

Supporting Information

Chemistry Letters

Oxidative Ring Opening of Benzocyclobutenone Oximes: Novel Access to Stable Nitrile Oxides

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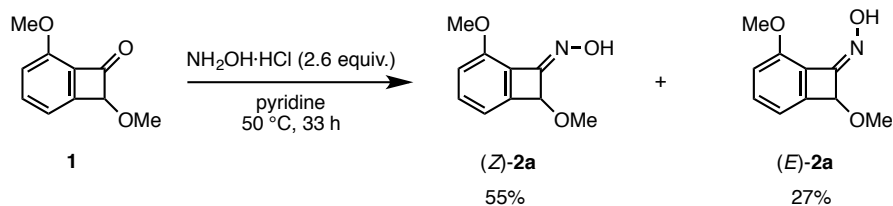
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General experimental procedures

All reactions utilizing air- or moisture-sensitive reagents were performed in dried glassware under an atmosphere of dry argon. Ethereal solvents (anhydrous; *Kanto Chemical Co., Inc.*) were used as received. MeOH was distilled from MgSO₄, and stored over 4A molecular sieves. Other reagents were used without further purification as received from commercial. For thin-layer chromatography (TLC) analysis, Merck pre-coated plates (TLC silica gel 60 F254, Art 5715, 0.25 nm) were used. Silica-gel preparative thin-layer chromatography (PTLC) was performed using plates prepared from Merck silica gel 60 PF254 (Art 7747). For flash column chromatography, silica gel 60N (Spherical, neutral, 63–210 μm) from Kanto Chemical was used. Melting point (mp) determinations were performed using a Yanaco MP-500 instrument or METTLER TOLEDO MP70 melting point system, and are uncorrected. ¹H- and ¹³C-NMR were measured on a Bruker Avance III (600 MHz) spectrometer in the solvent indicated; Chemical shifts (δ) are expressed in parts per million (ppm) downfield from internal standard (tetramethylsilane, 0.00 ppm), and coupling constants (*J*) are reported as hertz (Hz). Splitting patterns are indicated as follows: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, br = broad. Infrared (IR) spectra were recorded on a Thermo Scientific Nicolet iS5 FT-IR spectrometer. Attenuated total reflectance Fourier transform infrared (ATR-FTIR) spectra were recorded by using a Thermo Scientific Nicolet iS5 FT-IR spectrometer equipped with a universal ATR sampling accessory (iD5 ATR). Elemental analyses were recorded on an Elementar vario MICRO cube analyzer. High-resolution mass spectra (HRMS) were obtained with a Bruker micrOTOF-QII spectrometer.

Equation 1



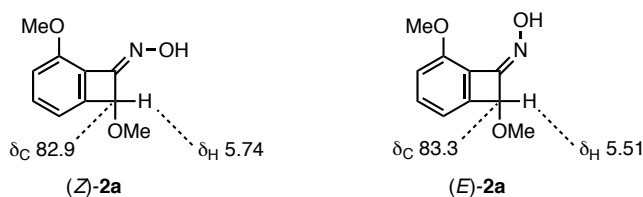
To a solution of benzocyclobutenone **1** (192 mg, 1.08 mmol) in pyridine (5.4 mL) was added $\text{NH}_2\text{OH}\cdot\text{HCl}$ (113 mg, 1.63 mmol) at $0\text{ }^\circ\text{C}$. After stirring for 8 h, the reaction was quenched by adding water, and the mixture was extracted with EtOAc (x3). The combined organic extracts were washed with 10% aqueous CuSO_4 (x1), water, and brine, dried (Na_2SO_4), and concentrated in vacuo. The residue was purified by flash column chromatography (SiO_2 , hexane/EtOAc = 3/1) to give oxime **(Z)-2a** (115 mg, 55%) and oxime **(E)-2a** (55.6 mg, 27%) both as white solids.

Oxime **(Z)-2a**: mp $144\text{--}145\text{ }^\circ\text{C}$ (hexane/EtOAc, colorless needle); R_f 0.60 (hexane/EtOAc = 1/1); $^1\text{H NMR}$ (600 MHz, acetone- d_6) δ 3.45 (s, 3H), 4.03 (s, 3H), 5.59 (s, 1H), 6.91 (d, 1H, $J = 8.4$ Hz), 7.06 (d, 1H, $J = 7.2$ Hz), 7.36 (dd, 1H, $J = 8.4, 7.2$ Hz), 10.02 (s, 1H, OH); $^{13}\text{C NMR}$ (150 MHz, acetone- d_6) δ 55.8, 58.3, 83.9, 116.4, 117.8, 126.7, 133.9, 149.8, 154.2, 154.3; **IR** (neat) 3183, 3122, 2997, 2952, 2881, 2830, 1690, 1604, 1485, 1454, 1272, 1134, 1047, 933 cm^{-1} ; **HRMS** (ESI) calcd for $\text{C}_{10}\text{H}_{12}\text{NO}_3$ ($[\text{M}+\text{H}]^+$) m/z 194.0812, found m/z 194.0813.

Oxime **(E)-2a**: mp $123\text{--}124\text{ }^\circ\text{C}$ (hexane/EtOAc, colorless needle); R_f 0.37 (hexane/EtOAc = 1/1); $^1\text{H NMR}$ (600 MHz, acetone- d_6) δ 3.44 (s, 3H), 3.90 (s, 3H), 5.37 (s, 1H), 7.04 (d, 1H, $J = 8.4$ Hz), 7.09 (d, 1H, $J = 7.2$ Hz), 7.48 (dd, 1H, $J = 8.4, 7.2$ Hz), 9.76 (s, 1H, OH); $^{13}\text{C NMR}$ (150 MHz, acetone- d_6) δ 56.3, 56.4, 83.9, 113.8, 116.1, 128.6, 135.0, 150.9, 151.2, 153.3; **IR** (ATR) 3177, 3104, 2988, 2934, 2843, 1697, 1592, 1487, 1281, 1050, 952 cm^{-1} ; **HRMS** (ESI) calcd for $\text{C}_{10}\text{H}_{12}\text{NO}_3$ ($[\text{M}+\text{H}]^+$) m/z 194.0812, found m/z 194.0816.

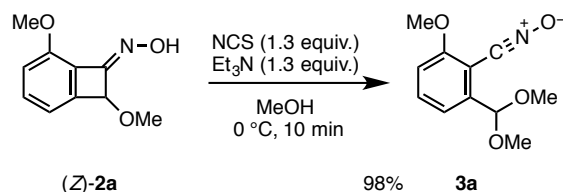
Based on the relation of the oximes geometry and $^{13}\text{C NMR}$ chemical shifts, the structures of **(Z)-2a** and **(E)-2a** were assigned as such by $^{13}\text{C NMR}$ analysis: The α -carbon to oxime moiety in **(Z)-2a** resonates at higher fields (δ_{C} 82.9) than that of **(E)-2a** (δ_{C} 83.3).

In addition, the α -proton in **(Z)-2a** resonates at lower field (δ_{H} 5.74) than that in **(E)-2a** (δ_{H} 5.51) due to the deshielding effect of the *N*-hydroxy group.*



*For the relation of *E/Z* geometries of oximes and $^{13}\text{C NMR}$ chemical shifts, see: G. C. Levy, G. L. Nelson, *J. Am. Chem. Soc.* **1972**, *94*, 4897.

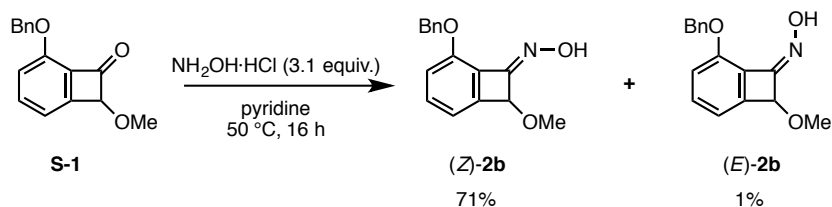
Scheme 2



To a solution of oxime (*Z*)-**2a** (57.9 mg, 0.300 mmol) and Et₃N (55.0 μL, 0.395 mmol) in MeOH (3.0 mL) was added NCS (52.1 mg, 0.390 mmol) at 0 °C. After stirring for 10 min at 0 °C, the reaction was stopped by adding water and brine, and the mixture was extracted with EtOAc (x3). The combined organic extracts were washed with brine, dried (MgSO₄), and concentrated in vacuo. The residue was purified by flash column chromatography (silica gel, hexane/EtOAc = 4/1) to afford nitrile oxide **3a** (65.9 mg, 98%) as colorless oil (solidified in a refrigerator as a white solid).

R_f 0.66 (hexane/EtOAc/CH₂Cl₂ = 2/1/1); ¹H NMR (600 MHz, CDCl₃) δ 3.38 (s, 6H), 3.91 (s, 3H), 5.45 (s, 1H), 6.91 (d, 1H, *J* = 8.4 Hz), 7.21 (d, 1H, *J* = 7.8 Hz), 7.42 (dd, 1H, *J* = 8.4, 7.8 Hz); ¹³C NMR (150 MHz, CDCl₃) δ 33.0, 53.6, 53.7, 56.1, 101.8, 111.1, 119.3, 131.5, 142.7, 161.9; IR (ATR) 2935, 2294, 1471, 1367, 1331, 1277, 1120, 1103, 1050, 990, 932, 815, 777 cm⁻¹; HRMS (ESI) calcd for C₁₁H₁₄NO₄ ([M+H]⁺) *m/z* 224.0917, found *m/z* 224.0921.

Table 1

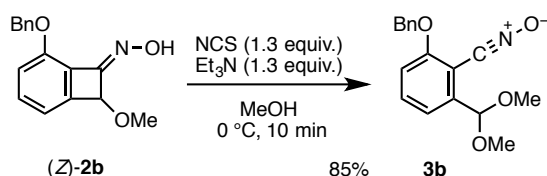


To a solution of benzocyclobutenone **S-1** (3.75 g, 14.7 mmol) in pyridine (49 mL) was added NH₂OH·HCl (1.22 g, 17.6 mmol). After stirring for 3 h at 50 °C, additional portions of NH₂OH·HCl (307 mg, 4.42 mmol; 309 mg 4.45 mmol; 1.01 g, 14.5 mmol; 303 mg 4.36 mmol) were added with 3 h intervals until the starting material was consumed. The reaction was stopped by adding 1 M aqueous HCl, and the mixture was extracted with EtOAc (x3). The combined extracts were washed with 1 M aqueous HCl (x2) and brine, dried (Na₂SO₄), and concentrated in vacuo. The residue was purified by trituration with hexane to give oxime (*Z*)-**2b** (2.56 g, 64%) as a white solid. The mother liquor was concentrated in vacuo, and the residue was purified by flash column chromatography (silica gel, hexane/EtOAc = 5/2) to give oxime (*Z*)-**2b** (278 mg, 7%; total yield = 71%) and oxime (*E*)-**2b** (46 mg, 1%) both as a white solid. The structures of (*Z*)-**2b** and (*E*)-**2b** were assigned by analogy to oximes (*Z*)-**2a** and (*E*)-**2a**.

Oxime (*Z*)-**2b**: *R_f* 0.45 (hexane/EtOAc = 2/1); *mp* 117–119 °C (hexane/EtOAc, colorless needle); ¹H NMR (600 MHz, acetone-*d*₆) δ 3.46 (s, 3H), 5.45 (d, 1H, *J* = 12.0 Hz), 5.50 (d, 1H, *J* = 12.0

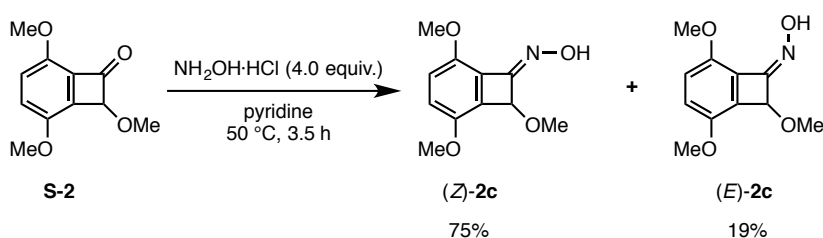
Hz), 5.60 (s, 1H), 6.99 (d, 1H, $J = 8.4$ Hz), 7.08 (d, 1H, $J = 7.2$ Hz), 7.32 (t, 1H, $J = 7$. Hz), 7.36–7.39 (m, 3H), 7.46 (d, 2H, $J = 7.2$ Hz), 10.15 (s, 1H, OH); ^{13}C NMR (150 MHz, acetone- d_6) δ 55.9, 72.6, 83.8, 116.7, 119.0, 127.0, 128.7, 128.8, 129.3, 134.0, 138.3, 149.8, 153.2 154.4; IR (ATR) 3266, 3036, 2924, 2839, 1939, 1688, 1600, 1576, 1484, 1452 cm^{-1} ; HRMS (ESI) calcd for $\text{C}_{16}\text{H}_{16}\text{NO}_3$ ($[\text{M}+\text{H}]^+$) m/z 270.1125, found m/z 270.1121.

Oxime (*E*)-**2b** [a mixture of (*E*)-**2b** and (*Z*)-**2b** (*E/Z* = 14/1)]; The major isomer (*E*)-**2b** had the following characteristics: R_f 0.13 (hexane/EtOAc = 2/1); ^1H NMR (600 MHz, acetone- d_6) δ 3.46 (s, 3H), 5.30 (s, 2H), 5.40 (s, 1H), 7.11 (d, 1H, $J = 7.2$ Hz), 7.11(d, 1H, $J = 7.2$ Hz), 7.30 (dd, 1H, $J = 7.2, 7.2$ Hz), 7.37 (dd, 1 H, $J = 7.8, 7.8$ Hz), 7.47 (t, 1H, $J = 7.8$ Hz), 7.57 (d, 2H, $J = 7.8$ Hz), 9.90 (s, 1H, OH); ^{13}C NMR (150 MHz, acetone- d_6) δ 56.5, 71.0, 84.0, 115.6, 116.6, 127.8, 128.4, 129.1, 129.2, 134.9, 138.2, 151.0, 151.3 152.3; IR (ATR) 3292, 3064, 2930, 2829, 1692, 1593, 1483, 1452, 1277, 1053, 938 cm^{-1} ; HRMS (ESI) calcd for $\text{C}_{16}\text{H}_{16}\text{NO}_3$ ($[\text{M}+\text{H}]^+$) m/z 270.1125, found m/z 270.1122.



To a solution of oxime (*Z*)-**2b** (80.1 mg, 0.297 mmol) and Et_3N (55.0 μL , 0.395 mmol) in MeOH (3.0 mL) was added NCS (52.1 mg, 0.390 mmol) at 0 °C. After stirring for 10 min at 0 °C, the reaction was stopped by adding water, and the mixture was extracted with EtOAc (x3). The combined organic extracts were washed with brine, dried (MgSO_4), and concentrated in vacuo. The residue was purified by PTLC (hexane/EtOAc/ $\text{CH}_2\text{Cl}_2 = 5/1/1$) to give nitrile oxide **3b** (75.7 mg, 85%) as colorless oil.

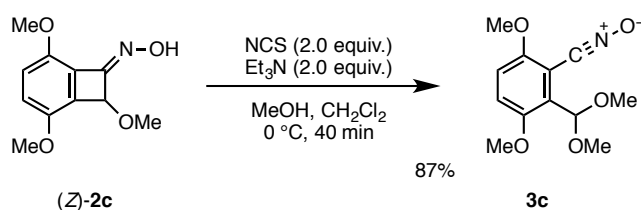
R_f 0.67 (hexane/EtOAc / $\text{CH}_2\text{Cl}_2 = 2/1/1$); ^1H NMR (600 MHz, CDCl_3) δ 3.39 (s, 6H), 5.19 (s, 2H), 5.47 (s, 1H), 6.95 (d, 1H, $J = 8.4$ Hz), 7.22 (d, 1H, $J = 8.4$ Hz), 7.33–7.44 (m, 6H); ^{13}C NMR (150 MHz, CDCl_3) δ 53.8, 70.7, 101.8, 102.3, 112.6, 119.5, 127.0, 128.2, 128.8, 131.4, 135.7, 142.9, 160.8 (A signal of the carbon in the nitrile oxide moiety is broadened.); IR (neat): 2934, 2829, 2310, 1275, 1061, 795 cm^{-1} ; HRMS (ESI) calcd for $\text{C}_{17}\text{H}_{18}\text{NO}_4$ ($[\text{M}+\text{H}]^+$) m/z 300.1230, found m/z 300.1239.



To a solution of benzocyclobutenone **S-2** (700 mg, 3.36 mmol) in pyridine (11 mL) was added $\text{NH}_2\text{OH}\cdot\text{HCl}$ (933 mg, 13.4 mmol). After stirring for 3.5 h, the reaction was quenched by adding 1 M aqueous HCl, and the mixture was extracted with EtOAc (x3). The combined organic extracts were washed with 1 M aqueous HCl (x3) and brine, dried (Na_2SO_4), and concentrated in vacuo. The residue was purified by trituration with hexane and EtOAc to give oxime (*Z*)-**2c** (417 mg, 56%) as a white solid. The mother liquor was concentrated in vacuo, and the residue was purified by flash column chromatography (silica gel, hexane/EtOAc = 2/1) to give oxime (*Z*)-**2c** (142 mg, 19%; total yield = 75%) and oxime (*E*)-**2c** (145 mg, 19%) both as a white solid. The structures of (*Z*)-**2c** and (*E*)-**2c** were assigned by analogy to oximes (*Z*)-**2a** and (*E*)-**2a**.

Oxime (*Z*)-**2c**: R_f 0.26 (hexane/EtOAc = 4/1); **mp** 164–166 °C (hexane/EtOAc, colorless needle); $^1\text{H NMR}$ (600 MHz, acetone- d_6) δ 3.45 (s, 3H), 3.91 (s, 3H), 3.96 (s, 3H), 5.77 (s, 1H), 6.82 (d, 1H, $J = 8.4$ Hz) 6.86 (d, 1H, $J = 8.4$ Hz), 10.11 (s, 1H, OH); $^{13}\text{C NMR}$ (150 MHz, acetone- d_6) δ 55.3, 57.3, 58.3, 83.4, 119.8, 120.5, 127.5, 131.2, 147.9, 150.7, 153.9; **IR** (ATR) 3196, 2954, 2887, 2837, 1497, 1260. 1019, 808 cm^{-1} ; **Anal.** calcd for $\text{C}_{12}\text{H}_{14}\text{O}_5$: C, 59.19; H, 5.87; N, 6.27. found: C, 59.06; H, 5.78; N, 6.17.

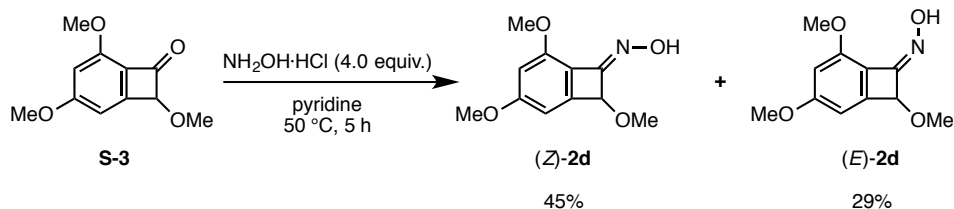
Oxime (*E*)-**2c**: R_f 0.10 (hexane/EtOAc = 4/1); **mp** 112–113 °C (hexane/EtOAc, colorless needle); $^1\text{H NMR}$ (600 MHz, acetone- d_6) δ 3.45 (s, 3H), 3.84 (s, 3H), 3.91 (s, 3H), 5.54 (s, 1H), 6.93 (d, 1H, $J = 9.0$ Hz) 6.98 (d, 1H, $J = 9.0$ Hz), 9.80 (s, 1H, OH); $^{13}\text{C NMR}$ (150 MHz, acetone- d_6) δ 55.3, 57.3, 58.3, 83.4, 119.8, 120.5, 127.5, 131.2, 147.9, 150.7, 153.9; **IR** (ATR) 3112, 2951, 2843, 1695, 1499, 1433, 1275, 1258, 960 cm^{-1} ; **HRMS** (ESI) calcd for $\text{C}_{11}\text{H}_{14}\text{NO}_4$ ($[\text{M}+\text{H}]^+$) m/z 224.0917, found m/z 224.0913.



To a solution of oxime (*Z*)-**2c** (66.9 mg, 0.300 mmol) and Et_3N (55.0 μL , 0.395 mmol) in MeOH (2.0 mL) and CH_2Cl_2 (1.0 mL) was added NCS (54.0 mg, 0.387 mmol) at 0 °C. After stirring for 30 min at 0 °C, additional portion of Et_3N (29.0 μL , 0.208 mmol) and NCS (28.2 mg, 0.211 mmol) were added. After stirring for 10 min at 0 °C, the reaction was stopped by adding water, and the mixture was extracted with EtOAc (x3). The combined organic extracts were washed with brine, dried (Na_2SO_4), and concentrated in vacuo. The residue was purified by flash column chromatography (silica gel, hexane/EtOAc = 2/1) to give nitrile oxide **3c** (79.4 mg, 94%) as colorless oil.

R_f 0.37 (hexane/EtOAc = 2/1); $^1\text{H NMR}$ (600 MHz, CDCl_3) δ 3.43 (s, 3H), 3.43 (s, 3H), 3.82 (s, 3H), 3.85 (s, 3H), 5.68 (s, 1H), 6.85 (d, 1H, $J = 9.0$ Hz), 6.96 (d, 1H, $J = 9.0$ Hz); $^{13}\text{C NMR}$ (150

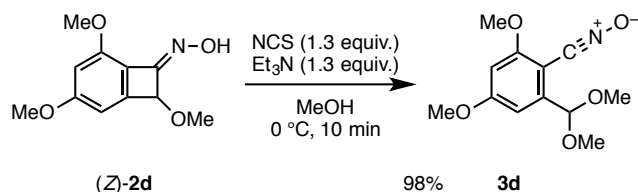
MHz, CDCl₃) δ 54.0, 55.3, 55.6, 98.2, 101.2, 110.5, 113.4, 129.6, 150.2, 155.4 (A signal of the carbon in the nitrile oxide moiety is broadened.); **IR** (neat) 2940, 2839, 2358, 2308, 1738, 1588, 1485, 1452, 1343, 1258, 1075 1052, cm⁻¹; **HRMS** (ESI) calcd for C₁₂H₁₅NNaO₅ ([M+Na]⁺) m/z 276.0842, found m/z 276.0852.



To a solution of benzocyclobutenone **S-3** (1.08 g, 5.19 mmol) in pyridine (17 mL) was added NH₂OH·HCl (1.44 g, 20.7 mmol). After stirring for 5 h at 50 °C, the reaction was quenched by adding 1 M aqueous HCl, and the mixture was extracted with EtOAc (x2). The combined extracts were washed with 1 M aqueous HCl (x3) and brine, dried (Na₂SO₄), and concentrated in vacuo. The residue was purified by flash column chromatography (silica gel, hexane/EtOAc = 1/1) to give oxime **(Z)-2d** (524 mg, 45%) and oxime **(E)-2d** (338 mg, 29%) both as a white solid. The structures of **(Z)-2d** and **(E)-2d** were assigned by analogy to oximes **(Z)-2a** and **(E)-2a**.

Oxime **(Z)-2d**: *R_f* 0.57 (hexane/EtOAc = 1/1); **mp** 109–110 °C (hexane/EtOAc, colorless needle); ¹H NMR (600 MHz, acetone-*d*₆) δ 3.43 (s, 3H), 3.84 (s, 3H), 4.01 (s, 3H), 5.52 (s, 1H), 6.44 (d, 1H, *J* = 1.8 Hz), 6.68 (d, 1H, *J* = 1.8 Hz), 9.73 (s, 1H, OH); ¹³C NMR (150 MHz, acetone-*d*₆) δ 55.7, 56.2, 58.5, 83.3, 102.1, 104.5, 119.4, 150.8, 153.4, 155.6, 165.3; **IR** (ATR) 3252, 3079, 2935, 2835, 1694, 1603, 1575, 1447, 1363, 1132 cm⁻¹; **Anal.** calcd for C₁₁H₁₃NO₄: C, 59.19; H, 5.87; N, 6.27, found: C, 59.21; H, 5.79; N, 6.25.

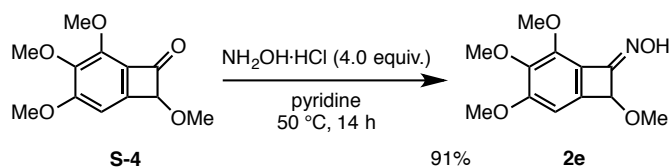
Oxime **(E)-2d**: *R_f* 0.17 (hexane/EtOAc = 1/1); **mp** 144–145 °C (hexane/EtOAc, colorless needle); ¹H NMR (400 MHz, acetone-*d*₆) δ 3.42 (s, 3H), 3.88 (s, 3H), 3.88 (s, 3H), 5.29 (s, 1H) 6.54 (d, 1H, *J* = 1.2 Hz), 6.70 (d, 1H, *J* = 1.2 Hz), 9.44 (s, 1H, OH); ¹³C NMR (150 MHz, acetone-*d*₆) δ 56.2, 56.3, 83.3, 100.4, 102.0, 121.9, 150.1, 152.4, 154.8, 166.3 (several signals overlapped); **IR** (ATR) 3220, 3132, 2898, 1702, 1584, 1494, 1336, 1313, 1207, 1148, 952, 826 cm⁻¹; **HRMS** (ESI) calcd for C₁₁H₁₄NO₄ ([M+H]⁺) m/z 224.0917, found m/z 224.0914.



To a solution of oxime **(Z)-2d** (67.0 mg, 0.300 mmol) and Et₃N (52.2 μL, 0.391 mmol) in MeOH (3.0 mL) was added NCS (55.0 mg, 0.395 mmol) at 0 °C. After stirring for 10 min at 0 °C, the reaction was stopped by adding water and brine, and the mixture was extracted with EtOAc (x3).

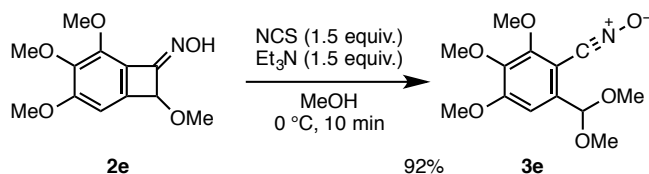
The combined organic extracts were washed with brine, dried (Na₂SO₄), and concentrated in vacuo. The residue was purified by flash column chromatography (silica gel, hexane/EtOAc = 3/1) to give nitrile oxide **3d** (74.2 mg, 98%) as a colorless oil.

R_f 0.56 (hexane/EtOAc/CH₂Cl₂ = 2/1/1); **¹H NMR** (600 MHz, CDCl₃) δ 3.39 (s, 6H), 3.86 (s, 3H), 3.87 (s, 3H), 5.41 (s, 1H), 6.43 (d, 1H, *J* = 2.4 Hz), 6.77 (d, 1H, *J* = 2.4 Hz); **¹³C NMR** (150 MHz, CDCl₃) δ 33.8, 53.9, 55.8, 56.1, 93.8, 98.7, 101.7, 103.9, 144.2, 162.6, 163.1; **IR** (neat) 3102, 2953, 2892, 2835, 2305, 1601, 1465, 1342, 861 cm⁻¹; **HRMS** (ESI) calcd for C₁₂H₁₆NO₅ ([M+H]⁺) *m/z* 254.1023, found *m/z* 254.1028.



To a solution of benzocyclobutenone **S-4** (480 mg, 2.02 mmol) in pyridine (6.7 mL) was added NH₂OH·HCl (280 mg, 4.03 mmol). After stirring for 1 h at 50 °C, an additional portion of NH₂OH·HCl (281 mg, 4.04 mmol) was added, and the mixture was stirred for 13 h at 50 °C. The reaction was stopped by adding 1 M aqueous HCl, and the mixture was extracted with EtOAc (x3). The combined organic extracts were washed with 1 M aqueous HCl (x3) and brine, dried (Na₂SO₄), and concentrated in vacuo. The residue was purified by trituration with hexane and EtOAc to give oxime **2e** (425 mg, 83%) as a white solid. The mother liquor was concentrated in vacuo, and the residue was purified by flash column chromatography (silica gel, hexane/EtOAc = 2/1) to give oxime **2e** (41.7 mg, 8%; total yield = 91%).

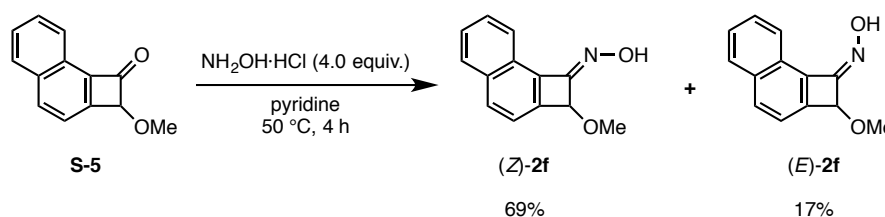
Oxime **2e**: **R_f** 0.59 (hexane/EtOAc = 1/1); **mp** 124–125 °C (hexane/EtOAc, colorless needle); **¹H NMR** (600 MHz, acetone-*d*₆) δ 3.43 (s, 3H), 3.70 (s, 3H), 3.89 (s, 3H), 4.10 (s, 3H), 5.50 (s, 1H), 6.82 (s, 1H), 9.80 (s, 1H, OH); **¹³C NMR** (150 MHz, acetone-*d*₆) δ 55.6, 56.8, 59.8, 60.7, 83.3, 101.6, 119.8, 141.1, 143.7, 149.1, 153.7, 158.7; **IR** (ATR) 3327, 2940, 2830, 1675, 1591, 1479, 1415, 1330, 1243, 1199, 1132 cm⁻¹; **HRMS** (ESI) calcd for C₁₂H₁₆NO₅ ([M+H]⁺) *m/z* 254.1023, found *m/z* 254.1023.



To a solution of oxime **2e** (76.0 mg, 0.300 mmol) and Et₃N (63.0 μL, 0.452 mmol) in MeOH (3.0 mL) was added NCS (60.4 mg, 0.452 mmol) at 0 °C. After stirring for 10 min at 0 °C, the reaction was stopped by adding water and brine, and the mixture was extracted with EtOAc (x3).

The combined organic extracts were washed with brine, dried (Na₂SO₄), and concentrated in vacuo. The residue was purified by flash column chromatography (silica gel, hexane/EtOAc/CH₂Cl₂ = 4/1/1) to give nitrile oxide **3e** (78.6 mg, 92%) as colorless oil.

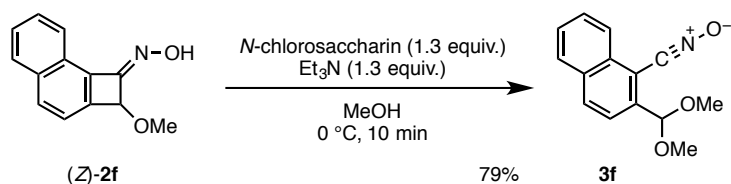
R_f 0.54 (hexane/EtOAc/CH₂Cl₂ = 2/1/1); **¹H NMR** (600 MHz, CDCl₃) δ 3.40 (s, 6H), 3.86 (s, 3H), 3.92 (s, 3H), 4.00 (s, 3H), 5.38 (s, 1H), 6.93 (s, 1H); **¹³C NMR** (150 MHz, CDCl₃) δ 54.1, 56.3, 61.1, 61.8, 99.3, 101.8, 105.9, 137.9, 141.9, 155.86 (A signal of the carbon in the nitrile oxide moiety is broadened.); **IR** (neat) 3019, 2360, 2342, 1348, 1215, 756 cm⁻¹; **HRMS** (ESI) calcd for C₁₃H₁₇NO₆ ([M+H]⁺) 284.1129, found m/z 284.1134.



To a solution of naphthocyclobutenone **S-5** (1.30 g, 6.56 mmol) in pyridine (22 mL) was added NH₂OH·HCl (1.82 g, 26.2 mmol). After stirring for 4 h at 50 °C, the reaction was quenched by adding 1 M aqueous HCl, and the mixture was extracted with EtOAc (x2). The combined extracts were washed with 1 M aqueous HCl (x3) and brine, dried (Na₂SO₄), and concentrated in vacuo. The residue was purified by flash column chromatography (silica gel, hexane/EtOAc = 4/1) to give oxime (*Z*)-**2f** (966 mg, 69%) and oxime (*E*)-**2f** (236 mg, 17%) both as a white solid. The structures of (*Z*)-**2f** and (*E*)-**2f** were assigned by analogy to oximes (*Z*)-**2a** and (*E*)-**2a**.

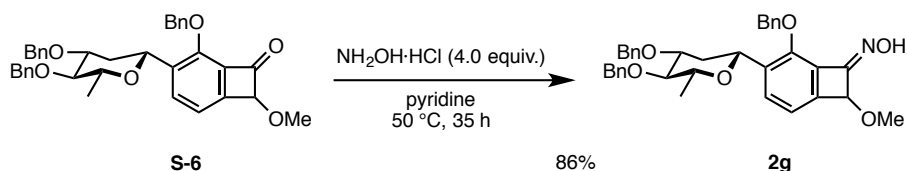
Oxime (*Z*)-**2f**: **R_f** 0.39 (hexane/EtOAc = 2/1); **mp** 149–150 °C (hexane/EtOAc, colorless needle); **¹H NMR** (600 MHz, acetone-*d*₆) δ 3.52 (s, 3H), 5.57 (s, 1H), 7.62 (dd, 1H, *J* = 7.8 Hz, 8.4 Hz), 7.65 (d, *J* = 8.4 Hz), 7.67 (dd, 1H, *J* = 7.8 Hz, 7.8 Hz), 8.02 (d, 1H, *J* = 8.4 Hz), 8.09 (d, 1H, *J* = 8.4 Hz), 8.74 (d, 1H, *J* = 7.8 Hz) 10.42 (s, 1H, OH); **¹³C NMR** (150 MHz, acetone-*d*₆) δ 56.6, 83.8, 121.4, 127.0, 127.6, 128.0, 128.6, 130.2, 134.4, 135.8, 139.2, 149.8, 153.2; **IR** (ATR) 3247, 3135, 2995, 2929, 2894, 2819, 1705, 1437, 1334, 1083, 923 cm⁻¹; **Anal.** calcd for C₁₃H₁₁NO₂: C, 73.23; H, 5.26; N 6.57, found: C, 73.16; H, 5.26; N 6.61.

Oxime (*E*)-**2f**: **R_f** 0.30 (hexane/EtOAc = 2/1); **mp** 111–112 °C (hexane/EtOAc, white solid); **¹H NMR** (600 MHz, acetone-*d*₆) δ 3.51 (s, 3H), 5.80 (s, 1H), 7.62 (dd, 1H, *J* = 7.2, 8.4 Hz), 7.63 (d, 1H, *J* = 7.8 Hz), 7.67 (dd, 1H, *J* = 7.2, 8.4 Hz), 7.99 (d, 1H, *J* = 8.4 Hz), 8.01 (d, 1H, *J* = 7.8 Hz), 8.04 (d, 1H, *J* = 8.4 Hz), 10.14 (s, 1H, OH); **¹³C NMR** (150 MHz, acetone-*d*₆) δ 55.9, 84.9, 21.8, 125.5, 126.7, 128.0, 128.9, 130.4, 132.9, 135.8, 139.0, 147.9, 155.4; **IR** (ATR) 3176, 3105, 2995, 2955, 2878, 2826, 1681, 1457, 1343, 1153, 1086 cm⁻¹; **HRMS** (ESI) calcd for C₁₃H₁₂NO₂ ([M+H]⁺) m/z 214.0863, found m/z 214.0857.



To a solution of oxime (**Z**)-**2f** (64.0 mg, 0.300 mmol) and Et₃N (54.5. μL, 0.391 mmol) in MeOH (3.0 mL) was added *N*-chlorosaccharin (55.0 mg, 0.395 mmol) at 0 °C. After stirring for 10 min at 0 °C, the reaction was stopped by adding water, and the mixture was extracted with EtOAc (x3). The combined organic extracts were washed with brine, dried (Na₂SO₄), and concentrated in vacuo. The residue was purified by flash column chromatography (silica gel, hexane/EtOAc = 3/1) to give nitrile oxide **3f** (59.4 mg, 79%) as colorless oil.

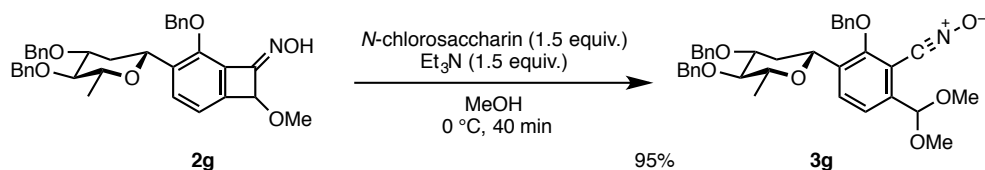
R_f 0.79 (hexane/EtOAc/CH₂Cl₂ = 2/1/1); **¹H NMR** (600 MHz, CDCl₃) δ 3.44 (s, 6H), 5.70 (s, 1H), 7.60 (dd, 1H, *J* = 8.4, 8.4 Hz), 7.66 (dd, 1H, *J* = 8.4, 7.8 Hz), 7.76 (d, 1H, *J* = 8.4 Hz), 7.91 (d, 1H, *J* = 7.8 Hz), 7.98 (d, 1H, *J* = 8.4 Hz), 8.15 (d, 1H, *J* = 8.4 Hz); **¹³C NMR** (150 MHz, CDCl₃) δ 54.0, 102.4, 110.0, 124.0, 125.2, 127.6, 128.4, 128.6, 130.9, 133.2, 133.4, 141.6 (A signal of the carbon in the nitrile oxide moiety is broadened.); **IR** (neat) 3060, 2935, 2828, 2292, 1308, 1113, 1048 cm⁻¹; **HRMS** (ESI) calcd for C₁₄H₁₃NNaO₃ ([M+Na]⁺) *m/z* 266.0788, found *m/z* 226.0789.



To a solution of benzocyclobutenone **S-6** (2.21 g, 3.91 mmol) in pyridine (13 mL) was added NH₂OH·HCl (545 mg, 7.84 mmol). After stirring for 20 h at 50 °C, additional portions of NH₂OH·HCl (271 mg, 3.90 mmol; 277 mg, 3.99 mmol) was added with 6 h intervals. After the stirring for 8.5 h at 50 °C, the reaction was stopped by adding 1 M aqueous HCl. The mixture was extracted with EtOAc (x3), and the combined extracts were washed with 1 M aqueous HCl (x2), and brine, dried (Na₂SO₄), and concentrated in vacuo. The residue was purified by trituration with hexane and EtOAc to give oxime **2g** (542 mg, 24%) as a white solid. The mother liquor was concentrated in vacuo, and the residue was purified by flash column chromatography (silica gel, hexane/EtOAc = 5/2) to give oxime **2g** (1.41 g, 62%; total yield = 86%, a mixture of two stereoisomers, ratio = 1:1).

Oxime **2g**: **R_f** 0.48 (hexane/EtOAc = 2/1); **mp** 91–93 °C (hexane/EtOAc, colorless needle); **¹H NMR** (600 MHz, acetone-*d*₆, ratio of diastereomers = 1/1, signals for the isomer are marked with an asterisk) δ 1.320 (d, 3H, *J* = 6.0 Hz), 1.322* (d, 3H, *J* = 6.0 Hz), 1.34–1.40 (m, 1H), 1.34–1.40* (m, 1H), 2.50–2.55 (m, 1H), 2.50–2.55* (m, 1H), 3.16 (dd, 1H, *J* = 9.0, 9.0 Hz), 3.16* (dd,

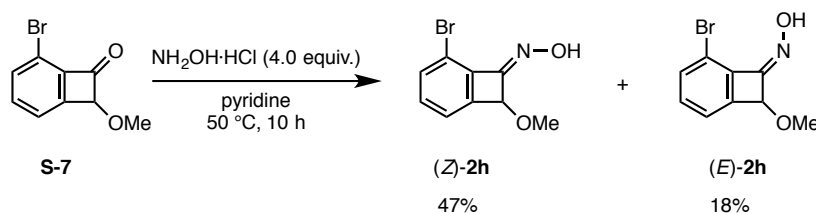
1H, $J = 9.0, 9.0$ Hz), 3.44* (s, 3H), 3.46–3.50 (m, 1H), 3.46–3.50* (m, 1H), 3.48 (s, 3H), 3.72–3.76 (m, 1H), 3.72–3.76* (m, 1H), 4.54 (d, 1H, $J = 11.4$ Hz), 4.54* (d, 1H, $J = 11.4$ Hz), 4.65 (d, 1H, $J = 11.4$ Hz), 4.67* (d, 1H, $J = 11.4$ Hz), 4.71 (d, 1H, $J = 10.8$ Hz), 4.71* (d, 1H, $J = 10.8$ Hz), 4.80 (dd, 1H, $J = 10.2, 7.2$ Hz), 4.80* (dd, 1H, $J = 10.2, 7.2$ Hz), 4.99 (d, 1H, $J = 10.8$ Hz), 4.99* (d, 1H, $J = 10.8$ Hz), 5.52 (d, 1H, $J = 12.6$ Hz), 5.59 (s, 1H), 5.59* (s, 1H), 5.61 (d, 1H, 12.6 Hz), 5.61* (d, 1H, 12.6 Hz), 5.67* (d, 1H, $J = 12.6$ Hz), 7.107* (d, 1H, $J = 7.2$ Hz), 7.111 (d, 1H, $J = 7.2$ Hz), 7.24–7.41 (m, 13H), 7.24–7.41* (m, 13H), 7.50 (d, 2H, $J = 7.8$ Hz), 7.50* (d, 2H, $J = 7.8$ Hz), 7.58 (d, 1H, $J = 7.2$ Hz), 7.58* (d, 1H, $J = 7.2$ Hz), 10.23 (s, 1H, OH), 10.23* (s, 1H, OH); ^{13}C NMR (150 MHz, acetone- d_6) δ 19.0, 38.6, 38.7, 55.7, 56.0, 71.29, 71.32, 72.6, 72.7, 73.4, 75.5, 76.4, 81.66, 81.69, 83.2, 83.4, 84.8, 116.9, 126.5, 128.2, 128.50, 128.53, 128.6, 128.7, 128.92, 128.93, 129.0, 129.1, 129.4, 131.1, 133.9, 134.0, 138.39, 138.41, 134.0, 140.2, 148.35, 148.44, 149.1, 154.3 (several signals overlapped); IR (ATR) 3318, 3031, 2925, 2850, 1591, 1086, 730, 694 cm^{-1} ; Anal. calcd for $\text{C}_{36}\text{H}_{37}\text{NO}_6$: C, 74.59; H, 6.43; N 2.42, found: C, 74.69; H, 6.35; N 2.42.



