

Efficient Total Synthesis of (-)-Stemoamide

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ELECTRONIC SUPPLEMENTARY INFORMATION (ESI)

1) General Experimental Procedures	S-2
2) Experimental Procedures	
(5 <i>S</i>)-({ <i>tert</i> -Butyl(dimethyl)silyl}oxy)methyl-1-[4-(tetrahydro-2 <i>H</i> -pyran-2-yloxy)butyl]pyrrolidin-2-one (6)	S-3
(5 <i>S</i>)-(Hydroxymethyl)-1-[4-(tetrahydro-2 <i>H</i> -pyran-2-yloxy)butyl]-pyrrolidin-2-one (7)	S-3
(2 <i>S</i>)-5-Oxo-1-[4-(tetrahydro-2 <i>H</i> -pyran-2-yloxy)butyl]pyrrolidine-2-carbaldehyde (8)	S-3
Methyl (2 <i>E</i>)-3-{(2 <i>S</i>)-5-oxo-1-[4-(tetrahydro-2 <i>H</i> -pyran-2-yloxy)butyl]pyrrolidin-2-yl}acrylate (9)	S-3
Methyl (2 <i>E</i>)-3-[(2 <i>S</i>)-1-[4-(hydroxybutyl)-5-oxopyrrolidin-2-yl]acrylate (10)	S-4
Methyl (2 <i>E</i>)-3-{(2 <i>S</i>)-5-oxo-1-[4-oxobutyl]pyrrolidin-2-yl}acrylate (11)	S-4
SmI ₂ -promoted cyclization of 11 to 12 and 13 in the absence of HMPA	S-5
(3 <i>aR</i> ,10 <i>aS</i> ,10 <i>bS</i>)-Octahydro-2 <i>H</i> -furo[3,2- <i>c</i>]pyrrolo[1,2- <i>a</i>]azepine-2,8(1 <i>H</i>)-dione (12)	S-5
(3 <i>aS</i> ,10 <i>aS</i> ,10 <i>bS</i>)-Octahydro-2 <i>H</i> -furo[3,2- <i>c</i>]pyrrolo[1,2- <i>a</i>]azepine-2,8(1 <i>H</i>)-dione (13)	S-5
(3 <i>aR</i> ,10 <i>aS</i>)-3 <i>a</i> ,4,5,6,10,10 <i>a</i> -Hexahydro-2 <i>H</i> -furo[3,2- <i>c</i>]pyrrolo[1,2- <i>a</i>]azepine-2,8(9 <i>H</i>)-dione (15)	S-5
SmI ₂ -promoted cyclization of 11 to 16 in the presence of HMPA	S-6
(3 <i>aS</i> ,10 <i>aS</i>)-3 <i>a</i> ,4,5,6,10,10 <i>a</i> -Hexahydro-2 <i>H</i> -furo[3,2- <i>c</i>]pyrrolo[1,2- <i>a</i>]azepine-	

2,8(9 <i>H</i>)-dione (16)	S-6
SmI ₂ -promoted cyclization of 11 to 16 in the presence of HMPA	S-6
SmI ₂ -promoted cyclization of 18 to 12 and 13 in the absence of HMPA	S-6
SmI ₂ -promoted cyclization of 18 to 16 in the presence of HMPA	S-7
(1 <i>S</i> ,3 <i>aR</i> ,10 <i>aS</i> ,10 <i>bR</i>)-1-Methyloctahydro-2 <i>H</i> -furo[3,2- <i>c</i>]pyrrolo[1,2- <i>a</i>]azepine-2,8(1 <i>H</i>)-dione; (-)-Stemoamide (1)	S-7
3) NMR spectra	
¹ H NMR Spectrum of 6	S-8
¹³ C NMR Spectrum of 6	S-9
¹ H NMR Spectrum of 7	S-10
¹³ C NMR Spectrum of 7	S-11
¹ H NMR Spectrum of 9	S-12
¹³ C NMR Spectrum of 9	S-13
¹ H NMR Spectrum of 10	S-14
¹³ C NMR Spectrum of 10	S-15
¹ H NMR Spectrum of 11	S-16
¹³ C NMR Spectrum of 11	S-17
¹ H NMR Spectrum of 15	S-18
¹³ C NMR Spectrum of 15	S-19
¹ H NMR Spectrum of 16	S-20
¹³ C NMR Spectrum of 16	S-21
¹ H NMR Spectrum of 1	S-22

General Experimental Procedures. Melting points were measured with a melting point apparatus and are uncorrected. IR spectra were recorded as thin films on sodium chloride plates. Otherwise noted, ¹H and ¹³C NMR spectra were obtained for solutions in CDCl₃, and chemical shifts are reported on the δ scale using TMS as an internal standard of δ 0.00 for ¹H-NMR spectra, and CDCl₃ as an internal standard or δ 77.00 for ¹³C NMR spectra, respectively (¹H-NMR: 400 MHz, ¹³C NMR: 100 MHz). Reagents were purchased from commercial sources.

(5S)-({*tert*-Butyl(dimethyl)silyloxy}methyl)-1-[4-(tetrahydro-2*H*-pyran-2-yloxy)-butyl]pyrrolidin-2-one (6). To a stirred solution of the amide (5) (2.00 g, 8.73 mmol), 2-(4-bromobutoxy)tetrahydro-2*H*-pyran (4.10 g, 17.37 mmol), and tetrabutylammonium iodide (323 mg, 0.84 mmol) in DMF (88 mL) was added dropwise NaHMDS (1.9M in THF solution, 6.9 mL, 13.1 mmol) at -15 °C. The resulting mixture was stirred at the same temperature for 10 min, and at ambient temperature for 3 h. The mixture was treated with saturated aqueous NH₄Cl solution, diluted with H₂O, and extracted three times with CHCl₃. The combined organic extracts were washed twice with H₂O and brine, dried over Na₂SO₄ and concentrated under reduced pressure. The residue was purified by column chromatography on silica gel. Elution with hexane-EtOAc (3:7, v/v) gave **6** (2.96 g, 88%) as a colorless oil. $[\alpha]_D^{20} +5.76$ (c 1.0 CHCl₃); IR ν_{max} : 3473, 2938, 2857, 1687, 1460 cm⁻¹; ¹H NMR δ : 4.50 (t, *J* = 4.2 Hz, 1H), 3.85-3.76 (m, 1H), 3.75-3.50 (m, 5H), 3.48-3.40 (m, 1H), 3.38-3.32 (m, 1H), 3.10-2.92 (m, 1H), 2.44-2.34 (m, 1H), 2.28-2.20 (m, 1H), 2.08-1.96 (m, 1H), 1.84-1.73 (m, 2H), 1.58-1.42 (m, 9H), 0.90 (s, 9H), 0.05 (s, 6H); ¹³C NMR δ : 175.2, 98.8, 67.0, 63.8, 62.3, 58.8, 40.5, 30.6, 30.5, 27.0, 25.6, 25.3, 24.2, 21.4, 19.5, 18.0, -5.7; HRMS Calcd for C₂₀H₄₀NO₄Si [M+H]⁺ 386.2726. Found 386.2721.

(5S)-(Hydroxymethyl)-1-[4-(tetrahydro-2*H*-pyran-2-yloxy)butyl]pyrrolidin-2-one (7). To a stirred solution of **6** (2.00 g, 5.19 mmol) in MeOH (26 mL) was added NH₄F (962 mg, 25.98 mmol) at room temperature and the resulting mixture was heated at reflux for 10 h. After concentration of the mixture in vacuo, a residue was taken up into 5% methanol in CHCl₃ and the whole solution was filtrated to remove the insoluble materials. The filtrate was concentrated to leave a residue, which was purification by column chromatography on silica gel. Elution with MeOH-EtOAc (5:95, v/v) furnished **7** as a colorless oil (1.34 g, 95%). $[\alpha]_D^{20} +6.4$ (c 1.0); IR ν_{max} : 3409, 2938, 1660, 1464 cm⁻¹; ¹H NMR δ : 4.50 (t, *J* = 4.2 Hz, 1H), 3.85-3.50 (m, 6H), 3.52-3.44 (m, 1H), 3.43-3.36 (m, 1H), 3.11-3.02 (m, 1H), 2.51-2.40 (m, 1H), 2.34-2.25 (m, 1H), 2.14-2.04 (m, 1H), 2.00-1.90 (m, 1H), 1.80-1.74 (m, 1H), 1.74-1.46 (m, 9H); ¹³C NMR δ : 175.8, 99.0, 67.0, 62.8, 62.6, 59.1, 40.5, 30.7, 30.5, 27.0, 25.4, 24.3, 21.2, 19.8; HRMS Calcd for C₁₄H₂₆NO₄ [M+H]⁺ 272.1862. Found 272.1846.

Methyl (2*E*)-3-{{(2*S*)-5-oxo-1-[4-(tetrahydro-2*H*-pyran-2-yloxy)butyl]pyrrolidin-2-yl}acrylate (9). To a stirred solution of oxalyl chloride (1.52 mL, 17.96 mmol) in CH₂Cl₂ (22 mL) was added a solution of DMSO (1.9 mL, 26.75 mmol) in CH₂Cl₂ (4.5 mL) at -78 °C under argon, and the resulting solution was stirred at the same

temperature for 10 min. A solution of **7** (2.40 g, 8.86 mmol) in CH₂Cl₂ (15 mL) was added to the solution, and the whole was stirred at the same temperature for further 1 h. The mixture was treated with triethylamine (8.7 mL, 62.13 mmol), and warmed to room temperature over the period of 20 min. The solution was treated with H₂O and extracted with CHCl₃. The organic layer was washed with brine and dried over Na₂SO₄. Removal of the solvent under reduced pressure afforded the aldehyde **8**, which without further purification, was used directly in the next step.

