

Supplementary Information

Synthesis of phosphonoacetate analogues of the second messenger adenosine 5'-diphosphate ribose (ADPR)

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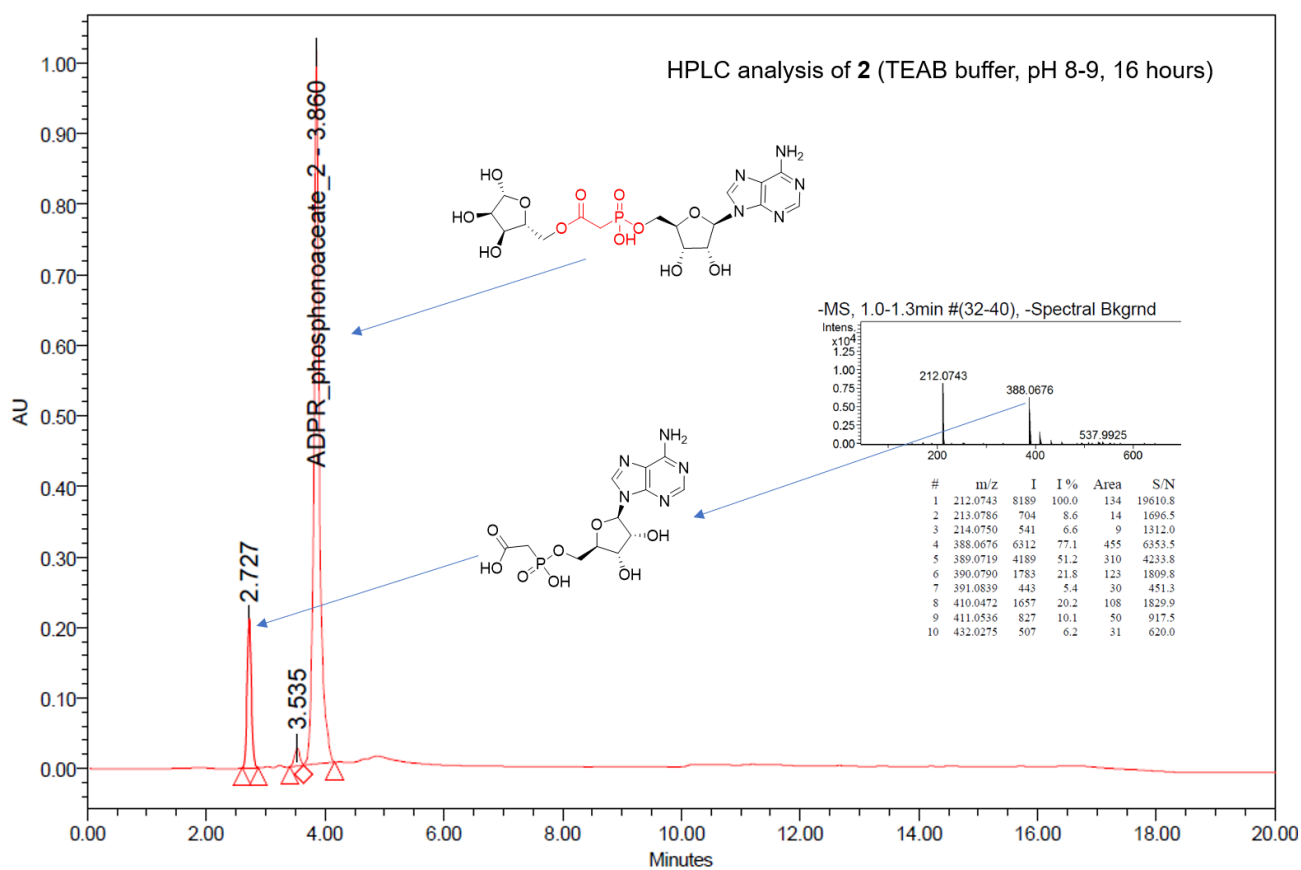
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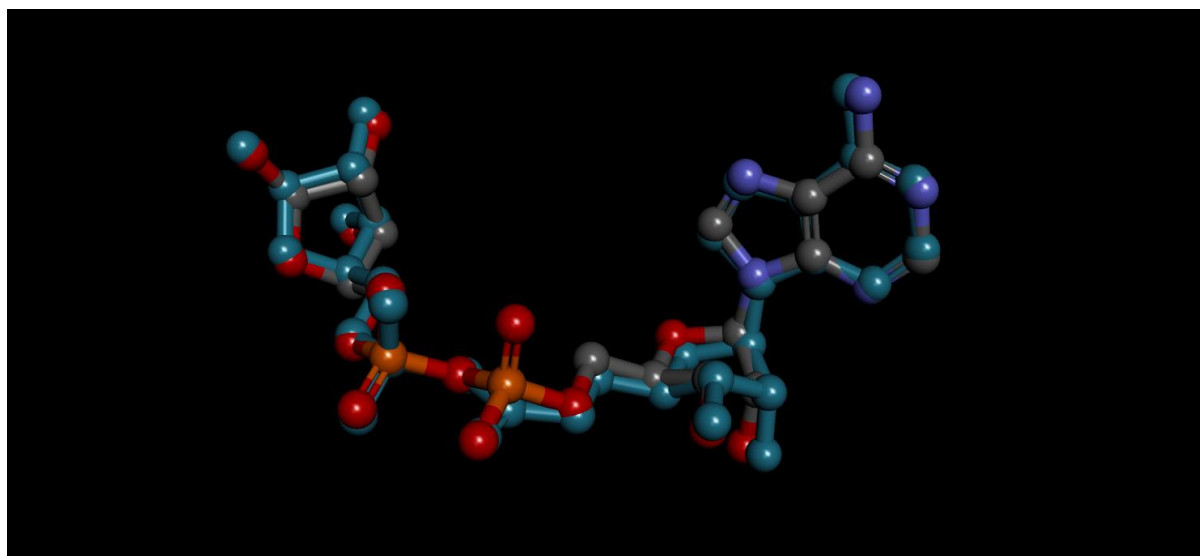
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Figure S1: Partial decomposition of compound **2** in TEAB (0.1M), after 16 hours



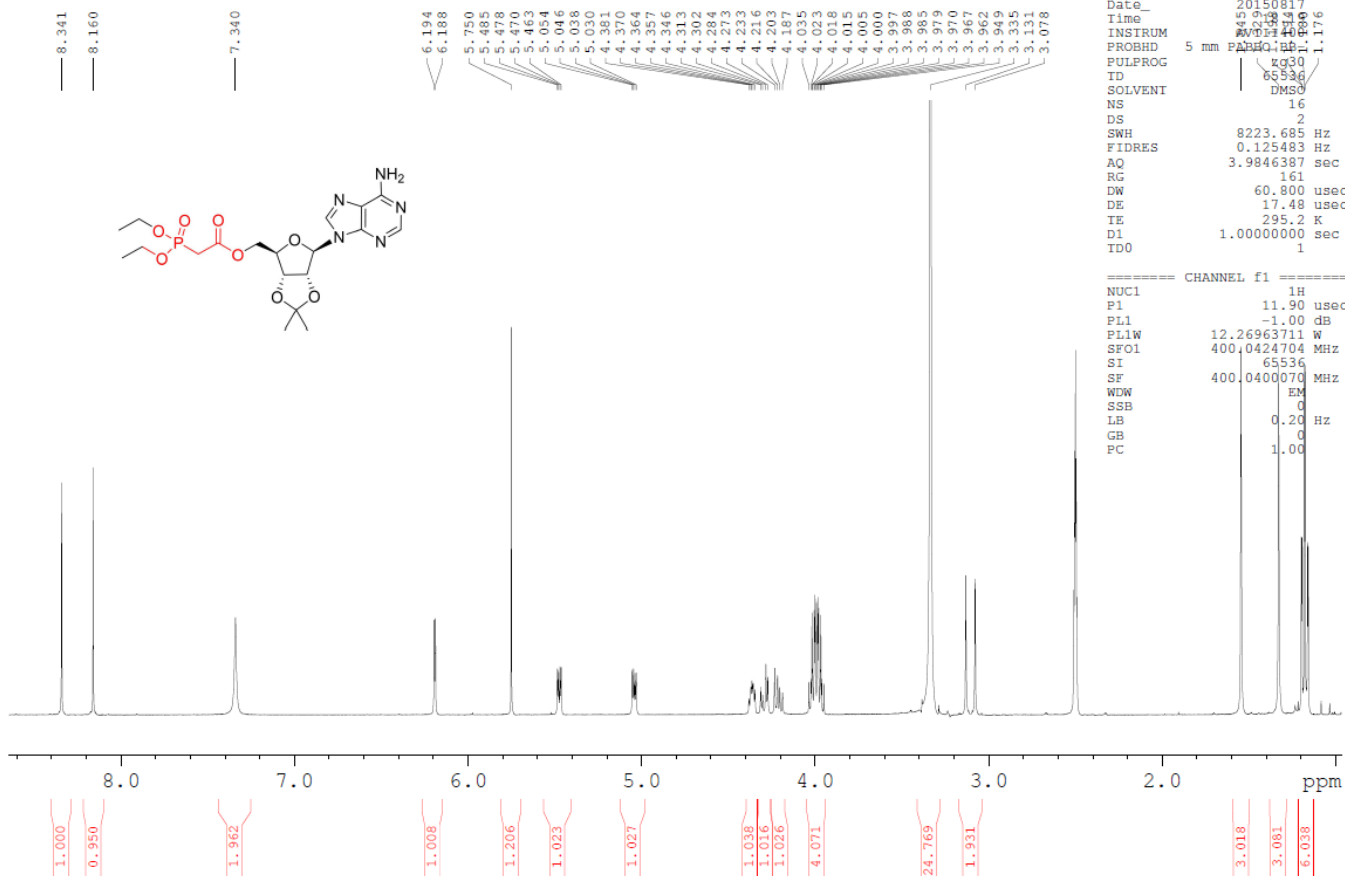
2

Figure S2. Comparison of phosphonoacetate analogue **1** with ADPR



Superposition of **1** (all atoms shown in blue) with ADPR (coloured), Discovery studio (Accelrys)

compound-5 ¹H NMR



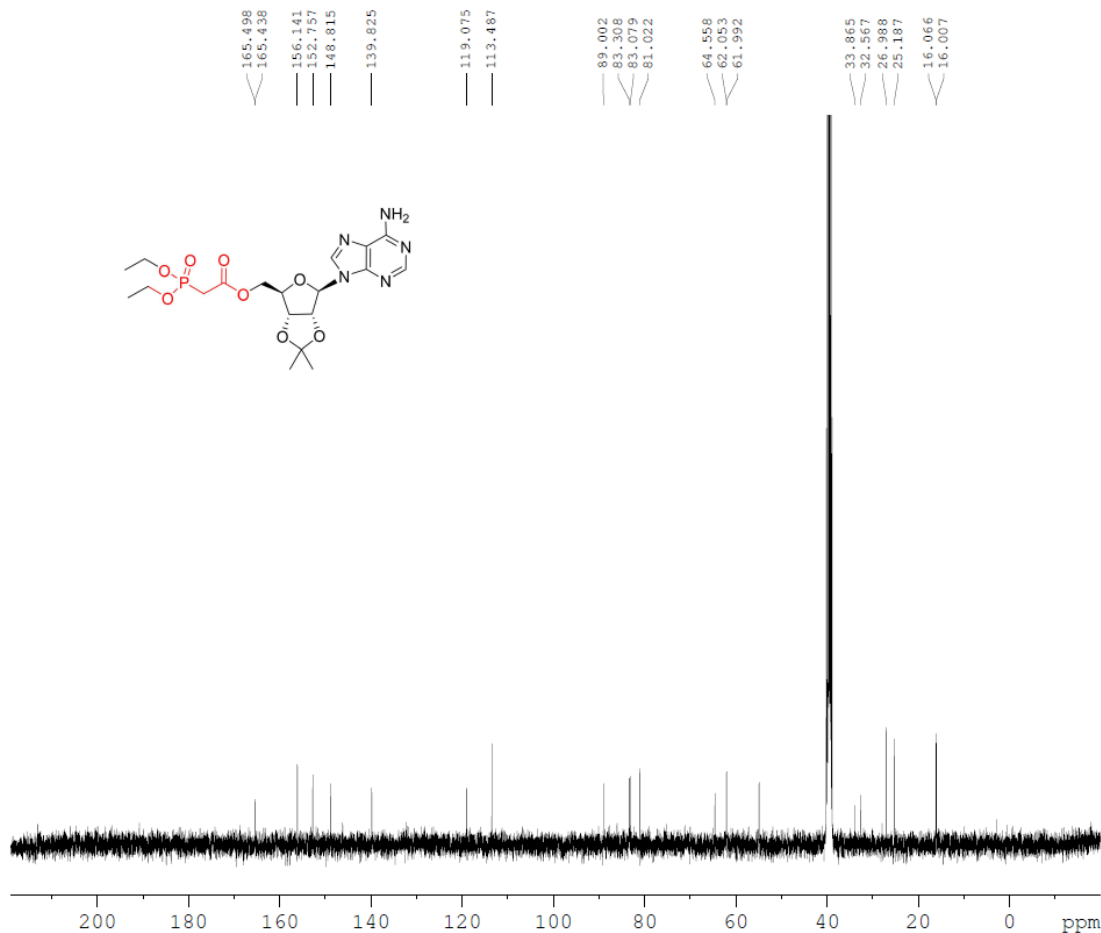
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NAME      Aug17-2015-ob6263
EXPNO    10
PROCNO   1
Date_    20150817
Time     15:08:34
INSTRUM  AVI1400
PROBHD   5 mm PABBO 1H
PULPROG  zgpg30
TD       65536
SOLVENT  DMSO
NS       16
DS       2
SWH      8223.685 Hz
FIDRES   0.125483 Hz
AQ       3.9846387 sec
RG       161
DW       60.800 usec
DE       17.48 usec
TE       295.2 K
D1       1.0000000 sec
D11      1
TD0      1
    
```

```

===== CHANNEL f1 =====
NUC1     1H
P1       11.90 usec
PL1      -1.00 dB
PL1W    12.26963711 W
SFO1     400.0424704 MHz
SI       65536
SF       400.0400070 MHz
WDW      EM
SSB      0
LB       0.20 Hz
GB       0
PC       1.00
    