To a solution of oxime **2g** (87.1 mg, 0.150 mmol) and Et_3N (27.2 μL , 0.195 mmol) in MeOH (1.5 mL) was added *N*-chlorosaccharin (42.2 mg, 0.195 mmol) at 0 °C. After stirring for 35 min at 0 °C, an additional portion of Et_3N (4.2 μL , 0.030 mmol) and *N*-chlorosaccharin (6.6 mg, 0.031 mmol) was added. After stirring for 5 min at 0 °C, the reaction was stopped by adding water, and the mixture was extracted with EtOAc (x3). The combined organic extracts were washed with brine, dried (Na_2SO_4), and concentrated in vacuo. The residue was purified by flash column chromatography (silica gel, hexane/EtOAc = 3/1) to give nitrile oxide **3g** (86.6 mg, 95%) as colorless oil. At 5 °C, nitrile oxide **3g** gradually underwent decomposition.

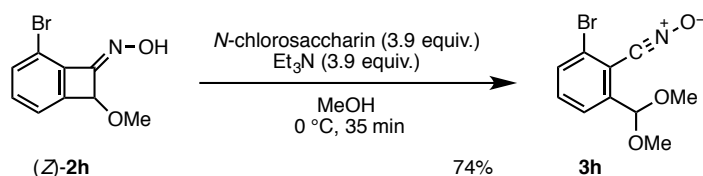
R_f 0.21 (hexane/EtOAc/ $\text{CH}_2\text{Cl}_2 = 16/1/2$); ^1H NMR (600 MHz, CDCl_3) δ 1.33 (d, 3H, $J = 6.0$ Hz), 1.57–1.62 (m, 1H), 2.23 (ddd, 1H, 12.6, 4.8, 1.2 Hz), 3.16 (dd, 1H, $J = 9.0, 9.0$ Hz), 3.36 (s, 3H), 3.38–3.41 (m, 1H), 3.41 (s, 3H), 3.63 (ddd, 1H, $J = 12.6, 9.0, 4.8$ Hz), 4.54 (d, 1H, $J = 12.0$ Hz), 4.57 (dd, 1H, $J = 12.6, 1.2$ Hz), 4.60 (d, 1H, $J = 12.0$ Hz), 4.68 (d, 1H, $J = 10.8$ Hz), 4.97 (d, 1H, $J = 10.8$ Hz), 5.03 (d, 1H, 10.8 Hz), 5.11 (d, 1H, 10.8 Hz), 5.46 (s, 1H), 7.28–7.43 (m, 16H), 7.60 (d, 1H, $J = 7.8$ Hz); ^{13}C NMR (150 MHz, CDCl_3) δ 18.6, 37.6, 53.4, 53.9, 71.3, 71.4, 75.4, 75.9, 77.8, 80.7, 83.8, 101.5, 107.1, 123.4, 127.65, 127.68, 128.1, 128.4, 128.5, 128.8, 129.3, 135.8, 136.1, 138.3, 138.4, 142.1, 158.4 (A signal of the carbon in the nitrile oxide moiety is broadened, and several signals overlapped.); IR (neat) 3629, 3088, 3063, 3030, 2933, 2880, 2298,

1952, 1870, 1810, 1736, 1604, 1497, 1454, 1335, 1251, 1113, 737, 698 cm^{-1} ; **HRMS** (ESI) calcd for $\text{C}_{37}\text{H}_{40}\text{NO}_7$ ($[\text{M}+\text{H}]^+$) m/z 610.2799, found m/z 610.2805.



To a solution of benzocyclobutenone **S-7** (250 mg, 1.10 mmol) in pyridine (3.7 mL) was added $\text{NH}_2\text{OH}\cdot\text{HCl}$ (306 mg, 4.40 mmol). After stirring for 10 h at 50 °C, the reaction was quenched by adding 1 M aqueous HCl, and the mixture was extracted with EtOAc (x3). The combined extracts were washed with 1 M aqueous HCl (x2) and brine, dried (Na_2SO_4), and concentrated in vacuo. The residue was purified by flash column chromatography (silica gel, hexane/EtOAc/ CHCl_3 = 3/1/2) to give oxime **(Z)-2h** (126.2 mg, 47%) and oxime **(E)-2h** (48.7 mg 18%) both as a white solid. The structures of **(Z)-2h** and **(E)-2h** were assigned by analogy to oximes **(Z)-2a** and **(E)-2a**. Oxime **(Z)-2h**: R_f 0.34 (hexane/EtOAc = 3/1); **mp** 119–120 °C (hexane/EtOAc, colorless needle); $^1\text{H NMR}$ (600 MHz, acetone- d_6) δ 3.47 (s, 3H), 5.67 (s, 1H), 7.37 (dd, 1H, J = 8.4, 7.8 Hz), 7.52 (d, 1H, J = 7.8 Hz), 7.62 (d, 1H, J = 8.4 Hz), 10.4 (s, 1H, OH); $^{13}\text{C NMR}$ (150 MHz, acetone- d_6) δ 56.2, 84.6, 113.0, 123.5, 133.4, 134.7, 142.1, 150.8, 154.1; **IR** (ATR) 3303, 3072, 2938, 2822, 1440, 1341, 1327, 1201, 1137, 1097, 1017, 933, 789 cm^{-1} ; **HRMS** (ESI) calcd for $\text{C}_9\text{H}_9\text{BrNO}_2$ ($[\text{M}+\text{H}]^+$) m/z 241.9811, found m/z 241.9802.

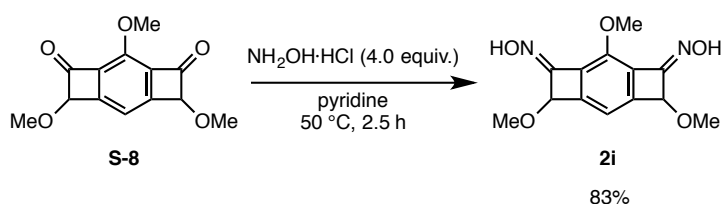
Oxime **(E)-2h**: R_f 0.24 (hexane/EtOAc = 3/1); **mp** 139–140 °C (hexane/EtOAc, colorless needle); $^1\text{H NMR}$ (600 MHz, acetone- d_6) δ 3.47 (s, 3H), 5.43 (s, 1H), 7.41 (dd, 1H, J = 7.8, 7.2 Hz), 7.55 (d, 1H, J = 7.2 Hz), 7.68 (d, 1H, J = 7.8 Hz), 10.50 (s, 1H, OH); $^{13}\text{C NMR}$ (150 MHz, CDCl_3) δ 57.5, 84.1, 115.8, 124.0, 135.0, 136.1, 142.6, 150.6, 153.0; **IR** (ATR) 3188, 3069, 2998, 2830, 1687, 1565, 1450, 1320, 1199, 1138 cm^{-1} ; **HRMS** (ESI) calcd for $\text{C}_9\text{H}_9\text{BrNO}_2$ ($[\text{M}+\text{H}]^+$) m/z 241.9811, found m/z 241.9803.



To a solution of oxime **(Z)-2h** (72.6 mg, 0.300 mmol) and Et_3N (55.0 μL , 0.395 mmol) in MeOH (3.0 mL) was added *N*-chlorosaccharin (42.2 mg, 0.195 mmol) at 0 °C. The reaction mixture was stirred for 15 min at 0 °C, and additional portions of Et_3N (55.0 μL , 0.395 mmol) and *N*-chlorosaccharin (84.6 mg, 0.390 mmol; 84.7 mg, 0.390 mmol) were added with 15 min intervals. After stirring for 5 min at 0 °C, the reaction was stopped by adding water, and the

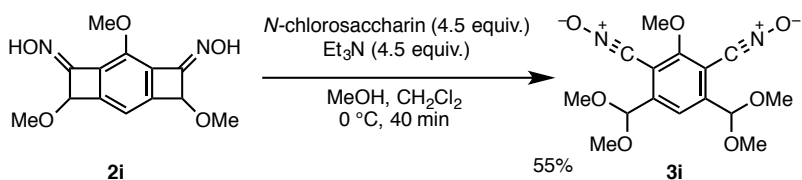
mixture was extracted with EtOAc (x3). The combined organic extracts were washed with brine, dried (Na₂SO₄), and concentrated in vacuo. The residue was purified by flash column chromatography (silica gel, hexane/EtOAc = 8/1) to give nitrile oxide **3h** (60.4 mg, 74%) as colorless oil.

R_f 0.63 (hexane/EtOAc = 3/1); **¹H NMR** (600 MHz, CDCl₃) δ 3.39 (s, 6H), 5.47 (s, 1H), 7.34 (dd, 1H, *J* = 9.0, 9.0 Hz), 7.60 (d, 1H, *J* = 9.0 Hz), 7.62 (d, 1H, *J* = 9.0 Hz); **¹³C NMR** (150 MHz, CDCl₃) δ 53.8, 101.6, 115.9, 126.1, 127.0, 131.1, 132.9, 143.9 (A signal of the carbon in the nitrile oxide moiety is broadened.); **IR** (neat) 2995, 2935, 2832, 2298, 1448, 1370, 1341, 1145, 1116, 1053, cm⁻¹; **HRMS** (ESI) calcd for C₁₀H₁₁BrNO₃ ([M+H]⁺) *m/z* 271.9917, found *m/z* 271.9915.



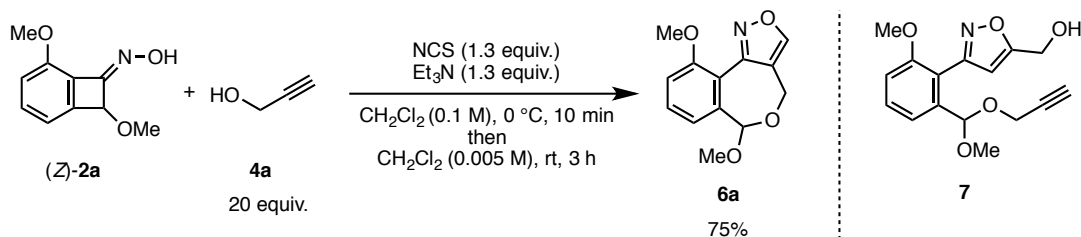
To a solution of diketone **S-8** (449 mg, 1.81 mmol) in pyridine (6.0 mL) was added NH₂OH·HCl (503 mg, 7.24 mmol). After stirring for 2.5 h at 50 °C, the reaction was quenched by adding 1 M aqueous HCl, and the mixture was extracted with EtOAc (x3). The combined organic extracts were washed with 1 M aqueous HCl (x3) and brine, dried (Na₂SO₄), and concentrated in vacuo. The residue was purified by trituration with hexane and EtOAc to give oxime **2i** (354 mg, 70%) as a white solid. The mother liquor was concentrated in vacuo, and the residue was purified by flash column chromatography (silica gel, hexane/EtOAc = 1/1) to give oxime **2i** (67.1 mg, 13%; total yield = 83%, a mixture of four stereoisomers, ratio = 1:1:1:1) as a white solid.

R_f 0.32 (hexane/EtOAc = 1/1); **mp** 168–173 °C (decomp.) (hexane/EtOAc, white solid); **¹H NMR** (600 MHz, acetone-*d*₆, without distinction of the four stereoisomers) δ 3.45–3.49 (m, 6H), 4.19 (s, 0.75H), 4.19 (s, 0.75H), 4.21 (s, 0.75H), 4.21 (s, 0.75H), 5.32 (s, 0.25H), 5.33 (s, 0.25H), 5.55 (s, 0.25H), 5.556 (s, 0.25H), 5.560 (s, 0.25H), 5.57 (s, 0.25H), 7.20 (s, 0.25H), 7.21 (s, 0.25H), 7.22 (s, 0.25H), 7.23 (s, 0.25H), 9.95 (s, 0.5H, OH), 10.17 (s, 1H, OH), 10.17 (s, 0.5H, OH), 10.26 (s, 0.5H, OH); **¹³C NMR** (150 MHz, acetone-*d*₆) δ 56.07, 56.11, 56.2, 56.61, 56.64, 60.2, 60.3, 82.5, 82.7, 82.8, 83.0, 33.0, 83.2, 112.2, 112.3, 112.6, 112.7, 127.5, 127.6, 128.9, 129.0, 129.8, 130.0, 147.3, 147.4, 147.46, 147.50, 149.9, 150.1, 153.4, 153.48, 153.54, 153.6, 153.9, 153.9, 154.0, 154.6, 154.7, 155.06, 155.14; **IR** (ATR) 3297, 2934, 2895, 2832, 2360, 2342, 1674, 1583, 1294, 1062 cm⁻¹; **HRMS** (ESI) calcd for C₁₃H₁₅N₂O₅ ([M+H]⁺) *m/z* 279.0976, found *m/z* 279.0973.



To a solution of oxime **2i** (41.7 mg, 0.150 mmol) and Et₃N (73.0 μL, 0.524 mmol) in MeOH (0.75 mL) and CH₂Cl₂ (0.75 mL) was added *N*-chlorosaccharin (42.2 mg, 0.524 mmol) at 0 °C. After stirring for 30 min at 0 °C, an additional portion of Et₃N (20.9 μL, 0.150 mmol) and *N*-chlorosaccharin (32.5 mg, 0.150 mmol) was added. After stirring for 10 min at 0 °C, the reaction was stopped by adding water, and the mixture was extracted with EtOAc (x3). The combined organic extracts were washed with brine, dried (Na₂SO₄), and concentrated in vacuo. The residue was purified by flash column chromatography (silica gel, hexane/EtOAc = 3/1) to give nitrile oxide **3i** (27.8 mg, 55%) as colorless oil.

R_f 0.62 (hexane/EtOAc = 1/1); **¹H NMR** (600 MHz, CDCl₃) δ 3.39 (s, 12H), 4.13 (s, 3H), 5.44 (s, 2H), 7.64 (s, 1H); **¹³C NMR** (150 MHz, CDCl₃) δ 30.5, 53.9, 62.9, 101.1, 107.5, 121.5, 144.6, 164.8; **IR** (neat) 2939, 2833, 2360, 2341, 1507, 1373, 1345, 1115, 1056, 668 cm⁻¹; **HRMS** (ESI) calcd for C₁₅H₁₉N₂O₇ ([M+H]⁺) m/z 339.1187, m/z found 339.1191.

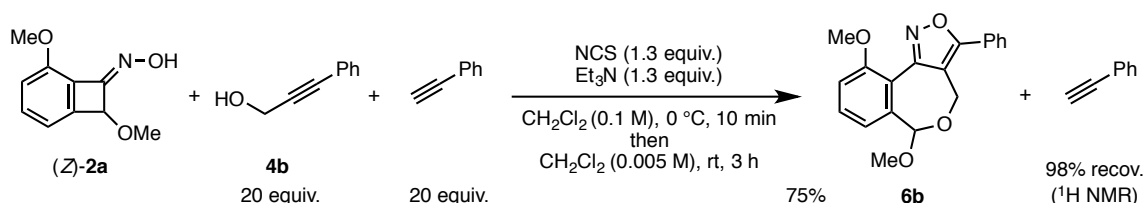


To a solution of oxime (*Z*)-**2a** (29.0 mg, 0.150 mmol), Et₃N (27.2 μL, 0.195 mmol), and propargylalcohol (**4a**) (177 μL, 3.02 mmol) in CH₂Cl₂ (1.5 mL) was added NCS (26.1 mg, 0.195 mmol) at 0 °C. After stirring for 10 min at 0 °C, the solution was diluted with CH₂Cl₂ (30 mL). After kept standing for 3 h at room temperature, the reaction was stopped by adding water, and the mixture was extracted with EtOAc (x3). The combined organic extracts were washed with brine, dried (Na₂SO₄), and concentrated in vacuo. The residue was purified by PTLC (hexane/EtOAc/CHCl₃ = 1/1/1) to give isoxazole **6a** (27.8 mg, 75%) as colorless oil.

Isoxazole **6a**: **R_f** 0.56 (hexane/EtOAc/CHCl₃ = 1/1/1); **¹H NMR** (600 MHz, CDCl₃) δ 3.32 (s, 3H), 3.95 (s, 3H), 4.49 (d, 1H, *J* = 12.6 Hz), 4.67 (d, 1H, *J* = 12.6 Hz), 5.47 (s, 1H), 7.10 (d, 1H, *J* = 8.4 Hz), 7.14 (d, 1H, *J* = 7.2 Hz), 7.46 (dd, 1H, *J* = 8.4, 7.2 Hz), 8.37 (s, 1H); **¹³C NMR** (150 MHz, CDCl₃) δ 55.4, 56.3, 56.4, 103.5, 112.7, 115.4, 117.4, 120.8, 131.1, 139.2, 152.9, 157.2, 159.1; **IR** (neat) 3106, 2937, 2897, 2838, 2236, 1620, 1600, 1579, 1476, 1272, 1073, 1044 cm⁻¹; **HRMS** (ESI) calcd for C₁₃H₁₄NO₄ ([M+H]⁺) m/z 248.0917, found m/z 248.0914.

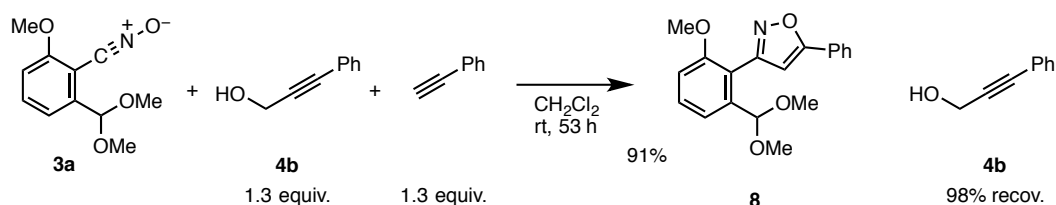
Isoxazole **7**: **R_f** 0.38 (hexane/EtOAc/CHCl₃ = 1/1/1); **¹H NMR** (600 MHz, CDCl₃) δ 2.29 (br,

1H), 2.36 (dd, 1H, $J = 2.4, 2.4$ Hz), 3.30 (s, 3H), 3.78 (s, 3H), 4.10 (dd, 1H, $J = 15.6, 2.4$ Hz), 4.19 (dd, 1H, $J = 15.6, 2.4$ Hz), 4.83 (, 1H), 5.65 (s, 1H), 7.98 (d, 1H, $J = 7.8$ Hz), 7.35 (d, 1H, $J = 7.8$ Hz), 7.45 (dd, 1H, $J = 7.8, 7.8$ Hz); $^{13}\text{C NMR}$ (150 MHz, CDCl_3) δ 53.6, 54.1, 55.9, 56.7, 74.3, 79.3, 99.5, 105.0, 111.4, 117.6, 118.9, 130.6, 138.4, 157.6, 158.7, 170.4; **IR** (neat) 3408, 3286, 2936, 2838, 1604, 1473, 1269, 1116, 1068 cm^{-1} ; **HRMS** (ESI) calcd for $\text{C}_{16}\text{H}_{17}\text{NNaO}_5$ ($[\text{M}+\text{Na}]^+$) m/z 326.09989, found m/z 326.10042.



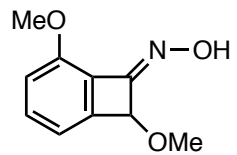
To a solution of oxime (*Z*)-**2a** (29.1 mg, 0.151 mmol), Et_3N (26.8 μL , 0.193 mmol), propargylalcohol **4b** (370 μL , 3.03 mmol), and phenylacetylene (330 μL , 3.02 mmol) in CH_2Cl_2 (1.5 mL) was added NCS (26.0 mg, 0.195 mmol) at 0 °C. After stirring for 10 min at 0 °C, the solution was diluted with CH_2Cl_2 (30 mL). After kept standing for 3 h at room temperature, the reaction was stopped by adding water, and the mixture was extracted with EtOAc (x3). The combined organic extracts were washed with brine, dried (Na_2SO_4), and concentrated in vacuo. The residue was purified by PTLC (hexane/ $\text{EtOAc} = 2/1$) to give isoxazole **6b** (36.4 mg, 75%) as colorless oil. The yield of unreacted phenylacetylene (98%) was determined by $^1\text{H NMR}$ analysis of the crude sample using mesitylene as an internal standard.

R_f 0.43 (hexane/ $\text{EtOAc}/\text{CH}_2\text{Cl}_2 = 2/1/1$); $^1\text{H NMR}$ (600 MHz, CDCl_3) δ 3.38 (s, 3H), 3.98 (s, 3H), 4.64 (d, 1H, $J = 13.2$ Hz), 4.85 (d, 1H, $J = 13.2$ Hz), 5.54 (s, 1H), 7.12 (d, 1H, $J = 8.4$ Hz), 7.18 (d, $J = 7.2$ Hz), 7.46–7.76 (m, 4H), 7.77 (d, 2H, $J = 8.4$ Hz); $^{13}\text{C NMR}$ (150 MHz, CDCl_3) δ 55.6, 56.4, 58.2, 103.4, 112.7, 112.8, 115.9, 120.5, 127.2, 127.7, 129.1, 130.1, 131.1, 139.3, 157.3, 160.7, 163.8; **IR** (neat) 3066, 3000, 2944, 2844, 1597, 1474, 1413, 1353, 1270, 1084, 1061, 942, 698 cm^{-1} ; **HRMS** (ESI) calcd for $\text{C}_{19}\text{H}_{18}\text{NO}_4$ ($[\text{M}+\text{H}]^+$) m/z 324.1232, Found m/z 324.1230.



To a solution of nitrile oxide **3a** (50.0 mg, 0.224 mmol) in CH_2Cl_2 (0.90 mL) was added propargylalcohol **4b** (36.0 μL , 0.291 mmol) and phenylacetylene (32.0 μL , 0.291 mmol) at room temperature. After stirring for 53 h at room temperature, the mixture was concentrated in vacuo. The residue was purified by PTLC (hexane/ $\text{EtOAc} = 1/1$) to give isoxazole **9** (66.3 mg, 91%) as colorless oil.

R_f 0.34 (hexane/EtOAc/CH₂Cl₂ = 2/1/1); **¹H NMR** (600 MHz, CDCl₃) δ 3.30 (s, 6H), 3.80 (s, 3H), 5.43 (s, 1H), 6.63 (s, 1H) 6.99 (d, 1H, *J* = 8.4 Hz), 7.36 (d, 1H, *J* = 7.8 Hz), 7.43–7.50 (m, 4H), 7.85 (d, 2H, *J* = 7.8 Hz); **¹³C NMR** (150 MHz, CDCl₃) δ 54.1, 56.0, 101.7, 102.5, 111.2, 117.8, 118.7, 125.9, 127.7, 129.0, 130.0, 130.5, 157.7, 159.4, 169.1; **IR** (neat) 2932, 2906, 2827, 1601, 1586, 1575, 1470, 1269, 1076, 1053, 763, 696 cm⁻¹; **HRMS** (ESI) calcd for C₁₉H₁₉NNaO₄ ([M+Na]⁺) *m/z* 348.1206, Found *m/z* 348.1209.



(Z)-2a

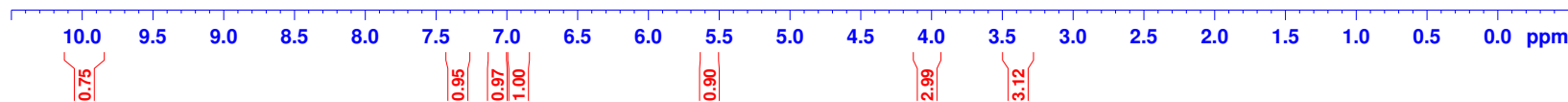
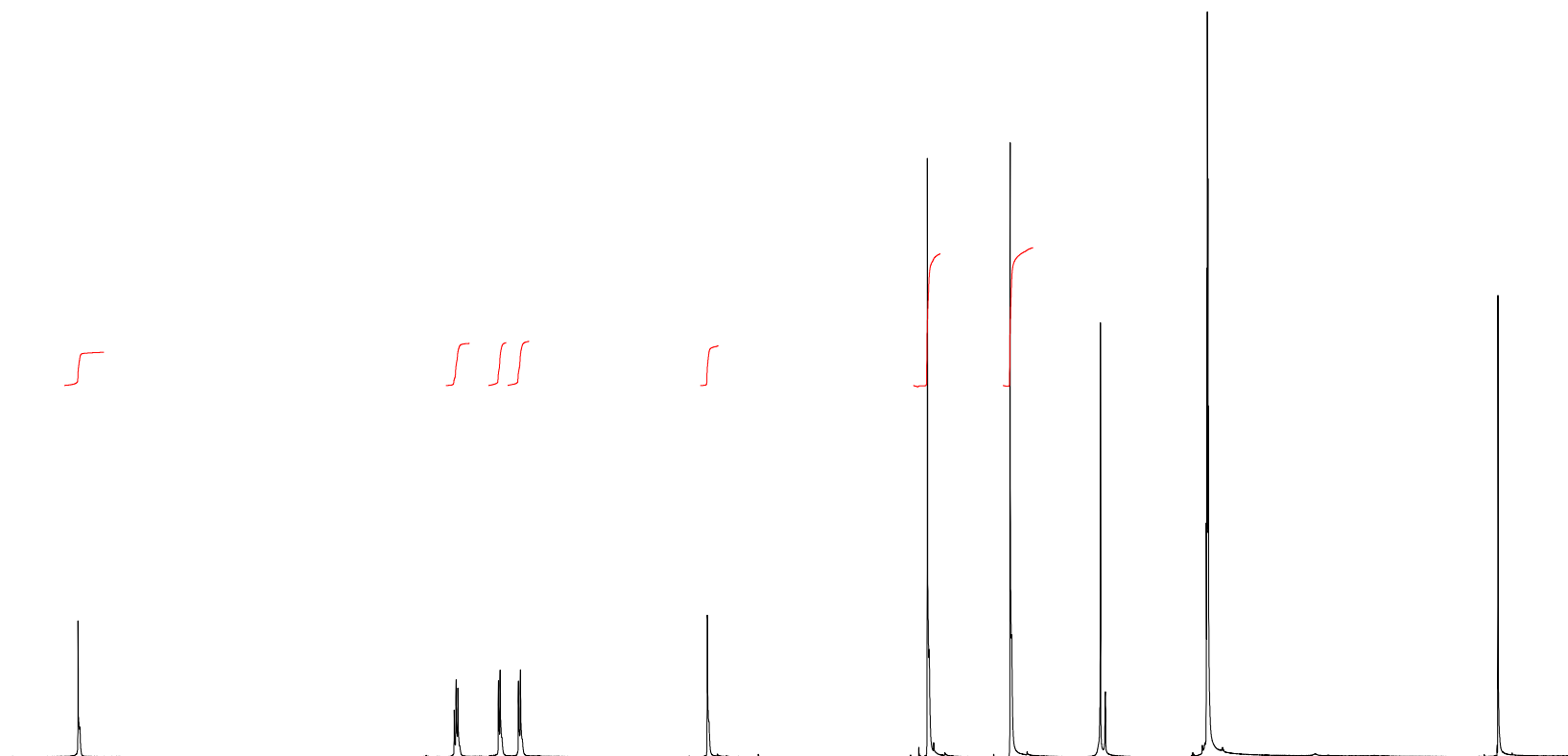


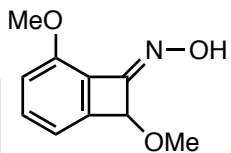
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 PROCNO 1

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 TD 65536
 SOLVENT Acetone
 NS 16
 DS 2
 SWH 12019.230 Hz
 FIDRES 0.183399 Hz
 AQ 2.7262976 sec
 RG 99.18
 DW 41.600 use
 DE 6.50 use
 TE 298.0 K
 D1 1.0000000 sec
 TD0 1

==== CHANNEL f1 =====
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 P1 12.00 use
 PLW1 22.0000000 W

F2 - Processing parameters
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(Z)-2a



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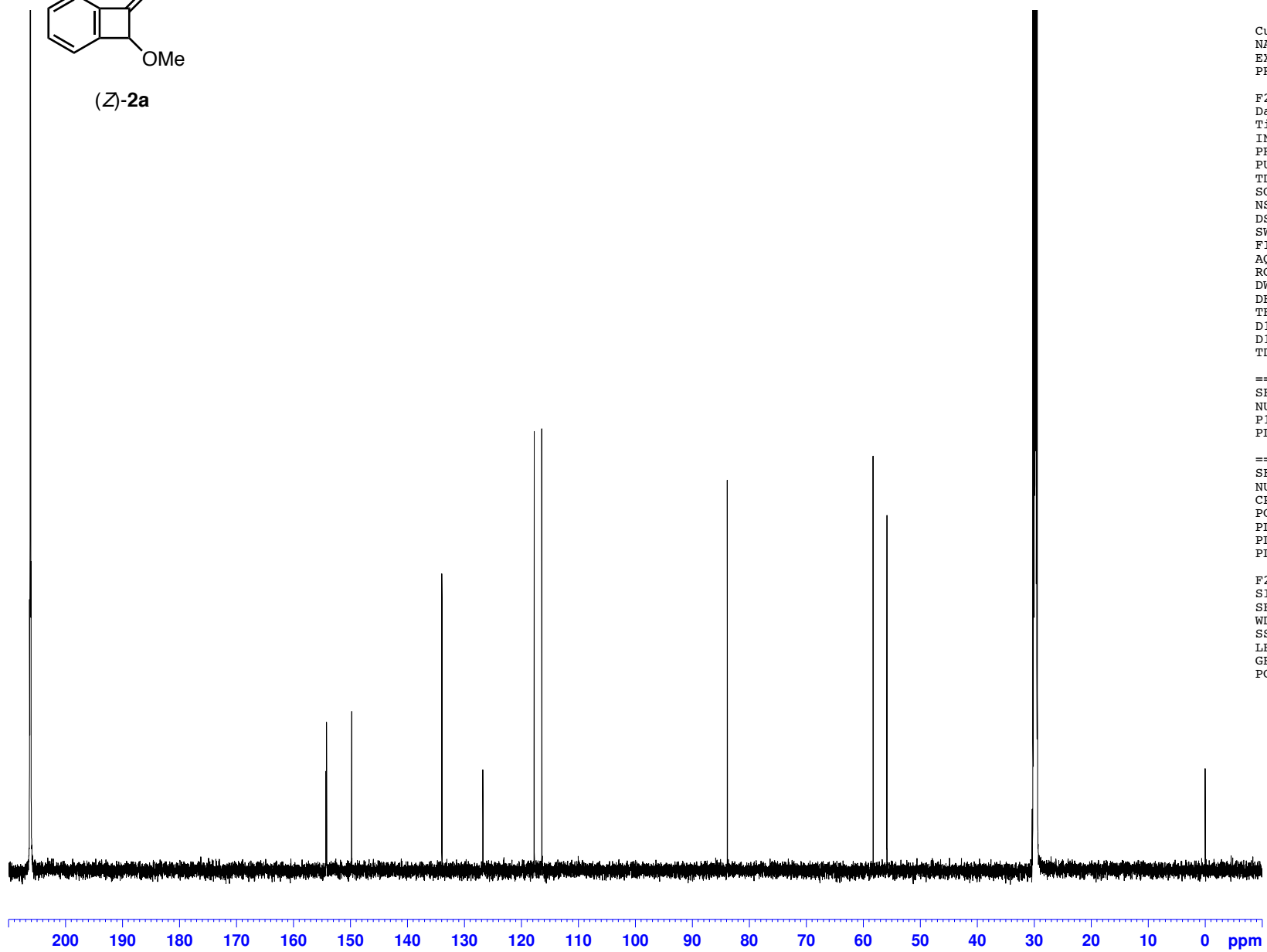
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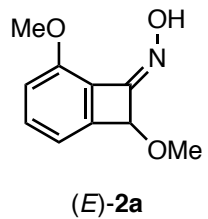
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FIDRES        0.550197 Hz
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DW            13.867 use
DE            6.50 use
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NUC1           13C
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PLW1          93.00000000 W

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NUC2            1H
CPDPRG[2]     waltz16
PCPD2         70.00 use
PLW2          22.00000000 W
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PLW13         0.31680000 W

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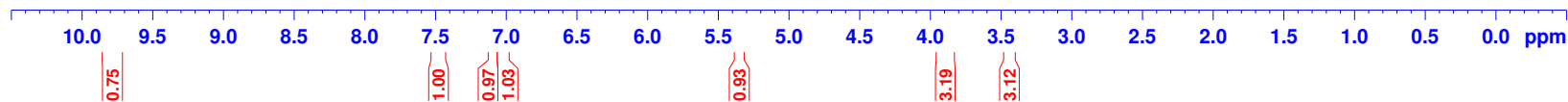
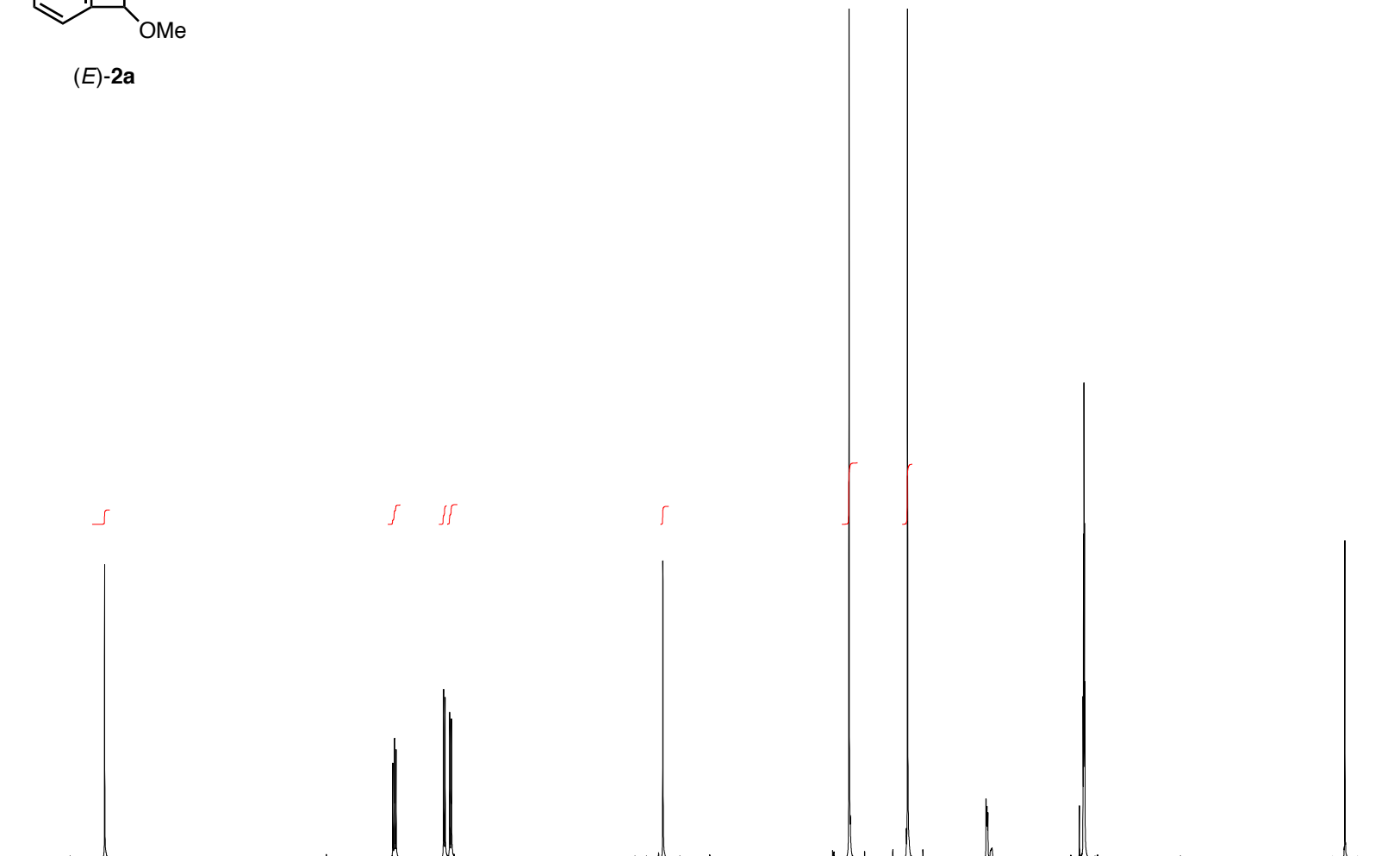


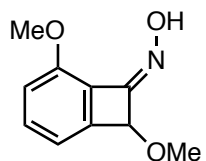
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 DS 2
 SWH 12019.230 Hz
 FIDRES 0.183399 Hz
 AQ 2.7262976 sec
 RG 87.68
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 D1 1.0000000 sec
 TD0 1

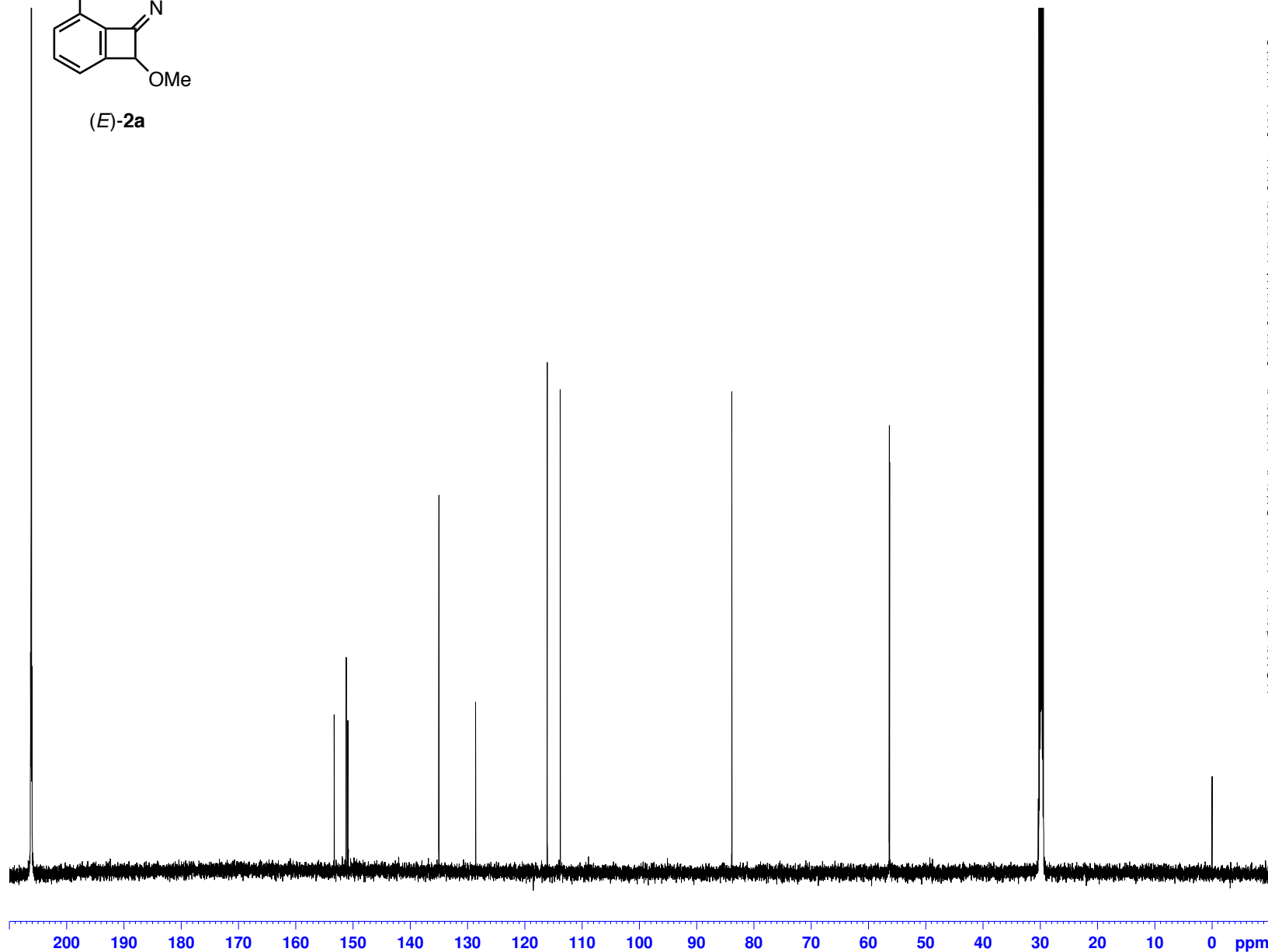
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(E)-2a



