Supplementary Material (ESI) for Green Chemistry

The Supporting Information

Microwave Irradiation C6-Functionalization of 6-Chloropurine Nucleosides with Various Mild Nucleophiles under Solvent-Free Condition

Hai-Ming Guo,*[a] Peng-Yang Xin,[a] Hong-Ying Niu,[b] Dong-Chao Wang,[a] Yi Jiang,[a] and Gui-Rong Qu*[a]

[a] *College of Chemistry and Environmental Science, Key Laboratory of Green Chemical Media and Reactions of Ministry of Education, Henan Normal University, Xinxiang, 453007, China. Fax: +86-3733329276; E-mail: guohm518@hotmail.com; quguir@sina.com*
[b] *School of Chemistry and Chemical Engineering, Henan Institute of Science and Technology, Xinxiang 453003, China*

General.....S1
Typical experimental procedure.....S2
Characterization of compounds.....S3
Copies of ^{1}H and ^{13}C NMR spectra.....S11

General: Melting points were recorded with a micro melting point apparatus and uncorrected. NMR spectra were recorded with a 400 NMR spectrometer for ^{1}H-NMR, 100 MHz for ^{13}C-NMR. Proton chemical shifts δ were given in ppm relative to tetramethylsilane (0.00 ppm) in $CDCl_3$ or to the residual proton signals of the deuterated solvent DMSO-d6 (2.50 ppm) for ^{1}H and ^{13}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 (GF254) was used as the stationary phase. All reactions were monitored by thin layer chromatography (TLC). All regents were purchased from commercial sources and purified commonly before used.

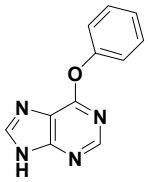
All microwave irradiation experiments were carried out in the cavity of a commercially available single-mode microwave synthesis apparatus equipped with a highly sensitive infrared sensor for temperature control and measurement (MAS-I, Sineo Microwave Chemical Technology Co. Ltd., Shanghai, P. R. of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

Typical Experimental Procedure for the Reaction of 6-Halopurine Nucleosides and various Nucleophiles under Microwave Irradiation without Solvent.

6-Chloropurine **1a** (1 mmol) and phenol (1.5 mmol), K_2CO_3 (2 mmol) were put in a ceramic mortar and then sufficient rubbing was needed to make the reactant well-distributed. Then the mixture was put into the cavity of the microwave synthesis apparatus and irradiated at 400 W at 90 ^{o}C for 5 min. After completion of the reaction, the mortar was cooled to room temperature, followed by TLC examination, the colored material was dissolved in ethyl acetate and adsorbed on silica gel and purified by silica gel column chromatography to give **3a** using CH_2Cl_2:CH_3OH = 30:1 (v/v) as the eluent.

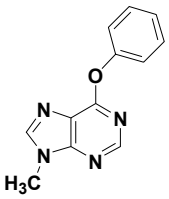
Characterization of compounds

6-phenoxy-9H-purine (3a)



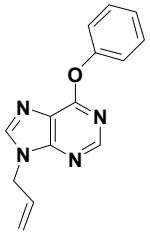
White powder, mp 197-200 °C. ^{1}H NMR (DMSO-d_6, 400 MHz) δ 13.55 (s, 1H), 8.46 (s, 1H), 8.40 (s, 1H), 7.48-7.29 (m, 5H). ^{13}C NMR (DMSO-d_6, 100 MHz) δ 152.7, 151.6, 130.1, 125.9, 122.4 . HRMS: calcd for $C_{11}H_9N_4O$ [M + H^+] 213.0776, found 213.0779.

9-methyl-6-phenoxy-9H-purine (3b)



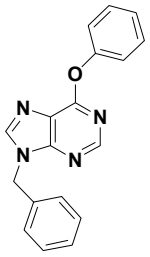
White powder, mp 150-152 °C. ^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.52 (s, 1H), 8.00 (s, 1H), 7.48-7.27 (m, 5H), 3.92 (s, 3H). ^{13}C NMR ($CDCl_3$, 100 MHz) δ 160.2, 153.4, 152.4, 152.1, 143.6, 129.6, 125.7, 121.8, 121.5, 30.1. HRMS: calcd for $C_{12}H_{10}N_4NaO$ [M + Na^+] 249.0752, found 249.0754.

9-allyl-6-phenoxy-9H-purine (3c)



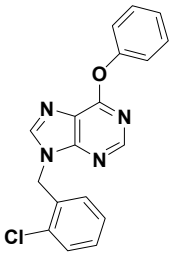
White powder, mp 89-91 °C. ^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.51 (s, 1H), 8.03 (s, 1H), 7.47-7.30 (m, 5H), 6.11-6.02 (m, 1H), 5.35 (d, J = 10.4 Hz, 1H), 5.24(d, J = 17.2 Hz, 2H), 4.90 (d, J = 5.6Hz, 2H). ^{13}C NMR ($CDCl_3$, 100 MHz) δ 160.3, 152.9, 152.4, 152.1, 142.9, 131.4, 129.6, 125.8, 121.8, 121.5, 119.3, 46.1. HRMS: calcd for $C_{14}H_{12}N_4NaO$ [M + Na^+] 275.0909, found 275.0918.

9-benzyl-6-phenoxy-9H-purine (3d)



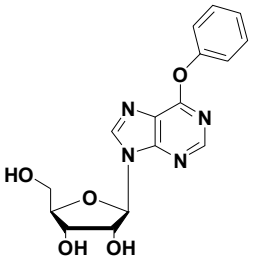
White powder, mp 111-113 ^{o}C. ^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.55 (s, 1H), 8.01(s, 1H), 7.48-7.27 (m, 10H), 5.45 (s, 2H). ^{13}C NMR ($CDCl_3$, 100 MHz) δ 160.3, 153.2, 152.4, 152.3, 142.9, 135.1, 129.6, 129.1, 128.6, 127.9, 125.8, 121.8, 121.5, 47.6. HRMS: calcd for $C_{18}H_{15}N_4O$ [M + H^+] 303.1246, found 303.1239.

9-(2-chlorobenzyl)-6-phenoxy-9H-purine (3e)



White powder, mp 120-121 ^{o}C. ^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.54 (s, 1H), 8.09 (s, 1H), 7.48-7.23 (m, 9H), 5.57 (s, 2H). ^{13}C NMR ($CDCl_3$, 100 MHz) δ 160.3, 153.2, 152.4, 152.3, 143.2, 133.5, 132.7, 130.3, 130.1, 130.0, 129.6, 127.5, 125.8, 121.8, 121.4, 45.2. HRMS: calcd for $C_{18}H_{14}ClN_4O$ [M + H^+] 337.0856, found 337.0847.

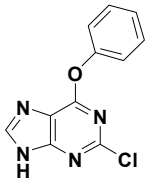
6-phenoxy-9-(beta-D-ribofuranosyl) purine (3f)



Yellow oily liquid. ^{1}H NMR (DMSO-d_6, 400 MHz) δ 8.77 (s, 1H), 8.48 (s, 1H), 7.50-7.28 (m, 5H), 6.04 (d, J = 5.6 Hz, 1H), 5.56 (d, J = 6 Hz, 1H), 5.27 (d, J = 5.2 Hz, 1H), 5.15(t, J =5.6 Hz, 1H), 4.62 (q, J = 8.4 Hz, 1H), 4.19 (q, J = 6.4 Hz, 1H), 3.99 (d, J = 3.6 Hz, 1H), 3.73-3.55 (m, 2H). ^{13}C NMR (DMSO-d_6, 100 MHz) δ 159.4, 152.7, 152.1, 151.4, 143.3, 129.6, 125.5, 121.8, 118.7, 115.1,

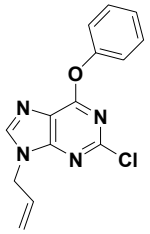
87.8, 85.6, 73.7, 70.2, 61.2. HRMS: calcd for $C_{16}H_{16}N_4NaO_5$ [M + Na^+] 367.1018, found 367.1009.

2-chloro-6-phenoxy-9H-purine (3g)



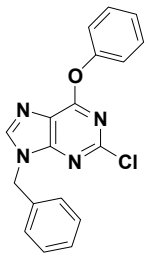
White powder, mp 212-215 °C. ^{1}H NMR (DMSO-d_6, 400 MHz) δ 13.55 (s, 1H), 8.46 (s, 1H), 8.40 (s, 1H), 7.48-7.29 (m, 5H). ^{13}C NMR (DMSO-d_6, 100 MHz) δ 152.3, 151.1, 130.3, 126.4, 122.1. HRMS: calcd for $C_{11}H_8ClN_4O$ [M + H^+] 247.0387, found 247.0393.

9-allyl-2-chloro-6-phenoxy-9H-purine (3h)



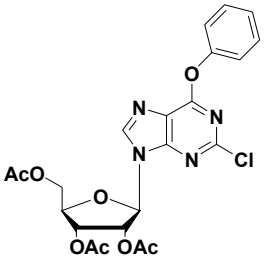
White powder, mp 99-101 °C. ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.87 (s, 1H), 7.46-7.27 (m, 5H), 6.09-5.99 (m, 1H), 5.36 (d, J = 10 Hz, 1H), 5.31(d, J = 18.8 Hz, 1H), 4.85 (d, J = 6 Hz, 2H). ^{13}C NMR ($CDCl_3$, 100 MHz) δ 160.1, 154.1, 153.0, 152.1, 143.2, 131.1, 129.5, 125.9, 121.5, 120.5, 119.8, 46.1. HRMS: calcd for $C_{14}H_{12}ClN_4O$ [M + H^+] 287.0700, found 287.0699.

9-benzyl-2-chloro-6-phenoxy-9H-purine (3i)



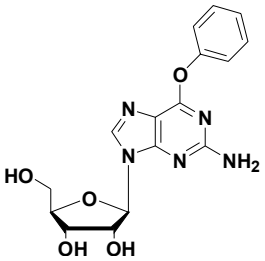
White powder, mp 118-120 °C. ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.93 (s, 1H), 7.47-7.28 (m, 10H), 5.41 (s, 2H). ^{13}C NMR ($CDCl_3$, 100 MHz) δ 152.4, 152.3, 142.9, 135.1, 129.6, 129.1, 128.6, 127.8, 125.8, 121.8, 121.5, 47.6. HRMS: calcd for $C_{18}H_{14}ClN_4O$ [M + H^+] 337.0856, found 337.0854.

9-(β-D-triacetoxyribofuranosyl)-2-chloro-6-phenoxy-9H-purine (3j)



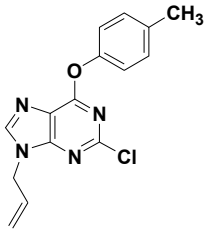
Colorless oil. ^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.19 (s, 1H), 7.44-7.15 (m, 5H), 6.22 (d, J =5.2 Hz, 1H), 5.83 (t, J = 5.6 Hz, 1H), 5.60 (t, J = 5.2 Hz, 1H), 4.45 (t, J = 4 Hz, 1H), 4.40 (t, J = 4.4 Hz, 2H), 2.17 (s, 3H), 2.12 (s, 3H), 2.07 (s, 3H). ^{13}C NMR ($CDCl_3$, 100 MHz) δ 170.4, 169.7, 169.5, 160.2, 156.3, 153.5, 151.9, 141.7, 129.6, 129.5, 126.1, 121.4, 120.0, 115.4, 86.3, 80.5, 73.3, 70.6, 63.0, 20.8, 20.5, 20.4. HRMS: calcd for $C_{22}H_{21}ClN_4NaO_8$ [M + Na^+] 527.0946, found 527.0921.

2-amino-6-phenoxy-9-(β-D-ribofuranosyl) purine (3k)



White powder, mp 110-111 ^{o}C. ^{1}H NMR (DMSO-d_6, 400 MHz) δ 8.22 (s, 1H), 7.45-7.22 (m, 5H), 6.44 (s, 2H), 5.82 (d, J = 6 Hz, 1H), 5.42 (d, J = 6 Hz, 1H), 5.15 (d, J = 4.8 Hz, 1H), 5.07 (t, J = 5.6 Hz, 1H), 4.49 (q, J = 8.4 Hz, 1H), 4.12 (q, J = 6.8 Hz, 1H), 3.90 (d, J = 6 Hz, 1H), 3.67-3.53 (m, 2H). ^{13}C NMR (DMSO-d_6, 100 MHz) δ 160.2, 155.7, 153.0, 139.4, 130.0, 125.6, 122.1, 114.5, 87.0, 85.7, 74.0, 70.8, 61.8. HRMS: calcd for $C_{16}H_{17}N_5NaO_5$ [M + Na^+] 382.1127, found 382.1115.

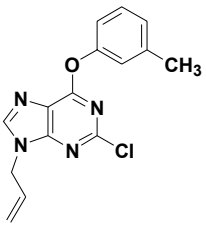
9-allyl-2-chloro-6-(p-tolyloxy)-9H-purine (4a)



White powder, mp 125-127 ^{o}C. ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.98 (s, 1H), 7.24-7.10 (m, 4H), 6.08-6.0 1(m, 1H), 5.37 (d, J = 10.4 Hz, 1H), 5.27 (d, J = 16.8 Hz, 1H), 4.86 (d, J = 5.6 Hz, 2H),

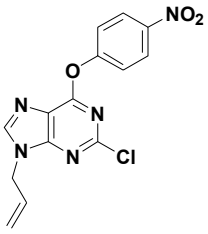
2.39 (s, 3H). ^{13}C NMR ($CDCl_3$, 100 MHz) δ 160.3, 154.0, 153.0, 149.8, 143.2, 135.5, 131.1, 130.0, 121.1, 120.5, 119.7, 46.1, 20.9. HRMS: calcd for $C_{15}H_{13}ClN_4NaO$ [M + Na^+] 323.0676, found 323.0677.

9-allyl-2-chloro-6-(m-tolyloxy)-9H-purine (4b)



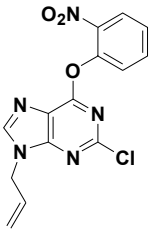
White powder, mp 64-65 ^{o}C. ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.98 (s, 1H), 7.34-7.09 (m, 4H), 6.08-6.01 (m, 1H), 5.37 (d, J=10.4 Hz, 1H), 5.27 (d, J=17.2 Hz, 1H), 4.86 (d, J=5.6 Hz, 2H), 2.40 (s, 3H). ^{13}C NMR ($CDCl_3$, 100 MHz) δ 160.2, 154.1, 153.1, 152.1, 143.2, 139.7, 131.1, 129.2, 126.7, 121.9, 120.5, 119.8, 118.4, 46.1, 21.4. HRMS: calcd for $C_{15}H_{13}ClN_4NaO$ [M + Na^+] 323.0676, found 323.0675.

9-allyl-2-chloro-6-(4-nitrophenoxy)-9H-purine (4c)



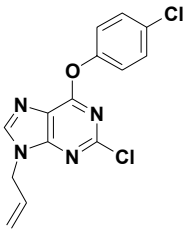
White powder, mp 131-133 ^{o}C. ^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.34 (d, J=9.2 Hz, 2H), 8.04 (s, 1H), 7.48 (d, J=9.2 Hz, 2H), 6.10-6.00 (m, 1H), 5.39 (d, J=10 Hz, 1H), 5.29 (d, J=16.8 Hz, 1H), 4.88 (d, J=6 Hz, 2H). ^{13}C NMR ($CDCl_3$, 100 MHz) δ 158.8, 156.7, 154.6, 152.7, 145.3, 144.1, 130.8, 125.4, 122.2, 120.4, 120.1, 46.3. HRMS: calcd for $C_{14}H_{11}ClN_5O_3$ [M + H^+] 332.0550, found 332.0553.

9-allyl-2-chloro-6-(2-nitrophenoxy)-9H-purine (4d)



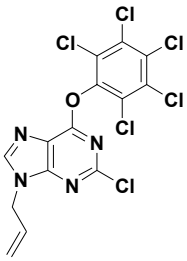
White powder, mp 177-179 ^{o}C. ^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.20 (q, J=4.8 Hz, 1H), 8.03 (s, 1H), 7.77-7.73 (m, 1H), 7.52-7.44 (m, 2H), 6.11-6.01 (m, 1H), 5.39 (d, J=10 Hz, 1H), 5.31(d, J=17.2 Hz, 1H), 4.87(d, J=6 Hz, 2H). ^{13}C NMR ($CDCl_3$, 100 MHz) δ 159.1, 154.5, 152.7, 145.1, 143.9, 141.9, 135.1, 130.9, 126.9, 126.1, 125.3, 120.1, 120.1, 46.3. HRMS: calcd for $C_{14}H_{10}ClN_5NaO_3$ [M + Na^+] 354.0370, found 354.0356.

9-allyl-2-chloro-6-(4-chlorophenoxy)-9H-purine (4e)



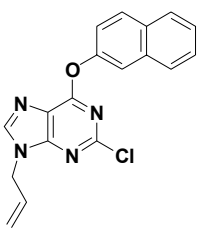
White powder, mp 101-103 ^{o}C. ^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.00 (s, 1H), 7.42-7.22 (m, 4H), 6.08-5.99 (m, 1H), 5.36 (d, J=10.4 Hz, 1H), 5.26 (d, J=17.2 Hz, 1H), 4.85 (d, J=5.6 Hz, 2H). ^{13}C NMR ($CDCl_3$, 100 MHz) δ 159.7, 154.2, 152.9, 150.5, 143.5, 131.2, 131.0, 129.6, 122.9, 120.3, 119.9, 46.2. HRMS: calcd for $C_{14}H_{11}Cl_2N_4O$ [M + H^+] 321.0304, found 321.0300.

9-allyl-2-chloro-6-(perchlorophenoxy)-9H-purine (4f)



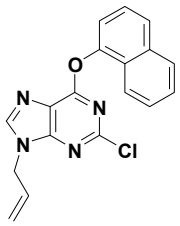
White powder, mp 209-211 ^{o}C. ^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.06 (s, 1H), 6.11-6.01 (m, 1H), 5.41 (d, J=10.4 Hz, 1H), 5.34 (d, J=17.2 Hz, 1H), 4.89 (d, J=6 Hz, 2H). ^{13}C NMR ($CDCl_3$, 100 MHz) δ 157.6, 154.7, 152.8, 145.0, 144.1, 132.1, 131.8, 130.7, 128.0, 120.4, 119.7, 46.4. HRMS: calcd for $C_{14}H_6Cl_6N_4NaO$ [M + Na^+] 478.8565, found 478.8559.

9-allyl-2-chloro-6-(naphthalen-2-yloxy)-9H-purine (4g)



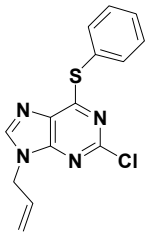
White powder, mp 143-144 ^{o}C. ^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.01 (s, 1H), 7.93-7.83 (m, 3H), 7.75 (d, J=2 Hz, 1H), 7.52-7.41 (m, 3H), 6.11-6.01 (m, 1H), 5.38 (d, J=10.4 Hz, 1H), 5.29 (d, J=17.2 Hz, 1H), 4.87 (d, J=5.6 Hz, 2H). ^{13}C NMR ($CDCl_3$, 100 MHz) δ 160.2, 154.1, 153.0, 149.8, 143.4, 133.9, 131.5, 131.1, 129.5, 127.8, 127.7, 126.6, 125.8, 121.2, 120.5, 119.8, 118.4, 46.2. HRMS: calcd for $C_{18}H_{14}ClN_4O$ [M + H^+] 337.0851, found 337.0838.

