

Highly Regio- and Stereoselective Nitro-Oxoamination of Mono-Substituted Allenes

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Supporting Information

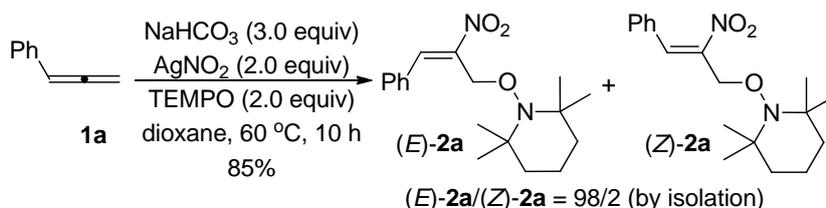
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General: Mono-substituted allenes were prepared according to the literature procedure.¹ Dioxane was distilled from Na wire/benzophenone. The other commercially available chemicals were purchased and used without additional purification. The reactions were performed under an atmosphere of nitrogen using standard Schlenk tubes unless otherwise stated. Petroleum ether with a boiling point ranging from 30 to 60 °C was used. Flash-column chromatography was carried out with silica gel H (10–40 μ). ¹H, ¹³C, and ¹⁹F NMR spectra were recorded with a Bruker AN 300 MHz spectrometer. ¹H NMR spectra (300 MHz) were recorded using TMS (δ 0 ppm) or CDCl₃ (δ 7.26 ppm) as the internal standard. ¹³C NMR spectra (75 MHz) were recorded using CDCl₃ as the internal standard (δ 77.00 ppm). ¹⁹F NMR spectra (282 MHz) were recorded using CFCl₃ as the internal standard (δ 0 ppm). IR spectra were recorded with a Perkin-Elmer 983G instrument. ESI-Mass spectrometry was performed with an Agilent 1100 LC/MSD SL system. ESI-High-resolution mass spectrometry was determined with a Bruker Daltonics APEXIIITM ESI-FTICRMS instrument. The configuration of the C=C bond in (*E*)-**2a** ~ (*E*)-**2v**, (*Z*)-**2f**, (*Z*)-**2h**, (*Z*)-**2i**, (*Z*)-**2j**, (*Z*)-**2n**, (*Z*)-**2s** and (*Z*)-**2v** were established by the NOE study.

Nitro-oxoamination reactions of mono-allenes without degassing operation

1. (*E*)- and (*Z*)-3-(2,2,6,6-Tetramethylpiperidinyloxy)-2-nitro-1-phenylprop-1-ene
(*E*)- and (*Z*)-**2a** (xc-9-176-1)



Typical Procedure I: To a dried Schlenk tube were added 2,2,6,6-tetramethyl-1-piperidineoxyl (TEMPO, 98%, 0.3193 g, 2.01 mmol), AgNO₂ (99%, 0.3121 g, 2.01 mmol), NaHCO₃ (0.2528 g, 3.01 mmol), 1-phenylpropa-1,2-diene **1a** (0.1162 g, 1.00 mmol), and 10 mL of anhydrous 1,4-dioxane at room temperature under N₂ atmosphere. The resulting mixture was then placed in a pre-heated oil bath of 60 °C and stirred for 10 h as monitored by TLC. After cooling to room temperature, the crude reaction mixture was filtrated through a short column of silica gel (Et₂O 3 × 15 mL). After concentration, the ratio of (*E*)-**2a**/*Z*-**2a** was 97/3 as determined by the ¹H NMR analysis. Column chromatography on silica gel (eluent: petroleum ether/ethyl ether = 100/1 then petroleum ether/ethyl acetate = 100/1) afforded **2a** (0.2702 g, 85%, (*E*)/(*Z*) = 98/2) as an oil.

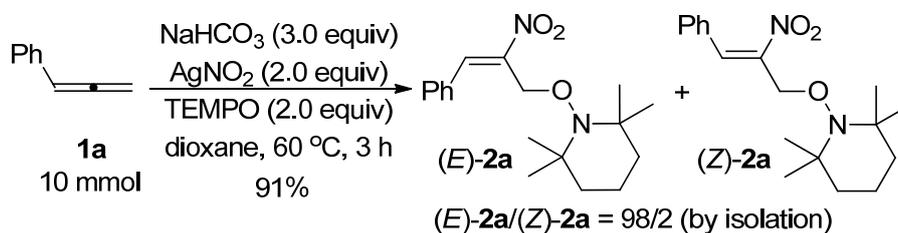
(*E*)-**2a**: ¹H NMR (300 MHz, CDCl₃) δ 8.15 (s, 1 H, =CH), 7.65-7.54 (m, 2 H, ArH), 7.49-7.40 (m, 3 H, ArH), 4.95 (s, 2 H, CH₂), 1.58-1.38 (m, 5 H, 2 × CH₂ and one proton of CH₂), 1.36-1.27 (m, 1 H, one proton of CH₂), 1.10 (s, 6 H, 2 × CH₃),

1.08 (s, 6 H, 2 × CH₃); ¹³C NMR (75 MHz, CDCl₃) δ 148.3, 137.8, 131.6, 130.4, 129.9, 128.7, 69.1, 60.0, 39.8, 32.8, 20.0, 16.9; IR (neat) ν (cm⁻¹) 3062, 2972, 2932, 2870, 1646, 1601, 1527, 1468, 1451, 1376, 1325, 1261, 1243, 1210, 1184, 1133, 1030; MS (ESI): *m/z* = 319 [M+H]⁺; HRMS (ESI): calcd. for C₁₈H₂₇N₂O₃ [M+H]⁺ 319.2016; found 319.2014. The following signals are discernible for (*Z*)-**2a**: ¹H NMR (300 MHz, CDCl₃) δ 6.68 (s, 1 H, =CH), 4.75 (s, 2 H, CH₂).

The following compounds were prepared following Typical Procedure I.

A 10 mmol scale nitro-oxoamination reaction of **1a**.

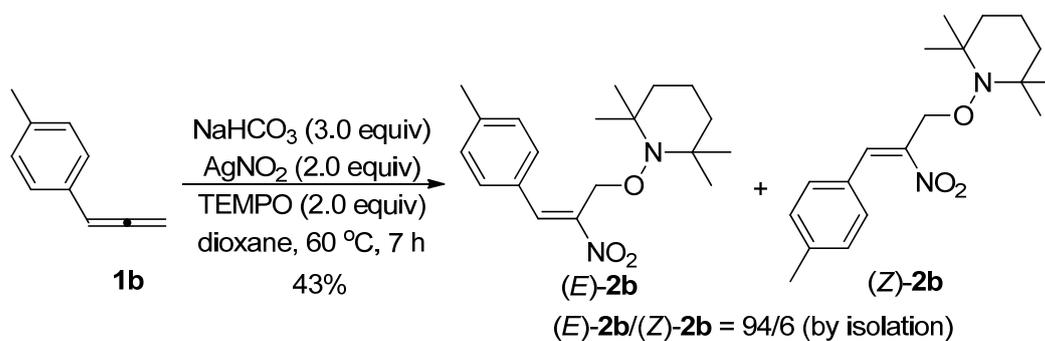
(*E*)- and (*Z*)-3-(2,2,6,6-Tetramethylpiperidinyloxy)-2-nitro-1-phenylprop-1-ene
(*E*)- and (*Z*)-**2a** (xc-10-159, 9-133)



The reaction of 1-phenylprop-1,2-diene **1a** (1.1615 g, 10.01 mmol), TEMPO (98%, 3.1845 g, 20.01 mmol), AgNO₂ (99%, 3.1112 g, 20.00 mmol), and NaHCO₃ (2.5213 g, 30.02 mmol) in dioxane (80 mL) at 60 °C for 3 h afforded a crude product. The ratio of (*E*)-**2a**/(*Z*)-**2a** was 98/2 as determined by the ¹H NMR analysis of the crude product. Further purification via column chromatography on silica gel (eluent: petroleum ether/ethyl ether = 150/1) afforded **2a** (2.8910 g, 91%, (*E*)/(*Z*) > 98/2) as an oil. (*E*)-**2a**: ¹H NMR (300 MHz, CDCl₃) δ 8.17 (s, 1 H, =CH), 7.67-7.53 (m, 2 H, ArH), 7.52-7.38 (m, 3 H, ArH), 4.96 (s, 2 H, CH₂), 1.57-1.23 (m, 6 H, 3 × CH₂), 1.11

(s, 6 H, 2 × CH₃), 1.08 (s, 6 H, 2 × CH₃); ¹³C NMR (75 MHz, CDCl₃) δ 148.3, 137.9, 131.6, 130.4, 129.9, 128.7, 69.1, 60.0, 39.8, 32.8, 20.0, 16.9; Elemental analysis calcd for C₁₈H₂₆N₂O₃: N, 8.80; C, 67.90; H, 8.23; Found: N, 8.84; C, 67.96; H, 8.03. The following signals are discernible for (*Z*)-**2a**: ¹H NMR (300 MHz, CDCl₃) δ 6.68 (s, 1 H, =CH), 4.74 (s, 2 H, CH₂).

2. (*E*)- and (*Z*)-1-(4-Methylphenyl)-3-(2,2,6,6-tetramethylpiperidinyl-1-oxy)-2-nitro prop-1-ene ((*E*)- and (*Z*)-**2b**) (xc-10-177-1,2)

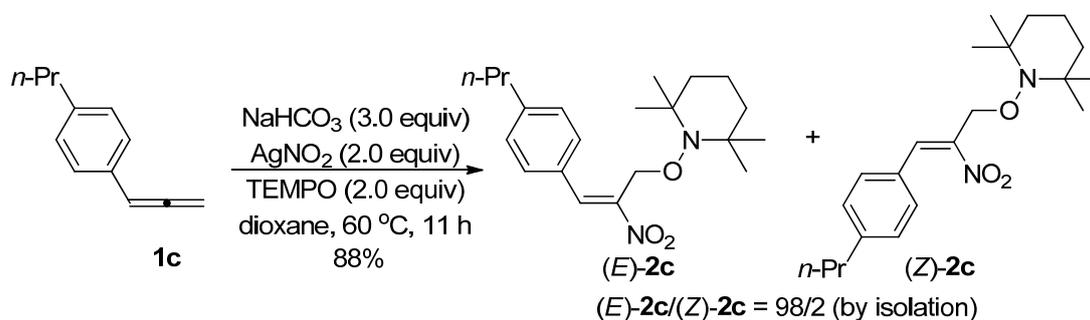


The reaction of 1-(4-methylphenyl)propa-1,2-diene **1b** (0.1302 g, 1.00 mmol), TEMPO (98%, 0.3197 g, 2.01 mmol), AgNO₂ (99%, 0.3115 g, 2.00 mmol), and NaHCO₃ (0.2522 g, 3.00 mmol) in dioxane (10 mL) at 60 °C for 7 h afforded a crude product. The ratio of (*E*)-**2b**/*Z*)-**2b** was 96/4 as determined by the ¹H NMR analysis of the crude product. Further purification via column chromatography on silica gel (eluent: petroleum ether/ethyl acetate = 150/1) afforded **2b** (0.1471 g, (*E*)/(*Z*) = 94/6; purity of **2b**: 96% as determined by using mesitylene as the internal standard, 43% yield) as an oil.

(*E*)-**2b**: ¹H NMR (300 MHz, CDCl₃) δ 8.15 (s, 1 H, =CH), 7.55 (d, *J* = 8.1 Hz, 2

H, ArH), 7.26 (d, $J = 8.1$ Hz, 2 H, ArH), 4.98 (s, 2 H, CH₂), 2.41 (s, 3 H, CH₃), 1.54-1.38 (m, 5 H, 2 × CH₂ and one proton of CH₂), 1.38-1.27 (m, 1 H, one proton of CH₂), 1.14 (s, 6 H, 2 × CH₃), 1.09 (s, 6 H, 2 × CH₃); ¹³C NMR (75 MHz, CDCl₃) δ 147.6, 141.2, 138.2, 130.3, 129.6, 128.8, 69.4, 60.1, 39.9, 33.0, 21.5, 20.1, 17.0; IR (neat) ν (cm⁻¹) 2974, 2933, 2869, 1644, 1608, 1557, 1526, 1468, 1454, 1376, 1361, 1322, 1243, 1186, 1132, 1036; MS (ESI): $m/z = 333$ [M+H]⁺; HRMS (ESI): calcd. for C₁₉H₂₉N₂O₃ [M+H]⁺ 333.2173; found 333.2164. The following signals are discernible for (*Z*)-**2b**: ¹H NMR (300 MHz, CDCl₃) δ 6.52 (s, 1 H, =CH), 4.73 (s, 2 H, CH₂).

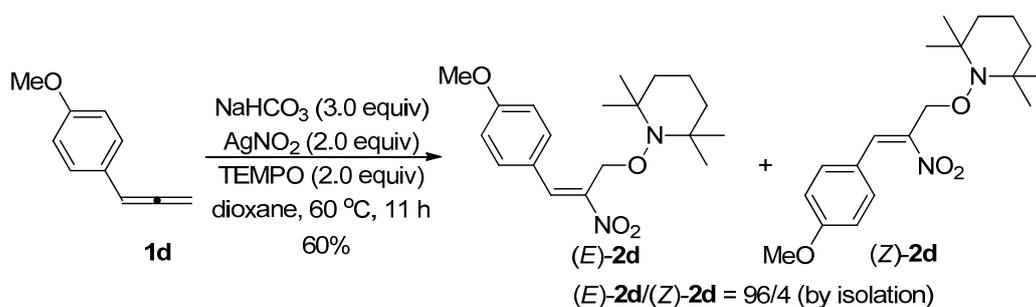
3. (*E*)- and (*Z*)-3-(2,2,6,6-Tetramethylpiperidinyl-1-oxy)-2-nitro-1-(4-propylphenyl)prop-1-ene ((*E*)- and (*Z*)-**2c**) (xc-9-184)



The reaction of 1-(4-propylphenyl)prop-1,2-diene **1c** (0.1585 g, 1.00 mmol), TEMPO (98%, 0.3189 g, 2.00 mmol), AgNO₂ (99%, 0.3121 g, 2.01 mmol), and NaHCO₃ (0.2532 g, 3.01 mmol) in dioxane (10 mL) at 60 °C for 11 h afforded a crude product. The ratio of (*E*)-**2c**/**(Z)**-**2c** was 96/4 as determined by the ¹H NMR analysis of the crude product. Further purification via column chromatography on silica gel (eluent: petroleum ether/ethyl ether= 100/1) afforded **2c** (0.3175 g, 88%, (*E*)/(*Z*) = 98/2) as an oil.

(E)-**2c**: ^1H NMR (300 MHz, CDCl_3) δ 8.08 (s, 1 H, =CH), 7.47 (d, $J = 8.4$ Hz, 2 H, ArH), 7.18 (d, $J = 8.1$ Hz, 2 H, ArH), 4.90 (s, 2 H, CH_2), 2.56 (t, $J = 7.5$ Hz, 2 H, CH_2), 1.58 (sext, $J = 7.4$ Hz, 2 H, CH_2), 1.49-1.30 (m, 5 H, $2 \times \text{CH}_2$ and one proton of CH_2), 1.29-1.20 (m, 1 H, one proton of CH_2), 1.05 (s, 6 H, $2 \times \text{CH}_3$), 1.01 (s, 6 H, $2 \times \text{CH}_3$), 0.87 (t, $J = 7.2$ Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, CDCl_3) δ 147.6, 145.9, 138.2, 130.2, 129.0, 128.9, 69.3, 60.0, 39.9, 37.8, 32.9, 24.2, 20.1, 17.0, 13.7; IR (neat) ν (cm^{-1}) 2931, 2871, 1644, 1608, 1527, 1467, 1376, 1360, 1323, 1260, 1243, 1186, 1133, 1034; MS (ESI): $m/z = 361$ $[\text{M}+\text{H}]^+$; HRMS (ESI): calcd. for $\text{C}_{21}\text{H}_{33}\text{N}_2\text{O}_3$ $[\text{M}+\text{H}]^+$ 361.2486; found 361.2476. The following signals are discernible for *(Z)*-**2c**: ^1H NMR (300 MHz, CDCl_3) δ 6.55 (s, 1 H, =CH), 4.65 (s, 2 H, CH_2).

4. *(E)*- and *(Z)*-1-(4-Methoxyphenyl)-3-(2,2,6,6-tetramethylpiperidinyl-1-oxy)-2-nitroprop-1-ene (*(E)*- and *(Z)*-**2d**) (xc-9-178,185)

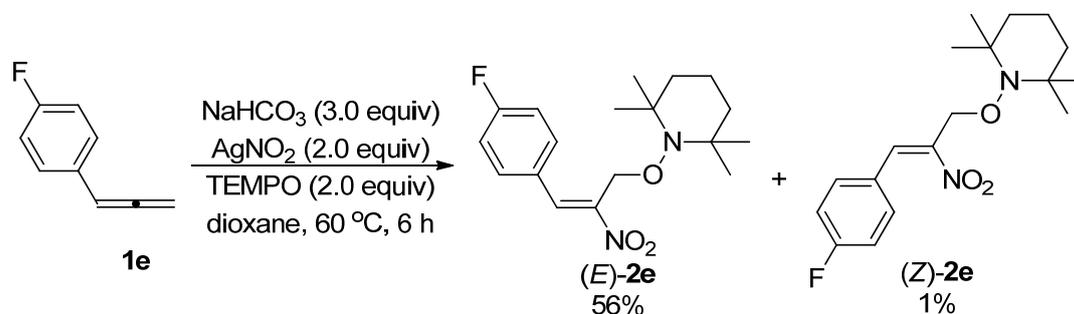


The reaction of 1-(4-methoxyphenyl)propa-1,2-diene **1d** (0.1458 g, 1.00 mmol), TEMPO (98%, 0.3188 g, 2.00 mmol), AgNO_2 (99%, 0.3123 g, 2.01 mmol), and NaHCO_3 (0.2533 g, 3.02 mmol) in dioxane (10 mL) at 60°C for 11 h afforded a crude product. The ratio of *(E)*-**2d**/*(Z)*-**2d** was 97/3 as determined by the ^1H NMR analysis of the crude product. Further purification via column chromatography on silica gel

(eluent: petroleum ether/ethyl acetate = 150/1, then petroleum ether/ethyl acetate = 100/1) afforded **2d** (0.2192 g, (*E*)/(*Z*)-**2d** = 96/4; purity of **2d**: 94% as determined by using mesitylene as the internal standard, 60% yield) as an oil.

(*E*)-**2d**: $^1\text{H NMR}$ (300 MHz, CDCl_3) δ 8.17 (s, 1 H, =CH), 7.68 (d, $J = 8.7$ Hz, 2 H, ArH), 6.99 (d, $J = 8.7$ Hz, 2 H, ArH), 5.03 (s, 2 H, CH_2), 3.89 (s, 3 H, CH_3), 1.57-1.45 (m, 5 H, $2 \times \text{CH}_2$ and one proton of CH_2), 1.40-1.31 (m, 1 H, one proton of CH_2), 1.18 (s, 6 H, $2 \times \text{CH}_3$), 1.13 (s, 6 H, $2 \times \text{CH}_3$); $^{13}\text{C NMR}$ (75 MHz, CDCl_3) δ 161.4, 146.0, 137.7, 132.0, 123.6, 113.9, 69.2, 59.6, 55.0, 39.5, 32.6, 19.7, 16.6; IR (neat) ν (cm^{-1}) 2972, 2933, 2843, 1641, 1604, 1520, 1466, 1376, 1306, 1260, 1180, 1132, 1030; MS (ESI): $m/z = 349$ $[\text{M}+\text{H}]^+$; HRMS (ESI): calcd. for $\text{C}_{19}\text{H}_{29}\text{N}_2\text{O}_4$ $[\text{M}+\text{H}]^+$ 349.2122; found 349.2109. The following signals are discernible for (*Z*)-**2d**: $^1\text{H NMR}$ (300 MHz, CDCl_3) δ 6.62 (s, 1 H, =CH), 4.74 (s, 2 H, CH_2), 3.84 (s, 3 H, CH_3).

5. (*E*)- and (*Z*)-1-(4-Fluorophenyl)-3-(2,2,6,6-tetramethylpiperidinyl-1-oxy)-2-nitro prop-1-ene ((*E*)- and (*Z*)-**2e**) (xc-9-183)



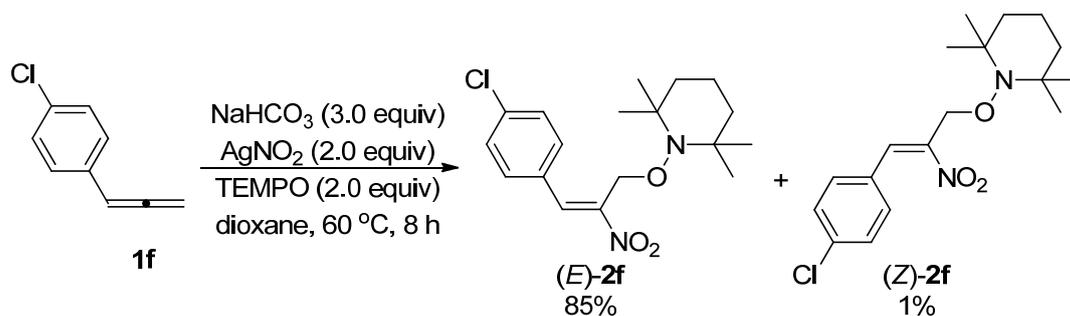
The reaction of 1-(4-fluorophenyl)propa-1,2-diene **1e** (0.1351 g, 1.01 mmol), TEMPO (98%, 0.3188 g, 2.00 mmol), AgNO_2 (99%, 0.3121 g, 2.01 mmol), and

NaHCO₃ (0.2529 g, 3.01 mmol) in dioxane (10 mL) at 60 °C for 6 h afforded a crude product. The ratio of (*E*)-**2e**/*Z*-**2e** was 96/4 as determined by the ¹H NMR analysis of the crude product. Further purification via column chromatography on silica gel (eluent: petroleum ether/ethyl ether = 100/1) afforded (*E*)-**2e** (0.1932 g, purity = 98% as determined by using mesitylene as the internal standard, 56% yield) as a liquid and (*Z*)-**2e** (0.0162 g, purity = 27% as determined by using mesitylene as the internal standard, 1% yield) as a solid.

(*E*)-**2e** (less polar): ¹H NMR (300 MHz, CDCl₃) δ 8.12 (s, 1 H, =CH), 7.72-7.53 (m, 2 H, ArH), 7.19-7.06 (m, 2 H, ArH), 4.95 (s, 2 H, CH₂), 1.60-1.38 (m, 5 H, 2 × CH₂ and one proton of CH₂), 1.37-1.26 (m, 1 H, one proton of CH₂), 1.11 (s, 6 H, 2 × CH₃), 1.08 (s, 6 H, 2 × CH₃); ¹³C NMR (75 MHz, CDCl₃) δ 164.0 (d, *J* = 251.3 Hz), 148.1, 136.8, 132.2 (d, *J* = 9.0 Hz), 127.8 (d, *J* = 3.6 Hz), 116.1 (d, *J* = 22.1 Hz), 69.2, 60.1, 39.9, 32.9, 20.1, 17.0; ¹⁹F NMR (282 MHz, CDCl₃) δ -108.9 ~ -109.4 (m, 1 F); IR (neat) ν (cm⁻¹) 2970, 2933, 1649, 1602, 1529, 1468, 1377, 1324, 1239, 1163, 1133, 1084, 1016; MS (ESI): *m/z* = 337 [M+H]⁺; HRMS (ESI): calcd. for C₁₈H₂₆FN₂O₃ [M+H]⁺ 337.1922; found 337.1919.

The following signals are discernible for (*Z*)-**2e** (more polar): ¹H NMR (300 MHz, CDCl₃) δ 6.57 (s, 1 H, =CH), 4.66 (s, 2 H, CH₂); ¹⁹F NMR (282 MHz, CDCl₃) δ -108.7 ~ -109.0 (m, 1 F).

6. (*E*)- and (*Z*)-1-(4-Chlorophenyl)-3-(2,2,6,6-tetramethylpiperidinyl-1-oxy)-2-nitro prop-1-ene ((*E*)- and (*Z*)-**2f**) (xc-10-162-1,2)



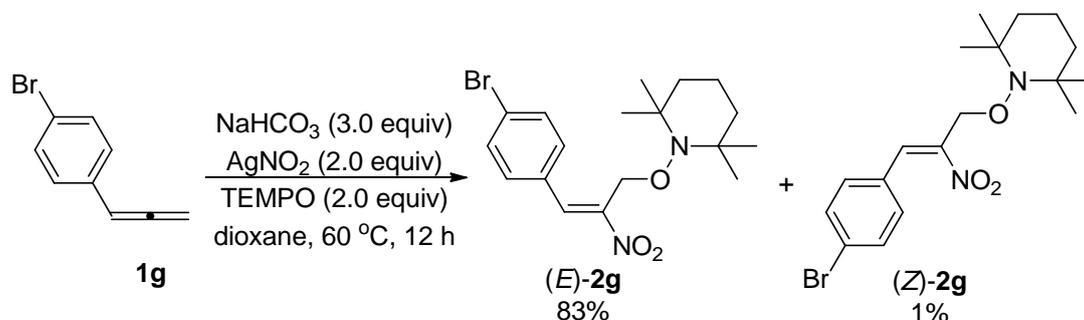
The reaction of 1-(4-chlorophenyl)propa-1,2-diene **1f** (0.1503 g, 1.00 mmol), TEMPO (98%, 0.3188 g, 2.00 mmol), AgNO_2 (99%, 0.3117 g, 2.00 mmol), and NaHCO_3 (0.2533 g, 3.02 mmol) in dioxane (10 mL) at 60 °C for 8 h afforded a crude product. The ratio of **(E)-2f**/**(Z)-2f** was 95/5 as determined by the ^1H NMR analysis of the crude product. Further purification via column chromatography on silica gel (eluent: petroleum ether/ethyl acetate = 150/1 then petroleum ether/ethyl acetate = 100/1) afforded **(E)-2f** (0.2997 g, 85%) as a solid and **(Z)-2f** (0.0112 g, purity = 47% as determined by using mesitylene as the internal standard, 1% yield) as an oil.

(E)-2f (less polar): m.p. 65.3-66.0 °C (*n*-hexane/ethyl acetate); ^1H NMR (300 MHz, CDCl_3) δ 8.10 (s, 1 H, =CH), 7.59 (d, $J = 8.4$ Hz, 2 H, ArH), 7.43 (d, $J = 8.4$ Hz, 2 H, ArH), 4.94 (s, 2 H, CH_2), 1.63-1.39 (m, 5 H, $2 \times \text{CH}_2$ and one proton of CH_2), 1.37-1.26 (m, 1 H, one proton of CH_2), 1.11 (s, 6 H, $2 \times \text{CH}_3$), 1.08 (s, 6 H, $2 \times \text{CH}_3$); ^{13}C NMR (75 MHz, CDCl_3) δ 148.6, 136.8, 136.6, 131.3, 130.1, 129.1, 69.2, 60.1, 39.9, 32.9, 20.1, 17.0; IR (KBr) ν (cm^{-1}) 2974, 2935, 2869, 1593, 1557, 1527, 1492, 1467, 1380, 1361, 1244, 1180, 1132, 1092, 1015; MS (ESI): $m/z = 353$ [$\text{M}(^{35}\text{Cl})+\text{H}$] $^+$, 355 [$\text{M}(^{37}\text{Cl})+\text{H}$] $^+$; Elemental analysis calcd for $\text{C}_{18}\text{H}_{25}\text{ClN}_2\text{O}_3$: N, 7.94; C, 61.27; H, 7.14; Found: N, 7.66; C, 61.06; H, 7.05.

The following signals are discernible for **(Z)-2f** (more polar): ^1H NMR (300 MHz,

CDCl₃) δ 7.34 (d, J = 8.7 Hz, 2 H, ArH), 7.25 (d, J = 7.5 Hz, 2 H, ArH), 6.64 (s, 1 H, =CH), 4.73 (s, 2 H, CH₂), 1.20 (s, 6 H, 2 \times CH₃), 1.11 (s, 6 H, 2 \times CH₃); IR (neat) ν (cm⁻¹) 2974, 2932, 2869, 1646, 1593, 1527, 1491, 1468, 1376, 1339, 1092, 1015; MS (ESI): m/z = 355 [M(³⁷Cl)+H]⁺, 353 [M(³⁵Cl)+H]⁺; HRMS (ESI): calcd. for C₁₈H₂₆ClN₂O₃ [M(³⁵Cl)+H]⁺ 353.1626; found 353.1621.

7. (*E*)- and (*Z*)-1-(4-Bromophenyl)-3-(2,2,6,6-tetramethylpiperidinyl-1-oxy)-2-nitro prop-1-ene ((*E*)- and (*Z*)-**2g**) (xc-10-005-2,3)



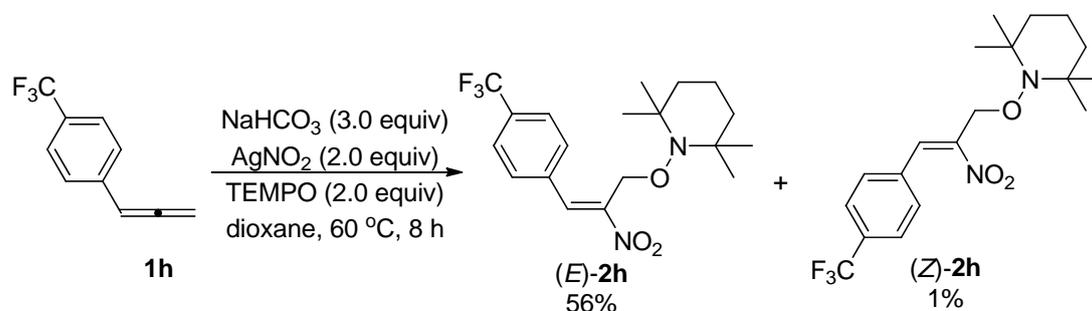
The reaction of 1-(4-bromophenyl)propa-1,2-diene **1g** (0.1959 g, 1.00 mmol), TEMPO (98%, 0.3192 g, 2.01 mmol), AgNO₂ (99%, 0.3125 g, 2.01 mmol), and NaHCO₃ (0.2515 g, 2.99 mmol) in dioxane (10 mL) at 60 °C for 12 h afforded a crude product. The ratio of (*E*)-**2g**/**(Z)-2g** was 97/3 as determined by the ¹H NMR analysis of the crude product. Further purification via column chromatography on silica gel (eluent: petroleum ether/ethyl ether = 100/1 then petroleum ether/ethyl acetate = 100/1) afforded (*E*)-**2g** (0.3295 g, 83%) as a solid and (*Z*)-**2g** (0.0390 g, purity = 10% as determined by using mesitylene as the internal standard, 1% yield) as an oil.

(*E*)-**2g** (less polar): m.p. 95.4-96.1 °C (*n*-hexane/ethyl acetate); ¹H NMR (300 MHz, CDCl₃) δ 8.07 (s, 1 H, =CH), 7.59 (d, J = 8.4 Hz, 2 H, ArH), 7.51 (d, J = 8.4 Hz,

2 H, ArH), 4.93 (s, 2 H, CH₂), 1.55-1.39 (m, 5 H, 2 × CH₂ and one proton of CH₂), 1.38-1.27 (m, 1 H, one proton of CH₂), 1.10 (s, 12 H, 4 × CH₃); ¹³C NMR (75 MHz, CDCl₃) δ 148.8, 136.6, 132.1, 131.5, 130.6, 125.2, 69.3, 60.2, 39.9, 33.0, 20.1, 17.0; IR (KBr) ν (cm⁻¹) 2973, 2932, 1646, 1588, 1531, 1488, 1468, 1451, 1375, 1361, 1325, 1261, 1243, 1209, 1183, 1133, 1075, 1012; MS (ESI): *m/z* = 397 [M(⁷⁹Br)+H]⁺, 399 [M(⁸¹Br)+H]⁺; Elemental analysis calcd for C₁₈H₂₅BrN₂O₃: N, 7.05; C, 54.41; H, 6.34; Found: N, 6.94; C, 54.55; H, 6.41.

The following signals are discernible for (*Z*)-**2g** (more polar): ¹H NMR (300 MHz, CDCl₃) δ 7.18 (d, *J* = 8.4 Hz, 2 H, ArH), 6.62 (s, 1 H, =CH), 4.73 (s, 2 H, CH₂).

8. (*E*)- and (*Z*)-1-(4-Trifluoromethylphenyl)-3-(2,2,6,6-tetramethylpiperidinyl-1-oxy)-2-nitroprop-1-ene ((*E*)- and (*Z*)-**2h**) (xc-10-166-1,2; xc-12-060-1,2)



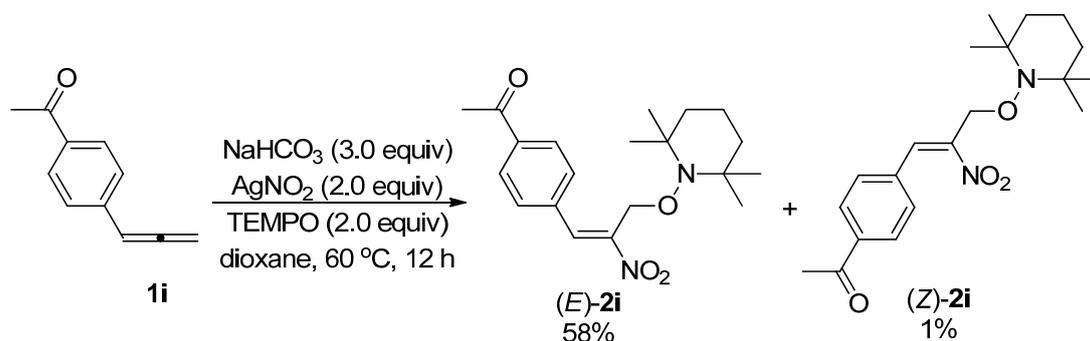
The reaction of 1-(4-trifluoromethylphenyl)prop-1,2-diene **1h** (0.1841 g, 1.00 mmol), TEMPO (98%, 0.3187 g, 2.00 mmol), AgNO₂ (99%, 0.3120 g, 2.01 mmol), and NaHCO₃ (0.2532 g, 3.01 mmol) in dioxane (10 mL) at 60 °C for 8 h afforded a crude product. The ratio of (*E*)-**2h**/**(Z)-2h** was 96/4 as determined by the ¹H NMR analysis of the crude product. Further purification via column chromatography on silica gel (eluent: petroleum ether/ethyl acetate = 150/1; then 100/1; and then 10/1)

afforded (*E*)-**2h** (0.2211 g, purity = 97%, 56%) as an oil and (*Z*)-**2h** (0.0325 g, purity = 15%, 1%) as an oil.

(*E*)-**2h** (less polar): ^1H NMR (300 MHz, CDCl_3) δ 8.14 (s, 1 H, =CH), 7.78-7.68 (m, 4 H, ArH), 4.93 (s, 2 H, CH_2), 1.56-1.40 (m, 5 H, $2 \times \text{CH}_2$ and one proton of CH_2), 1.35-1.27 (m, 1 H, one proton of CH_2), 1.07 (s, 12 H, $4 \times \text{CH}_3$); ^{13}C NMR (75 MHz, CDCl_3) δ 149.8, 135.9, 135.2, 132.0 (q, $J = 32.9$ Hz), 130.1, 125.7 (q, $J = 3.7$ Hz), 123.6 (q, $J = 270.6$ Hz), 69.0, 60.1, 39.8, 32.8, 20.1, 16.9; ^{19}F NMR (282 MHz, CDCl_3) δ -63.5 (s, 3 F); IR (neat) ν (cm^{-1}) 2975, 2935, 2869, 1618, 1533, 1469, 1376, 1361, 1324, 1262, 1170, 1131, 1069, 1019; MS (ESI): $m/z = 387$ [$\text{M}+\text{H}$] $^+$; HRMS (ESI): calcd. for $\text{C}_{19}\text{H}_{26}\text{F}_3\text{N}_2\text{O}_3$ [$\text{M}+\text{H}$] $^+$ 387.1890; found 387.1874.

The following signals are discernible for (*Z*)-**2h** (more polar): ^1H NMR (300 MHz, CDCl_3) δ 6.99 (s, 1 H, =CH), 4.92 (s, 2 H, CH_2).

9. (*E*)- and (*Z*)-1-(4-Acetylphenyl)-3-(2,2,6,6-tetramethylpiperidinyl-1-oxy)-2-nitro prop-1-ene ((*E*)- and (*Z*)-**2i**) (xc-11-039-1,2)



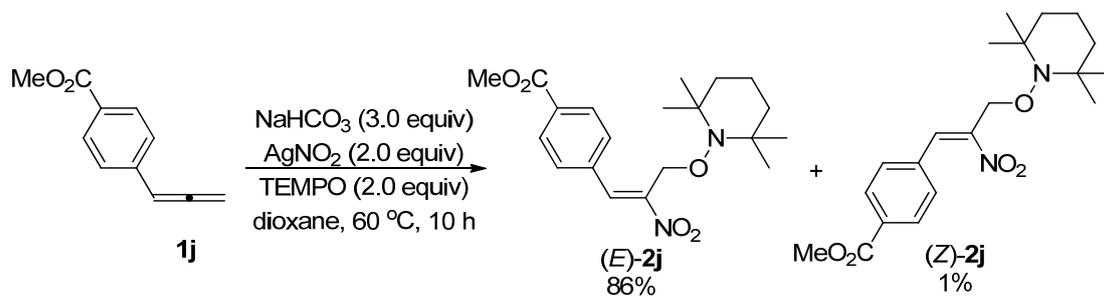
The reaction of 1-(4-acetylphenyl)propa-1,2-diene **1i** (0.1587 g, 1.00 mmol), TEMPO (98%, 0.3188 g, 2.00 mmol), AgNO_2 (99%, 0.3115 g, 2.00 mmol), and

NaHCO₃ (0.2527 g, 3.01 mmol) in dioxane (10 mL) at 60 °C for 12 h afforded a crude product. The ratio of (*E*)-**2i**/*(Z)*-**2i** was 97/3 as determined by the ¹H NMR analysis of the crude product. Further purification via column chromatography on silica gel (eluent: petroleum ether/ethyl acetate = 20/1 then petroleum ether/ethyl acetate = 15/1) afforded (*E*)-**2i** (0.2101 g, 58%) as a solid and (*Z*)-**2i** (0.0163 g, purity = 13% as determined by using mesitylene as the internal standard, 1% yield) as an oil.

(*E*)-**2i** (less polar): m.p. 71.3-71.8 °C (*n*-hexane/ethyl acetate); ¹H NMR (300 MHz, CDCl₃) δ 8.15 (s, 1 H, =CH), 8.03 (d, *J* = 8.1 Hz, 2 H, ArH), 7.71 (d, *J* = 8.4 Hz, 2 H, ArH), 4.94 (s, 2 H, CH₂), 2.65 (s, 3 H, CH₃), 1.54-1.39 (m, 5 H, 2 × CH₂ and one proton of CH₂), 1.37-1.25 (m, 1 H, one proton of CH₂), 1.09 (s, 6 H, 2 × CH₃), 1.07 (s, 6 H, 2 × CH₃); ¹³C NMR (75 MHz, CDCl₃) δ 197.2, 149.6, 138.0, 136.22, 136.17, 130.1, 128.6, 69.1, 60.2, 39.9, 32.9, 26.7, 20.1, 17.0; IR (KBr) ν (cm⁻¹) 2973, 2932, 1687, 1605, 1529, 1468, 1360, 1326, 1265, 1133, 1016; MS (ESI): *m/z* = 361 [M+H]⁺; Elemental analysis calcd for C₂₀H₂₈N₂O₄: N, 7.50; C, 66.64; H, 7.90; Found: N, 7.50; C, 66.57; H, 7.90.

The following signals are discernible for (*Z*)-**2i** (more polar): ¹H NMR (300 MHz, CDCl₃) δ 7.95 (d, *J* = 8.1 Hz, 2 H, ArH), 7.40 (d, *J* = 7.8 Hz, 2 H, ArH), 6.75 (s, 1 H, =CH), 4.77 (s, 2 H, CH₂), 2.61 (s, 3 H, CH₃), 1.35 (s, 6 H, 2 × CH₃), 1.26 (s, 6 H, 2 × CH₃).

10. (*E*)- and (*Z*)-1-(4-Methoxycarbonylphenyl)-3-(2,2,6,6-tetramethylpiperidinyl-1-oxy)-2-nitroprop-1-ene ((*E*)- and (*Z*)-**2j**) (xc-11-038-1,2)



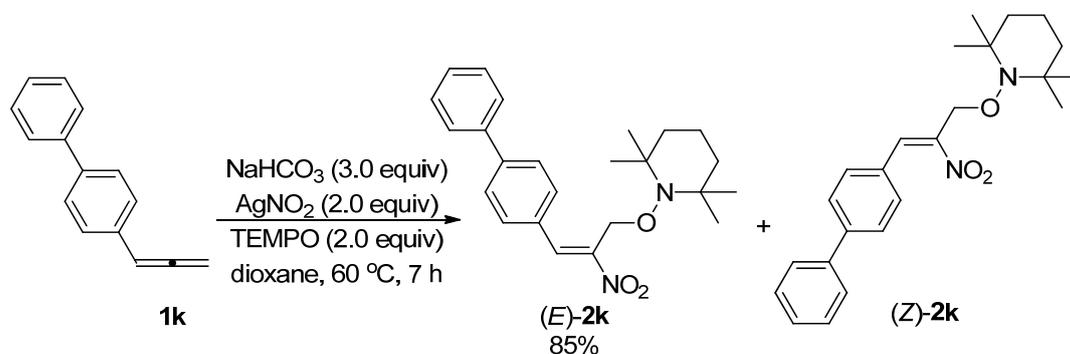
The reaction of 1-(4-methoxycarbonylphenyl)propa-1,2-diene **1j** (0.1745 g, 1.00 mmol), TEMPO (98%, 0.3192 g, 2.01 mmol), AgNO₂ (99%, 0.3118 g, 2.00 mmol), and NaHCO₃ (0.2527 g, 3.01 mmol) in dioxane (10 mL) at 60 °C for 10 h afforded a crude product. The ratio of *(E)*-**2j**/*(Z)*-**2j** was 98/2 as determined by the ¹H NMR analysis of the crude product. Further purification via column chromatography on silica gel (eluent: petroleum ether/ethyl acetate = 20/1) afforded *(E)*-**2j** (0.3306 g, purity = 98% as determined by using mesitylene as the internal standard, 86% yield) as a solid and *(Z)*-**2j** (0.0102 g, purity = 41% as determined by using mesitylene as the internal standard, 1% yield) as an oil.

(E)-**2j** (less polar): m.p. 99.7-101.0 °C (*n*-hexane/ethyl acetate); ¹H NMR (300 MHz, CDCl₃) δ 8.15 (s, 1 H, =CH), 8.11 (d, *J* = 8.4 Hz, 2 H, ArH), 7.68 (d, *J* = 8.1 Hz, 2 H, ArH), 4.94 (s, 2 H, CH₂), 3.96 (s, 3 H, CH₃), 1.57-1.23 (m, 6 H, 3 × CH₂), 1.09 (s, 6 H, 2 × CH₃), 1.07 (s, 6 H, 2 × CH₃); ¹³C NMR (75 MHz, CDCl₃) δ 166.1, 149.3, 136.3, 135.9, 131.4, 129.72, 129.66, 68.9, 60.0, 52.3, 39.7, 32.7, 19.9, 16.8; IR (KBr) ν (cm⁻¹) 2977, 2933, 2869, 2843, 1727, 1648, 1608, 1531, 1436, 1361, 1326, 1280, 1185, 1133, 1110, 1020; MS (ESI): *m/z* = 377 [M+H]⁺; Elemental analysis calcd for C₂₀H₂₈N₂O₅: N, 7.44; C, 63.81; H, 7.50; Found: N, 7.27; C, 63.92; H, 7.55.

The following signals are discernible for *(Z)*-**2j** (more polar): ¹H NMR (300 MHz, S15

CDCl₃) δ 8.03 (d, J = 8.4 Hz, 2 H, ArH), 7.37 (d, J = 8.4 Hz, 2 H, ArH), 6.75 (s, 1 H, =CH), 4.76 (s, 2 H, CH₂), 3.93 (s, 3 H, CH₃); ¹³C NMR (75 MHz, CDCl₃) δ 129.9, 128.3, 125.9, 60.3, 39.7; IR (neat) ν (cm⁻¹) 3012, 2927, 2852, 1728, 1608, 1532, 1436, 1376, 1279, 1109, 1016; MS (ESI): m/z = 377 [M+H]⁺; HRMS (ESI): calcd. for C₂₀H₂₉N₂O₅ [M+H]⁺ 377.2071; found 377.2058.

11. (*E*)- and (*Z*)-3-(2,2,6,6-Tetramethylpiperidinyl-1-oxy)-2-nitro-1-((1,1'-biphenyl)-4-yl)prop-1-ene ((*E*)- and (*Z*)-**2k**) (xc-10-172-1,2)



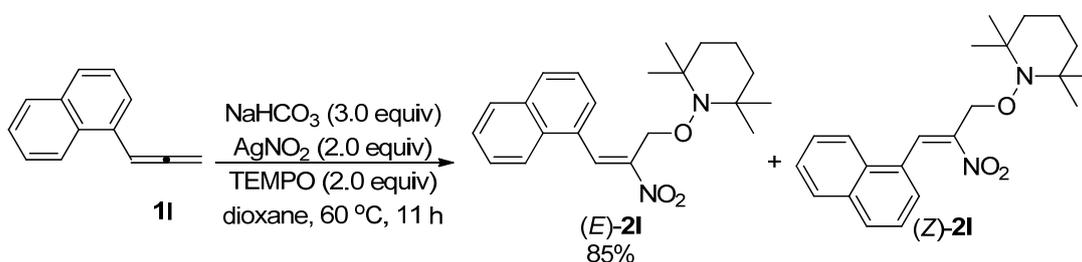
The reaction of 1-((1,1'-diphenyl)-4-yl)propa-1,2-diene **1k** (0.1922 g, 1.00 mmol), TEMPO (98%, 0.3188 g, 2.00 mmol), AgNO₂ (99%, 0.3113 g, 2.00 mmol), and NaHCO₃ (0.2527 g, 3.01 mmol) in dioxane (10 mL) at 60 °C for 7 h afforded a crude product. The ratio of (*E*)-**2k**/**(Z)-2k** was 96/4 as determined by the ¹H NMR analysis of the crude product. Further purification via column chromatography on silica gel (eluent: petroleum ether/ethyl acetate = 150/1) afforded pure (*E*)-**2k** (0.3337 g, 85%) as a solid and **2k** (0.0063 g, 1.6%, *E/Z* = 77/23) as an oil.

(*E*)-**2k** (less polar): m.p. 105.4-106.7 °C (*n*-hexane/ethyl acetate); ¹H NMR (300 MHz, CDCl₃) δ 8.21 (s, 1 H, =CH), 7.78-7.58 (m, 6 H, ArH), 7.53-7.37 (m, 3 H, ArH), 5.02

(s, 2 H, CH₂), 1.54-1.27 (m, 6 H, 3 × CH₂), 1.15 (s, 6 H, 2 × CH₃), 1.10 (s, 6 H, 2 × CH₃); ¹³C NMR (75 MHz, CDCl₃) δ 148.1, 143.4, 139.8, 137.8, 130.8, 130.5, 128.9, 128.1, 127.4, 127.1, 69.4, 60.1, 39.9, 33.0, 20.1, 17.0; IR (KBr) ν (cm⁻¹) 2973, 2932, 1644, 1605, 1526, 1488, 1450, 1375, 1361, 1323, 1262, 1133, 1008; MS (ESI): *m/z* = 395 [M+H]⁺; Elemental analysis calcd for C₂₄H₃₀N₂O₃: N, 7.10; C, 73.07; H, 7.66; Found: N, 6.82; C, 72.85; H, 7.74.

The following signals are discernible for (*Z*)-**2k** (more polar): ¹H NMR (300 MHz, CDCl₃) δ 6.71 (s, 1 H, =CH), 4.76 (s, 2 H, CH₂).

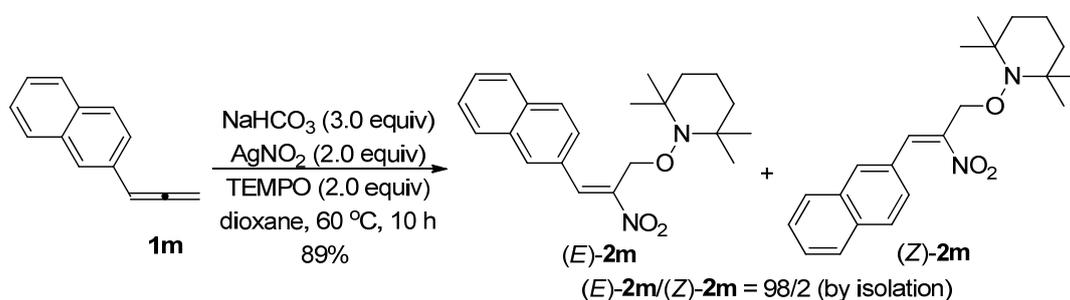
12. (*E*)- and (*Z*)-3-(2,2,6,6-Tetramethylpiperidinyl-1-oxy)-1-(1-naphthyl)-2-nitroprop-1-ene ((*E*)- and (*Z*)-**2l**) (xc-10-078-2)



The reaction of 1-(1-naphthyl)propa-1,2-diene **11** (0.1665 g, 1.00 mmol), TEMPO (98%, 0.3193 g, 2.01 mmol), AgNO₂ (99%, 0.3122 g, 2.01 mmol), and NaHCO₃ (0.2533 g, 3.02 mmol) in dioxane (10 mL) at 60 °C for 11 h afforded a crude product. The ratio of (*E*)-**2l**/*Z*-**2l** was 99/1 as determined by the ¹H NMR analysis of the crude product. Further purification via column chromatography on silica gel (eluent: petroleum ether/ethyl ether = 100/1 then petroleum ether/ethyl acetate = 100/1) afforded (*E*)-**2l** (0.3138 g, 85%) as a liquid: ¹H NMR (300 MHz, CDCl₃) δ 8.69 (s, 1

H, =CH), 7.98-7.81 (m, 3 H, ArH), 7.72-7.63 (m, 1 H, ArH), 7.63-7.46 (m, 3 H, ArH), 4.82 (s, 2 H, CH₂), 1.54-1.33 (m, 5 H, 2 × CH₂ and one proton of CH₂), 1.30-1.19 (m, 1 H, one proton of CH₂), 1.01 (s, 6 H, 2 × CH₃), 0.97 (s, 6 H, 2 × CH₃); ¹³C NMR (75 MHz, CDCl₃) δ 149.2, 136.4, 133.1, 131.1, 130.4, 128.8, 128.5, 127.5, 127.0, 126.4, 124.9, 124.0, 69.0, 59.8, 39.7, 32.4, 19.7, 16.7; IR (neat) ν (cm⁻¹) 3059, 2973, 2932, 2869, 1651, 1531, 1470, 1450, 1375, 1360, 1334, 1244, 1133, 1038; MS (ESI): *m/z* = 369 [M+H]⁺; HRMS (ESI): calcd. for C₂₂H₂₉N₂O₃ [M+H]⁺ 369.2173; found 369.2168.

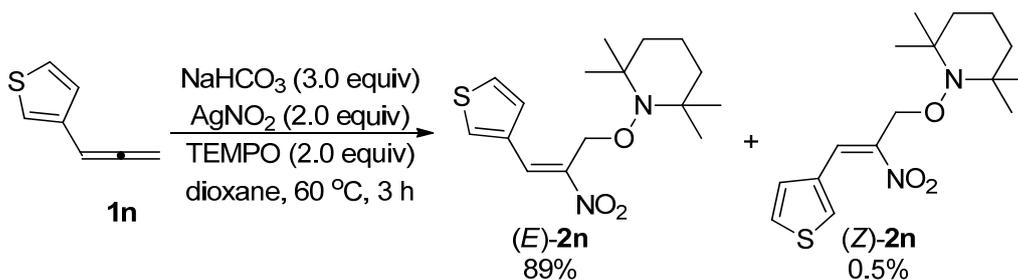
13. (*E*)- and (*Z*)-3-(2,2,6,6-Tetramethylpiperidinyl-1-oxy)-1-(2-naphthyl)-2-nitroprop-1-ene ((*E*)- and (*Z*)-**2m**) (xc-10-161-1,2)



The reaction of 1-(2-naphthyl)prop-1,2-diene **1m** (0.1665 g, 1.00 mmol), TEMPO (98%, 0.3188 g, 2.00 mmol), AgNO₂ (99%, 0.3115 g, 2.00 mmol), and NaHCO₃ (0.2533 g, 3.02 mmol) in dioxane (10 mL) at 60 °C for 10 h afforded a crude product. The ratio of (*E*)-**2m**/**(Z)-2m** was 97/3 as determined by the ¹H NMR analysis of the crude product. Further purification via column chromatography on silica gel (eluent: petroleum ether/ethyl acetate = 150/1) afforded **2m** (0.3285 g, (*E*)/(*Z*) = 98/2, 89% yield) as a solid.

(*E*)-**2m**: m.p. 57.7-58.9 °C (*n*-hexane/ethyl acetate); ¹H NMR (300 MHz, CDCl₃) δ 8.32 (s, 1 H, =CH), 8.21 (s, 1 H, ArH), 7.96-7.84 (m, 3 H, ArH), 7.71-7.63 (m, 1 H, ArH), 7.62-7.51 (m, 2 H, ArH), 5.06 (s, 2 H, CH₂), 1.55-1.40 (m, 5 H, 2 × CH₂ and one proton of CH₂), 1.39-1.28 (m, 1 H, one proton of CH₂), 1.15 (s, 12 H, 4 × CH₃); ¹³C NMR (75 MHz, CDCl₃) δ 148.3, 138.2, 133.9, 132.9, 130.6, 129.1, 128.6, 127.8, 127.7, 126.9, 126.6, 69.6, 60.2, 39.9, 33.0, 20.2, 17.0; IR (KBr) ν (cm⁻¹) 3057, 2973, 2932, 1645, 1625, 1598, 1525, 1467, 1375, 1361, 1319, 1272, 1242, 1181, 1132, 1018; MS (ESI): *m/z* = 369 [M+H]⁺; Elemental analysis calcd for C₂₂H₂₈N₂O₃: N, 7.60; C, 71.71; H, 7.66; Found: N, 7.32; C, 71.68; H, 7.72. The following signals are discernible for (*Z*)-**2m**: ¹H NMR (300 MHz, CDCl₃) δ 6.86 (s, 1 H, =CH), 4.80 (s, 2 H, CH₂).

14. (*E*)- and (*Z*)-3-(2,2,6,6-Tetramethylpiperidinyl-1-oxy)-2-nitro-1-(3-thienyl)prop-1-ene ((*E*)- and (*Z*)-**2n**) (xc-11-026-1,2)



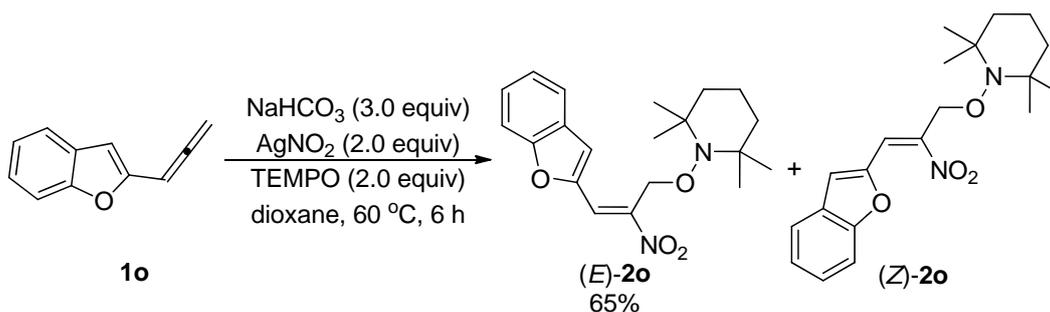
The reaction of 1-(3-thienyl)prop-1,2-diene **1n** (0.1225 g, 1.00 mmol), TEMPO (98%, 0.3191 g, 2.00 mmol), AgNO₂ (99%, 0.3118 g, 2.00 mmol), and NaHCO₃ (0.2527 g, 3.01 mmol) in dioxane (10 mL) at 60 °C for 3 h afforded a crude product. The ratio of (*E*)-**2n**/**(Z)-2n** was 97/3 as determined by the ¹H NMR analysis of the

crude product. Further purification via column chromatography on silica gel (eluent: petroleum ether/ethyl acetate = 150/1) afforded (*E*)-**2n** (0.2887 g, (*E*)/(*Z*) = 99/1, 89% yield) as a solid and (*Z*)-**2n** (0.0103 g, purity = 16% as determined by using mesitylene as the internal standard, 0.5% yield) as an oil.

(*E*)-**2n** (less polar): m.p. 63.7-64.8 °C (*n*-hexane/ethyl acetate); ¹H NMR (300 MHz, CDCl₃) δ 8.14 (s, 1 H, =CH), 7.92 (s, 1 H, ArH), 7.44 (s, 2 H, ArH), 5.07 (s, 2 H, CH₂), 1.59-1.26 (m, 6 H, 3 × CH₂), 1.16 (s, 6 H, 2 × CH₃), 1.08 (s, 6 H, 2 × CH₃); ¹³C NMR (75 MHz, CDCl₃) δ 147.1, 132.9, 131.5, 130.9, 128.5, 126.9, 69.6, 60.0, 39.7, 33.0, 20.1, 17.0; IR (KBr) ν (cm⁻¹) 3103, 2973, 2932, 1640, 1525, 1468, 1450, 1375, 1361, 1317, 1258, 1133, 1012; MS (ESI): *m/z* = 325 [M+H]⁺; Elemental analysis calcd for C₁₆H₂₄N₂O₃S: N, 8.63; C, 59.23; H, 7.46; Found: N, 8.50; C, 59.38; H, 7.46.

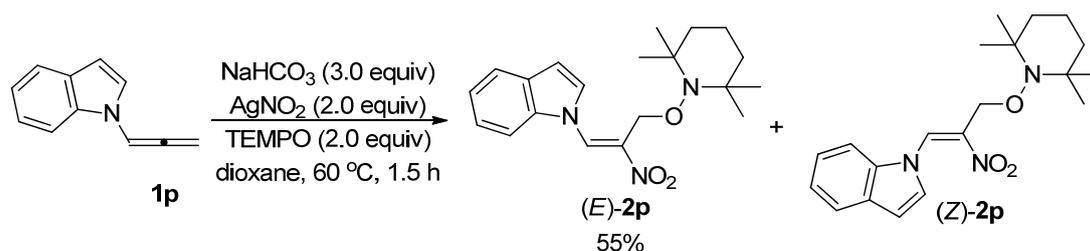
The following signals are discernible for (*Z*)-**2n** (more polar): ¹H NMR (300 MHz, CDCl₃) δ 7.69-7.62 (m, 1 H, ArH), 7.36-7.29 (m, 1 H, ArH), 7.20-7.14 (m, 1 H, ArH), 6.70 (s, 1 H, =CH), 4.72 (s, 2 H, CH₂); MS (ESI): *m/z* = 325 [M+H]⁺; HRMS (ESI): calcd. for C₁₆H₂₅N₂O₃S [M+H]⁺ 325.1580; found 325.1569.

15. (*E*)- and (*Z*)-1-(Benzofuran-2-yl)-3-(2,2,6,6-tetramethylpiperidinyl-1-oxy)-2-nitro prop-1-ene ((*E*)- and (*Z*)-**2o**) (xc-11-071-2)



The reaction of 1-(benzofuran-2-yl)propa-1,2-diene **1o** (0.1565 g, 1.00 mmol), TEMPO (98%, 0.3187 g, 2.00 mmol), AgNO_2 (99%, 0.3121 g, 2.01 mmol), and NaHCO_3 (0.2528 g, 3.01 mmol) in dioxane (10 mL) at 60 °C for 6 h afforded a crude product. The ratio of **(E)-2o**/**(Z)-2o** was 97/3 as determined by the ^1H NMR analysis of the crude product. Further purification via column chromatography on silica gel (eluent: petroleum ether/ethyl acetate = 150/1 then petroleum ether/ethyl acetate = 100/1) afforded **(E)-2o** (0.2324 g, 65%) as a solid: m.p. 100.9-102.3 °C (*n*-hexane/ethyl acetate); ^1H NMR (300 MHz, CDCl_3) δ 7.97 (s, 1 H, =CH), 7.66 (d, J = 7.2 Hz, 1 H, ArH), 7.51-7.39 (m, 2 H, ArH), 7.36-7.23 (m, 2 H, ArH), 5.38 (s, 2 H, CH_2), 1.65-1.43 (m, 6 H, 3 \times CH_2), 1.37 (s, 6 H, 2 \times CH_3), 1.06 (s, 6 H, 2 \times CH_3); ^{13}C NMR (75 MHz, CDCl_3) δ 156.3, 148.2, 145.7, 127.8, 127.5, 123.8, 123.5, 122.2, 117.5, 111.5, 68.8, 60.1, 40.0, 32.9, 20.0, 17.0; IR (KBr) ν (cm^{-1}) 2971, 2931, 2869, 1651, 1612, 1547, 1518, 1469, 1449, 1360, 1315, 1293, 1259, 1134, 1084, 1032; MS (ESI): m/z = 359 $[\text{M}+\text{H}]^+$; Elemental analysis calcd for $\text{C}_{20}\text{H}_{26}\text{N}_2\text{O}_4$: N, 7.82; C, 67.02; H, 7.31; Found: N, 7.17; C, 67.26; H, 7.46.

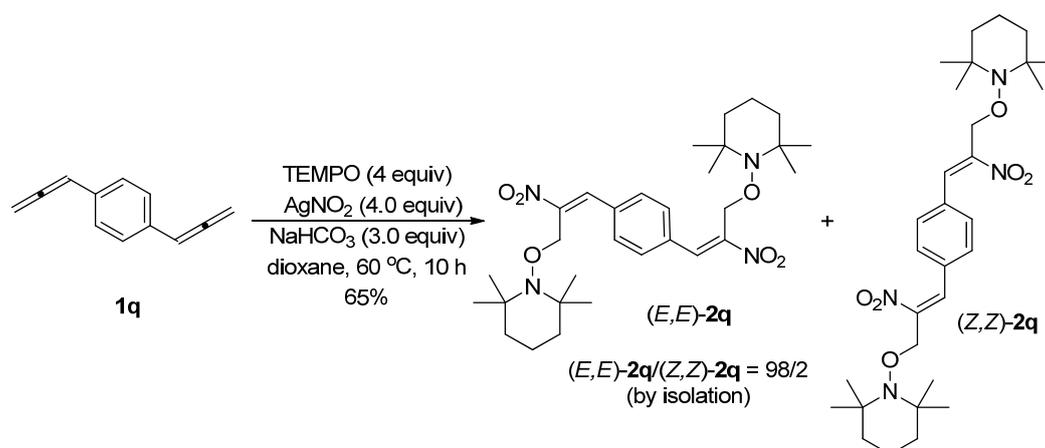
16. (*E*)- and (*Z*)-1-(1-Indolyl)-3-(2,2,6,6-tetramethylpiperidinyl-1-oxy)-2-nitroprop-1-ene (**(E)-** and **(Z)-2p**) (xc-11-057-1, 059-1)



The reaction of 1-(1-indolyl)propa-1,2-diene **1p** (0.1547 g, 1.00 mmol), TEMPO (98%, 0.3189 g, 2.00 mmol), AgNO₂ (99%, 0.3121 g, 2.01 mmol), and NaHCO₃ (0.2533 g, 3.02 mmol) in dioxane (10 mL) at 60 °C for 1.5 h afforded a crude product. The ratio of **(E)-2p**/**(Z)-2p** was 99/1 as determined by the ¹H NMR analysis of the crude product. Further purification via column chromatography on silica gel (eluent: petroleum ether/ethyl acetate = 150/1 then petroleum ether/ethyl acetate = 100/1) afforded **(E)-2p** (0.1963 g, 55%) as a solid: m.p. 127.5-128.6 °C (*n*-hexane/ethyl acetate); ¹H NMR (300 MHz, CDCl₃) δ 9.02 (s, 1 H, =CH), 8.13 (d, *J* = 3.3 Hz, 1 H, ArH), 7.70-7.52 (m, 2 H, ArH), 7.43-7.27 (m, 2 H, ArH), 6.84 (d, *J* = 3.3 Hz, 1 H, ArH), 5.20 (s, 2 H, CH₂), 1.60-1.42 (m, 5 H, 2 × CH₂ and one proton of CH₂), 1.39-1.31 (m, 1 H, one proton of CH₂), 1.23 (s, 6 H, 2 × CH₃), 1.13 (s, 6 H, 2 × CH₃); ¹³C NMR (75 MHz, CDCl₃) δ 137.1, 134.9, 134.0, 129.7, 126.8, 124.3, 123.5, 121.5, 110.6, 110.1, 69.3, 60.0, 39.8, 33.1, 20.2, 16.9; IR (KBr) ν (cm⁻¹) 2973, 2932, 2869, 1644, 1588, 1533, 1514, 1463, 1405, 1376, 1362, 1336, 1286, 1250, 1197, 1126, 1098, 1012; MS (ESI): *m/z* = 358 [M+H]⁺; Elemental analysis calcd for C₂₀H₂₇N₃O₃: N, 11.76; C, 67.20; H, 7.61; Found: N, 11.50; C, 67.58; H, 7.77.

17. 1,4-Bis(*E*)- and (*Z*)-3-((2,2,6,6-tetramethylpiperidin-1-yl)oxy)-2-nitroprop-1-

en-1-yl)benzene ((*E,E*)- and (*Z,Z*)-**2q**) (xc-11-033-2)



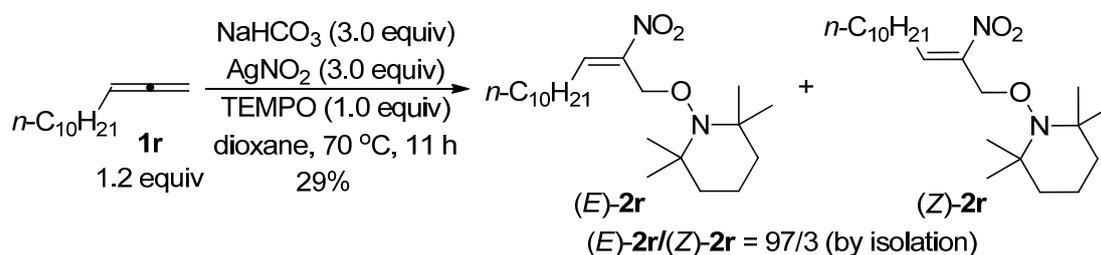
The reaction of 1,4-di(propa-1,2-dien-1-yl)benzene **1q** (0.1542 g, 1.00 mmol), TEMPO (98%, 0.6372 g, 4.00 mmol), AgNO₂ (99%, 0.6233 g, 4.01 mmol), and NaHCO₃ (0.5051 g, 6.01 mmol) in dioxane (20 mL) at 60 °C for 10 h afforded a crude product. The ratio of (*E,E*)-**2q**/*Z,Z*-**2q** was 98/2 as determined by the ¹H NMR analysis of the crude product. Further purification via column chromatography on silica gel (eluent: petroleum ether/ethyl acetate = 150/1) afforded **2q** (0.3624 g, (*E,E*)/(*Z,Z*) = 98/2, 65% yield) as a solid.

(*E,E*)-**2q**: m.p. 124.7-126.8 °C (*n*-hexane/ethyl acetate); ¹H NMR (300 MHz, CDCl₃) δ 8.15 (s, 2 H, 2 × =CH), 7.71 (s, 4 H, ArH), 4.97 (s, 4 H, 2 × CH₂), 1.63-1.40 (m, 10 H, 4 × CH₂ and two protons of 2 × CH₂), 1.37-1.26 (m, 2 H, two protons of 2 × CH₂), 1.11 (s, 12 H, 4 × CH₃), 1.08 (s, 12 H, 4 × CH₃); ¹³C NMR (75 MHz, CDCl₃) δ 149.2, 136.4, 133.6, 130.3, 69.2, 60.1, 39.8, 32.9, 20.1, 16.9; IR (KBr) ν (cm⁻¹) 3006, 2977, 2931, 2869, 1651, 1607, 1537, 1532, 1470, 1376, 1361, 1325, 1261, 1243, 1133, 1036; MS (ESI): *m/z* = 559 [M+H]⁺; Elemental analysis calcd for C₃₀H₄₆N₄O₆: N, 10.03; C, 64.49; H, 8.30; Found: N, 9.82; C, 64.60; H, 8.39. The following signals are

discernible for (Z,Z)-**2q**: $^1\text{H NMR}$ (300 MHz, CDCl_3) δ 6.73 (s, 2 H, $2 \times =\text{CH}$), 4.77 (s, 4 H, $2 \times \text{CH}_2$).

Nitro-oxoamination reactions of mono-allenes that need the degassing operation

1. (*E*)- and (*Z*)-1-(2,2,6,6-Tetramethylpiperidinyl-1-oxy)-2-nitrotridec-2-ene ((*E*)- and (*Z*)-**2r**) (xc-11-099-1,2; 11-151-1,2)



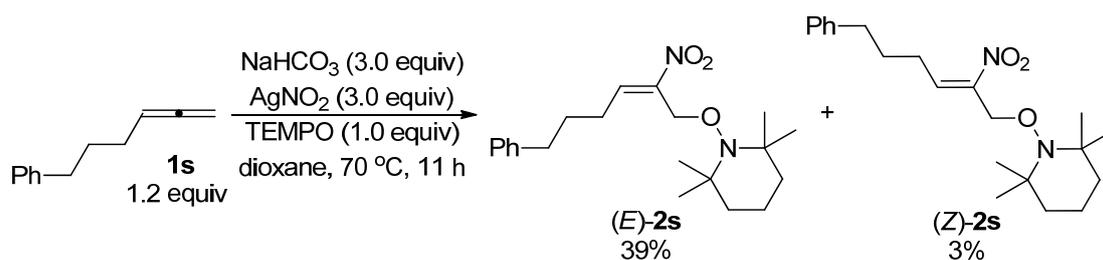
Typical Procedure II: To a dried flask were added AgNO_2 (99%, 0.0935 g, 0.60 mmol), NaHCO_3 (0.0507 g, 0.60 mmol), TEMPO (98%, 0.0321 g, 0.20 mmol), trideca-1,2-diene **1r** (0.0437 g, 0.24 mmol), and 2 mL of anhydrous 1,4-dioxane at room temperature under N_2 atmosphere. The resulting mixture was then degassed for three times before the reaction flask was placed in a pre-heated oil bath of 70°C . The reaction was finished in 11 h as monitored by TLC. After cooling to room temperature, the crude reaction mixture was filtrated through a short column of silica gel (ethyl acetate 3×15 mL). After concentration, the ratio of (*E*)-**2r**/(*Z*)-**2r** was 98/2 as determined by the $^1\text{H NMR}$ analysis. Column chromatography on silica gel (eluent: petroleum ether/ethyl ether = 100/1) afforded **2r** (0.0225 g, (*E*)/(*Z*) = 97/3, 29% yield) as an oil.

(*E*)-**2r** (more polar): $^1\text{H NMR}$ (300 MHz, CDCl_3) δ 7.27 (t, $J = 8.1$ Hz, 1 H, $=\text{CH}$),

4.75 (s, 2 H, CH₂), 2.36 (q, *J* = 7.7 Hz, 2 H, CH₂), 1.59-1.42 (m, 7 H, 3 × CH₂ and one proton of CH₂), 1.38-1.20 (m, 21 H, one proton of CH₂, 7 × CH₂ and 2 × CH₃), 1.06 (s, 6 H, 2 × CH₃), 0.88 (t, *J* = 6.6 Hz, 3 H, CH₃); ¹³C NMR (75 MHz, CDCl₃) δ 147.9, 141.4, 68.2, 60.0, 39.8, 32.9, 31.8, 29.5, 29.4, 29.30, 29.28, 29.2, 28.6, 28.3, 22.6, 19.9, 17.0, 14.1; IR (neat) ν (cm⁻¹) 2926, 2855, 1669, 1639, 1558, 1530, 1468, 1375, 1360, 1337, 1261, 1246, 1209, 1183, 1132, 1039; MS (ESI): *m/z* = 538 [M+TEMPO]⁺, 326 [M+H-Bu]⁺; Elemental analysis calcd for C₂₂H₄₂N₂O₃: N, 7.32; C, 69.07; H, 11.07; Found: N, 7.29; C, 69.04; H, 11.12.

The following signals are discernible for (*Z*)-**2r** (less polar): ¹H NMR (300 MHz, CDCl₃) δ 6.09 (t, *J* = 7.5 Hz, 1 H, =CH), 4.61 (d, *J* = 0.9 Hz, 2 H, CH₂), 2.49 (d, *J* = 7.5 Hz, 2 H, CH₂).

2. (*E*)- and (*Z*)-1-(2,2,6,6-Tetramethylpiperidinyl-1-oxy)-2-nitro-6-phenylhex-2-ene ((*E*)- and (*Z*)-**2s**) (xc-11-152-1,2)



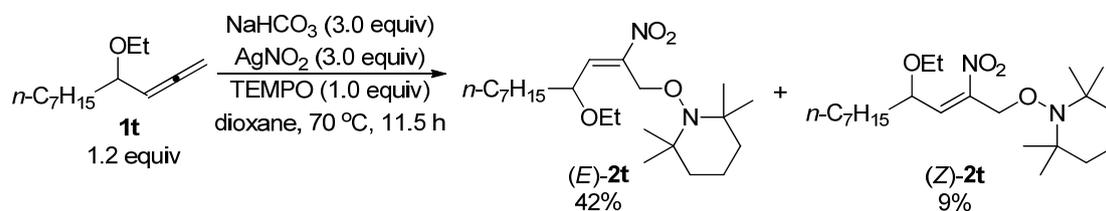
Following Typical Procedure II: The reaction of 6-phenylhexa-1,2-diene **1s** (0.0381 g, 0.24 mmol), AgNO₂ (99%, 0.0935 g, 0.60 mmol), NaHCO₃ (0.0512 g, 0.61 mmol), and TEMPO (98%, 0.0318 g, 0.20 mmol) in dioxane (2 mL) at 70 °C for 11 h afforded a crude product. The ratio of (*E*)-**2s**/*Z*)-**2s** was 95/5 as determined by the ¹H

NMR analysis of the crude product. Further purification via column chromatography on silica gel (eluent: petroleum ether/ethyl ether = 80/1 then 50/1) afforded (*E*)-**2s** (0.0277 g, 39% yield) as an oil and (*Z*)-**2s** (0.0039 g, purity of (*Z*)-**2s**: 55% as determined by using mesitylene as the internal standard, 3% yield) as an oil.

(*E*)-**2s** (more polar): ^1H NMR (300 MHz, CDCl_3) δ 7.35-7.10 (m, 6 H, ArH and =CH), 4.71 (s, 2 H, CH_2), 2.69 (t, $J = 7.5$ Hz, 2 H, CH_2), 2.38 (q, $J = 7.7$ Hz, 2 H, CH_2), 1.87 (quint, $J = 7.5$ Hz, 2 H, CH_2), 1.60-1.39 (m, 5 H, $2 \times \text{CH}_2$ and one proton of CH_2), 1.37-1.27 (m, 1 H, one proton of CH_2), 1.21 (s, 6 H, $2 \times \text{CH}_3$), 1.04 (s, 6 H, $2 \times \text{CH}_3$); ^{13}C NMR (75 MHz, CDCl_3) δ 148.1, 140.9, 140.7, 128.5, 128.3, 126.1, 68.3, 60.0, 39.8, 35.4, 32.9, 30.1, 27.8, 20.0, 17.0; IR (neat) ν (cm^{-1}) 2972, 2932, 2867, 1667, 1637, 1526, 1497, 1470, 1453, 1375, 1360, 1335, 1261, 1246, 1132, 1038; MS (ESI): $m/z = 361$ $[\text{M}+\text{H}]^+$; HRMS (ESI): calcd. for $\text{C}_{21}\text{H}_{33}\text{N}_2\text{O}_3$ $[\text{M}+\text{H}]^+$ 361.2486; found 361.2492.

