

## Efficient synthesis of triazole-containing spiro dilactones

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### Experimental part

**General remarks.**  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectra were recorded on Varian Mercury-300 MHz in DMSO- $\text{CCl}_4$  mixture (1:3) or on Bruker AVANCE 400 MHz spectrometer in  $\text{CDCl}_3$ , acetone- $d_6$ , DMSO- $d_6$ . Chemical shifts ( $\delta$ ) in ppm are reported as quoted relative to the residual signals of chloroform- $d$  (7.25 for  $^1\text{H}$  NMR and 77.0 for  $^{13}\text{C}$  NMR), acetone- $d_6$  (2.04 for  $^1\text{H}$  NMR and 29.8 for  $^{13}\text{C}$  NMR), or DMSO- $d_6$  (2.49 for  $^1\text{H}$  NMR and 39.5 for  $^{13}\text{C}$  NMR) as internal references. The coupling constants ( $J$ ) are given in Hertz. IR spectra were recorded on ThermoNicolet IR 200. ESI-MS spectra were measured with a MicroTof Bruker Daltonics instrument. The silica gel used for column chromatography was 230-400 Mesh. TLC analysis was performed on “Silufol UV-254” plates. All reagents were of reagent grade and were used as such or distilled prior to use. Starting ethyl tetrahydro-2-oxofuran-3-carboxylates were prepared as previously reported. [V. S. Harutyunyan, T. V. Kochikyan, E. V. Harutyunyan and A. A. Avetisyan, *Arm. Khim. Zh.*, 2000, **53**, 121; V. S. Harutyunyan, T. V. Kochikyan, N.S. Egiazaryan and A. A. Avetisyan, *Arm. Khim. Zh.*, 1994, **47**, 91].

**Synthesis of ethyl 3-allyl-2-oxotetrahydrofuran-3-carboxylates 1-3 (general procedure).** Flask was charged with 20 mL of absolute ethanol and 2.3 g (0.1 mol) of sodium to give a solution. Next, 0.1 mol of corresponding ethyl tetrahydro-2-oxofuran-3-carboxylate was added dropwise at cooling. Reaction mixture was stirred for 15 min and then 0.11 mol of appropriate alkenyl halogenide was added. Stirring was continued for 2 h at room temperature and then at 75-80 °C until neutral reaction was achieved. Ethanol was evaporated and the residue was acidified by dropwise addition of diluted hydrochloric acid until pH 2-3. Reaction mixture was extracted with ether, combined extracts were dried over  $\text{MgSO}_4$ . Volatiles were evaporated and the residue was distilled at reduced pressure.

**Ethyl 3-allyl-5,5-dimethyl-2-oxotetrahydrofuran-3-carboxylate (1).** Yield 91%; b.p. 85-86/1 Torr;  $R_f=0.64$  (EtOH: $\text{C}_6\text{H}_6$ :hexane 3:3:10);  $n_D^{20}$  1.4550;  $d_4^{20}$  1.0497;  $^1\text{H}$  NMR (300 MHz, DMSO- $d_6$ )  $\delta$  1.30 (t,  $J = 7.6$  Hz, 3H,  $\text{CH}_3\text{CH}_2$ ), 1.45 (s, 6H, 2 $\text{CH}_3$ ), 2.20 and 2.45 (both m,  $\text{CH}_2$  in cycle, 2H), 2.56 and 2.82 (both m,  $\text{CH}_2$  non cyclic, 2H), 4.22 (m,  $\text{CH}_3\text{CH}_2$ , 2H), 5.05 (m,  $\text{C}=\text{CH}_2$ , 1H), 5.10 (m,  $\text{C}=\text{CH}_2$ , 1H), 5.85 (m,  $\text{CH}=\text{CH}_2$ , 1H). Anal. Calcd for  $\text{C}_{12}\text{H}_{18}\text{O}_4$ : C 63.70; H 8.02. Found: C 63.60; H 8.05.

**Ethyl 3-allyl-5-methyl-2-oxotetrahydrofuran-3-carboxylate (2).** Yield 84%; b.p. 86-87 °C /2 Torr;  $R_f=0.59$  (EtOH: $\text{C}_6\text{H}_6$ :hexane 3:3:10);  $n_D^{20}$  1.4565;  $d_4^{20}$  1.0754;  $^1\text{H}$  NMR (300 MHz, DMSO- $d_6$ )  $\delta$  1.30 (t,  $J = 7.6$  Hz, 3H,  $\text{CH}_3\text{CH}_2$ ), 1.40 (m, CHO, 1H), 2.30 and 2.50 (both m,  $\text{CH}_2$  in cycle, 2H), 2.55 and 2.60 (both m,  $\text{CH}_2$  non cyclic, 2H), 4.20 (m,  $\text{CH}_3\text{CH}_2$ , 2H), 5.00 (m,  $\text{C}=\text{CH}_2$ , 1H), 5.10 (m,  $\text{C}=\text{CH}_2$ , 1H), 5.90 (m,  $\text{CH}=\text{CH}_2$ , 1H); Anal. Calcd for  $\text{C}_{11}\text{H}_{16}\text{O}_4$ : C 62.26; H 7.55. Found: C 62.35; H 7.45.

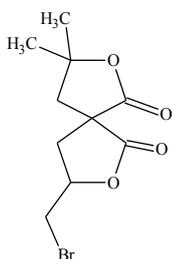
**Ethyl 5,5-dimethyl-3-(2-methylallyl)-2-oxotetrahydrofuran-3-carboxylate (3).** Yield 83%; b.p. 83-84 °C /1 Torr;  $R_f=0.67$  (EtOH: $\text{C}_6\text{H}_6$ :hexane 3:3:10);  $n_D^{20}$  1.4575;  $d_4^{20}$  1.0538;  $^1\text{H}$  NMR (300 MHz, DMSO- $d_6$ )  $\delta$  1.30 (t,  $J = 7.6$  Hz, 3H,  $\text{CH}_3\text{CH}_2$ ), 1.45 (s, 2 $\text{CH}_3$ , 6H), 1.80 (s,  $\text{CH}_3\text{C}=\text{CH}$ , 3H), 2.25 and 2.50 (both m,  $\text{CH}_2$  in cycle, 2H), 2.55 and 2.60

(both m, CH<sub>2</sub> non cyclic, 2H), 4.25 (m, CH<sub>3</sub>CH<sub>2</sub>, 2H); 5.05 (m, C=CH<sub>2</sub>, 1H), 5.10 (m, C=CH<sub>2</sub>, 1H), 5.85 (m, CH=CH<sub>2</sub>, 1H). Anal. Calcd for C<sub>13</sub>H<sub>20</sub>O<sub>4</sub>: C 65.00; H 8.33. Found: C 65.10; H 8.25.

IR of **1-3**, (cm<sup>-1</sup>)  $\nu$  1730 (C=O from CO<sub>2</sub>Et); 1775, 1755 (C=O from lactone); 1178, 1264 (C-O-C); 1640 (C=C); 3050 (C=CH).

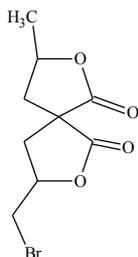
**Synthesis of 8-bromomethyl-2,7-dioxaspiro[4.4]nonane-1,6-diones 4-6 (general procedure).** 0.03 Mol of corresponding ethyl 3-allyl-tetrahydro-2-oxofuran-3-carboxylate **1-3** was dissolved in 15 ml of CCl<sub>4</sub> and solution of 4.8 g (0.03 mol) Br<sub>2</sub> in 5 mL of CCl<sub>4</sub> was added dropwise at room temperature keeping the reaction mixture colorless. Reaction mixture was additionally stirred for 15 min, volatiles were evaporated at reduced pressure. After that cyclization was carried out at 15-20 Torr and the residue was distilled to give a final product.

**8-Bromomethyl-3,3-dimethyl-2,7-dioxaspiro[4.4]nonane-1,6-dione (4).**



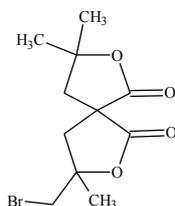
White powder, (83% yield); b.p. 170 °C/2 Torr, R<sub>f</sub>=0.63 (EtOH:C<sub>6</sub>H<sub>6</sub>:hexane 3:3:10), m.p. 100-101 °C (EtOH-H<sub>2</sub>O, 4:1); <sup>1</sup>H NMR (400.1 MHz, CDCl<sub>3</sub>)  $\delta$  1.47 (s, 3H, CH<sub>3</sub>), 1.59 (s, 3H, CH<sub>3</sub>), 2.11-2.24 (m, 1.6H), 2.51 (dd,  $J = 13.8$  Hz,  $J = 7.8$  Hz, 0.4H, minor), 2.72-2.83 (m, 1.4H), 2.90 (dd,  $J = 13.1$  Hz,  $J = 6.5$  Hz, 0.6H, major), 3.56-3.69 (m, 2H), 4.70-4.78 (m, 0.4H, minor), 5.04-5.09 (m, 0.6H, major). <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100.6 MHz)  $\delta$  28.6 (CH<sub>3</sub>), 29.3 (CH<sub>3</sub>), 33.1, 39.5, 44.6, 54.2 (C<sub>q</sub>), 75.6 (CH), 84.3 (C<sub>q</sub>), 172.9 (C=O), 173.1 (C=O) (major); 28.5 (CH<sub>3</sub>), 29.4 (CH<sub>3</sub>), 32.0, 38.9, 46.2, 53.3 (C<sub>q</sub>), 76.8 (CH), 84.1 (C<sub>q</sub>), 173.4 (C=O), 173.7 (C=O) (minor). Anal. Calcd for C<sub>10</sub>H<sub>13</sub>O<sub>4</sub>Br: C 43.34; H 4.73; Br 28.83. Found: C 43.25; H 4.84; Br 29.00.

**3-Bromomethyl-8-methyl-2,7-dioxaspiro[4.4]nonane-1,6-dione (5).**



Yield 86%; b.p. 167 °C /1 Torr; R<sub>f</sub>=0.60 (EtOH:C<sub>6</sub>H<sub>6</sub>:hexane 3:3:10); m.p. 58-60 °C (EtOH-H<sub>2</sub>O – 1:1); <sup>1</sup>H NMR (300 MHz, DMSO-*d*<sub>6</sub>)  $\delta$  1.35-1.53 (m, CH<sub>3</sub>, 3H), 1.96-2.12 (m, CH<sub>2</sub>, 1H), 2.12-2.28 (m, CH<sub>2</sub>, 0.5H), 2.36-2.70 (m, CH<sub>2</sub>, 1H), 2.75-2.99 (m, CH<sub>2</sub>, 1.5H), 3.61-3.84 (m, CH<sub>2</sub>Br, 2H), 4.70-5.09 (m, CHO, 2H). <sup>13</sup>C NMR (75 MHz, DMSO-*d*<sub>6</sub>)  $\delta$  20.0, 20.3, 32.9, 33.8, 35.6, 35.9, 39.1, 40.0, 52.9, 53.0, 74.6, 75.2, 76.3, 76.8, 172.2, 172.6, 172.7. Anal. Calcd for C<sub>9</sub>H<sub>11</sub>O<sub>4</sub>Br: C 41.06; H 4.18; Br 30.42. Found: C 41.20; H 4.15; Br 30.55.

**3-Bromomethyl-3,8,8-trimethyl-2,7-dioxaspiro[4.4]nonane-1,6-dione (6).**



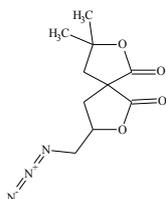
Yield 71%, b.p. 174 °C /1 Torr, R<sub>f</sub>=0.59 (EtOH:C<sub>6</sub>H<sub>6</sub>:hexane 3:3:10), m.p. 89-91 °C (EtOH-H<sub>2</sub>O – 1:1); <sup>1</sup>H NMR (300 MHz, DMSO-*d*<sub>6</sub>)  $\delta$  1.43-1.51 (m, CH<sub>3</sub>, 3H), 1.53-1.59 (m, CH<sub>3</sub>, 3H), 1.59-1.74 (m, CH<sub>3</sub>, 3H),

2.24-2.43 (m, CH<sub>2</sub>, 1.5H), 2.46-2.56 (m, CH<sub>2</sub>, 0.5H), 2.62-2.81 (m, CH<sub>2</sub>, 1.5H), 2.85-3.02 (m, CH<sub>2</sub>, 0.5H), 3.64-3.90 (m, CH<sub>2</sub>Br, 2H). <sup>13</sup>C NMR (75 MHz, DMSO-*d*<sub>6</sub>) δ 25.0, 25.1, 27.98, 28.01, 28.4, 28.5, 38.6, 40.8, 43.0, 43.4, 46.07, 46.10, 46.3, 54.1, 54.4, 82.1, 82.4, 83.0, 83.1, 172.7, 173.0, 173.2. Anal. Calcd for C<sub>11</sub>H<sub>15</sub>O<sub>4</sub>Br: C 45.36; H 5.16; Br 27.49. Found: C 45.25; H 5.30; Br 27.60.

IR of **4-6**, (cm<sup>-1</sup>) ν 1775, 1755 (C=O from lactone); 1264, 1178 (C-O-C); 665 (C-Br).

**Synthesis of 8-azidomethyl-3,3-dimethyl-2,7-dioxaspiro[4.4]nonane-1,6-dione (7).** A 3 ml vial with a screw cup was charged with 0.139 g (0.5 mmol) of 8-bromomethyl-3,3-dimethyl-2,7-dioxaspiro[4.4]nonane-1,6-dione **4**, 0.044 g (0.6 mmol) of NaN<sub>3</sub> and 1 ml of DMSO and stirred for 12 h at room temperature. Reaction mixture was poured into 30 ml of water, extracted with CH<sub>2</sub>Cl<sub>2</sub> (3×10 ml). Combined extracts were dried over Na<sub>2</sub>SO<sub>4</sub>, volatiles were evaporated and the residue was passed through a short silica gel pad in hexane-CH<sub>2</sub>Cl<sub>2</sub> 1:1 to give pure product.

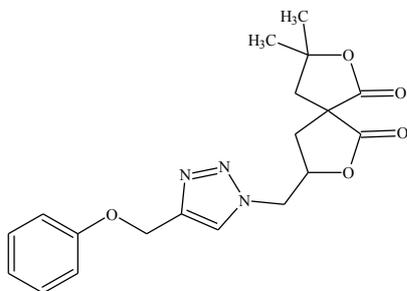
**8-Azidomethyl-3,3-dimethyl-2,7-dioxaspiro[4.4]nonane-1,6-dione (7).**



White solid, (102 mg, 85% yield); m.p. 45-47 °C; R<sub>f</sub>=0.39 (CH<sub>2</sub>Cl<sub>2</sub>); IR (cm<sup>-1</sup>) ν 1749 (O-C=O), 2090 (N<sub>3</sub>); <sup>1</sup>H NMR (400.1 MHz, CDCl<sub>3</sub>) δ 1.46 (s, 3H, CH<sub>3</sub>), 1.57 (s, 3H, CH<sub>3</sub>), 2.12-2.24 (m, 1.6H), 2.43 (dd, *J* = 13.6 Hz, *J* = 7.5 Hz, 0.4H, minor), 2.64-2.80 (m, 2H), 3.45 (dd, *J* = 13.6 Hz, *J* = 4.5 Hz, 0.6H, major), 3.57 (dd, *J* = 13.2 Hz, *J* = 4.7 Hz, 0.4H, minor), 3.70-3.76 (m, 1H), 4.62-4.68 (m, 0.4H, minor), 4.96-5.00 (m, 0.6H, major). <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100.6 MHz) δ 28.5 (CH<sub>3</sub>), 29.1 (CH<sub>3</sub>), 37.2, 44.6, 52.7, 53.9 (C<sub>q</sub>), 76.1 (CH), 84.3 (C<sub>q</sub>), 173.1 (C=O), 173.2 (C=O) (major); 28.3 (CH<sub>3</sub>), 29.2 (CH<sub>3</sub>), 37.4, 45.8, 53.1 (C<sub>q</sub>), 53.5, 76.6 (CH), 84.0 (C<sub>q</sub>), 173.5 (C=O), 173.8 (C=O) (minor). HRMS (ESI): *m/z* calcd for C<sub>10</sub>H<sub>13</sub>N<sub>3</sub>O<sub>4</sub>Na<sup>+</sup> [*M*+Na<sup>+</sup>]: 262.0798; found: 262.0799.

**Synthesis of triazoles 8-16 (general procedure).** A 3 ml vial with a screw cup was charged with 0.139 g (0.5 mmol) of appropriate 8-bromomethyl-2,7-dioxaspiro[4.4]nonane-1,6-dione **4-6**, 0.044 g (0.6 mmol) of NaN<sub>3</sub> and 1 ml of DMSO and stirred for 12 h at room temperature. Next, 0.071g (0.55 mmol) of (*i*-Pr)<sub>2</sub>Net, 0.0096 g (0.05 mmol) of CuI and 0.6 mmol of corresponding alkyne were added and heated for 5 h at 65 °C. Reaction mixture was poured into 30 ml of 0.1 M HCl and extracted with CH<sub>2</sub>Cl<sub>2</sub> (3×10 ml). Combined extracts were dried over Na<sub>2</sub>SO<sub>4</sub>, volatiles were evaporated and the residue was purified via column chromatography on silica gel using mixture of CH<sub>2</sub>Cl<sub>2</sub> and MeOH (30:1) as an eluent.