```

compound-5 ¹³C NMR



```

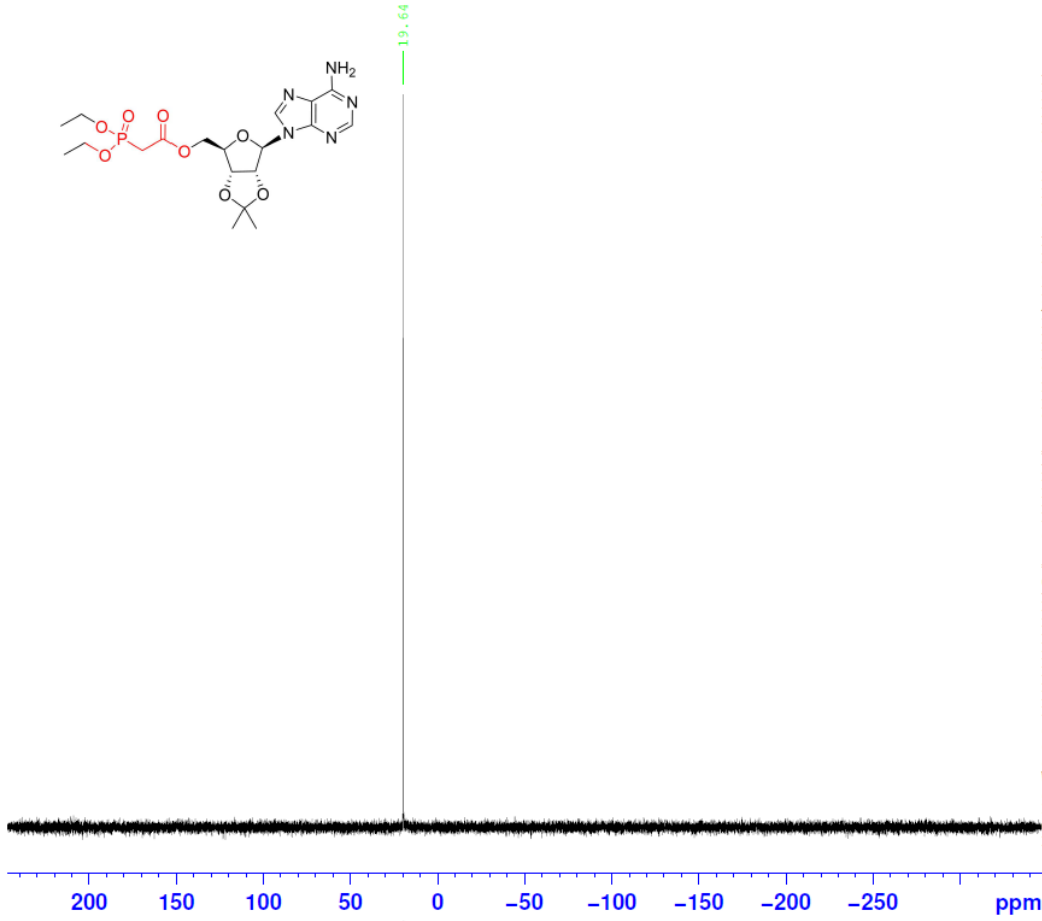
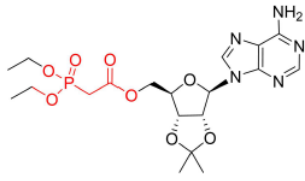
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EXPNO    14
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Date_    20150820
Time     6:09
INSTRUM  AVI1400
PROBHD   5 mm PABBO 5H
PULPROG  zgpg30
TU       65536
SOLVENT  DMSO
NS       512
DS       4
SWH      24038.461 Hz
FIDRES   0.366798 Hz
AQ       1.3631988 sec
RG       1820
DW       20.800 usec
DE       6.50 usec
TE       295.2 K
D1       2.0000000 sec
D11      0.0300000 sec
TD0      1
    
```

```

===== CHANNEL f1 =====
NUC1     13C
P1       9.25 usec
PL1      -2.00 dB
PL1W    58.9198684 W
SFO1     100.6001970 MHz

===== CHANNEL f2 =====
CPDPRG2  waltz16
NUC2     1H
PCPDZ    80.00 usec
PL2      -1.00 dB
PL12     15.55 dB
PL13     19.00 dB
PL2W    12.26963711 W
PL12W   0.27153867 W
PL13W   0.12269637 W
SFO2     400.0416002 MHz
SI       65536
SF       100.5901865 MHz
WDW      EM
SSB      0
LB       1.00 Hz
GB       0
PC       1.40
    
```

compound-5 ³¹P NMR



```

NAME      Aug17-2015-ob6263
EXPNO     1
PROCNO    1
Date_     20150817
Time      18.23
INSTRUM   AVIII400
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD         65536
SOLVENT   DMSO
NS         64
DS         4
SWH        96153.844 Hz
FIDRES     1.467191 Hz
AQ         0.3408372 sec
RG         2050
DW         5.200 usec
DE         6.50 usec
TE         295.2 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1
    
```

```

===== CHANNEL f1 =====
NUC1      31P
P1        9.40 usec
PL1       0.00 dB
PL1W      23.83780289 W
SFO1      161.9310633 MHz
    
```

```

===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2      1H
PCPD2     80.00 usec
PL2       -1.00 dB
PL12      15.55 dB
PL13      19.00 dB
PL12W     12.26963711 W
PL12W     0.27153867 W
PL13W     0.12269637 W
SFO2      400.0416002 MHz
SI        32768
SF        161.9391600 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.40
    
```

compound-6 ¹H NMR

8.586
8.157
7.325

6.130
6.123

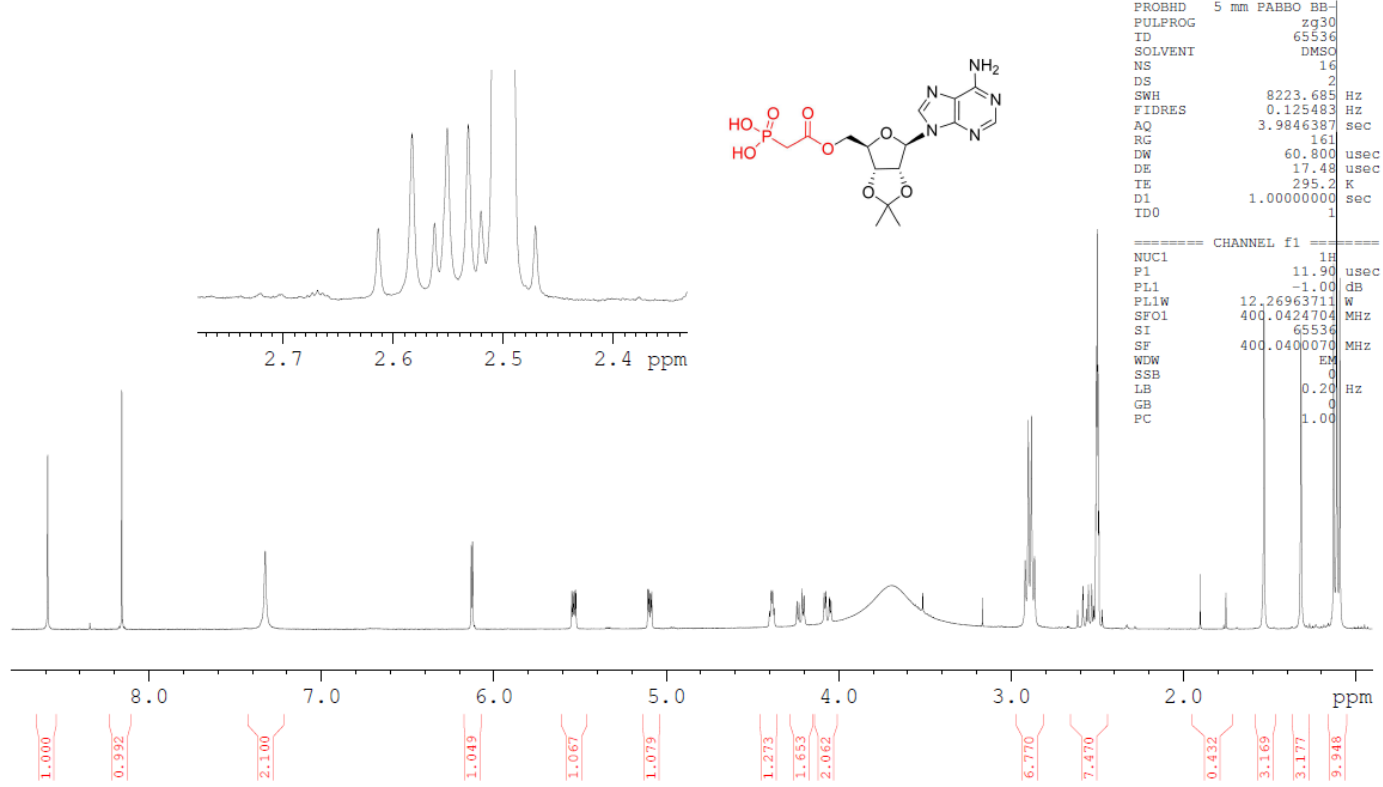
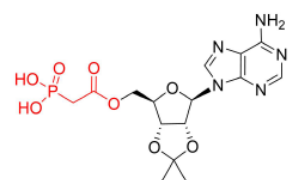
5.548
5.540
5.532
5.524

5.106
5.099
5.091
5.084

4.401
4.391
4.384
4.374
4.242
4.230
4.211
4.200
4.084
4.074
4.053
4.045

3.512
3.166
3.158
3.150
2.982
2.983
2.614
2.583
2.563
2.551
2.532
2.520
2.509

2.495
2.486
2.477
2.471
1.902
1.894
1.552
1.521
1.389
1.380
1.171
1.161
1.092
1.083



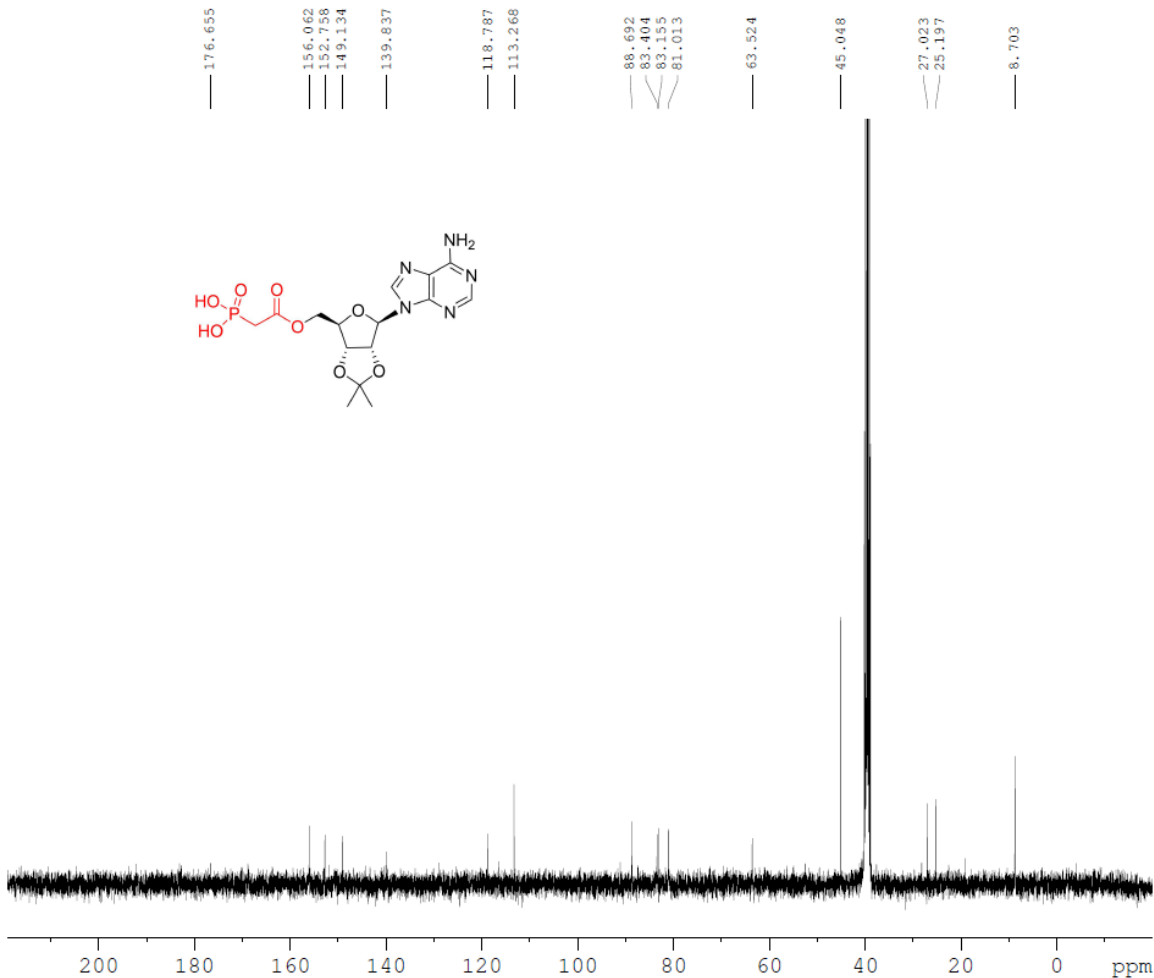
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Date_     20150817
Time      18.11
INSTRUM   AVIII400
PROBHD    5 mm PABBO BB-
PULPROG   zg30
TD         65536
SOLVENT   DMSO
NS         16
DS         2
SWH        8223.685 Hz
FIDRES     0.125483 Hz
AQ         3.9846387 sec
RG         161
DW         60.800 usec
DE         17.48 usec
TE         295.2 K
D1         1.00000000 sec
D10        1
    
```

```

===== CHANNEL f1 =====
NUC1      1H
P1        11.90 usec
PL1       -1.00 dB
PL1W      12.26963711 W
SFO1      400.0424704 MHz
SI        65536
SF        400.0400070 MHz
WDW        EM
SSB        0
LB         0.20 Hz
GB         0
PC         1.00
    
```

compound-6 ¹³CNMR



```

NAME      Aug17-2015-ob6262
EXPNO    12
PROCNO   1
Date_    20150820
Time     7.32
INSTRUM  AVIII400
PROBHD   5 mm PABBO BB-
PULPROG  zgpg30
TD       65536
SOLVENT  DMSO
NS       512
DS       4
SWH      24038.461 Hz
FIDRES   0.366798 Hz
AQ       1.3631988 sec
RG       1820
DW       20.800 usec
DE       6.50 usec
TE       295.2 K
D1       2.0000000 sec
D11      0.0300000 sec
TD0      1
    
```