To a stirred solution of the crude aldehyde (**8**) (2.36 g, 8.77 mmol) in acetonitrile (43 mL) was added methyl (triphenylphosphoranylidene)acetate (3.82 g, 11.44 mmol), and the resulting mixture was stirred at room temperature for 12 h. Removal of the solvent under reduced pressure gave a residue, which was purified by column chromatography on silica gel. Elution with hexane-EtOAc (1:9, v/v) gave **9** (2.36 g, 82%) as a colorless oil. $[\alpha]_D^{20} +5.3$ (c 0.9); IR ν_{max} : 2947, 1725, 1687, 1437 cm⁻¹; ¹H NMR δ : 6.75 (dd, $J = 8.1, 15.6$ Hz, 1H), 5.95 (d, $J = 15.6$ Hz, 1H), 4.50 (t, $J = 4.2$ Hz, 1H), 4.27-4.20 (m, 1H), 3.87-3.80 (m, 1H), 3.77-3.70 (m, 4H), 3.68-3.60 (m, 1H), 3.52-3.46 (m, 1H), 3.41-3.35 (m, 1H), 2.90-2.82 (m, 1H), 2.50-2.20 (m, 3H), 1.86-1.75 (m, 2H), 1.74-1.46 (m, 9H); ¹³C NMR δ : 174.6, 166.0, 146.6, 122.7, 98.9, 66.9, 62.4, 58.7, 51.8, 40.8, 30.7, 29.6, 27.0, 25.4, 24.9, 24.2, 19.7; HRMS Calcd for C₁₇H₂₈NO₅ [M+H]⁺ 326.1967. Found 326.1945.

Methyl (2E)-3-[(2S)-1-[4-(hydroxybutyl)-5-oxopyrrolidin-2-yl]acrylate (10). To a stirred solution of **9** (2.84 g, 8.74 mmol) in MeOH (87 mL) was added *p*-TSA (166 mg, 0.87 mmol) at room temperature, and the resulting mixture was stirred at the same temperature for further 12 h. The reaction was quenched by addition of saturated aqueous NaHCO₃ solution and the aqueous layer was extracted with CHCl₃. The extract was washed with brine and dried over Na₂SO₄. Removal of the solvent under reduced pressure afforded the alcohol (**10**) (1.94 g, 92%) as a colorless oil. $[\alpha]_D^{20} +7.5$ (c 1.0); IR ν_{max} : 3401, 2946, 1720, 1666, 1436 cm⁻¹; ¹H NMR δ : 6.75 (dd, $J = 8.3, 15.7$ Hz, 1H), 5.95 (d, $J = 15.7$ Hz, 1H), 4.25 (dt, $J = 4.8, 8.0$ Hz, 1H), 3.75 (s, 3H), 3.65-3.55 (m, 3H), 2.94-2.83 (m, 1H), 2.50-2.25 (m, 4H), 1.86-1.77 (m, 1H), 1.60–1.45 (m, 4H); ¹³C NMR δ : 175.0, 165.9, 146.4, 122.8, 62.1, 58.9, 51.8, 40.7, 29.6, 29.4, 24.8, 23.8; HRMS Calcd for C₁₂H₂₀NO₄ [M+H]⁺ 242.1392. Found 242.1369.

Methyl (2E)-3-[(2S)-5-oxo-1-[4-oxobutyl]pyrrolidin-2-yl]acrylate (11). To a solution of oxalyl chloride (0.15 mL, 1.77 mmol) in CH₂Cl₂ (2 mL) was added a solution of DMSO (0.18 mL, 2.49 mmol) in CH₂Cl₂ (0.4 mL) at -78 °C under argon, and the

resulting solution was stirred at the same temperature for 10 min. A solution of alcohol **10** (200 mg, 0.83 mmol) in CH₂Cl₂ (2 mL) was added to the solution, and the whole was stirred at the same temperature for 1 h. After the mixture was treated with triethylamine (0.82 mL, 5.88 mmol), the whole was warmed up to room temperature over the period of 20 min. The solution was treated with H₂O, and extracted with CHCl₃. The organic layer was washed with brine, dried over Na₂SO₄, and concentrated under reduced pressure to leave a residue, which was subjected to column chromatography on silica gel. Elution with MeOH-CHCl₃ (2:98, v/v) afforded the aldehyde (**11**) (168 mg, 85%) as a colorless oil. [α]_D²⁰ +9.5 (c 1.0); IR ν_{max} : 2915, 1720, 1670, 1435 cm⁻¹; ¹H NMR δ : 9.75 (s, 1H), 6.75 (dd, J = 8.4, 15.5 Hz, 1H), 5.95 (d, J = 15.5 Hz, 1H), 4.25 (dt, J = 4.7, 8.1 Hz, 1H), 3.75 (s, 3H), 3.60-3.50 (m, 1H), 2.92-2.84 (m, 1H), 2.52-2.20 (m, 5H), 1.85-1.74 (m, 3H); ¹³C NMR δ : 201.1, 175.0, 165.9, 146.1, 123.1, 58.8, 51.8, 41.1, 40.2, 29.4, 24.8, 19.7; HRMS Calcd for C₁₂H₁₇NO₄ [M]⁺ 239.1157. Found 239.1185.

SmI₂-promoted cyclization of 11 to 12 and 13 in the absence of HMPA. To a stirred solution of the aldehyde (**11**) (100 mg, 0.42 mmol) in THF (21 mL) in the presence of MeOH (85 μ L, 2.1 mmol) was added SmI₂ (0.2 M in THF solution, 10.5 mL, 2.1 mmol) at 0 °C under argon. After being stirred for 3 h, the resulting mixture was treated saturated aqueous NH₄Cl solution and extracted with CHCl₃. The organic layer was washed with brine and dried over Na₂SO₄. Evaporation of the solvent gave a mixture of **12** and **13** in 60% yield, in a ratio of *ca.* 0.9 : 1, based on the analysis of its ¹H NMR spectrum. Spectroscopic data of **12**¹ and **13**^{2,3} were similar to those reported in the literature.

(3a*S*,10a*S*)-3a,4,5,6,10,10a-Hexahydro-2*H*-furo[3,2-*c*]pyrrolo[1,2-*a*]azepine-2,8(9*H*)-dione (15**).** To a stirred solution of **12** (293 mg, 1.40 mmol) in THF (37 mL) was added LiHMDS (1.6M in THF solution, 3.5 mL, 5.60 mmol) at -78 °C. After being stirred at the same temperature for 30 min, phenylselenenyl bromide (828 mg, 3.51 mmol) was added, and the resulting mixture was stirred for 1 h. After the reaction mixture was quenched by addition of 1 M hydrochloric acid, the aqueous phase was extracted with CHCl₃. The extract was washed with brine and dried over Na₂SO₄, and concentrated under reduced pressure to leave the selenide (**14**), which was then dissolved in CH₂Cl₂ (18 mL). The solution was cooled to 0 °C and treated with 30% hydrogen peroxide (27 mL). After the mixture was vigorously stirred for 1 h at the same temperature, the aqueous layer was extracted with CHCl₃. The organic layer was washed with brine and dried over Na₂SO₄, and concentrated under reduced pressure to leave a residue, which

was subjected to column chromatography on silica gel. Elution with MeOH-CHCl₃ (2:98, v/v) afforded **15** (347mg, 85 %) as a white solid: Mp 156-157 °C (lit.⁴ mp 157-158 °C); [α]_D -204 (*c* = 0.4, CHCl₃) {lit.⁴ [α]_D -224 (*c* = 0.4, CHCl₃)}. The spectroscopic data of **15** were comparable to those reported in the literature.⁴

(3aR,10aS,10bR)-Octahydro-2H-furo[3,2-c]pyrrolo[1,2-a]azepine-2,8(1H)-dione (16).

To a stirred solution of **15** (26.8 mg, 0.13 mmol) in MeOH (2.5 mL) was added NiCl₂ · 6H₂O (7.7 mg, 32.3 μ mol) followed by NaBH₄ (20 mg, 0.53 mmol) at -30 °C. After being stirred for 3 h at the same temperature, the solution was treated with 1M HCl. The aqueous layer was extracted with CHCl₃, and the organic layer was washed with brine and dried over Na₂SO₄, and concentrated under reduced pressure to leave a residue, which was purified by column chromatography on silica gel. Elution with MeOH-CHCl₃ (2:98, v/v) afforded **16** (24.6 mg, 91%) as a colorless oil. [α]_D²⁰ -97.8 (*c* 0.3) {lit.⁴ [α]_D²⁰ -94.0 (*c* 0.4 CHCl₃)}. The spectroscopic data of **16** were comparable to those reported in the literature.

SmI₂-promoted cyclization of 11 to 16 in the presence of HMPA. To a stirred solution of the aldehyde (**11**) (80.9 mg, 0.34 mmol) in THF (17 mL) in the presence of MeOH (68.6 μ L, 1.70 mmol) and HMPA (1.17 mL, 6.72 mmol) was added SmI₂ (0.2 M in THF solution, 8.5 mL, 1.69 mmol) at 0 °C under argon. After being stirred for 3 h, the resulting mixture was treated saturated aqueous NH₄Cl solution and extracted with CHCl₃. The organic layer was washed with brine and dried over Na₂SO₄. Evaporation of the solvent gave a residue, which was purified by column chromatography on silica gel. Elution with MeOH-CHCl₃ (2:98, v/v) gave **16** (39.2 mg, 55%) as a colorless oil, together with a small amount of its diastereoisomer (**17**). The spectroscopic data of **16** were identical with those of the authentic specimen obtained above. Although isolation of a minor product (**17**) was attempted, we could not obtain it in pure form, unfortunately, due to its small amount available.

SmI₂-promoted cyclization of 18 to 12 and 13 in the absence of HMPA. To a stirred solution of the aldehyde (**11**) (50 mg, 0.20 mmol) in THF (10.5 mL) in the presence of EtOH (58 μ L, 0.99 mmol) was added SmI₂ (0.2 M in THF solution, 5.2 mL, 0.99 mmol) at 0 °C under argon. After being stirred for 3 h, the resulting mixture was treated saturated aqueous NH₄Cl solution and extracted with CHCl₃. The organic layer was

washed with brine and dried over Na₂SO₄. Evaporation of the solvent gave a mixture of **12** and **13** in 53% yield, in a ratio of *ca.* 0.7 : 1, based on the analysis of its ¹H NMR spectrum. Spectroscopic data of **12**¹ and **13**^{2,3} were similar to those reported in the literature.

SmI₂-promoted cyclization of 18 to 16 in the presence of HMPA. To a stirred solution of the aldehyde (**18**) (32 mg, 0.13 mmol) in THF (6.4 mL) in the presence of EtOH (38 μL, 0.65 mmol) and HMPA (0.44 mL, 2.53 mmol) was added SmI₂ (0.2 M in THF solution, 3.2 mL, 0.64 mmol) at 0 °C under argon. After being stirred for 3 h, the resulting mixture was treated saturated aqueous NH₄Cl solution and extracted with CHCl₃. The organic layer was washed with brine and dried over Na₂SO₄. Evaporation of the solvent gave a residue, which was purified by column chromatography on silica gel. Elution with MeOH-CHCl₃ (2:98, v/v) gave **16** and **17** in 49% yield in a ratio of 3.5 : 1.

