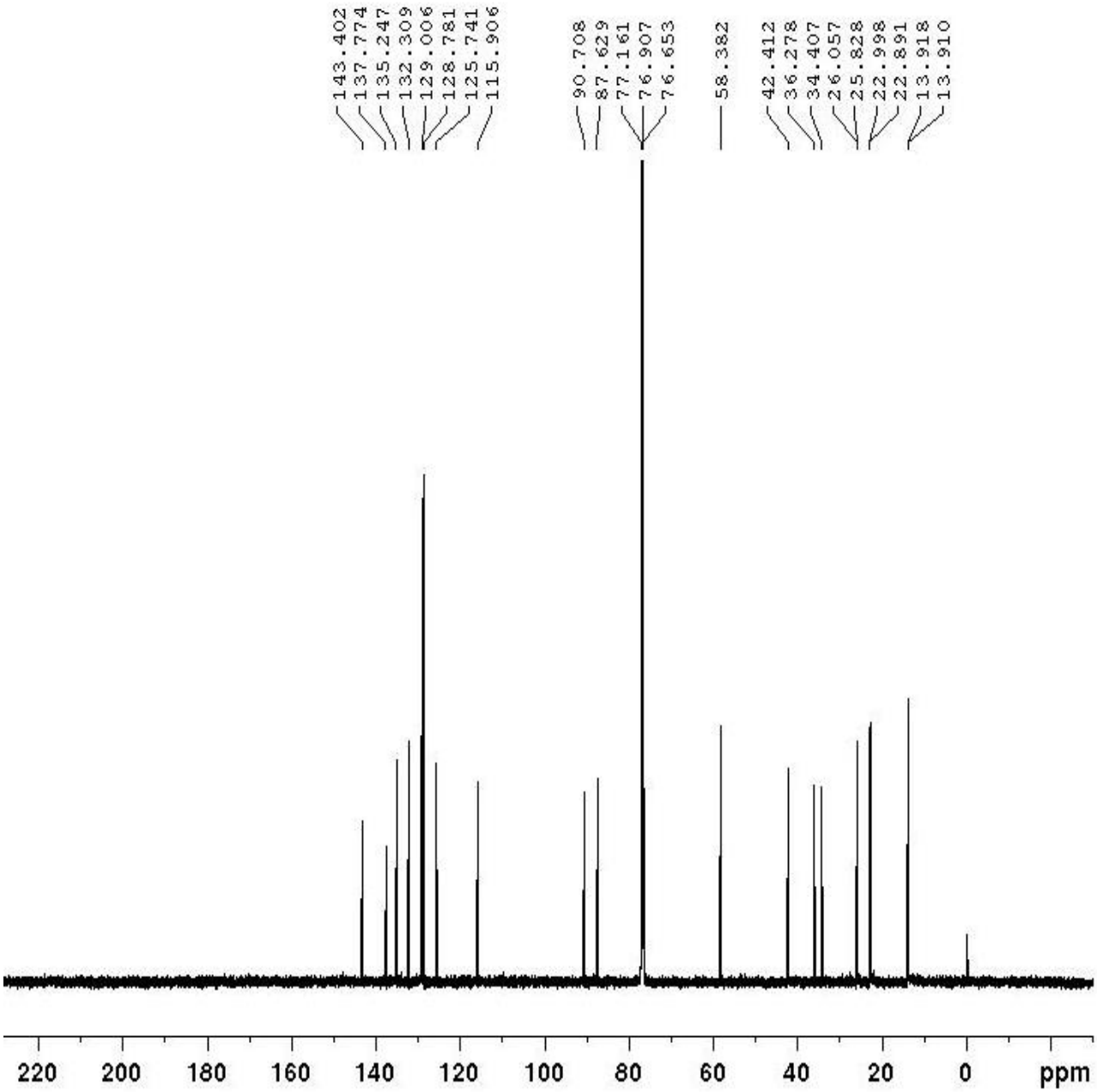
16d


7.395
7.379
7.299
7.287
7.284
7.268
7.199
7.184
7.170
5.926
5.601
5.598
5.033
3.856
2.531
2.502
2.349
2.321
2.213
2.207
1.731
1.723
1.711
1.705
1.700
1.692
1.685
1.679
1.673
1.576
1.457
1.394
1.380
1.366
1.352
1.339
1.328
1.317
1.307
1.300
1.287
1.278
1.272
1.258
1.245
1.233
0.952
0.938
0.923
0.866
0.852
0.838
0.030