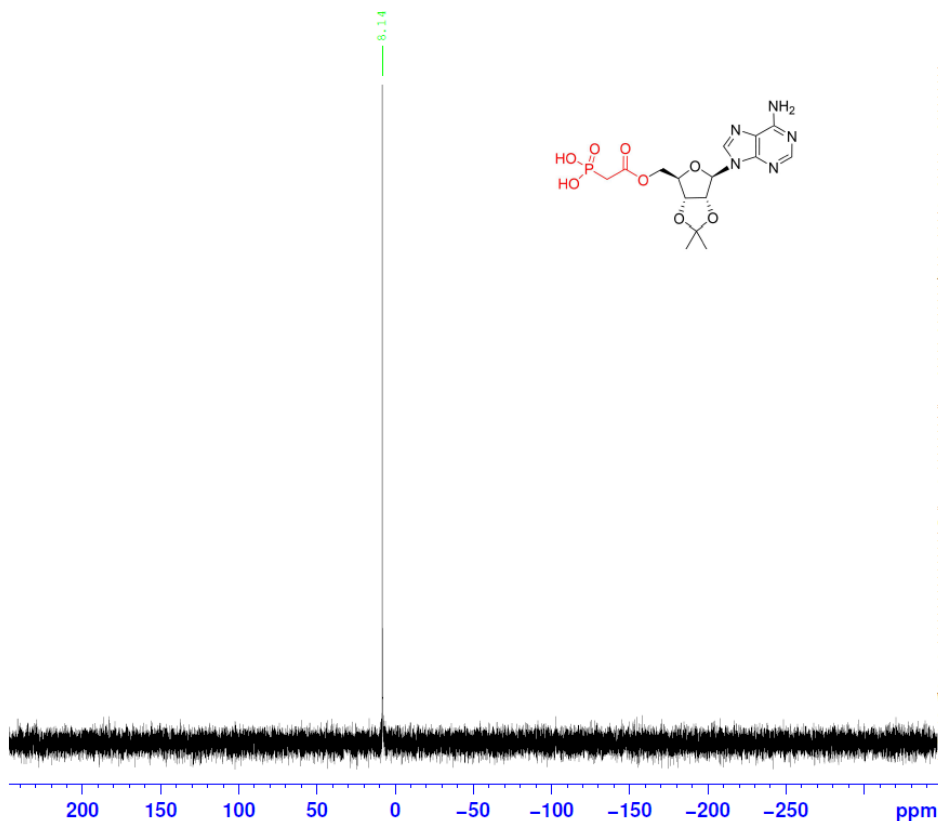
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===== CHANNEL f1 =====
NUC1     13C
P1       9.25 usec
PL1     -2.00 dB
PL1W    58.9198684 W
SFO1    100.6001970 MHz
    
```

```

===== CHANNEL f2 =====
CPDPRG2  waltz16
NUC2     1H
PCPD2   80.00 usec
PL2     -1.00 dB
PL12    15.55 dB
PL13    19.00 dB
PL2W    12.2696371 W
PL12W   0.27153867 W
PL13W   0.12269637 W
SFO2    400.0416002 MHz
SI       65536
SF      100.5901868 MHz
WDW     EM
SSB     0
LB      1.00 Hz
GB      0
PC      1.40
    
```

compound-6 ³¹P NMR



```

NAME      Aug17-2015-ob6262
EXPNO    11
PROCNO   1
Date_    20150817
Time     18.15
INSTRUM  AVIII400
PROBHD   5 mm PABBO BB-
PULPROG  zgpg30
TD       65536
SOLVENT  DMSO
NS       64
DS       4
SWH      96153.844 Hz
FIDRES   1.467191 Hz
AQ       0.3408372 sec
RG       2050
DW       5.200 usec
DE       6.50 usec
TE       295.2 K
D1       2.0000000 sec
D11      0.0300000 sec
TD0      1
    
```

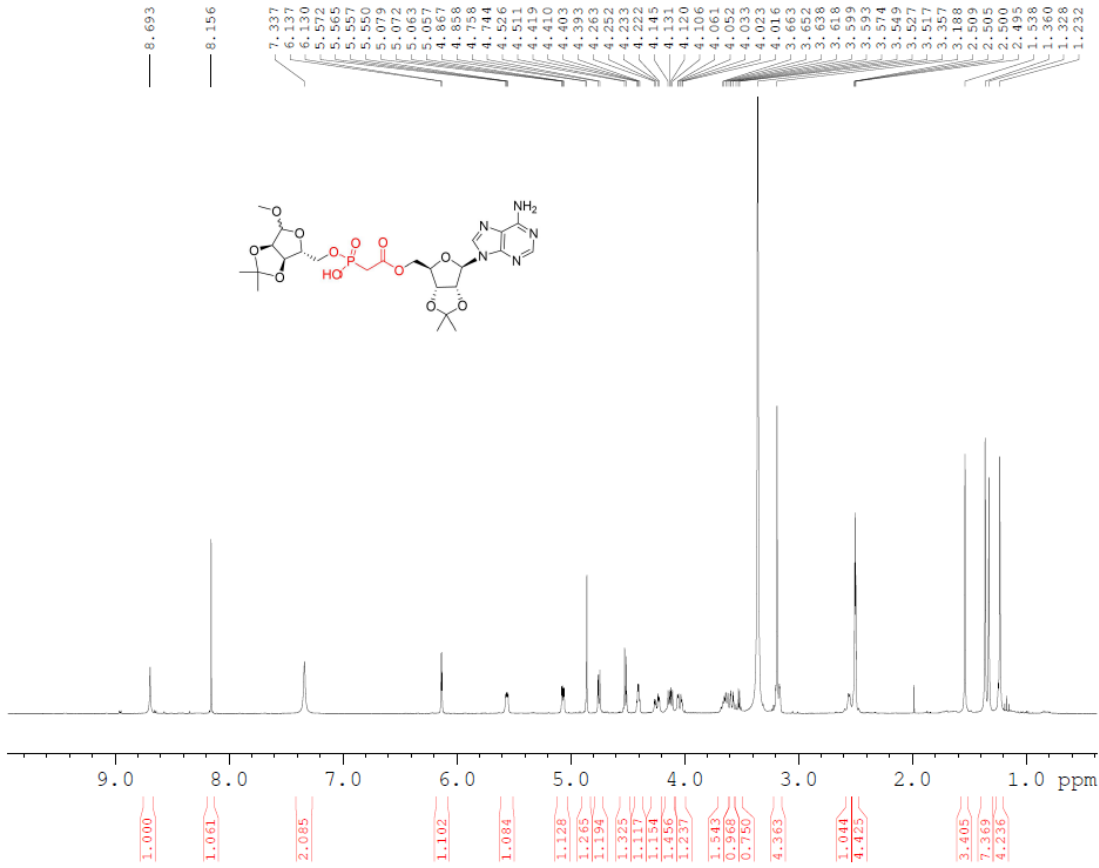
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===== CHANNEL f1 =====
NUC1     31P
P1       9.40 usec
PL1     0.00 dB
PL1W    23.83780289 W
SFO1    161.9310633 MHz
    
```

```

===== CHANNEL f2 =====
CPDPRG2  waltz16
NUC2     1H
PCPD2   80.00 usec
PL2     -1.00 dB
PL12    15.55 dB
PL13    19.00 dB
PL2W    12.2696371 W
PL12W   0.27153867 W
PL13W   0.12269637 W
SFO2    400.0416002 MHz
SI       32768
SF      161.9391600 MHz
WDW     EM
SSB     0
LB      1.00 Hz
GB      0
PC      1.40
    
```

compound-7 ¹HNMR

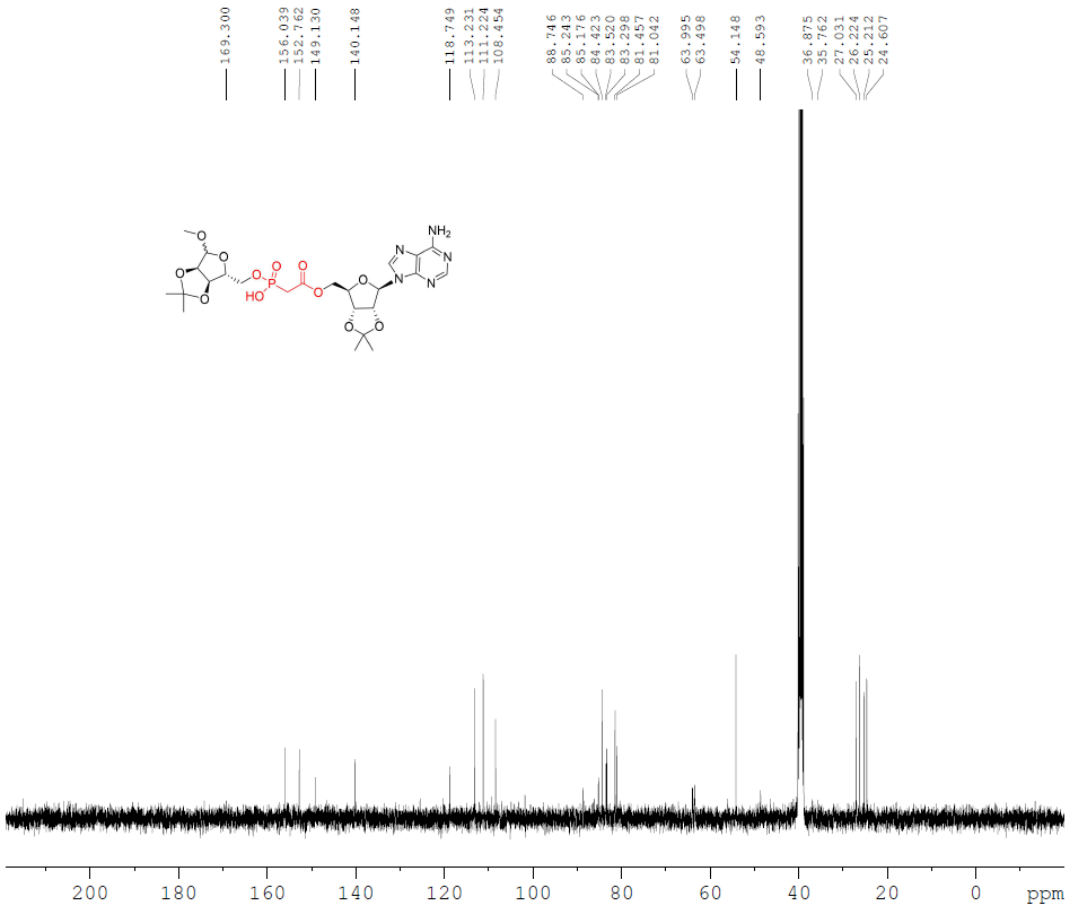


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EXPNO    1
PROCNO   1
Date_    20150817
Time     18.03
INSTRUM  AVIII400
PROBHD   5 mm PABBO BB-
PULPROG  zg30
TD        65536
SOLVENT  DMSO
NS        16
DS        2
SWH       8223.695 Hz
FIDRES    0.125483 Hz
AQ        3.9846387 sec
RG        144
DW        60.800 usec
DE        17.48 usec
TE        295.2 K
D1        1.00000000 sec
TD0       1

===== CHANNEL f1 =====
NUC1      1H
P1        11.90 usec
PL1       -1.00 dB
PL1W      12.26963711 W
SFO1      400.0424704 MHz
SI        65536
SF        400.0400070 MHz
WDW       EM
SSB       0
LB        0.20 Hz
GB        0
PC        1.00
    
```

compound-7 ¹³CNMR



```

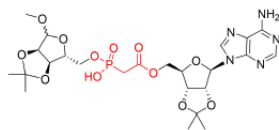
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EXPNO    1
PROCNO   1
Date_    20150817
Time     21.31
INSTRUM  AVIII400
PROBHD   5 mm PABBO BB-
PULPROG  zgpg30
TD        65536
SOLVENT  DMSO
NS        512
DS        4
SWH       24039.461 Hz
FIDRES    0.366798 Hz
AQ        1.3631988 sec
RG        1820
DW        20.800 usec
DE        6.50 usec
TE        295.2 K
D1        2.00000000 sec
D11       0.03000000 sec
TD0       1

===== CHANNEL F1 =====
NUC1      13C
P1        9.25 usec
PL1       -2.00 dB
PL1W      58.91986084 W
SFO1      100.6001970 MHz

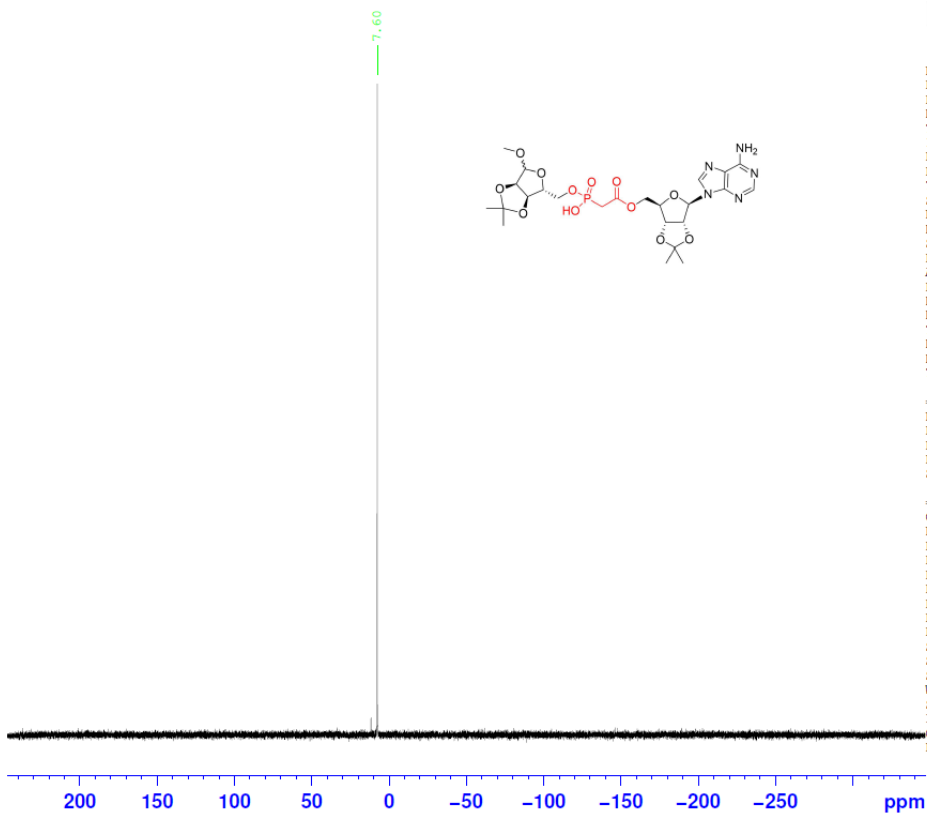
===== CHANNEL F2 =====
CPDPRG2  waltz16
NUC2      1H
PCPD2    80.00 usec
PL2       -1.00 dB
PL12     15.55 dB
PL13     19.00 dB
PL1W     12.26963711 W
SFO2     0.27153867 W
PL13W    0.12269637 W
SFO2     400.0416002 MHz
SI        65536
SF        100.5901857 MHz
WDW       EM
SSB       0
LB        1.00 Hz
GB        0
PC        1.40
    