9-allyl-2-chloro-6-(naphthalen-1-yloxy)-9H-purine (4h)



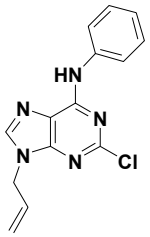
Light yellow powder, mp 116-117 ^{o}C. ^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.02 (s, 1H), 7.97 (d, J=8.4 Hz, 1H), 7.91 (d, J=8 Hz, 1H), 7.81 (d, J=8 Hz, 1H), 7.55-7.41 (m, 4H), 6.10-6.00 (m, 1H), 5.37(d, J=10.4 Hz, 1H), 5.29 (d, J=17.2 Hz, 1H), 4.86 (d, J=6 Hz, 2H). ^{13}C NMR ($CDCl_3$, 100 MHz) δ 160.7, 154.3, 153.2, 148.1, 143.4, 134.8, 131.1, 128.0, 126.9, 126.5, 126.4, 126.1, 125.5, 121.5, 120.4, 119.9, 118.0, 46.2. HRMS: calcd for $C_{18}H_{14}ClN_4O$ [M + H^+] 337.0851, found 337.0843.

9-allyl-2-chloro-6-(phenylthio)-9H-purine (4j)

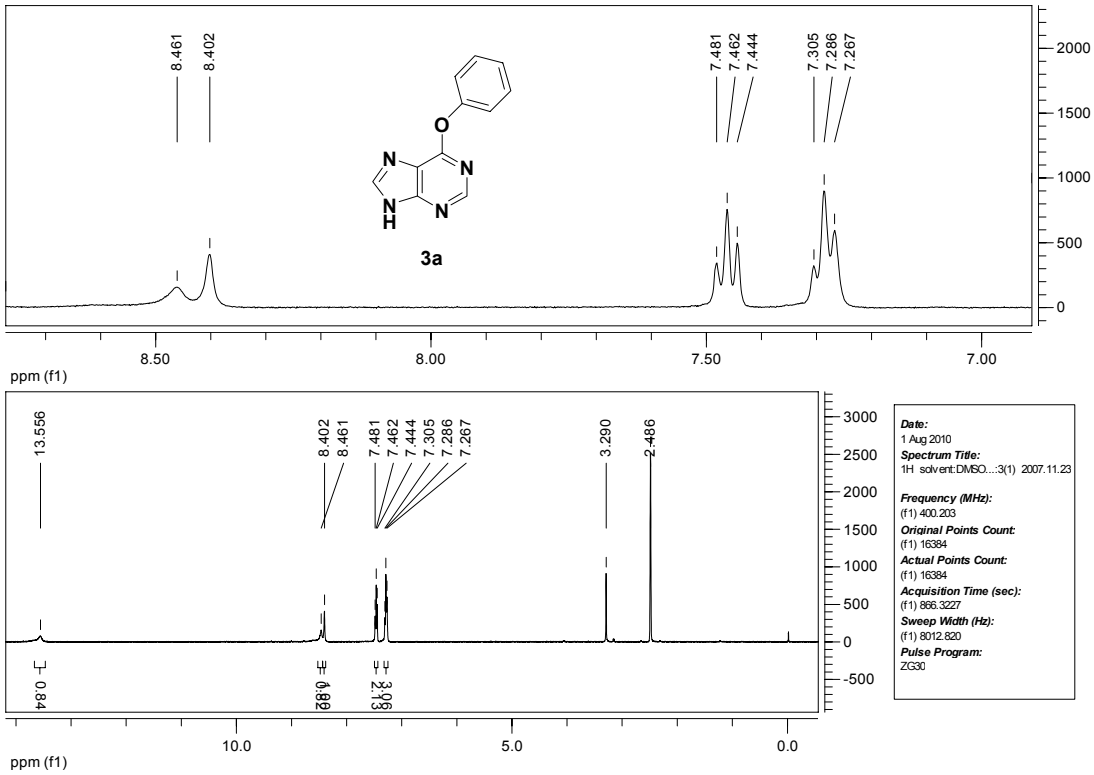


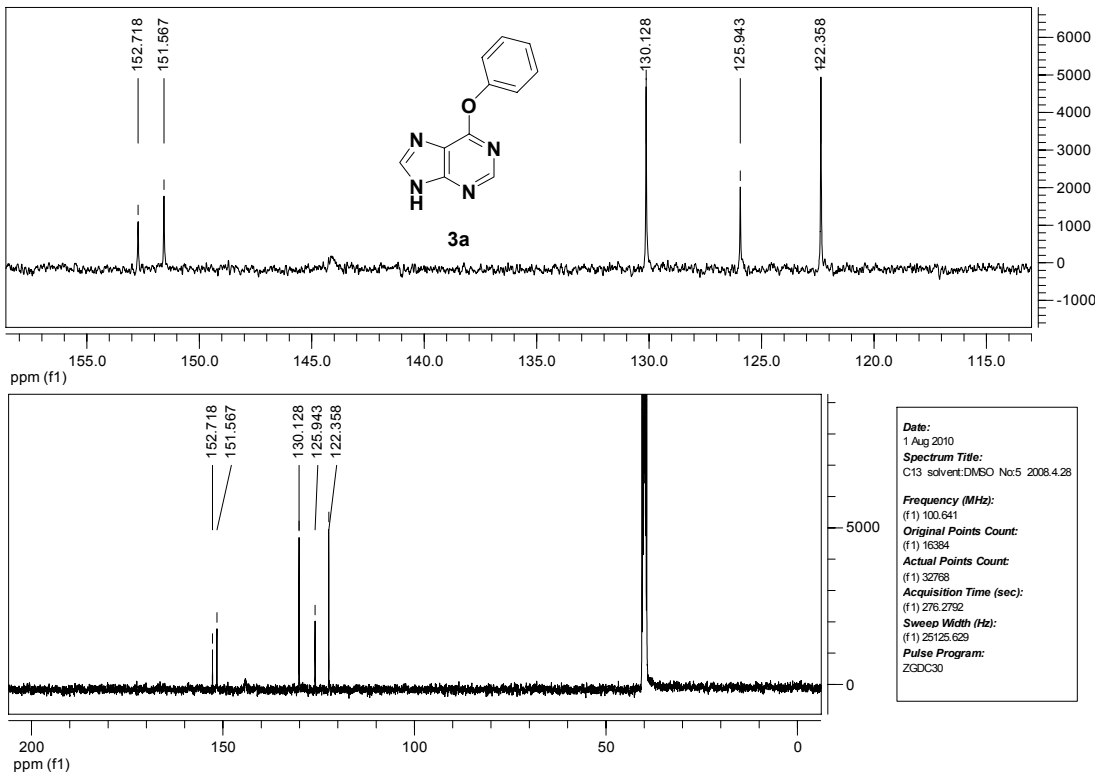
White powder, mp 89-91 ^{o}C. ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.95 (s, 1H), 7.66-7.45 (m, 5H), 6.06-5.96 (m, 1H), 5.35 (d, J=10.4 Hz, 1H), 5.24 (d, J=16.8 Hz, 1H), 4.81 (d, J=5.6 Hz, 2H). ^{13}C NMR ($CDCl_3$, 100 MHz) δ 162.7, 153.8, 150.3, 143.3, 135.3, 131.0, 129.7, 129.2, 126.5, 119.9, 46.0. HRMS: calcd for $C_{14}H_{11}ClN_4NaS$ [M + Na^+] 325.0285, found 325.0284.

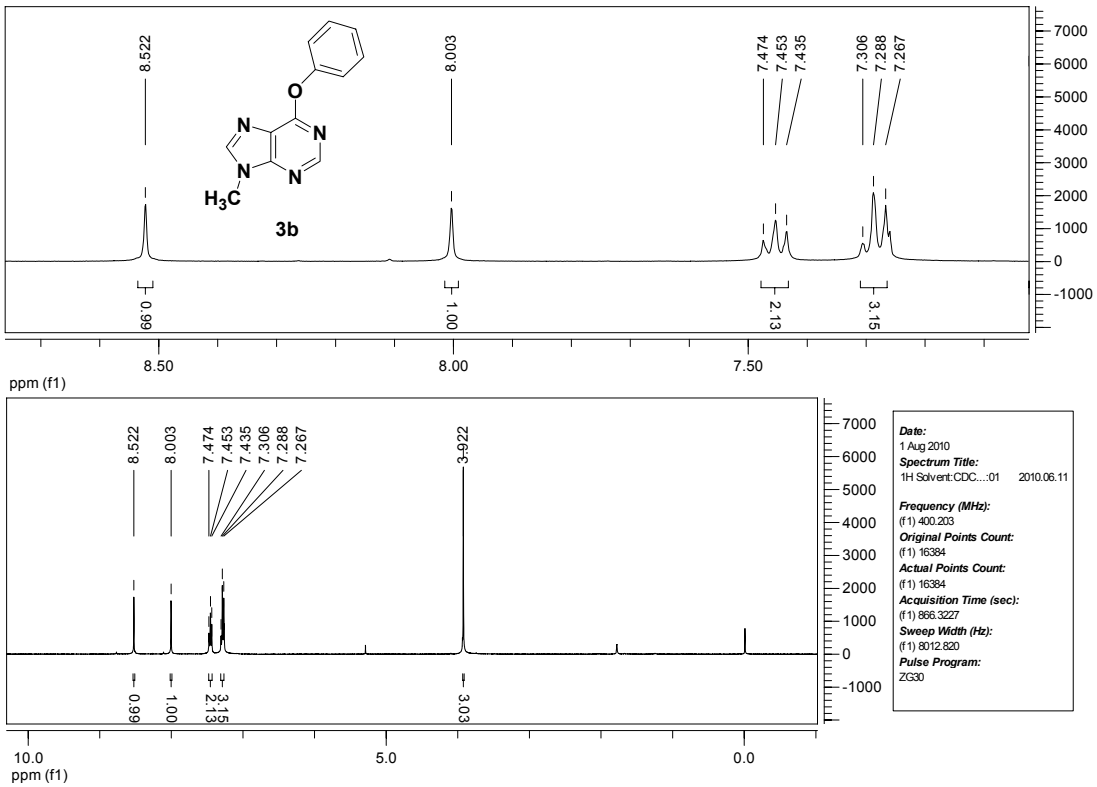
9-allyl-2-chloro-N-phenyl-9H-purin-6-amine (4k)

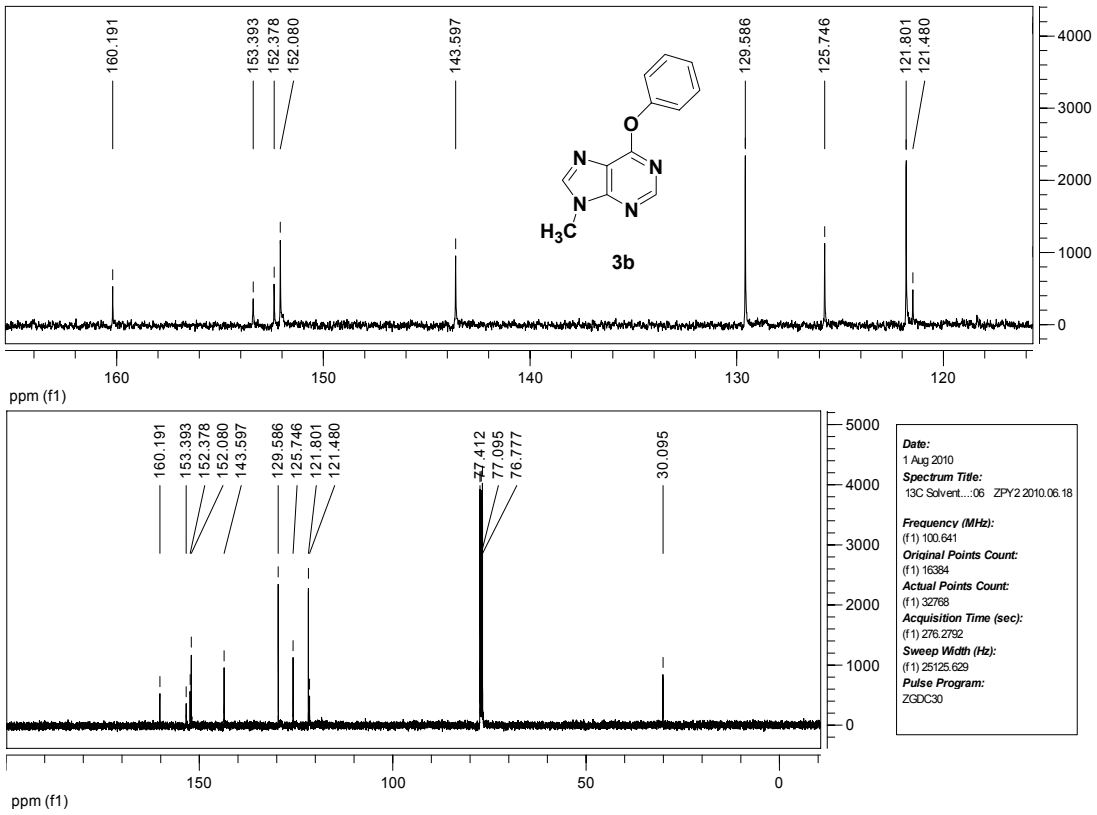


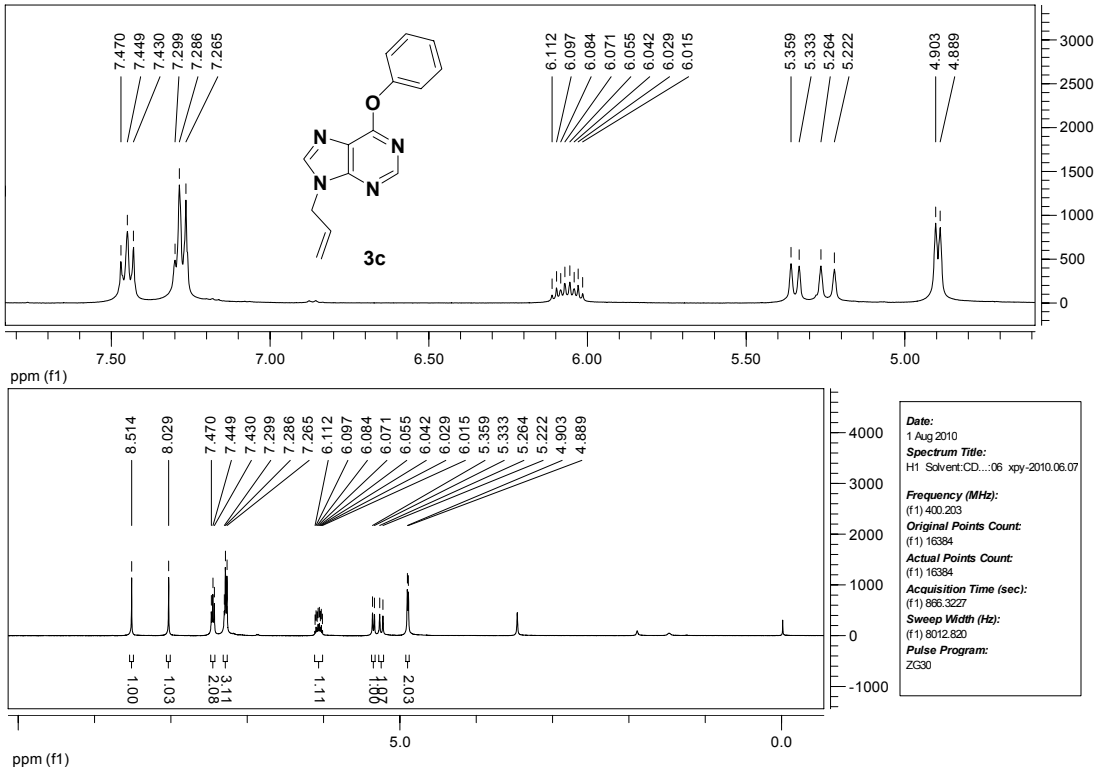
Light yellow powder, mp 144-145 ^{o}C. ^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.03 (s, 1H), 7.76 (d, J=7.6 Hz, 3H), 7.38 (t, J=8 Hz, 2H), 7.13 (t, J=7.2 Hz, 1H), 6.06-5.97 (m, 1H), 5.33 (d, J=10 Hz, 1H), 5.23 (d, J=17.2 Hz, 1H), 4.79 (d, J=6 Hz, 2H). ^{13}C NMR ($CDCl_3$, 100 MHz) δ 154.2, 152.3, 150.5, 140.7, 138.0, 131.4, 129.1, 124.0, 120.2, 119.5, 119.0, 45.9. HRMS: calcd for $C_{14}H_{13}ClN_5$ [M + H^+] 286.0854, found 286.0850.

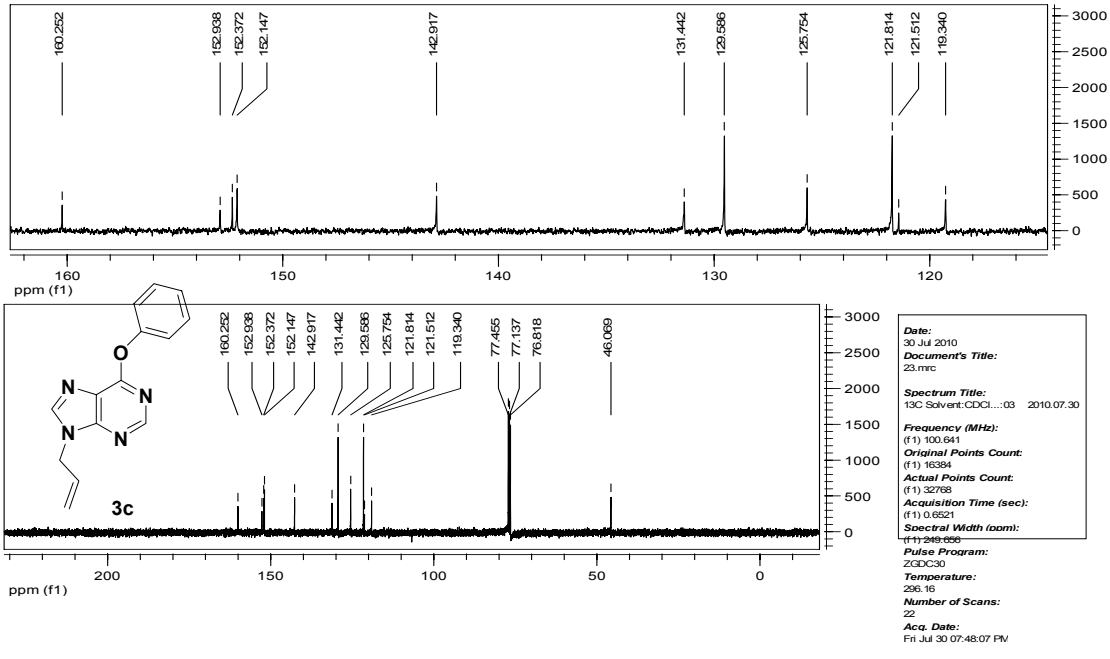

8.461
8.402
7.481
7.462
7.444
7.305
7.286
7.267
3a
ppm (f1)
13.556
8.402
8.461
7.481
7.462
7.444
7.305
7.286
7.267
3.290
2.486
0.84
2.00
2.19
3.06
Date:
1 Aug 2010
Spectrum Title:
1H solvent:DMSO...:3(1) 2007.11.23
Frequency (MHz):
(f1) 400.203
Original Points Count:
(f1) 16384
Actual Points Count:
(f1) 16384
Acquisition Time (sec):
(f1) 866.3227
Sweep Width (Hz):
(f1) 8012.820
Pulse Program:
ZG30
ppm (f1)

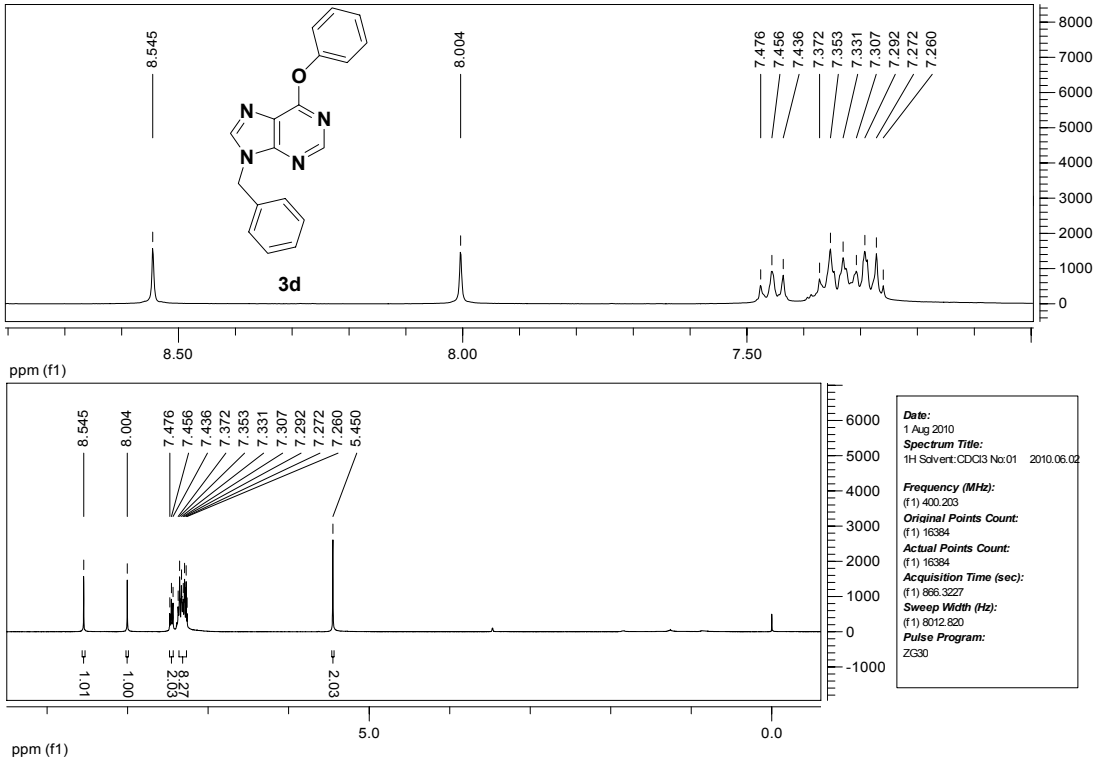

152.718
151.567
130.128
125.943
122.358
3a
ppm (f1)
152.718
151.567
130.128
125.943
122.358
Date:
1 Aug 2010
Spectrum Title:
C13 solvent:DMSO No:5 2008.4.28
Frequency (MHz):
(f1) 100.641
Original Points Count:
(f1) 16384
Actual Points Count:
(f1) 32768
Acquisition Time (sec):
(f1) 276.2792
Sweep Width (Hz):
(f1) 25125.629
Pulse Program:
ZGDC30
ppm (f1)

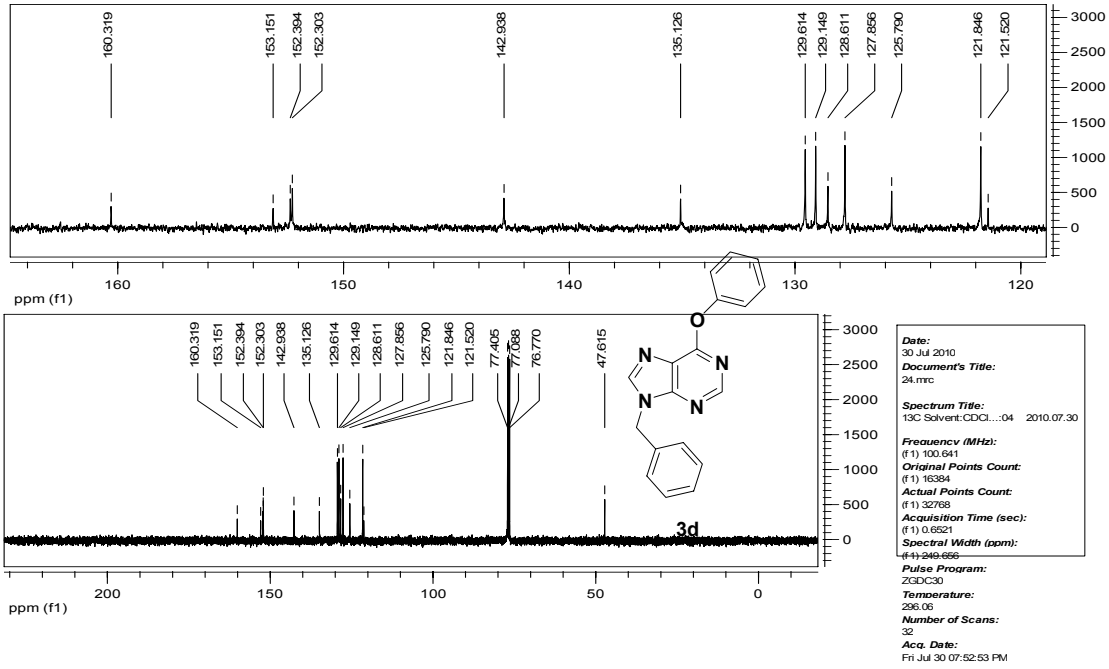

8.522
8.003
7.474
7.453
7.435
7.306
7.288
7.267
3.922
3b
0.99
1.00
2.13
3.15
3.03
ppm (f1)
Date:
1 Aug 2010
Spectrum Title:
1H Solvent:CDC...::01 2010.06.11
Frequency (MHz):
(f1) 400.203
Original Points Count:
(f1) 16384
Actual Points Count:
(f1) 16384
Acquisition Time (sec):
(f1) 866.3227
Sweep Width (Hz):
(f1) 8012.820
Pulse Program:
ZG30

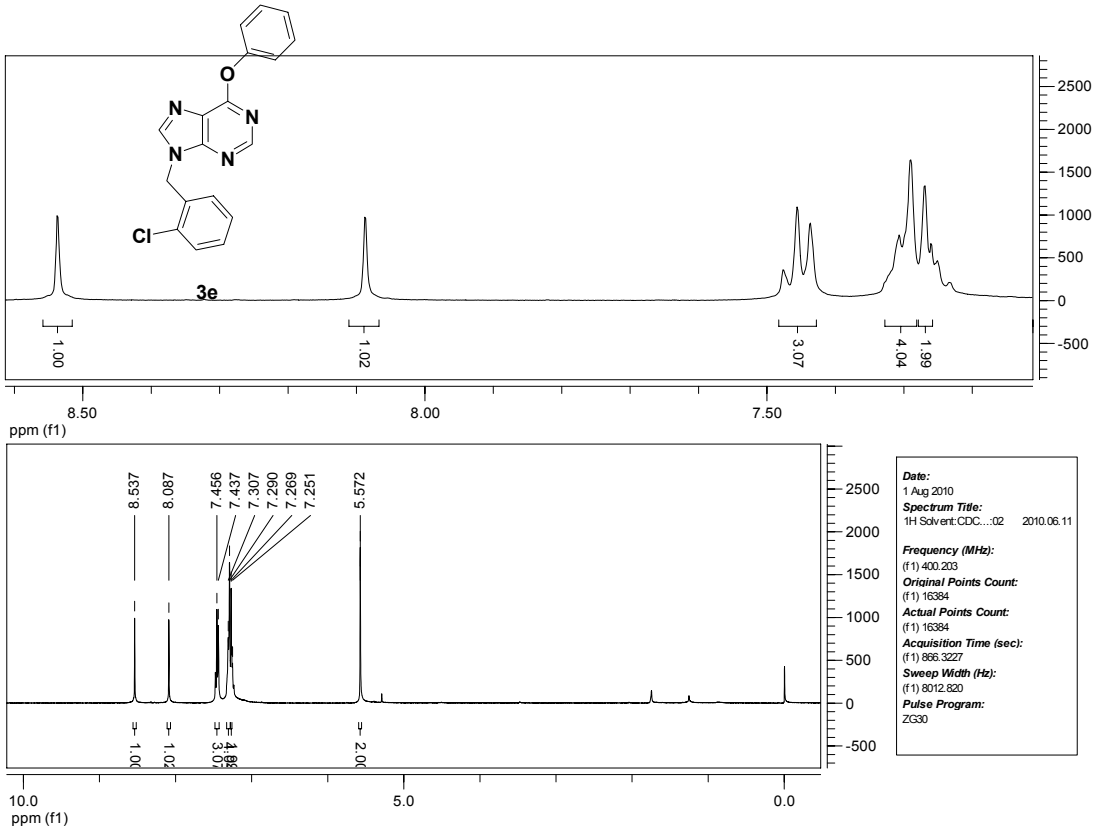

160.191
153.393
152.378
152.080
143.597
129.586
125.746
121.801
121.480
77.412
77.095
76.777
30.095
3b
ppm (f1)
Date:
1 Aug 2010
Spectrum Title:
13C Solvent...::06 ZPY2 2010.06.18
Frequency (MHz):
(f1) 100.641
Original Points Count:
(f1) 16384
Actual Points Count:
(f1) 32768
Acquisition Time (sec):
(f1) 276.2792
Sweep Width (Hz):
(f1) 25125.629
Pulse Program:
ZGDC30

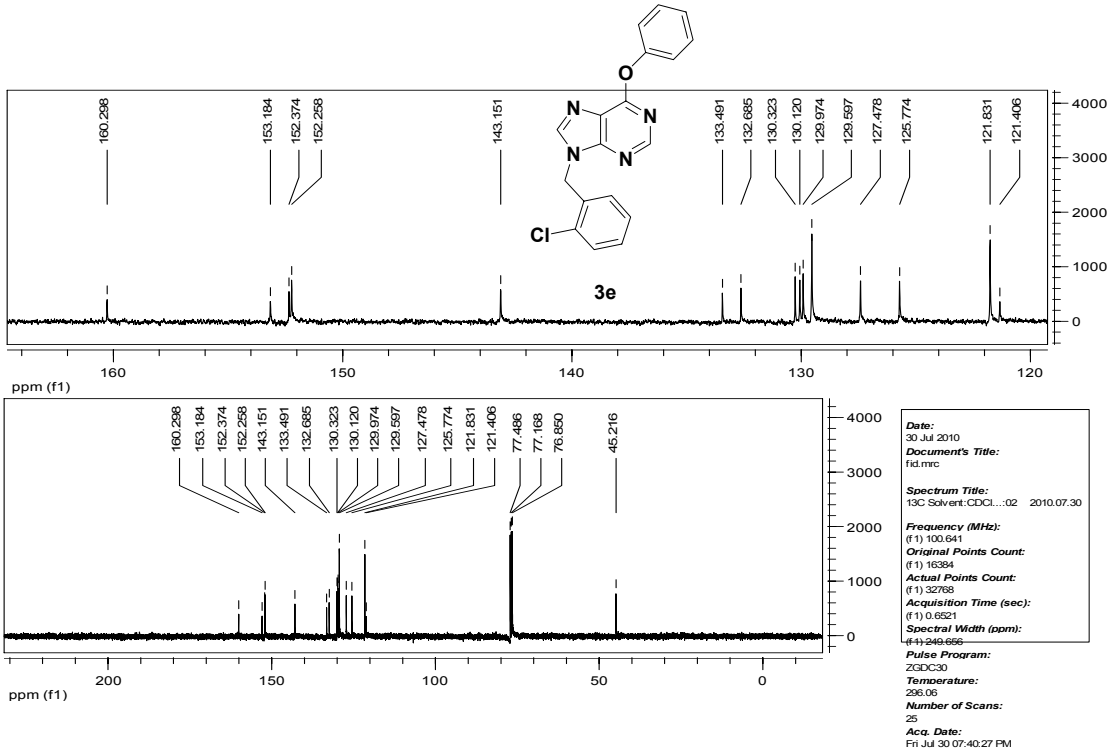


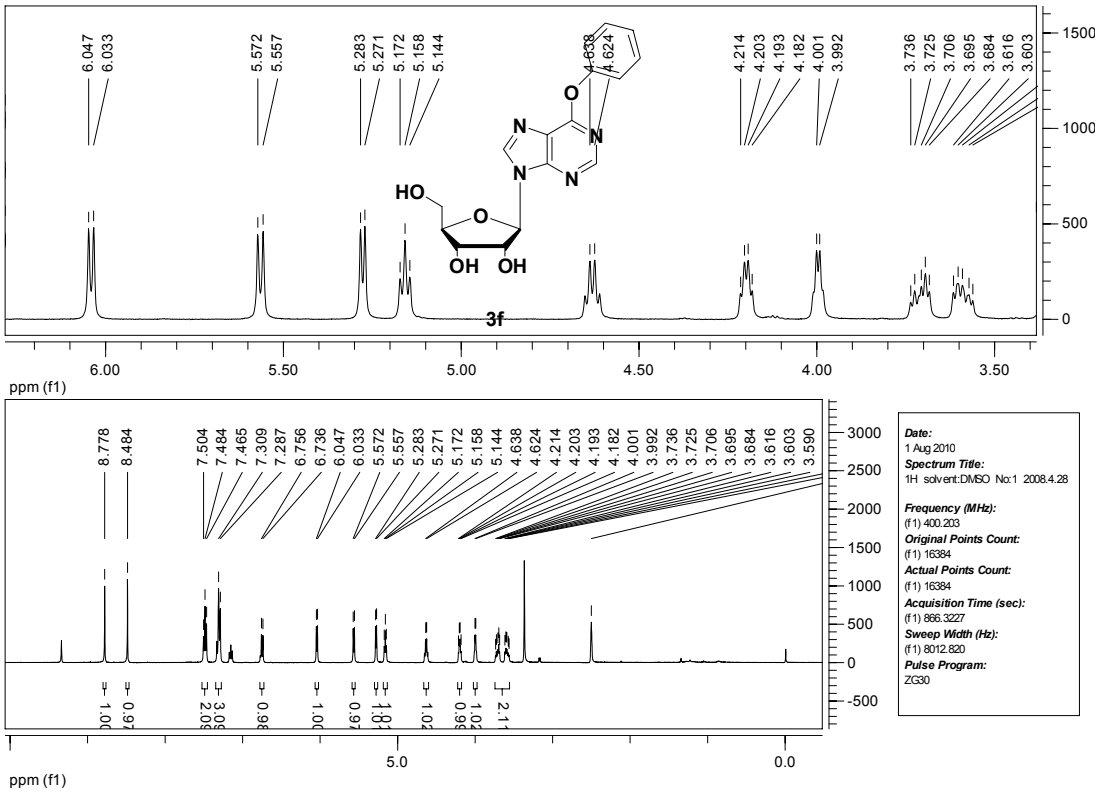


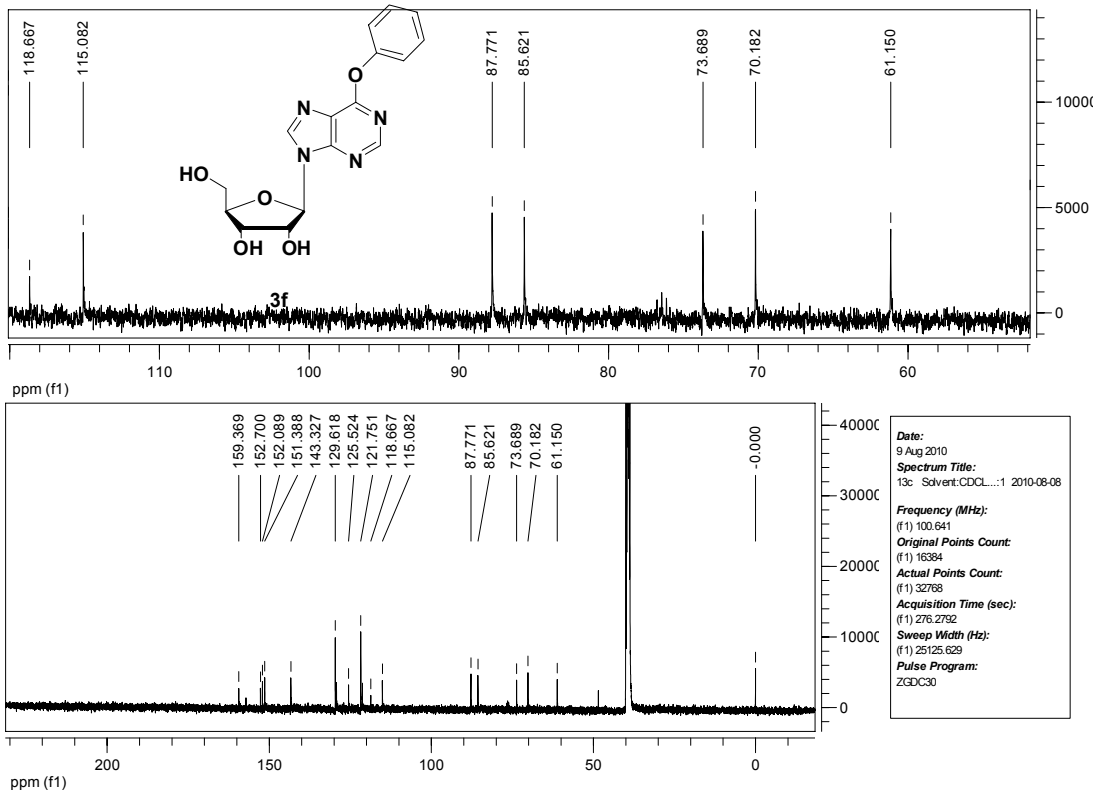

8.545
8.004
7.476
7.456
7.436
7.372
7.353
7.331
7.307
7.292
7.272
7.260
5.450
3d
ppm (f1)
1.01
1.00
2.27
2.03
Date:
1 Aug 2010
Spectrum Title:
1H Solvent:CDCl3 No:01 2010.06.02
Frequency (MHz):
(f1) 400.203
Original Points Count:
(f1) 16384
Actual Points Count:
(f1) 16384
Acquisition Time (sec):
(f1) 866.3227
Sweep Width (Hz):
(f1) 8012.820
Pulse Program:
ZG30

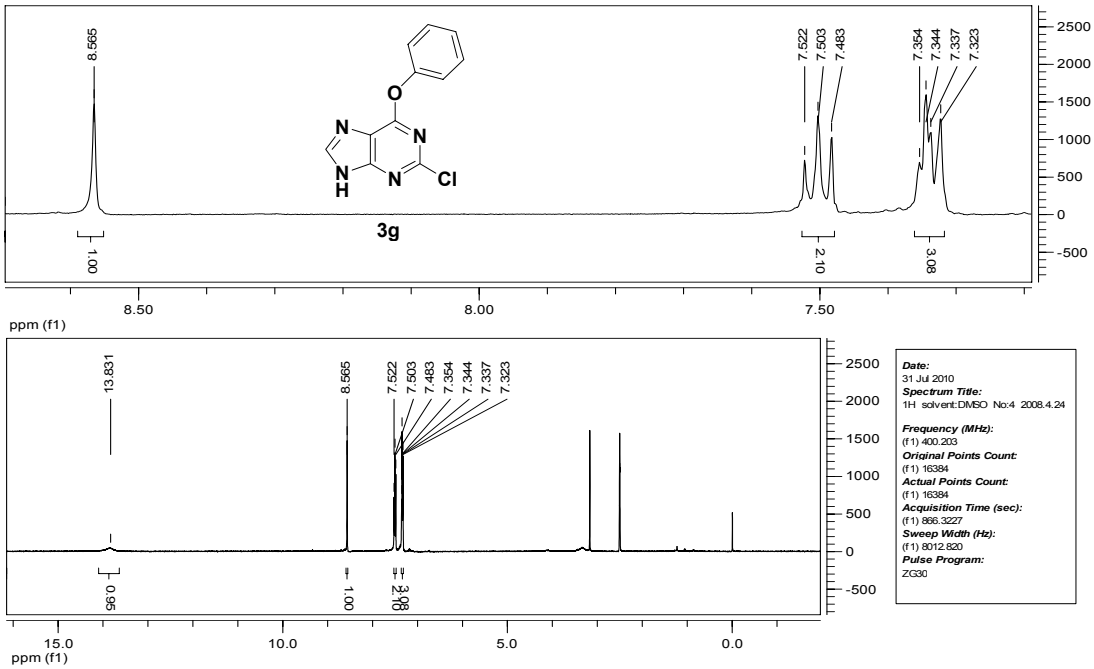

160.319
153.151
152.394
152.303
142.938
135.126
129.614
129.149
128.611
127.856
125.790
121.846
121.520
77.405
77.088
76.770
47.615
3d
ppm (f1)
Date:
30 Jul 2010
Document's Title:
24.mrc
Spectrum Title:
13C Solvent:CDCl...:04 2010.07.30
Freauencv (MHz):
(f1) 100.641
Original Points Count:
(f1) 16384
Actual Points Count:
(f1) 32768
Acquisition Time (sec):
(f1) 0.8521
Spectral Width (ppm):
(f1) 249.656
Pulse Program:
ZGDC30
Temperature:
296.06
Number of Scans:
32
Acq. Date:
Fri Jul 30 07:52:53 PM

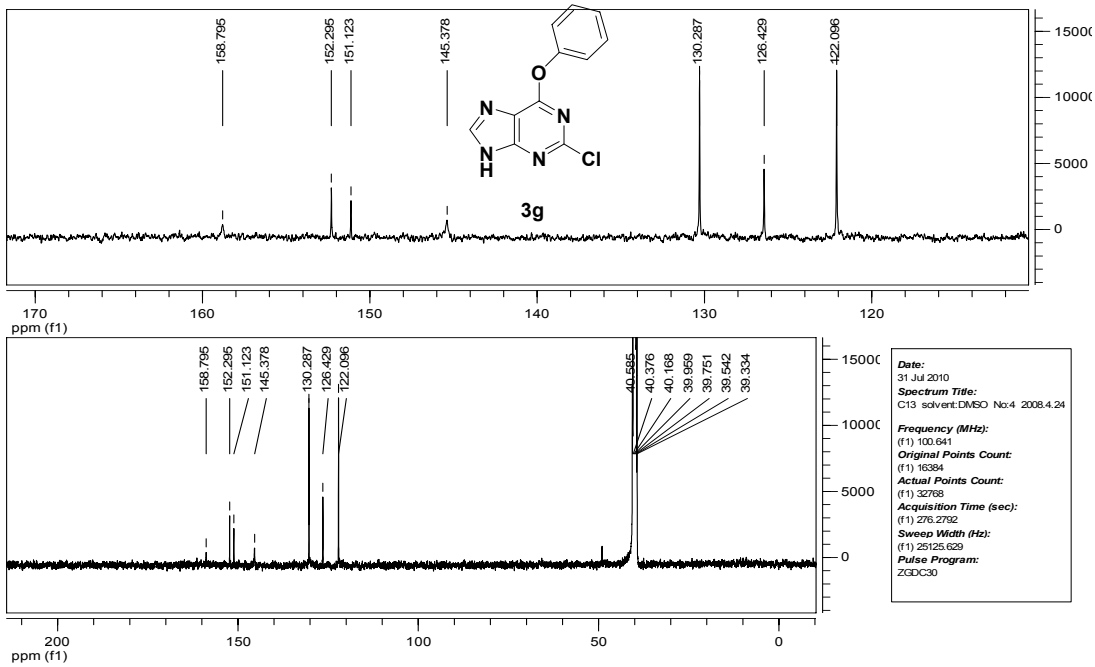

3e
1.00
1.02
3.07
4.04
1.99
8.50
8.00
7.50
ppm (f1)
8.537
8.087
7.456
7.437
7.307
7.290
7.269
7.251
5.572
10.0
5.0
0.0
ppm (f1)
Date:
1 Aug 2010
Spectrum Title:
1H Solvent:CDC...:02 2010.06.11
Frequency (MHz):
(f1) 400.203
Original Points Count:
(f1) 16384
Actual Points Count:
(f1) 16384
Acquisition Time (sec):
(f1) 866.3227
Sweep Width (Hz):
(f1) 8012.820
Pulse Program:
ZG30

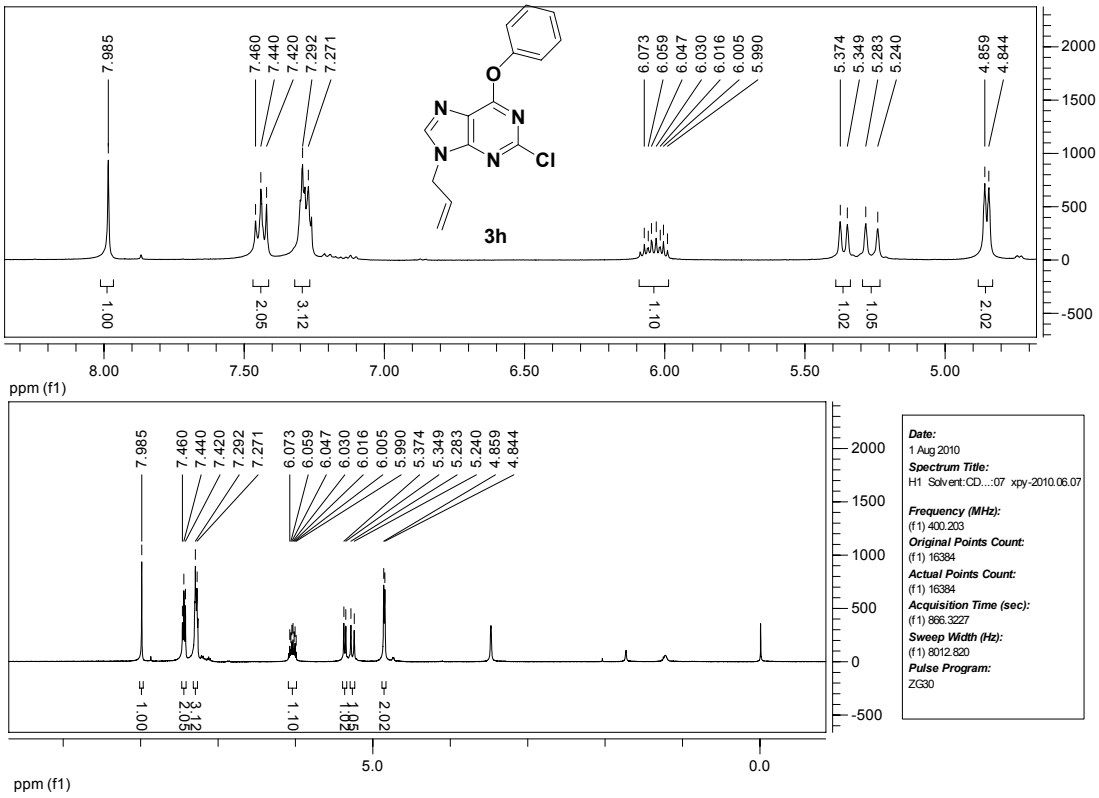

3e
160.298
153.184
152.374
152.258
143.151
133.491
132.685
130.323
130.120
129.974
129.597
127.478
125.774
121.831
121.406
160
150
140
130
120
ppm (f1)
77.486
77.168
76.850
45.216
200
150
100
50
0
ppm (f1)
Date:
30 Jul 2010
Document's Title:
fid.mrc
Spectrum Title:
13C Solvent:CDCl...:02 2010.07.30
Frequency (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:
296.06
Number of Scans:
25
Acq. Date:
Fri Jul 30 07:40:27 PM

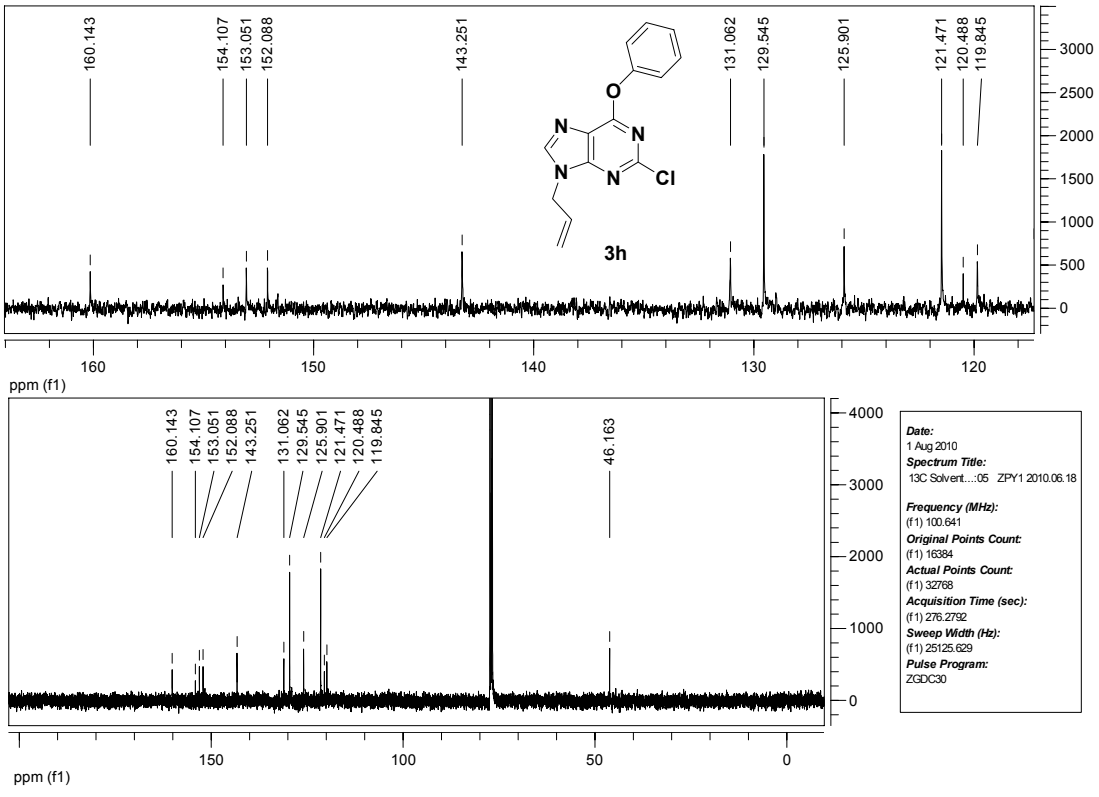

6.047
6.033
5.572
5.557
5.283
5.271
5.172
5.158
5.144
4.638
4.624
4.214
4.203
4.193
4.182
4.001
3.992
3.736
3.725
3.706
3.695
3.684
3.616
3.603
HO
OH OH
3f
ppm (f1)
8.778
8.484
7.504
7.484
7.465
7.309
7.287
6.756
6.736
6.047
6.033
5.572
5.557
5.283
5.271
5.172
5.158
5.144
4.638
4.624
4.214
4.203
4.193
4.182
4.001
3.992
3.736
3.725
3.706
3.695
3.684
3.616
3.603
3.590
Date:
1 Aug 2010
Spectrum Title:
1H solvent:DMSO No:1 2008.4.28
Frequency (MHz):
(f1) 400.203
Original Points Count:
(f1) 16384
Actual Points Count:
(f1) 16384
Acquisition Time (sec):
(f1) 866.3227
Sweep Width (Hz):
(f1) 8012.820
Pulse Program:
ZG30
ppm (f1)

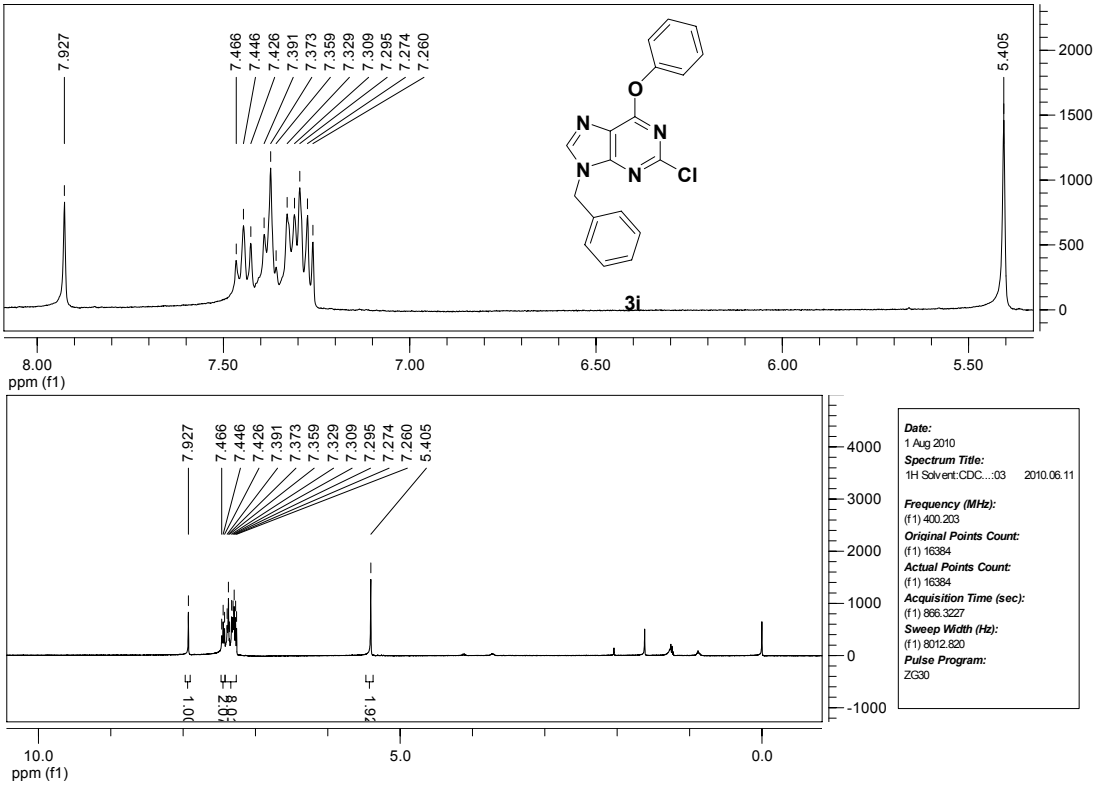

118.667
115.082
87.771
85.621
73.689
70.182
61.150
HO
OH OH
3f
ppm (f1)
159.369
152.700
152.089
151.388
143.327
129.618
125.524
121.751
118.667
115.082
87.771
85.621
73.689
70.182
61.150
-0.000
Date:
9 Aug 2010
Spectrum Title:
13c Solvent:CDCL...:1 2010-08-08
Frequency (MHz):
(f1) 100.641
Original Points Count:
(f1) 16384
Actual Points Count:
(f1) 32768
Acquisition Time (sec):
(f1) 276.2792
Sweep Width (Hz):
(f1) 25125.629
Pulse Program:
ZGDC30
ppm (f1)

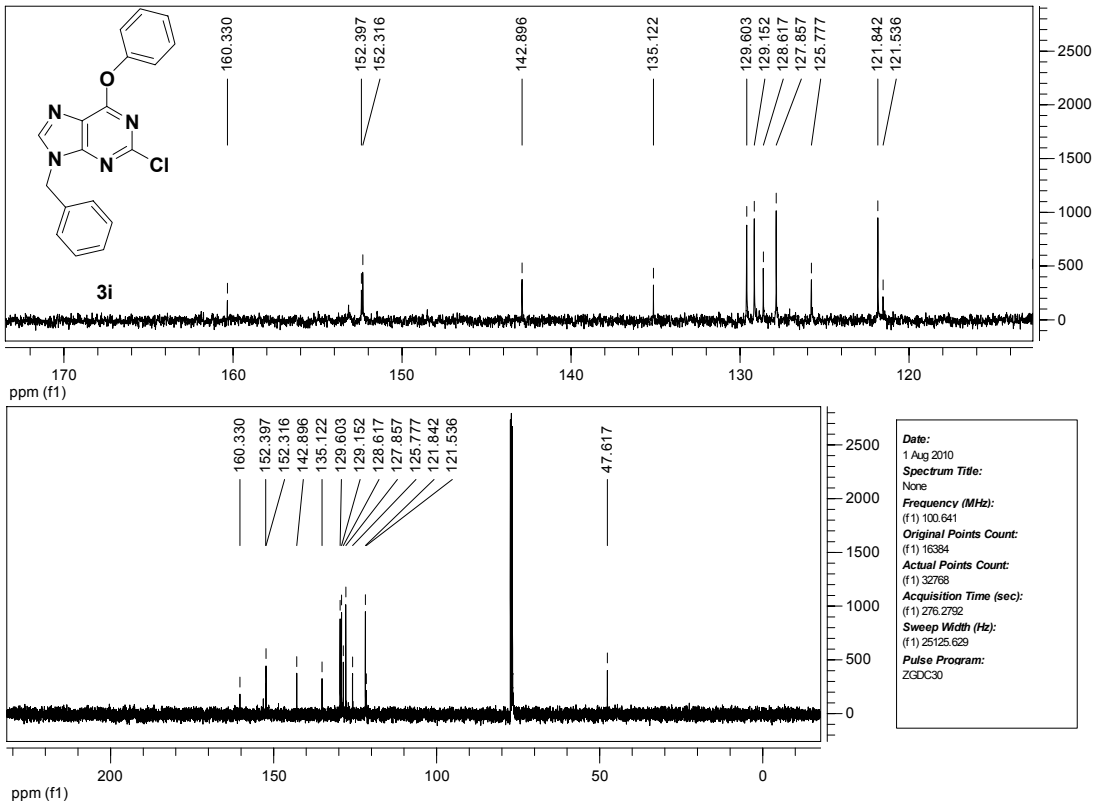

8.565
7.522
7.503
7.483
7.354
7.344
7.337
7.323
3g
1.00
2.10
3.08
ppm (f1)
13.831
0.96
2.08
Date:
31 Jul 2010
Spectrum Title:
1H solvent:DMSO No:4 2008.4.24
Frequency (MHz):
(f1) 400.203
Original Points Count:
(f1) 16384
Actual Points Count:
(f1) 16384
Acquisition Time (sec):
(f1) 866.3227
Sweep Width (Hz):
(f1) 8012.820
Pulse Program:
ZG30

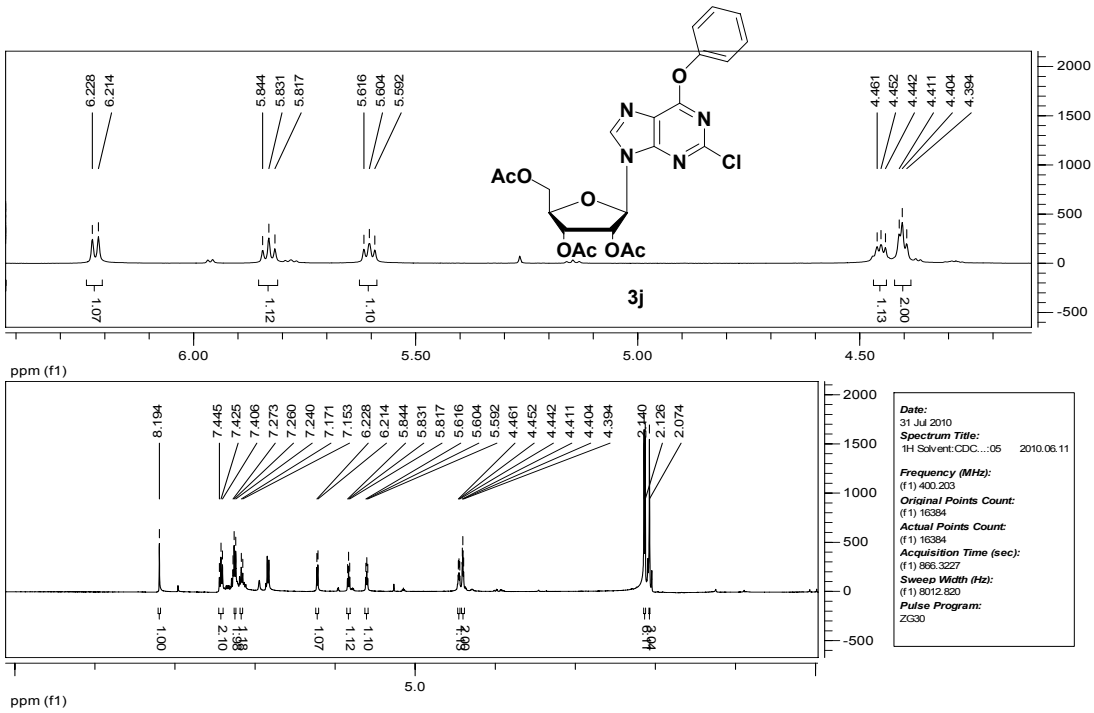

158.795
152.295
151.123
145.378
130.287
126.429
122.096
3g
ppm (f1)
40.585
40.376
40.168
39.959
39.751
39.542
39.334
Date:
31 Jul 2010
Spectrum Title:
C13 solvent:DMSO No:4 2008.4.24
Frequency (MHz):
(f1) 100.641
Original Points Count:
(f1) 16384
Actual Points Count:
(f1) 32768
Acquisition Time (sec):
(f1) 276.2792
Sweep Width (Hz):
(f1) 25125.629
Pulse Program:
ZGDC30

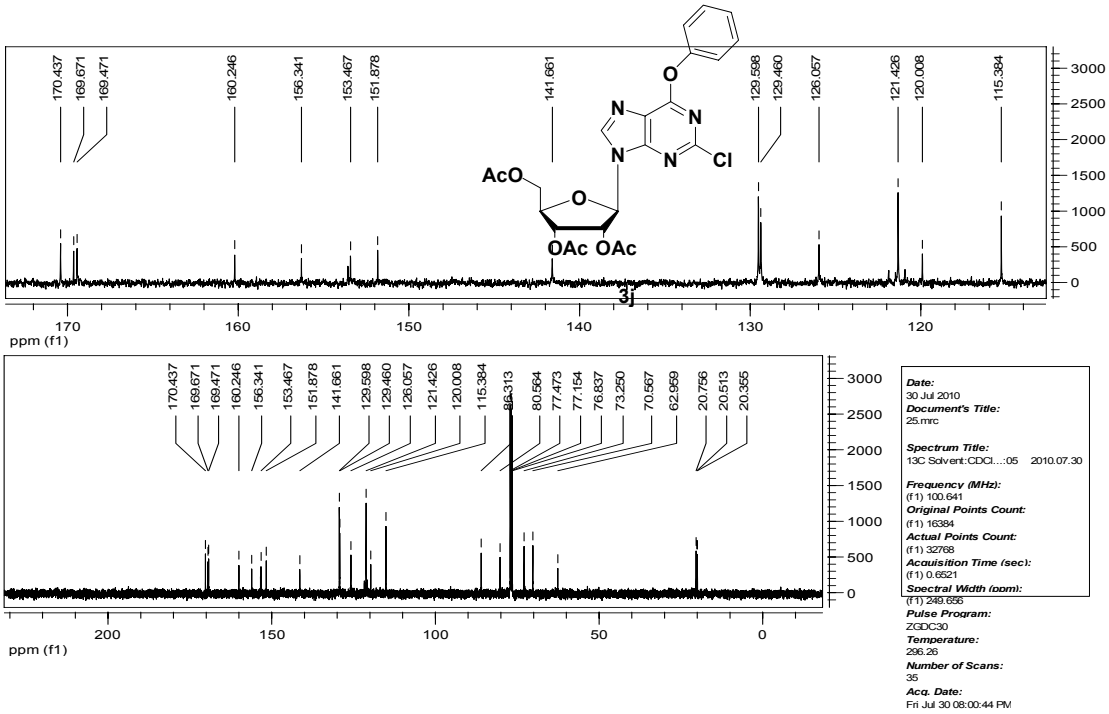

7.985
7.460
7.440
7.420
7.292
7.271
6.073
6.059
6.047
6.030
6.016
6.005
5.990
5.374
5.349
5.283
5.240
4.859
4.844
3h
1.00
2.05
3.12
1.10
1.02
1.05
2.02
ppm (f1)
Date:
1 Aug 2010
Spectrum Title:
H1 Solvent:CD...:07 xpy-2010.06.07
Frequency (MHz):
(f1) 400.203
Original Points Count:
(f1) 16384
Actual Points Count:
(f1) 16384
Acquisition Time (sec):
(f1) 866.3227
Sweep Width (Hz):
(f1) 8012.820
Pulse Program:
ZG30

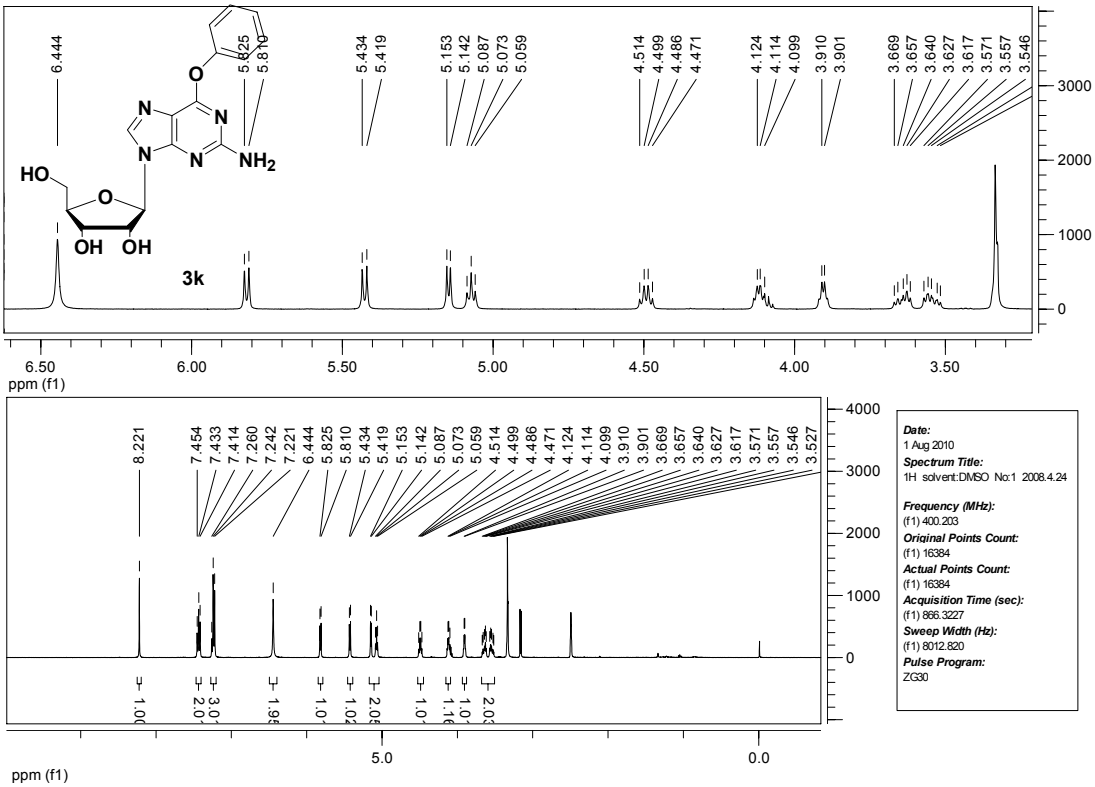

160.143
154.107
153.051
152.088
143.251
131.062
129.545
125.901
121.471
120.488
119.845
3h
ppm (f1)
46.163
Date:
1 Aug 2010
Spectrum Title:
13C Solvent...:05 ZPY1 2010.06.18
Frequency (MHz):
(f1) 100.641
Original Points Count:
(f1) 16384
Actual Points Count:
(f1) 32768
Acquisition Time (sec):
(f1) 276.2792
Sweep Width (Hz):
(f1) 25125.629
Pulse Program:
ZGDC30

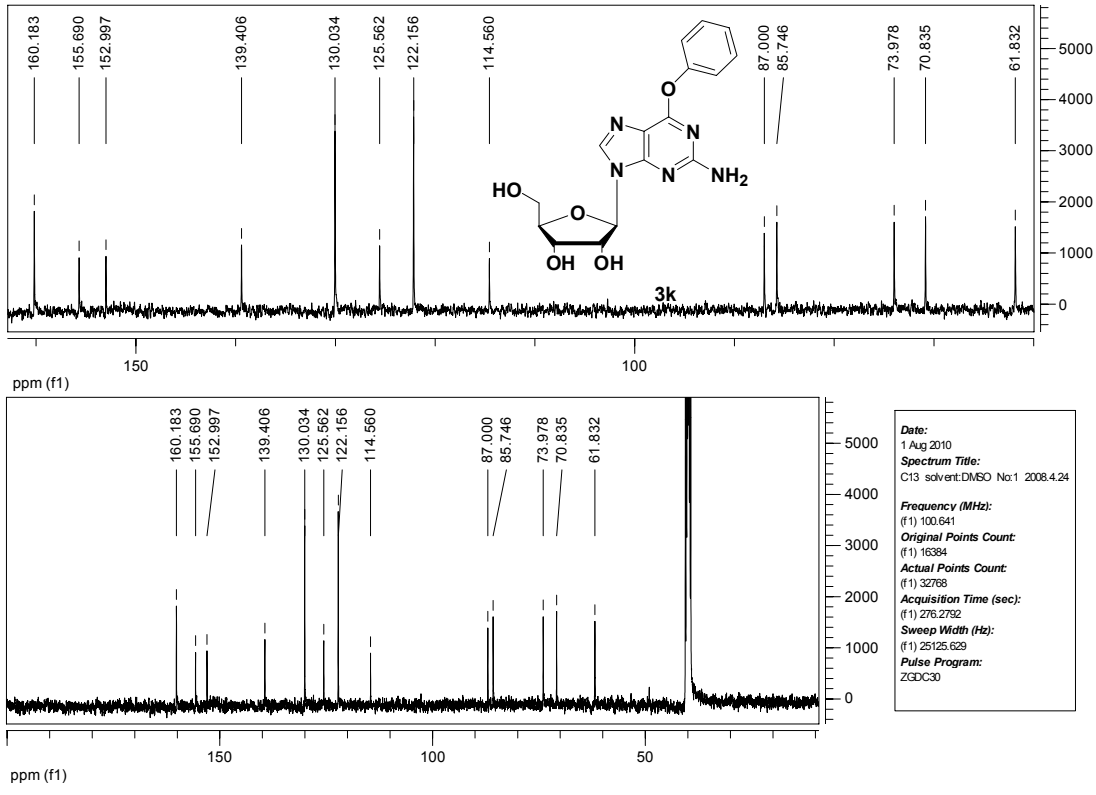

7.927
7.466
7.446
7.426
7.391
7.373
7.359
7.329
7.309
7.295
7.274
7.260
5.405
3i
ppm (f1)
Date:
1 Aug 2010
Spectrum Title:
1H Solvent:CDC...:03 2010.06.11
Frequency (MHz):
(f1) 400.203
Original Points Count:
(f1) 16384
Actual Points Count:
(f1) 16384
Acquisition Time (sec):
(f1) 866.3227
Sweep Width (Hz):
(f1) 8012.820
Pulse Program:
ZG30

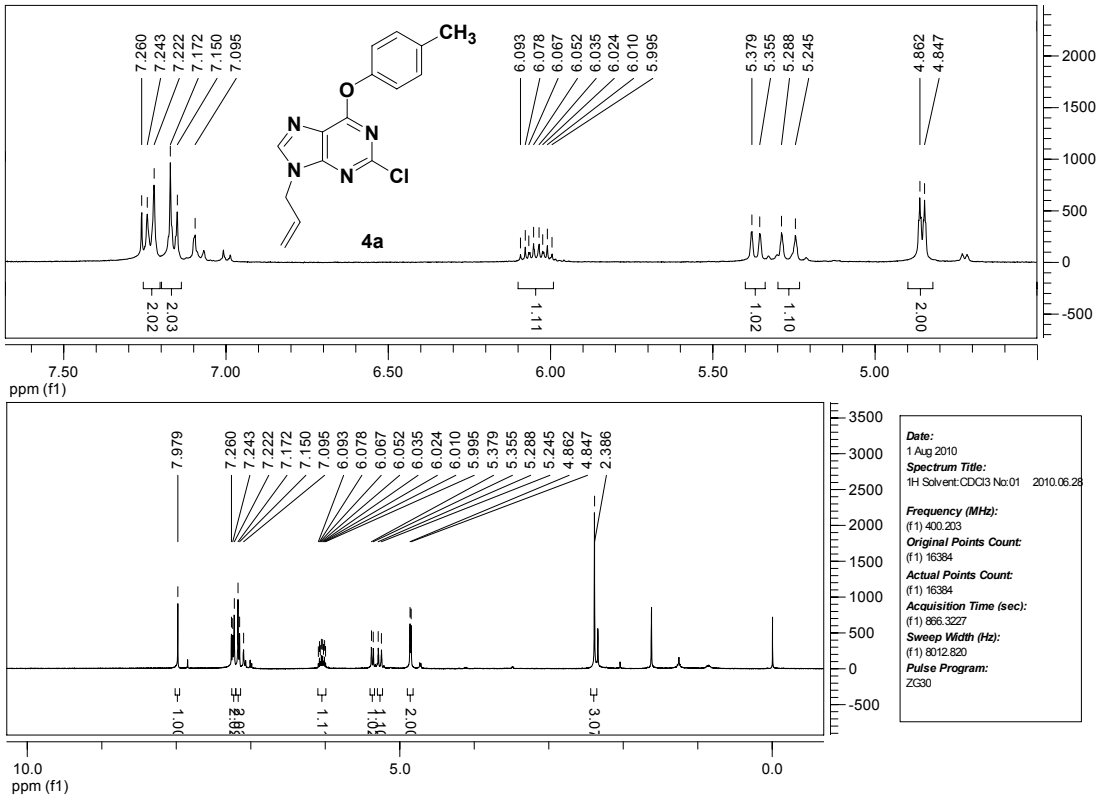

160.330
152.397
152.316
142.896
135.122
129.603
129.152
128.617
127.857
125.777
121.842
121.536
47.617
3i
ppm (f1)
Date:
1 Aug 2010
Spectrum Title:
None
Frequency (MHz):
(f1) 100.641
Original Points Count:
(f1) 16384
Actual Points Count:
(f1) 32768
Acquisition Time (sec):
(f1) 276.2792
Sweep Width (Hz):
(f1) 25125.629
Pulse Program:
ZGDC30

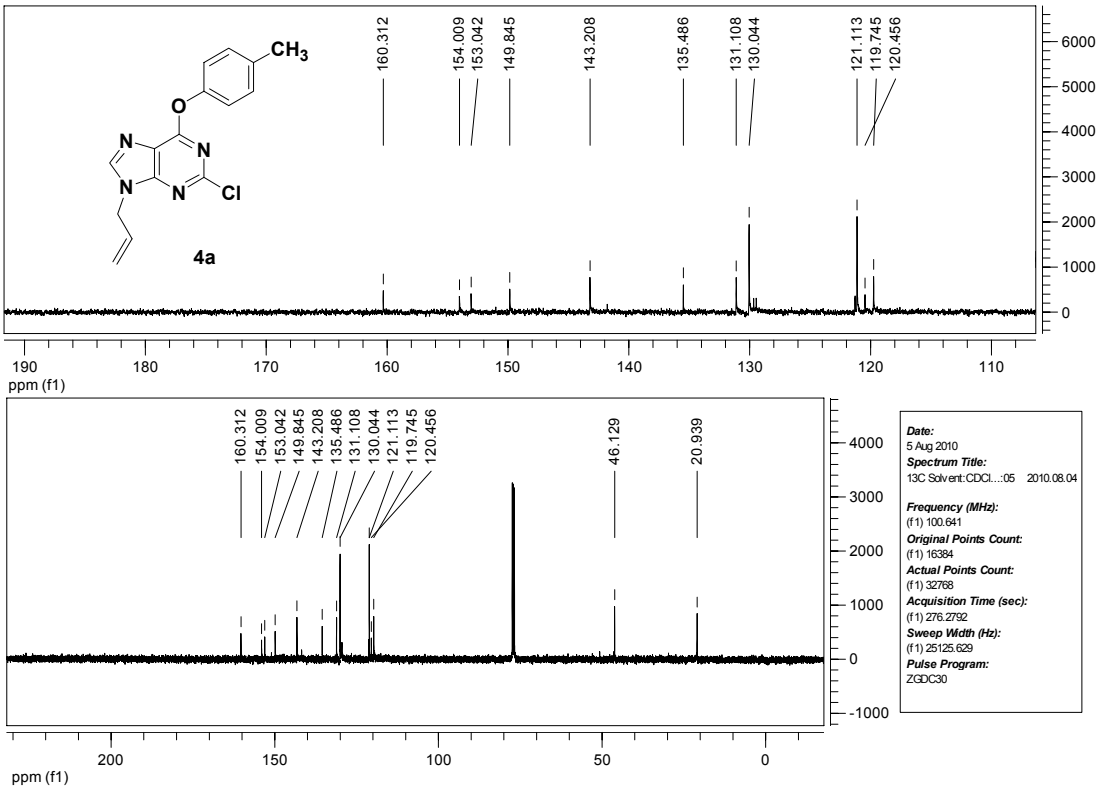

6.228
6.214
5.844
5.831
5.817
5.616
5.604
5.592
4.461
4.452
4.442
4.411
4.404
4.394
AcO
OAc OAc
Cl
3j
1.07
1.12
1.10
1.13
2.00
ppm (f1)
8.194
7.445
7.425
7.406
7.273
7.260
7.240
7.171
7.153
2.140
2.126
2.074
Date:
31 Jul 2010
Spectrum Title:
1H Solvent:CDC...::05 2010.06.11
Frequency (MHz):
(f1) 400.203
Original Points Count:
(f1) 16384
Actual Points Count:
(f1) 16384
Acquisition Time (sec):
(f1) 866.3227
Sweep Width (Hz):
(f1) 8012.820
Pulse Program:
ZG30

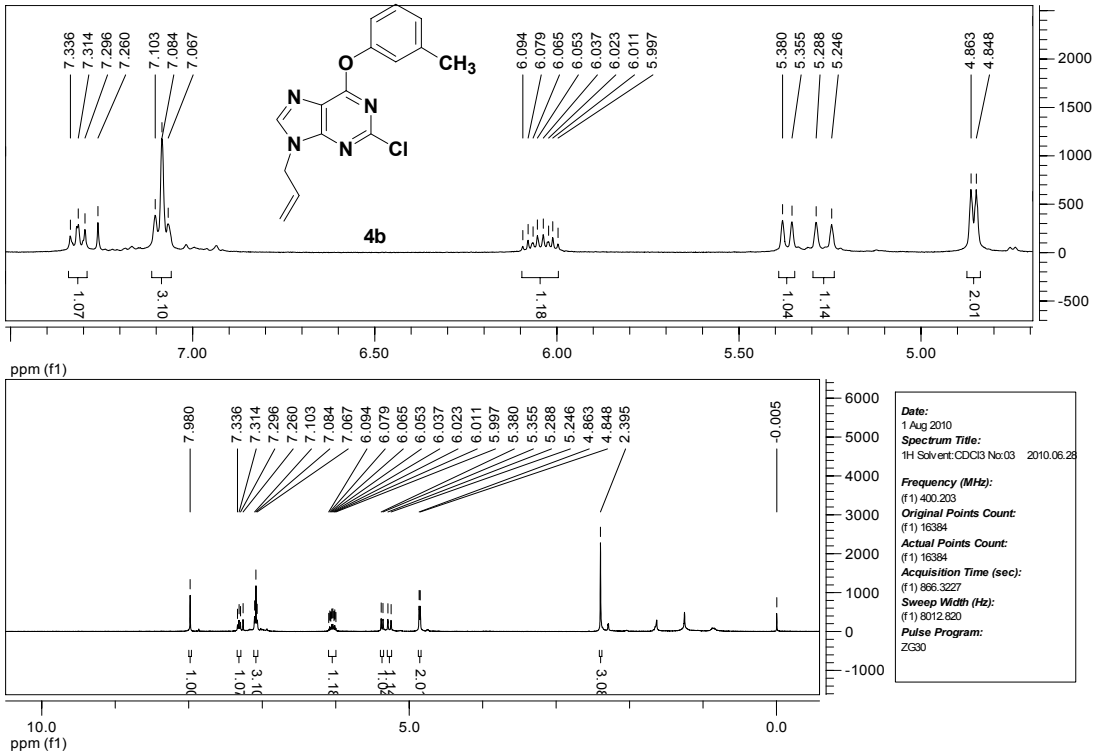

170.437
169.671
169.471
160.246
156.341
153.467
151.878
141.661
129.598
129.460
126.057
121.426
120.008
115.384
AcO
OAc OAc
Cl
3j
ppm (f1)
86.313
80.564
77.473
77.154
76.837
73.250
70.567
62.959
20.756
20.513
20.355
Date:
30 Jul 2010
Document's Title:
25.mrc
Spectrum Title:
13C Solvent:CDCl...::05 2010.07.30
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:
296.26
Number of Scans:
35
Acq. Date:
Fri Jul 30 08:00:44 PM

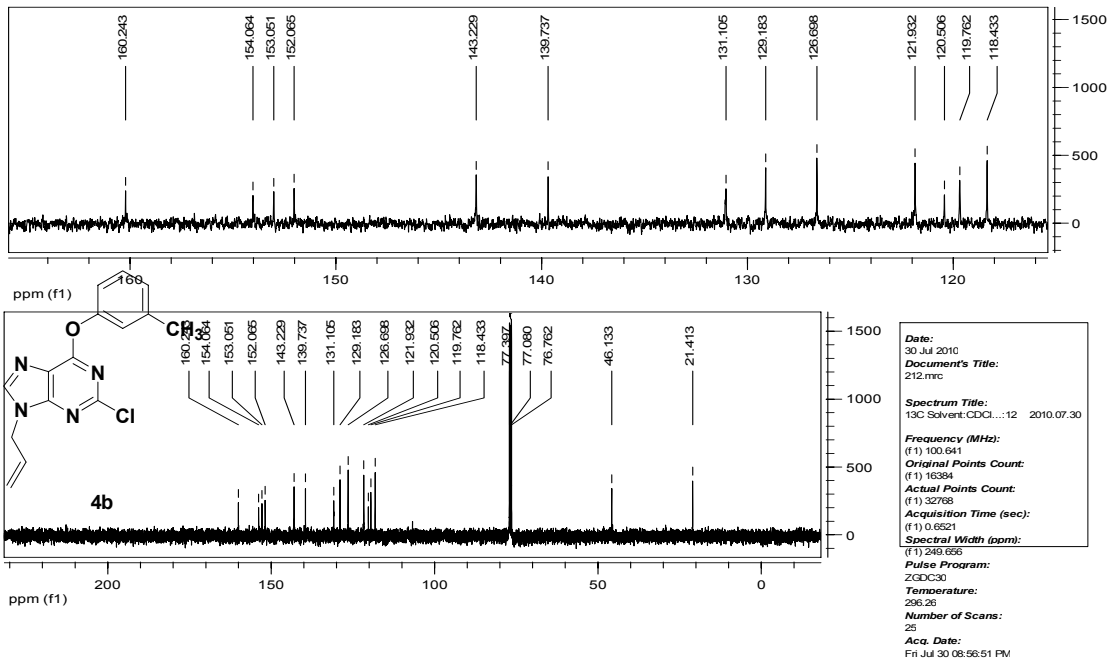

6.444
5.825
5.810
5.434
5.419
5.153
5.142
5.087
5.073
5.059
4.514
4.499
4.486
4.471
4.124
4.114
4.099
3.910
3.901
3.669
3.657
3.640
3.627
3.617
3.571
3.557
3.546
HO
O
N
N
N
N
NH2
OH OH
3k
6.50
6.00
5.50
5.00
4.50
4.00
3.50
ppm (f1)
8.221
7.454
7.433
7.414
7.260
7.242
7.221
6.444
5.825
5.810
5.434
5.419
5.153
5.142
5.087
5.073
5.059
4.514
4.499
4.486
4.471
4.124
4.114
4.099
3.910
3.901
3.669
3.657
3.640
3.627
3.617
3.571
3.557
3.546
3.527
1.00
2.07
3.07
1.93
1.07
2.09
1.02
2.09
1.07
1.16
1.07
2.03
5.0
0.0
ppm (f1)
Date:
1 Aug 2010
Spectrum Title:
1H solvent:DMSO No:1 2008.4.24
Frequency (MHz):
(f1) 400.203
Original Points Count:
(f1) 16384
Actual Points Count:
(f1) 16384
Acquisition Time (sec):
(f1) 866.3227
Sweep Width (Hz):
(f1) 8012.820
Pulse Program:
ZG30

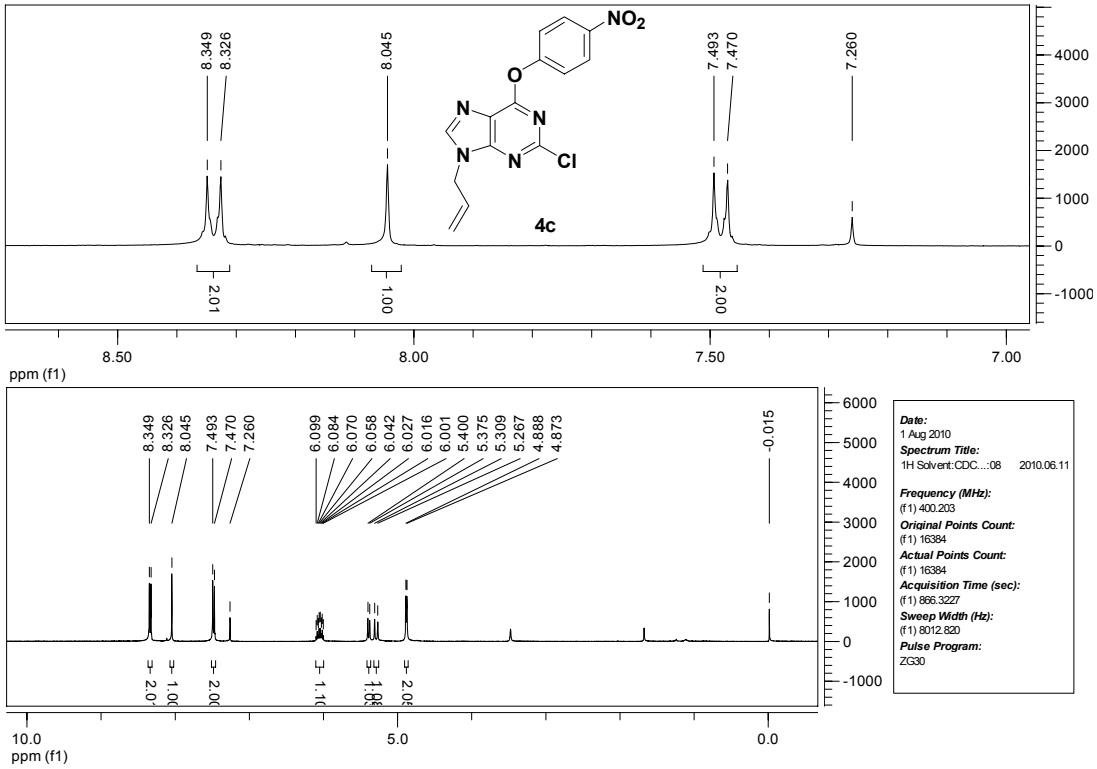

160.183
155.690
152.997
139.406
130.034
125.562
122.156
114.560
87.000
85.746
73.978
70.835
61.832
HO
O
N
N
N
N
NH2
OH OH
3k
150
100
ppm (f1)
160.183
155.690
152.997
139.406
130.034
125.562
122.156
114.560
87.000
85.746
73.978
70.835
61.832
150
100
50
ppm (f1)
Date:
1 Aug 2010
Spectrum Title:
C13 solvent:DMSO No:1 2008.4.24
Frequency (MHz):
(f1) 100.641
Original Points Count:
(f1) 16384
Actual Points Count:
(f1) 32768
Acquisition Time (sec):
(f1) 276.2792
Sweep Width (Hz):
(f1) 25125.629
Pulse Program:
ZGDC30

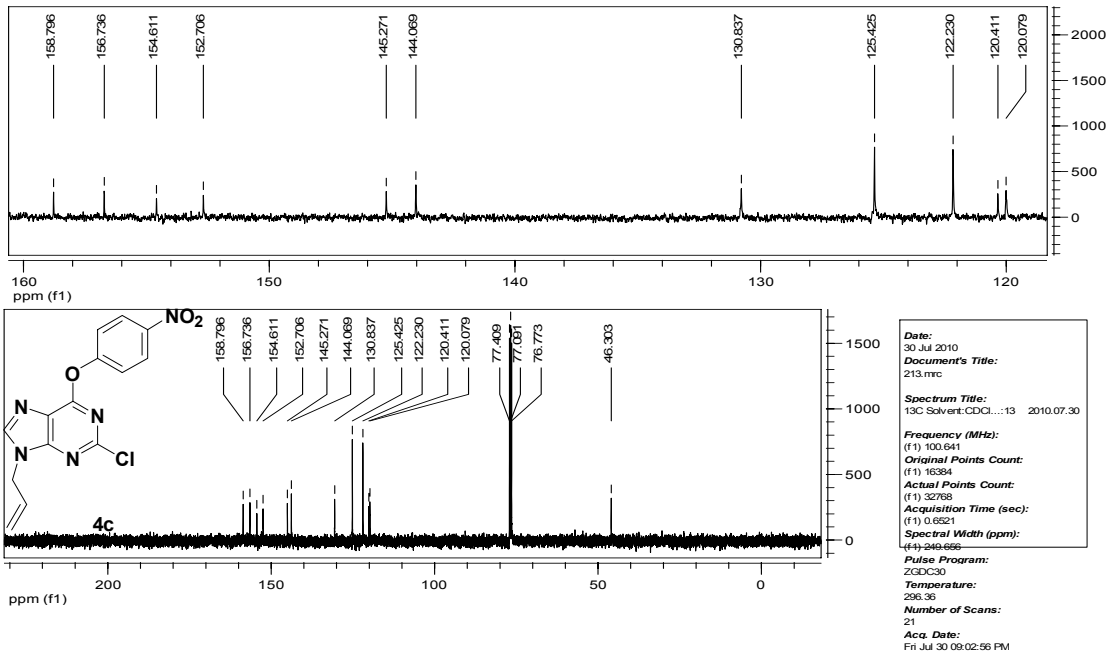

7.260
7.243
7.222
7.172
7.150
7.095
6.093
6.078
6.067
6.052
6.035
6.024
6.010
5.995
5.379
5.355
5.288
5.245
4.862
4.847
CH3
O
N
N
N
N
Cl
4a
2.02
2.03
1.11
1.02
1.10
2.00
ppm (f1)
7.979
2.386
3.07
Date:
1 Aug 2010
Spectrum Title:
1H Solvent:CDCl3 No:01 2010.06.28
Frequency (MHz):
(f1) 400.203
Original Points Count:
(f1) 16384
Actual Points Count:
(f1) 16384
Acquisition Time (sec):
(f1) 866.3227
Sweep Width (Hz):
(f1) 8012.820
Pulse Program:
ZG30

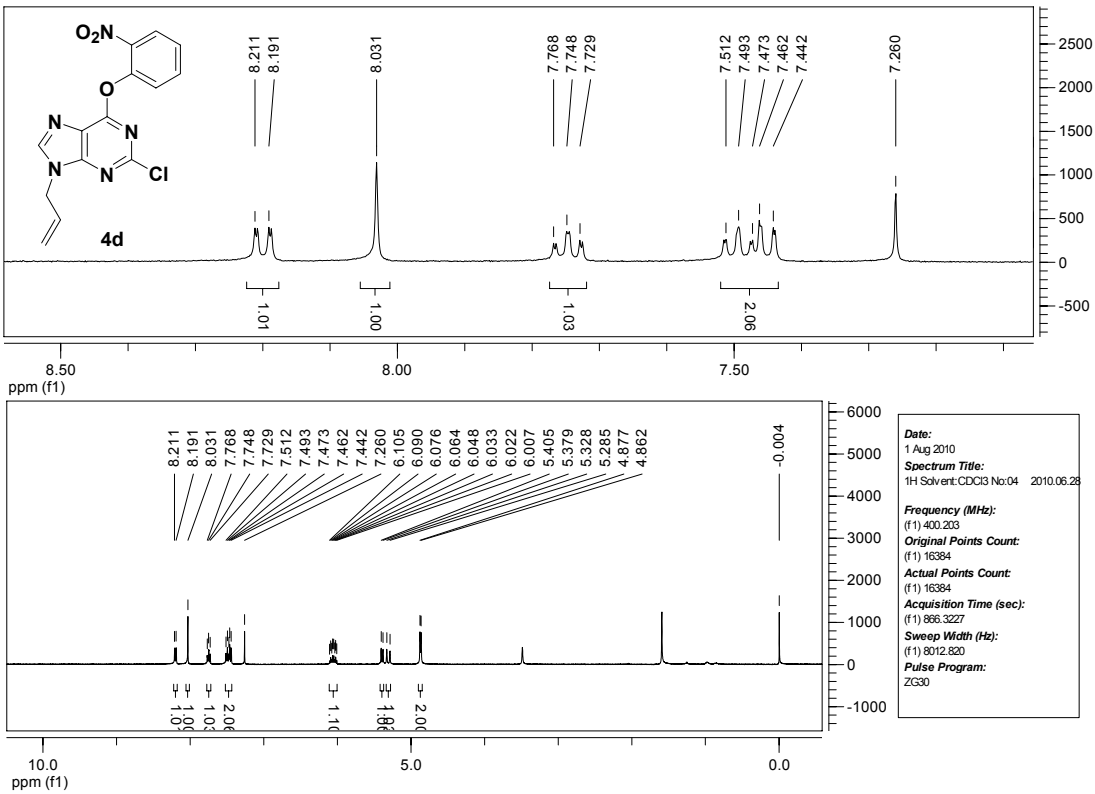

160.312
154.009
153.042
149.845
143.208
135.486
131.108
130.044
121.113
119.745
120.456
CH3
O
N
N
N
N
Cl
4a
ppm (f1)
46.129
20.939
Date:
5 Aug 2010
Spectrum Title:
13C Solvent:CDCl...:05 2010.08.04
Frequency (MHz):
(f1) 100.641
Original Points Count:
(f1) 16384
Actual Points Count:
(f1) 32768
Acquisition Time (sec):
(f1) 276.2792
Sweep Width (Hz):
(f1) 25125.629
Pulse Program:
ZGDC30

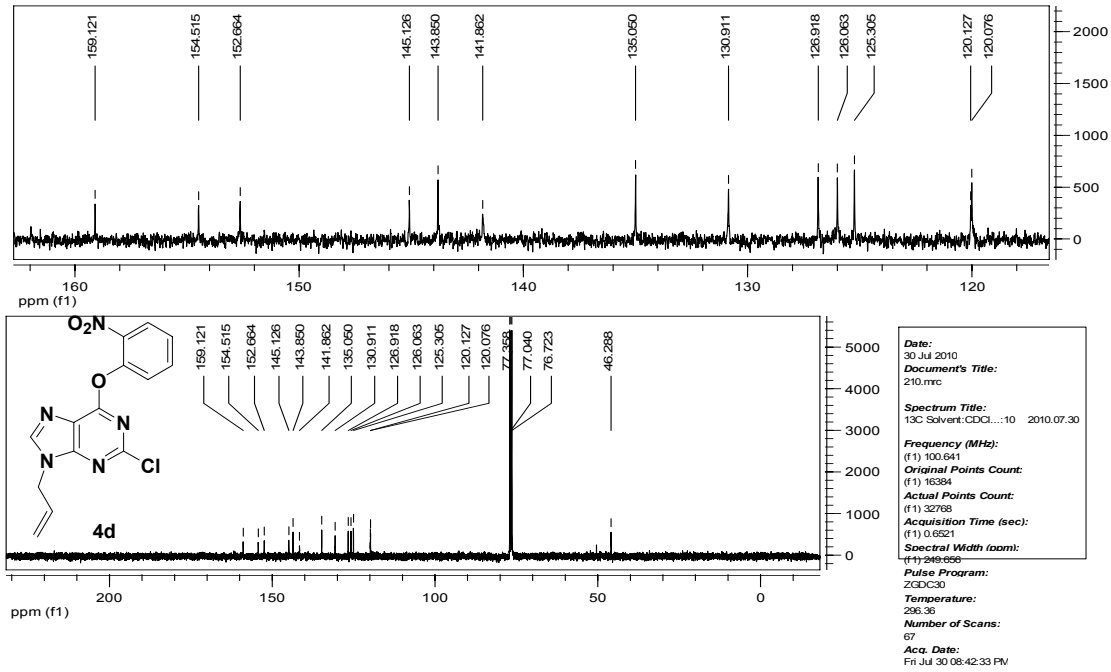

7.336
7.314
7.296
7.260
7.103
7.084
7.067
6.094
6.079
6.065
6.053
6.037
6.023
6.011
5.997
5.380
5.355
5.288
5.246
4.863
4.848
O
CH3
N
N
N
N
Cl
4b
1.07
3.10
1.18
1.04
1.14
2.01
ppm (f1)
7.980
2.395
-0.005
Date:
1 Aug 2010
Spectrum Title:
1H Solvent:CDCl3 No:03 2010.06.28
Frequency (MHz):
(f1) 400.203
Original Points Count:
(f1) 16384
Actual Points Count:
(f1) 16384
Acquisition Time (sec):
(f1) 866.3227
Sweep Width (Hz):
(f1) 8012.820
Pulse Program:
ZG30

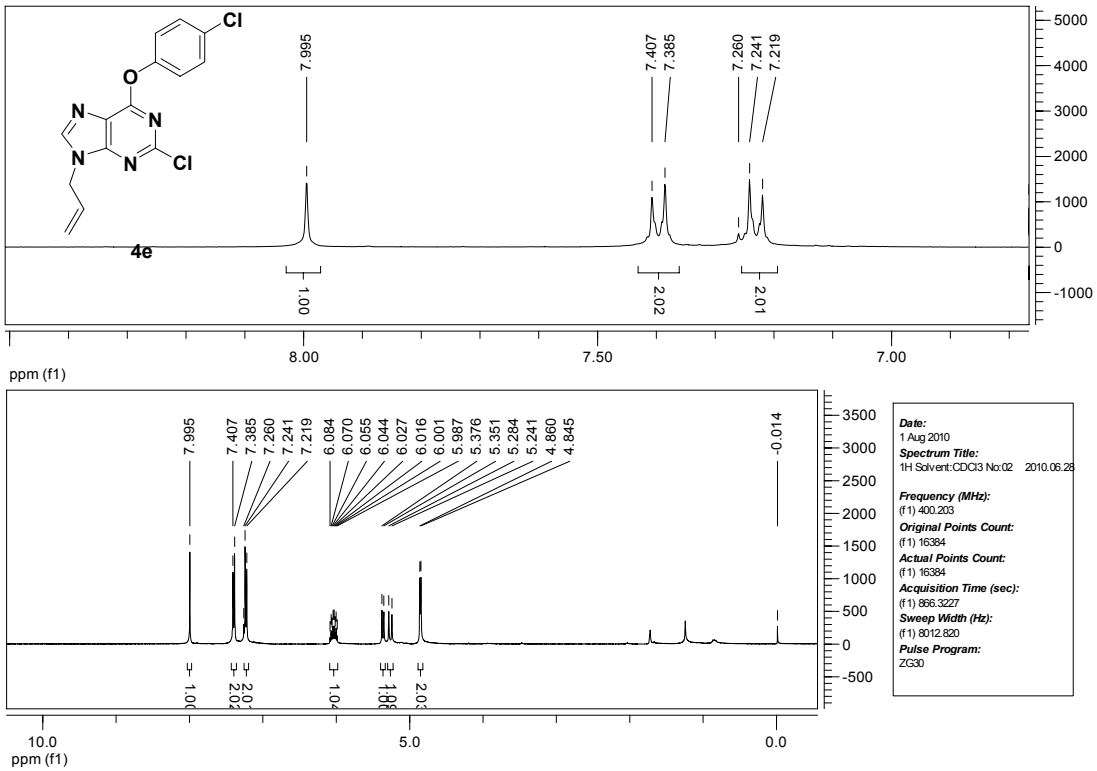

160.243
154.064
153.051
152.065
143.229
139.737
131.105
129.183
126.698
121.932
120.506
119.762
118.433
ppm (f1)
O
CH3
N
N
N
N
Cl
4b
77.397
77.080
76.762
46.133
21.413
Date:
30 Jul 2010
Document's Title:
212.mrc
Spectrum Title:
13C Solvent:CDCl...:12 2010.07.30
Frequency (MHz):
(f1) 100.641
Original 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:
296.26
Number of Scans:
25
Acq. Date:
Fri Jul 30 08:56:51 PM

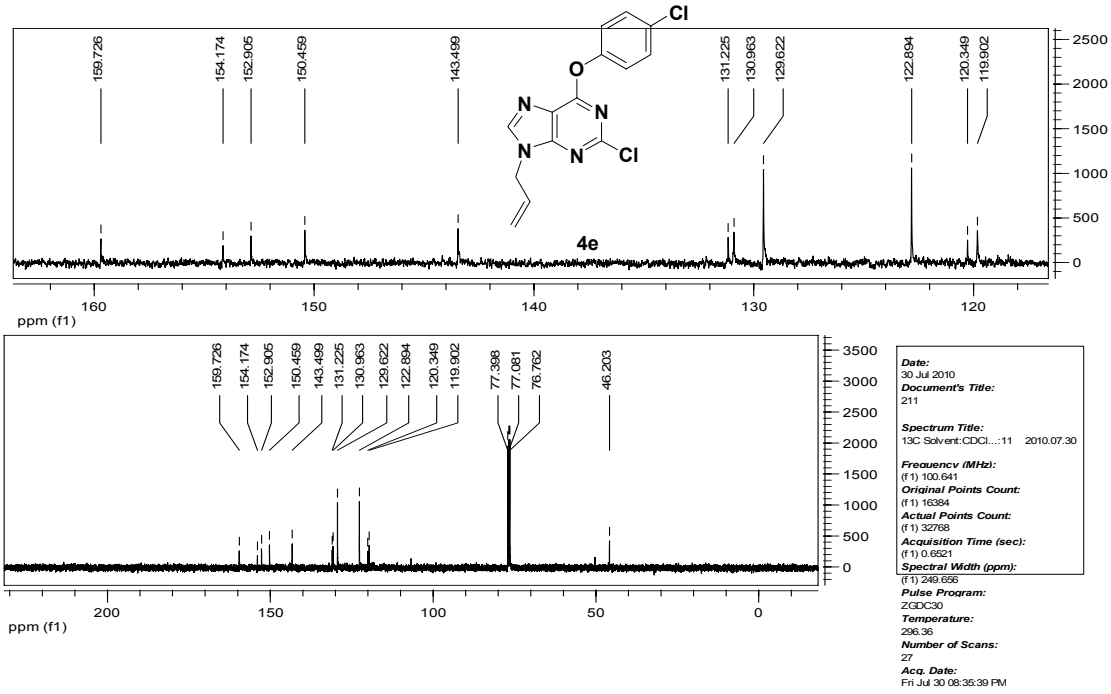

8.349
8.326
8.045
7.493
7.470
7.260
NO2
O
N
N
N
N
Cl
4c
2.01
1.00
2.00
ppm (f1)
-0.015
6.099
6.084
6.070
6.058
6.042
6.027
6.016
6.001
5.400
5.375
5.309
5.267
4.888
4.873
Date:
1 Aug 2010
Spectrum Title:
1H Solvent:CDC...:08 2010.06.11
Frequency (MHz):
(f1) 400.203
Original Points Count:
(f1) 16384
Actual Points Count:
(f1) 16384
Acquisition Time (sec):
(f1) 866.3227
Sweep Width (Hz):
(f1) 8012.820
Pulse Program:
ZG30

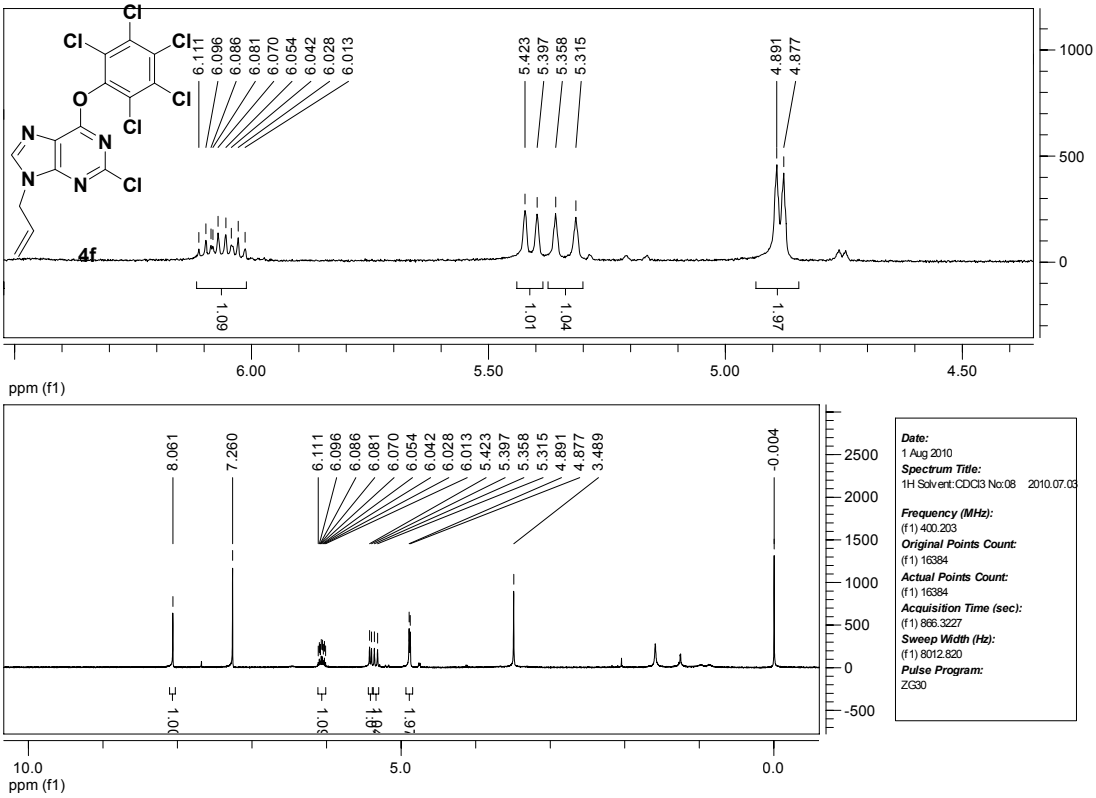

158.796
156.736
154.611
152.706
145.271
144.069
130.837
125.425
122.230
120.411
120.079
ppm (f1)
NO2
4c
77.409
77.091
76.773
46.303
Date:
30 Jul 2010
Document's Title:
213.mrc
Spectrum Title:
13C Solvent:CDCl...:13 2010.07.30
Frequency (MHz):
(f1) 100.641
Original Points Count:
(f1) 16384
Actual Points Count:
(f1) 32768
Acquisition Time (sec):
(f1) 0.6521
Spectral Width (ppm):
(f1) 249.656
Pulse Program:
ZGDC30
Temperature:
296.36
Number of Scans:
21
Acq. Date:
Fri Jul 30 09:02:56 PM

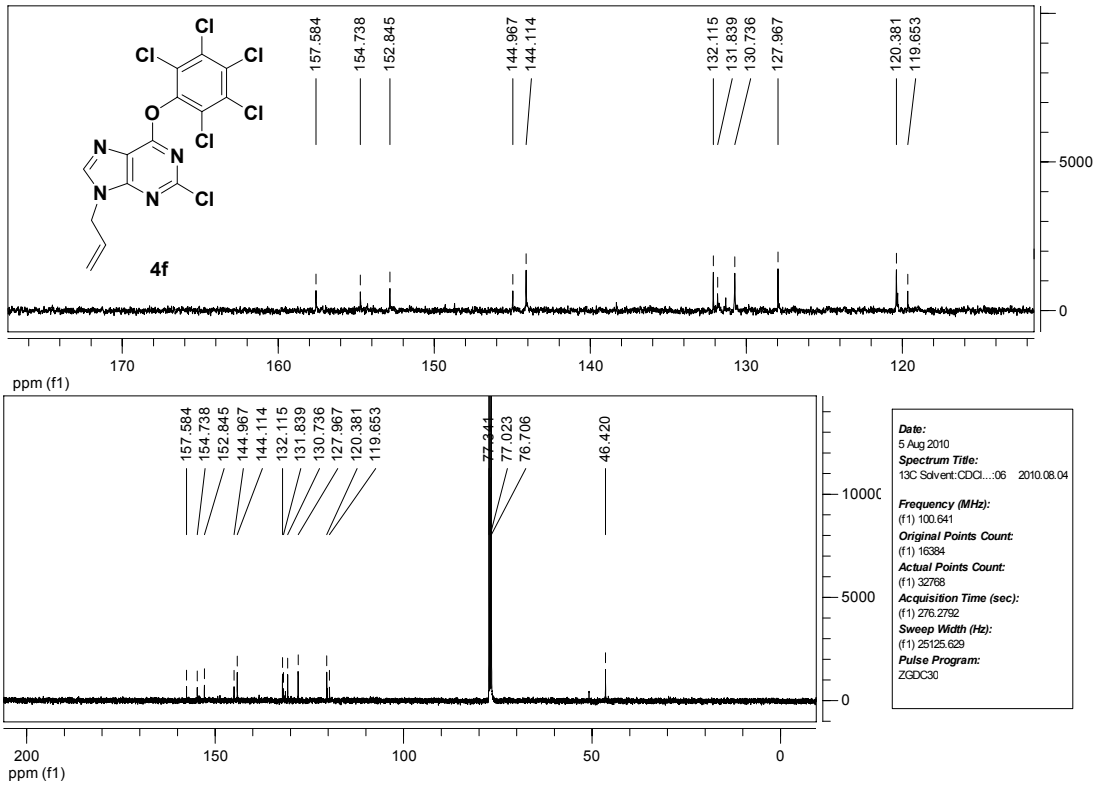

4d
8.211
8.191
8.031
7.768
7.748
7.729
7.512
7.493
7.473
7.462
7.442
7.260
1.01
1.00
1.03
2.06
ppm (f1)
-0.004
Date:
1 Aug 2010
Spectrum Title:
1H Solvent:CDCl3 No:04 2010.06.28
Frequency (MHz):
(f1) 400.203
Original Points Count:
(f1) 16384
Actual Points Count:
(f1) 16384
Acquisition Time (sec):
(f1) 866.3227
Sweep Width (Hz):
(f1) 8012.820
Pulse Program:
ZG30

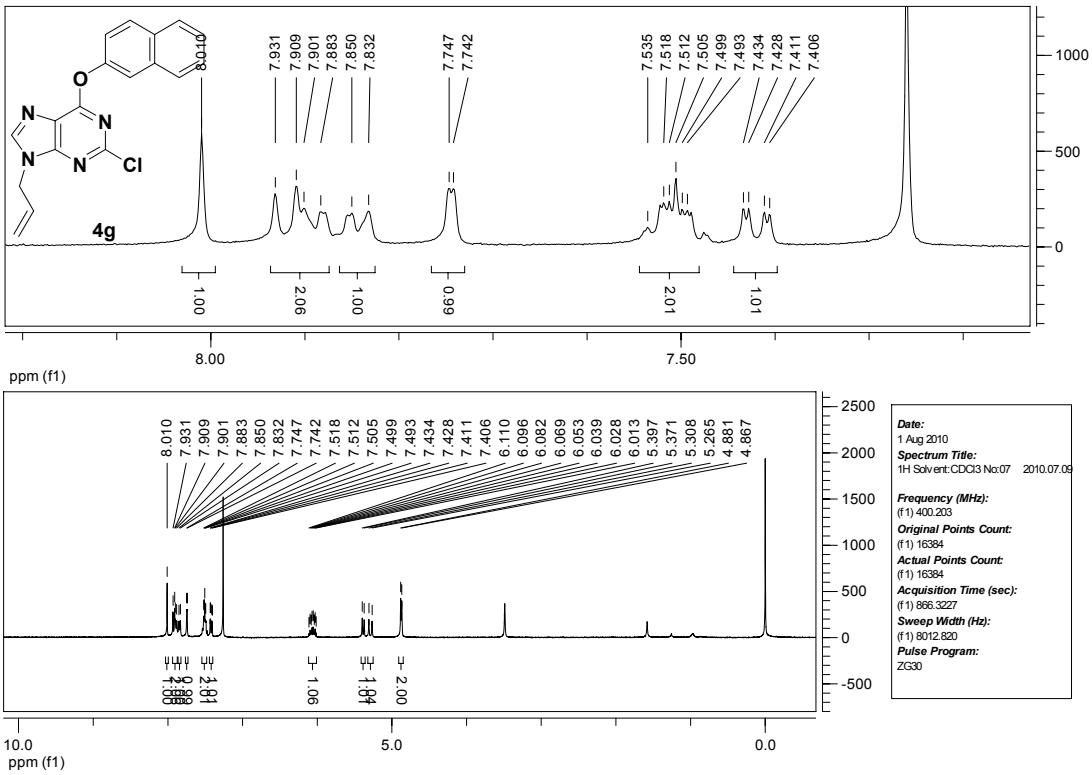

4d
159.121
154.515
152.664
145.126
143.850
141.862
135.050
130.911
126.918
126.063
125.305
120.127
120.076
77.358
77.040
76.723
46.288
ppm (f1)
Date:
30 Jul 2010
Document's Title:
210.mrc
Spectrum Title:
13C Solvent:CDCl...:10 2010.07.30
Frequency (MHz):
(f1) 100.641
Original Points Count:
(f1) 16384
Actual Points Count:
(f1) 32768
Acquisition Time (sec):
(f1) 0.6521
Spectral Width (ppm):
(f1) 249.656
Pulse Program:
ZGDC30
Temperature:
296.36
Number of Scans:
67
Acq. Date:
Fri Jul 30 08:42:33 PM

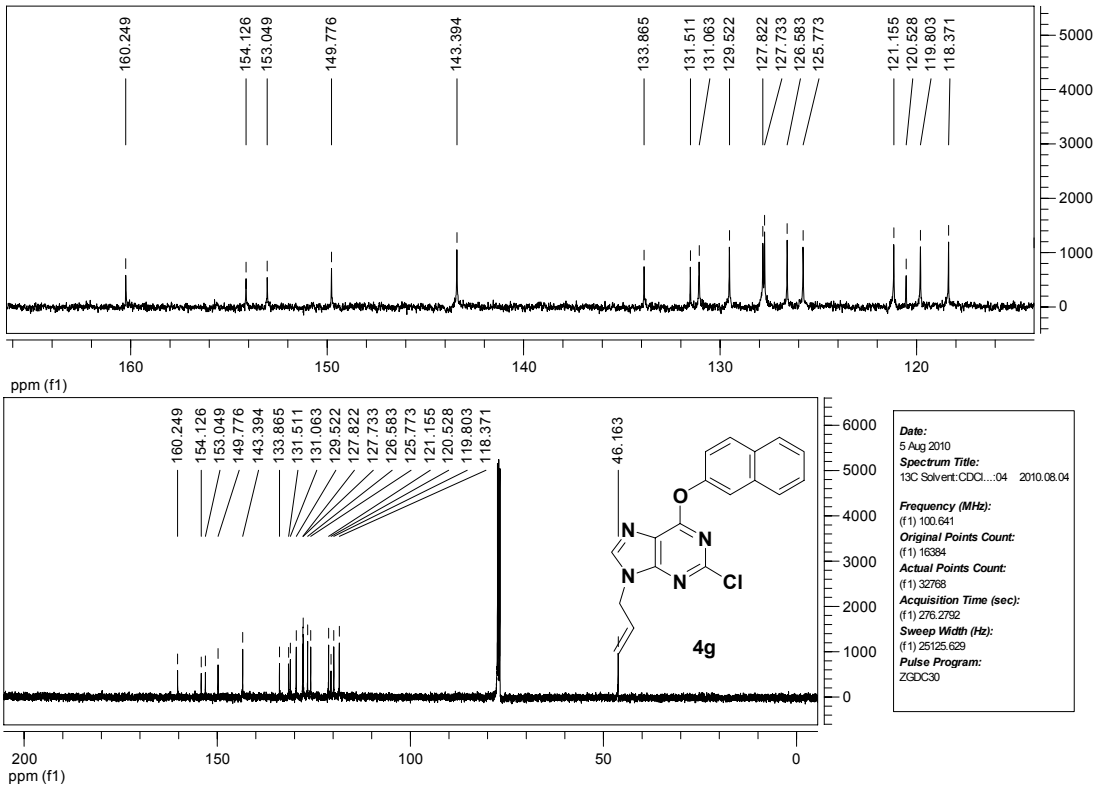

Cl
O
N
N
N
N
Cl
4e
7.995
7.407
7.385
7.260
7.241
7.219
1.00
2.02
2.01
ppm (f1)
6.084
6.070
6.055
6.044
6.027
6.016
6.001
5.987
5.376
5.351
5.284
5.241
4.860
4.845
-0.014
Date:
1 Aug 2010
Spectrum Title:
1H Solvent:CDCl3 No:02 2010.06.28
Frequency (MHz):
(f1) 400.203
Original Points Count:
(f1) 16384
Actual Points Count:
(f1) 16384
Acquisition Time (sec):
(f1) 866.3227
Sweep Width (Hz):
(f1) 8012.820
Pulse Program:
ZG30

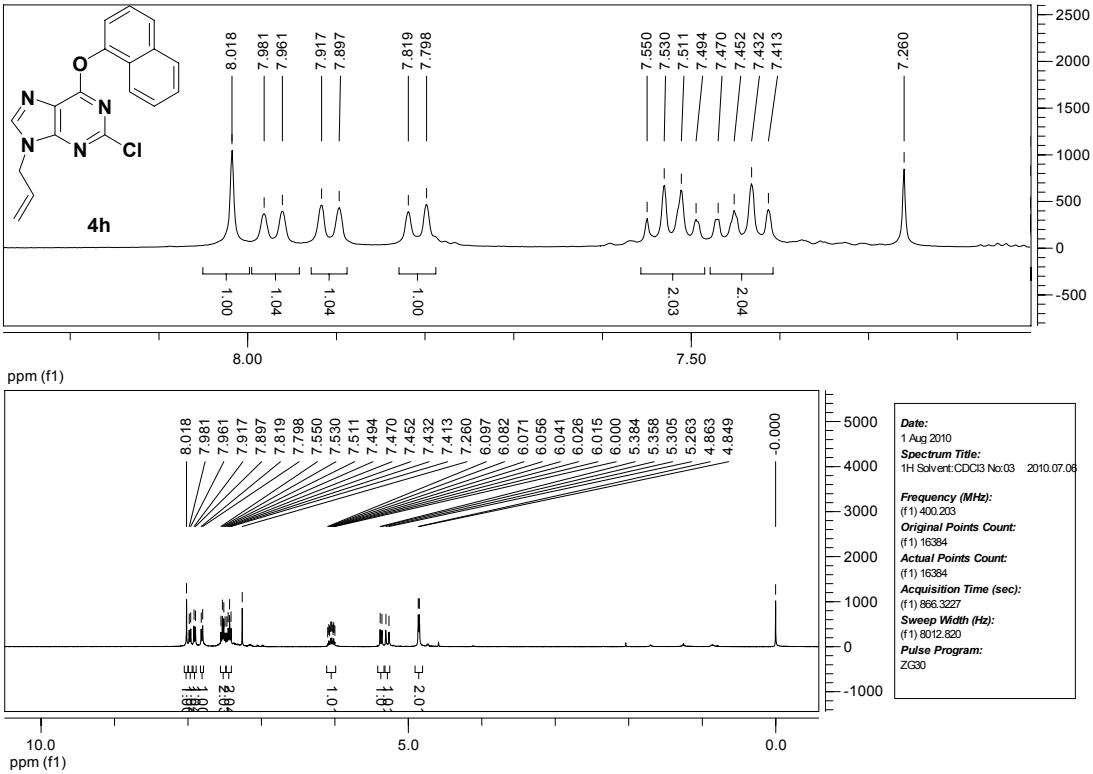

Cl
O
N
N
N
N
Cl
4e
159.726
154.174
152.905
150.459
143.499
131.225
130.963
129.622
122.894
120.349
119.902
ppm (f1)
77.398
77.081
76.762
46.203
Date:
30 Jul 2010
Document's Title:
211
Spectrum Title:
13C Solvent:CDCl...:11 2010.07.30
Frequency (MHz):
(f1) 100.641
Original Points Count:
(f1) 16384
Actual Points Count:
(f1) 32768
Acquisition Time (sec):
(f1) 0.8521
Spectral Width (ppm):
(f1) 249.656
Pulse Program:
ZGDC30
Temperature:
296.36
Number of Scans:
27
Acq. Date:
Fri Jul 30 08:35:39 PM

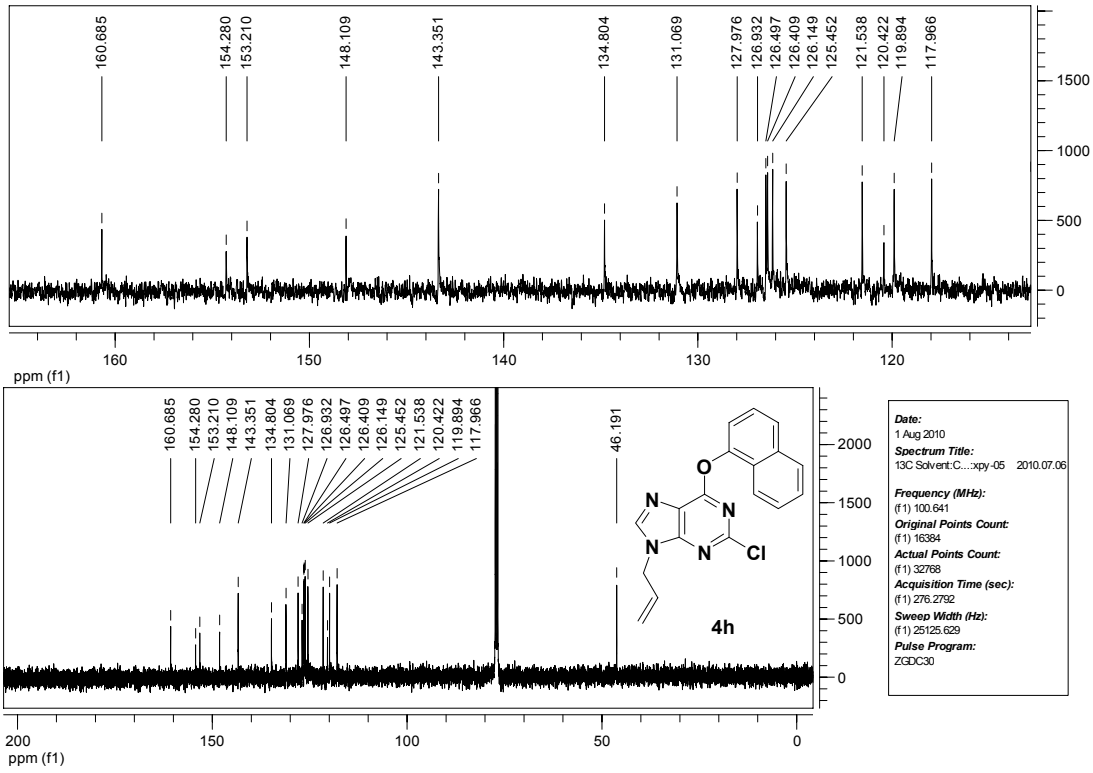

4f
6.111
6.096
6.086
6.081
6.070
6.054
6.042
6.028
6.013
5.423
5.397
5.358
5.315
4.891
4.877
1.09
1.01
1.04
1.97
ppm (f1)
8.061
7.260
-0.004
3.489
Date:
1 Aug 2010
Spectrum Title:
1H Solvent:CDCl3 No:08 2010.07.03
Frequency (MHz):
(f1) 400.203
Original Points Count:
(f1) 16384
Actual Points Count:
(f1) 16384
Acquisition Time (sec):
(f1) 866.3227
Sweep Width (Hz):
(f1) 8012.820
Pulse Program:
ZG30

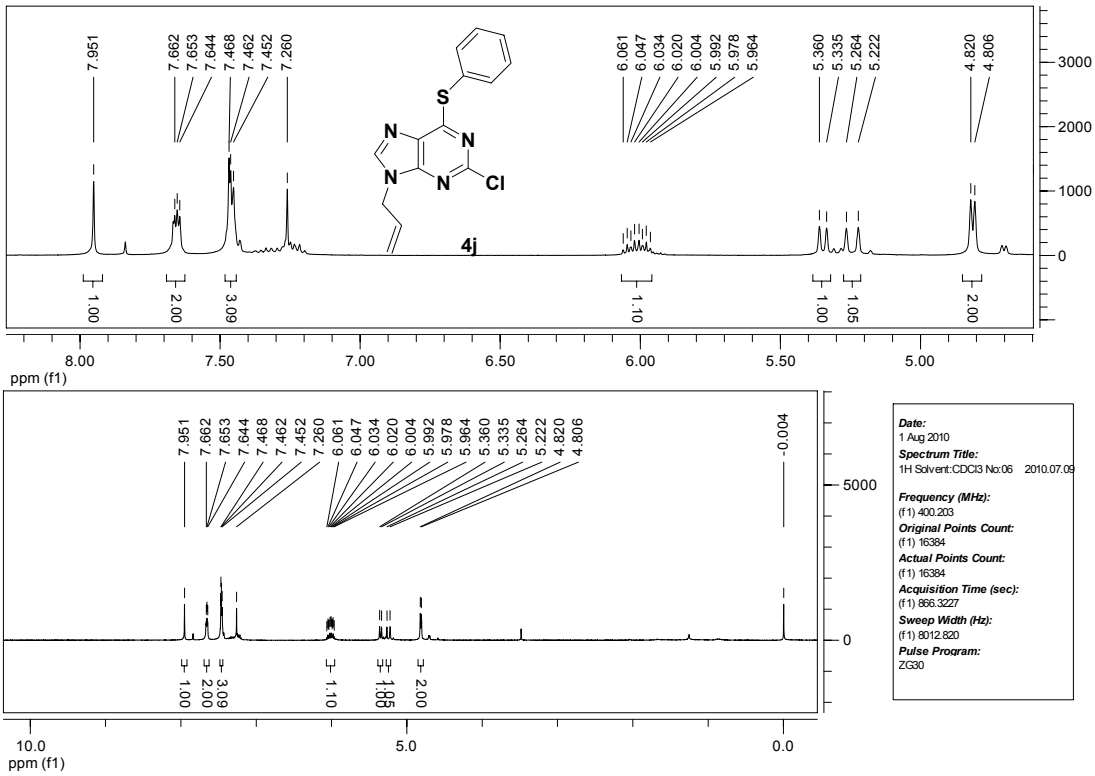

4f
157.584
154.738
152.845
144.967
144.114
132.115
131.839
130.736
127.967
120.381
119.653
ppm (f1)
77.344
77.023
76.706
46.420
Date:
5 Aug 2010
Spectrum Title:
13C Solvent:CDCl...:06 2010.08.04
Frequency (MHz):
(f1) 100.641
Original Points Count:
(f1) 16384
Actual Points Count:
(f1) 32768
Acquisition Time (sec):
(f1) 276.2792
Sweep Width (Hz):
(f1) 25125.629
Pulse Program:
ZGDC30

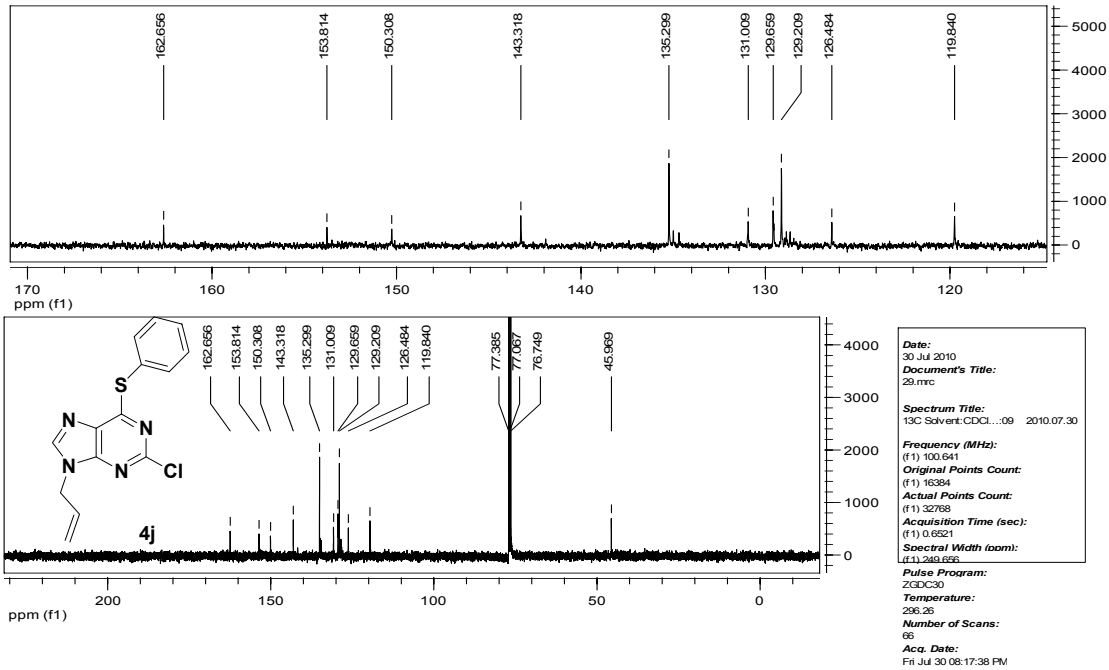

8.010
7.931
7.909
7.901
7.883
7.850
7.832
7.747
7.742
7.535
7.518
7.512
7.505
7.499
7.493
7.434
7.428
7.411
7.406
1.00
2.06
1.00
0.99
2.01
1.01
4g
8.00
7.50
ppm (f1)
6.110
6.096
6.082
6.069
6.053
6.039
6.028
6.013
5.397
5.371
5.308
5.265
4.881
4.867
1.06
1.04
2.00
10.0
5.0
0.0
Date:
1 Aug 2010
Spectrum Title:
1H Solvent:CDCl3 No:07 2010.07.09
Frequency (MHz):
(f1) 400.203
Original Points Count:
(f1) 16384
Actual Points Count:
(f1) 16384
Acquisition Time (sec):
(f1) 866.3227
Sweep Width (Hz):
(f1) 8012.820
Pulse Program:
ZG30


160.249
154.126
153.049
149.776
143.394
133.865
131.511
131.063
129.522
127.822
127.733
126.583
125.773
121.155
120.528
119.803
118.371
160
150
140
130
120
ppm (f1)
46.163
4g
200
150
100
50
0
Date:
5 Aug 2010
Spectrum Title:
13C Solvent:CDCl...:04 2010.08.04
Frequency (MHz):
(f1) 100.641
Original Points Count:
(f1) 16384
Actual Points Count:
(f1) 32768
Acquisition Time (sec):
(f1) 276.2792
Sweep Width (Hz):
(f1) 25125.629
Pulse Program:
ZGDC30


4h
8.018
7.981
7.961
7.917
7.897
7.819
7.798
7.550
7.530
7.511
7.494
7.470
7.452
7.432
7.413
7.260
1.00
1.04
1.04
1.00
2.03
2.04
ppm (f1)
Date:
1 Aug 2010
Spectrum Title:
1H Solvent:CDCl3 No:03 2010.07.06
Frequency (MHz):
(f1) 400.203
Original Points Count:
(f1) 16384
Actual Points Count:
(f1) 16384
Acquisition Time (sec):
(f1) 866.3227
Sweep Width (Hz):
(f1) 8012.820
Pulse Program:
ZG30


160.685
154.280
153.210
148.109
143.351
134.804
131.069
127.976
126.932
126.497
126.409
126.149
125.452
121.538
120.422
119.894
117.966
46.191
4h
ppm (f1)
Date:
1 Aug 2010
Spectrum Title:
13C Solvent:C...:xpy-05 2010.07.06
Frequency (MHz):
(f1) 100.641
Original Points Count:
(f1) 16384
Actual Points Count:
(f1) 32768
Acquisition Time (sec):
(f1) 276.2792
Sweep Width (Hz):
(f1) 25125.629
Pulse Program:
ZGDC30


7.951
7.662
7.653
7.644
7.468
7.462
7.452
7.260
6.061
6.047
6.034
6.020
6.004
5.992
5.978
5.964
5.360
5.335
5.264
5.222
4.820
4.806
4j
1.00
2.00
3.09
1.10
1.00
1.05
2.00
ppm (f1)
-0.004
Date:
1 Aug 2010
Spectrum Title:
1H Solvent:CDCl3 No:06 2010.07.09
Frequency (MHz):
(f1) 400.203
Original Points Count:
(f1) 16384
Actual Points Count:
(f1) 16384
Acquisition Time (sec):
(f1) 866.3227
Sweep Width (Hz):
(f1) 8012.820
Pulse Program:
ZG30


162.656
153.814
150.308
143.318
135.299
131.009
129.659
129.209
126.484
119.840
77.385
77.067
76.749
45.969
4j
ppm (f1)
Date:
30 Jul 2010
Document's Title:
29.mrc
Spectrum Title:
13C Solvent:CDCl...:09 2010.07.30
Frequency (MHz):
(f1) 100.641
Original Points Count:
(f1) 16384
Actual Points Count:
(f1) 32768
Acquisition Time (sec):
(f1) 0.6521
Pulse Program:
ZGDC30
Temperature:
296.26
Number of Scans:
66
Acq. Date:
Fri Jul 30 08:17:38 PM