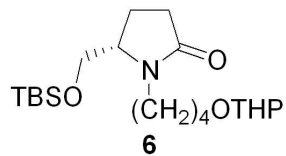
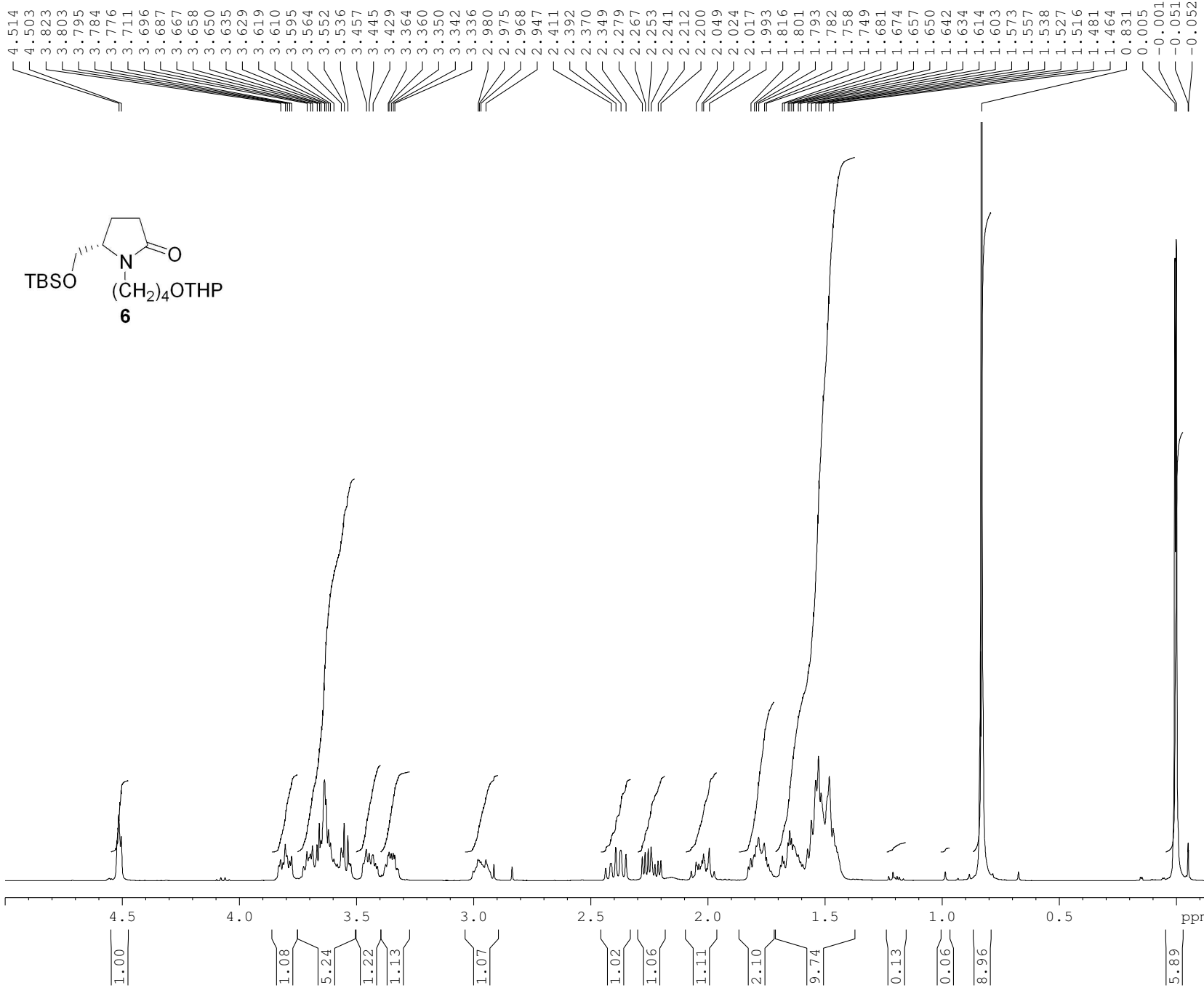
(1S,3aR,10aS,10bR)-1-Methyloctahydro-2H-furo[3,2-c]pyrrolo[1,2-a]azepine-2,8(1H)-dione; (-)-Stemoamide (1). To a stirred solution of the lactone (**16**) (37 mg, 0.18 mmol) in THF (2.3 mL) was added LiHMDS (1.6M in THF solution, 0.2 mL, 0.19 mmol) at -78 °C. After being stirred for 30 min, MeI (55 μL, 0.88 mmol) was added and the resulting mixture was stirred for 3 h at the same temperature. The mixture was allowed to warm to room temperature gradually and was stirred at room temperature for 20 h. After treatment with saturated aqueous NH₄Cl solution, the whole was extracted with EtOAc. The extract was washed with brine and dried over Na₂SO₄. Evaporation of the solvent gave a residue, which was purified by column chromatography on silica gel. Elution with MeOH-CHCl₃ (3:97, v/v) gave (-)-stemoamide (**1**) (32.2 mg, 82%) as a white solid: Mp 184-185 °C (lit.⁴ mp 185-186 °C); [α]²⁰_D -138 (c 0.2, MeOH) {lit.⁴ [α]²⁰_D -141 (c 0.3 MeOH)}. The spectroscopic data of **1** were comparable to those reported in the literature.⁴

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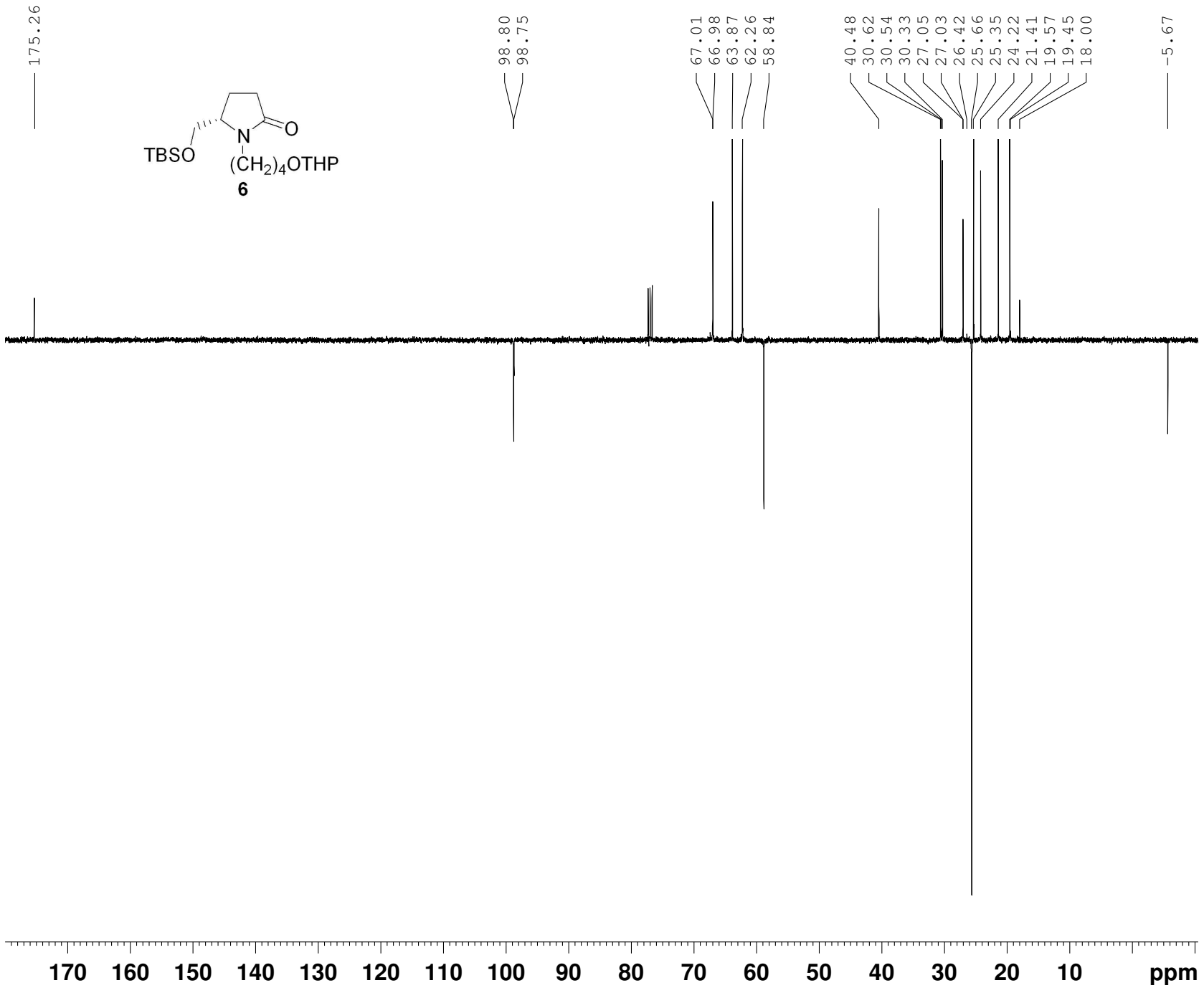


