

## Supporting information

### Synthesis of Novel 6-[N, N-Bis(2-hydroxyethyl)amino]purine Nucleosides under Microwave Irradiation in Neat Water

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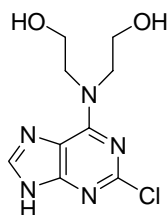
**General:** Melting points are recorded with a micro melting point apparatus and uncorrected. NMR spectra were recorded with a 400 NMR spectrometer for <sup>1</sup>H-NMR, 100 MHz for <sup>13</sup>C-NMR. Proton chemical shifts are given in ppm relative to tetramethylsilane (0.00 ppm) in CDCl<sub>3</sub> or to the residual proton signals of the deuterated solvent DMSO-d<sub>6</sub> (2.50 ppm) for <sup>1</sup>H and <sup>13</sup>C NMR. High resolution mass spectra were taken with a 3000 mass spectrometer, using Waters Q-ToFMS/MS system. For column chromatography 200-300 mesh silica gel (GF<sub>254</sub>) was used as the stationary phase. All reactions were monitored by thin layer chromatography (TLC). All reagents and solvents were purchased from commercial sources and purified commonly before used.

All microwave irradiation experiments were carried out in the cavity of a commercially available single-mode microwave synthesis apparatus equipped with a high sensitivity infrared sensor for temperature control and measurement (MAS-I, Sineo Microwave Chemical Technology Co. Ltd., Shanghai, P. R. of China) with continuous irradiation power from 0 to 600 W. The reactions were carried out in open glass vials. The temperature was measured with an IR sensor on the outer surface of the reaction vials.

**Typical Experimental Procedure for the Reaction of Purines with Diethanolamine under Microwave Irradiation.**

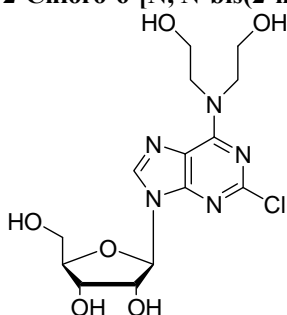
Purine nucleoside **1a** (1 mmol) and water (5 mL) were put in a 25 mL glass vial equipped with a small magnetic stirring bar. To this was added diethanolamine **2a** (1.5 mmol). Then the mixture was put into the cavity of the microwave synthesis apparatus and irradiated at 400 W at 100 °C for 10 min. After completion of the reaction, the vial was cooled to room temperature. After evaporation of the water, the crude product was purified by column chromatography over silica gel using CH<sub>2</sub>Cl<sub>2</sub>:CH<sub>3</sub>OH =17:1 (v/v) as the eluent, to give **3a**.

### 2-Chloro-6-[N, N-bis(2-hydroxyethyl)amino]purine (3a)



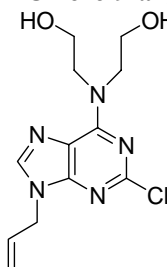
Light yellow powder, mp 239-241 °C.  $^1\text{H}$  NMR (DMSO- $d_6$ , 400 MHz)  $\delta$  13.08 (s, 1H), 8.10 (s, 1H), 4.78 (s, 2H), 4.29 (s, 2H), 3.77 (s, 2H), 3.67 (s, 4H).  $^{13}\text{C}$  NMR (DMSO- $d_6$ , 100 MHz)  $\delta$  154.1, 152.3, 152.1, 138.6, 117.6, 59.9, 58.1, 52.1, 51.2. HRMS: calcd for  $\text{C}_9\text{H}_{12}\text{ClN}_5\text{NaO}_2$  [ $\text{M} + \text{Na}^+$ ] 280.0578, found 280.0578.

### 2-Chloro-6-[N, N-bis(2-hydroxyethyl)amino]-9-( $\beta$ -D-ribofuranosyl)purine (3b)



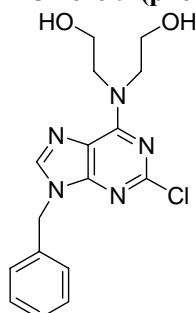
White powder, mp 100-102 °C.  $^1\text{H}$  NMR (DMSO- $d_6$ , 400 MHz)  $\delta$  8.40 (s, 1H), 5.82 (d,  $J = 5.6$  Hz, 1H), 5.50 (d,  $J = 6.0$  Hz, 1H), 5.22 (d,  $J = 4.8$  Hz, 1H), 5.07 (t,  $J = 5.6$  Hz, 1H), 4.83-4.82 (m, 2H), 4.50-4.46 (m, 1H), 4.26-4.25 (m, 2H), 4.11-4.09 (m, 1H), 3.92 (d,  $J = 3.6$  Hz, 1H), 3.76-3.75 (m, 2H), 3.65-3.62 (m, 5H), 3.55-3.51 (m, 1H).  $^{13}\text{C}$  NMR (DMSO- $d_6$ , 100 MHz)  $\delta$  154.2, 152.4, 151.1, 138.8, 118.3, 87.2, 85.5, 73.6, 70.2, 61.2, 59.7, 58.0, 52.1, 51.3. HRMS: calcd for  $\text{C}_{14}\text{H}_{21}\text{ClN}_5\text{O}_6$  [ $\text{M} + \text{H}^+$ ] 390.1180, found 390.1180.

### 2-Chloro-9-allyl-6-[N, N-bis(2-hydroxyethyl)amino]purine (3c)



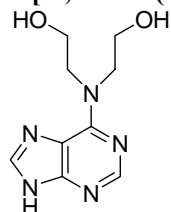
White powder, mp 137-139 °C.  $^1\text{H}$  NMR (DMSO- $d_6$ , 400 MHz)  $\delta$  8.14 (s, 1H), 6.07-6.01 (m, 1H), 5.20 (d,  $J = 10.0$  Hz, 1H), 5.02 (d,  $J = 16.8$  Hz, 1H), 4.80-4.75 (m, 4H), 4.29 (s, 2H), 3.77 (s, 2H), 3.67 (s, 4H).  $^{13}\text{C}$  NMR (DMSO- $d_6$ , 100 MHz)  $\delta$  154.2, 152.5, 151.2, 140.4, 133.0, 117.8, 117.4, 59.8, 58.1, 52.1, 51.4, 45.0. HRMS: calcd for  $\text{C}_{12}\text{H}_{16}\text{ClN}_5\text{NaO}_2$  [ $\text{M} + \text{Na}^+$ ] 320.0891, found 320.0891.

**2-Chloro-9-(phenylmethyl)-6-[N, N-bis(2-hydroxyethyl)amino]purine (3d)**



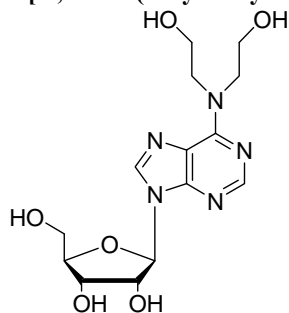
White powder, mp 144-146 °C.  $^1\text{H}$  NMR (DMSO- $d_6$ , 400 MHz)  $\delta$  8.28 (s, 1H), 7.37-7.26 (m, 5H), 5.35 (s, 2H), 4.81 (s, 2H), 4.28 (s, 2H), 3.77 (s, 2H), 3.66 (d,  $J$  = 4.8 Hz, 4H).  $^{13}\text{C}$  NMR (DMSO- $d_6$ , 100 MHz)  $\delta$  154.2, 152.6, 151.3, 140.5, 136.6, 128.7, 127.7, 127.2, 118.0, 59.8, 58.0, 52.1, 51.4, 46.1. HRMS: calcd for  $\text{C}_{16}\text{H}_{18}\text{ClN}_5\text{NaO}_2$  [ $\text{M} + \text{Na}^+$ ] 370.1047, found 370.1049.