```



NAME Aug17-2015-ob6261
 EXPNO 11
 PROCNO 1
 Date_ 20150817
 Time 18.07
 INSTRUM AVIII400
 PROBHD 5 mm PABBO BB-
 PULPROG zgpg30
 TD 65536
 SOLVENT DMSO
 NS 64
 DS 4
 SWH 96153.844 Hz
 FIDRES 1.467191 Hz
 AQ 0.3408372 sec
 RG 2050
 DW 5.200 usec
 DE 6.50 usec
 TE 295.2 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TD0 1



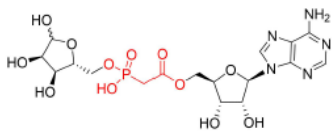
===== CHANNEL f1 =====
 NUC1 31P
 P1 9.40 usec
 PL1 0.00 dB
 PL1W 23.83780289 W
 SFO1 161.9310633 MHz
 ===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 -1.00 dB
 PL12 15.55 dB
 PL13 19.00 dB
 PL2W 12.26963711 W
 PL12W 0.27153867 W
 PL13W 0.12269637 W
 SFO2 400.0416002 MHz
 S1 32768
 SF 161.9391600 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40



compound-1 ¹H NMR (DOSY)

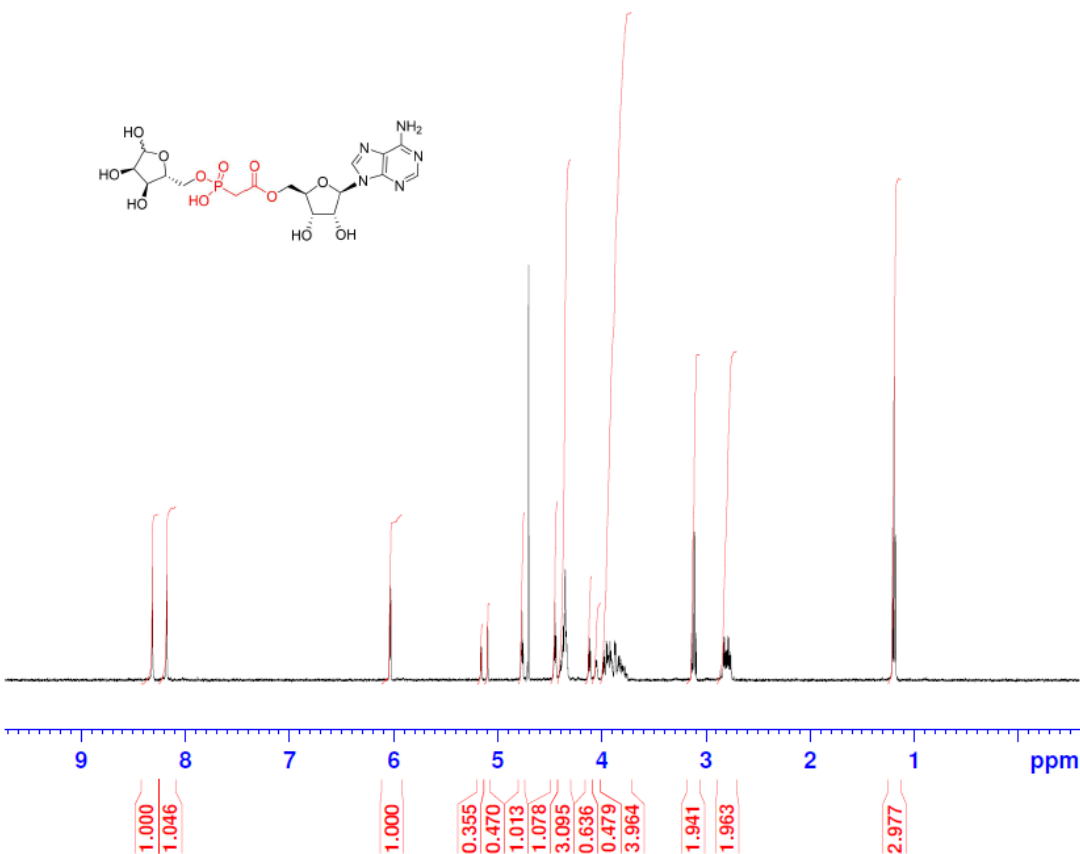
NAME OBB44
 EXPNO 15
 PROCNO 1
 Date_ 20151119
 Time 17.29
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG ledbpp2s1d
 TD 65536
 SOLVENT D2O
 NS 16
 DS 2
 SWH 10330.578 Hz
 FIDRES 0.157632 Hz
 AQ 3.1719923 sec
 RG 406
 DW 48.400 usec
 DE 9.73 usec
 TE 298.2 K
 D1 3.00000000 sec
 D16 0.00020000 sec
 D20 0.05000000 sec
 D21 0.00500000 sec
 TD0 1

8.316
 8.181
 8.178
 6.037
 6.028
 5.164
 5.156
 5.100
 5.095
 4.779
 4.768
 4.759
 4.704
 4.460
 4.450
 4.440
 4.380
 4.371
 4.361
 4.353
 4.343
 4.337
 4.120
 4.109
 3.982
 3.957
 3.949
 3.923
 3.914
 3.880
 3.876
 3.870
 3.866
 3.140
 3.125
 3.110
 3.096
 2.834
 2.824
 2.812
 2.803
 2.792
 2.783
 2.771
 1.210
 1.195
 1.180



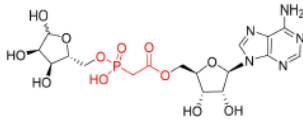
===== CHANNEL f1 =====
 NUC1 1H
 P1 10.00 usec
 P2 20.00 usec
 PL1 -0.12 dB
 PL1W 19.35150909 W
 SFO1 500.1330885 MHz

===== GRADIENT CHANNEL =====
 GPNAM6 sine.100
 GPNAM7 sine.100



compound-1 ¹³CNMR

169.698
155.526
152.729
149.019
144.599
139.985
118.868
101.053
96.275
87.581
81.850
81.115
75.056
73.442
70.649
70.438
69.997
69.853
64.025
46.645
35.301
8.190



```

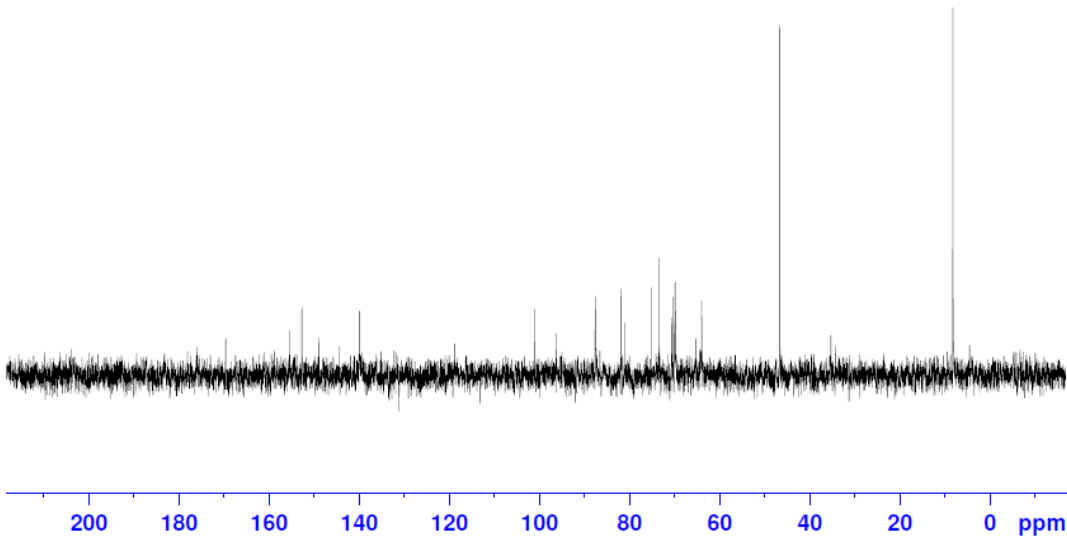
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EXPNO     12
PROCNO    1
Date_     20151119
Time      23.22
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD        65536
SOLVENT   D2O
NS        4096
DS        4
SWH       29761.904 Hz
FIDRES    0.454131 Hz
AQ        1.1010548 sec
RG        2050
DW        16.800 usec
DE        8.45 usec
TE        298.2 K
D1        2.0000000 sec
D11       0.0300000 sec
TD0       1
    
```

```

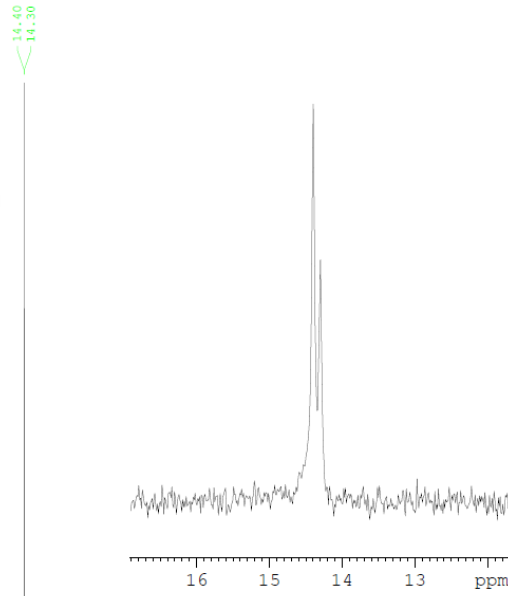
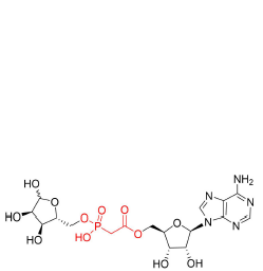
===== CHANNEL f1 =====
NUC1      13C
P1        9.40 usec
PL1       -0.51 dB
PL1W     99.92730713 W
SFO1     125.7703643 MHz
    
```

```

===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2      1H
PCPD2     80.00 usec
PL2       -0.12 dB
PL12      17.94 dB
PL13      21.00 dB
PL2W     19.35150909 W
PL12W    0.30249262 W
PL13W    0.14952536 W
SFO2     500.1320005 MHz
SI        131072
SF        125.7577890 MHz
WDW       EM
SSB       0
LB        3.00 Hz
GB        0
PC        1.40
    
```



compound-1 ³¹P NMR



```

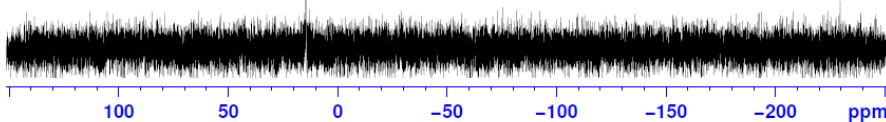
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EXPNO     15
PROCNO    1
Date_     20151119
Time      6.55
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD        65536
SOLVENT   D2O
NS        256
DS        4
SWH       81521.742 Hz
FIDRES    1.243923 Hz
AQ        0.4020041 sec
RG        2050
DW        6.133 usec
DE        6.50 usec
TE        298.2 K
D1        2.0000000 sec
D11       0.0300000 sec
TD0       1
    
```

```

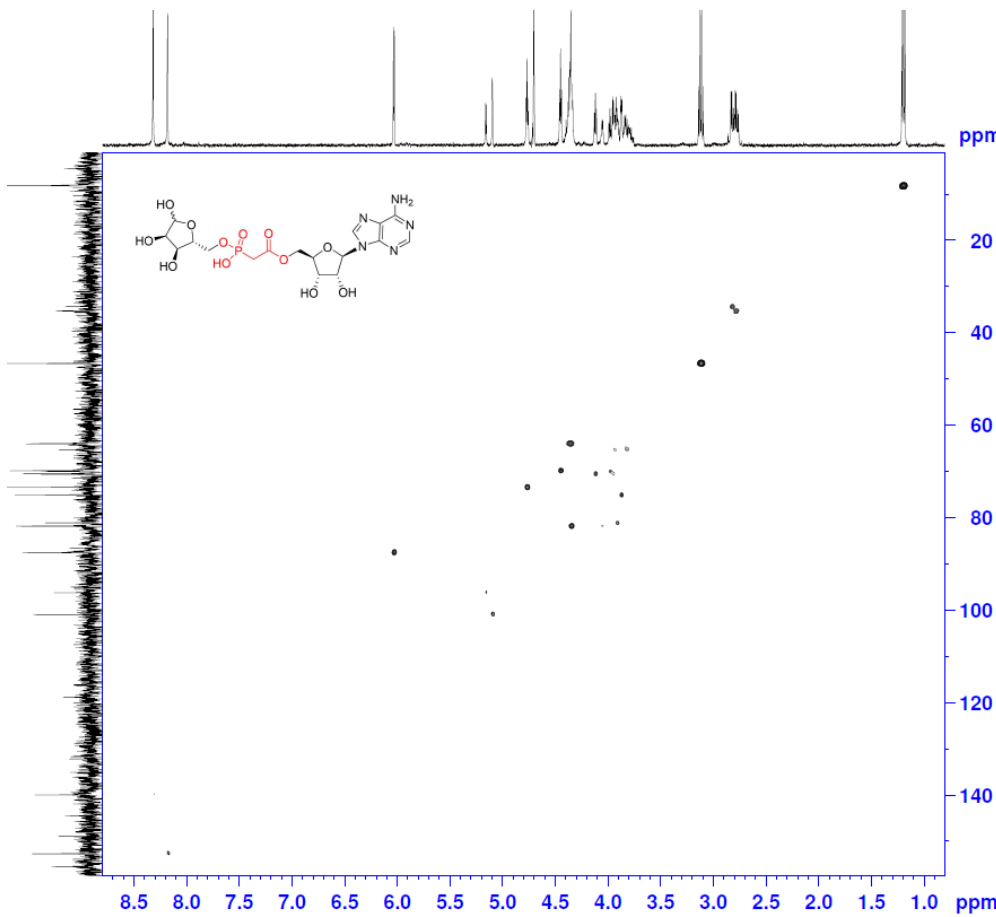
===== CHANNEL f1 =====
NUC1      31P
P1        10.50 usec
PL1       0.00 dB
PL1W     81.20777893 W
SFO1     202.4462121 MHz
    
```

```

===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2      1H
PCPD2     80.00 usec
PL2       -0.12 dB
PL12      17.94 dB
PL13      21.00 dB
PL2W     19.35150909 W
PL12W    0.30249262 W
PL13W    0.14952536 W
SFO2     500.1320005 MHz
SI        32768
SF        202.4563350 MHz
WDW       EM
SSB       0
LB        1.00 Hz
GB        0
PC        1.40
    