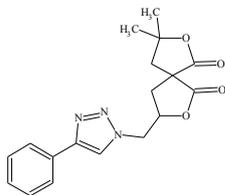
**3,3-Dimethyl-8-[(4-phenoxyethyl-1*H*-1,2,3-triazol-1-yl)methyl]-2,7-dioxaspiro[4.4]nonane-1,6-dione (8).**



White powder, (180 mg, 97% yield); m.p. 126-128 °C; R<sub>f</sub>=0.25 (CH<sub>2</sub>Cl<sub>2</sub>:MeOH 30:1); IR (cm<sup>-1</sup>) ν 1749, 1772 (O-C=O); <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400.1 MHz,) δ 1.38 (s, 2H, CH<sub>3</sub>, major), 1.43 (s, 1H, CH<sub>3</sub>, minor), 1.49 (s, 2H, CH<sub>3</sub>, major), 1.55 (s, 1H, CH<sub>3</sub>, minor), 1.89 (d, *J* = 13.7 Hz, 0.67H), 2.04-2.19 (m, 1H), 2.51-2.69 (m, 1.7H), 2.84 (dd, *J* = 13.3 Hz, *J* = 6.4 Hz, 0.67H), 4.59 (dd, *J* = 14.9 Hz, *J* = 5.4 Hz, 0.67H),

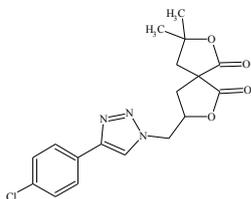
4.70-4.77 (m, 1.1H), 4.94-5.01 (m, 0.33H, minor), 5.14 (s, 2H, PhOCH<sub>2</sub>), 5.16-5.23 (m, 0.67H, major), 6.91-6.96 (m, 3H, Ph), 7.23-7.27 (m, 2H, Ph), 7.86-7.88 (m, 2H, Ph), 7.77 (s, 0.67H, C<sub>2</sub>N<sub>3</sub>H, major), 8.62 (s, 0.33H, C<sub>2</sub>N<sub>3</sub>H, minor). <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100.6 MHz) δ 28.4 (CH<sub>3</sub>), 28.8 (CH<sub>3</sub>), 36.9, 43.9, 51.8, 53.1 (C<sub>q</sub>), 61.3 (PhOCH<sub>2</sub>), 75.6 (CH), 84.4 (C<sub>q</sub>), 114.6, 121.1, 124.3 (C=CH-N), 129.4, 144.4 (C=CH-N), 157.9 (C<sub>q</sub>, Ph), 172.7 (C=O), 172.9 (C=O) (major); 28.3 (CH<sub>3</sub>), 29.0 (CH<sub>3</sub>), 37.1, 45.4, 53.0 (C<sub>q</sub>), 53.8, 61.4 (PhOCH<sub>2</sub>), 76.2 (CH), 84.3 (C<sub>q</sub>), 144.1 (C=CH-N), 158.0 (C<sub>q</sub>, Ph), 173.6 (C=O) (minor). Anal. Calcd for C<sub>19</sub>H<sub>21</sub>N<sub>3</sub>O<sub>5</sub>: C 61.45; H 5.70; N 11.31. Found: C 61.44; H 5.73; N 11.15.

**3,3-Dimethyl-8-[(4-phenyl-1H-1,2,3-triazol-1-yl)methyl]-2,7-dioxaspiro[4.4]nonane-1,6-dione (9).**

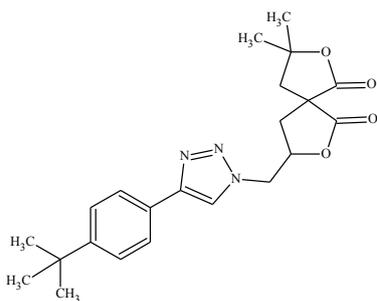


White powder, (158 mg, 93% yield); m.p. 159-162 °C; R<sub>f</sub>=0.29 (CH<sub>2</sub>Cl<sub>2</sub>:MeOH 30:1); IR (cm<sup>-1</sup>) ν 1751, 1772 (O-C=O); <sup>1</sup>H NMR (DMSO-*d*<sub>6</sub>, 400.1 MHz,) δ 1.43 (s, 3H, CH<sub>3</sub>), 1.48 (s, 3H, CH<sub>3</sub>), 2.25 (d, *J* = 13.8 Hz, 0.67H), 2.36-2.41 (m, 1H), 2.56-2.75 (m, 1.6H), 3.02 (dd, *J* = 13.3 Hz, *J* = 6.7 Hz, 0.67H), 4.73-4.89 (m, 2H), 5.07-5.13 (m, 0.3H, minor), 5.14-5.21 (m, 0.7H, major), 7.32-7.36 (m, 1H, Ph), 7.43-7.47 (m, 2H, Ph), 7.86-7.88 (m, 2H, Ph), 8.61 (s, 0.3H, C<sub>2</sub>N<sub>3</sub>H, minor), 8.62 (s, 0.7H, C<sub>2</sub>N<sub>3</sub>H, major). <sup>13</sup>C NMR (DMSO-*d*<sub>6</sub>, 100.6 MHz) δ 28.3 (CH<sub>3</sub>), 28.4 (CH<sub>3</sub>), 36.5, 43.7, 52.3, 52.4 (C<sub>q</sub>), 76.5 (CH), 84.5 (C<sub>q</sub>), 122.0 (C=CH-N), 125.2, 127.9, 128.9, 130.5 (C<sub>q</sub>, Ph), 146.5 (C=CH-N), 173.4 (C=O), 173.6 (C=O) (major); 28.1 (CH<sub>3</sub>), 28.5 (CH<sub>3</sub>), 36.8, 44.4, 53.5 (C<sub>q</sub>), 53.7, 76.8 (CH), 84.2 (C<sub>q</sub>), 122.1 (C=CH-N), 125.2, 127.9, 128.9, 130.5 (C<sub>q</sub>, Ph), 146.5 (C=CH-N), 173.8 (C=O), 174.5 (C=O) (minor). Anal. Calcd for C<sub>18</sub>H<sub>19</sub>N<sub>3</sub>O<sub>4</sub>: C 63.33; H 5.61; N 12.31. Found: C 63.57; H 5.68; N 12.03.

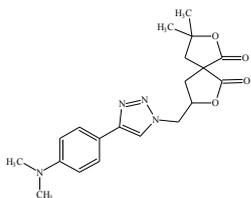
**8-[[4-(4-Chlorophenyl)-1H-1,2,3-triazol-1-yl]methyl]-3,3-dimethyl-2,7-dioxaspiro[4.4]nonane-1,6-dione (10).**



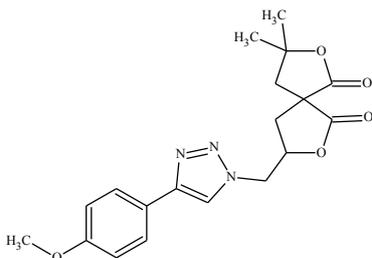
White powder, (178 mg, 95% yield); m.p. 183-184 °C; R<sub>f</sub>=0.43 (CH<sub>2</sub>Cl<sub>2</sub>:MeOH 30:1); IR (cm<sup>-1</sup>) ν 1751, 1776 (O-C=O); <sup>1</sup>H NMR (acetone-*d*<sub>6</sub>, 400.1 MHz,) δ 1.47 (s, 1.8H, CH<sub>3</sub>, major), 1.49 (s, 1.2H, CH<sub>3</sub>, minor), 1.52 (s, 1.8H, CH<sub>3</sub>, major), 1.55 (s, 1.2H, CH<sub>3</sub>, minor), 2.29 (d, *J* = 13.8 Hz, 0.6H), 2.43-2.52 (m, 1.4H), 2.64-2.88 (m, 1.4H), 3.09 (dd, *J* = 13.4 Hz, *J* = 6.5 Hz, 0.6H), 4.81-4.97 (m, 2H), 5.12-5.18 (m, 0.4H, minor), 5.25-5.32 (m, 0.6H, major), 7.44-7.49 (m, 2H, Ar), 7.91-7.94 (m, 2H, Ar), 8.45 (s, 0.6H, C<sub>2</sub>N<sub>3</sub>H, major), 8.46 (s, 0.4H, C<sub>2</sub>N<sub>3</sub>H, minor). <sup>13</sup>C NMR (acetone-*d*<sub>6</sub>, 100.6 MHz) δ 28.9 (CH<sub>3</sub>), 29.0 (CH<sub>3</sub>), 38.0, 44.8, 53.3, 54.9 (C<sub>q</sub>), 77.3 (CH), 84.8 (C<sub>q</sub>), 122.6 (C=CH-N), 127.9, 129.7, 130.8 (C<sub>q</sub>, Ph), 133.9 (C<sub>q</sub>, Ph), 147.1 (C=CH-N), 174.1 (C=O), 174.2 (C=O) (major); 28.7 (CH<sub>3</sub>), 29.2 (CH<sub>3</sub>), 38.1, 45.0, 53.9, 54.5 (C<sub>q</sub>), 77.6 (CH), 84.7 (C<sub>q</sub>), 122.7 (C=CH-N), 127.9, 129.7, 130.9 (C<sub>q</sub>, Ph), 133.8 (C<sub>q</sub>, Ph), 146.9 (C=CH-N), 174.7 (C=O), 175.1 (C=O) (minor). Anal. Calcd for C<sub>18</sub>H<sub>18</sub>ClN<sub>3</sub>O<sub>4</sub>: C 57.53; H 4.83; N 11.18. Found: C 57.52; H 4.95; N 11.14.

**8-[[4-(4-*tert*-Butylphenyl)-1*H*-1,2,3-triazol-1-yl]methyl]-3,3-dimethyl-2,7-dioxaspiro[4.4]nonane-1,6-dione****(11).**White powder, (183 mg, 92% yield); m.p. 129-132 °C;  $R_f=0.25$ 

(CH<sub>2</sub>Cl<sub>2</sub>:MeOH 30:1); <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400.1 MHz,)  $\delta$  1.33 (s, 9H, C(CH<sub>3</sub>)<sub>3</sub>), 1.41 (s, 2.1H, CH<sub>3</sub>, major), 1.46 (s, 0.9H, CH<sub>3</sub>, minor), 1.51 (s, 2.1H, CH<sub>3</sub>, major), 1.56 (s, 0.9H, CH<sub>3</sub>, minor), 1.92 (d,  $J = 13.6$  Hz, 0.7H), 2.07-2.20 (m, 1H), 2.58 (d,  $J = 13.4$  Hz, 1H), 2.67-2.75 (m, 0.8H), 2.82-2.93 (m, 0.8H), 4.65-4.95 (m, 2H), 5.02-5.12 (m, 0.33H, minor), 5.24-5.34 (m, 0.67H, major), 7.44 (br s, 2H, Ar), 7.80 (br s, 2H, Ar), 8.10 (br s, 1H, C<sub>2</sub>N<sub>3</sub>H). <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100.6 MHz)  $\delta$  28.5 (CH<sub>3</sub>), 29.1 (CH<sub>3</sub>), 31.2 (C(CH<sub>3</sub>)<sub>3</sub>), 34.6 (C(CH<sub>3</sub>)<sub>3</sub>), 37.1, 44.1, 51.8, 53.0 (C<sub>q</sub>), 75.5 (CH), 84.5 (C<sub>q</sub>), 125.4, 125.6 (C=CH-N), 125.8 127.2 (C<sub>q</sub>, Ar), 131.6 (C<sub>q</sub>, Ar), 151.6 (C=CH-N), 172.7 (C=O), 172.9 (C=O) (major); 28.4 (CH<sub>3</sub>), 29.3 (CH<sub>3</sub>), 37.4, 45.8, 51.8 (C<sub>q</sub>), 53.9, 76.3 (CH), 84.4 (C<sub>q</sub>), 125.7 (C=CH-N), 151.5 (C=CH-N), 173.6 (C=O), 173.7 (C=O) (minor). Anal. Calcd for C<sub>22</sub>H<sub>27</sub>N<sub>3</sub>O<sub>4</sub>: C 66.48; H 6.85; N 10.57. Found: C 65.68; H 6.74; N 10.14.

**8-[[4-(4-Dimethylaminophenyl)-1*H*-1,2,3-triazol-1-yl]methyl]-3,3-dimethyl-2,7-dioxaspiro[4.4]nonane-1,6-dione (12).**White powder, (179 mg, 93% yield); m.p. 189-192 °C;  $R_f=0.22$  (CH<sub>2</sub>Cl<sub>2</sub>:MeOH 30:1); <sup>1</sup>H

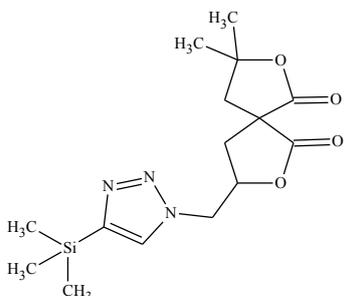
NMR (CDCl<sub>3</sub>, 400.1 MHz,)  $\delta$  1.40 (s, 2.1H, CH<sub>3</sub>, major), 1.46 (s, 0.9H, CH<sub>3</sub>, minor), 1.51 (s, 2.1H, CH<sub>3</sub>, major), 1.59 (s, 0.9H, CH<sub>3</sub>, minor), 1.91 (d,  $J = 13.7$  Hz, 0.7H), 2.07-2.18 (m, 1H), 2.51-2.86 (m, 2.3H), 2.97 (s, 6H, NMe<sub>2</sub>), 4.62-4.80 (m, 2H), 4.99-5.05 (m, 0.33H, minor), 5.23-5.28 (m, 0.67H, major), 6.77 (d,  $J = 8.2$  Hz, 2H, Ar), 7.67 (d,  $J = 8.2$  Hz, 2H, Ar), 7.76 (s, 0.67H, C<sub>2</sub>N<sub>3</sub>H, major), 7.79 (s, 0.33H, C<sub>2</sub>N<sub>3</sub>H, minor). <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100.6 MHz)  $\delta$  28.5 (CH<sub>3</sub>), 29.1 (CH<sub>3</sub>), 37.1, 40.5 (NMe<sub>2</sub>), 44.1, 51.5, 53.3 (C<sub>q</sub>), 76.6 (CH), 84.5 (C<sub>q</sub>), 112.6, 119.6 (C=CH-N), 126.7 148.8 (C<sub>q</sub>, Ar), 150.3 (C=CH-N), 172.3 (C=O), 172.9 (C=O) (major); 28.4 (CH<sub>3</sub>), 29.3 (CH<sub>3</sub>), 37.4, 45.8, 53.0 (C<sub>q</sub>), 53.9, 76.4 (CH), 84.4 (C<sub>q</sub>), 119.7 (C=CH-N), 150.2 (C=CH-N), 173.6 (C=O), 173.7 (C=O) (minor). Anal. Calcd for C<sub>20</sub>H<sub>24</sub>N<sub>4</sub>O<sub>4</sub>: C 62.49; H 6.29; N 14.57. Found: C 61.89; H 6.32; N 14.14.

**8-[[4-(4-Methoxyphenyl)-1*H*-1,2,3-triazol-1-yl]methyl]-3,3-dimethyl-2,7-dioxaspiro[4.4]nonane-1,6-dione (13).**White powder, (178 mg, 96% yield); m.p. 167-169 °C;  $R_f=0.30$ 

(CH<sub>2</sub>Cl<sub>2</sub>:MeOH 30:1); IR (cm<sup>-1</sup>)  $\nu$  1749 (O-C=O); <sup>1</sup>H NMR (DMSO-*d*<sub>6</sub>, 400.1 MHz,)  $\delta$  1.43 (s, 3H, CH<sub>3</sub>), 1.48 (s, 3H, CH<sub>3</sub>), 2.25 (d,  $J = 13.8$  Hz, 0.65H), 2.23-2.41 (m, 1H), 2.55-2.64 (m, 1.3H), 2.71 (dd,  $J = 13.5$  Hz,  $J = 7.1$  Hz, 0.35H),

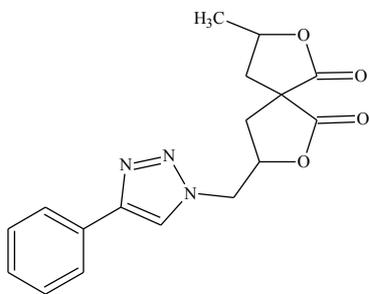
3.01 (dd,  $J = 13.2$  Hz,  $J = 6.6$  Hz, 0.65H), 3.78 (s, 3H, MeO), 4.71-4.83 (m, 2H), 5.06-5.11 (m, 0.35H, minor), 5.13-5.19 (m, 0.65H, major), 7.01 (d,  $J = 8.8$  Hz, 2H, Ar), 7.01 (d,  $J = 8.8$  Hz, 2H, Ar), 8.49 (s, 0.35H, C<sub>2</sub>N<sub>3</sub>H, minor), 8.50 (s, 0.65H, C<sub>2</sub>N<sub>3</sub>H, major). <sup>13</sup>C NMR (DMSO-*d*<sub>6</sub>, 100.6 MHz)  $\delta$  28.3 (CH<sub>3</sub>), 28.4 (CH<sub>3</sub>), 36.5, 43.7, 52.3, 52.5 (C<sub>q</sub>), 55.1 (MeO), 76.6 (CH), 84.5 (C<sub>q</sub>), 114.3, 121.0 (C=CH-N), 123.1 (C<sub>q</sub>, Ar), 126.6, 146.5 (C=CH-N), 159.1 (C<sub>q</sub>, Ph), 173.4 (C=O), 173.6 (C=O) (major); 28.1 (CH<sub>3</sub>), 28.5 (CH<sub>3</sub>), 36.8, 44.4, 53.5 (C<sub>q</sub>), 53.7, 76.9 (CH), 84.2 (C<sub>q</sub>), 122.1 (C=CH-N), 146.4 (C=CH-N), 173.8 (C=O), 174.5 (C=O) (minor). HRMS (ESI):  $m/z$  calcd for C<sub>19</sub>H<sub>21</sub>N<sub>3</sub>O<sub>5</sub>Na<sup>+</sup> [M+Na<sup>+</sup>]: 394.1373; found: 394.1371.

**3,3-Dimethyl-8-[(4-trimethylsilyl-1H-1,2,3-triazol-1-yl)methyl]-2,7-dioxaspiro[4.4]nonane-1,6-dione (14).**



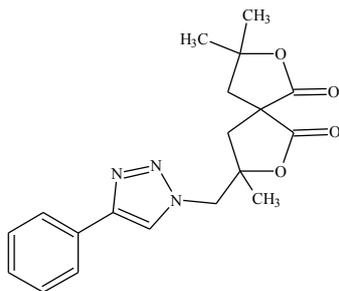
Slightly greenish oil, (165 mg, 98% yield);  $R_f=0.15$  (CH<sub>2</sub>Cl<sub>2</sub>:MeOH 30:1); IR (cm<sup>-1</sup>)  $\nu$  1749, 1780 (O=C=O); <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400.1 MHz)  $\delta$  0.23 (s, 9H, Si(CH<sub>3</sub>)<sub>3</sub>), 1.35 (s, 2H, CH<sub>3</sub>, major), 1.40 (s, 1H, CH<sub>3</sub>, minor), 1.45 (s, 2H, CH<sub>3</sub>, major), 1.51 (s, 1H, CH<sub>3</sub>, minor), 1.88 (d,  $J = 13.7$  Hz, 0.8H), 2.10-2.19 (m, 1.2H), 2.46-2.66 (m, 2H), 2.83-2.88 (m, 0.8H), 4.56-4.81 (m, 2H), 4.92-5.01 (m, 0.33H, minor), 5.12-5.22 (m, 0.67H, major), 7.66 (br s, 1H, C<sub>2</sub>N<sub>3</sub>H). <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100.6 MHz)  $\delta$  -1.4 (Si(CH<sub>3</sub>)<sub>3</sub>), 28.4 (CH<sub>3</sub>), 28.8 (CH<sub>3</sub>), 37.1, 43.9, 51.3, 53.0 (C<sub>q</sub>), 75.8 (CH), 84.3 (C<sub>q</sub>), 130.4 (C=CH-N), 146.8 (C=CH-N), 172.8 (C=O), 172.9 (C=O) (major); 28.3 (CH<sub>3</sub>), 29.0 (CH<sub>3</sub>), 37.2, 45.4, 52.6 (C<sub>q</sub>), 53.7, 76.5 (CH), 84.3 (C<sub>q</sub>), 130.3 (C=CH-N), 146.7 (C=CH-N), 173.6 (C=O), 173.7 (C=O) (minor). Anal. Calcd for C<sub>15</sub>H<sub>23</sub>N<sub>3</sub>O<sub>4</sub>Si: C, 53.39; H, 6.87; N, 12.45. Found: C 53.01; H 6.91; N 12.27.