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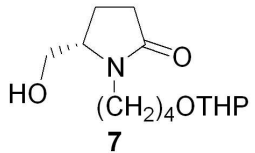
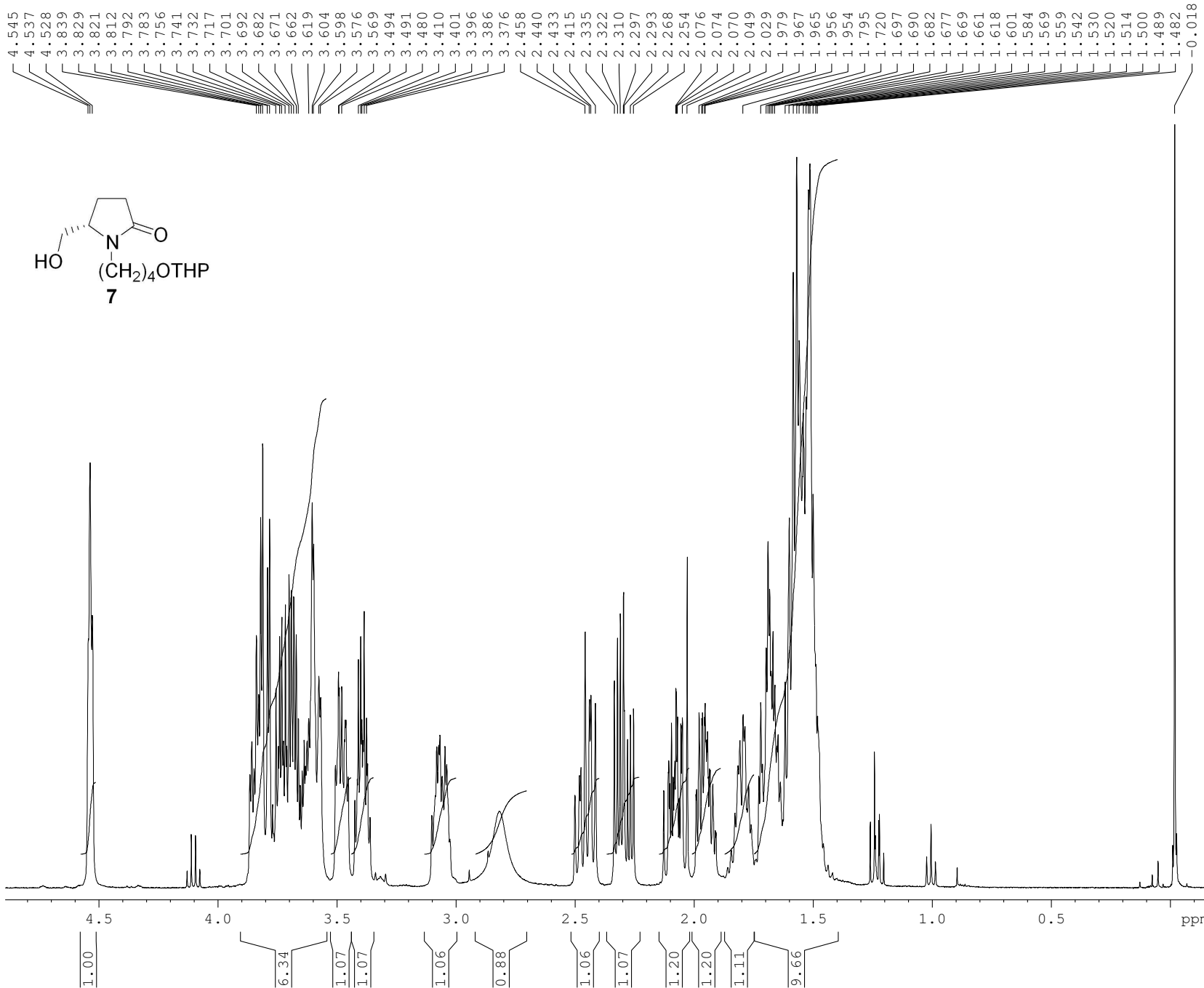
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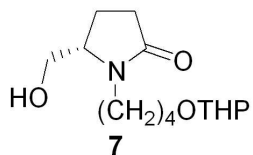
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 EXPNO 1
 PROCNO 1

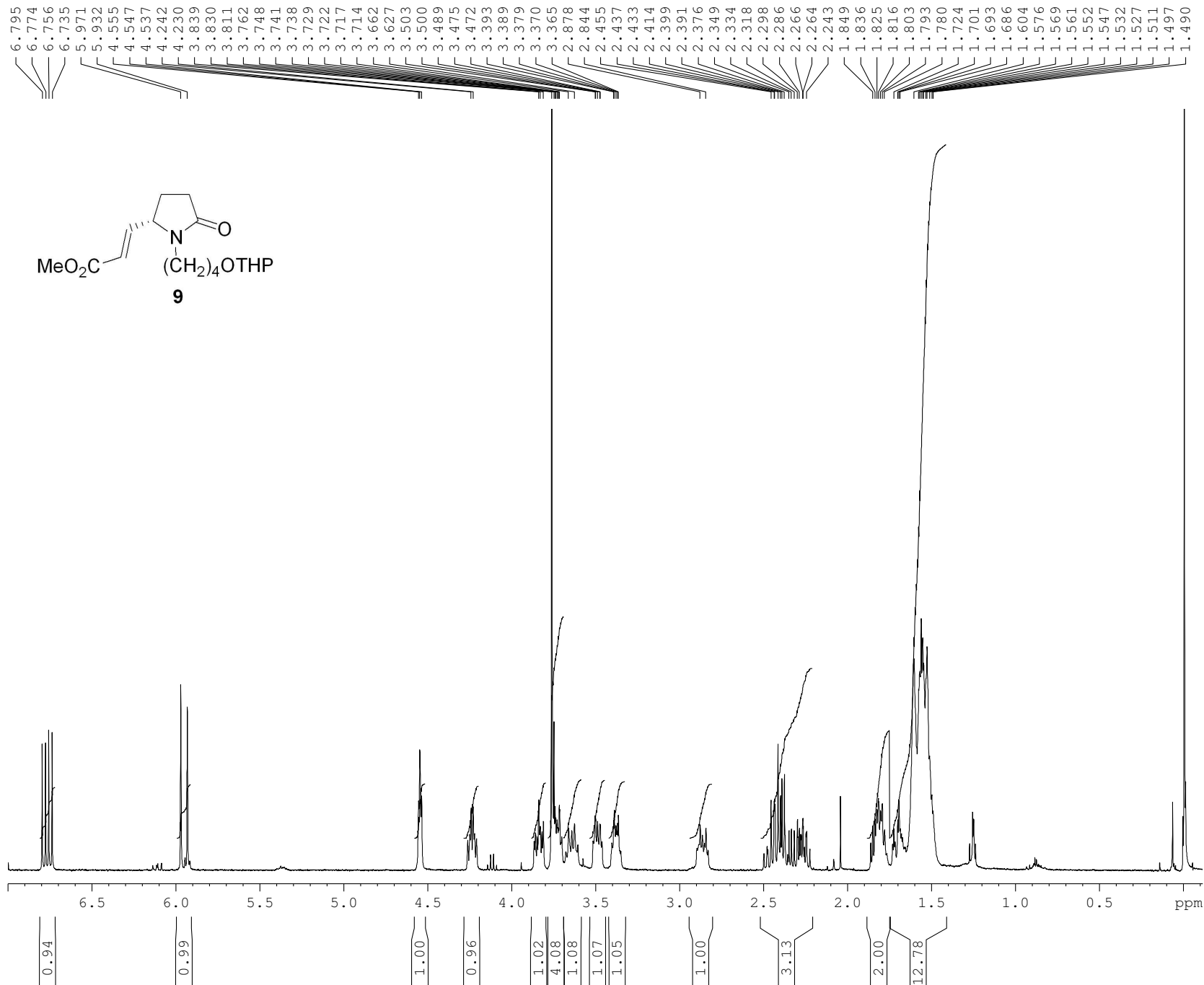
F2 - Acquisition Parameters
 Date_ 20090929
 Time 16.48
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG jmod
 TD 65536
 SOLVENT CDC13
 NS 125
 DS 4
 SMH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631988 sec
 RG 2050
 DW 20.800 usec
 DE 6.00 usec
 TE 673.2 K
 CNST2 145.0000000
 CNST11 1.0000000
 D1 2.0000000 sec
 D20 0.00689655 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 13C
 P1 8.80 usec
 P2 17.60 usec
 PL1 -2.00 dB
 PL1W 54.93878937 W
 SFO1 100.6228298 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 0.00 dB
 PL12 14.30 dB
 PL2W 7.98779869 W
 PL12W 0.29677486 W
 SFO2 400.1316005 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6127728 MHz
 NDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

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



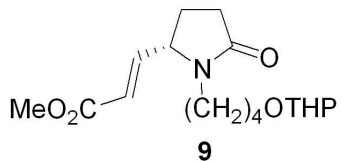
Current Data Parameters
 NAME 20091006 thp pure
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20091006
 Time 20.36
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 8223.685 Hz
 FIDRES 0.125483 Hz
 AQ 3.9846387 sec
 RG 228
 DW 60.800 usec
 DE 6.00 usec
 TE 673.2 K
 D1 1.00000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 12.80 usec
 PL1 -2.00 dB
 PL1W 12.65980816 W
 SFO1 400.1324710 MHz

F2 - Processing parameters
 SI 32768
 SF 400.1300099 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