```



compound-1 HSQC



```

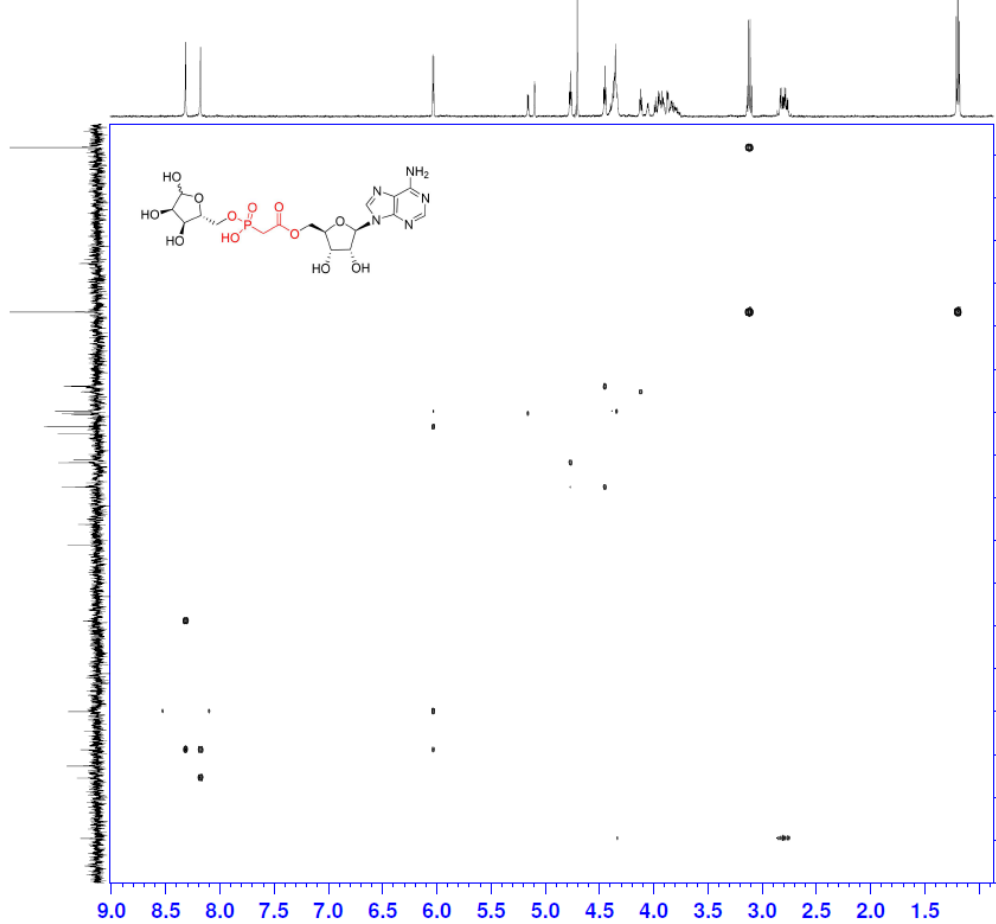
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EXPNO 1
PROCNO 1
Date_ 20151119
Time 8:55
INSTRUM spect
PROBHD 5 mm F4BBO BB-
PULPROG hsqcattppp_3
TD 1024
SOLVENT D2O
NS 2
DS 16
SWH 5980.861 Hz
FIDRES 5.84658 Hz
AQ 0.0856564 sec
RG 350
DM 83.600 usec
DE 6.50 usec
TE 298.2 K
CNS1 148.000000 sec
DO 0.0000000 sec
D1 1.5000000 sec
D11 0.00172414 sec
D12 0.0300000 sec
D16 0.0002000 sec
D21 0.0036000 sec
INO 0.00001805 sec

----- CHANNEL f1 -----
NUC1 13
P1 10.00 usec
P2 20.00 usec
P3 0.00 usec
P4 0.00 usec
PL1 -0.12 dB
PL1W 19.35150909 W
SFO1 500.1323521 MHz

----- CHANNEL f2 -----
CPDPRG2 gpcp2
NUC2 13C
P3 8.40 usec
P4 500.00 usec
P14 1900.00 usec
P31 74.00 usec
PCPD2 74.00 usec
PL0 120.00 dB
PL2 -0.51 dB
PL12 17.41 dB
PL1W 0.0000000 W
PL2W 99.92730713 W
PL3W 1.41318505 W
SFO2 125.7665916 MHz
SF3 8.19 dB
SF7 14.39 dB
SPNAM3 Crp60, 0.5, 20.1
SPNAM18 crp60_xr11.2
SFOAL3 0.500
SFOAL18 0.500
SPOFF3 0.00 Hz
SPOFF18 0.00 Hz

----- GRADIENT CHANNEL -----
CPNAM1 SMSG10.100
CPNAM2 SMSG10.100
CP1 80.00 %
CP2 20.10 %
F16 1000.00 usec
ND0 2
TD 256
SFO1 125.7665 MHz
FIDRES 108.080666 Hz
SW 240.000 ppm
F2MODE Echo-Antiecho
SI 1024
SF 500.1300000 MHz
WVW Q5INE
SBB 2
LB 0.00 Hz
GB 0
PC 1.40
SI 1024
MC2 echo-antiecho
SF 125.7571980 MHz
WVW Q5INE
SBB 2
LB 0.00 Hz
GB 0
    
```

compound-1 HMBC



```

NAME OR844_mora
EXPNO 11
PROCNO 1
Date_ 20151119
Time 17:36
INSTRUM spect
PROBHD 5 mm F4BBO BB-
PULPROG hmbcattpp3ind
TD 4096
SOLVENT D2O
NS 2
DS 16
SWH 6510.417 Hz
FIDRES 1.589451 Hz
AQ 0.3146228 sec
RG 2050
DM 76.800 usec
DE 6.50 usec
TE 298.2 K
CNS1 120.000000 sec
CNS17 170.000000 sec
CNS13 5.0000000 sec
CNS130 0.5981126 sec
DO 0.0000000 sec
D1 1.5000000 sec
D6 0.0625000 sec
D16 0.0012000 sec
INO 0.00001655 sec

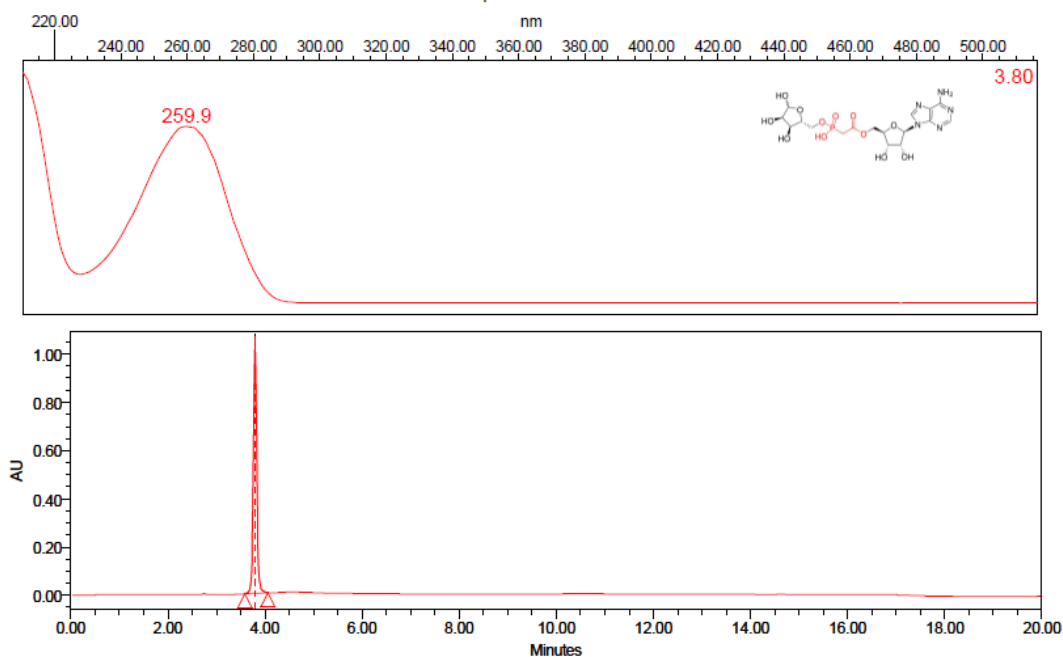
----- CHANNEL f1 -----
NUC1 13
P1 10.00 usec
P2 20.00 usec
P3 0.00 usec
P4 0.00 usec
PL1 -0.12 dB
PL1W 19.35150909 W
SFO1 500.1331508 MHz

----- CHANNEL f2 -----
NUC2 13C
P3 8.40 usec
P4 2000.00 usec
P14 1900.00 usec
P31 74.00 usec
PCPD2 74.00 usec
PL0 120.00 dB
PL2 -0.51 dB
PL12 17.41 dB
PL1W 0.0000000 W
PL2W 99.92730713 W
PL3W 1.41318505 W
SFO2 125.7716219 MHz
SF7 8.19 dB
SPNAM7 Crp60com4
SFOAL7 0.500
SPOFF7 0.00 Hz

----- GRADIENT CHANNEL -----
CPNAM1 SMSG10.100
CPNAM3 SMSG10.100
CPNAM6 SMSG10.100
CPNAM5 SMSG10.100
CPNAM6 SMSG10.100
CP1 80.00 %
CP2 20.10 %
CP3 14.00 %
CP24 -8.00 %
CP25 -4.00 %
CP26 -2.00 %
F16 1000.00 usec
ND0 2
TD 256
SFO1 125.7716 MHz
FIDRES 117.910896 Hz
SW 240.000 ppm
F2MODE Echo-Antiecho
SI 2048
SF 500.1300000 MHz
WVW Q5INE
SBB 2
LB 0.00 Hz
GB 0
PC 1.40
SI 1024
MC2 echo-antiecho
SF 125.7571980 MHz
WVW Q5INE
SBB 2
LB 0.00 Hz
GB 0
    
```

Spectrum Index Plot

Compound-1 UV/HPLC



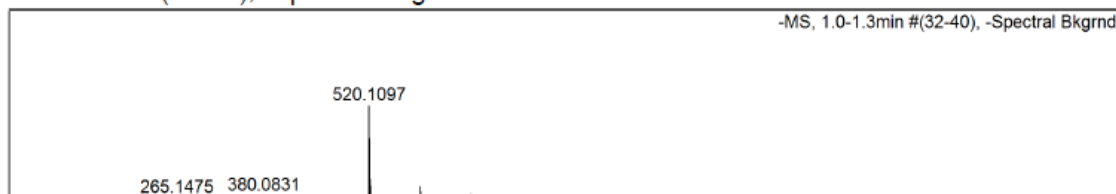
PDA Result Table

Name	RT	Area	Purity (#1) Angle	Purity (#1) Threshold	Purity Flag	Match (#1) Spect. Name	Match (#1) Angle	Match (#1) Threshold	PDA Match Flag
1 Compound-1	3.798	5141072			No				No

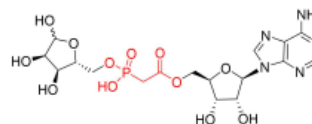
Confirmation of Expected Formula

Sample-ID	compound-1	Submitter	Ondrej Baszczynski
Analysis Name	po_ob_obb44_345634_43_01_50210.d	Supervisor	Barry Potter
Method used	Confirm Formula Negative 50to1500 loop inj.m	Acquisition Date	26/11/2015 10:33:36
Ionisation Mode	negative electrospray (ESI)		

-MS, 1.0-1.3min #(32-40), -Spectral Bkgrnd



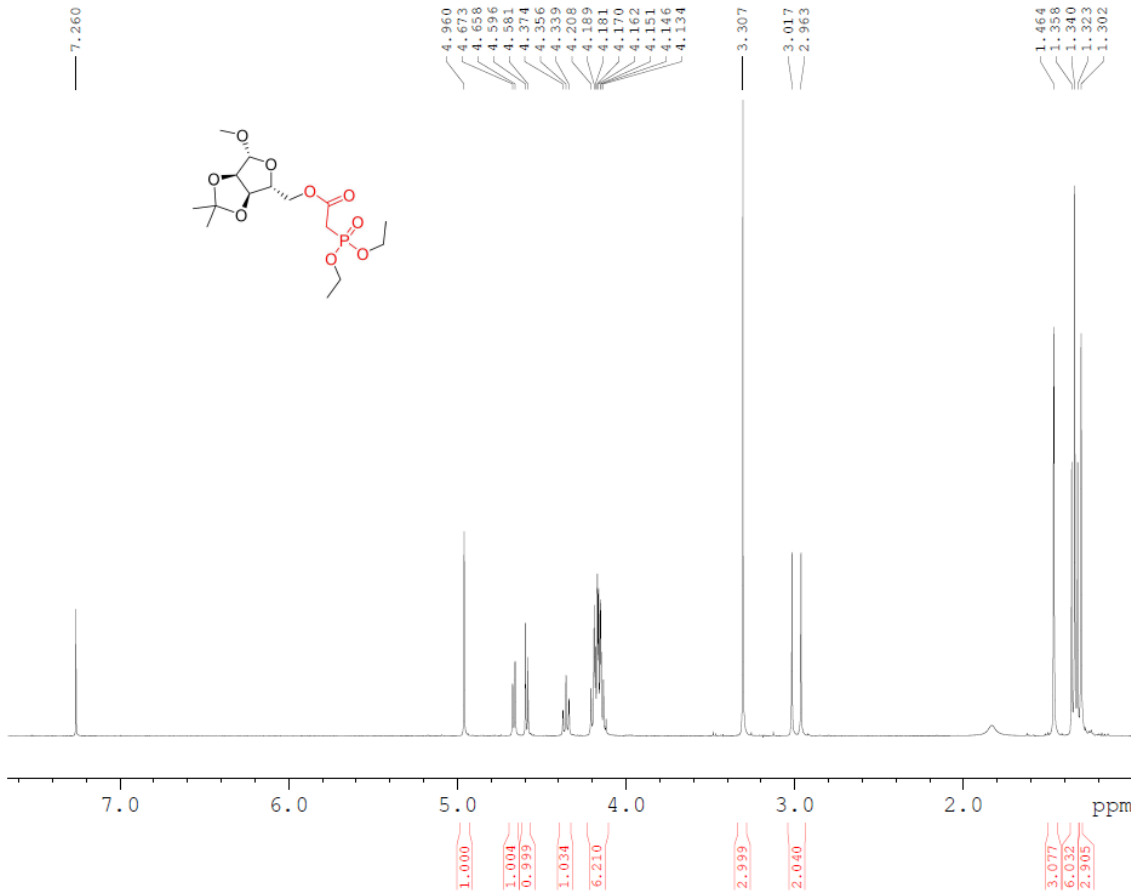
#	m/z	I	I%	Area	S/N
1	265.1475	142	2.2	6	3780.9
2	380.0831	328	5.0	20	14138.4
3	520.1097	6522	100.0	496	21988.3
4	520.9155	285	4.4	12	968.0
5	521.1132	1445	22.2	101	4923.3
6	522.1150	292	4.5	22	1006.4
7	588.0950	939	14.4	78	8695.3
8	589.1039	239	3.7	14	2221.1
9	656.0836	395	6.0	32	4196.9
10	724.0699	140	2.1	10	2161.1



Generate Molecular Formula Parameters

Charge	Tolerance	SearchRadius	H/C Ratio min.	H/C Ratio max.	Electron Conf.	Nitrogen Rule	sigma limit
negative	10 ppm	0.05 m/z	0	3	both	true	0.05
Expected Formula	C17 H24 N5 O12 P		Adduct(s):		H, Na		
#	meas. m/z	theo. m/z	Err[ppm]	Sigma	Formula		
1	520.1097	520.108083	-2.10	0.0078	C 17 H 23 N 5 O 12 P 1		

compound-8 ¹HNMR

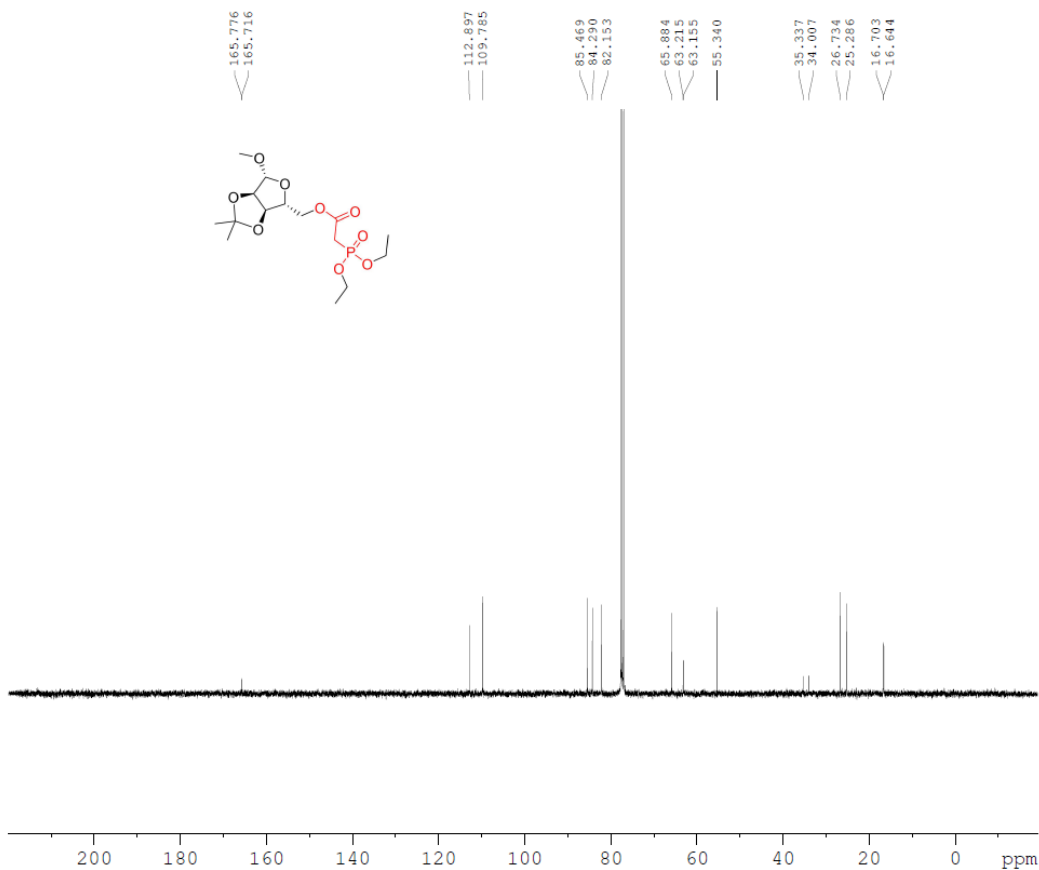