**3-Methyl-8-[(4-phenyl-1H-1,2,3-triazol-1-yl)methyl]-2,7-dioxaspiro[4.4]nonane-1,6-dione (15).**



White powder, (154 mg, 94% yield); m.p. 179-181 °C;  $R_f=0.33$  (CH<sub>2</sub>Cl<sub>2</sub>:MeOH 30:1); <sup>1</sup>H NMR (DMSO-*d*<sub>6</sub>, 400.1 MHz)  $\delta$  1.35-1.37 (m, 3H, CH<sub>3</sub>), 2.03-2.16 (m, 1H), 2.30 (dd,  $J = 13.5$  Hz,  $J = 8.5$  Hz, 0.5H), 2.56-2.58 (m, 1H), 2.72 (dd,  $J = 13.2$  Hz,  $J = 6.5$  Hz, 0.5H), 2.87-2.94 (m, 1H), 4.74-4.92 (m, 3H), 5.10-5.16 (m, 0.5H), 5.17-5.24 (m, 0.5H), 7.32-7.35 (m, 1H, Ph), 7.43-7.47 (m, 2H, Ph), 7.85-7.87 (m, 2H, Ph), 8.60 (s, 0.5H, C<sub>2</sub>N<sub>3</sub>H), 8.62 (s, 0.5H, C<sub>2</sub>N<sub>3</sub>H). <sup>13</sup>C NMR (DMSO-*d*<sub>6</sub>, 100.6 MHz)  $\delta$  20.1 (CH<sub>3</sub>), 20.5 (CH<sub>3</sub>), 34.2, 34.3, 38.7, 52.48, 52.53, 52.9, 53.2, 75.3, 76.1, 76.9, 77.2, 122.0 (C=CH-N), 122.1 (C=CH-N), 125.17, 125.20, 127.92, 127.94, 128.9, 130.50 (C<sub>q</sub>, Ph), 130.52 (C<sub>q</sub>, Ph), 146.4 (C=CH-N), 146.5 (C=CH-N), 173.4 (C=O), 173.6 (C=O), 173.66 (C=O), 173.72 (C=O). Anal. Calcd for C<sub>17</sub>H<sub>17</sub>N<sub>3</sub>O<sub>4</sub>: C 62.38; H 5.23; N 12.84. Found: C 62.31; H 5.36; N 12.87.

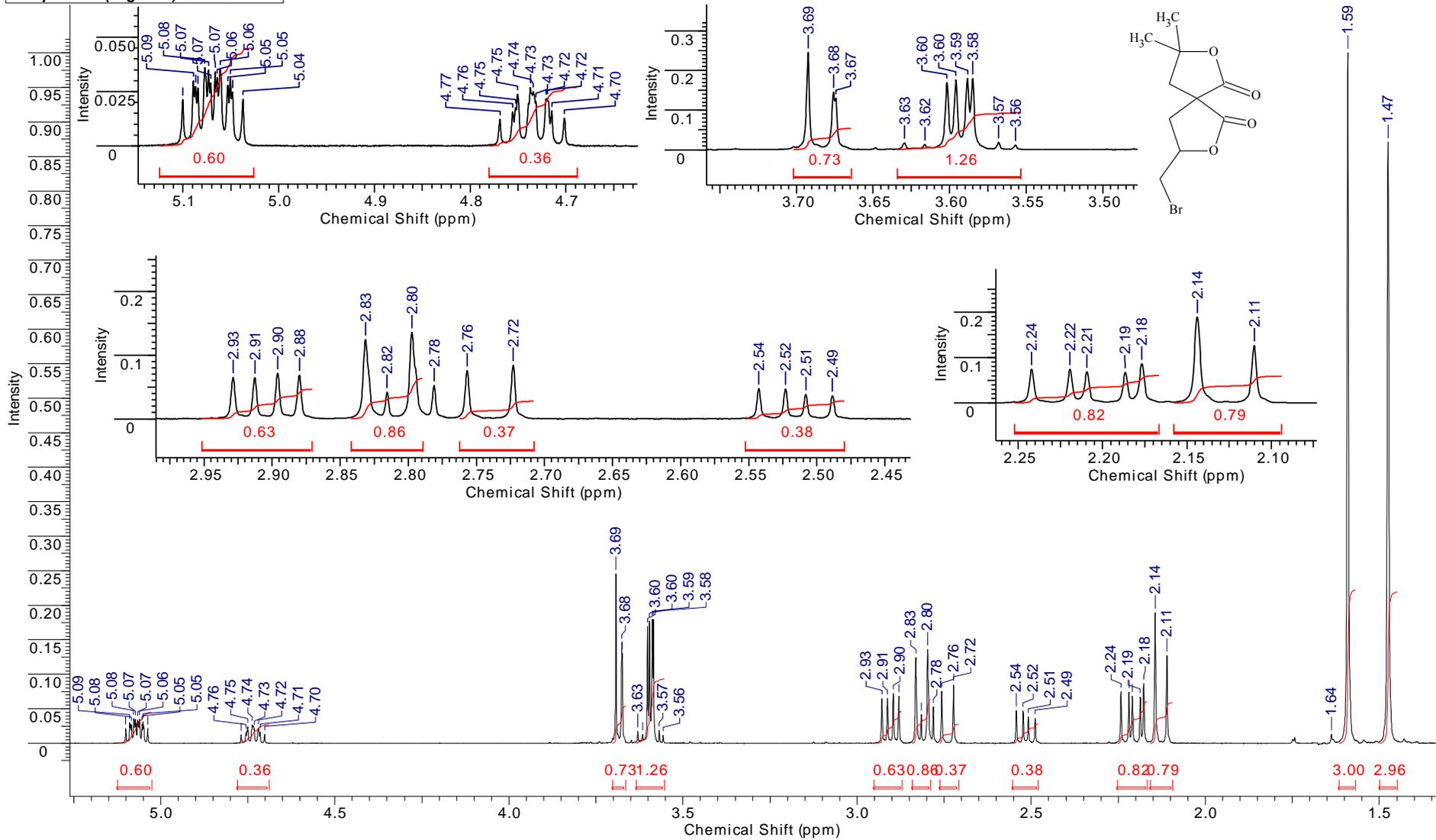
**3,3,8-Trimethyl-8-[(4-phenyl-1*H*-1,2,3-triazol-1-yl)methyl]-2,7-dioxaspiro[4.4]nonane-1,6-dione (16).**



Pale brownish viscous oil, (128 mg, 72% yield);  $R_f=0.37$  ( $\text{CH}_2\text{Cl}_2:\text{MeOH}$  30:1);  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400.1 MHz,)  $\delta$  1.31 (s, 1.8H,  $\text{CH}_3$ , major), 1.38 (s, 1.2H,  $\text{CH}_3$ , minor), 1.41 (s, 1.8H,  $\text{CH}_3$ , major), 1.44 (s, 1.2H,  $\text{CH}_3$ , minor), 1.58 (s, 1.2H,  $\text{CH}_3$ , minor), 1.65 (d,  $J = 13.7$  Hz, 0.7H), 1.69 (s, 1.8H,  $\text{CH}_3$ , major), 2.16-2.25 (m, 1.3H), 2.56 (d,  $J = 14.1$  Hz, 0.6H, major), 2.71 (d,  $J = 14.1$  Hz, 0.6H, major), 2.81 (d,  $J = 13.7$  Hz, 0.4H, minor), 2.95 (d,  $J = 13.7$  Hz, 0.4H, minor), 4.55 (d,  $J = 14.7$  Hz, 0.6H, major), 4.60 (d,  $J = 14.7$  Hz, 0.6H, major), 4.78 (d,  $J = 14.6$  Hz, 0.4H, minor), 4.90 (d,  $J = 14.6$  Hz, 0.4H, minor), 7.28-7.40 (m, 3H, Ph), 7.77-7.83 (m, 2H, Ph), 7.89 (s, 0.6H,  $\text{C}_2\text{N}_3\text{H}$ , major), 7.95 (s, 0.4H,  $\text{C}_2\text{N}_3\text{H}$ , minor).  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100.6 MHz)  $\delta$  25.2 ( $\text{CH}_3$ ), 28.2 ( $\text{CH}_3$ ), 28.9 ( $\text{CH}_3$ ), 42.0, 46.1, 54.7, 57.5, 82.9, 84.2, 121.5 ( $\text{C}=\text{CH}-\text{N}$ ), 125.72, 128.5, 128.9, 129.7 ( $\text{C}_q$ , Ph), 148.3 ( $\text{C}=\text{CH}-\text{N}$ ), 173.3 ( $\text{C}=\text{O}$ ), 173.8 ( $\text{C}=\text{O}$ ) (major); 24.9 ( $\text{CH}_3$ ), 28.3 ( $\text{CH}_3$ ), 29.2 ( $\text{CH}_3$ ), 44.0, 47.0, 54.4, 57.2, 83.1, 84.4, 121.3 ( $\text{C}=\text{CH}-\text{N}$ ), 125.70, 128.2, 128.8, 130.1 ( $\text{C}_q$ , Ph), 148.0 ( $\text{C}=\text{CH}-\text{N}$ ), 173.2 ( $\text{C}=\text{O}$ ), 173.84 ( $\text{C}=\text{O}$ ) (minor). Anal. Calcd for  $\text{C}_{19}\text{H}_{21}\text{N}_3\text{O}_4$ : C 64.21; H 5.96; N 11.82. Found: C 63.84; H 6.00; N 11.76.

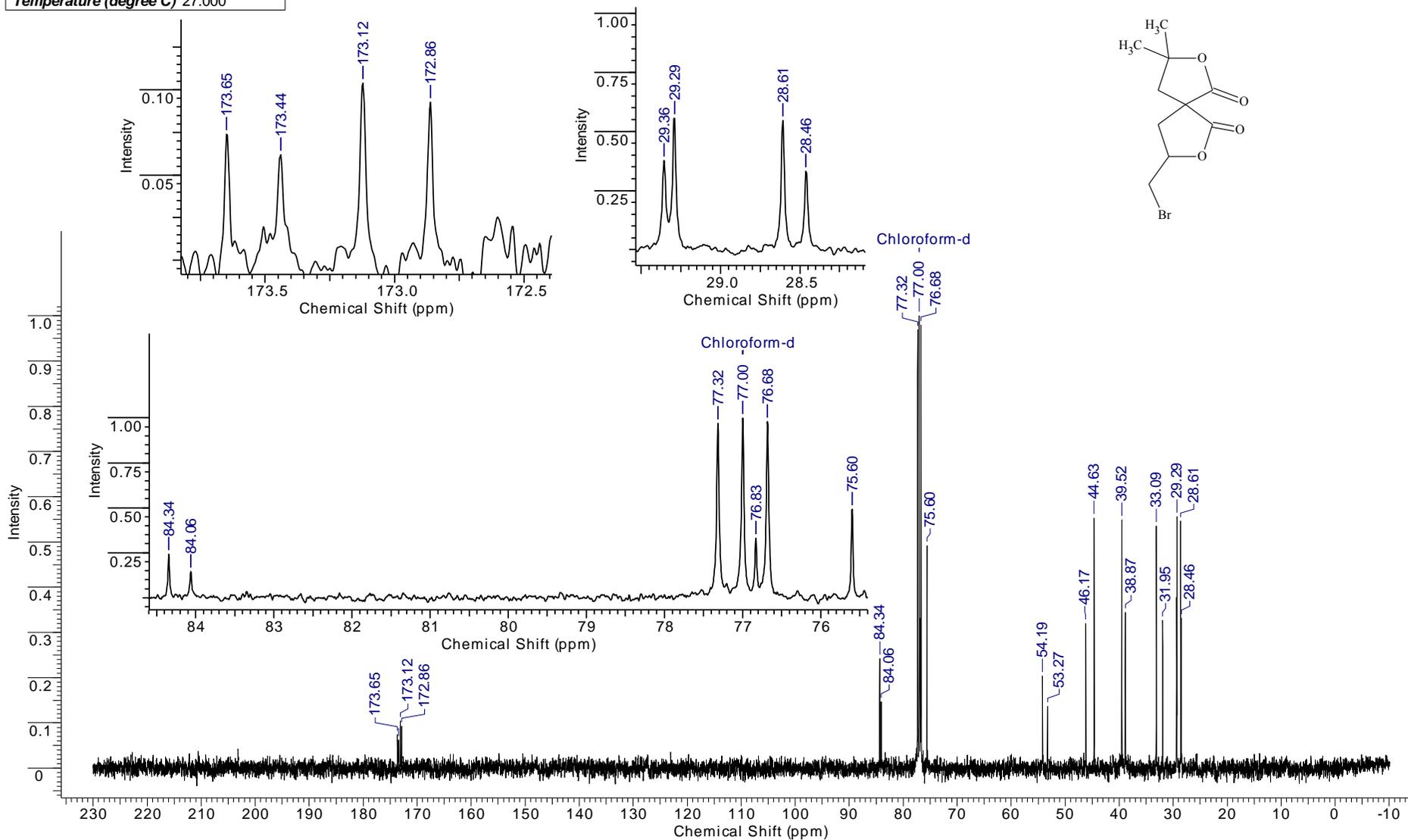
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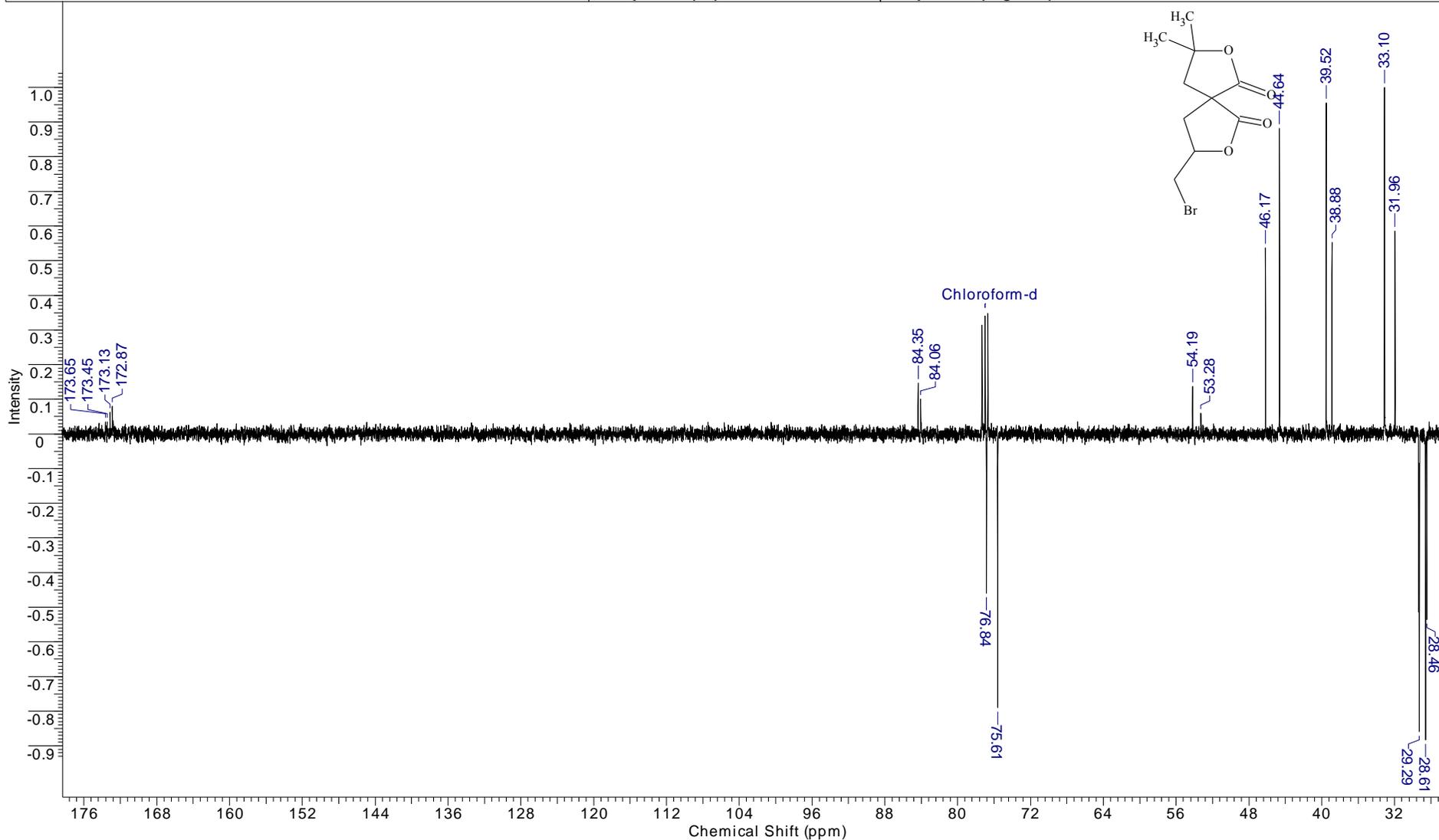
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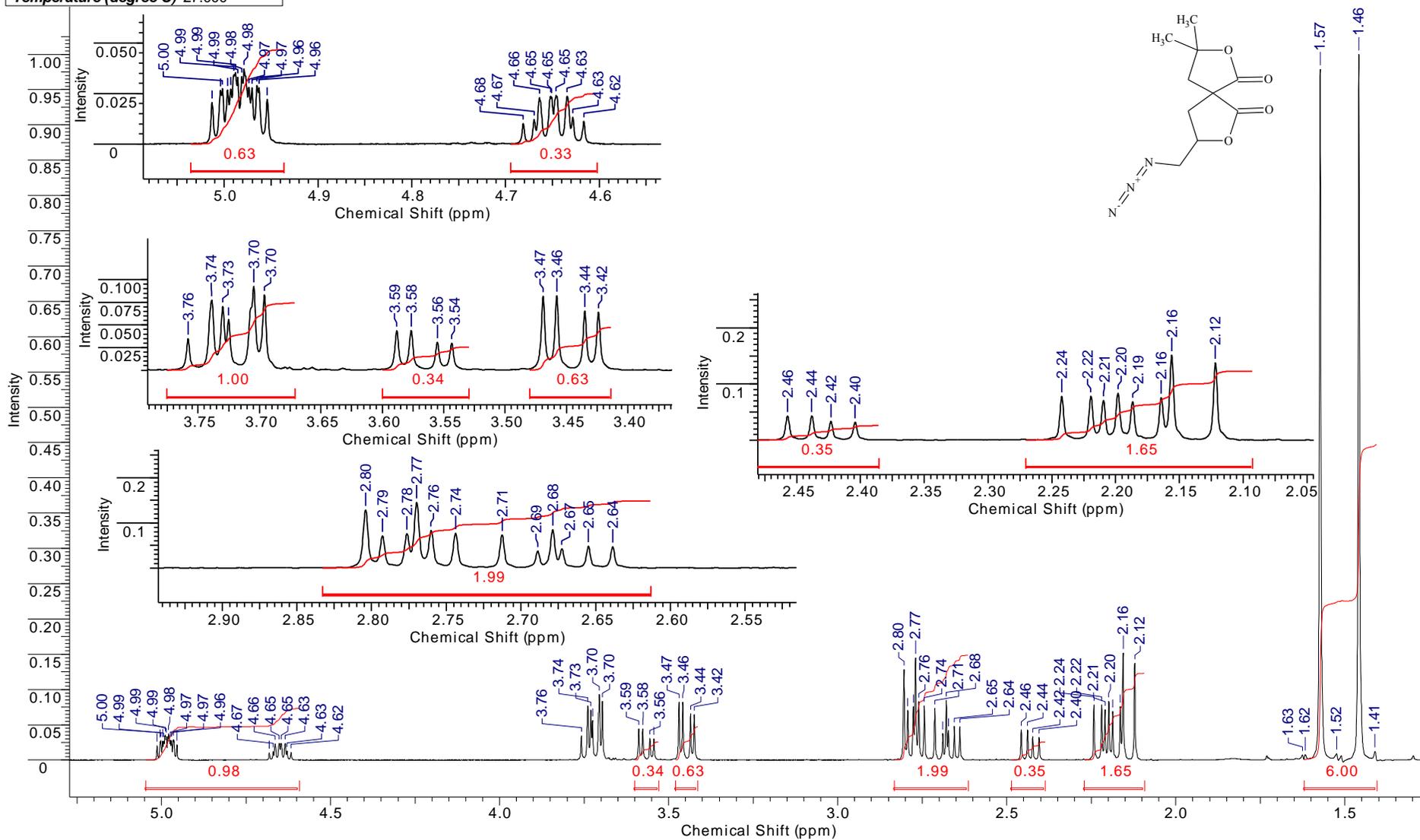
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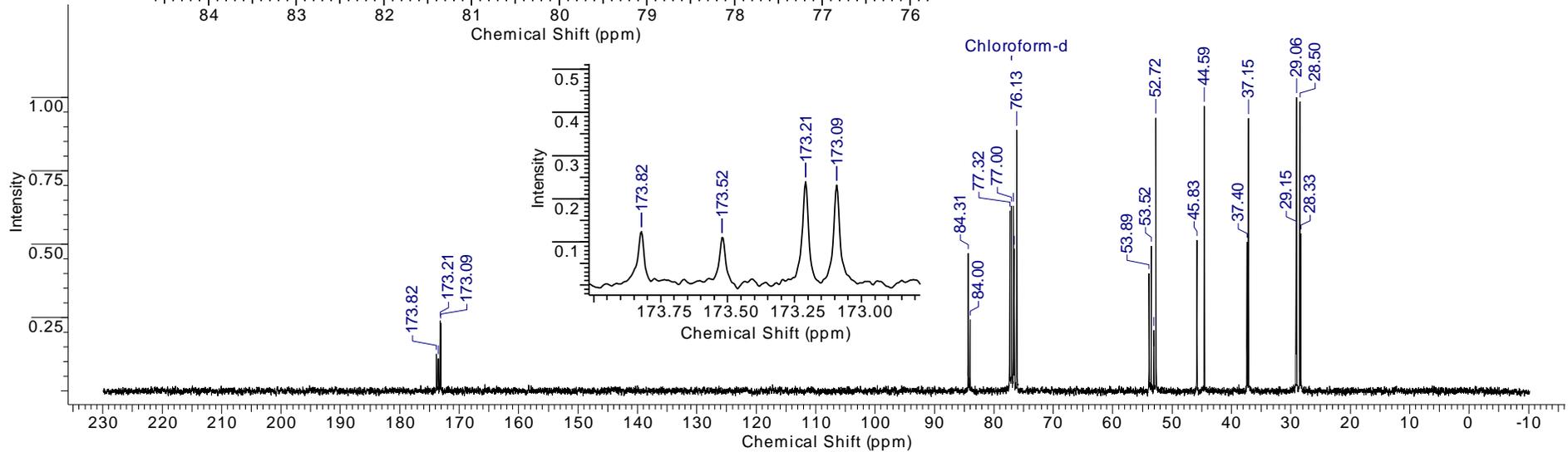
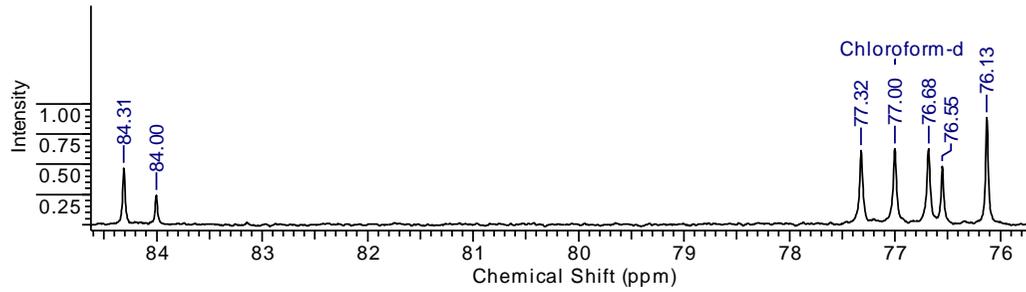
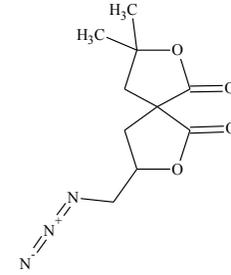
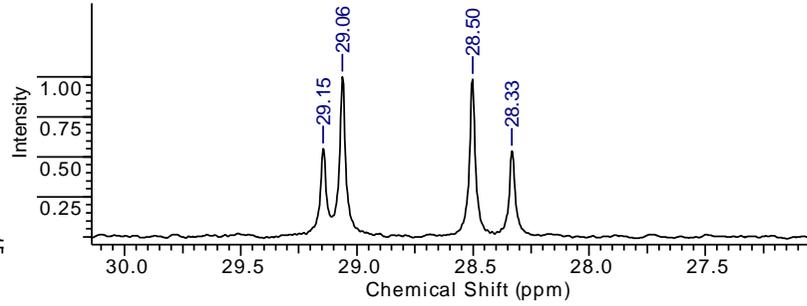
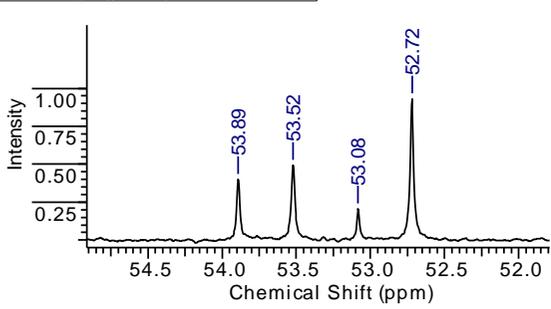
FW 239.2280 Formula C<sub>10</sub>H<sub>13</sub>N<sub>3</sub>O<sub>4</sub>