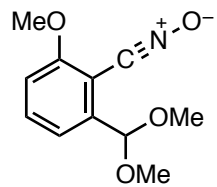
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 DS 4
 SWH 36057.691 Hz
 FIDRES 0.550197 Hz
 AQ 0.9087659 sec
 RG 175.56
 DW 13.867 use
 DE 6.50 use
 TE 298.0 K
 D1 2.0000000 sec
 D11 0.0300000 sec
 TD0 1

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 NUC1 13C
 P1 11.00 use
 PLW1 93.0000000 W

==== CHANNEL f2 =====
 SFO2 600.1324005 MHz
 NUC2 1H
 CPDPRG[2] waltz16
 PCPD2 70.00 use
 PLW2 22.0000000 W
 PLW12 0.64652997 W
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 SSB 0
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3a

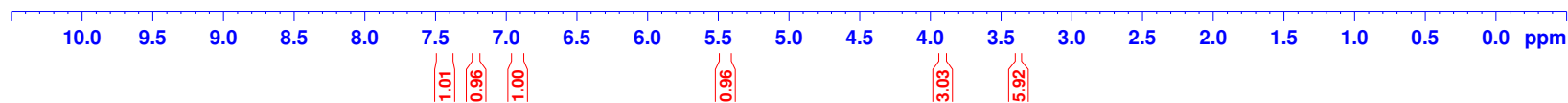
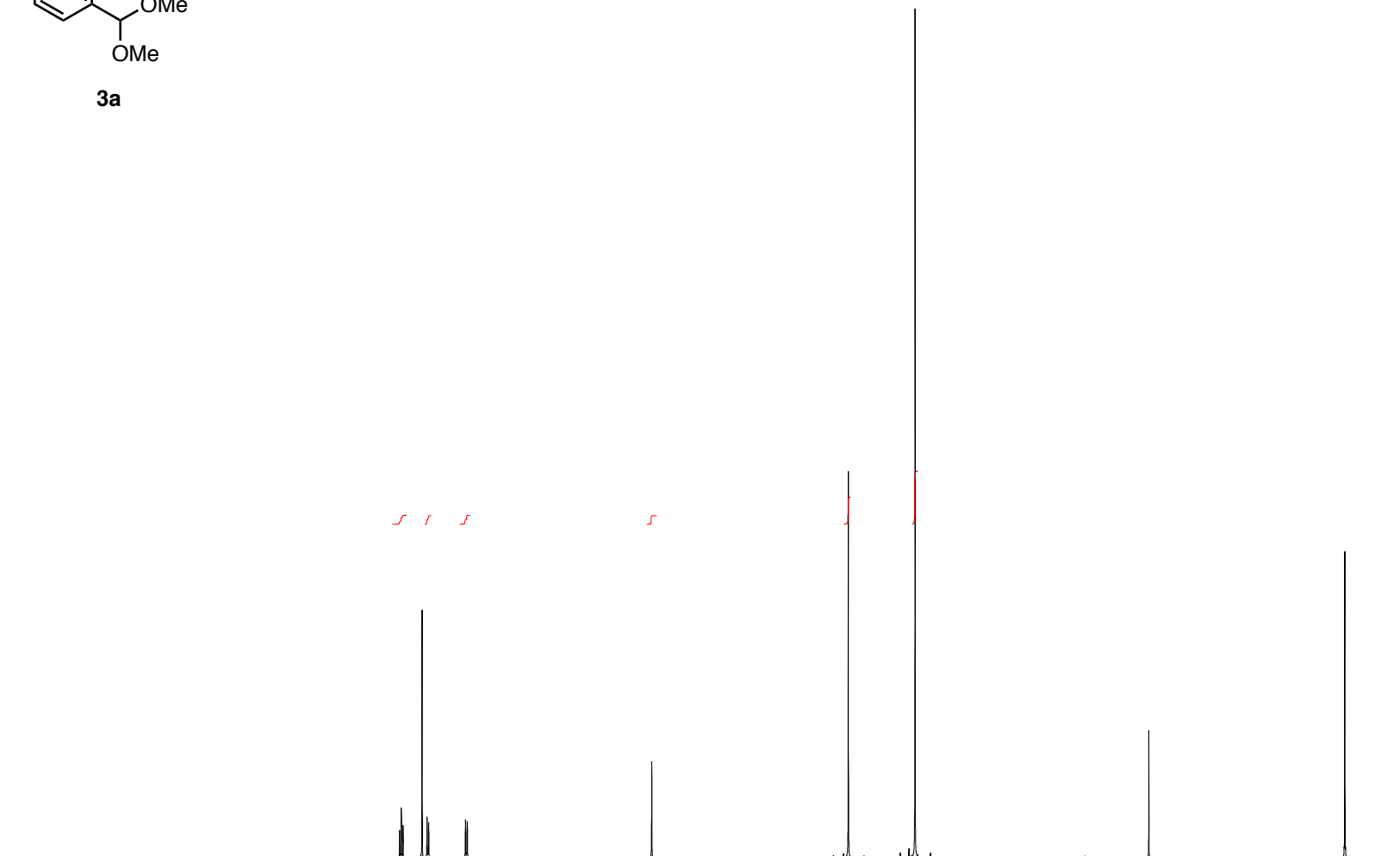


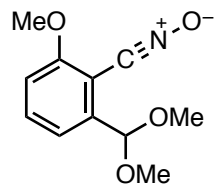
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 SOLVENT CDC13
 NS 16
 DS 2
 SWH 12019.230 Hz
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 AQ 2.7262976 sec
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 TD0 1

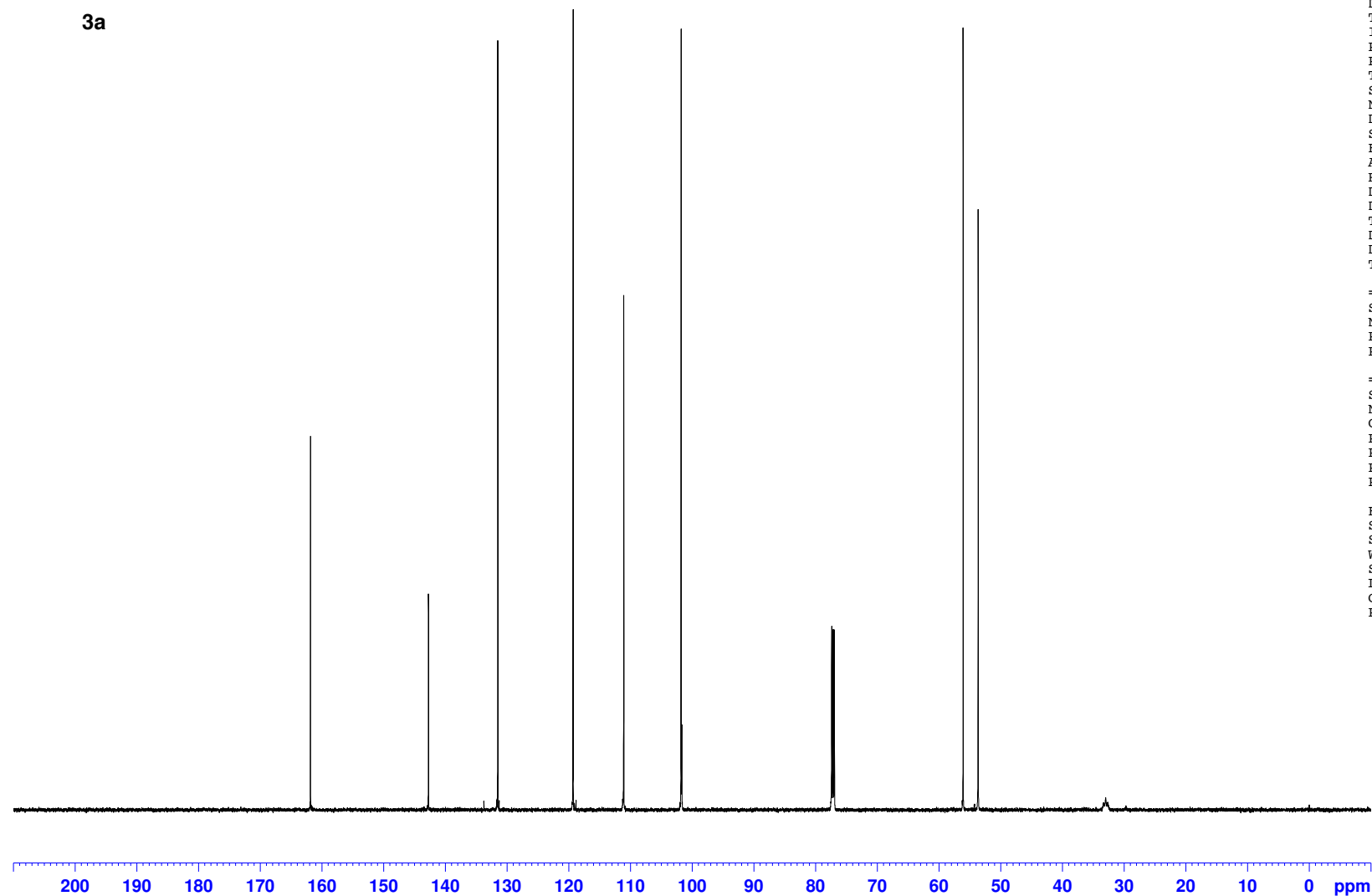
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3a



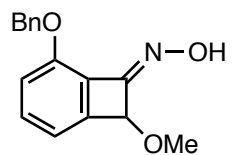
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 SOLVENT CDC13
 NS 600
 DS 4
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 FIDRES 0.550197 Hz
 AQ 0.9087659 sec
 RG 175.56
 DW 13.867 use
 DE 6.50 use
 TE 298.0 K
 D1 2.0000000 sec
 D11 0.0300000 sec
 TD0 1

==== CHANNEL f1 =====
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 NUC1 13C
 P1 11.00 use
 PLW1 93.0000000 W

==== CHANNEL f2 =====
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 CPDPRG[2] waltz16
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(Z)-2b

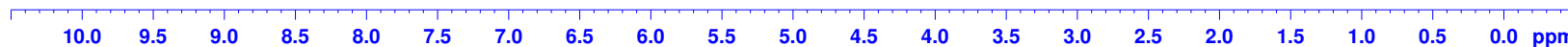
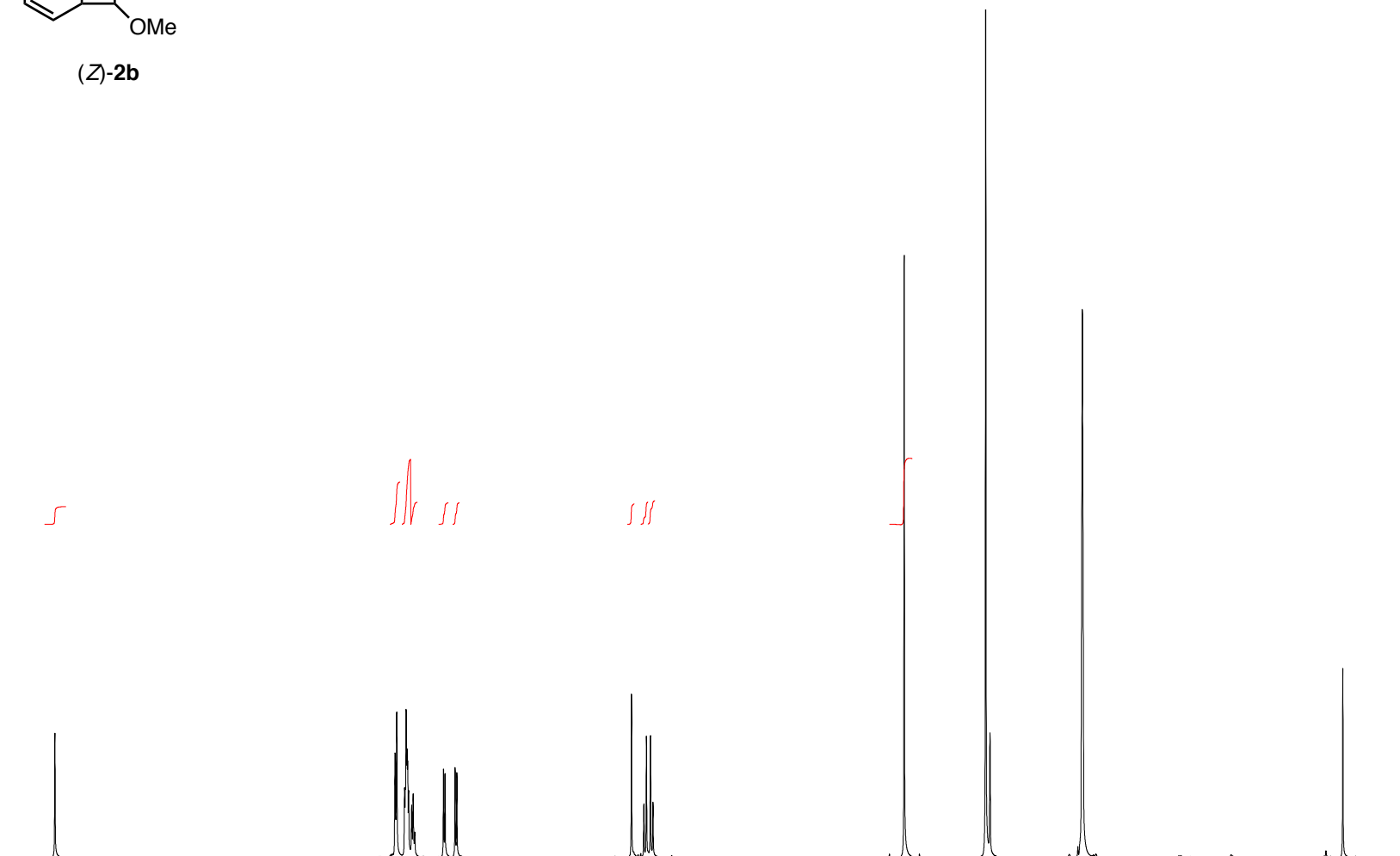


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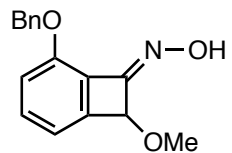
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 NS 16
 DS 2
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 FIDRES 0.183399 Hz
 AQ 2.7262976 sec
 RG 31.94
 DW 41.600 use
 DE 10.00 use
 TE 300.0 K
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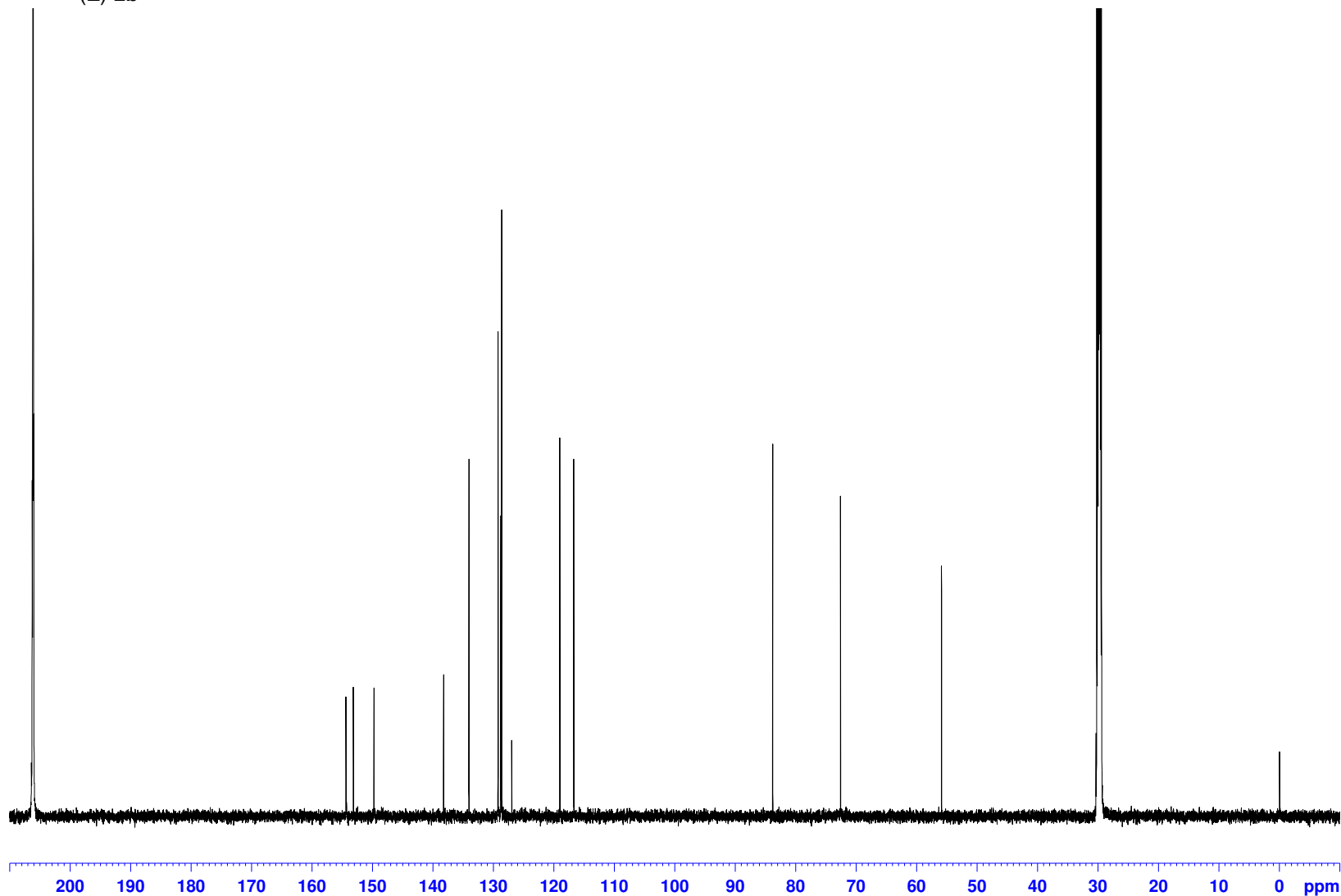
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 0.95 1.04 1.10
 3.04



(Z)-2b



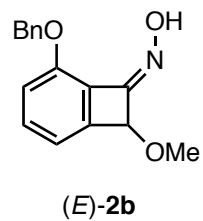
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 SOLVENT Acetone
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 DS 4
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 FIDRES 0.550197 Hz
 AQ 0.9087659 sec
 RG 175.56
 DW 13.867 use
 DE 18.00 use
 TE 300.0 K
 D1 2.0000000 sec
 D11 0.0300000 sec
 TD0 1

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 NUC1 13C
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 PLW1 70.0000000 W

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 NUC2 1H
 CPDPRG[2] waltz16
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 PLW12 0.76407999 W
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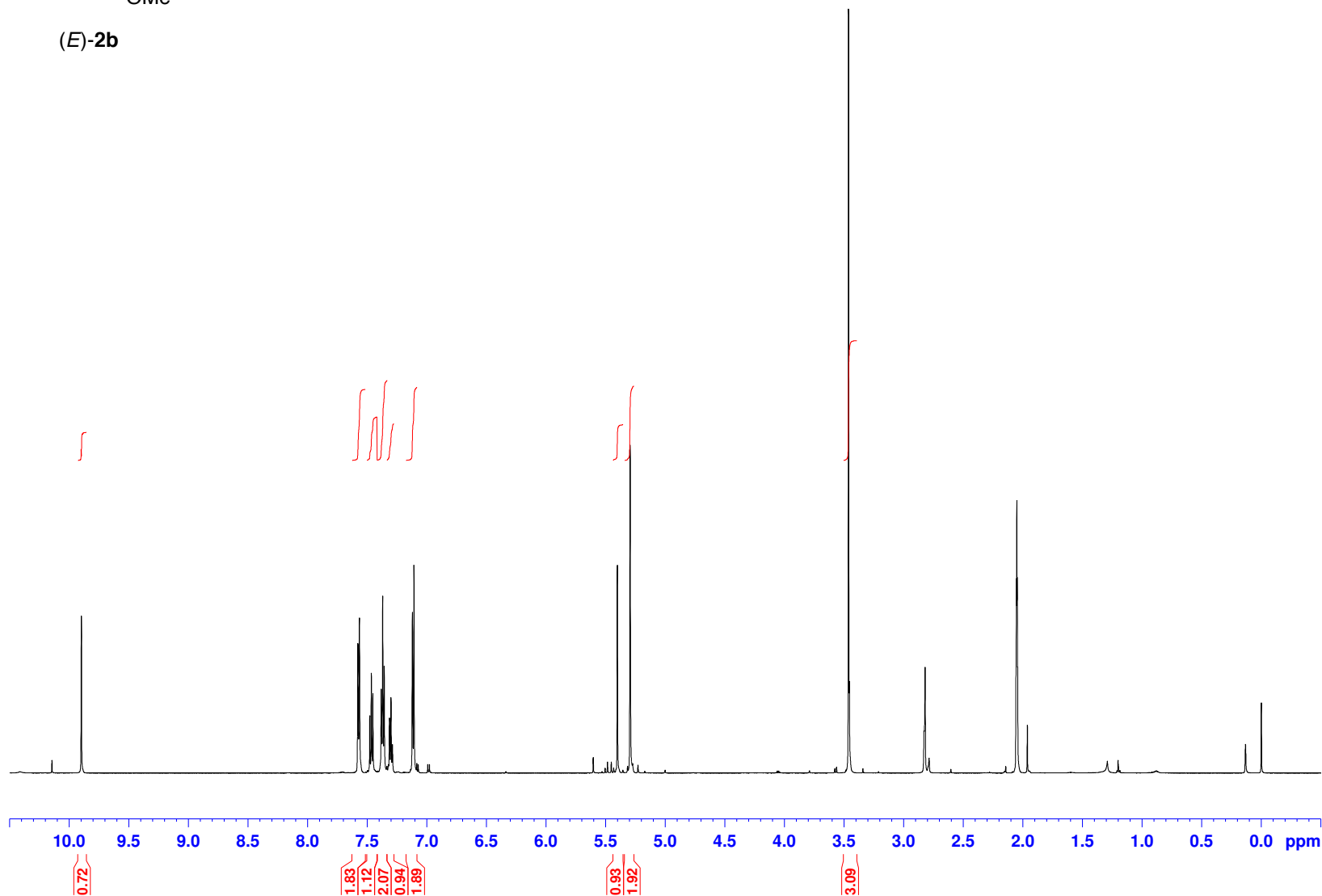


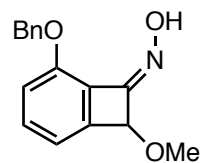
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 SOLVENT Acetone
 NS 16
 DS 2
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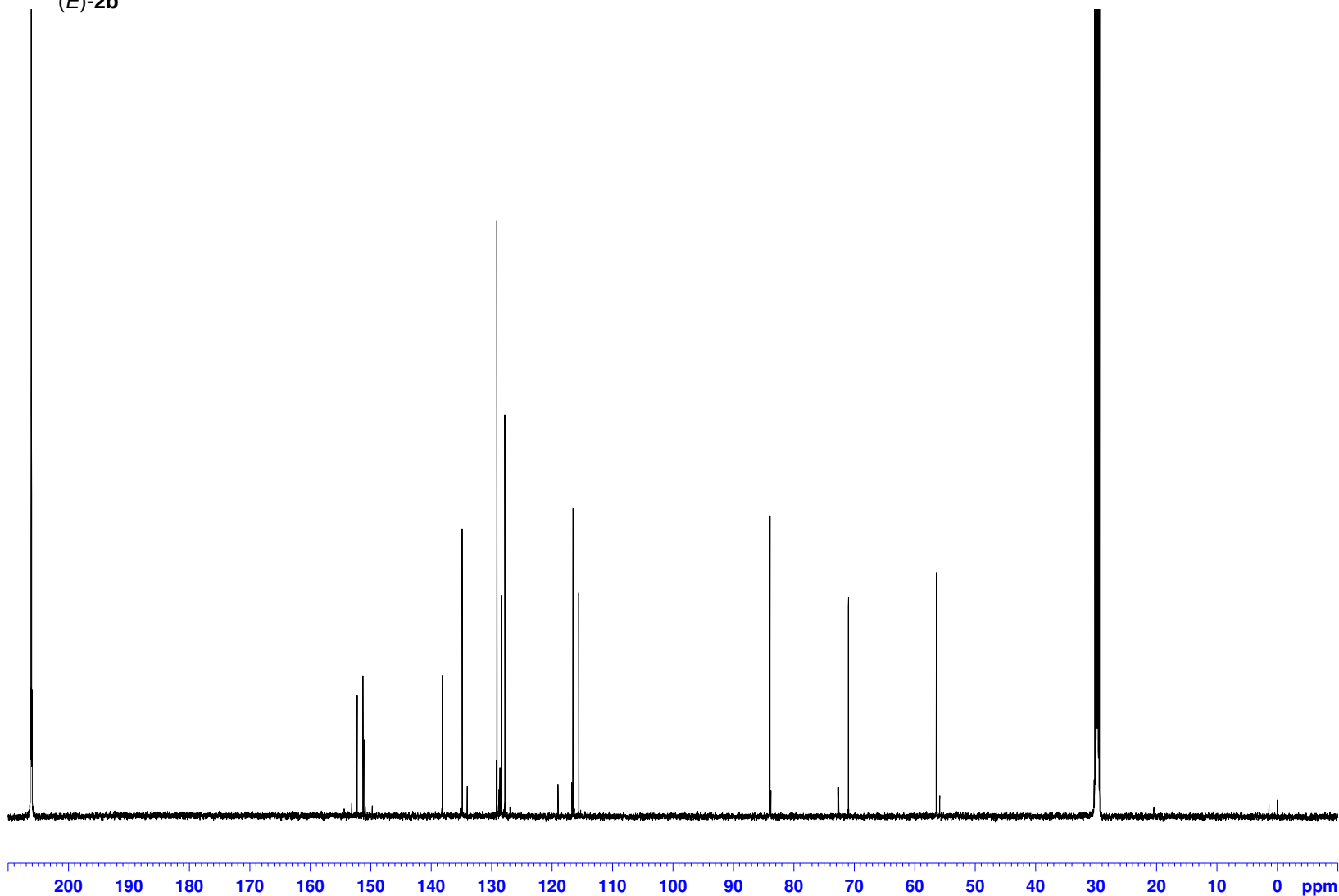
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(E)-2b



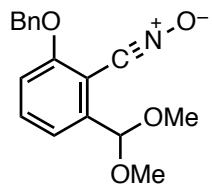
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 DE 6.50 use
 TE 298.0 K
 D1 2.0000000 sec
 D11 0.0300000 sec
 TD0 1

==== CHANNEL f1 =====
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 PLW1 93.0000000 W

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 CPDPRG[2] waltz16
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 PLW12 0.64652997 W
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3b

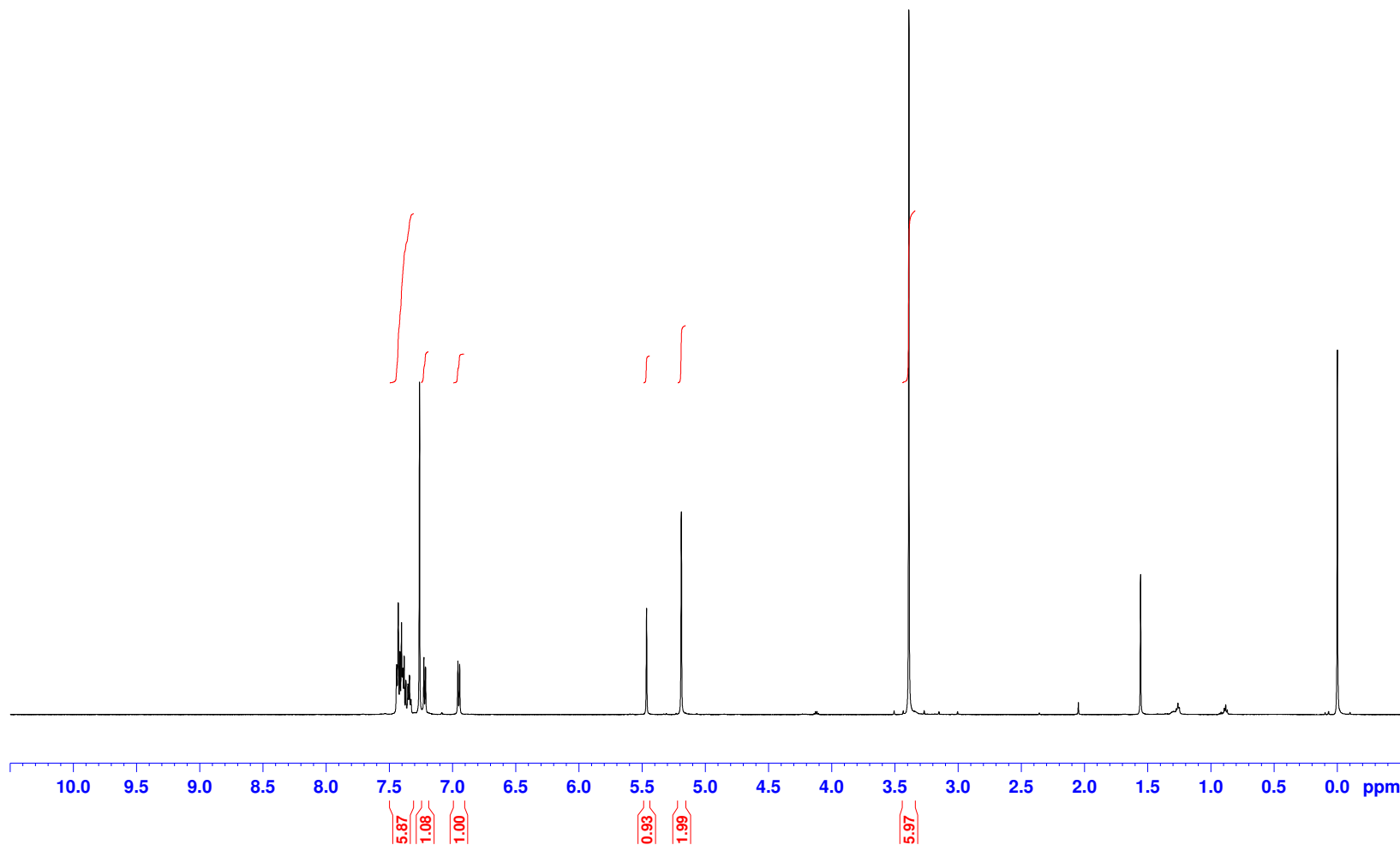


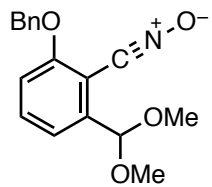
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 SOLVENT CDC13
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 RG 31.94
 DW 41.600 use
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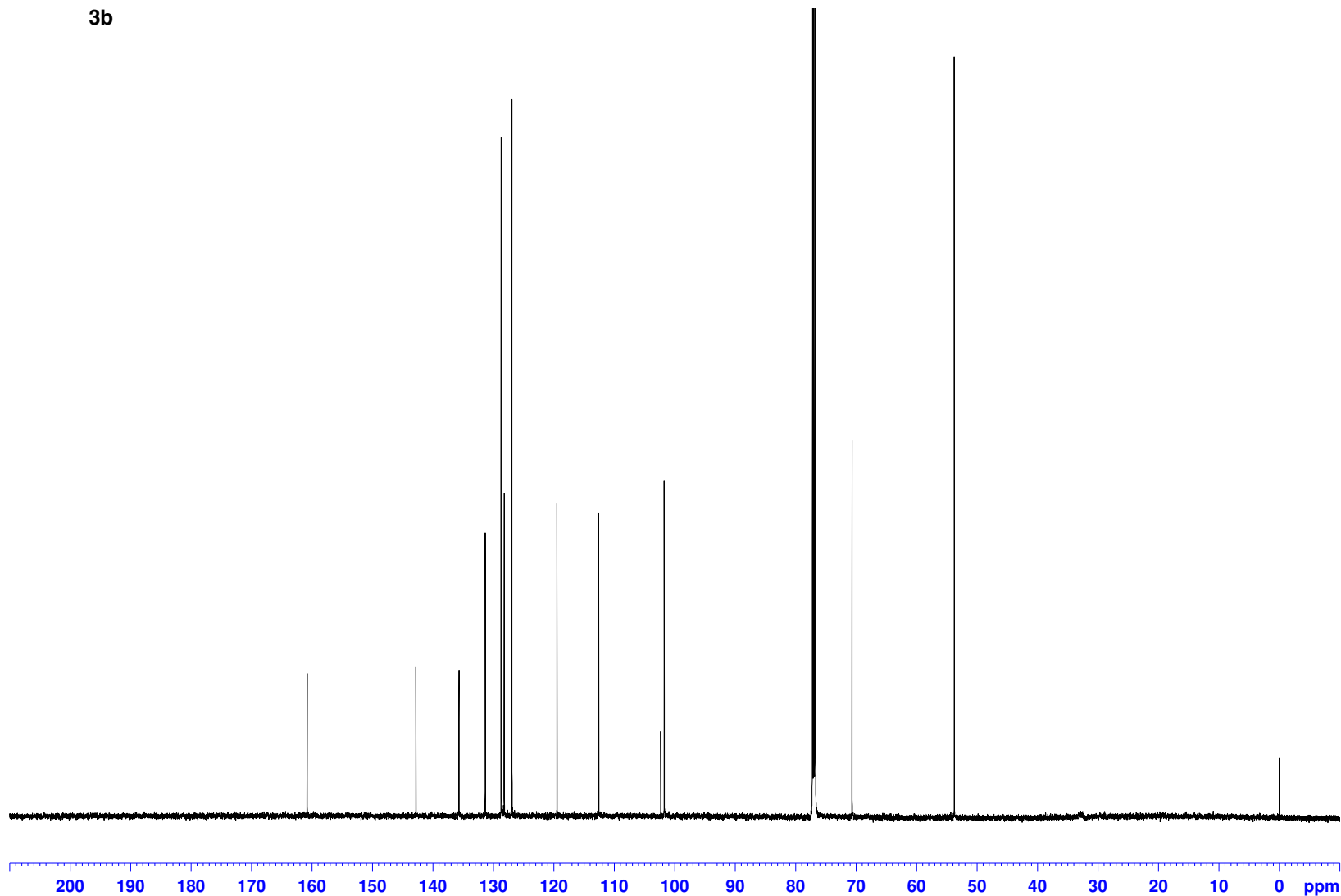
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3b



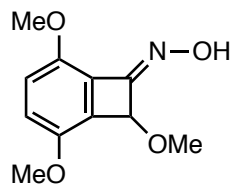
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 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 1024
 DS 4
 SWH 36057.691 Hz
 FIDRES 0.550197 Hz
 AQ 0.9087659 sec
 RG 175.56
 DW 13.867 use
 DE 18.00 use
 TE 291.5 K
 D1 2.0000000 sec
 D11 0.0300000 sec
 TD0 1

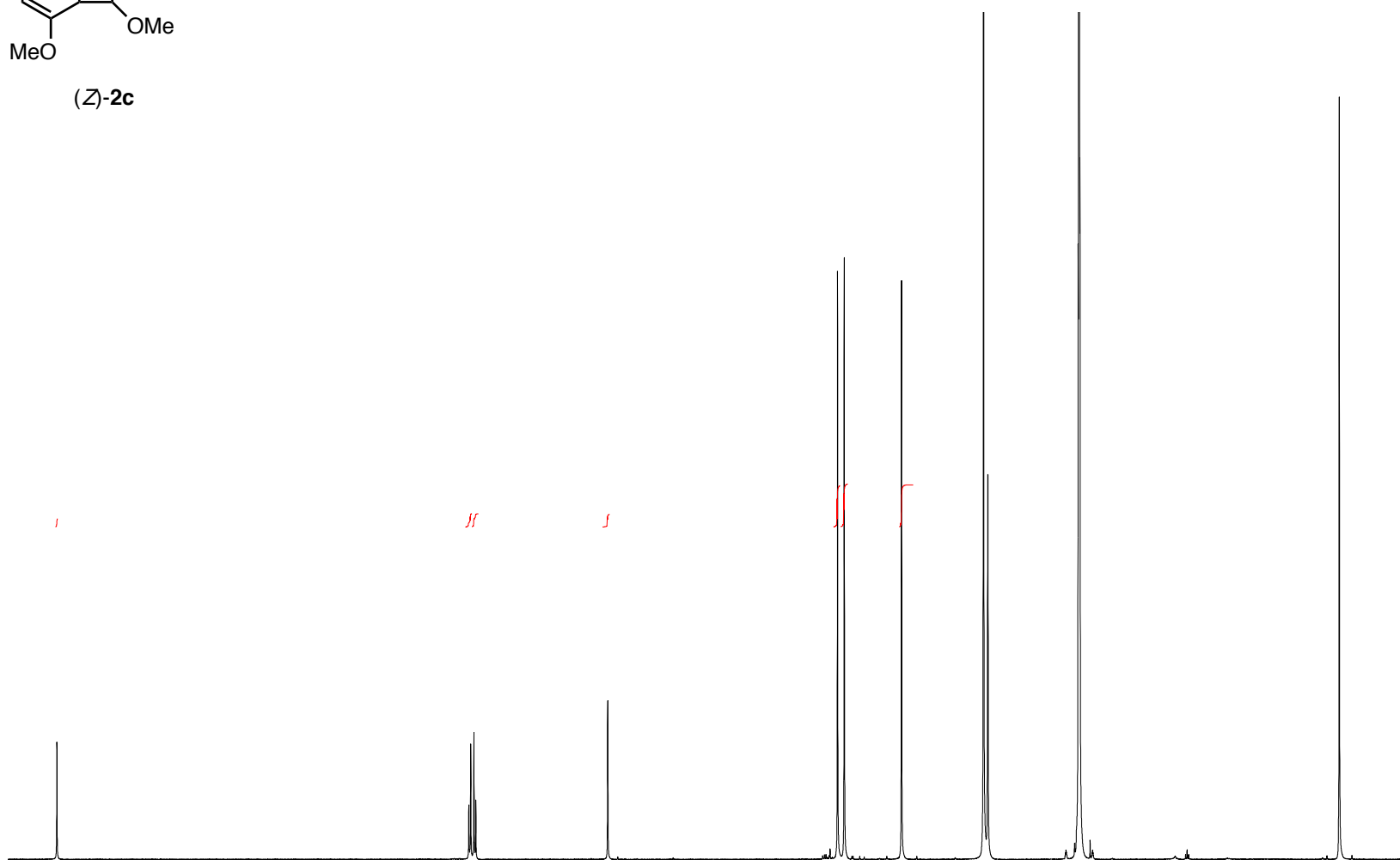
==== CHANNEL f1 =====
 SFO1 150.9178981 MHz
 NUC1 13C
 P1 10.00 use
 PLW1 70.0000000 W

==== CHANNEL f2 =====
 SFO2 600.1324005 MHz
 NUC2 1H
 CPDPRG[2] waltz16
 PCPD2 70.00 use
 PLW2 26.0000000 W
 PLW12 0.76407999 W
 PLW13 0.37439999 W

F2 - Processing parameters
 SI 32768
 SF 150.9028128 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40



(Z)-2c

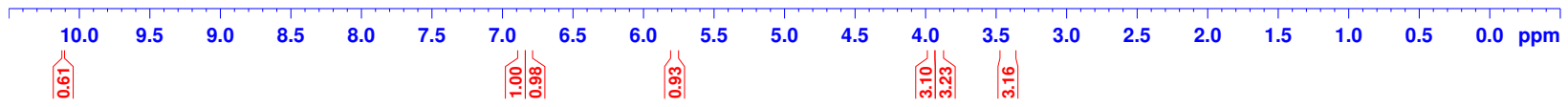


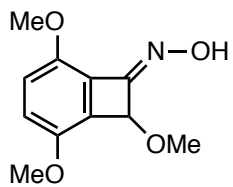
Current Data Parameters
 NAME rs3-236-1 acetone
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20150807
 Time 21.00
 INSTRUM spect
 PROBHD 5 mm CPPBBO BB
 PULPROG zg30
 TD 65536
 SOLVENT Acetone
 NS 16
 DS 2
 SWH 12019.230 Hz
 FIDRES 0.183399 Hz
 AQ 2.7262976 sec
 RG 31.94
 DW 41.600 use
 DE 10.00 use
 TE 300.0 K
 D1 1.00000000 sec
 TD0 1