```

NAME      Jul17-2015-086088
EXPNO    10
PROCNO   1
Date_    20150717
Time     16.37
INSTRUM  AVIII400
PROBHD   5 mm PABBO BB-
PULPROG  zg30
TD       65536
SOLVENT  CDCl3
NS       16
DS       2
SWH      8223.685 Hz
FIDRES   0.125483 Hz
AQ       3.9846387 sec
RG       144
DW       60.800 usec
DE       17.48 usec
TE       295.2 K
D1       1.00000000 sec
TD0      1

===== CHANNEL f1 =====
NUC1     1H
P1       11.90 usec
PL1     -1.00 dB
PL1W    12.26963711 W
SFO1    400.0424704 MHz
SI       65536
SF      400.0399813 MHz
WDW      EM
SSB      0
LB       0.20 Hz
GB       0
PC       1.00
    
```

compound-8 ¹³CNMR



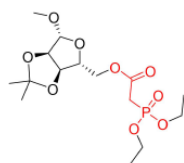
```

NAME      Jul17-2015-086088
EXPNO    13
PROCNO   1
Date_    20150718
Time     2.00
INSTRUM  AVIII400
PROBHD   5 mm PABBO BB-
PULPROG  zgpg30
TD       65536
SOLVENT  CDCl3
NS       512
DS       4
SWH      24038.461 Hz
FIDRES   0.366798 Hz
AQ       1.3631988 sec
RG       1620
DW       20.800 usec
DE       6.50 usec
TE       295.2 K
D1       2.00000000 sec
D11      0.03000000 sec
TD0      1

===== CHANNEL F1 =====
NUC1     13C
P1       9.25 usec
PL1     -2.00 dB
PL1W    58.91986484 W
SFO1    100.6001970 MHz

===== CHANNEL F2 =====
CPDPRG2  waltz16
NUC2     1H
PCPD2    80.00 usec
PL2     -1.00 dB
PL12    15.55 dB
PL13    19.00 dB
PL1W    12.26963711 W
SFO2    0.27153867 W
PL13W   0.12269637 W
SFO2    400.0416002 MHz
SI       65536
SF      100.5900987 MHz
WDW      RM
SSB      0
LB       1.00 Hz
GB       0
PC       1.40
    
```

compound-8 ³¹P NMR



```

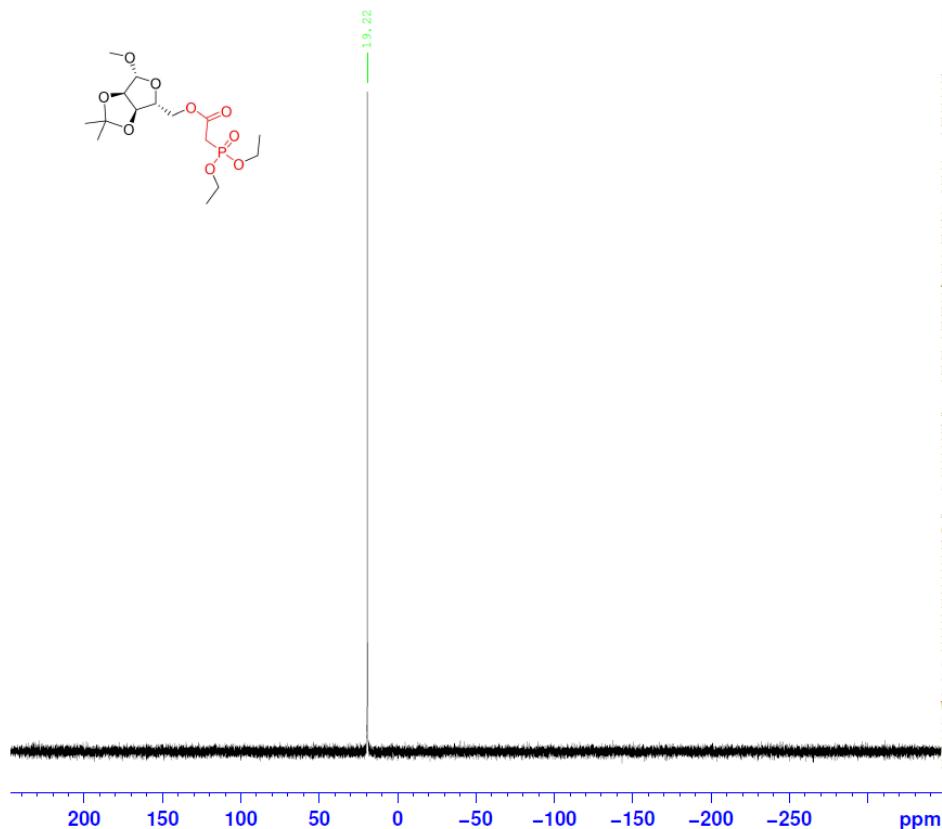
NAME      Jul17-2015-OB6088
EXPNO     11
PROCNO    1
Date_     20150717
Time      16.40
INSTRUM   AVIII400
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD         65536
SOLVENT   CDC13
NS         64
DS         4
SWH       96153.844 Hz
FIDRES    1.467191 Hz
AQ         0.3408372 sec
RG         2050
DW         5.200 usec
DE         6.50 usec
TE         295.2 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1
    
```

```

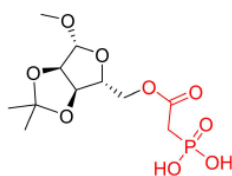
===== CHANNEL f1 =====
NUC1      31P
P1         9.40 usec
PL1        0.00 dB
PL1W       23.83780289 W
SFO1      161.9310633 MHz
    
```

```

===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2       1H
PCPD2     80.00 usec
PL2        -1.00 dB
PL12       15.55 dB
PL13       19.00 dB
PL12W     12.26963711 W
PL13W     0.27153867 W
PL13W     0.12269637 W
SFO2      400.0416002 MHz
SI         32768
SF         161.9391600 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.40
    
```



compound-9 ¹H NMR

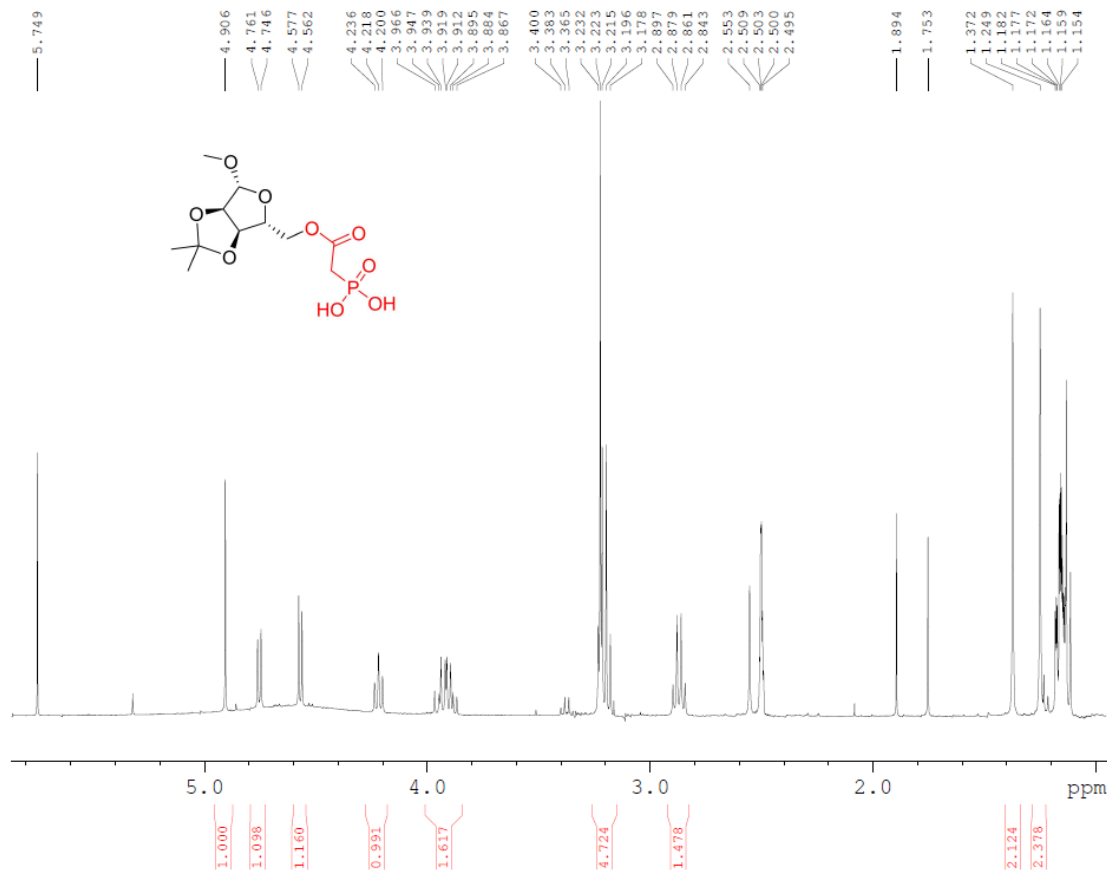


```

NAME      Aug10-2015-OB6081
EXPNO     10
PROCNO    1
Date_     20150810
Time      11.22
INSTRUM   AVIII400
PROBHD    5 mm PABBO BB-
PULPROG   zg30
TD         65536
SOLVENT   DMSO
NS         16
DS         2
SWH       8223.685 Hz
FIDRES    0.125483 Hz
AQ         3.9846387 sec
RG         114
DW         60.800 usec
DE         17.48 usec
TE         295.2 K
D1         1.00000000 sec
TD0        1
    
```

```

===== CHANNEL f1 =====
NUC1      1H
P1         11.90 usec
PL1        -1.00 dB
PL1W       12.26963711 W
SFO1      400.0424704 MHz
SI         65536
SF         400.0400070 MHz
WDW        EM
SSB        0
LB         0.20 Hz
GB         0
PC         1.00
    
```



compound-9 ¹³CNMR

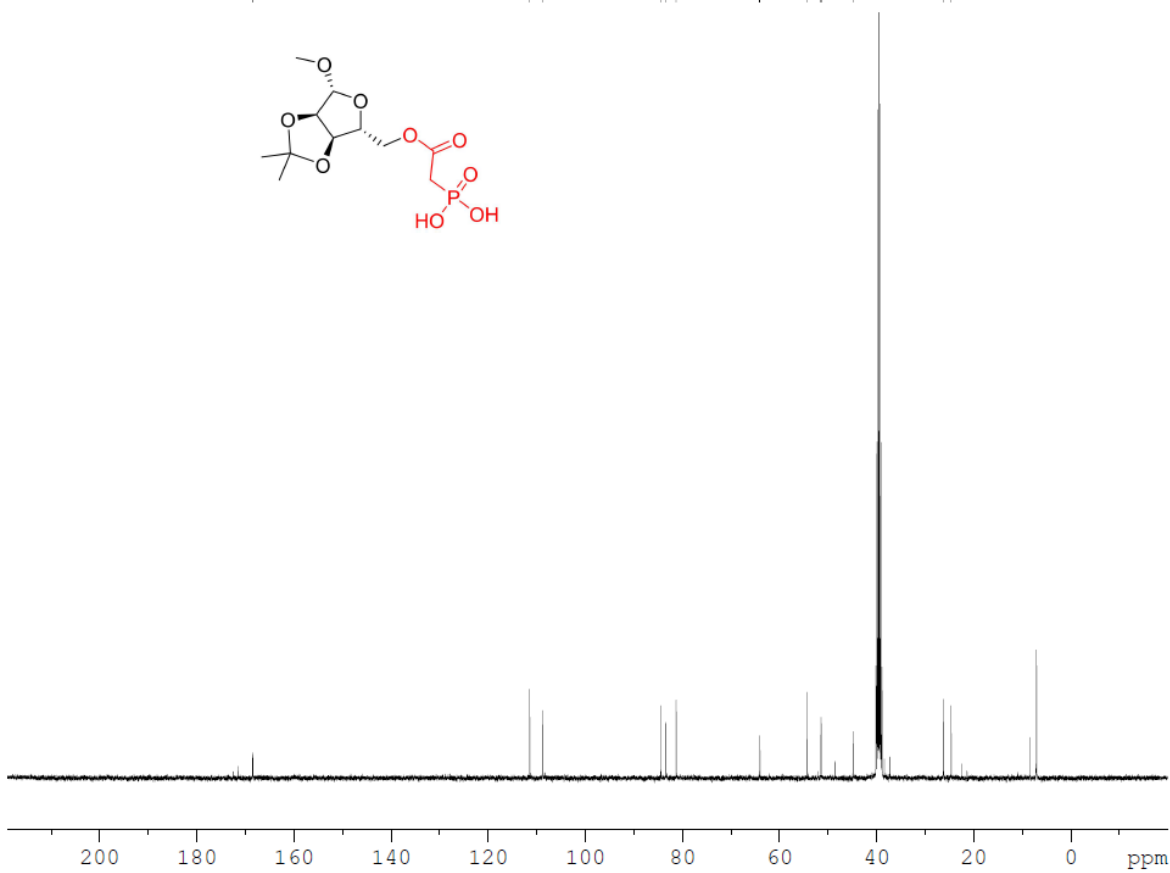
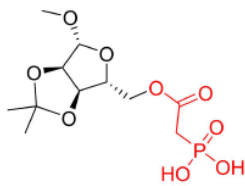
166.537
166.472

111.470
108.763

84.470
83.403
81.276

64.054
54.285
51.475
51.445
51.415
44.844

26.224
24.656