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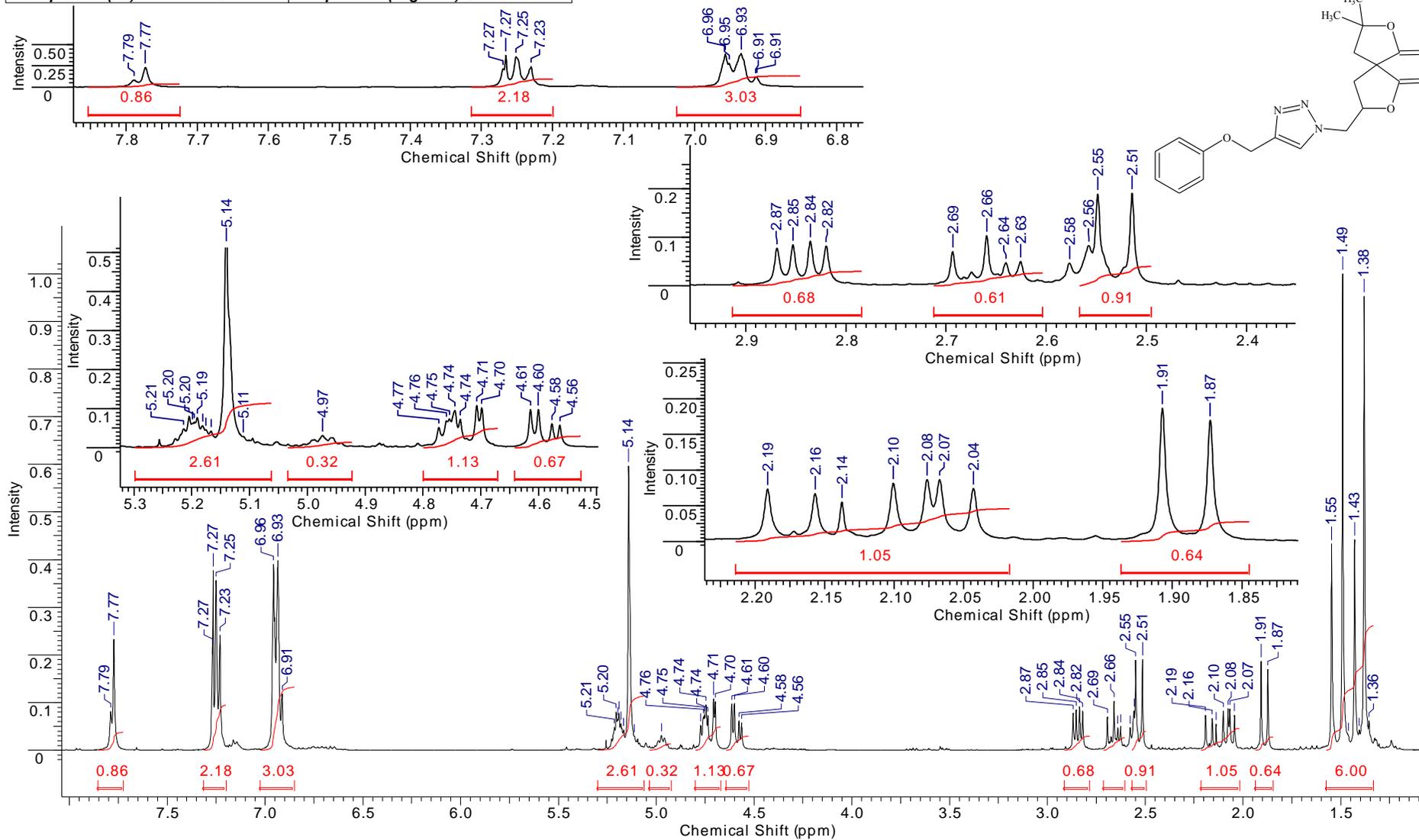
**FW** 239.2280 **Formula** C<sub>10</sub>H<sub>13</sub>N<sub>3</sub>O<sub>4</sub>

<b>Acquisition Time (sec)</b>	0.4999	<b>Comment</b>	Imported from UXNMR.		<b>Date</b>	06 Jun 2015 19:17:26			
<b>File Name</b>	D:\BN\output\2015\06.июнь\BM-791.C_002001r		<b>Frequency (MHz)</b>	100.61	<b>Nucleus</b>	13C	<b>Number of Transients</b>	245	
<b>Original Points Count</b>	12076	<b>Points Count</b>	65536	<b>Pulse Sequence</b>	zgpg30	<b>Solvent</b>	DMSO-D6	<b>Sweep Width (Hz)</b>	24154.59
<b>Temperature (degree C)</b>	27.000								



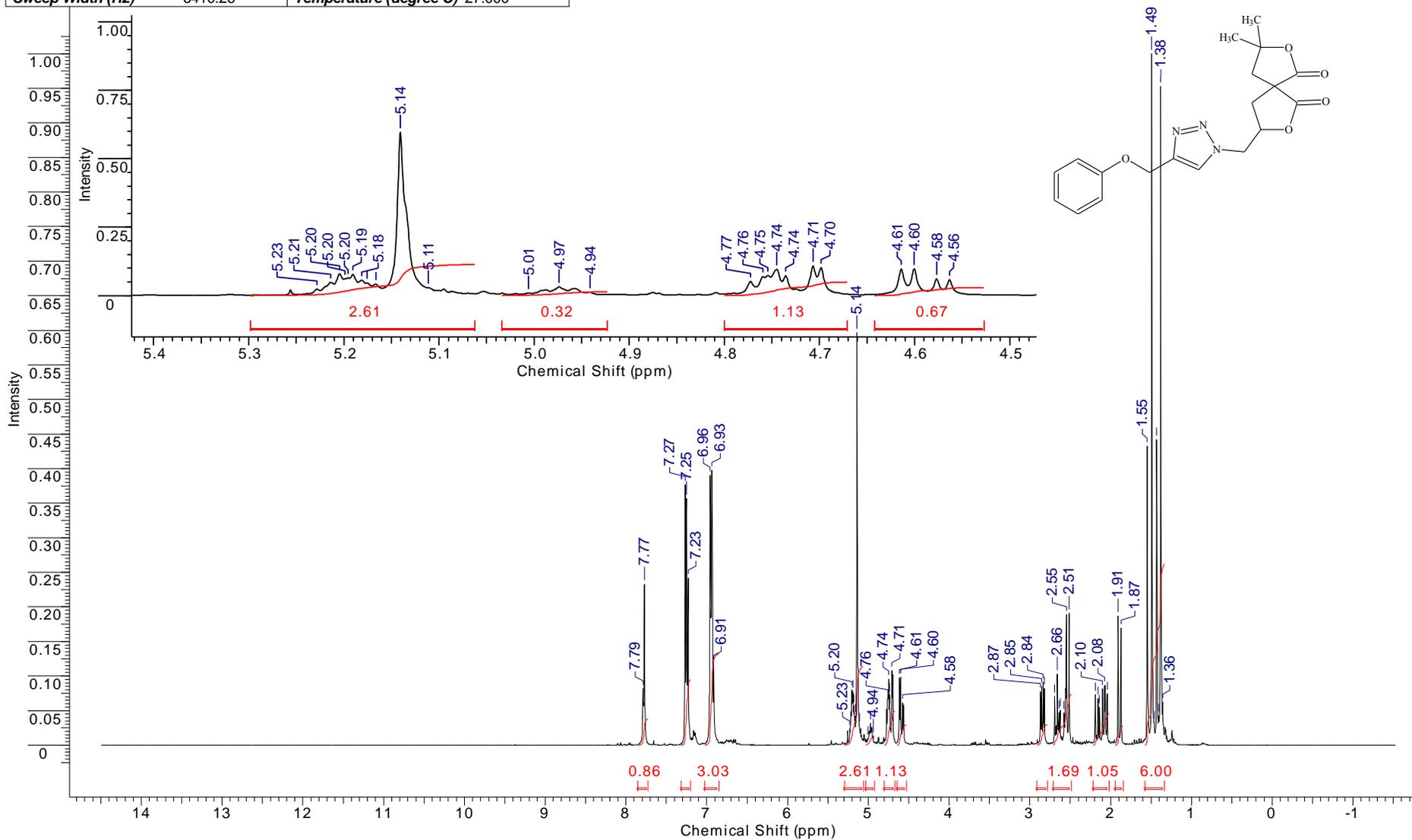
FW 371.3873 Formula C<sub>19</sub>H<sub>21</sub>N<sub>3</sub>O<sub>5</sub>

Acquisition Time (sec)	2.5559	Comment	Imported from UXNMR.	Date	11 Jun 2015 19:42:00
File Name	D:\BN\output\2015\06.июнь\BM-797.H_001001r	Frequency (MHz)	400.13	Nucleus	<sup>1</sup> H
Original Points Count	16384	Points Count	65536	Pulse Sequence	zg30
Sweep Width (Hz)	6410.26	Temperature (degree C)	27.000	Solvent	CHLOROFORM-D
				Number of Transients	5



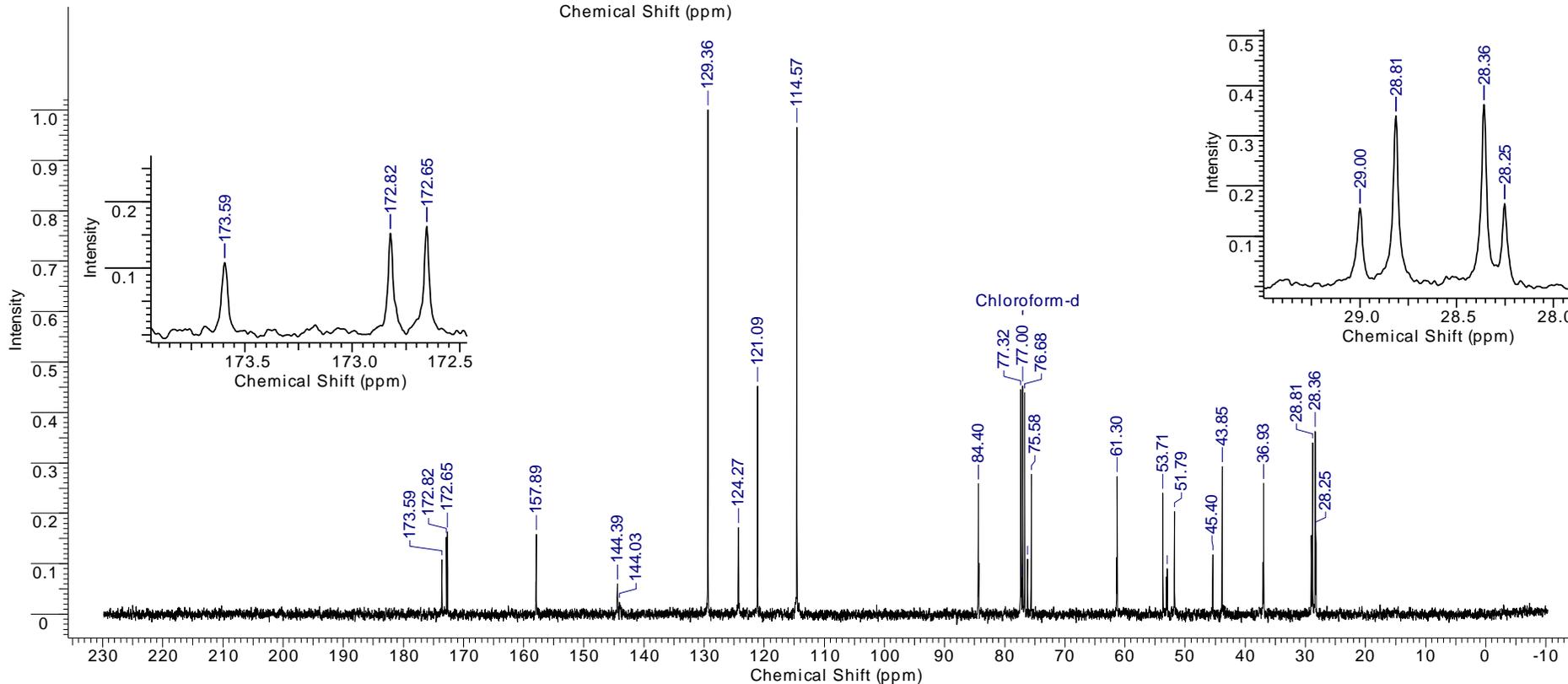
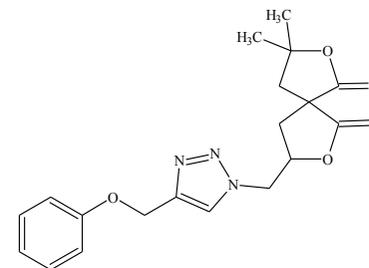
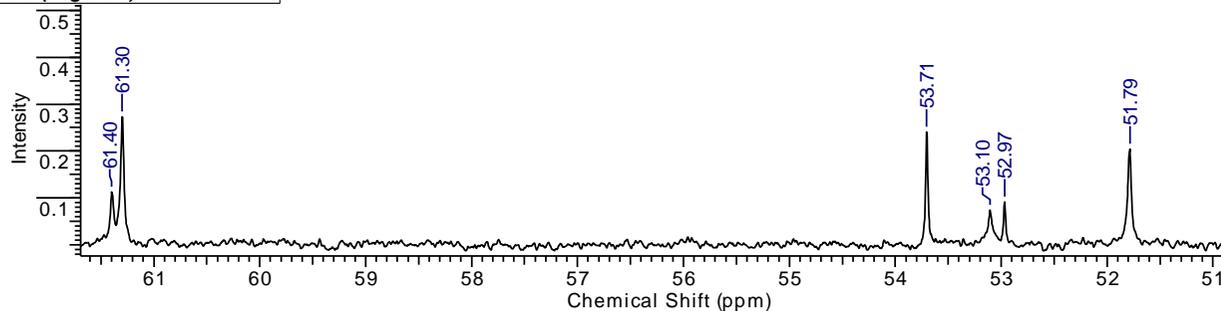
FW 371.3873 Formula C<sub>19</sub>H<sub>21</sub>N<sub>3</sub>O<sub>5</sub>