The following signals are discernible for (*Z*)-**2s** (less polar): ^1H NMR (300 MHz, CDCl_3) δ 6.08 (t, $J = 7.5$ Hz, 1 H, =CH), 4.60 (d, $J = 0.9$ Hz, 2 H, CH_2), 2.67 (t, $J = 7.7$ Hz, 2 H, CH_2), 2.54 (q, $J = 7.3$ Hz, 2 H, CH_2), 1.84 (quint, $J = 7.7$ Hz, 2 H, CH_2), 1.16 (s, 6 H, $2 \times \text{CH}_3$), 1.08 (s, 6 H, $2 \times \text{CH}_3$); ^{13}C NMR (75 MHz, CDCl_3) δ 147.1, 141.4, 135.7, 128.42, 128.38, 126.0, 74.3, 60.1, 39.7, 35.5, 32.8, 30.5, 27.9, 20.1, 17.0; IR (neat) ν (cm^{-1}) 2971, 2930, 2867, 1640, 1578, 1526, 1453, 1332, 1242, 1132; MS (ESI): $m/z = 361$ $[\text{M}+\text{H}]^+$; HRMS (ESI): calcd. for $\text{C}_{21}\text{H}_{33}\text{N}_2\text{O}_3$ $[\text{M}+\text{H}]^+$ 361.2486; found 361.2496.

3. (*E*)- and (*Z*)-4-Ethoxy-1-(2,2,6,6-tetramethylpiperidinyl-1-oxy)-2-nitroundec-2-ene
(*E*)- and (*Z*)-**2t** (xc-12-025)



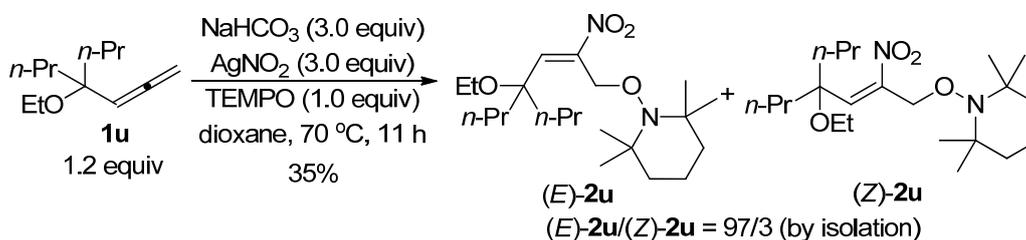
Following Typical Procedure II: The reaction of 4-ethoxyundeca-1,2-diene **1t** (0.0482 g, 0.25 mmol), AgNO₂ (99%, 0.0935 g, 0.60 mmol), NaHCO₃ (0.0507 g, 0.60 mmol), and TEMPO (98%, 0.0320 g, 0.20 mmol) in dioxane (2 mL) at 70 °C for 11.5 h afforded a crude product. The ratio of (*E*)-**2t**/*Z*)-**2t** was 83/17 as determined by the ¹H NMR analysis of the crude product. Further purification via column chromatography on silica gel (eluent: petroleum ether/ethyl ether = 80/1 then 50/1) afforded (*E*)-**2t** (0.0333 g, 42% yield) as an oil and (*Z*)-**2t** (0.0086 g, purity of (*Z*)-**2t**: 83% as determined by using mesitylene as the internal standard, 9% yield) as an oil.

(*E*)-**2t** (less polar): ¹H NMR (300 MHz, CDCl₃) δ 7.06 (d, *J* = 9.3 Hz, 1 H, =CH), 4.80 (AB, *J* = 12.0 Hz, 1 H, one proton of NOCH₂), 4.74 (AB, *J* = 11.70 Hz, 1 H, one proton of NOCH₂), 4.22-4.11 (m, 1 H, CH), 3.58-3.31 (m, 2 H, OCH₂), 1.82-1.68 (m, 1 H, one proton of CH₂), 1.61-1.40 (m, 7 H, 3 × CH₂ and one proton of CH₂), 1.38-1.18 (m, 19 H, 5 × CH₂ and 3 × CH₃), 1.06 (s, 6 H, 2 × CH₃), 0.88 (t, *J* = 6.9 Hz, 3 H, CH₃); ¹³C NMR (75 MHz, CDCl₃) δ 148.3, 141.3, 75.4, 68.6, 65.1, 60.2, 60.1, 39.8, 35.4, 33.0, 32.9, 31.7, 29.5, 29.1, 25.2, 22.6, 20.05, 20.00, 17.0, 15.3, 14.1; IR (neat) ν (cm⁻¹) 2929, 2871, 1537, 1469, 1453, 1375, 1361, 1335, 1261, 1244, 1209,

1184, 1132, 1114, 1093, 1045; MS (ESI): $m/z = 399$ $[M+H]^+$; HRMS (ESI): calcd. for $C_{22}H_{43}N_2O_4$ $[M+H]^+$ 399.3217; found 399.3222.

The following signals are discernible for (*Z*)-**2t** (more polar): 1H NMR (300 MHz, $CDCl_3$) δ 6.07 (d, $J = 8.4$ Hz, 1 H, =CH), 4.68 (AB, $J = 12.9$ Hz, 1 H, one proton of $NOCH_2$), 4.59 (AB, $J = 13.2$ Hz, 1 H, one proton of $NOCH_2$), 4.53-4.43 (m, 1 H, CH), 3.57-3.33 (m, 2 H, OCH_2); IR (neat) ν (cm^{-1}) 2928, 2852, 1531, 1467, 1450, 1376, 1361, 1262, 1132, 1089; MS (ESI): $m/z = 399$ $[M+H]^+$; HRMS (ESI): calcd. for $C_{22}H_{43}N_2O_4$ $[M+H]^+$ 399.3217; found 399.3224.

4. (*E*)- and (*Z*)-4-Ethoxy-1-(2,2,6,6-tetramethylpiperidinyloxy)-2-nitro-4-propyl hept-2-ene ((*E*)- and (*Z*)-**2u**) (xc-12-030-1)



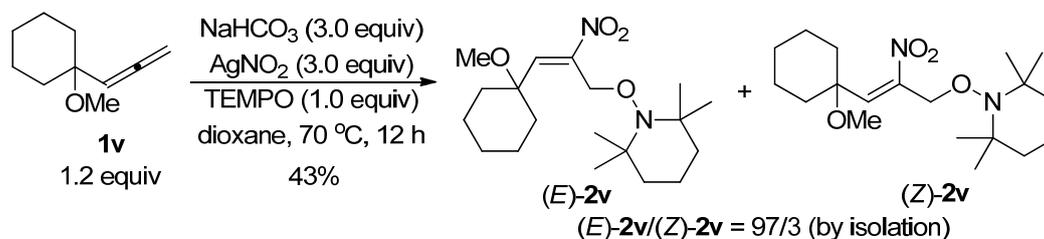
Following Typical Procedure II: The reaction of 4-ethoxy-4-propylhepta-1,2-diene **1u** (0.0439 g, 0.24 mmol), AgNO₂ (99%, 0.0938 g, 0.60 mmol), NaHCO₃ (0.0512 g, 0.61 mmol), and TEMPO (98%, 0.0322 g, 0.20 mmol) in dioxane (2 mL) at 70 °C for 11 h afforded a crude product. The ratio of (*E*)-**2u**/**(Z)-2u** was 96/4 as determined by the 1H NMR analysis of the crude product. Further purification via column chromatography on silica gel (eluent: petroleum ether/ethyl ether = 100/1 then 50/1) afforded **2u** (0.0273 g, (*E*)/(*Z*) = 97/3, 35% yield) as an oil.

(*E*)-**2u**: 1H NMR (300 MHz, $CDCl_3$) δ 6.69 (s, 1 H, =CH), 5.08 (s, 2 H, $NOCH_2$),

3.31 (q, $J = 6.9$ Hz, 2 H, OCH₂), 1.87-1.72 (m, 2 H, CH₂), 1.56-1.41 (m, 7 H, 3 × CH₂ and one proton of CH₂), 1.37-1.18 (m, 11 H, 2 × CH₃, 2 × CH₂ and one proton of CH₂), 1.17 (t, $J = 6.9$ Hz, 3 H, CH₃), 1.04 (s, 6 H, 2 × CH₃), 0.90 (t, $J = 7.2$ Hz, 6 H, 2 × CH₃); ¹³C NMR (75 MHz, CDCl₃) δ 149.9, 141.1, 79.7, 68.2, 60.0, 56.6, 40.1, 39.2, 33.1, 19.9, 17.0, 16.8, 15.5, 14.3; IR (neat) ν (cm⁻¹) 2959, 2933, 2874, 1532, 1469, 1455, 1376, 1361, 1347, 1326, 1262, 1245, 1166, 1133, 1116, 1074, 1033; MS (ESI): $m/z = 385$ [M+H]⁺; HRMS (ESI): calcd. for C₂₁H₄₁N₂O₄ [M+H]⁺ 385.3061; found 385.3063.

The following signals are discernible for (*Z*)-**2u**: ¹H NMR (300 MHz, CDCl₃) δ 5.34 (s, 1 H, =CH), 4.51 (s, 2 H, NOCH₂), 3.24 (q, $J = 7.1$ Hz, 2 H, OCH₂).

5. (*E*)- and (*Z*)-3-(1-Methoxycyclohexyl)-1-(2,2,6,6-tetramethylpiperidinyl-1-oxy)-2-nitroprop-2-ene ((*E*)- and (*Z*)-**2v**) (xc-12-022-1)



Following Typical Procedure II: The reaction of 1-(1-methoxycyclohexyl)prop-1,2-diene **1v** (0.0367 g, 0.24 mmol), AgNO₂ (99%, 0.0935 g, 0.60 mmol), NaHCO₃ (0.0508 g, 0.60 mmol), and TEMPO (98%, 0.0319 g, 0.20 mmol) in dioxane (2 mL) at 70 °C for 12 h afforded a crude product. The ratio of (*E*)-**2v**/*Z*-**2v** was 96/4 as determined by the ¹H NMR analysis of the crude product. Further purification via column chromatography on silica gel (eluent: petroleum

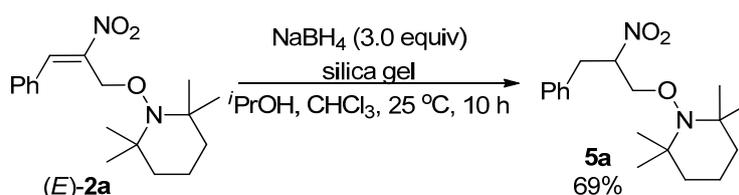
ether/ethyl ether = 80/1 then 50/1) afforded **2v** (0.0306 g, (*E*)/(*Z*) = 97/3, 43% yield) as an oil.

(*E*)-**2v**: $^1\text{H NMR}$ (300 MHz, CDCl_3) δ 6.88 (s, 1 H, =CH), 4.99 (s, 2 H, NOCH_2), 3.14 (s, 3 H, OCH_3), 1.98-1.88 (m, 2 H, CH_2), 1.68-1.42 (m, 14 H, $7 \times \text{CH}_2$), 1.26 (s, 6 H, $2 \times \text{CH}_3$), 1.05 (s, 6 H, $2 \times \text{CH}_3$); $^{13}\text{C NMR}$ (75 MHz, CDCl_3) δ 149.4, 141.3, 75.6, 67.9, 60.2, 50.2, 40.2, 35.5, 32.8, 25.0, 21.3, 20.0, 17.0; IR (neat) ν (cm^{-1}) 2934, 2860, 1532, 1470, 1450, 1376, 1361, 1348, 1326, 1262, 1244, 1183, 1147, 1133, 1074, 1046; MS (ESI): $m/z = 355$ $[\text{M}+\text{H}]^+$; HRMS (ESI): calcd. for $\text{C}_{19}\text{H}_{35}\text{N}_2\text{O}_4$ $[\text{M}+\text{H}]^+$ 355.2591; found 355.2600.

The following signals are discernible for (*Z*)-**2v**: $^1\text{H NMR}$ (300 MHz, CDCl_3) δ 5.46 (s, 1 H, =CH), 4.54 (d, $J = 1.2$ Hz, 2 H, CH_2), 3.10 (s, 3 H, CH_3).

Synthetic applications of nitro-oxoamination product (*E*)-**2a**.

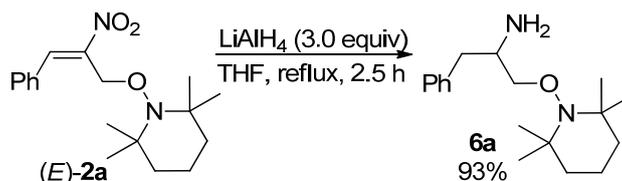
1. 2,2,6,6-Tetramethyl-1-(2-nitro-3-phenylpropoxy)piperidine (**5a**)² (xc-12-056)



To a dried Schlenk tube were added (*E*)-3-(2,2,6,6-tetramethylpiperidinyl-1-oxy)-2-nitro-1-phenylprop-1-ene (**5a**) (0.0957 g, 0.30 mmol), silica gel (100 ~ 200 mesh, 1.0012 g), CHCl_3 (1.5 mL), and $i\text{PrOH}$ (7.5 mL) at room temperature under N_2 atmosphere. After stirring for 5 min, NaBH_4 (98%, 0.0351 g, 0.91 mmol) was added. The resulting mixture was stirred at

25 °C for 10 h as monitored by TLC. The crude reaction mixture was filtrated through a short column of silica gel (eluted with ethyl acetate 3 × 10 mL). After evaporation, the crude product was purified through column chromatography on silica gel (eluent: petroleum ether/ethyl acetate = 100/1 then 80/1) to afford **5a** (0.0663 g, 69%) as an oil: ¹H NMR (300 MHz, CDCl₃) δ 7.35-7.23 (m, 3 H, ArH), 7.21-7.13 (m, 2 H, ArH), 4.96-4.83 (m, 1 H, CH), 4.31 (dd, *J*₁ = 10.4 Hz, *J*₂ = 8.9 Hz, 1 H, one proton of OCH₂), 3.96 (dd, *J*₁ = 10.2 Hz, *J*₂ = 3.3 Hz, 1 H, one proton of OCH₂), 3.26 (dd, *J*₁ = 14.3 Hz, *J*₂ = 8.6 Hz, 1 H, one proton of CH₂), 3.02 (dd, *J*₁ = 14.1 Hz, *J*₂ = 6.3 Hz, 1 H, one proton of CH₂), 1.54-1.36 (m, 5 H, 2 × CH₂ and one proton of CH₂), 1.35-1.26 (m, 1 H, one proton of CH₂), 1.12 (s, 3 H, CH₃), 1.06 (s, 3 H, CH₃), 1.03 (s, 6 H, 2 × CH₃); ¹³C NMR (75 MHz, CDCl₃) δ 135.0, 128.8, 128.7, 127.5, 87.7, 75.7, 60.3, 60.1, 39.6, 39.5, 36.5, 32.7, 20.1, 19.9, 16.9; IR (neat) ν (cm⁻¹) 3065, 3030, 2974, 2932, 1605, 1557, 1497, 1470, 1455, 1376, 1361, 1310, 1262, 1245, 1209, 1184, 1133, 1071, 1047; MS (ESI): *m/z* = 321 [M+H]⁺; HRMS (ESI): calcd. For C₁₈H₂₉N₂O₃ [M+H]⁺ 321.2173; found 321.2164.

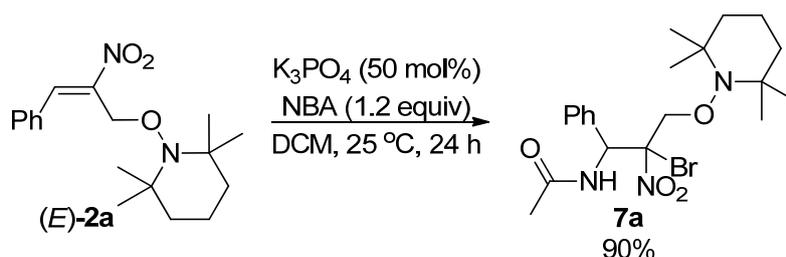
2. 1-Phenyl-3-((2,2,6,6-tetramethylpiperidin-1-yl)oxy)prop-2-yl amine (**6a**)³
(xc-12-038)



To a dried Schlenk tube were added LiAlH₄ (98%, 0.1165 g, 3.0 mmol) and THF

(6 mL) at room temperature under N₂ atmosphere. After stirring for 5 min, (*E*)-**2a** (0.3175 g, 1.0 mmol) and THF (4 mL) were added. Then the Schlenk tube was placed in a pre-heated oil bath of 90 °C, the resulting mixture was stirred and refluxed for 2.5 h as monitored by TLC. After cooling to room temperature H₂O (20 mL) and NaOH (1 M, 3 mL) were added to quench the reaction in an ice-water bath. The resulting mixture was extracted with ethyl acetate (20 mL × 3), washed with brine and dried over anhydrous Na₂SO₄. After filtration and concentration, the crude product was purified through column chromatography (6 cm high) on silica gel (eluent: petroleum ether/ethyl acetate = 10/1 then 5/1) to afford **6a** (0.2697 g, 93%) as an oil: ¹H NMR (300 MHz, CDCl₃) δ 7.34-7.25 (m, 2 H, ArH), 7.24-7.16 (m, 3 H, ArH), 3.82-3.65 (m, 2 H, OCH₂), 3.32-3.20 (m, 1 H, CH), 2.80 (dd, *J*₁ = 13.4 Hz, *J*₂ = 4.7 Hz, 1 H, one proton of CH₂), 2.53 (dd, *J*₁ = 13.5 Hz, *J*₂ = 9.0 Hz, 1 H, one proton of CH₂), 1.61-1.38 (m, 7 H, NH₂, 2 × CH₂ and one proton of CH₂), 1.37-1.27 (m, 1 H, one proton of CH₂), 1.19 (s, 3 H, CH₃), 1.13 (s, 6 H, 2 × CH₃), 1.11 (s, 3 H, CH₃); ¹³C NMR (75 MHz, CDCl₃) δ 139.0, 129.2, 128.4, 126.2, 81.3, 59.8, 52.2, 40.9, 39.6, 33.2, 33.0, 20.14, 20.06, 17.0; IR (neat) ν (cm⁻¹) 3378, 3062, 3026, 2973, 2931, 2871, 1727, 1603, 1495, 1470, 1454, 1374, 1359, 1262, 1245, 1208, 1184, 1133, 1049; MS (ESI): *m/z* = 291 [M+H]⁺; HRMS (ESI): calcd. For C₁₈H₃₁N₂O: [M+H]⁺ 291.2431; found 291.2439.

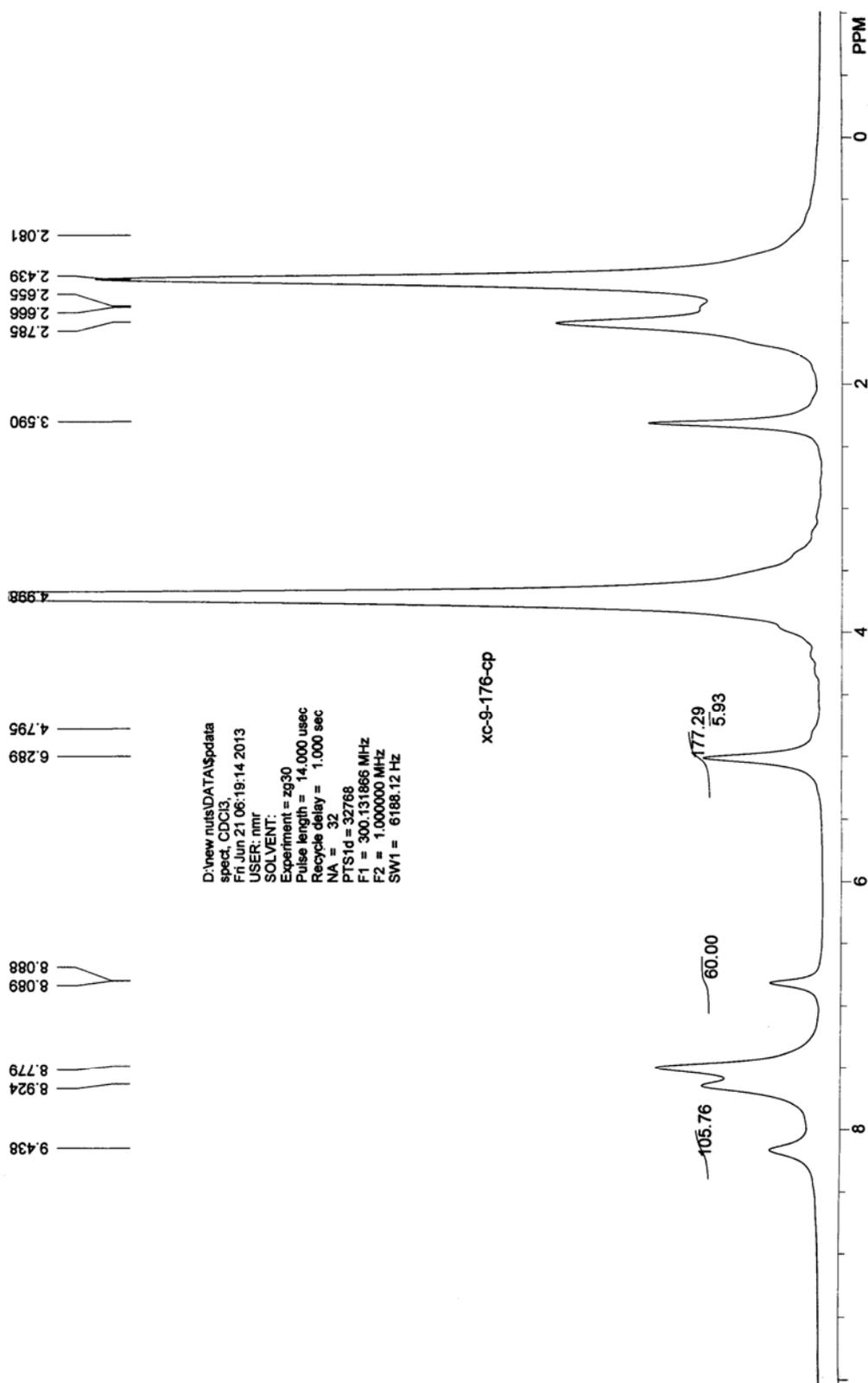
3. *N*-(2-Bromo-2-nitro-1-phenyl-3-((2,2,6,6-tetramethylpiperidin-1-yl)oxy)propyl)acetamide (**7a**)⁴ (xc-12-037)

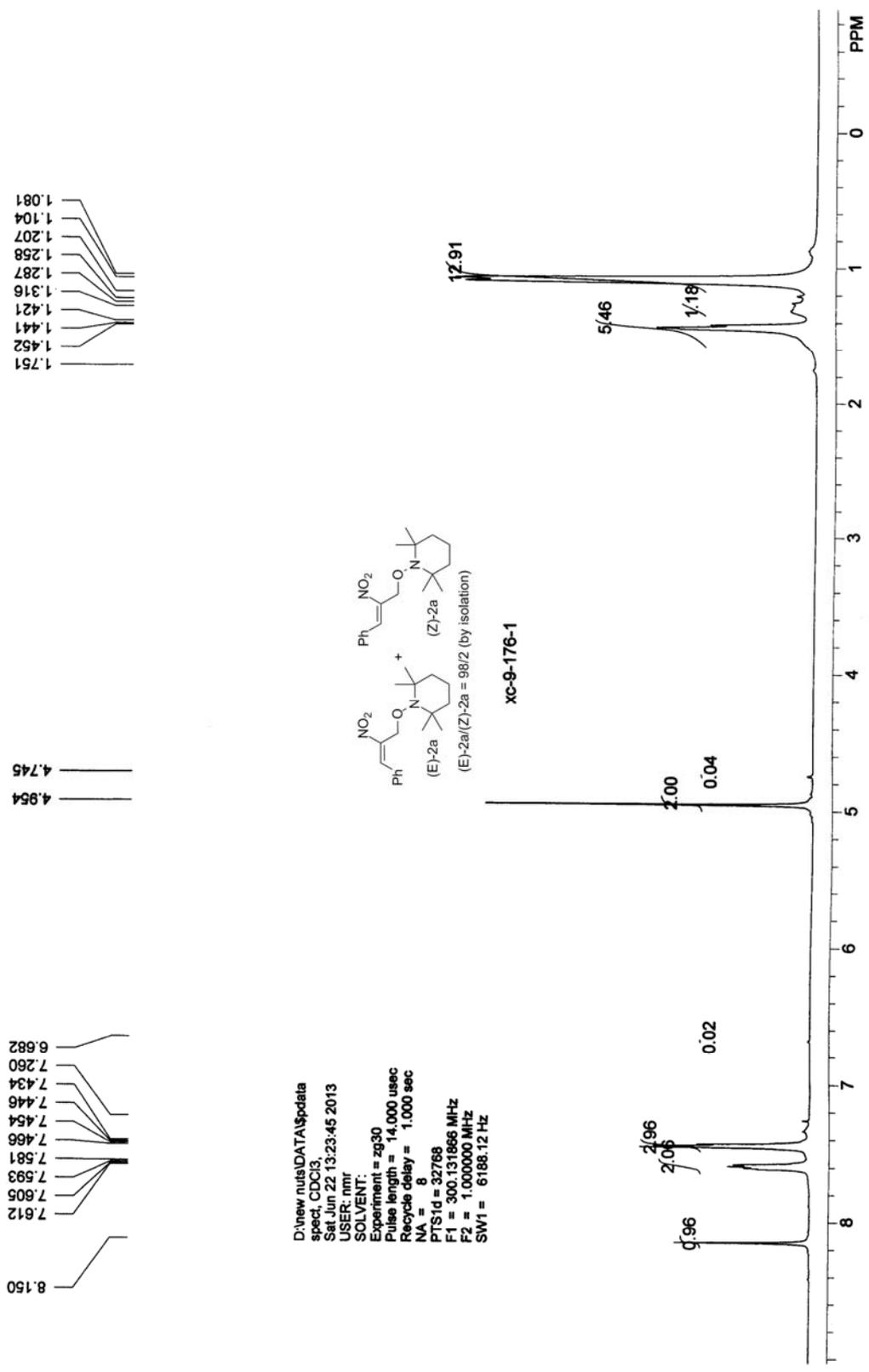


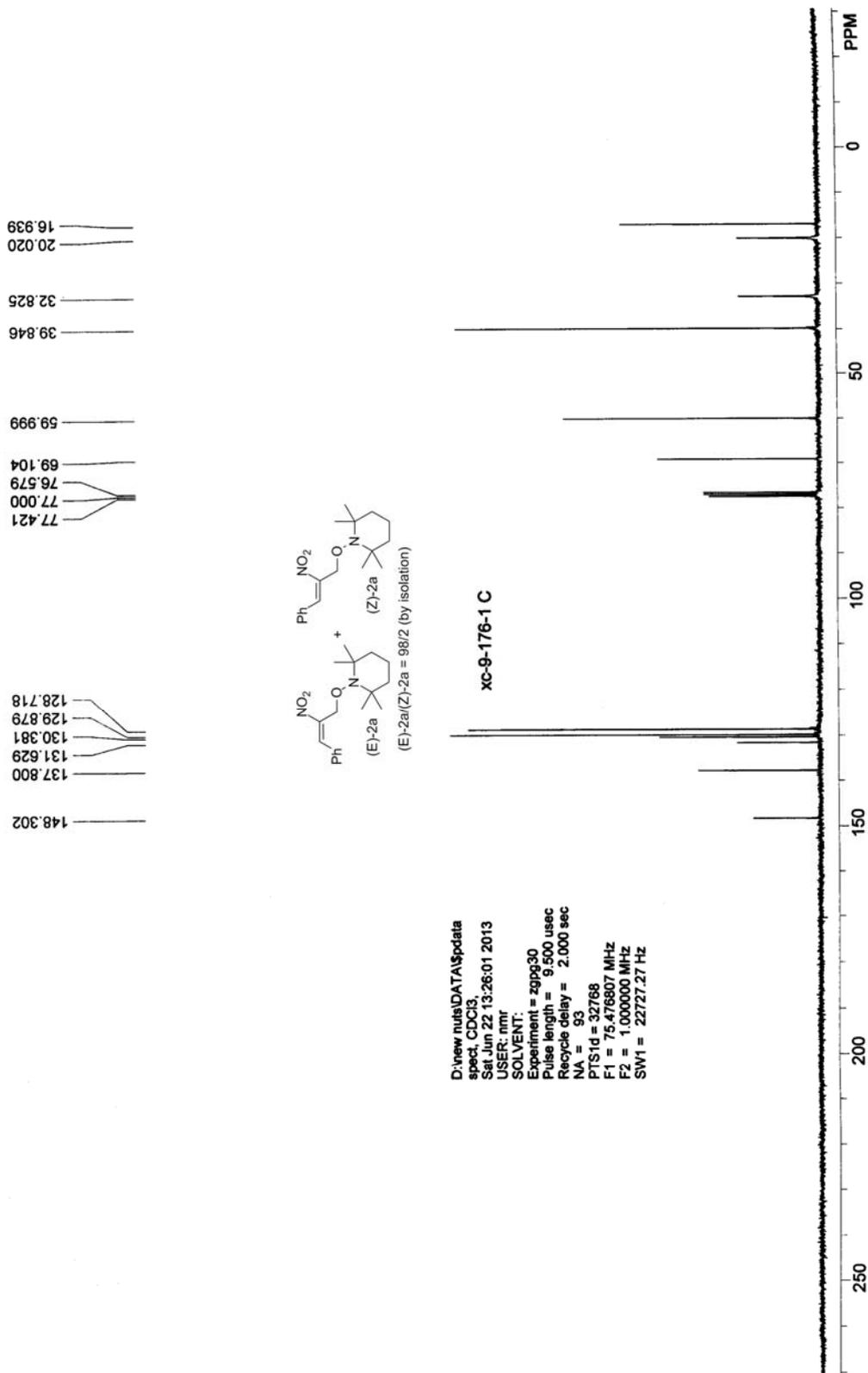
To a dried Schlenk tube were added (*E*)-**2a** (0.3178 g, 1.0 mmol), *N*-bromoacetamide (NBA, 97%, 0.1711 g, 1.2 mmol), K_3PO_4 (0.1065 g, 0.5 mmol) and DCM (10 mL). The resulting mixture was stirred at 25 °C for 24 h as monitored by TLC. The resulting mixture was transfer to a separation funnel with ethyl acetate (20 mL), washed with 10 mL each of water and brine, and dried over anhydrous Na_2SO_4 . After filtration and concentration, the crude product was purified through column chromatography on silica gel (eluent: petroleum ether/ethyl acetate = 50/1 then 5/1) to afford **7a** (0.4121 g, 90%) as a viscous oil: ^1H NMR (300 MHz, CDCl_3) δ 7.38-7.28 (m, 5 H, ArH), 6.33 (d, $J = 9.9$ Hz, 1 H, NH), 5.97 (d, $J = 9.9$ Hz, 1 H, CH), 4.77 (d, $J = 10.8$ Hz, 1 H, one proton of CH_2), 4.20 (d, $J = 10.8$ Hz, 1 H, one proton of CH_2), 2.06 (s, 3 H, CH_3), 1.54-1.35 (m, 5 H, 2 $\times\text{CH}_2$ and one proton of CH_2), 1.33-1.24 (m, 1 H, one proton of CH_2), 1.18 (s, 3 H, CH_3), 1.12 (s, 6 H, 2 $\times\text{CH}_3$), 1.00 (s, 3 H, CH_3); ^{13}C NMR (75 MHz, CDCl_3) δ 169.0, 133.9, 129.3, 128.6, 128.3, 105.8, 79.7, 60.9, 60.7, 56.9, 39.9, 32.4, 23.3, 20.3, 20.2, 16.8; IR (neat) ν (cm^{-1}) 3296, 2974, 2933, 2872, 1738, 1660, 1568, 1532, 1471, 1453, 1374, 1328, 1245, 1207, 1131, 1101, 1083, 1046; MS (ESI): $m/z = 458$ $[\text{M}+\text{H}]^+$; HRMS (ESI): calcd. For $\text{C}_{20}\text{H}_{31}\text{BrN}_3\text{O}_4$: $[\text{M}+\text{H}]^+$ 456.1492; found 456.1500.

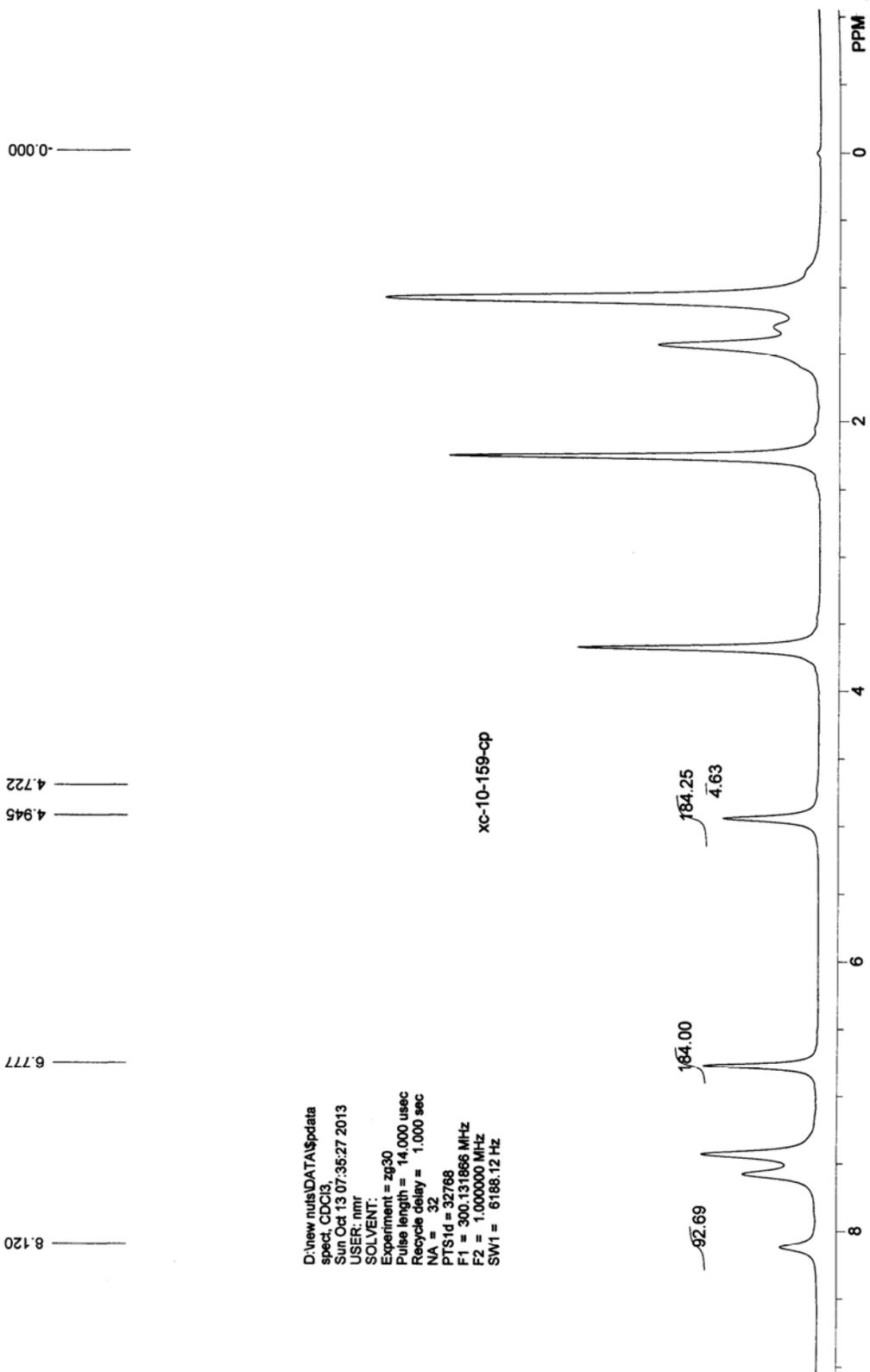
Reference:

1. Kuang, J.-Q.; Ma, S.-M. *J. Org. Chem.*, 2009, **74**, 1763.
2. S. P. Waters, M. W. Fennie, M. C. Kozlowski, *Org. Lett.*, 2006, **8**, 3243.
3. N. Kise, S. Isemoto, T. Sakurai, *J. Org. Chem.* 2011, **76**, 9856.
4. Z.-G. Chen, Y. Wang, J.-F. Wei, P.-F. Zhao, X.-Y. Shi, *J. Org. Chem.*, 2010, **75**, 2085.

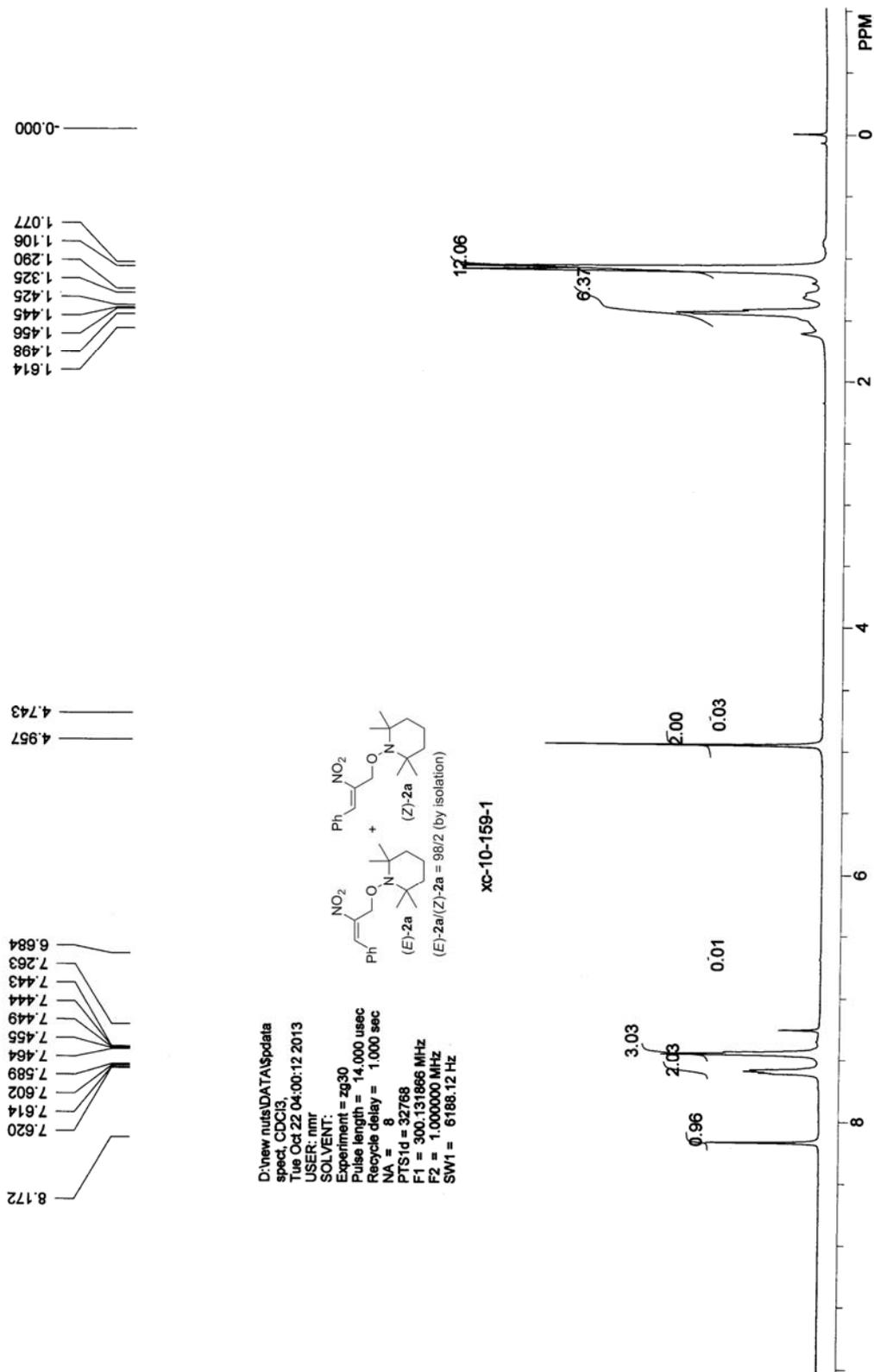




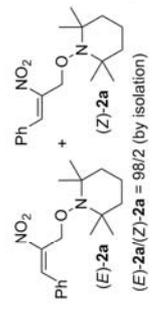




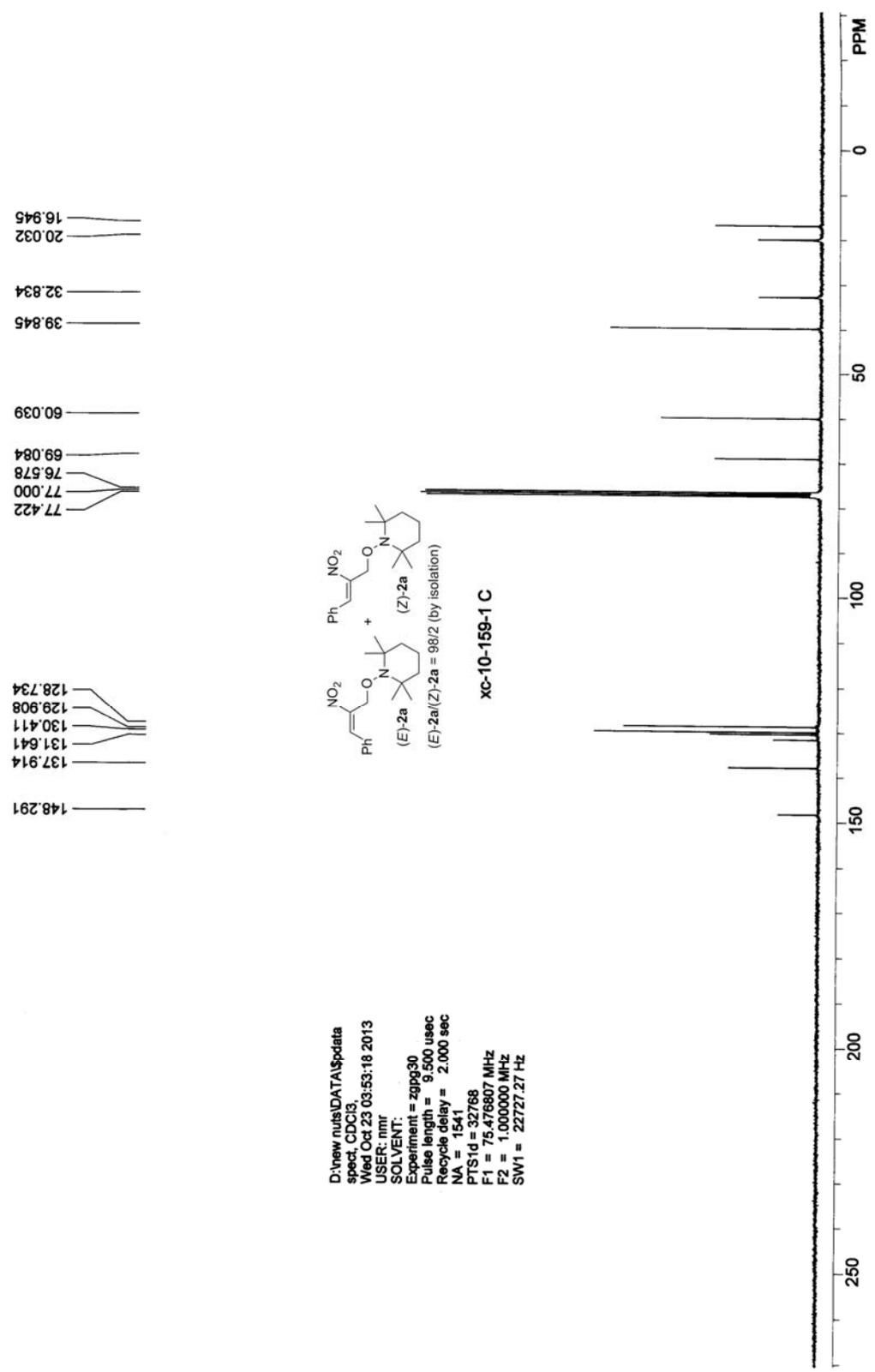
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 F2 = 1,000,000 MHz
 SW1 = 6188.12 Hz



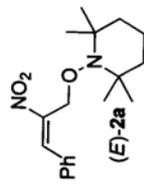
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 F2 = 1.000000 MHz
 SW1 = 22727.27 Hz



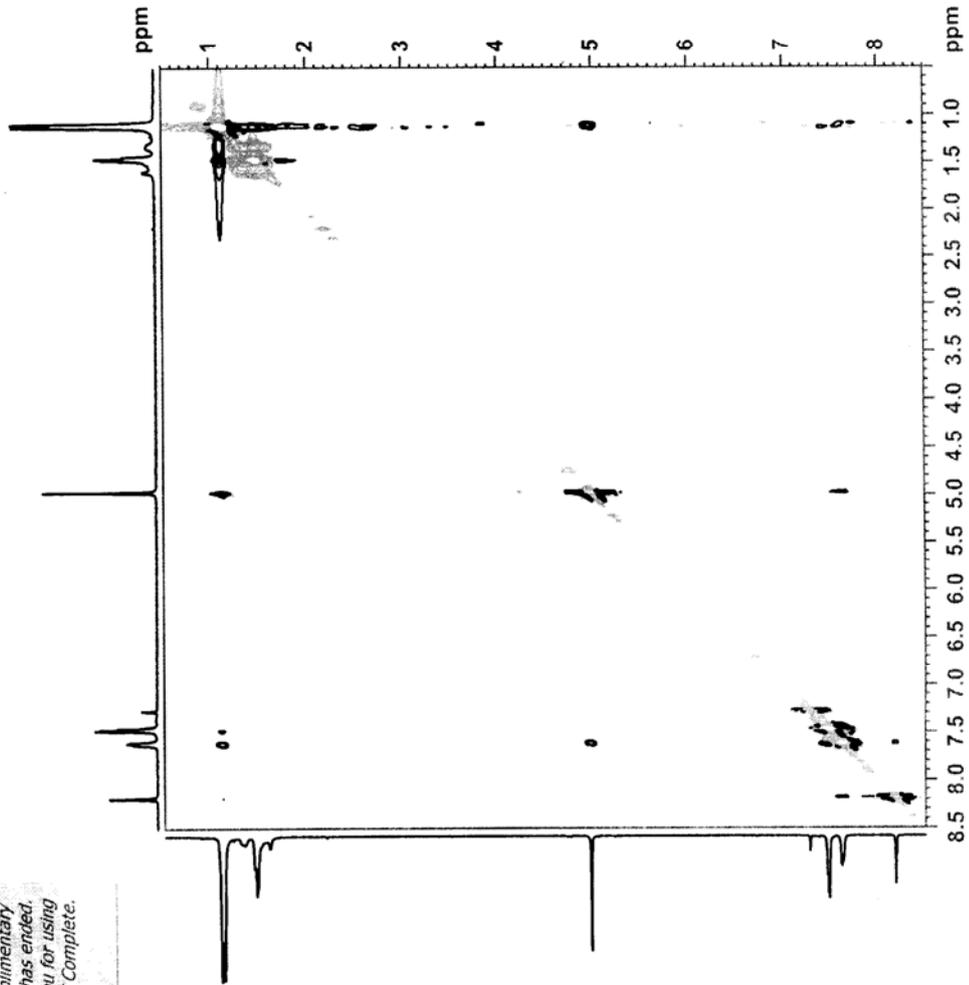
xc-10-159-1 C

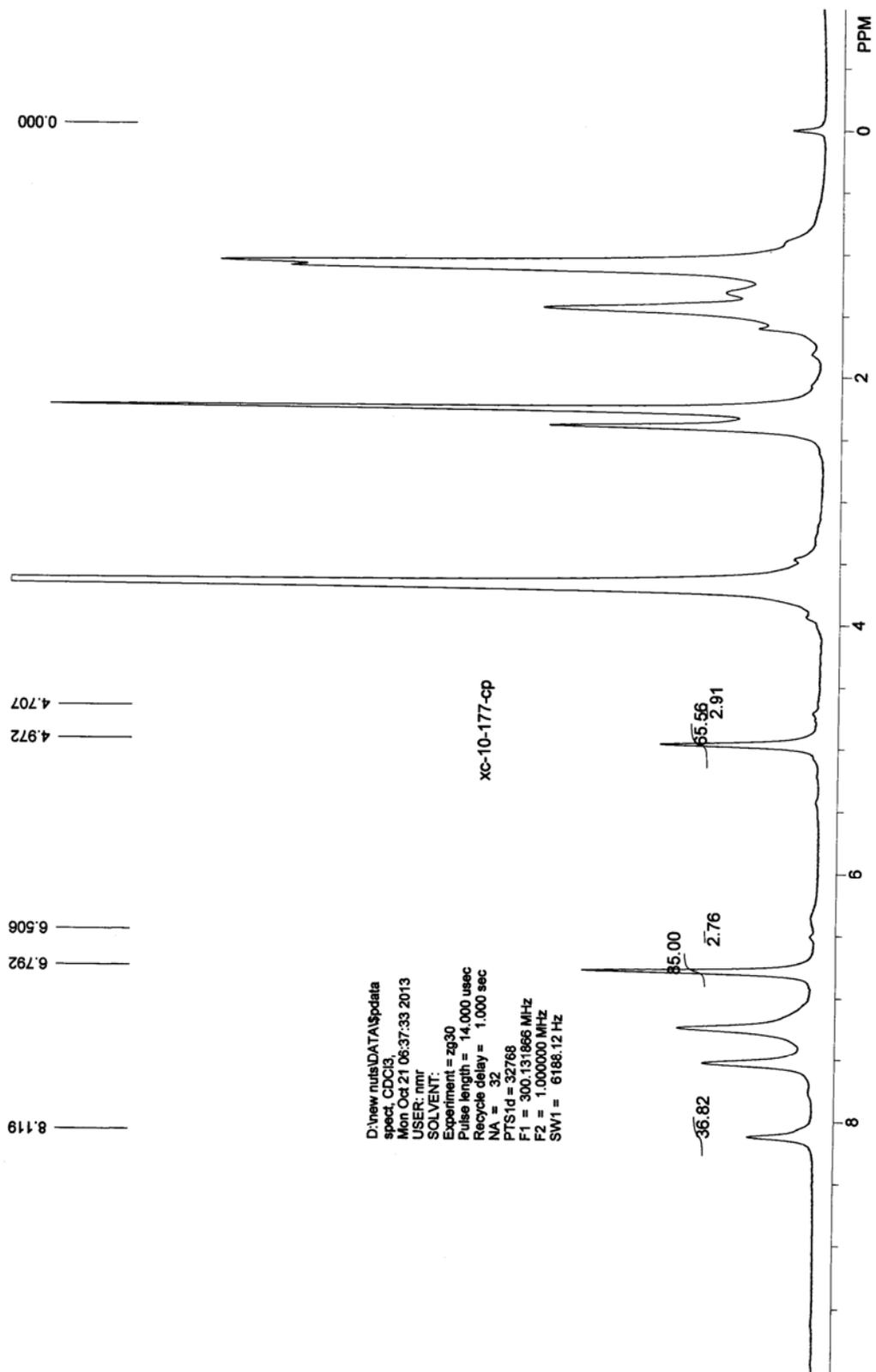


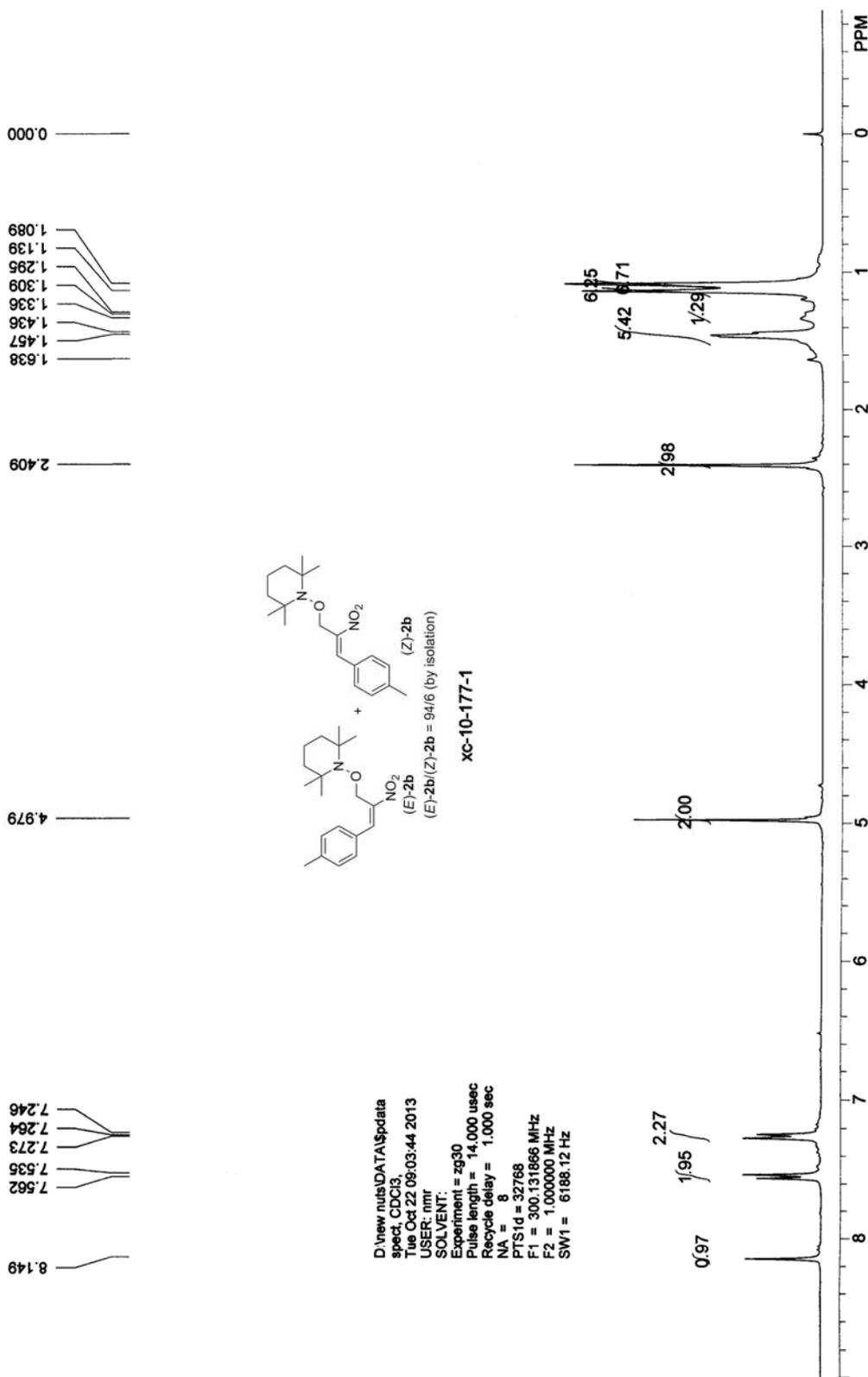
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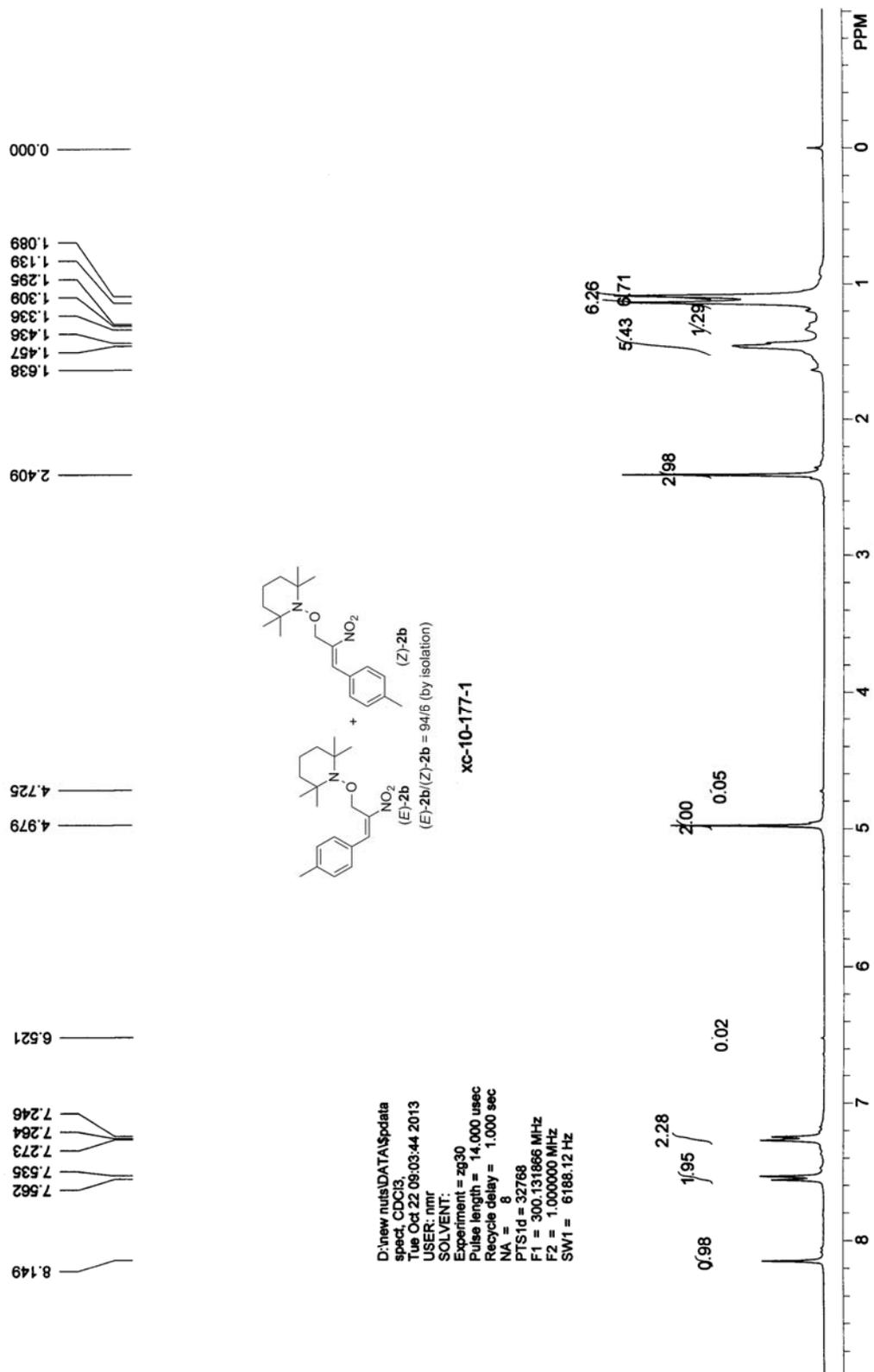


xc-10-159-1-noe

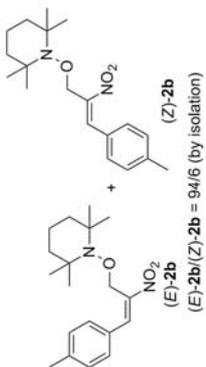




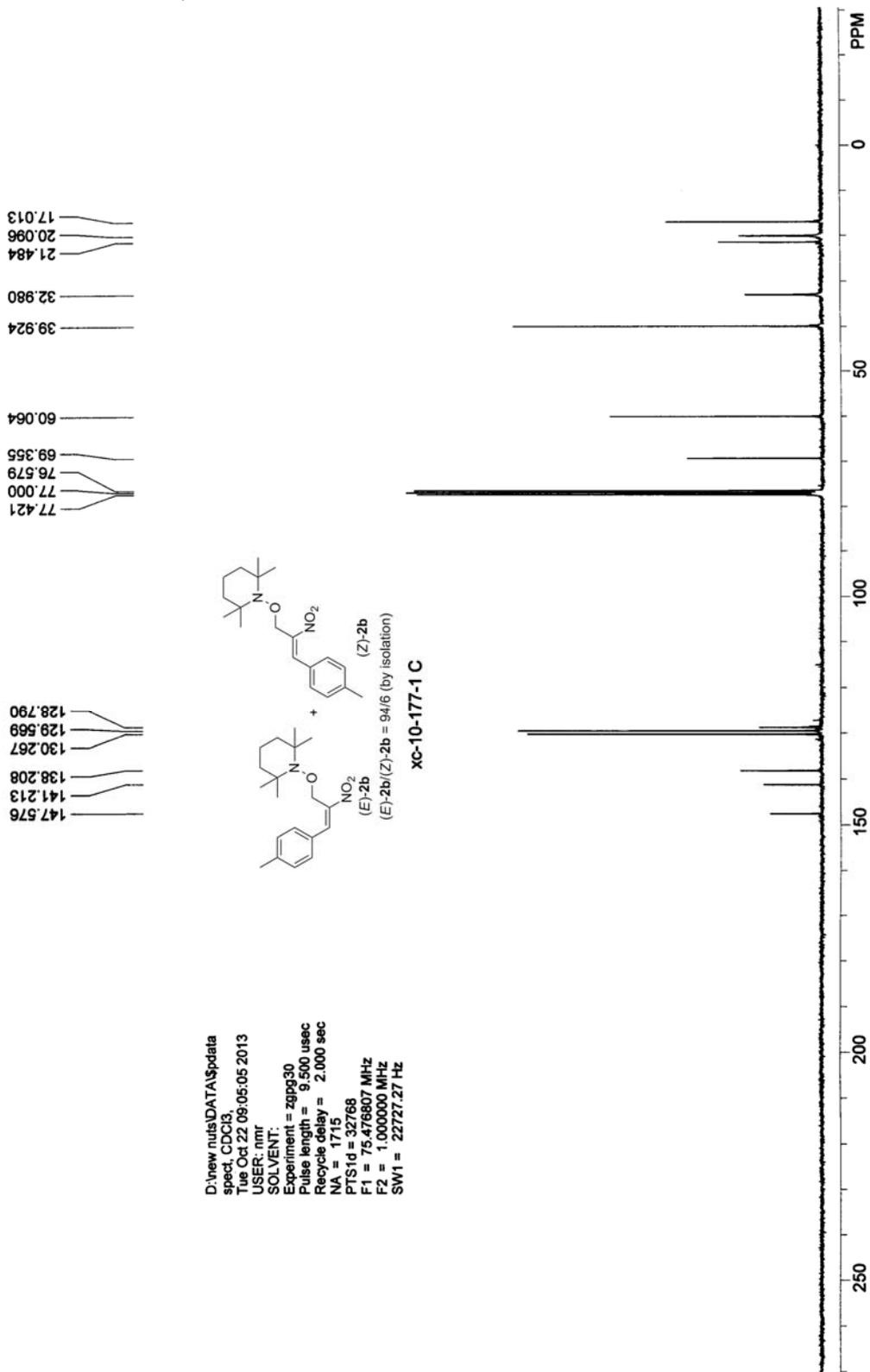


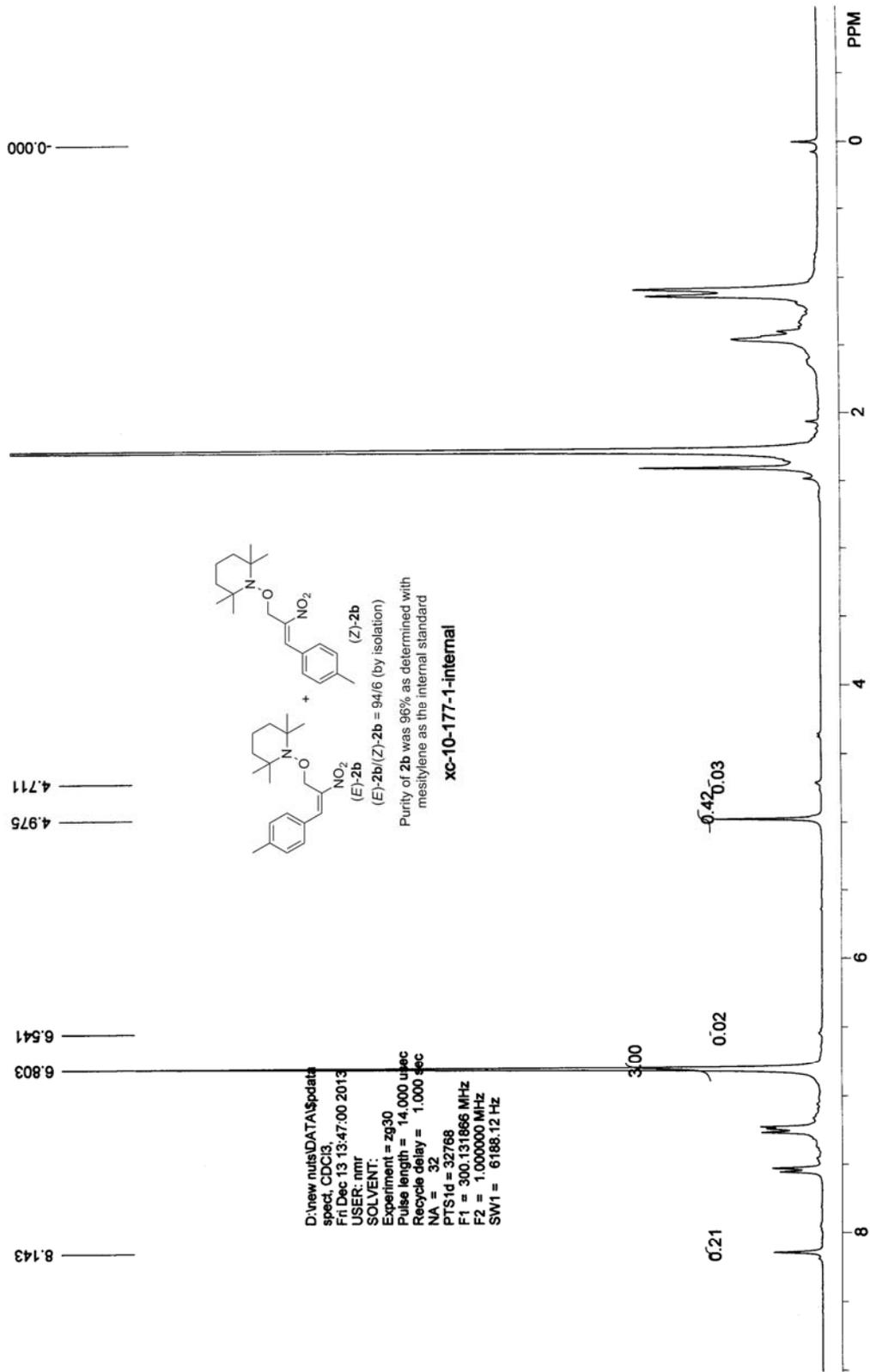


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 SW1 = 22727.27 Hz

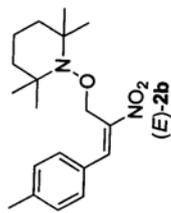
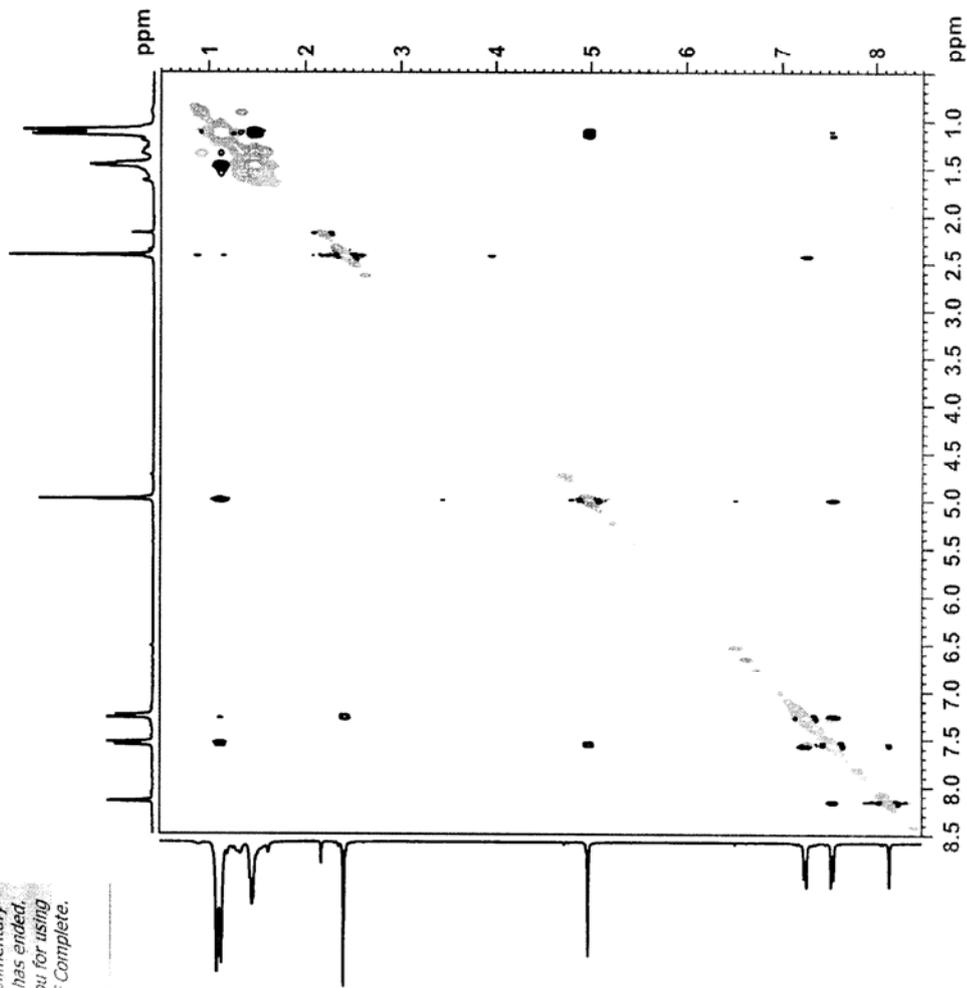


xc-10-177-1 C

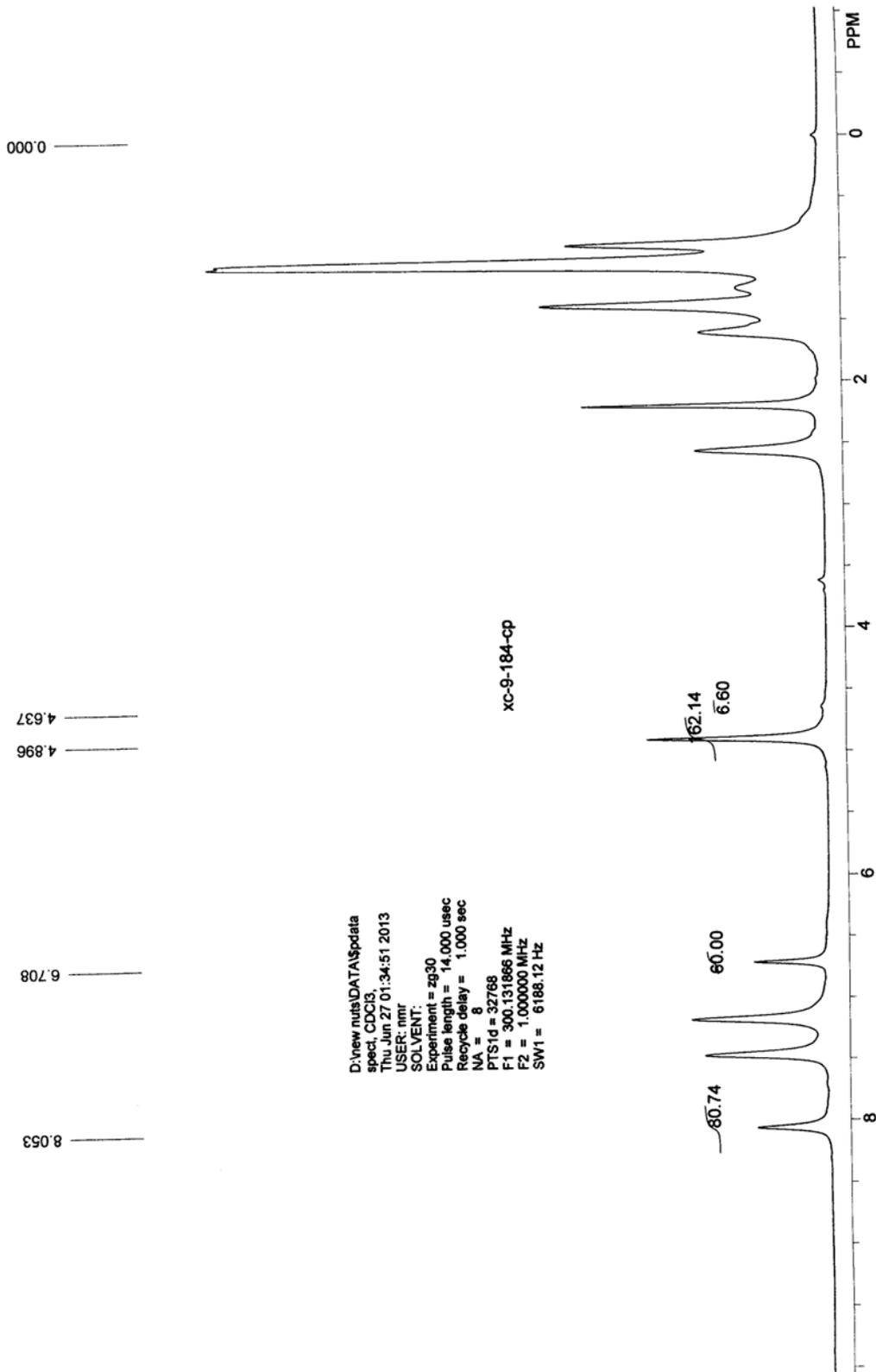


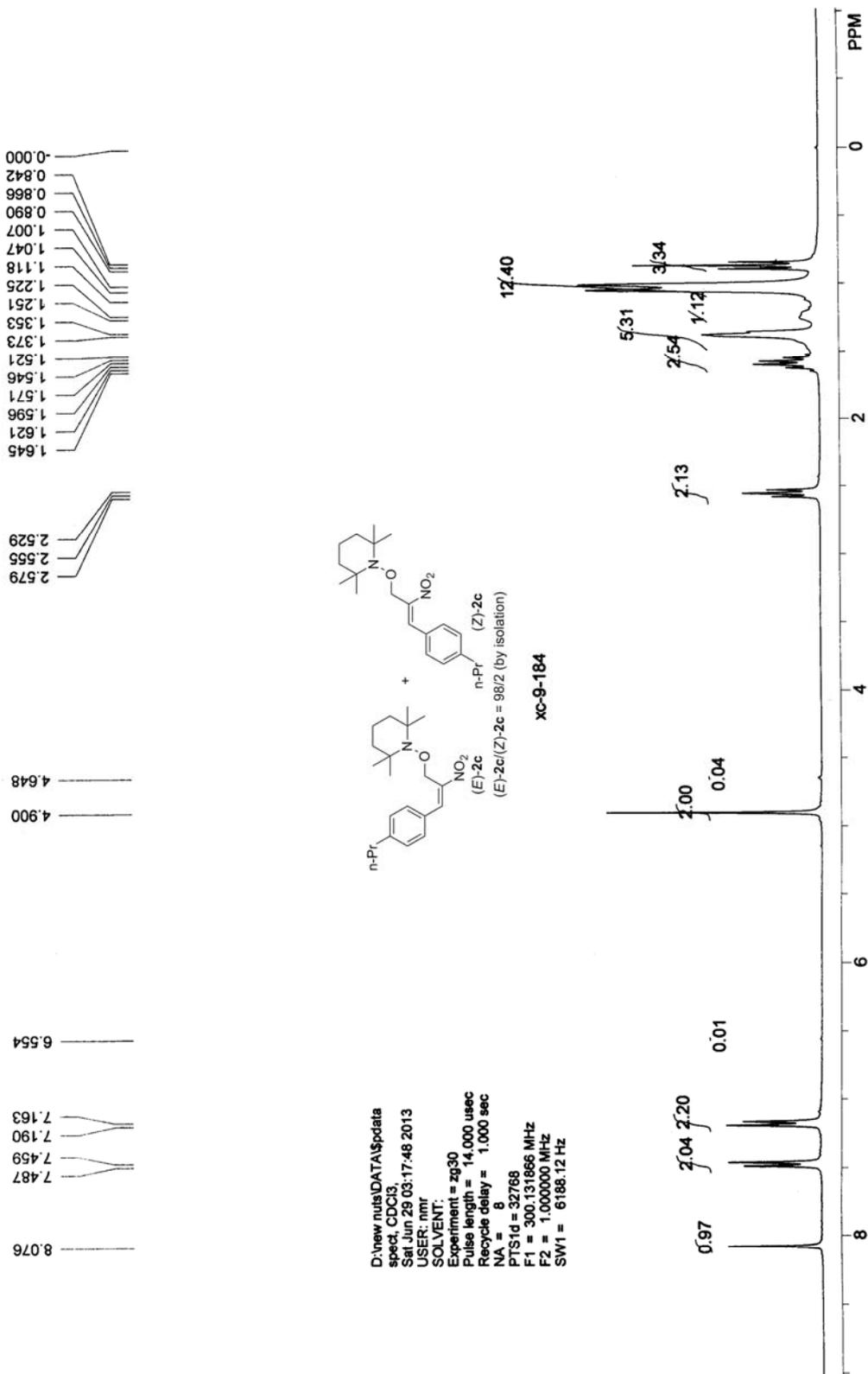


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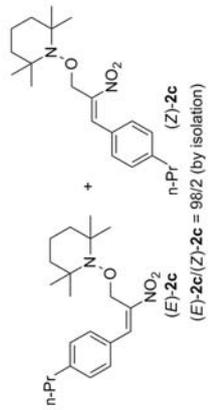