```

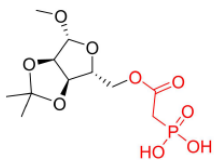
NAME      Dec19-2015_OBB14_RR
EXPNO     12
PROCNO    1
Date_     20151219
Time      18.16
INSTRUM   AVIII400
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD         65536
SOLVENT   DMSO
NS         1024
DS         4
SWH        24038.461 Hz
FIDRES     0.366798 Hz
AQ         1.3631988 sec
RG         1620
DW         20.800 usec
DE         6.50 usec
TE         298.2 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1

===== CHANNEL f1 =====
NUC1       13C
P1         9.25 usec
PL1        -2.00 dB
PL1W       58.91986084 W
SFO1       100.6001970 MHz

===== CHANNEL f2 =====
CPDPRG2    waltz16
NUC2        1H
PCPD2       80.00 usec
PL2         -1.00 dB
PL12        15.55 dB
PL13        19.00 dB
PL2W       12.26963711 W
PL12W       0.27153867 W
PL13W       0.12269637 W
SFO2       400.0416002 MHz
SI          65536
SF         100.5901854 MHz
WDW         EM
SSB         0
LB          1.00 Hz
GB          0
PC          1.40
    
```

compound-9 ³¹P NMR

12.47
12.39
8.36



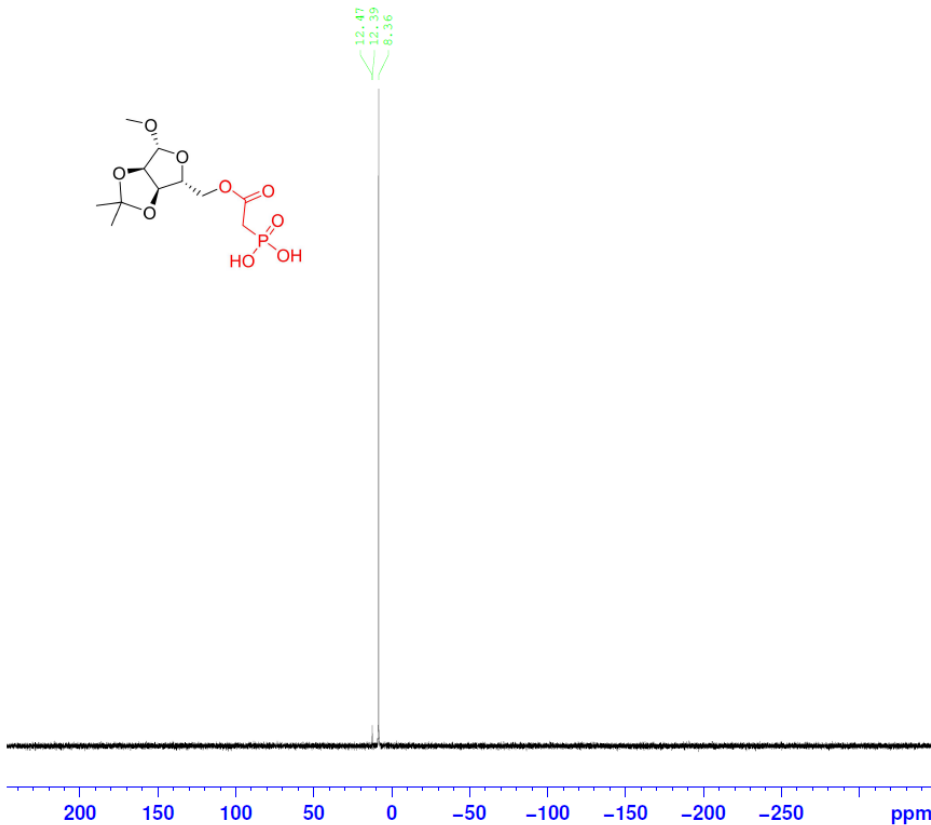
```

NAME      Aug10-2015-OB6081
EXPNO     11
PROCNO    1
Date_     20150810
Time      11.26
INSTRUM   AVIII400
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD         65536
SOLVENT   DMSO
NS         64
DS         4
SWH        96153.844 Hz
FIDRES     1.467191 Hz
AQ         0.3408372 sec
RG         2050
DW         5.200 usec
DE         6.50 usec
TE         295.2 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1
    
```

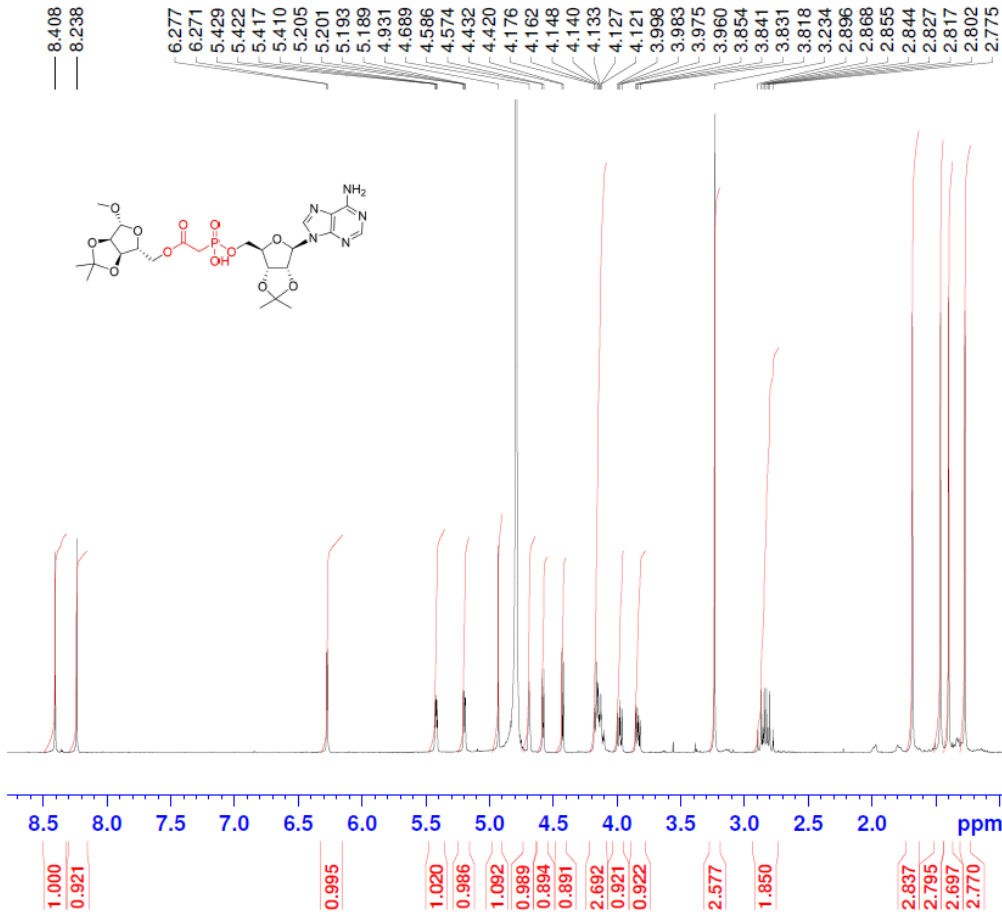
```

===== CHANNEL f1 =====
NUC1       31P
P1         9.40 usec
PL1         0.00 dB
PL1W       23.83780289 W
SFO1       161.9310633 MHz

===== CHANNEL f2 =====
CPDPRG2    waltz16
NUC2        1H
PCPD2       80.00 usec
PL2         -1.00 dB
PL12        15.55 dB
PL13        19.00 dB
PL2W       12.26963711 W
PL12W       0.27153867 W
PL13W       0.12269637 W
SFO2       400.0416002 MHz
SI          32768
SF         161.9391600 MHz
WDW         EM
SSB         0
LB          1.00 Hz
GB          0
PC          1.40
    
```



compound-10 ¹H NMR

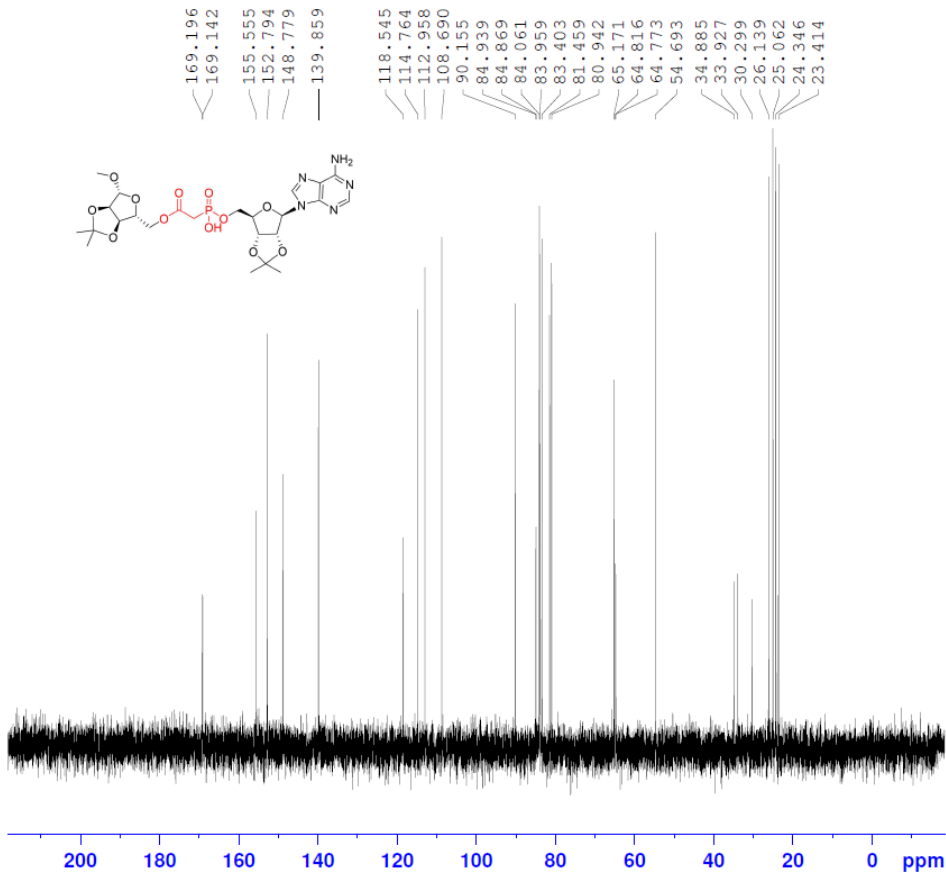


```

NAME          OBB-24
EXPNO         10
PROCNO        1
Date_         20151021
Time         11.51
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zg30
ID            65536
SOLVENT       D2O
NS            16
DS            2
SWH           10330.578 Hz
FIDRES        0.157632 Hz
AQ            3.1719923 sec
RG            114
DW            48.400 usec
DE            14.00 usec
TE            298.2 K
D1            1.00000000 sec
TD0           1

===== CHANNEL f1 =====
NUC1          1H
P1            10.00 usec
PL1           -0.12 dB
PL1W          19.35150909 W
SFO1          500.1330885 MHz
SI            32768
SF            500.1299569 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
    
```

compound-10 ¹³C NMR



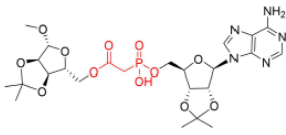
```

NAME          OBB-24
EXPNO         13
PROCNO        1
Date_         20151021
Time         13.04
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zgpg30
ID            65536
SOLVENT       D2O
NS            2227
DS            4
SWH           29761.904 H:
FIDRES        0.454131 H:
AQ            1.1010548 s:
RG            2050
DW            16.800 u:
DE            8.45 u:
TE            298.3 K
D1            2.00000000 s:
D11           0.03000000 s:
TD0           1

===== CHANNEL f1 =====
NUC1          13C
P1            9.40 u:
PL1           -0.51 dF
PL1W          99.92730713 W
SFO1          125.7703643 M:

===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2          1H
PCPD2         80.00 u:
PL2           -0.12 dF
PL12          17.94 dF
PL13          21.00 dF
PL2W          19.35150909 W
PL12W         0.30249262 W
PL13W         0.14952536 W
SFO2          500.1320005 M:
SI            32768
SF            125.7577890 M:
WDW           EM
SSB           0
LB            1.00 H:
GB            0
PC            1.40
    
```

compound-10 ³¹PNMR



14.11

```

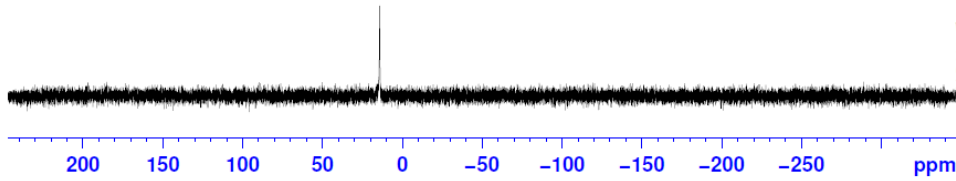
NAME      Dec08-2015-0BB24_PUR
EXPNO     11
PROCNO    1
Date_     20151208
Time      16.43
INSTRUM   AVII1400
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD         65536
SOLVENT   D2O
NS         64
DS         4
SWH        96153.844 Hz
FIDRES     1.467191 Hz
AQ         0.3408372 sec
RG         2050
DW         5.200 usec
DE         6.50 usec
TE         298.2 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1
    
```

```

===== CHANNEL f1 =====
NUC1      31P
P1        9.40 usec
PL1       0.00 dB
PL1W      23.83780289 W
SFO1      161.9310633 MHz
    
```

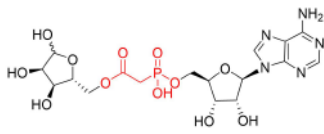
```

===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2      1H
PCPD2     80.00 usec
PL2       -1.00 dB
PL12      15.55 dB
PL13      19.00 dB
PL2W      12.26963711 W
PL12W     0.27153867 W
PL13W     0.12269637 W
SFO2      400.0416002 MHz
SI         32768
SF         161.9391600 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.40
    
```



compound-2 ¹H NMR (DOSY)

8.365 8.148 6.043 6.032 5.106 5.103 4.683 4.673 4.662 4.430 4.427 4.420 4.412 4.295 4.231 4.224 4.167 4.158 4.154 4.144 4.115 4.091 4.085 4.082 4.073 4.049 3.987 3.975 3.971 3.967 3.895 3.892 3.886 3.883 3.130 3.115 3.101 3.086 2.866 2.859 2.842 2.825 2.818 2.801 1.201 1.186 1.172