Acquisition Time (sec)	2.5559	Comment	Imported from UXMNR.		Date	11 Jun 2015 19:42:00	
File Name	D:\BN\output\2015\06.июнь\BM-797.H_001001r	Frequency (MHz)	400.13	Nucleus	1H	Number of Transients	5
Original Points Count	16384	Points Count	65536	Pulse Sequence	zg30	Solvent	CHLOROFORM-D
Sweep Width (Hz)	6410.26	Temperature (degree C)	27.000				



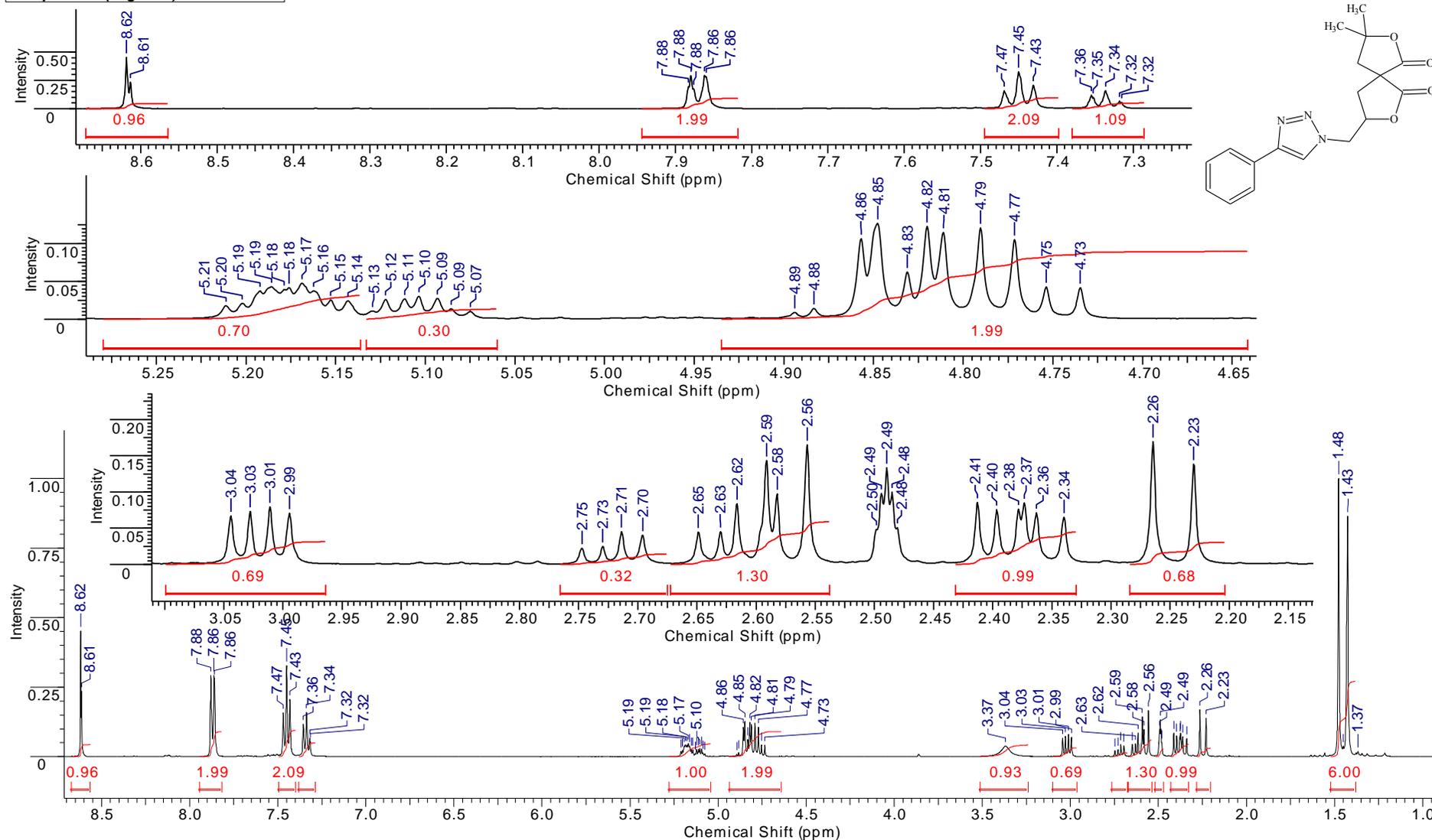
FW 371.3873 Formula C<sub>19</sub>H<sub>21</sub>N<sub>3</sub>O<sub>5</sub>

Acquisition Time (sec)	0.4999	Comment	Imported from UXMNR.		Date	11 Jun 2015 19:44:24			
File Name	D:\BN\output\2015\06.июнь\BM-797.C_002001r		Frequency (MHz)	100.61	Nucleus	13C	Number of Transients	64	
Original Points Count	12076	Points Count	65536	Pulse Sequence	zpgg30	Solvent	DMSO-D6	Sweep Width (Hz)	24154.59
Temperature (degree C)	27.000								



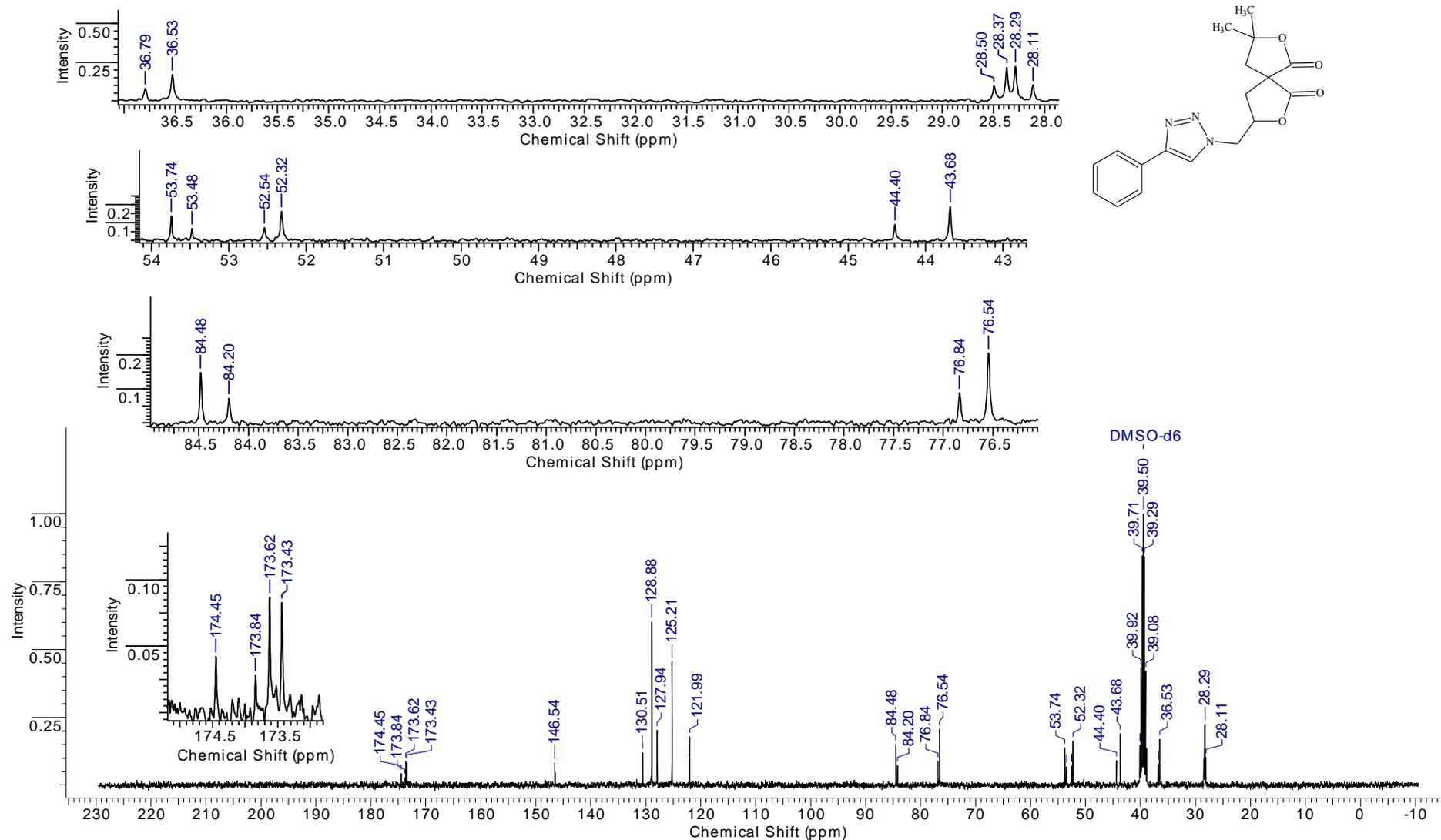
FW 341.3613 Formula C<sub>18</sub>H<sub>19</sub>N<sub>3</sub>O<sub>4</sub>

Acquisition Time (sec)	2.5559	Comment	Imported from UXMNR.	Date	23 Jun 2015 15:52:08		
File Name	D:\BN\output\2015\06.июнь\BM-800.H_001001r	Frequency (MHz)	400.13	Nucleus	1H	Number of Transients	8
Original Points Count	16384	Points Count	65536	Pulse Sequence	zg30	Solvent	DMSO-D6
Temperature (degree C)	27.000					Sweep Width (Hz)	6410.26



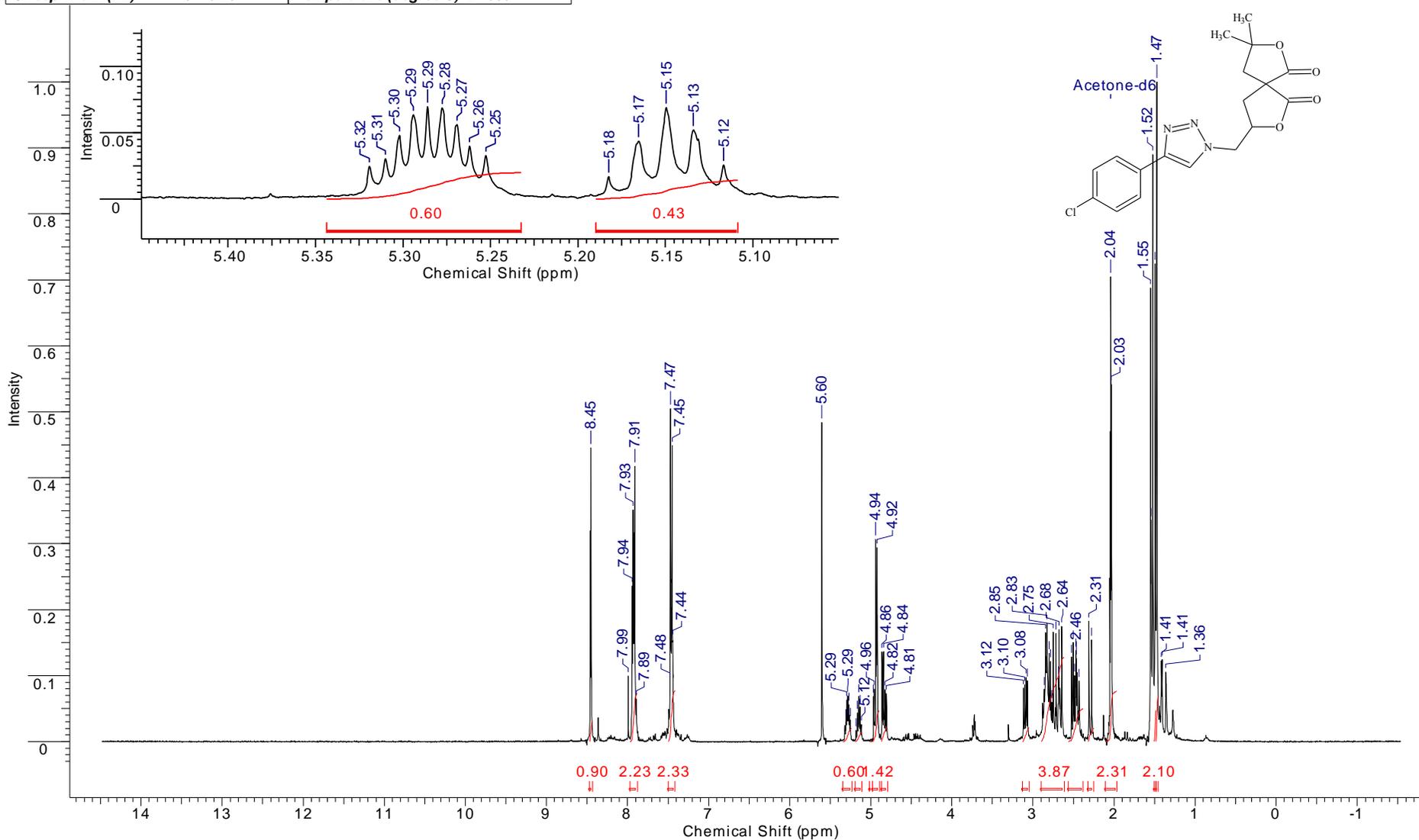
**FW** 341.3613 **Formula** C<sub>18</sub>H<sub>19</sub>N<sub>3</sub>O<sub>4</sub>

<b>Acquisition Time (sec)</b>	0.4999	<b>Comment</b>	Imported from UXMNR.		<b>Date</b>	23 Jun 2015 15:55:08	
<b>File Name</b>	D:\BN\output\2015\06.июнь\BM-800.C_002001r	<b>Frequency (MHz)</b>	100.61	<b>Nucleus</b>	13C	<b>Number of Transients</b>	128
<b>Original Points Count</b>	12076	<b>Points Count</b>	65536	<b>Pulse Sequence</b>	zgpg30	<b>Sweep Width (Hz)</b>	24154.59
<b>Temperature (degree C)</b>	27.000						



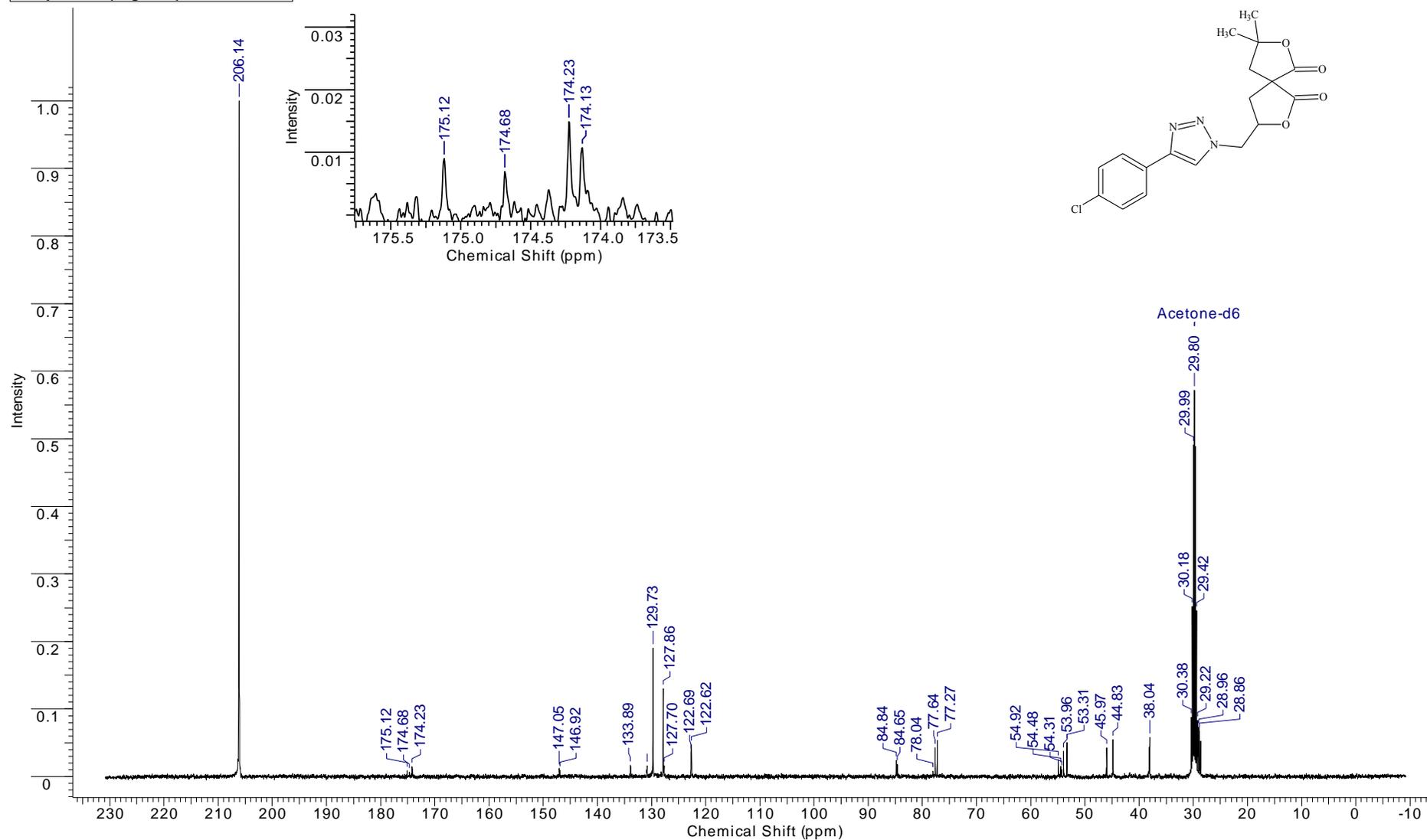
<b>FW</b>	375.8060	<b>Formula</b>	C <sub>18</sub> H <sub>18</sub> ClN <sub>3</sub> O <sub>4</sub>
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<b>Acquisition Time (sec)</b>	2.5559	<b>Comment</b>	Imported from UXNMR.	<b>Date</b>	23 Jun 2015 14:54:08
<b>File Name</b>	D:\BN\output\2015\06.июнь\BM-803.H_001001r	<b>Frequency (MHz)</b>	400.13	<b>Nucleus</b>	1H
<b>Original Points Count</b>	16384	<b>Points Count</b>	65536	<b>Pulse Sequence</b>	zg30
<b>Sweep Width (Hz)</b>	6410.26	<b>Temperature (degree C)</b>	27.000	<b>Solvent</b>	CHLOROFORM-D