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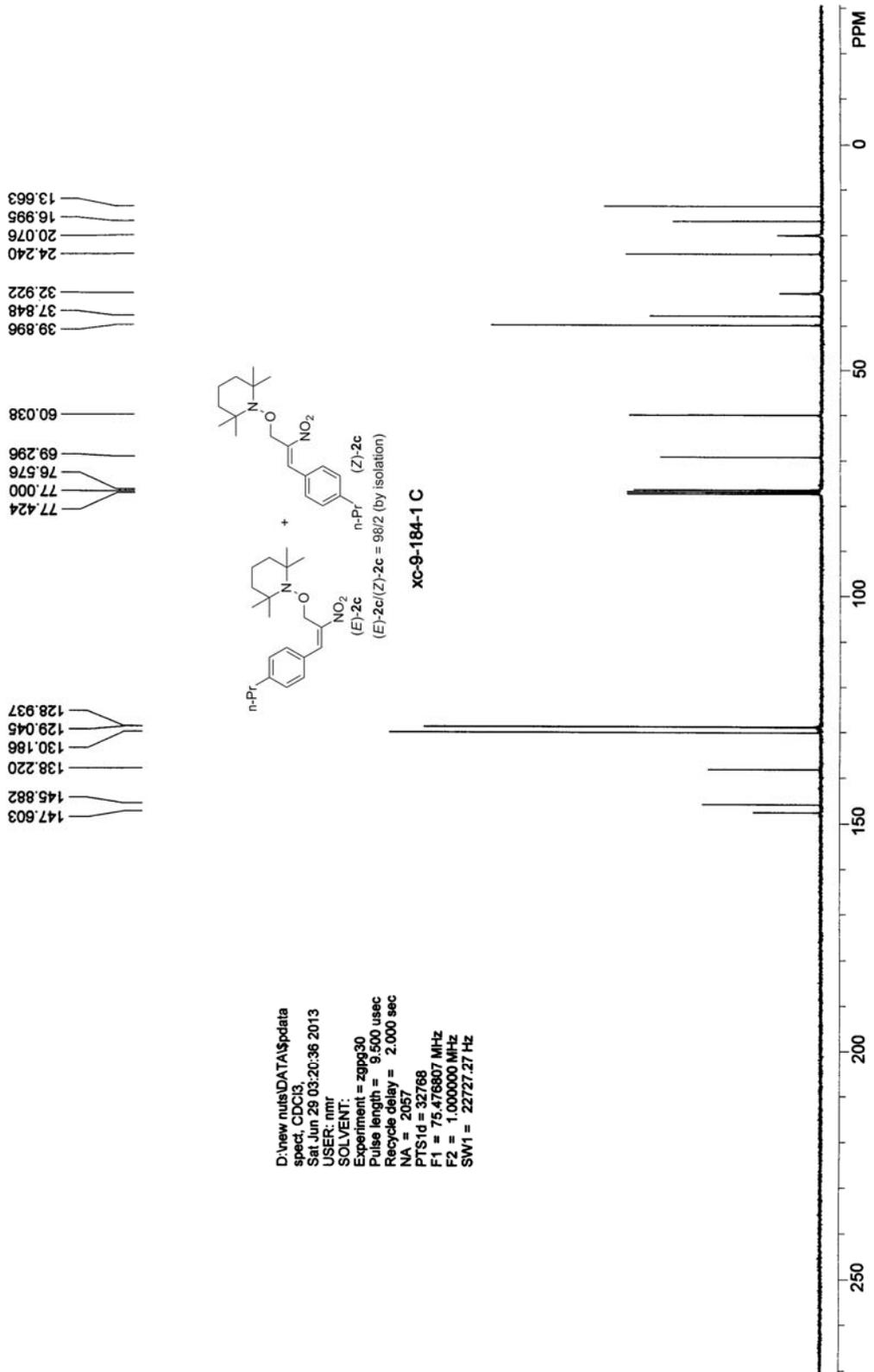




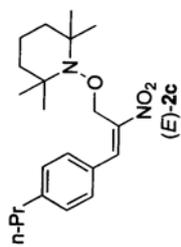
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 Sat Jun 29 03:20:36 2013
 USER: nmr
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 SW1 = 22727.27 Hz



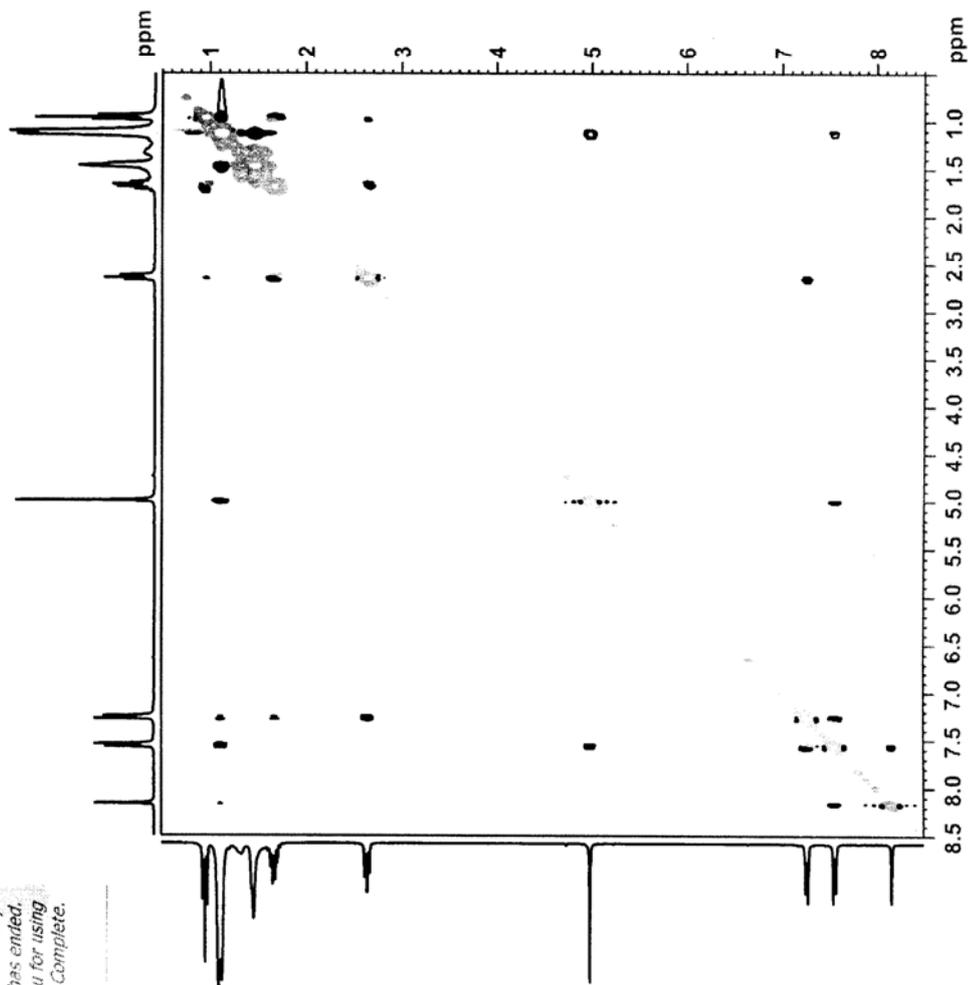
xc-9-184-1 C

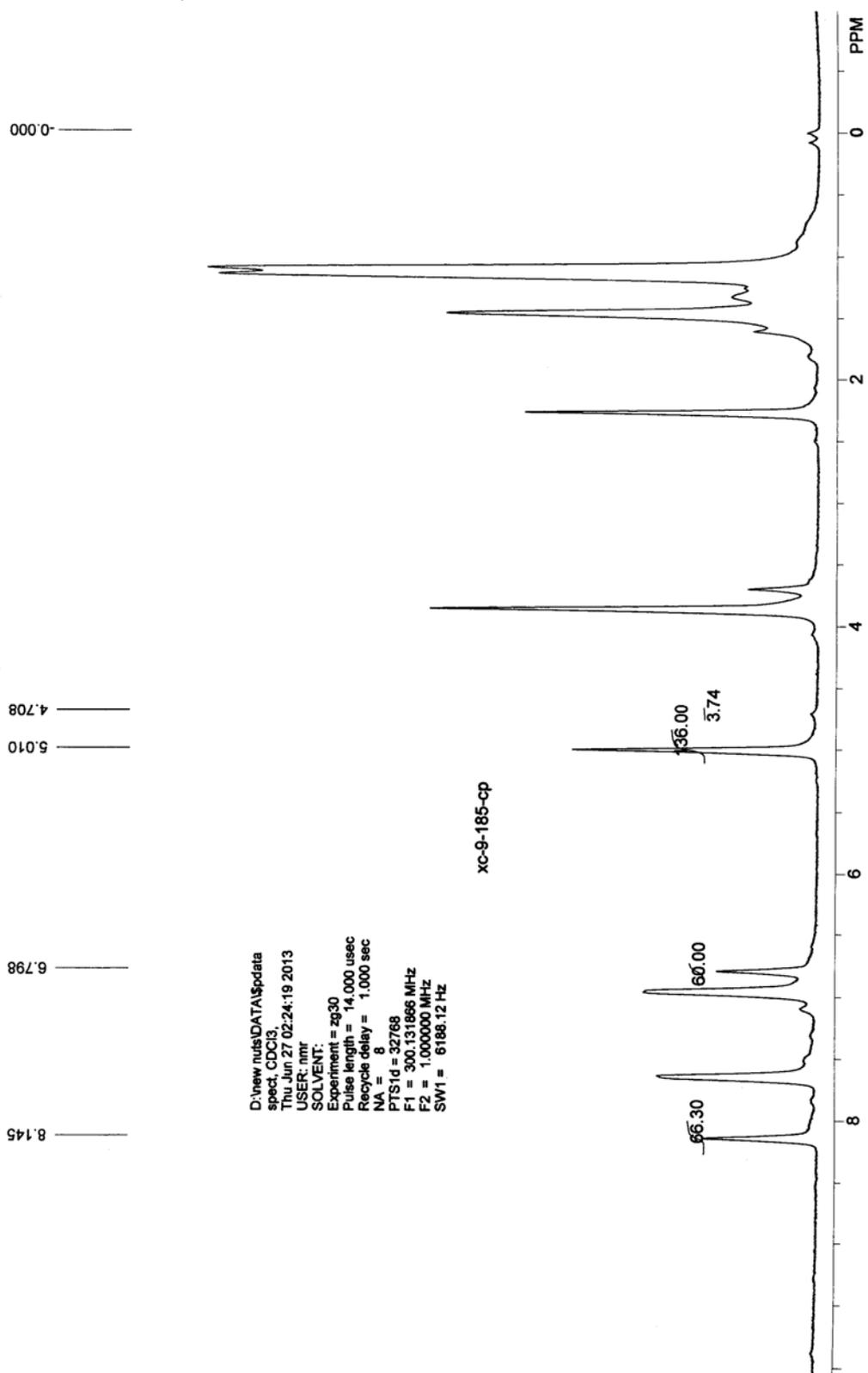


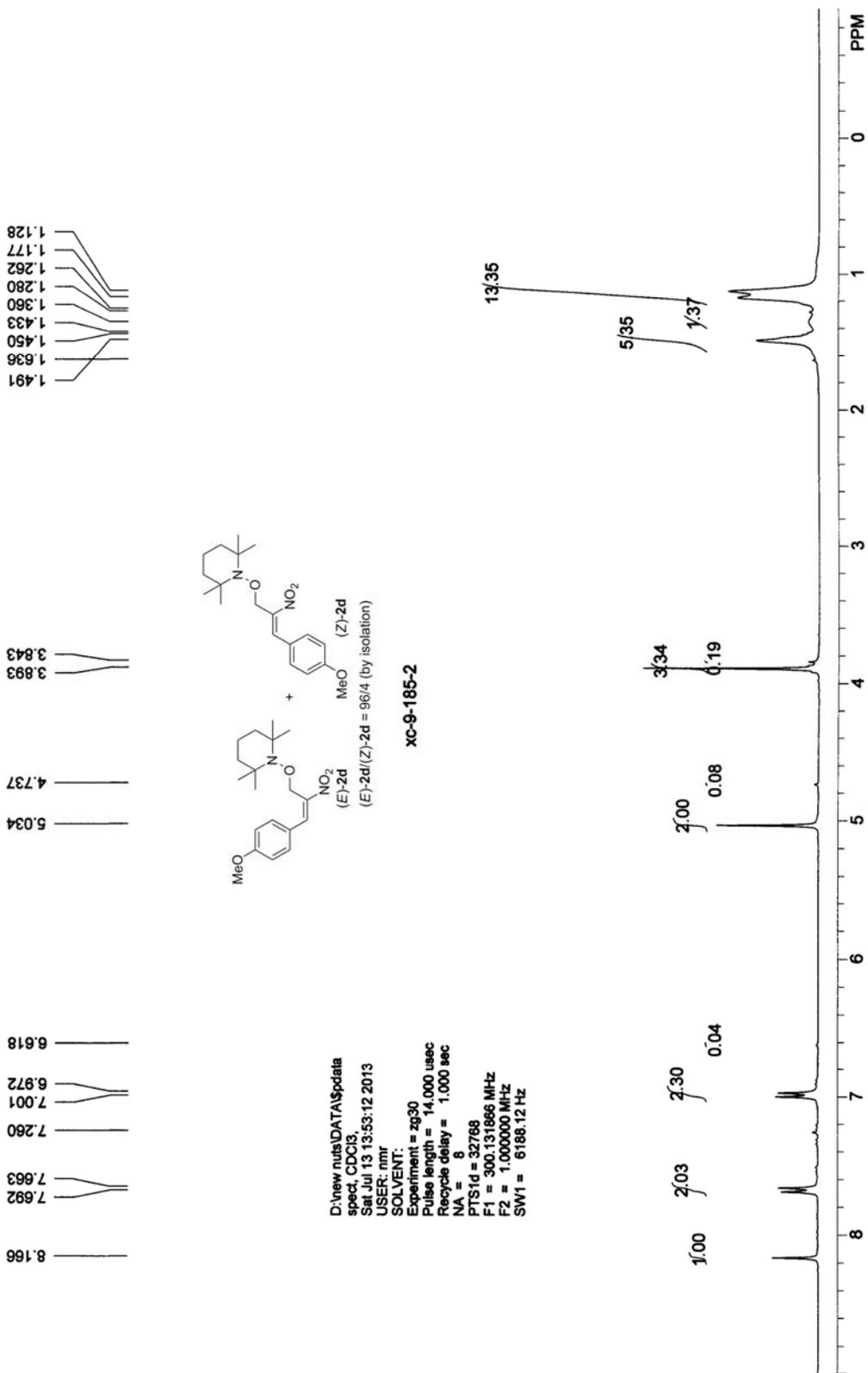
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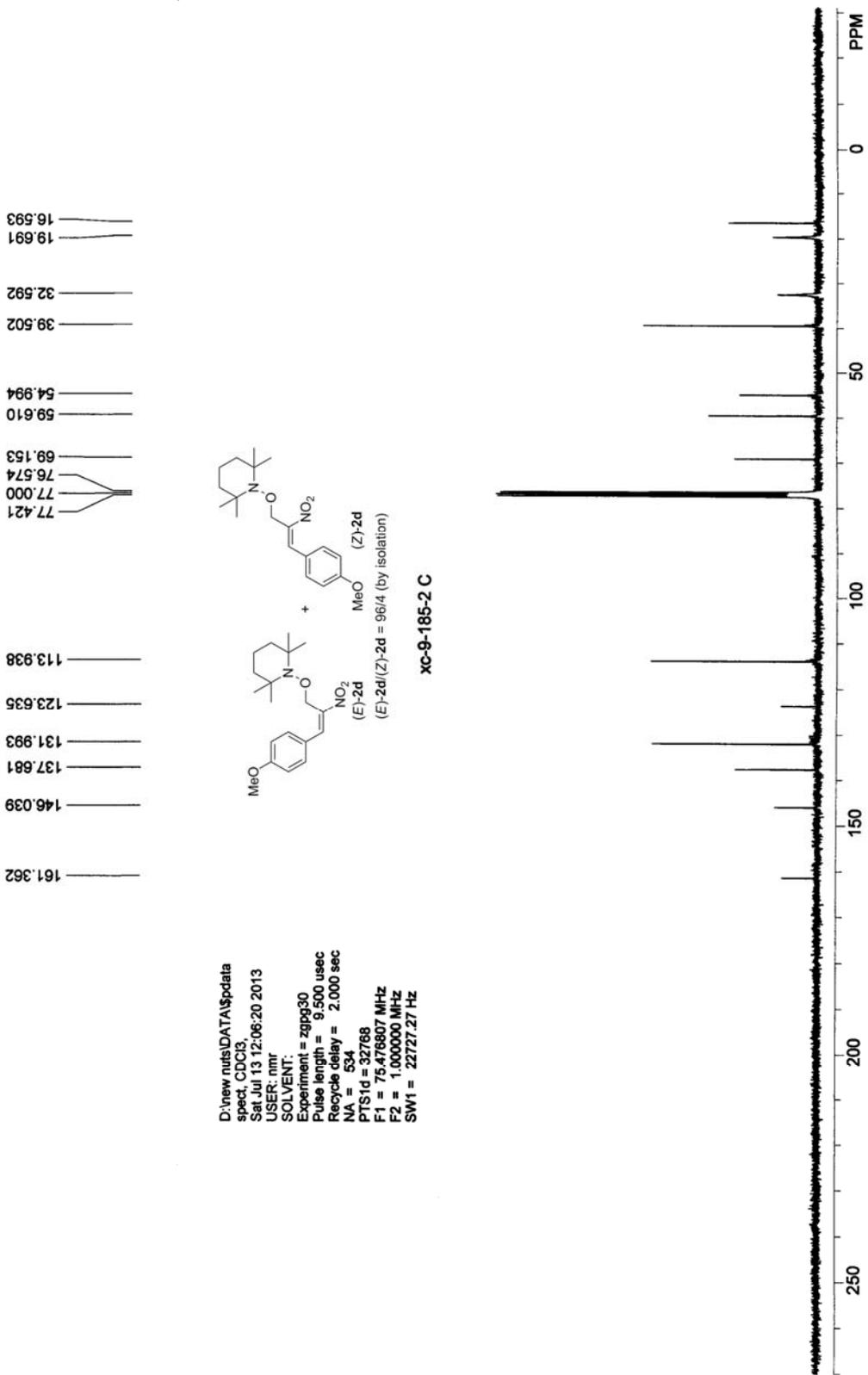


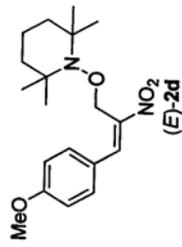
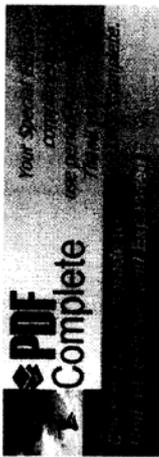
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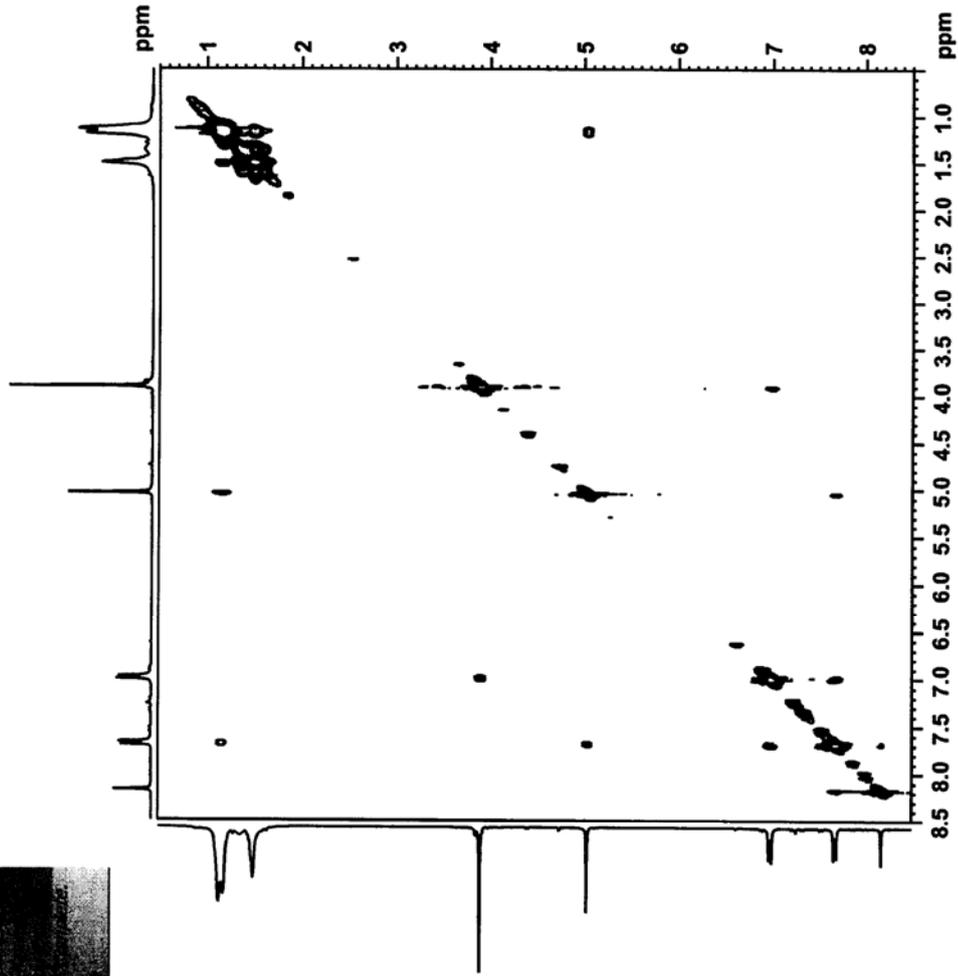


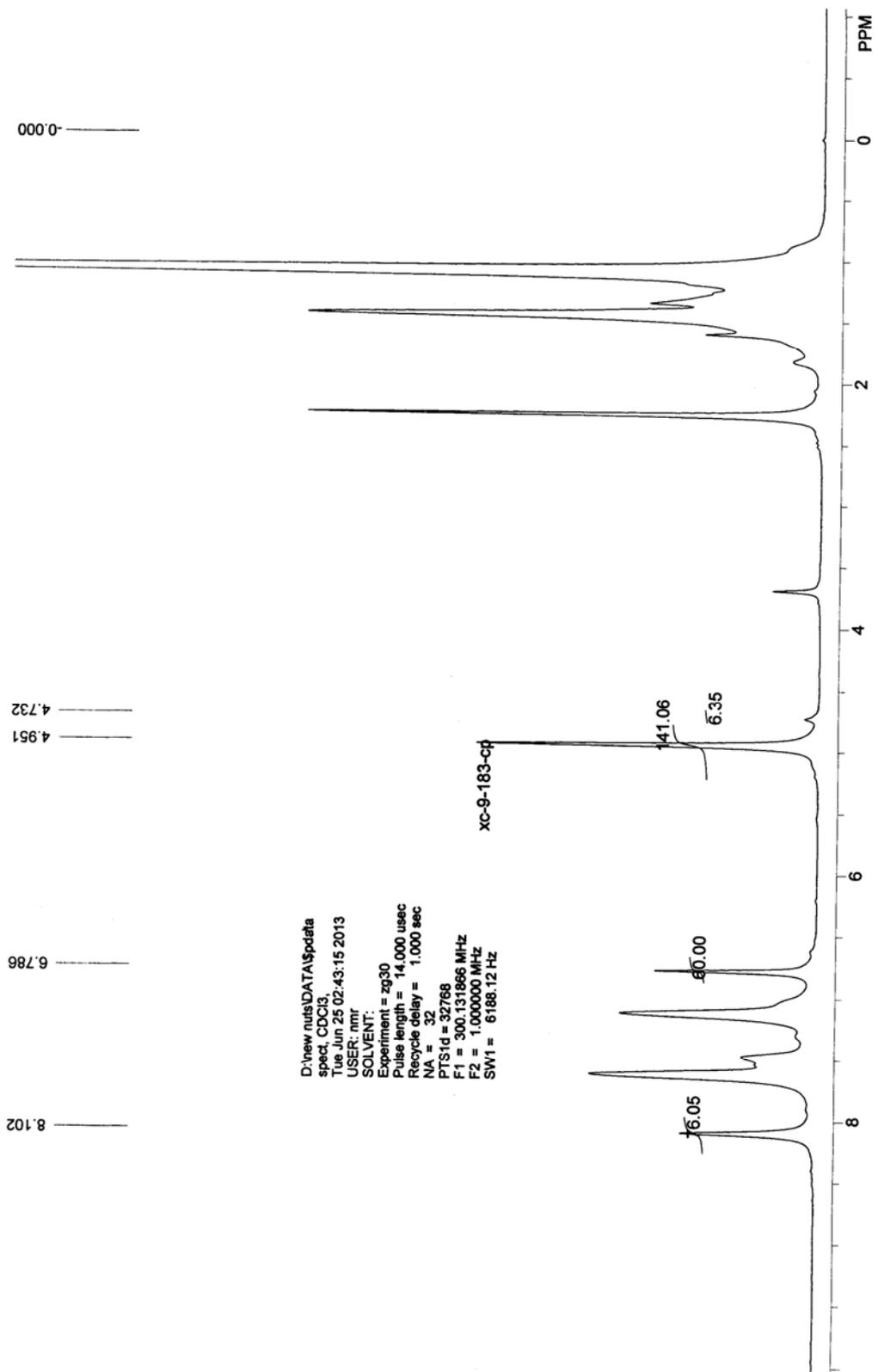


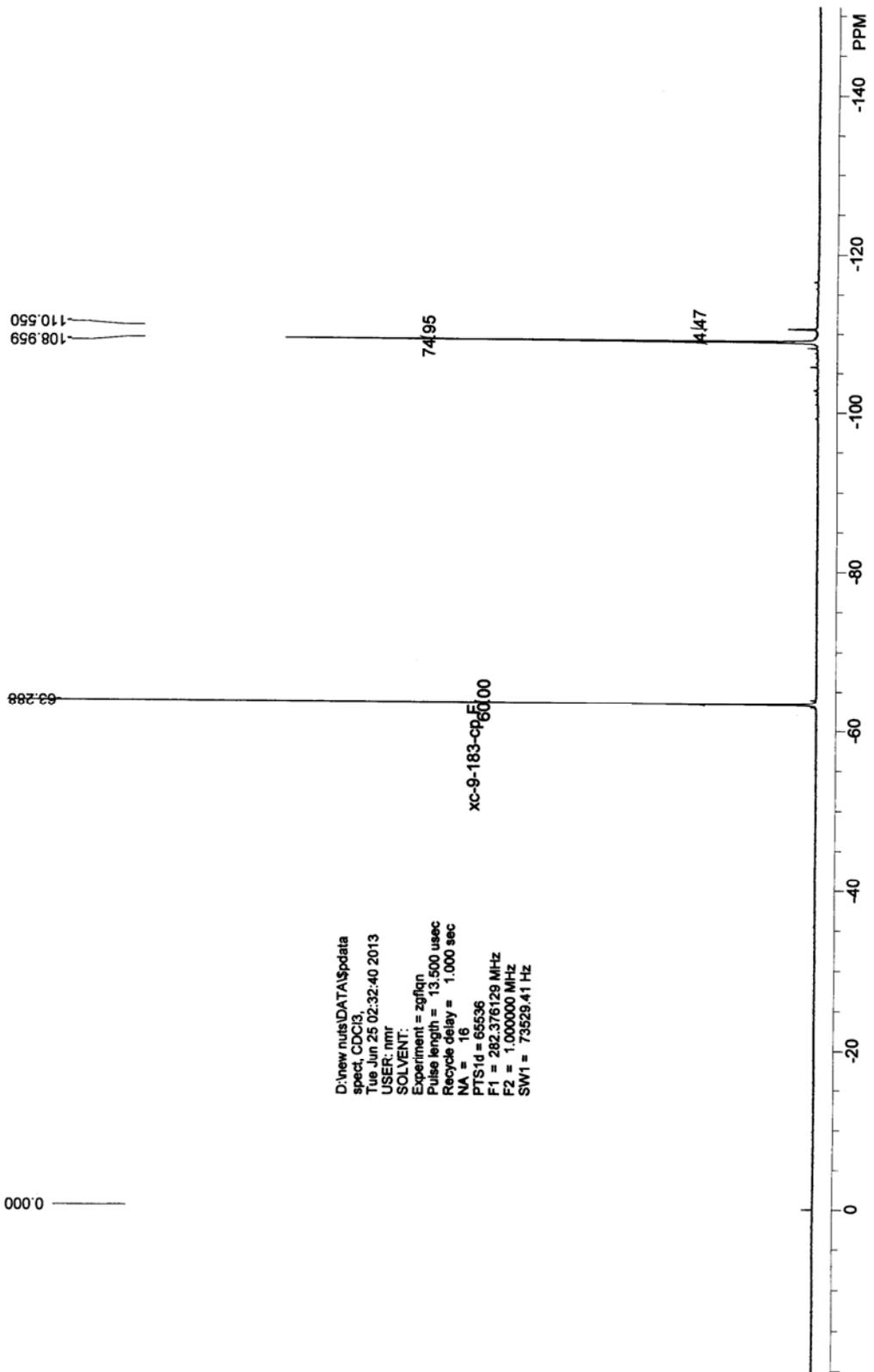


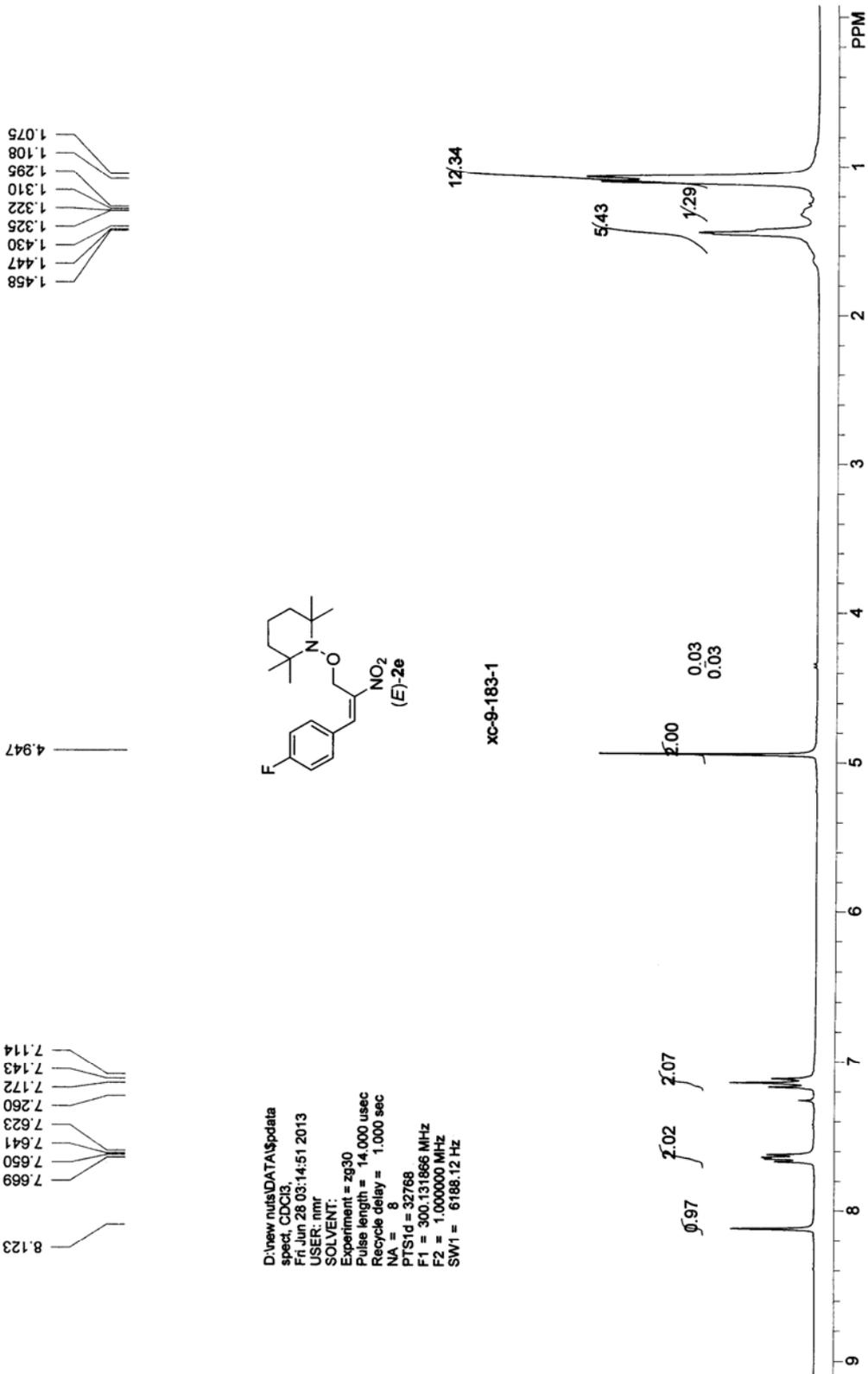


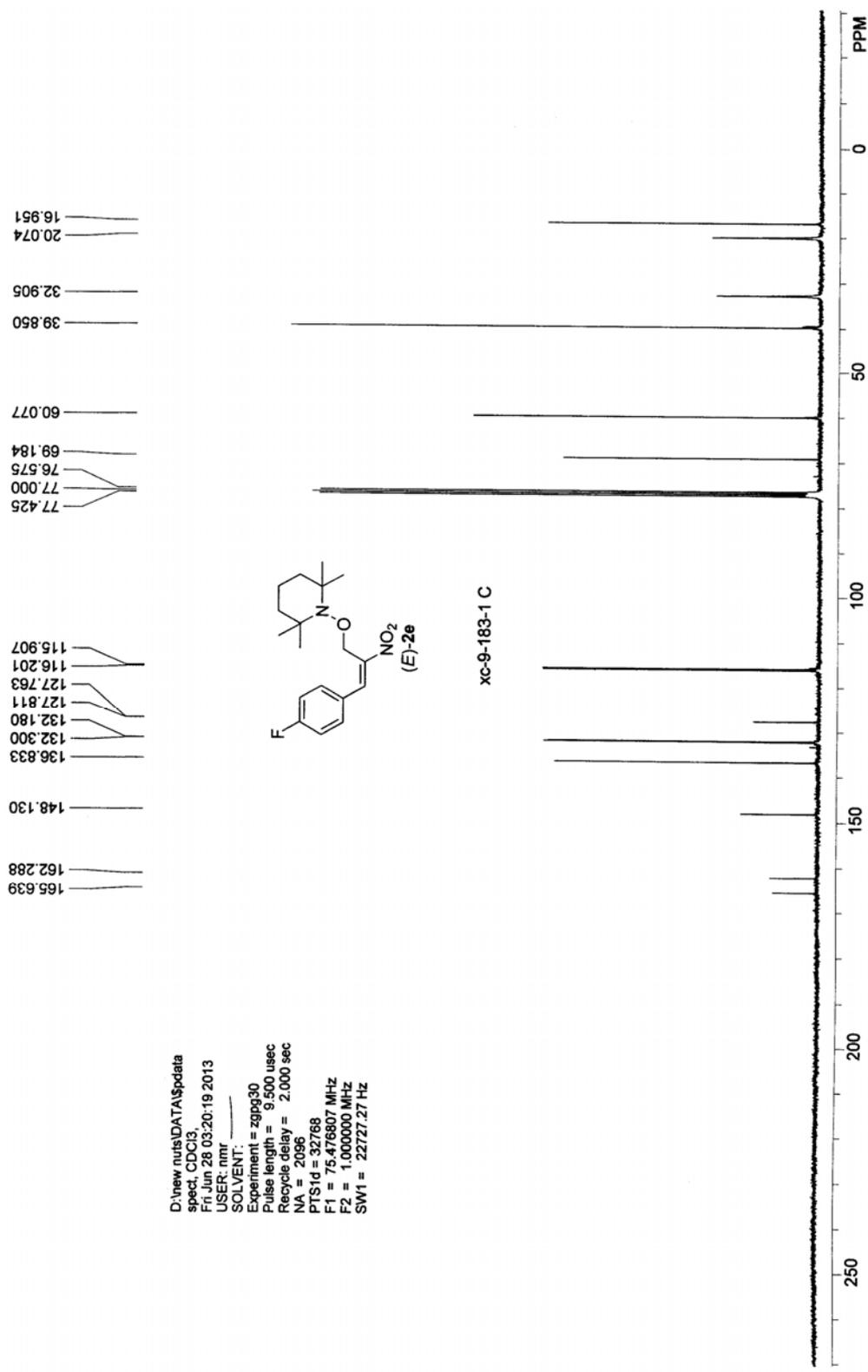
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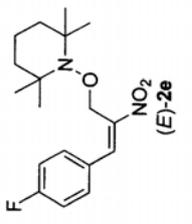




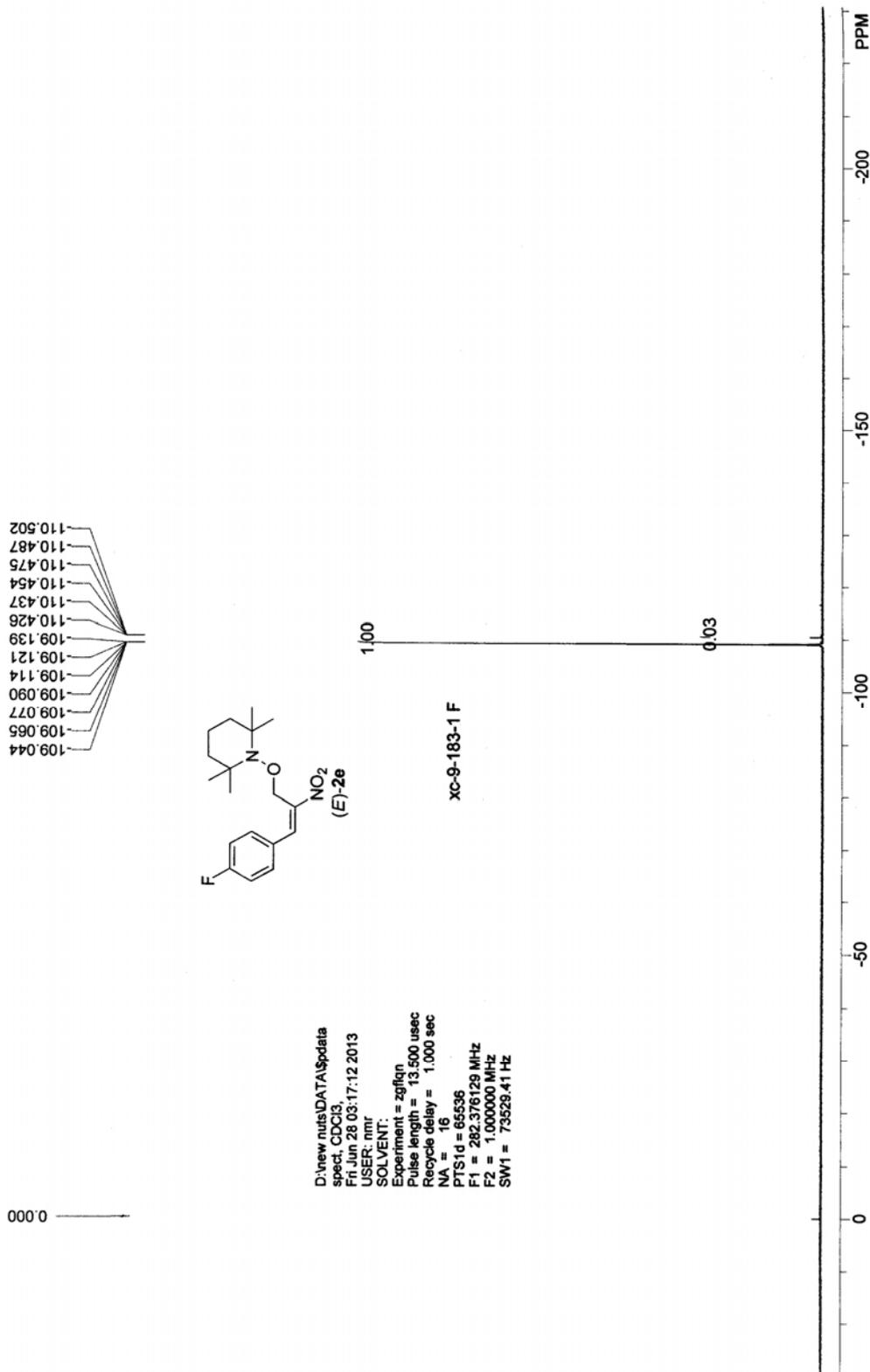


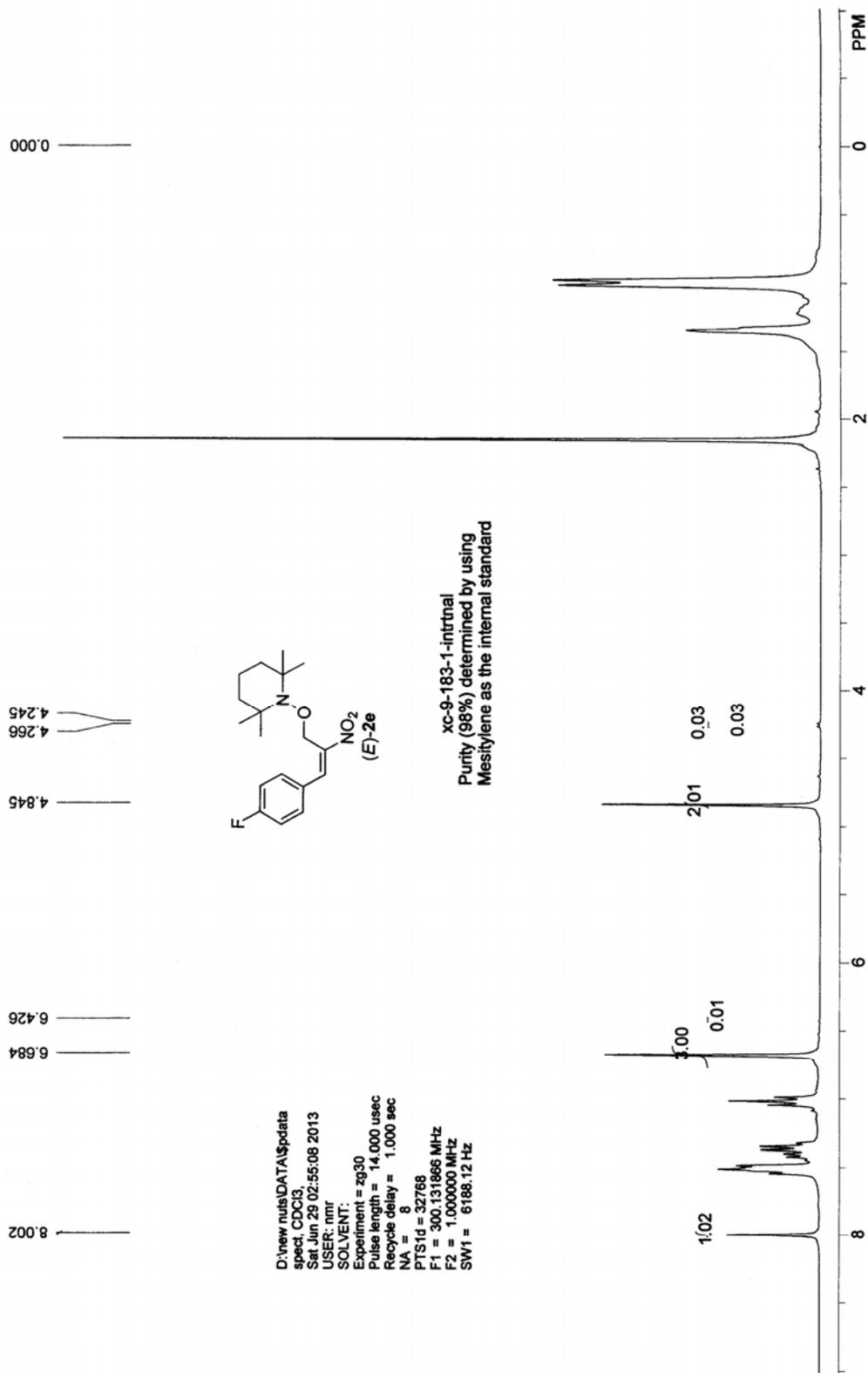


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F2 = 1.000000 MHz
SW1 = 22727.27 Hz

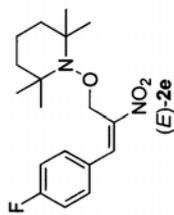
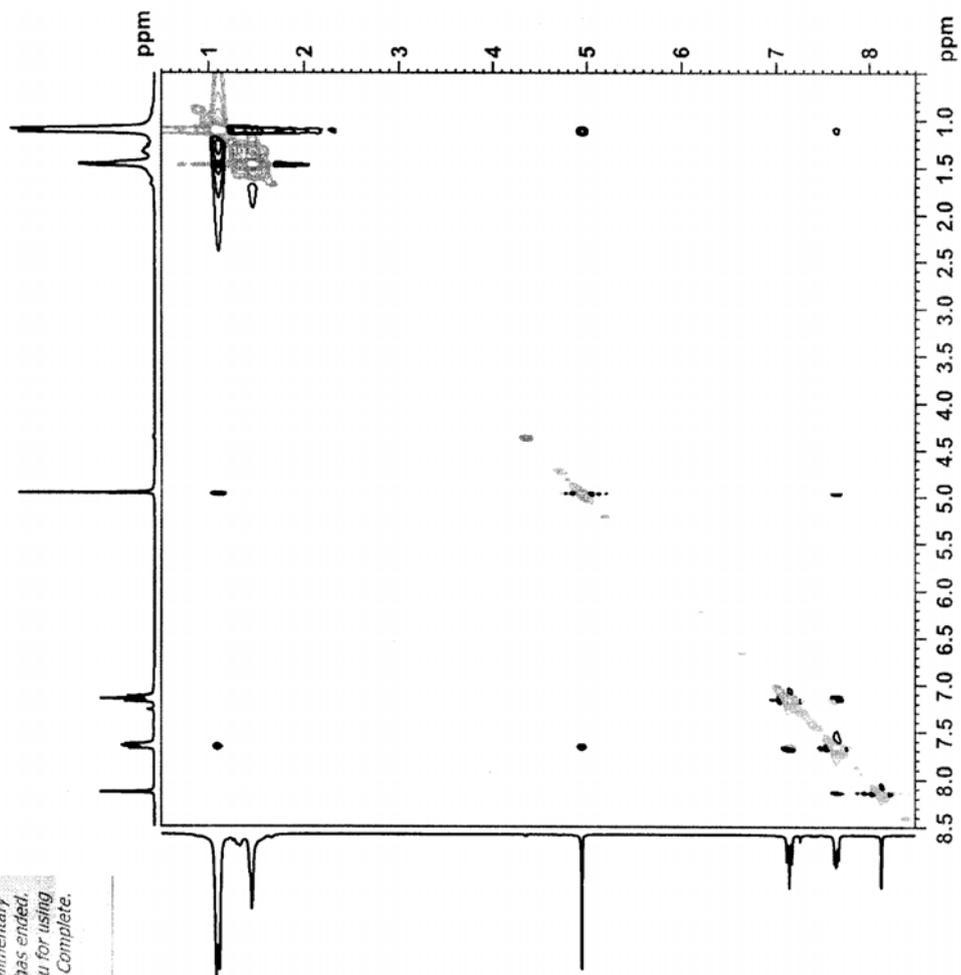


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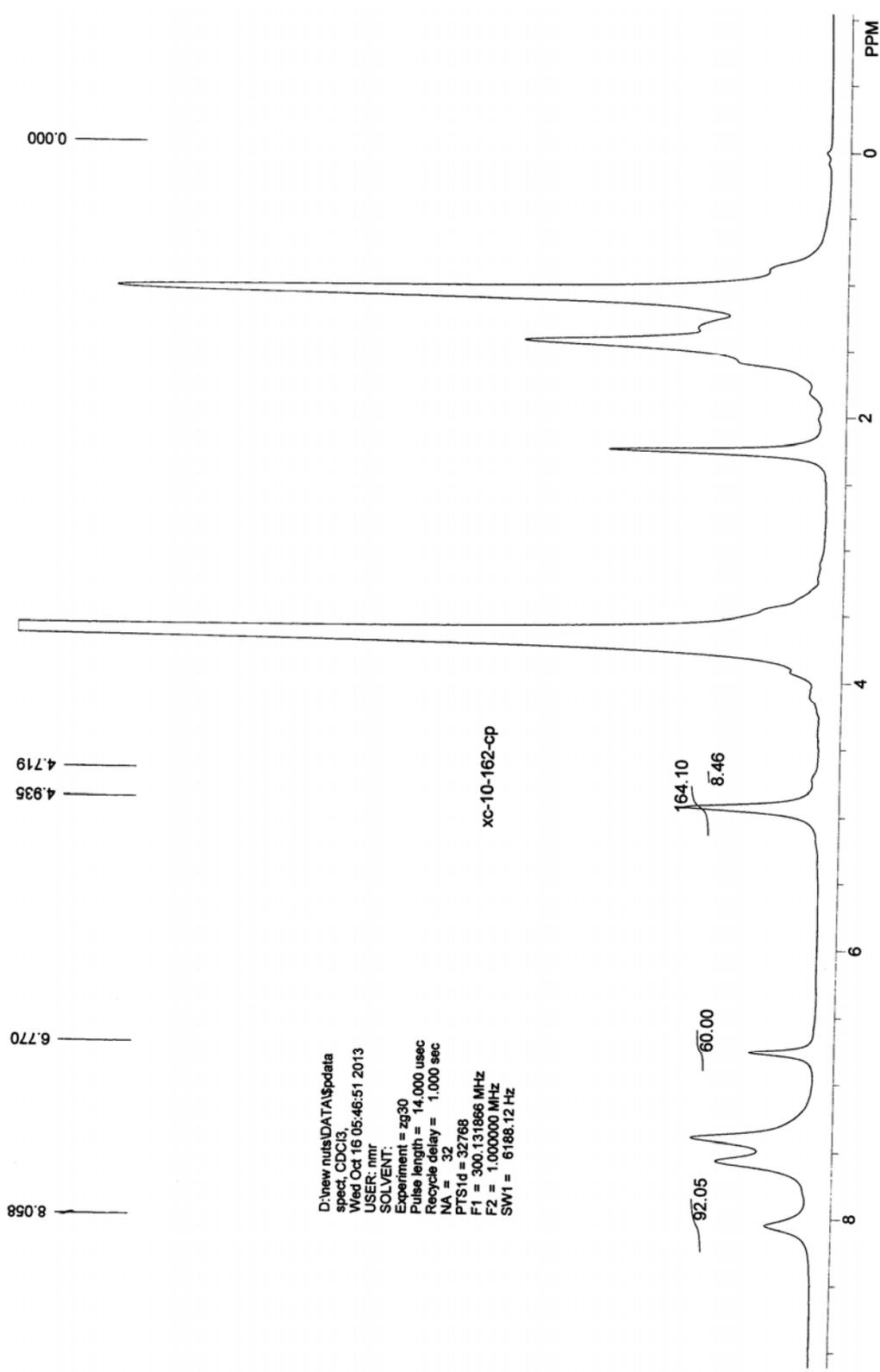




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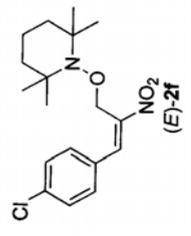
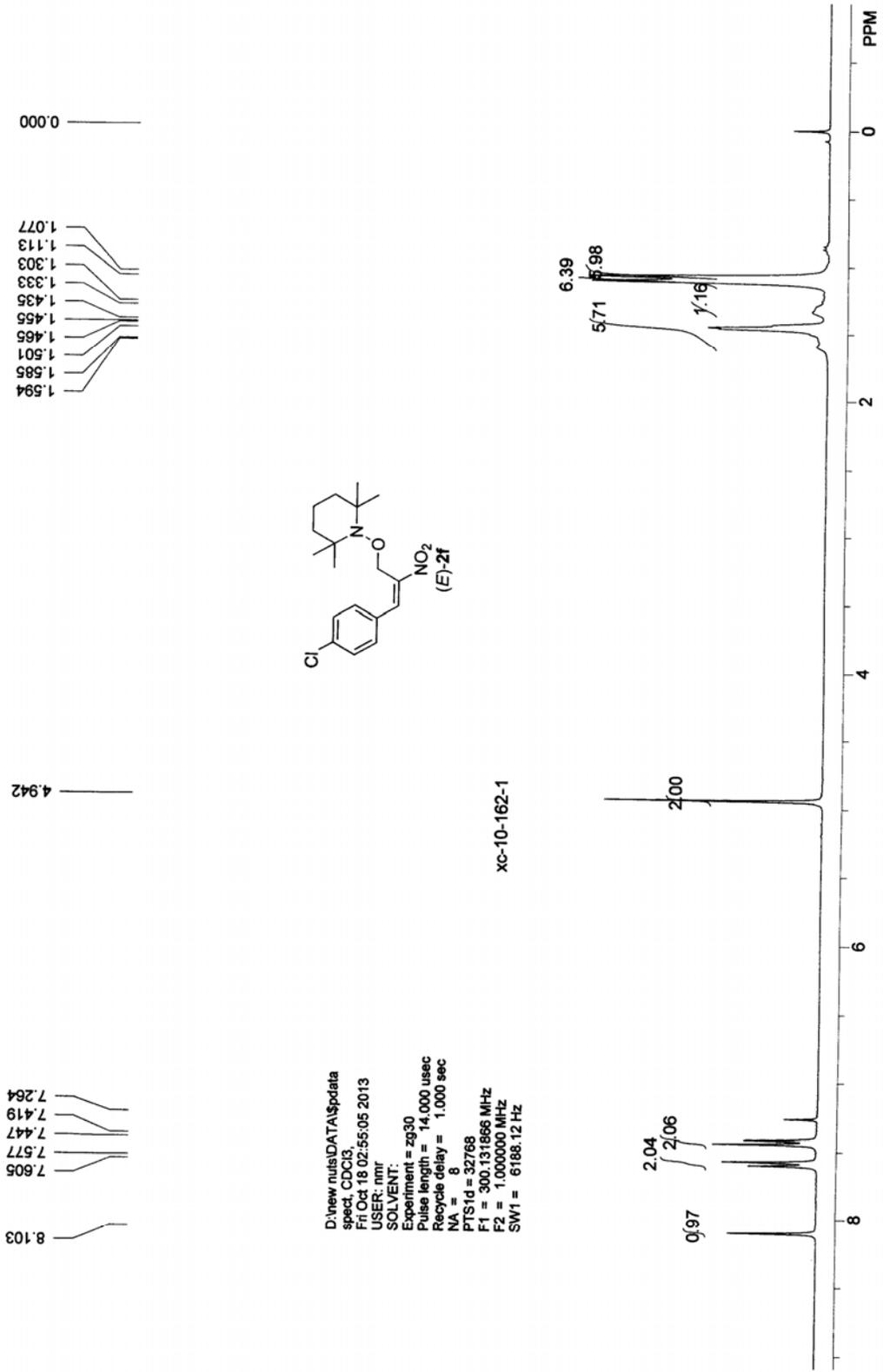


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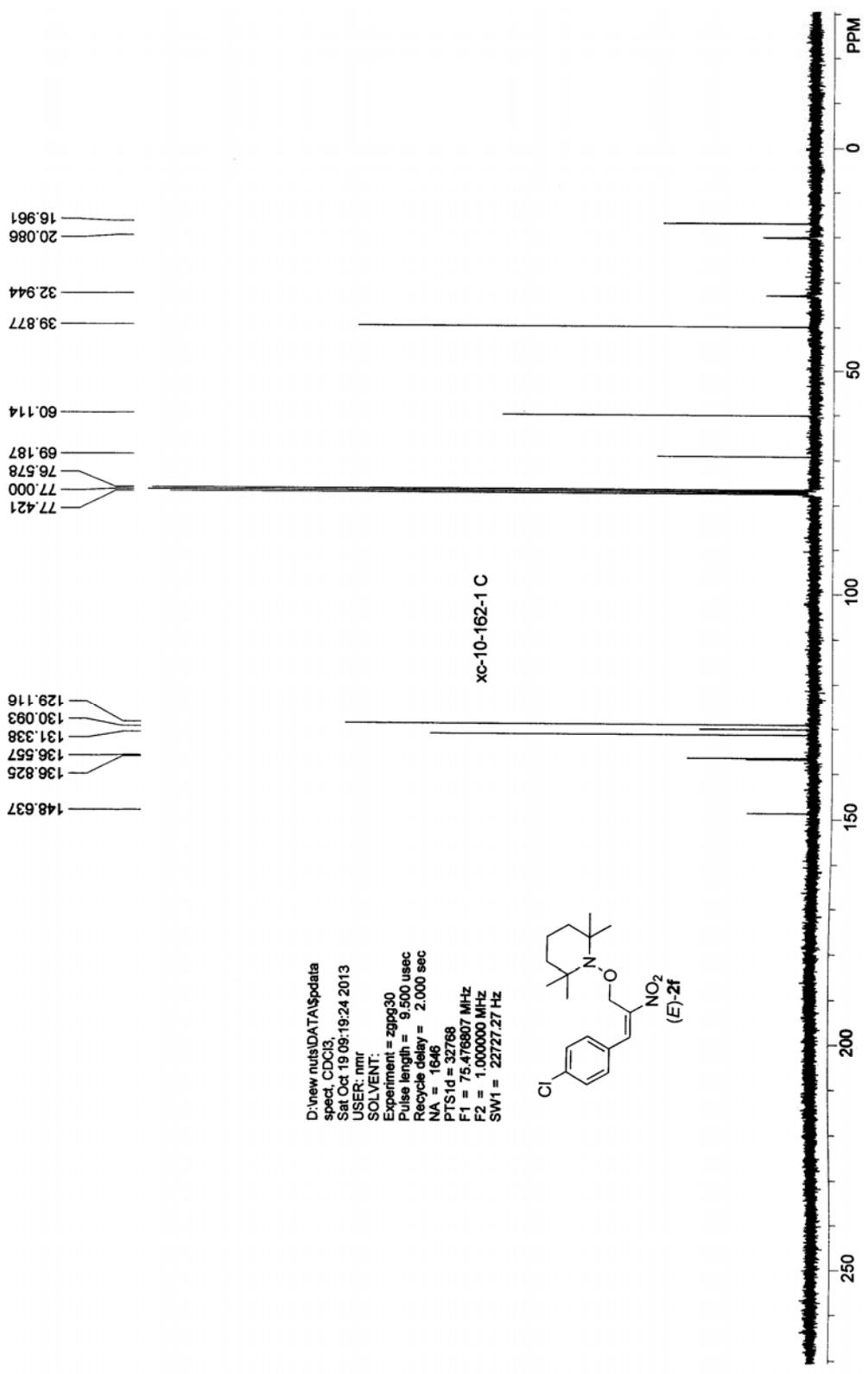
xc-10-162-cp

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 F2 = 1,000000 MHz
 SW1 = 6188.12 Hz

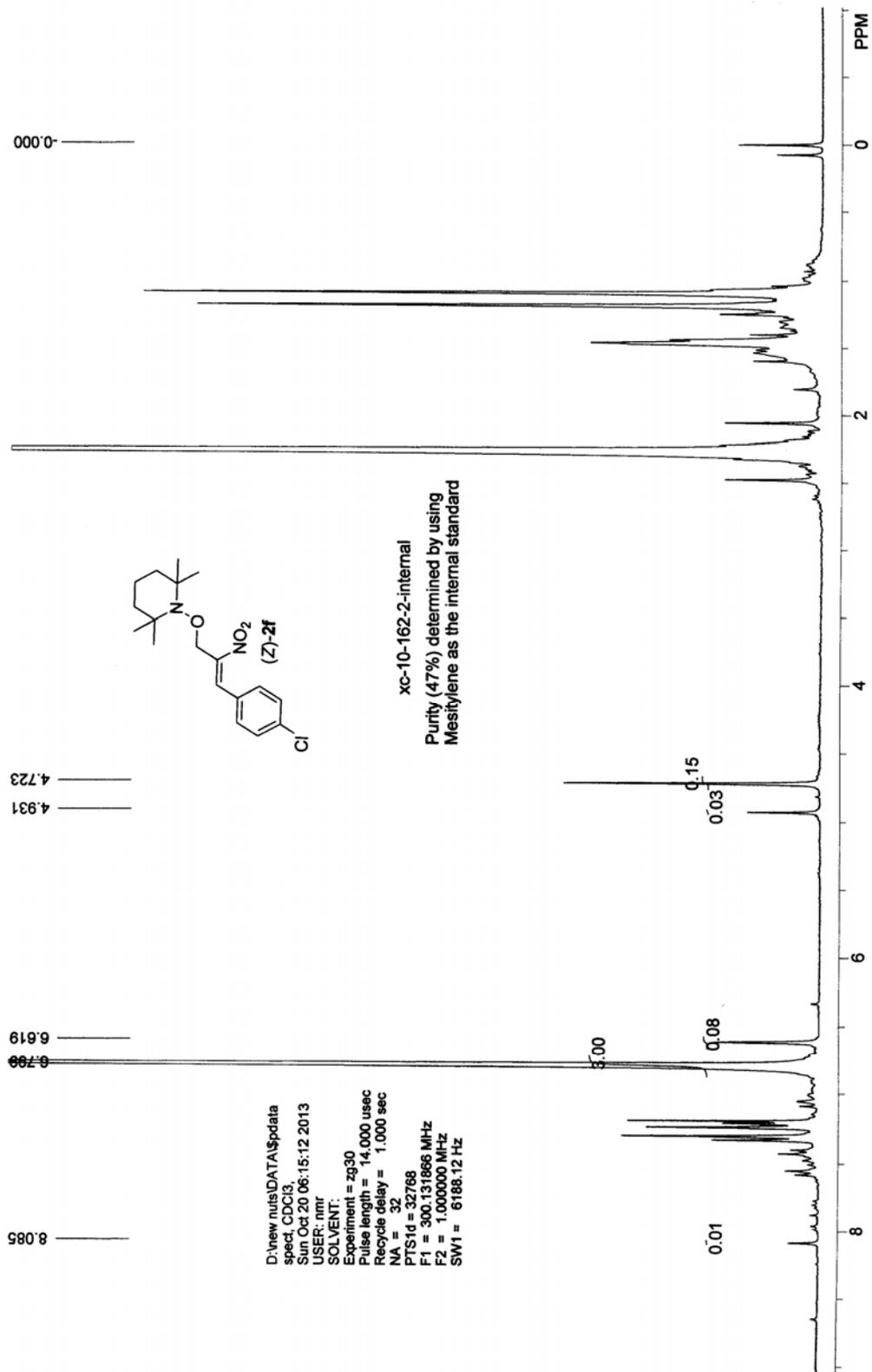


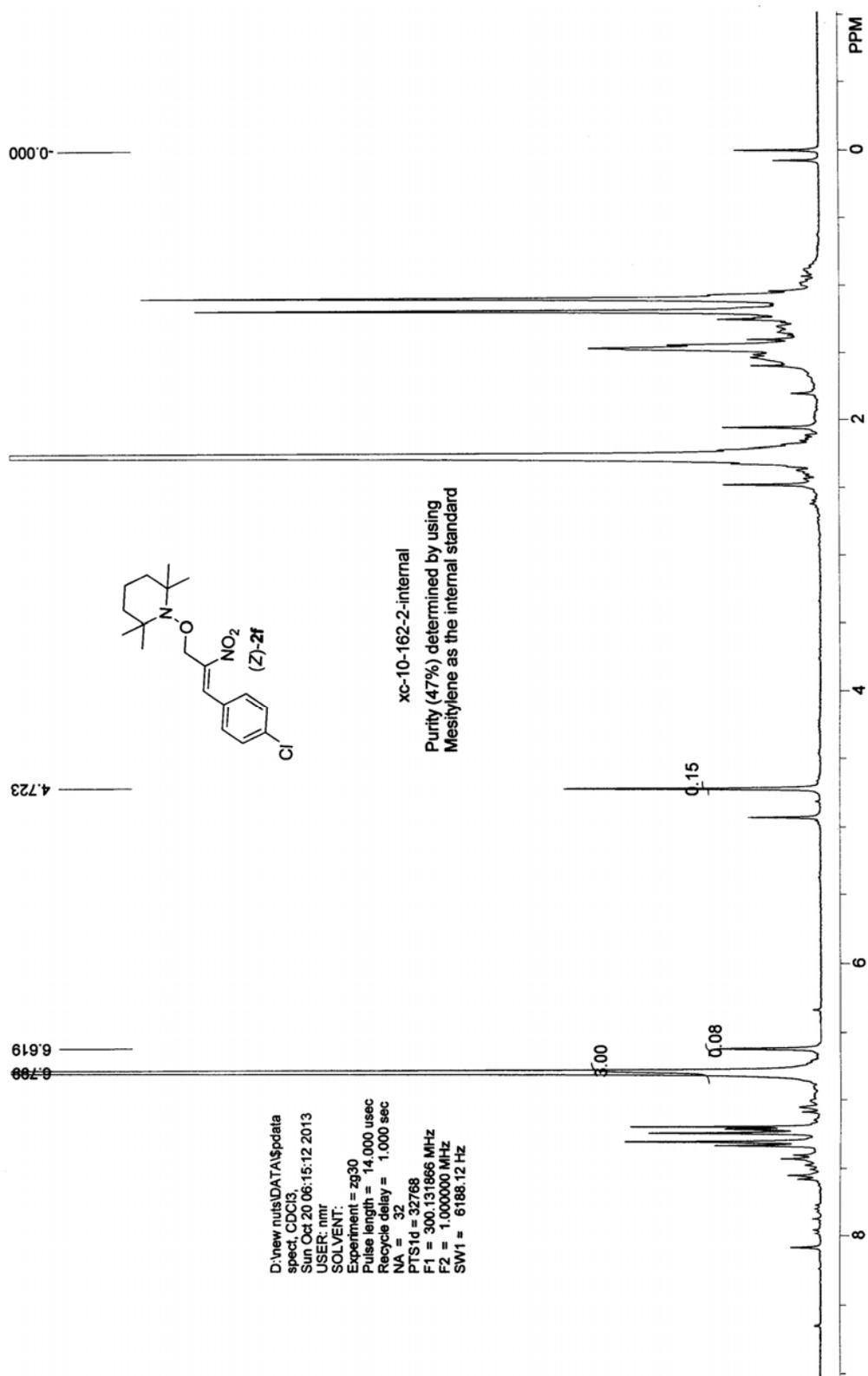
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 spect_CDCl3
 Fri Oct 18 02:55:05 2013
 USER: nmr
 SOLVENT:
 Experiment = zg30
 Pulse length = 14.000 usec
 Recycle delay = 1.000 sec
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 SWH1 = 6168.12 Hz

xc-10-162-1

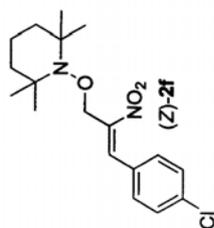
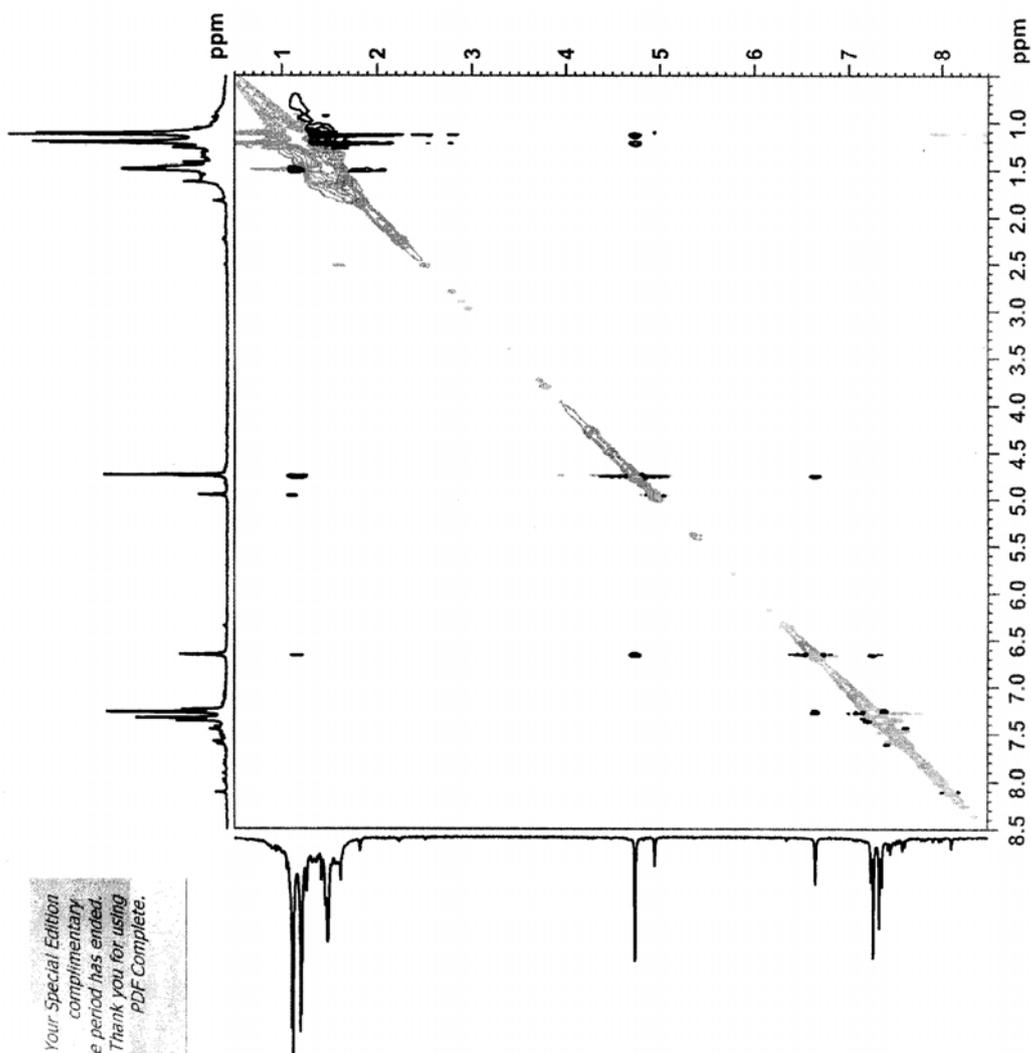


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 USER: mnr
 SOLVENT:
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 SW1 = 22727.27 Hz