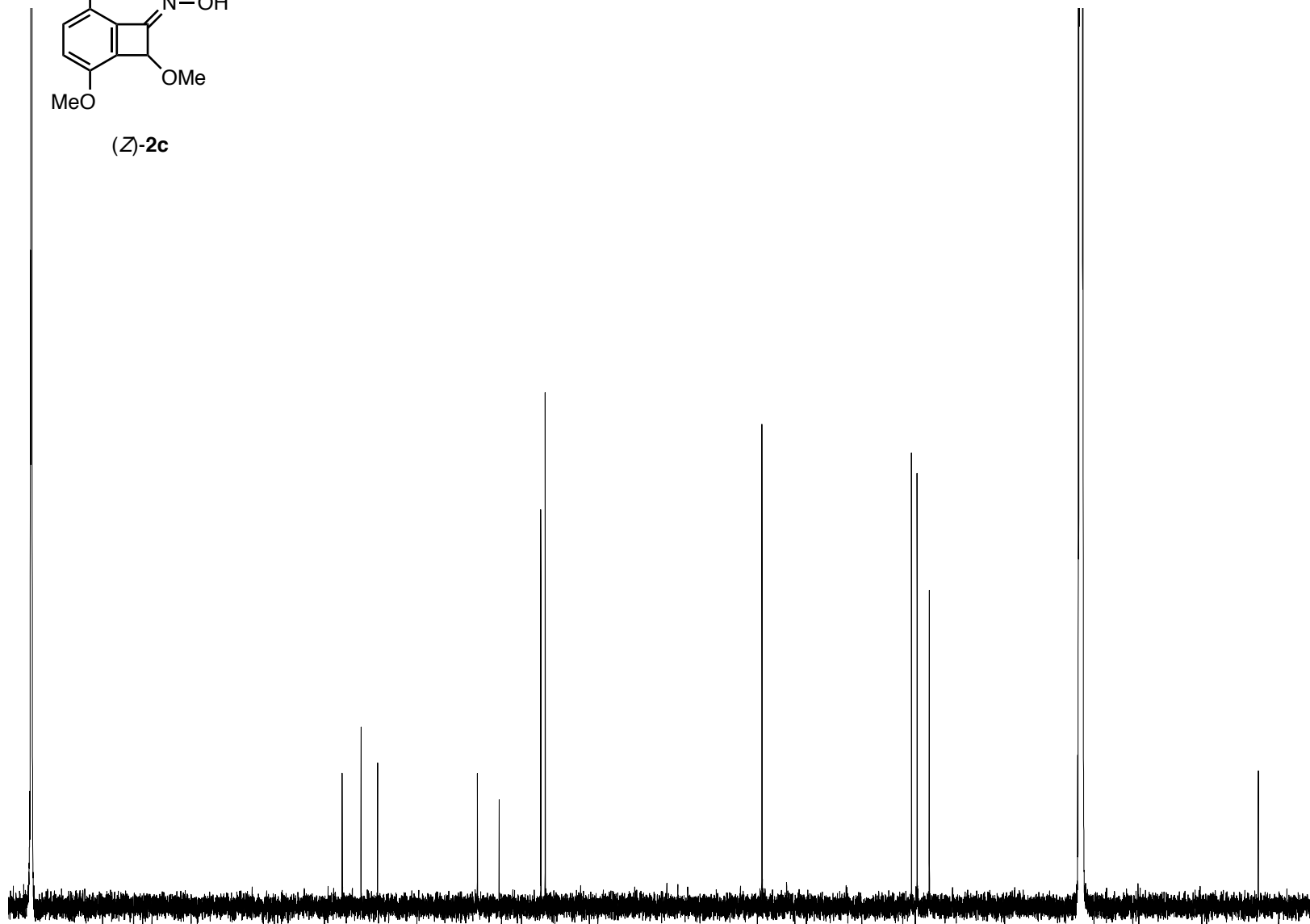
==== CHANNEL f1 =====
 SF01 600.1337060 MHz
 NUC1 1H
 P1 12.00 use
 PLW1 23.00000000 W

F2 - Processing parameters
 SI 65536
 SF 600.1300109 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00





(Z)-2c



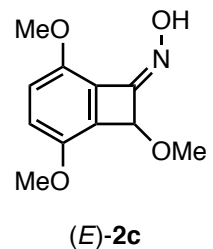
Current Data Parameters
NAME rs3-238-2
EXPNO 21
PROCNO 1

F2 - Acquisition Parameters
Date_ 20150905
Time_ 22.54
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT Acetone
NS 1024
DS 4
SWH 36057.691 Hz
FIDRES 0.550197 Hz
AQ 0.9087659 sec
RG 175.56
DW 13.867 use
DE 18.00 use
TE 300.0 K
D1 2.0000000 sec
D11 0.0300000 sec
TD0 1

==== CHANNEL f1 =====
SFO1 150.9178981 MHz
NUC1 13C
P1 10.00 use
PLW1 70.0000000 W

==== CHANNEL f2 =====
SFO2 600.1324005 MHz
NUC2 1H
CPDPRG[2] waltz16
PCPD2 70.00 use
PLW2 26.0000000 W
PLW12 0.76407999 W
PLW13 0.37439999 W

F2 - Processing parameters
SI 32768
SF 150.9026726 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

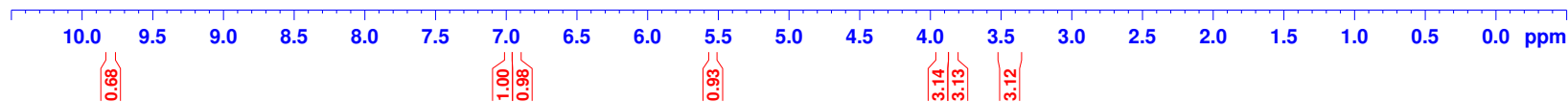
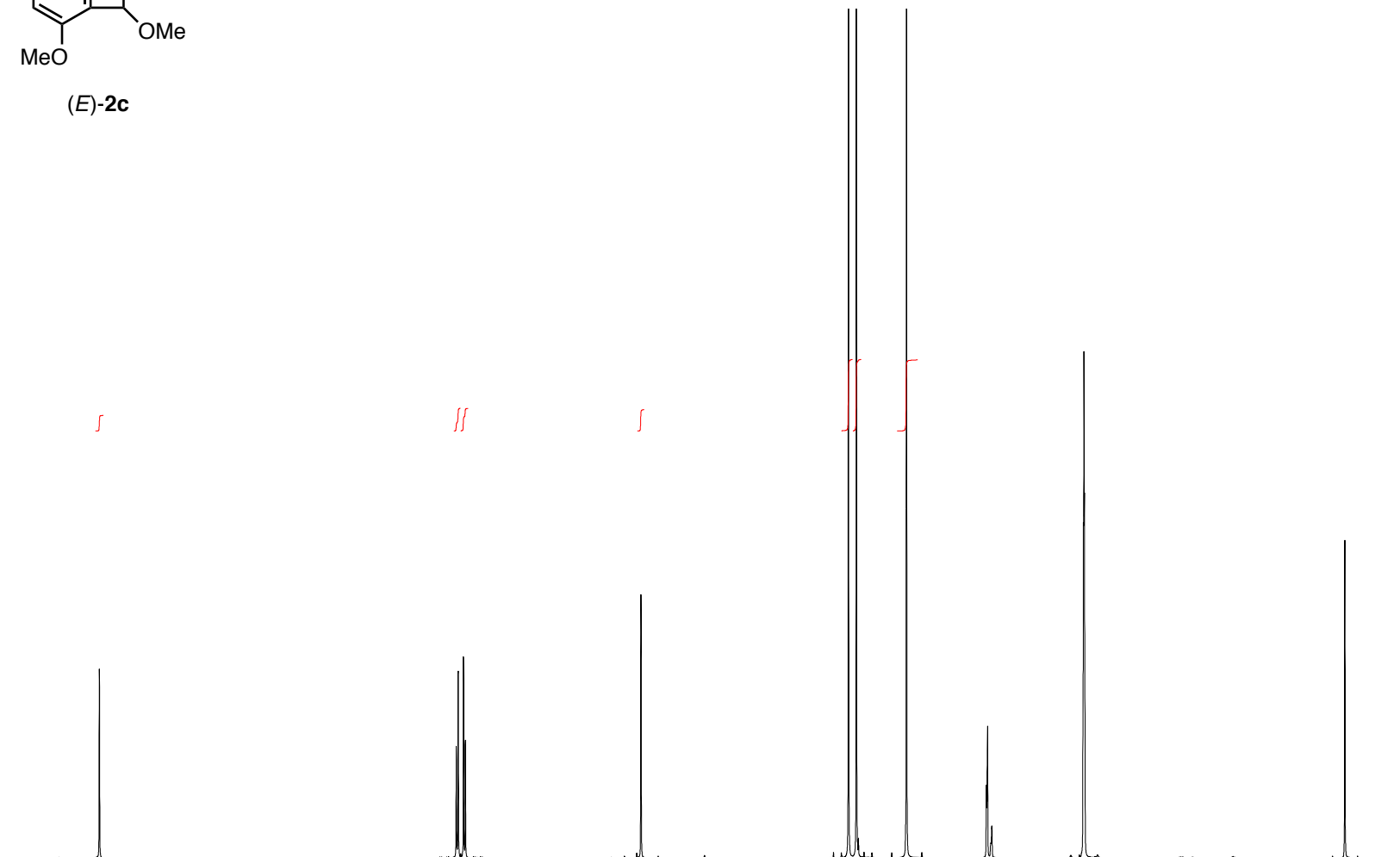


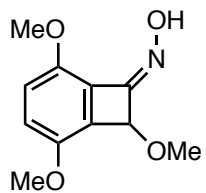
Current Data Parameters
 NAME rs3-238-3 paradi-E
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20161229
 Time_ 6.06
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT Acetone
 NS 16
 DS 2
 SWH 12019.230 Hz
 FIDRES 0.183399 Hz
 AQ 2.7262976 sec
 RG 99.18
 DW 41.600 use
 DE 6.50 use
 TE 298.0 K
 D1 1.00000000 sec
 TD0 1

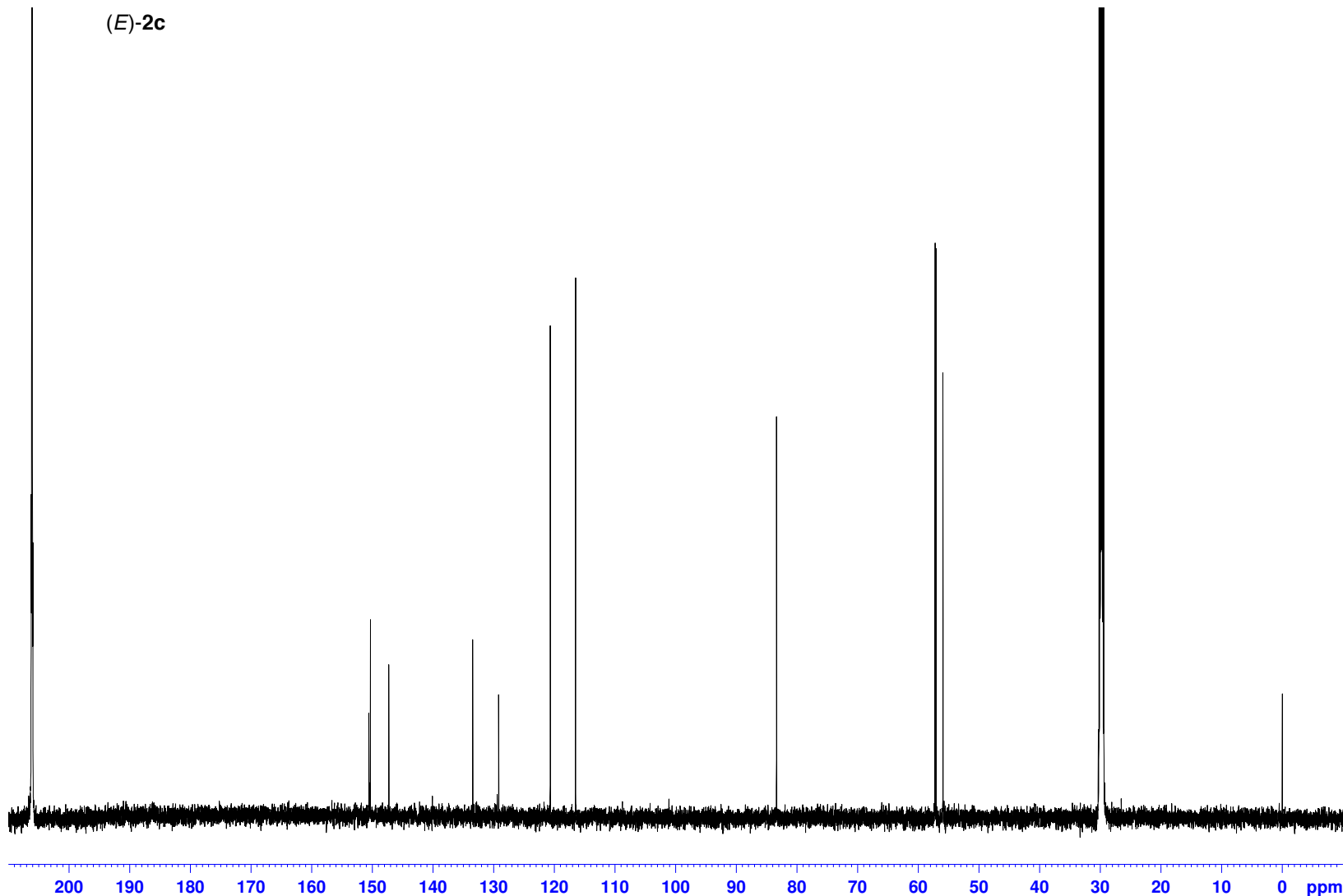
==== CHANNEL f1 =====
 SFO1 600.1337060 MHz
 NUC1 1H
 P1 12.00 use
 PLW1 22.00000000 W

F2 - Processing parameters
 SI 65536
 SF 600.1300126 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00





(E)-2c



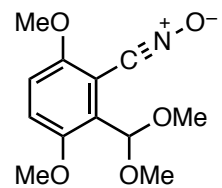
Current Data Parameters
 NAME rs3-238-3 paradi-E
 EXPNO 11
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20161229
 Time 6.05
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT Acetone
 NS 3072
 DS 4
 SWH 36057.691 Hz
 FIDRES 0.550197 Hz
 AQ 0.9087659 sec
 RG 175.56
 DW 13.867 use
 DE 6.50 use
 TE 298.0 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TD0 1

===== CHANNEL f1 =====
 SFO1 150.9178981 MHz
 NUC1 13C
 P1 11.00 use
 PLW1 93.00000000 W

===== CHANNEL f2 =====
 SFO2 600.1324005 MHz
 NUC2 1H
 CPDPRG[2] waltz16
 PCPD2 70.00 use
 PLW2 22.00000000 W
 PLW12 0.64652997 W
 PLW13 0.31680000 W

F2 - Processing parameters
 SI 32768
 SF 150.9026726 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40



3c

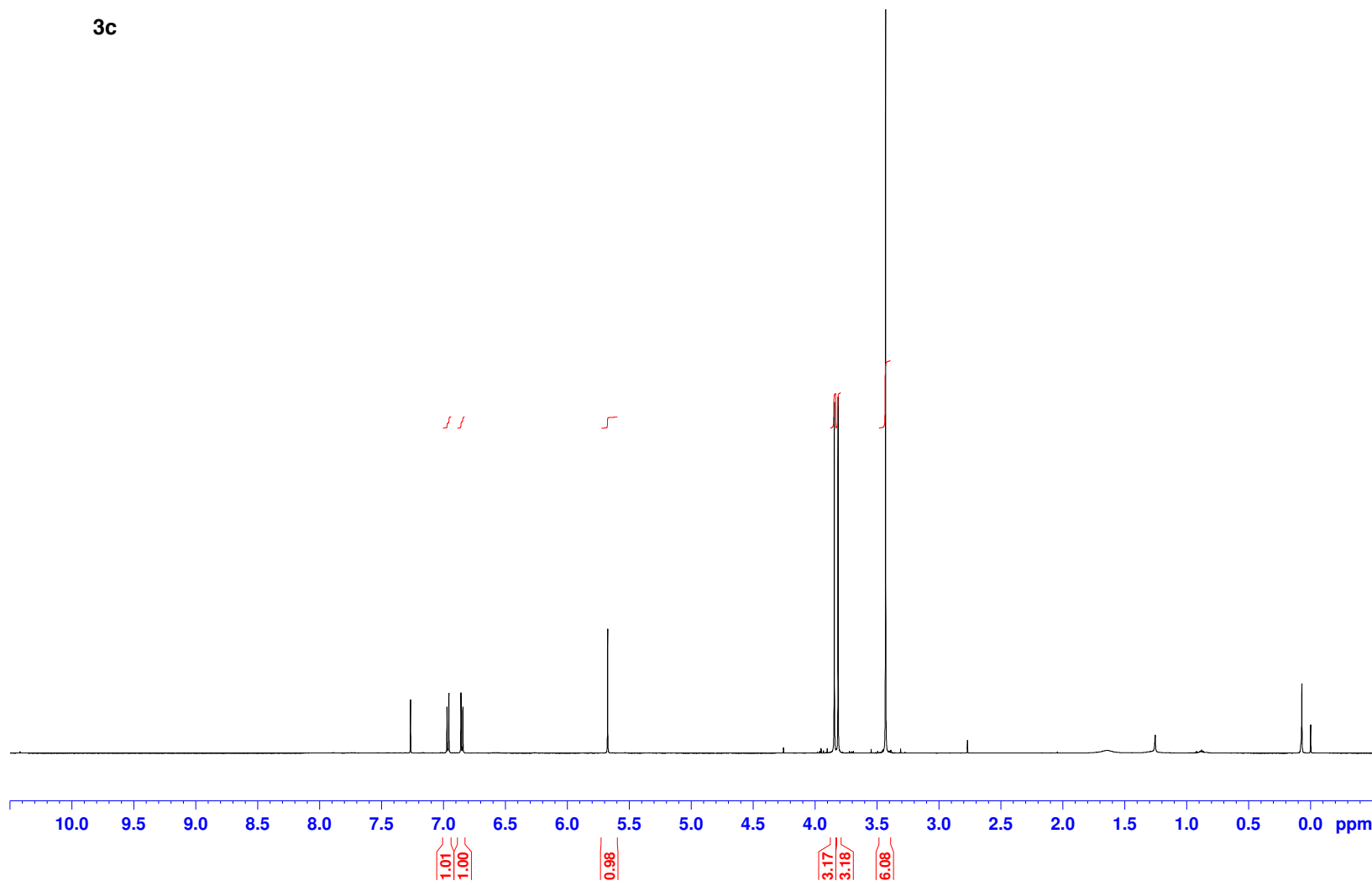


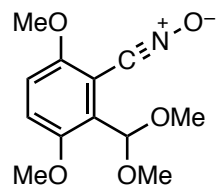
Current Data Parameters
 NAME paradimethoxy nito
 EXPNO 20
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20170220
 Time_ 17.32
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDC13
 NS 16
 DS 2
 SWH 12019.230 Hz
 FIDRES 0.183399 Hz
 AQ 2.7262976 sec
 RG 75.66
 DW 41.600 use
 DE 6.50 use
 TE 298.0 K
 D1 1.0000000 sec
 TD0 1

==== CHANNEL f1 =====
 SFO1 600.1337060 MHz
 NUC1 1H
 P1 12.00 use
 PLW1 22.0000000 W

F2 - Processing parameters
 SI 65536
 SF 600.1300145 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00





3c



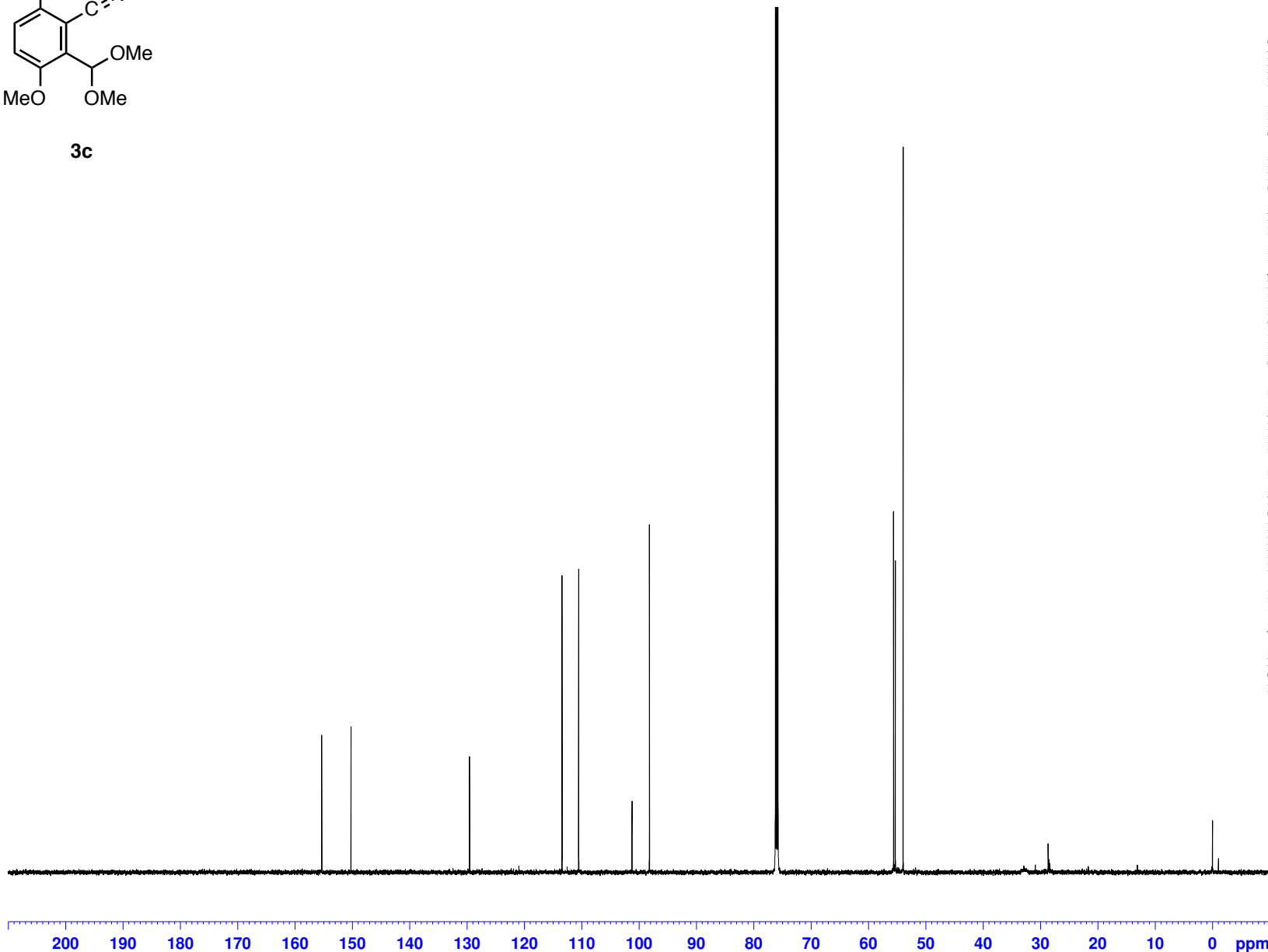
Current Data Parameters
 NAME paradimethoxy nito
 EXPNO 21
 PROCNO 1

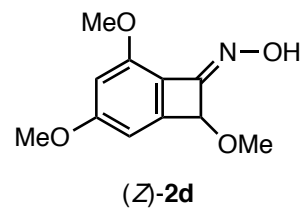
F2 - Acquisition Parameters
 Date_ 20170221
 Time_ 5.00
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 6000
 DS 4
 SWH 36057.691 Hz
 FIDRES 0.550197 Hz
 AQ 0.9087659 sec
 RG 175.56
 DW 13.867 use
 DE 6.50 use
 TE 298.0 K
 D1 2.0000000 sec
 D11 0.0300000 sec
 TD0 1

==== CHANNEL f1 =====
 SFO1 150.9178981 MHz
 NUC1 13C
 P1 11.00 use
 PLW1 93.0000000 W

==== CHANNEL f2 =====
 SFO2 600.1324005 MHz
 NUC2 1H
 CPDPRG[2] waltz16
 PCPD2 70.00 use
 PLW2 22.0000000 W
 PLW12 0.64652997 W
 PLW13 0.31680000 W

F2 - Processing parameters
 SI 32768
 SF 150.9029630 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40



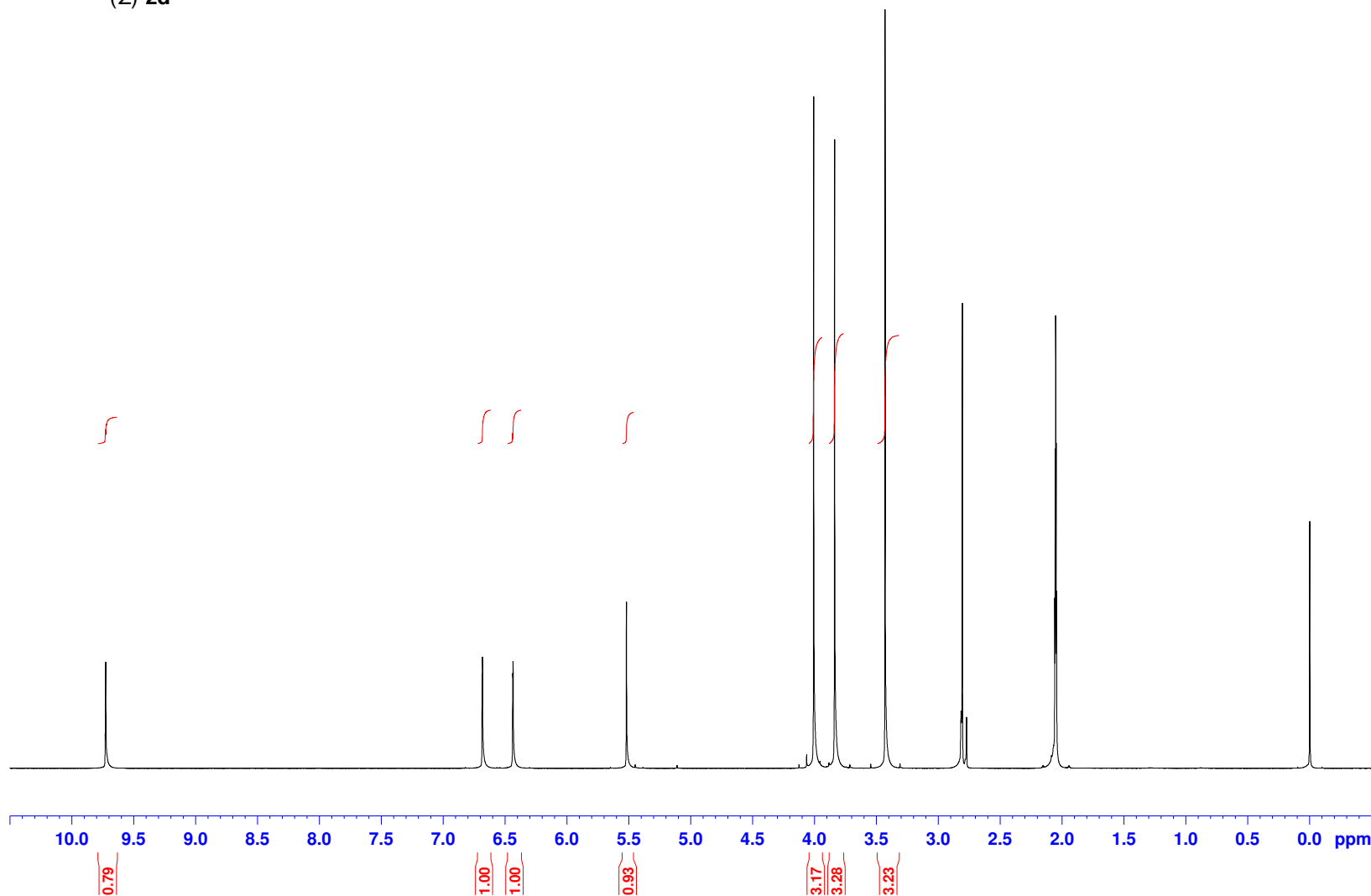


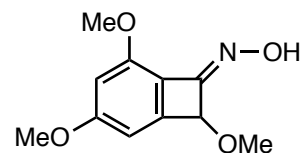
Current Data Parameters
 NAME rs3-166-1
 EXPNO 20
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20150421
 Time_ 17.10
 INSTRUM spect
 PROBHD 5 mm CPPBBO BB
 PULPROG zg30
 TD 65536
 SOLVENT Acetone
 NS 16
 DS 2
 SWH 12019.230 Hz
 FIDRES 0.183399 Hz
 AQ 2.7262976 sec
 RG 31.94
 DW 41.600 use
 DE 10.00 use
 TE 300.0 K
 D1 1.00000000 sec
 TD0 1

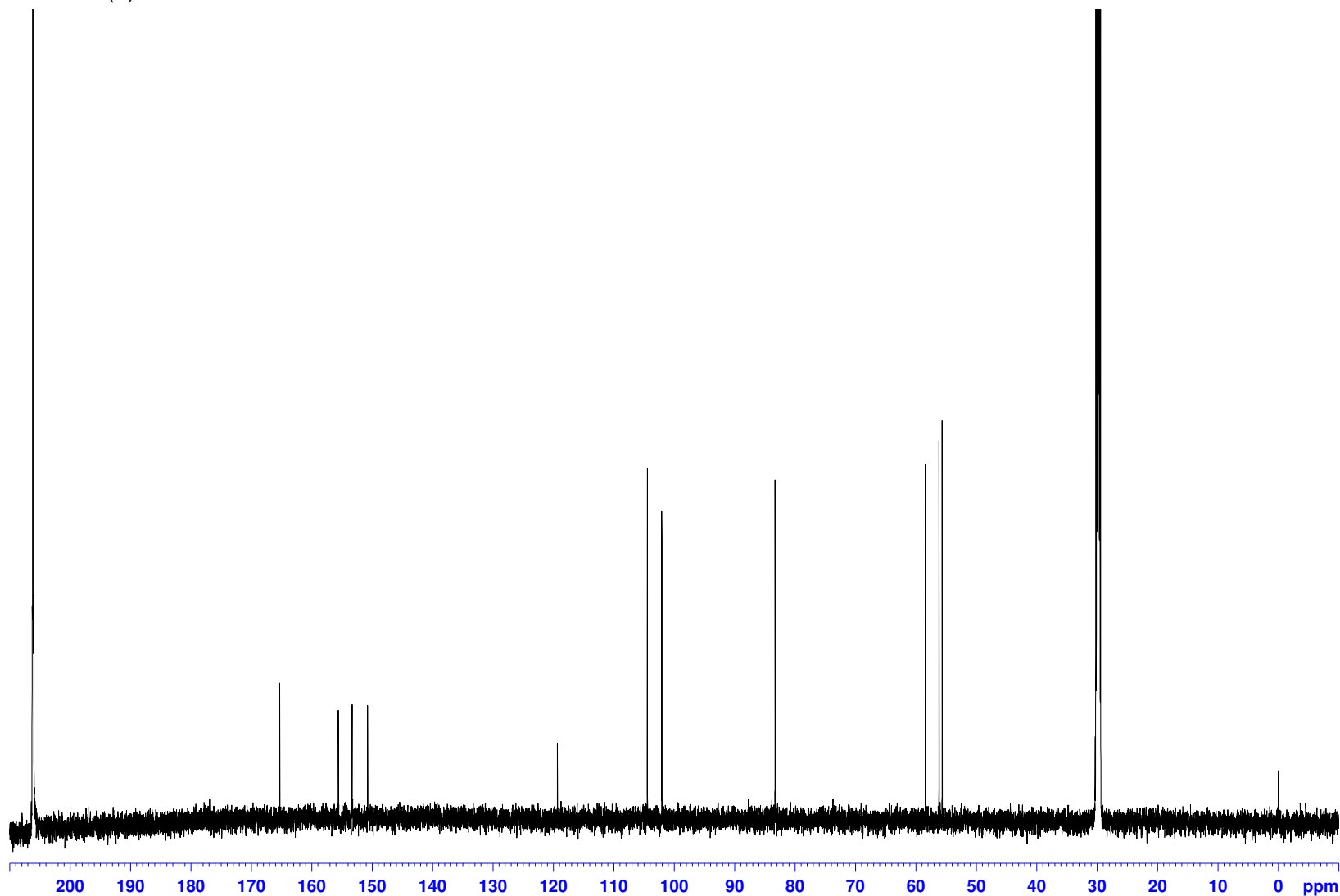
===== CHANNEL f1 =====
 SFO1 600.1337060 MHz
 NUC1 1H
 P1 12.00 use
 PLW1 23.00000000 W

F2 - Processing parameters
 SI 65536
 SF 600.1300110 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00





(Z)-2d



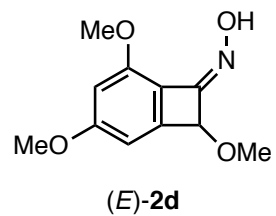
Current Data Parameters
 NAME rs3-166-1
 EXPNO 21
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20150421
 Time_ 17.18
 INSTRUM spect
 PROBHD 5 mm CPPBBO BB
 PULPROG zgpg30
 TD 65536
 SOLVENT Acetone
 NS 128
 DS 4
 SWH 36057.691 Hz
 FIDRES 0.550197 Hz
 AQ 0.9087659 sec
 RG 175.56
 DW 13.867 use
 DE 18.00 use
 TE 300.0 K
 D1 2.0000000 sec
 D11 0.0300000 sec
 TD0 1

==== CHANNEL f1 =====
 SFO1 150.9178981 MHz
 NUC1 13C
 P1 10.00 use
 PLW1 70.0000000 W

==== CHANNEL f2 =====
 SFO2 600.1324005 MHz
 NUC2 1H
 CPDPRG[2] waltz16
 PCPD2 70.00 use
 PLW2 26.0000000 W
 PLW12 0.76407999 W
 PLW13 0.37439999 W

F2 - Processing parameters
 SI 32768
 SF 150.9026714 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

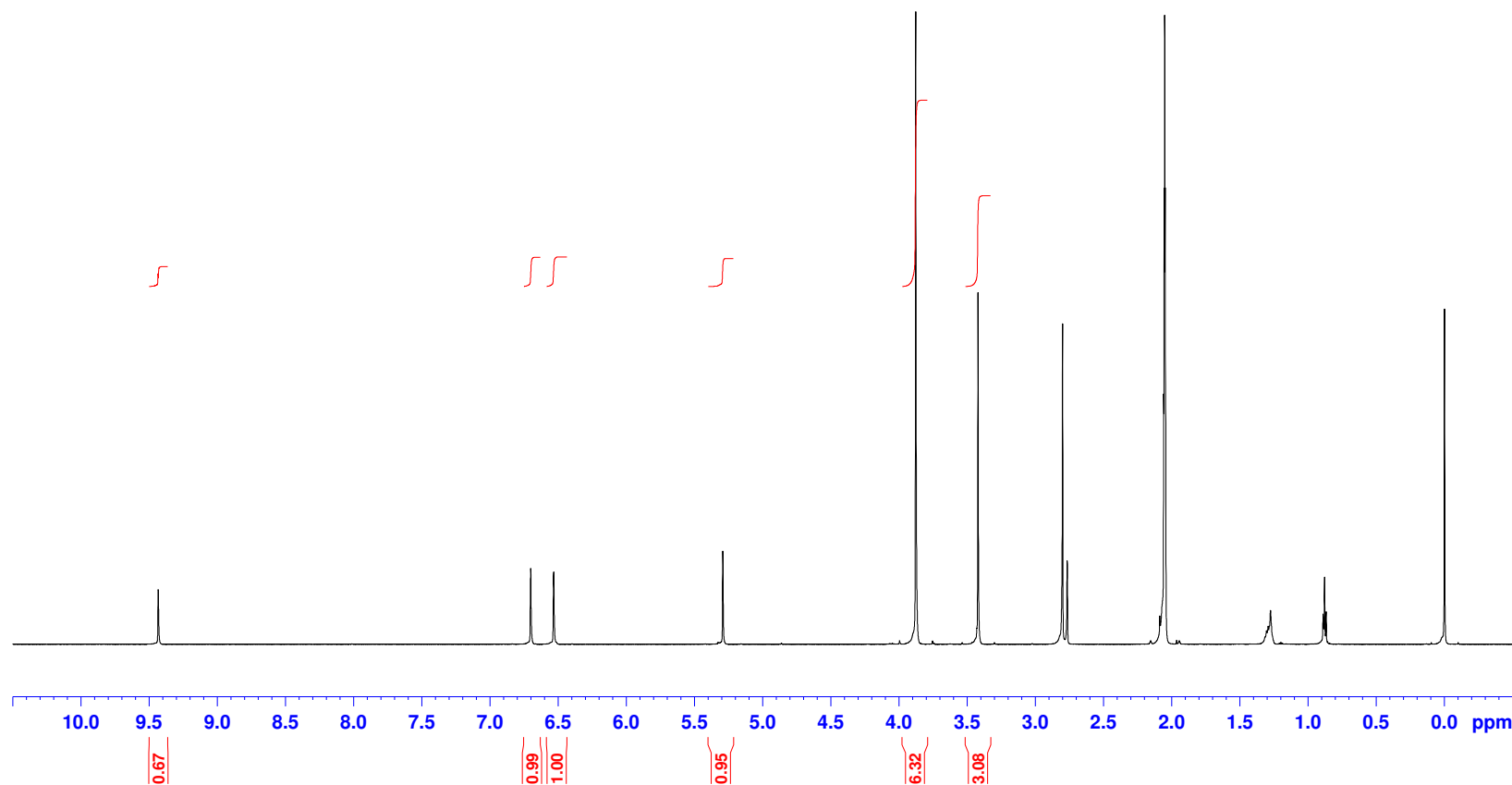


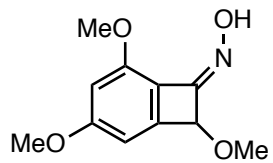
Current Data Parameters
 NAME rs3-166-2
 EXPNO 11
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20150414
 Time_ 20.59
 INSTRUM spect
 PROBHD 5 mm CPPBBO BB
 PULPROG zg30
 TD 65536
 SOLVENT Acetone
 NS 16
 DS 2
 SWH 12019.230 Hz
 FIDRES 0.183399 Hz
 AQ 2.7262976 sec
 RG 31.94
 DW 41.600 use
 DE 10.00 use
 TE 300.0 K
 D1 1.00000000 sec
 TD0 1

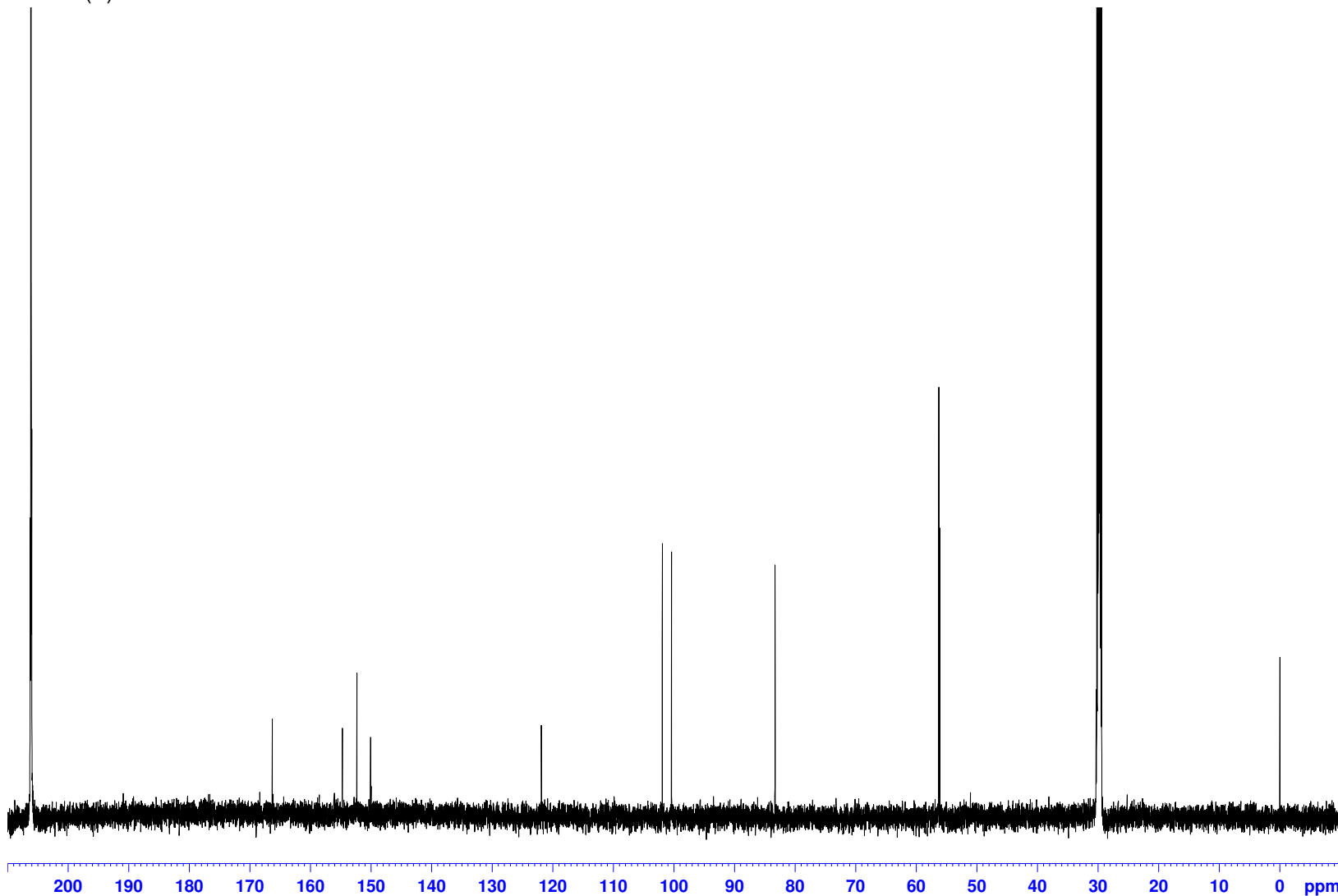
==== CHANNEL f1 =====
 SFO1 600.1337060 MHz
 NUC1 1H
 P1 12.00 use
 PLW1 23.00000000 W

F2 - Processing parameters
 SI 65536
 SF 600.1300110 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00