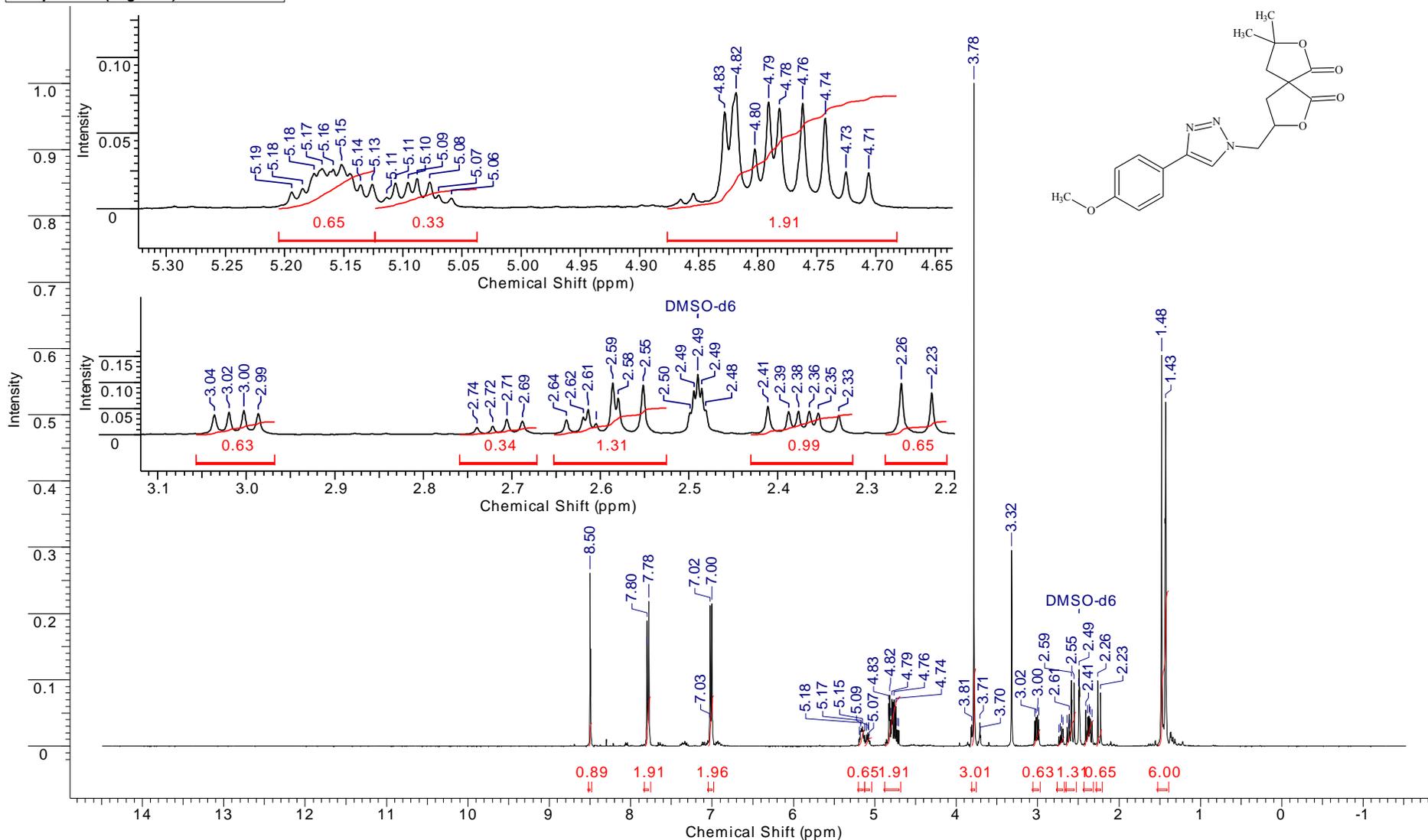
<b>FW</b>	375.8060	<b>Formula</b>	C <sub>18</sub> H <sub>18</sub> ClN <sub>3</sub> O <sub>4</sub>
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<b>Acquisition Time (sec)</b>	0.4999	<b>Comment</b>	Imported from UXMNR.		<b>Date</b>	23 Jun 2015 15:14:28			
<b>File Name</b>	D:\BN\output\2015\06.июнь\BM-803.C_002001r		<b>Frequency (MHz)</b>	100.61	<b>Nucleus</b>	13C	<b>Number of Transients</b>	680	
<b>Original Points Count</b>	12076	<b>Points Count</b>	65536	<b>Pulse Sequence</b>	zgpg30	<b>Solvent</b>	DMSO-D6	<b>Sweep Width (Hz)</b>	24154.59
<b>Temperature (degree C)</b>	27.000								



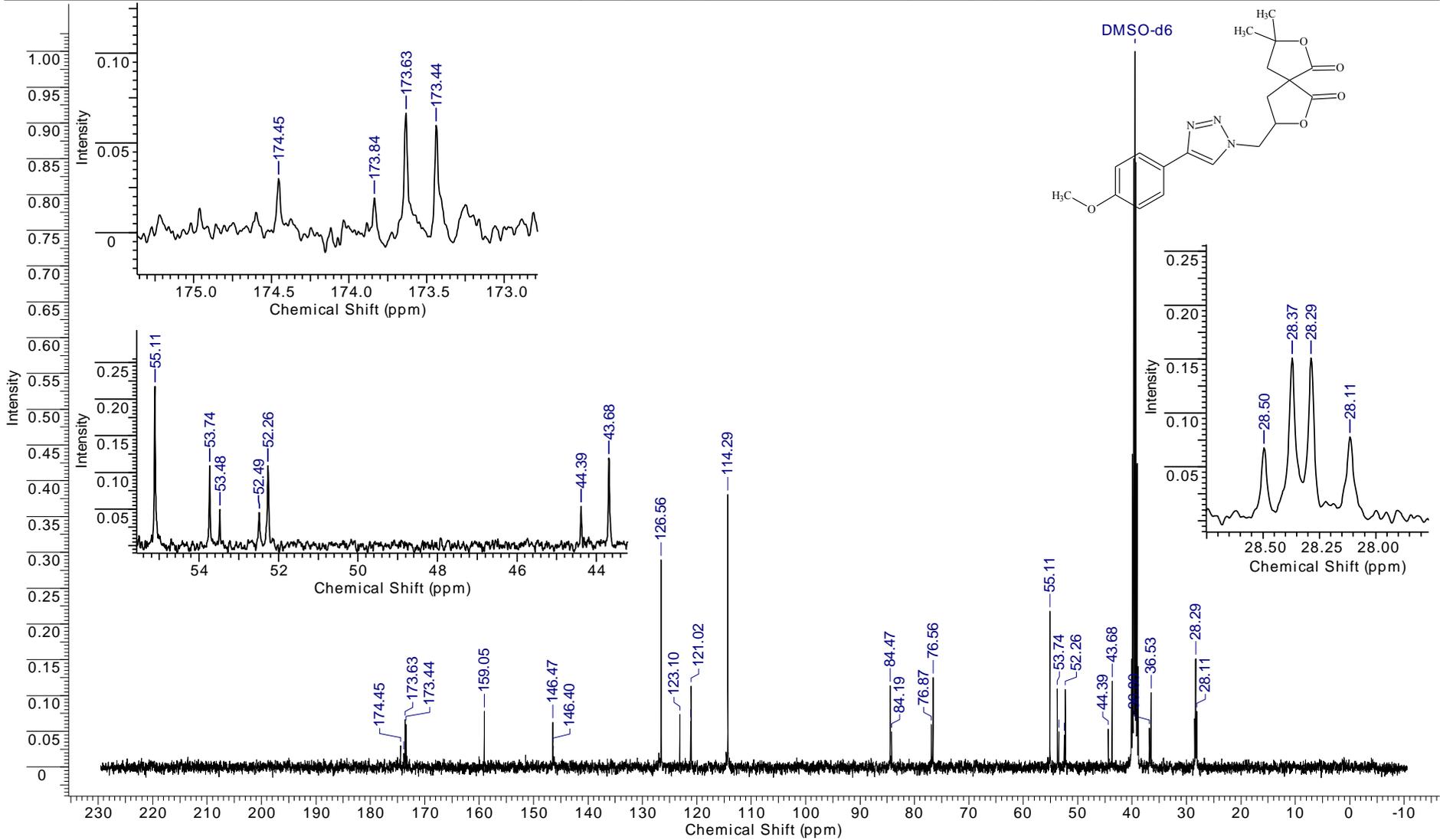
FW 371.3873 Formula C<sub>19</sub>H<sub>21</sub>N<sub>3</sub>O<sub>5</sub>

Acquisition Time (sec)	2.5559	Comment	Imported from UXNMR.	Date	25 Jun 2015 17:07:52				
File Name	D:\BN\output\2015\06.июнь\BM-804.H_001001r	Frequency (MHz)	400.13	Nucleus	1H	Number of Transients	4		
Original Points Count	16384	Points Count	65536	Pulse Sequence	zg30	Solvent	DMSO-D6	Sweep Width (Hz)	6410.26
Temperature (degree C)	27.000								



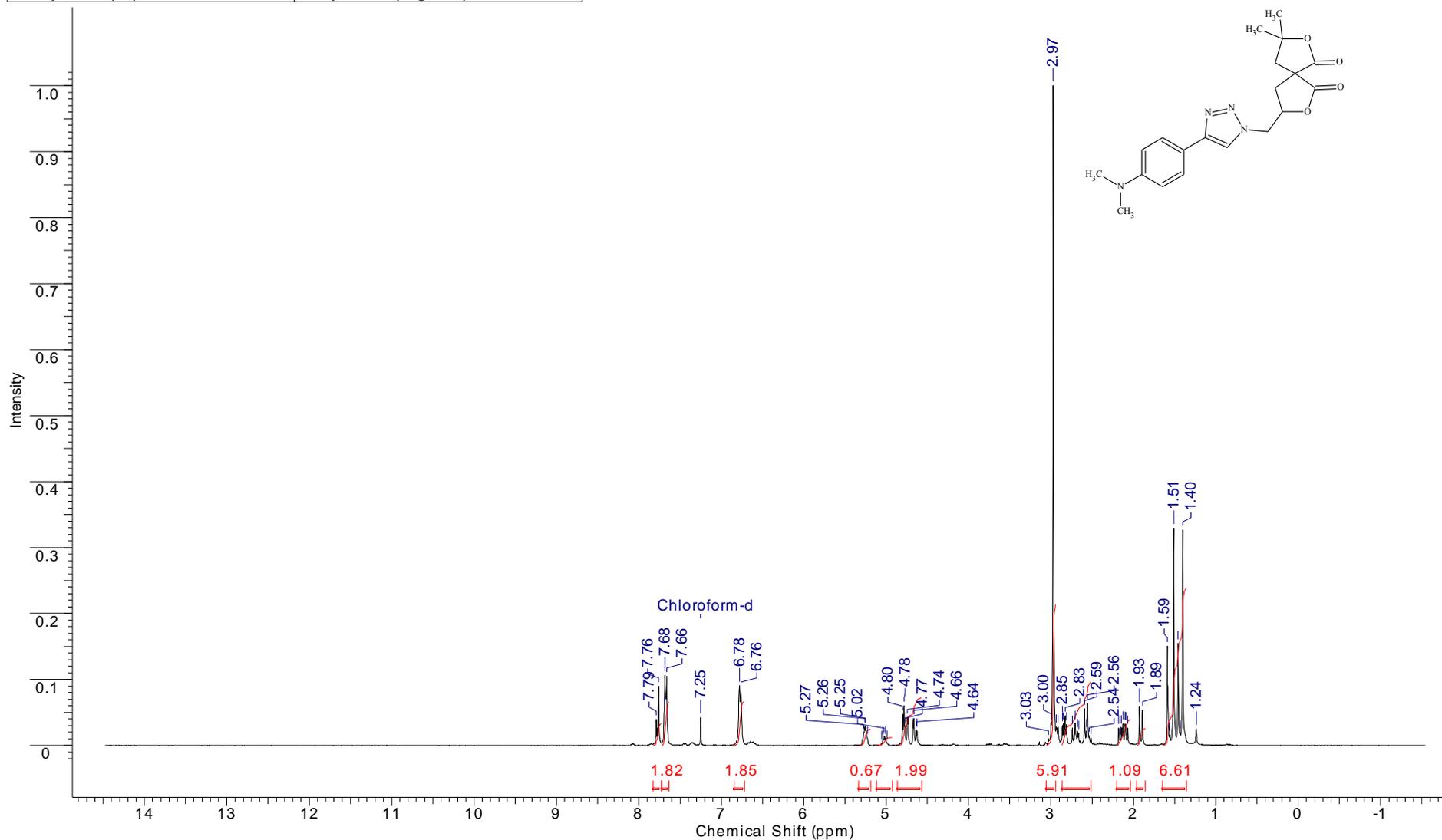
**FW** 371.3873 **Formula** C<sub>19</sub>H<sub>21</sub>N<sub>3</sub>O<sub>5</sub>

<b>Acquisition Time (sec)</b>	0.4999	<b>Comment</b>	Imported from UXNMR.		<b>Date</b>	25 Jun 2015 17:15:38	
<b>File Name</b>	D:\BN\output\2015\06.июнь\BM-804.C_002001r		<b>Frequency (MHz)</b>	100.61	<b>Nucleus</b>	13C	
<b>Number of Transients</b>	230	<b>Original Points Count</b>	12076	<b>Points Count</b>	65536	<b>Pulse Sequence</b>	zgpg30
<b>Solvent</b>	CHLOROFORM-D		<b>Sweep Width (Hz)</b>	24154.59	<b>Temperature (degree C)</b>	27.000	



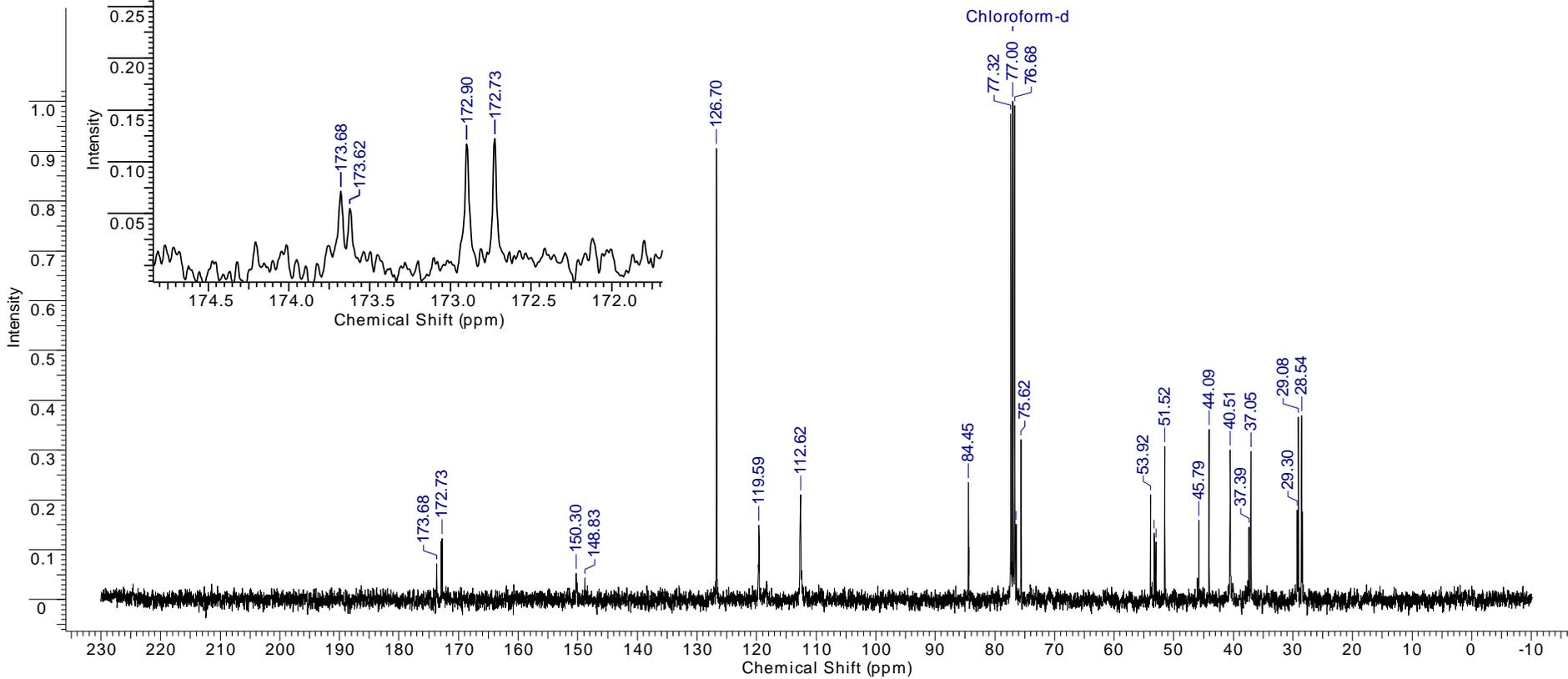
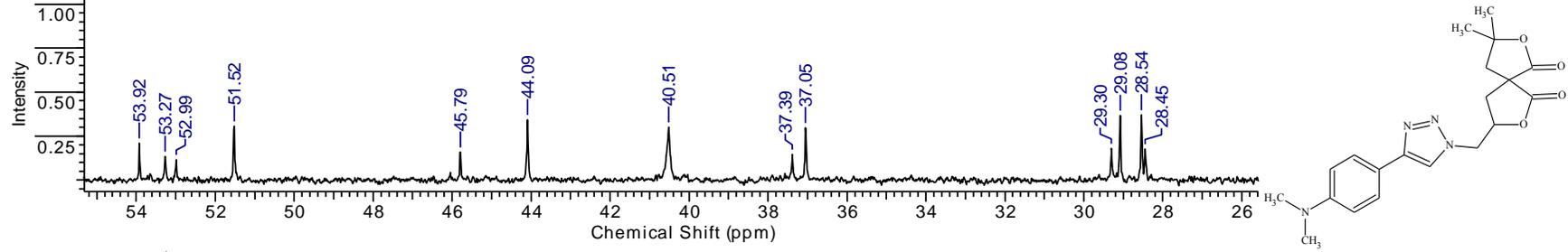
**FW** 384.4291 **Formula** C<sub>20</sub>H<sub>24</sub>N<sub>4</sub>O<sub>4</sub>