**6-[N, N-Bis(2-hydroxyethyl)amino]purine (3e)**



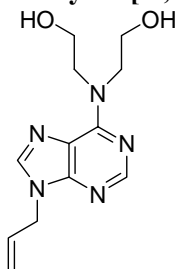
White powder, mp 214-215 °C.  $^1\text{H}$  NMR (DMSO- $d_6$ , 400 MHz)  $\delta$  12.97 (s, 1H), 8.17 (s, 1H), 8.10 (s, 1H), 4.83 (s, 2H), 4.27-4.21 (m, 2H), 3.83-3.81 (m, 2H), 3.67 (s, 4H).  $^{13}\text{C}$  NMR (DMSO- $d_6$ , 100 MHz)  $\delta$  153.6, 151.7, 151.1, 138.1, 118.5, 60.1, 58.6, 52.0, 51.4. HRMS: calcd for  $\text{C}_9\text{H}_{14}\text{N}_5\text{O}_2$  [ $\text{M} + \text{H}^+$ ] 224.1147, found 224.1147.

**6-[N, N-Bis(2-hydroxyethyl)amino]-9-( $\beta$ -D-ribofuranosyl)purine (3f)**



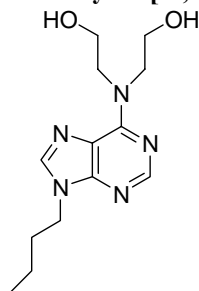
White powder, mp 221-223 °C.  $^1\text{H}$  NMR (DMSO- $d_6$ , 400 MHz)  $\delta$  8.36 (s, 1H), 8.19 (s, 1H), 5.88 (d,  $J$  = 6.0 Hz, 1H), 5.46 (d,  $J$  = 6.4 Hz, 1H), 5.39-5.36 (m, 1H), 5.19 (d,  $J$  = 4.4 Hz, 1H), 4.81-4.79 (m, 2H), 4.59-4.55 (m, 1H), 4.26-4.23 (m, 2H), 4.14-4.11 (m, 1H), 3.94 (d,  $J$  = 3.2 Hz, 1H), 3.81-3.80 (m, 2H), 3.66-3.64 (m, 5H), 3.56-3.52 (m, 1H).  $^{13}\text{C}$  NMR (DMSO- $d_6$ , 100 MHz)  $\delta$  153.7, 151.5, 149.8, 138.7, 119.4, 87.7, 85.6, 73.3, 70.4, 61.4. HRMS: calcd for  $\text{C}_{14}\text{H}_{21}\text{N}_5\text{NaO}_6$  [ $\text{M} + \text{Na}^+$ ] 378.1390, found 378.1389.

**9-Allyl-6-[N, N-bis(2-hydroxyethyl)amino]purine (3g)**



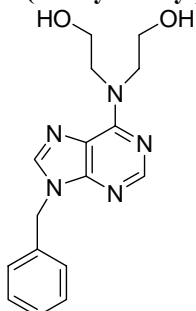
White powder, mp 144-146 °C. <sup>1</sup>H NMR (DMSO-d<sub>6</sub>, 400 MHz) δ 8.20 (s, 1H), 8.13 (s, 1H), 6.10-6.00 (m, 1H), 5.18 (dd, *J* = 10.4 Hz, 1.2 Hz, 1H), 5.01 (dd, *J* = 17.2 Hz, 1.2 Hz, 1H), 4.83-4.78 (m, 4H), 4.27-4.26 (m, 2H), 3.81 (s, 1H), 3.67 (s, 4H). <sup>13</sup>C NMR (DMSO-d<sub>6</sub>, 100 MHz) δ 153.7, 151.7, 150.1, 139.6, 133.2, 118.7, 117.3, 58.7, 51.7, 44.7. HRMS: calcd for C<sub>12</sub>H<sub>17</sub>N<sub>5</sub>NaO<sub>2</sub> [M + Na<sup>+</sup>] 286.1280, found 286.1280.

**9-n-Butyl-6-[N, N-bis(2-hydroxyethyl)amino]purine (3h)**



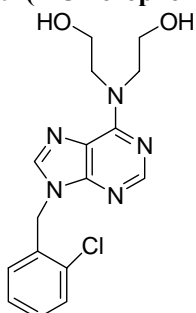
Light yellow crystal, mp 93-94 °C. <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz) δ 8.31 (s, 1H), 7.76 (s, 1H), 4.90-4.87 (m, 2H), 4.19 (t, *J* = 7.2 Hz, 2H), 4.10 (s, 4H), 3.98-3.96 (m, 4H), 1.89-1.82 (m, 2H), 1.39-1.34 (m, 2H), 0.96 (t, *J* = 7.2 Hz, 3H). <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz) δ 155.7, 151.9, 150.6, 138.9, 119.7, 61.4, 52.2, 43.7, 31.9, 19.8, 13.4. HRMS: calcd for C<sub>13</sub>H<sub>21</sub>N<sub>5</sub>NaO<sub>2</sub> [M + Na<sup>+</sup>] 302.1593, found 302.1592.

**9-(Phenylmethyl)-6-[N, N-bis(2-hydroxyethyl)amino]purine (3i)**



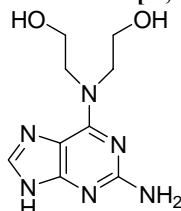
White powder, mp 93-95 °C. <sup>1</sup>H NMR (CDCl<sub>3</sub>, 400 MHz) δ 8.34 (s, 1H), 7.72 (s, 1H), 7.36-7.26 (m, 5H), 5.35 (s, 2H), 4.88 (s, 2H), 4.09 (s, 4H), 3.97-3.95 (m, 4H). <sup>13</sup>C NMR (CDCl<sub>3</sub>, 100 MHz) δ 155.9, 152.3, 150.8, 138.8, 135.4, 129.0, 128.4, 127.8, 119.6, 61.5, 58.3, 52.3, 47.2. HRMS: calcd for C<sub>16</sub>H<sub>19</sub>N<sub>5</sub>NaO<sub>2</sub> [M + Na<sup>+</sup>] 336.1437, found 336.1436.

**9-(2-Chlorophenylmethyl)-6-[N, N-bis(2-hydroxyethyl)amino]purine (3j)**



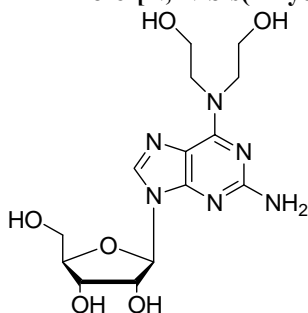
White crystal, mp 140-141 °C.  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz)  $\delta$  8.27 (s, 1H), 7.75 (s, 1H), 7.37-7.15 (m, 4H), 5.41 (s, 2H), 4.68 (s, 2H), 4.03 (s, 4H), 3.91-3.89 (m, 4H).  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz)  $\delta$  155.9, 152.2, 150.8, 139.0, 133.3, 132.9, 130.1, 129.9, 129.8, 127.4, 119.5, 61.5, 52.2, 44.8. HRMS: calcd for  $\text{C}_{16}\text{H}_{18}\text{ClN}_5\text{NaO}_2$  [ $\text{M} + \text{Na}^+$ ] 370.1047, found 370.1047.