```

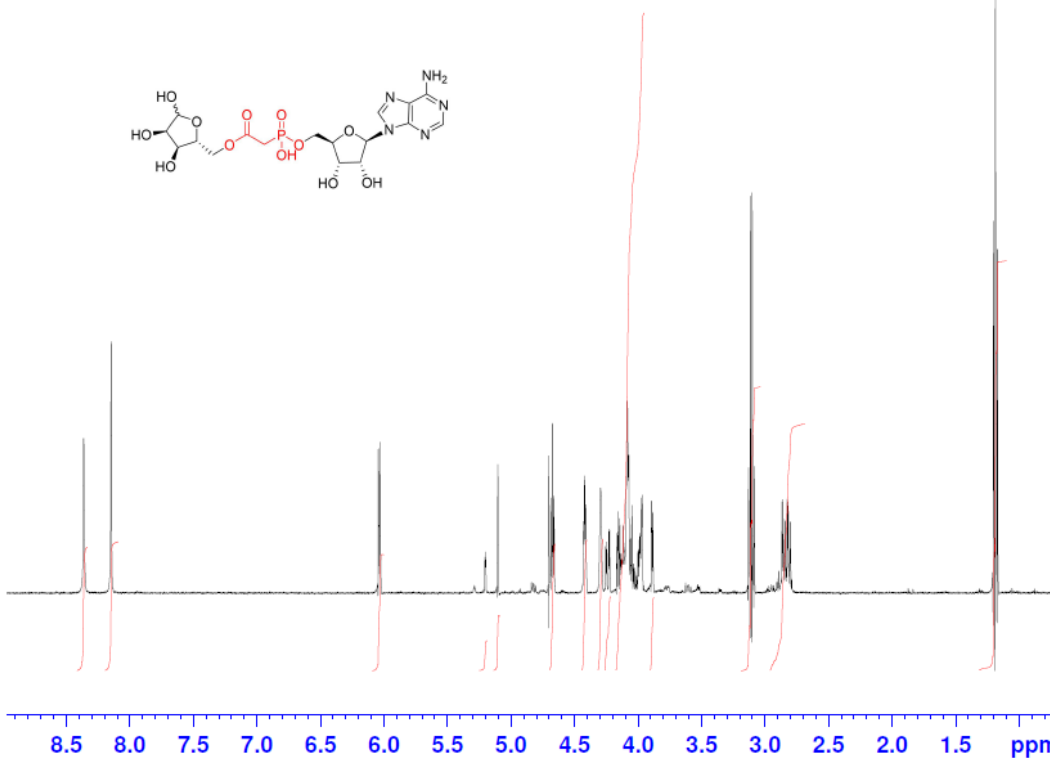
NAME      OBB46
EXPNO     50
PROCNO    1
Date_     20151218
Time      11.50
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   ledbpgp2s1d
TD         65536
SOLVENT   D2O
NS         16
DS         2
SWH        10330.578 Hz
FIDRES     0.157632 Hz
AQ         3.1719923 sec
RG         22.6
DW         48.400 usec
DE         9.73 usec
TE         298.2 K
D1         1.00000000 sec
D16        0.00020000 sec
D20        0.05000000 sec
D21        0.00500000 sec
TD0        1
    
```

```

===== CHANNEL f1 =====
NUC1      1H
P1        10.00 usec
P2        20.00 usec
PL1       -0.12 dB
PL1W      19.35150909 W
SFO1      500.1330885 MHz
    
```

```

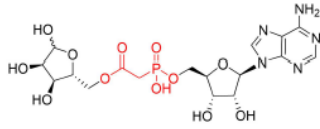
===== GRADIENT CHANNEL =====
GPAM6     sine.100
GPAM7     sine.100
    
```



1.068 1.112 1.000 0.256 0.467 1.098 1.128 1.136 0.638 5.690 0.629 2.454 2.135 3.545

compound-2 ¹³CNMR

169.715
155.524
152.768
149.089
139.740
118.692
101.183
96.336
87.022
83.757
79.827
79.437
74.931
74.203
70.503
70.281
70.100
65.304
64.581
63.839
46.634
35.138
34.175



```

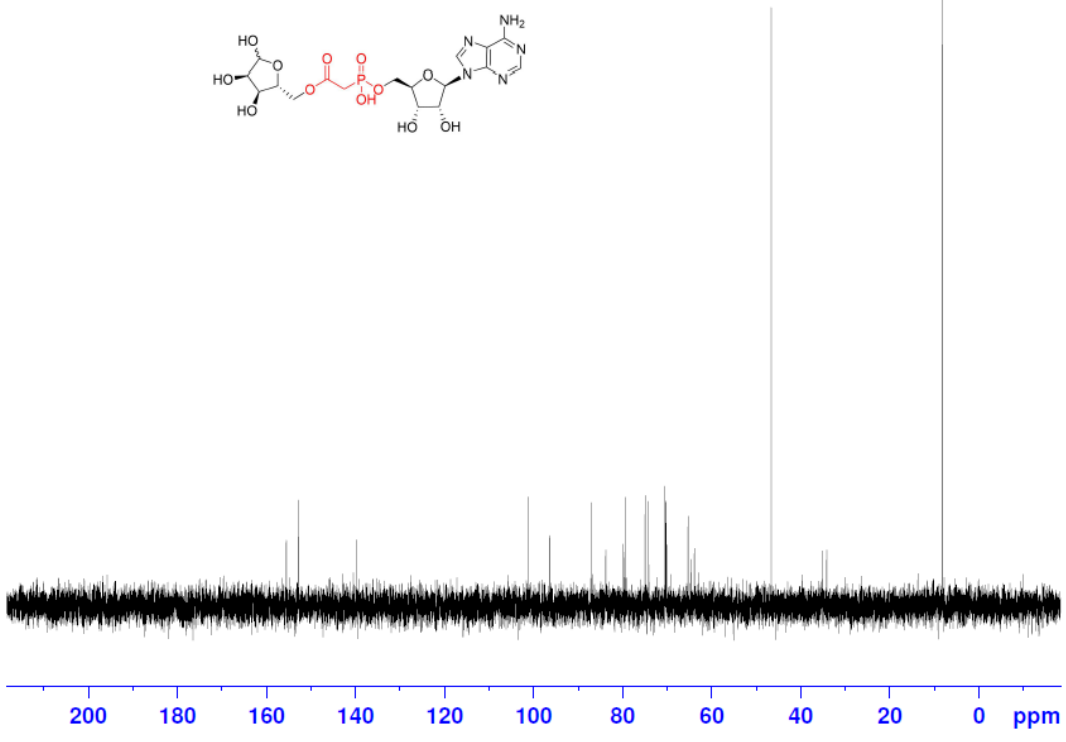
NAME OBB46
EXPNO 22
PROCNO 1
Date_ 20151218
Time 9.41
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT D2O
NS 512
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 2050
DW 16.800 usec
DE 8.45 usec
TE 298.2 K
D1 2.0000000 sec
D11 0.0300000 sec
TD0 1
    
```

```

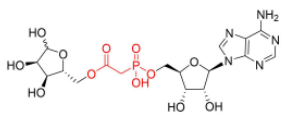
===== CHANNEL f1 =====
NUC1 13C
P1 9.40 usec
PL1 -0.51 dB
PL1W 99.92730713 W
SFO1 125.7703643 MHz
    
```

```

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -0.12 dB
PL12 17.94 dB
PL13 21.00 dB
PL2W 19.35150909 W
PL12W 0.30249262 W
PL13W 0.14952536 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7577890 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
    
```



compound-2 ³¹P NMR



14.61
14.36



```

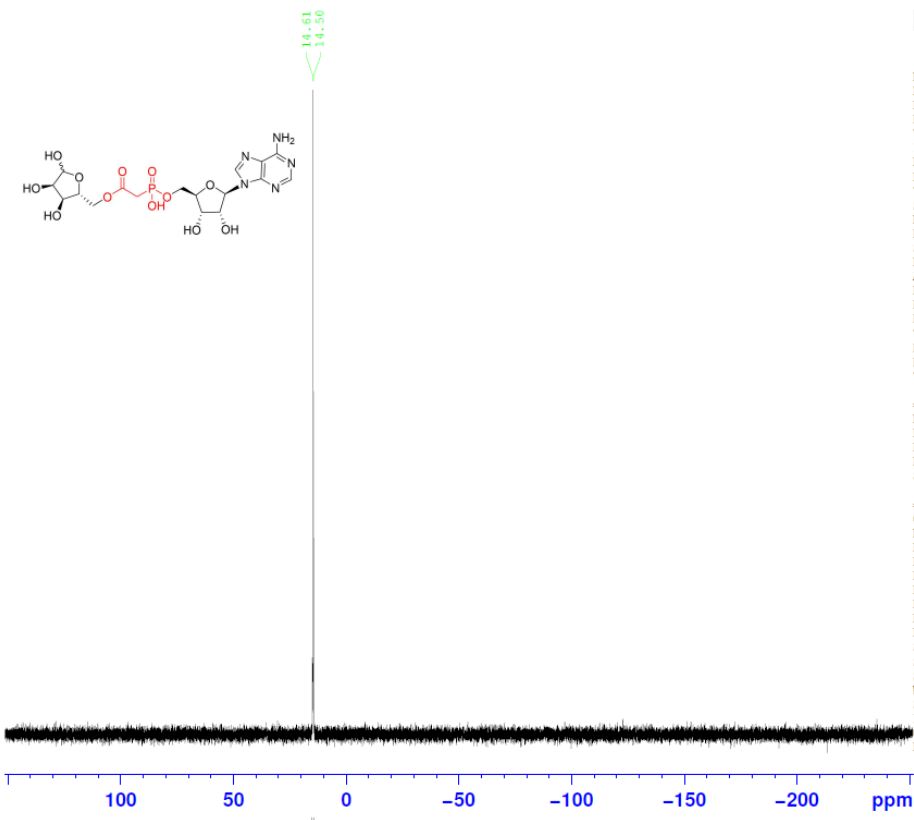
NAME OBB46
EXPNO 24
PROCNO 1
Date_ 20151218
Time 10.09
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT D2O
NS 256
DS 4
SWH 81521.742 Hz
FIDRES 1.243923 Hz
AQ 0.4020041 sec
RG 2050
DW 6.133 usec
DE 6.50 usec
TE 298.2 K
D1 2.0000000 sec
D11 0.0300000 sec
TD0 1
    
```

```

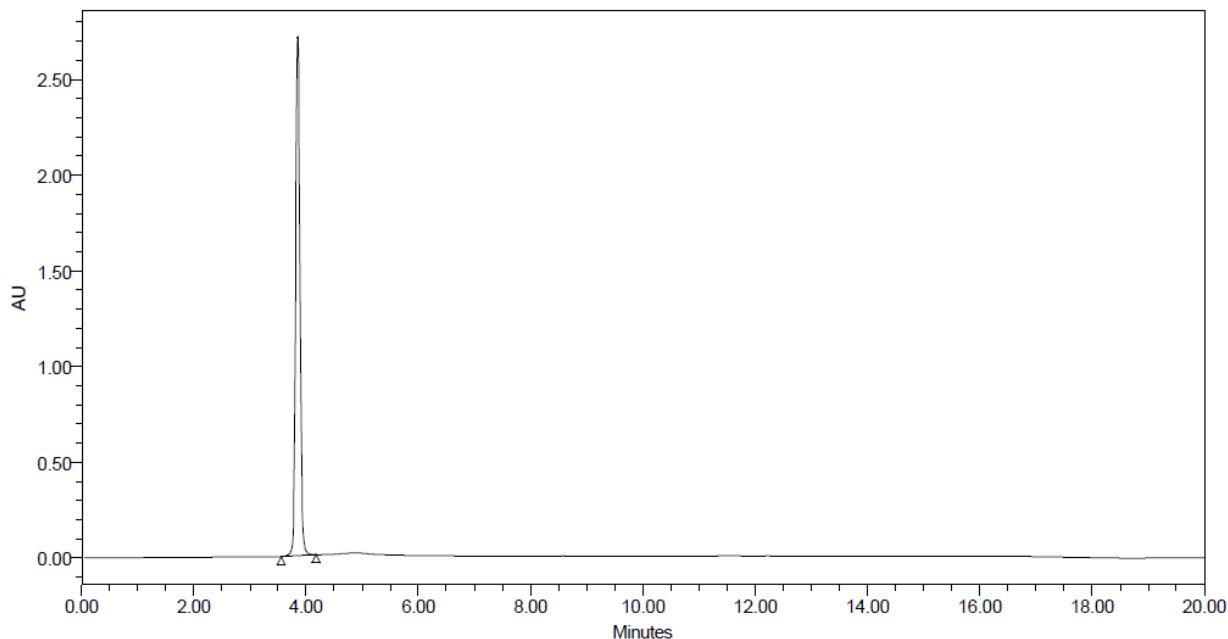
===== CHANNEL f1 =====
NUC1 31P
P1 10.50 usec
PL1 0.00 dB
PL1W 81.20777893 W
SFO1 202.4462121 MHz
    
```

```

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -0.12 dB
PL12 17.94 dB
PL13 21.00 dB
PL2W 19.35150909 W
PL12W 0.30249262 W
PL13W 0.14952536 W
SFO2 500.1320005 MHz
SI 32768
SF 202.4563350 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
    
```



compound-2 HPLC



	Peak Name	RT	Area	% Area	Height
1	compound-2	3.854	14751722	100.00	2724648

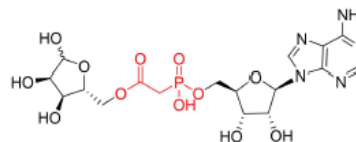
Confirmation of Expected Formula

Sample-ID	compound-2	Submitter	Ondrej Baszczynski
Analysis Name	po_ob_obb45_345635_44_01_50211.d	Supervisor	Barry Potter
Method used	Confirm Formula Negative 50to1500 loop inj.m	Acquisition Date	26/11/2015 10:36:59
Ionisation Mode	negative electrospray (ESI)		

-MS, 1.0-1.3min #(32-40), -Spectral Bkgrnd



#	m/z	I	I %	Area	S/N
1	212.0876	3329	33.3	42	71219.7
2	520.1086	10006	100.0	790	16233.1
3	521.1114	2135	21.3	164	3467.2
4	522.1139	446	4.5	32	725.4
5	542.0918	1811	18.1	155	3018.5
6	543.0931	359	3.6	30	599.9
7	564.0744	837	8.4	73	1434.8
8	670.0365	314	3.1	29	1168.4
9	1063.2048	361	3.6	49	4160.5
10	1085.1838	266	2.7	35	3488.5



Generate Molecular Formula Parameters

Charge	Tolerance	SearchRadius	H/C Ratio min.	H/C Ratio max.	Electron Conf.	Nitrogen Rule	sigma limit
negative	10 ppm	0.05 m/z	0	3	both	true	0.05

Expected Formula C17 H24 N5 O12 P Adduct(s): H, Na

#	meas. m/z	theo. m/z	Err[ppm]	Sigma	Formula
1	520.1086	520.108083	0.20	0.0042	C 17 H 23 N 5 O 12 P 1