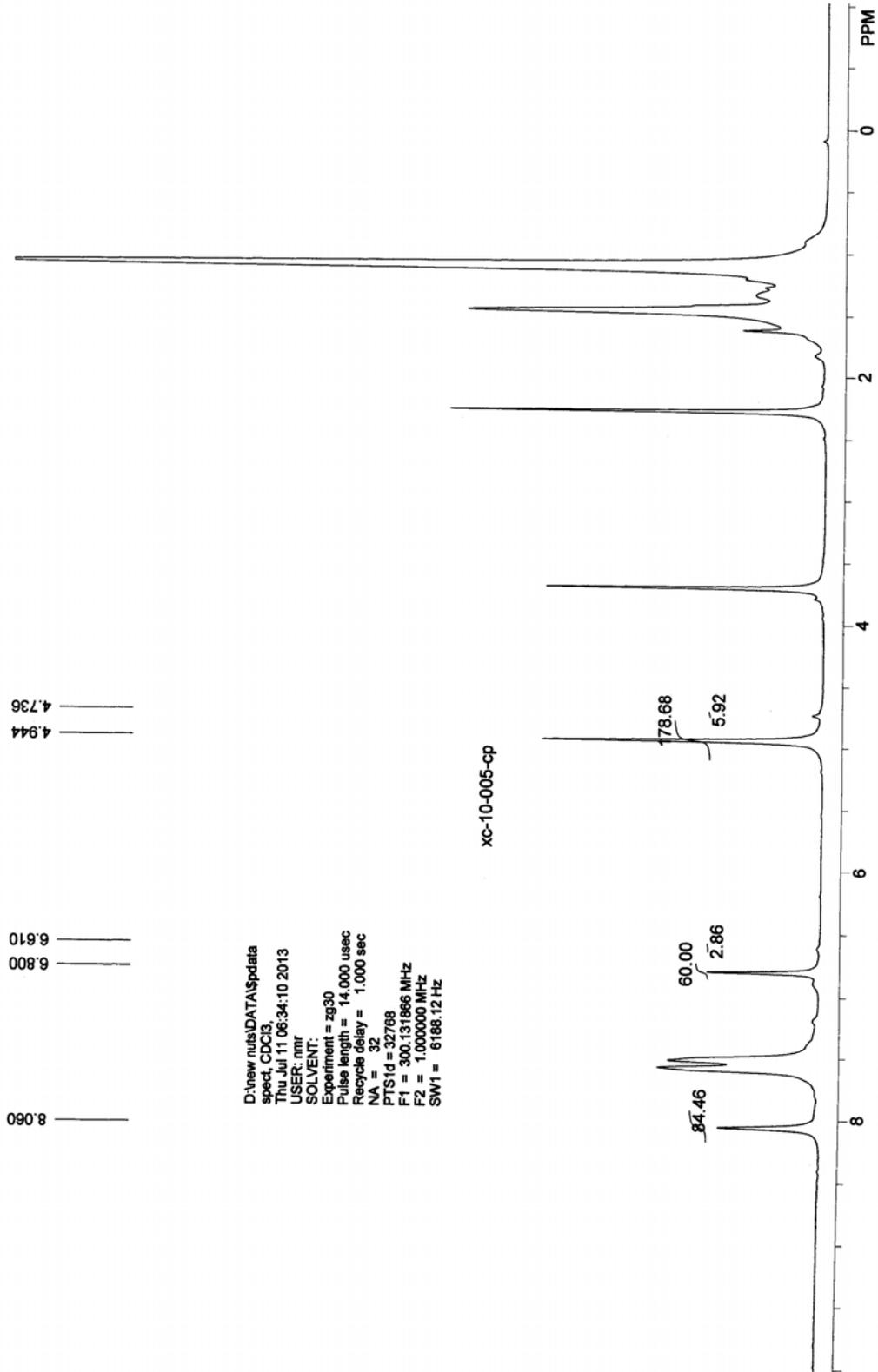




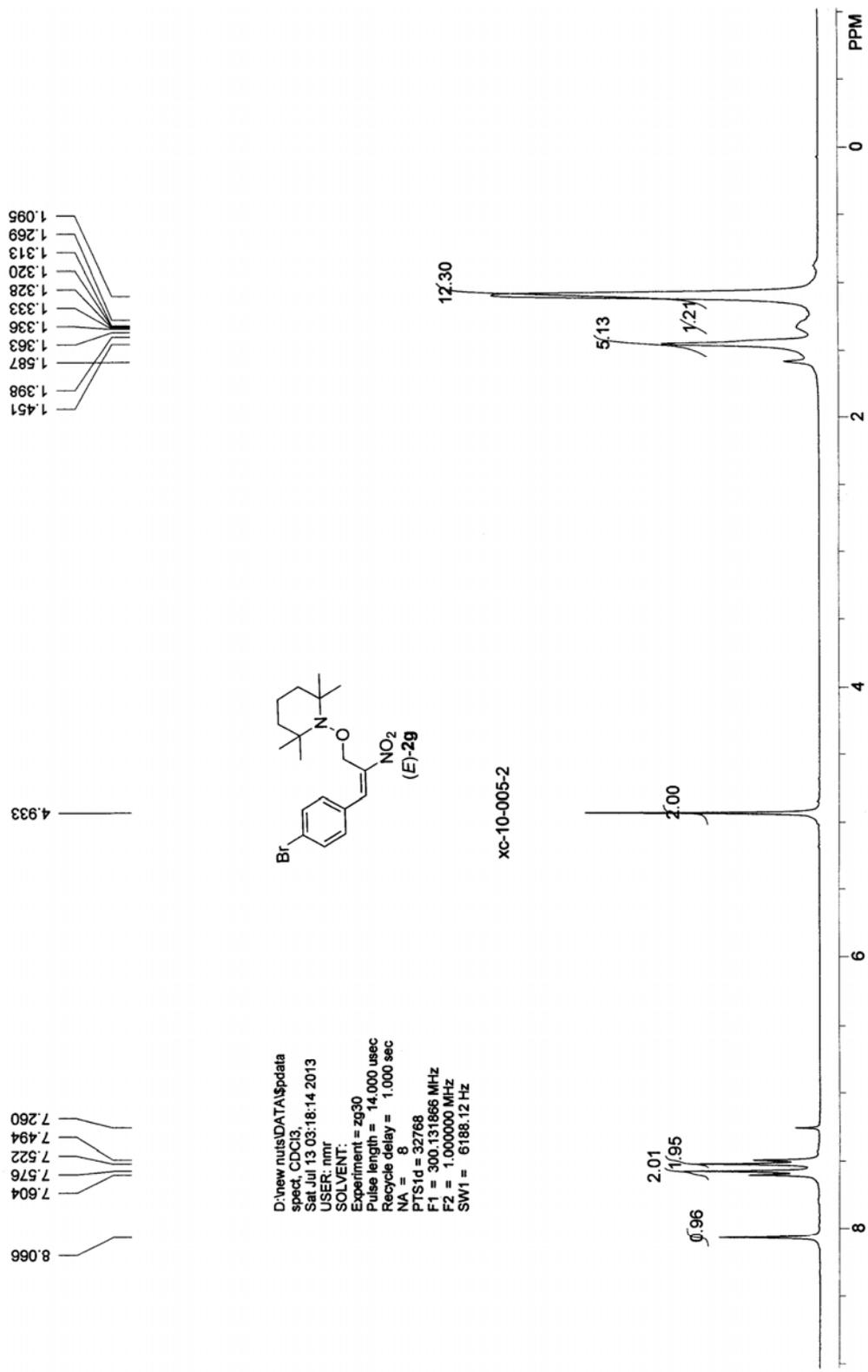
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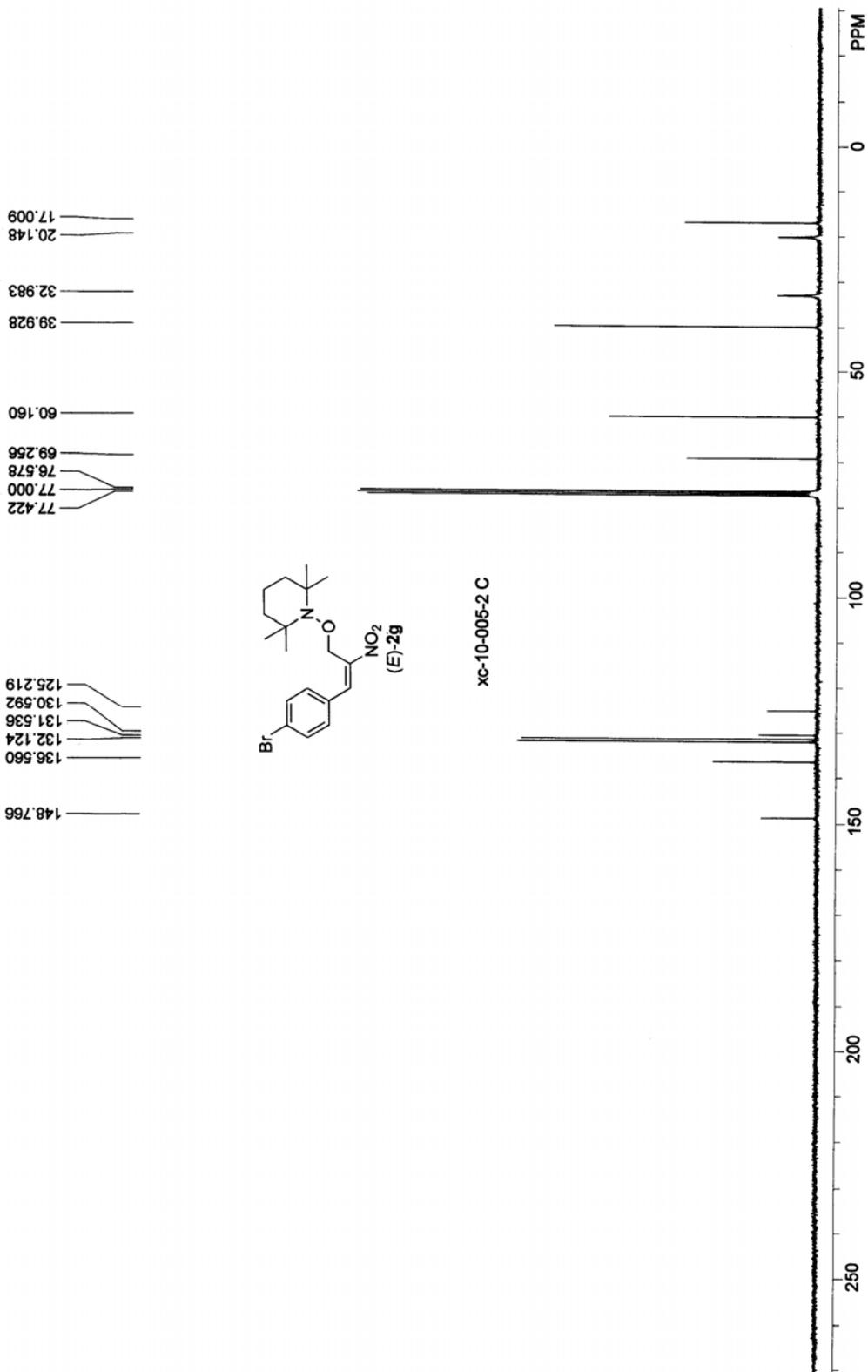
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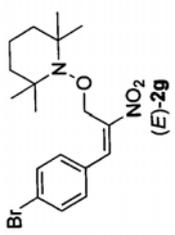
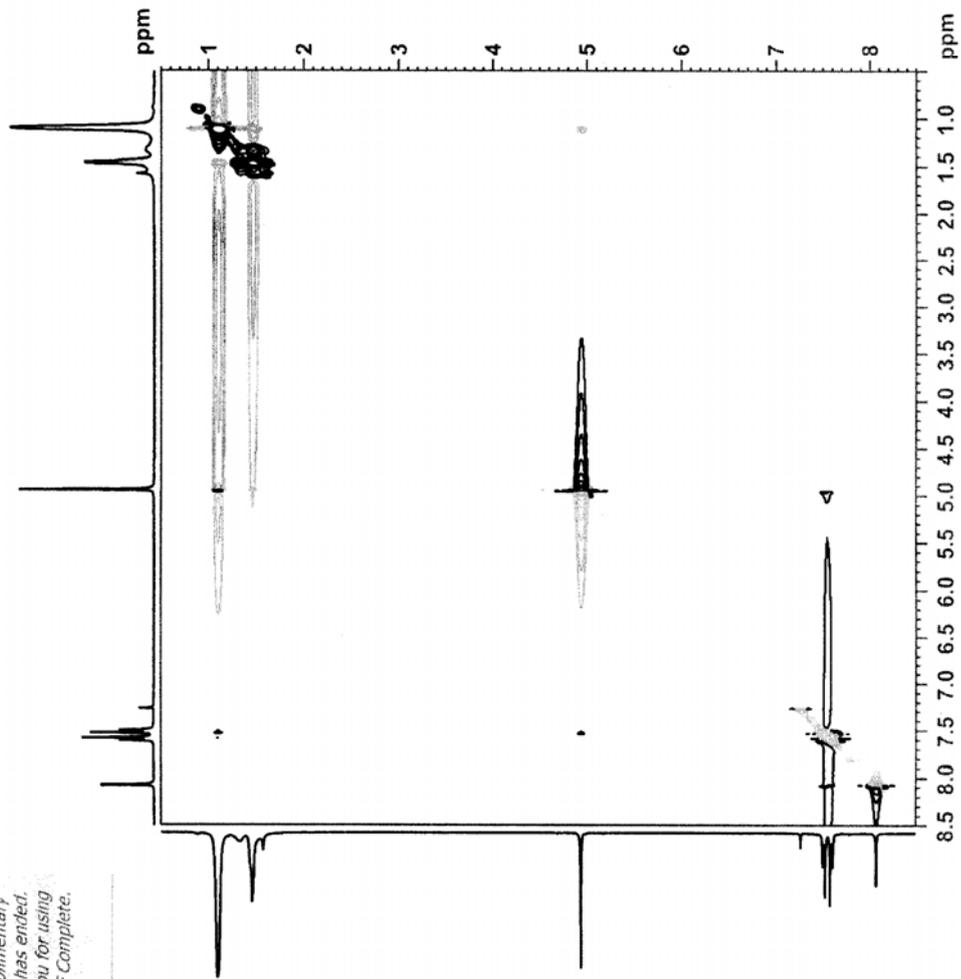
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SOLVENT:
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Recycle delay = 1,000 sec
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F1 = 300.131866 MHz
F2 = 1,000000 MHz
SW1 = 6188.12 Hz



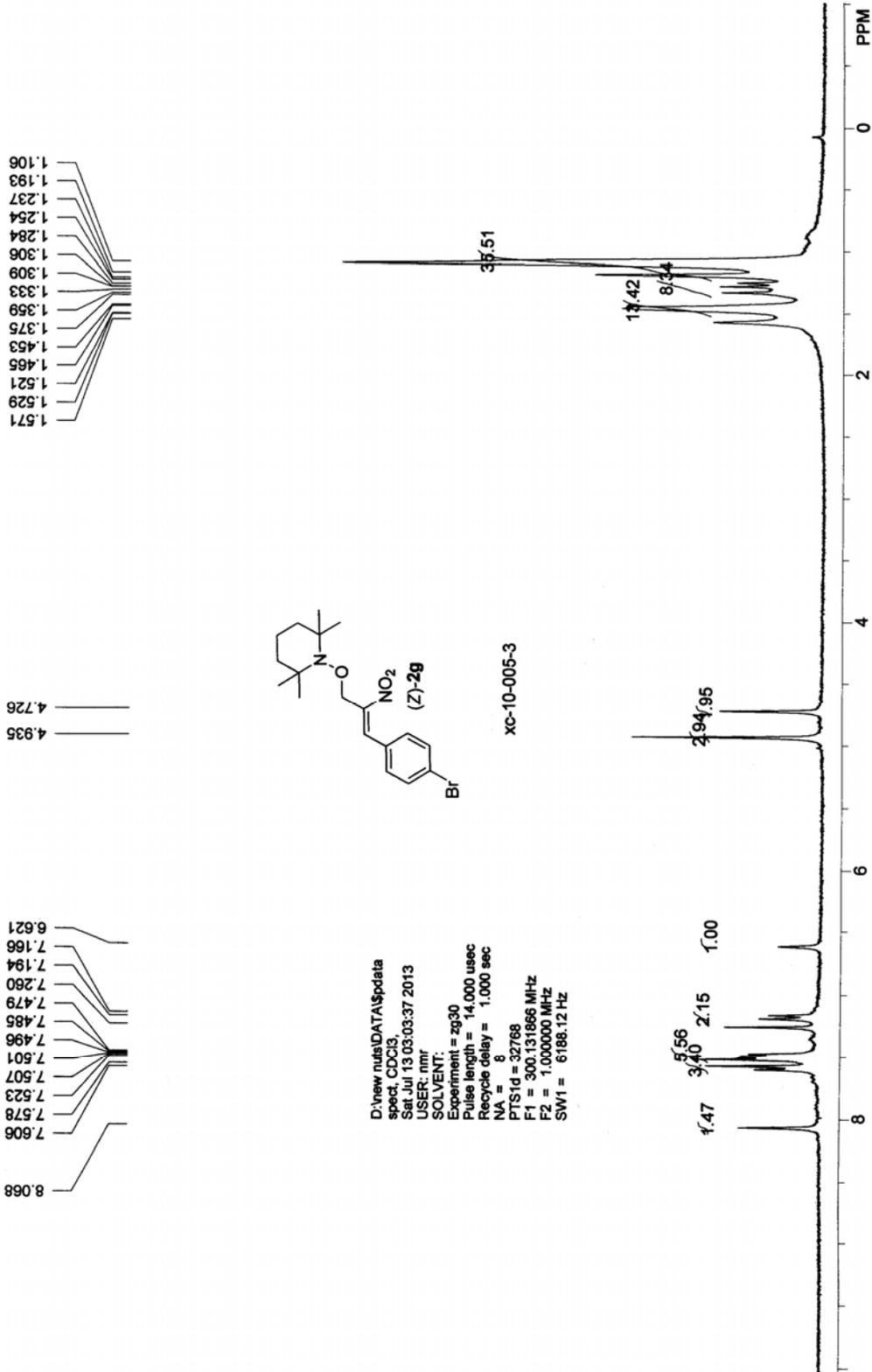
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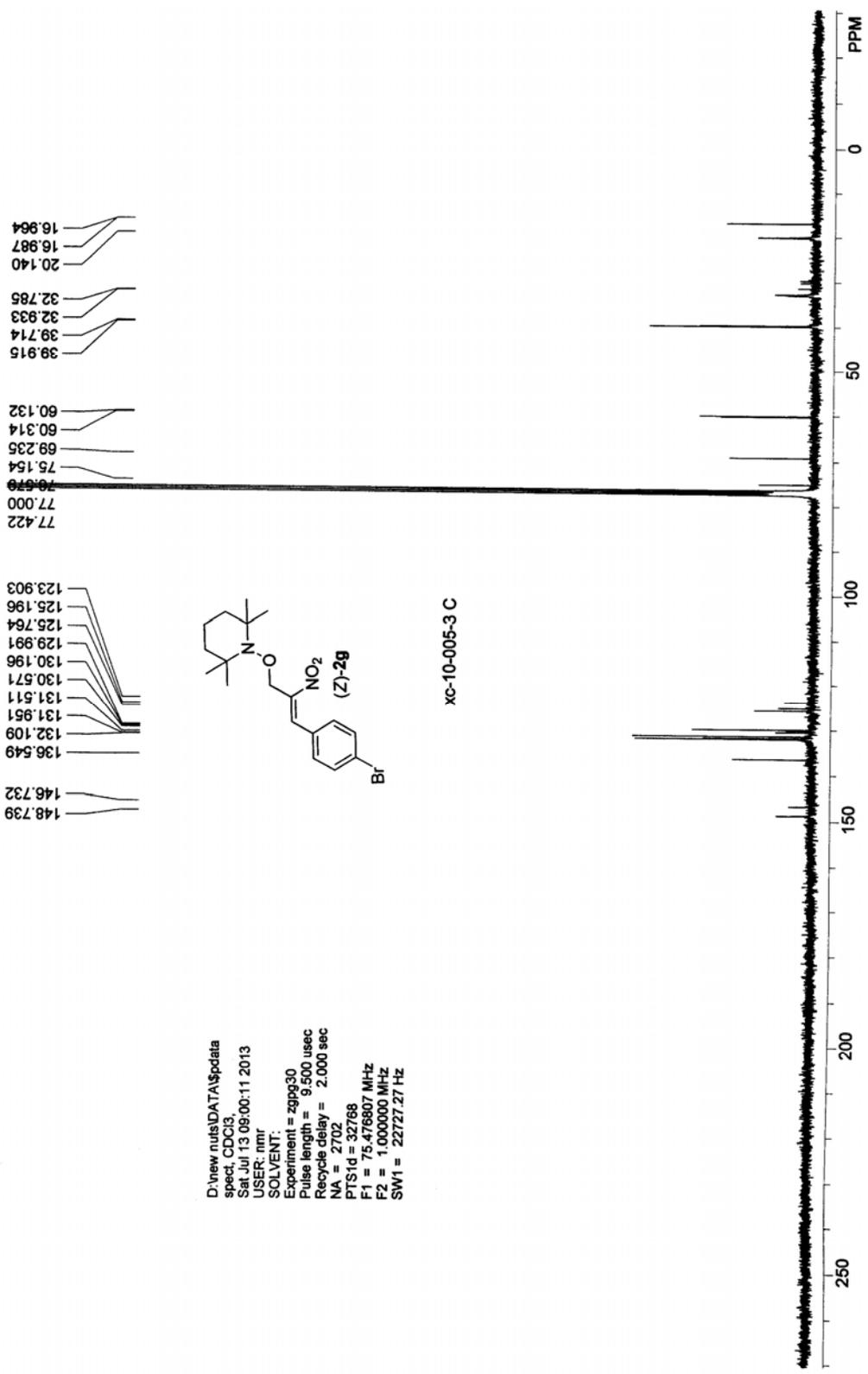


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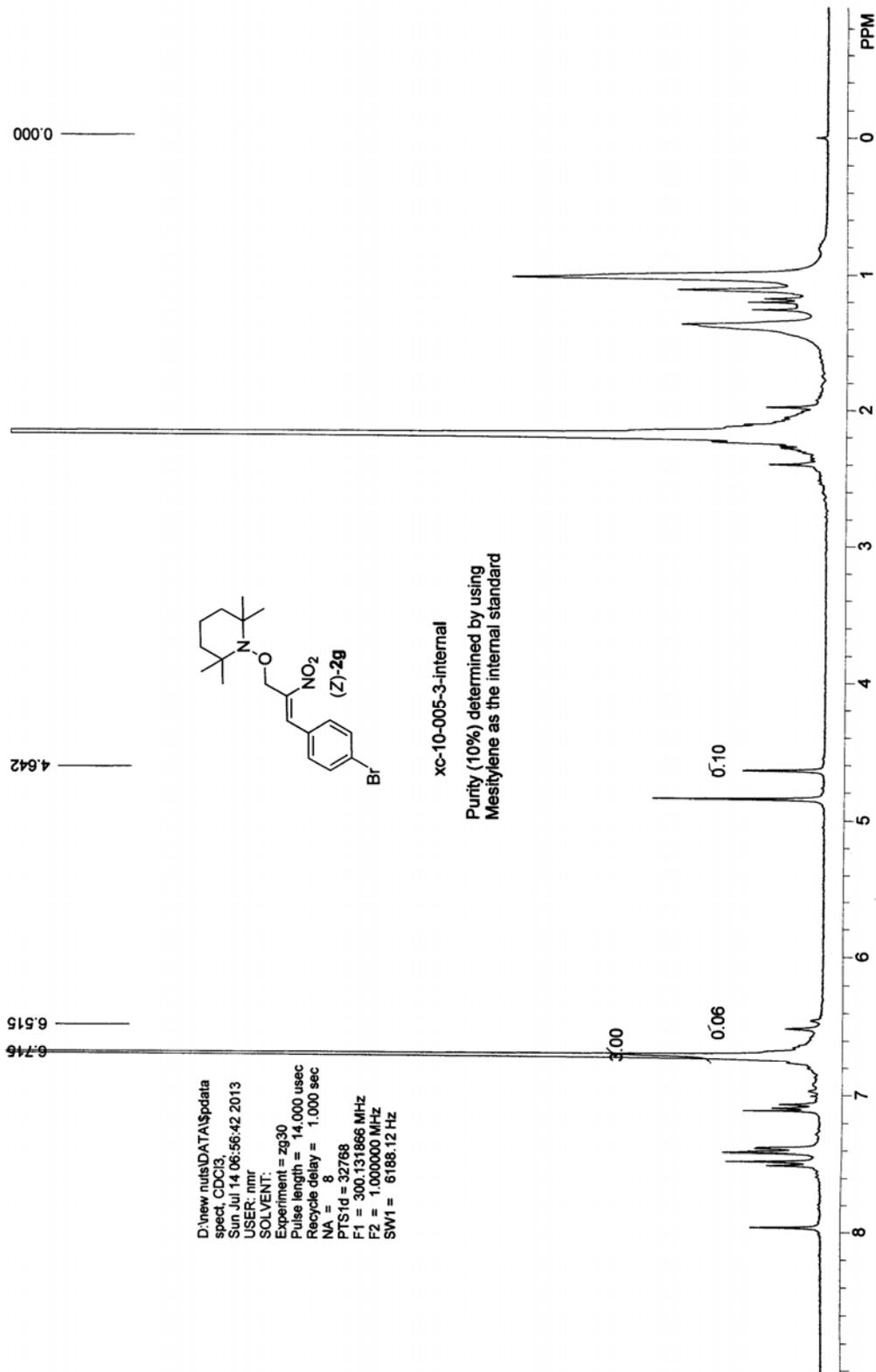


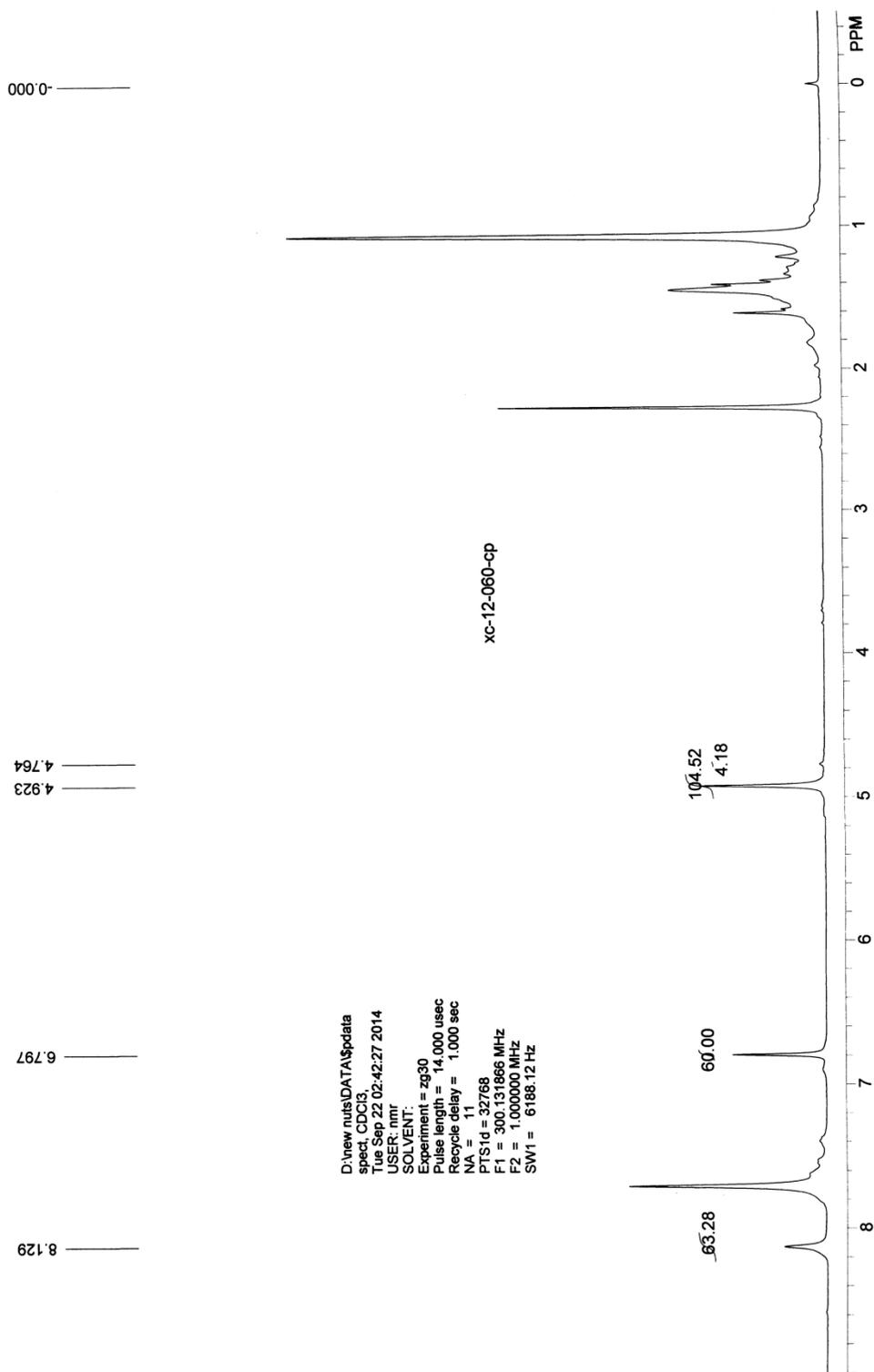
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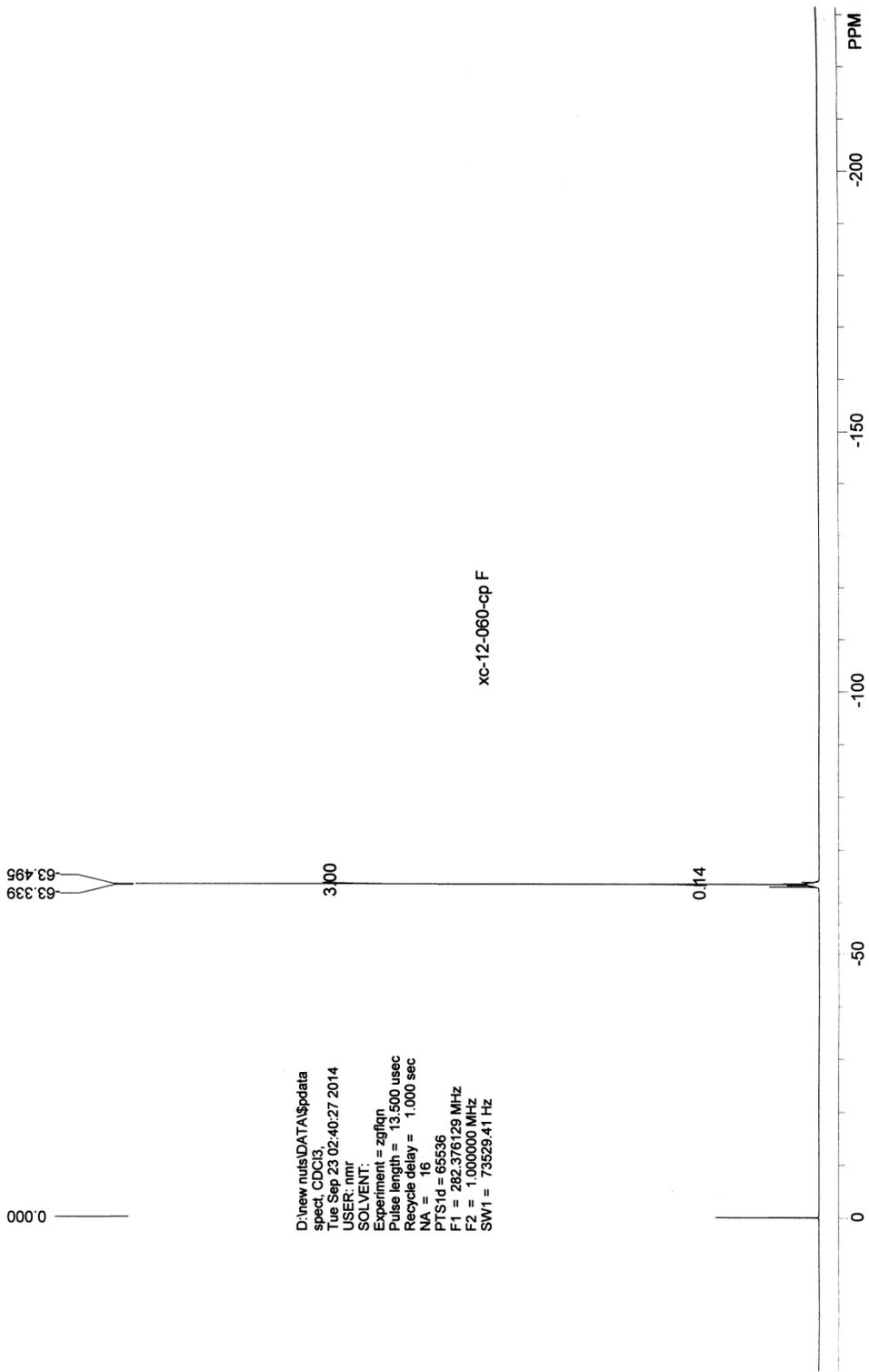


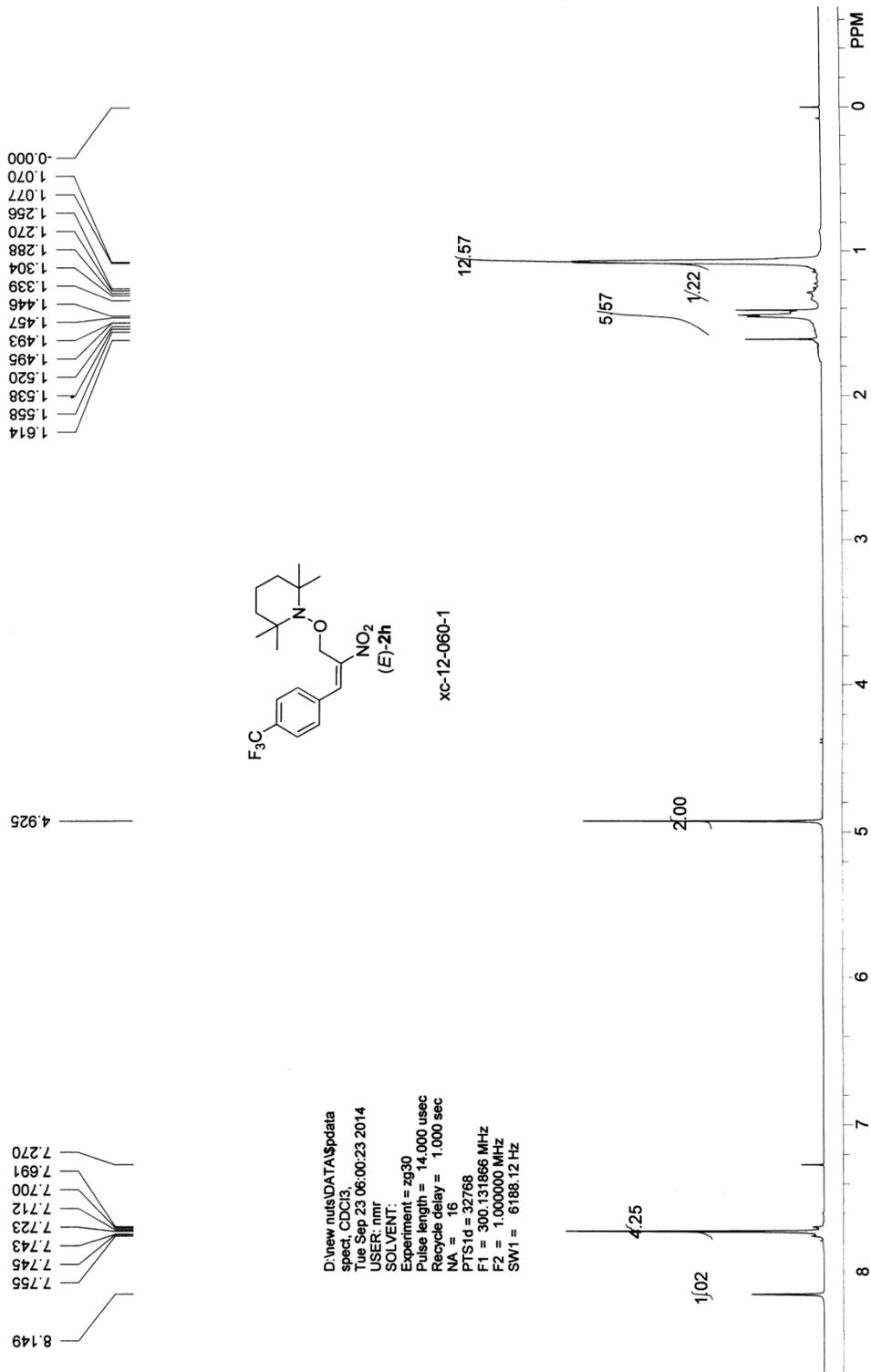
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Sat Jul 13 09:00:11 2013
USER: nmr
SOLVENT:
Experiment = zpgg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 2702
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F2 = 1.000000 MHz
SW1 = 22727.27 Hz

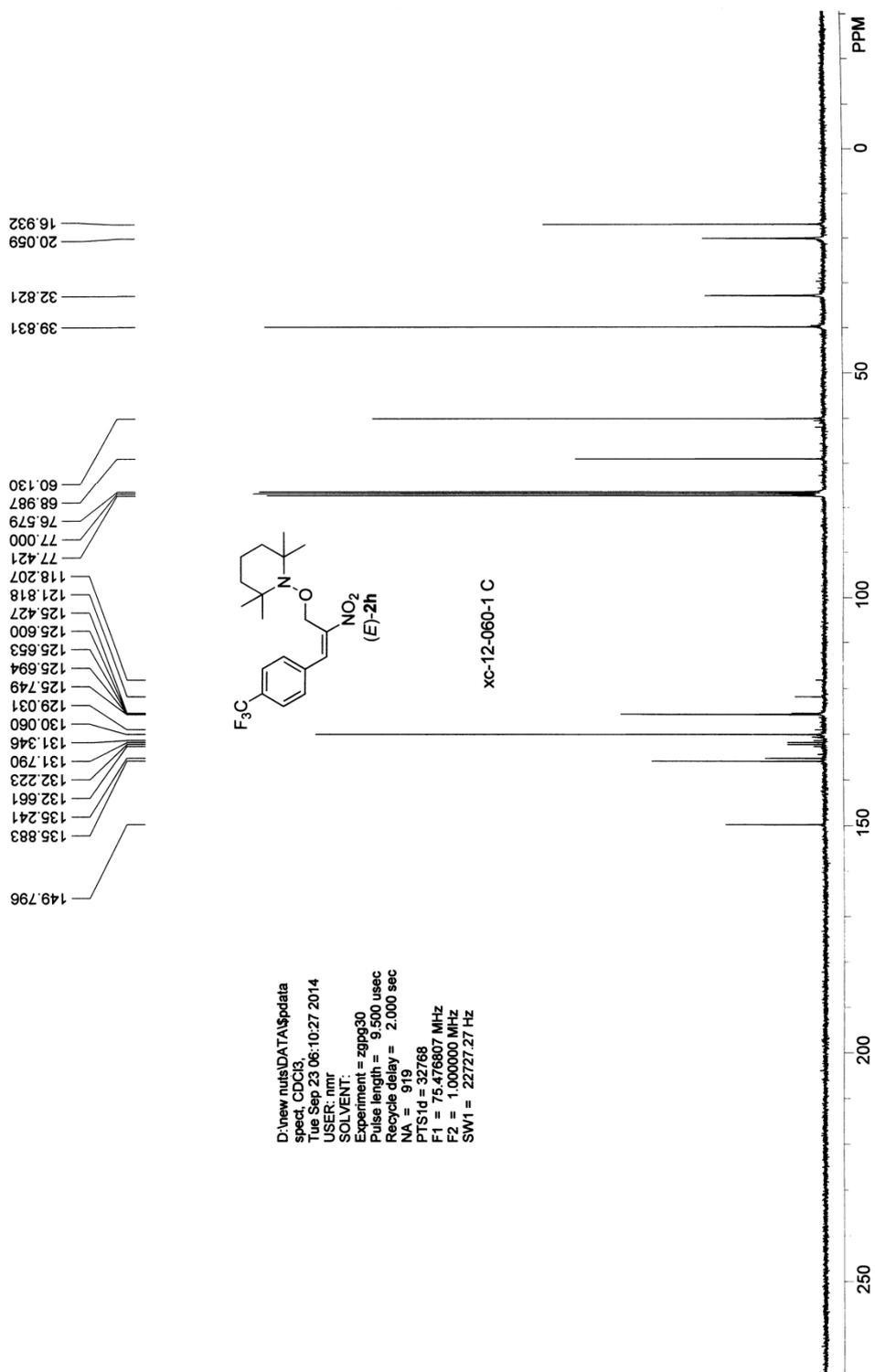




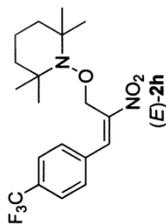
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 Tue Sep 22 02:42:27 2014
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 SOLVENT:
 Experiment = z930
 Pulse length = 14.000 usec
 Recycle delay = 1.000 sec
 NA = 11
 P1S1d = 32768
 F1 = 300.137866 MHz
 F2 = 1.000000 MHz
 SW1 = 6188.12 Hz







D:\new nui\DATA\Spdata
spect, CDCl3,
Tue Sep 23 06:10:27 2014
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 919
PTS1d = 32768
F1 = 75.476807 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz



xc-12-060-1 F

D:\new nuts\DATA\Spdata
 spect, CDCI3,
 Tue Sep 23 03:30:38 2014
 USER: nmr
 SOLVENT:
 Experiment = zoffin
 Pulse length = 13.500 usec
 Recycle delay = 1.000 sec
 NA = 16
 P1Std = 65536
 F1 = 282.376129 MHz
 F2 = 1.000000 MHz
 SW1 = 73529.41 Hz

0000

63.450

3.00

PPM

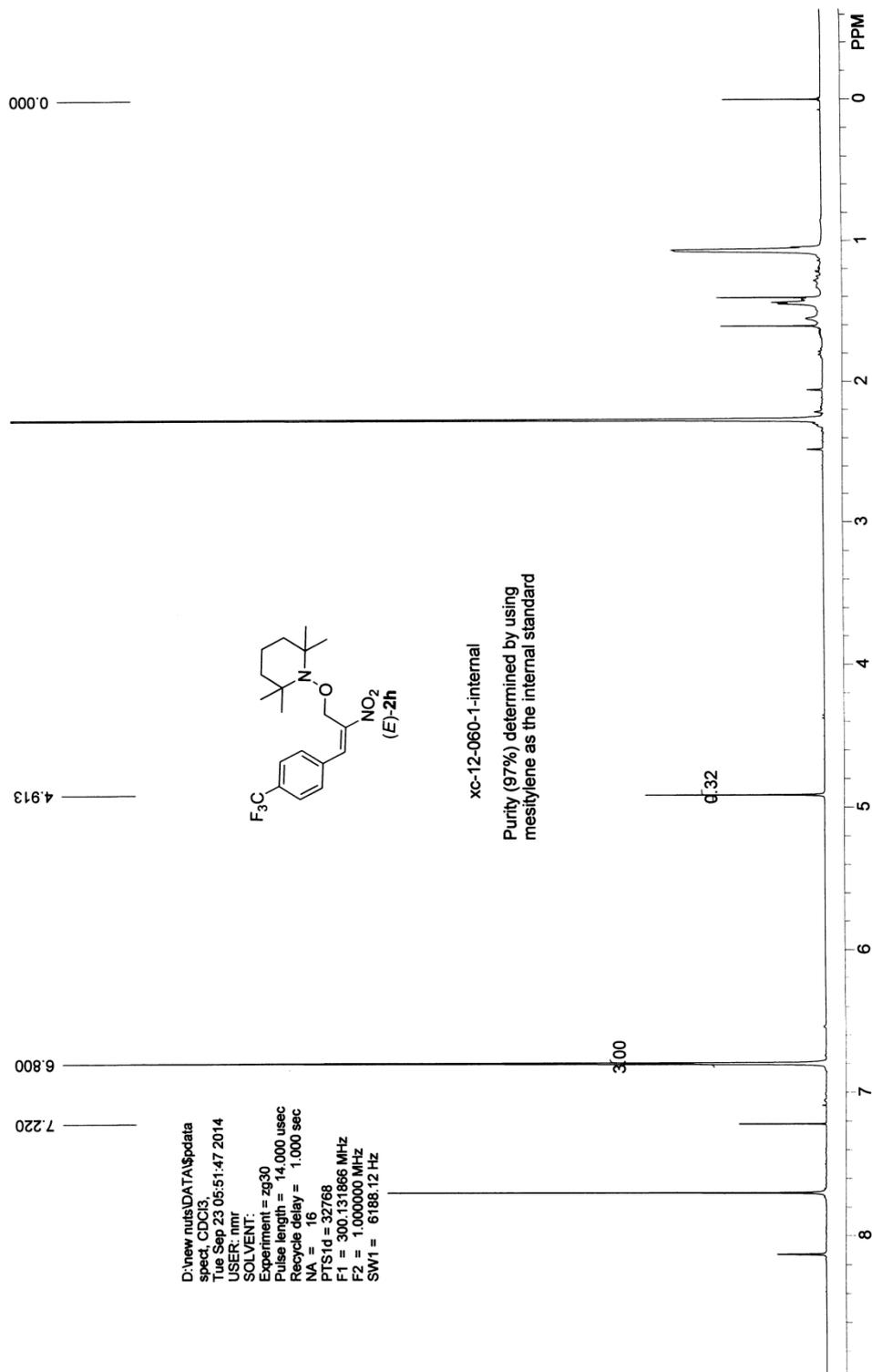
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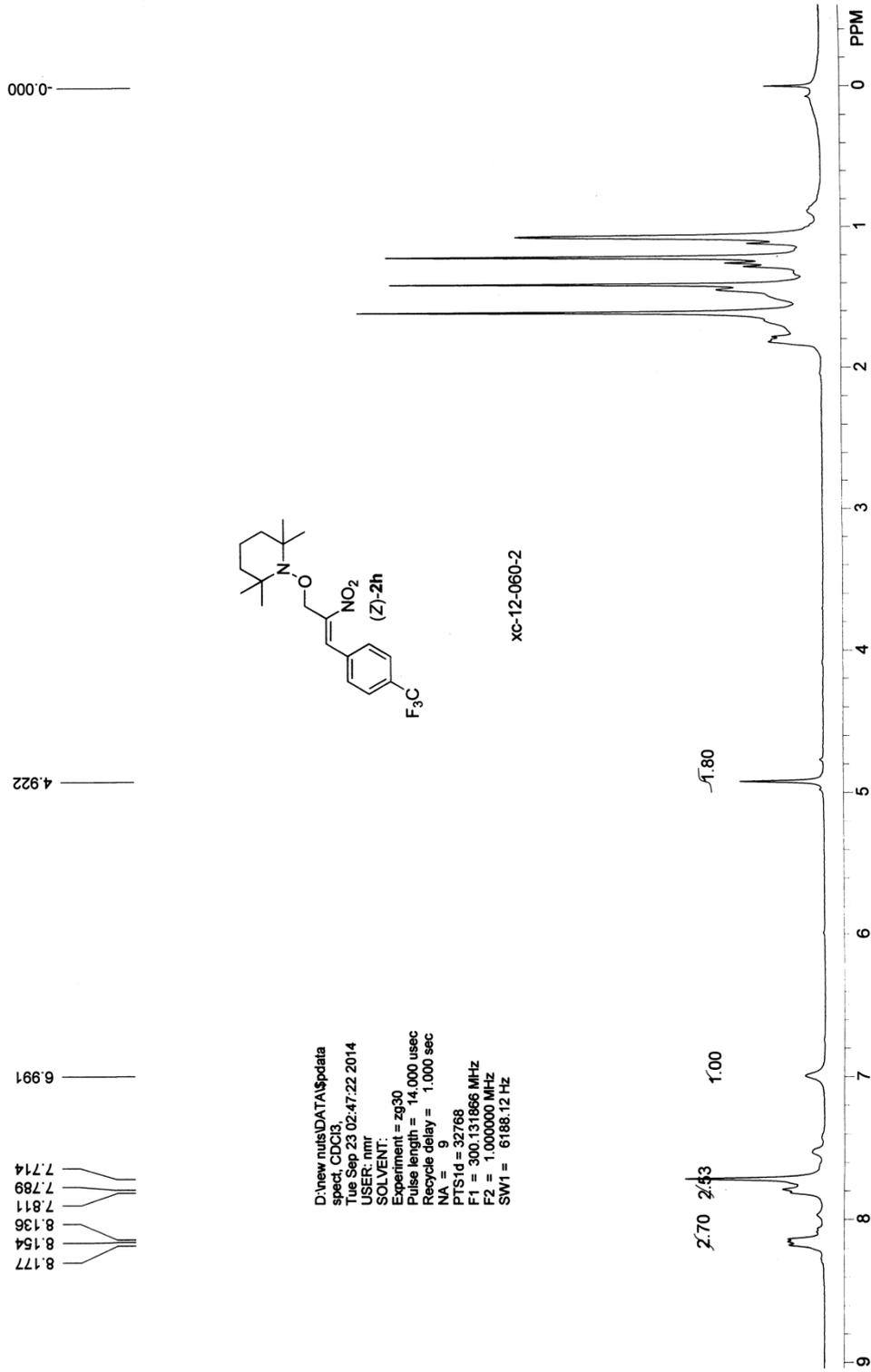
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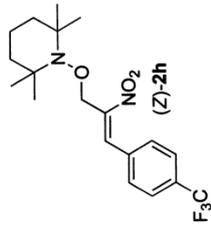




63.900
63.363

0.000

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spect, CDCI3,
Tue Sep 23 12:25:16 2014
USER: nmr
SOLVENT:
Experiment = zoflon
Pulse length = 13.500 usec
Recycle delay = 1.000 sec
NA = 16
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F1 = 282.376129 MHz
F2 = 1.000000 MHz
SW1 = 73529.41 Hz



xc-12-060-2 F

3.100
2.139

PPM

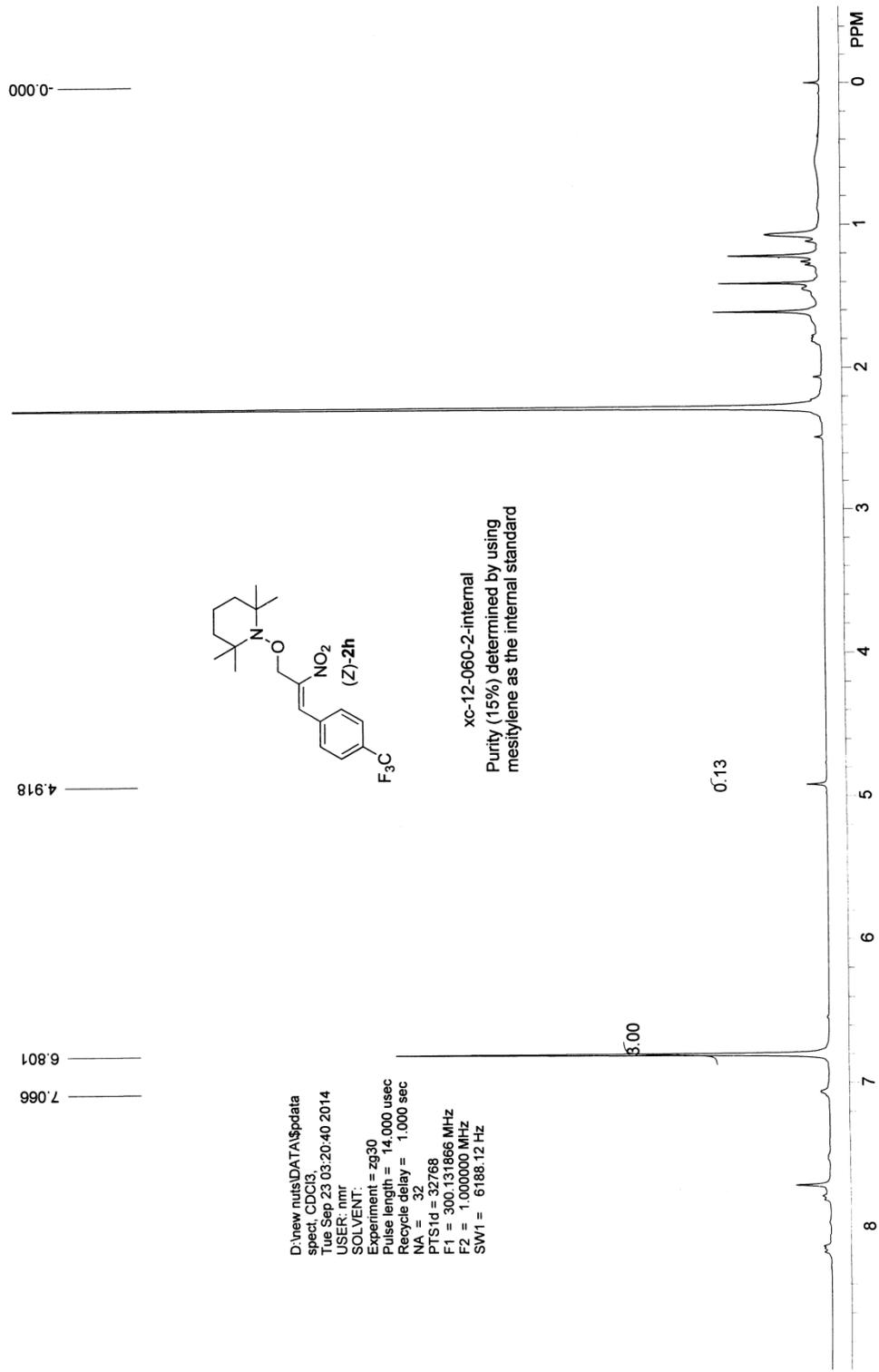
-200

-150

-100

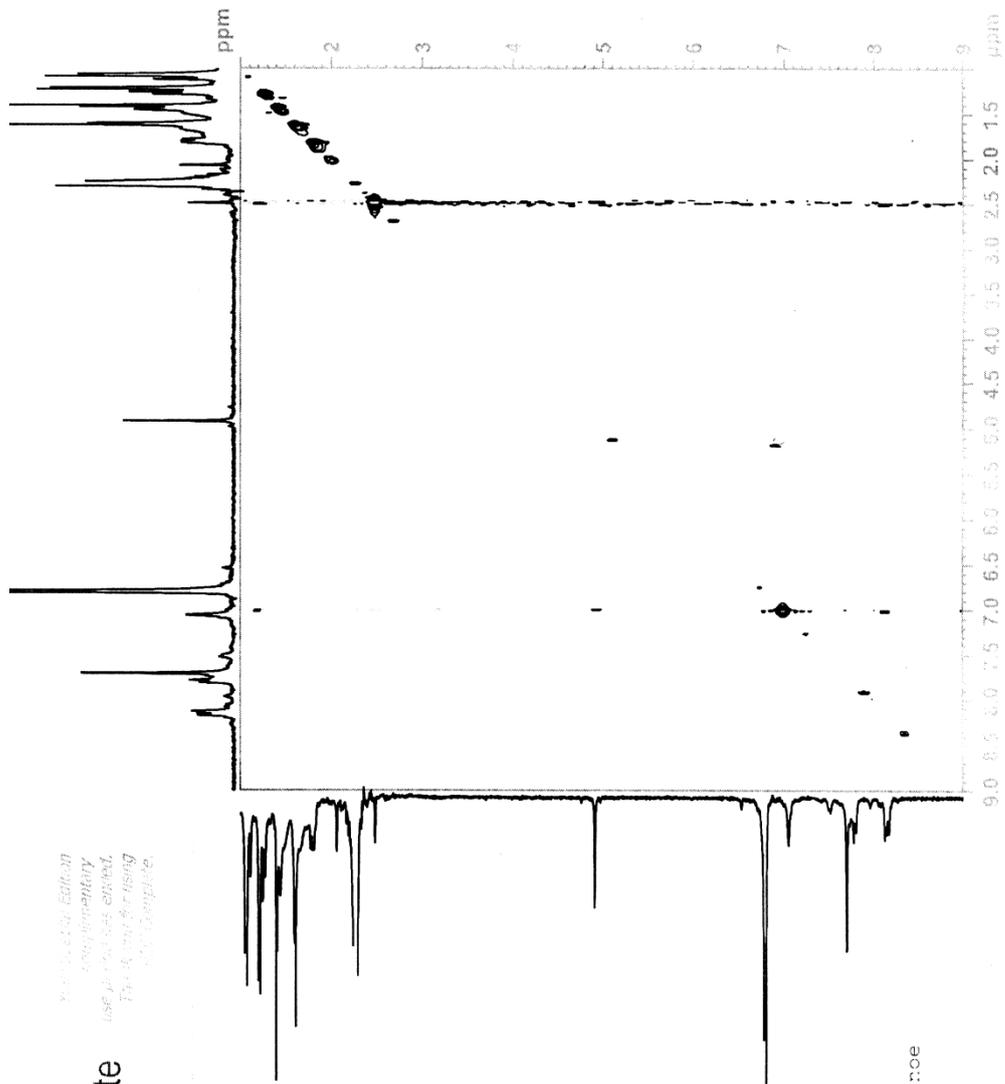
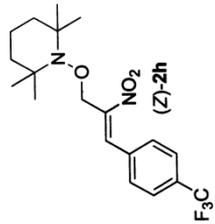
-50

0

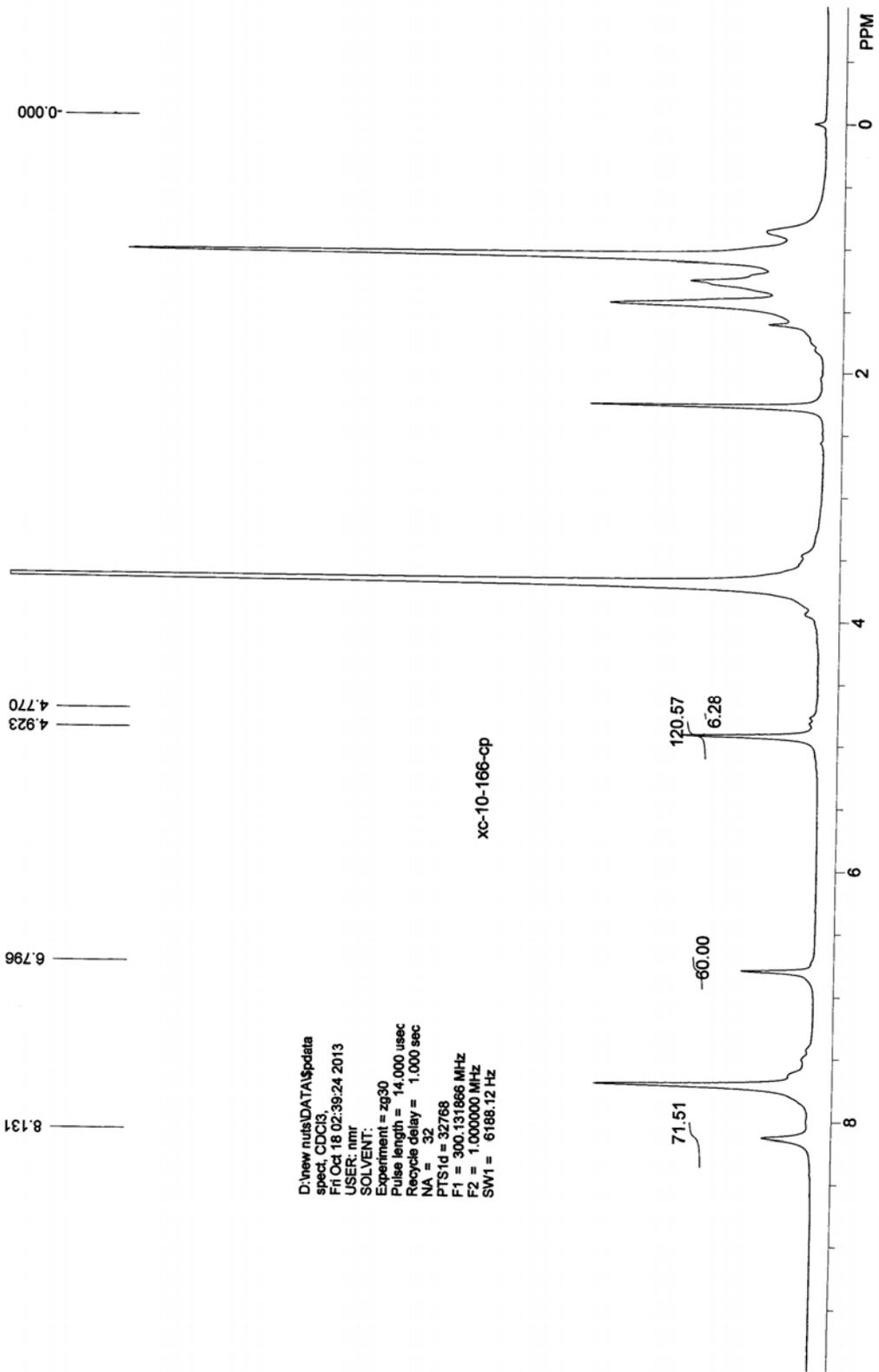


13C NMR
Complete
100% complementary
use for 13C NMR
To 4.0 ppm for using
13C NMR

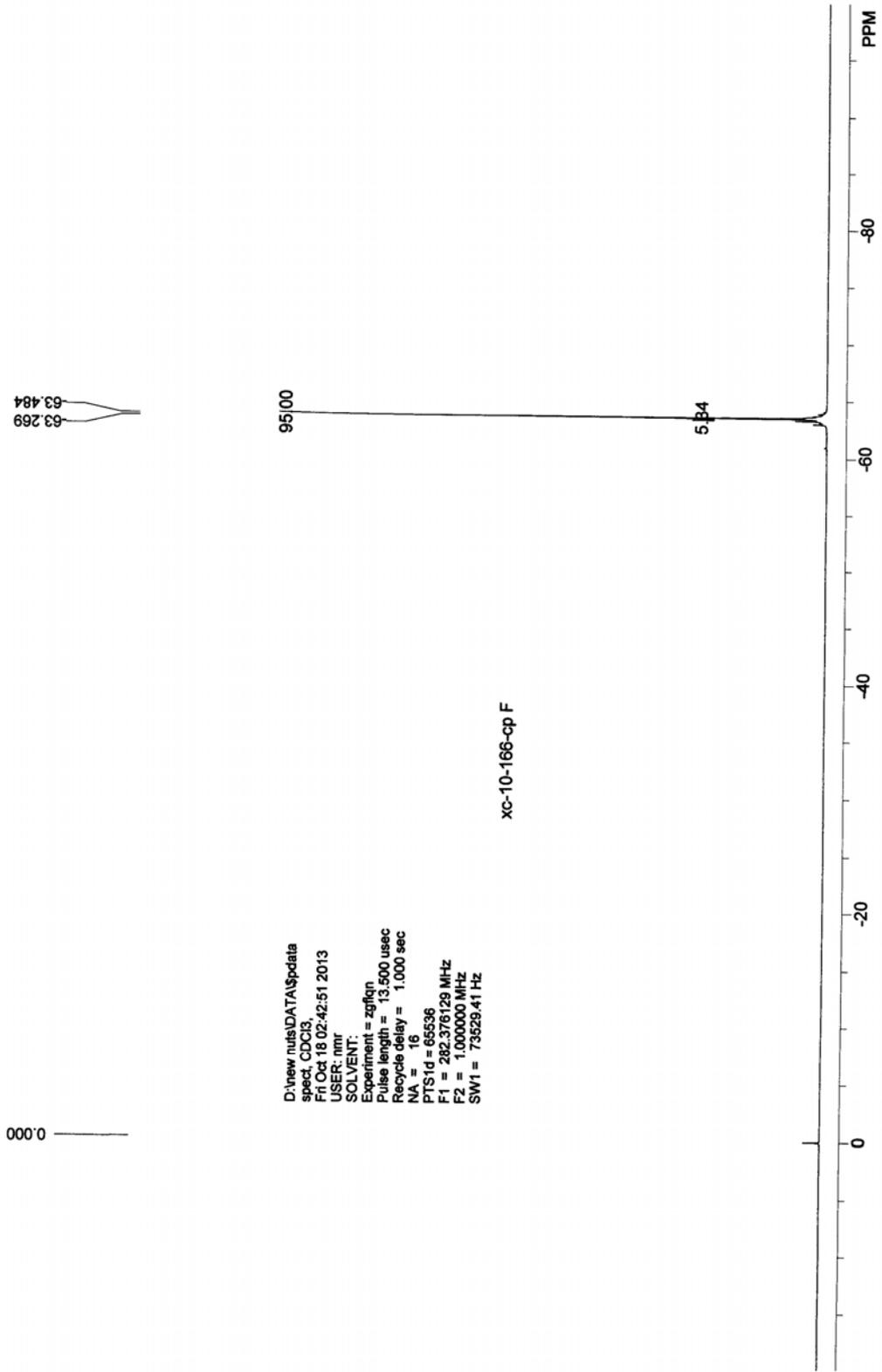
Complete

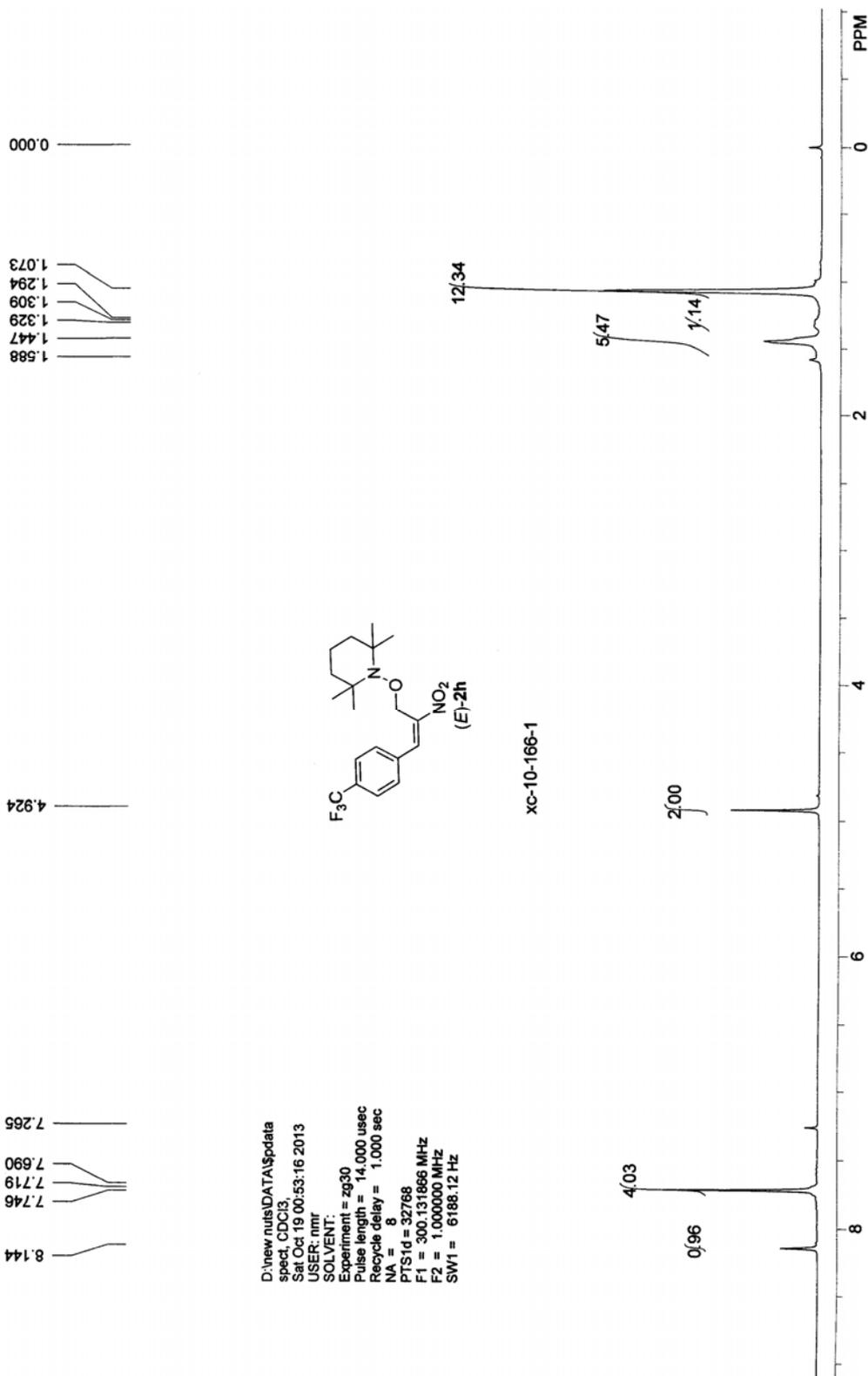


XC-12-060-2-10e

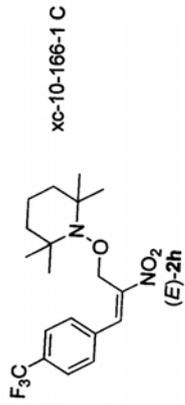
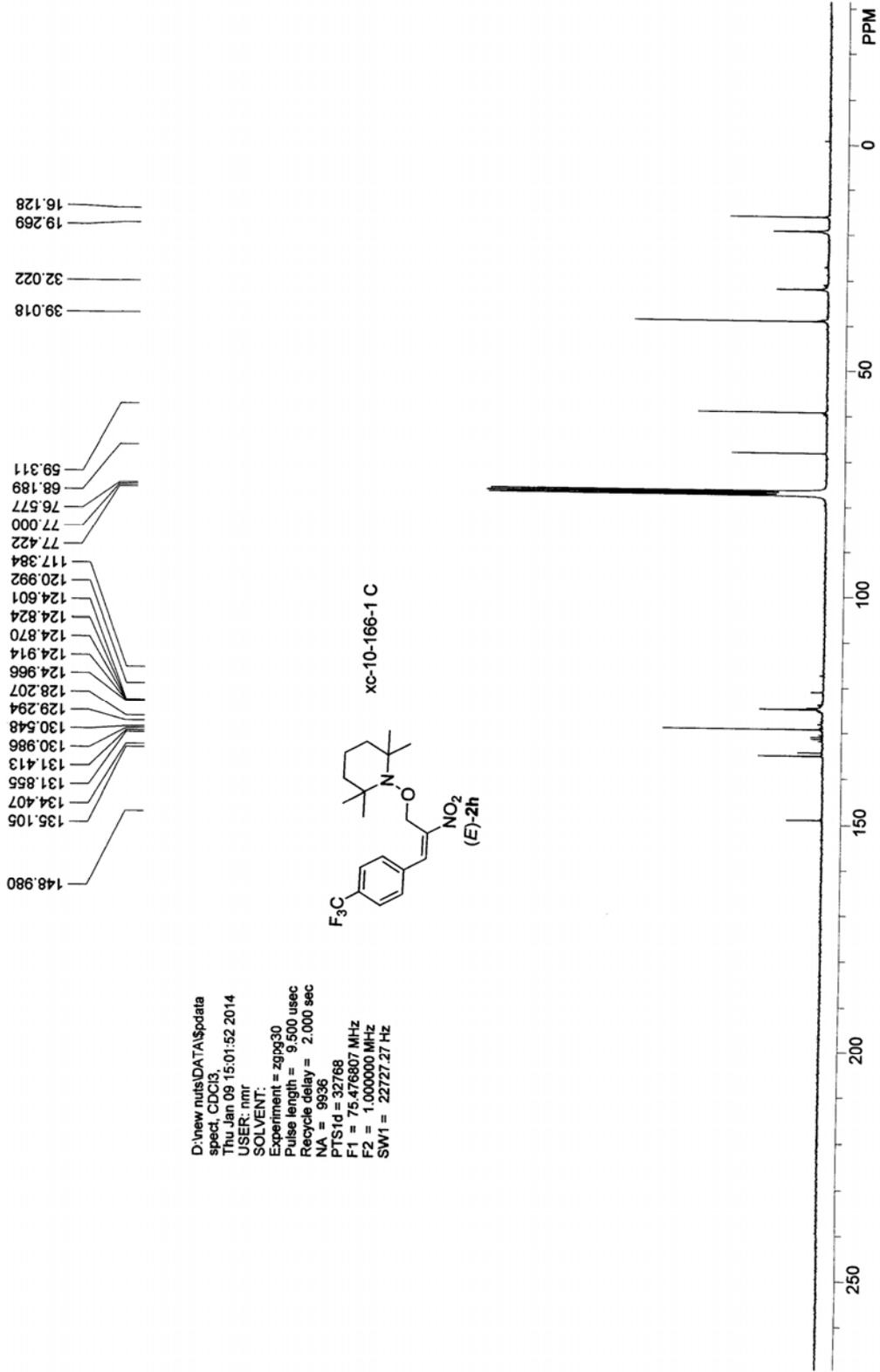


D:\new nutisDATA\spdata
 spect_CDCl3
 Fri Oct 18 02:39:24 2013
 USER: nmir
 SOLVENT:
 Experiment = zg30
 Pulse length = 14.000 usec
 Recycle delay = 1.000 sec
 NA = 32
 P1 = 300.131866 MHz
 F2 = 1.000000 MHz
 SW1 = 6188.12 Hz





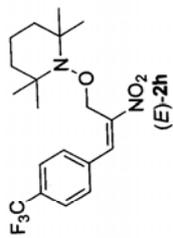
D:\new nuts\DATA\spdata
 spect, CDC13,
 Sat Oct 19 00:53:16 2013
 USER: nmr
 SOLVENT:
 Experiment = zg30
 Pulse length = 14,000 usec
 Recycle delay = 1,000 sec
 NA = 8
 P1 = 32768
 F1 = 300.131866 MHz
 F2 = 1,000,000 MHz
 SWH = 6188.12 Hz



D:\new nuts\DATA\pdata
 spect, CDCl₃,
 Thu Jan 09 15:01:52 2014
 USER: nmr
 SOLVENT:
 Experiment = zqpg30
 Pulse length = 9.500 usec
 Recycle delay = 2.000 sec
 NA = 9936
 P1 = 32768
 F1 = 75.476807 MHz
 F2 = 1.000000 MHz
 SW1 = 22727.27 Hz

62.922

D:\new nutis\DATA\spdata
spect, CDCI3,
Fri Dec 13 14:15:35 2013
USER: nmr
SOLVENT: nmr
Experiment = zgfgn
Pulse length = 13.500 usec
Recycle delay = 1.000 sec
NA = 16
PTStd = 65536
F1 = 282.376129 MHz
F2 = 1.000000 MHz
SW1 = 73529.41 Hz



xc-10-166-1 F

300

PPM

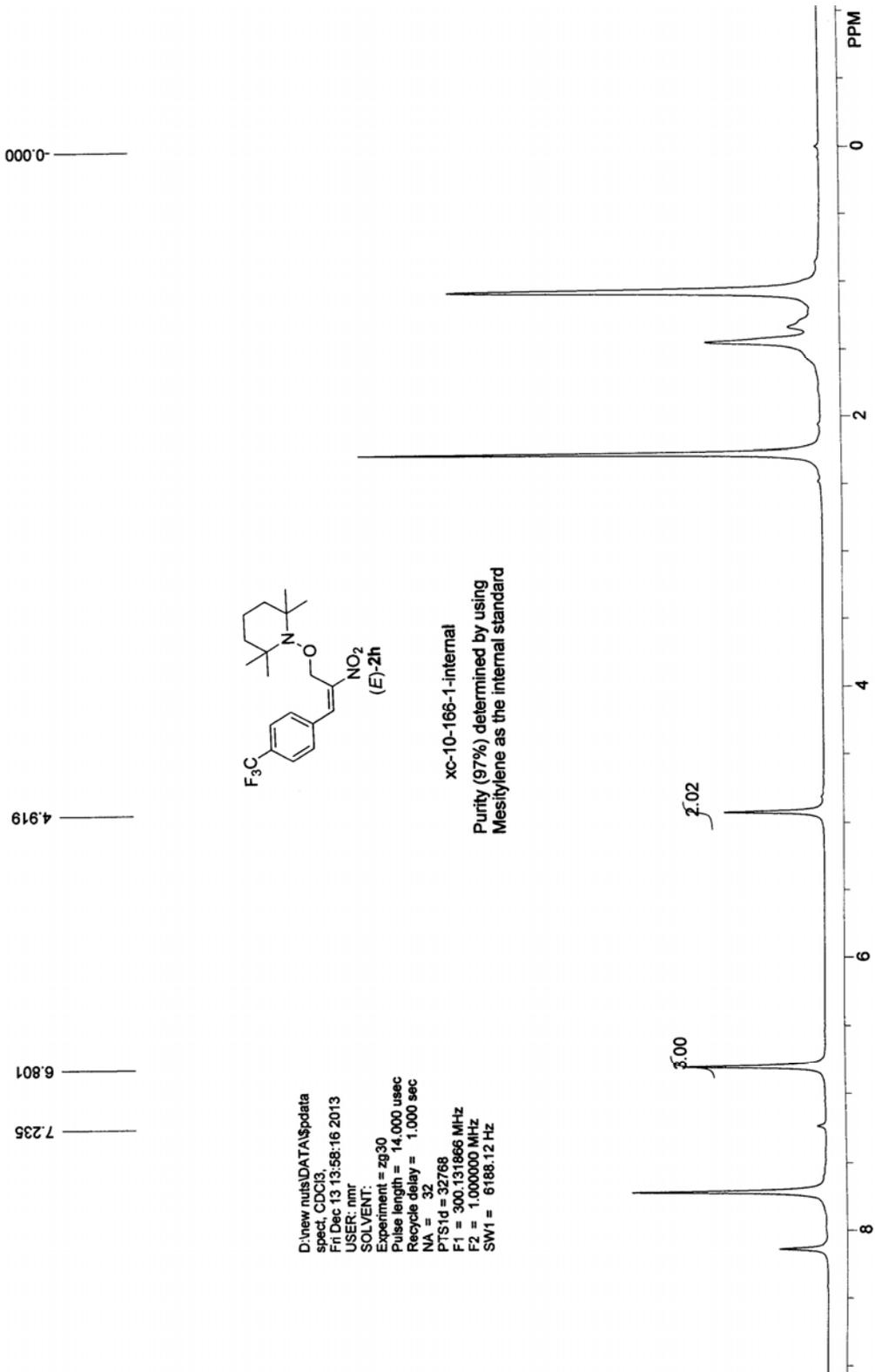
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-150

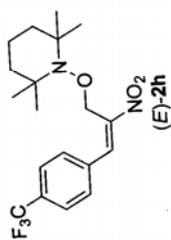
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-50