**2-Amino-6-[N, N-bis(2-hydroxyethyl)amino]purine (3k)**



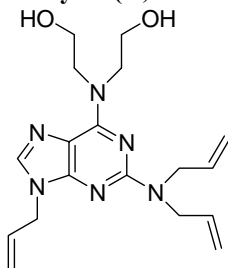
White powder, mp 196-198 °C.  $^1\text{H}$  NMR ( $\text{DMSO-d}_6$ , 400 MHz) 12.14-12.11 (m, 1H), 7.66 (s, 1H), 5.66 (s, 2H), 4.80-4.78 (m, 2H), 4.16-4.10 (m, 4H), 3.80-3.75 (m, 4H).  $^{13}\text{C}$  NMR ( $\text{DMSO-d}_6$ , 100 MHz)  $\delta$  159.3, 154.0, 153.3, 134.6, 112.9, 59.8, 51.2. HRMS: calcd for  $\text{C}_9\text{H}_{15}\text{N}_6\text{O}_2$  [ $\text{M} + \text{H}^+$ ] 239.1256, found 239.1254.

**2-Amino-6-[N, N-bis(2-hydroxyethyl)amino]-9-( $\beta$ -D-ribofuranosyl)purine (3l)**

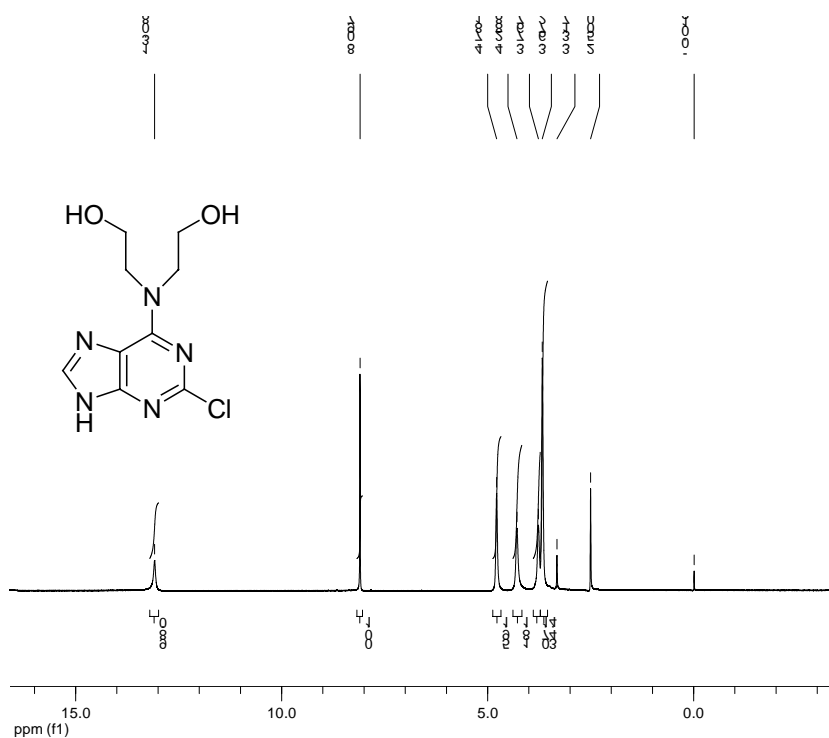


White crystal, mp 100-102 °C.  $^1\text{H}$  NMR ( $\text{DMSO-d}_6$ , 400 MHz) 7.95 (s, 1H), 5.79-5.74 (m, 3H), 5.40-5.35 (m, 2H), 5.13 (d,  $J = 4.4$  Hz, 1H), 4.77 (t,  $J = 5.2$  Hz, 2H), 4.49-4.45 (m, 1H), 4.10-4.07 (m, 3H), 3.88 (d,  $J = 3.2$  Hz, 1H), 3.65-3.61 (m, 7H), 3.54-3.52 (m, 1H).  $^{13}\text{C}$  NMR ( $\text{DMSO-d}_6$ , 100 MHz)  $\delta$  159.1, 154.1, 152.4, 135.2, 113.5, 86.7, 85.2, 73.1, 70.4, 61.5. HRMS: calcd for  $\text{C}_{14}\text{H}_{23}\text{N}_6\text{O}_6$  [ $\text{M} + \text{H}^+$ ] 371.1679, found 371.1678

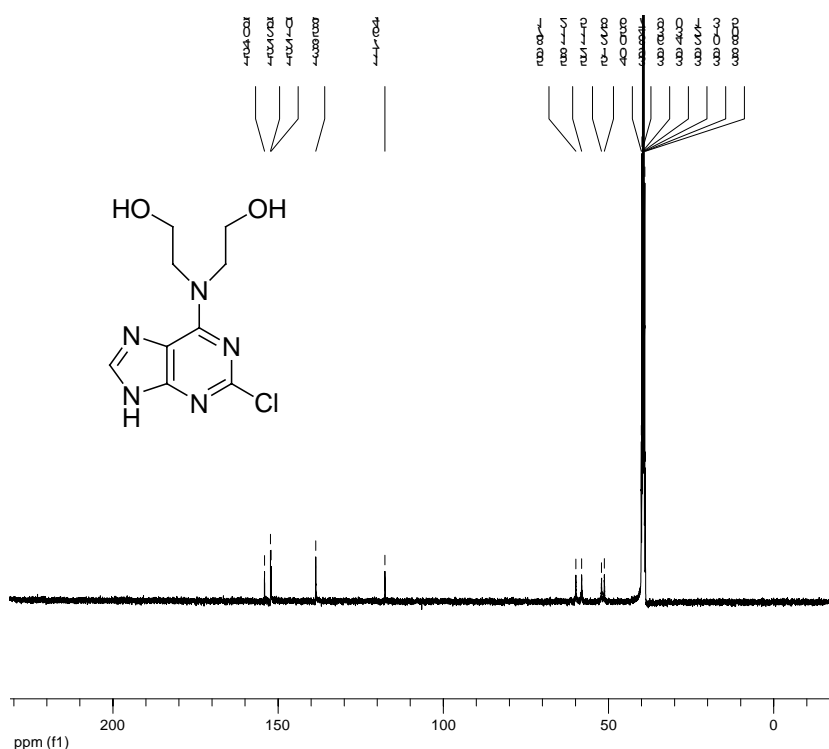
**9-Allyl-2-(N, N-diallyl)-6-[N, N-bis(2-hydroxyethyl)amino]purine (3m)**



White powder, mp 107-108 °C. <sup>1</sup>H NMR (DMSO-d<sub>6</sub>, 400 MHz) δ 7.45 (s, 1H), 6.00-5.95 (m, 1H), 5.90-5.83 (m, 2H), 5.28-5.11 (m, 6H), 4.63 (d, *J* = 6 Hz, 2H), 4.20 (d, *J* = 5.6 Hz, 4H), 4.03 (s, 4H), 3.94-3.92 (m, 4H). <sup>13</sup>C NMR (DMSO-d<sub>6</sub>, 100 MHz) δ 158.0, 156.3, 153.2, 136.0, 134.6, 132.2, 118.6, 115.9, 113.2, 61.4, 51.0, 49.3, 45.2. HRMS: calcd for C<sub>18</sub>H<sub>27</sub>N<sub>6</sub>O<sub>2</sub> [M + H<sup>+</sup>] 359.2195, found 359.2192