(E)-2d



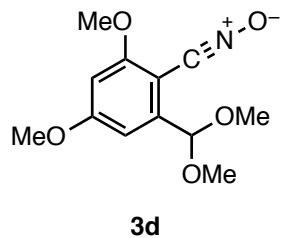
Current Data Parameters
 NAME rs3-166-2 dimethox
 EXPNO 21
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20161227
 Time 3.44
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT Acetone
 NS 2048
 DS 4
 SWH 36057.691 Hz
 FIDRES 0.550197 Hz
 AQ 0.9087659 sec
 RG 175.56
 DW 13.867 use
 DE 6.50 use
 TE 298.0 K
 D1 2.0000000 sec
 D11 0.0300000 sec
 TD0 1

==== CHANNEL f1 =====
 SFO1 150.9178981 MHz
 NUC1 13C
 P1 11.00 use
 PLW1 93.0000000 W

==== CHANNEL f2 =====
 SFO2 600.1324005 MHz
 NUC2 1H
 CPDPRG[2] waltz16
 PCPD2 70.00 use
 PLW2 22.0000000 W
 PLW12 0.64652997 W
 PLW13 0.31680000 W

F2 - Processing parameters
 SI 32768
 SF 150.9026725 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

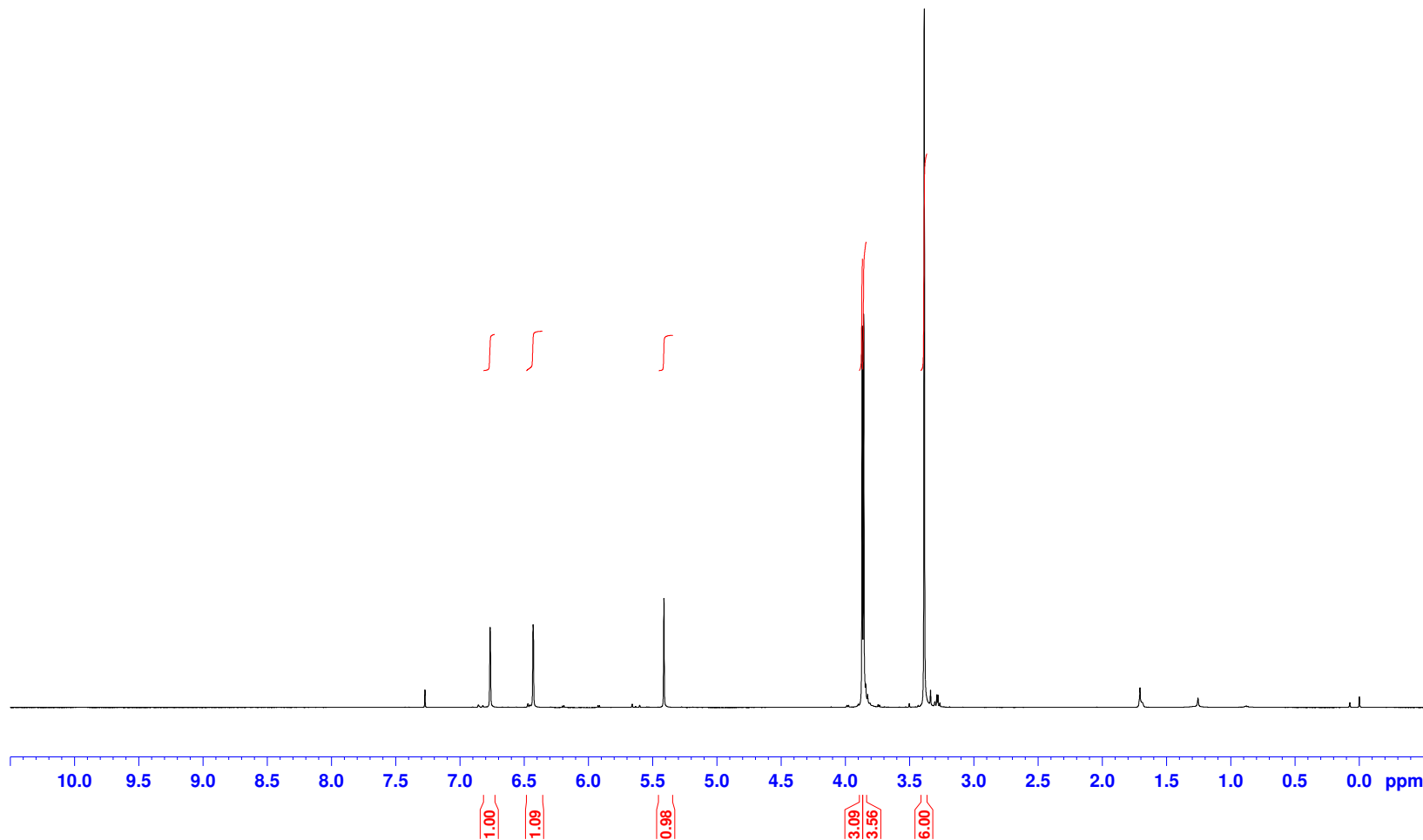


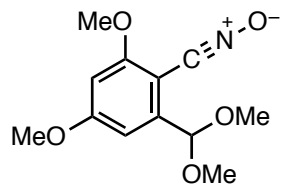
Current Data Parameters
 NAME rs3-170-1
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20150430
 Time_ 23.15
 INSTRUM spect
 PROBHD 5 mm CPPBBO BB
 PULPROG zg30
 TD 65536
 SOLVENT CDC13
 NS 16
 DS 2
 SWH 12019.230 Hz
 FIDRES 0.183399 Hz
 AQ 2.7262976 sec
 RG 17.5
 DW 41.600 use
 DE 10.00 use
 TE 300.0 K
 D1 1.00000000 sec
 TD0 1

==== CHANNEL f1 =====
 SFO1 600.1337060 MHz
 NUC1 1H
 P1 12.00 use
 PLW1 23.00000000 W

F2 - Processing parameters
 SI 65536
 SF 600.1300089 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00





3d



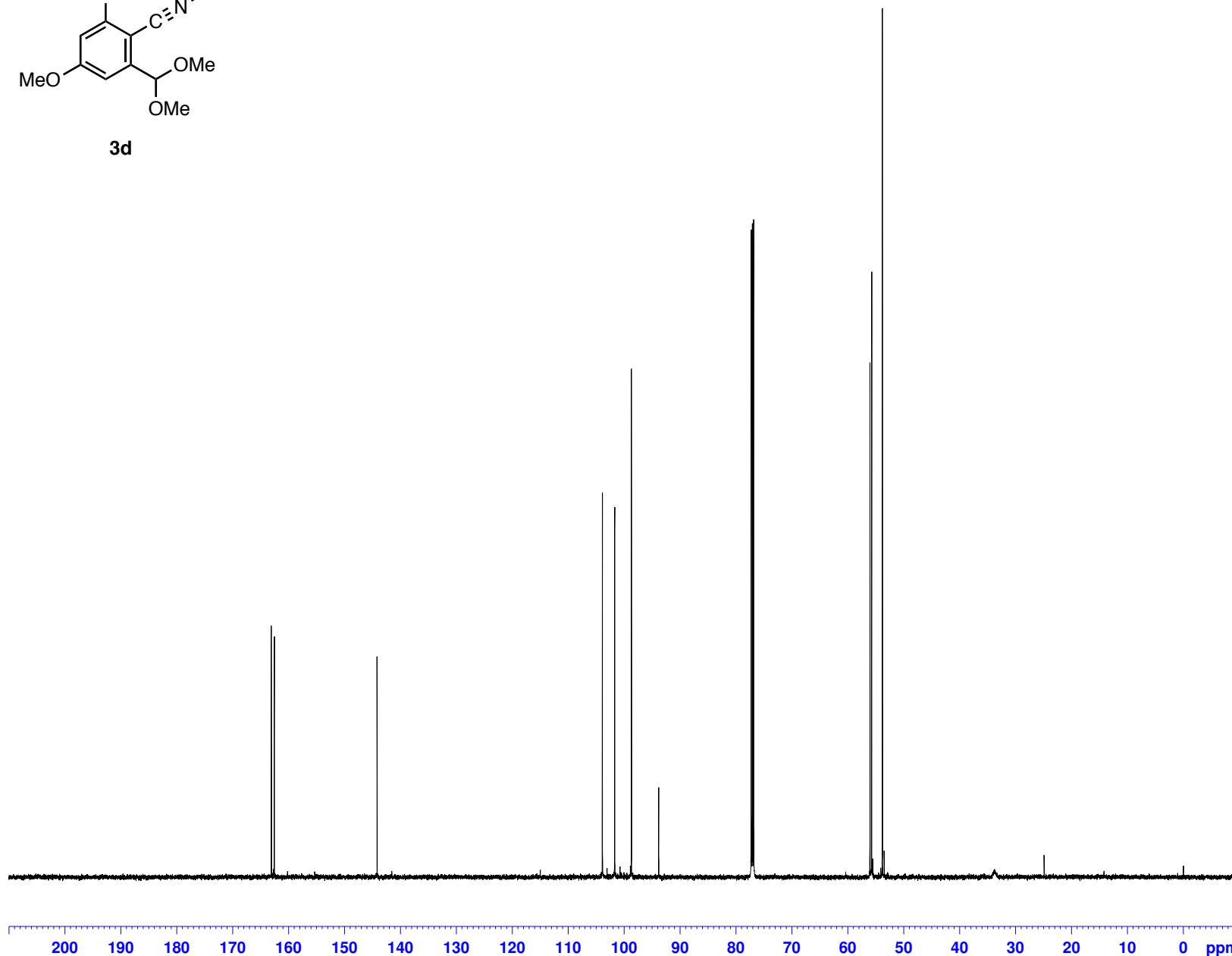
Current Data Parameters
 NAME rs3-170-1
 EXPNO 21
 PROCNO 1

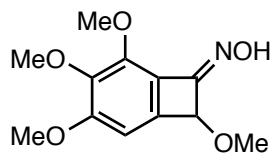
F2 - Acquisition Parameters
 Date_ 20150516
 Time_ 19.39
 INSTRUM spect
 PROBHD 5 mm CPPBBO BB
 PULPROG zgpg30
 TD 65536
 SOLVENT CDC13
 NS 128
 DS 4
 SWH 36057.691 Hz
 FIDRES 0.550197 Hz
 AQ 0.9087659 sec
 RG 175.56
 DW 13.867 use
 DE 18.00 use
 TE 300.0 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TD0 1

==== CHANNEL f1 =====
 SFO1 150.9178981 MHz
 NUC1 13C
 P1 10.00 use
 PLW1 70.00000000 W

==== CHANNEL f2 =====
 SFO2 600.1324005 MHz
 NUC2 1H
 CPDPRG[2] waltz16
 PCPD2 70.00 use
 PLW2 26.00000000 W
 PLW12 0.76407999 W
 PLW13 0.37439999 W

F2 - Processing parameters
 SI 32768
 SF 150.9028056 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40





2e

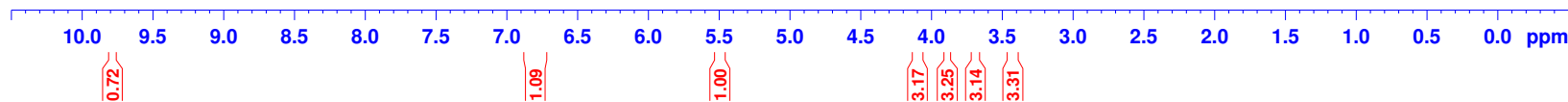
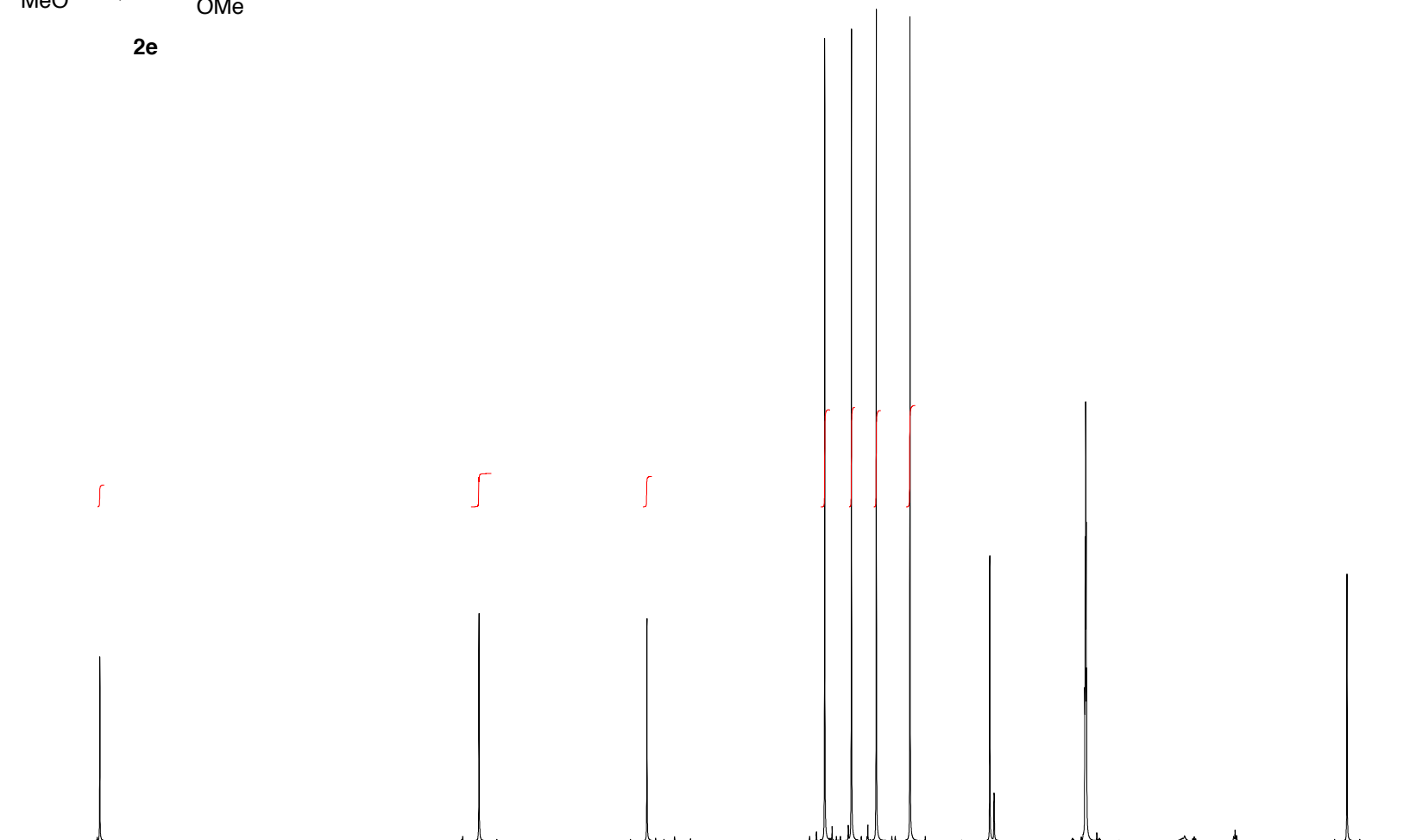


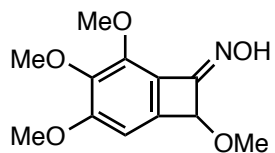
Current Data Parameters
 NAME rs3-108-1
 EXPNO 20
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20150411
 Time_ 0.10
 INSTRUM spect
 PROBHD 5 mm CPPBBO BB
 PULPROG zg30
 TD 65536
 SOLVENT Acetone
 NS 16
 DS 2
 SWH 12019.230 Hz
 FIDRES 0.183399 Hz
 AQ 2.7262976 sec
 RG 31.94
 DW 41.600 use
 DE 10.00 use
 TE 300.0 K
 D1 1.0000000 sec
 TD0 1

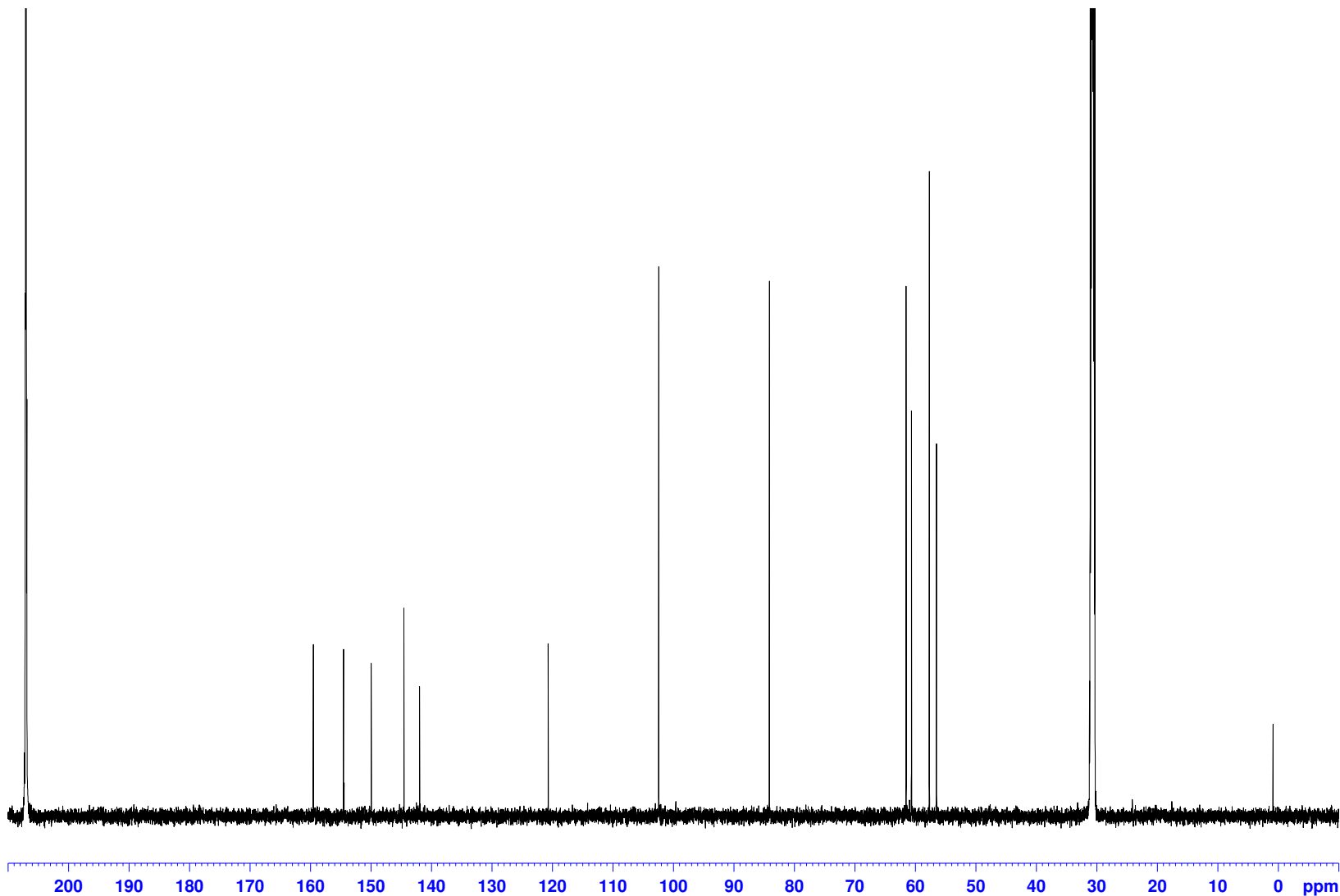
==== CHANNEL f1 =====
 SFO1 600.1337060 MHz
 NUC1 1H
 P1 12.00 use
 PLW1 23.0000000 W

F2 - Processing parameters
 SI 65536
 SF 600.1300109 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00





2e



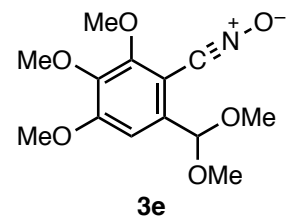
Current Data Parameters
 NAME rs3-108-1
 EXPNO 21
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20150411
 Time_ 7.53
 INSTRUM spect
 PROBHD 5 mm CPPBBO BB
 PULPROG zgpg30
 TD 65536
 SOLVENT Acetone
 NS 1024
 DS 4
 SWH 36057.691 Hz
 FIDRES 0.550197 Hz
 AQ 0.9087659 sec
 RG 175.56
 DW 13.867 use
 DE 18.00 use
 TE 300.0 K
 D1 2.0000000 sec
 D11 0.0300000 sec
 TD0 1

==== CHANNEL f1 =====
 SFO1 150.9178981 MHz
 NUC1 13C
 P1 10.00 use
 PLW1 70.0000000 W

==== CHANNEL f2 =====
 SFO2 600.1324005 MHz
 NUC2 1H
 CPDPRG[2] waltz16
 PCPD2 70.00 use
 PLW2 26.0000000 W
 PLW12 0.76407999 W
 PLW13 0.37439999 W

F2 - Processing parameters
 SI 32768
 SF 150.9025361 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

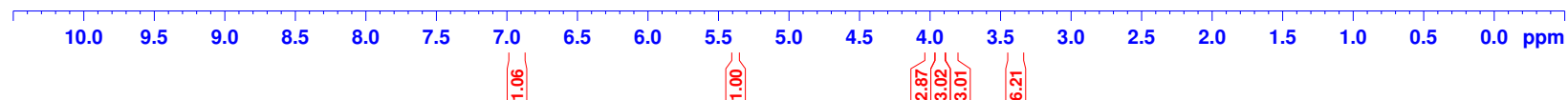
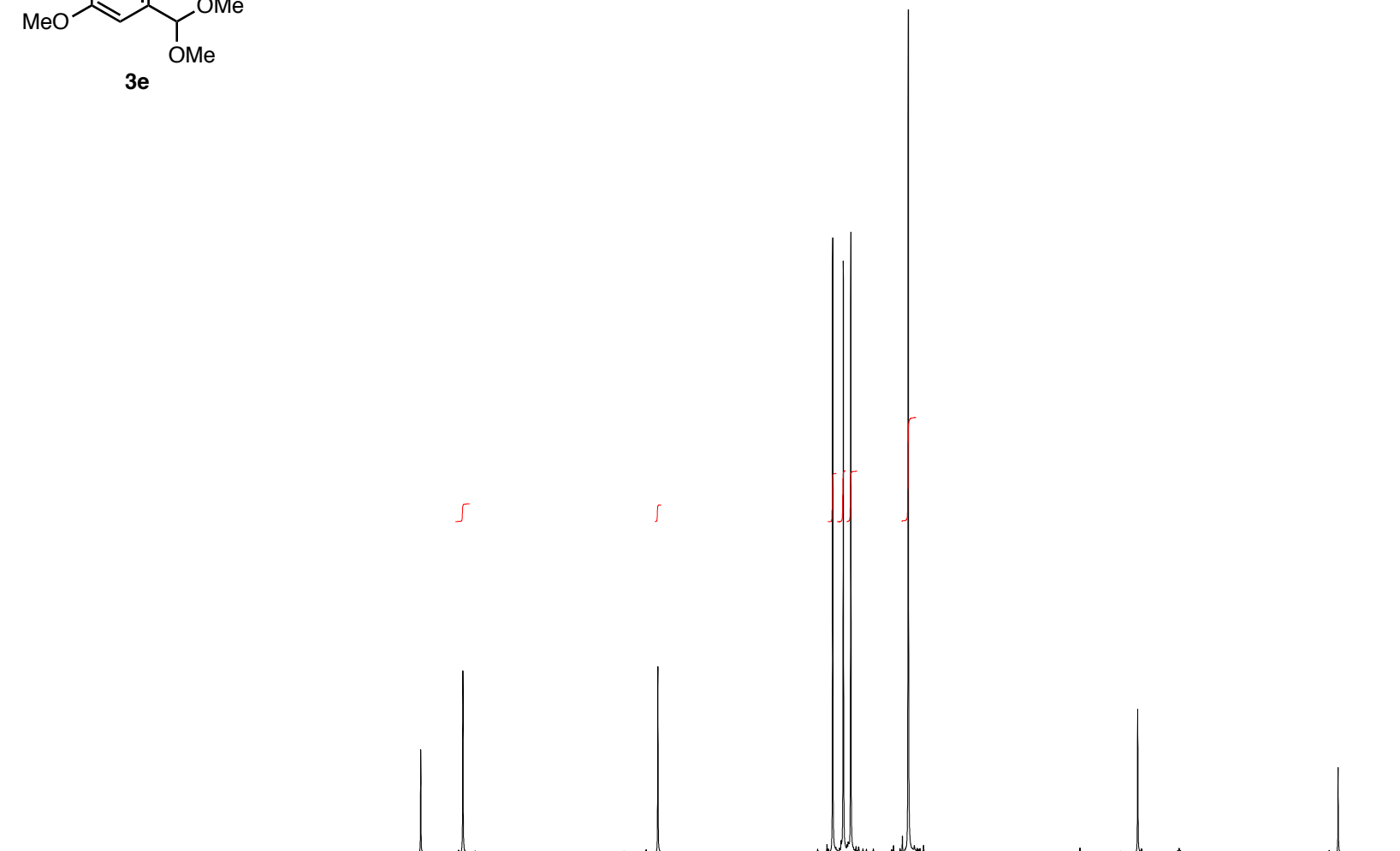


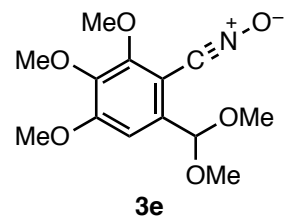
Current Data Parameters
 NAME tri
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20150407
 Time_ 23.14
 INSTRUM spect
 PROBHD 5 mm CPPBBO BB
 PULPROG zg30
 TD 65536
 SOLVENT CDC13
 NS 16
 DS 2
 SWH 12019.230 Hz
 FIDRES 0.183399 Hz
 AQ 2.7262976 sec
 RG 31.94
 DW 41.600 use
 DE 10.00 use
 TE 300.0 K
 D1 1.00000000 sec
 TD0 1

==== CHANNEL f1 =====
 SFO1 600.1337060 MHz
 NUC1 1H
 P1 12.00 use
 PLW1 23.00000000 W

F2 - Processing parameters
 SI 65536
 SF 600.1300161 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00





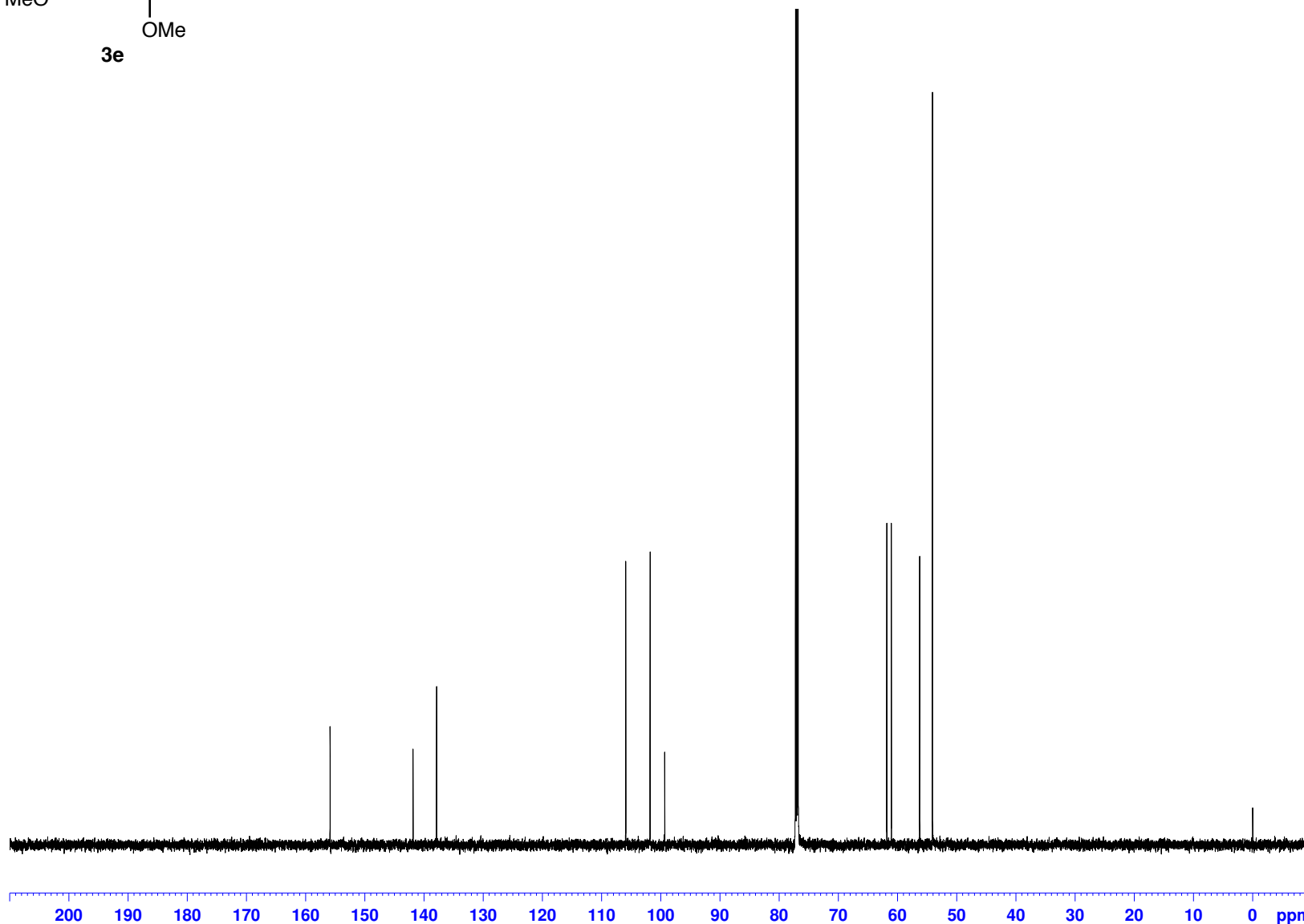
Current Data Parameters
 NAME tri
 EXPNO 11
 PROCNO 1

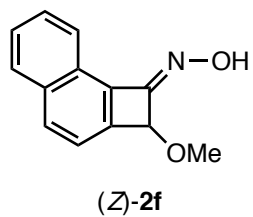
F2 - Acquisition Parameters
 Date_ 20150407
 Time_ 23.31
 INSTRUM spect
 PROBHD 5 mm CPPBBO BB
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 128
 DS 4
 SWH 36057.691 Hz
 FIDRES 0.550197 Hz
 AQ 0.9087659 sec
 RG 175.56
 DW 13.867 use
 DE 18.00 use
 TE 300.0 K
 D1 2.0000000 sec
 D11 0.0300000 sec
 TD0 1

==== CHANNEL f1 =====
 SFO1 150.9178981 MHz
 NUC1 13C
 P1 10.00 use
 PLW1 70.0000000 W

==== CHANNEL f2 =====
 SFO2 600.1324005 MHz
 NUC2 1H
 CPDPRG[2] waltz16
 PCPD2 70.00 use
 PLW2 26.0000000 W
 PLW12 0.76407999 W
 PLW13 0.37439999 W

F2 - Processing parameters
 SI 32768
 SF 150.9028090 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40



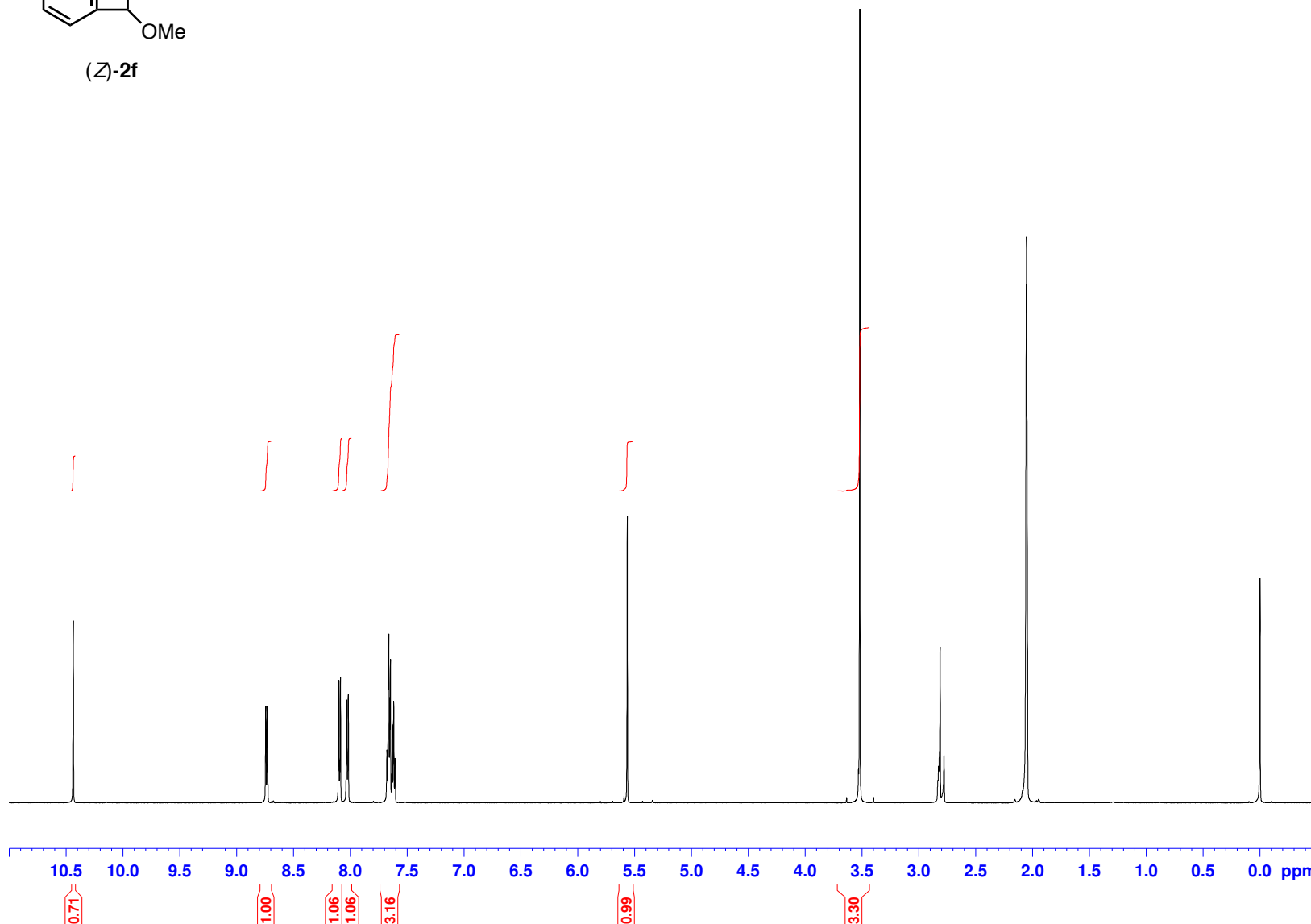


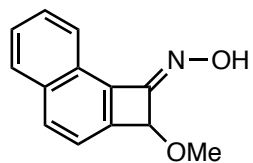
Current Data Parameters
 NAME naphtho oxine-Z
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20170119
 Time_ 13.25
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT Acetone
 NS 16
 DS 2
 SWH 12019.230 Hz
 FIDRES 0.183399 Hz
 AQ 2.7262976 sec
 RG 114
 DW 41.600 use
 DE 6.50 use
 TE 298.0 K
 D1 1.00000000 sec
 TD0 1

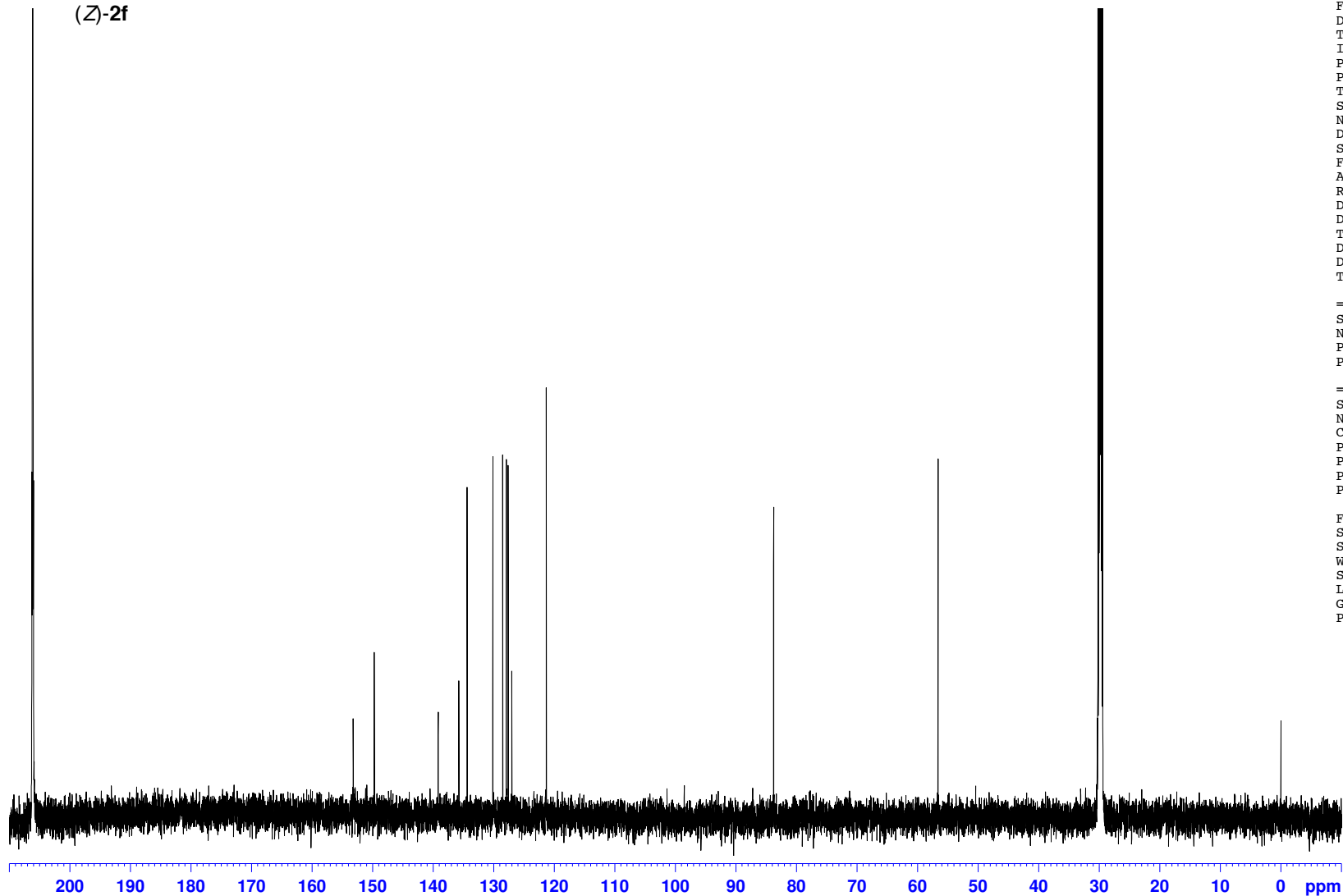
==== CHANNEL f1 =====
 SFO1 600.1337060 MHz
 NUC1 1H
 P1 12.00 use
 PLW1 22.00000000 W

F2 - Processing parameters
 SI 65536
 SF 600.1300113 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00





(Z)-2f



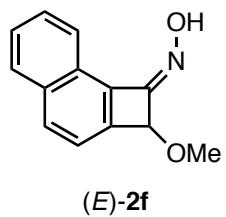
Current Data Parameters
 NAME naphtho oxine-Z
 EXPNO 11
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20170119
 Time_ 13.23
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT Acetone
 NS 1024
 DS 4
 SWH 36057.691 Hz
 FIDRES 0.550197 Hz
 AQ 0.9087659 sec
 RG 175.56
 DW 13.867 use
 DE 6.50 use
 TE 298.0 K
 D1 2.0000000 sec
 D11 0.0300000 sec
 TD0 1

==== CHANNEL f1 =====
 SFO1 150.9178981 MHz
 NUC1 13C
 P1 11.00 use
 PLW1 93.0000000 W

==== CHANNEL f2 =====
 SFO2 600.1324005 MHz
 NUC2 1H
 CPDPRG[2] waltz16
 PCPD2 70.00 use
 PLW2 22.0000000 W
 PLW12 0.64652997 W
 PLW13 0.31680000 W

F2 - Processing parameters
 SI 32768
 SF 150.9026725 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