174.69
165.97
146.57

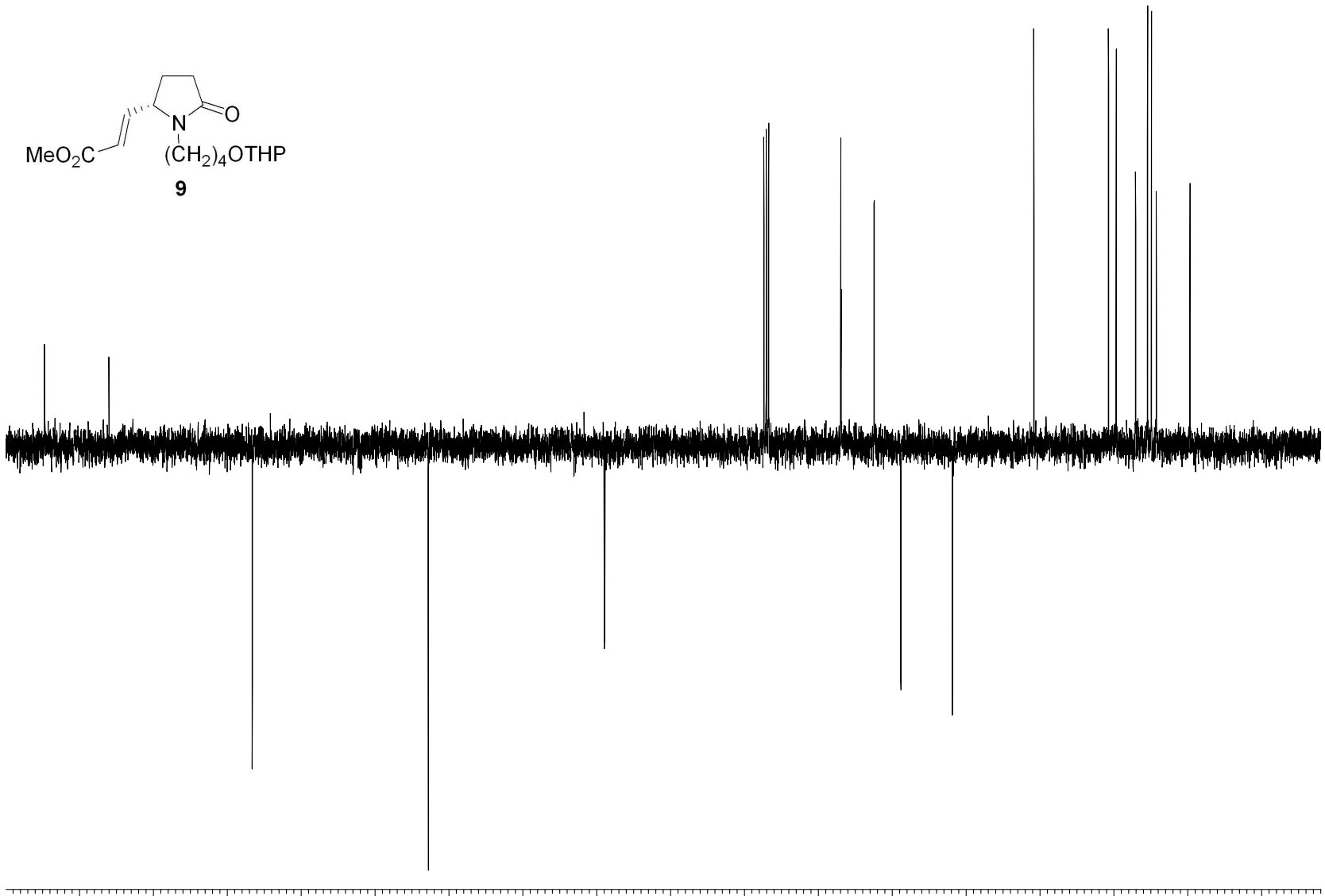


122.74

98.93
98.85

66.92
66.87
62.40
62.38
58.79
58.76
51.81

40.78
30.68
29.63
27.01
26.98
25.40
24.86
24.20
19.65



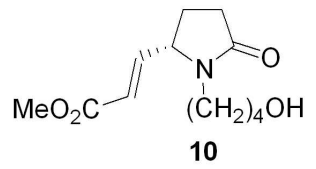
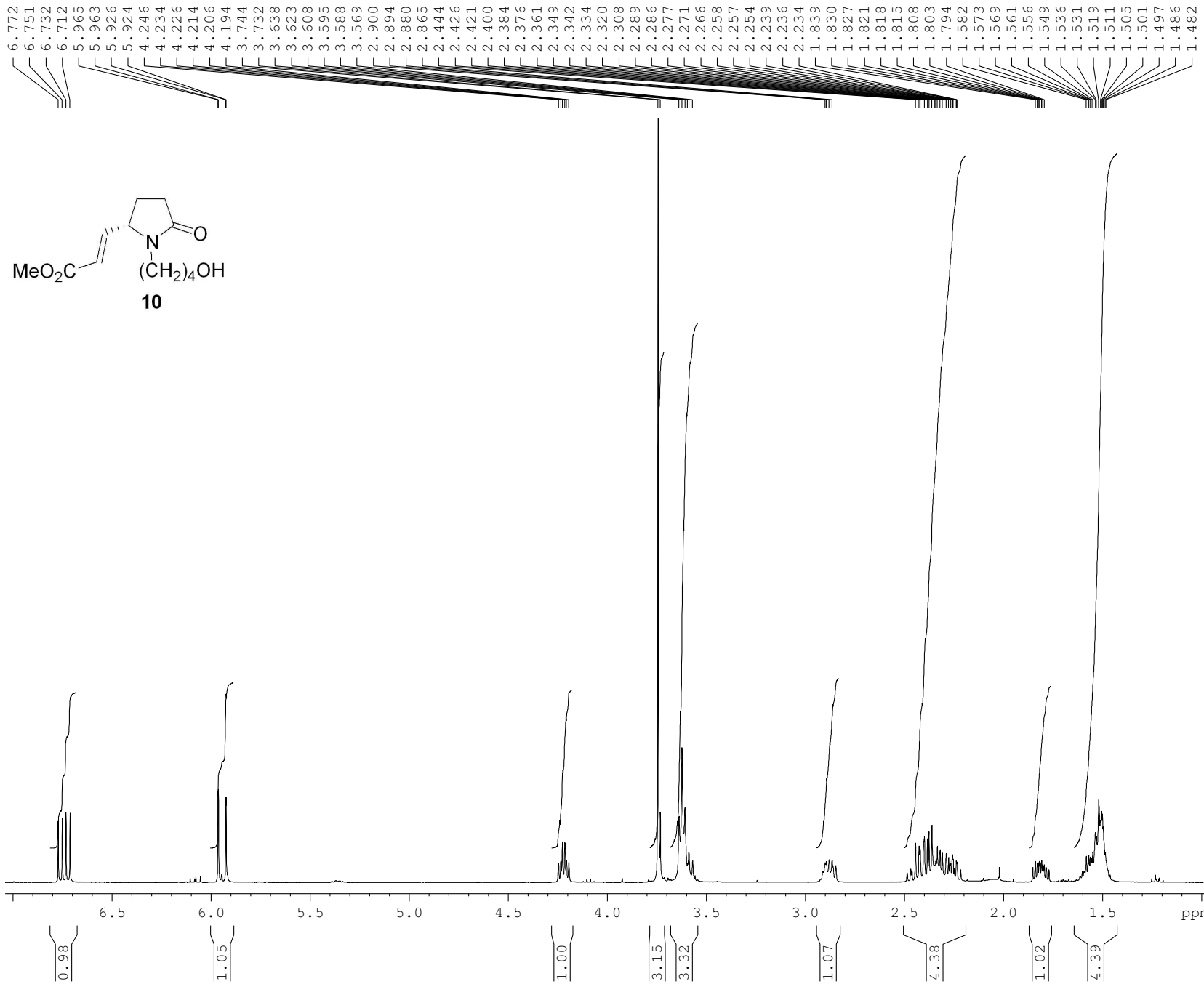
Current Data Parameters
NAME 20090930 pure carbon
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20090930
Time 22.20
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG jmod
TD 65536
SOLVENT CDC13
NS 135
DS 4
SMH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DW 20.800 usec
DE 6.00 usec
TE 673.2 K
CNST2 145.0000000
CNST11 1.0000000
D1 2.0000000 sec
D20 0.00689655 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 8.80 usec
P2 17.60 usec
PL1 -2.00 dB
PL1W 54.93878937 W
SFO1 100.6228298 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 14.30 dB
PL2W 7.98779869 W
PL12W 0.29677486 W
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127735 MHz
NDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



```

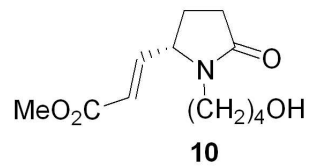
Current Data Parameters
NAME      20091003 wit alcohol
EXPNO    1
PROCNO   1

F2 - Acquisition Parameters
Date_    20091003
Time     13.59
INSTRUM  spect
PROBHD   5 mm PABBO BB-
PULPROG  zg30
TD       65536
SOLVENT  CDCl3
NS       16
DS       2
SWH      8223.685 Hz
FIDRES   0.125483 Hz
AQ       3.9846387 sec
RG       114
DW       60.800 usec
DE       6.00 usec
TE       673.2 K
D1       1.00000000 sec
TD0      1

===== CHANNEL f1 =====
NUC1     1H
P1       12.80 usec
PL1      -2.00 dB
PL1W     12.65980816 W
SFO1     400.1324710 MHz

F2 - Processing parameters
SI       32768
SF       400.1300096 MHz
WDW      EM
SSB      0
LB       0.30 Hz
GB       0
PC       1.00
  
```

175.00
165.98
146.40
122.88



62.08
58.91
51.83
40.68
29.64
29.46
24.86
23.84



Current Data Parameters
NAME 20091003 wit alcohol carbon
EXPNO 1
PROCNO 1

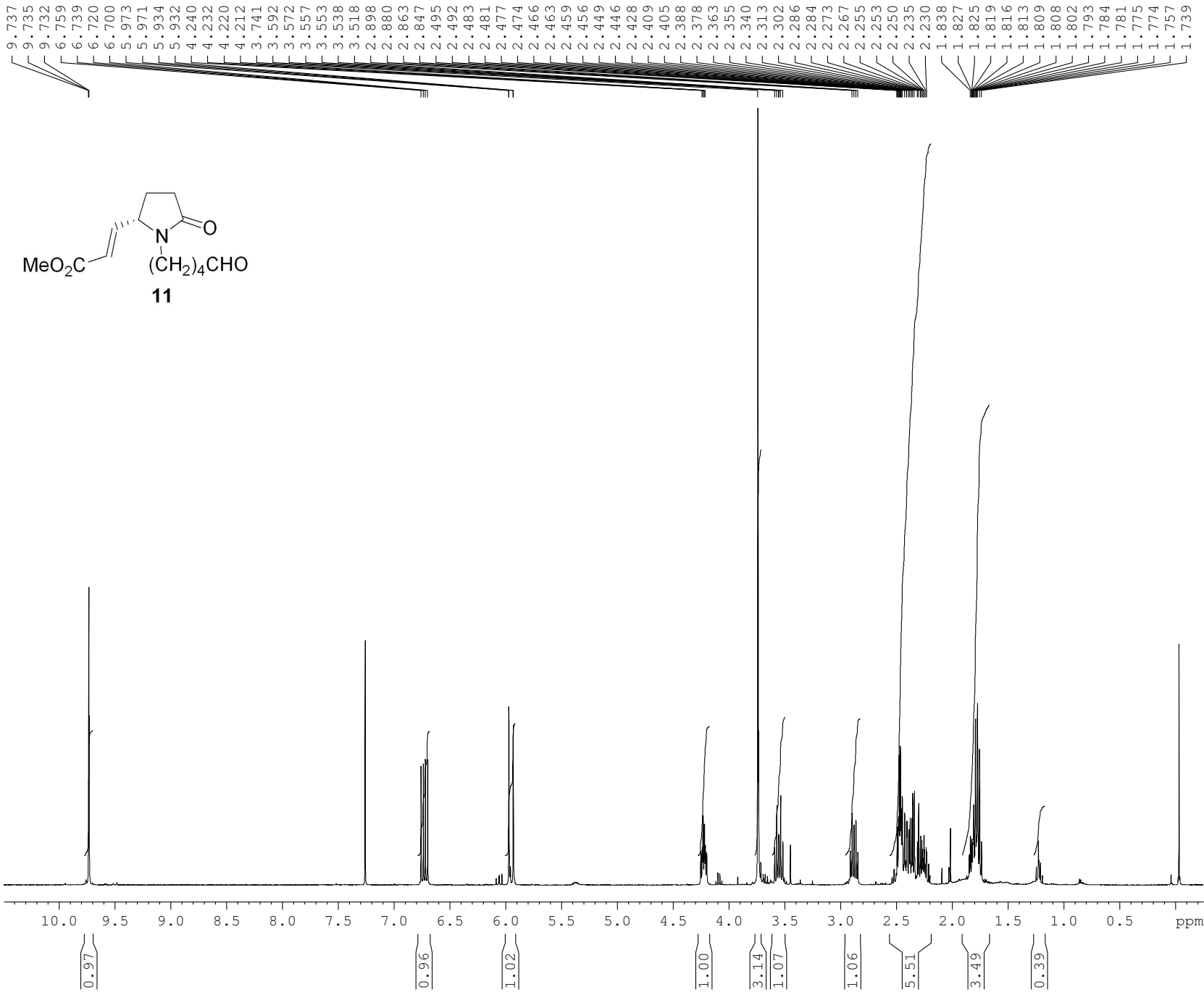
F2 - Acquisition Parameters
Date_ 20091003
Time 14.08
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG jmod
TD 65536
SOLVENT CDC13
NS 109
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DW 20.800 usec
DE 6.00 usec
TE 673.2 K
CNST2 145.000000
CNST11 1.000000
D1 2.0000000 sec
D20 0.00689655 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 8.80 usec
P2 17.60 usec
PL1 -2.00 dB
PL1W 54.93878937 W
SFO1 100.6228298 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 14.30 dB
PL2W 7.98779869 W
PL12W 0.29677486 W
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127745 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