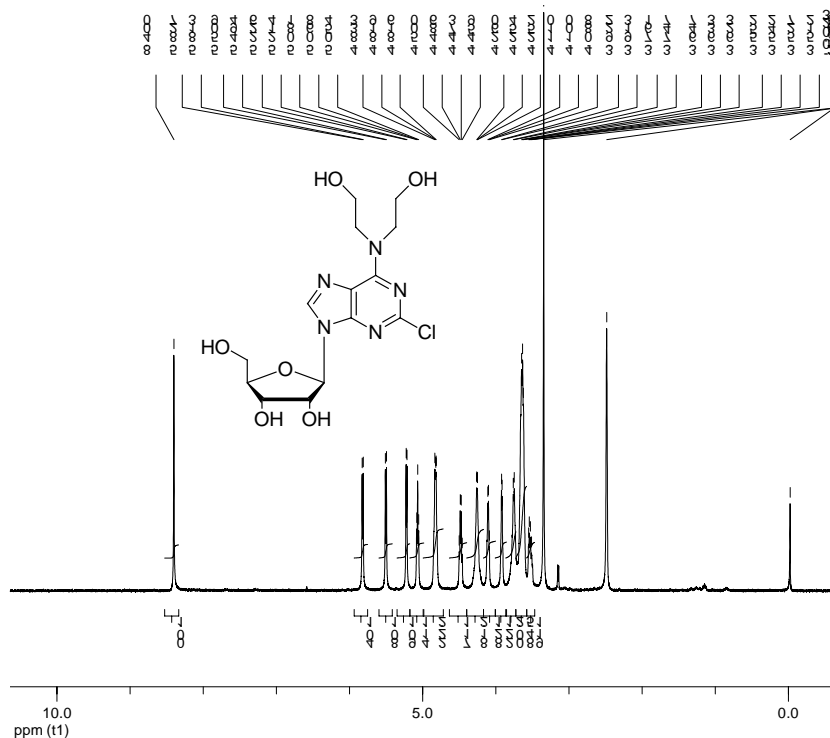


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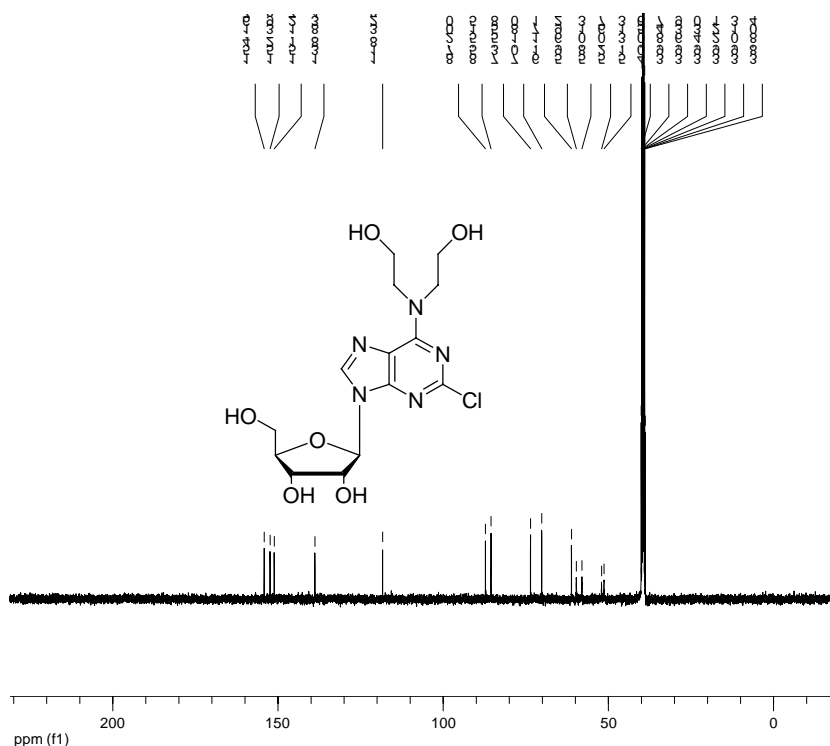


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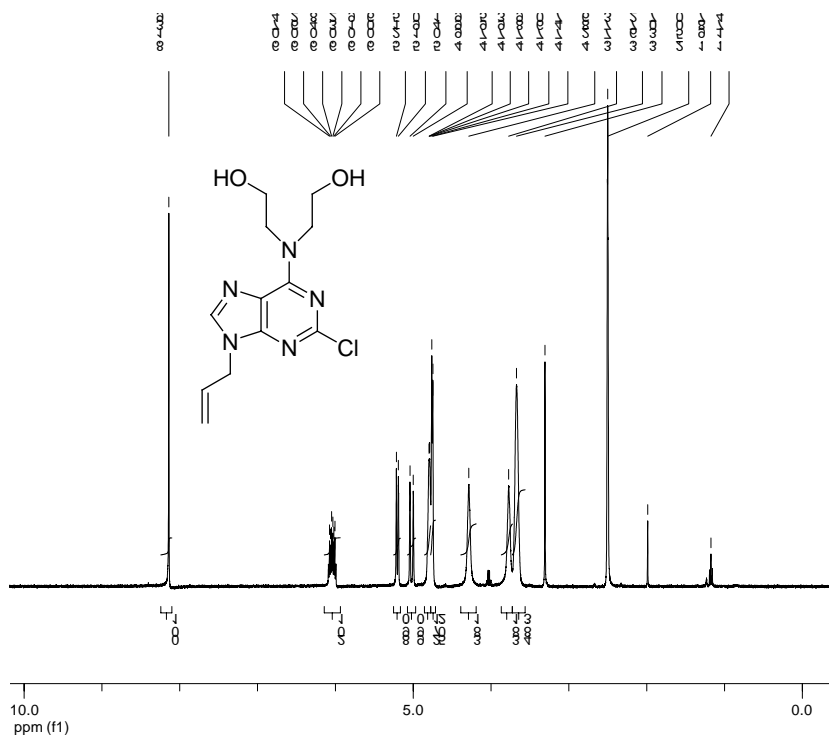