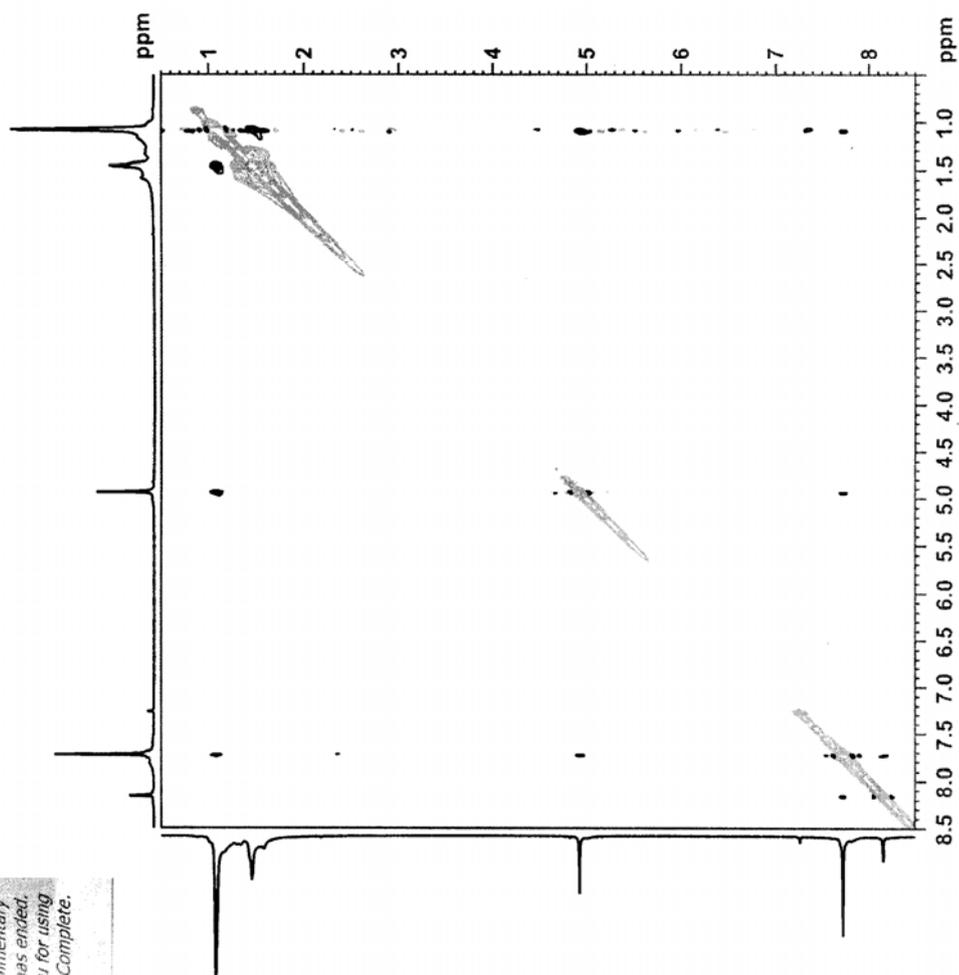
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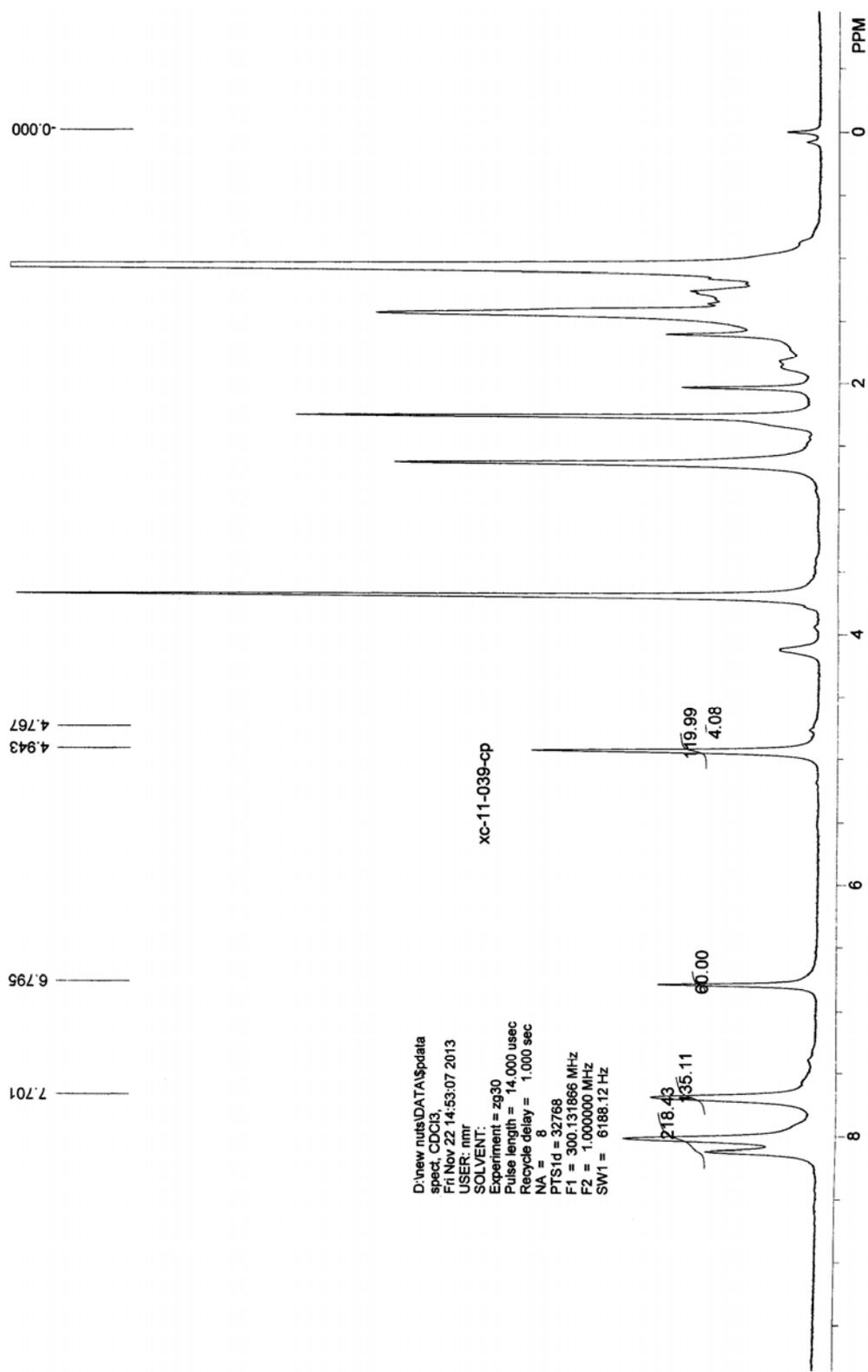


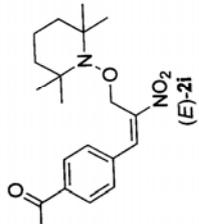
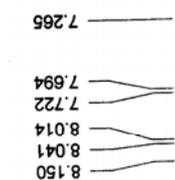
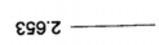
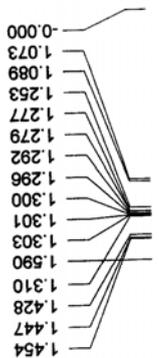
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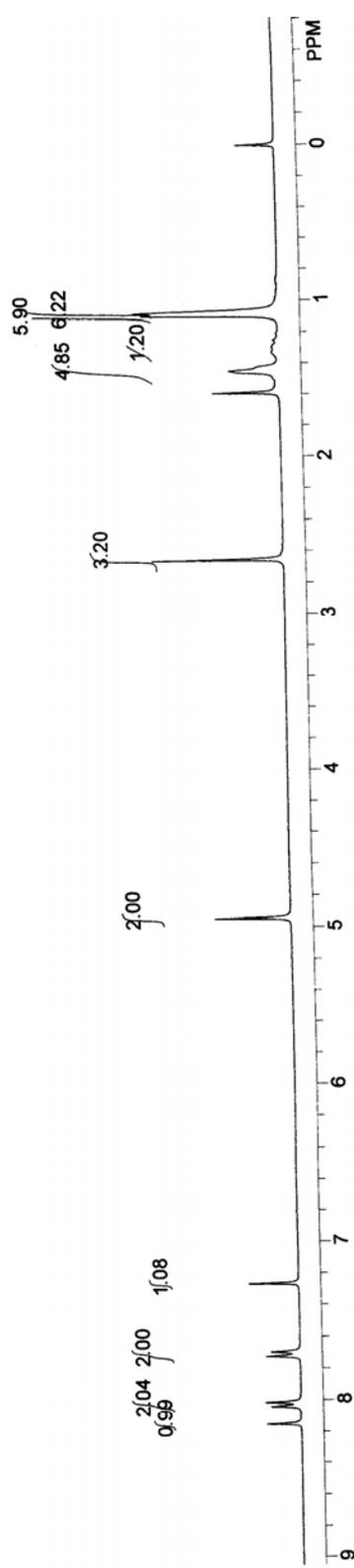


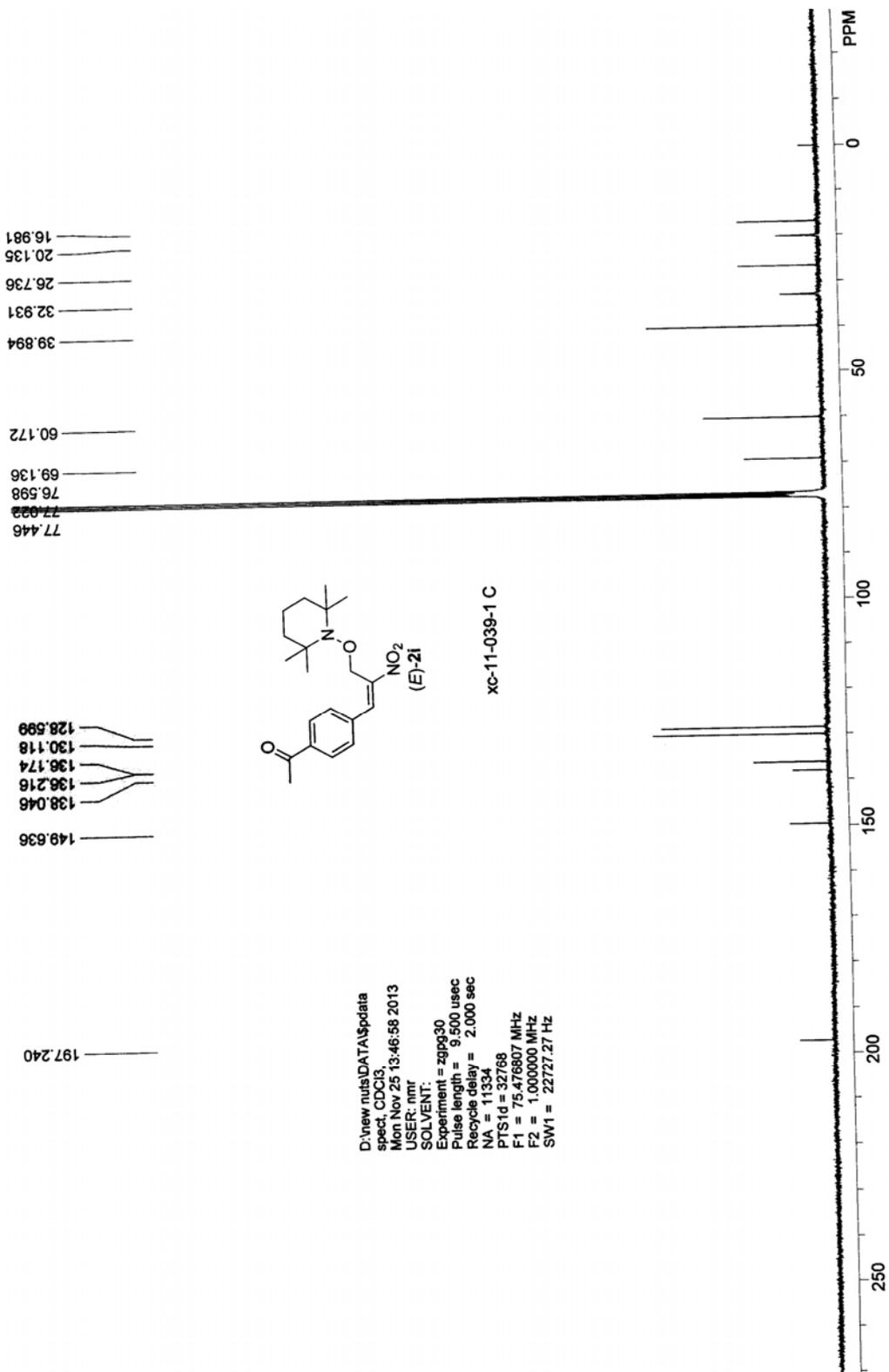




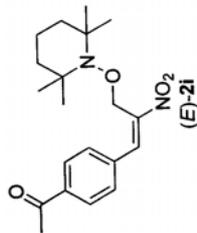
D:\new nuts\DATA\p\data
 spect. CDCl3
 Mon Nov 25 13:42:53 2013
 USER: nmr
 SOLVENT:
 Experiment = zg30
 Pulse length = 14.000 usec
 Recycle delay = 1.000 sec
 NA = 8
 PTS1d = 32768
 F1 = 300.131866 MHz
 F2 = 1.000000 MHz
 SW1 = 6188.12 Hz

xc-11-039-1

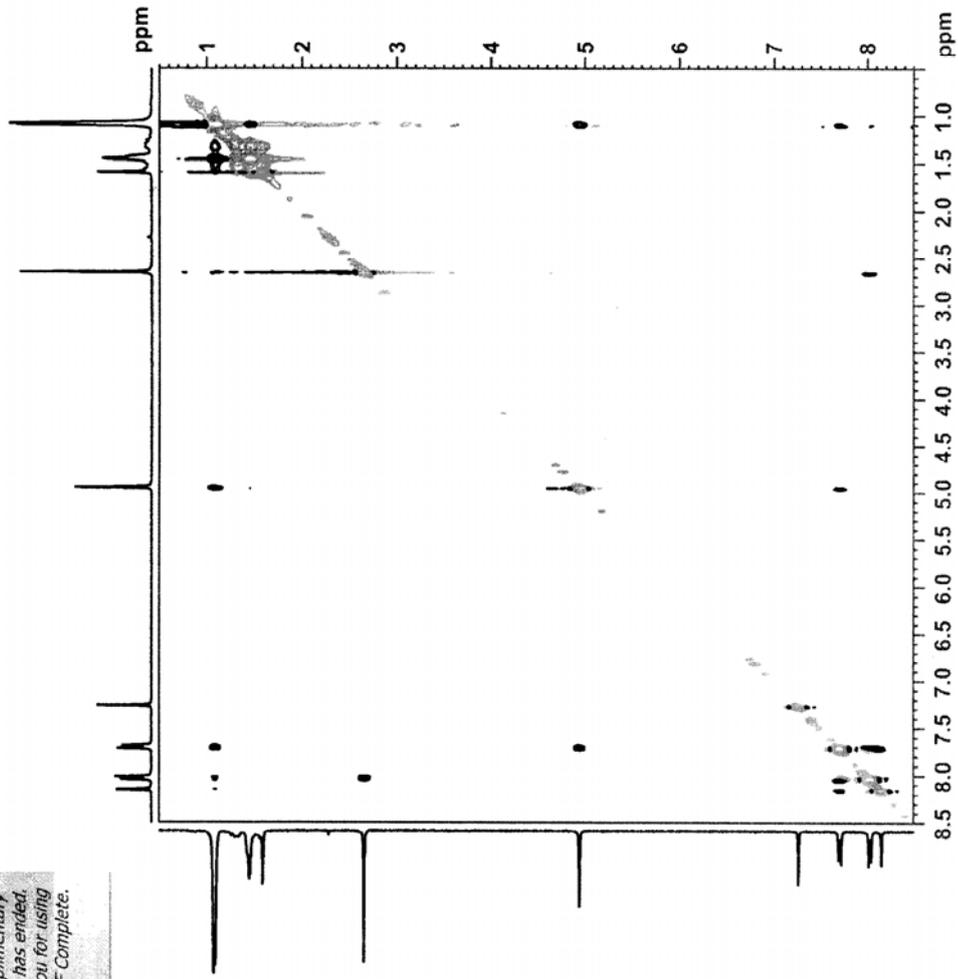


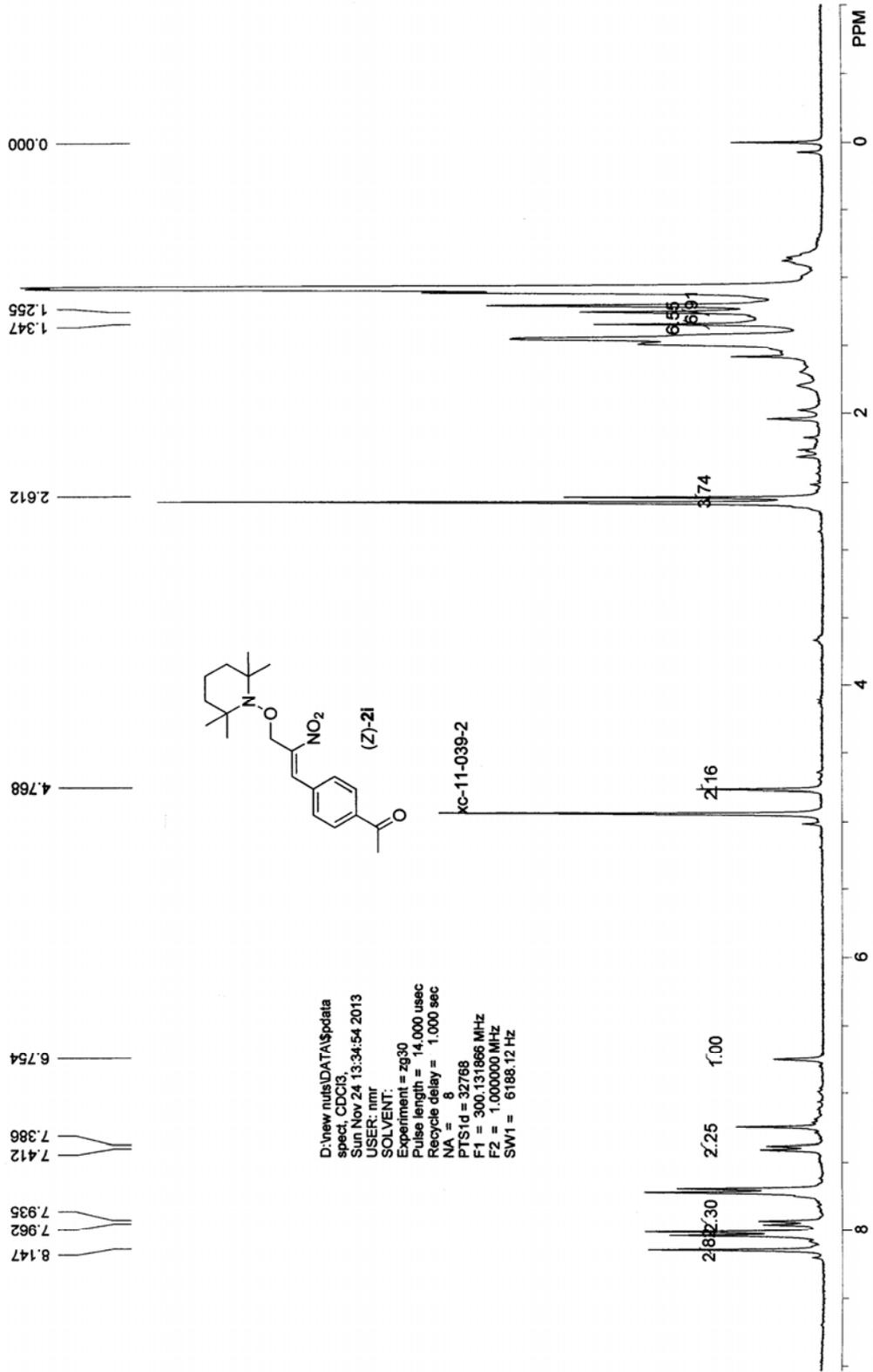


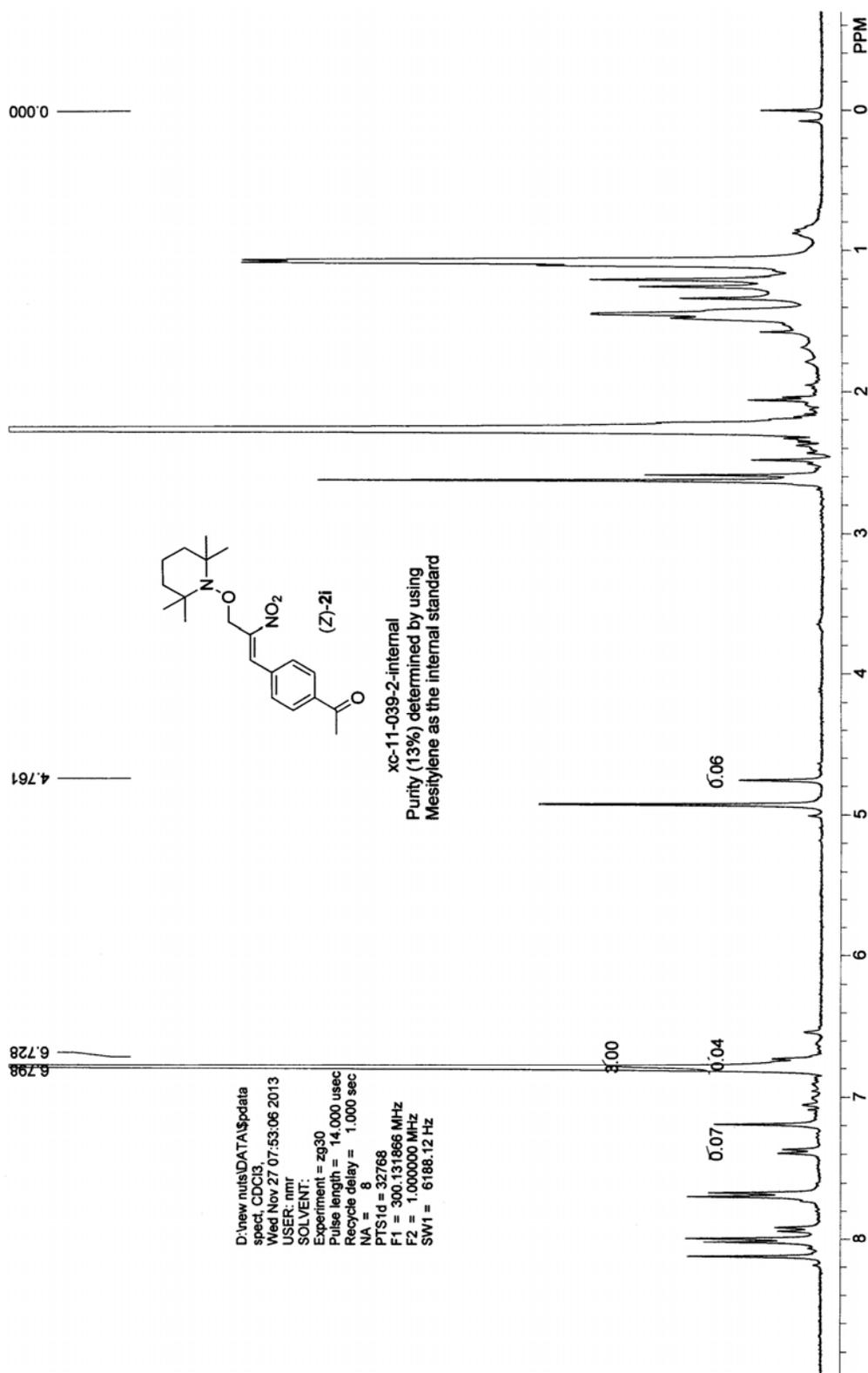
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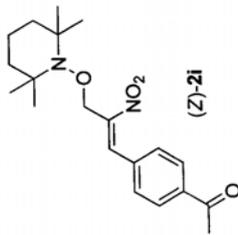
xc-11-039-1-noe



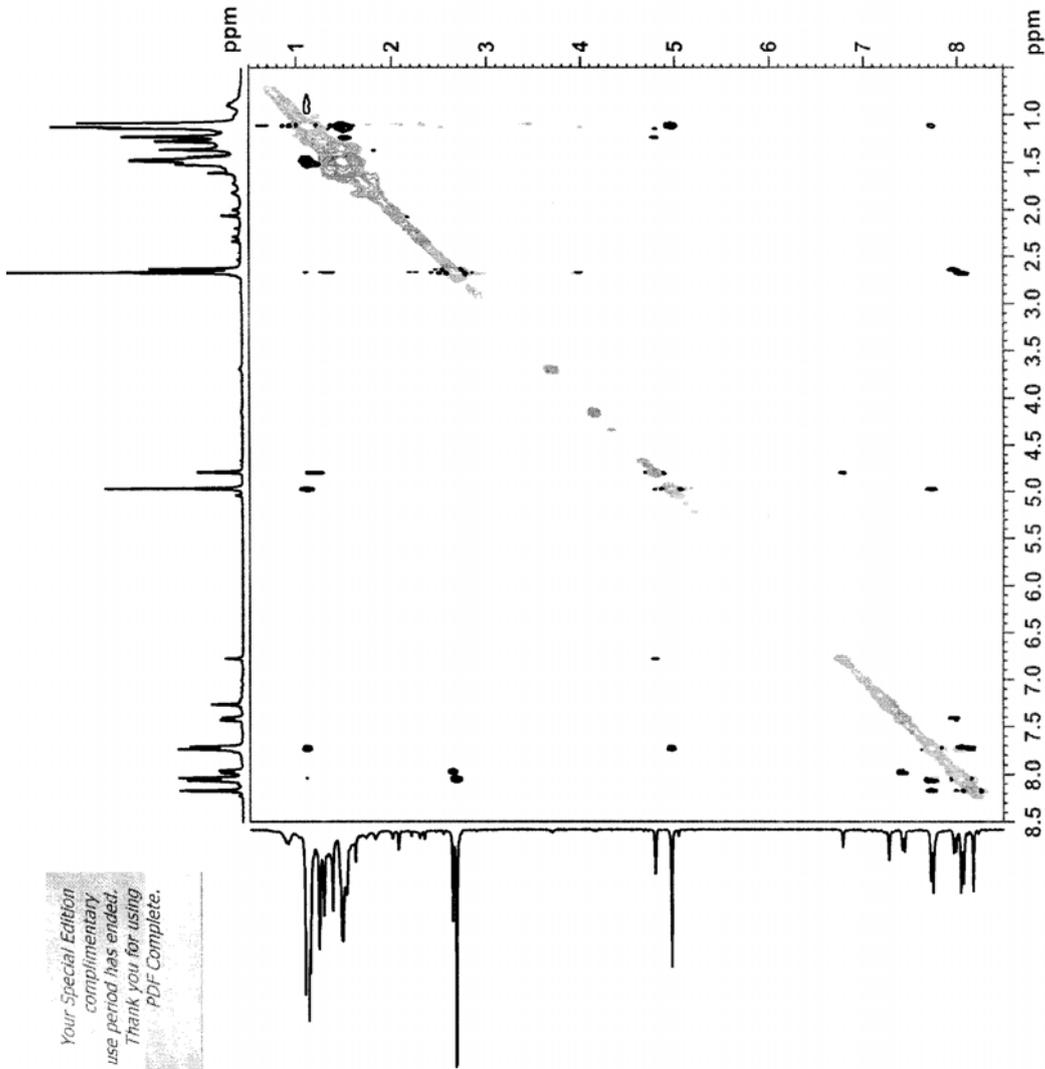


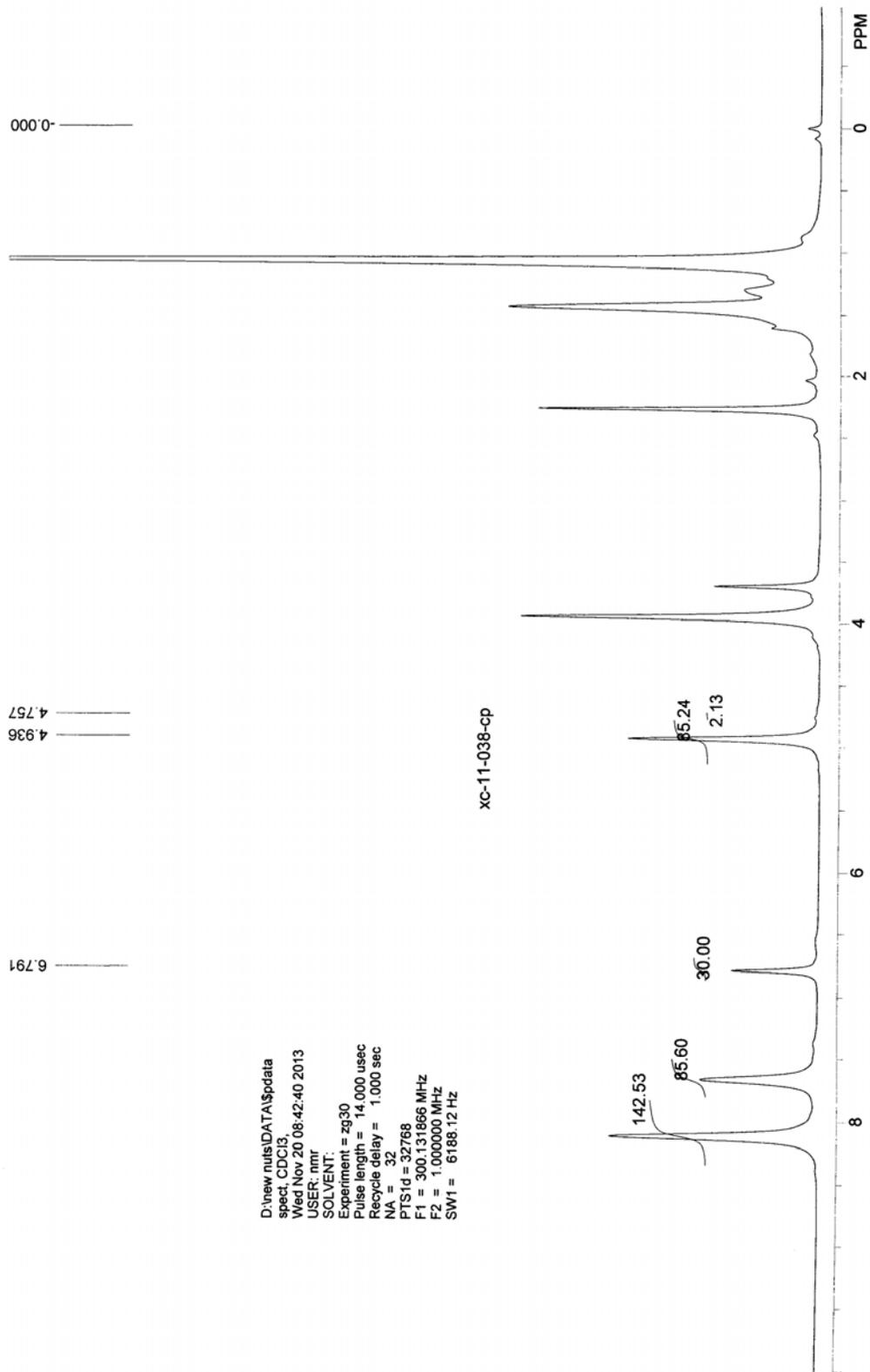


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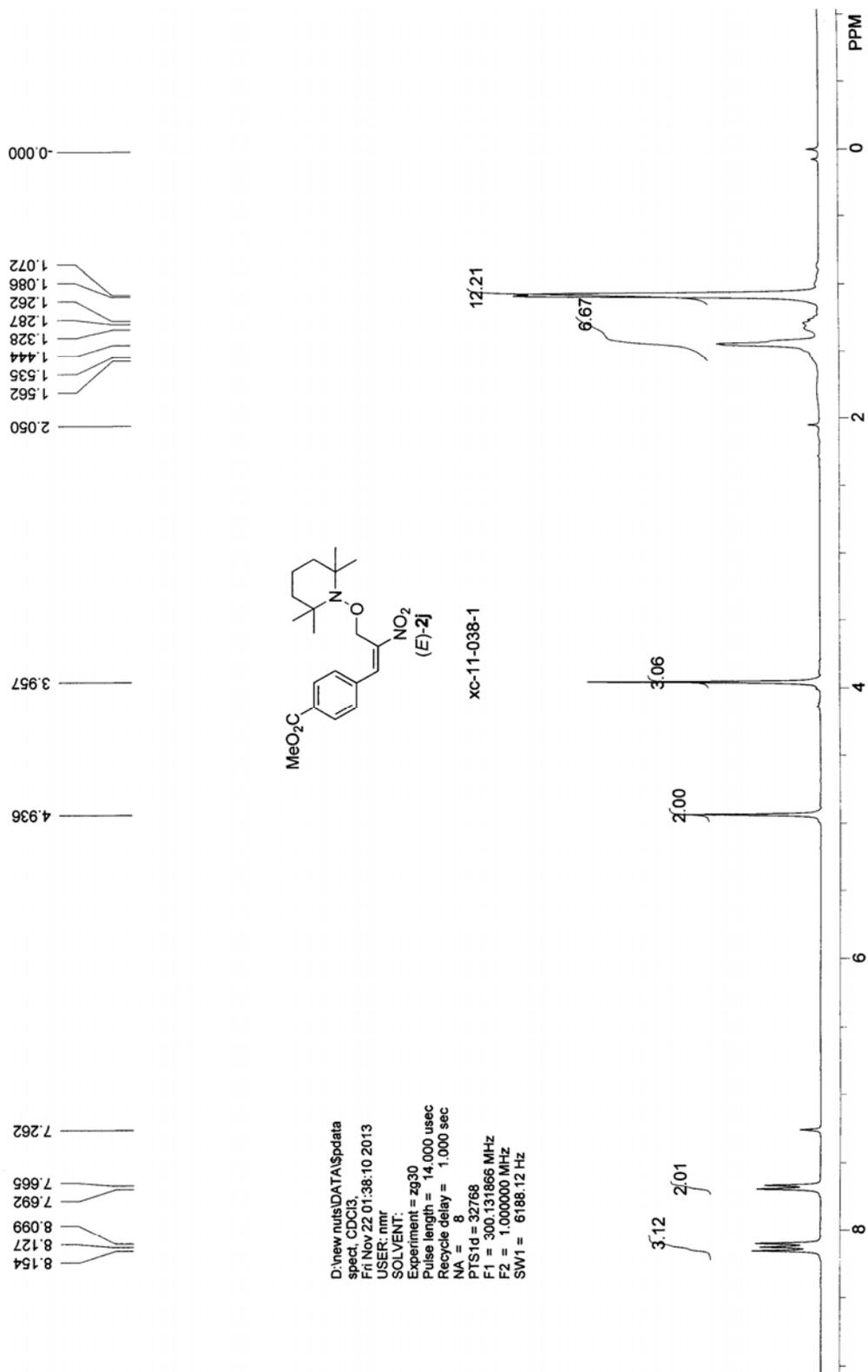


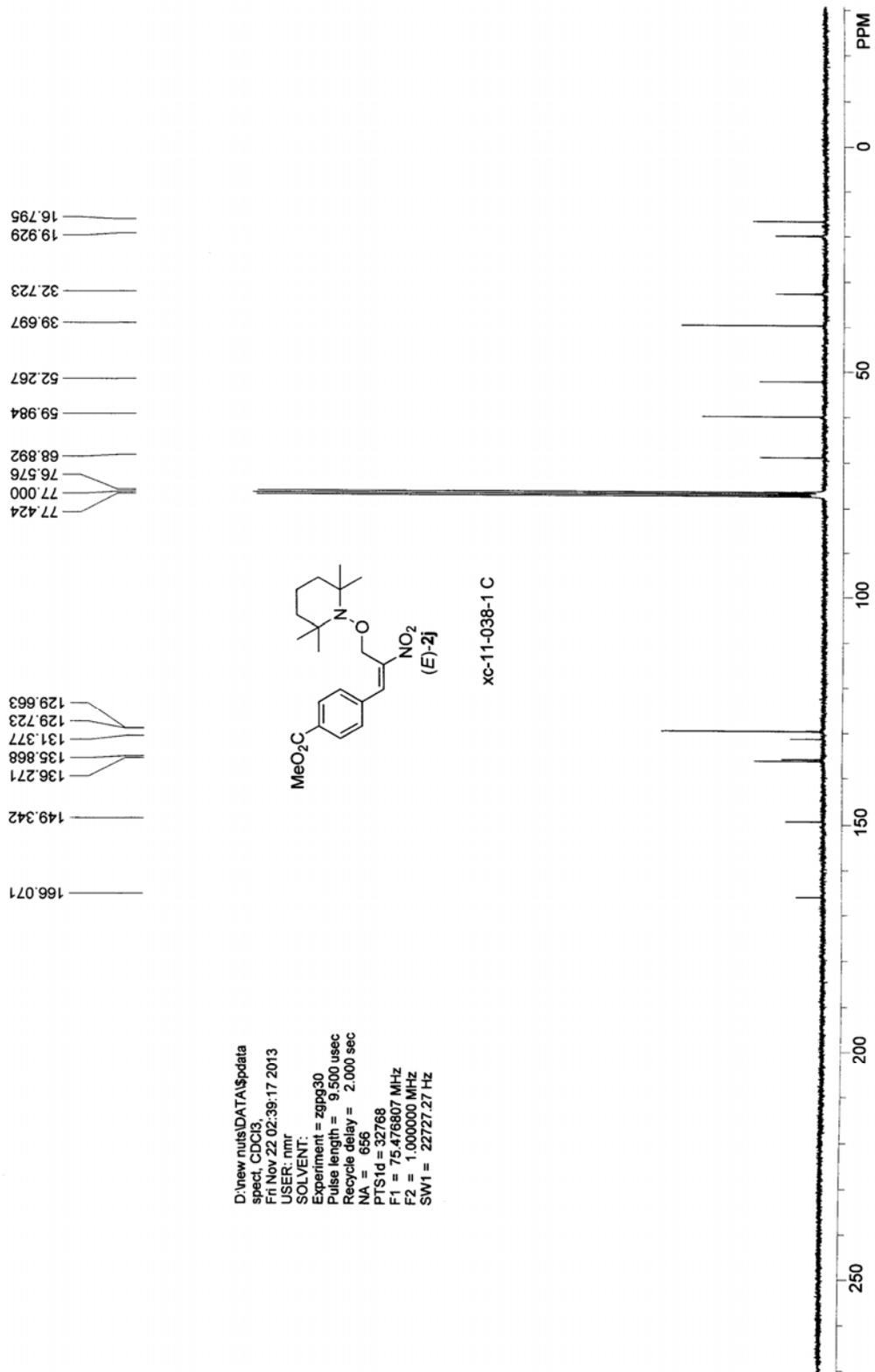
xc-11-039-2-noe

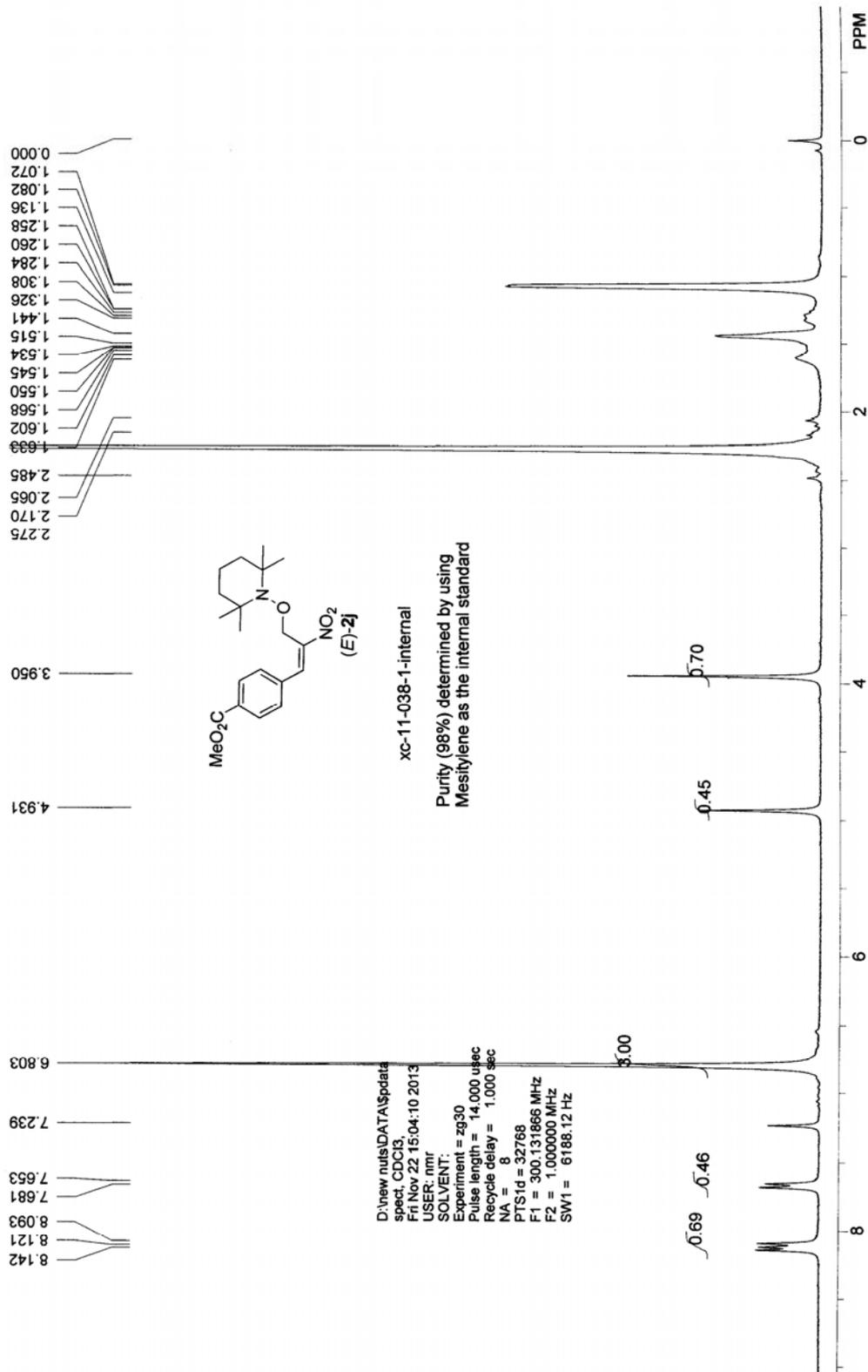




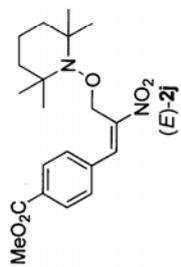
D:\new nuts\DATA\Spdata
 spect, CDC18,
 Wed Nov 20 08:42:40 2013
 USER: nmr
 SOLVENT:
 Experiment = zg30
 Pulse length = 14.000 usec
 Recycle delay = 1.000 sec
 NA = 32
 P1 = 32768
 F1 = 300.131866 MHz
 F2 = 1.000000 MHz
 SW1 = 6188.12 Hz



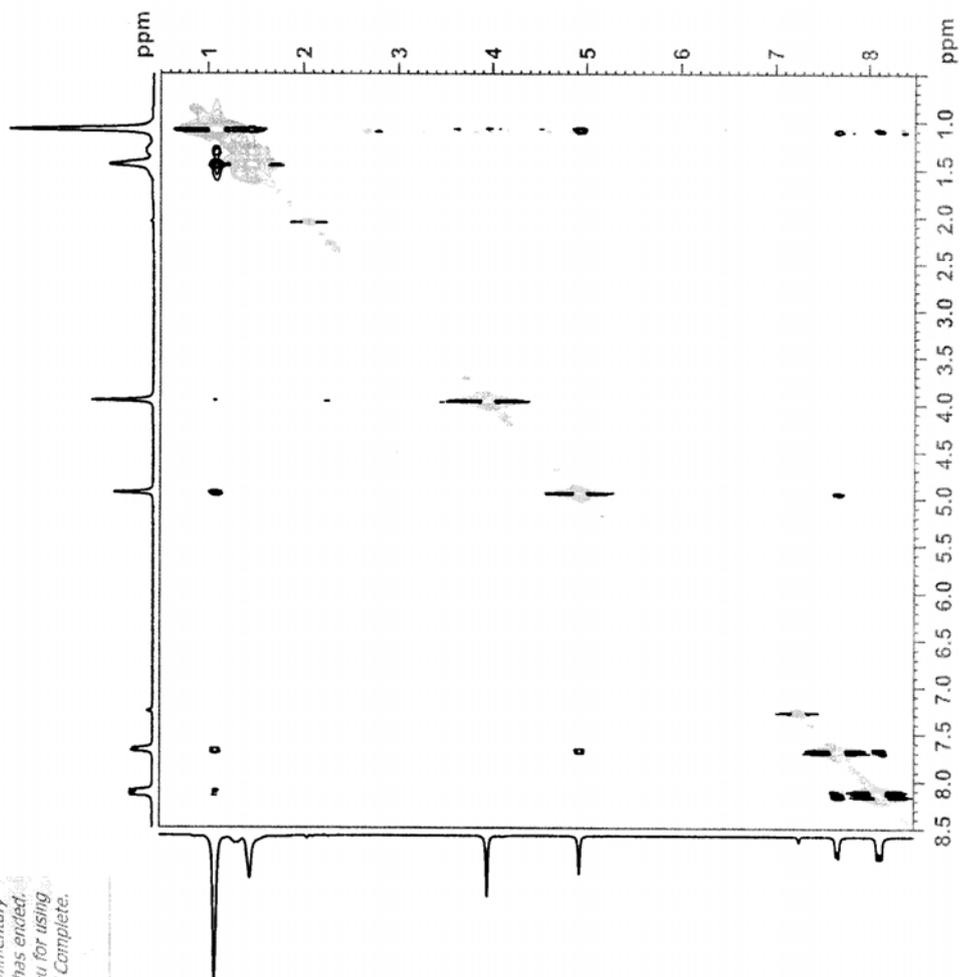


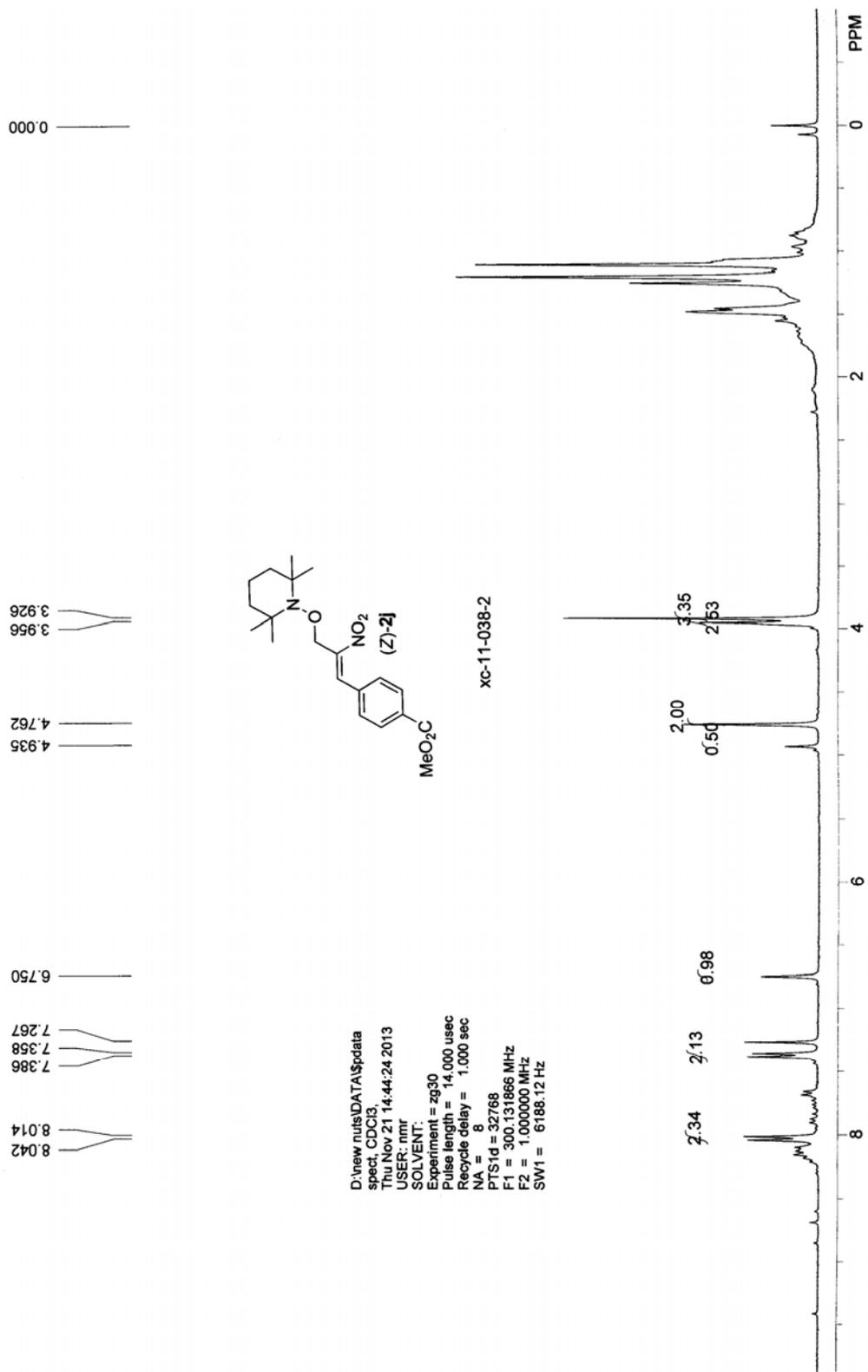


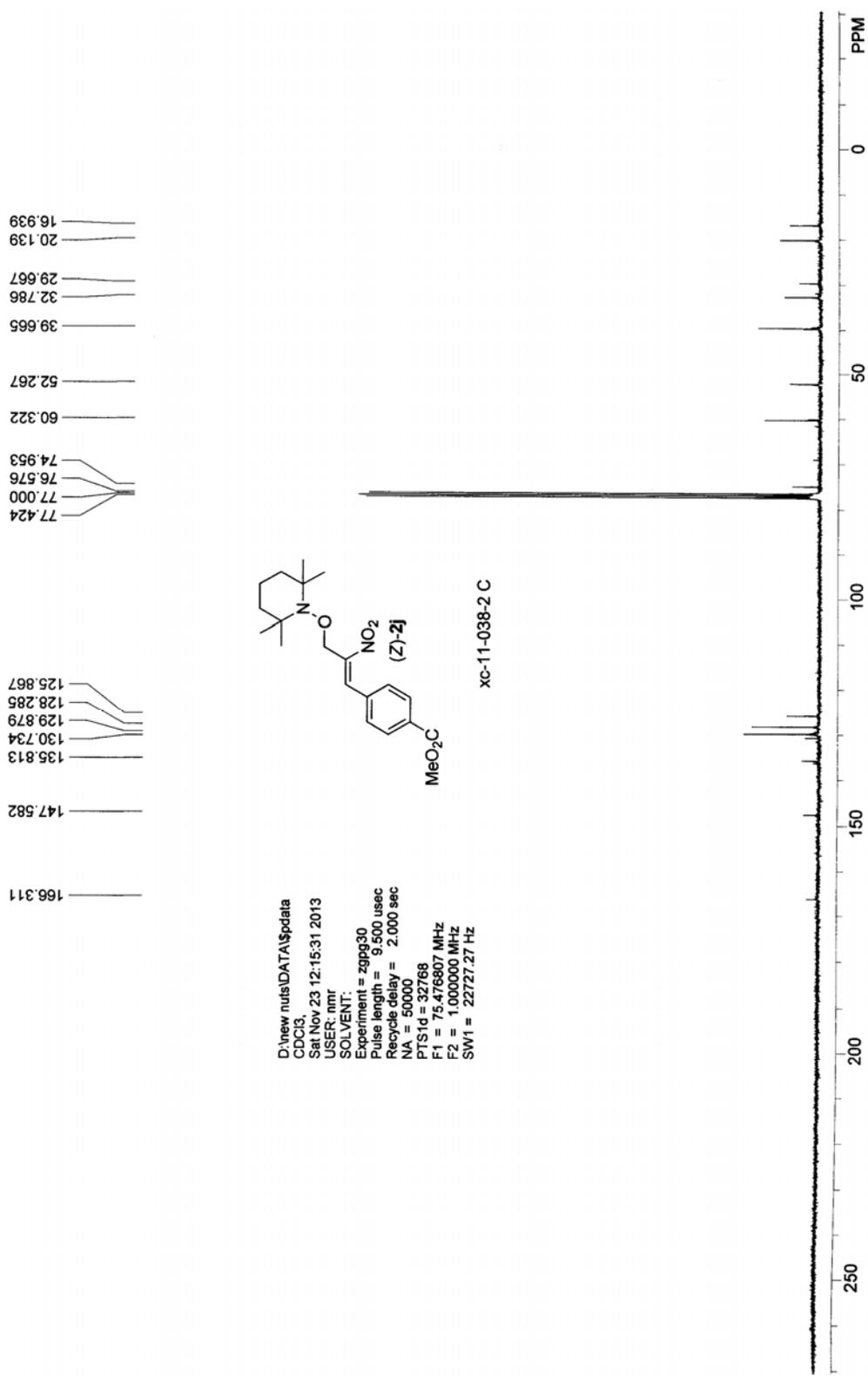
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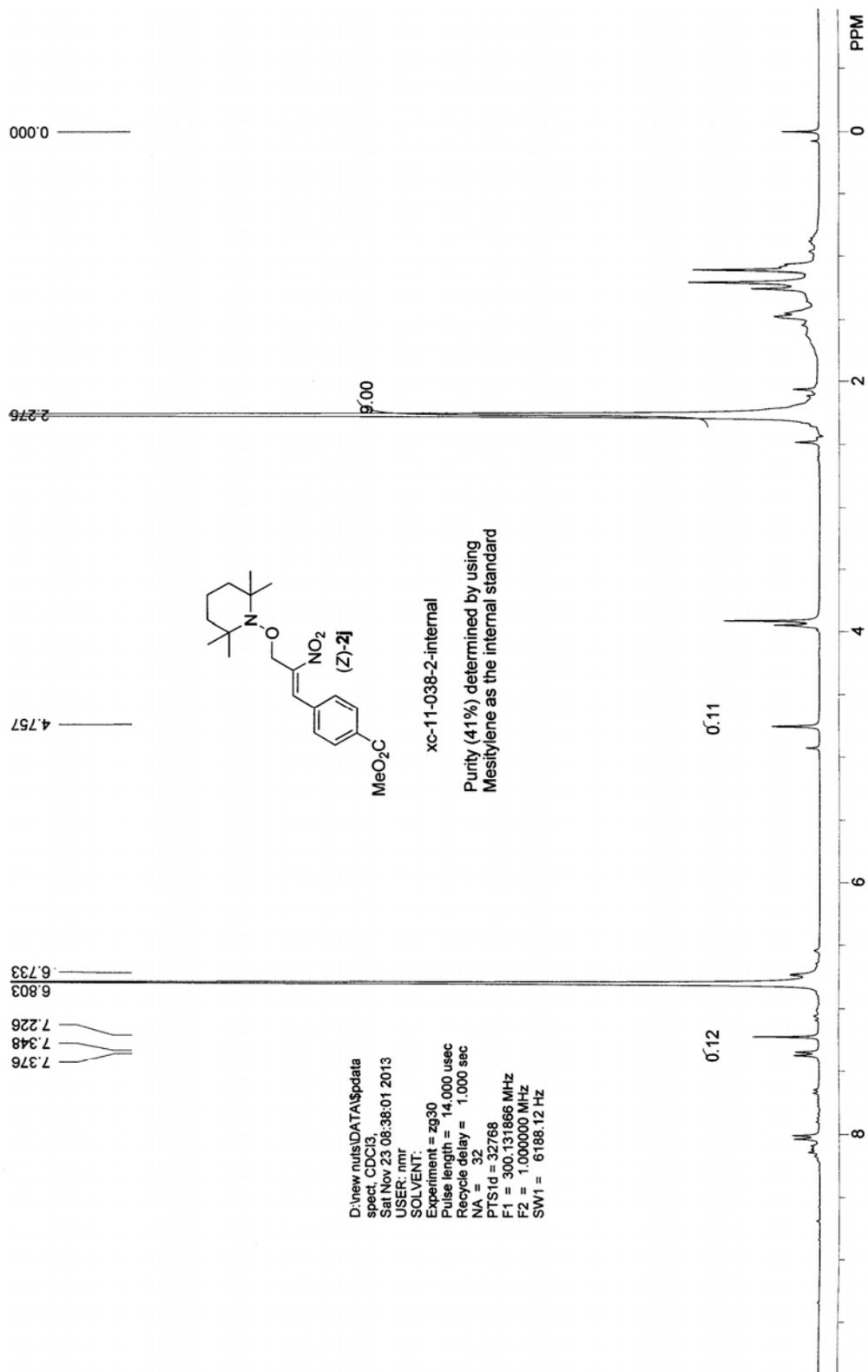


xc-11-038-1-noe

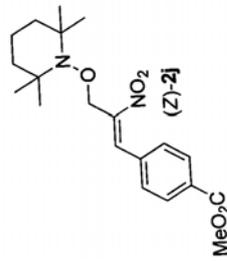




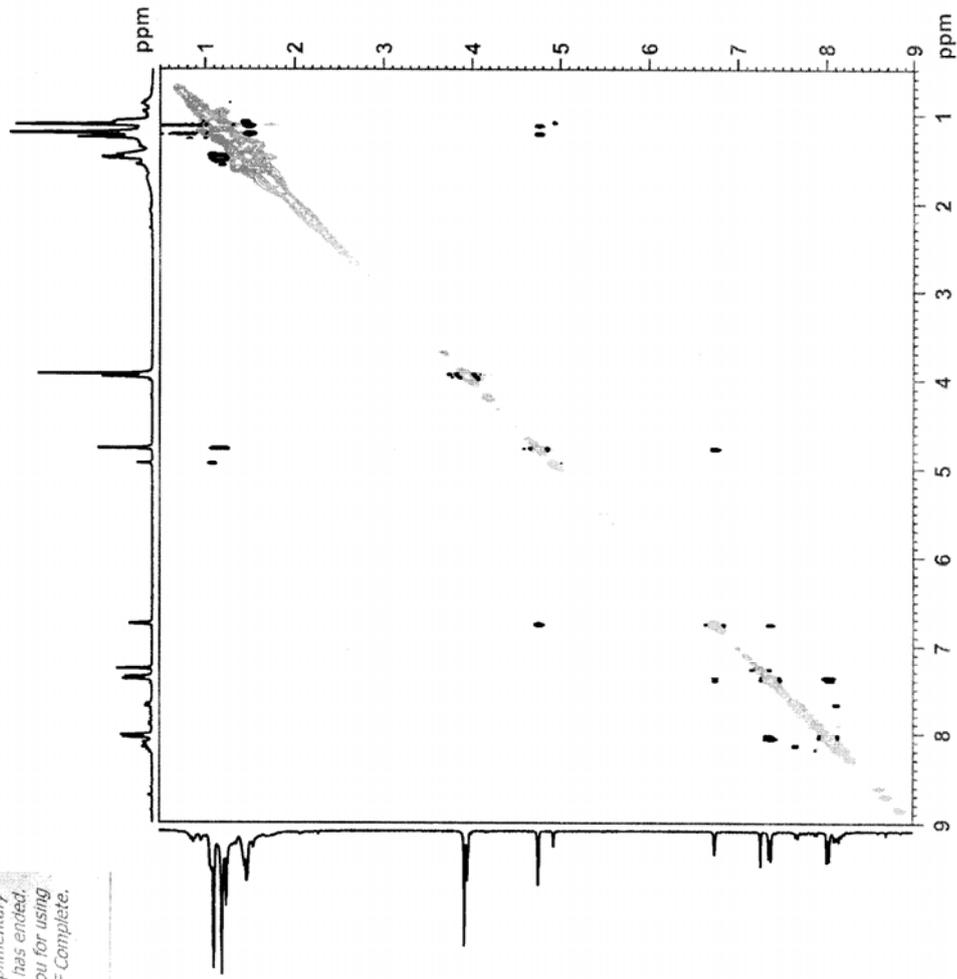


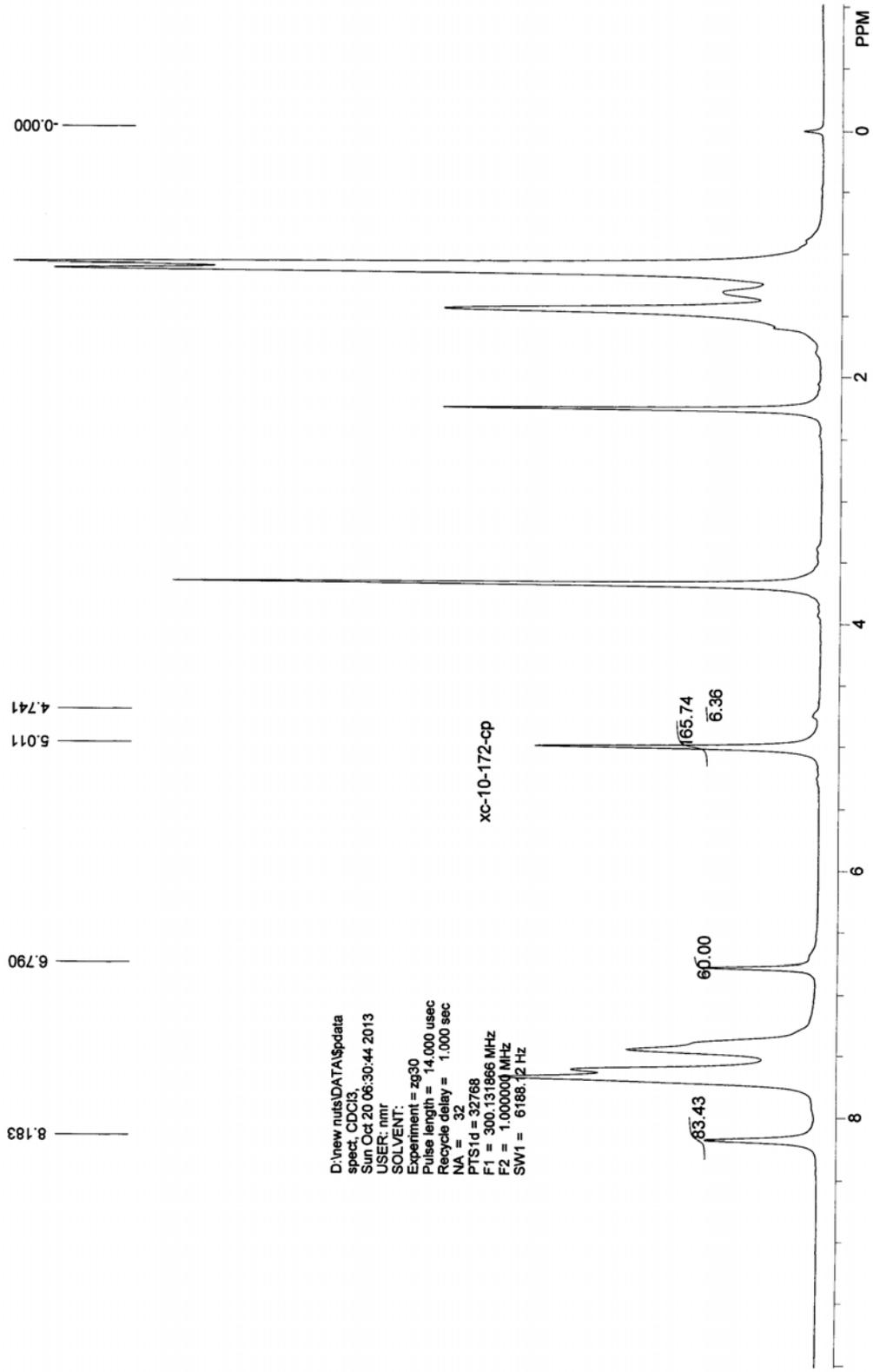


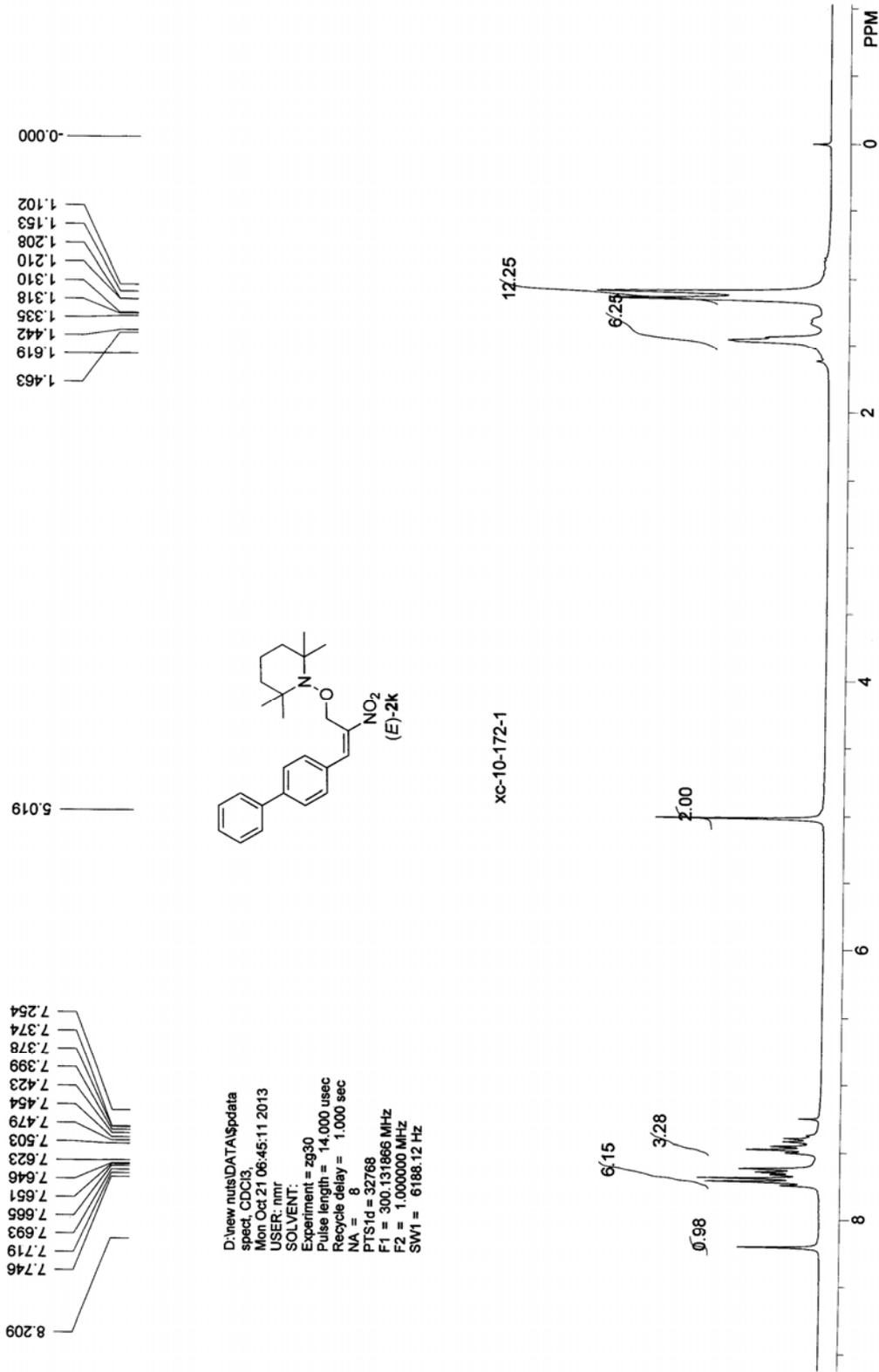
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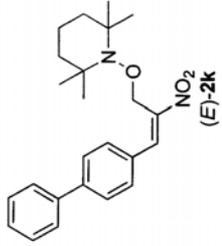
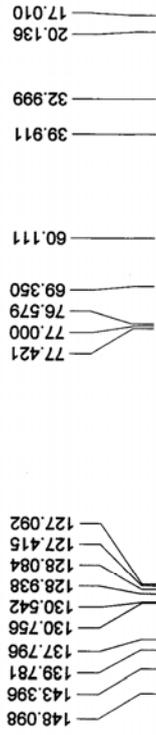


xc-11-038-2-noe



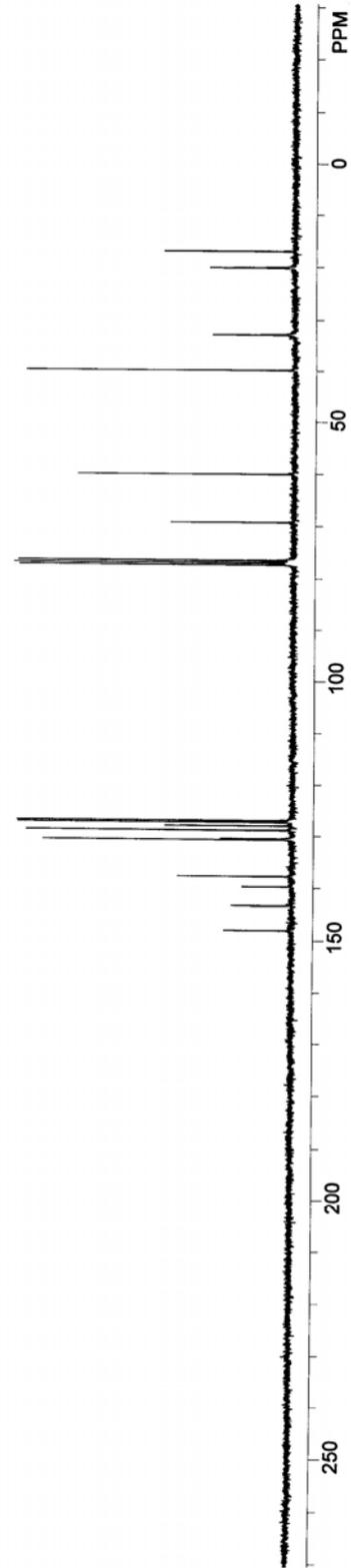




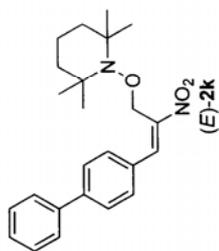
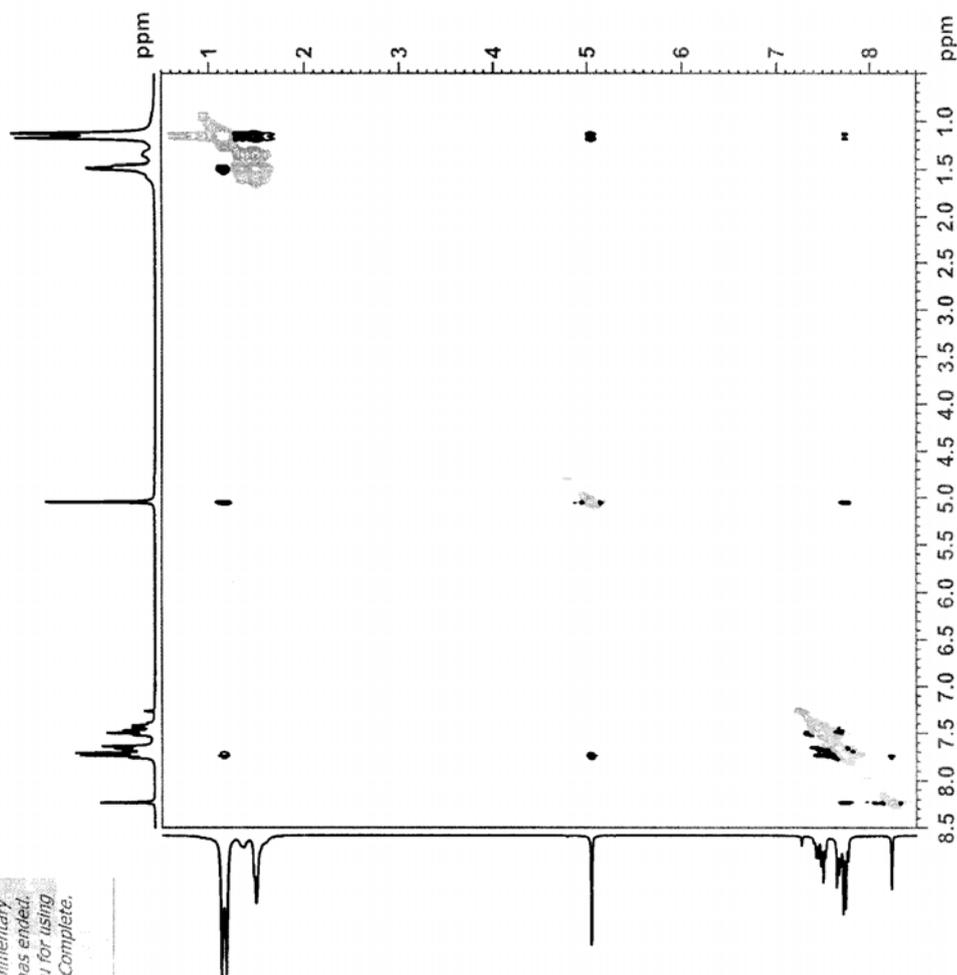


D:\new nuts\DATA\Sp\data
spect, CDC13,
Mon Oct 21 06:47:21 2013
USER: nmr
SOLVENT:
Experiment = z9p930
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 444
PTS1d = 32768
F1 = 75.476807 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz

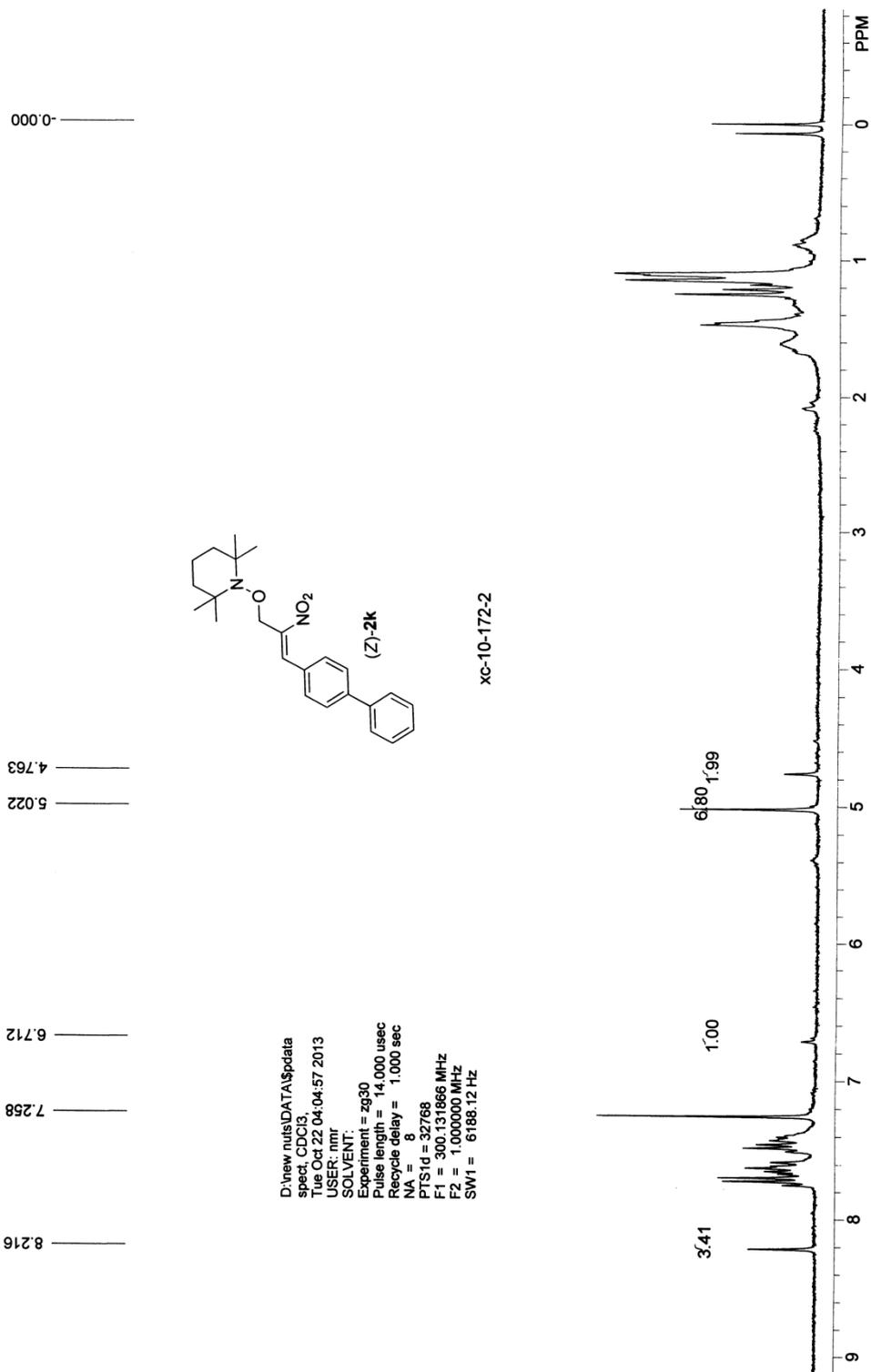
xc-10-172-1 C

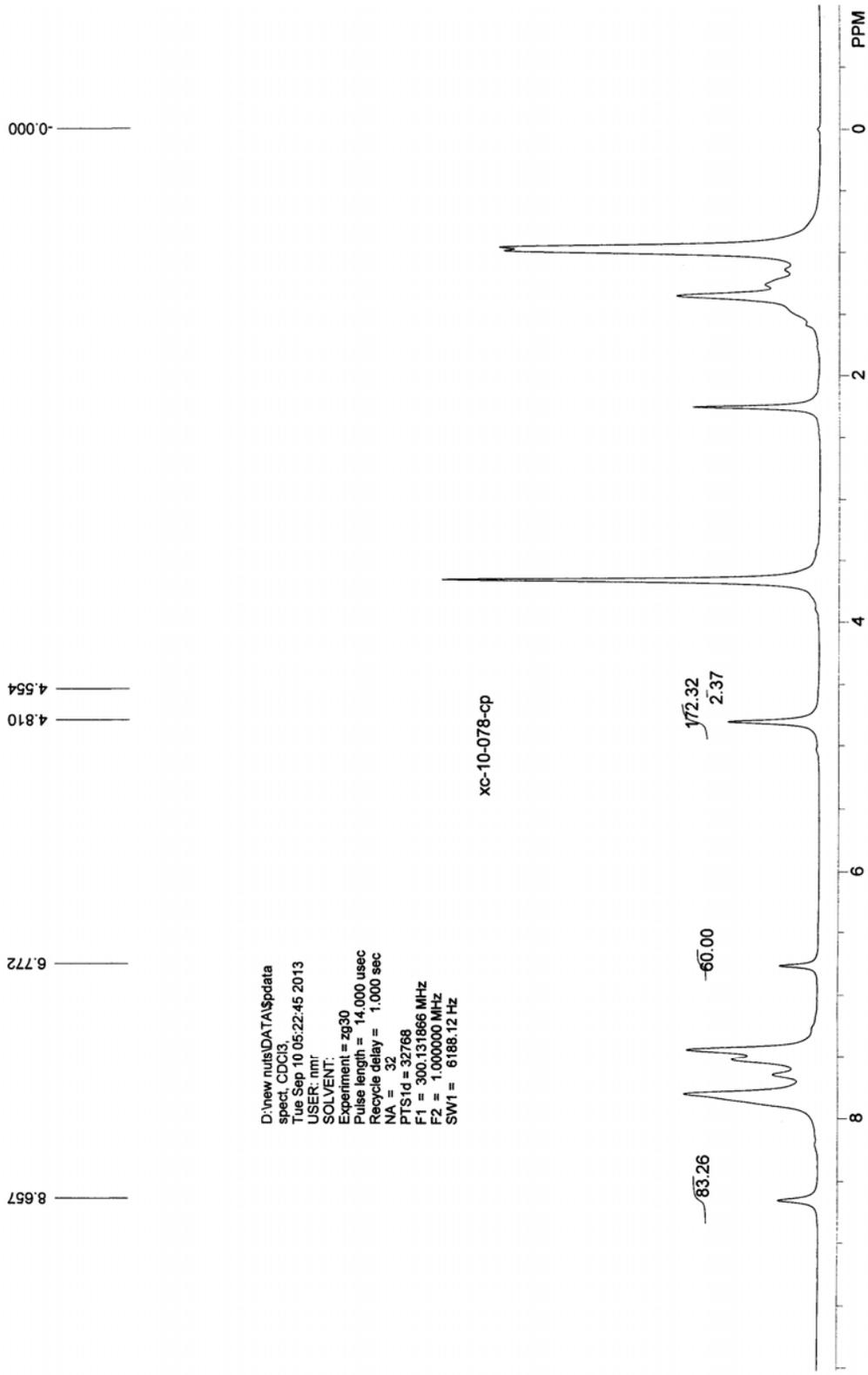


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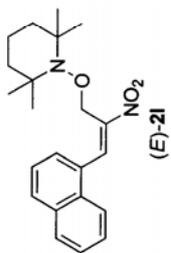
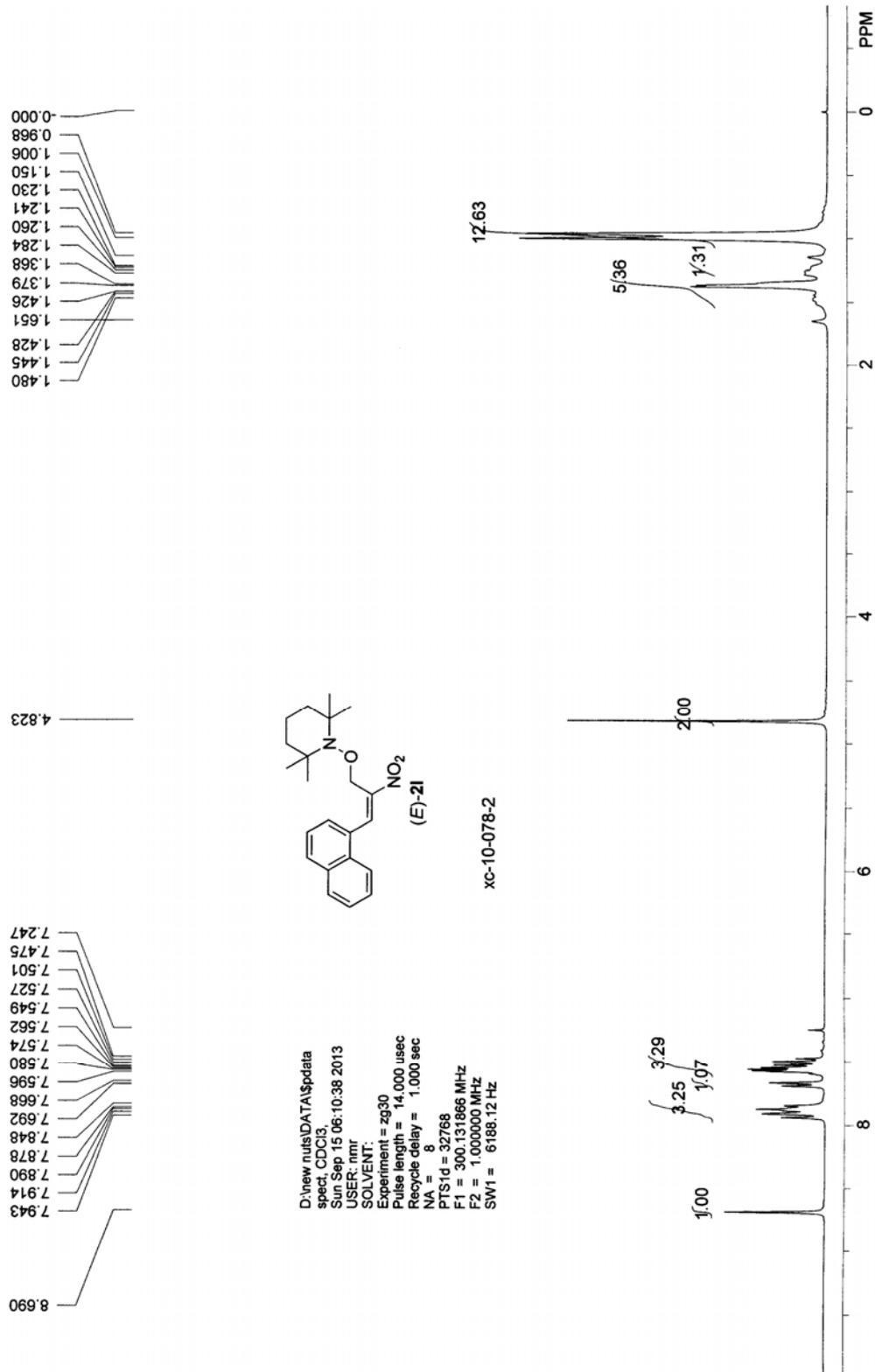


xc-10-172-1-noe



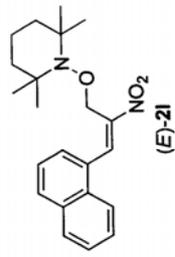
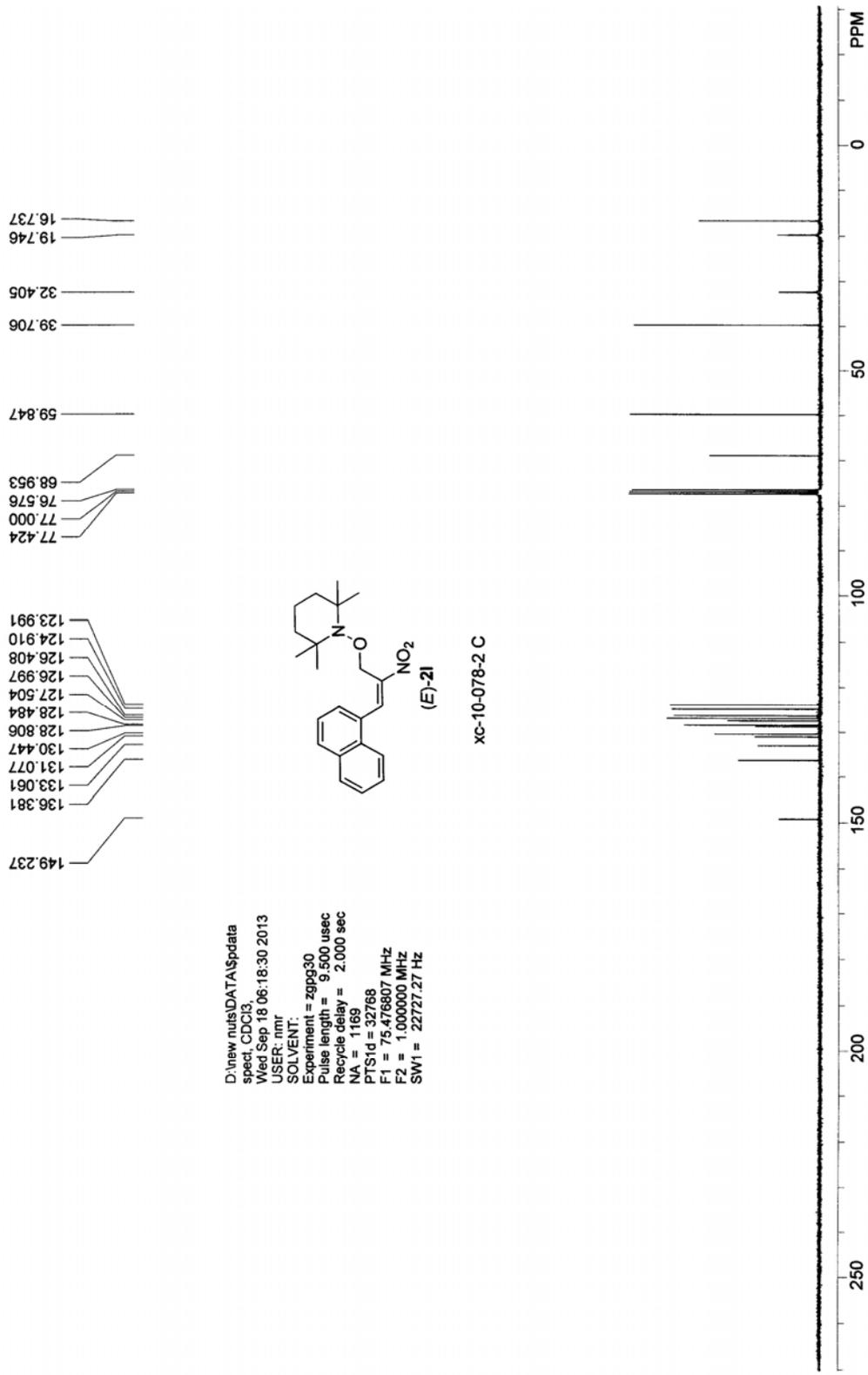


D:\new nulis\DATA\Spdata
 spect, CDC13,
 Tue Sep 10 05:22:45 2013
 USER: nmr
 SOLVENT:
 Experiment = zg30
 Pulse length = 14.000 usec
 Recycle delay = 1.000 sec
 NA = 32
 P1 = 32768
 F1 = 300.131866 MHz
 F2 = 1.000000 MHz
 SW1 = 6188.12 Hz



xc-10-078-2

D:\new_nuis\DATA\spdata
 spect_CDCl3
 Sun Sep 15 06:10:38 2013
 USER: nmr
 SOLVENT:
 Experiment = zg30
 Pulse length = 14.000 usec
 Recycle delay = 1.000 sec
 NA = 8
 PTS1d = 32768
 F1 = 300.131866 MHz
 F2 = 1.000000 MHz
 SW1 = 6188.12 Hz

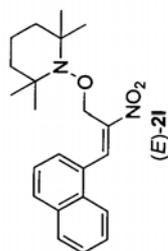
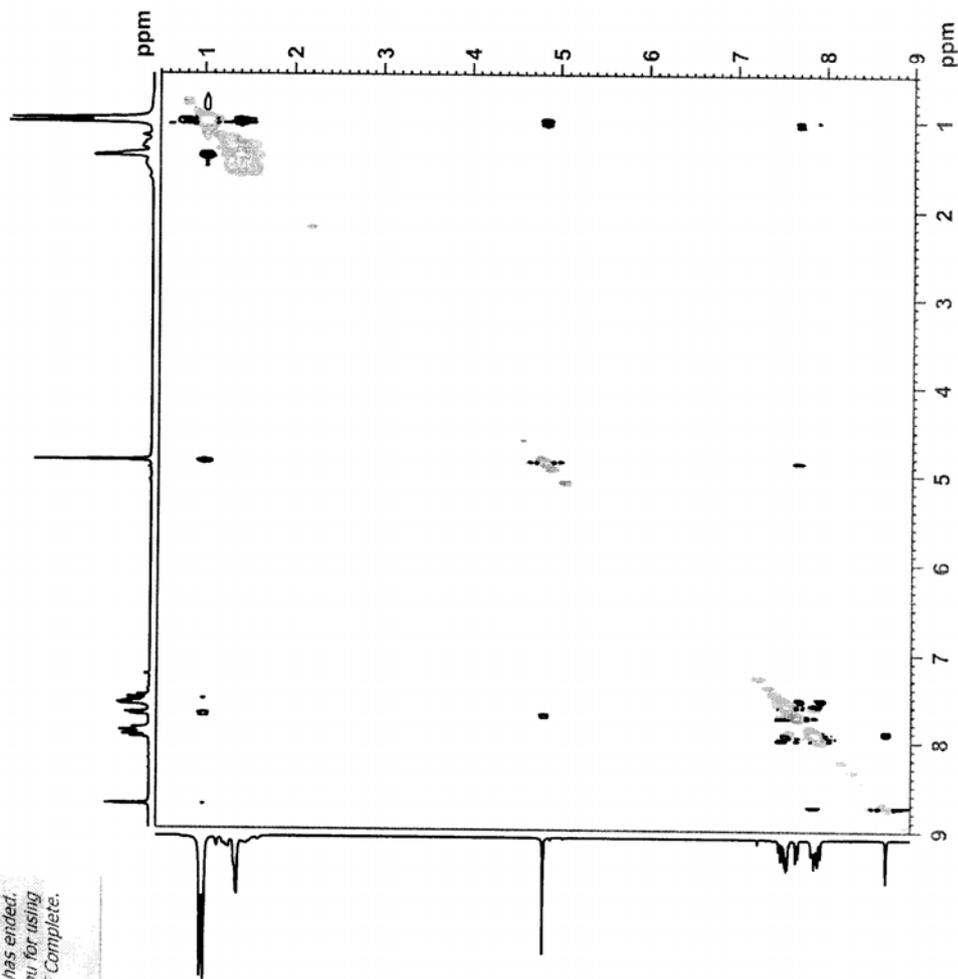


xc-10-078-2 C

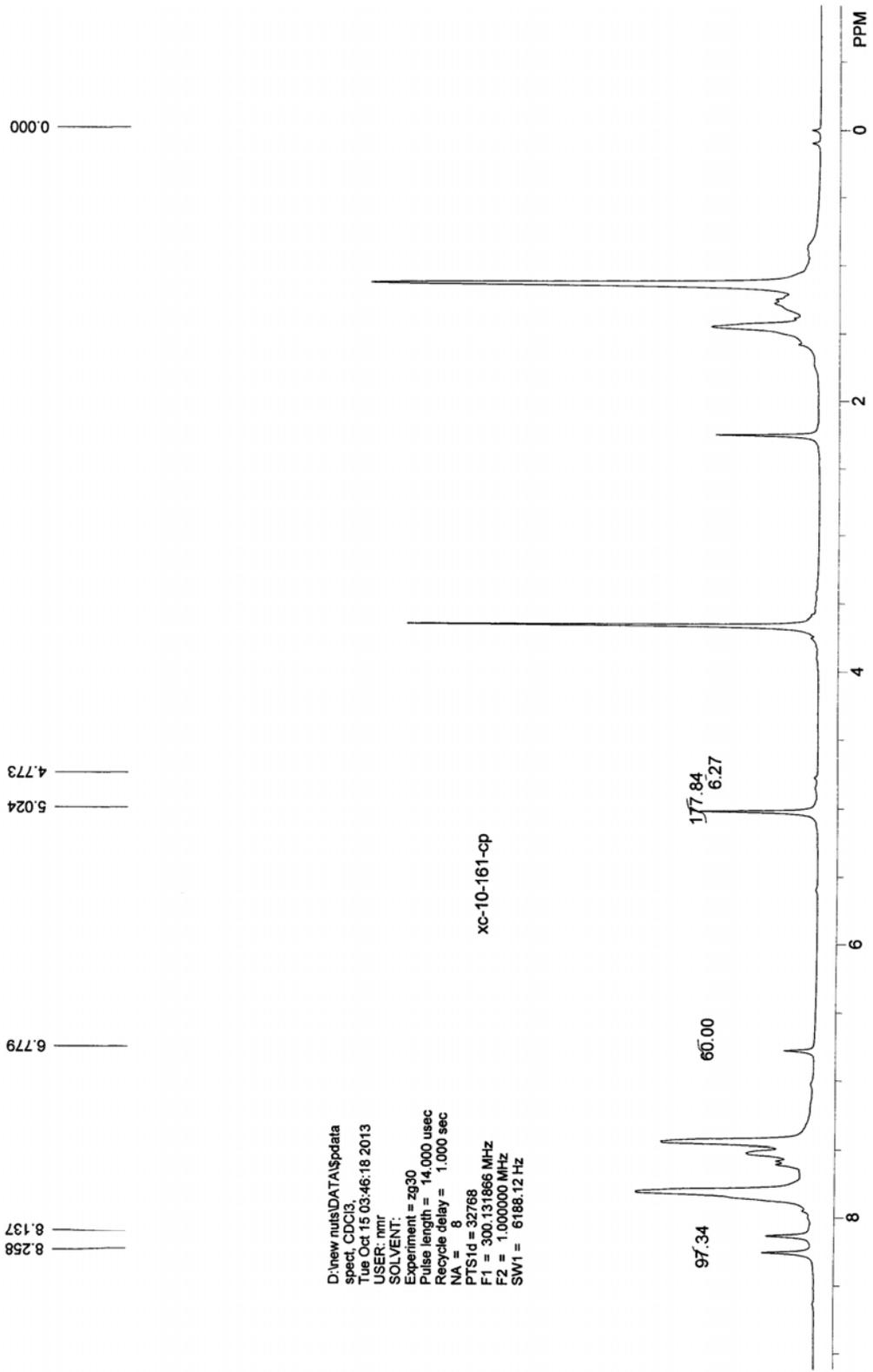
D:\new nuts\DATA\spdata
 spect, CDC13
 Wed Sep 18 06:18:30 2013
 USER: nmr
 SOLVENT:
 Experiment = zgpg30
 Pulse length = 9.500 usec
 Recycle delay = 2.000 sec
 NA = 1169
 PTS1d = 32768
 F1 = 75.476807 MHz
 F2 = 1.000000 MHz
 SW1 = 22727.27 Hz

- 16.737
- 19.746
- 32.405
- 39.706
- 59.847
- 68.953
- 76.576
- 77.000
- 77.424
- 123.991
- 124.910
- 126.408
- 126.997
- 127.504
- 128.484
- 128.806
- 130.447
- 131.077
- 133.061
- 136.381
- 149.237

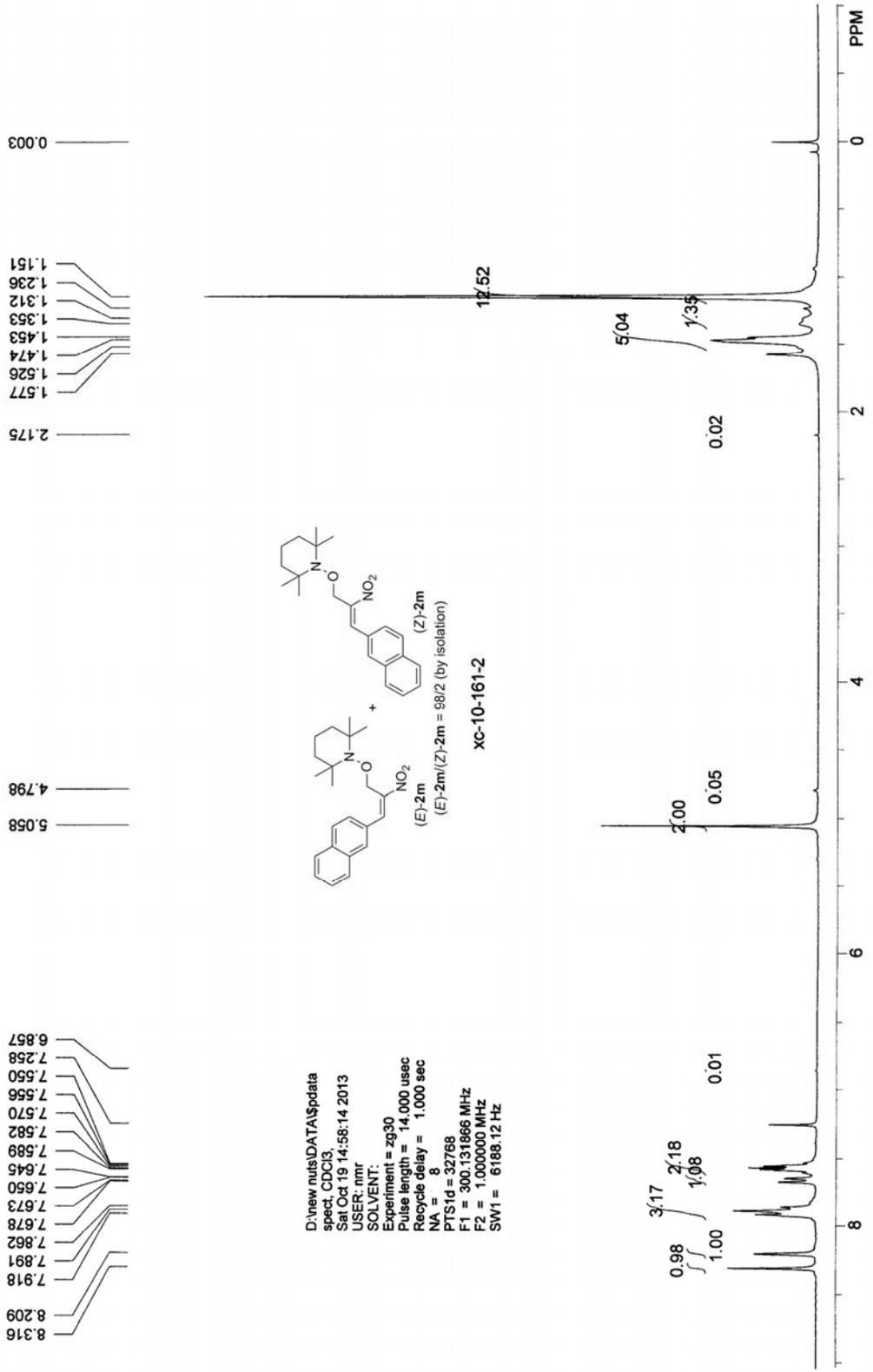
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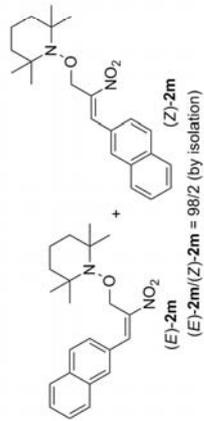
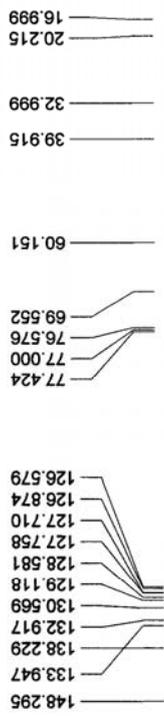
xc-10-078-2-noe



D:\new_nuis\DATA\spdata
 spect, CDCl3,
 Tue Oct 15 03:46:18 2013
 USER: nmr
 SOLVENT:
 Experiment = zg30
 Pulse length = 14.000 usec
 Recycle delay = 1.000 sec
 NA = 8
 P1 = 32768
 F1 = 300.131866 MHz
 F2 = 1.000000 MHz
 SW1 = 6188.12 Hz



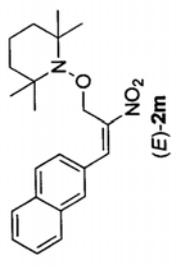
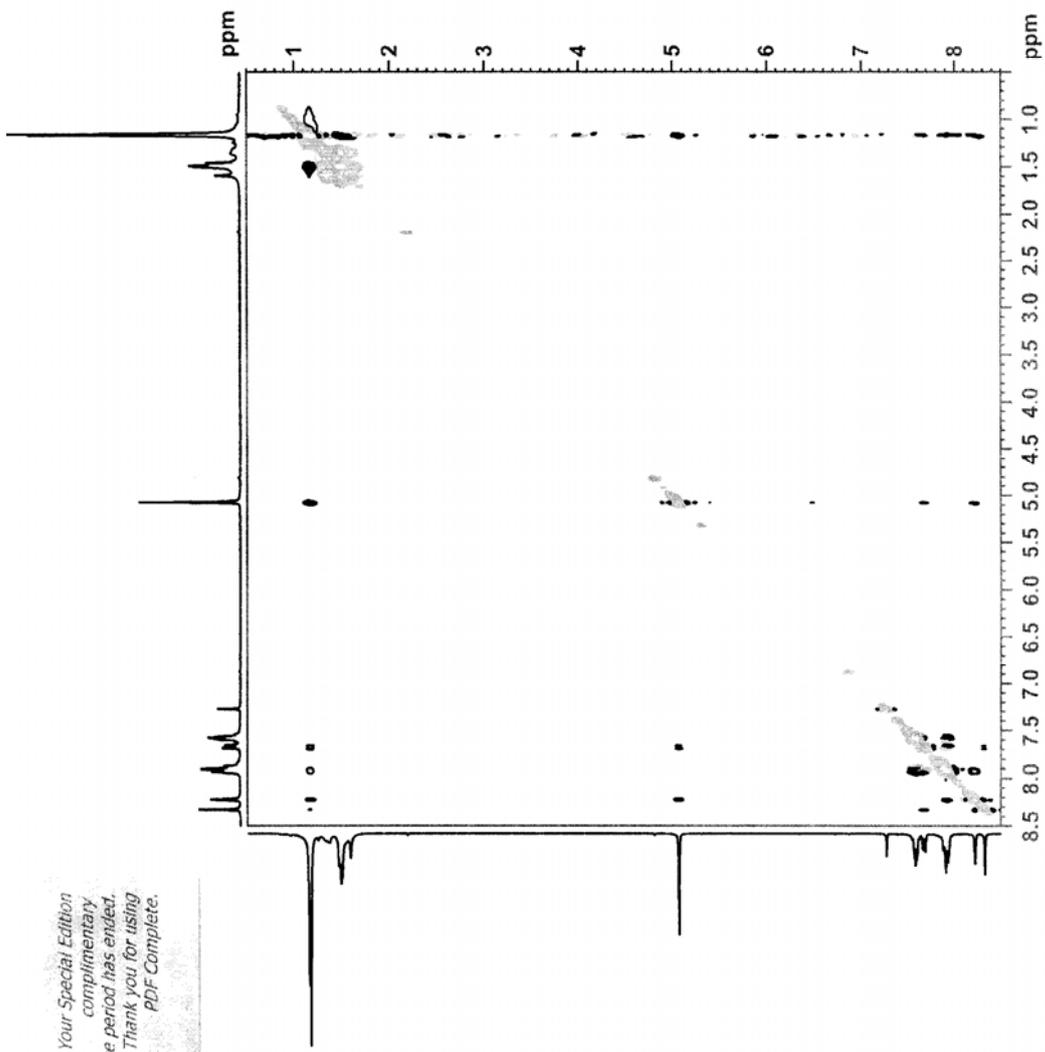
D:\new nuis\DATA\spdata
 spec1, CDCl3,
 Sat Oct 19 05:07:20 2013
 USER: nmr
 SOLVENT:
 Experiment = zgpg30
 Pulse length = 9.500 usec
 Recycle delay = 2.000 sec
 NA = 980
 P1 = 32768
 F1 = 75.476807 MHz
 F2 = 1.000000 MHz
 SW1 = 22727.27 Hz



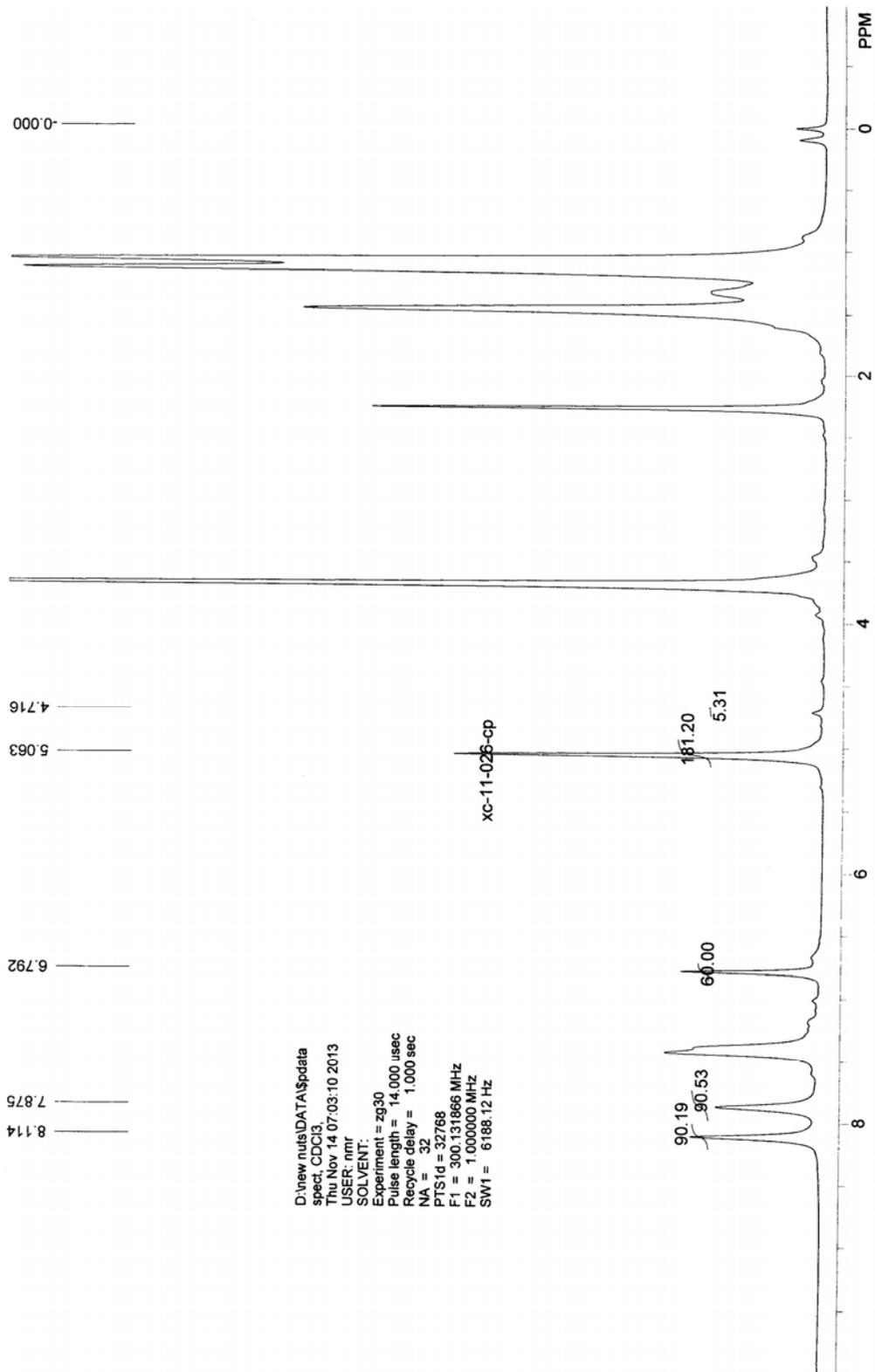
xc-10-161-2 C

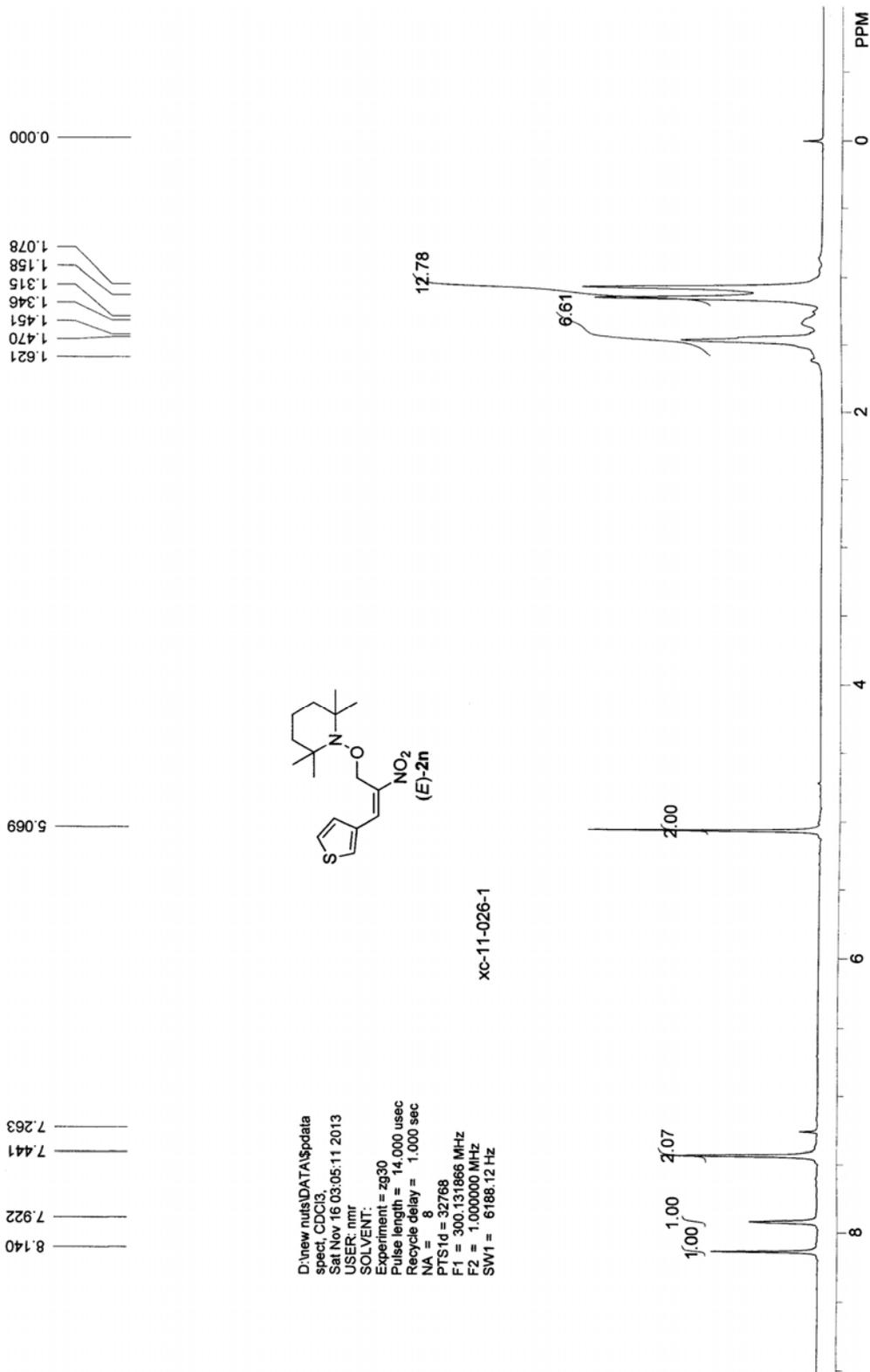


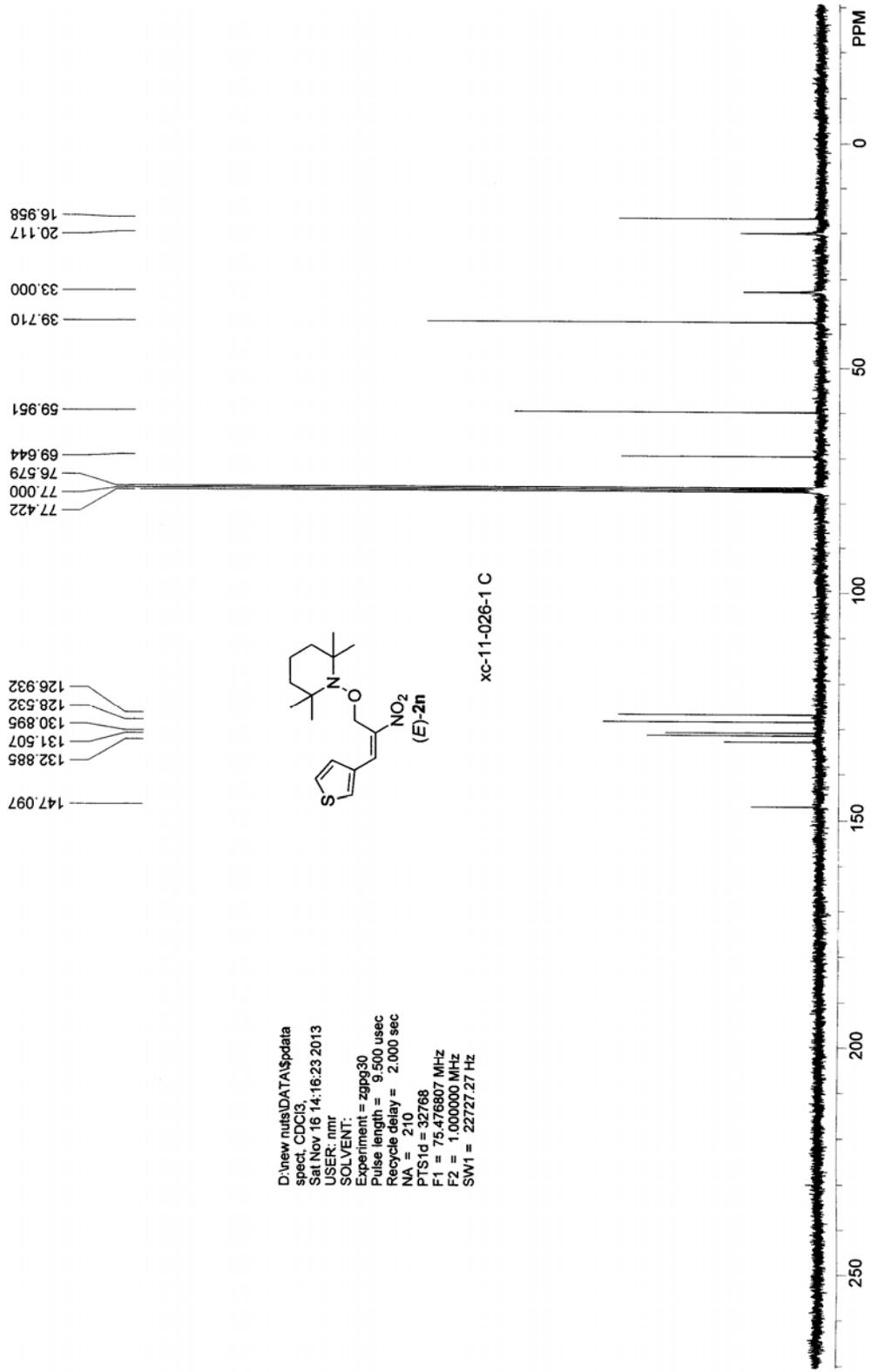
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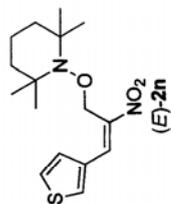




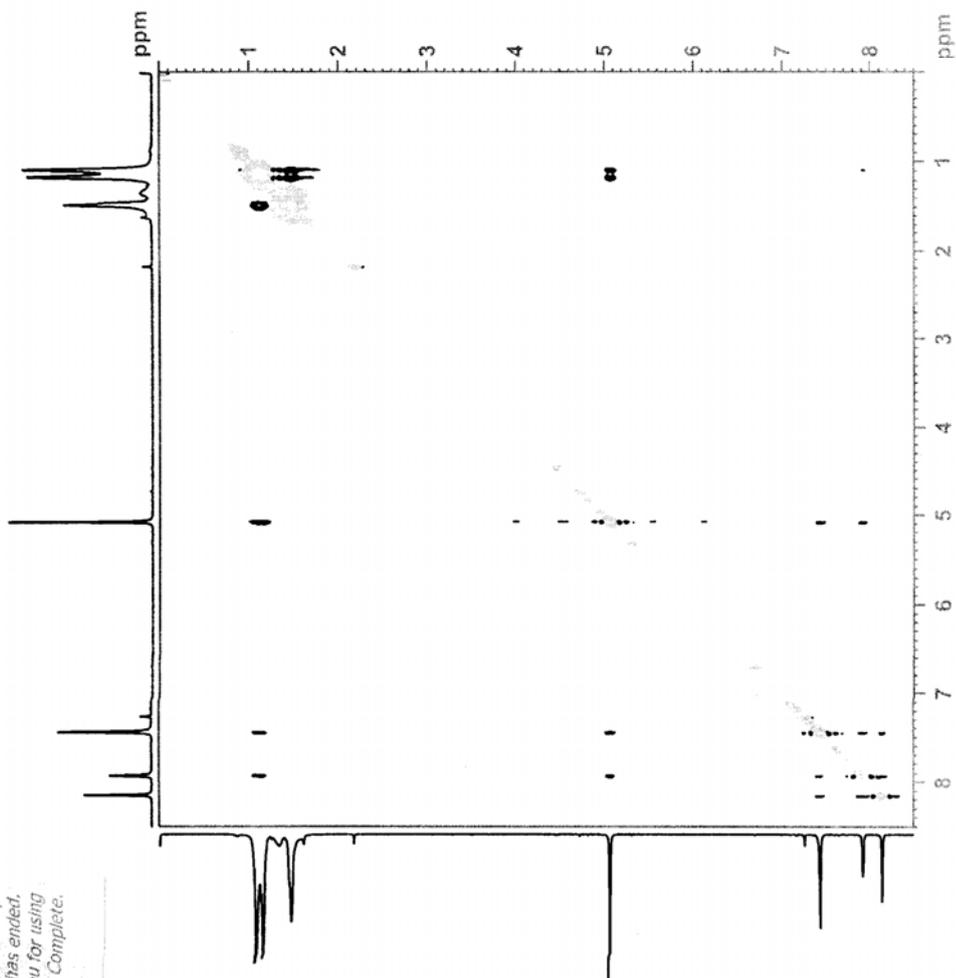


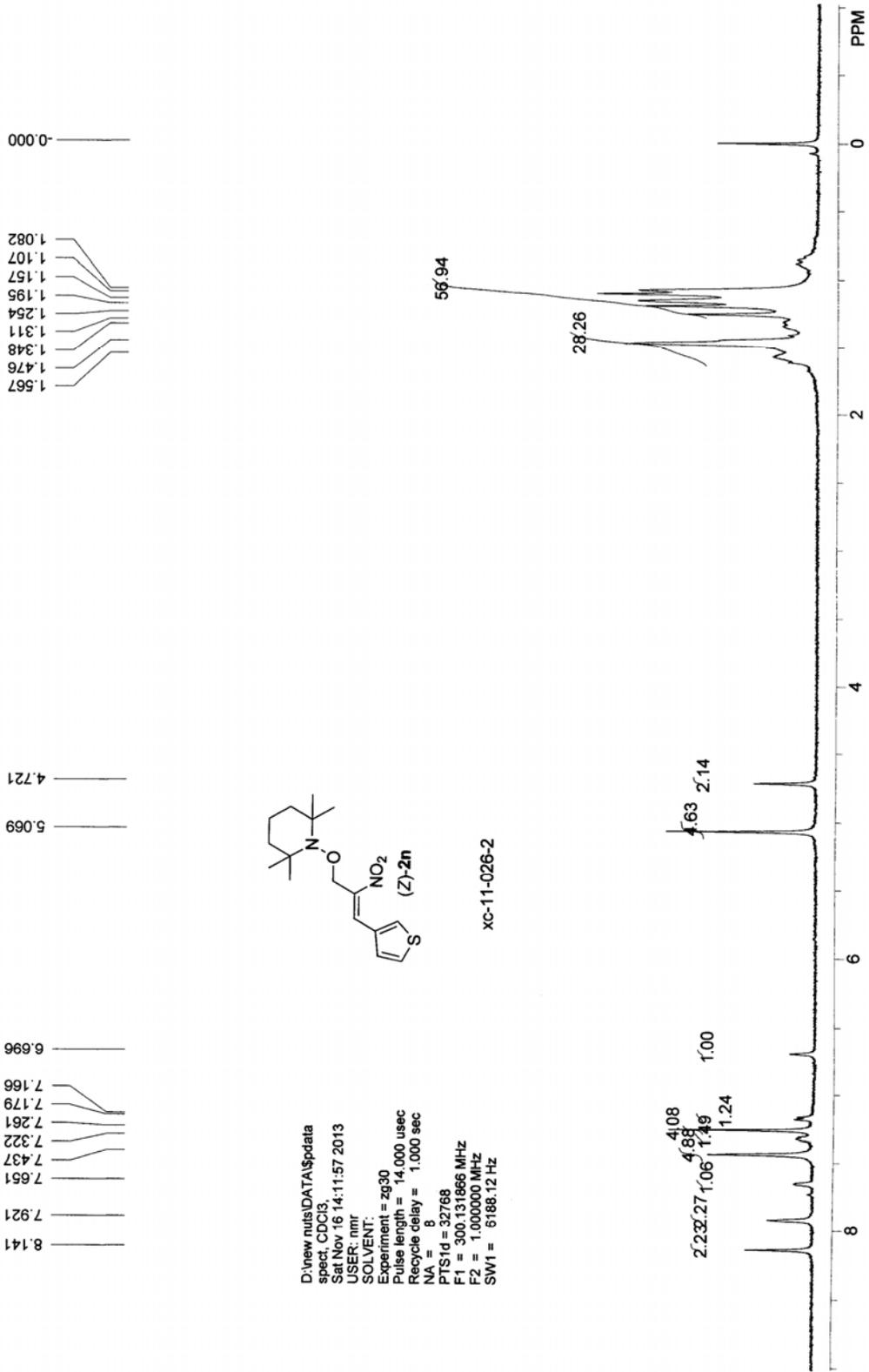
D:\new nuts\DATA\data
spect, CDCI3,
Sat Nov 16 14:16:23 2013
USER: nmr
SOLVENT:
Experiment = zppg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 210
PTS1d = 32768
F1 = 75.476807 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz

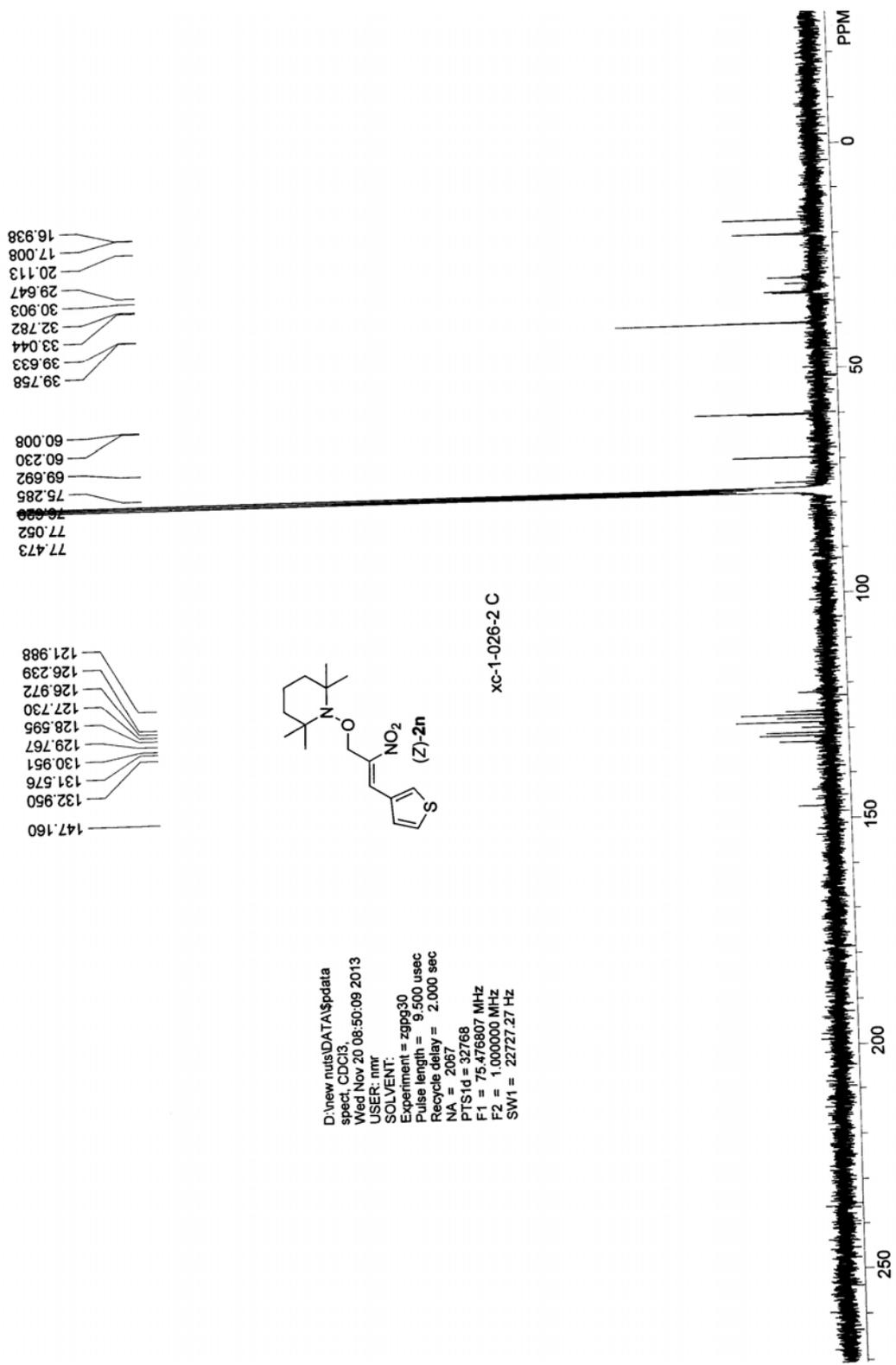
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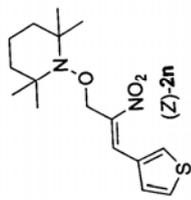
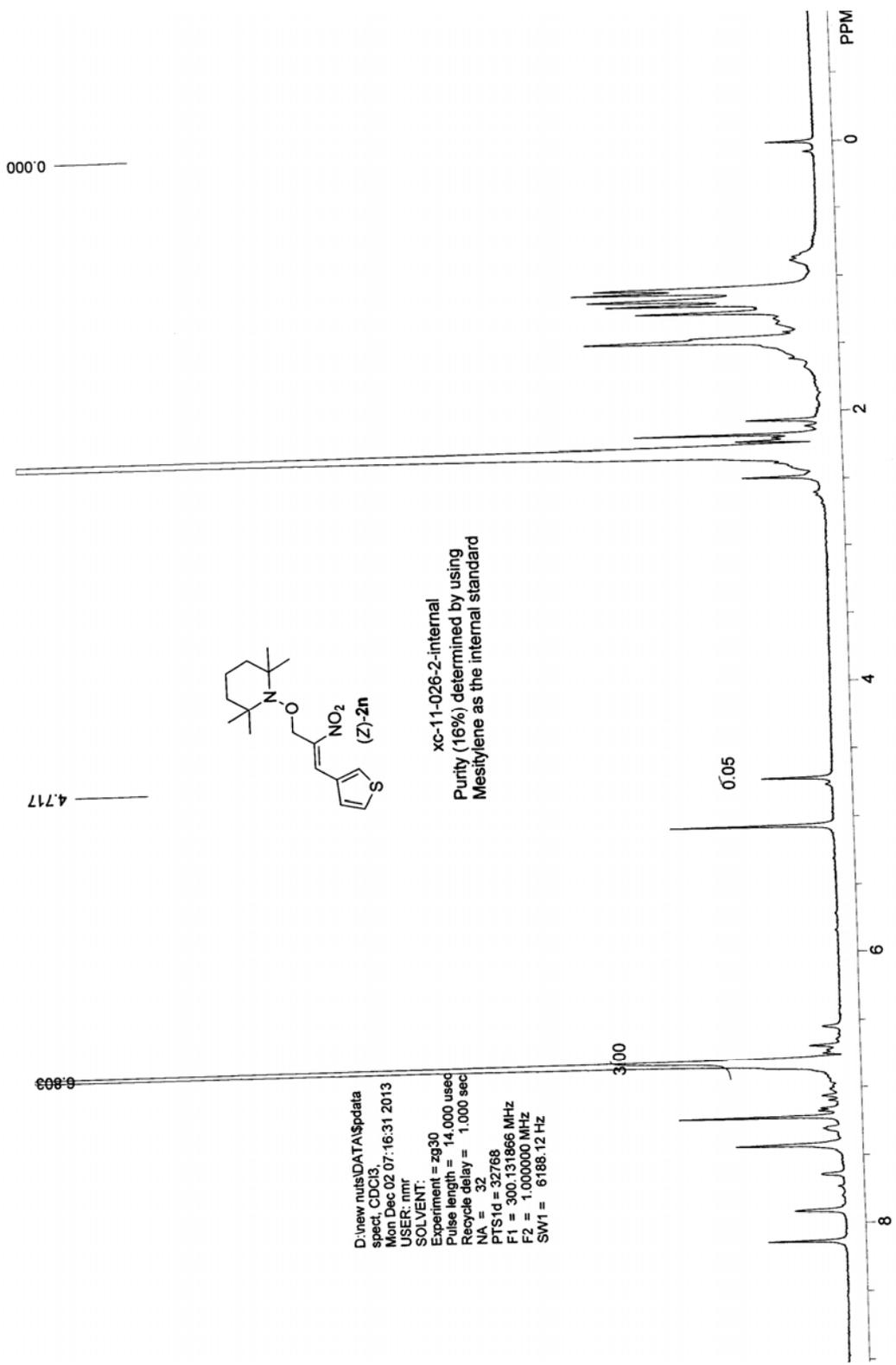


xc-11-026-1-noe





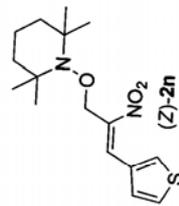




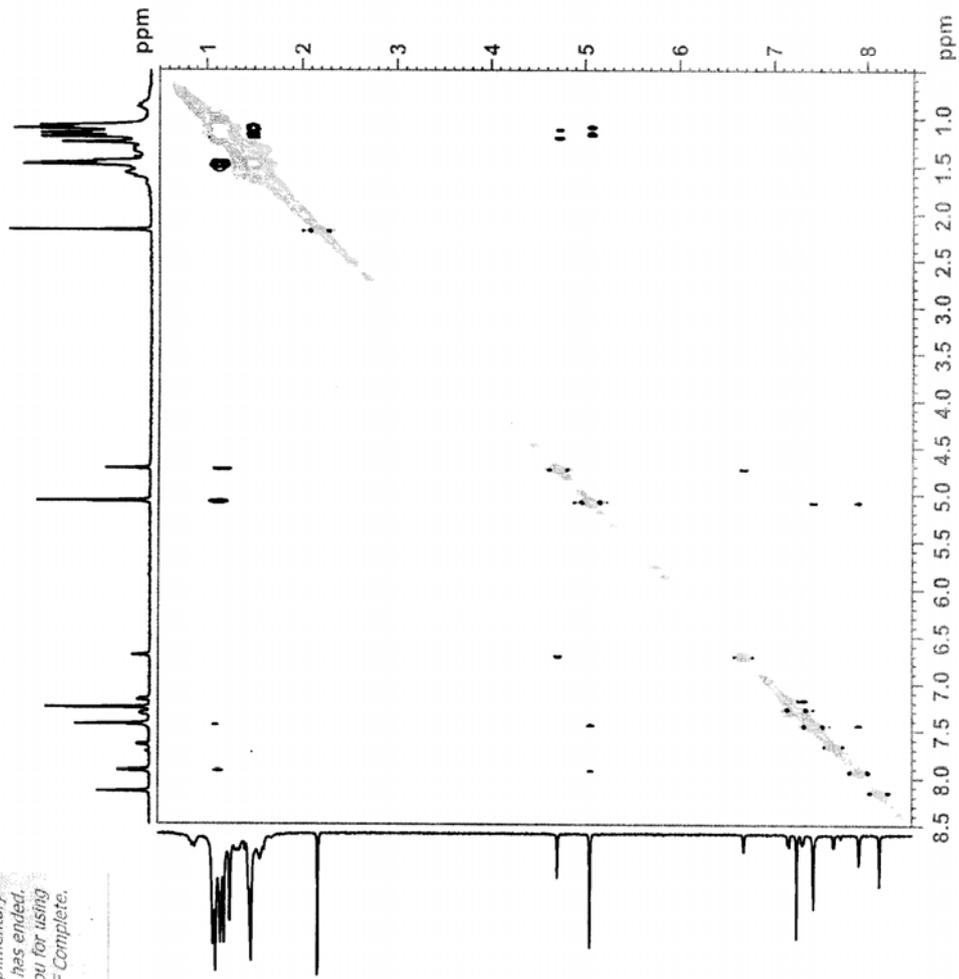
xc-11-026-2-internal
 Purity (16%) determined by using
 Mesitylene as the internal standard

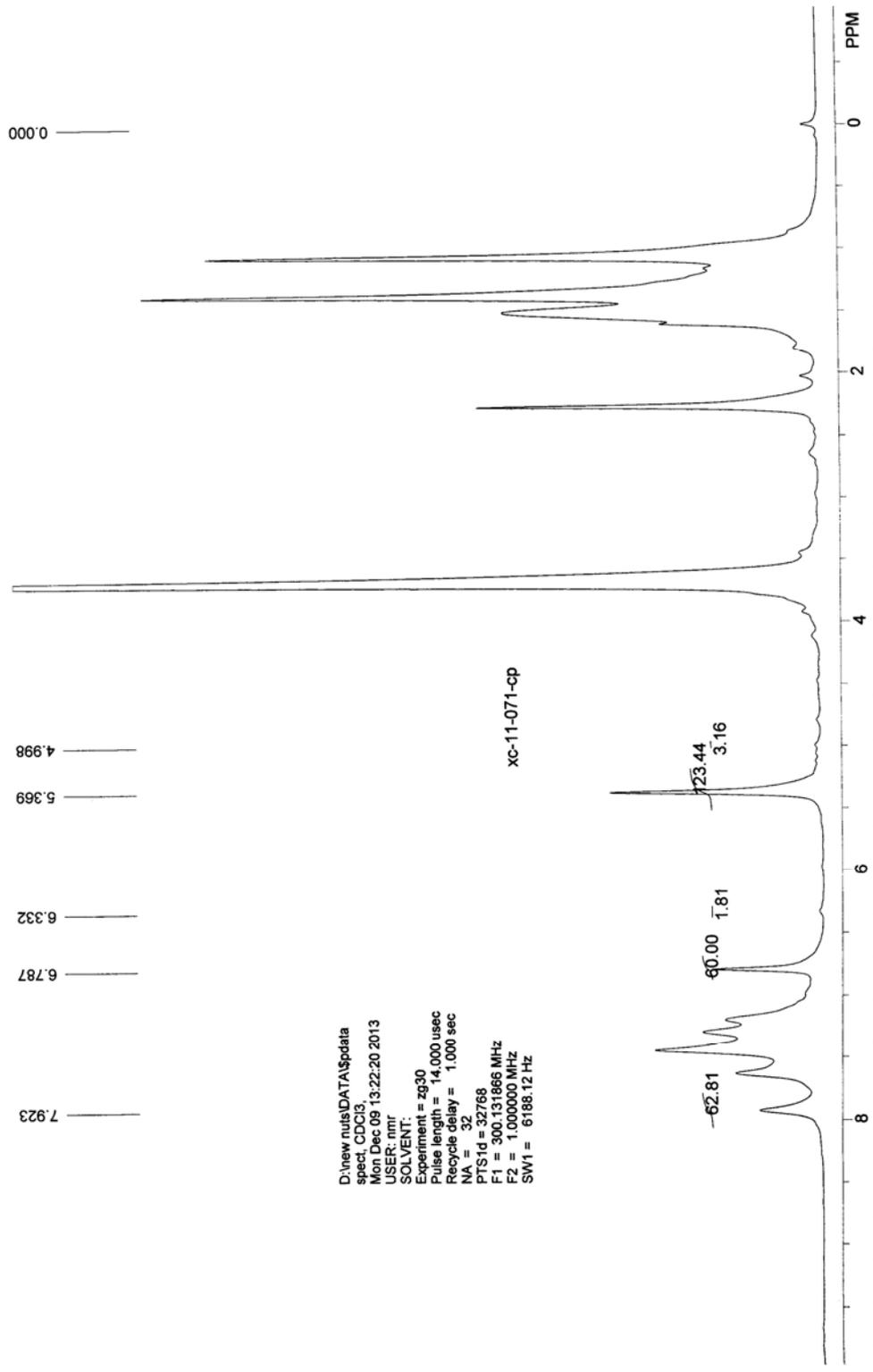
D:\new nuts\DATA\Spdata
 spect, CDC18,
 Mon Dec 02 07:16:31 2013
 USER: nmr
 SOLVENT:
 Experiment = zg30
 Pulse length = 14.000 usec
 Recycle delay = 1.000 sec
 NA = 32
 P1 = 32768
 F1 = 300.131866 MHz
 F2 = 1.000000 MHz
 SW1 = 6188.12 Hz

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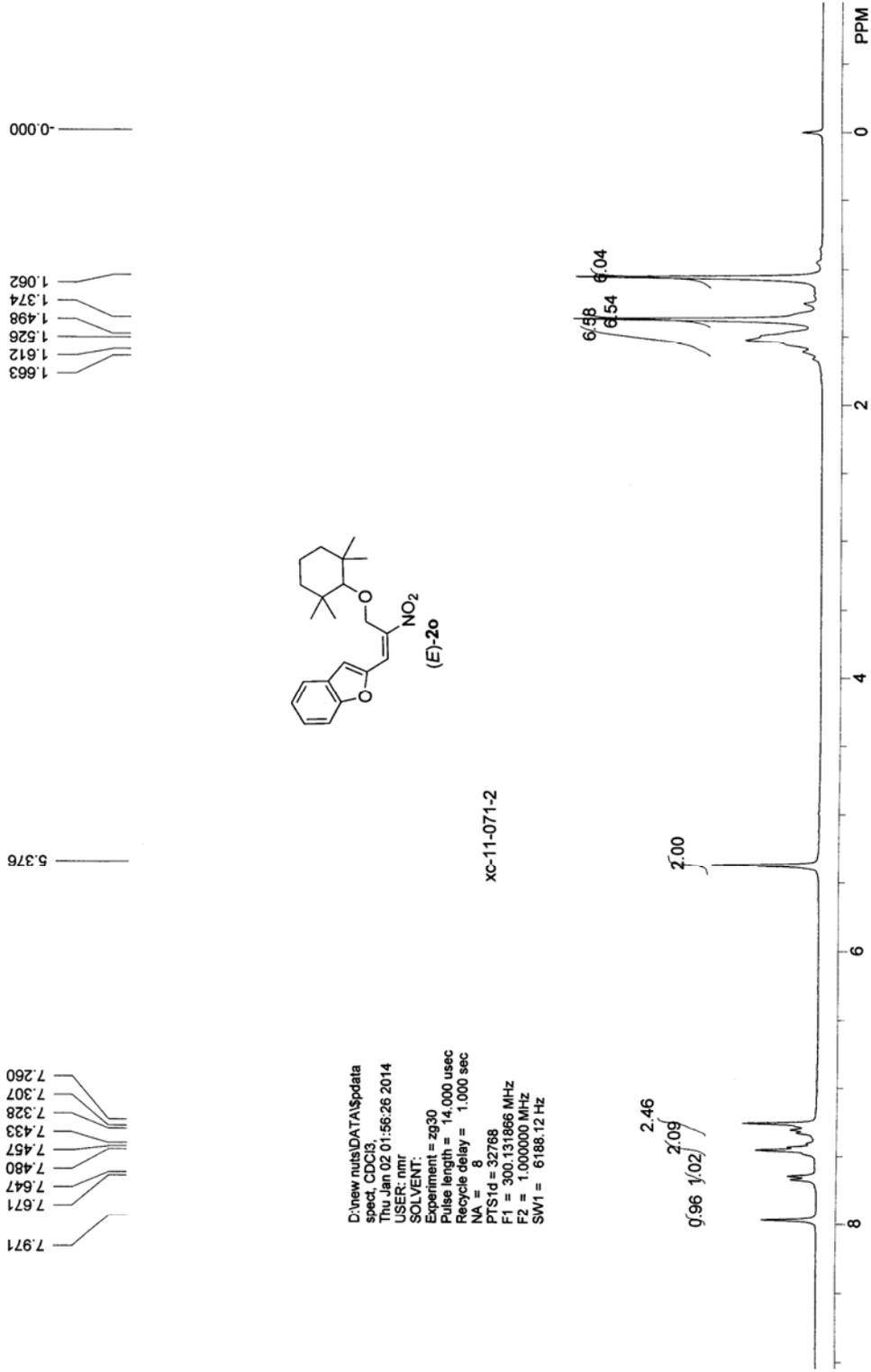
xc-11-026-2-noe

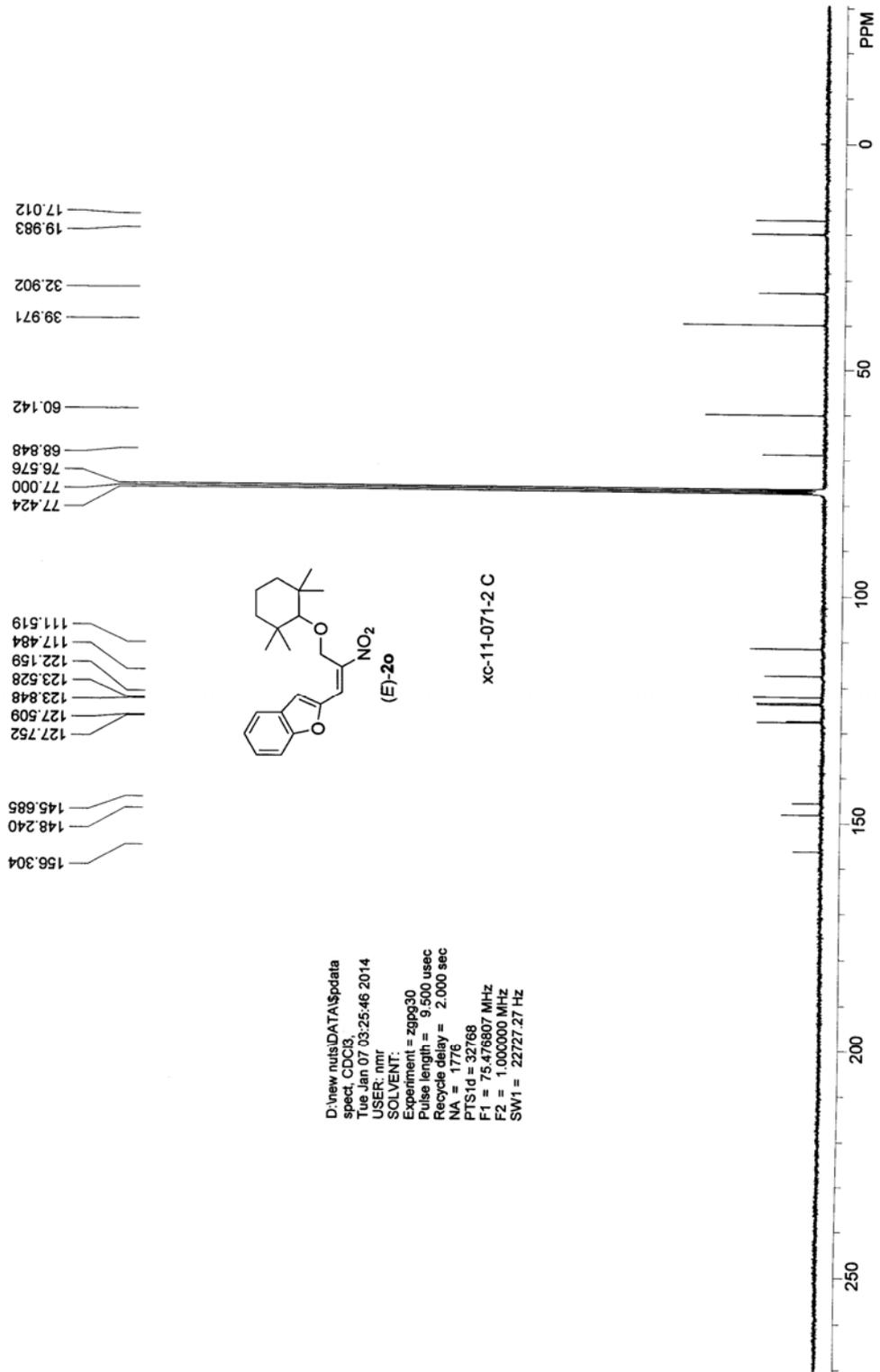




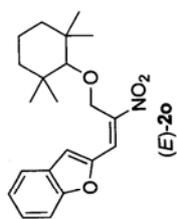
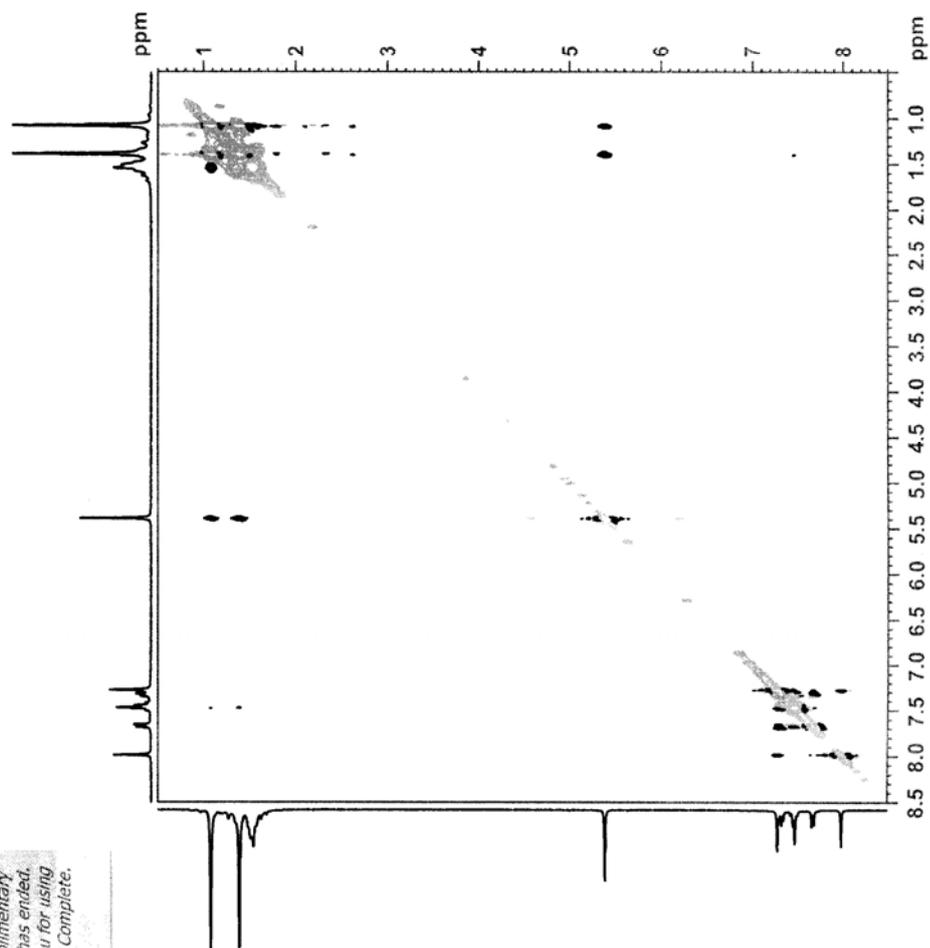
D:\new nui\DATA\1\data
spect, CDC13,
Mon Dec 09 13:22:20 2013
USER: nmr
SOLVENT:
Experiment = zg30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 32
PTSD1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz

xc-11-071-cp

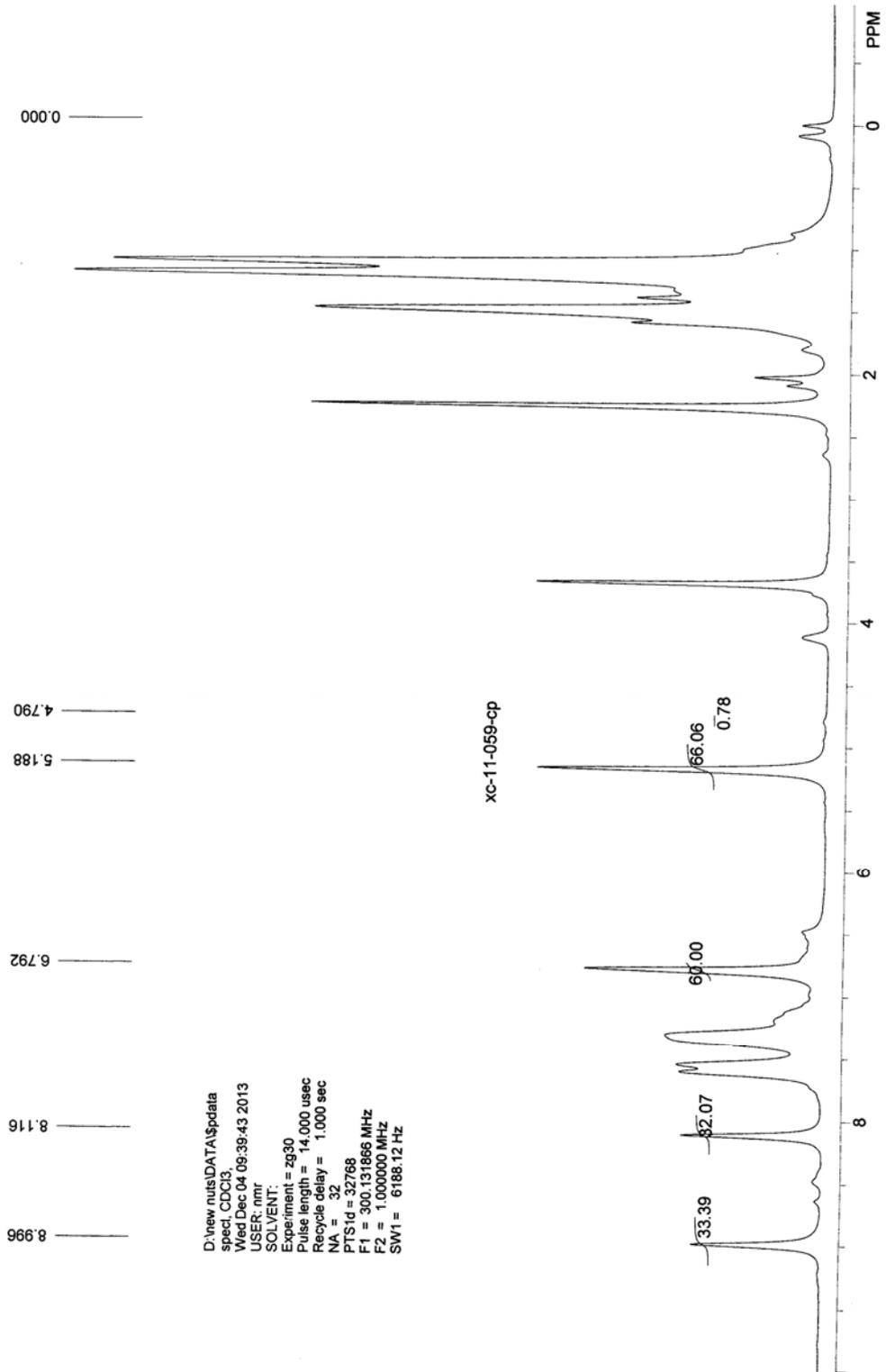




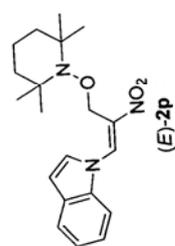
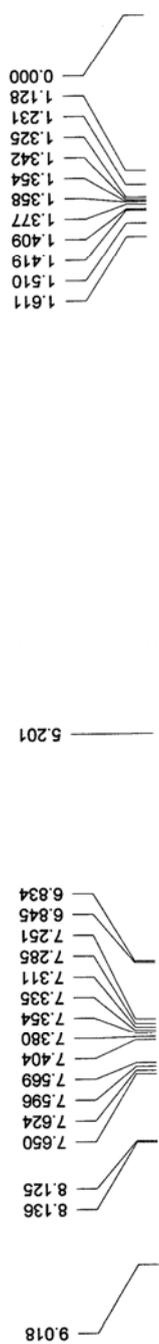
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xc-11-071-2-noe