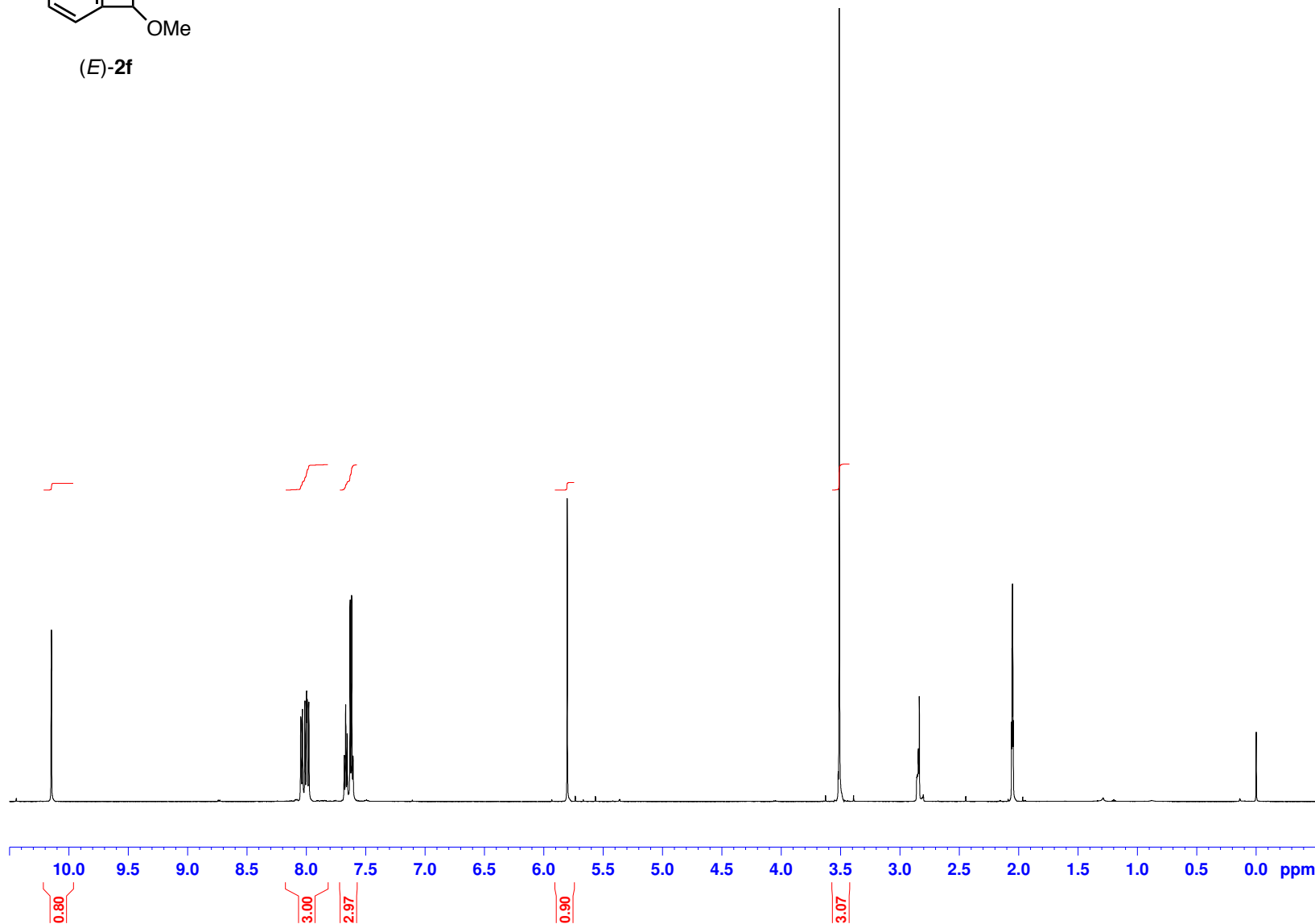


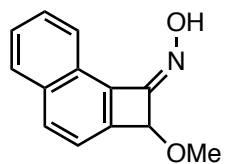
Current Data Parameters
 NAME rs3-441-3
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20161129
 Time_ 20.09
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT Acetone
 NS 16
 DS 2
 SWH 12019.230 Hz
 FIDRES 0.183399 Hz
 AQ 2.7262976 sec
 RG 83.7
 DW 41.600 use
 DE 6.50 use
 TE 298.0 K
 D1 1.0000000 sec
 TD0 1

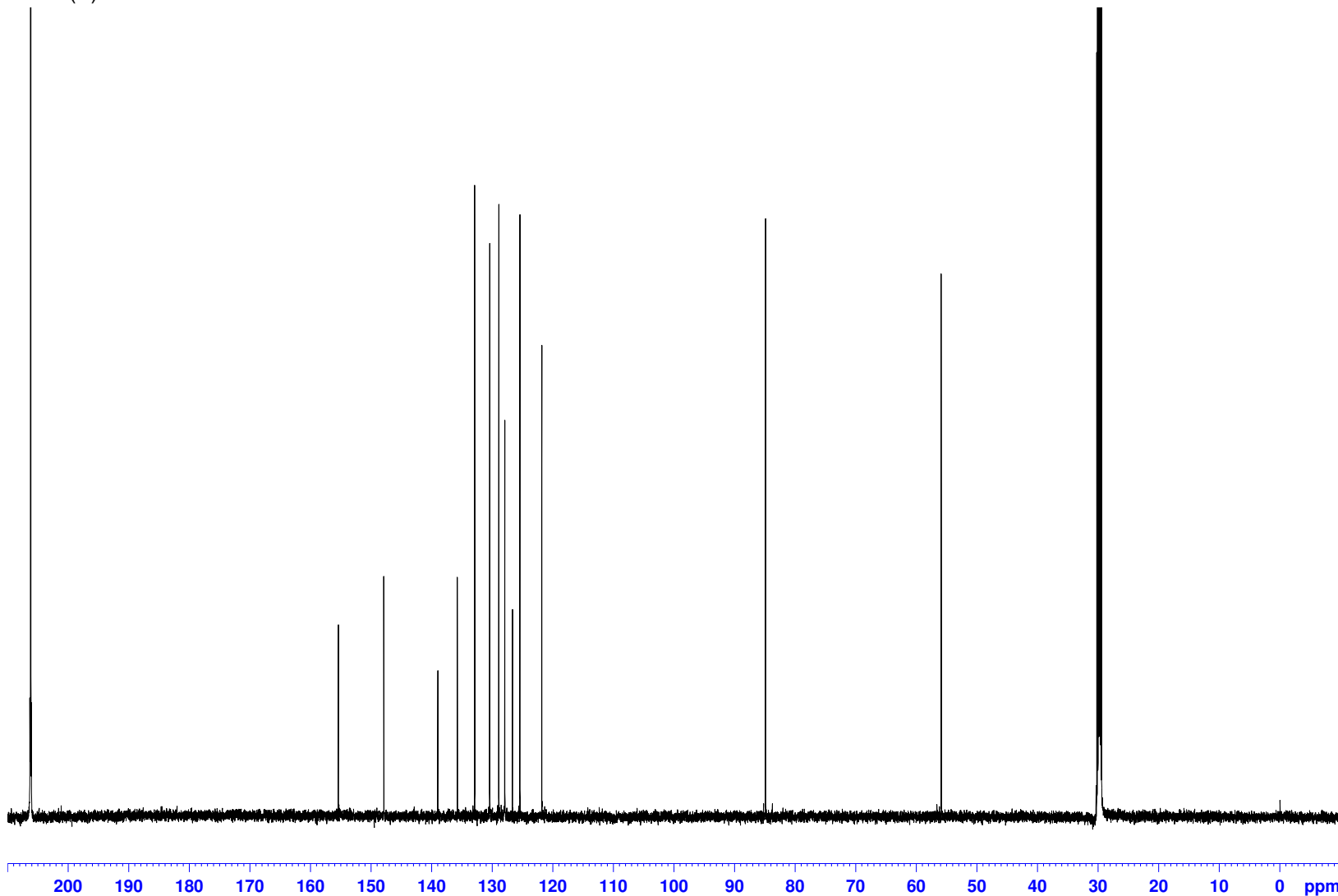
==== CHANNEL f1 =====
 SFO1 600.1337060 MHz
 NUC1 1H
 P1 12.00 use
 PLW1 22.0000000 W

F2 - Processing parameters
 SI 65536
 SF 600.1300111 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00





(E)-2f



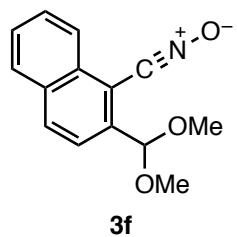
Current Data Parameters
 NAME rs3-441-3
 EXPNO 20
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20161202
 Time 0.54
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT Acetone
 NS 1024
 DS 4
 SWH 36057.691 Hz
 FIDRES 0.550197 Hz
 AQ 0.9087659 sec
 RG 175.56
 DW 13.867 use
 DE 6.50 use
 TE 298.0 K
 D1 2.0000000 sec
 D11 0.0300000 sec
 TD0 1

==== CHANNEL f1 =====
 SFO1 150.9178981 MHz
 NUC1 13C
 P1 11.00 use
 PLW1 93.0000000 W

==== CHANNEL f2 =====
 SFO2 600.1324005 MHz
 NUC2 1H
 CPDPRG[2] waltz16
 PCPD2 70.00 use
 PLW2 22.0000000 W
 PLW12 0.64652997 W
 PLW13 0.31680000 W

F2 - Processing parameters
 SI 32768
 SF 150.9026737 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

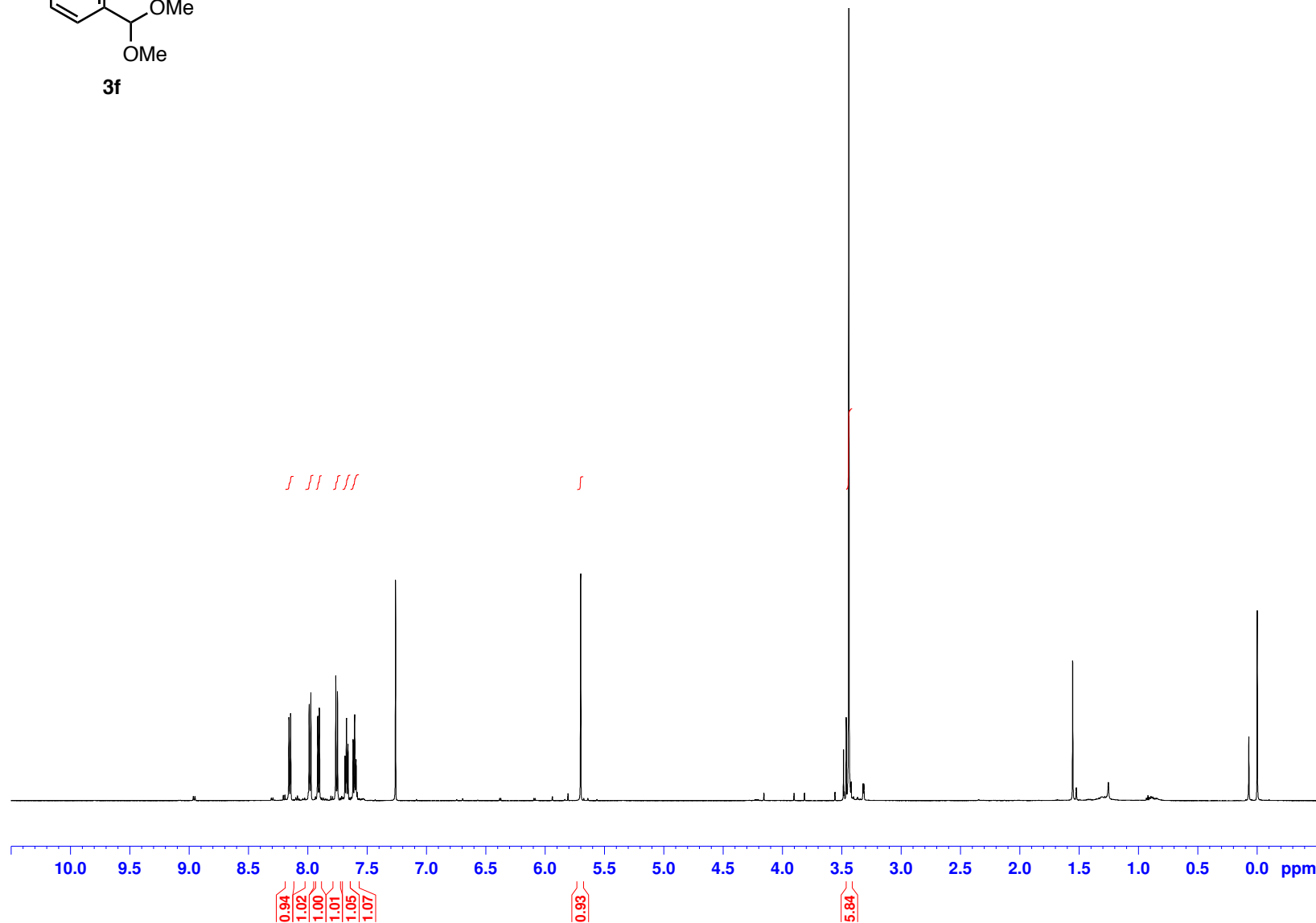


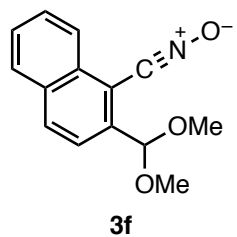
Current Data Parameters
 NAME nitri 1
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20170118
 Time_ 23.34
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDC13
 NS 16
 DS 2
 SWH 12019.230 Hz
 FIDRES 0.183399 Hz
 AQ 2.7262976 sec
 RG 87.68
 DW 41.600 use
 DE 6.50 use
 TE 298.0 K
 D1 1.00000000 sec
 TD0 1

==== CHANNEL f1 =====
 SFO1 600.1337060 MHz
 NUC1 1H
 P1 12.00 use
 PLW1 22.00000000 W

F2 - Processing parameters
 SI 65536
 SF 600.1300183 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00





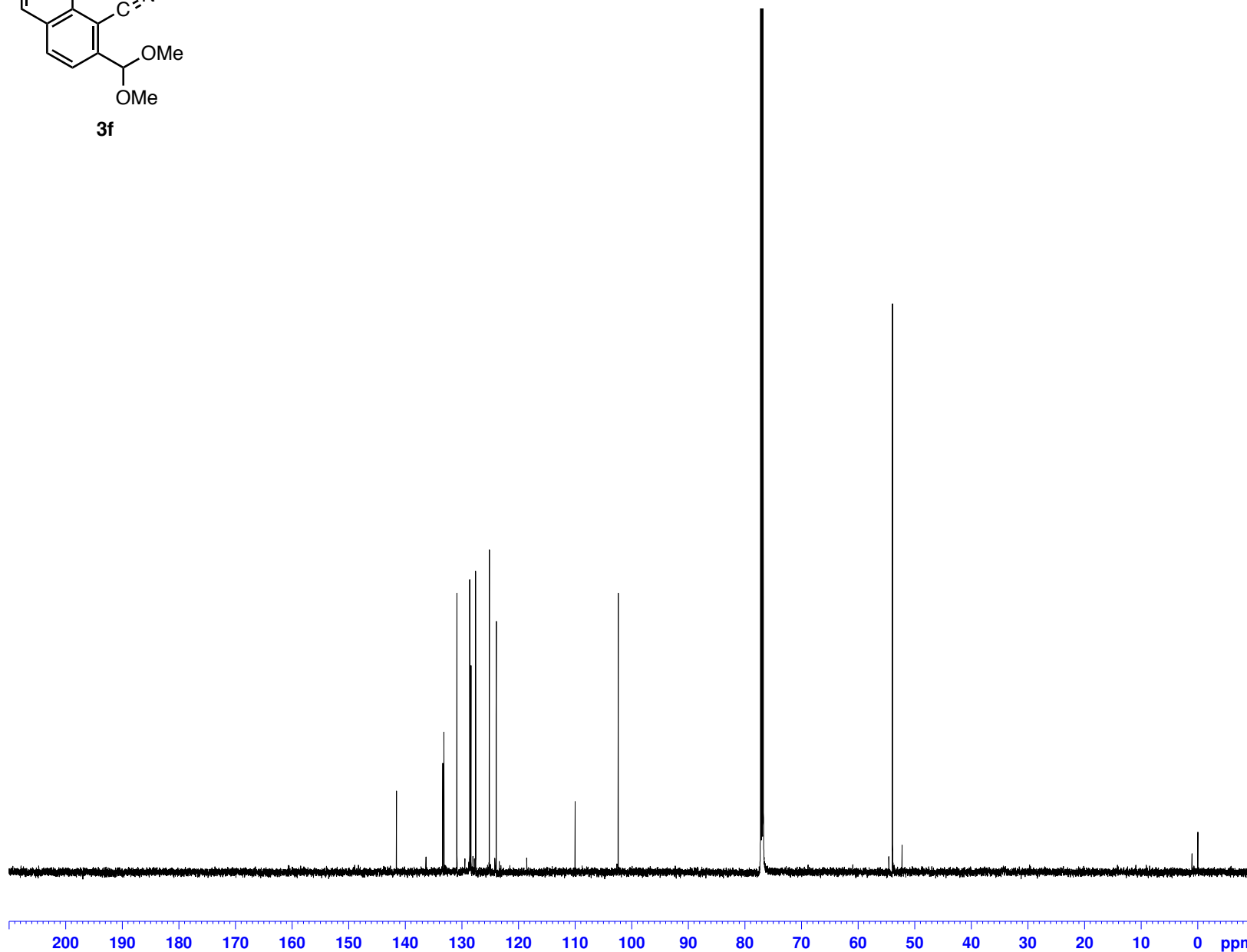
Current Data Parameters
 NAME nitri 1
 EXPNO 11
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20170119
 Time_ 5.56
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDC13
 NS 3500
 DS 4
 SWH 36057.691 Hz
 FIDRES 0.550197 Hz
 AQ 0.9087659 sec
 RG 175.56
 DW 13.867 use
 DE 6.50 use
 TE 298.0 K
 D1 2.0000000 sec
 D11 0.0300000 sec
 TD0 1

==== CHANNEL f1 =====
 SFO1 150.9178981 MHz
 NUC1 13C
 P1 11.00 use
 PLW1 93.0000000 W

==== CHANNEL f2 =====
 SFO2 600.1324005 MHz
 NUC2 1H
 CPDPRG[2] waltz16
 PCPD2 70.00 use
 PLW2 22.0000000 W
 PLW12 0.64652997 W
 PLW13 0.31680000 W

F2 - Processing parameters
 SI 32768
 SF 150.9028096 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40



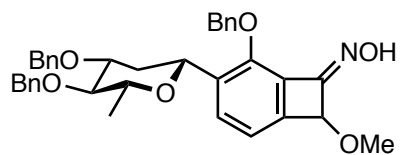


Current Data Parameters
NAME 70-1
EXPNO 10
PROCNO 1

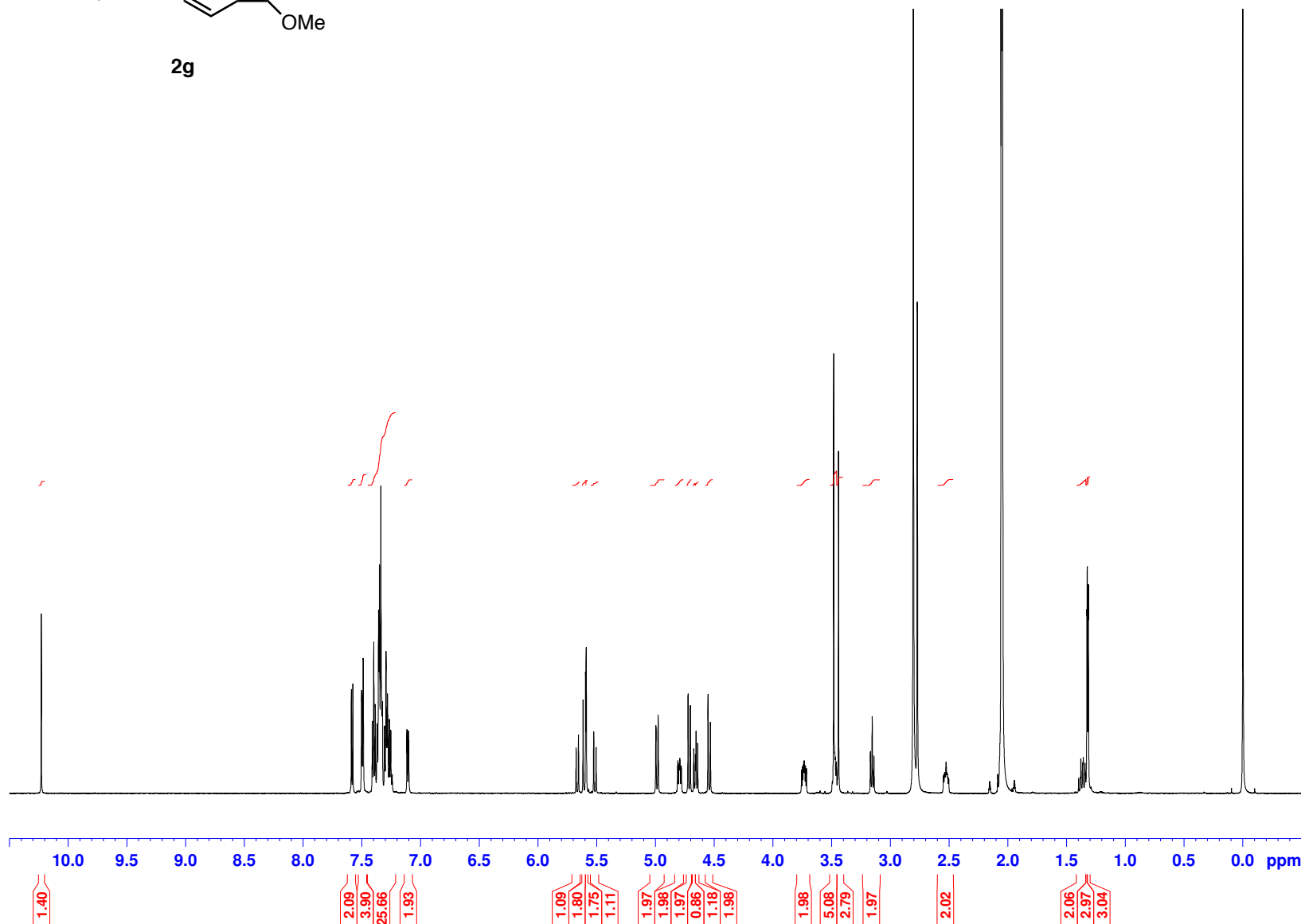
F2 - Acquisition Parameters
Date_ 20150603
Time_ 22.43
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT Acetone
NS 16
DS 2
SWH 12019.230 Hz
FIDRES 0.183399 Hz
AQ 2.7262976 sec
RG 31.94
DW 41.600 use
DE 10.00 use
TE 300.0 K
D1 1.0000000 sec
TD0 1

==== CHANNEL f1 =====
SFO1 600.1337060 MHz
NUC1 1H
P1 12.00 use
PLW1 23.0000000 W

F2 - Processing parameters
SI 65536
SF 600.1300115 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



2g





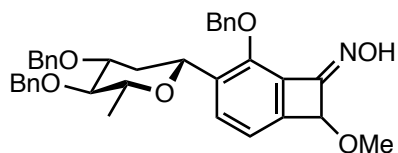
Current Data Parameters
NAME 70-1 13C
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20150604
Time_ 4.22
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zgpg30
TD 65536
SOLVENT Acetone
NS 1024
DS 4
SWH 36057.691 Hz
FIDRES 0.550197 Hz
AQ 0.9087659 sec
RG 175.56
DW 13.867 use
DE 18.00 use
TE 300.0 K
D1 2.0000000 sec
D11 0.0300000 sec
TD0 1

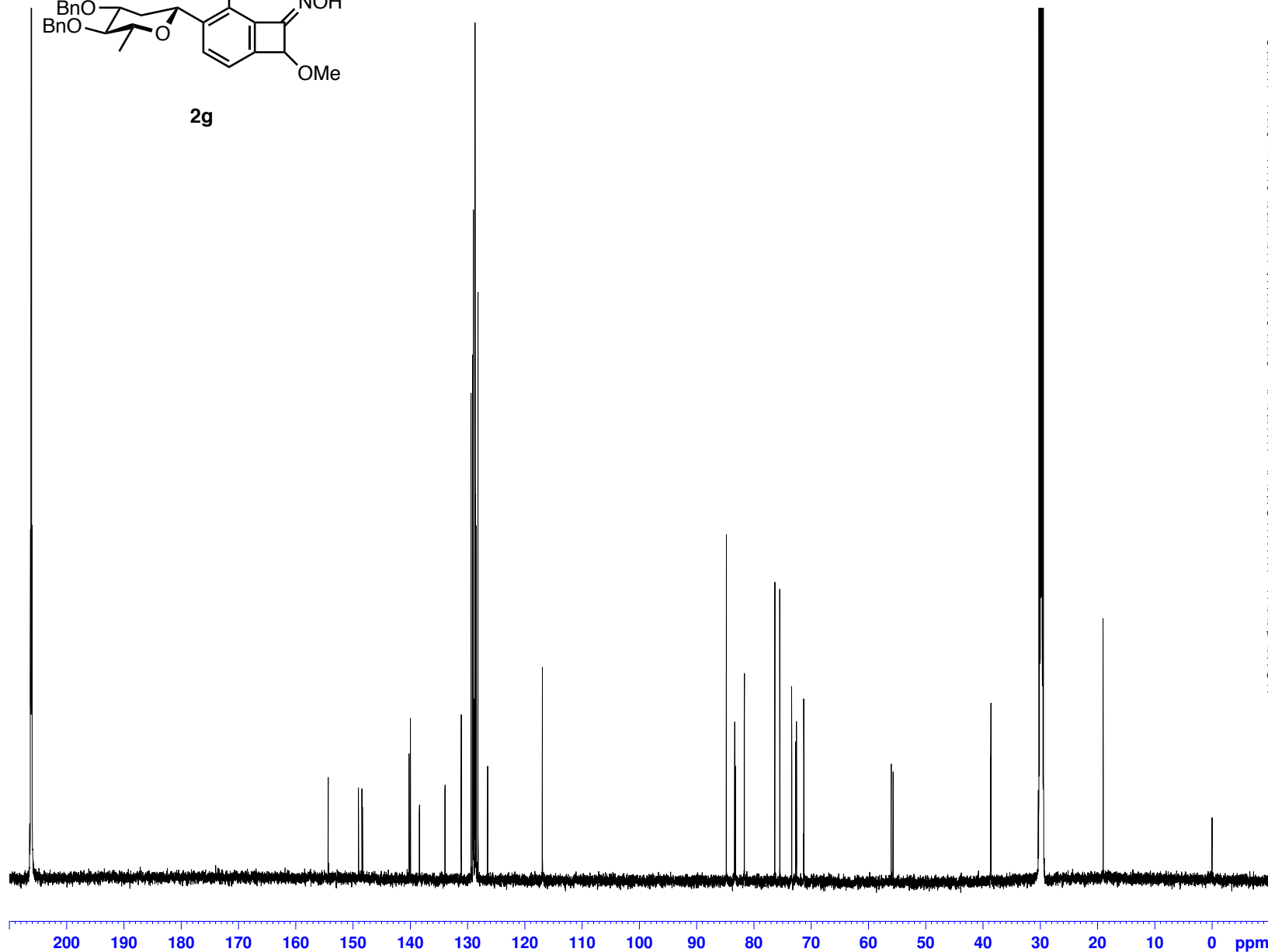
==== CHANNEL f1 =====
SFO1 150.9178981 MHz
NUC1 13C
P1 10.00 use
PLW1 70.0000000 W

==== CHANNEL f2 =====
SFO2 600.1324005 MHz
NUC2 1H
CPDPRG[2] waltz16
PCPD2 70.00 use
PLW2 26.0000000 W
PLW12 0.76407999 W
PLW13 0.37439999 W

F2 - Processing parameters
SI 32768
SF 150.9026725 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



2g



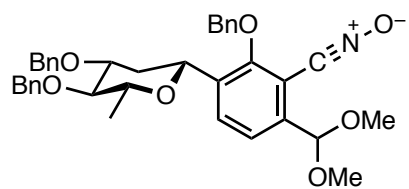


Current Data Parameters
NAME rs3-102-1-2
EXPNO 11
PROCNO 1

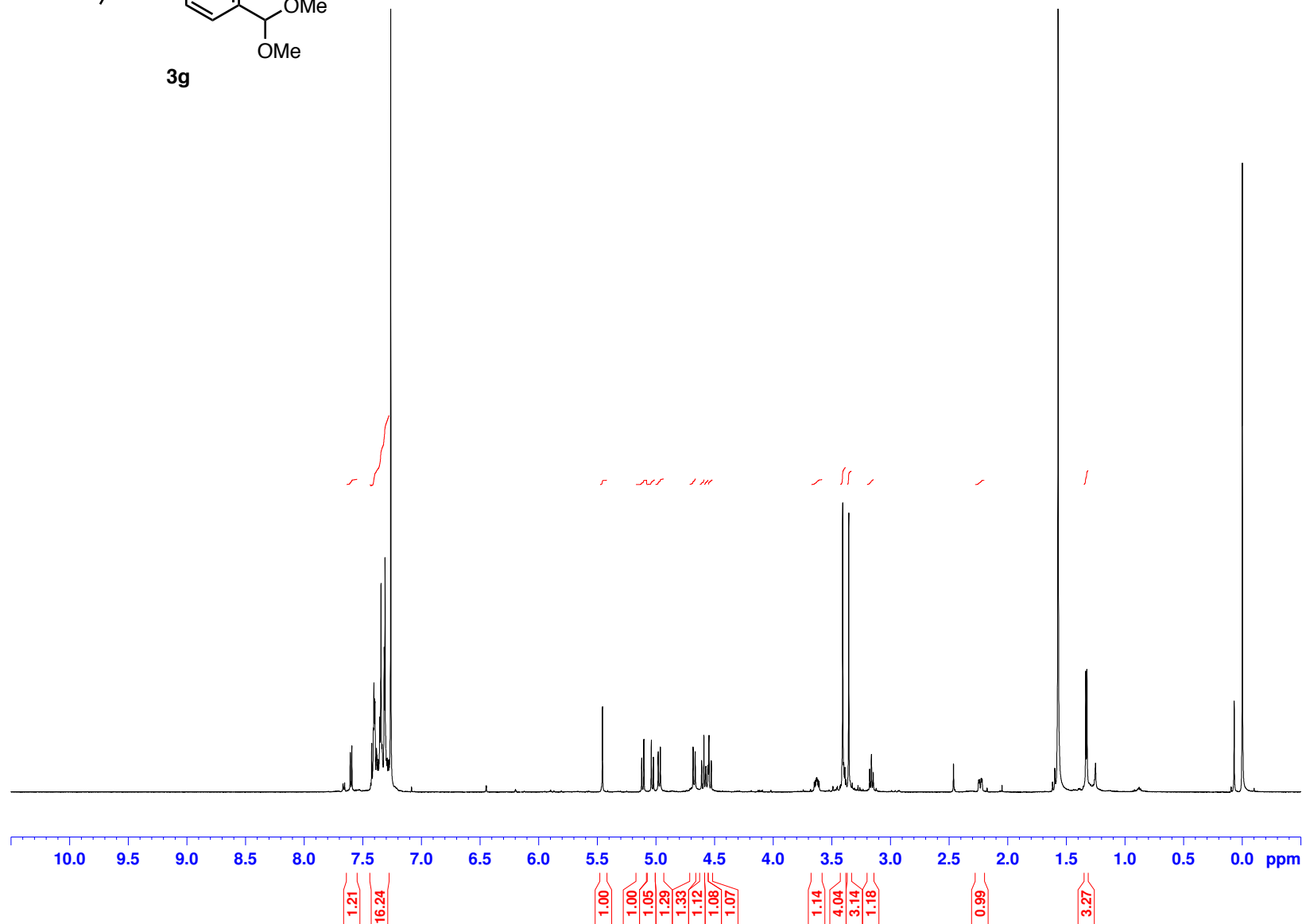
F2 - Acquisition Parameters
Date_ 20150528
Time 18.51
INSTRUM spect
PROBHD 5 mm CPPBBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 12019.230 Hz
FIDRES 0.183399 Hz
AQ 2.7262976 sec
RG 31.94
DW 41.600 use
DE 10.00 use
TE 291.5 K
D1 1.0000000 sec
TD0 1

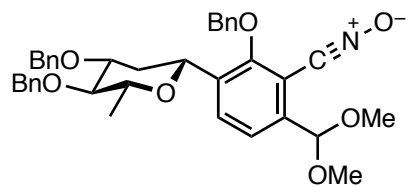
==== CHANNEL f1 =====
SFO1 600.1337060 MHz
NUC1 1H
P1 12.00 use
PLW1 23.0000000 W

F2 - Processing parameters
SI 65536
SF 600.1300142 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



3g





3g



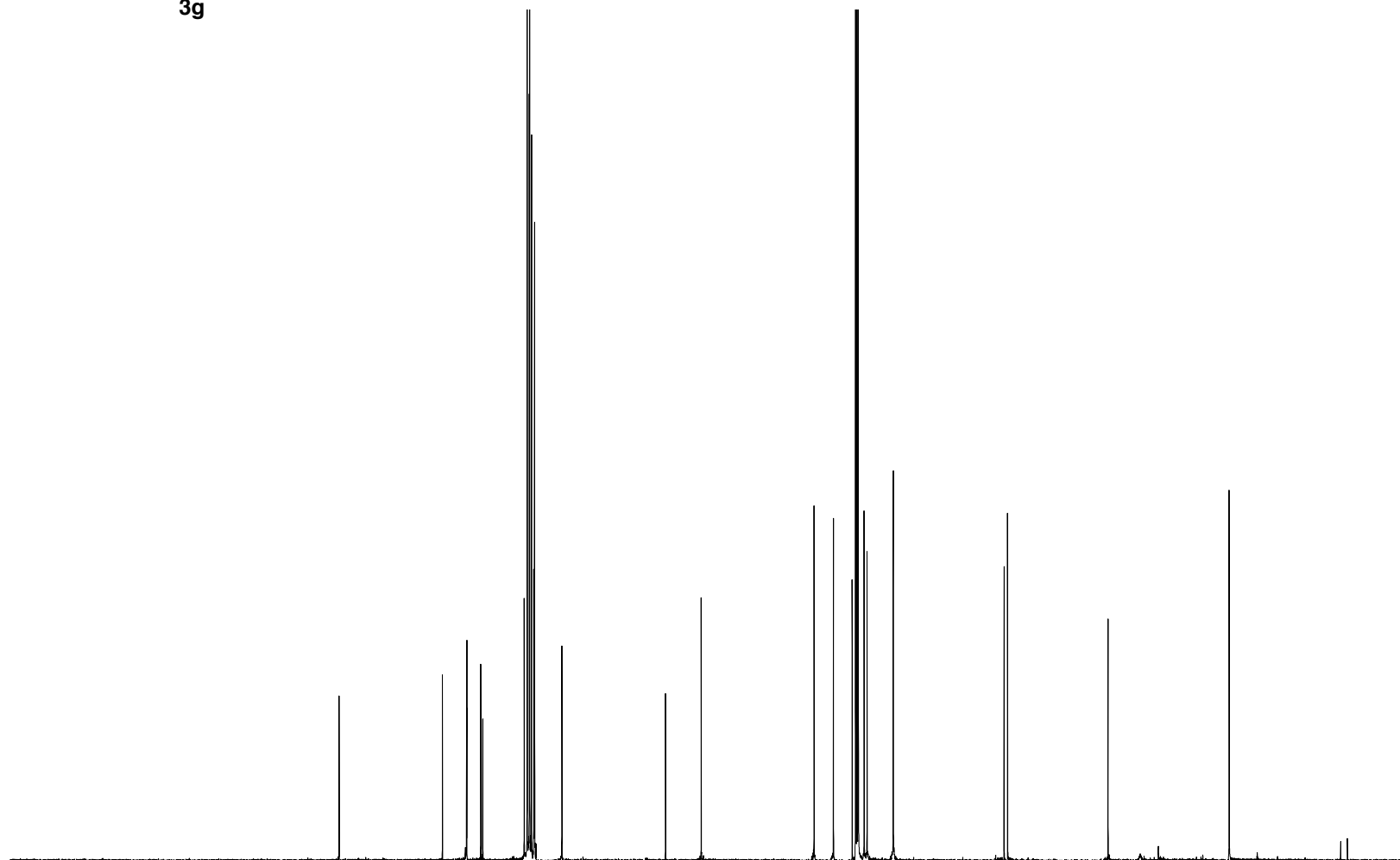
Current Data Parameters
 NAME rs3-102-1
 EXPNO 40
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20150529
 Time_ 4.56
 INSTRUM spect
 PROBHD 5 mm CPPBBO BB
 PULPROG zgpg30
 TD 65536
 SOLVENT CDC13
 NS 1024
 DS 4
 SWH 36057.691 Hz
 FIDRES 0.550197 Hz
 AQ 0.9087659 sec
 RG 175.56
 DW 13.867 use
 DE 18.00 use
 TE 291.5 K
 D1 2.0000000 sec
 D11 0.0300000 sec
 TD0 1

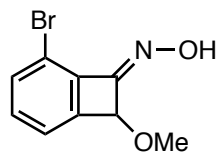
==== CHANNEL f1 =====
 SFO1 150.9178981 MHz
 NUC1 13C
 P1 10.00 use
 PLW1 70.0000000 W

==== CHANNEL f2 =====
 SFO2 600.1324005 MHz
 NUC2 1H
 CPDPRG[2] waltz16
 PCPD2 70.00 use
 PLW2 26.0000000 W
 PLW12 0.76407999 W
 PLW13 0.37439999 W

F2 - Processing parameters
 SI 32768
 SF 150.9028167 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40



200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm



(Z)-2h

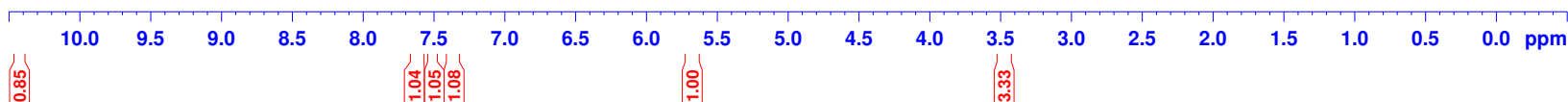
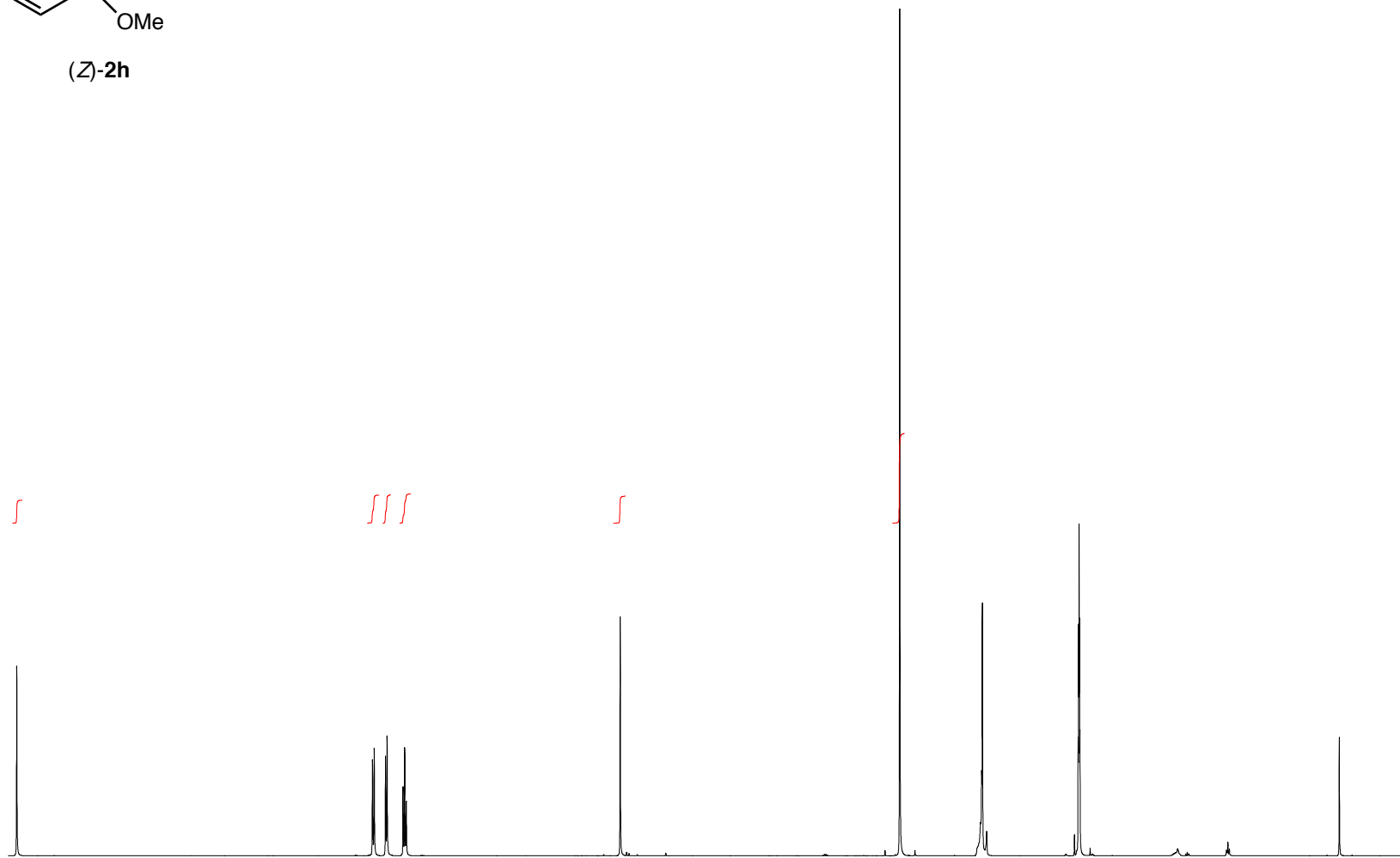


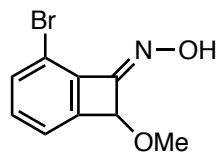
Current Data Parameters
 NAME rs3-148-1
 EXPNO 30
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20150331
 Time_ 21.52
 INSTRUM spect
 PROBHD 5 mm CPPBBO BB
 PULPROG zg30
 TD 65536
 SOLVENT Acetic
 NS 16
 DS 2
 SWH 12019.230 Hz
 FIDRES 0.183399 Hz
 AQ 2.7262976 sec
 RG 31.94
 DW 41.600 use
 DE 10.00 use
 TE 300.0 K
 D1 1.00000000 sec
 TD0 1