<b>Acquisition Time (sec)</b>	2.5559	<b>Comment</b>	Imported from UXNMR.		<b>Date</b>	30 Jun 2015 18:56:10	
<b>File Name</b>	D:\BN\output\2015\06.июнь\BM-807.H_001001r	<b>Frequency (MHz)</b>	400.13	<b>Nucleus</b>	1H	<b>Number of Transients</b>	4
<b>Original Points Count</b>	16384	<b>Points Count</b>	65536	<b>Pulse Sequence</b>	zg30	<b>Solvent</b>	CHLOROFORM-D
<b>Sweep Width (Hz)</b>	6410.26	<b>Temperature (degree C)</b>	27.000				



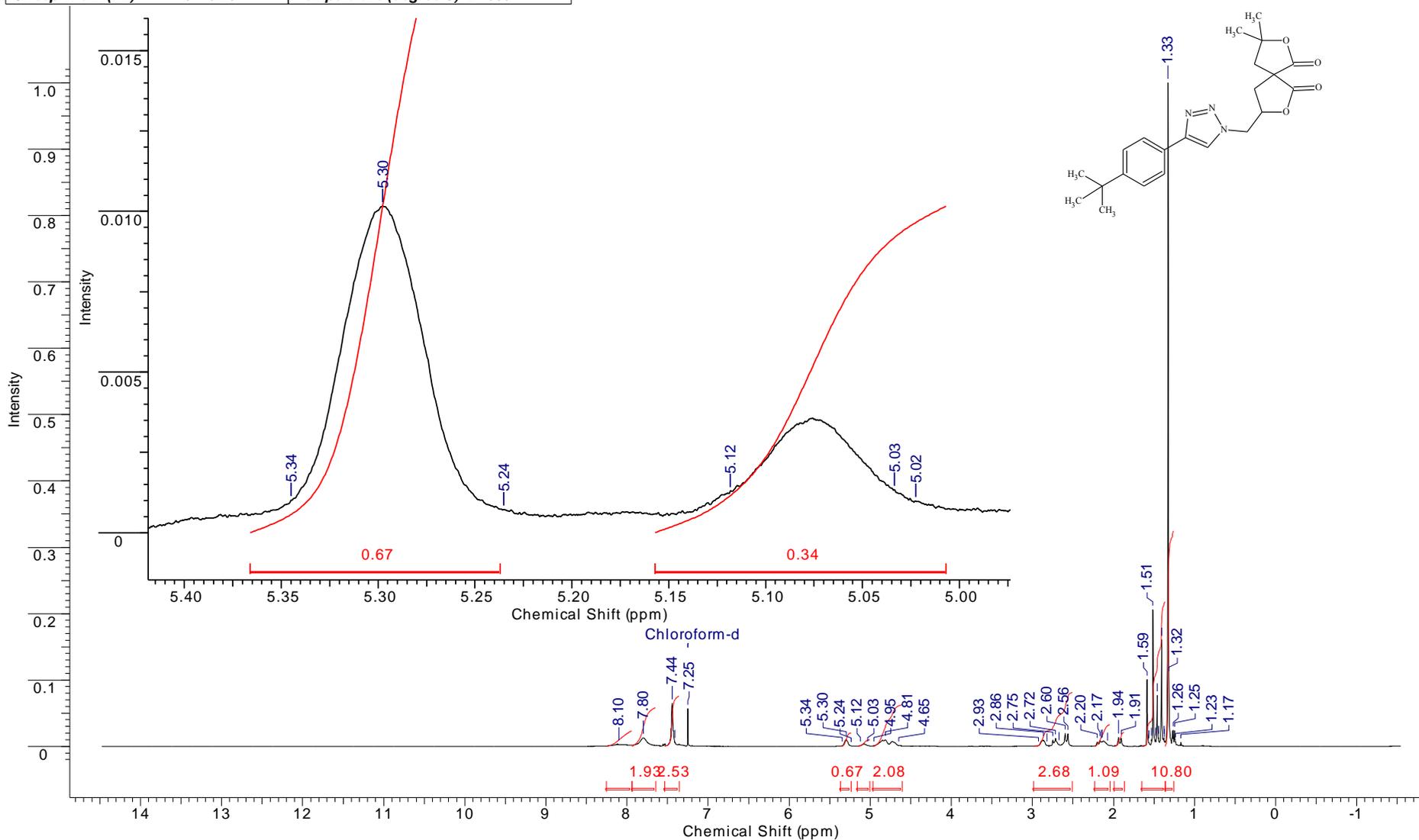
**FW** 384.4291 **Formula** C<sub>20</sub>H<sub>24</sub>N<sub>4</sub>O<sub>4</sub>

<b>Acquisition Time (sec)</b>	0.4999	<b>Comment</b>	Imported from UXMNR.		<b>Date</b>	30 Jun 2015 19:04:42	
<b>File Name</b>	D:\BN\output\2015\06.июнь\BM-807.C_002001r	<b>Frequency (MHz)</b>	100.61	<b>Nucleus</b>	13C	<b>Number of Transients</b>	303
<b>Original Points Count</b>	12076	<b>Points Count</b>	65536	<b>Pulse Sequence</b>	zgpg30	<b>Solvent</b>	DMSO-D6
<b>Temperature (degree C)</b>	27.000					<b>Sweep Width (Hz)</b>	24154.59



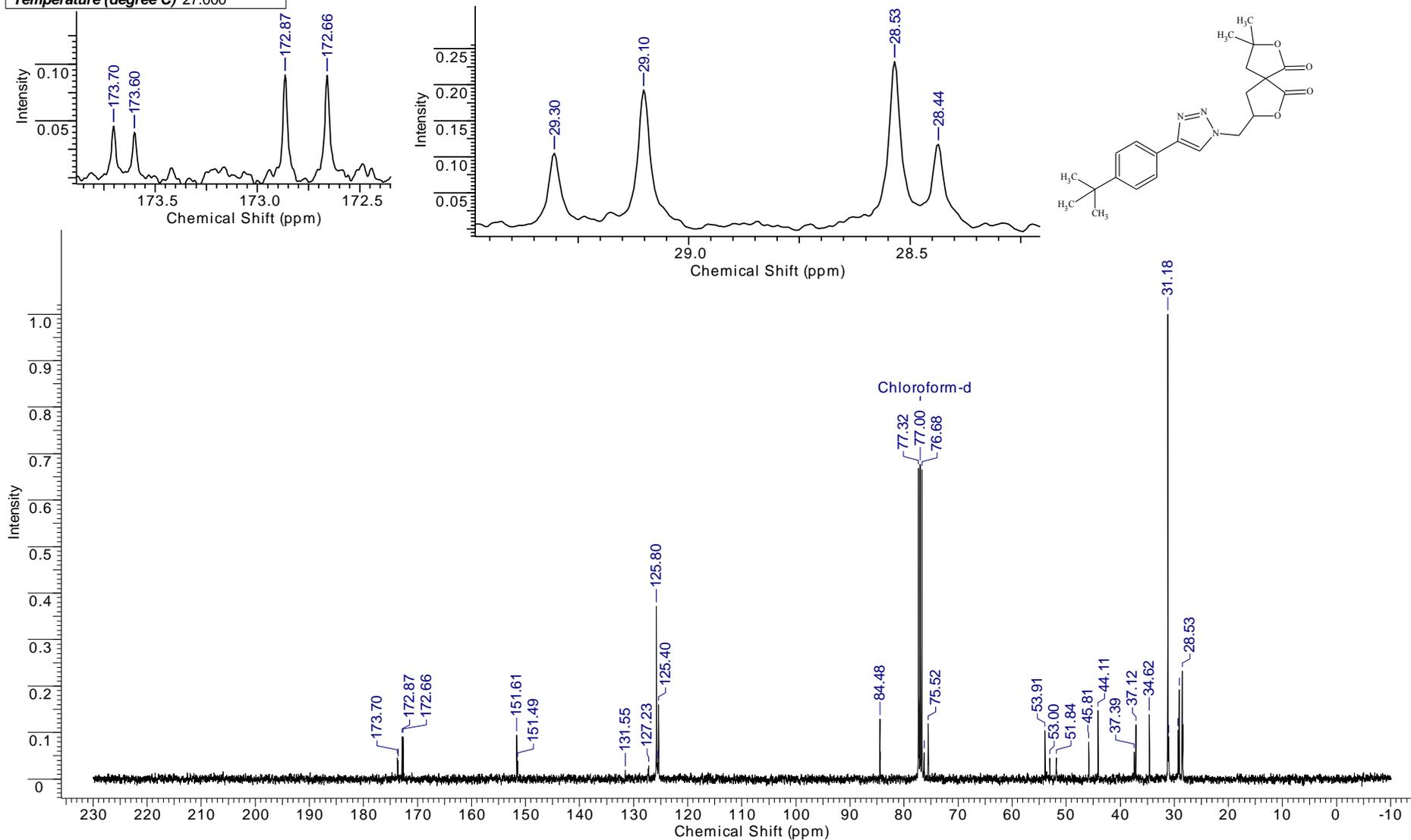
FW 397.4676 Formula C<sub>22</sub>H<sub>27</sub>N<sub>3</sub>O<sub>4</sub>

Acquisition Time (sec)	2.5559	Comment	Imported from UXMNR.		Date	30 Jun 2015 18:46:12		
File Name	D:\BN\output\2015\06.июнь\BM-808.H_001001r		Frequency (MHz)	400.13	Nucleus	1H	Number of Transients	4
Original Points Count	16384	Points Count	65536	Pulse Sequence	zg30	Solvent	CHLOROFORM-D	
Sweep Width (Hz)	6410.26	Temperature (degree C)	27.000					



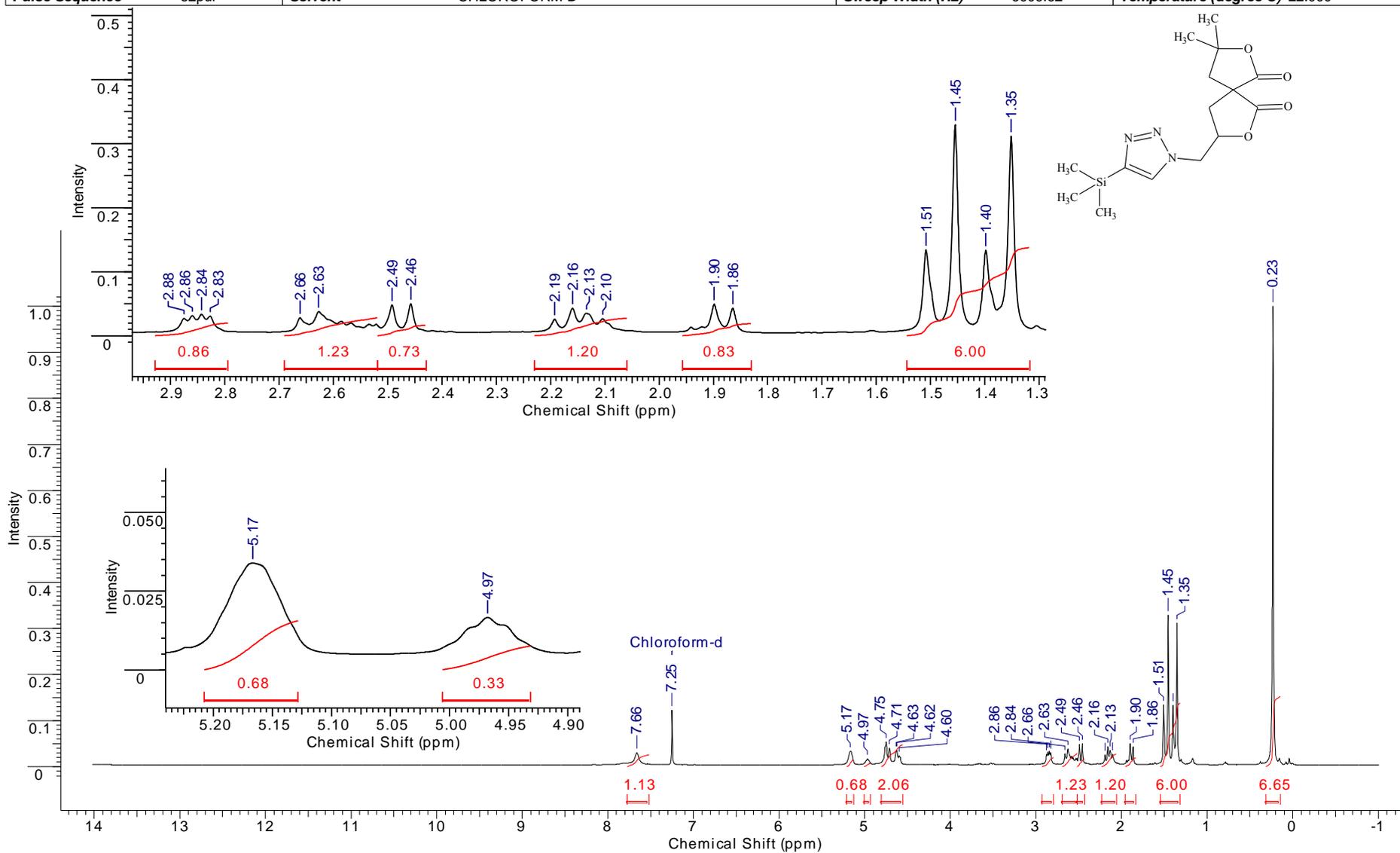
FW 397.4676 Formula C<sub>22</sub>H<sub>27</sub>N<sub>3</sub>O<sub>4</sub>

Acquisition Time (sec)	0.4999	Comment	Imported from UXMNR.		Date	30 Jun 2015 18:53:38			
File Name	D:\BN\output\2015\06.июнь\BM-808.C_002001r		Frequency (MHz)	100.61	Nucleus	13C	Number of Transients	256	
Original Points Count	12076	Points Count	65536	Pulse Sequence	zgpg30	Solvent	DMSO-D6	Sweep Width (Hz)	24154.59
Temperature (degree C)	27.000								



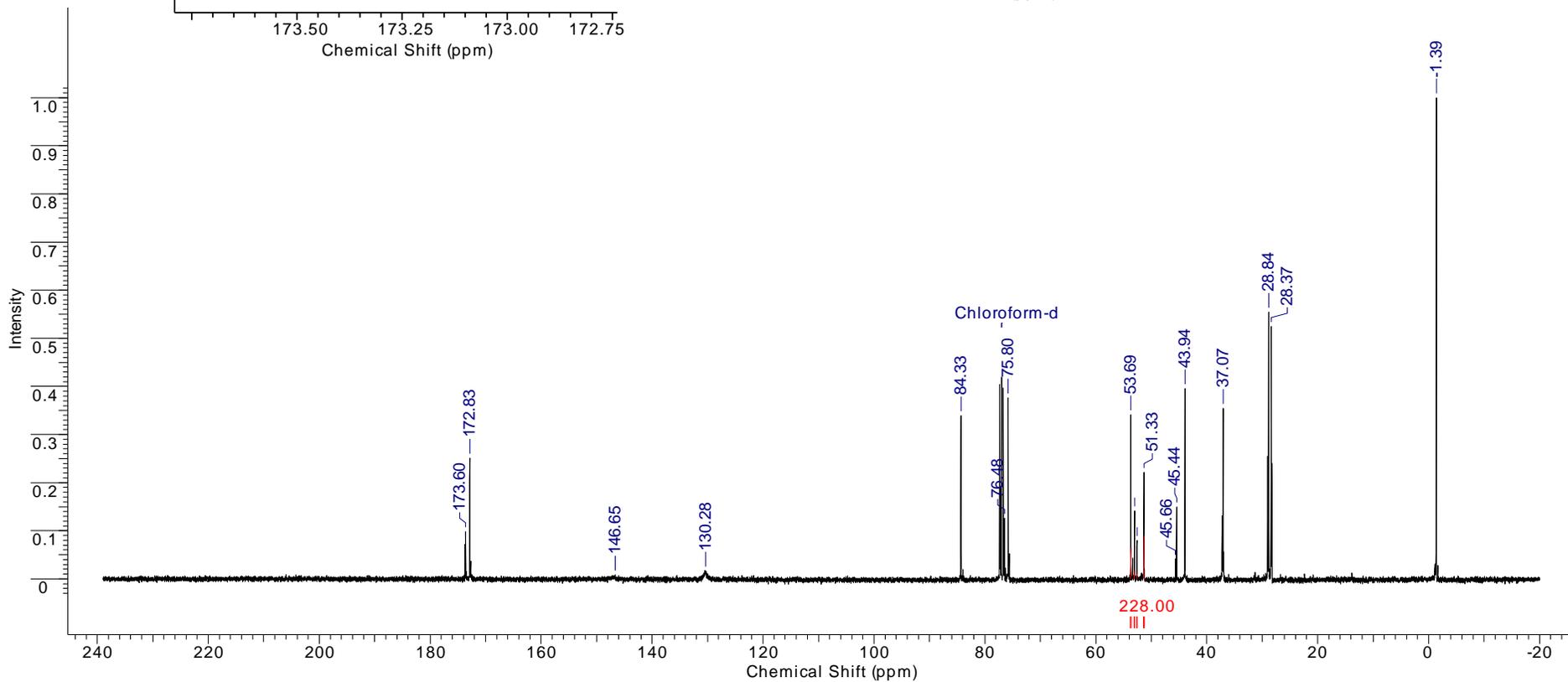
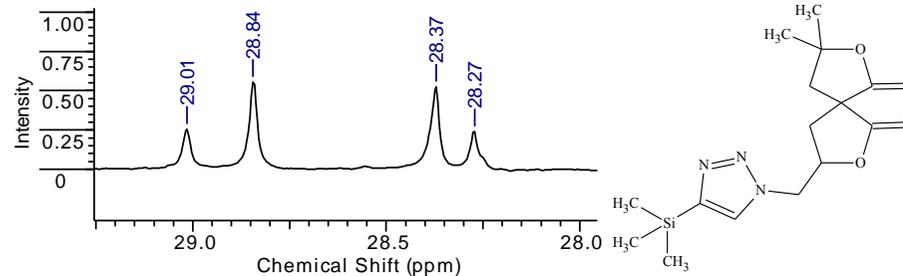
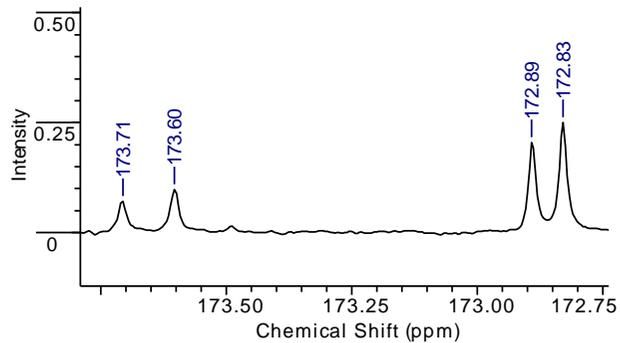
<b>FW</b>	337.4464	<b>Formula</b>	C <sub>15</sub> H <sub>23</sub> N <sub>3</sub> O <sub>4</sub> Si
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<b>Acquisition Time (sec)</b>	6.0001	<b>Date</b>	Jun 25 2015	<b>File Name</b>	D:\BN\output\2015\06.июнь\BM-801_20150625_01\PROTON_01				
<b>Frequency (MHz)</b>	399.96	<b>Nucleus</b>	1H	<b>Number of Transients</b>	8	<b>Original Points Count</b>	36058	<b>Points Count</b>	65536
<b>Pulse Sequence</b>	s2pul	<b>Solvent</b>	CHLOROFORM-D	<b>Sweep Width (Hz)</b>	6009.62	<b>Temperature (degree C)</b> 22.000			