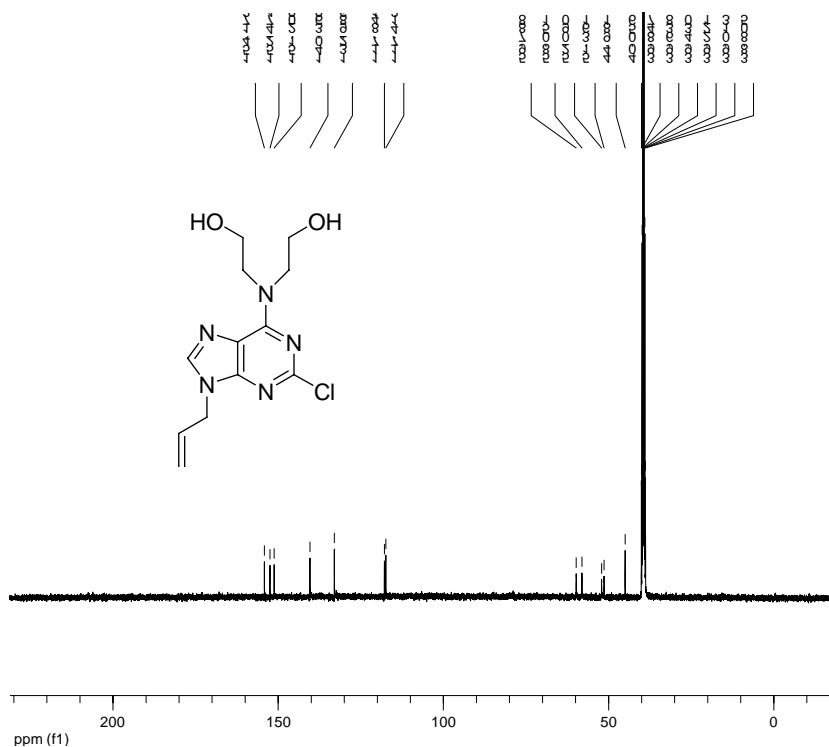
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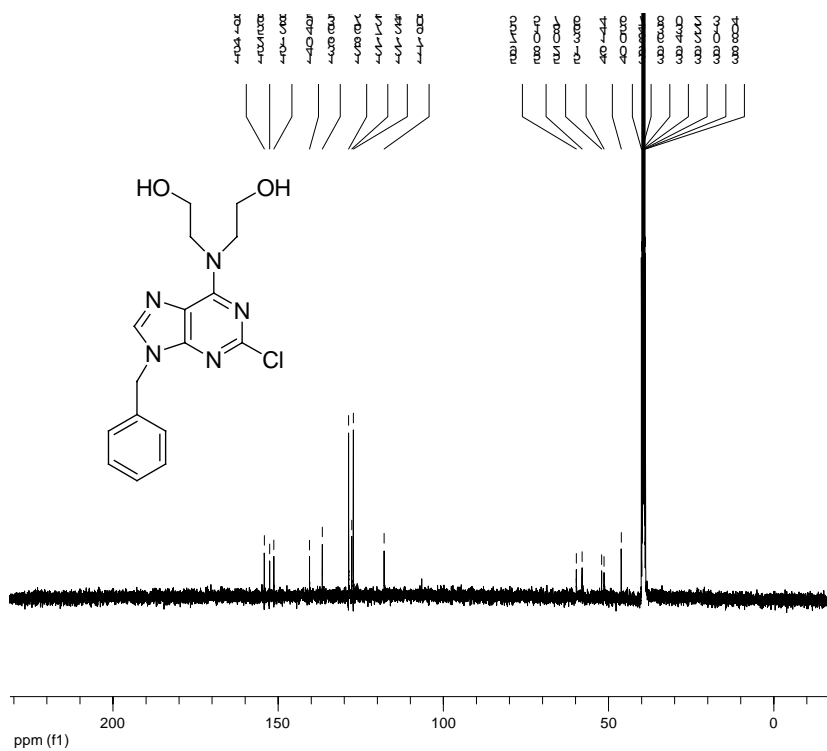
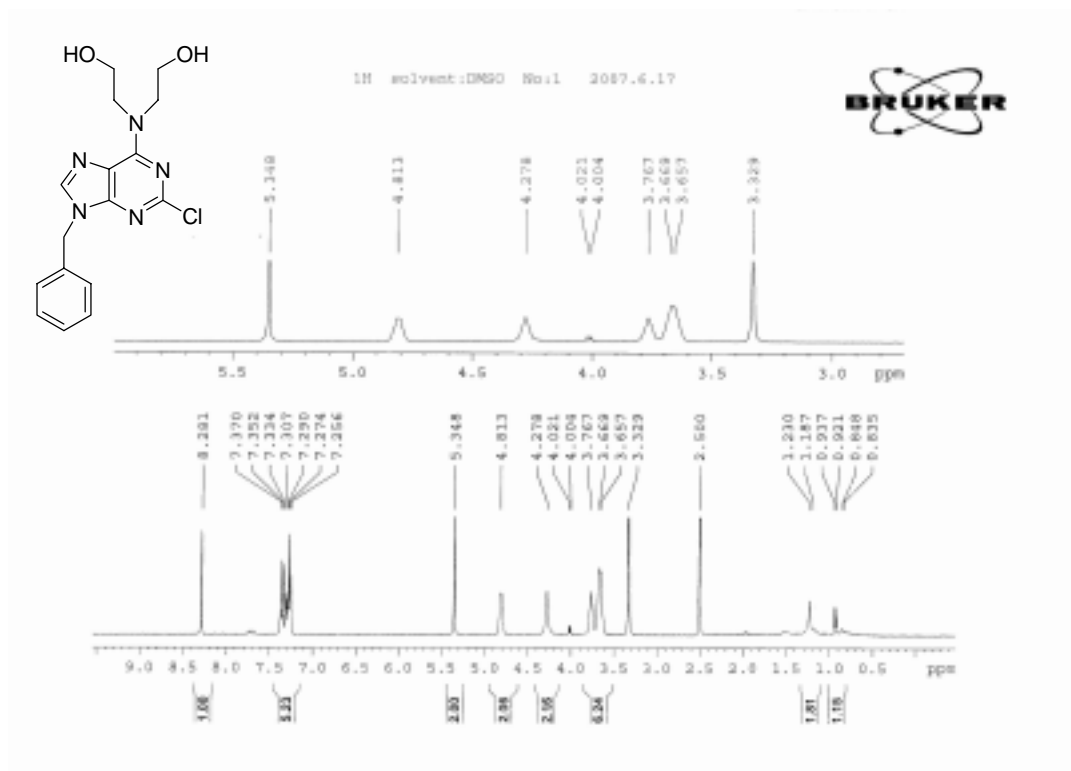
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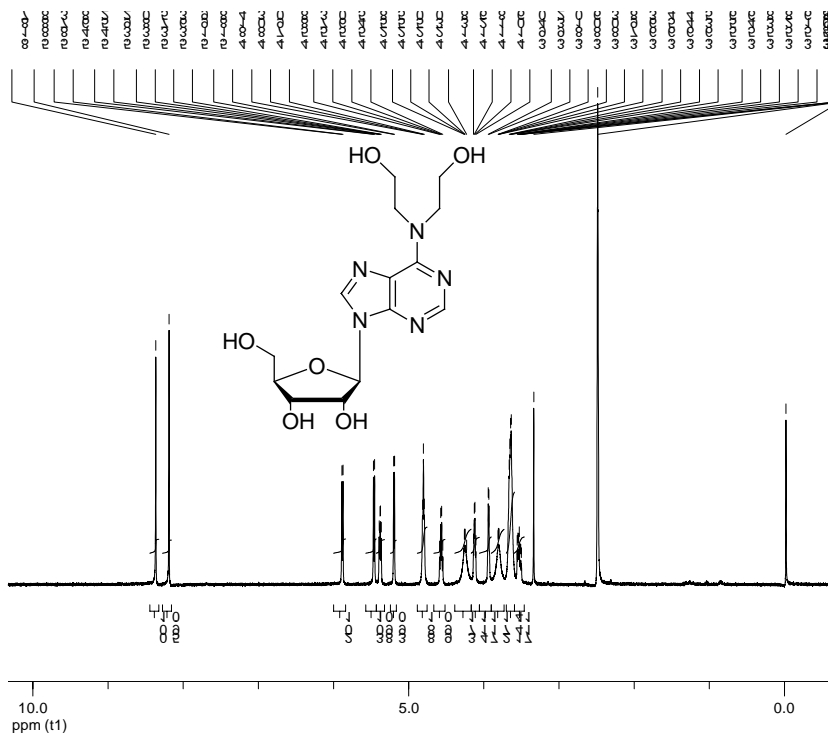


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Date: 3 Dec 2008  
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**Date:**  
3 Dec 2008

**Document's Title:**  
17

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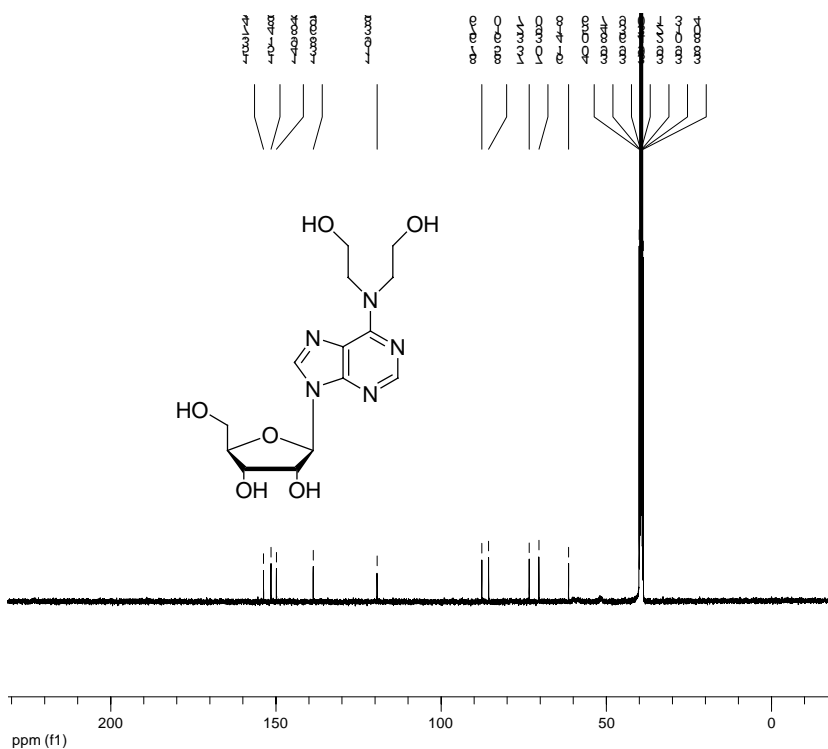
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**Acq. Date:**  
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**Date:**  
3 Dec 2008