9
8
7
6
5
4
3
2
1
ppm
2.11
2.15
1.11
2.14
1.02
1.02
1.00
1.16
1.02
1.06
3.16
8.06
3.04
3.03


PhS
H
O
Bu
Bu

16d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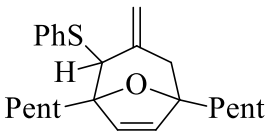
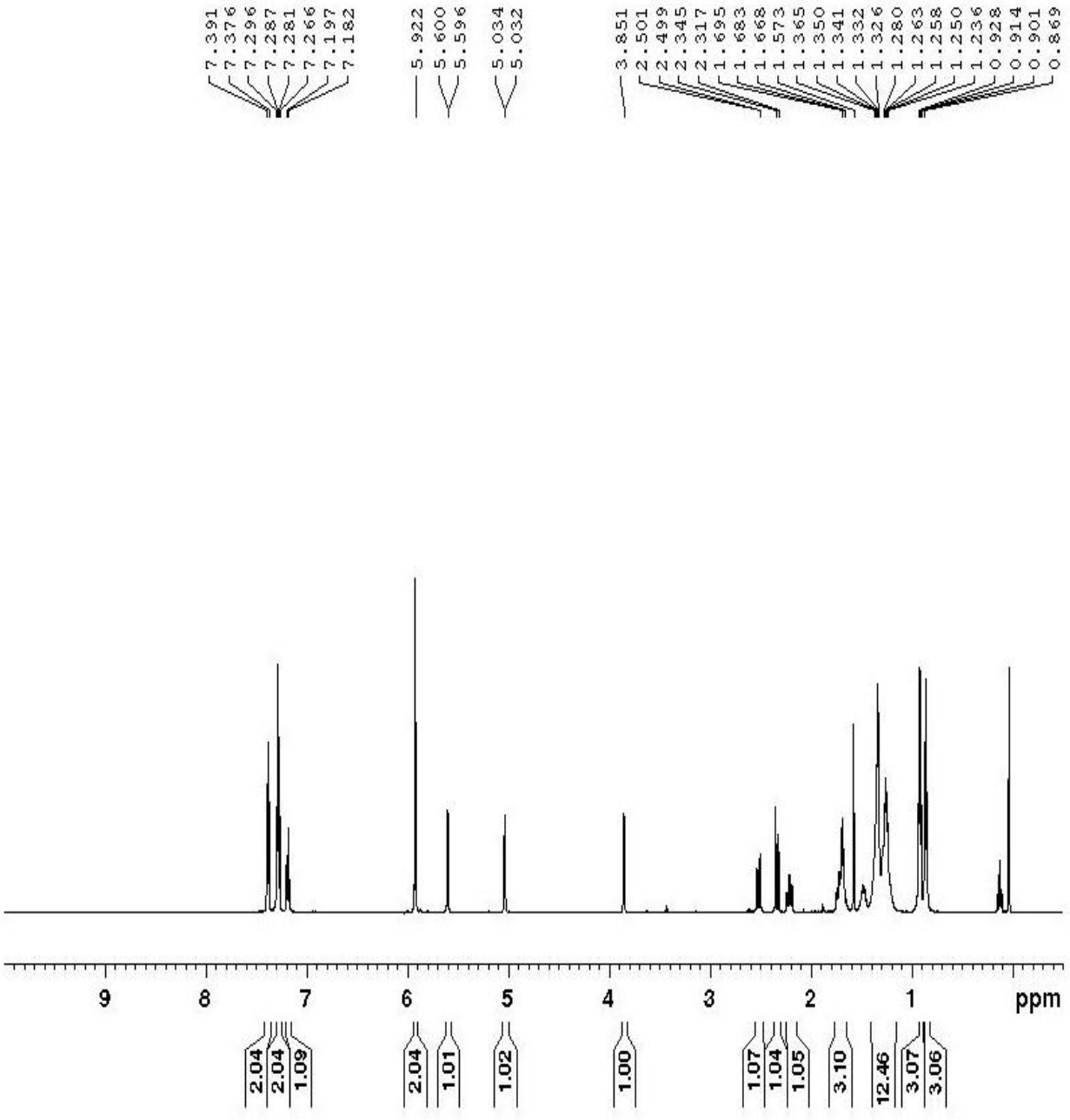
135.247
132.309
129.006
128.781
125.741
115.906
90.708
87.629
77.161
76.907
76.653
58.382
42.412
36.278
34.407
26.057
25.828
22.998
22.891
13.918
13.910
220
200
180
160
140
120
100
80
60
40
20
0
ppm