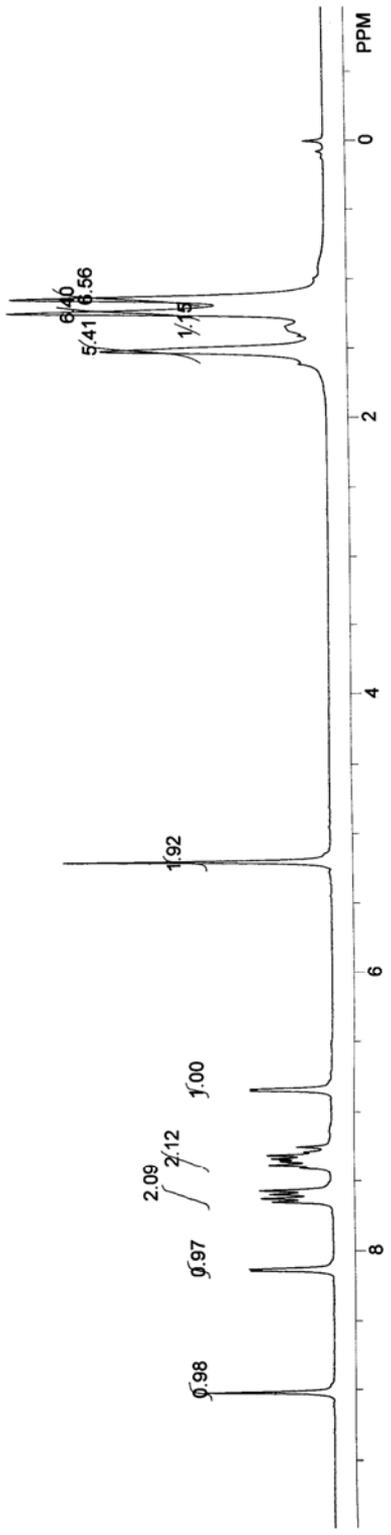


D:\new nuts\DATA\pdata
 spec1, CDC13,
 Wed Dec 04 09:39:43 2013
 USER: nmr
 SOLVENT:
 Experiment = z930
 Pulse length = 14,000 usec
 Recycle delay = 1,000 sec
 NA = 32
 FTSId = 32768
 F1 = 300.131866 MHz
 F2 = 1,000000 MHz
 SW1 = 6188.12 Hz



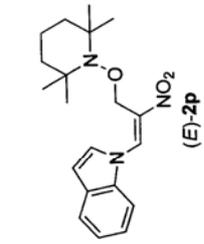
D:\new\nuis\DATA\spdata
 spectr_CDC18
 Thu Jan 02 10:46:28 2014
 USER: nmr
 SOLVENT:
 Experiment = zg30
 Pulse length = 14.000 usec
 Recycle delay = 1.000 sec
 NA = 8
 PTS1d = 32768
 F1 = 300.131866 MHz
 F2 = 1.000000 MHz
 SWH = 6188.12 Hz

xc-11-059-1

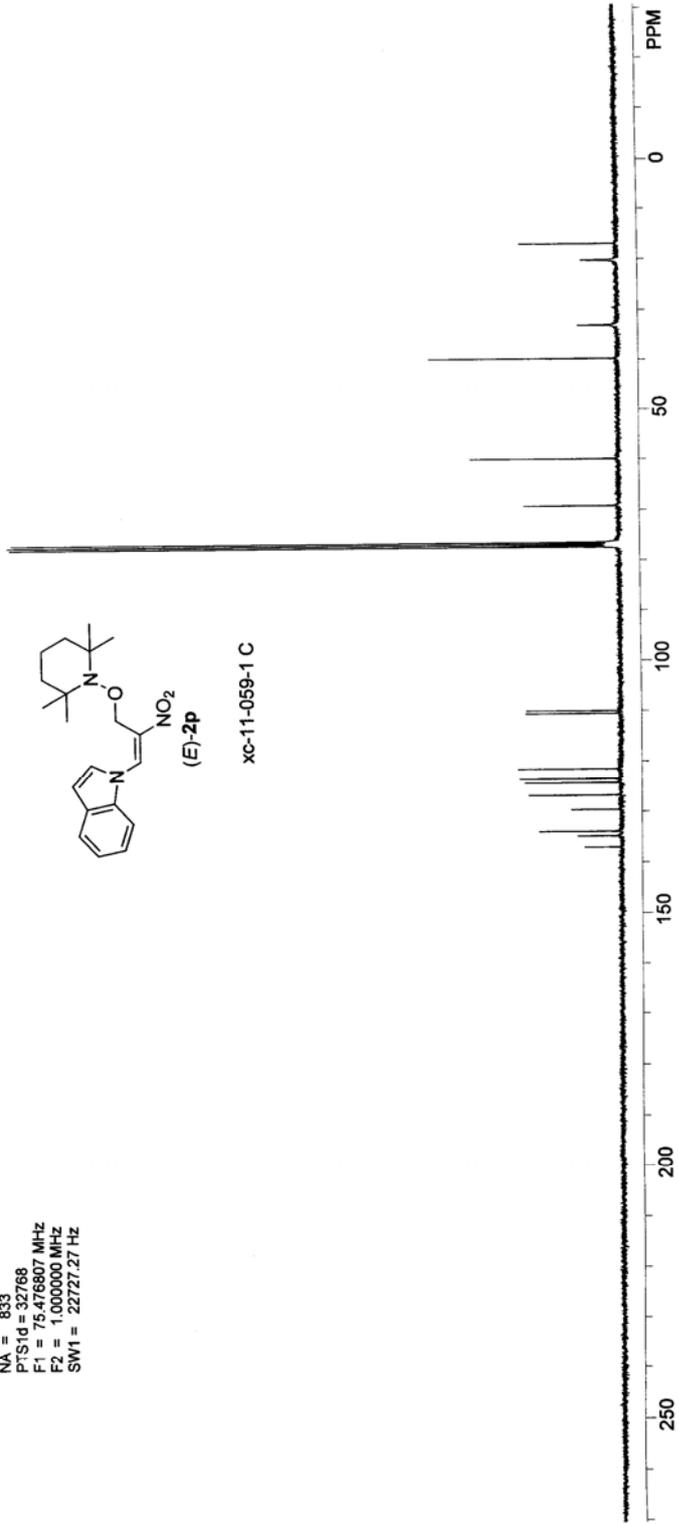


D:\new nuts\DATA\data
 spect, CDCI3,
 Thu Jan 02 10:49:36 2014
 USER: nmr
 SOLVENT:
 Experiment = zqpg30
 Pulse length = 9.500 usec
 Recycle delay = 2.000 sec
 NA = 833
 P1S1d = 32768
 F1 = 75.476807 MHz
 F2 = 1.000000 MHz
 SW1 = 22727.27 Hz

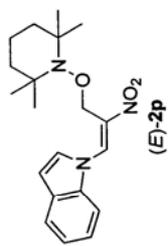
137.091
 134.888
 133.960
 129.662
 126.765
 124.265
 123.534
 121.541
 110.591
 110.056
 77.424
 77.000
 76.569
 69.281
 60.005
 39.757
 33.143
 20.217
 16.923



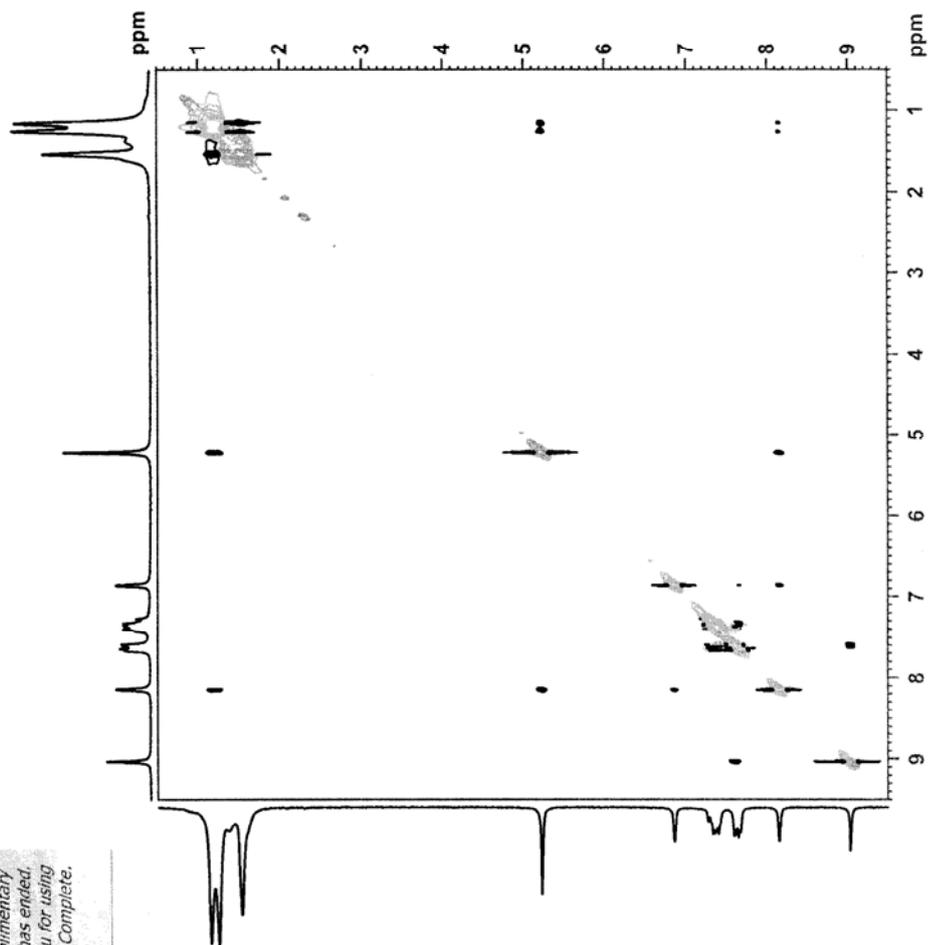
xc-11-059-1 C

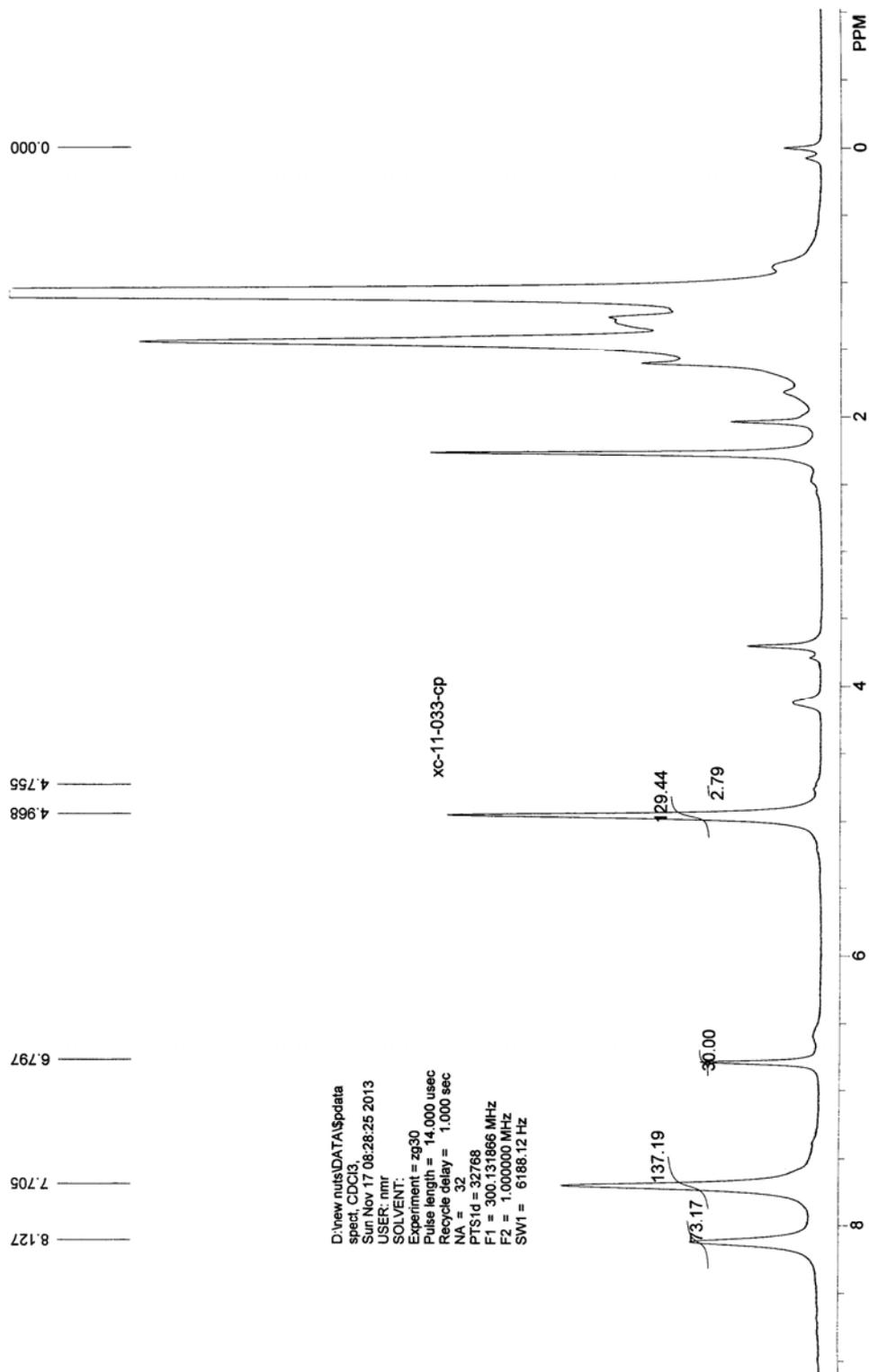


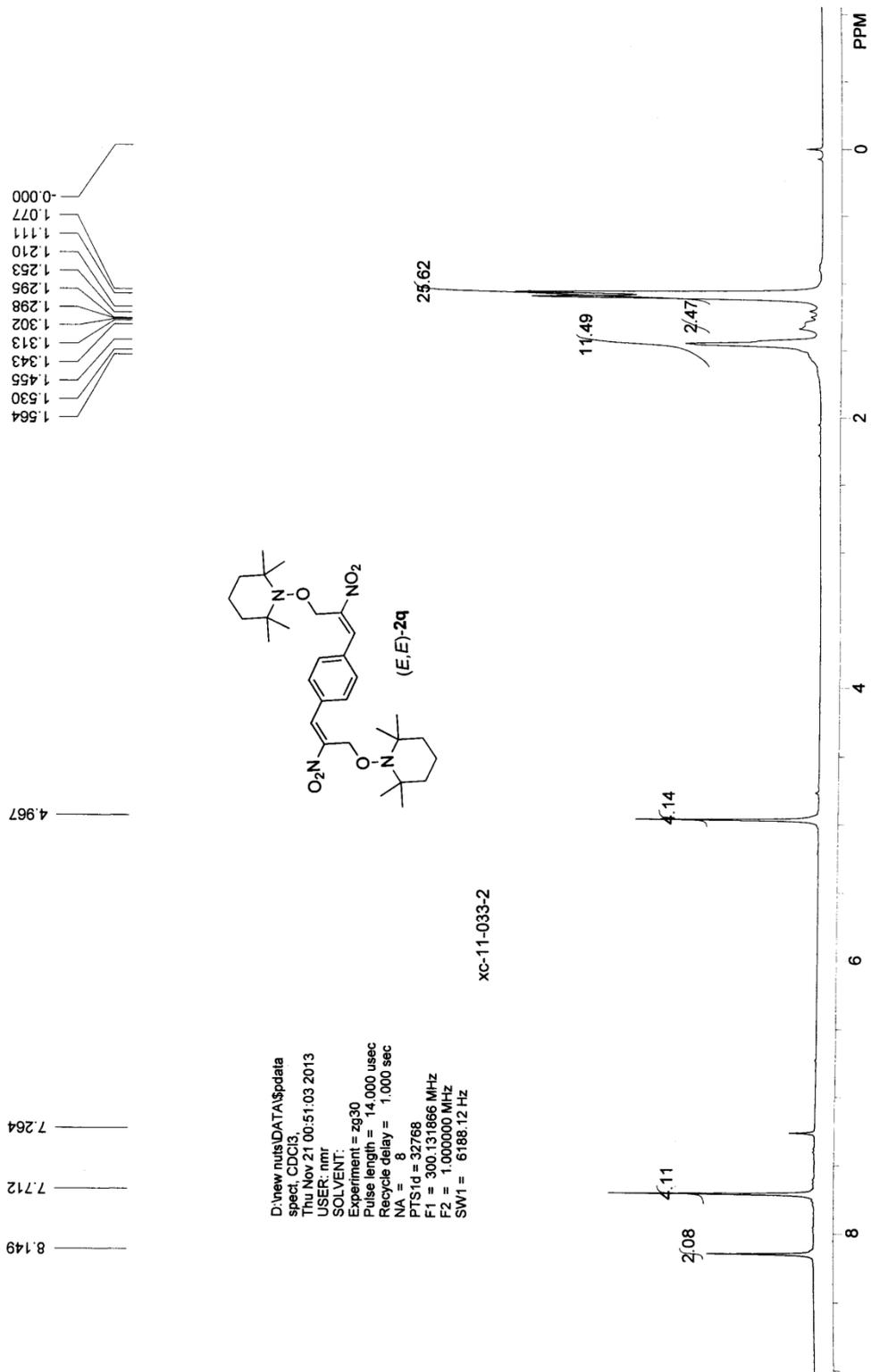
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xc-11-057-1-noe

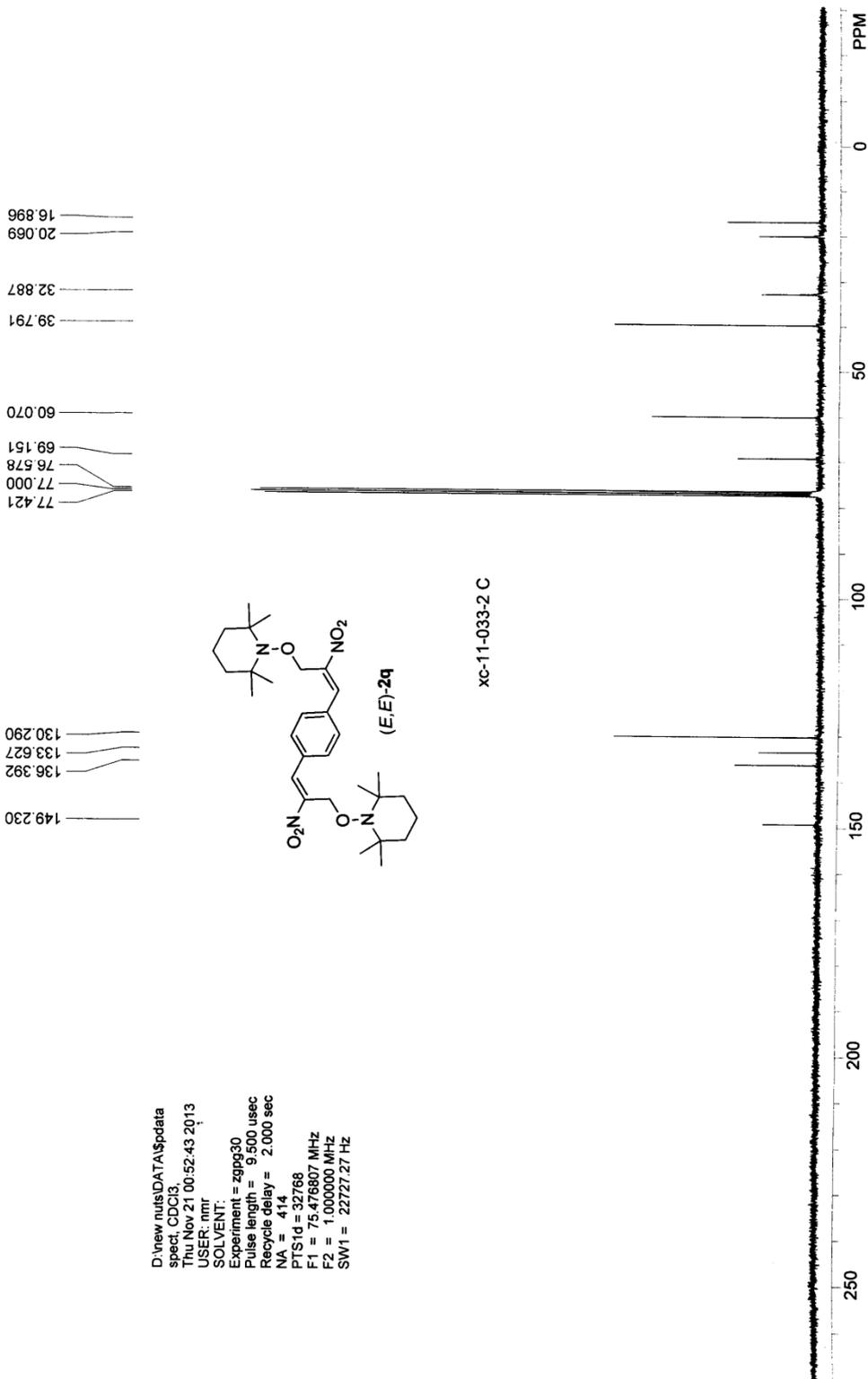
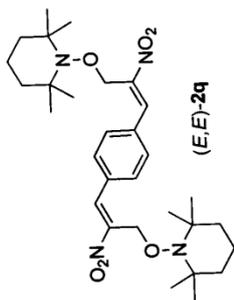


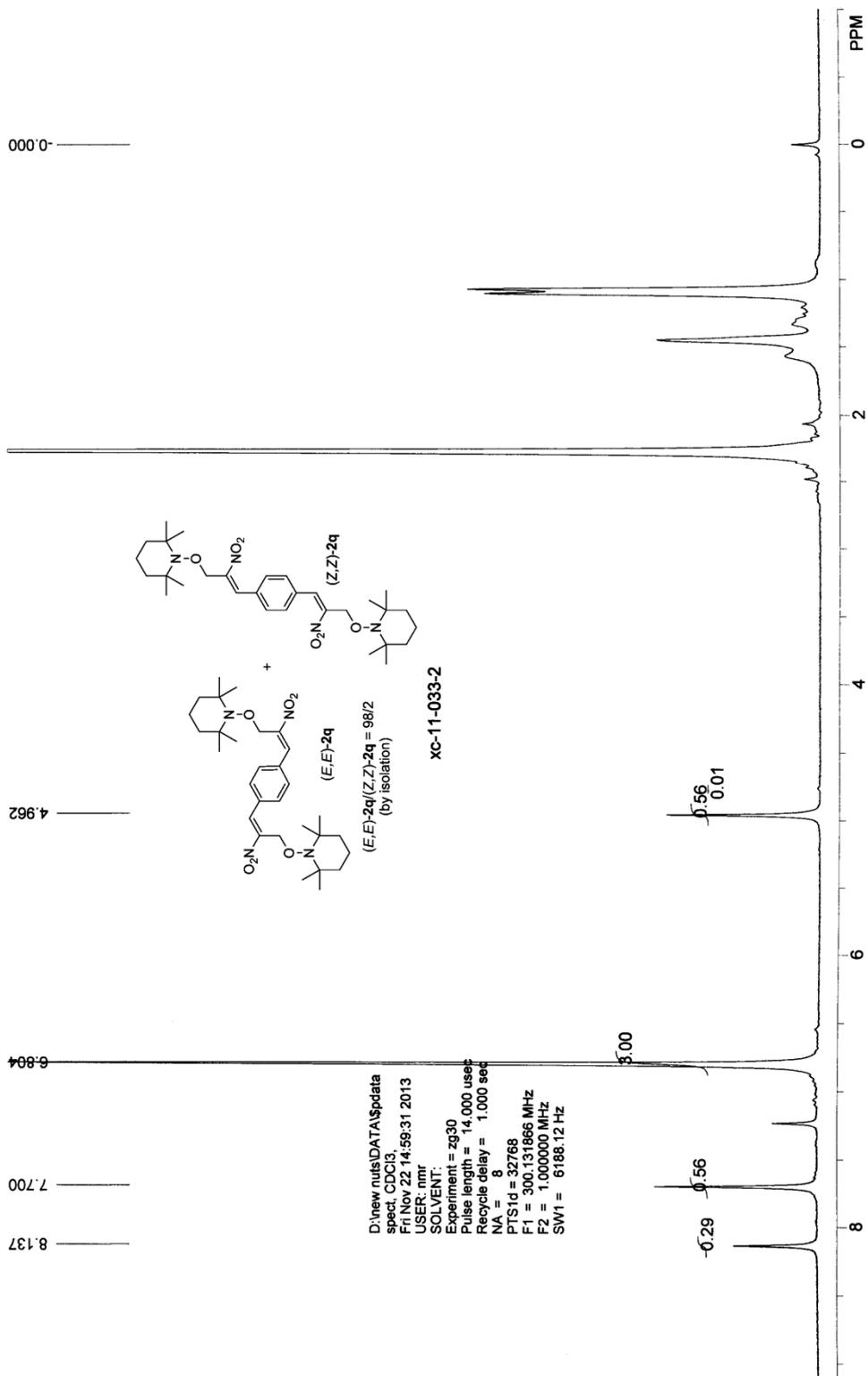




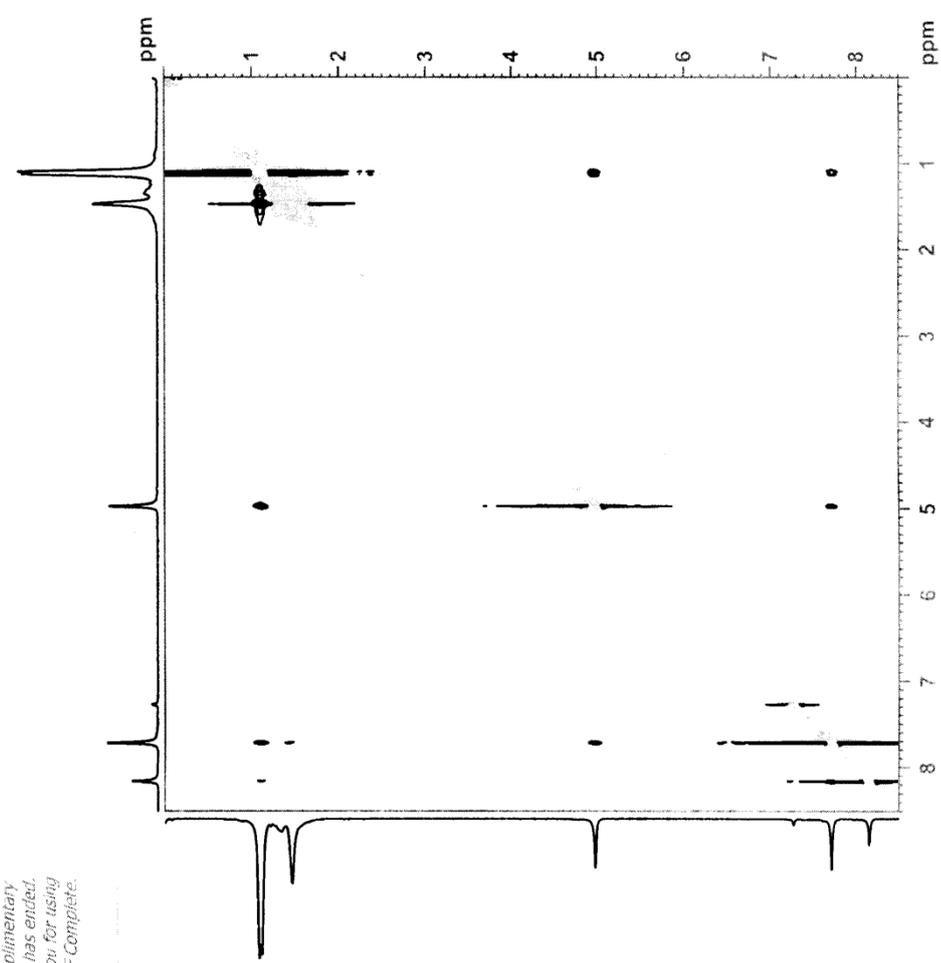
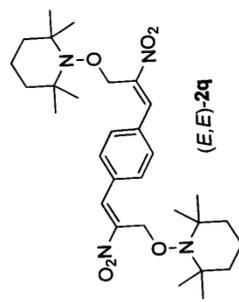
D:\new nuts\DATA\spdata
 spect, CDCI3,
 Thu Nov 21 00:51:03 2013
 USER: nmr
 SOLVENT: nmr
 Experiment = zg30
 Pulse length = 14.000 usec
 Recycle delay = 1.000 sec
 NA = 8
 P1 = 32768
 F1 = 300.131866 MHz
 F2 = 1.000000 MHz
 SW1 = 6188.12 Hz

D:\new nulis\DATA\spdata
 spect, CDCI3,
 Thu Nov 21 00:52:43 2013
 USER: nmr
 SOLVENT:
 Experiment = zpg630
 Pulse length = 9.500 usec
 Recycle delay = 2.000 sec
 NA = 414
 PTS1d = 32768
 F1 = 75.476807 MHz
 F2 = 1.000000 MHz
 SW1 = 22727.27 Hz

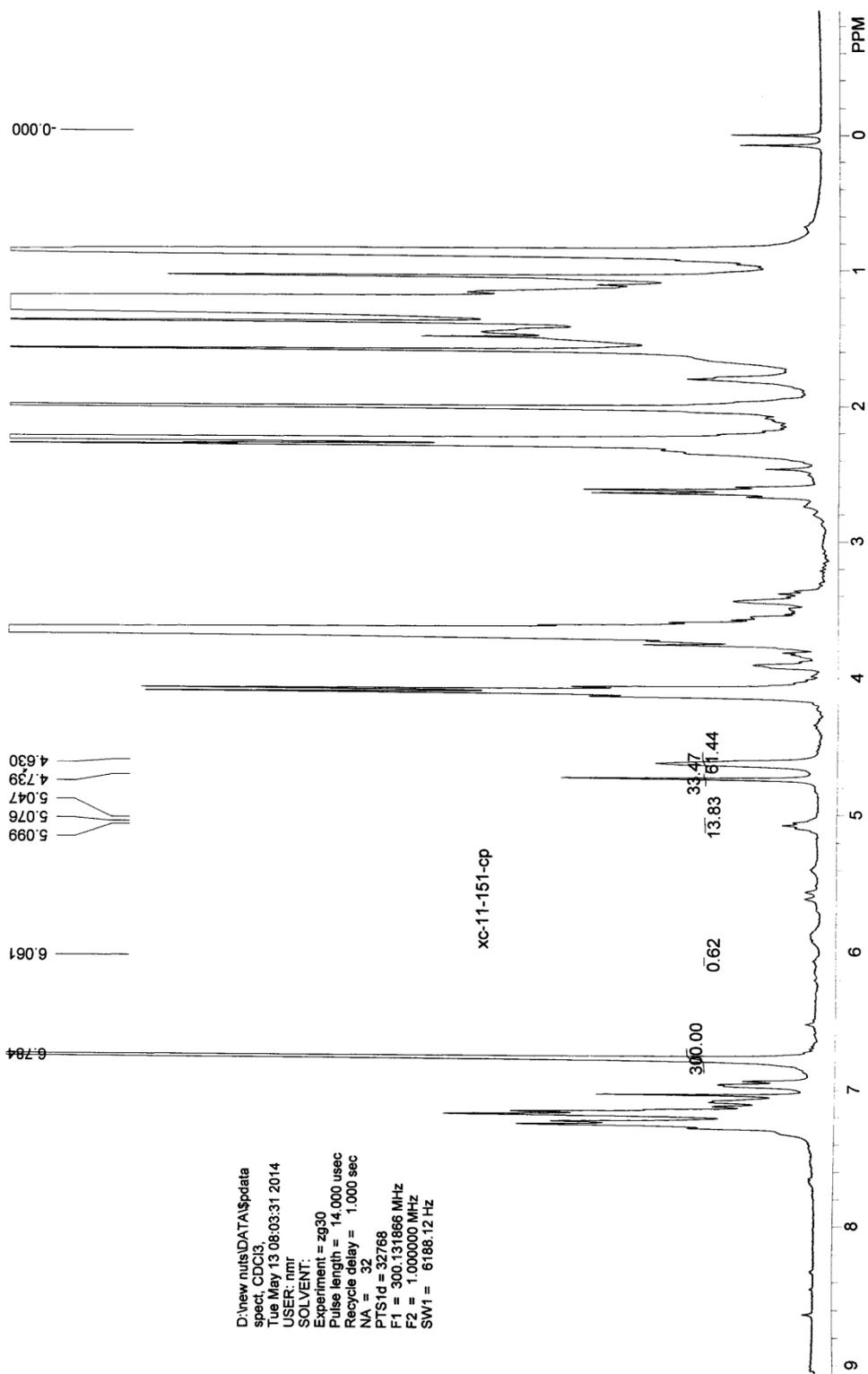




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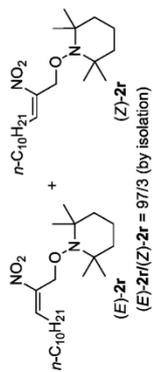


XC-11-033-2-noe

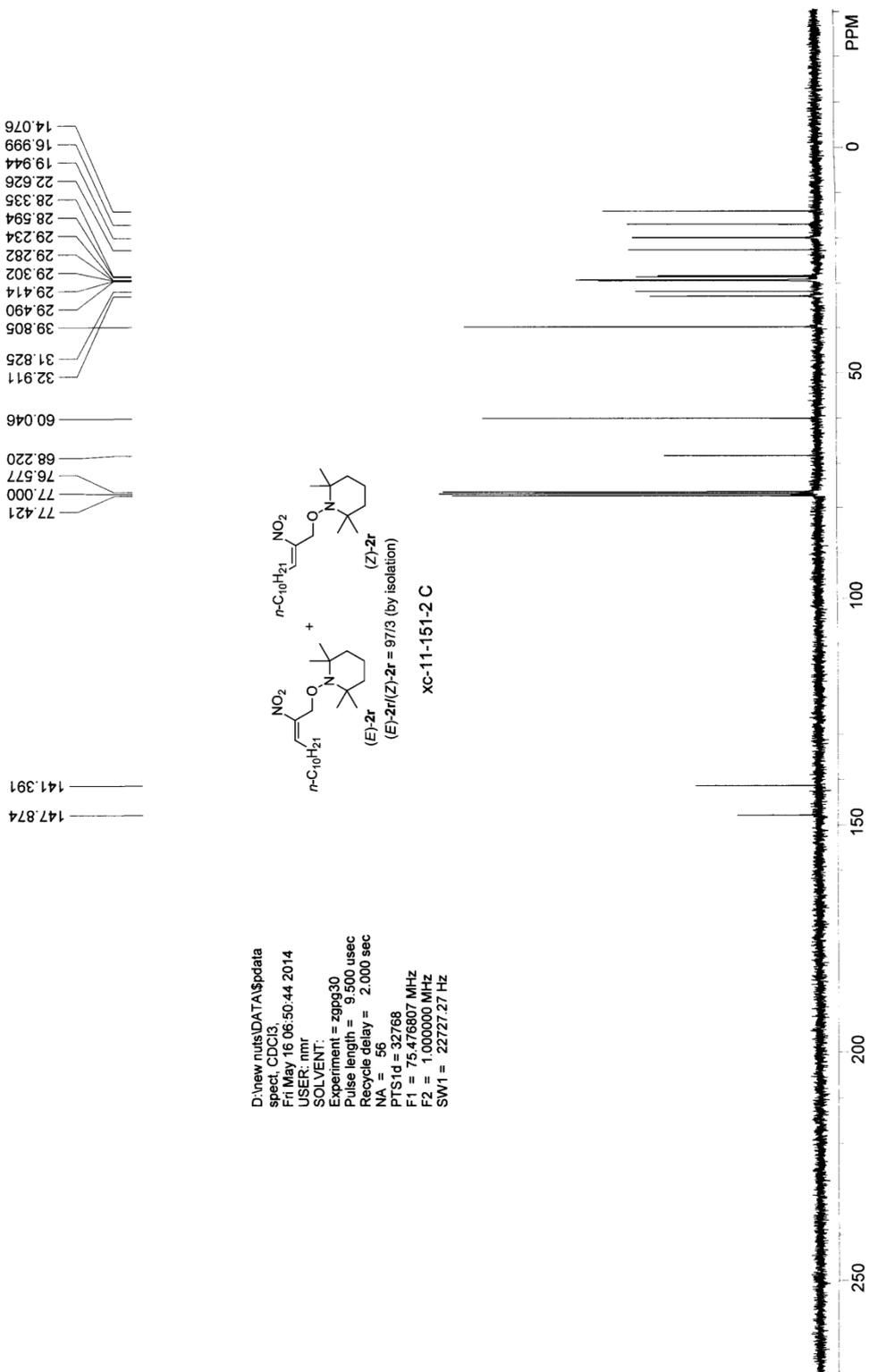


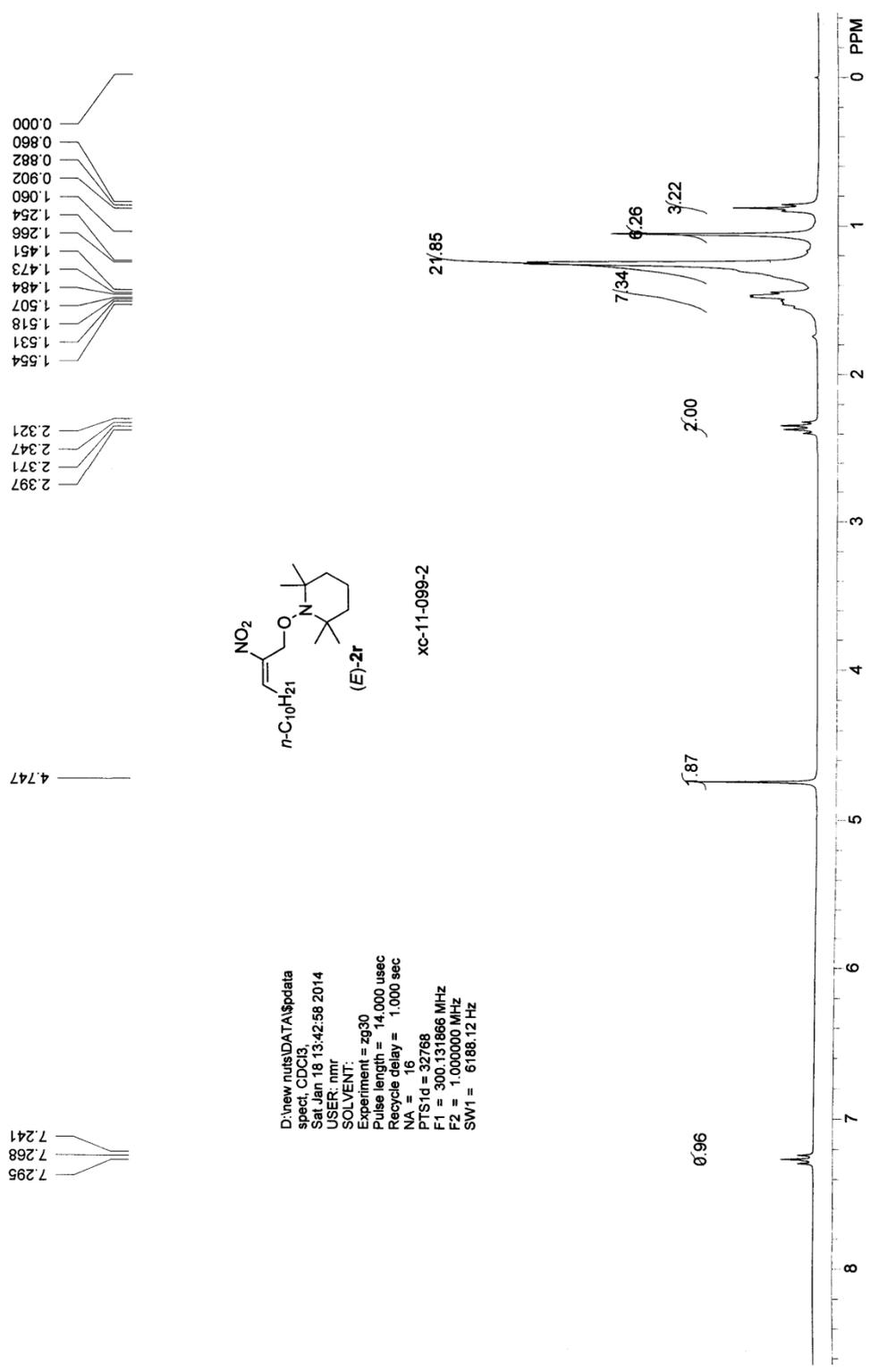
D:\new\nu1\data\spect_data
 spect_CDCl3
 Tue May 13 08:03:31 2014
 USER: nmr
 SOLVENT:
 Experiment = z930
 Pulse length = 14.000 usec
 Recycle delay = 1.000 sec
 NA = 32
 PTS1d = 32768
 F1 = 300.131866 MHz
 F2 = 1.000000 MHz
 SW1 = 6188.12 Hz

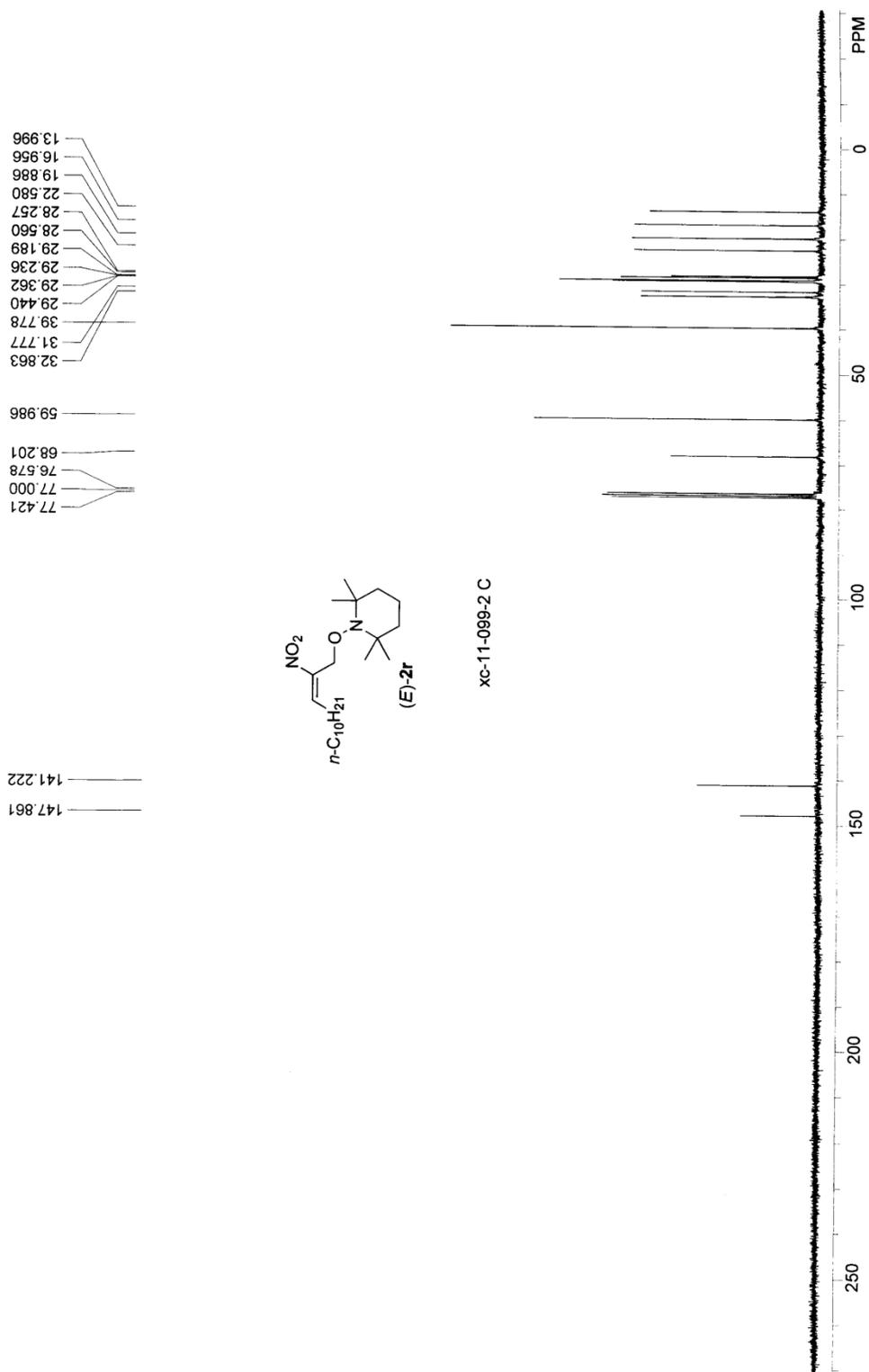
D:\new nuisDATA\spdata
 spect, CDCl3
 Fri May 16 06:50:44 2014
 USER: nmr
 SOLVENT:
 Experiment = zpgg30
 Pulse length = 9.500 usec
 Recycle delay = 2.000 sec
 NA = 56
 P1 = 32768
 F1 = 75.476807 MHz
 F2 = 1,000,000 MHz
 SW1 = 22727.27 Hz



XC-11-151-2 C

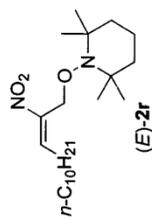
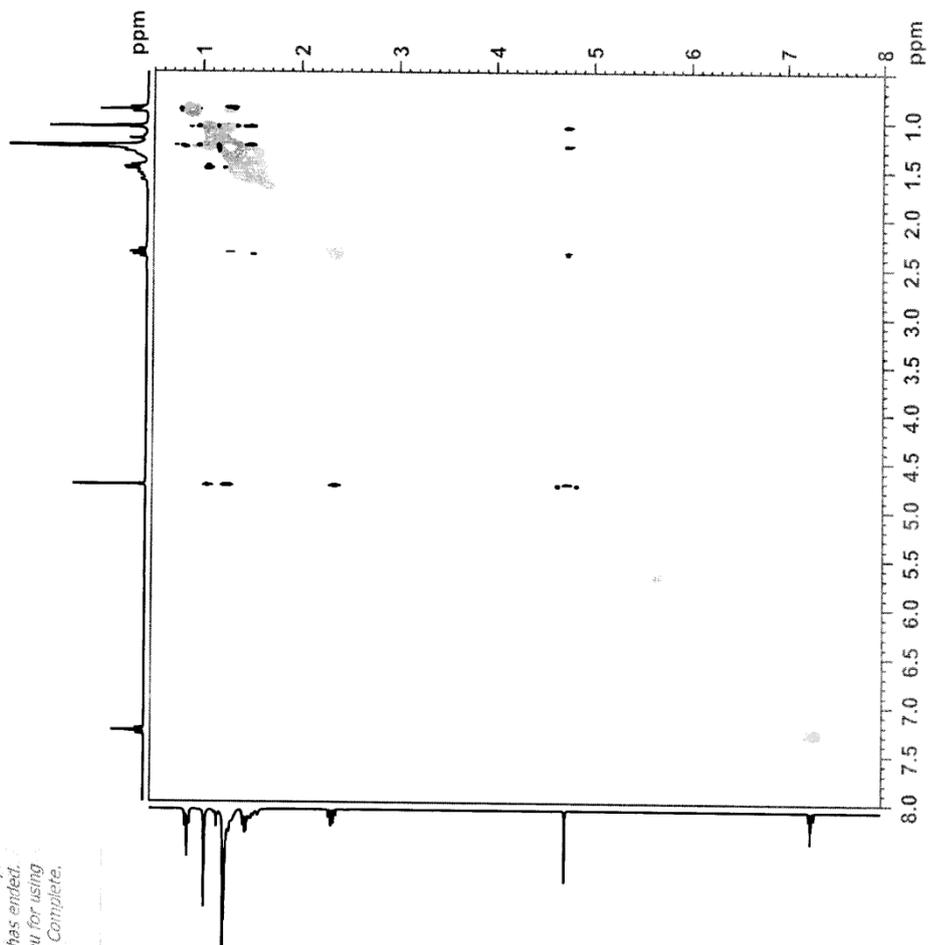




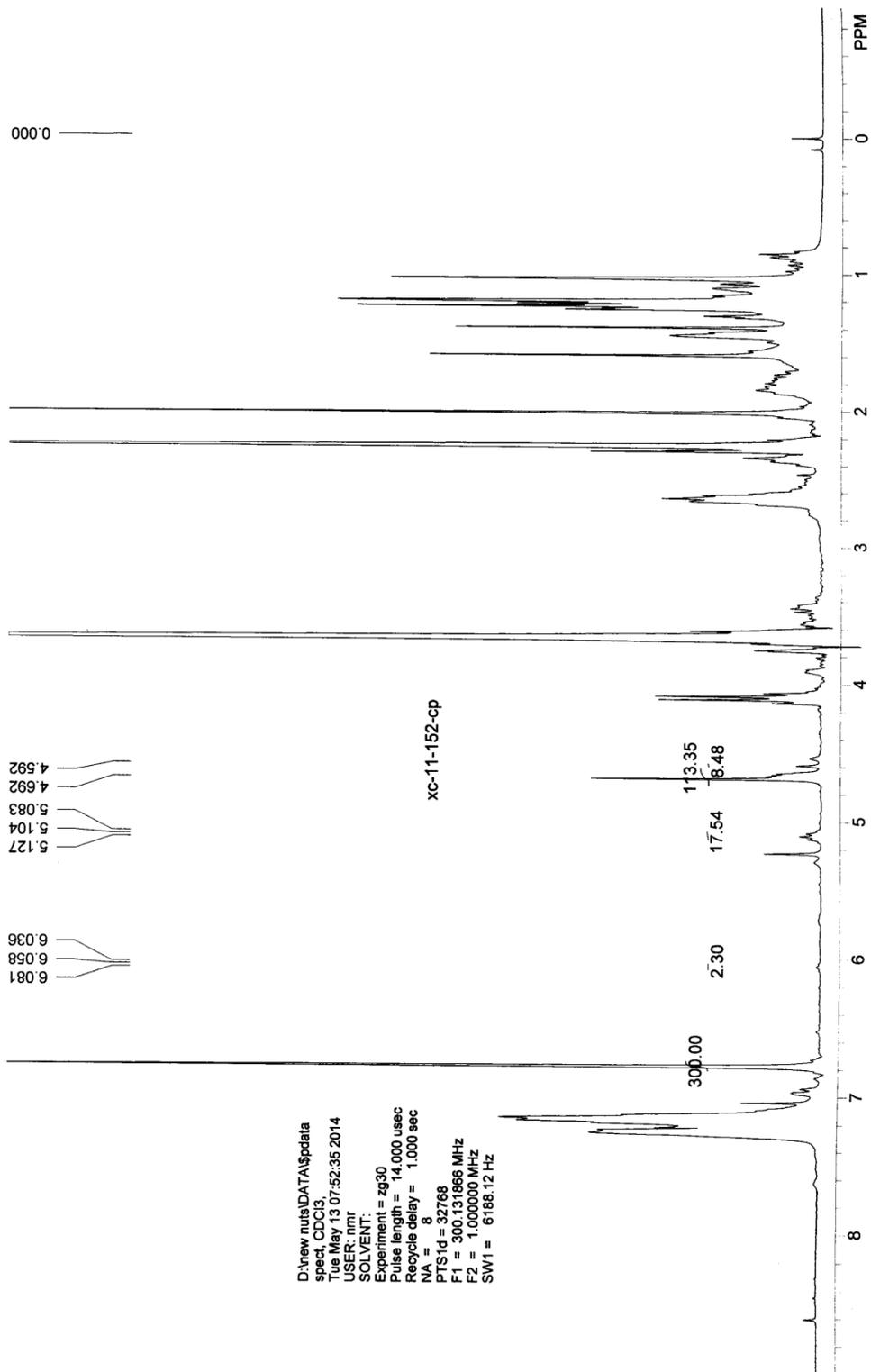


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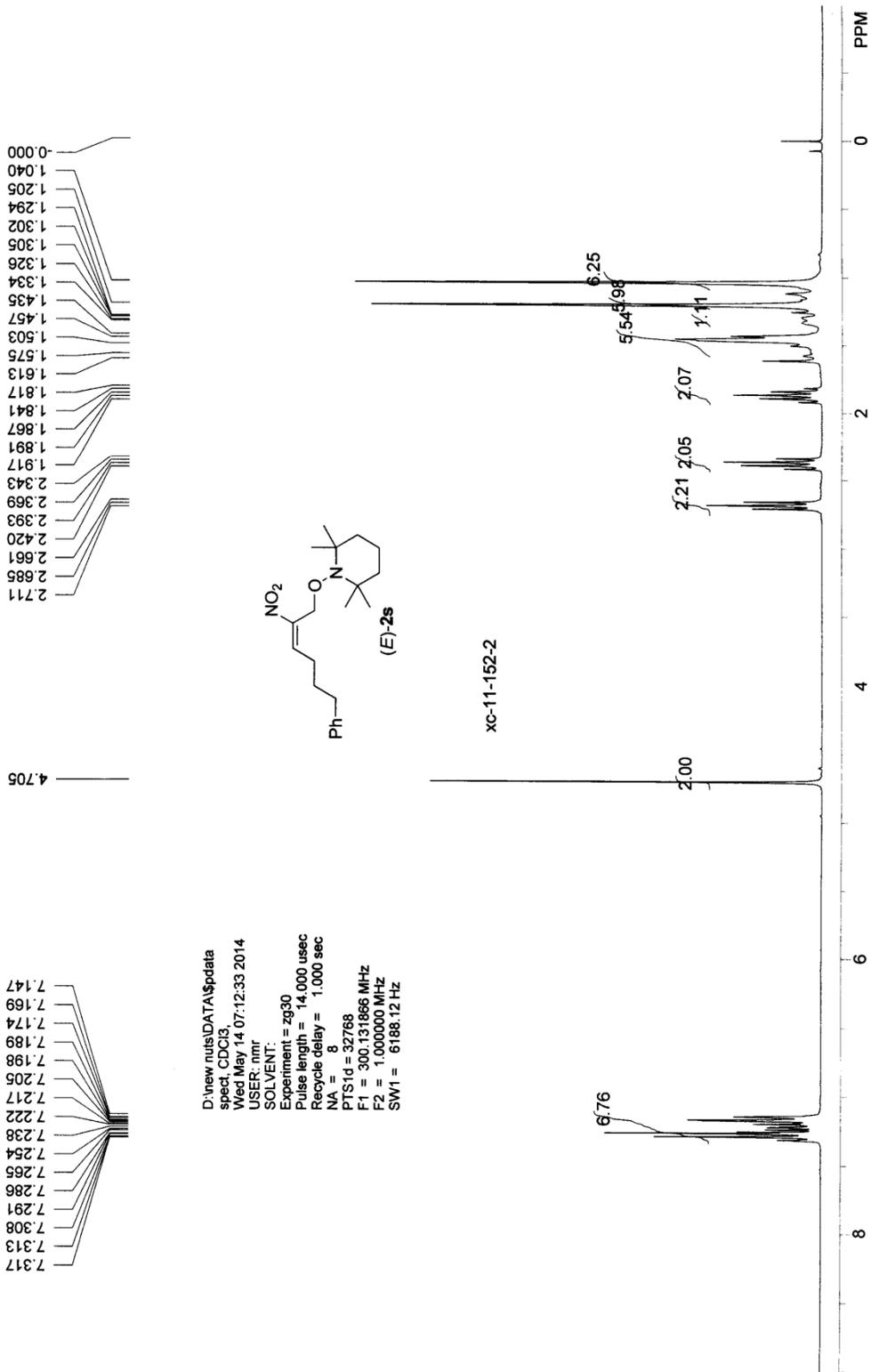
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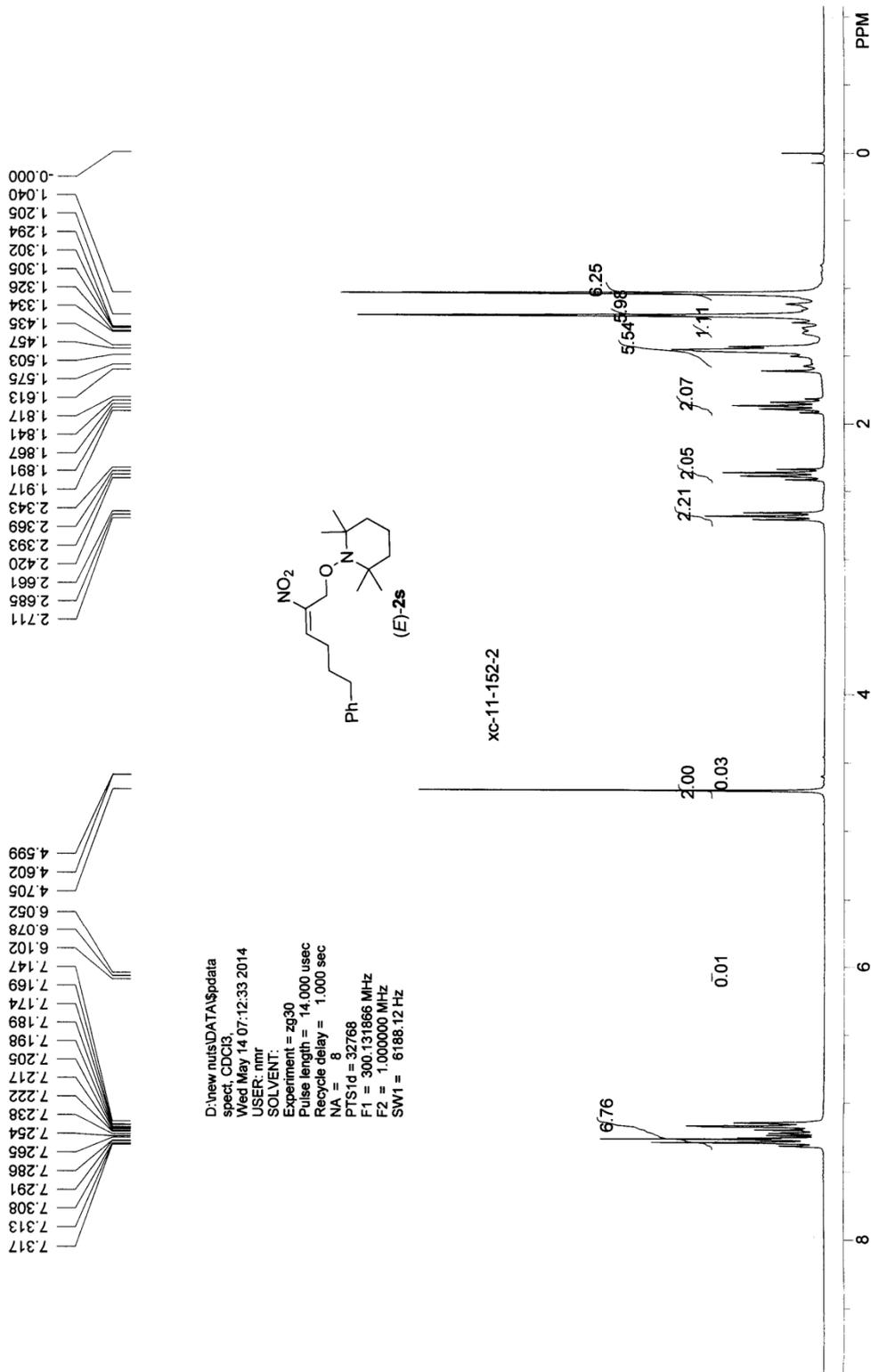
xc-11-099-2-noe



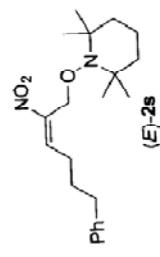
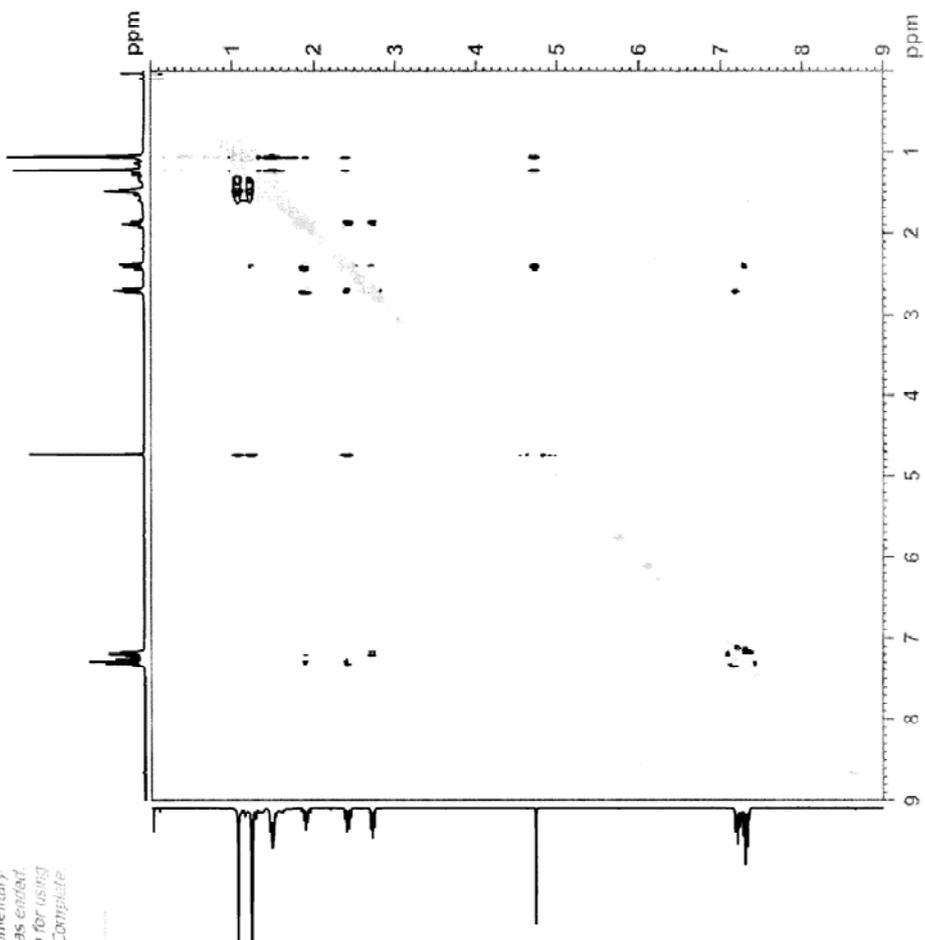
D:\new nuts\DATA\5\data
 spect, CDC13
 Tue May 13 07:52:35 2014
 USER: nmr
 SOLVENT:
 Experiment = zg30
 Pulse length = 14.000 usec
 Recycle delay = 1.000 sec
 NA = 8
 PTD1d = 32768
 F1 = 300.131866 MHz
 F2 = 1.000000 MHz
 SW1 = 6188.12 Hz



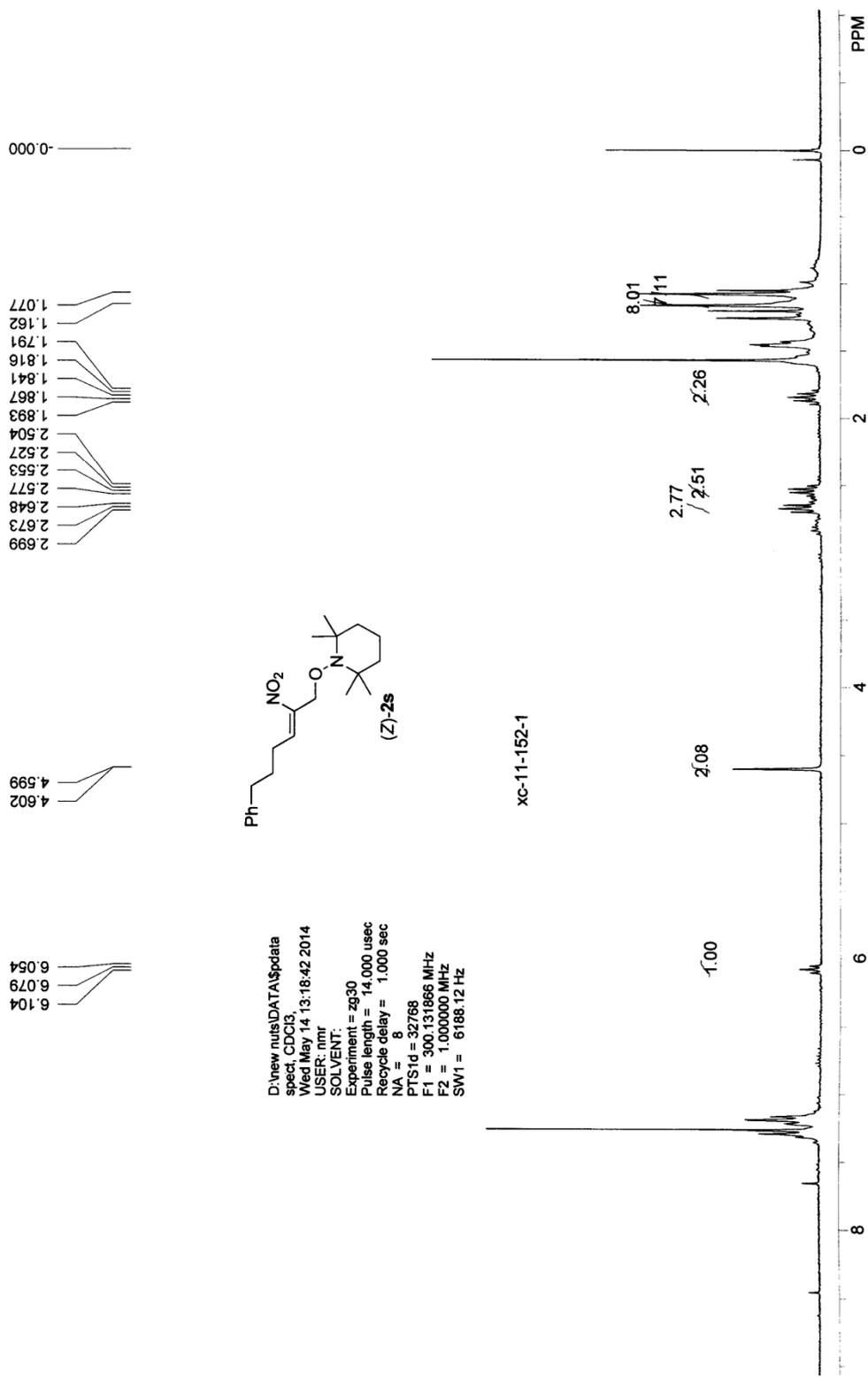
D:\new nuts\DATA\pdata
 spect, CDC13,
 Wed May 14 07:12:33 2014
 USER: nmr
 SOLVENT:
 Experiment = z930
 Pulse length = 14,000 usec
 Recycle delay = 1,000 sec
 NA = 8
 PTD1d = 32768
 F1 = 300.131866 MHz
 F2 = 1,000000 MHz
 SW1 = 6188.12 Hz



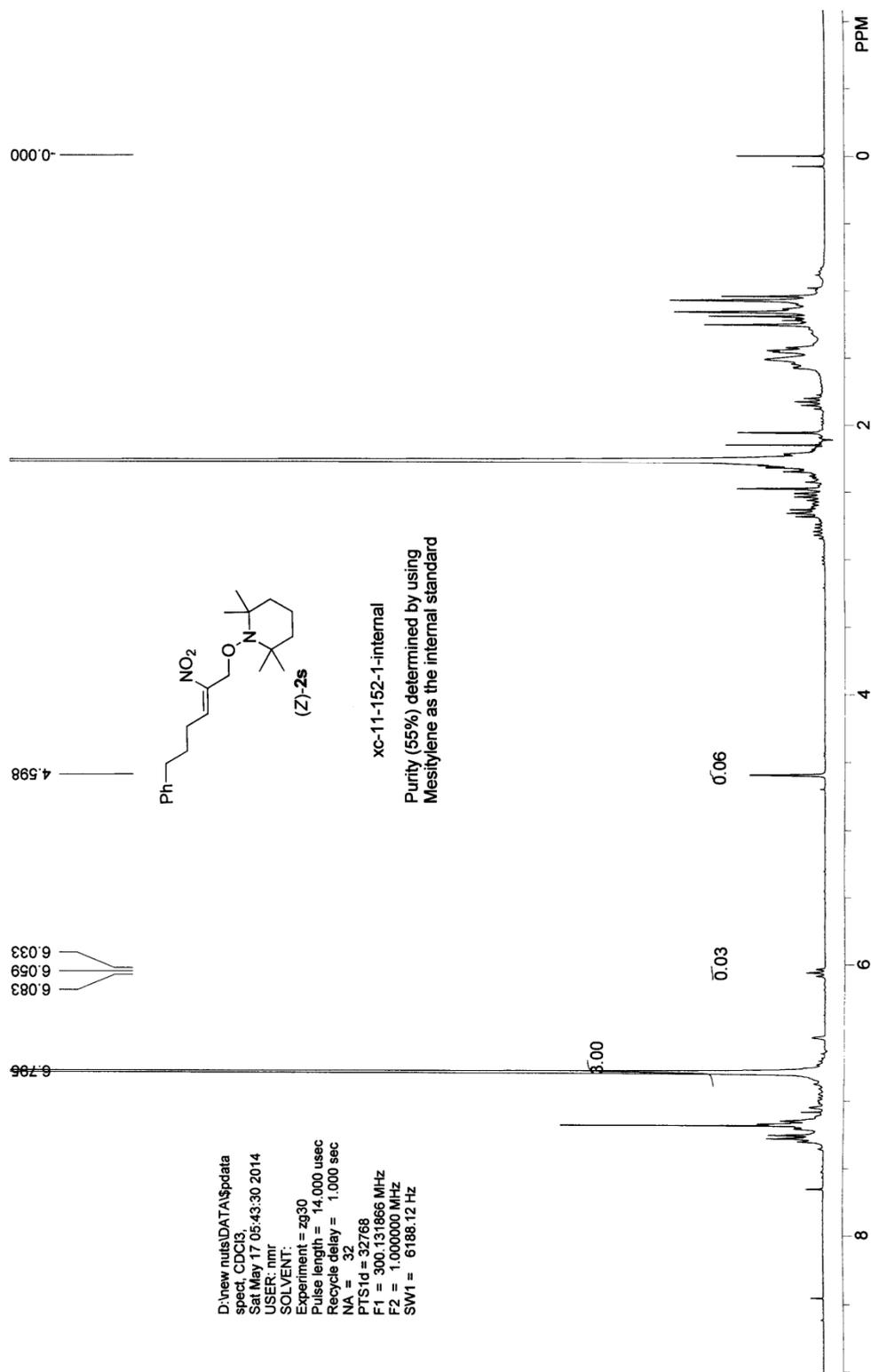
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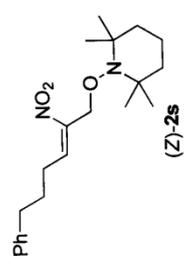
xc-11-152-2-noe



D:\new nuts\DATA\data
 spect, CDCI3,
 Wed May 14 13:18:42 2014
 USER: nmr
 SOLVENT:
 Experiment = z930
 Pulse length = 14,000 usec
 Recycle delay = 1,000 sec
 NA = 8
 P1S1d = 32788
 F1 = 300.131866 MHz
 F2 = 1,000000 MHz
 SW1 = 6188.12 Hz

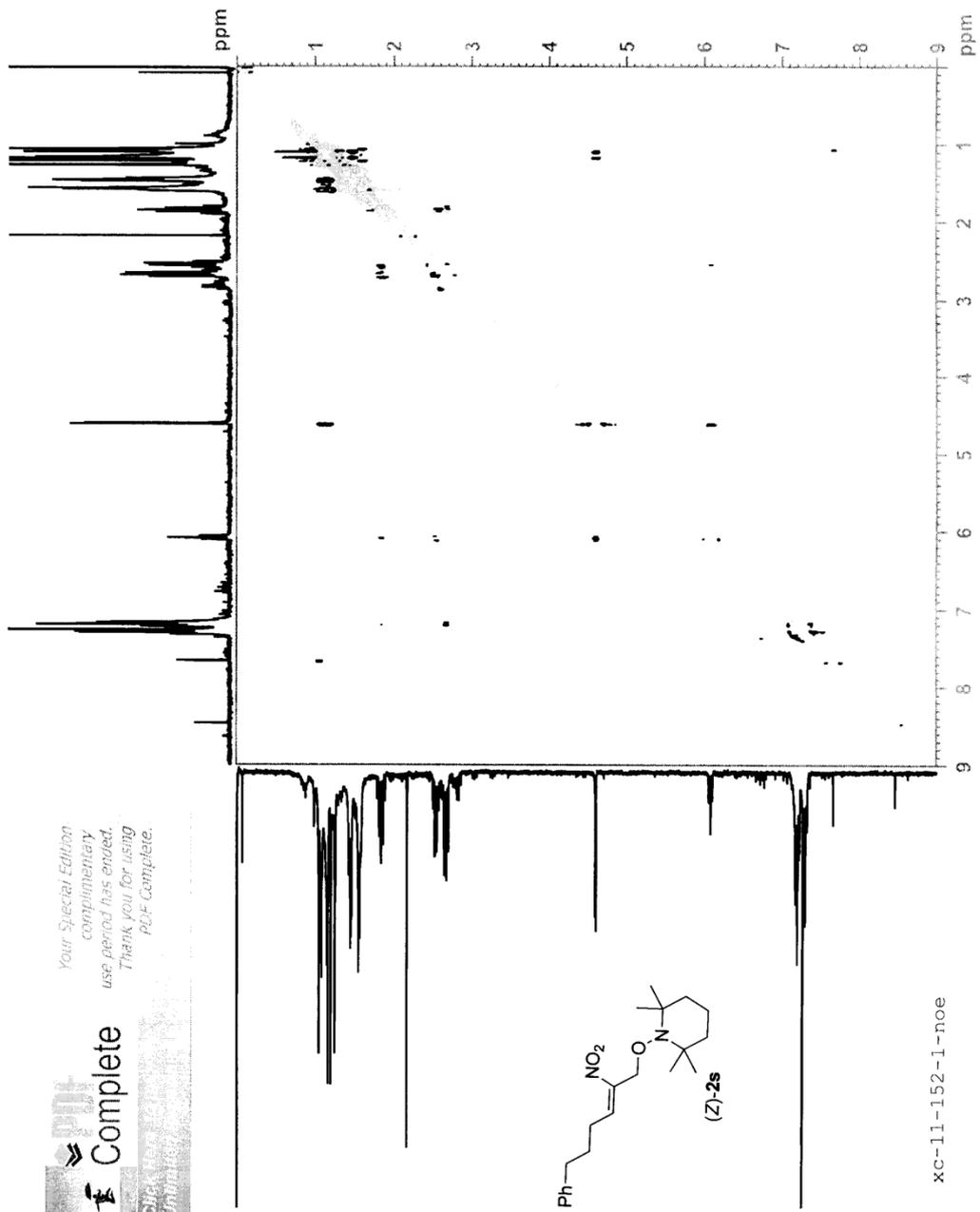


D:\new_nuis\DATA\data
 spect_CDCl3
 Sat May 17 05:43:30 2014
 USER: nmr
 SOLVENT:
 Experiment = zg30
 Pulse length = 14.000 usec
 Recycle delay = 1.000 sec
 NA = 32
 PTS1d = 32768
 F1 = 300.131866 MHz
 F2 = 1.000000 MHz
 SW1 = 6188.12 Hz