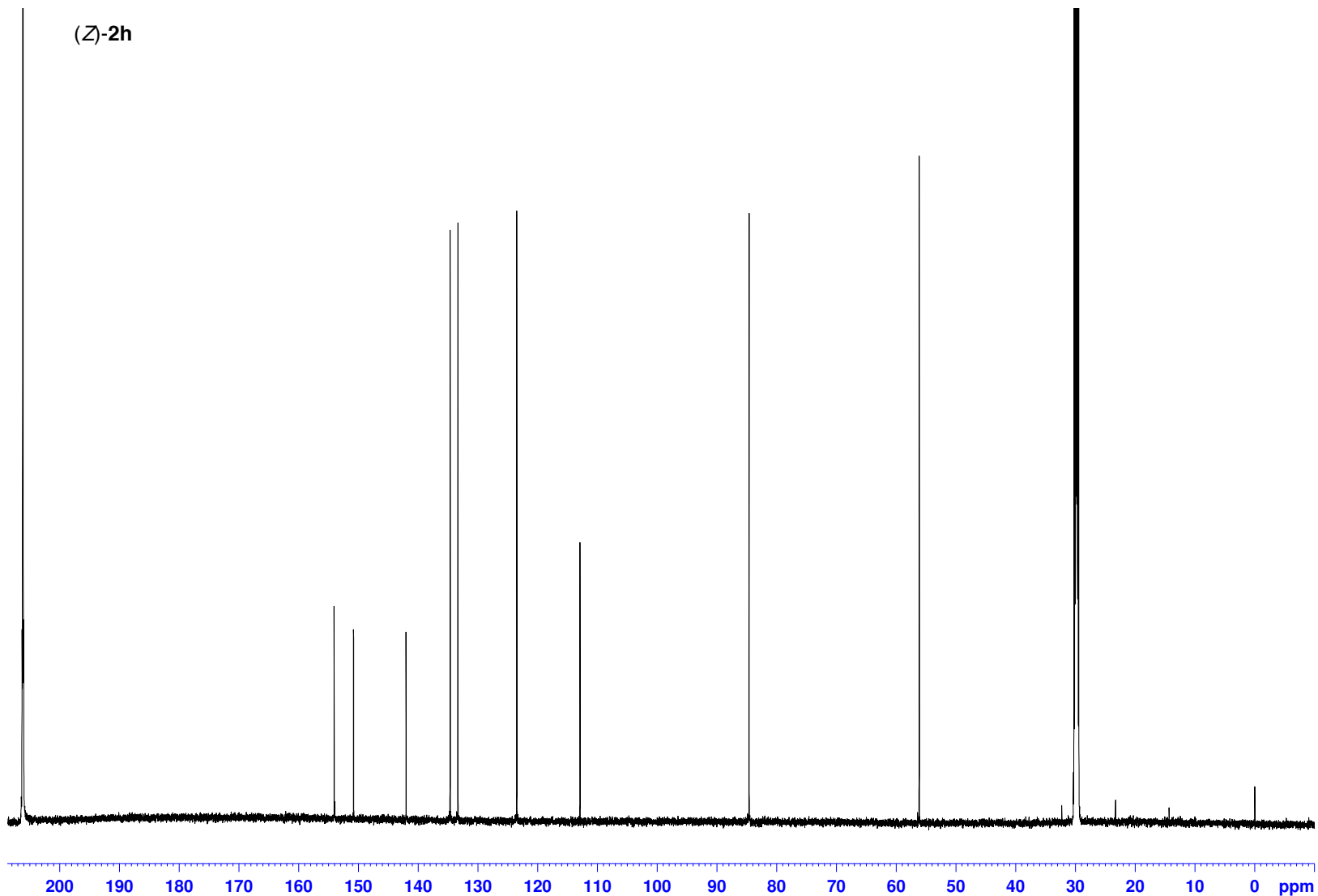
==== CHANNEL f1 =====
 SFO1 600.1337060 MHz
 NUC1 1H
 P1 12.00 use
 PLW1 23.00000000 W

F2 - Processing parameters
 SI 65536
 SF 600.1300045 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00





(Z)-2h



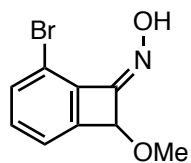
Current Data Parameters
 NAME rs3-148-1
 EXPNO 31
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20150401
 Time_ 4.28
 INSTRUM spect
 PROBHD 5 mm CPPBBO BB
 PULPROG zgpg30
 TD 65536
 SOLVENT Acetic
 NS 1024
 DS 4
 SWH 36057.691 Hz
 FIDRES 0.550197 Hz
 AQ 0.9087659 sec
 RG 175.56
 DW 13.867 use
 DE 18.00 use
 TE 300.0 K
 D1 2.0000000 sec
 D11 0.0300000 sec
 TD0 1

==== CHANNEL f1 =====
 SFO1 150.9178981 MHz
 NUC1 13C
 P1 10.00 use
 PLW1 70.0000000 W

==== CHANNEL f2 =====
 SFO2 600.1324005 MHz
 NUC2 1H
 CPDPRG[2] waltz16
 PCPD2 70.00 use
 PLW2 26.0000000 W
 PLW12 0.76407999 W
 PLW13 0.37439999 W

F2 - Processing parameters
 SI 32768
 SF 150.9026714 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40



(E)-2h

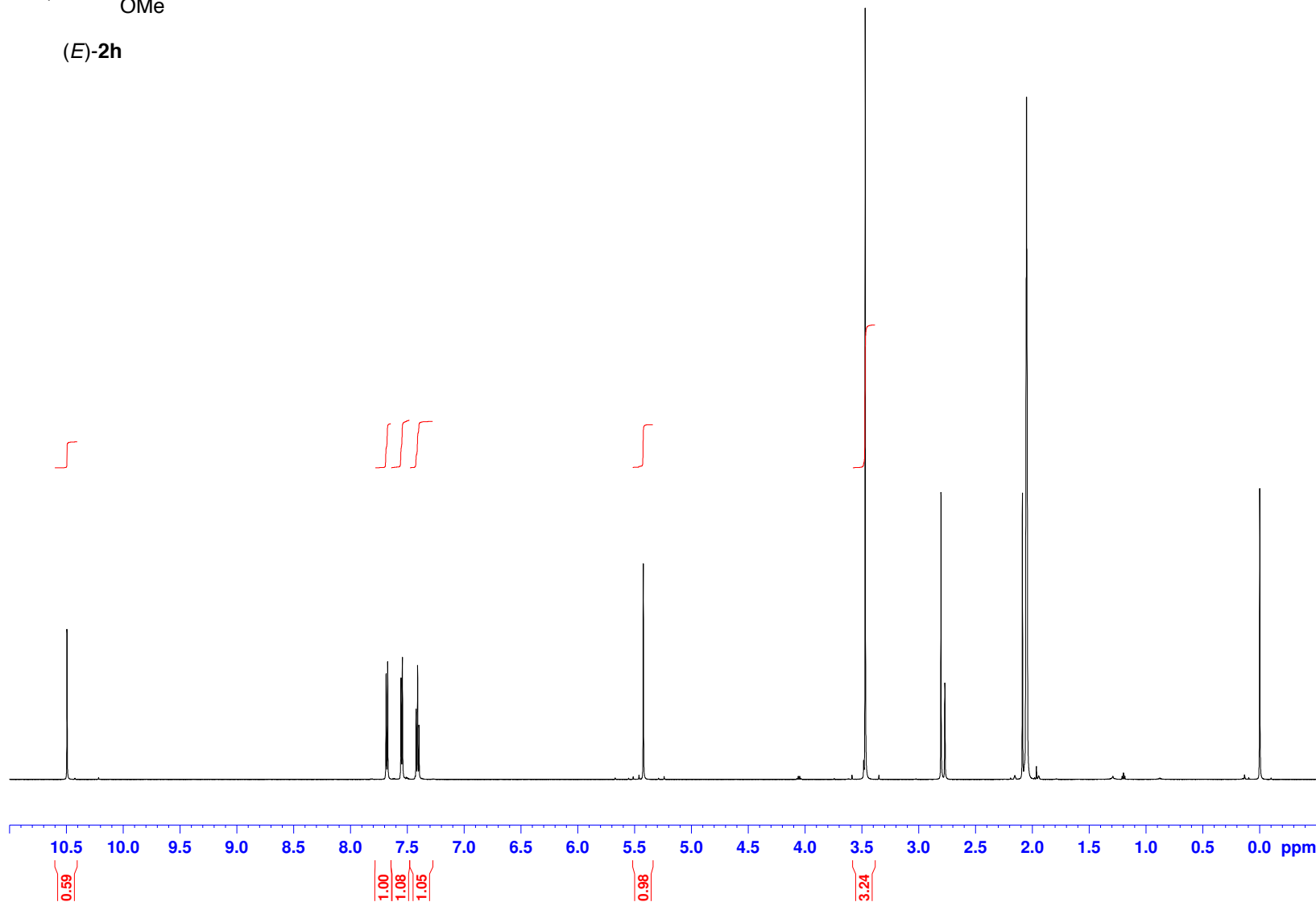


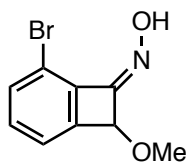
Current Data Parameters
 NAME rs3-148-3
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20150216
 Time_ 19.53
 INSTRUM spect
 PROBHD 5 mm CPPBBO BB
 PULPROG zg30
 TD 65536
 SOLVENT Acetone
 NS 16
 DS 2
 SWH 12019.230 Hz
 FIDRES 0.183399 Hz
 AQ 2.7262976 sec
 RG 31.94
 DW 41.600 use
 DE 10.00 use
 TE 300.0 K
 D1 1.00000000 sec
 TD0 1

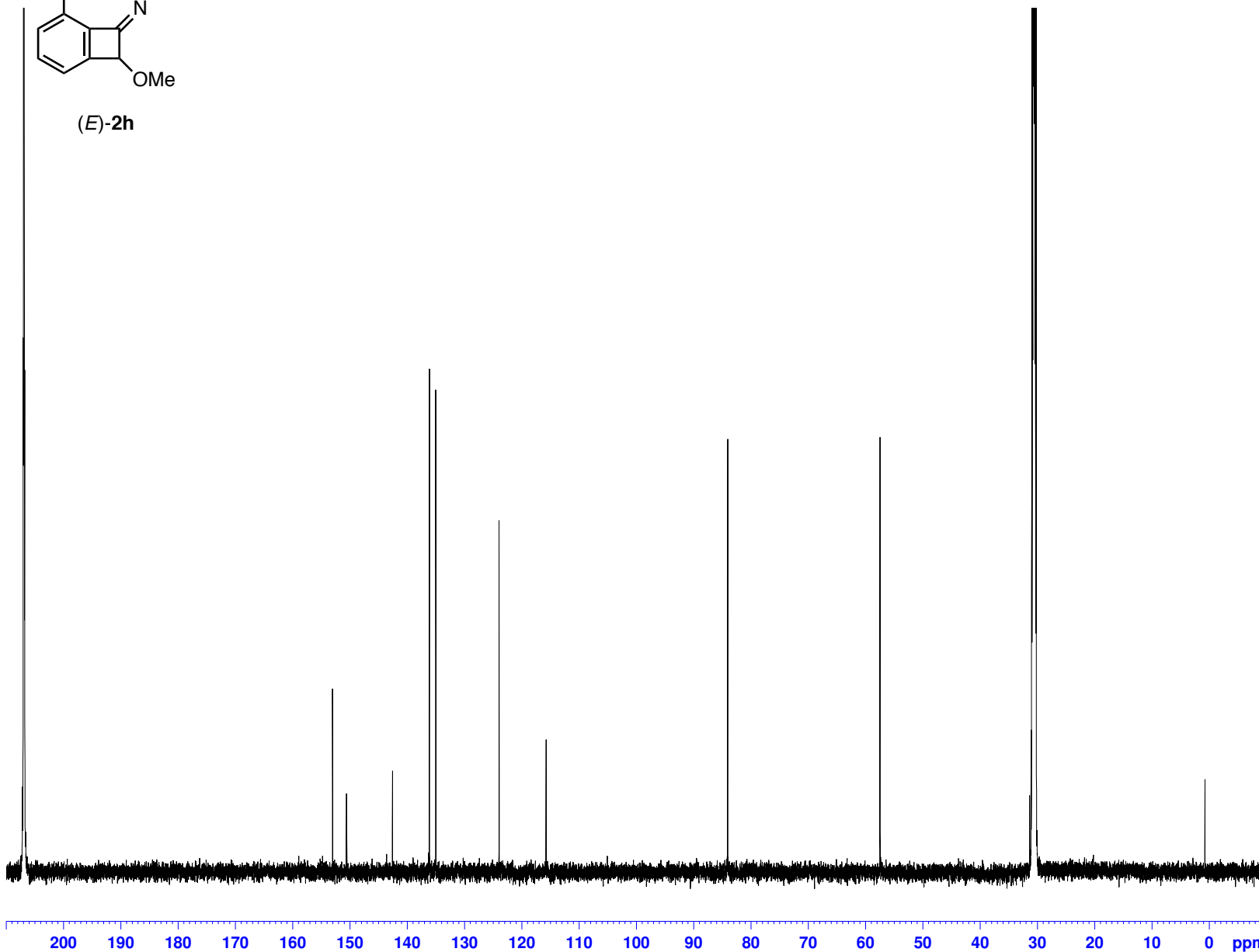
==== CHANNEL f1 =====
 SFO1 600.1337060 MHz
 NUC1 1H
 P1 12.00 use
 PLW1 23.00000000 W

F2 - Processing parameters
 SI 65536
 SF 600.1300108 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00





(E)-2h



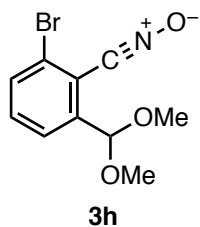
Current Data Parameters
 NAME rs3-148-3
 EXPNO 11
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20150217
 Time_ 6.00
 INSTRUM spect
 PROBHD 5 mm CPPBBO BB
 PULPROG zgpg30
 TD 65536
 SOLVENT Acetone
 NS 1024
 DS 4
 SWH 36057.691 Hz
 FIDRES 0.550197 Hz
 AQ 0.9087659 sec
 RG 175.56
 DW 13.867 use
 DE 18.00 use
 TE 300.0 K
 D1 2.0000000 sec
 D11 0.0300000 sec
 TD0 1

==== CHANNEL f1 =====
 SFO1 150.9178981 MHz
 NUC1 13C
 P1 10.00 use
 PLW1 70.0000000 W

==== CHANNEL f2 =====
 SFO2 600.1324005 MHz
 NUC2 1H
 CPDPRG[2] waltz16
 PCPD2 70.00 use
 PLW2 26.0000000 W
 PLW12 0.76407999 W
 PLW13 0.37439999 W

F2 - Processing parameters
 SI 32768
 SF 150.9025576 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

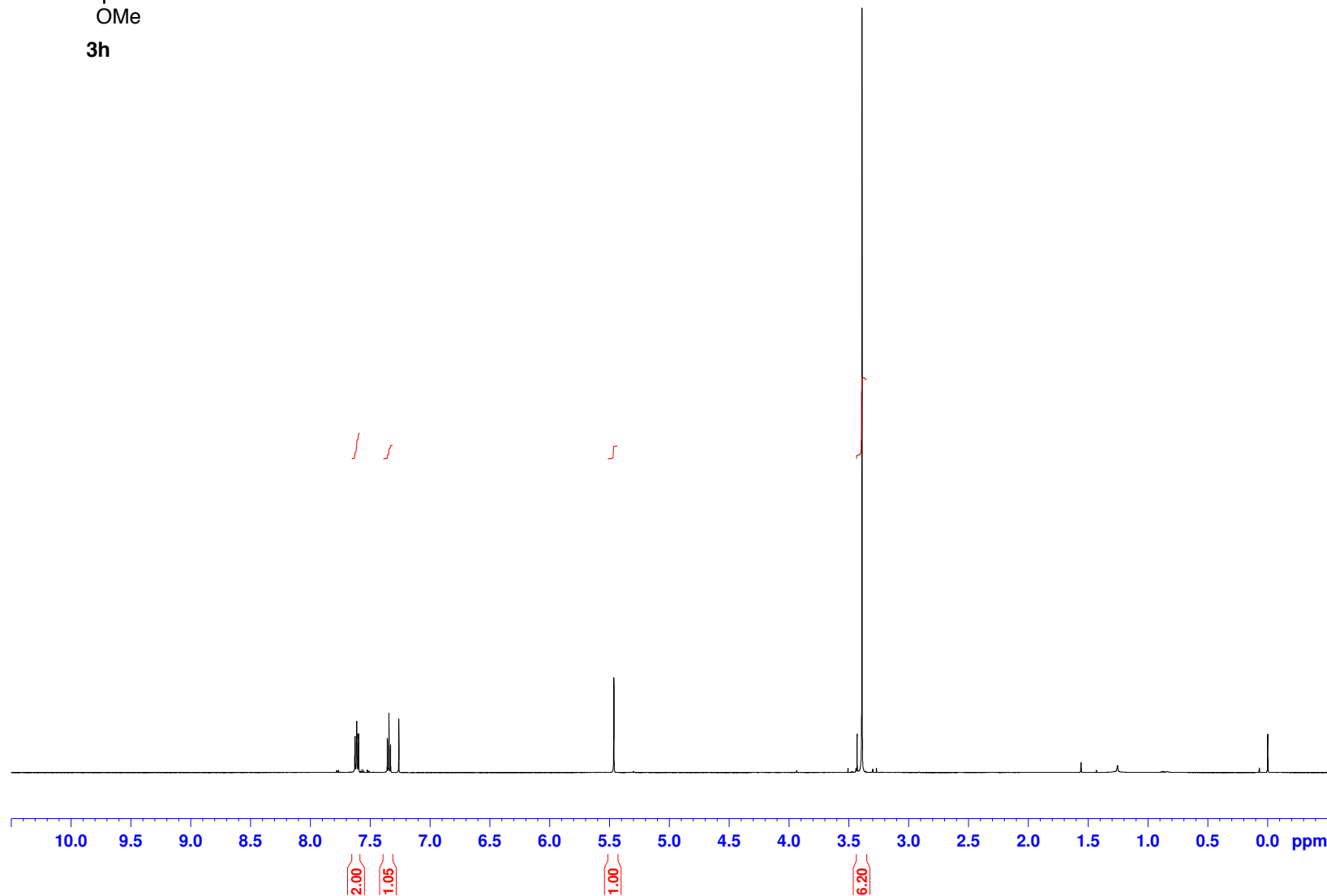


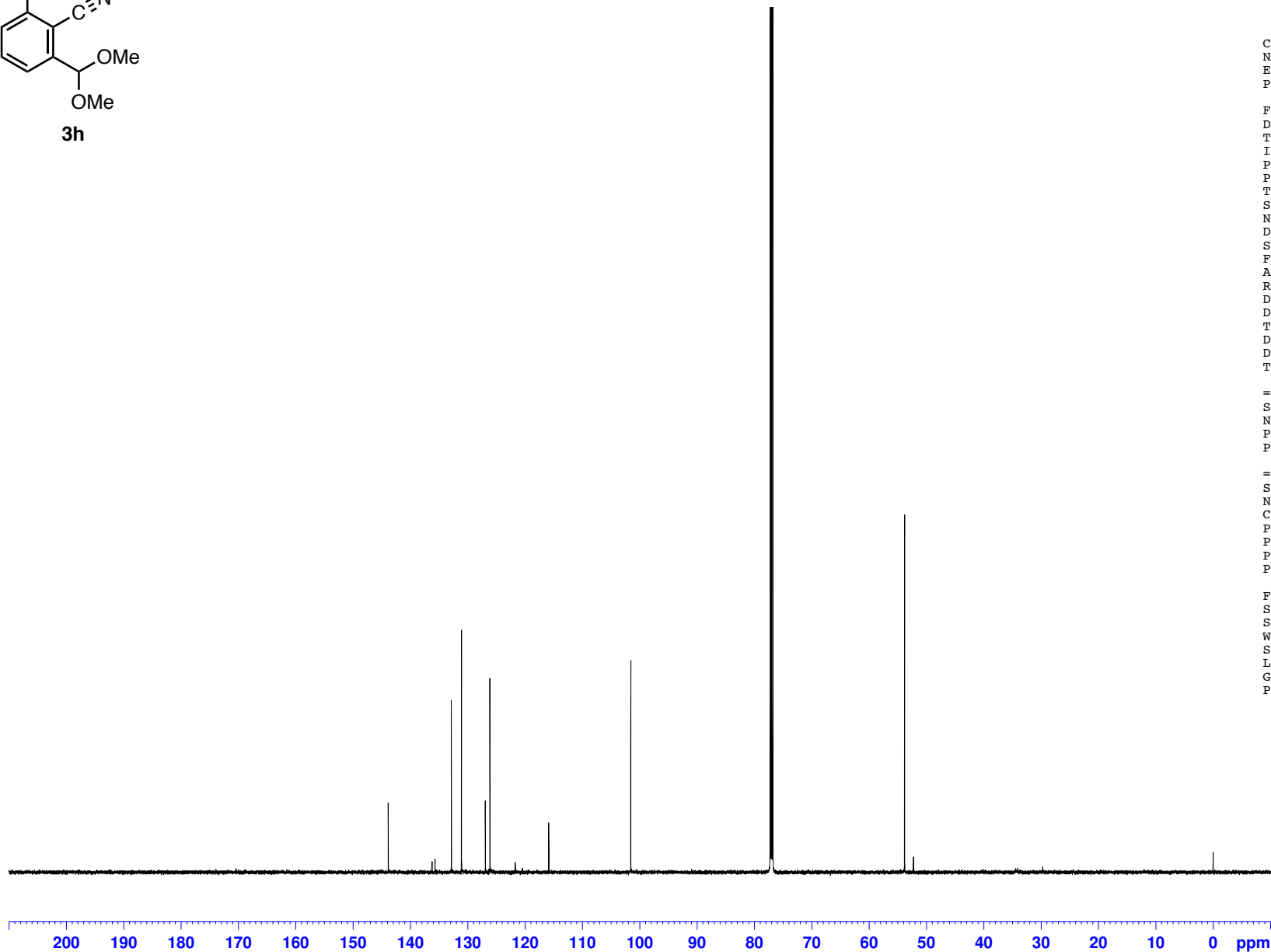
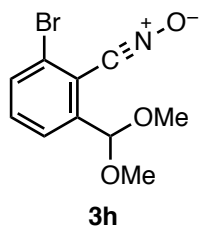
Current Data Parameters
 NAME Br nitori
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20170222
 Time_ 15.00
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDC13
 NS 16
 DS 2
 SWH 12019.230 Hz
 FIDRES 0.183399 Hz
 AQ 2.7262976 sec
 RG 87.68
 DW 41.600 use
 DE 6.50 use
 TE 298.0 K
 D1 1.0000000 sec
 TD0 1

==== CHANNEL f1 =====
 SFO1 600.1337060 MHz
 NUC1 1H
 P1 12.00 use
 PLW1 22.0000000 W

F2 - Processing parameters
 SI 65536
 SF 600.1300162 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00





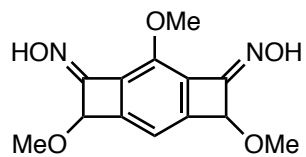
Current Data Parameters
 NAME Br nitori
 EXPNO 20
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20170223
 Time_ 2.34
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDC13
 NS 3000
 DS 4
 SWH 36057.691 Hz
 FIDRES 0.550197 Hz
 AQ 0.9087659 sec
 RG 175.56
 DW 13.867 use
 DE 6.50 use
 TE 298.0 K
 D1 2.0000000 sec
 D11 0.0300000 sec
 TD0 1

==== CHANNEL f1 =====
 SFO1 150.9178981 MHz
 NUC1 13C
 P1 11.00 use
 PLW1 93.0000000 W

==== CHANNEL f2 =====
 SFO2 600.1324005 MHz
 NUC2 1H
 CPDPRG[2] waltz16
 PCPD2 70.00 use
 PLW2 22.0000000 W
 PLW12 0.64652997 W
 PLW13 0.31680000 W

F2 - Processing parameters
 SI 32768
 SF 150.9028089 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40



2i

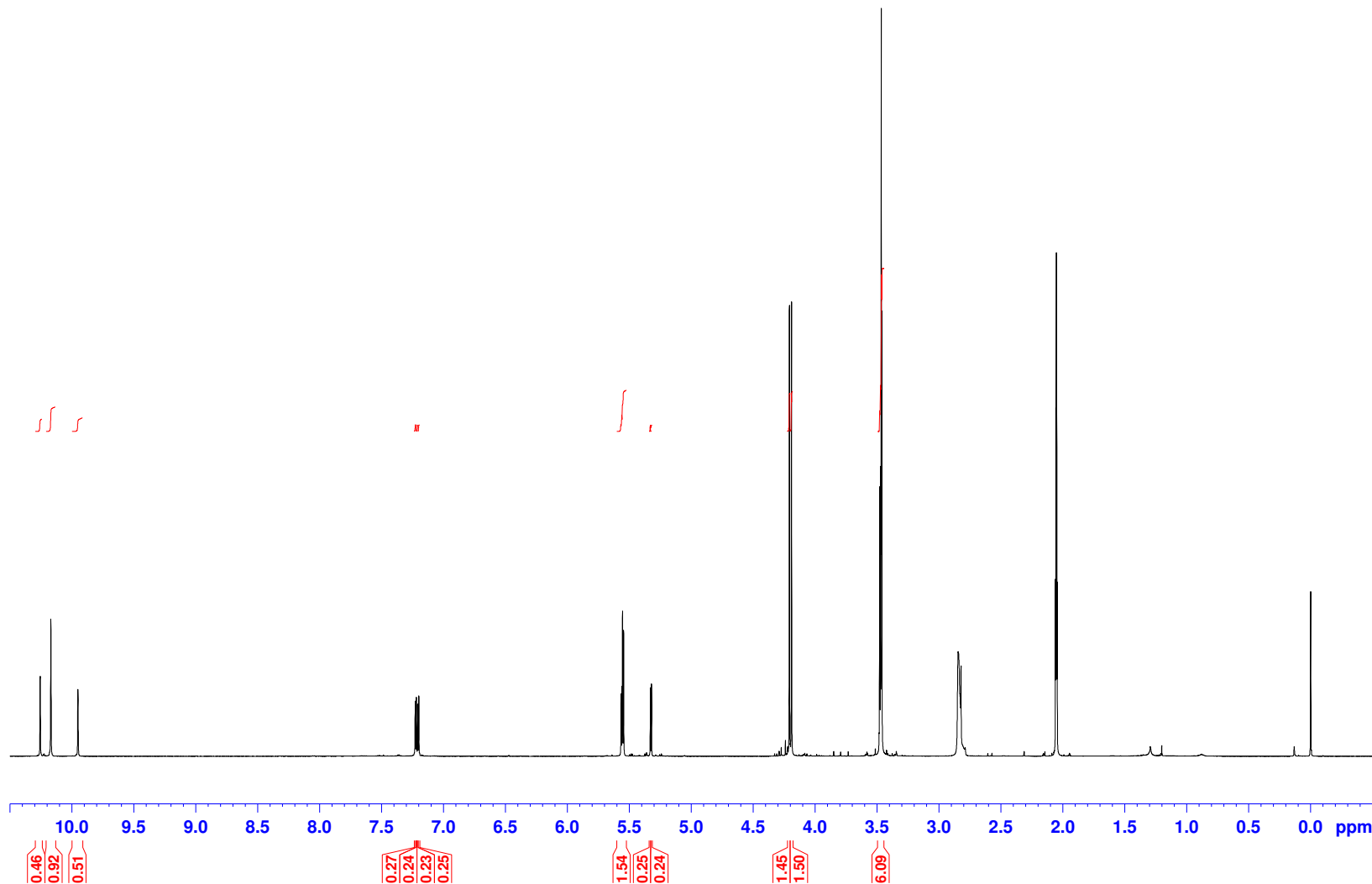


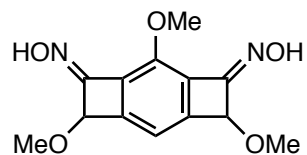
Current Data Parameters
 NAME dioxime
 EXPNO 70
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20170222
 Time_ 23.54
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT Acetone
 NS 16
 DS 2
 SWH 12019.230 Hz
 FIDRES 0.183399 Hz
 AQ 2.7262976 sec
 RG 87.68
 DW 41.600 use
 DE 6.50 use
 TE 298.0 K
 D1 1.00000000 sec
 TD0 1

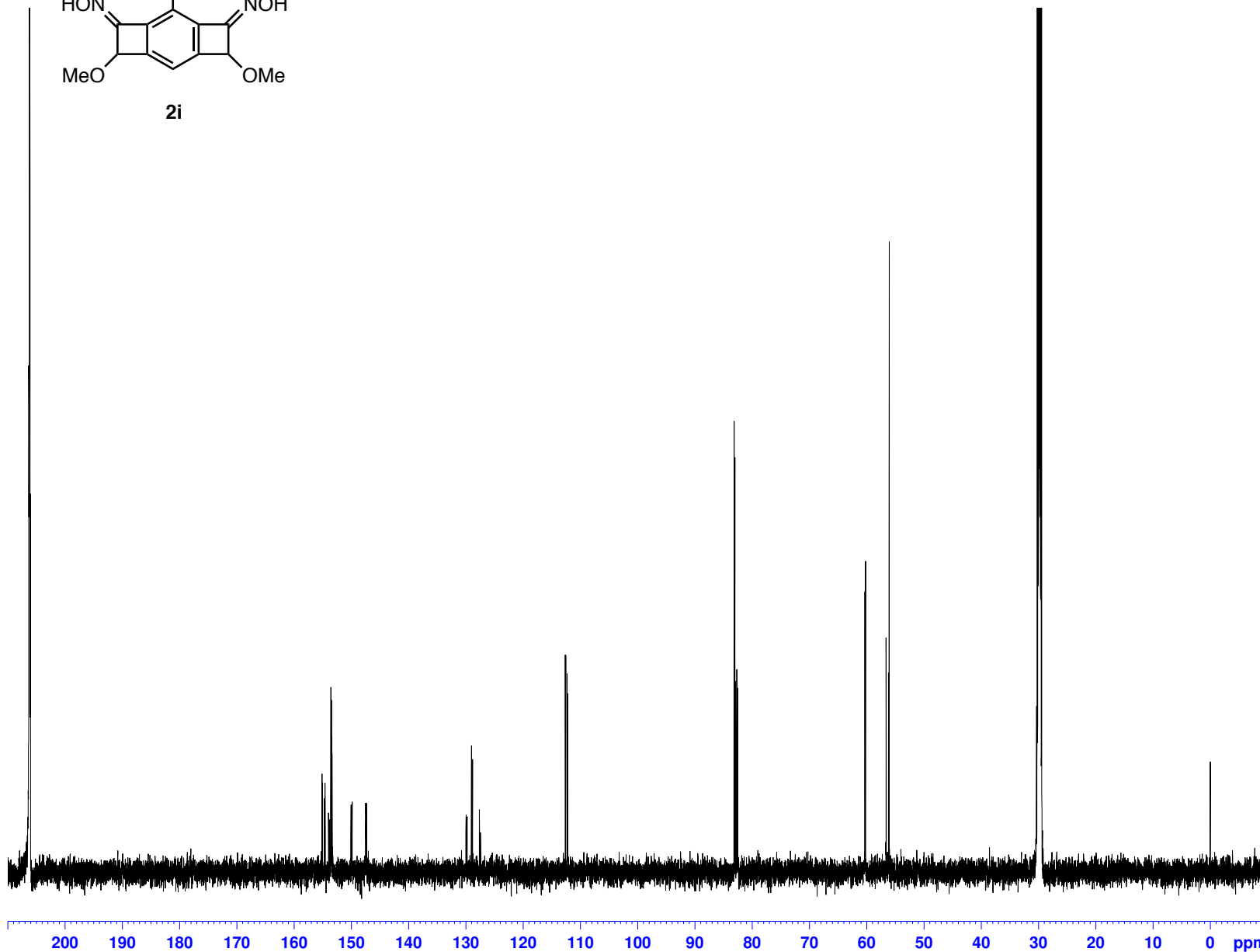
==== CHANNEL f1 =====
 SFO1 600.1337060 MHz
 NUC1 1H
 P1 12.00 use
 PLW1 22.00000000 W

F2 - Processing parameters
 SI 65536
 SF 600.1300108 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00





2i



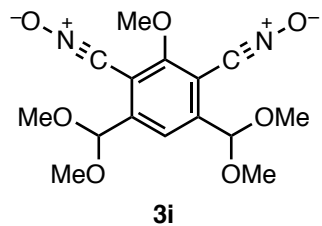
Current Data Parameters
NAME dioxime
EXPNO 71
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170223
Time_ 7.40
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT Acetone
NS 3000
DS 4
SWH 36057.691 Hz
FIDRES 0.550197 Hz
AQ 0.9087659 sec
RG 175.56
DW 13.867 use
DE 6.50 use
TE 298.0 K
D1 2.0000000 sec
D11 0.0300000 sec
TD0 1

==== CHANNEL f1 =====
SFO1 150.9178981 MHz
NUC1 13C
P1 11.00 use
PLW1 93.0000000 W

==== CHANNEL f2 =====
SFO2 600.1324005 MHz
NUC2 1H
CPDPRG[2] waltz16
PCPD2 70.00 use
PLW2 22.0000000 W
PLW12 0.64652997 W
PLW13 0.31680000 W

F2 - Processing parameters
SI 32768
SF 150.9026725 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

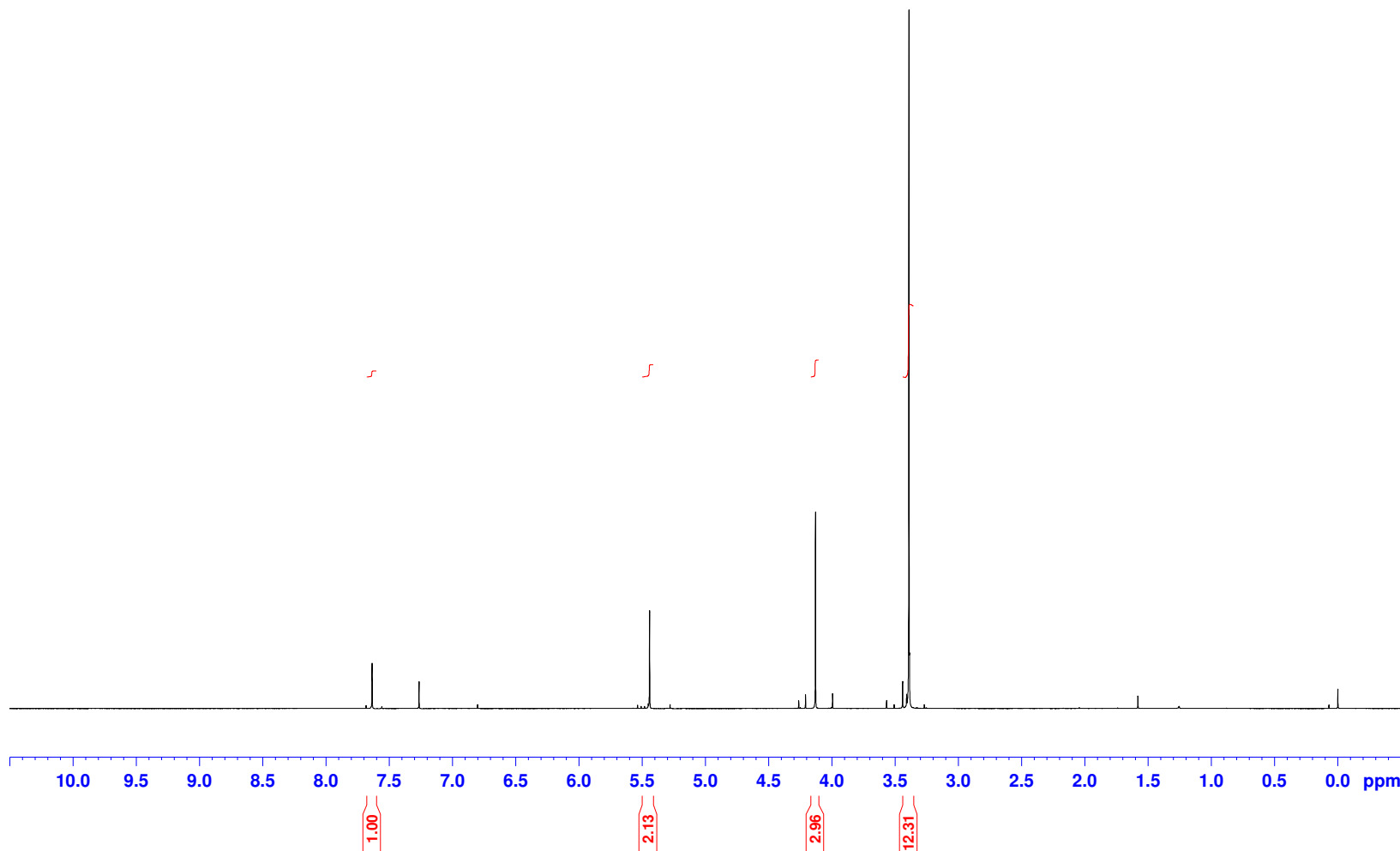


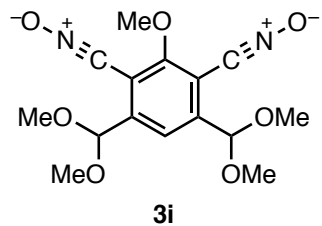
Current Data Parameters
 NAME dinitori
 EXPNO 60
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20170308
 Time_ 16.05
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDC13
 NS 16
 DS 2
 SWH 12019.230 Hz
 FIDRES 0.183399 Hz
 AQ 2.7262976 sec
 RG 75.66
 DW 41.600 use
 DE 6.50 use
 TE 298.0 K
 D1 1.0000000 sec
 TD0 1

==== CHANNEL f1 =====
 SFO1 600.1337060 MHz
 NUC1 1H
 P1 12.00 use
 PLW1 22.0000000 W

F2 - Processing parameters
 SI 65536
 SF 600.1300148 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00





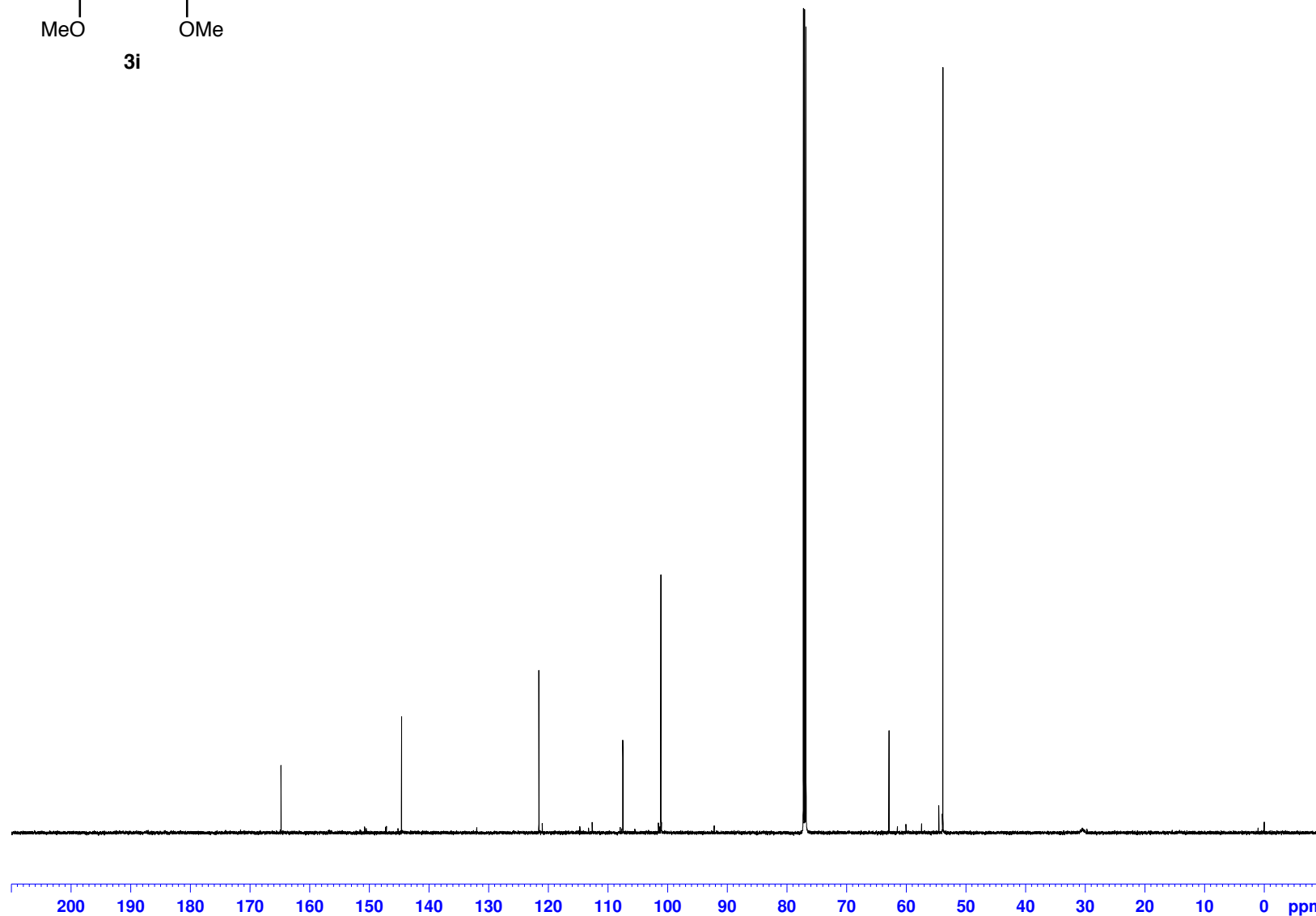
Current Data Parameters
 NAME dinitori
 EXPNO 61
 PROCNO 1