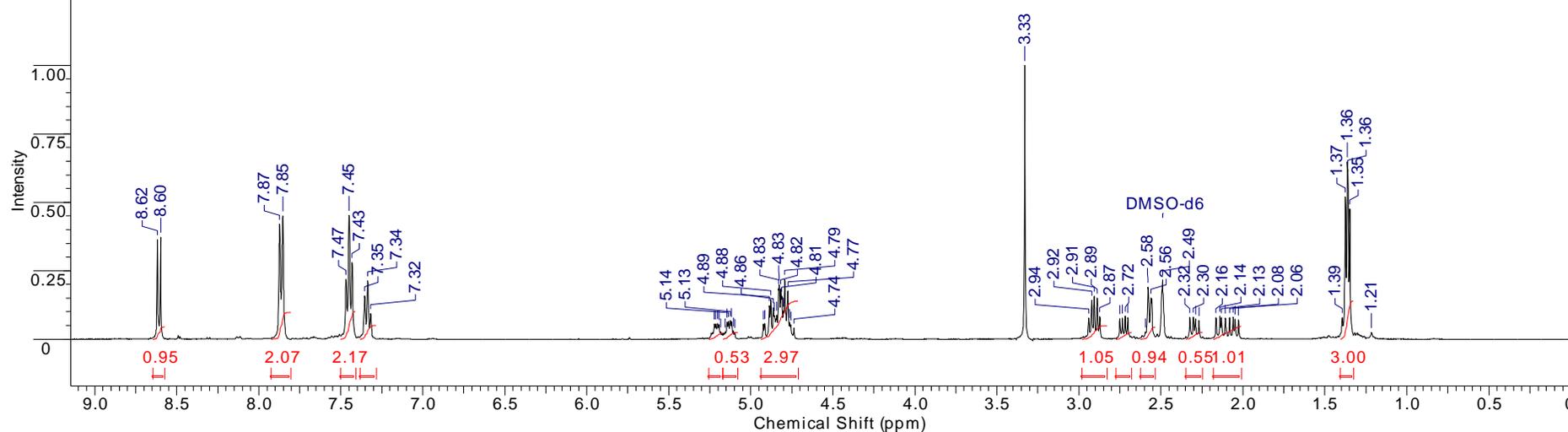
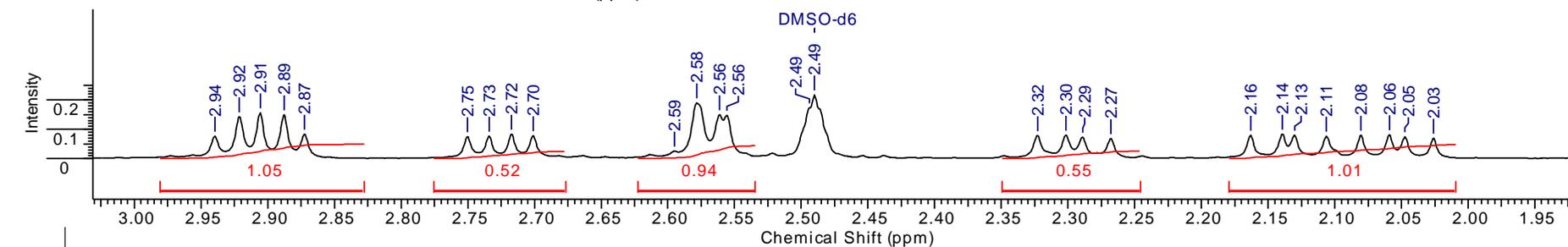
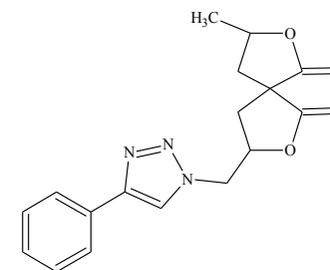
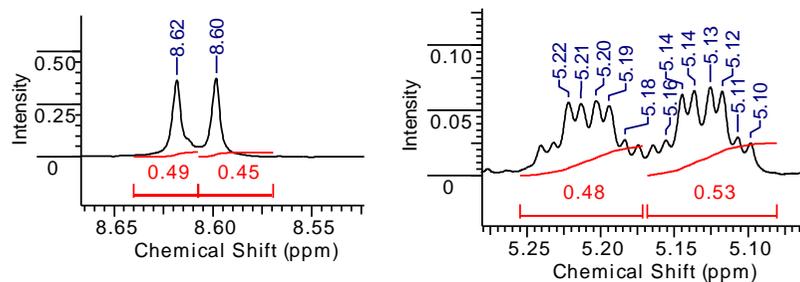
<b>FW</b>	337.4464	<b>Formula</b>	C <sub>15</sub> H <sub>23</sub> N <sub>3</sub> O <sub>4</sub> Si
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<b>Acquisition Time (sec)</b>	1.5000	<b>Date</b>	Jun 25 2015	<b>File Name</b>	D:\BN\output\2015\06.июнь\BM-801-C_20150625_01\CARBON_01		
<b>Frequency (MHz)</b>	100.58	<b>Nucleus</b>	13C	<b>Number of Transients</b>	256	<b>Original Points Count</b>	39063
<b>Points Count</b>	65536	<b>Pulse Sequence</b>	s2pul	<b>Solvent</b>	CHLOROFORM-D		
<b>Sweep Width (Hz)</b>	26041.67	<b>Temperature (degree C)</b>	22.000				



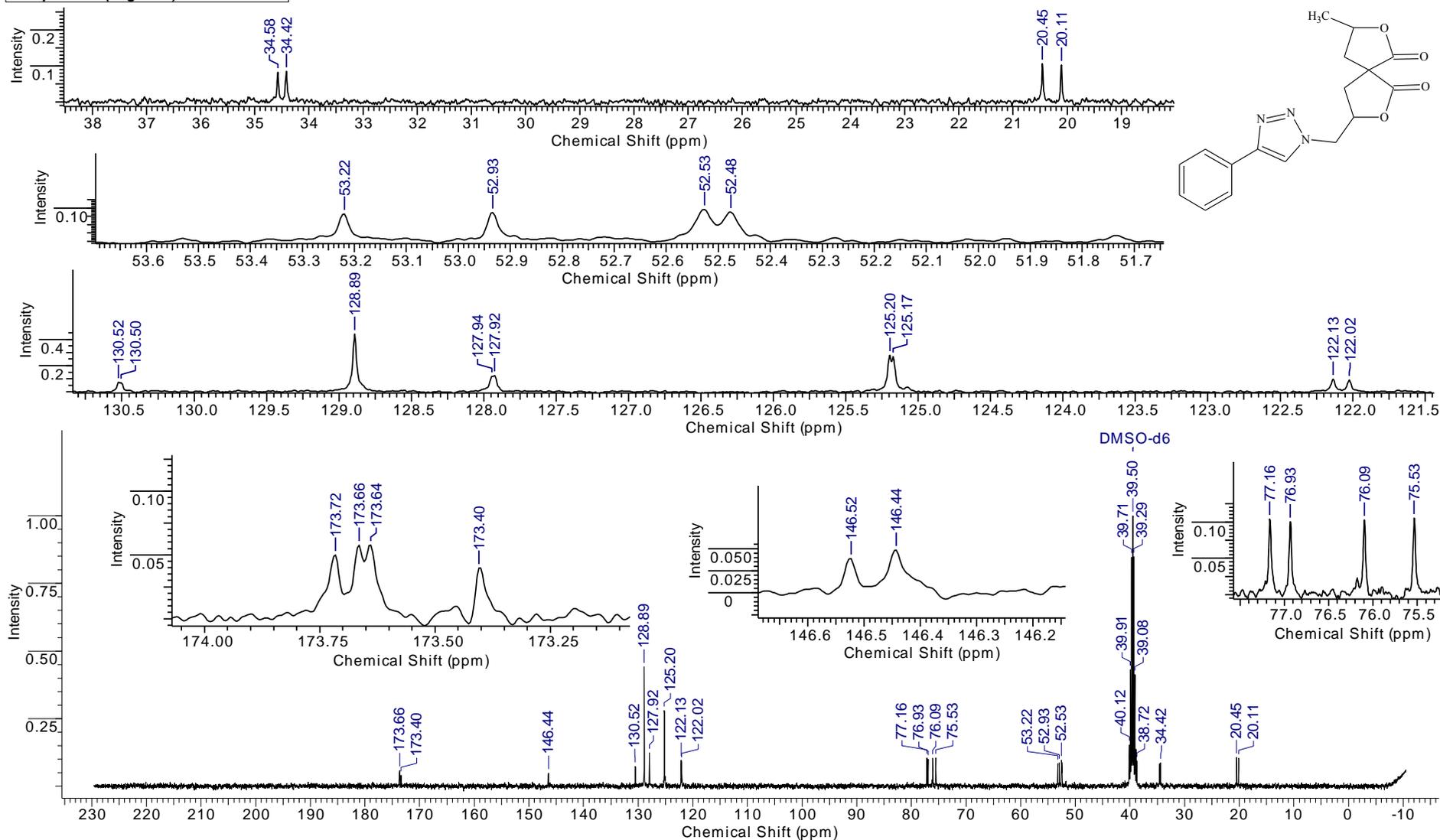
FW 327.3347 Formula C<sub>17</sub>H<sub>17</sub>N<sub>3</sub>O<sub>4</sub>

Acquisition Time (sec)	2.5559	Comment	Imported from UXNMR.		Date	02 Jul 2015 19:52:30	
File Name	D:\BN\output\2015\07.июль\BM-810.H_001001r	Frequency (MHz)	400.13	Nucleus	1H	Number of Transients	5
Original Points Count	16384	Points Count	65536	Pulse Sequence	zg30	Solvent	CHLOROFORM-D
Sweep Width (Hz)	6410.26	Temperature (degree C)	27.000				



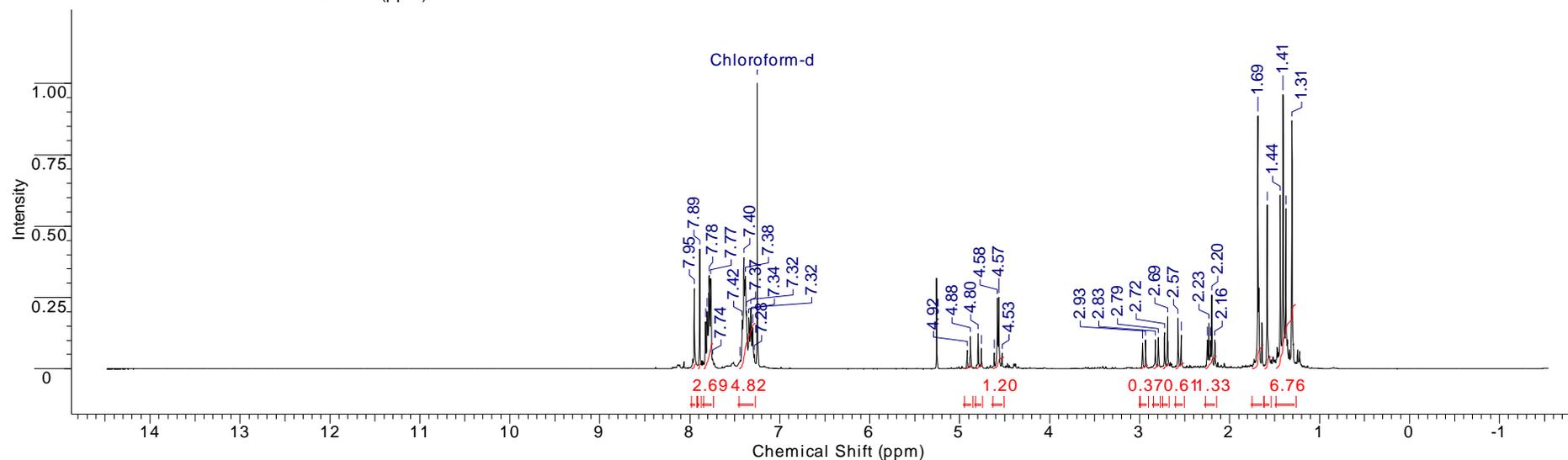
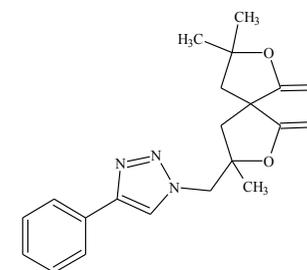
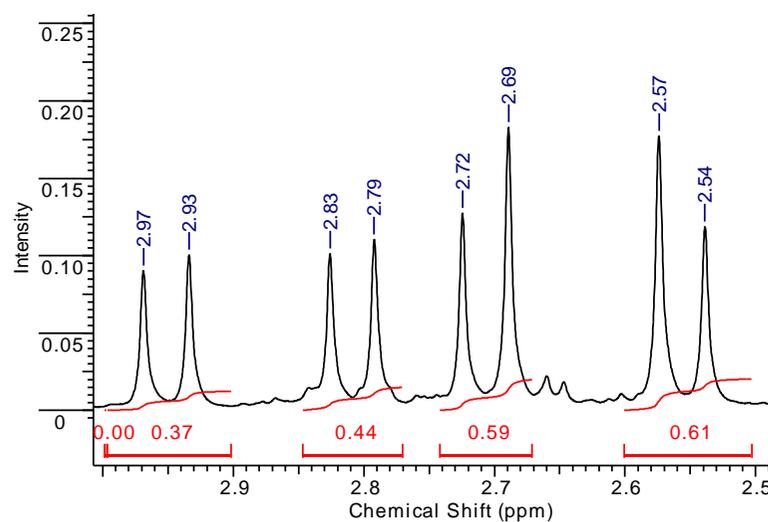
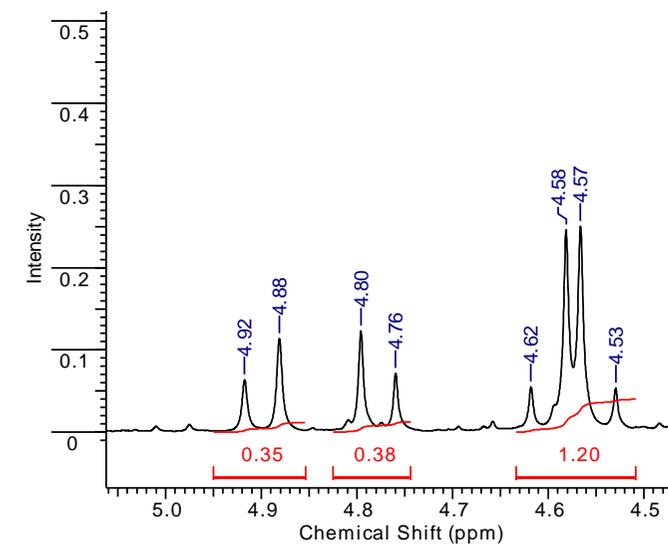
FW 327.3347 Formula C<sub>17</sub>H<sub>17</sub>N<sub>3</sub>O<sub>4</sub>

Acquisition Time (sec)	0.4999	Comment	Imported from UXNMR.	Date	02 Jul 2015 19:56:32				
File Name	D:\BN\output\2015\07\июль\BM-810.C_002001r	Frequency (MHz)	100.61	Nucleus	<sup>13</sup> C	Number of Transients	128		
Original Points Count	12076	Points Count	65536	Pulse Sequence	zgpg30	Solvent	DMSO-D6	Sweep Width (Hz)	24154.59
Temperature (degree C)	27.000								



FW 355.3879 Formula C<sub>19</sub>H<sub>21</sub>N<sub>3</sub>O<sub>4</sub>

Acquisition Time (sec)	2.5559	Comment	Imported from UXNMR.		Date	07 Jul 2015 18:44:08	
File Name	D:\BN\output\2015\07\июль\BM-811.H_001001r	Frequency (MHz)	400.13	Nucleus	1H	Number of Transients	4
Original Points Count	16384	Points Count	65536	Pulse Sequence	zg30	Solvent	CHLOROFORM-D
Sweep Width (Hz)	6410.26	Temperature (degree C)	27.000				



**FW** 355.3879 **Formula** C<sub>19</sub>H<sub>21</sub>N<sub>3</sub>O<sub>4</sub>

<b>Acquisition Time (sec)</b>	0.4999	<b>Comment</b>	Imported from UXMNR.		<b>Date</b>	07 Jul 2015 18:47:58			
<b>File Name</b>	D:\BN\output\2015\07.июль\BM-811.C_002001r		<b>Frequency (MHz)</b>	100.61	<b>Nucleus</b>	13C	<b>Number of Transients</b>	64	
<b>Original Points Count</b>	12076	<b>Points Count</b>	65536	<b>Pulse Sequence</b>	zgpg30	<b>Solvent</b>	DMSO-D6	<b>Sweep Width (Hz)</b>	24154.59
<b>Temperature (degree C)</b>	27.000								