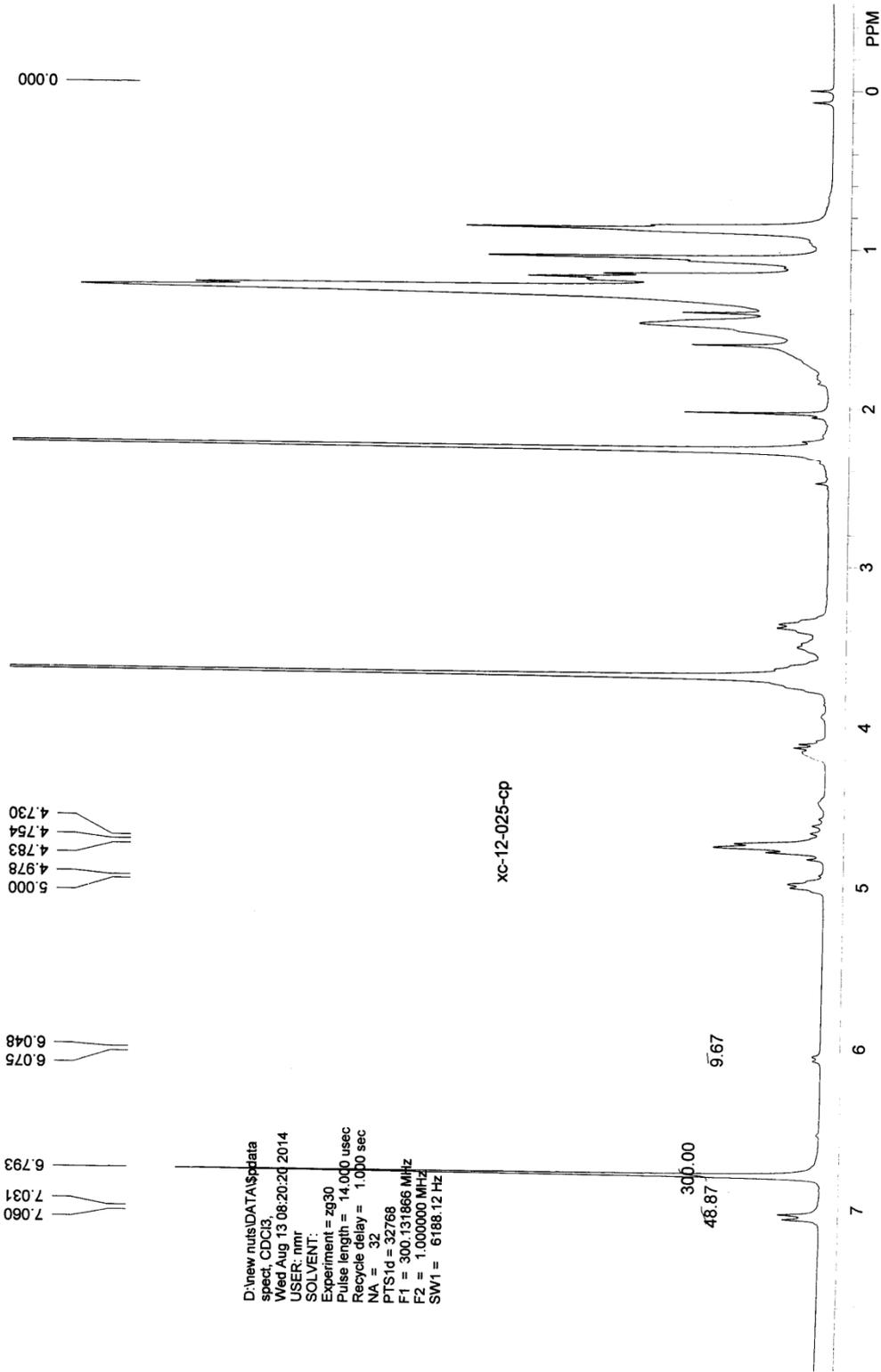


xc-11-152-1-internal
 Purity (55%) determined by using
 Mesitylene as the internal standard

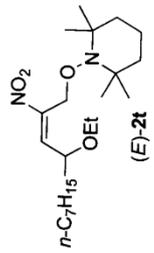
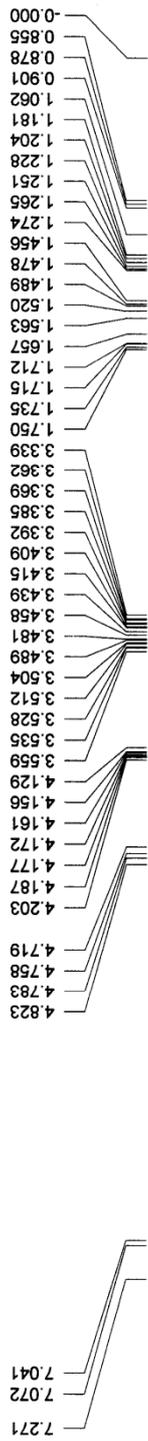
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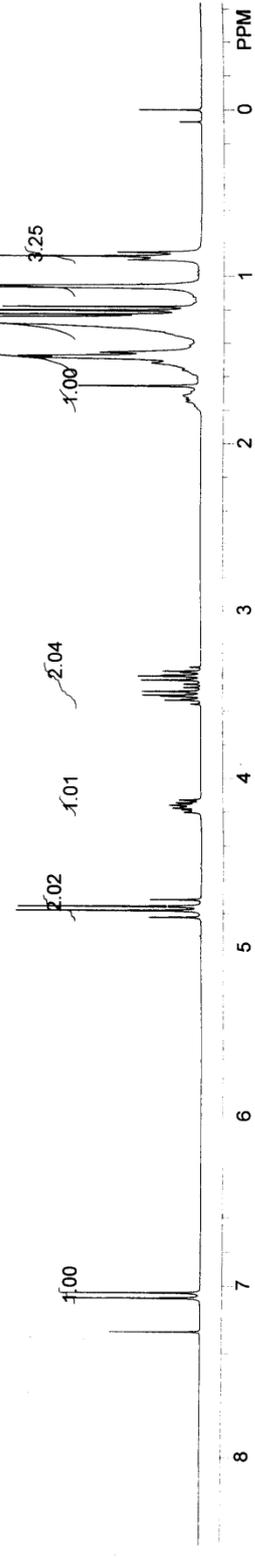


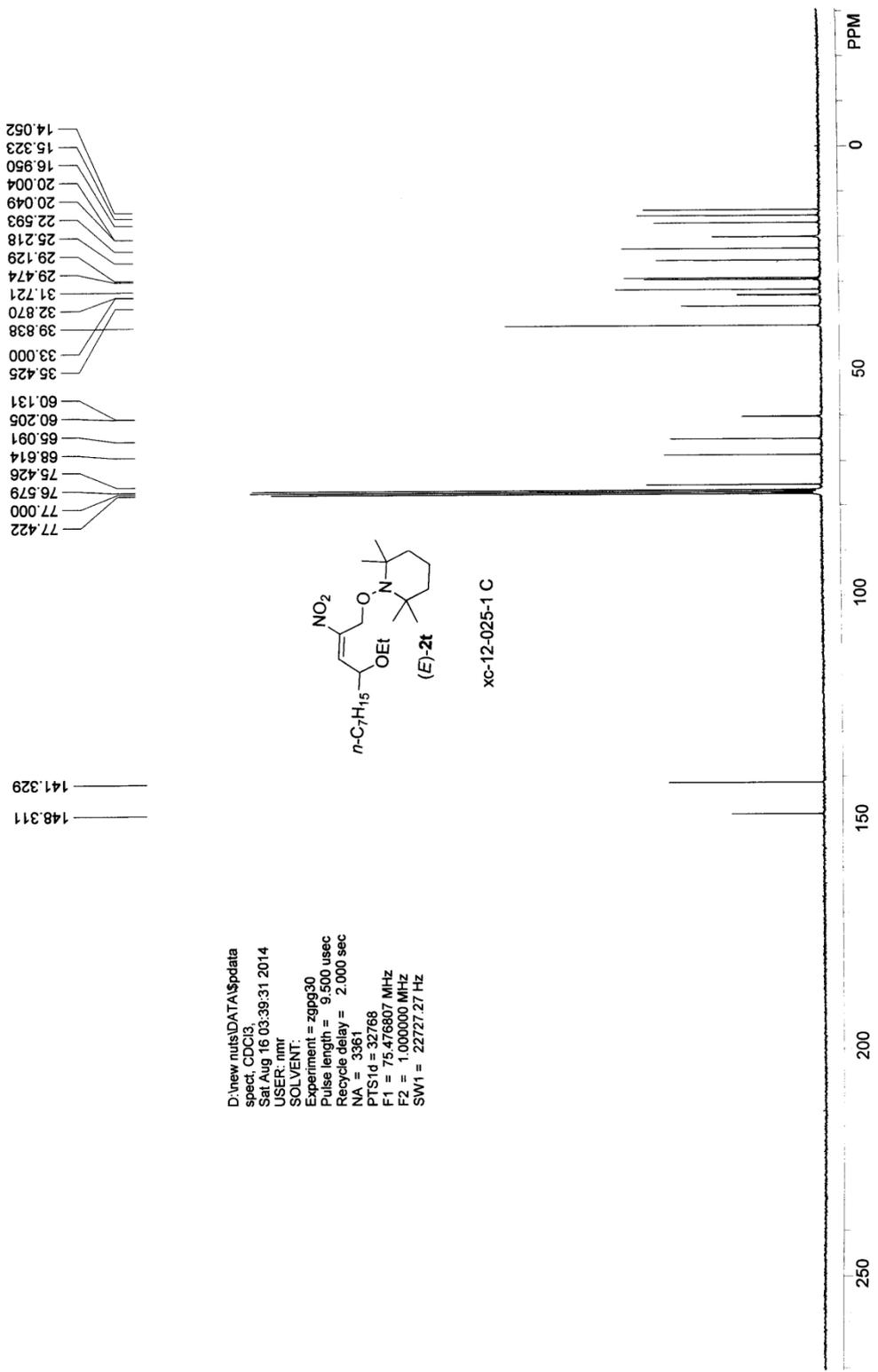
D:\new nuts\DATA\data
spect, CDC13,
Wed Aug 13 08:20:20 2014
USER: nmr
SOLVENT:
Experiment = zq30
Pulse length = 14.000 usec
Recycle delay = 1.000 sec
NA = 32
PTS1d = 32768
F1 = 300.131866 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz



D:\new nuts\DATA\Sp\data
 spect, CDC13,
 Fri Aug 15 06:00:52 2014
 USER: nmr
 SOLVENT:
 Experiment = zg30
 Pulse length = 14,000 usec
 Recycle delay = 1,000 sec
 NA = 8
 P1 = 32768
 F1 = 300.131866 MHz
 F2 = 1,000,000 MHz
 SW1 = 6188.12 Hz

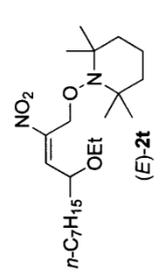
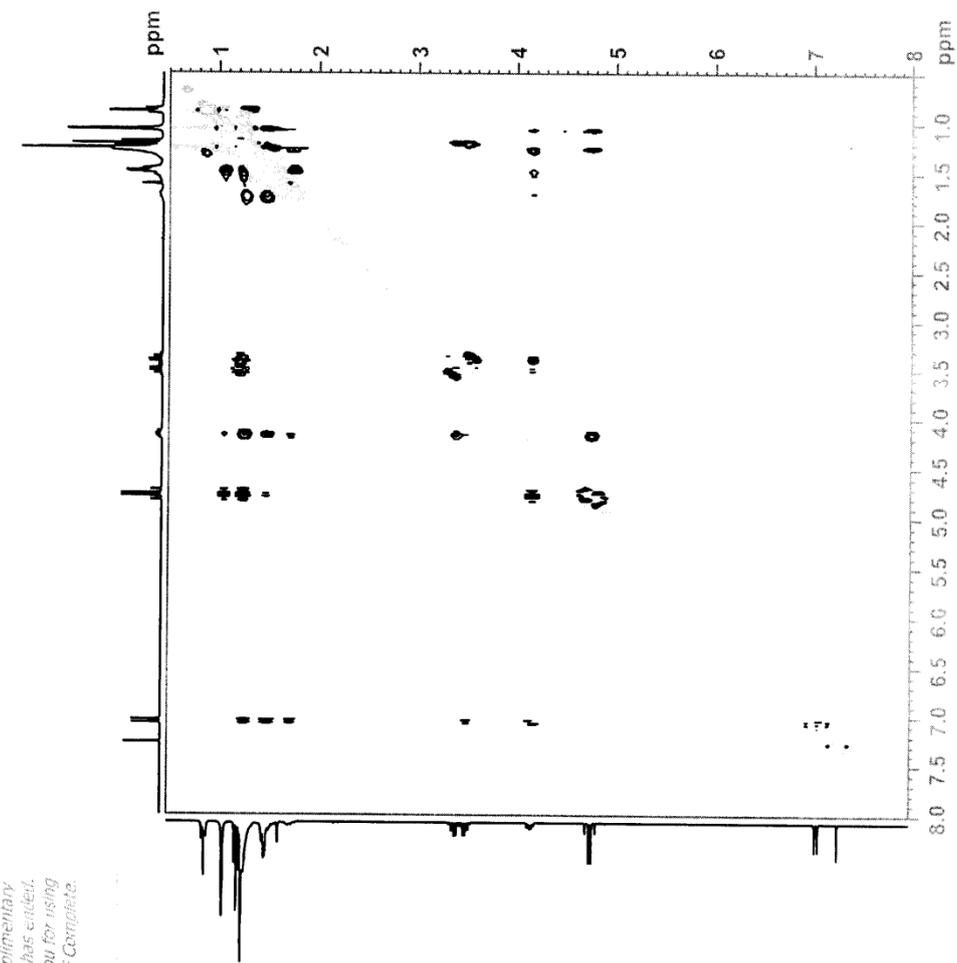
xc-12-025-1



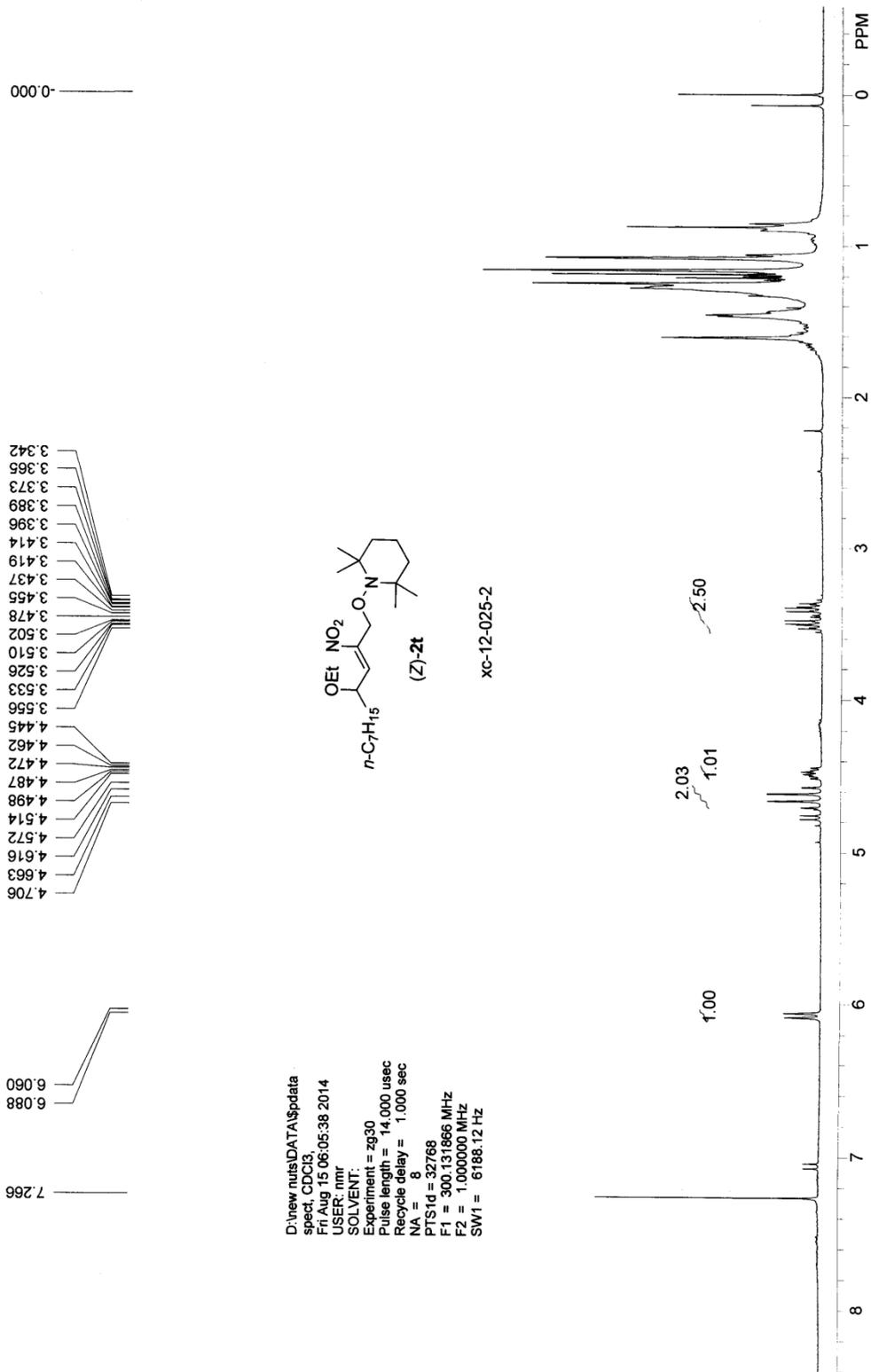


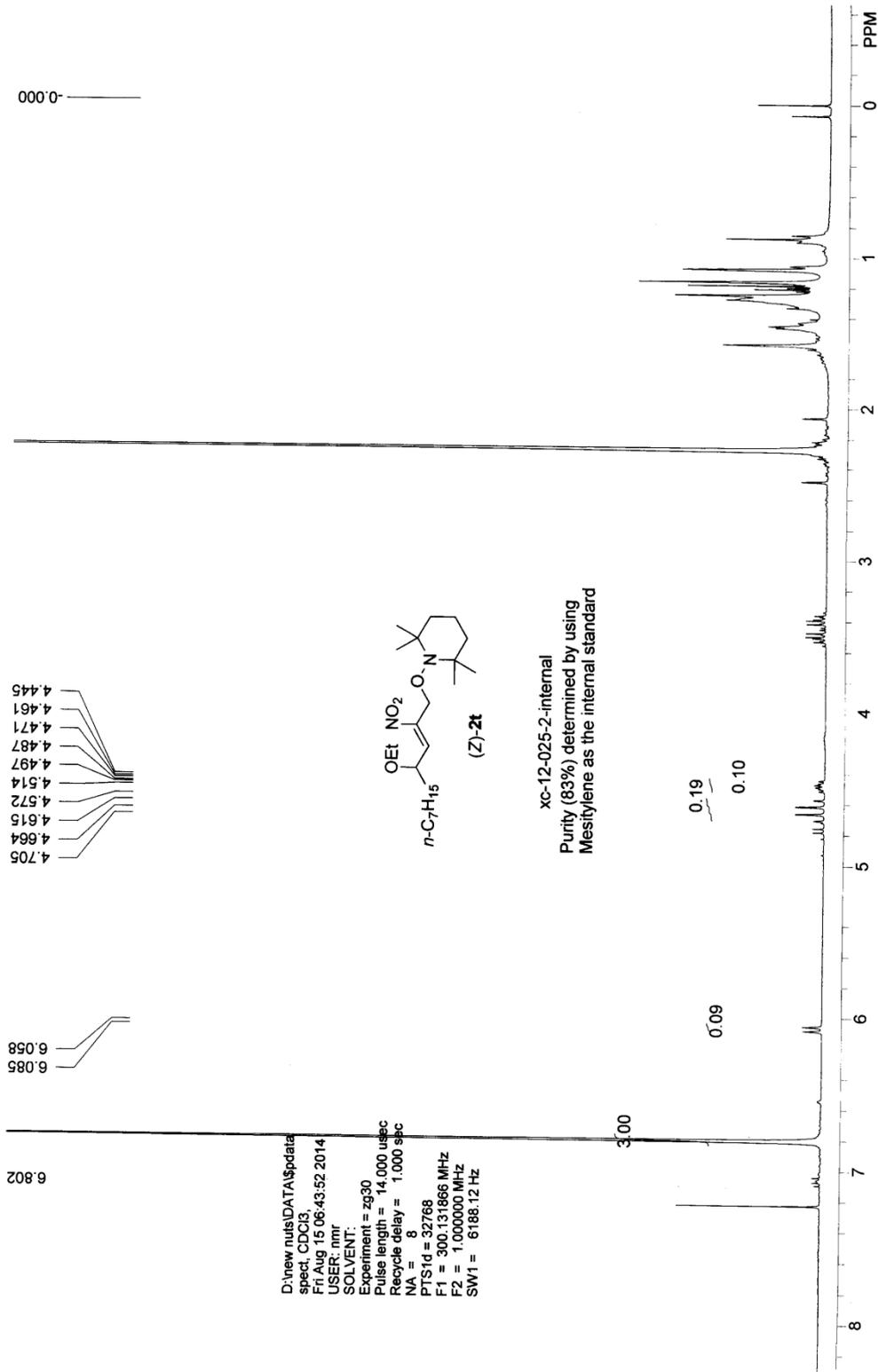
D:\new nuts\DATA\pdata
 spect, CDC13
 Sat Aug 16 03:39:31 2014
 USER: nmr
 SOLVENT:
 Experiment = zgpg30
 Pulse length = 9.500 usec
 Recycle delay = 2.000 sec
 NA = 3361
 PTS1d = 32768
 F1 = 75.476807 MHz
 F2 = 1.000000 MHz
 SW1 = 22727.27 Hz

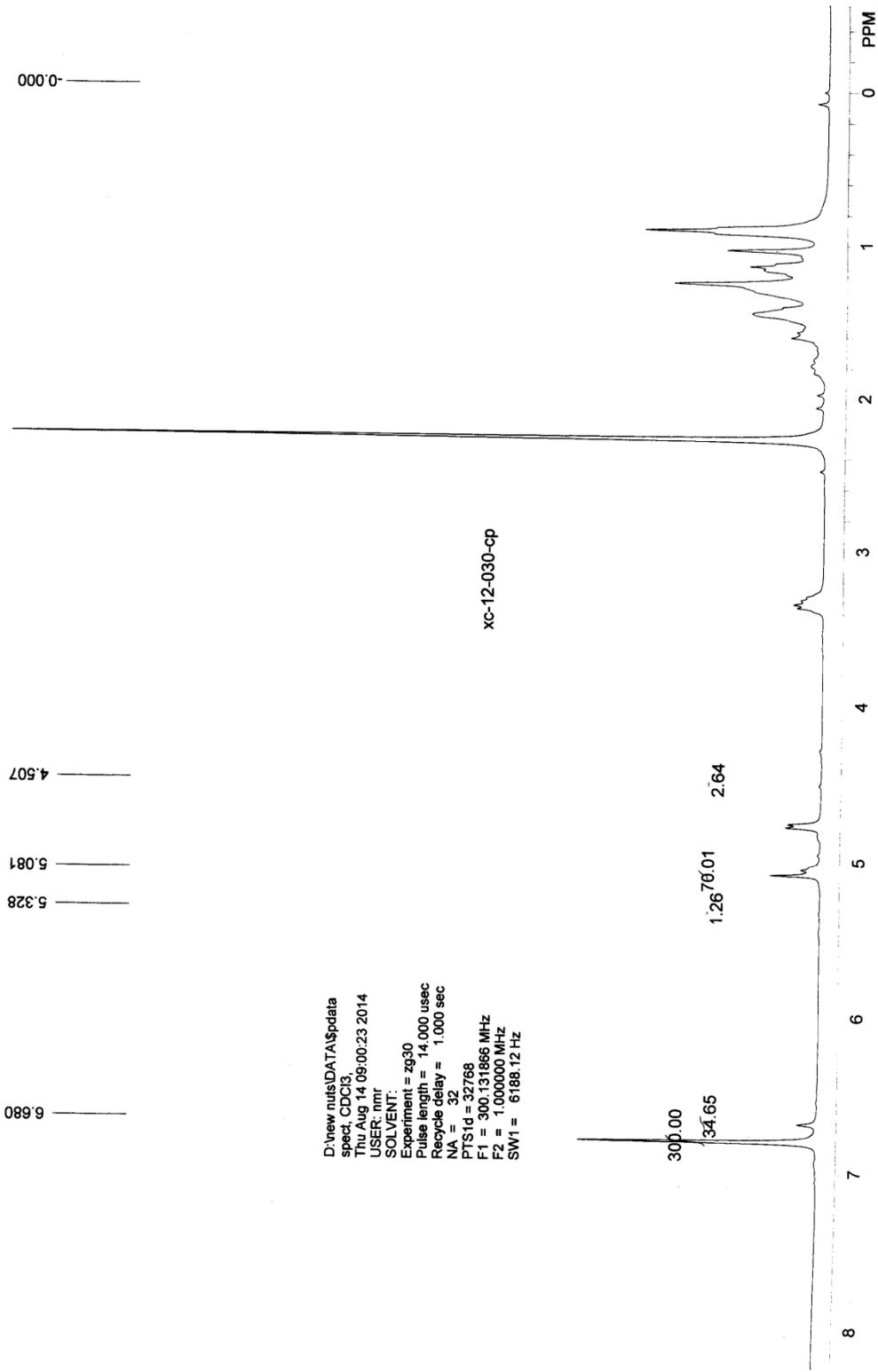

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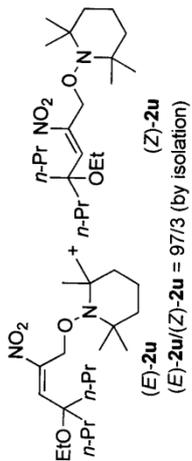
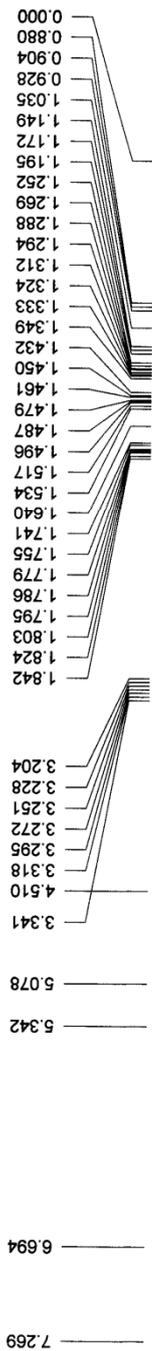
xc-12-025-1-noe





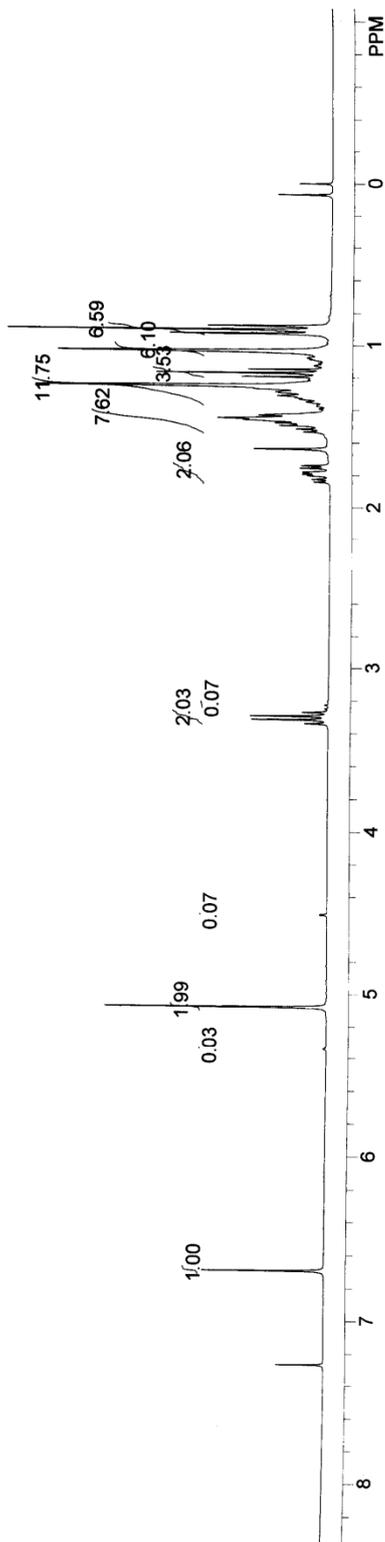


D:\new_nmr\DATA\pdata
 spect_CDCl3
 Thu Aug 14 09:00:23 2014
 USER: nmr
 SOLVENT:
 Experiment = zg30
 Pulse length = 14,000 usec
 Recycle delay = 1,000 sec
 NA = 32
 PTS1d = 32768
 F1 = 300,131866 MHz
 F2 = 1,000000 MHz
 SW1 = 6188.12 Hz

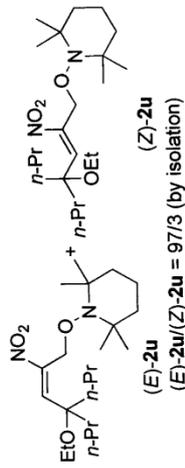


D:\new nuts\DATA\data
 spect, CDCI3,
 Fri Aug 15 05:56:06 2014
 USER: nmr
 SOLVENT:
 Experiment = zq30
 Pulse length = 14,000 usec
 Recycle delay = 1,000 sec
 NA = 8
 P1 = 32768
 F1 = 300.131866 MHz
 F2 = 1,000,000 MHz
 SWH = 6188.12 Hz

xc-12-030-1



D:\new nuts\DATA\data
 spect, CDC3,
 Fri Aug 15 07:52:00 2014
 USER: nmr
 SOLVENT:
 Experiment = z99g30
 Pulse length = 9.500 usec
 Recycle delay = 2.000 sec
 NA = 3518
 PTS1d = 32768
 F1 = 75.476807 MHz
 F2 = 1.000000 MHz
 SW1 = 22727.27 Hz



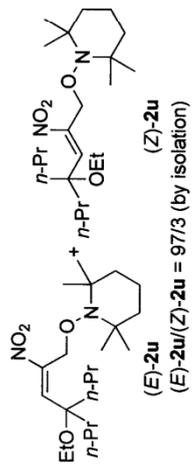
xc-12-030-1 C

79.684
 77.424
 77.000
 76.568
 68.226
 60.024
 56.643
 40.057
 39.187
 33.085
 19.913
 17.013
 16.830
 15.500
 14.268

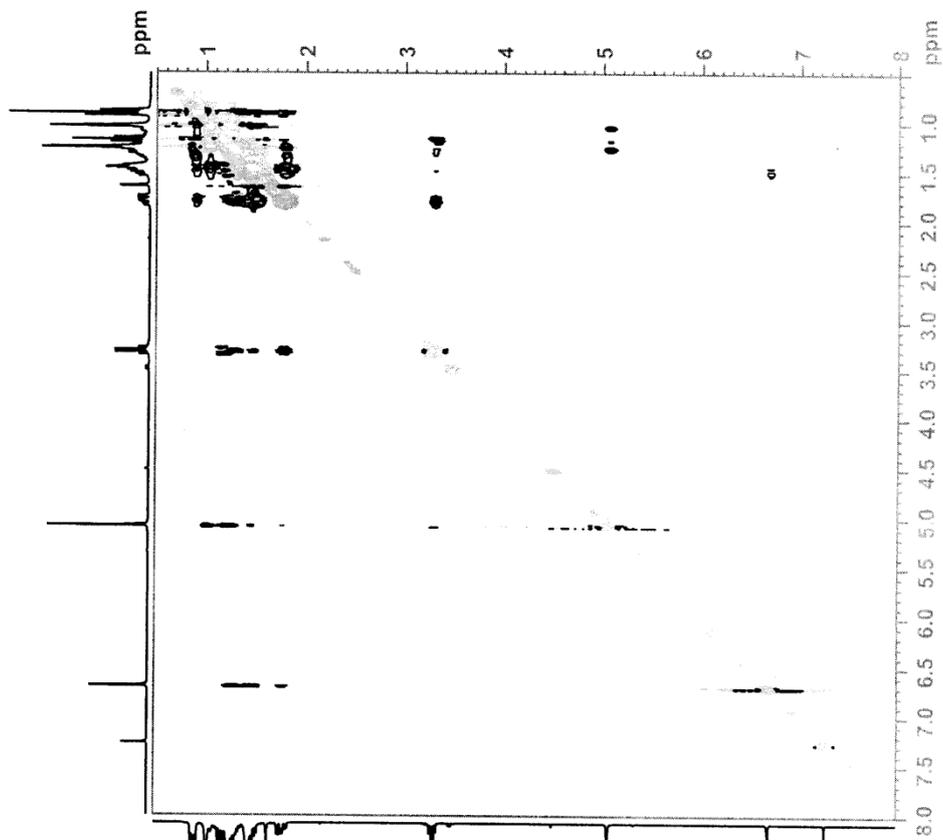
149.862
 141.083

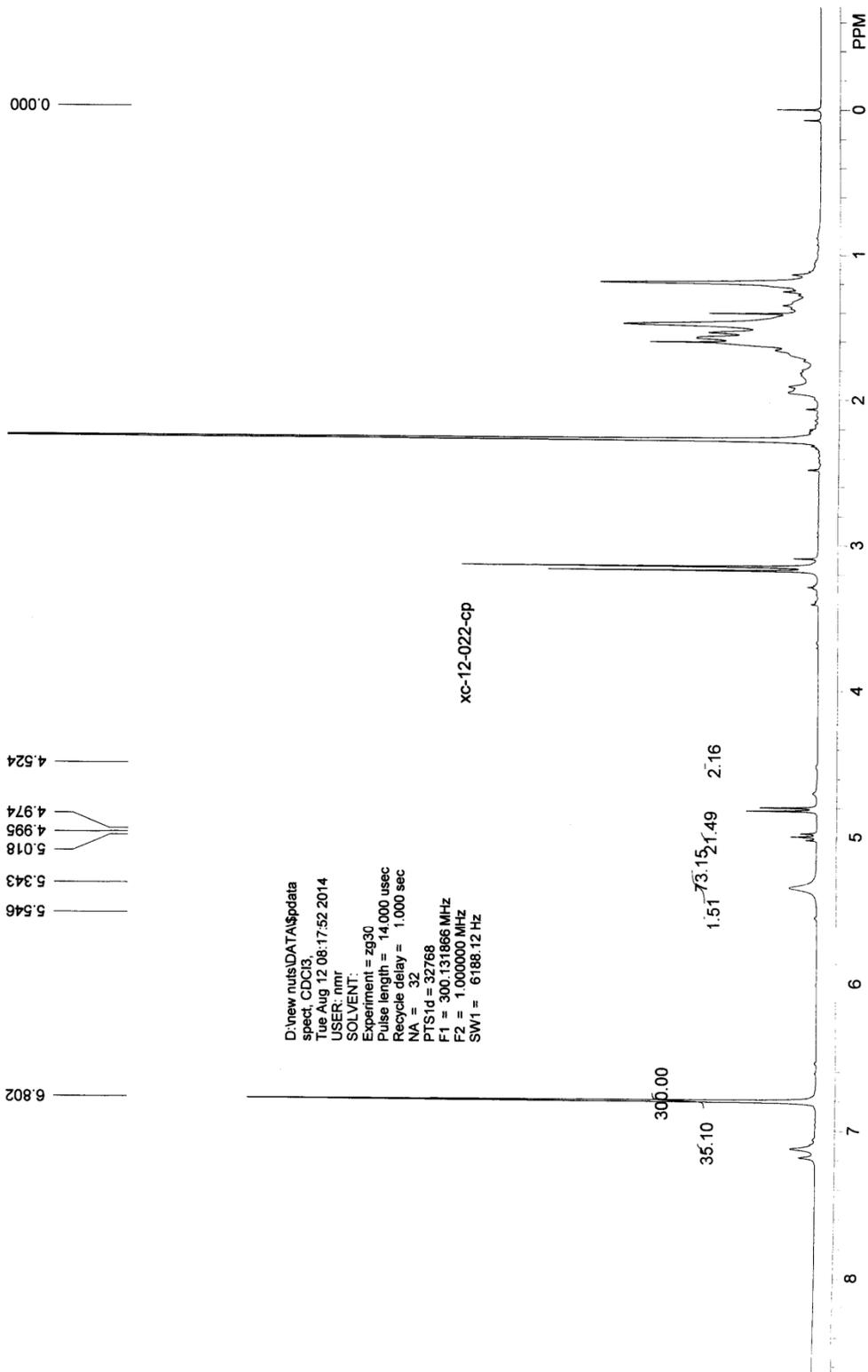
250
 200
 150
 100
 50
 0
 PPM

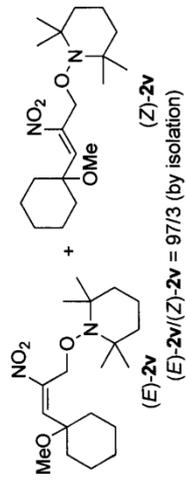
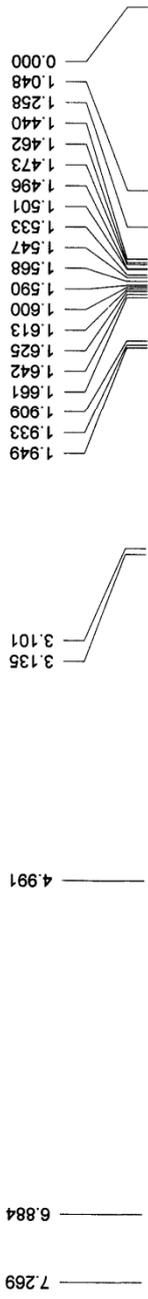

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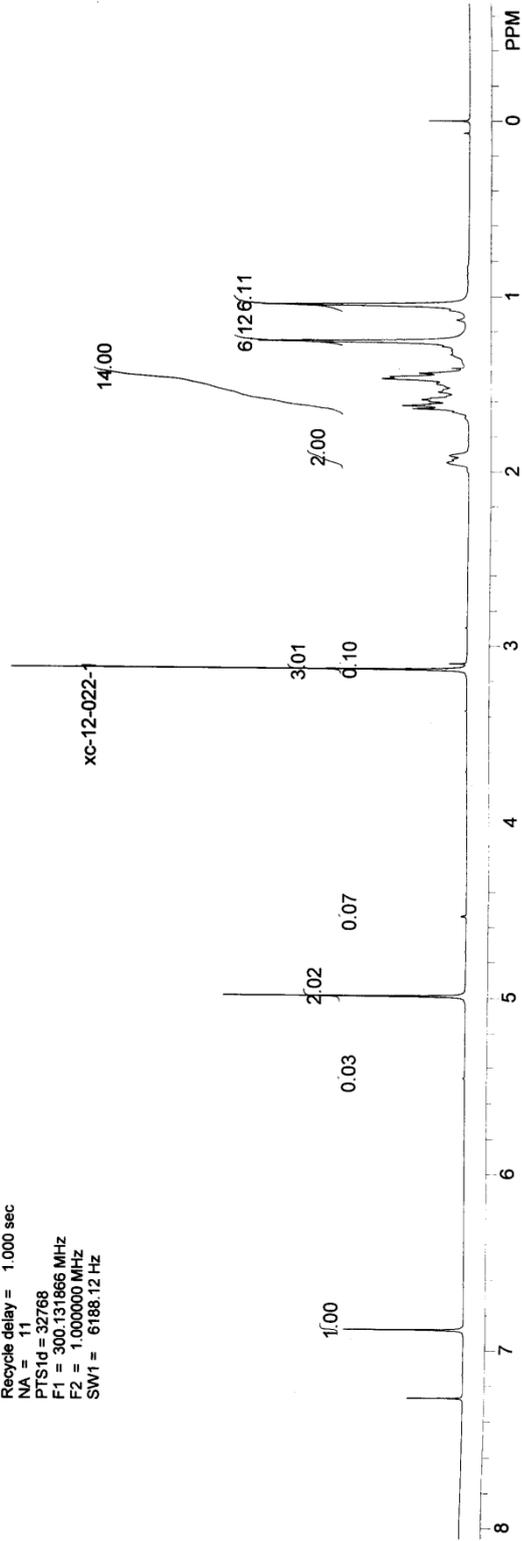
xc-12-030-1-noe





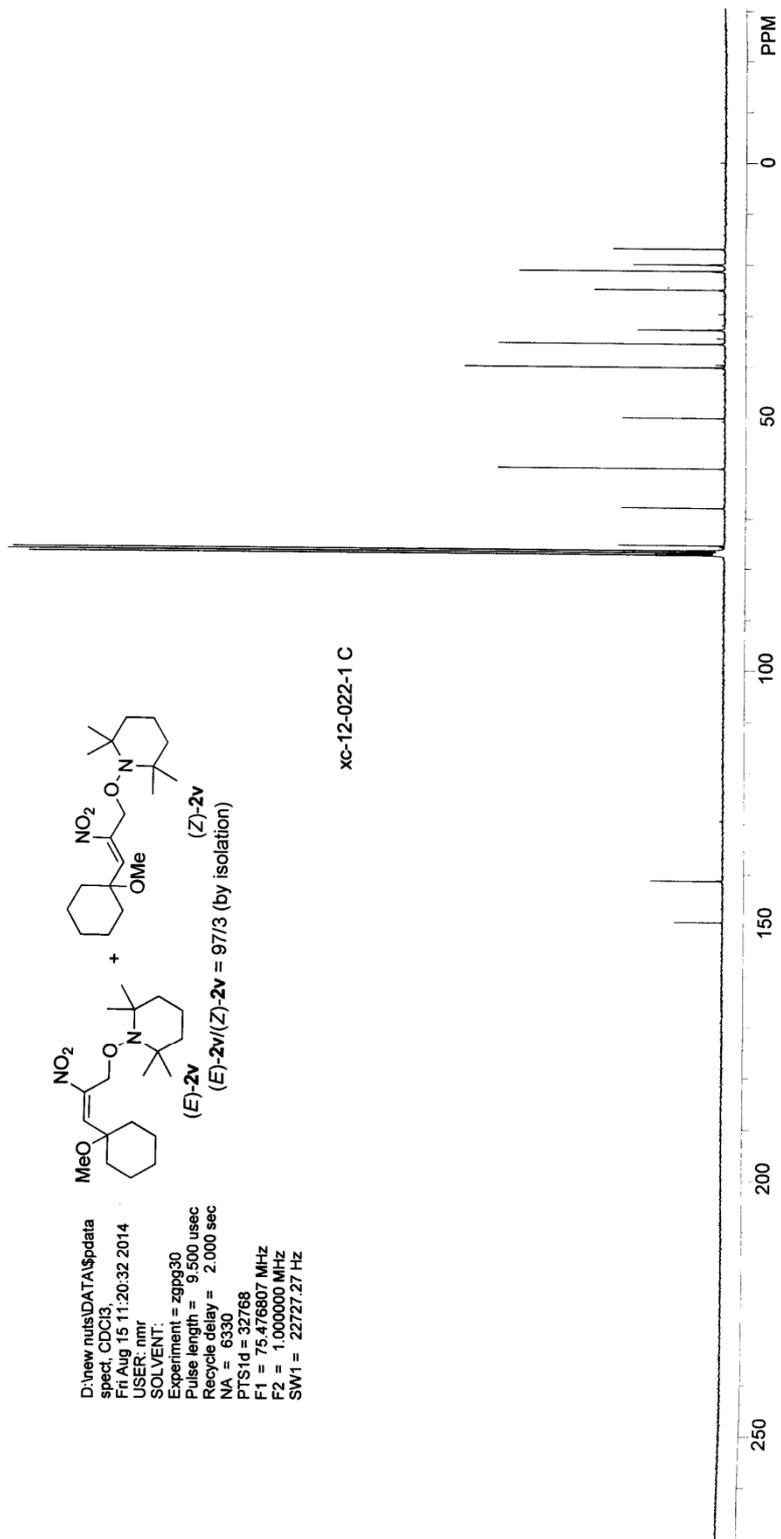


D:\new nuts\DATA\pdata
 spect, CDCl3
 Thu Aug 14 09:40:54 2014
 USER: nmr
 SOLVENT:
 Experiment = zg30
 Pulse length = 14.000 usec
 Recycle delay = 1.000 sec
 NA = 11
 PTS1d = 32788
 F1 = 300.131866 MHz
 F2 = 1.000000 MHz
 SW1 = 6188.12 Hz

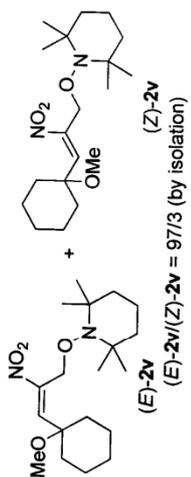


16.980
20.028
21.284
24.954
32.751
35.512
40.155
50.193
60.169
67.940
75.581
76.579
77.000
77.422

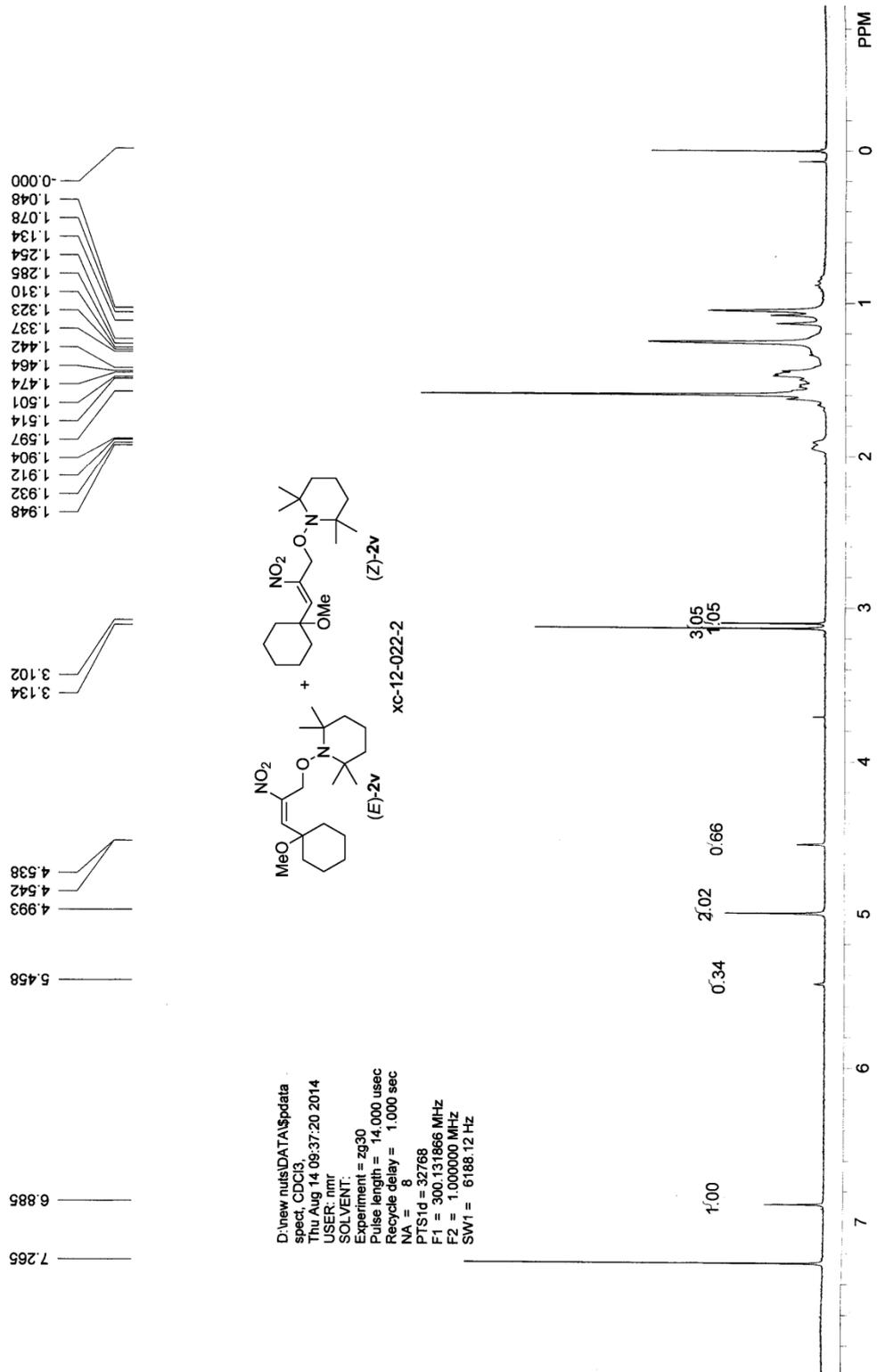
141.315
149.410

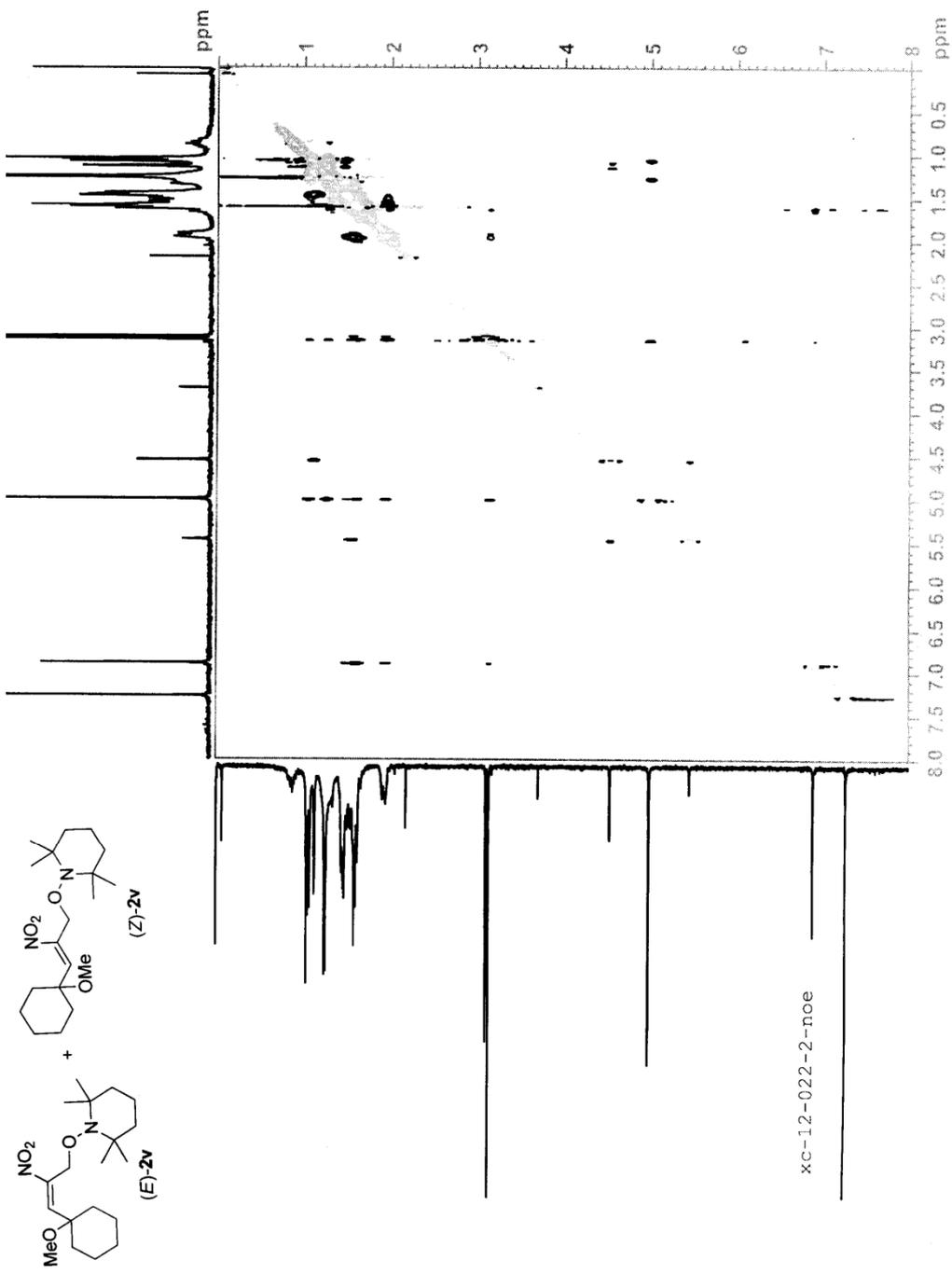


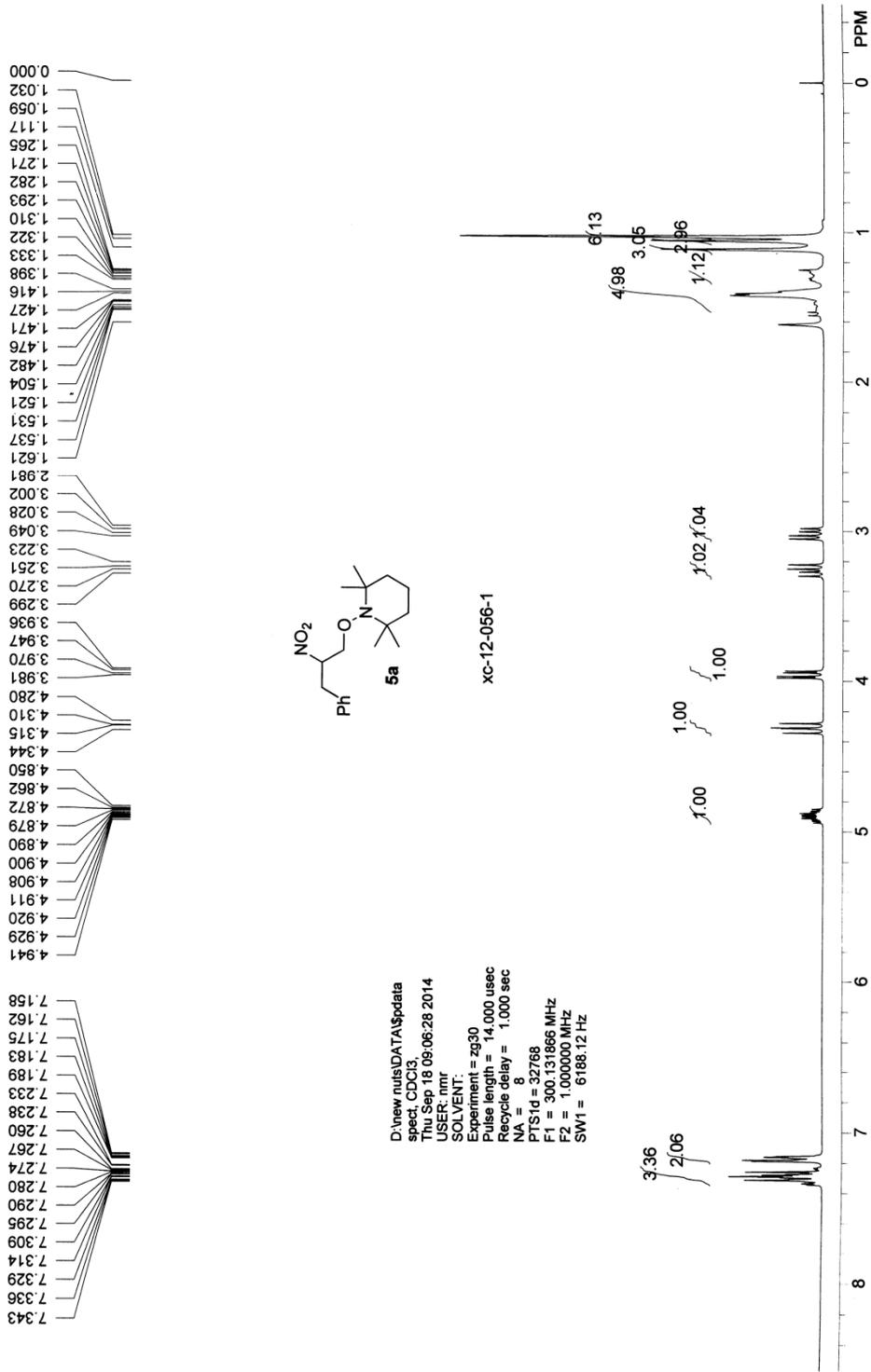
xc-12-022-1 C

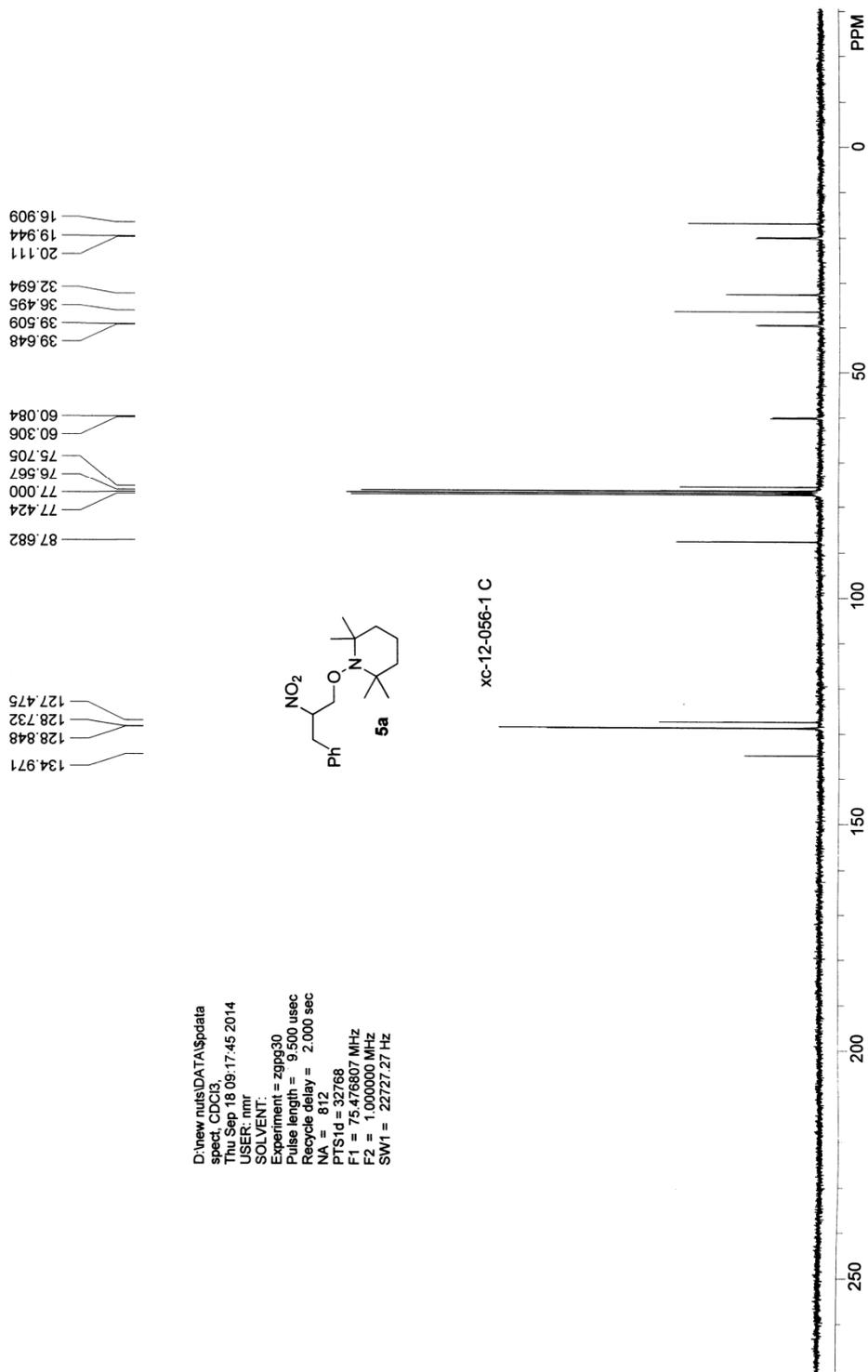


D:\new nuts\DATA\spdata
 spect, CDCl3
 Fri Aug 15 11:20:32 2014
 USER: nmr
 SOLVENT:
 Experiment = z9p930
 Pulse length = 9.500 usec
 Recycle delay = 2.000 sec
 NA = 6330
 PTS1d = 32768
 F1 = 75.476807 MHz
 F2 = 1.000000 MHz
 SW1 = 22727.27 Hz

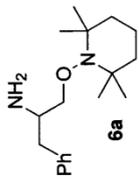
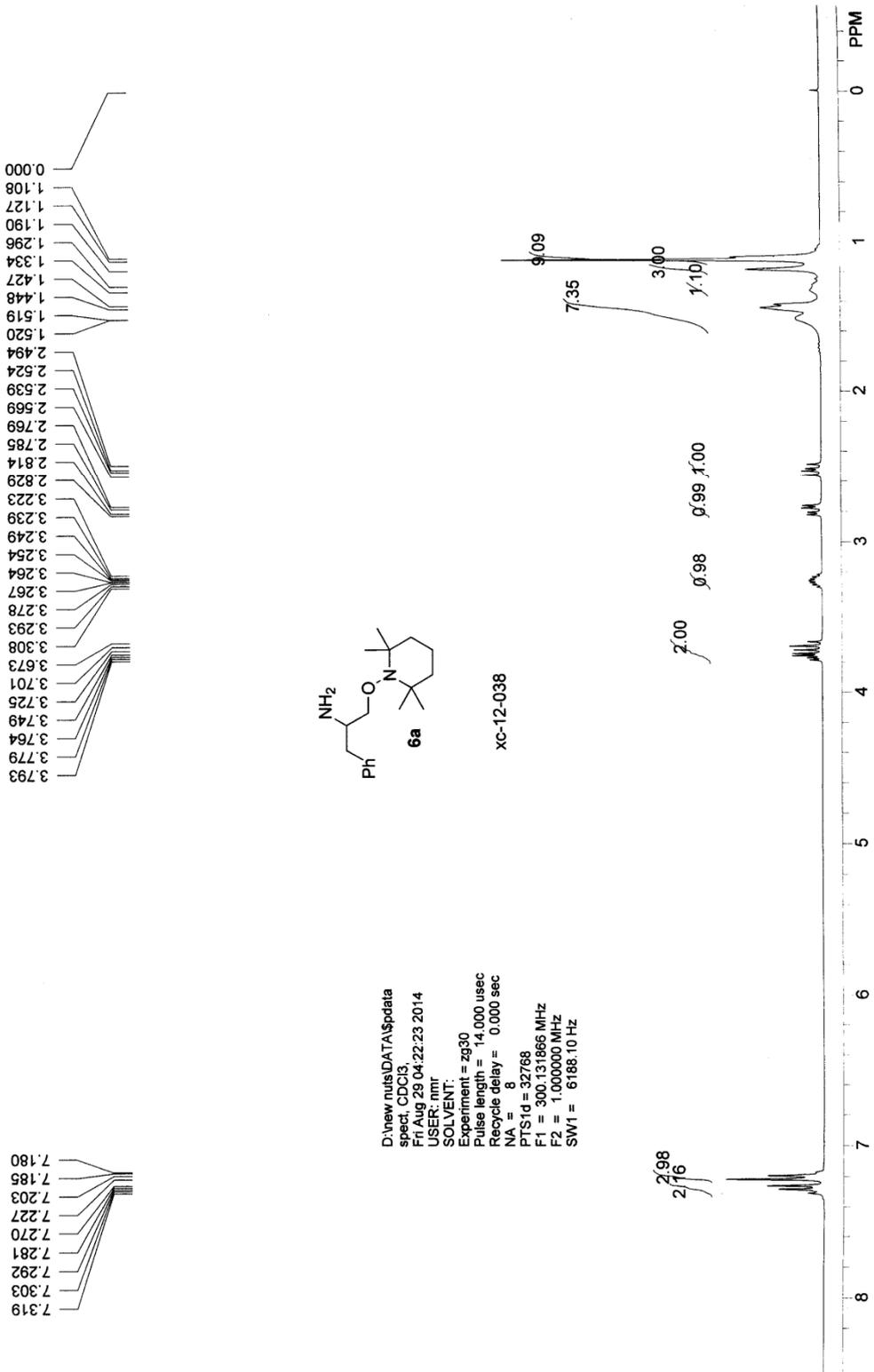








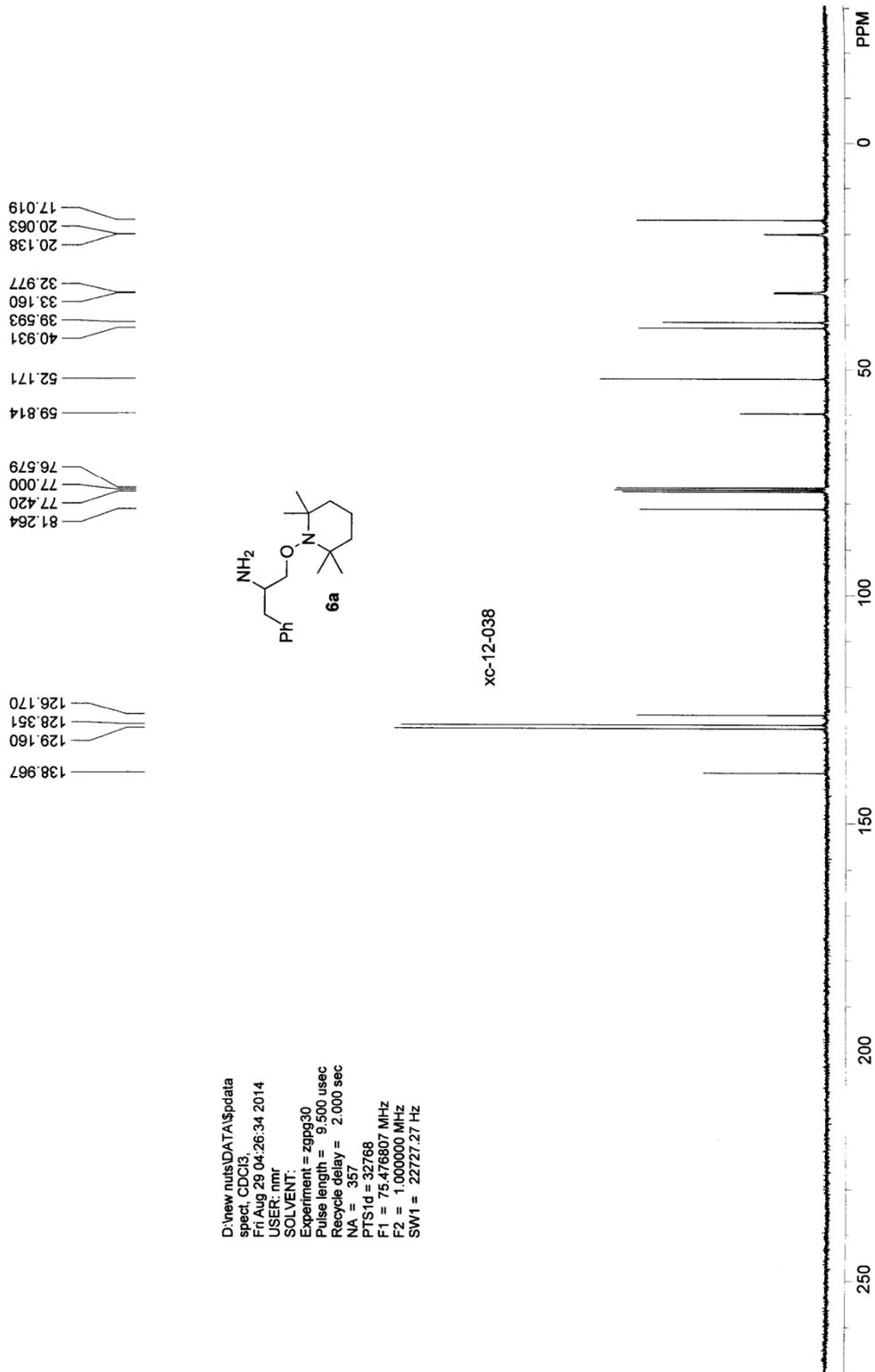
D:\new nuts\DATA\data
 spect, CDCI3,
 Thu Sep 18 09:17:45 2014
 USER: nmr
 SOLVENT:
 Experiment = zgpg30
 Pulse length = 9.500 usec
 Recycle delay = 2.000 sec
 NA = 812
 PTS1d = 32768
 F1 = 75.476807 MHz
 F2 = 1.000000 MHz
 SW1 = 22727.27 Hz



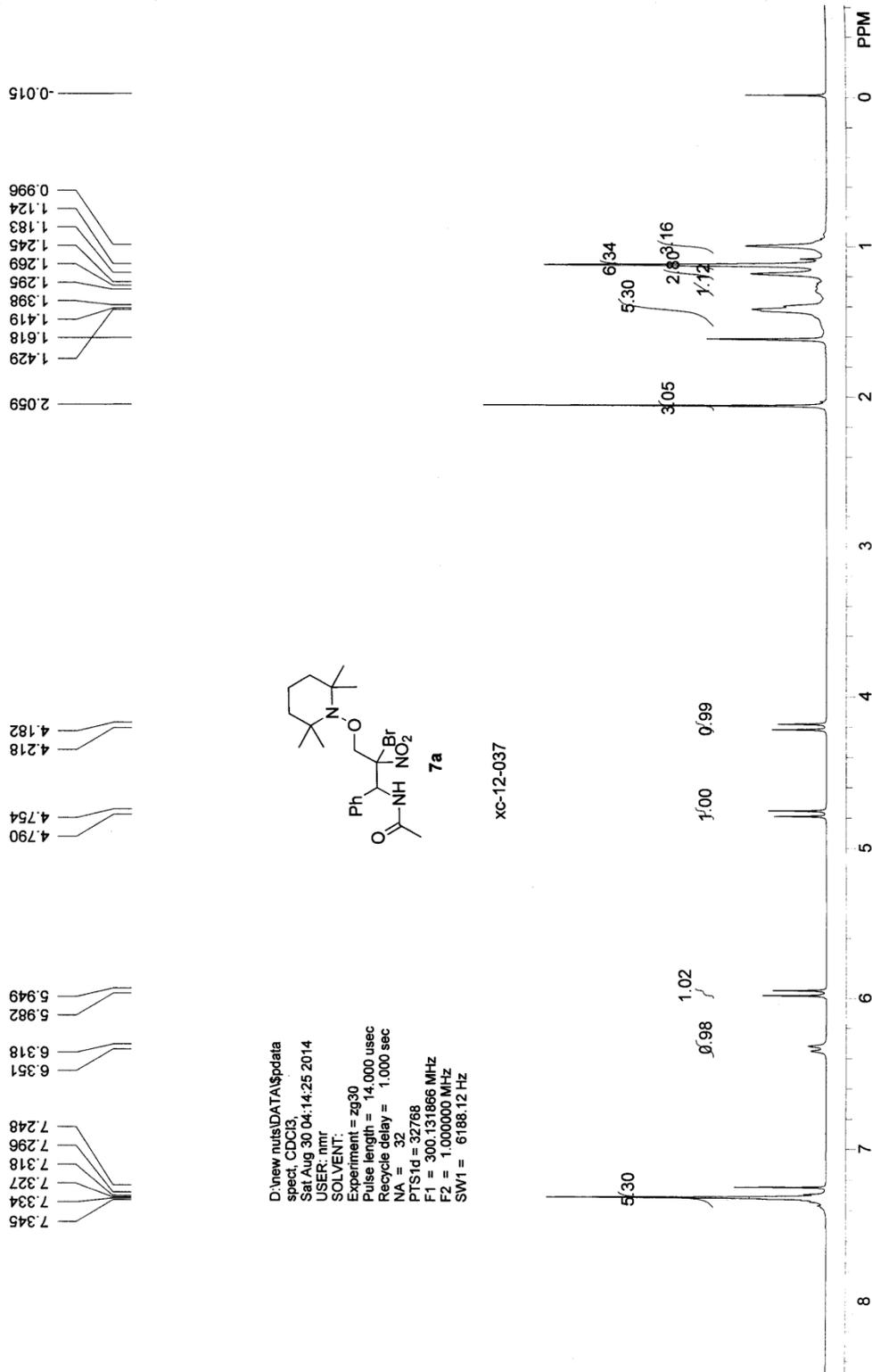
XC-12-038

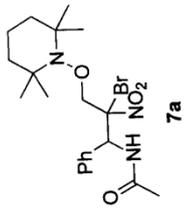
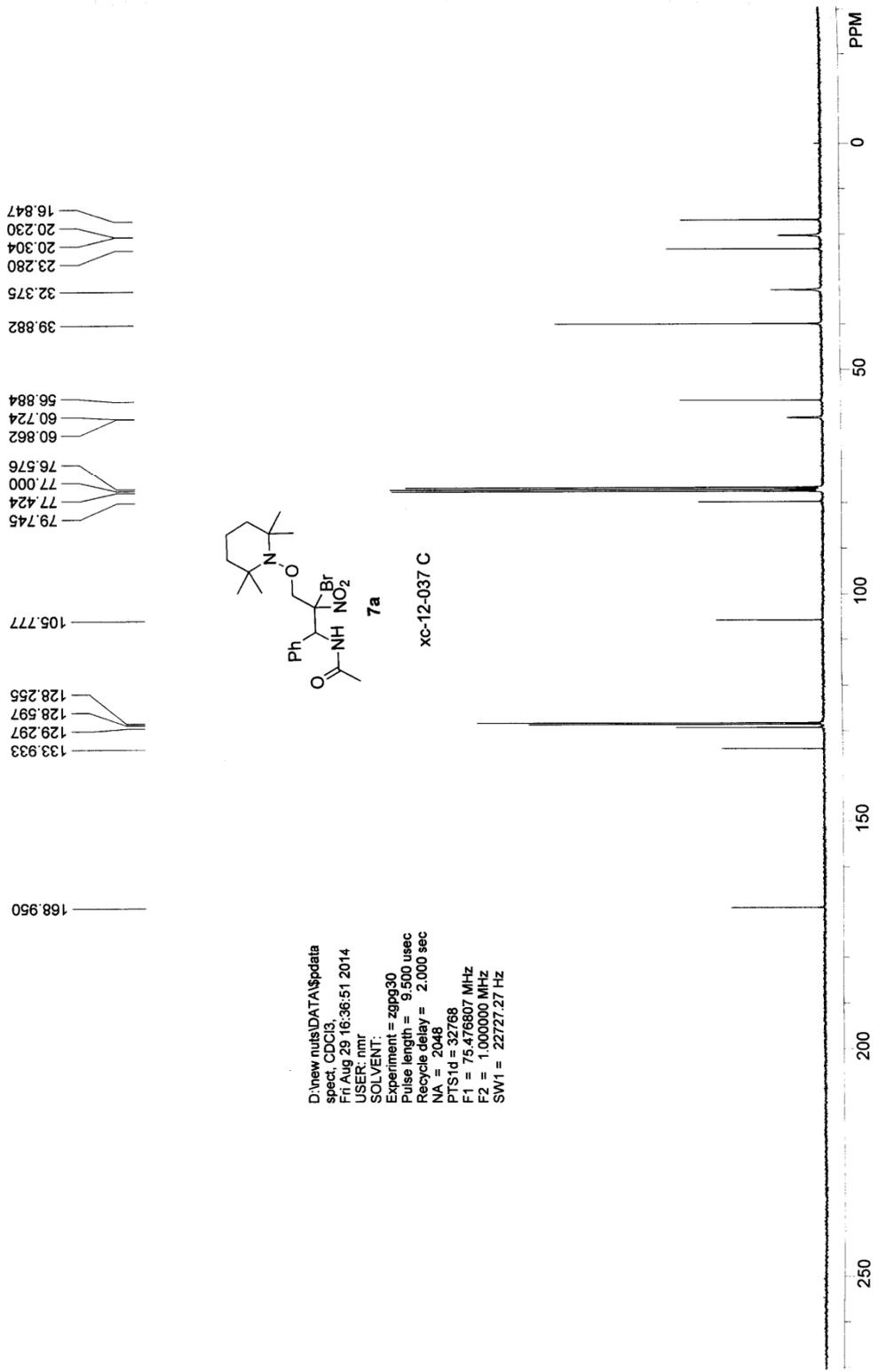
D:\new nus\DATA\Spdata
 spect, CDCl3,
 Fri Aug 29 04:22:23 2014
 USER: nmr
 SOLVENT:
 Experiment = zg30
 Pulse length = 14.000 usec
 Recycle delay = 0.000 sec
 NA = 8
 P1 = 32768
 F1 = 300.131866 MHz
 F2 = 1.000000 MHz
 SWH = 6186.10 Hz

7.319
 7.303
 7.292
 7.281
 7.270
 7.227
 7.203
 7.185
 7.180
 3.793
 3.779
 3.764
 3.749
 3.725
 3.701
 3.673
 3.308
 3.293
 3.278
 3.267
 3.264
 3.249
 3.239
 3.223
 2.829
 2.814
 2.785
 2.769
 2.569
 2.539
 2.524
 2.494
 1.520
 1.519
 1.448
 1.427
 1.334
 1.296
 1.190
 1.127
 1.108
 0.000



D:\new nuis\DATA\pdata
 spect_CDCl3
 Fri Aug 29 04:26:34 2014
 USER: rmi
 SOLVENT:
 Experiment = zgpg30
 Pulse length = 9.500 usec
 Recycle delay = 2.000 sec
 NA = 357
 PTS1d = 32768
 F1 = 75.476807 MHz
 F2 = 1.000000 MHz
 SW1 = 22727.27 Hz





D:\new nuis\DATA\pdata
spect. CDCl3.
Fri Aug 29 16:36:51 2014
USER: nmr
SOLVENT:
Experiment = zgpg30
Pulse length = 9.500 usec
Recycle delay = 2.000 sec
NA = 2048
PTS1d = 32768
F1 = 75.476807 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz