Synthesis of fluorophosphonylated acyclic nucleotide analogues *via* Copper (I)-catalyzed Huisgen 1-3 dipolar cycloaddition

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Supporting Information

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Experimental Part

General. All commercially available reagents were bought from Aldrich and used as received. For anhydrous conditions the glassware was put in the oven at 120°C the day before and cooled to room temperature under a continuous nitrogen flow. Anhydrous solvents (THF, CH_2Cl_2, CH_3CN and Et_2O) were dried at a solvent generator from "Innovative Technologies Inc.", which uses an activated alumina column to remove water. $BF_3.Et_2O$, DMF and NEt_3 were distilled under CaH_2.

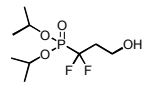
Flash column chromatographies were realized on silica gel 60 (40-63 µm) from Merck with air pressure and were detected by thin layer chromatography, on which the spots were visualized by UV-irradiation and/or $KMnO_4$ solution.

NMR spectra were recorded on 250 MHz, 400 MHz or 500 MHz apparatus in deutered solvent at 25°C. ^{31}P and ^{19}F NMR spectral lines are with respect to the internal references H_3PO_4 (capillary) and $CFCl_3$. All chemical shifts are reported in parts per million (ppm) and coupling constants are in hertz (Hz).

High-resolution mass data were recorded on a high-resolution mass spectrometer in the ESI mode.

IR spectra were recorded on a Perkin-Elmer ATR IR instrument.

1/ 1,2-Cyclic Sulfate Ring-Opening Reaction: Diisopropyl 1,1-difluoro-3-hydroxypropylphosphonate 2

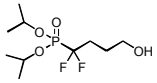


(2)

To a cooled solution of *t*-butyllithium (1.14 cm^3, 1.3 M in pentane, 1.49 mmol) in dried THF (10 cm^3) at -78 °C was added dropwise diisopropyl methylsulfanyl difluoromethyl-phosphonate **1** (0.30 g, 1.14 mmol). After 5 minutes of stirring, 1,2-cyclic sulfate (0.19 g, 1.49 mmol, in 0.5 cm^3 of THF) was slowly added. After 15 min the reaction mixture was quenched at -78 °C by addition of a 20% aqueous sulphuric acid (5 cm^3), and the mixture was slowly warmed up to room temperature. The aqueous layer was extracted with Et_2O (2 x 10 cm^3). Combined organic layers were washed with aqueous solution of $NaHCO_3$ and dried over $MgSO_4$. Solvents were evaporated under reduced pressure and crude product was purified by flash column chromatography on silica using ethyl acetate/pentane (1/1) as eluent to afford diisopropyl 1,1-difluoro-3-hydroxypropylphosphonate **2** (0.09 g, 30%) as a colourless oil;

ν_{max}(ATR)/cm^{-1} 3464, 2973, 1272 and 982; δ_H(250 MHz, $CDCl_3$) 1.39 (12 H, dd, *J* 6.2 and 1.9, 2 x (C*H*$_3$)$_2$CH), 2.27-2.35 (2 H, m, CH_2CF_2), 3.86 (2 H, t, *J* 5.5, C*H*$_2$OH), 4.87 (2 H, dsept, *J* 6.2, 2 x C*H*$(CH_3)_2$); δ_P(101 MHz, $CDCl_3$) 5.95 (1 P, t, *J* 106.0); δ_F(235 MHz $CDCl_3$) -110.07 (2 F, td, *J* 19.0 and 106.0); δ_C(63 MHz, $CDCl_3$) 24.0 (2 x d, *J* 5.0, (*C*H$_3$)$_2$CH), 24.4 (2 x d, *J* 3.5, (*C*H$_3$)$_2$CH), 38.7 (dt, *J* 15.0 and 19.9, CF_2*C*H$_2$), 56.3 (dt, *J* 6.2 and 12.2, CH_2O), 74.5 (2 x d, *J* 7.2, 2 x $(CH_3)_2$*C*H) and 120.0 (dt, *J* 215.9 and 259.4, CF_2); *m/z* (ESI) 261.1070 (M + H$^+$. $C_9H_{20}F_2O_4P$ requires 261.1067), 261 (55%), 219 (100%) and 177 (50%).

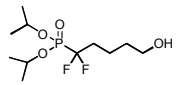
2/ Oxetane Ring-Opening Reaction: Diisopropyl 1,1-difluoro-4-hydroxybutylphosphonate 3



(3)

To a cooled solution of *t*-butyllithium (0.80 cm^3, 1.6 M in hexane, 1.26 mmol) in dried diethyl ether (10 cm^3) at -78 °C was added dropwise neat diisopropyl methylsulfanyl difluoromethylphosphonate **1** (0.30 g, 1.14 mmol). After 5 minutes of stirring, etheral solution of trimethylene oxide (0.08 cm^3, 1.26 mmol, in 0.5 cm^3 of diethyl ether) was slowly added followed by a dropwise addition of BF_3.Et_2O (0.16 cm^3, 1.26 mmol). After 3-5 minutes the reaction mixture was quenched at -78 °C by addition of saturated aqueous solution of NH_4Cl (1 cm^3), and the mixture was slowly warm-up to room temperature. The aqueous layer was extracted with CH_2Cl_2 (5 cm^3), and the organic layer was washed twice with aqueous solution of $NaHCO_3$, then with NaCl and dried over $MgSO_4$. Solvents were evaporated under reduced pressure and crude product was purified by flash column chromatography on silica using ethyl acetate-pentane (6/4) as eluent to give diisopropyl 1,1-difluoro-4-hydroxybutylphosphonate **3** (0.21 g, 63%) as a colourless oil; ν_{max}(ATR)/cm^{-1} 3444, 2982, 1261, 1103 and 1005; δ_H(400 MHz, $CDCl_3$) 1.38 (12 H, dd, *J* 6.2 and 2.0, 2 x (C*H*$_3$)$_2$CH), 1.75-2.03 (3 H, m, CH_2C*H*$_2$CH$_2$), 2.05-2.32 (2 H, m, CH_2CF_2), 3.70 (2 H, t, *J* 6.2, C*H*$_2$OH), 4.84 (2 H, dsept, *J* 6.2, 2 x C*H*$(CH_3)_2$); δ_P(101 MHz, $CDCl_3$) 6.74 (1 P, t, *J* 110.0); δ_F(235 MHz $CDCl_3$) -113.10 (2 F, td, *J* 20.9 and 110.0); δ_C(101 MHz, $CDCl_3$) 23.7 (2 x d, *J* 4.9, (*C*H$_3$)$_2$CH), 24.0 (dt, *J* 4.4 and 10.2, CF_2CH_2*C*H$_2$), 24.1 (2 x d, *J* 3.4, (*C*H$_3$)$_2$CH), 30.4 (dt, *J* 14.9 and 21.3, CF_2*C*H$_2$), 61.8 (s, CH_2O), 71.5 (2 x d, *J* 7.1, 2 x (*C*H$_3$)$_2$CH) and 120.6 (dt, *J* 218.0 and 259.0, CF_2); *m/z* (EI) 274.1157 (M$^+$. $C_{10}H_{21}F_2O_4P$ requires 274.1146), 274 (28%), 233 (37%), 191 (31%), 173 (100%), 172 (40%), 123 (69%), 101 (28%), 43 (43%) and 41 (41%).

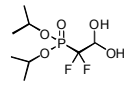
3/ Tetrahydrofuran Ring-Opening Reaction: Diisopropyl 1,1-difluoro-5-hydroxypentylphosphonate 4



(4)

To a cooled solution of *t*-butyllithium (6.05 cm^3, 1.6 M in hexane, 9.71 mmol) in dried THF (30 cm^3) at -78°C was added dropwise neat diisopropyl methylsulfanyl difluoromethylphosphonate **1** (2.30 g, 8.80 mmol). After 5 minutes of stirring, solution of BF_3.Et_2O (2.23 cm^3, 17.60 mmol) in THF (5 cm^3) was slowly added over 20 minutes period using a syringe-pump. After 15 minutes the reaction mixture was quenched at -78 °C by addition of saturated aqueous solution of NH_4Cl (5 cm^3), and the mixture was slowly warm-up to room temperature. The aqueous layer was extracted with CH_2Cl_2 (5 cm^3), and the organic layer was washed twice with aqueous solution of $NaHCO_3$, then with NaCl and dried over $MgSO_4$. Solvents were evaporated under reduced pressure and crude product was purified by flash column chromatography on silica using ethyl acetate-pentane (8/2) as eluent to give diisopropyl 1,1-difluoro-5-hydroxypentylphosphonate **4** (1.85 g, 71%) as a colourless oil; ν_{max}(ATR)/cm^{-1} 3456, 2984, 1268 and 987; δ_H(250 MHz, $CDCl_3$) 1.38 (12 H, dd, *J* 6.2 and 2.2, 2 x (C*H*$_3$)$_2$CH), 1.60 (1 H, br s, OH), 1.63-1.71 (4 H, m, CH_2C*H*$_2$C*H*$_2$CH$_2$), 2.04-2.10 (2 H, m, CH_2CF_2), 3.68 (2 H, t, *J* 6.1, C*H*$_2$OH), 4.80 (2 H, dsept, *J* 6.3, 2 x C*H*$(CH_3)_2$); δ_P(101 MHz, $CDCl_3$) 6.79 (1 P, t, *J* 108.1); δ_F(235 MHz $CDCl_3$) -113.20 (2 F, td, *J* 19.0 and 108.1); δ_C(63 MHz, $CDCl_3$) 17.5 (dt, *J* 5.0 and 10.5, CF_2CH_2*C*H$_2$), 24.1 (2 x d, *J* 5.1, (*C*H$_3$)$_2$CH), 24.4 (2 x d, *J* 3.4, (*C*H$_3$)$_2$CH), 32.6 (s, *C*H$_2$CH$_2$O), 34.0 (dt, *J* 14.4 and 21.0, CF_2*C*H$_2$), 62.4 (s, CH_2*C*H$_2$O), 73.9 (2 x d, *J* 6.9, 2 x $(CH_3)_2$*C*H) and 120.9 (dt, *J* 217.0 and 259.0, CF_2); *m/z* (EI) 288.1296 (M$^+$. $C_{11}H_{23}F_2O_4P$ requires 288.1302), 288 (8%), 204 (75%), 187 (100%), 123 (68%), 109 (35%), 99 (31%) and 43 (33%).

4/ Diisopropyl 1,1-difluoro-2,2-dihydroxyethylphosphonate 17



(17)

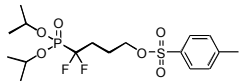
To a cooled solution of *t*-butyllithium (0.80 cm^3, 1.6 M in hexane, 1.26 mmol) in dried THF (10 cm^3) at -78 °C was added dropwise neat diisopropyl methylsulfanyl difluoromethylphosphonate **1** (0.30 g, 1.14 mmol). After 5 minutes of stirring, dimethyl

formamide (0.116 cm^3, 1.49 mmol) was added. After 1h of stirring at -78°C, the mixture was acidified by addition of 3 cm^3 of HCl (1 M) and was slowly warm-up to room temperature. The aqueous layer was extracted with CH_2Cl_2 (5 cm^3), and the organic layer was washed twice with aqueous solution of $NaHCO_3$, then with NaCl and dried over $MgSO_4$. Solvents were evaporated under reduced pressure and crude product was purified by flash column chromatography on silica using ethyl acetate-ether (7/3) as eluent to give diisopropyl 1,1-difluoro-2,2-dihydroxyethylphosphonate **17** (0.24 g, 80%) as a white solid, mp 78-80 °C; ν_{max}(ATR)/cm^{-1} 3600, 3200, 2950, 1470, 1390, 1220, 1180, 1100 and 1000; δ_H(250 MHz, $CDCl_3$) 1.38 (6 H, dd, *J* 5.9, $(CH_3)_2CH$), 1.41 (6 H, dd, *J* 5.9, $(CH_3)_2CH$), 4.91 (2 H, dsept, *J* 6.1, 2 x $CH(CH_3)_2$), 4.50-5.51 (2 H, m, OH), 5.15 (1 H, dt, *J* 6.7 and 11.6, $CH(OH)_2$); δ_P(101 MHz, $CDCl_3$) 4.60 (1 P, t, *J* 97.8); δ_F(235 MHz $CDCl_3$) -122.50 (2 F, dd, *J* 6.7 and 97.8); δ_C(101 MHz, $CDCl_3$) 23.5 (2 x d, *J* 5.6, $(CH_3)_2CH$), 24.2 (2 x d, *J* 2.9, $(CH_3)_2CH$), 74.7 (2 x d, *J* 7.1, 2 x $(CH_3)_2CH$), 89.7 (dt, *J* 15.2 and *J* 28.3, CF_2CH) and 115.7 (dt, *J* 202.3 and 268.2, CF_2); Anal. Calcd for $C_8H_{17}F_2O_5P$: C, 36.65; H, 6.54. Found: C, 36.69; H, 6.54%.

5/ Tosyl compounds: General procedure for the preparation of 6 and 7

In a 25 mL round bottom flask under N_2 atmosphere were placed the alcohol (1.00 mmol), tosyl chloride (1.50 mmol) and 10 cm^3 of anhydrous CH_2Cl_2. Triethylamine (1.50 mmol) was added dropwise and the mixture was stirred 24 hours at room temperature. The solution was hydrolyzed with 1N aqueous HCl solution and extracted with CH_2Cl_2. The organic layer was successively washed with saturated aqueous solution of $NaHCO_3$ and NaCl and dried over $MgSO_4$. Solvents were evaporated under reduced pressure and the crude product was purified by flash column chromatography on silica using ethyl acetate/pentane (6/4) as eluent to give products **6** and **7** in 78-85% yields as a colourless oil.

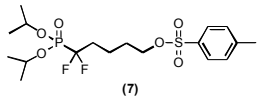
Diisopropyl 1,1-difluoro-4-(p-toluenesulfonyloxy)butylphosphonate (6) Yield: 78%



(6)

ν_{max}(ATR)/cm^{-1} 2975, 1376, 1265, 1164 and 986; δ_H(250 MHz, $CDCl_3$) 1.34 (12 H, dd, *J* 6.1 and 2.4, 2 x $(CH_3)_2CH$), 1.80-1.99 (2 H, m, $CH_2CH_2CH_2$), 2.01-2.20 (2 H, m, CH_2CF_2), 2.42 (3 H, s, CH_3Ph), 4.04 (2 H, t, *J* 6.0, CH_2OTs), 4.81 (2 H, dsept, *J* 6.3, 2 x $CH(CH_3)_2$), 7.31 (2 H, d, *J* 8.2, H_{ar}), 7.76 (2 H, d, *J* 8.2, H_{ar}); δ_P(101 MHz, $CDCl_3$) 5.16 (1 P, t, *J* 107.5); δ_F(235 MHz $CDCl_3$) -113.02 (2 F, td, *J* 19.0 and 107.5); δ_C(63 MHz, $CDCl_3$) 17.4 (dt, *J* 5.1 and 9.8, $CF_2CH_2CH_2$), 21.9 (s, $PhCH_3$), 24.0 (2 x d, *J* 4.7, $(CH_3)_2CH$), 24.4 (2 x d, *J* 3.4, $(CH_3)_2CH$), 33.4 (dt, *J* 15.0 and 21.3, CF_2CH_2), 70.2 (s, CH_2O), 73.8 (2 x d, *J* 7.0, 2 x $(CH_3)_2CH$), 120.7 (dt, *J* 218.1 and 259.6, CF_2), 127.9 (2 x s, 2 x C_{ar}), 129.9 (2 x s, 2 x C_{ar}), 133.2 (s, C_{ar}) and 144.33 (s, C_{ar}); *m/z* (ESI) 429.1312 (M + H^+. $C_{17}H_{28}F_2O_6PS$ requires 429.1315), 429 (22%), 387 (55%), 344 (100).

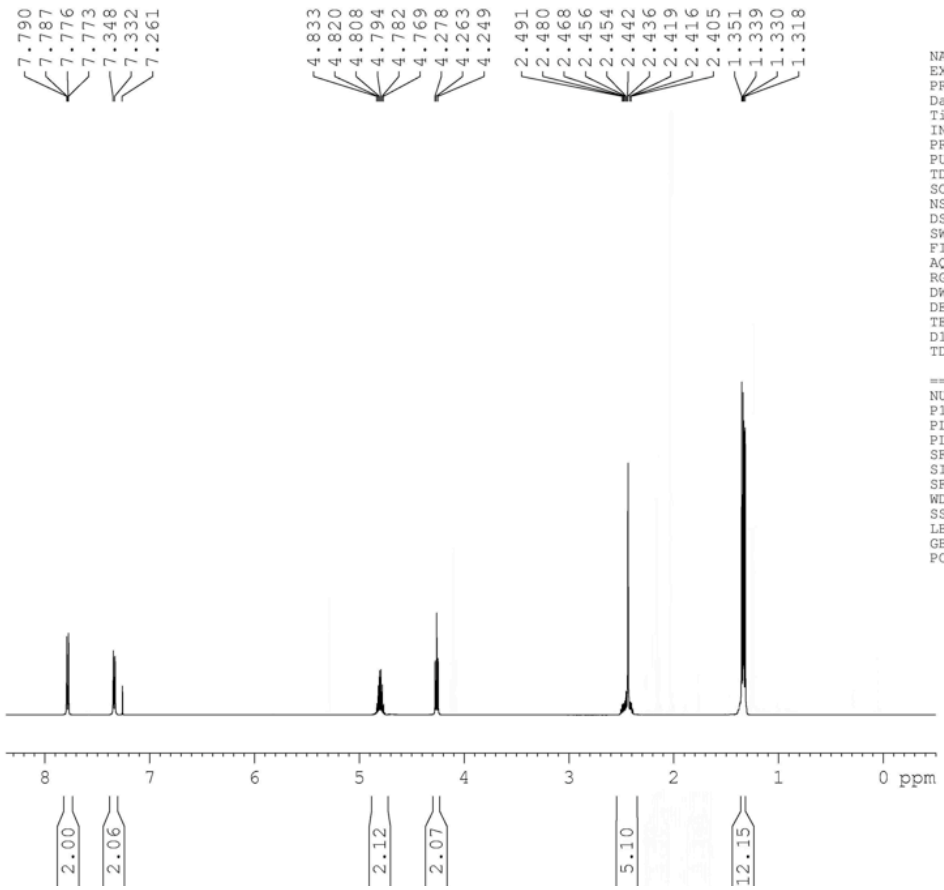
Diisopropyl 1,1-difluoro-5-(p-toluenesulfonyloxy)pentylphosphonate (7) Yield: 85%



(7)

ν_{max}(ATR)/cm^{-1} 2985, 1381, 1272, 1177 and 989; δ_H(400 MHz, $CDCl_3$) 1.35 (12 H, dd, *J* 6.1 and 2.9, 2 x $(CH_3)_2CH$), 1.50-1.72 (4 H, m, $CH_2CH_2CH_2CH_2$), 1.85-2.15 (2 H, m, CH_2CF_2), 2.43 (3 H, s, CH_3Ph), 4.02 (2 H, t, *J* 6.0, CH_2OTs), 4.81 (2 H, dsept, *J* 6.3, 2 x $CH(CH_3)_2$), 7.33 (2 H, d, *J* 8.2, H_{ar}), 7.77 (2 H, d, *J* 8.2, H_{ar}); δ_P(101 MHz, $CDCl_3$) 5.35 (1 P, t, *J* 108.0); δ_F(235 MHz $CDCl_3$) -113.10 (2 F, td, *J* 20.0 and 108.0); δ_C(63 MHz, $CDCl_3$) 17.3 (dt, *J* 5.0 and 10.3, $CF_2CH_2CH_2$), 21.6 (s, $PhCH_3$), 23.7 (2 x d, *J* 4.9, $(CH_3)_2CH$), 24.0 (2 x d, *J* 3.5, $(CH_3)_2CH$), 28.4 (s, CH_2CH_2O), 33.1 (dt, *J* 14.5 and 21.1, CF_2CH_2), 69.9 (s, CH_2CH_2O), 73.5 (2 x d, *J* 7.1, 2 x $(CH_3)_2CH$), 120.2 (dt, *J* 217.6 and 259.8, CF_2), 127.8 (2 x s, 2 x C_{ar}), 129.9 (2 x s, 2 x C_{ar}), 132.9 (s, C_{ar}) and 144.8 (s, C_{ar}); *m/z* (ESI) 443.1469 (M + H^+. $C_{18}H_{30}F_2O_6PS$ requires 443.1452), 443 (36%), 400 (70%), 359 (100).

5/ NMR Spectra :

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D1             1.00000000 sec
TD0                    1

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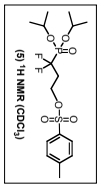

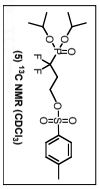


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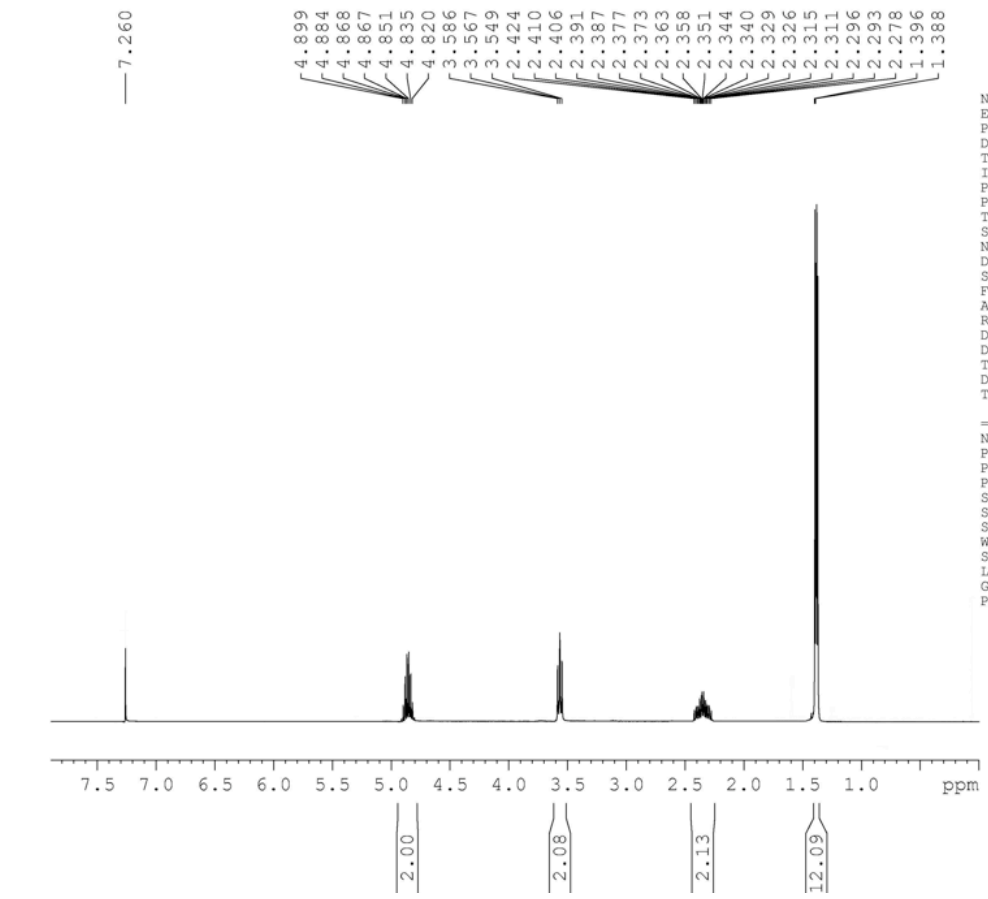


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TD0                    1

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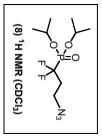

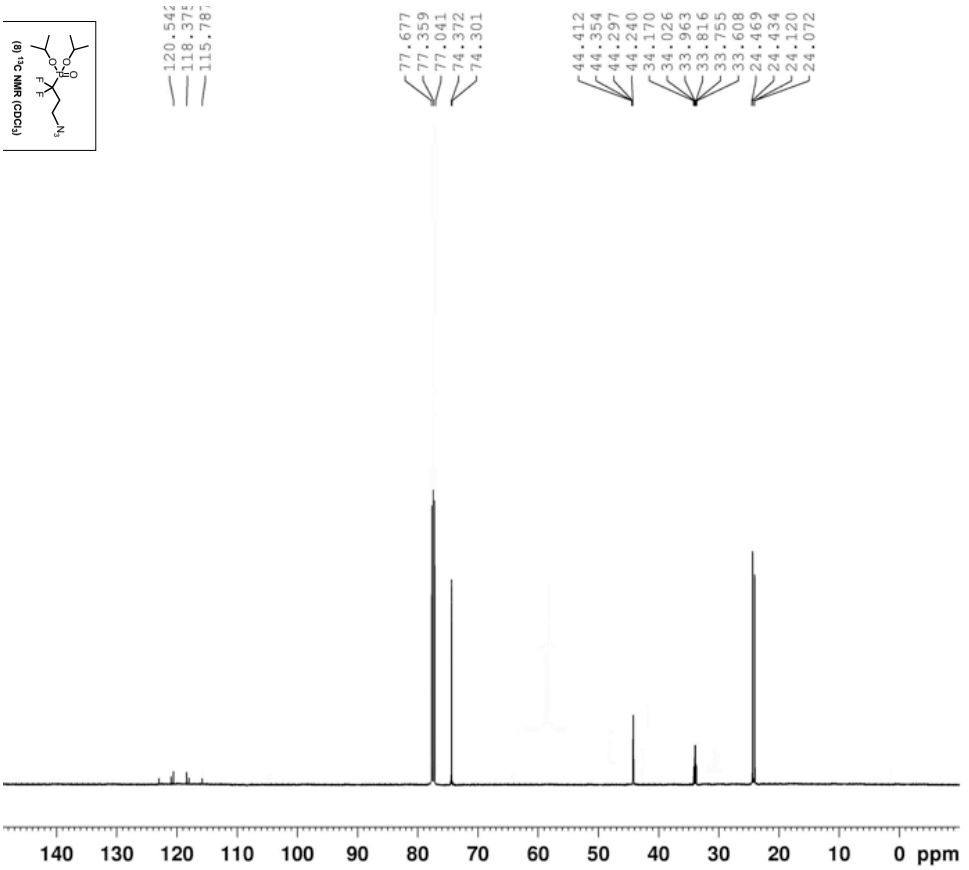


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DE                 8.00 usec
TE                295.0 K
D1             1.50000000 sec
D11            0.03000000 sec
TD0                    1

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SSB                    0
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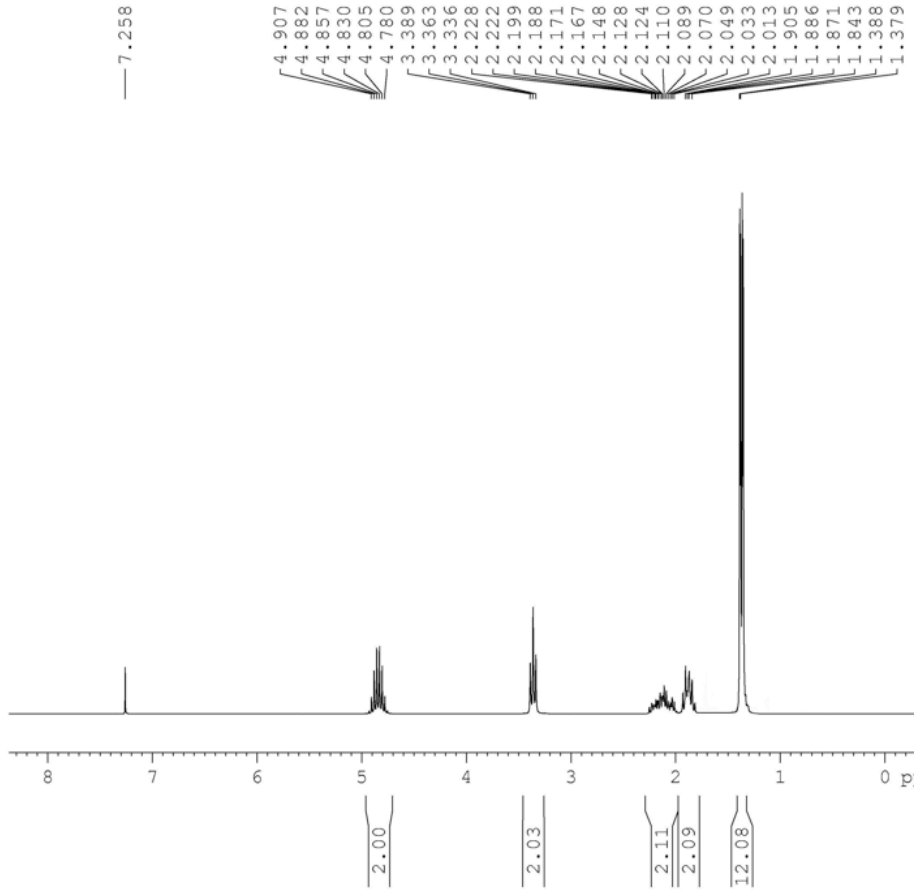
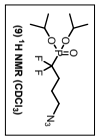
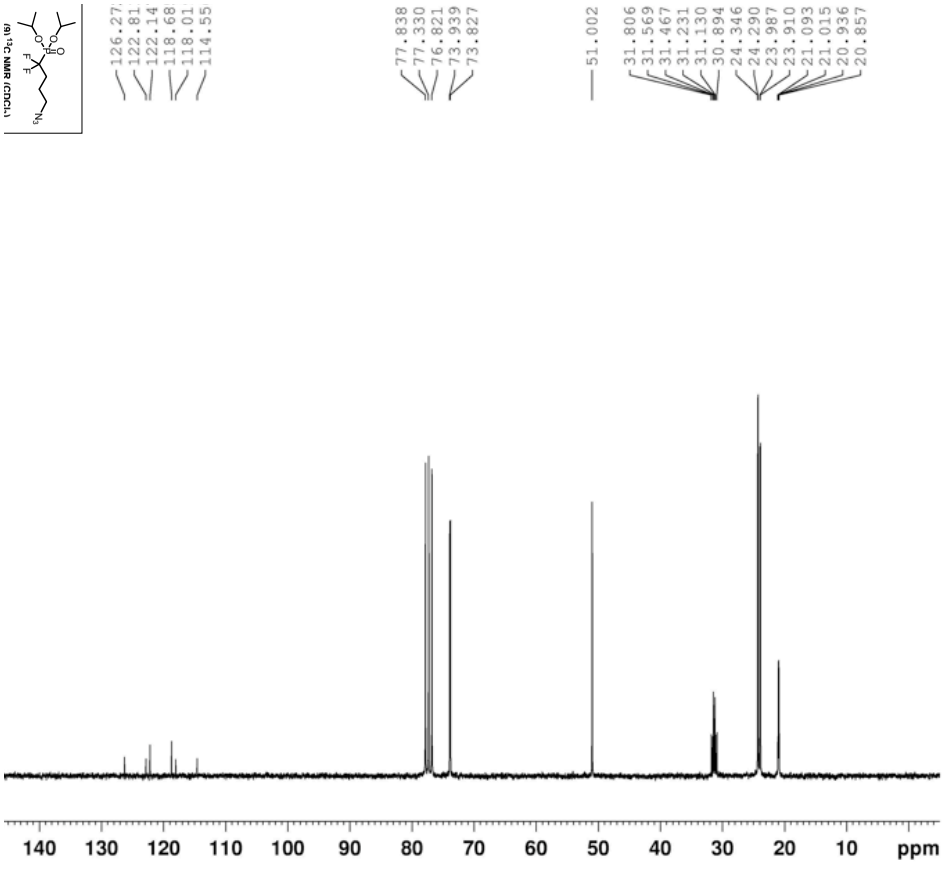
PC                  1.40
```




```
NAME                ah14
EXPNO                 10
PROCNO                 1
Date_           20061205
Time                6.48
INSTRUM            spect
PROBHD    5 mm QNP 1H/13
PULPROG             zg30
TD                 32768
SOLVENT            CDC13
NS                    16
DS                     0
SWH             4990.020 Hz
FIDRES          0.152283 Hz
AQ              3.2834036 sec
RG                   362
DW               100.200 usec
DE                10.00 usec
TE                683.2 K
D1             2.00000000 sec
MCREST         0.00000000 sec
MCWRK          0.01500000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                 7.50 usec
PL1                -6.00 dB
SFO1         250.1318626 MHz
SI                 16384
SF           250.1300006 MHz
WDW                   EM
SSB                    0
LB                 0.30 Hz
GB                     0
PC                 0.50
```

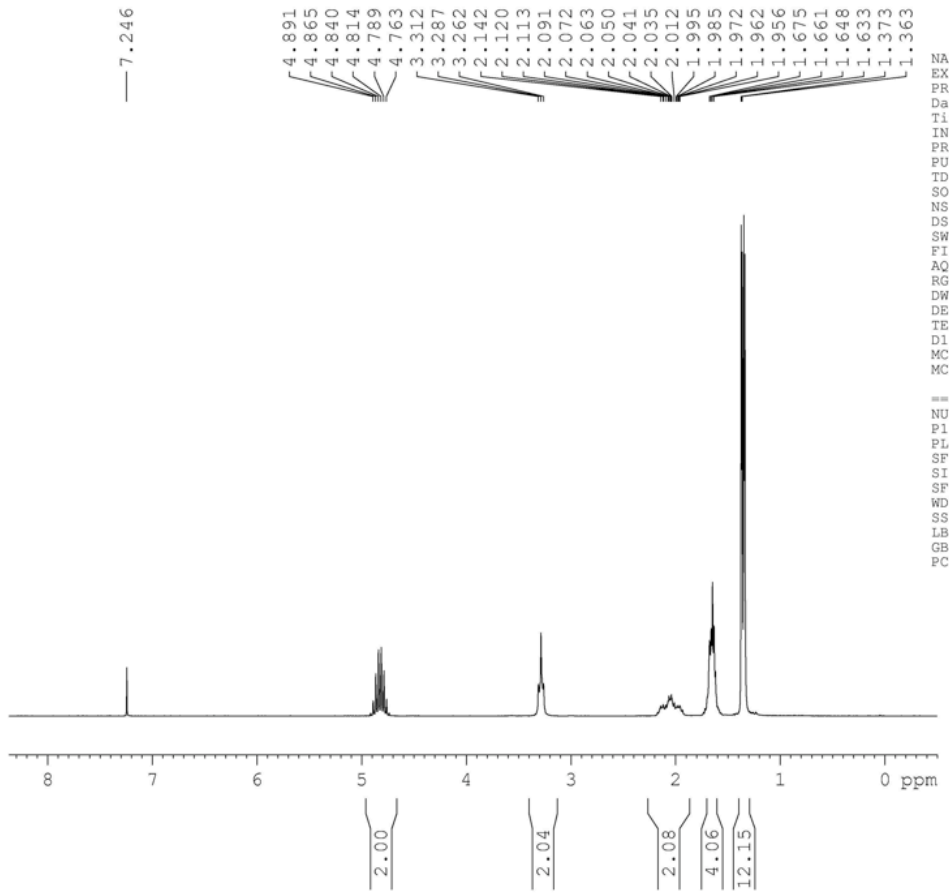





```
NAME                ah14
EXPNO                 20
PROCNO                 1
Date_           20061208
Time               21.15
INSTRUM            spect
PROBHD    5 mm QNP 1H/13
PULPROG          zgdc30
TD                 65536
SOLVENT            CDC13
NS                  1024
DS                     0
SWH            16339.869 Hz
FIDRES          0.249327 Hz
AQ              2.0054517 sec
RG                1149.4
DW               30.600 usec
DE                10.00 usec
TE                683.2 K
D1             2.00000000 sec
d11            0.03000000 sec
MCREST         0.00000000 sec
MCWRK          0.01500000 sec

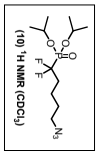
======== CHANNEL f1 ========
NUC1                 13C
P1                 5.00 usec
PL1                -6.00 dB
SFO1         62.9027614 MHz

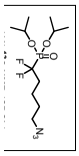
======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2            100.00 usec
PL2                -6.00 dB
PL12               18.00 dB
SFO2         250.1312506 MHz
SI                 32768
SF           62.8952212 MHz
WDW                   EM
SSB                    0
LB                 1.00 Hz
GB                     0
PC                 1.40
```

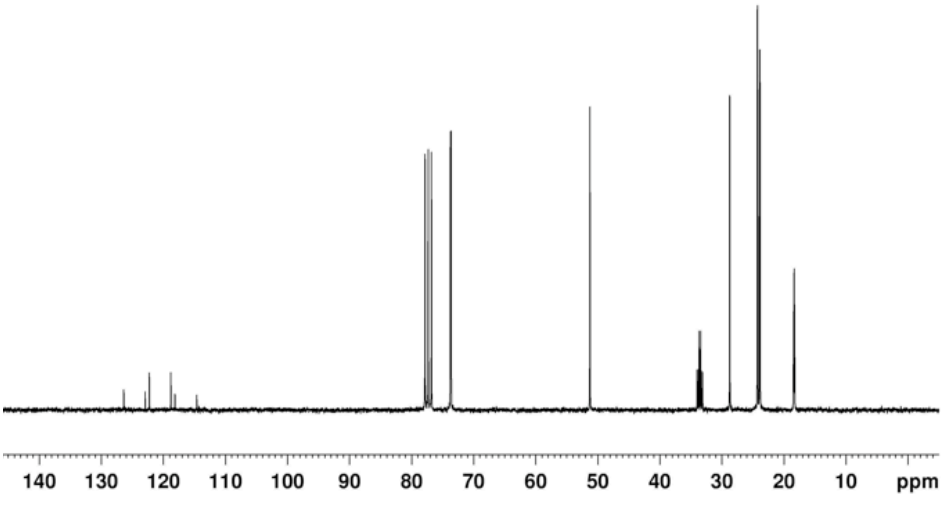



```
NAME                ah15
EXPNO                 10
PROCNO                 1
Date_           20061206
Time                10.16
INSTRUM             spect
PROBHD    5 mm QNP 1H/13
PULPROG              zg30
TD                  32768
SOLVENT             CDC13
NS                     16
DS                      0
SWH              4990.020 Hz
FIDRES           0.152283 Hz
AQ               3.2834036 sec
RG                   406.4
DW                 100.200 usec
DE                  10.00 usec
TE                  683.2 K
D1              2.00000000 sec
MCREST          0.00000000 sec
MCWRK           0.01500000 sec

======== CHANNEL f1 ========
NUC1                   1H
P1                   7.50 usec
PL1                 -6.00 dB
SFO1          250.1318626 MHz
SI                  16384
SF            250.1300037 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   0.50
```



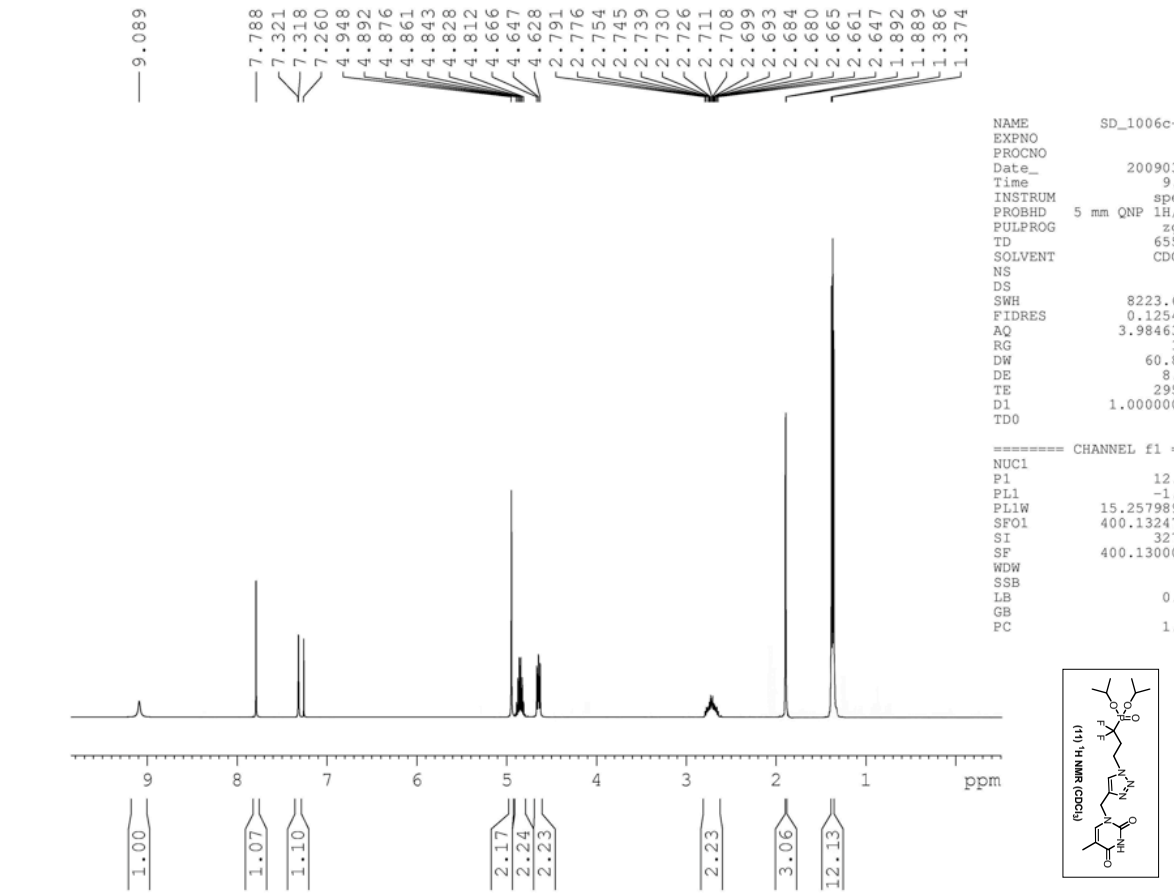





```
NAME                ah15
EXPNO                 20
PROCNO                 1
Date_           20061208
Time                22.31
INSTRUM             spect
PROBHD    5 mm QNP 1H/13
PULPROG           zgdc30
TD                  65536
SOLVENT             CDC13
NS                   1024
DS                      0
SWH             16339.869 Hz
FIDRES           0.249327 Hz
AQ               2.0054517 sec
RG                    512
DW                  30.600 usec
DE                  10.00 usec
TE                  683.2 K
D1              2.00000000 sec
d11             0.03000000 sec
MCREST          0.00000000 sec
MCWRK           0.01500000 sec

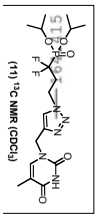
======== CHANNEL f1 ========
NUC1                  13C
P1                   5.00 usec
PL1                 -6.00 dB
SFO1          62.9027614 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                   1H
PCPD2              100.00 usec
PL2                 -6.00 dB
PL12                18.00 dB
SFO2          250.1312506 MHz
SI                  32768
SF            62.8952223 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.40
```




```
NAME          SD_1006c-F2
EXPNO                  12
PROCNO                  1
Date_            20090316
Time                 9.12
INSTRUM             spect
PROBHD    5 mm QNP 1H/13
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     32
DS                      2
SWH              8223.685 Hz
FIDRES           0.125483 Hz
AQ               3.9846387 sec
RG                    161
DW                 60.800 usec
DE                  8.00 usec
TE                 295.0 K
D1              1.00000000 sec
TD0                     1

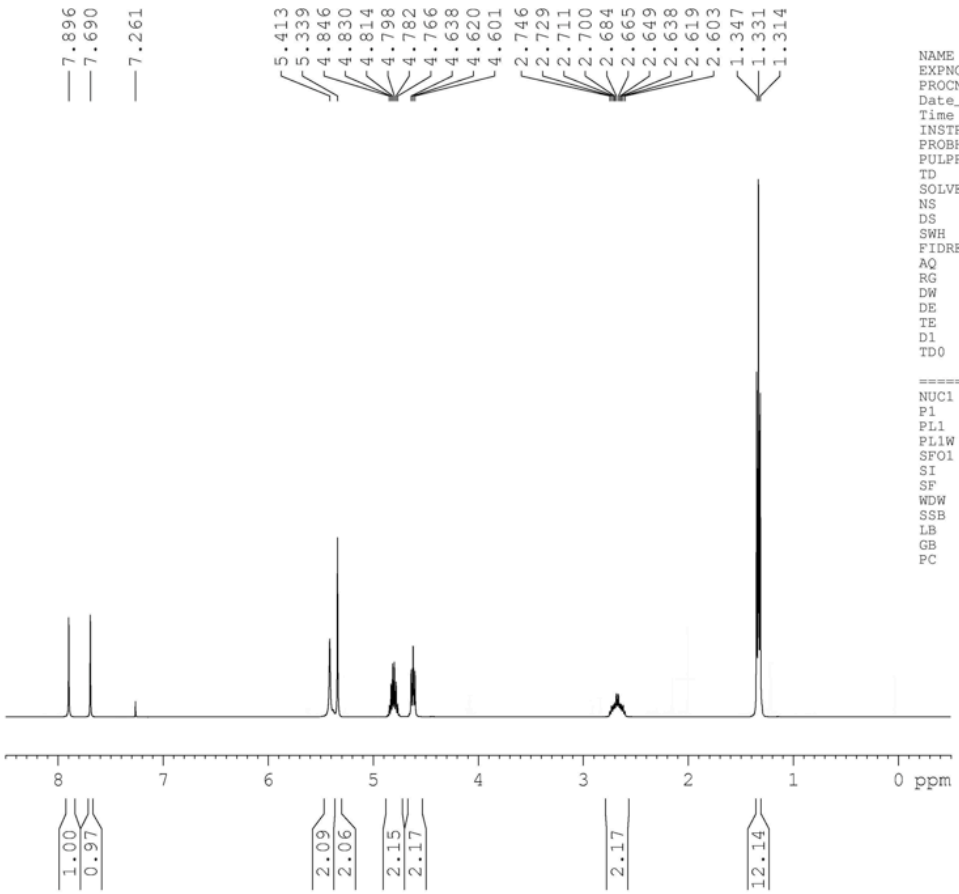
======== CHANNEL f1 ========
NUC1                   1H
P1                  12.00 usec
PL1                 -1.00 dB
PL1W           15.25798988 W
SFO1           400.1324710 MHz
SI                  32768
SF             400.1300097 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
```


```
NAME          SD_1006c_F2
EXPNO                   2
PROCNO                  1
Date_            20090317
Time                 5.55
INSTRUM             spect
PROBHD    5 mm PABBO BB-
PULPROG           zgpg30
TD                  65536
SOLVENT             CDCl3
NS                  10240
DS                      2
SWH             31250.000 Hz
FIDRES           0.476837 Hz
AQ               1.0486259 sec
RG                    575
DW                 16.000 usec
DE                  8.00 usec
TE                 295.0 K
D1              3.00000000 sec
D11             0.03000000 sec
TD0                     1

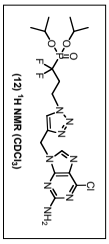
======== CHANNEL f1 ========
NUC1                  13C
P1                  10.50 usec
PL1                  1.50 dB
PL1W           64.32036591 W
SFO1           125.7703648 MHz

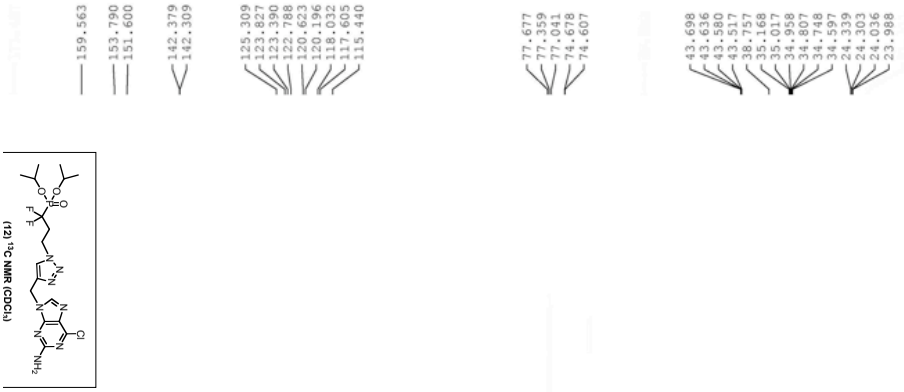
======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                   1H
PCPD2               80.00 usec
PL2                  1.90 dB
PL12                16.73 dB
PL13                17.00 dB
PL2W           16.07213783 W
PL12W           0.52853495 W
PL13W           0.49667653 W
SFO2           500.1316005 MHz
SI                 131072
SF             125.7577501 MHz
WDW                    EM
SSB                     0
LB                   2.50 Hz
GB                      0
PC                   1.00
```




```
NAME            SD_1048c
EXPNO                12
PROCNO                1
Date_          20090417
Time               6.12
INSTRUM           spect
PROBHD   5 mm QNP 1H/13
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ            3.9846387 sec
RG                 90.5
DW               60.800 usec
DE                8.00 usec
TE                295.0 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                12.00 usec
PL1               -1.00 dB
PL1W        15.25798988 W
SFO1       400.1324710 MHz
SI                 32768
SF         400.1300095 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                1.00
```

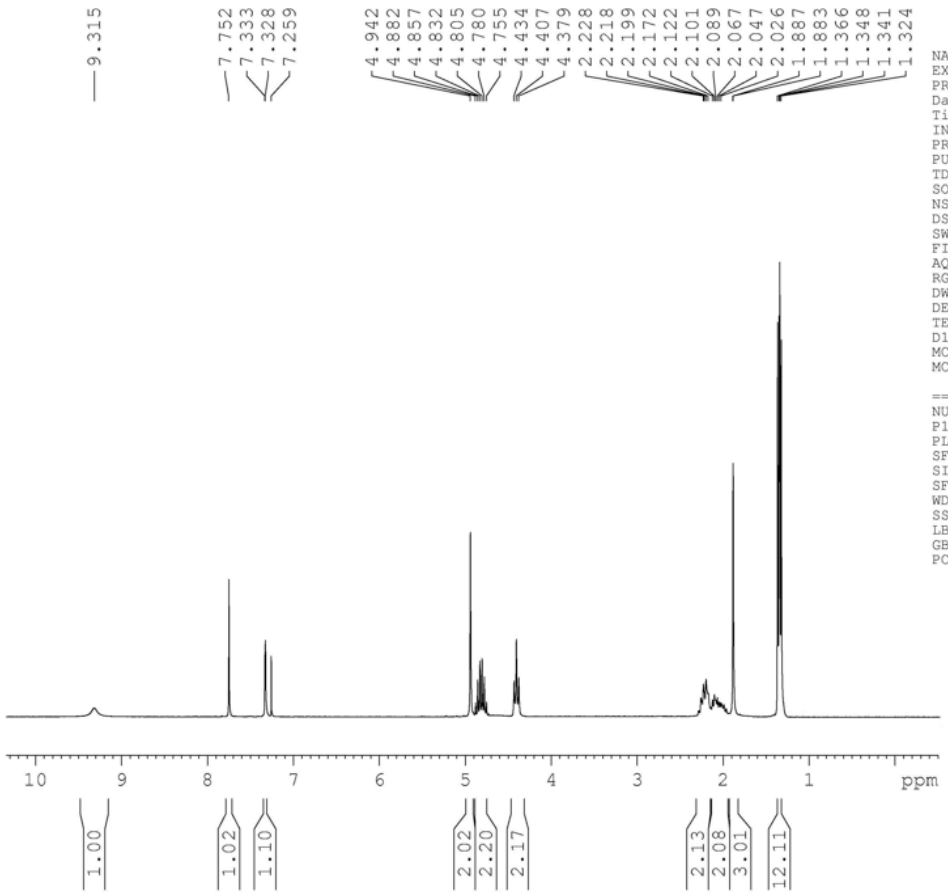





```
NAME            SD_1048c
EXPNO                20
PROCNO                1
Date_          20090418
Time              11.35
INSTRUM           spect
PROBHD   5 mm QNP 1H/13
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                 10240
DS                     2
SWH            26041.666 Hz
FIDRES          0.397364 Hz
AQ             1.2583412 se
RG                  2050
DW               19.200 us
DE                8.00 us
TE                295.0 K
D1           1.50000000 se
D11          0.03000000 se
TD0                    1

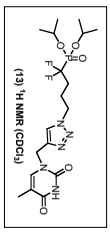
======== CHANNEL f1 ======
NUC1                 13C
P1                8.30 us
PL1               -2.00 dB
PL1W        58.36251068 W
SFO1       100.6238364 MH

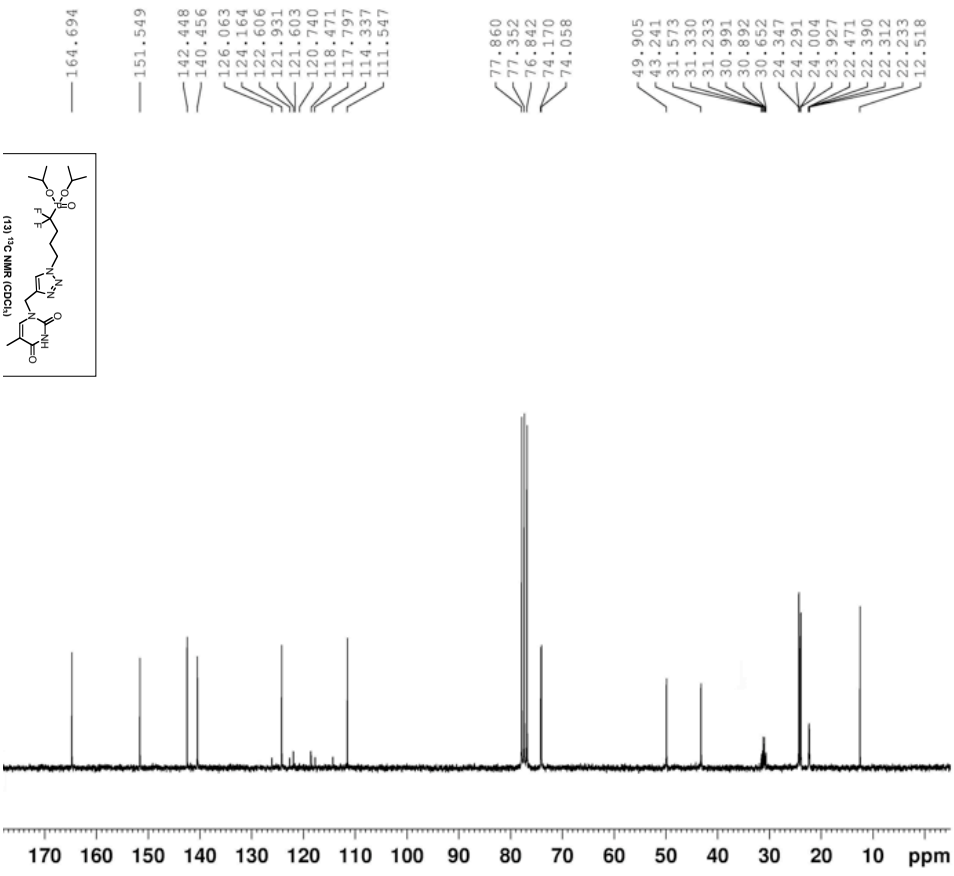
======== CHANNEL f2 ======
CPDPRG2          waltz16
NUC2                  1H
PCPD2            100.00 us
PL2               -1.00 dB
PL12              17.42 dB
PL13              20.00 dB
PL2W        15.25798988 W
PL12W        0.21953173 W
PL13W        0.12119852 W
SFO2       400.1316005 MH
SI                131072
SF         100.6127408 MH
WDW                   EM
SSB                    0
LB                1.00 Hz
GB                     0
PC                1.40
```




```
NAME                ah63
EXPNO                 10
PROCNO                 1
Date_           20070313
Time                11.14
INSTRUM             spect
PROBHD   5 mm QNP 1H/13
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    16
DS                     0
SWH             4990.020 Hz
FIDRES          0.152283 Hz
AQ            3.2834036 sec
RG                203.2
DW              100.200 usec
DE               10.00 usec
TE               683.2 K
D1           2.00000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                 7.50 usec
PL1               -6.00 dB
SFO1       250.1318626 MHz
SI                 16384
SF         250.1300003 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                0.50
```

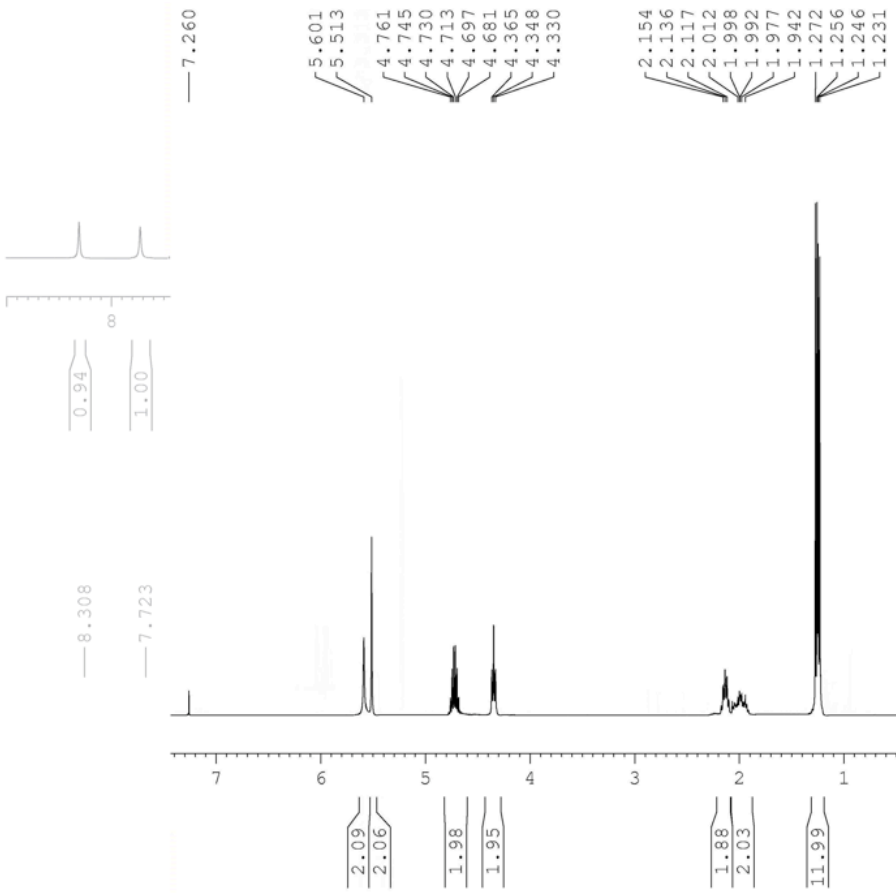





```
NAME                ah63
EXPNO                  1
PROCNO                 1
Date_           20070315
Time                14.14
INSTRUM             spect
PROBHD   5 mm QNP 1H/13
PULPROG          zgdc30
TD                 65536
SOLVENT            CDCl3
NS                  1024
DS                     0
SWH            16339.869 Hz
FIDRES          0.249327 Hz
AQ            2.0054517 sec
RG                  2048
DW               30.600 usec
DE               10.00 usec
TE               683.2 K
D1           2.00000000 sec
d11          0.03000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

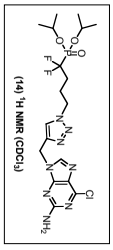
======== CHANNEL f1 ========
NUC1                 13C
P1                 5.00 usec
PL1               -6.00 dB
SFO1       62.9027614 MHz

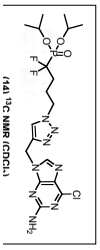
======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2            100.00 usec
PL2               -6.00 dB
PL12              18.00 dB
SFO2       250.1312506 MHz
SI                 32768
SF          62.8952207 MHz
WDW                   EM
SSB                    0
LB                1.00 Hz
GB                     0
PC                0.50
```




```
NAME            SD_659c-F2
EXPNO                   10
PROCNO                   1
Date_             20080924
Time                 8.00
INSTRUM             spect
PROBHD    5 mm QNP  1H/1
PULPROG              zg30
TD                  65536
SOLVENT             CDC13
NS                     16
DS                      2
SWH              8223.685 Hz
FIDRES           0.125483 Hz
AQ               3.9846387 se
RG                   28.5
DW                 60.800 us
DE                  8.00 us
TE                 295.0 K
D1              1.00000000 se
TD0                     1

======== CHANNEL f1 ======
NUC1                   1H
P1                  11.00 us
PL1                 -1.00 dB
PL1W          15.25798988 W
SFO1         400.1324710 MH
SI                  32768
SF           400.1300100 MH
WDW                    EM
SSB                     0
LB                 0.30 Hz
GB                      0
PC                   1.00
```

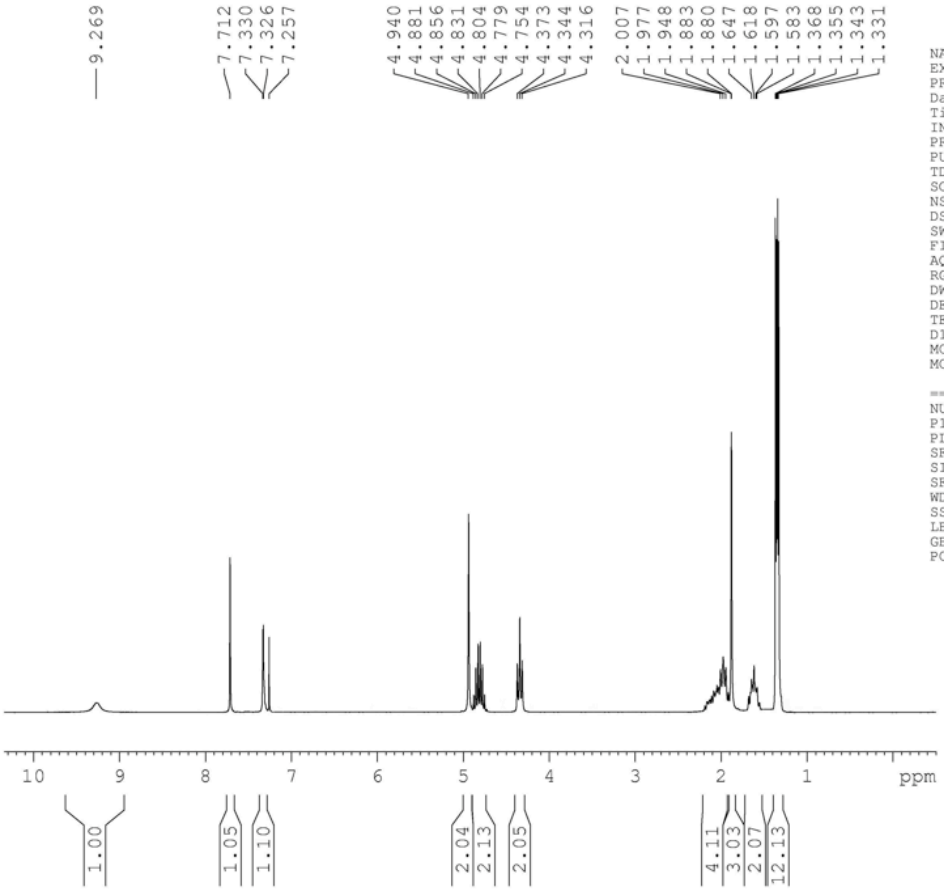





```
NAME            SD_659c-F2
EXPNO                   13
PROCNO                   1
Date_             20080924
Time                13.22
INSTRUM             spect
PROBHD    5 mm QNP  1H/1
PULPROG           zgpg30
TD                  65536
SOLVENT             CDC13
NS                   1024
DS                      2
SWH             24038.461 Hz
FIDRES           0.366798 Hz
AQ               1.3631988 sec
RG                   2050
DW                20.800 usec
DE                 8.00 usec
TE                 295.0 K
D1              1.00000000 sec
D11             0.03000000 sec
TD0                     1

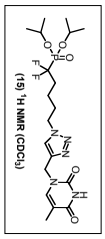
======== CHANNEL f1 ========
NUC1                  13C
P1                  8.30 usec
PL1                -2.00 dB
PL1W          58.36251068 W
SFO1         100.6228298 MHz

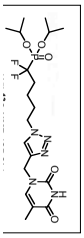
======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                   1H
PCPD2             100.00 usec
PL2                -1.00 dB
PL12               18.00 dB
PL13               20.00 dB
PL2W          15.25798988 W
PL12W          0.19208671 W
PL13W          0.12119852 W
SFO2         400.1316005 MHz
SI                  32768
SF           100.6127485 MHz
WDW                    EM
SSB                     0
LB                 1.00 Hz
GB                      0
PC                   1.40
```




```
NAME                ah62
EXPNO                 10
PROCNO                 1
Date_           20070315
Time                10.14
INSTRUM             spect
PROBHD   5 mm QNP 1H/13
PULPROG             zg30
TD                 32768
SOLVENT            CDC13
NS                    16
DS                     0
SWH           4990.020 Hz
FIDRES        0.152283 Hz
AQ            3.2834036 sec
RG                   181
DW             100.200 usec
DE              10.00 usec
TE              683.2 K
D1           2.00000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                1H
P1               7.50 usec
PL1             -6.00 dB
SFO1       250.1318626 MHz
SI                16384
SF         250.1300008 MHz
WDW                EM
SSB                 0
LB               0.30 Hz
GB                  0
PC               0.50
```

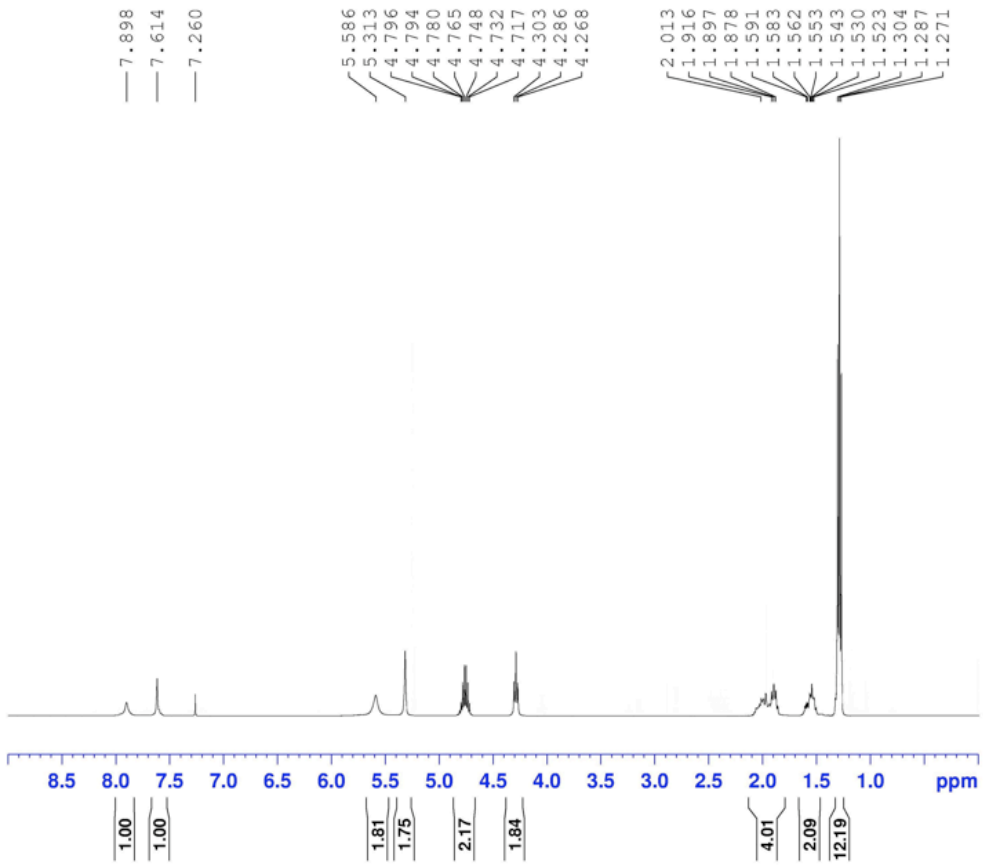





```
NAME                ah68
EXPNO                 40
PROCNO                 1
Date_           20070320
Time                13.35
INSTRUM             spect
PROBHD   5 mm QNP 1H/13
PULPROG           zgdc30
TD                 65536
SOLVENT            CDC13
NS                  1024
DS                     0
SWH          16339.869 Hz
FIDRES        0.249327 Hz
AQ            2.0054517 sec
RG                2896.3
DW              30.600 usec
DE              10.00 usec
TE              683.2 K
D1           2.00000000 sec
d11          0.03000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

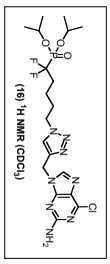
======== CHANNEL f1 ========
NUC1               13C
P1               5.00 usec
PL1             -6.00 dB
SFO1       62.9027614 MHz

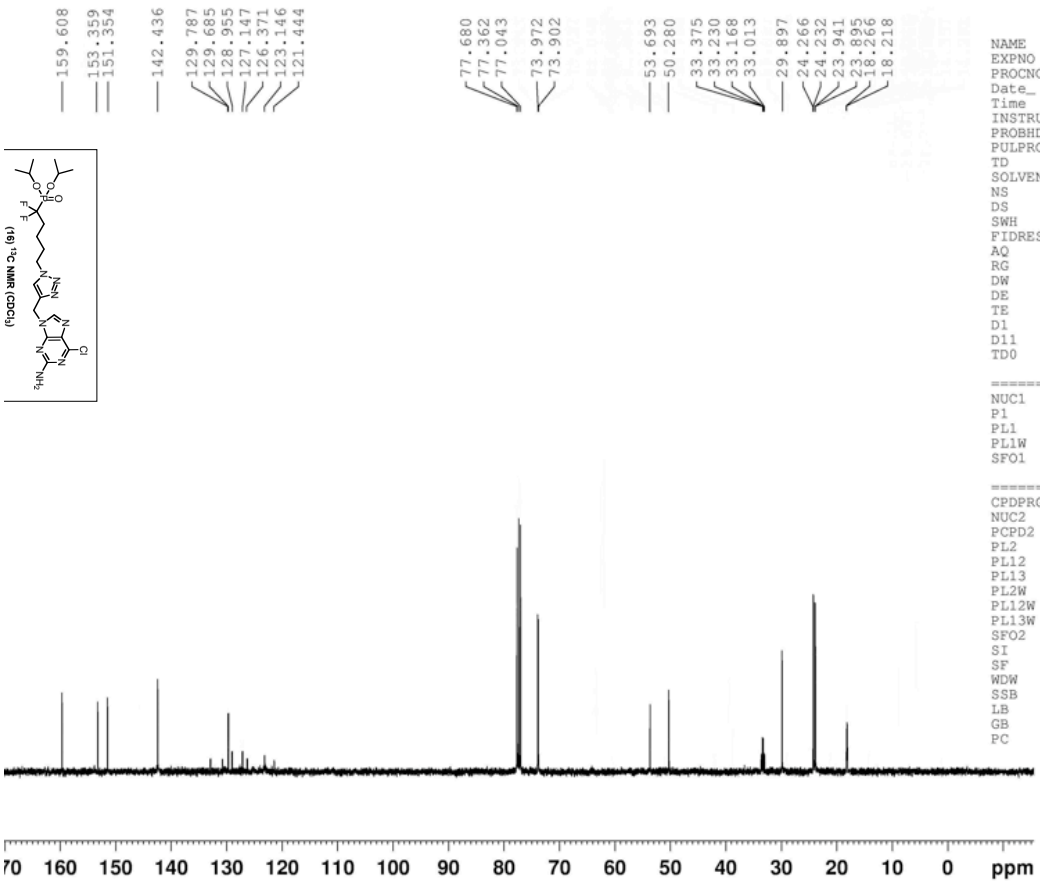
======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                1H
PCPD2           100.00 usec
PL2             -6.00 dB
PL12            18.00 dB
SFO2       250.1312506 MHz
SI                32768
SF         62.8952226 MHz
WDW                EM
SSB                 0
LB               1.00 Hz
GB                  0
PC               0.50
```




```
NAME          SD_736c-F2
EXPNO                 10
PROCNO                 1
Date_           20080929
Time                4.32
INSTRUM            spect
PROBHD   5 mm QNP  1H/1
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ              3.9846387 sec
RG                  28.5
DW                60.800 usec
DE                 8.00 usec
TE                295.0 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 11.00 usec
PL1                -1.00 dB
PL1W          15.25798988 W
SFO1         400.1324710 MHz
SI                 32768
SF           400.1300099 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

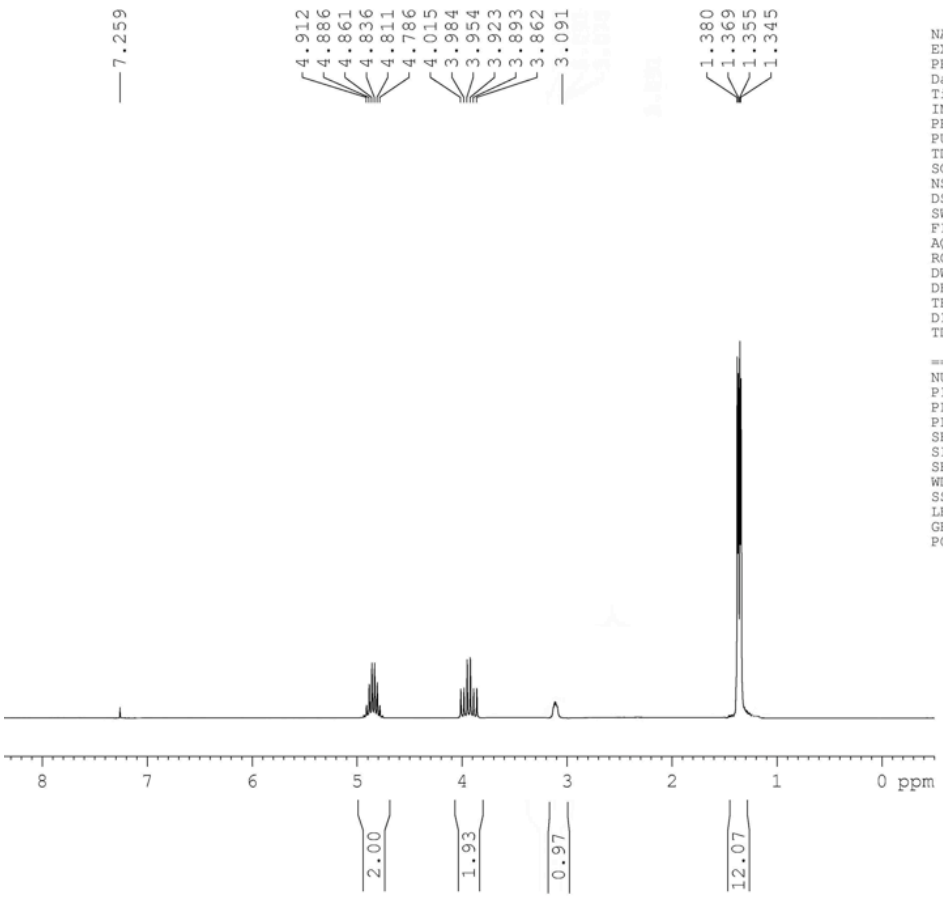





```
NAME          SD_736c-F2
EXPNO                 13
PROCNO                 1
Date_           20080929
Time                4.44
INSTRUM            spect
PROBHD   5 mm QNP  1H/1
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   128
DS                     2
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ              1.3631988 sec
RG                  2050
DW                20.800 usec
DE                 8.00 usec
TE                295.0 K
D1             1.50000000 sec
D11            0.03000000 sec
TD0                    1

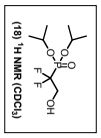
======== CHANNEL f1 ========
NUC1                 13C
P1                 8.30 usec
PL1                -2.00 dB
PL1W          58.36251068 W
SFO1         100.6228298 MHz

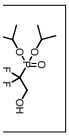
======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2            100.00 usec
PL2                -1.00 dB
PL12               18.00 dB
PL13               20.00 dB
PL2W          15.25798988 W
PL12W          0.19208671 W
PL13W          0.12119852 W
SFO2         400.1316005 MHz
SI                 32768
SF           100.6127474 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```




```
NAME            SD_1016b
EXPNO                 12
PROCNO                 1
Date_           20090323
Time                8.36
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG             zg30
TD                 65536
SOLVENT            CDC13
NS                    16
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ              3.9846387 sec
RG                   161
DW                60.800 usec
DE                 8.00 usec
TE                295.0 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 12.00 usec
PL1                -1.00 dB
PL1W         15.25798988 W
SFO1        400.1324710 MHz
SI                 32768
SF          400.1300096 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

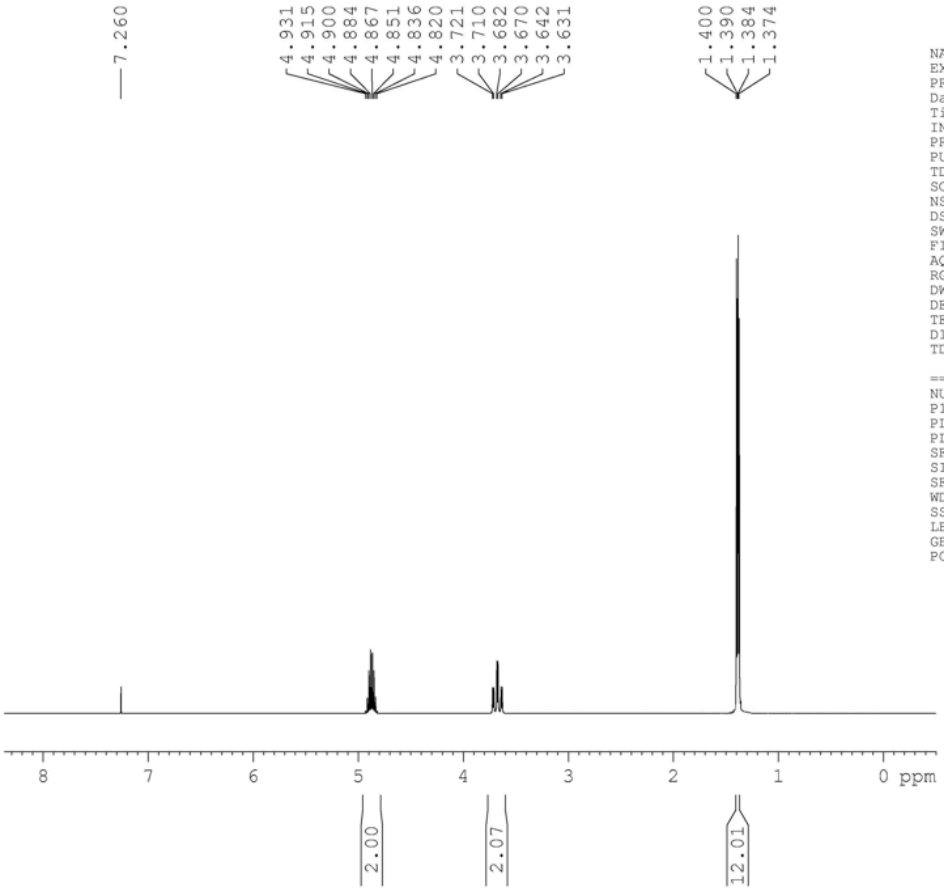





```
NAME            SD_1016
EXPNO                 10
PROCNO                 1
Date_           20090324
Time               23.42
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                 65536
SOLVENT            CDC13
NS                  1024
DS                     2
SWH            31250.000 Hz
FIDRES          0.476837 Hz
AQ              1.0486259 sec
RG                   128
DW                16.000 usec
DE                 8.00 usec
TE                295.0 K
D1             1.00000000 sec
D11            0.03000000 sec
TD0                    1

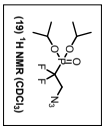
======== CHANNEL f1 ========
NUC1                 13C
P1                 10.50 usec
PL1                 1.50 dB
PL1W         64.32036591 W
SFO1        125.7703648 MHz

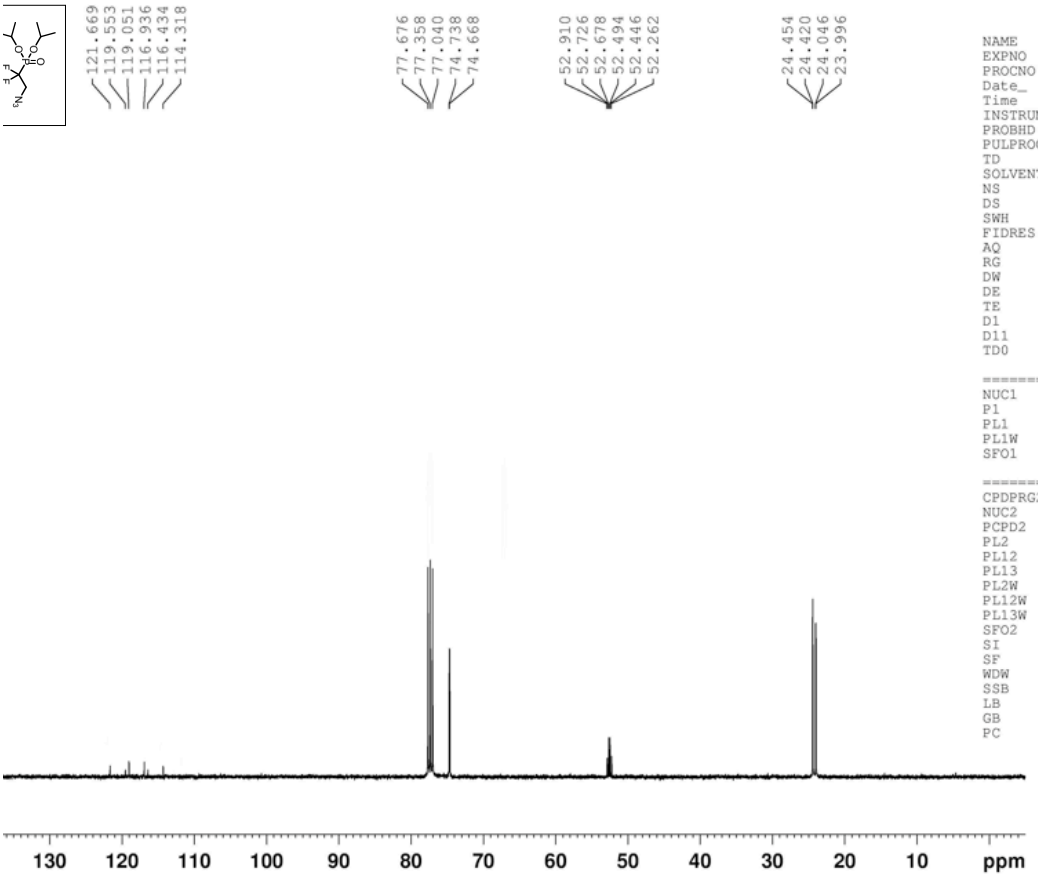
======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                 1.90 dB
PL12               16.73 dB
PL13               17.00 dB
PL2W         16.07213783 W
PL12W         0.52853495 W
PL13W         0.49667653 W
SFO2        500.1316005 MHz
SI                131072
SF          125.7577476 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```




```
NAME            SD_1020b
EXPNO                 12
PROCNO                 1
Date_           20090325
Time                4.21
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG             zg30
TD                 65536
SOLVENT            CDC13
NS                    16
DS                     2
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ              3.9846387 sec
RG                   161
DW                60.800 usec
DE                 8.00 usec
TE                295.0 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 12.00 usec
PL1                -1.00 dB
PL1W         15.25798988 W
SFO1         400.1324710 MHz
SI                 32768
SF           400.1300096 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

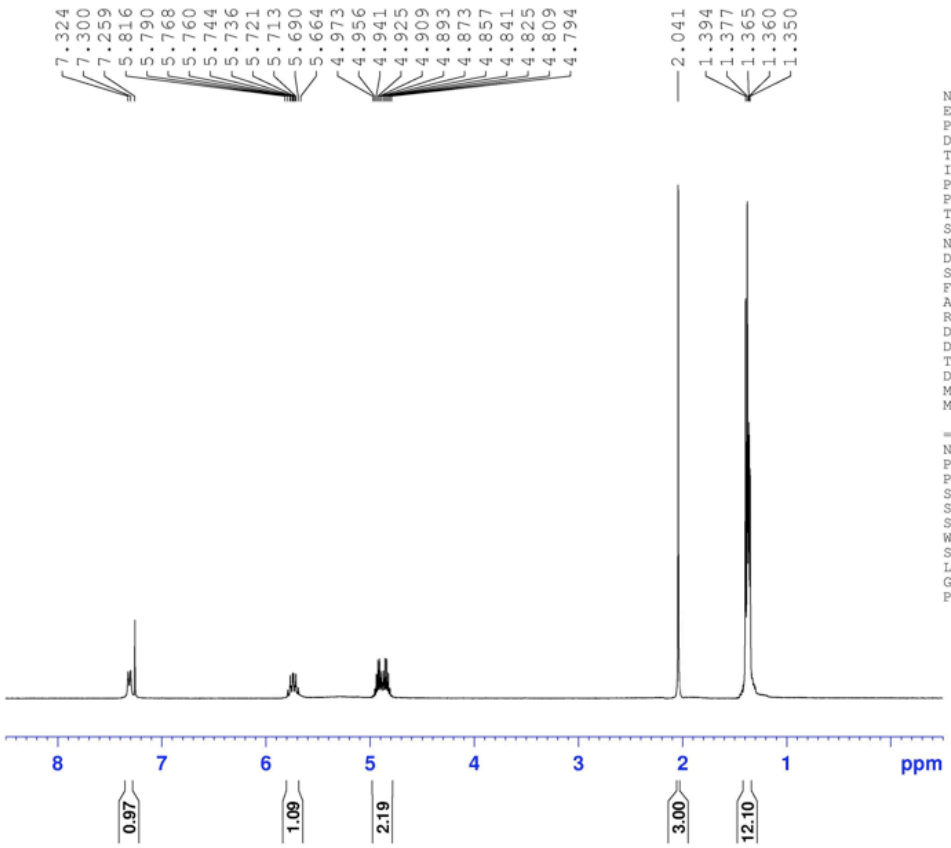





```
NAME            SD_1020b
EXPNO                 20
PROCNO                 1
Date_           20090325
Time               17.07
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG          zgpg30
TD                 65536
SOLVENT            CDC13
NS                  1024
DS                     2
SWH            26041.666 Hz
FIDRES          0.397364 Hz
AQ              1.2583412 sec
RG                  2050
DW                19.200 usec
DE                 8.00 usec
TE                295.0 K
D1           1.50000000 sec
D11          0.03000000 sec
TD0                    1

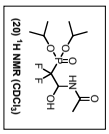
======== CHANNEL f1 ========
NUC1                 13C
P1                  8.30 usec
PL1                -2.00 dB
PL1W         58.36251068 W
SFO1         100.6238364 MHz

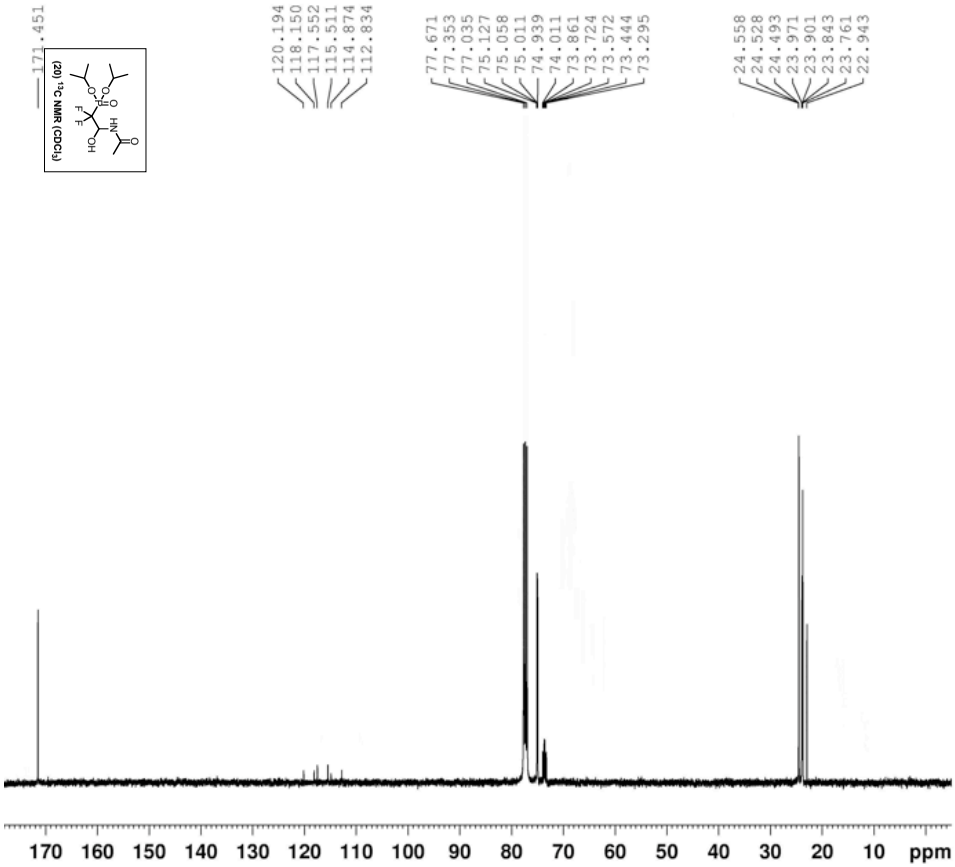
======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             100.00 usec
PL2                -1.00 dB
PL12               17.42 dB
PL13               20.00 dB
PL2W         15.25798988 W
PL12W         0.21953173 W
PL13W         0.12119852 W
SFO2         400.1316005 MHz
SI                131072
SF           100.6127359 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```




```
NAME            fles07c13
EXPNO                  10
PROCNO                  1
Date_            20080304
Time                6.13
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8223.685 Hz
FIDRES          0.250967 Hz
AQ              1.9923444 sec
RG                 912.3
DW                60.800 usec
DE                10.00 usec
TE                299.2 K
D1              1.00000000 sec
MCREST          0.00000000 sec
MCWRK           0.01500000 sec

======== CHANNEL f1 ========
NUC1                   1H
P1                   6.00 usec
PL1                 -2.00 dB
SFO1         400.1324710 MHz
SI                 65536
SF           400.1300094 MHz
WDW                   no
SSB                    0
LB                  0.00 Hz
GB                     0
PC                  1.00
```

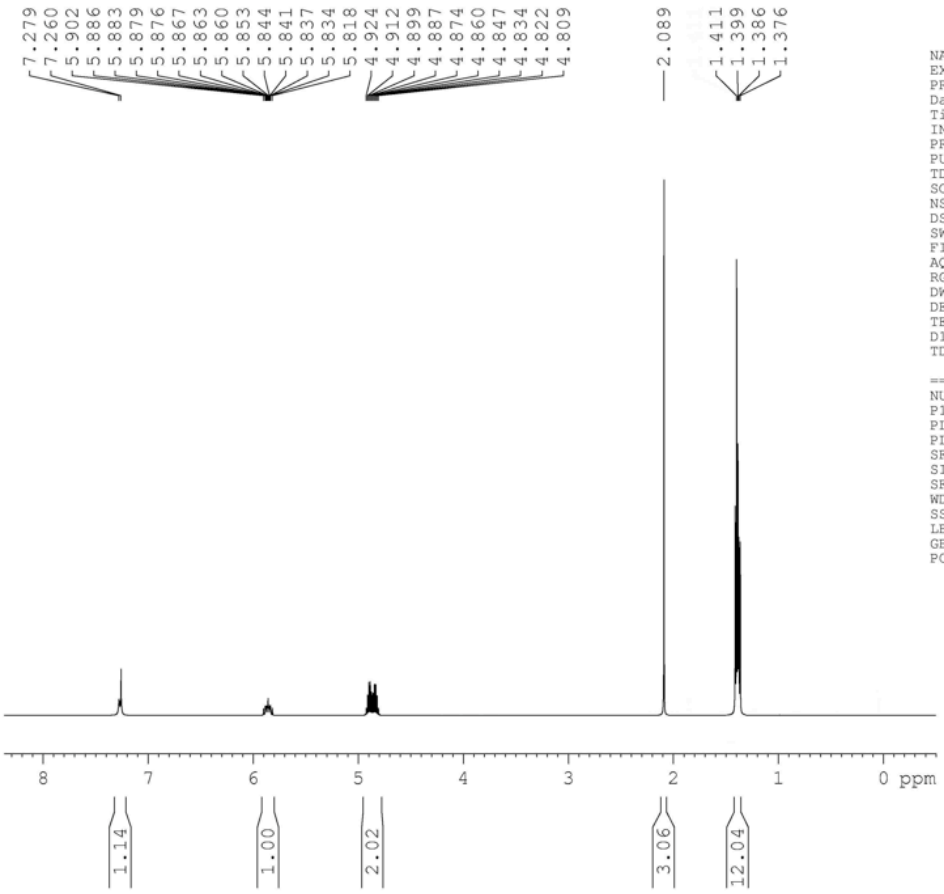





```
NAME            fles07c13
EXPNO                  14
PROCNO                  1
Date_            20080304
Time               22.00
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG          zgdc30
TD                 65536
SOLVENT            CDCl3
NS                 10240
DS                     2
SWH            25125.629 Hz
FIDRES          0.383387 Hz
AQ              1.3042164 sec
RG                 20642.5
DW                19.900 usec
DE                10.00 usec
TE                299.2 K
D1              3.00000000 sec
d11             0.03000000 sec
MCREST          0.00000000 sec
MCWRK           0.01500000 sec

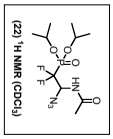
======== CHANNEL f1 ========
NUC1                  13C
P1                   7.70 usec
PL1                 -3.00 dB
SFO1         100.6237964 MHz

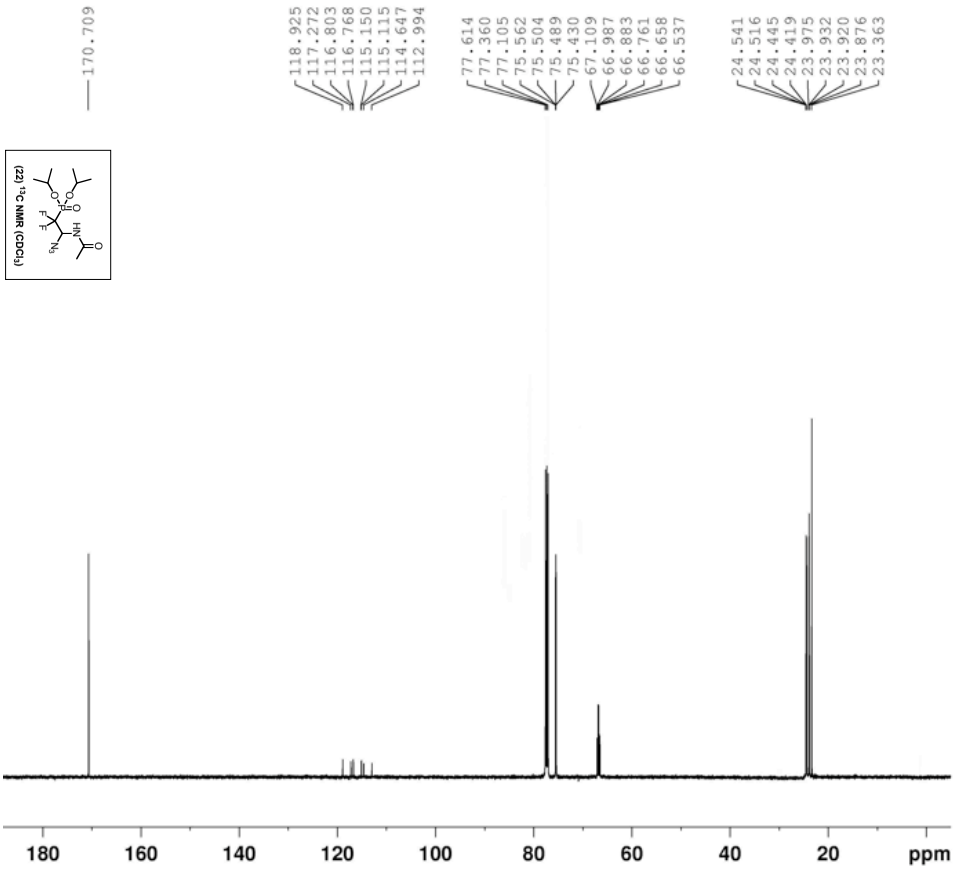
======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                   1H
PCPD2              100.00 usec
PL2                120.00 dB
PL12                16.79 dB
SFO2         400.1316005 MHz
SI                 32768
SF           100.6127363 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```




```
NAME            cyl415f1
EXPNO                 10
PROCNO                 1
Date_           20090416
Time               18.49
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT            CDC13
NS                    16
DS                     2
SWH            10330.578 Hz
FIDRES          0.157632 Hz
AQ              3.1719923 sec
RG                   114
DW                48.400 usec
DE                 6.50 usec
TE                295.0 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 14.70 usec
PL1                 1.00 dB
PL1W         19.77305031 W
SFO1        500.1330885 MHz
SI                 32768
SF          500.1300135 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

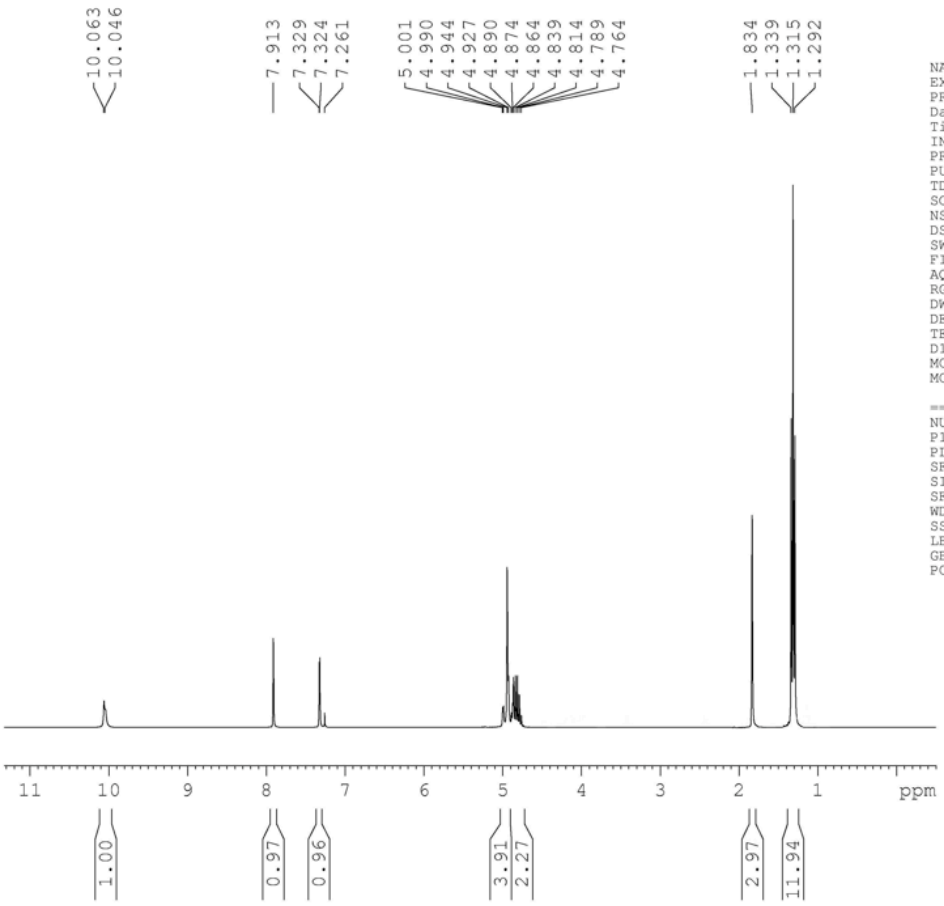





```
NAME            cyl415f1
EXPNO                 13
PROCNO                 1
Date_           20090416
Time               23.39
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT            CDC13
NS                  4096
DS                     2
SWH            31250.000 Hz
FIDRES          0.476837 Hz
AQ              1.0486259 sec
RG                   128
DW                16.000 usec
DE                 8.00 usec
TE                295.0 K
D1           3.00000000 sec
D11          0.03000000 sec
TD0                    1

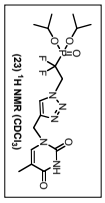
======== CHANNEL f1 ========
NUC1                 13C
P1                 10.50 usec
PL1                 1.50 dB
PL1W         64.32036591 W
SFO1        125.7703648 MHz

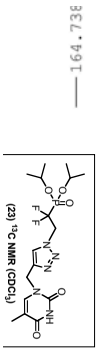
======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             100.00 usec
PL2                 1.00 dB
PL12               18.00 dB
PL13               20.00 dB
PL2W         19.77305031 W
PL12W         0.39452422 W
PL13W         0.24892795 W
SFO2        500.1316005 MHz
SI                131072
SF          125.7577501 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```




```
NAME            ah76_2md-1
EXPNO                   10
PROCNO                   1
Date_             20070403
Time                 12.58
INSTRUM              spect
PROBHD    5 mm QNP 1H/13
PULPROG              zg30
TD                  32768
SOLVENT             CDC13
NS                     16
DS                      0
SWH              4990.020 Hz
FIDRES           0.152283 Hz
AQ               3.2834036 sec
RG                   90.5
DW                100.200 usec
DE                 10.00 usec
TE                 683.2 K
D1            2.00000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1                   1H
P1                   7.50 usec
PL1                 -6.00 dB
SFO1         250.1318626 MHz
SI                  16384
SF           250.1299994 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  0.50
```

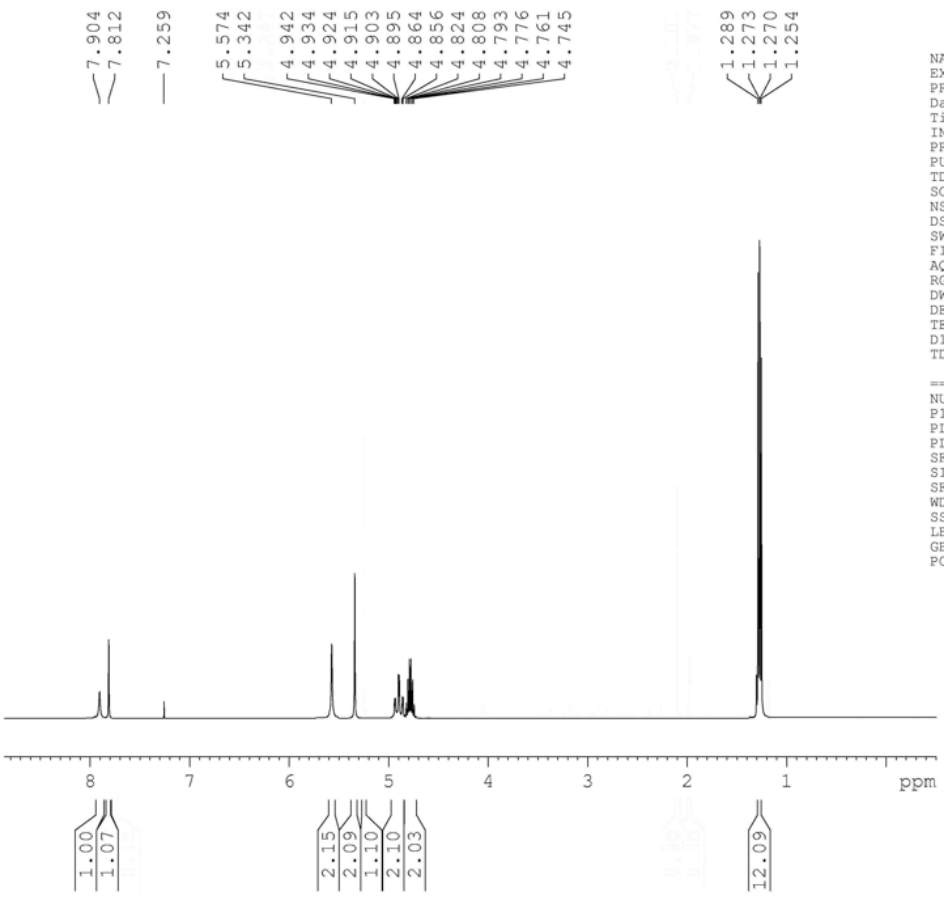





```
NAME            ah76_2nd-1
EXPNO                   10
PROCNO                   1
Date_             20070404
Time                 19.54
INSTRUM              spect
PROBHD    5 mm QNP 1H/13
PULPROG           zgdc30
TD                  65536
SOLVENT             CDC13
NS                   1024
DS                      0
SWH             16339.869 Hz
FIDRES           0.249327 Hz
AQ               2.0054517 sec
RG                 4597.6
DW                 30.600 usec
DE                 10.00 usec
TE                 683.2 K
D1            2.00000000 sec
d11           0.03000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

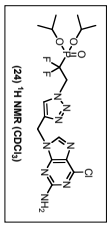
======== CHANNEL f1 ========
NUC1                  13C
P1                   5.00 usec
PL1                 -6.00 dB
SFO1         62.9027614 MHz

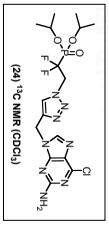
======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                   1H
PCPD2              100.00 usec
PL2                 -6.00 dB
PL12                18.00 dB
SFO2         250.1312506 MHz
SI                  32768
SF           62.8952227 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```




```
NAME          SD_1022c-F2
EXPNO                  11
PROCNO                  1
Date_            20090326
Time                18.12
INSTRUM             spect
PROBHD   5 mm QNP 1H/13
PULPROG              zg30
TD                  65536
SOLVENT             CDC13
NS                     16
DS                      2
SWH              8223.685 Hz
FIDRES           0.125483 Hz
AQ               3.9846387 sec
RG                     57
DW                 60.800 usec
DE                  8.00 usec
TE                 295.0 K
D1            1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                  12.00 usec
PL1                 -1.00 dB
PL1W         15.25798988 W
SFO1         400.1324710 MHz
SI                  32768
SF           400.1300101 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
```

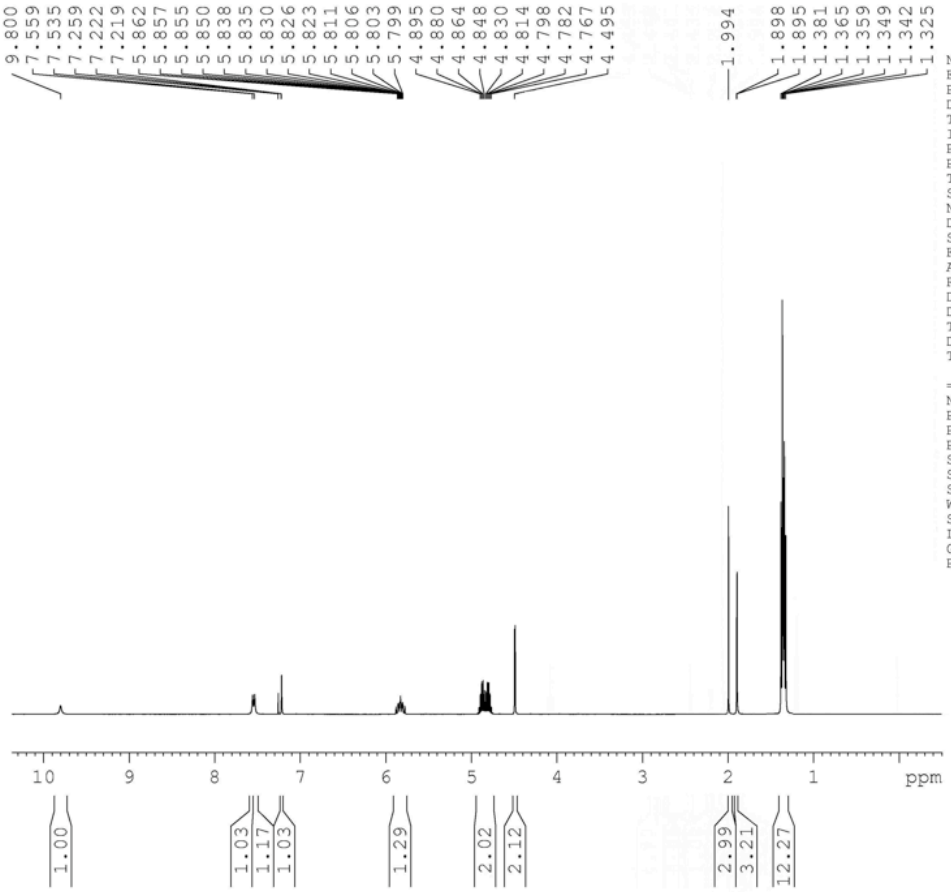





```
NAME          SD_1022c-F2
EXPNO                  10
PROCNO                  1
Date_            20090326
Time                18.09
INSTRUM             spect
PROBHD   5 mm QNP 1H/13
PULPROG           zgpg30
TD                  65536
SOLVENT             CDC13
NS                   2048
DS                      2
SWH             26041.666 Hz
FIDRES           0.397364 Hz
AQ               1.2583412 sec
RG                   2050
DW                 19.200 usec
DE                  8.00 usec
TE                 295.0 K
D1            1.50000000 sec
D11           0.03000000 sec
TD0                     1

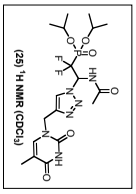
======== CHANNEL f1 ========
NUC1                  13C
P1                   8.30 usec
PL1                 -2.00 dB
PL1W         58.36251068 W
SFO1         100.6238364 MHz

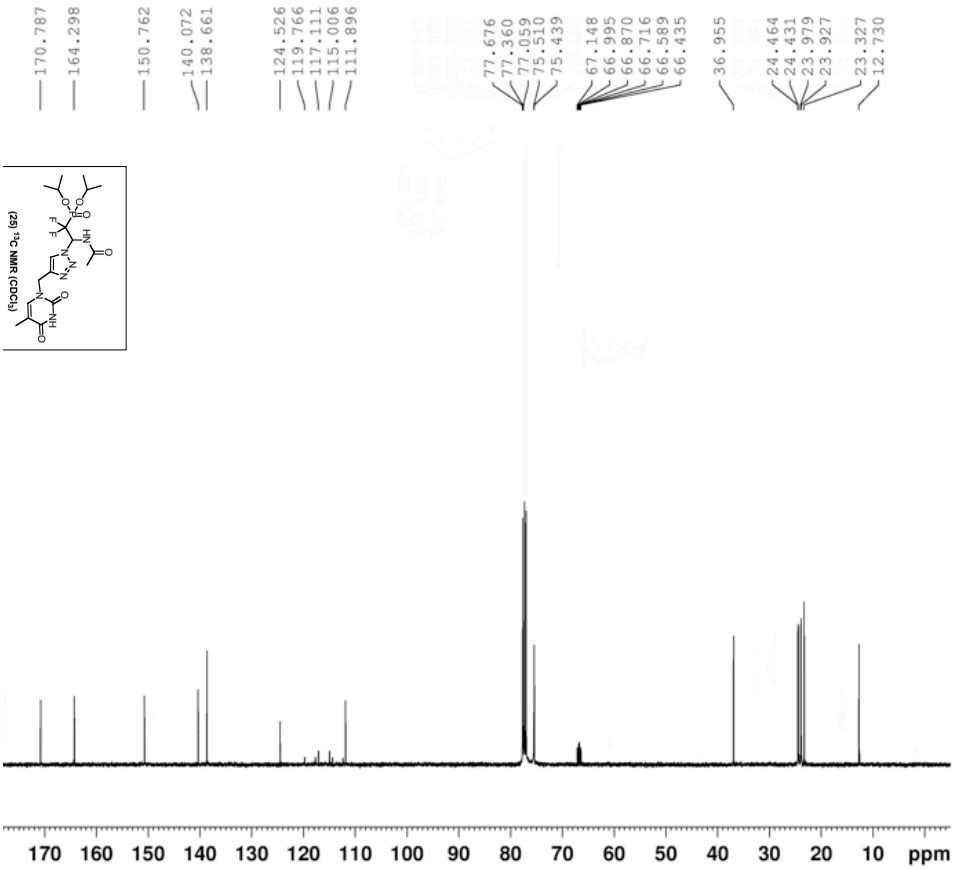
======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                   1H
PCPD2              100.00 usec
PL2                 -1.00 dB
PL12                17.42 dB
PL13                20.00 dB
PL2W         15.25798988 W
PL12W         0.21953173 W
PL13W         0.12119852 W
SFO2         400.1316005 MHz
SI                 131072
SF           100.6127454 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.40
```




```
NAME          SD_CYL366c-F1
EXPNO                    10
PROCNO                    1
Date_              20090422
Time                  12.23
INSTRUM               spect
PROBHD   5 mm QNP 1H/13
PULPROG                zg30
TD                    65536
SOLVENT               CDC13
NS                       16
DS                        2
SWH                8223.685 Hz
FIDRES            0.125483 Hz
AQ                3.9846387 sec
RG                      161
DW                   60.800 usec
DE                    8.00 usec
TE                   294.9 K
D1                1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                    1H
P1                   12.00 usec
PL1                  -1.00 dB
PL1W            15.25798988 W
SFO1           400.1324710 MHz
SI                    32768
SF             400.1300100 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```

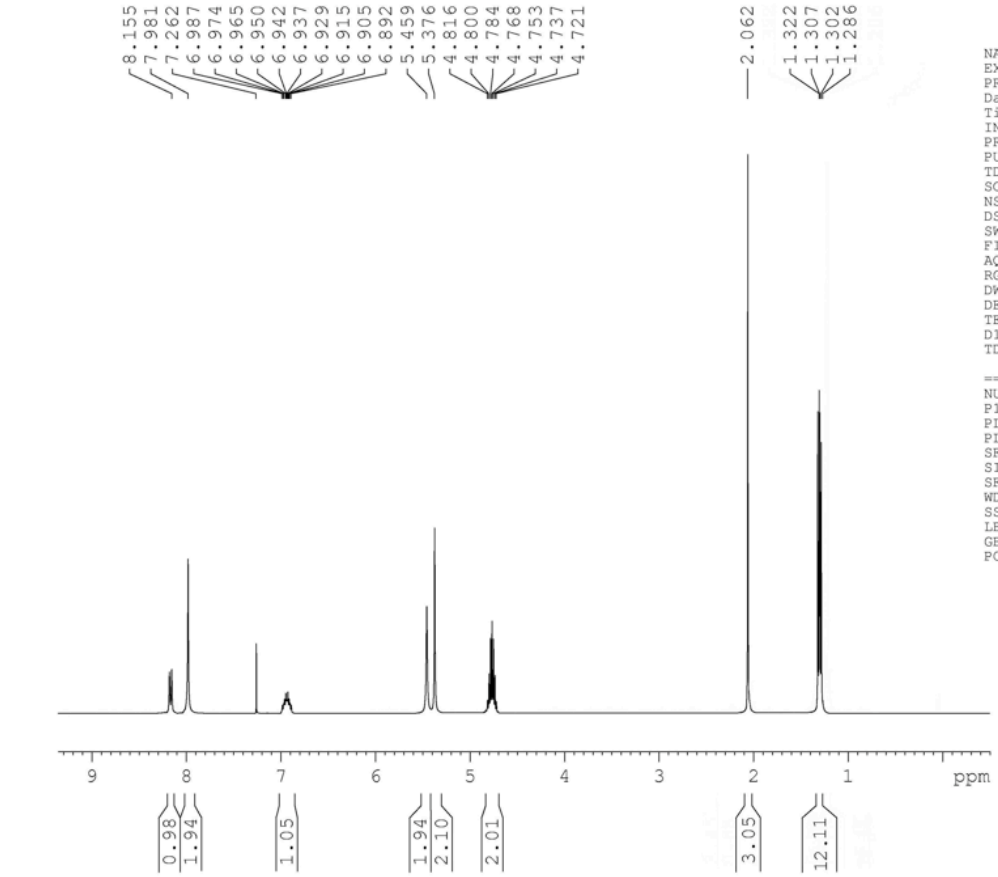





```
NAME          SD_CYL366c-F1
EXPNO                    13
PROCNO                    1
Date_              20090422
Time                  15.41
INSTRUM               spect
PROBHD   5 mm QNP 1H/13
PULPROG             zgpg30
TD                    65536
SOLVENT               CDC13
NS                     4096
DS                        2
SWH               26041.666 Hz
FIDRES            0.397364 Hz
AQ                1.2583412 sec
RG                     2050
DW                   19.200 usec
DE                    8.00 usec
TE                   295.0 K
D1                1.50000000 sec
D11               0.03000000 sec
TD0                       1

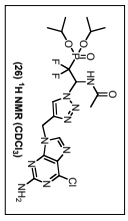
======== CHANNEL f1 ========
NUC1                   13C
P1                    8.30 usec
PL1                  -2.00 dB
PL1W            58.36251068 W
SFO1           100.6238364 MHz

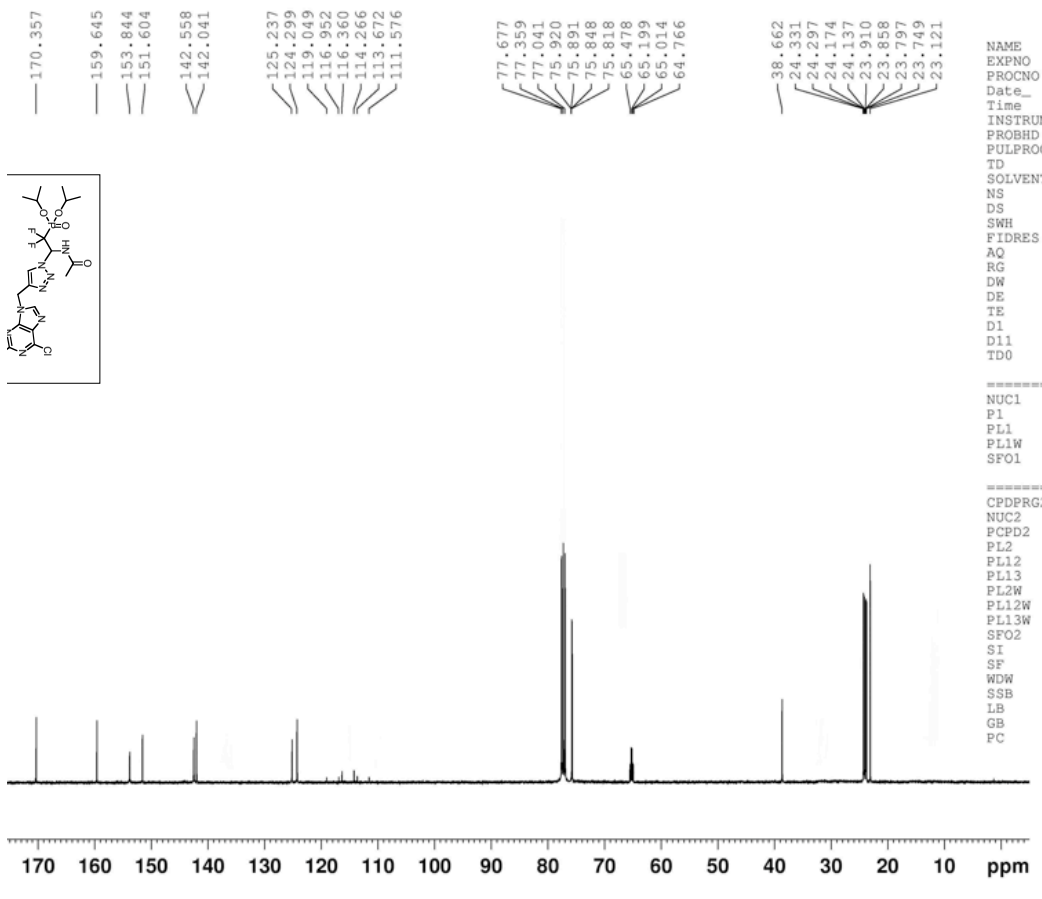
======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                    1H
PCPD2                100.00 usec
PL2                  -1.00 dB
PL12                 17.42 dB
PL13                 20.00 dB
PL2W            15.25798988 W
PL12W            0.21953173 W
PL13W            0.12119852 W
SFO2           400.1316005 MHz
SI                   131072
SF             100.6127368 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
```




```
NAME          SD_1028c-F2
EXPNO                  22
PROCNO                  1
Date_            20090418
Time                18.03
INSTRUM             spect
PROBHD   5 mm QNP 1H/13
PULPROG              zg30
TD                  65536
SOLVENT             CDC13
NS                     64
DS                      2
SWH              8223.685 Hz
FIDRES           0.125483 Hz
AQ               3.9846387 sec
RG                    161
DW                 60.800 usec
DE                  8.00 usec
TE                 295.0 K
D1            1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                  12.00 usec
PL1                 -1.00 dB
PL1W         15.25798988 W
SFO1         400.1324710 MHz
SI                  32768
SF           400.1300089 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

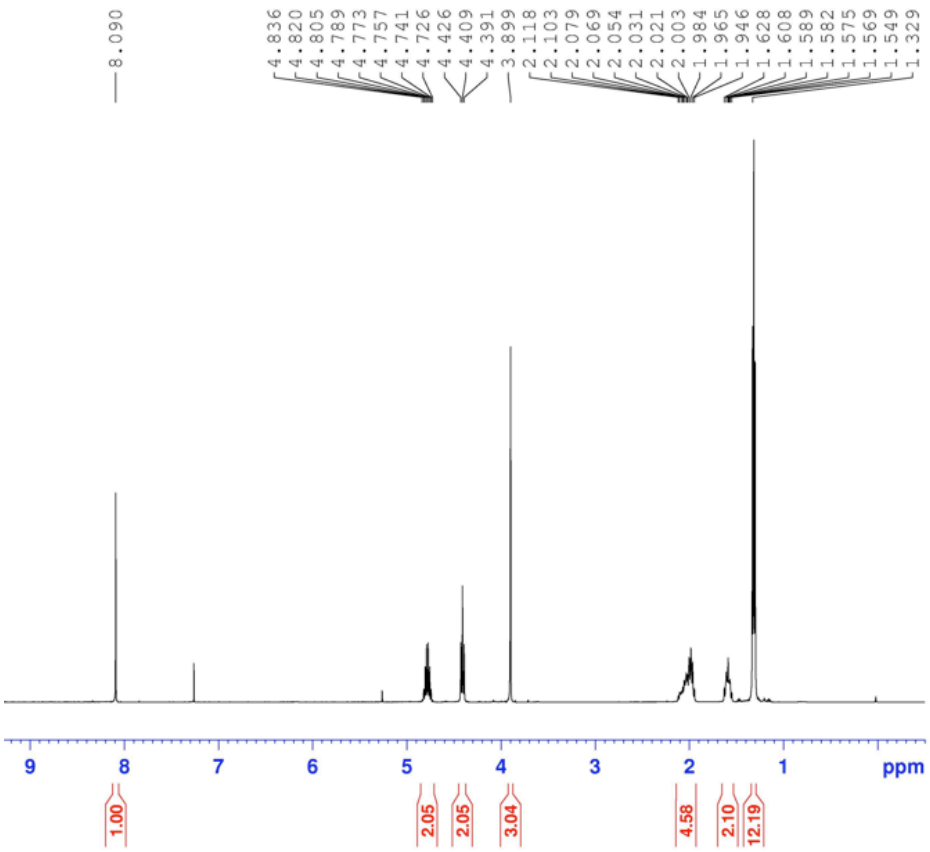





```
NAME          SD_1028c-F2
EXPNO                  10
PROCNO                  1
Date_            20090416
Time                20.02
INSTRUM             spect
PROBHD   5 mm QNP 1H/13
PULPROG            zgpg30
TD                  65536
SOLVENT             CDC13
NS                   6144
DS                      2
SWH             26041.666 Hz
FIDRES           0.397364 Hz
AQ               1.2583412 sec
RG                   2050
DW                 19.200 usec
DE                  8.00 usec
TE                 295.0 K
D1            1.50000000 sec
D11           0.03000000 sec
TD0                     1

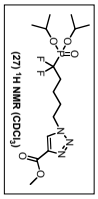
======== CHANNEL f1 ========
NUC1                  13C
P1                  8.30 usec
PL1                 -2.00 dB
PL1W         58.36251068 W
SFO1         100.6238364 MHz

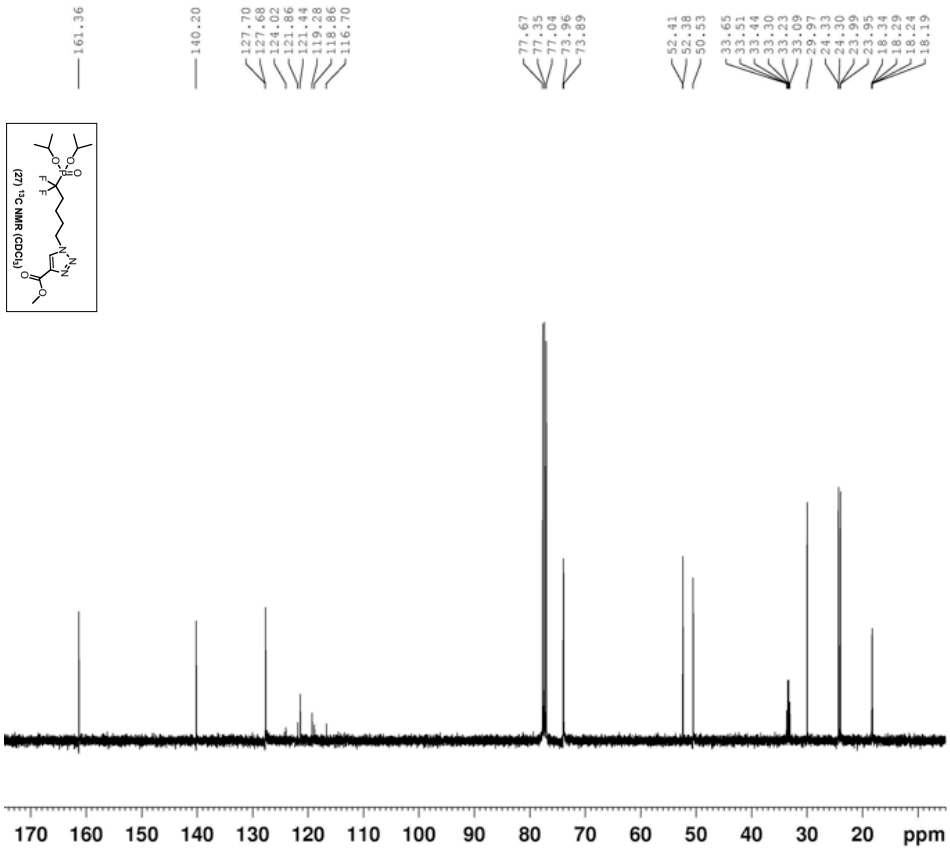
======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                   1H
PCPD2             100.00 usec
PL2                 -1.00 dB
PL12                17.42 dB
PL13                20.00 dB
PL2W         15.25798988 W
PL12W         0.21953173 W
PL13W         0.12119852 W
SFO2         400.1316005 MHz
SI                 131072
SF           100.6127380 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```




```
NAME            sg122bis
EXPNO                 10
PROCNO                 1
Date_           20060614
Time                9.30
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG             zg30
TD                 32768
SOLVENT            CDC13
NS                    16
DS                     2
SWH             8223.685 Hz
FIDRES          0.250967 Hz
AQ              1.9923444 sec
RG                 724.1
DW                60.800 usec
DE                10.00 usec
TE                296.2 K
D1             1.00000000 sec
MCREST         0.00000000 sec
MCWRK          0.01500000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                10.75 usec
PL1               -2.00 dB
SFO1        400.1324710 MHz
SI                 16384
SF          400.1300094 MHz
WDW                   no
SSB                    0
LB                0.00 Hz
GB                     0
PC                1.00
```

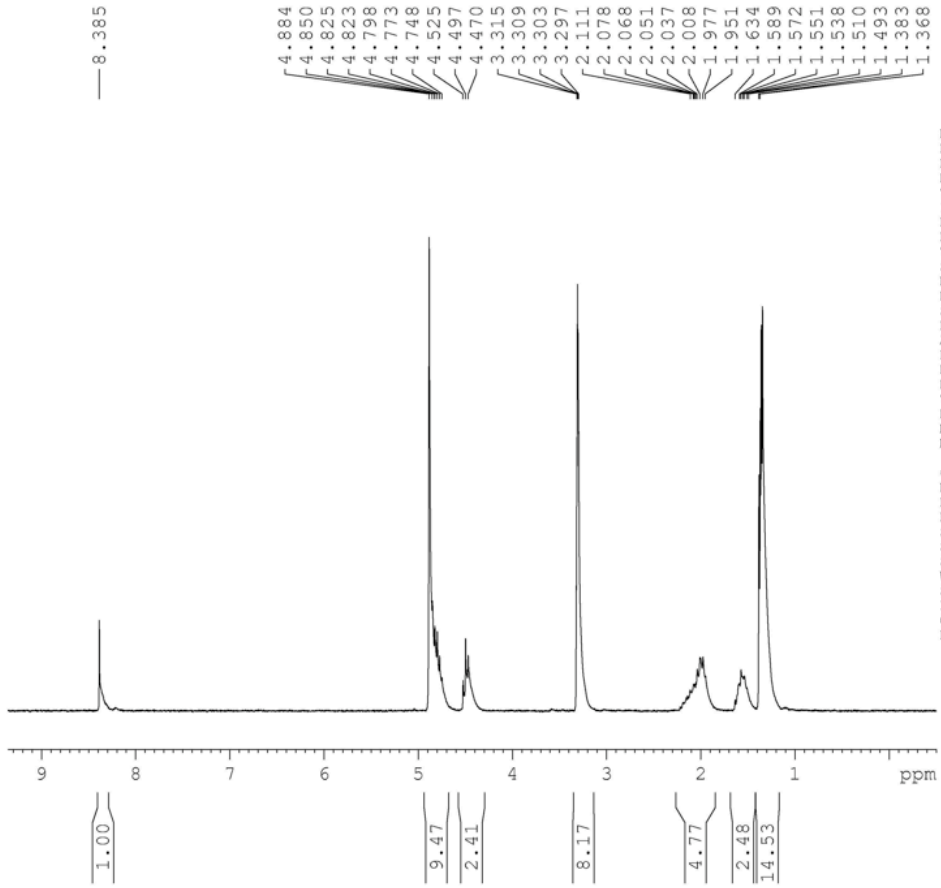





```
NAME            sg122bis
EXPNO                 11
PROCNO                 1
Date_           20060614
Time                9.38
INSTRUM            spect
PROBHD   5 mm QNP 1H/13
PULPROG           zgdc30
TD                 65536
SOLVENT            CDC13
NS                  3072
DS                     2
SWH            25125.629 Hz
FIDRES          0.383387 Hz
AQ              1.3042164 sec
RG                 18390.4
DW                19.900 usec
DE                10.00 usec
TE                296.2 K
D1             2.00000000 sec
d11            0.03000000 sec
MCREST         0.00000000 sec
MCWRK          0.01500000 sec

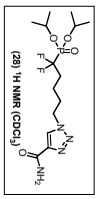
======== CHANNEL f1 ========
NUC1                 13C
P1                13.75 usec
PL1               -2.00 dB
SFO1        100.6248025 MHz

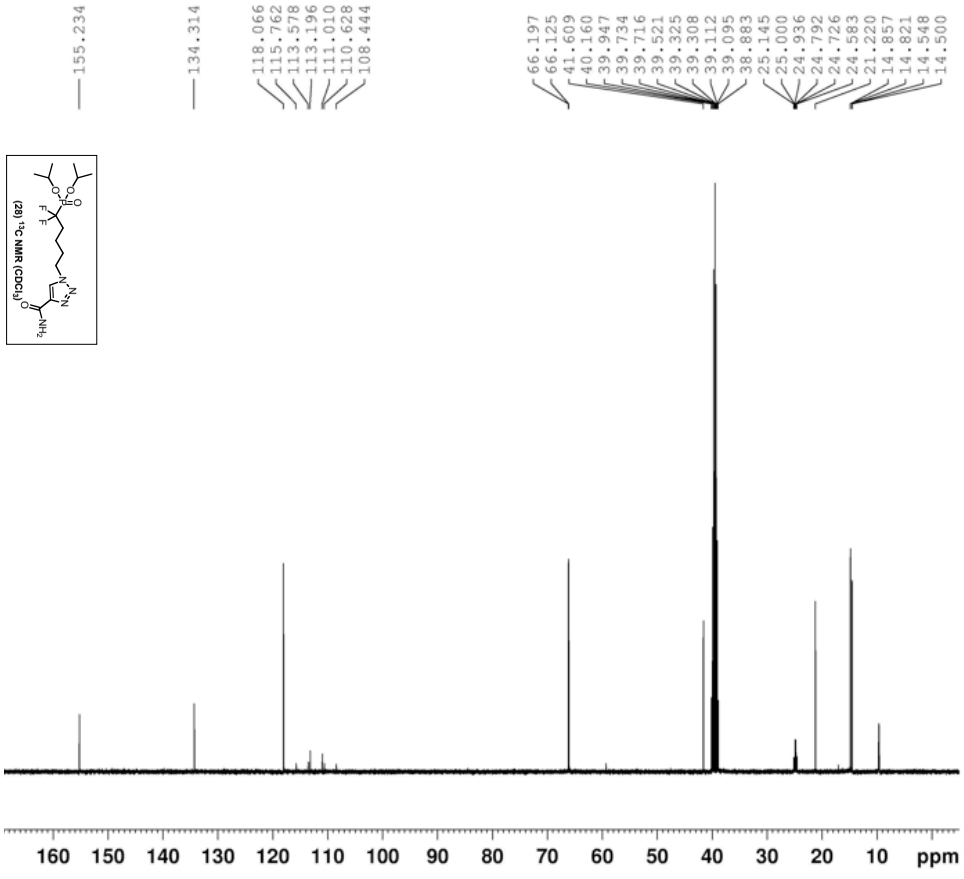
======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             100.00 usec
PL2               120.00 dB
PL12               17.37 dB
SFO2        400.1316005 MHz
SI                 32768
SF          100.6127425 MHz
WDW                   no
SSB                    0
LB                0.00 Hz
GB                     0
PC                1.40
```




```
NAME            sgbr125
EXPNO                10
PROCNO                1
Date_          20060620
Time               1.49
INSTRUM           spect
PROBHD   5 mm QNP 1H/13
PULPROG            zg30
TD                32768
SOLVENT            MeOH
NS                   16
DS                    0
SWH            4990.020 Hz
FIDRES        0.152283 Hz
AQ            3.2834036 sec
RG                724.1
DW              100.200 usec
DE               10.00 usec
TE               683.2 K
D1            2.00000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1                 1H
P1                 7.50 usec
PL1               -6.00 dB
SFO1       250.1318626 MHz
SI                16384
SF         250.1299998 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 0.50
```



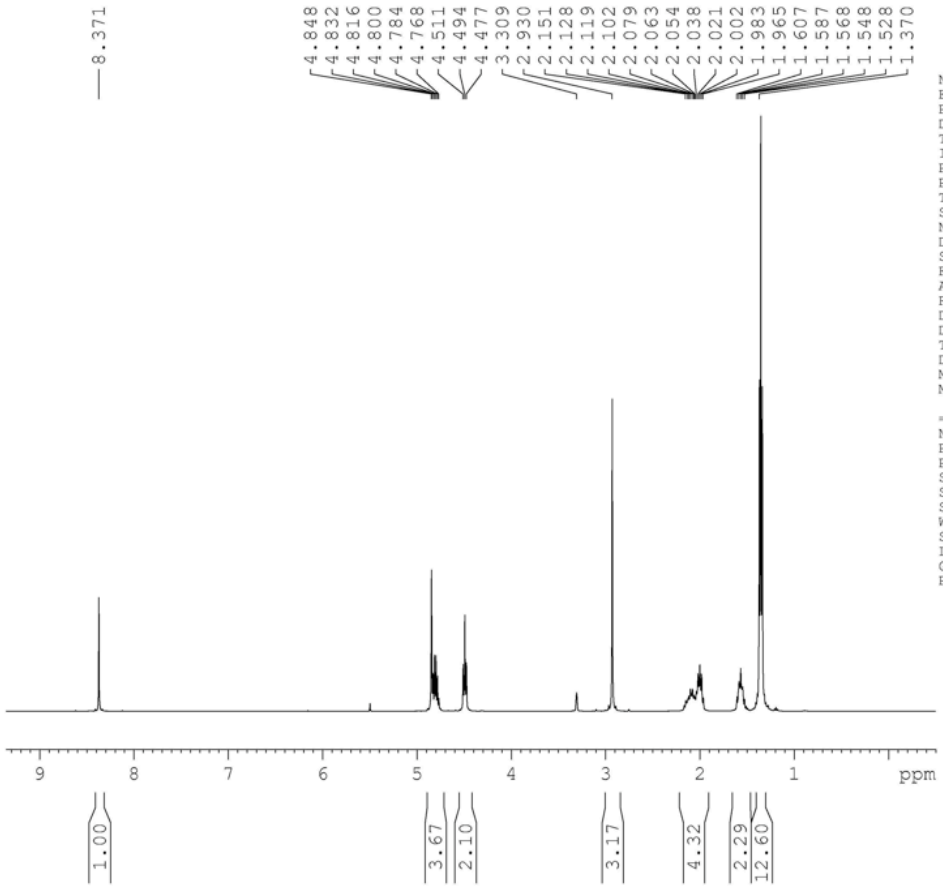



```
NAME            sg125bis
EXPNO                20
PROCNO                1
Date_          20060629
Time               0.28
INSTRUM           spect
PROBHD   5 mm QNP 1H/13
PULPROG        zgfbig.chb
TD                65536
SOLVENT            MeOD
NS                  750
DS                    0
SWH           25125.629 Hz
FIDRES        0.383387 Hz
AQ            1.3042164 sec
RG                26008
DW              19.900 usec
DE               10.00 usec
TE               294.2 K
D1            3.00000000 sec
d11           0.03000000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                 6.00 usec
PL1               -3.00 dB
SFO1       100.6237964 MHz

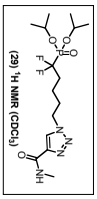
======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2            100.00 usec
PL2                0.00 dB
PL12              18.00 dB
SFO2       400.1328009 MHz

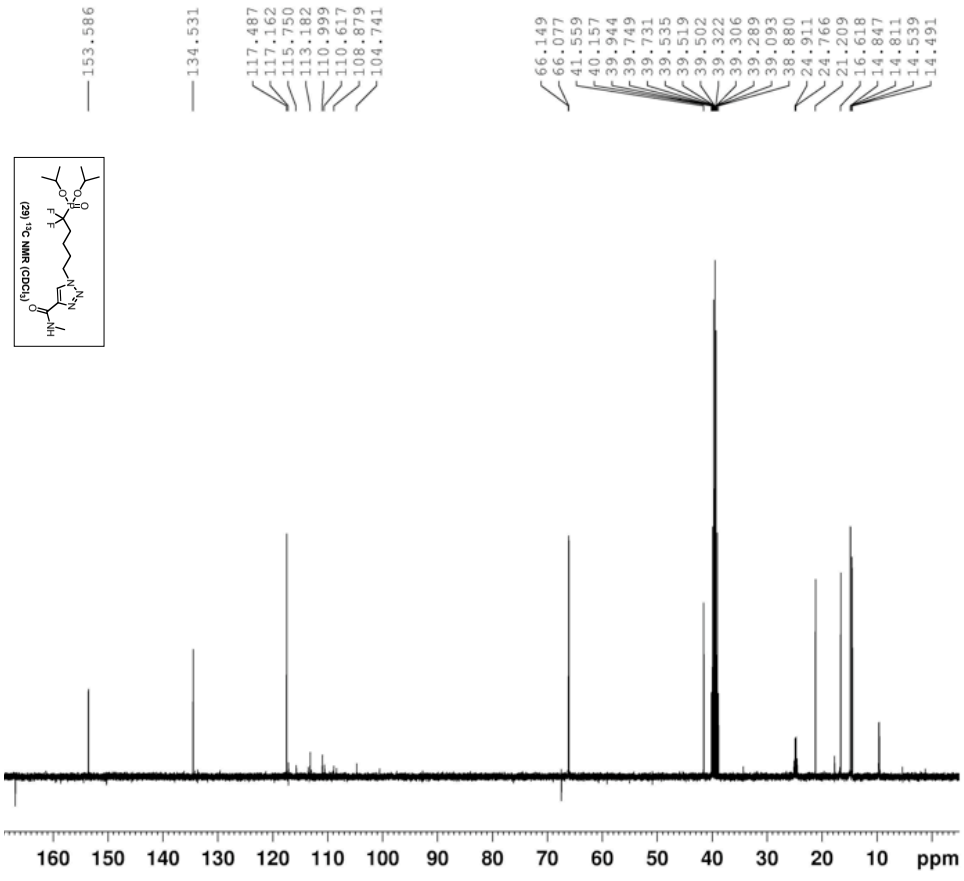
======== CHANNEL f3 ========
CPDPRG3          waltz16
NUC3                 31P
PCPD3              80.00 usec
PL3                9.00 dB
PL16              18.00 dB
SFO3       161.9788125 MHz
SI                65536
SF         100.6135853 MHz
WDW                  no
SSB                   0
LB                 0.00 Hz
GB                    0
PC                 1.00
```




```
NAME                sg135
EXPNO                  10
PROCNO                  1
Date_            20060712
Time                 7.02
INSTRUM             spect
PROBHD    5 mm QNP 1H/13
PULPROG              zg30
TD                  32768
SOLVENT              MeOD
NS                     16
DS                      2
SWH             8223.685 Hz
FIDRES          0.250967 Hz
AQ             1.9923444 sec
RG                  90.5
DW                60.800 usec
DE                10.00 usec
TE                294.2 K
D1           1.00000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                   1H
P1                 10.75 usec
PL1                -2.00 dB
SFO1        400.1324710 MHz
SI                  16384
SF          400.1300079 MHz
WDW                   no
SSB                    0
LB                  0.00 Hz
GB                     0
PC                  1.00
```



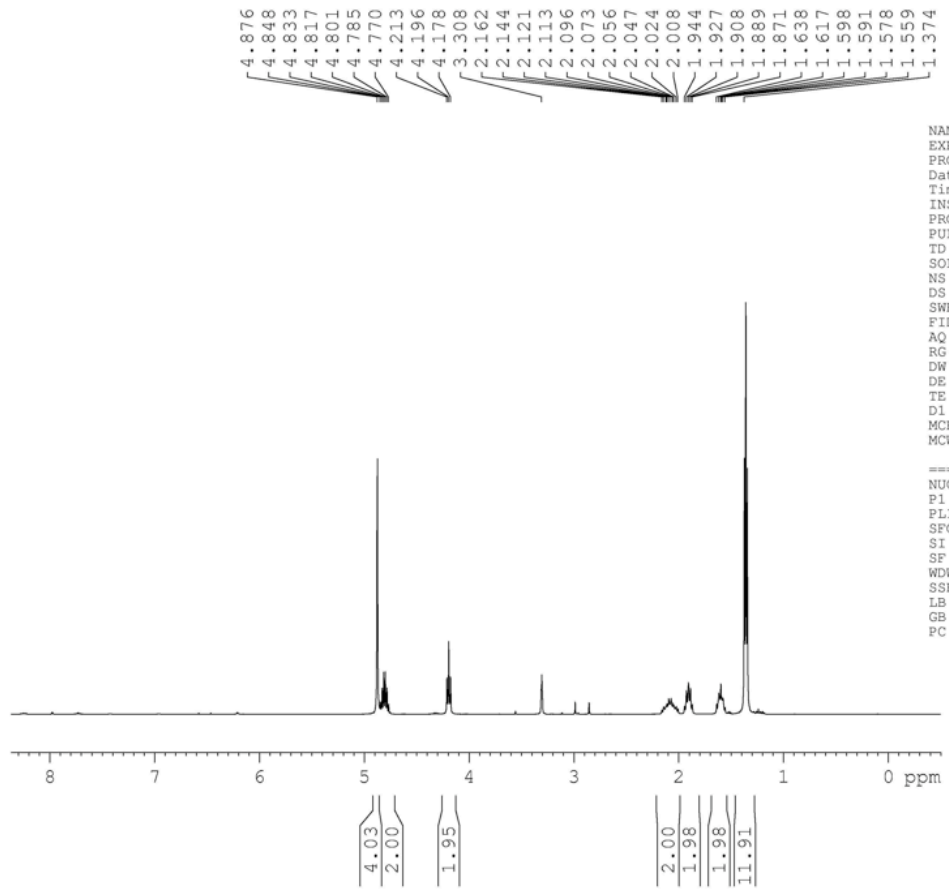



```
NAME                sg135
EXPNO                  20
PROCNO                  1
Date_            20060712
Time                 7.14
INSTRUM             spect
PROBHD    5 mm QNP 1H/13
PULPROG         zgfbig.chb
TD                  65536
SOLVENT              CDC13
NS                    244
DS                      0
SWH            25125.629 Hz
FIDRES          0.383387 Hz
AQ             1.3042164 sec
RG                  16384
DW                19.900 usec
DE                10.00 usec
TE                294.2 K
D1           3.00000000 sec
d11          0.03000000 sec

======== CHANNEL f1 ========
NUC1                  13C
P1                  6.00 usec
PL1                -3.00 dB
SFO1        100.6237964 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                   1H
PCPD2             100.00 usec
PL2                 0.00 dB
PL12               18.00 dB
SFO2        400.1328009 MHz

======== CHANNEL f3 ========
CPDPRG3          waltz16
NUC3                  31P
PCPD3              80.00 usec
PL3                 9.00 dB
PL16               18.00 dB
SFO3        161.9788125 MHz
SI                  65536
SF          100.6135875 MHz
WDW                   no
SSB                    0
LB                  0.00 Hz
GB                     0
PC                  1.00
```


4.876
4.848
4.833
4.817
4.801
4.785
4.770
4.213
4.196
4.178
3.308
2.162
2.144
2.121
2.113
2.096
2.073
2.056
2.047
2.024
2.008
1.944
1.927
1.908
1.889
1.871
1.638
1.617
1.598
1.591
1.578
1.559
1.374
NAME sg136
EXPNO 10
PROCNO 1
Date_ 20060718
Time 8.23
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 32768
SOLVENT MeOD
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.250967 Hz
AQ 1.9923444 sec
RG 362
DW 60.800 usec
DE 10.00 usec
TE 294.2 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 10.75 usec
PL1 -2.00 dB
SFO1 400.1324710 MHz
SI 16384
SF 400.1300082 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
8
7
6
5
4
3
2
1
0 ppm
4.03
2.00
1.95
2.00
1.98
1.98
11.91
(30) 1H NMR (CDCl3)