PhS
H
O
Pent
Pent

16e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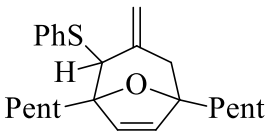
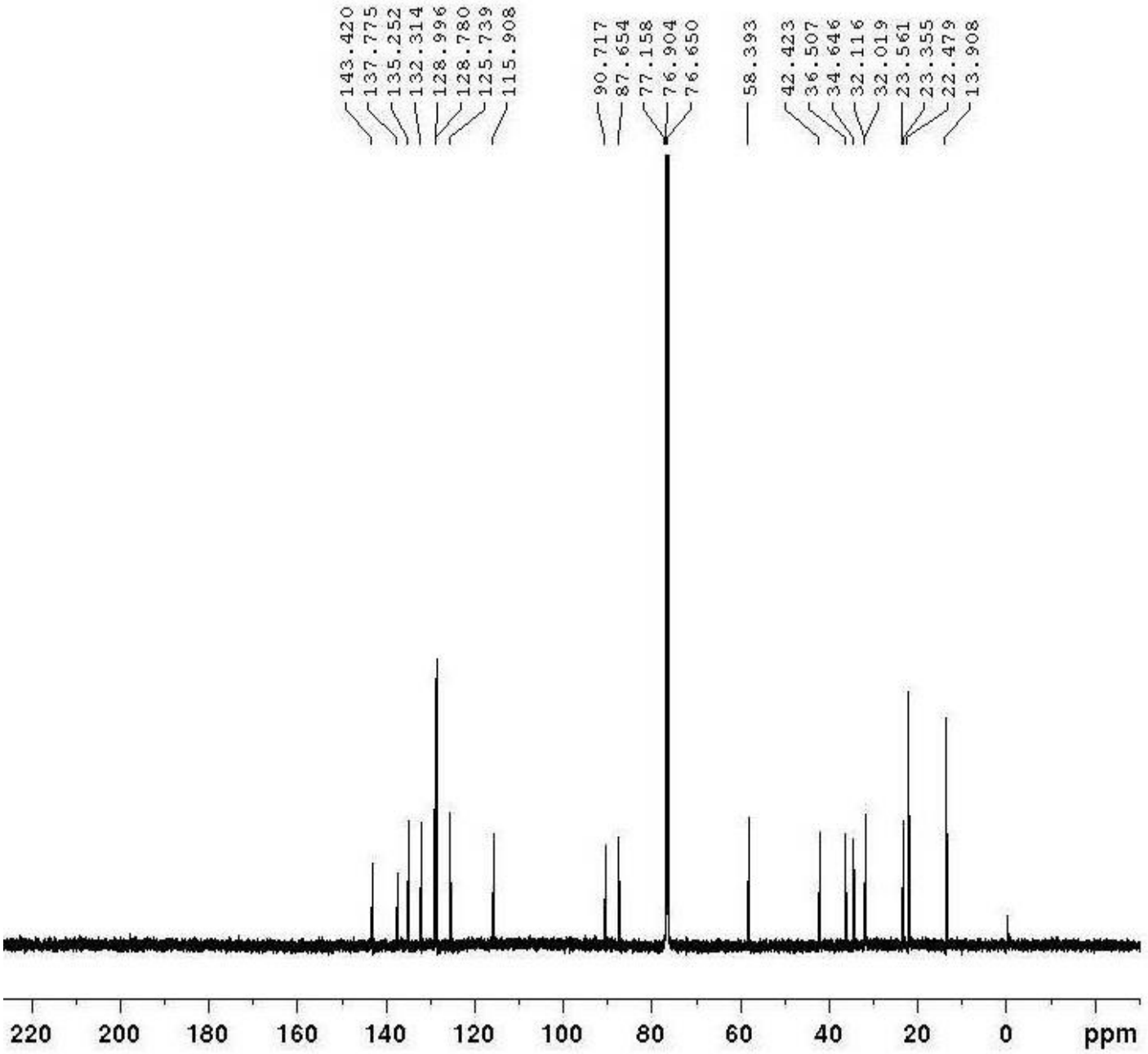
7.376
7.296
7.287
7.281
7.266
7.197
7.182
5.922
5.600
5.596
5.034
5.032
3.851
2.501
2.499
2.345
2.317
1.695
1.683
1.668
1.573
1.365
1.350
1.341
1.332
1.326
1.280
1.263
1.258
1.250
1.236
0.928
0.914
0.901
0.869
9
8
7
6
5
4
3
2
1
ppm
2.04
2.04
1.09
2.04
1.01
1.02
1.00
1.07
1.04
1.05
3.10
12.46
3.07
3.06


PhS
H
O
Pent
Pent

16e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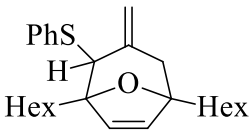
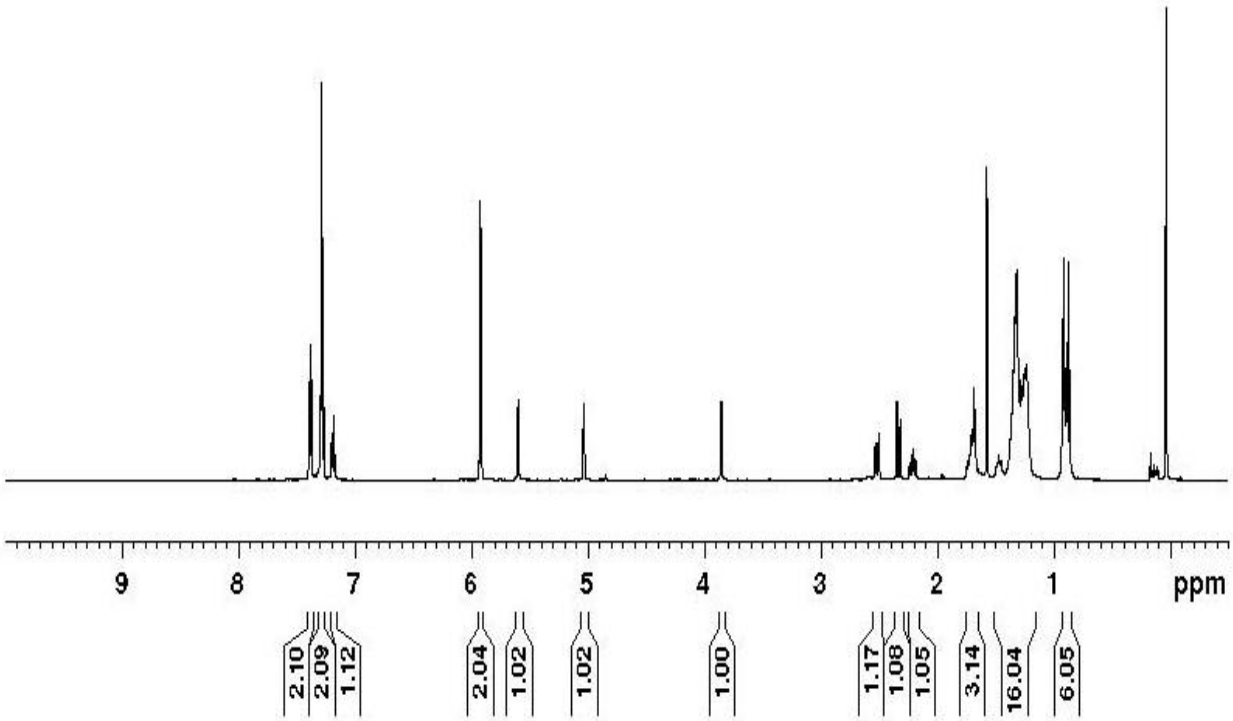
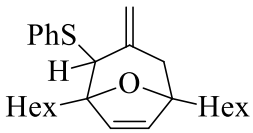
132.314
128.996
128.780
125.739
115.908
90.717
87.654
77.158
76.904
76.650
58.393
42.423
36.507
34.646
32.116
32.019
23.561
23.355
22.479
13.908
220
200
180
160
140
120
100
80
60
40
20
0
ppm


PhS
H
O
Hex
Hex

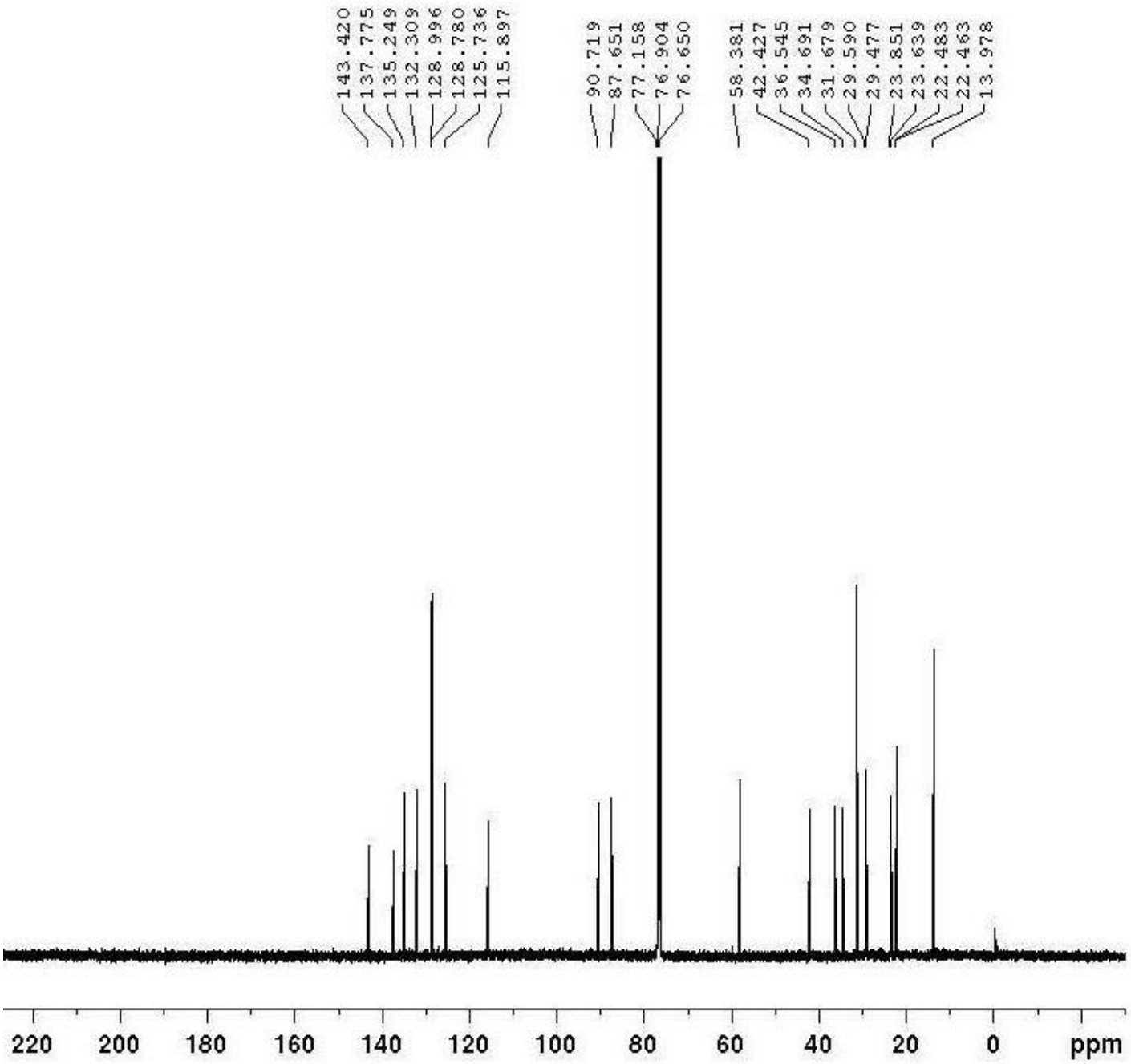
16f


7.390
7.375
7.296
7.287
7.281
7.266
7.196
7.182
5.919
5.596
5.594
5.031
3.850
2.498
2.342
2.314
1.699
1.696
1.682
1.676
1.667
1.569
1.342
1.330
1.313
1.283
1.271
1.256
1.245
1.233
1.226
0.921
0.908
0.894


9
8
7
6
5
4
3
2
1
ppm
2.10
2.09
1.12
2.04
1.02
1.02
1.00
1.17
1.08
1.05
3.14
16.04
6.05


PhS
H
O
Hex
Hex

16f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137.775
135.249
132.309
128.996
128.780
125.736
115.897
90.719
87.651
77.158
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76.650
58.381
42.427
36.545
34.691
31.679
29.590
29.477
23.851
23.639
22.483
22.463
13.978
220
200
180
160
140
120
100
80
60
40
20
0
ppm