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



```

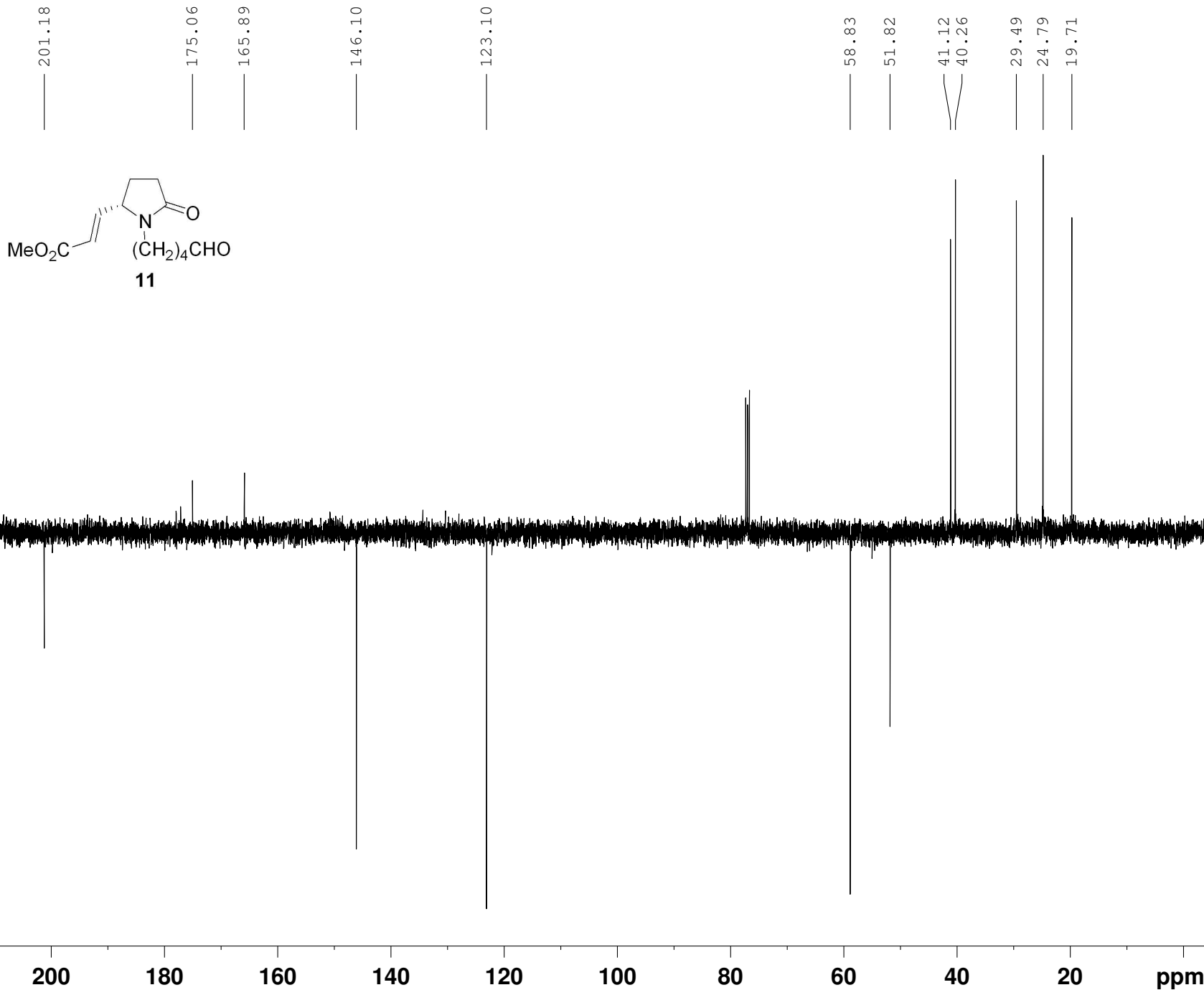
Current Data Parameters
NAME      20091005 pure
EXPNO    1
PROCNO   1

F2 - Acquisition Parameters
Date_    20091005
Time     18.39
INSTRUM spect
PROBHD   5 mm PABBO BB-
PULPROG zg30
TD       65536
SOLVENT  CDC13
NS       16
DS       2
SWH      8223.685 Hz
FIDRES   0.125483 Hz
AQ       3.9846387 sec
RG       144
DW       60.800 usec
DE       6.00 usec
TE       673.2 K
D1       1.00000000 sec
TD0      1

===== CHANNEL f1 =====
NUC1     1H
P1       12.80 usec
PL1      -2.00 dB
PL1W    12.65980816 W
SFO1    400.1324710 MHz

F2 - Processing parameters
SI       32768
SF       400.1300096 MHz
WDW      EM
SSB      0
LB       0.30 Hz
GB       0
PC       1.00

```

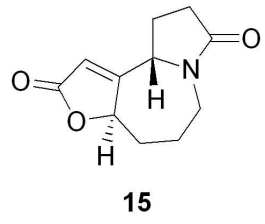
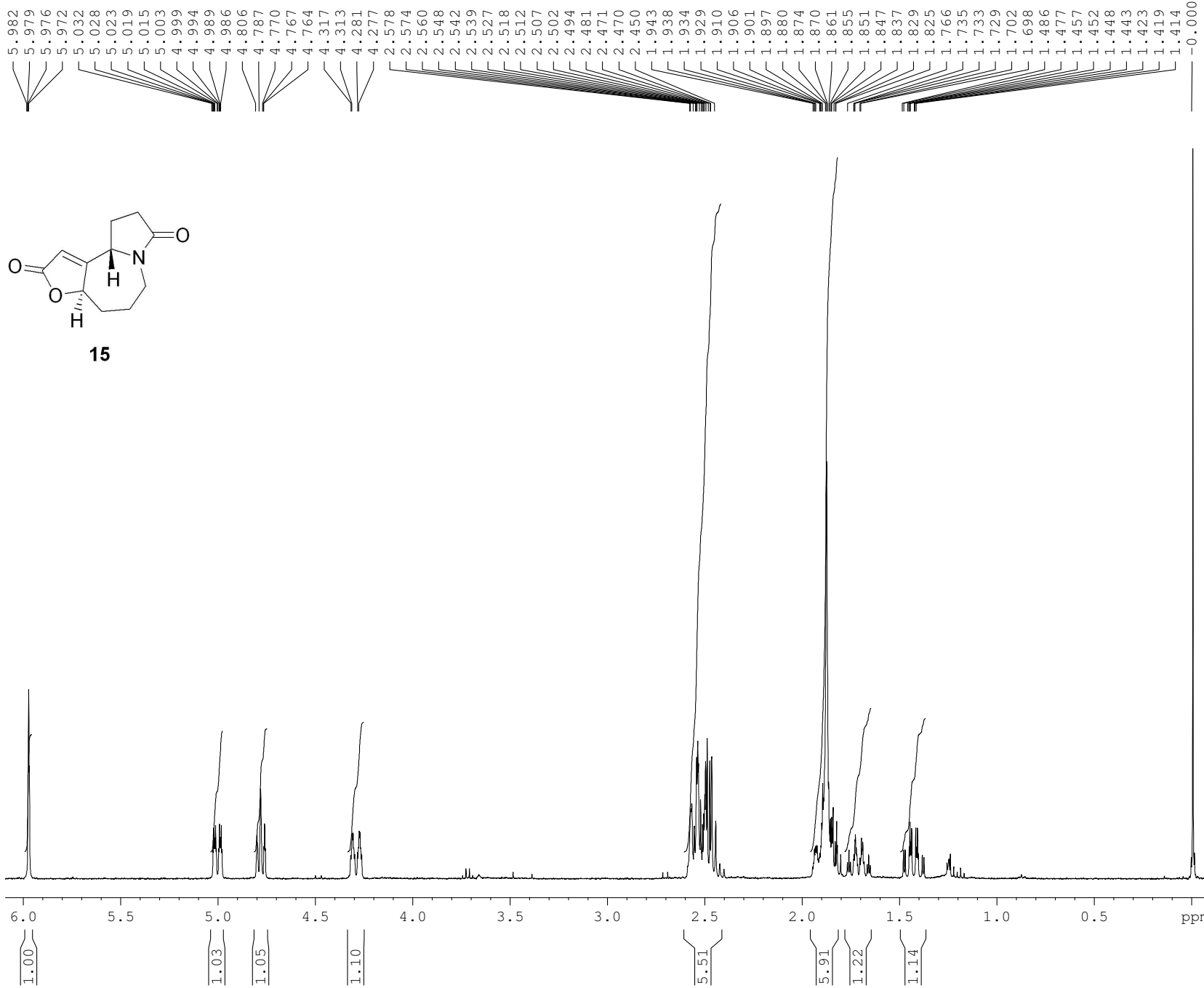
Current Data Parameters
 NAME 20091005 pure carbon
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20091005
 Time 19.07
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG jmod
 TD 65536
 SOLVENT CDCl3
 NS 63
 DS 4
 SWH 24038.461 Hz
 FIDRES 0.366798 Hz
 AQ 1.3631988 sec
 RG 2050
 DW 20.800 usec
 DE 6.00 usec
 TE 673.2 K
 CNST2 145.0000000
 CNST11 1.0000000
 D1 2.0000000 sec
 D20 0.00689655 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 13C
 P1 8.80 usec
 P2 17.60 usec
 PL1 -2.00 dB
 PL1W 54.93878937 W
 SFO1 100.6228298 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 0.00 dB
 PL12 14.30 dB
 PL2W 7.98779869 W
 PL12W 0.29677486 W
 SFO2 400.1316005 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6127758 MHz
 NDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40



Current Data Parameters
 NAME 100519 yellow2
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20100519
 Time 13.49
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 4
 DS 2
 SWH 8223.685 Hz
 FIDRES 0.125483 Hz
 AQ 3.9846387 sec
 RG 256
 DW 60.800 usec
 DE 6.00 usec
 TE 293.6 K
 D1 1.00000000 sec
 TD0 1

==== CHANNEL f1 =====
 NUC1 1H
 P1 13.30 usec
 PL1 -2.00 dB
 PL1W 12.65980816 W
 SFO1 400.1324710 MHz

F2 - Processing parameters
 SI 32768
 SF 400.1300098 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

174.48
174.13
171.71

115.88

82.85

58.12

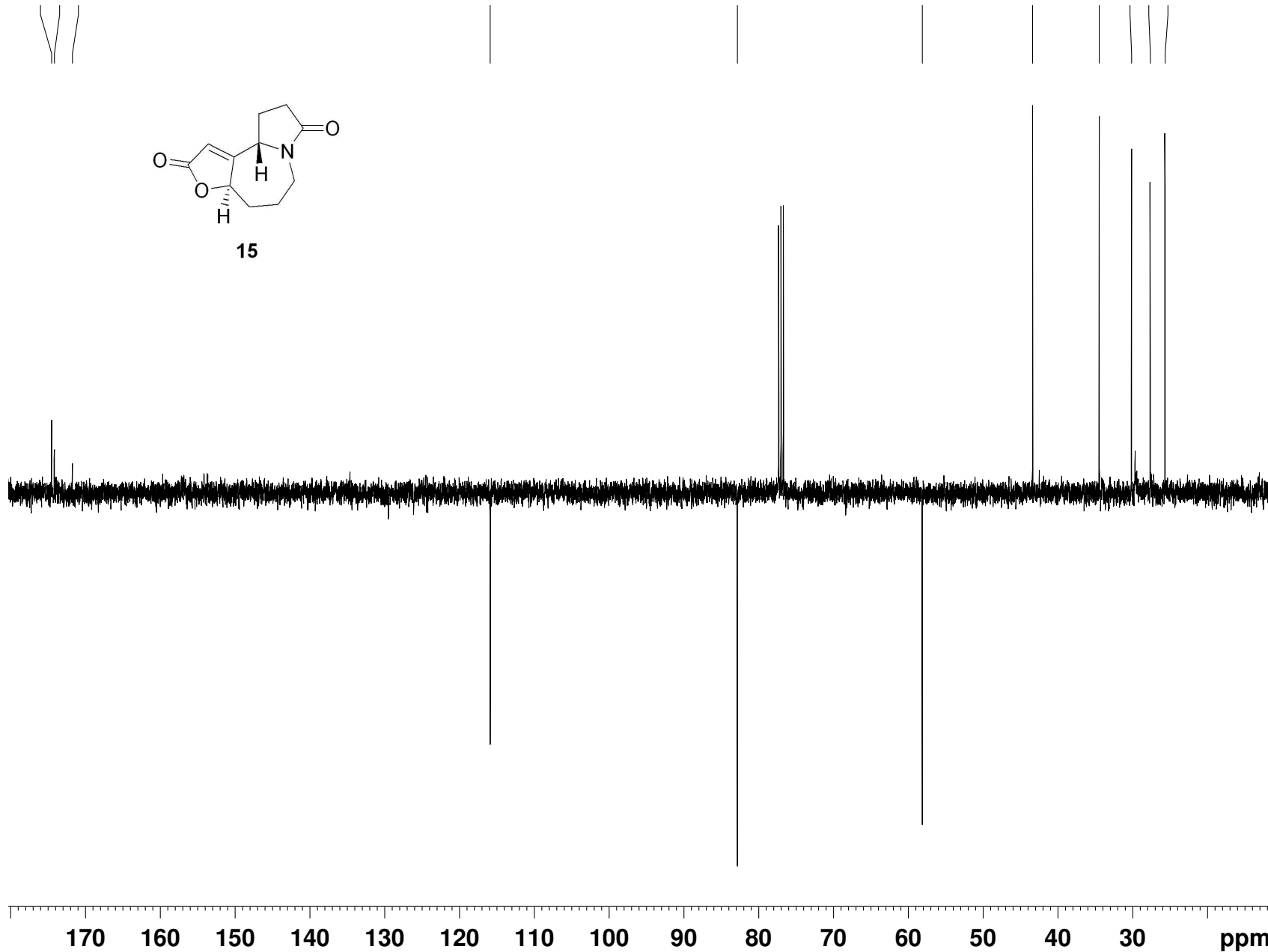
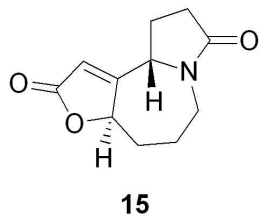
43.36

34.45

30.14

27.65

25.67



Current Data Parameters
NAME 100519 carbon
EXPNO 1
PROCNO 1

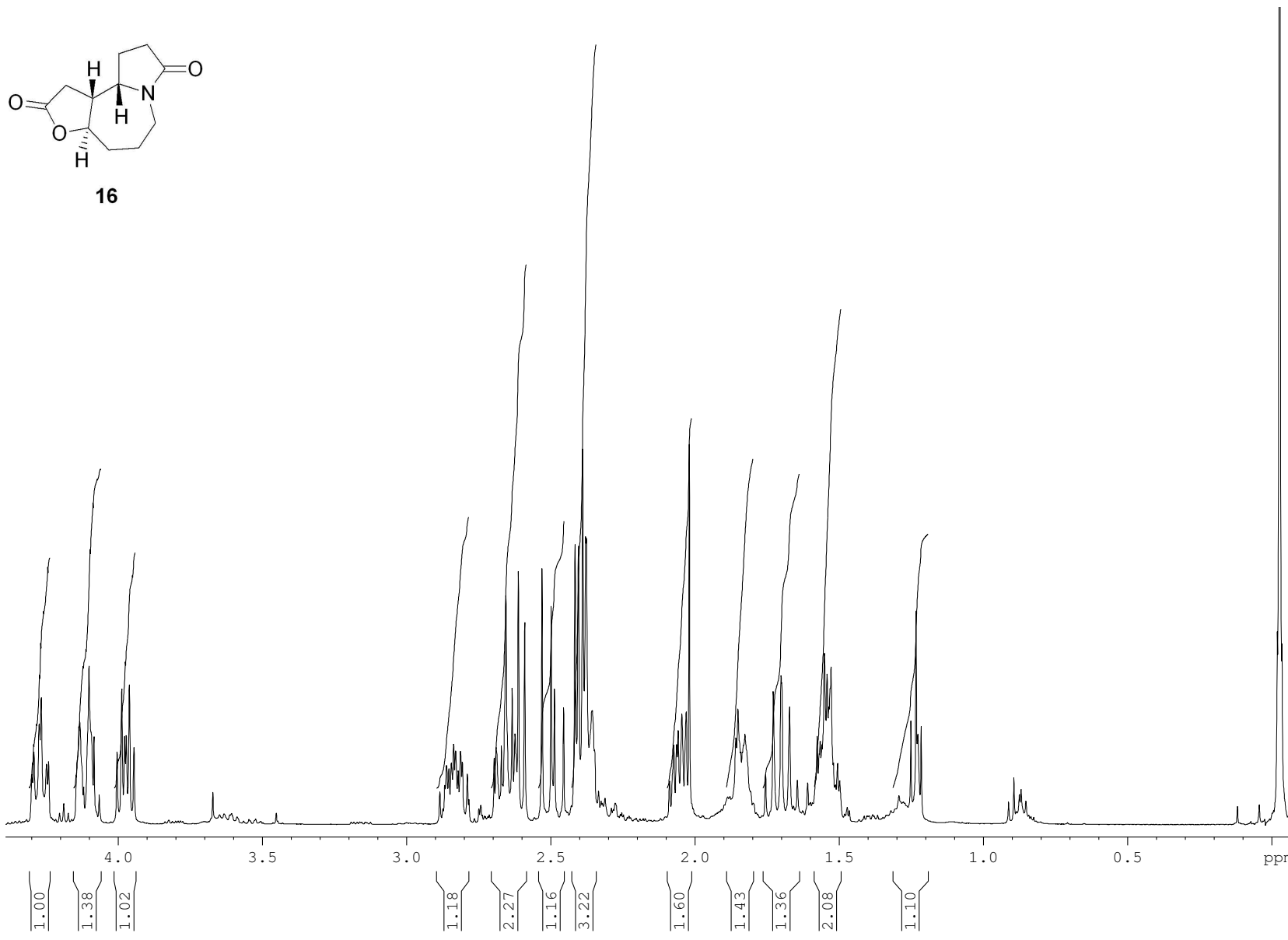
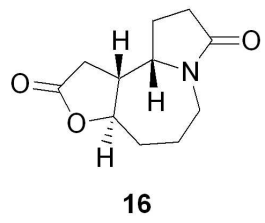
F2 - Acquisition Parameters
Date_ 20100519
Time 18.59
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG jmod
TD 65536
SOLVENT CDC13
NS 190
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DW 20.800 usec
DE 6.00 usec
TE 295.0 K
CNST2 145.0000000
CNST11 1.0000000
D1 2.00000000 sec
D20 0.00689655 sec
TD0 1

==== CHANNEL f1 =====
NUC1 13C
P1 8.60 usec
P2 17.20 usec
PL1 -2.00 dB
PL1W 54.93878937 W
SFO1 100.6228298 MHz

==== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -0.50 dB
PL12 14.10 dB
PL2W 8.96245766 W
PL12W 0.31076142 W
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127731 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

4.189
4.120
4.101
4.066
3.962
3.672
2.874
2.749
2.743
2.696
2.689
2.671
2.667
2.656
2.624
2.612
2.591
2.530
2.499
2.429
2.416
2.404
2.389
2.379
2.377
2.348
2.334
2.325
2.321
2.312
2.290
2.283
2.277
2.202
2.006
1.915
1.909
1.898
1.886
1.879
1.873
1.797
1.702
1.699
1.659
1.627
1.600
1.593
1.551
1.542
1.528
1.472
1.465
1.321
1.305
1.293
1.277
1.266
1.251
1.233
1.227
1.216
0.912
0.894
0.875
0.869
0.853
0.119
0.043
-0.007
-0.019
-0.027
-0.035



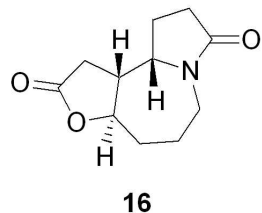
Current Data Parameters
NAME 100316 yellow
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20100316
Time 19.46
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 101
DW 60.800 usec
DE 6.00 usec
TE 293.6 K
D1 1.00000000 sec
TD0 1

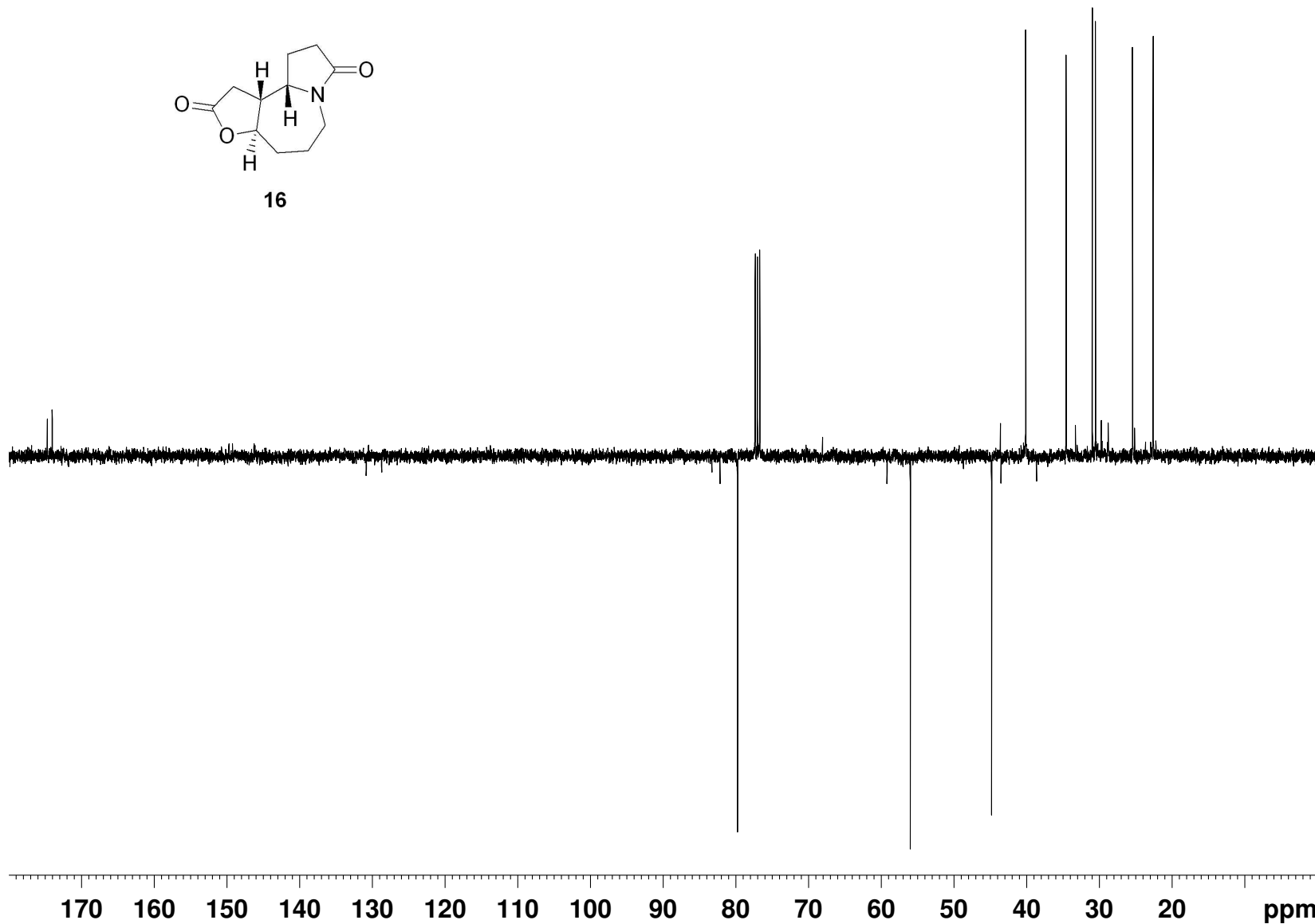
===== CHANNEL f1 =====
NUC1 1H
P1 13.30 usec
PL1 -2.00 dB
PL1W 12.65980816 W
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1300096 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

174.70
174.03



79.74
55.98
44.80
40.11
34.54
30.93
30.50
25.40
22.59



Current Data Parameters
NAME 100317 carbon
EXPNO 1
PROCNO 1

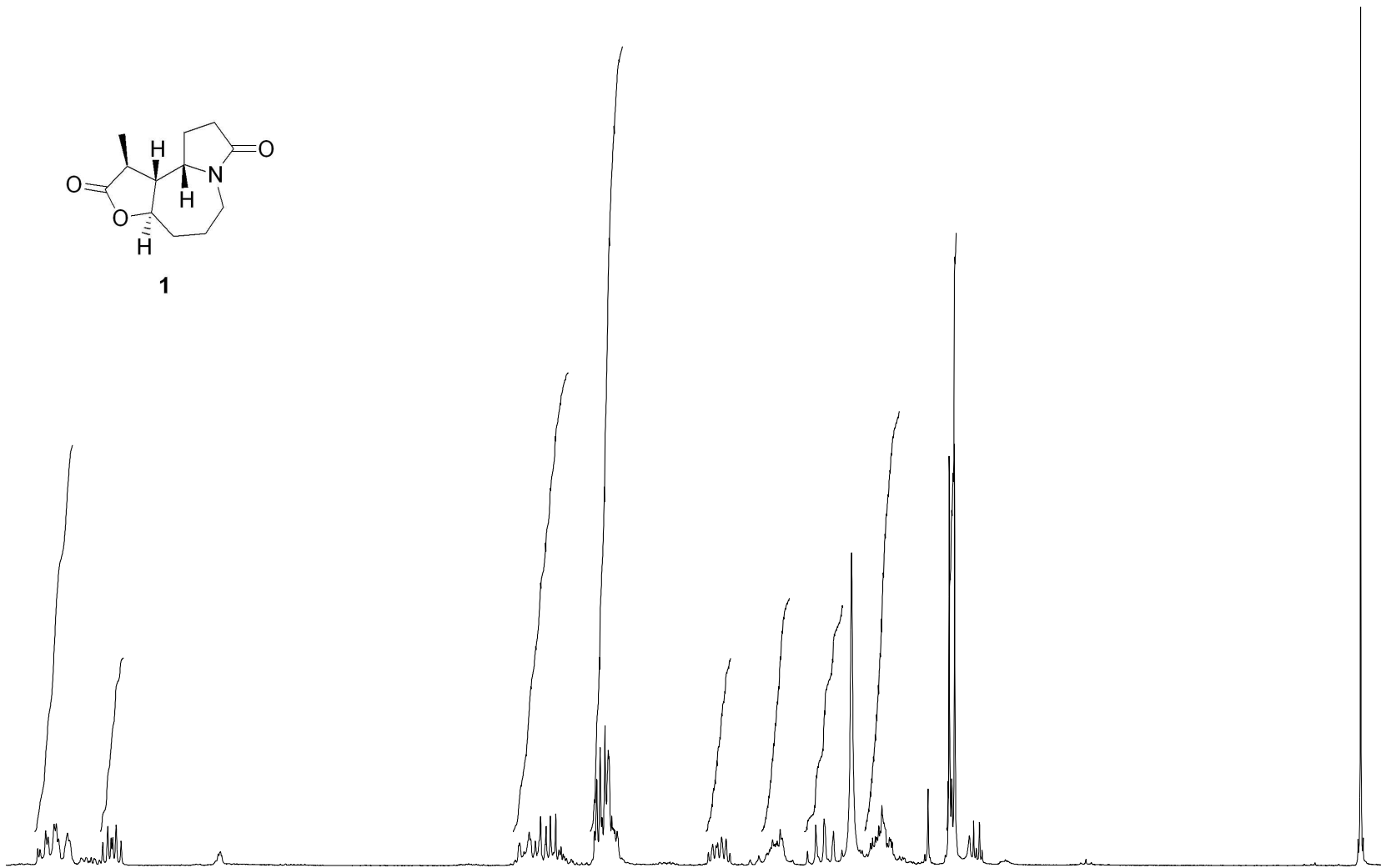
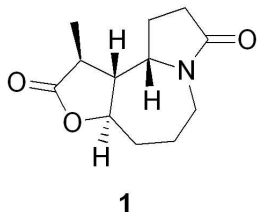
F2 - Acquisition Parameters
Date_ 20100317
Time 12.13
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG jmod
TD 65536
SOLVENT CDCl3
NS 276
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 2050
DW 20.800 usec
DE 6.00 usec
TE 294.9 K
CNST2 145.0000000
CNST11 1.0000000
D1 2.0000000 sec
D20 0.00689655 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 8.60 usec
P2 17.20 usec
PL1 -2.00 dB
PL1W 54.93878937 W
SFO1 100.6228298 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -0.50 dB
PL12 14.10 dB
PL2W 8.96245766 W
PL12W 0.31076142 W
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127766 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

4.231
4.224
4.206
4.198
4.179
4.172
4.165
4.136
4.130
4.023
4.007
3.996
3.991
3.980
3.964
2.688
2.673
2.657
2.652
2.637
2.620
2.603
2.589
2.578
2.572
2.561
2.555
2.447
2.440
2.429
2.423
2.414
2.404
2.401
2.392
2.389
2.386
2.382
2.375
2.083
2.069
2.058
2.053
2.040
2.026
2.014
1.894
1.887
1.883
1.877
1.870
1.866
1.862
1.853
1.846
1.765
1.738
1.712
1.682
1.655
1.563
1.557
1.548
1.544
1.537
1.532
1.527
1.509
1.502
1.498
1.494
1.311
1.294



Current Data Parameters
NAME 100903 yellow
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20100903
Time 20.25
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 256
DW 60.800 usec
DE 6.00 usec
TE 293.7 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 13.30 usec
PL1 -2.00 dB
PL1W 12.65980816 W
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1300098 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00