**Document's Title:**  
27

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(f1) 0.6521

**Spectral Width (ppm):**  
(f1) 249.656

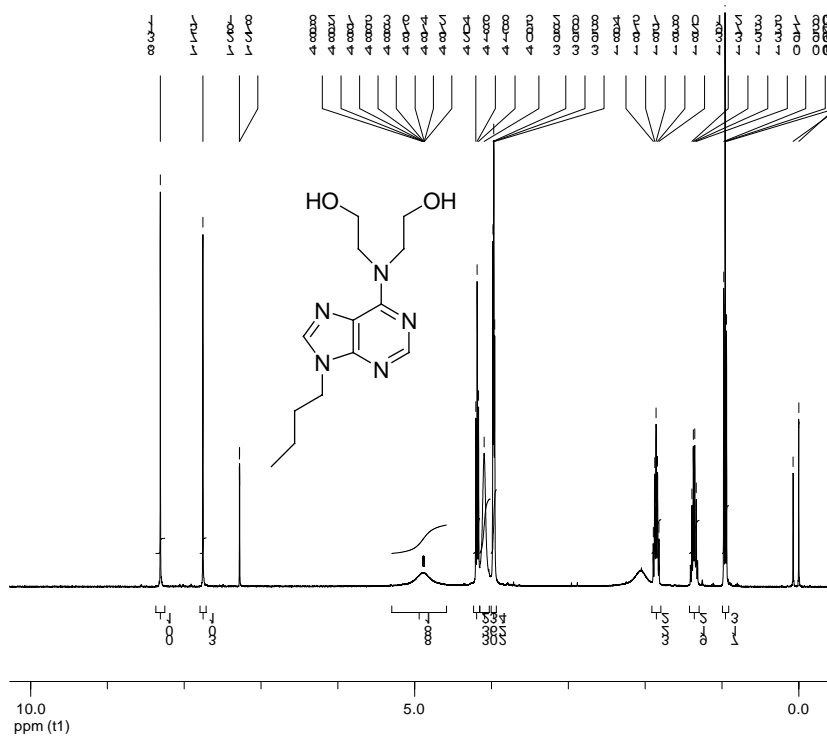
**Pulse Program:**  
ZGDC30

**Temperature:**  
303.16

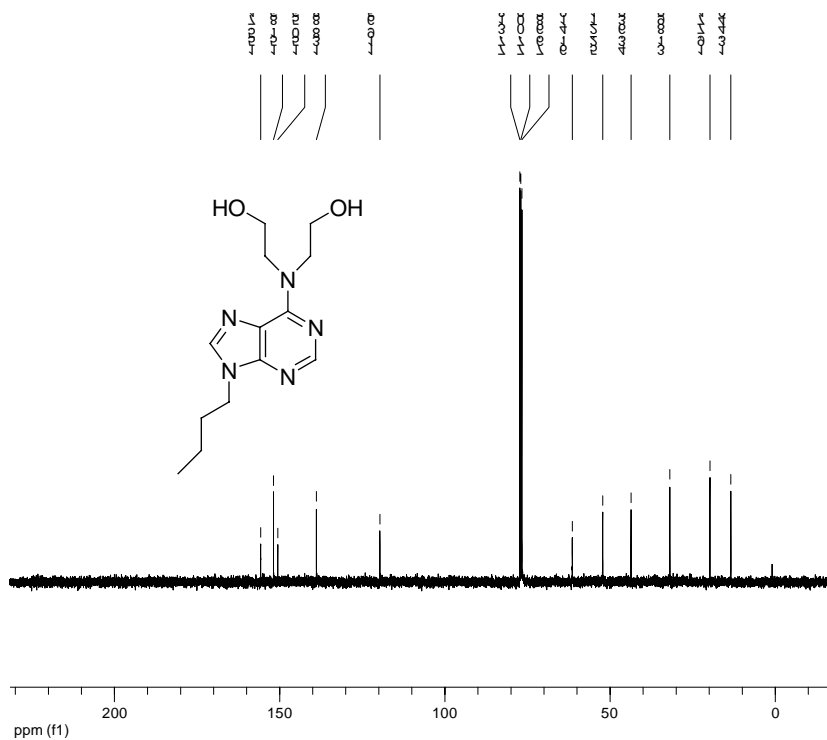
**Number of Scans:**  
2877

**Acq. Date:**  
Mon Nov 24 10:36:45 PM

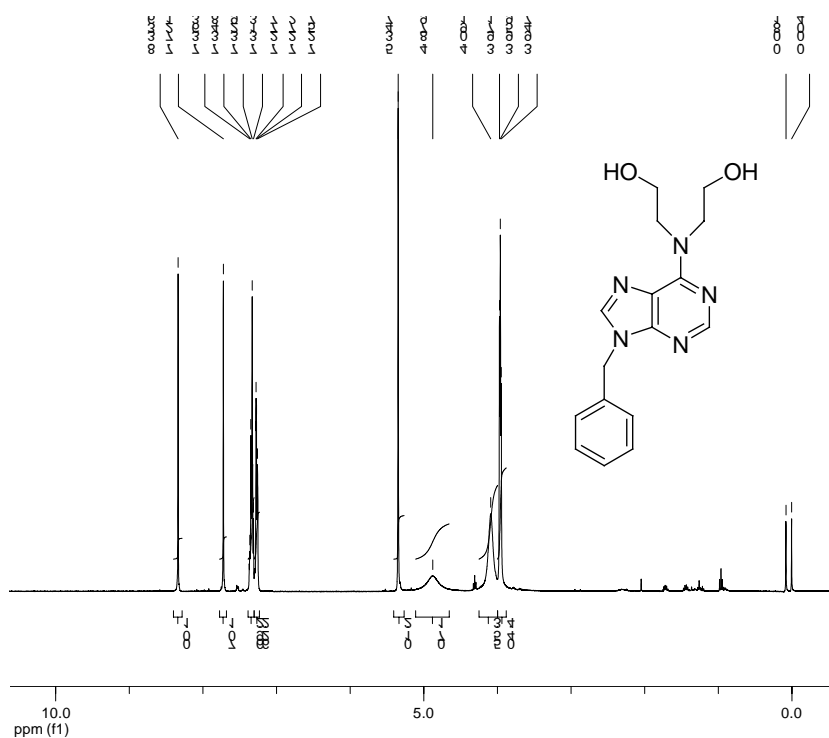




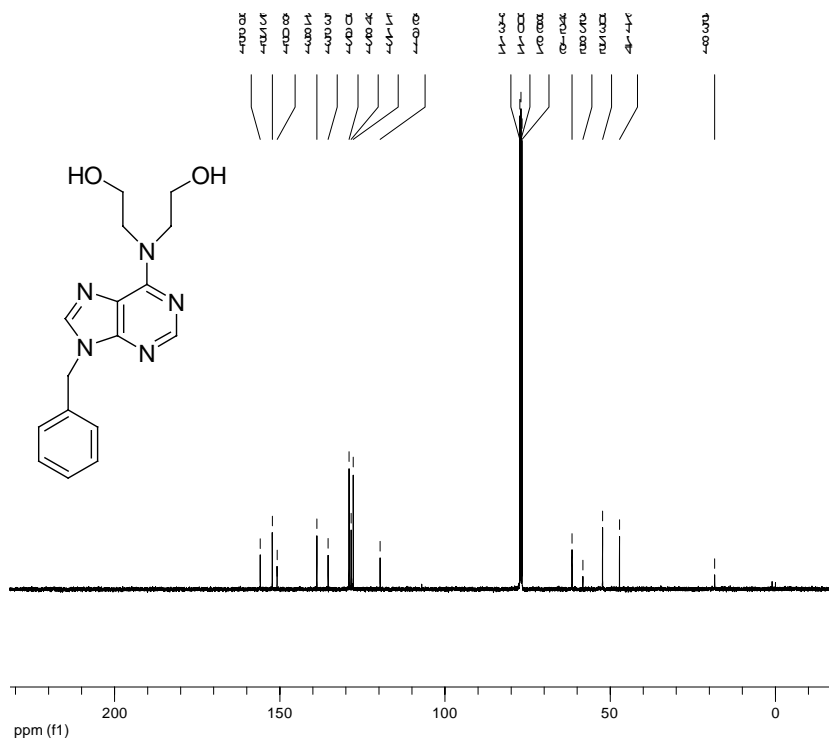
**Date:**  
3 Dec 2008  
**Document's Title:**  
12  
**Spectrum Title:**  
1H solvent:CDCl3 No:12(2) 2008.9.7  
**Frequency (MHz):**  
(f1) 400.203  
**Original Points Count:**  
(f1) 16384  
**Actual Points Count:**  
(f1) 32768  
**Acquisition Time (sec):**  
(f1) 2.0447  
**Spectral Width (ppm):**  
(f1) 20.022  
**Pulse Program:**  
ZG30  
**Temperature:**  
294.26  
**Number of Scans:**  
8  
**Acq. Date:**  
Sun Sep 07 02:37:13 PM



**Date:**  
3 Dec 2008  
**Document's Title:**  
22  
**Spectrum Title:**  
C13 solvent:CDCl3 No:2 2008.11.10  
**Frequency (MHz):**  
(f1) 100.641  
**Original Points Count:**  
(f1) 16384  
**Actual Points Count:**  
(f1) 32768  
**Acquisition Time (sec):**  
(f1) 0.6521  
**Spectral Width (ppm):**  
(f1) 249.656  
**Pulse Program:**  
ZGDC30  
**Temperature:**  
232.46  
**Number of Scans:**  
44  
**Acq. Date:**  
Mon Nov 10 11:01:29 PM



**Date:**  
3 Dec 2008  
**Document's Title:**  
11  
**Spectrum Title:**  
1H solvent:CDCl3 No:1 2008.11.10  
**Frequency (MHz):**  
(f1) 400.203  
**Original Points Count:**  
(f1) 16384  
**Actual Points Count:**  
(f1) 32768  
**Acquisition Time (sec):**  
(f1) 2.0447  
**Spectral Width (ppm):**  
(f1) 20.022  
**Pulse Program:**  
ZG30  
**Temperature:**  
291.96  
**Number of Scans:**  
8  
**Acq. Date:**  
Mon Nov 10 10:34:02 PM

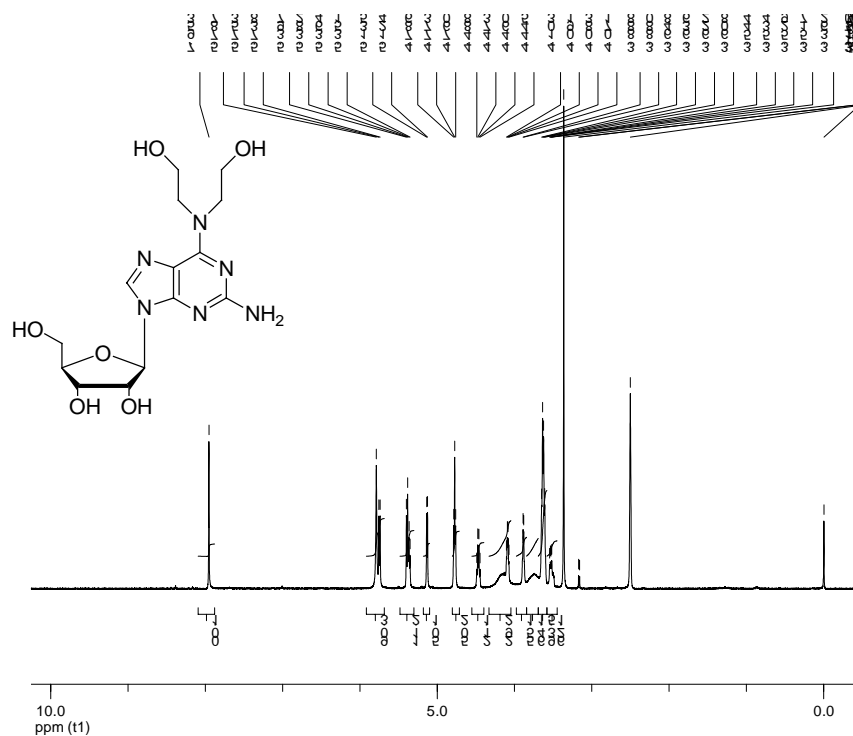


**Date:**  
3 Dec 2008  
**Document's Title:**  
28  
**Spectrum Title:**  
C13 solvent:CDCl3 No:8 2008.11.18  
**Frequency (MHz):**  
(f1) 100.641  
**Original Points Count:**  
(f1) 16384  
**Actual Points Count:**  
(f1) 32768  
**Acquisition Time (sec):**  
(f1) 0.6521  
**Spectral Width (ppm):**  
(f1) 249.656  
**Pulse Program:**  
ZGDC30  
**Temperature:**  
303.16  
**Number of Scans:**  
386  
**Acq. Date:**  
Tue Nov 25 07:58:51 AM

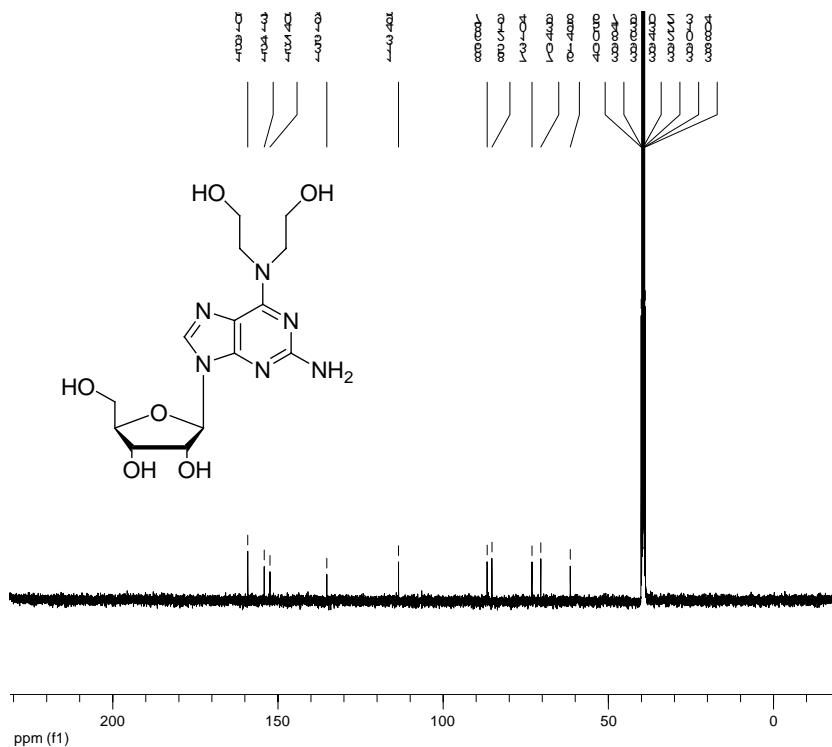




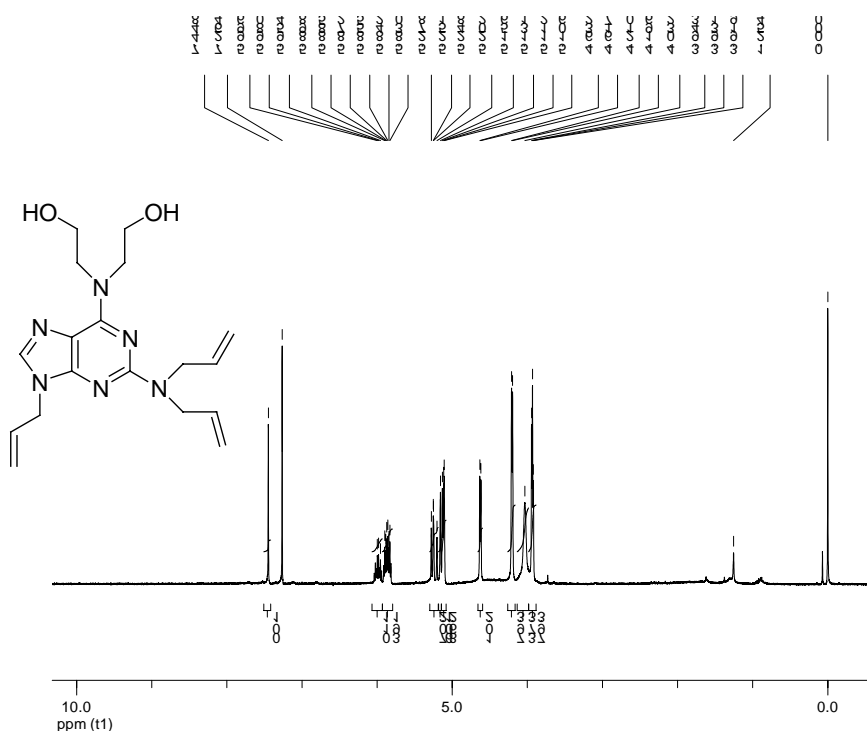




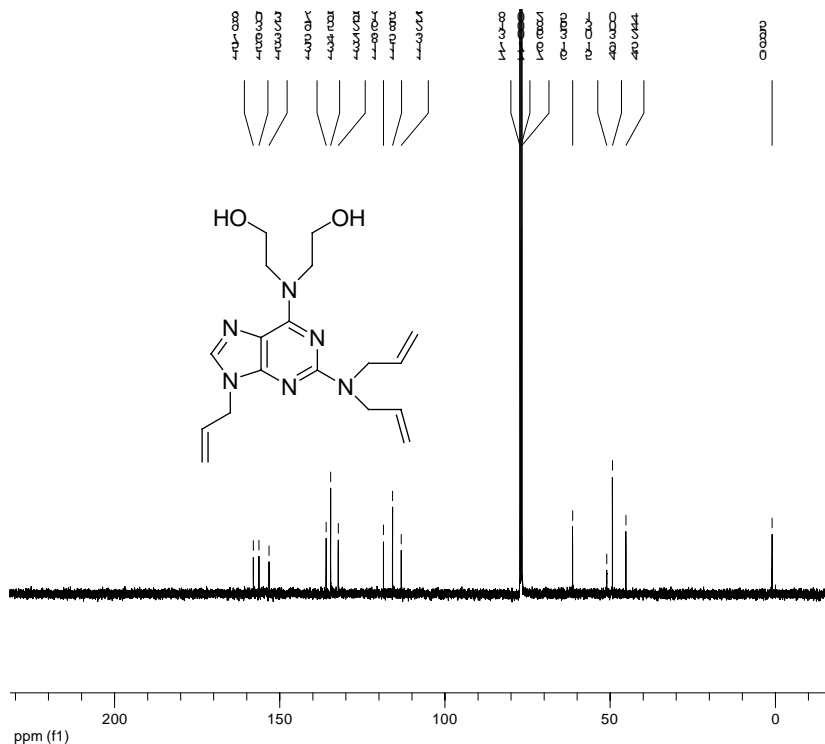
**Date:** 3 Dec 2008  
**Document's Title:** 14  
**Spectrum Title:** 1H solvent:CDCl3 No:1 2008.11.18  
**Frequency (MHz):** (f1) 400.203  
**Original Points Count:** (f1) 16384  
**Actual Points Count:** (f1) 32768  
**Acquisition Time (sec):** (f1) 2.0447  
**Spectral Width (ppm):** (f1) 20.022  
**Pulse Program:** ZG30  
**Temperature:** 290.36  
**Number of Scans:** 8  
**Acq. Date:** Tue Nov 18 09:08:35 PM



**Date:** 3 Dec 2008  
**Document's Title:** 24  
**Spectrum Title:** C13 solvent:DMSO No:4 2008.11.18  
**Frequency (MHz):** (f1) 100.641  
**Original Points Count:** (f1) 16384  
**Actual Points Count:** (f1) 32768  
**Acquisition Time (sec):** (f1) 0.6521  
**Spectral Width (ppm):** (f1) 249.656  
**Pulse Program:** ZGDC30  
**Temperature:** 303.16  
**Number of Scans:** 509  
**Acq. Date:** Mon Nov 24 08:16:11 PM



**Date:** 3 Dec 2008  
**Document's Title:** 14  
**Spectrum Title:** 1H solvent:CDCl3 No:4 2008.9.7  
**Frequency (MHz):** (f1) 400.203  
**Original Points Count:** (f1) 16384  
**Actual Points Count:** (f1) 32768  
**Acquisition Time (sec):** (f1) 2.0447  
**Spectral Width (ppm):** (f1) 20.022  
**Pulse Program:** Z330  
**Temperature:** 294.25  
**Number of Scans:** 8  
**Acq. Date:** Sun Sep 07 02:41:34 PM



**Date:** 3 Dec 2008  
**Document's Title:** 22  
**Spectrum Title:** C13 solvent:CDCl3 No:2 2008.11.12  
**Frequency (MHz):** (f1) 100.641  
**Original Points Count:** (f1) 16384  
**Actual Points Count:** (f1) 32768  
**Acquisition Time (sec):** (f1) 0.6521  
**Spectral Width (ppm):** (f1) 249.656  
**Pulse Program:** Z33DC3  
**Temperature:** 291.95  
**Number of Scans:** 252  
**Acq. Date:** Thu Nov 13 09:58:39 AM