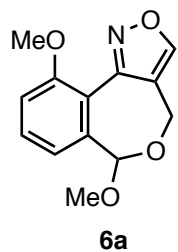
F2 - Acquisition Parameters
 Date_ 20170309
 Time_ 2.02
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 2400
 DS 4
 SWH 36057.691 Hz
 FIDRES 0.550197 Hz
 AQ 0.9087659 sec
 RG 175.56
 DW 13.867 use
 DE 6.50 use
 TE 298.0 K
 D1 2.0000000 sec
 D11 0.0300000 sec
 TD0 1

==== CHANNEL f1 =====
 SFO1 150.9178981 MHz
 NUC1 13C
 P1 11.00 use
 PLW1 93.0000000 W

==== CHANNEL f2 =====
 SFO2 600.1324005 MHz
 NUC2 1H
 CPDPRG[2] waltz16
 PCPD2 70.00 use
 PLW2 22.0000000 W
 PLW12 0.64652997 W
 PLW13 0.31680000 W

F2 - Processing parameters
 SI 32768
 SF 150.9028080 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40



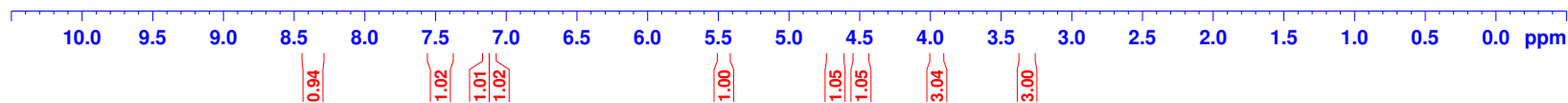
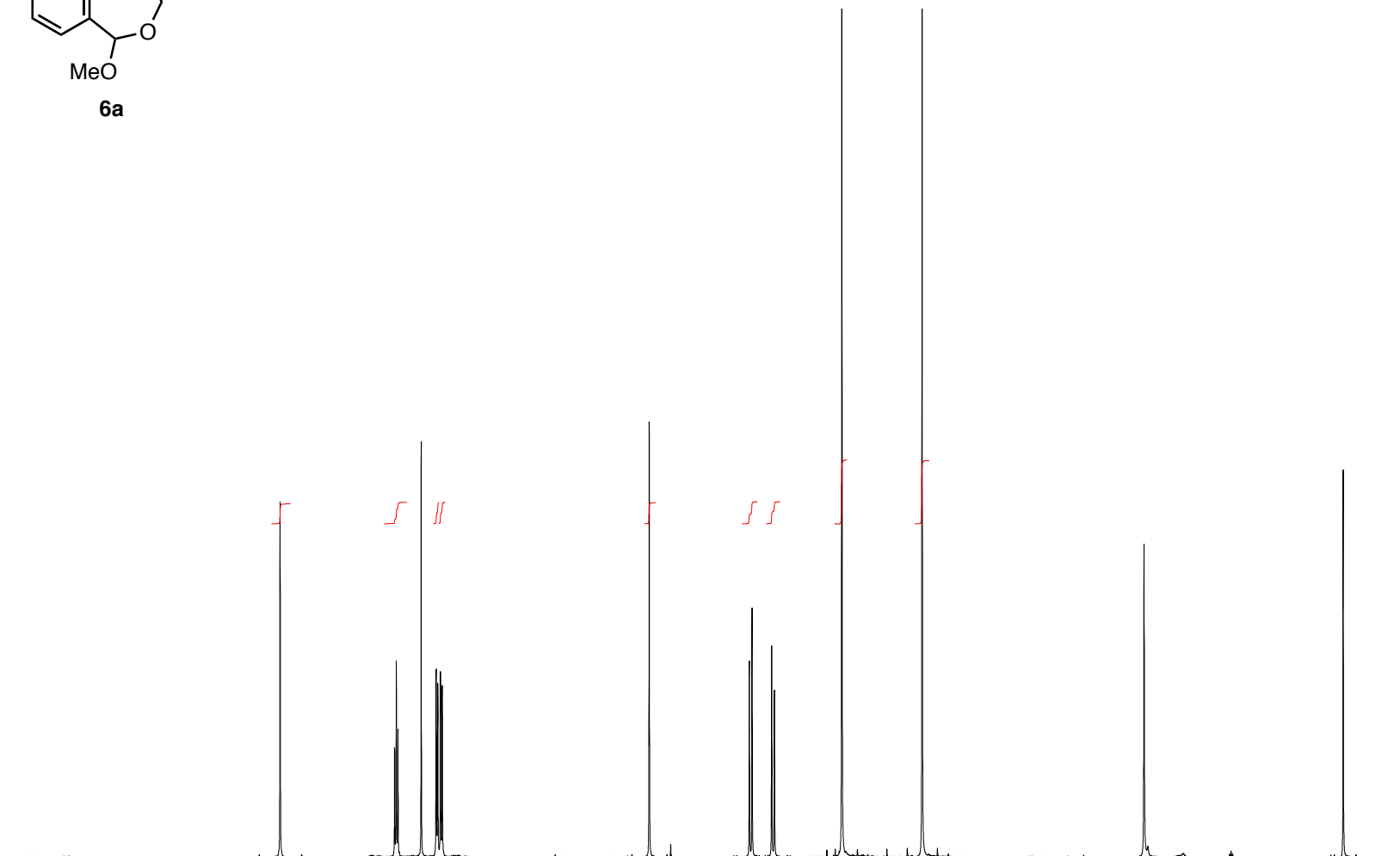


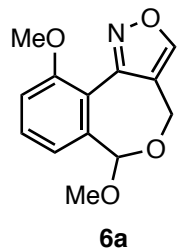
Current Data Parameters
 NAME 137-1
 EXPNO 11
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20150407
 Time_ 23.18
 INSTRUM spect
 PROBHD 5 mm CPPBBO BB
 PULPROG zg30
 TD 65536
 SOLVENT CDC13
 NS 16
 DS 2
 SWH 12019.230 Hz
 FIDRES 0.183399 Hz
 AQ 2.7262976 sec
 RG 31.94
 DW 41.600 use
 DE 10.00 use
 TE 300.0 K
 D1 1.0000000 sec
 TD0 1

==== CHANNEL f1 =====
 SFO1 600.1337060 MHz
 NUC1 1H
 P1 12.00 use
 PLW1 23.0000000 W

F2 - Processing parameters
 SI 65536
 SF 600.1300156 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00





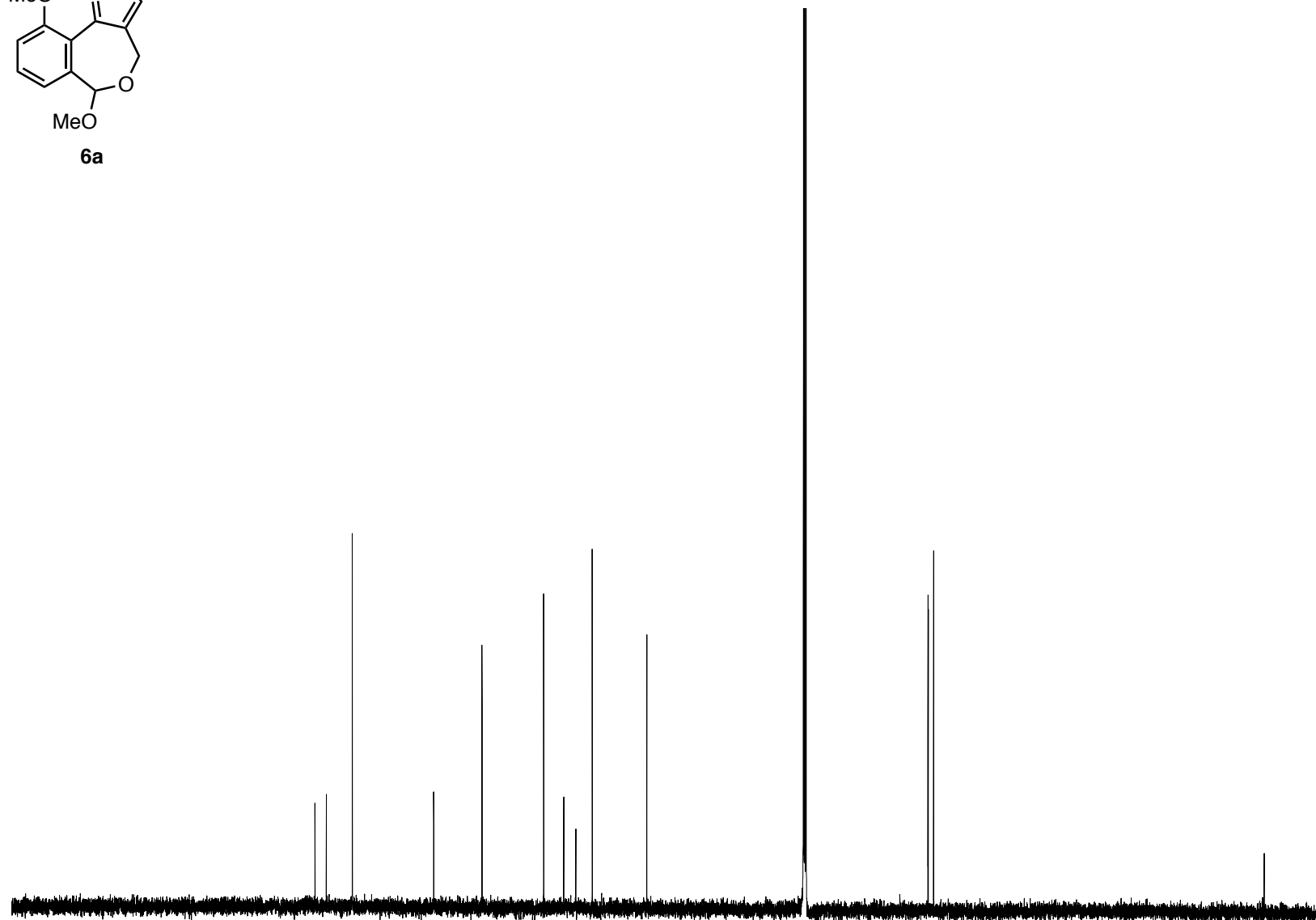
Current Data Parameters
 NAME 137-1
 EXPNO 12
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20150407
 Time_ 23.40
 INSTRUM spect
 PROBHD 5 mm CPPBBO BB
 PULPROG zgpg30
 TD 65536
 SOLVENT CDC13
 NS 128
 DS 4
 SWH 36057.691 Hz
 FIDRES 0.550197 Hz
 AQ 0.9087659 sec
 RG 175.56
 DW 13.867 use
 DE 18.00 use
 TE 300.0 K
 D1 2.0000000 sec
 D11 0.0300000 sec
 TD0 1

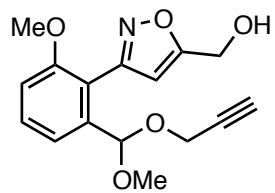
==== CHANNEL f1 =====
 SFO1 150.9178981 MHz
 NUC1 13C
 P1 10.00 use
 PLW1 70.0000000 W

==== CHANNEL f2 =====
 SFO2 600.1324005 MHz
 NUC2 1H
 CPDPRG[2] waltz16
 PCPD2 70.00 use
 PLW2 26.0000000 W
 PLW12 0.76407999 W
 PLW13 0.37439999 W

F2 - Processing parameters
 SI 32768
 SF 150.9028090 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40



200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm



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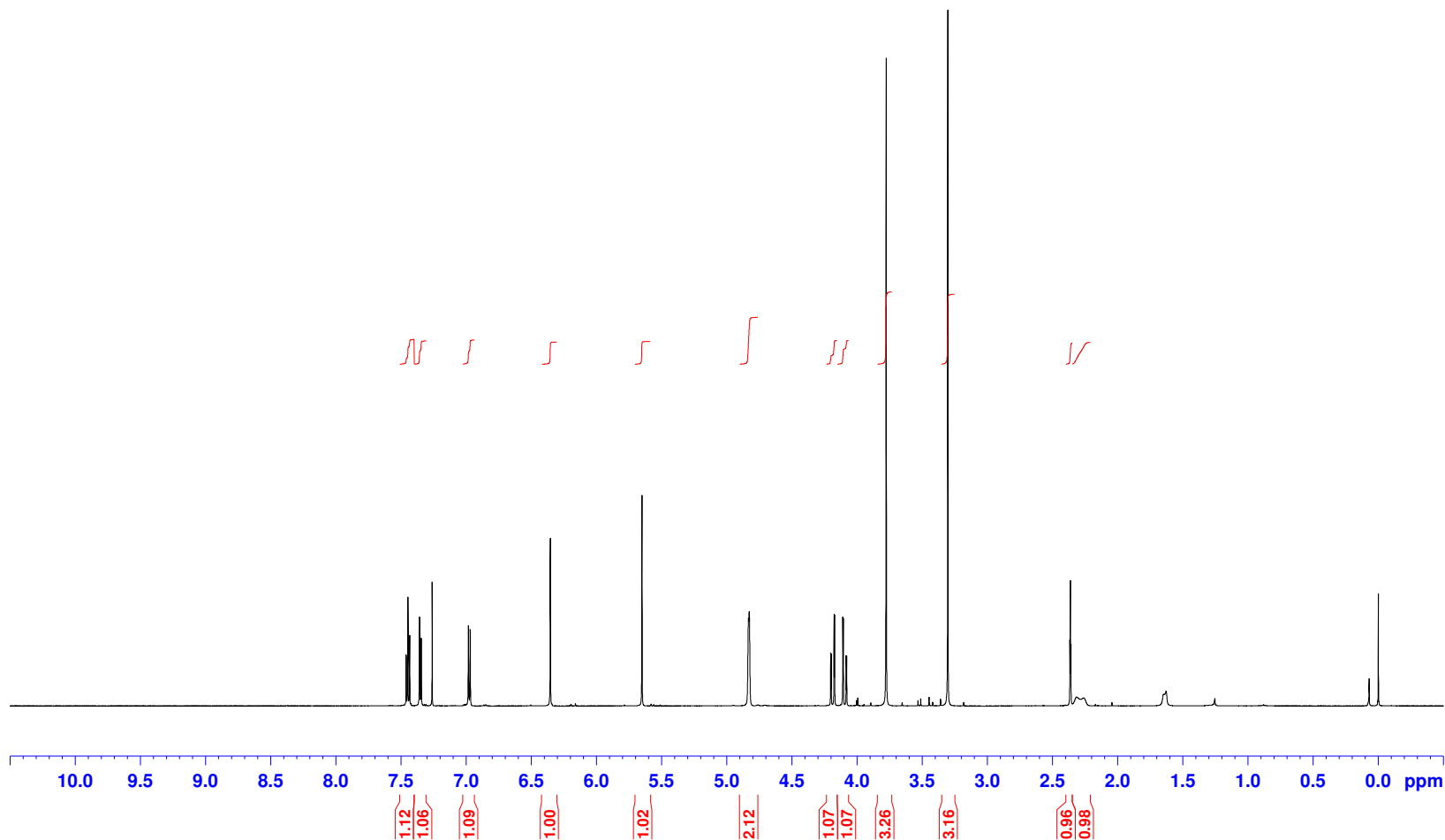


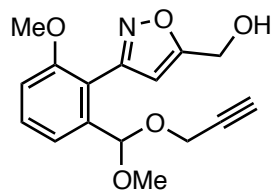
Current Data Parameters
 NAME iso 1-2
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20170304
 Time 17.12
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 12019.230 Hz
 FIDRES 0.183399 Hz
 AQ 2.7262976 sec
 RG 83.7
 DW 41.600 use
 DE 6.50 use
 TE 298.0 K
 D1 1.00000000 sec
 TD0 1

==== CHANNEL f1 =====
 SF01 600.1337060 MHz
 NUC1 1H
 P1 12.00 use
 PLW1 22.00000000 W

F2 - Processing parameters
 SI 65536
 SF 600.1300168 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00





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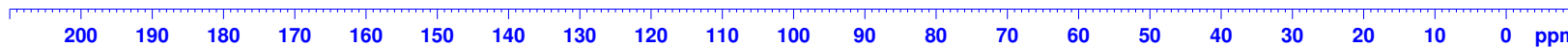
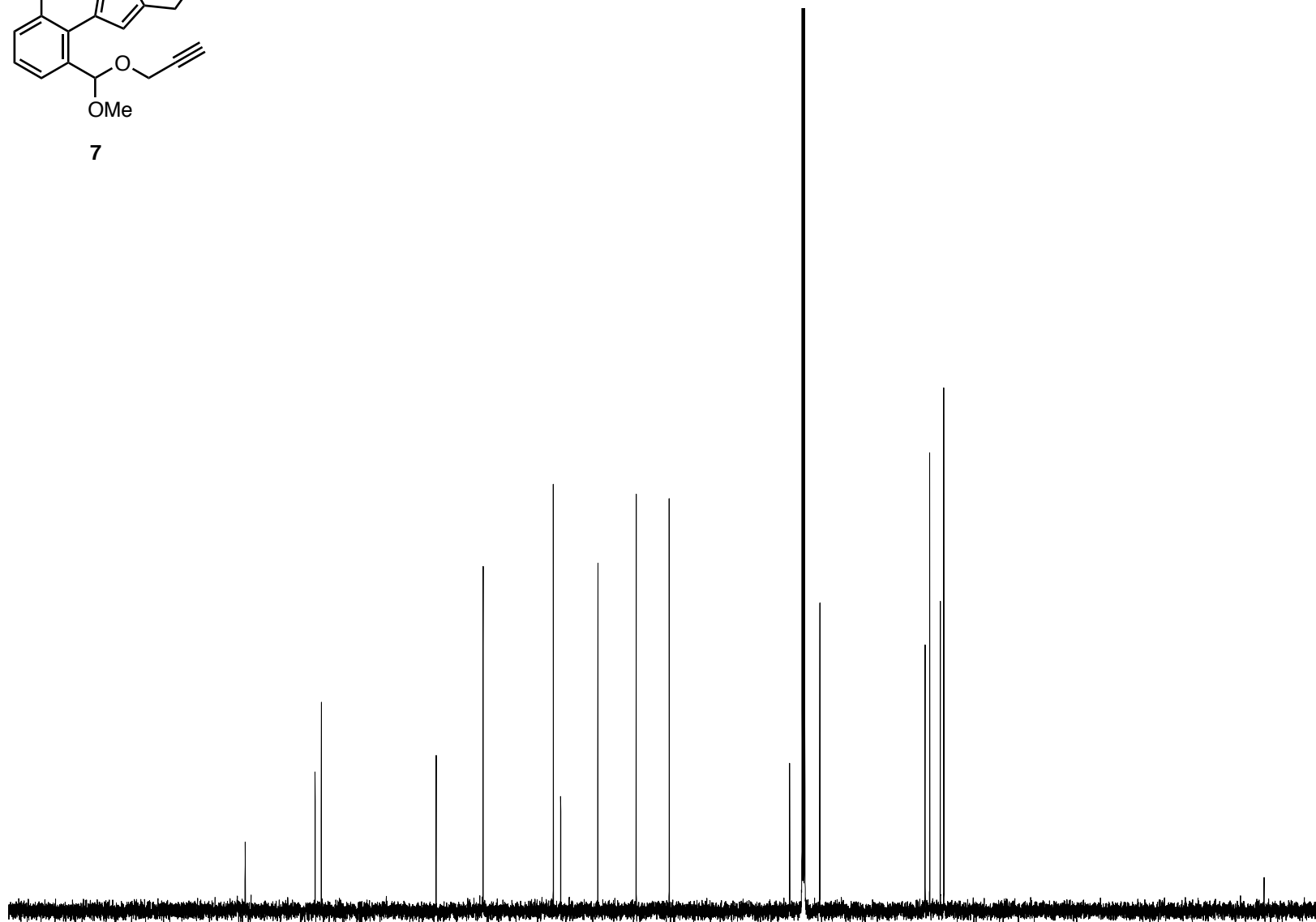
Current Data Parameters
 NAME iso 1-2
 EXPNO 11
 PROCNO 1

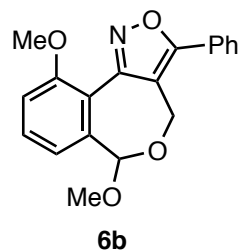
F2 - Acquisition Parameters
 Date_ 20170304
 Time_ 17.43
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDC13
 NS 600
 DS 4
 SWH 36057.691 Hz
 FIDRES 0.550197 Hz
 AQ 0.9087659 sec
 RG 175.56
 DW 13.867 use
 DE 6.50 use
 TE 298.0 K
 D1 2.0000000 sec
 D11 0.0300000 sec
 TD0 1

==== CHANNEL f1 =====
 SFO1 150.9178981 MHz
 NUC1 13C
 P1 11.00 use
 PLW1 93.0000000 W

==== CHANNEL f2 =====
 SFO2 600.1324005 MHz
 NUC2 1H
 CPDPRG[2] waltz16
 PCPD2 70.00 use
 PLW2 22.0000000 W
 PLW12 0.64652997 W
 PLW13 0.31680000 W

F2 - Processing parameters
 SI 32768
 SF 150.9028090 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40



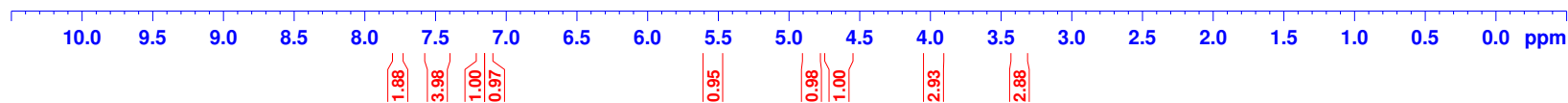
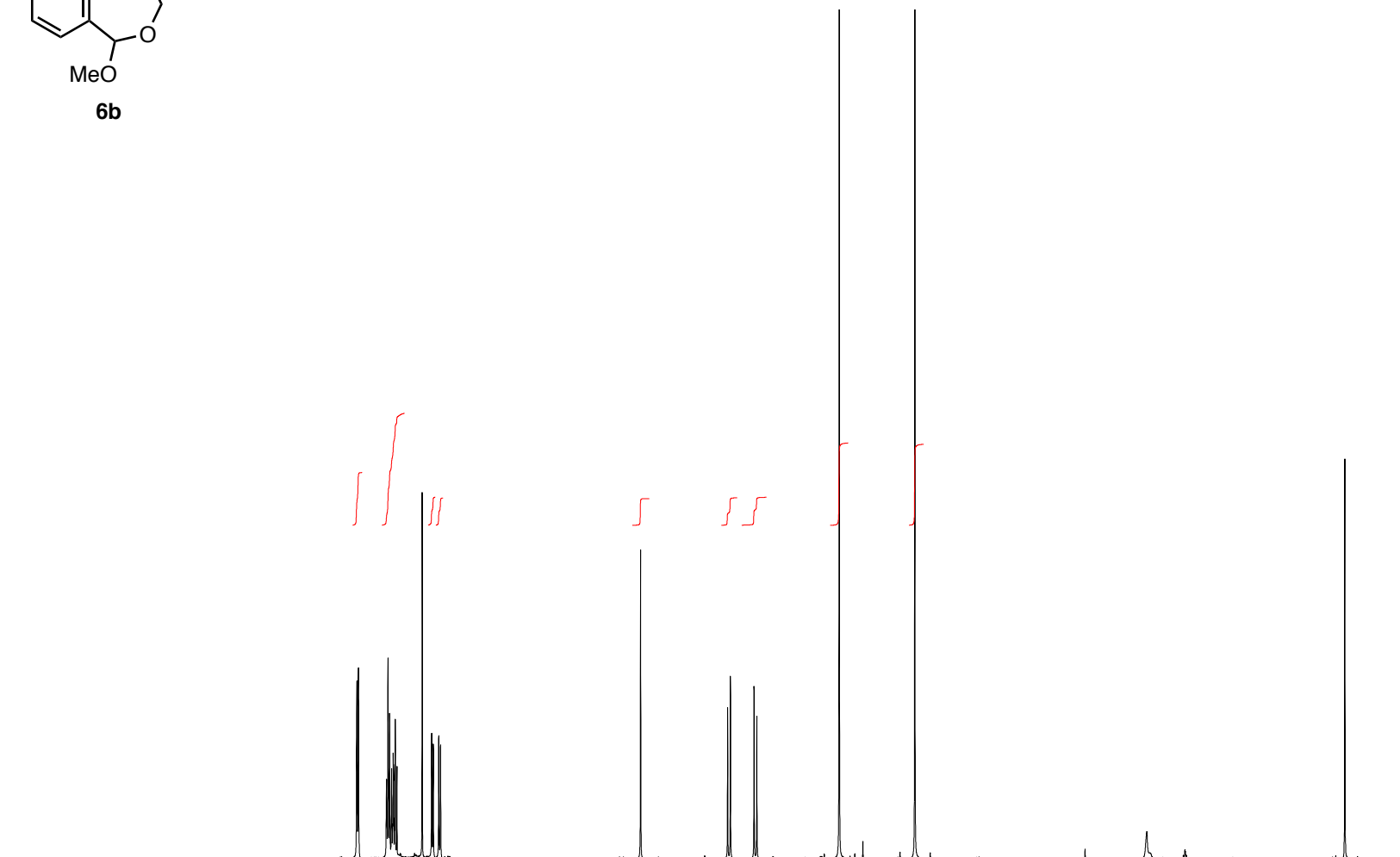


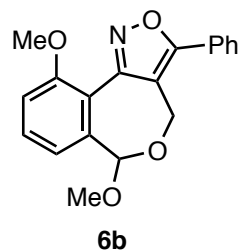
Current Data Parameters
 NAME rs3-448-1
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20161210
 Time_ 17.29
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDC13
 NS 16
 DS 2
 SWH 12019.230 Hz
 FIDRES 0.183399 Hz
 AQ 2.7262976 sec
 RG 99.18
 DW 41.600 use
 DE 6.50 use
 TE 298.0 K
 D1 1.0000000 sec
 TD0 1

==== CHANNEL f1 =====
 SFO1 600.1337060 MHz
 NUC1 1H
 P1 12.00 use
 PLW1 22.0000000 W

F2 - Processing parameters
 SI 65536
 SF 600.1300184 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00





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Current Data Parameters
NAME          rs3-447-1
EXPNO         10
PROCNO        1

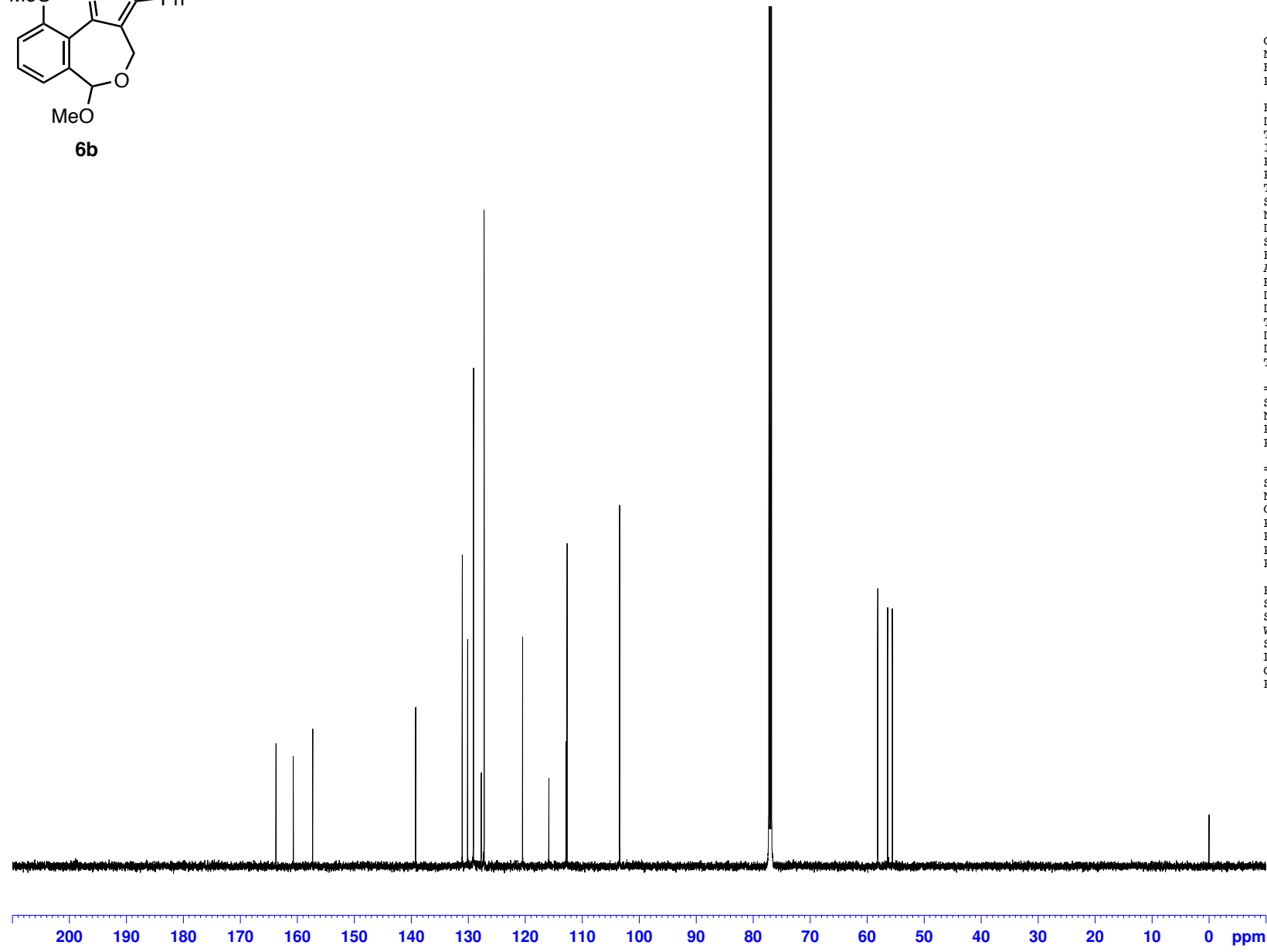
F2 - Acquisition Parameters
Date_         20161214
Time_         7.26
INSTRUM       spect
PROBHD        5 mm PABBO BB/
PULPROG       zgpg30
TD            65536
SOLVENT       CDC13
NS            4096
DS            4
SWH           36057.691 Hz
FIDRES        0.550197 Hz
AQ            0.9087659 sec
RG            175.56
DW            13.867 use
DE            6.50 use
TE            298.0 K
D1            2.0000000 sec
D11           0.0300000 sec
TD0           1

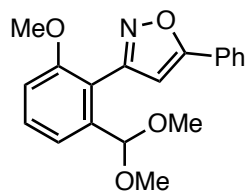
===== CHANNEL f1 =====
SFO1          150.9178981 MHz
NUC1           13C
P1             11.00 use
PLW1           93.0000000 W

===== CHANNEL f2 =====
SFO2          600.1324005 MHz
NUC2           1H
CPDPRG[2]     waltz16
PCPD2         70.00 use
PLW2          22.0000000 W
PLW12         0.64652997 W
PLW13         0.31680000 W

F2 - Processing parameters
SI             32768
SF            150.9028101 MHz
WDW            EM
SSB            0
LB             1.00 Hz
GB            0
PC             1.40

```





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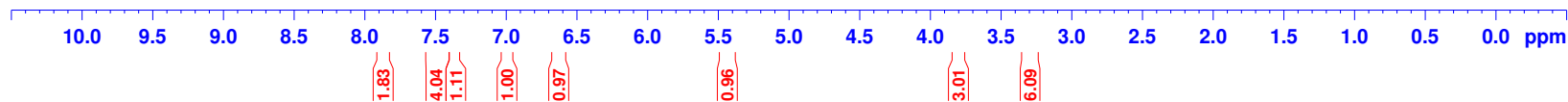
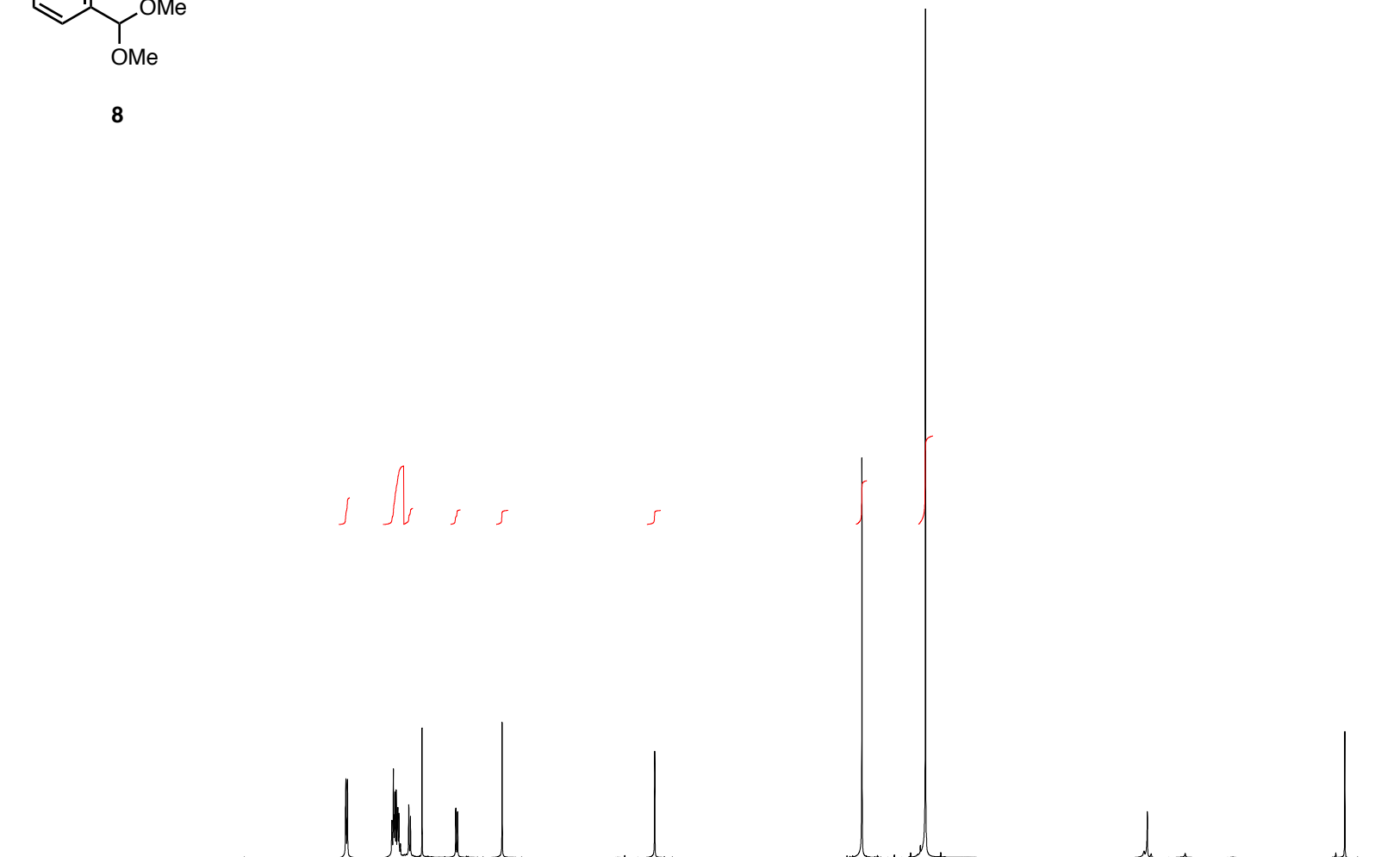


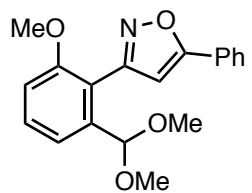
Current Data Parameters
 NAME rs3 1H
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20161126
 Time_ 18.23
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDC13
 NS 16
 DS 2
 SWH 12019.230 Hz
 FIDRES 0.183399 Hz
 AQ 2.7262976 sec
 RG 87.68
 DW 41.600 use
 DE 6.50 use
 TE 298.0 K
 D1 1.0000000 sec
 TD0 1

==== CHANNEL f1 =====
 SFO1 600.1337060 MHz
 NUC1 1H
 P1 12.00 use
 PLW1 22.0000000 W

F2 - Processing parameters
 SI 65536
 SF 600.1300187 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00





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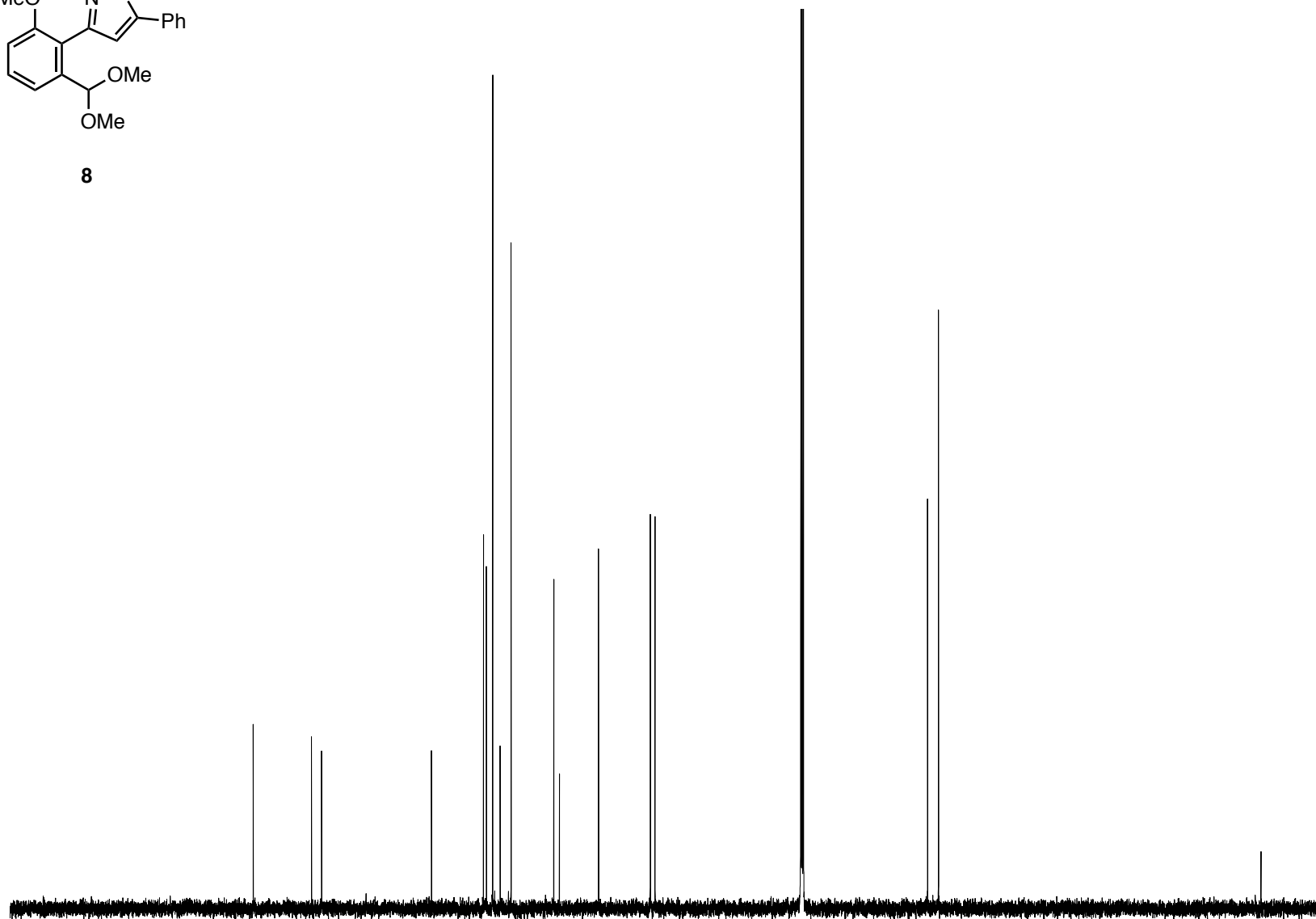
Current Data Parameters
 NAME rs3 13C
 EXPNO 10
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20161127
 Time_ 23.53
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 1024
 DS 4
 SWH 36057.691 Hz
 FIDRES 0.550197 Hz
 AQ 0.9087659 sec
 RG 175.56
 DW 13.867 use
 DE 6.50 use
 TE 298.0 K
 D1 2.0000000 sec
 D11 0.0300000 sec
 TD0 1

==== CHANNEL f1 =====
 SFO1 150.9178981 MHz
 NUC1 13C
 P1 11.00 use
 PLW1 93.0000000 W

==== CHANNEL f2 =====
 SFO2 600.1324005 MHz
 NUC2 1H
 CPDPRG[2] waltz16
 PCPD2 70.00 use
 PLW2 22.0000000 W
 PLW12 0.64652997 W
 PLW13 0.31680000 W

F2 - Processing parameters
 SI 32768
 SF 150.9028101 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40



200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm