Supplementary Information

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

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Contents:

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

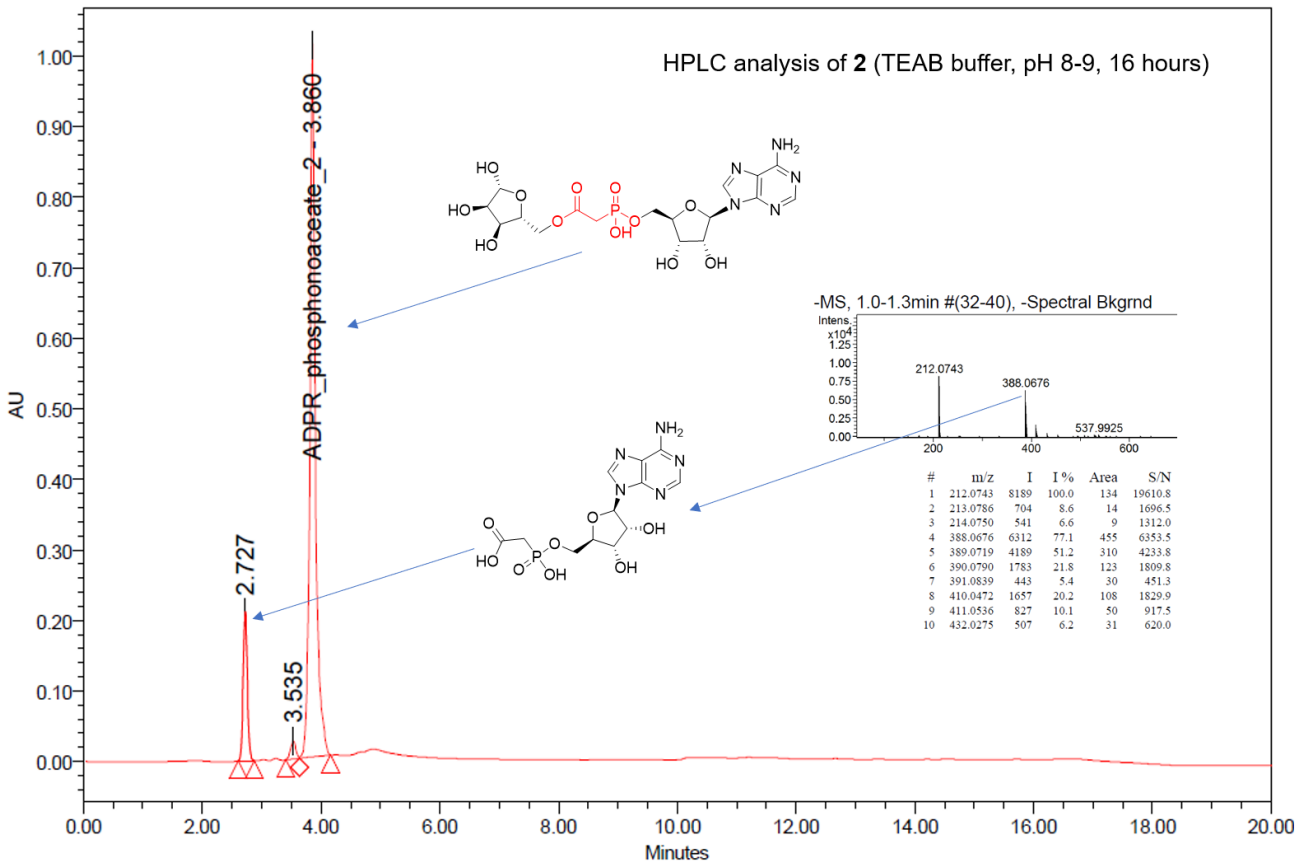
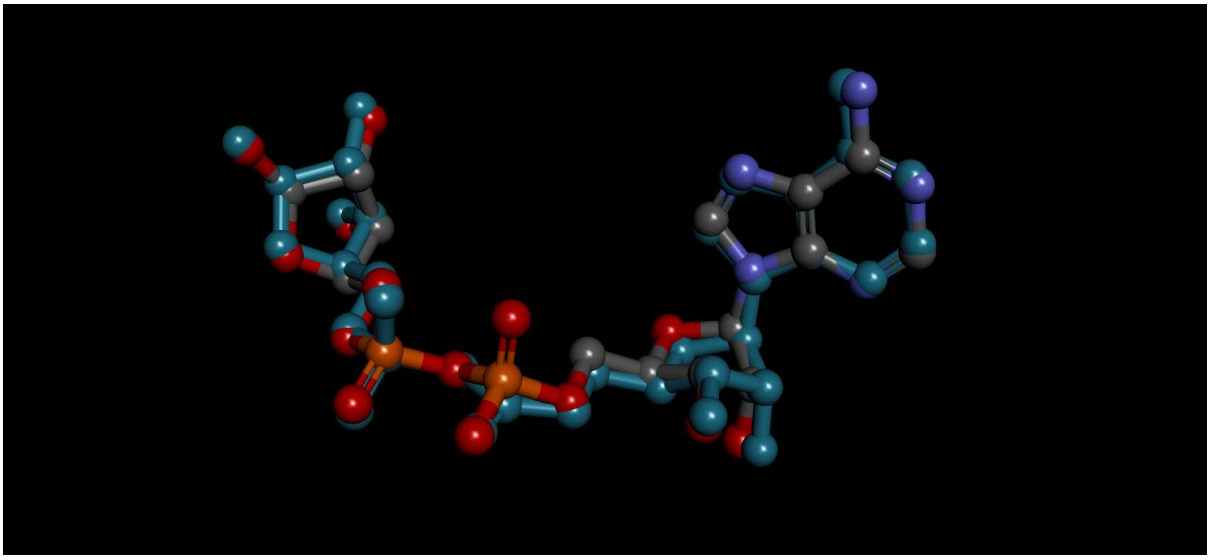
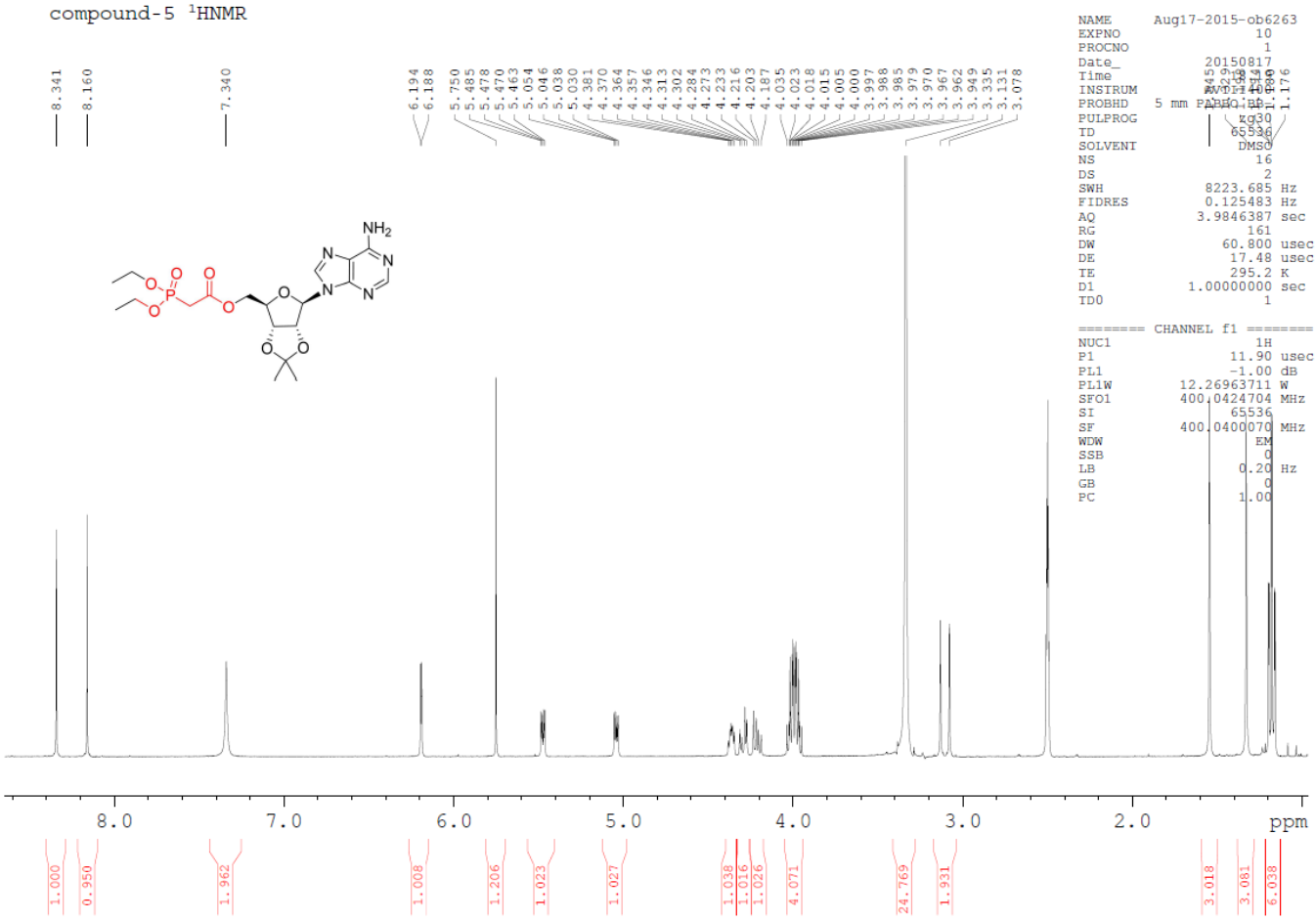


Figure S2. Comparison of phosphonoacetate analogue **1** with ADPR



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

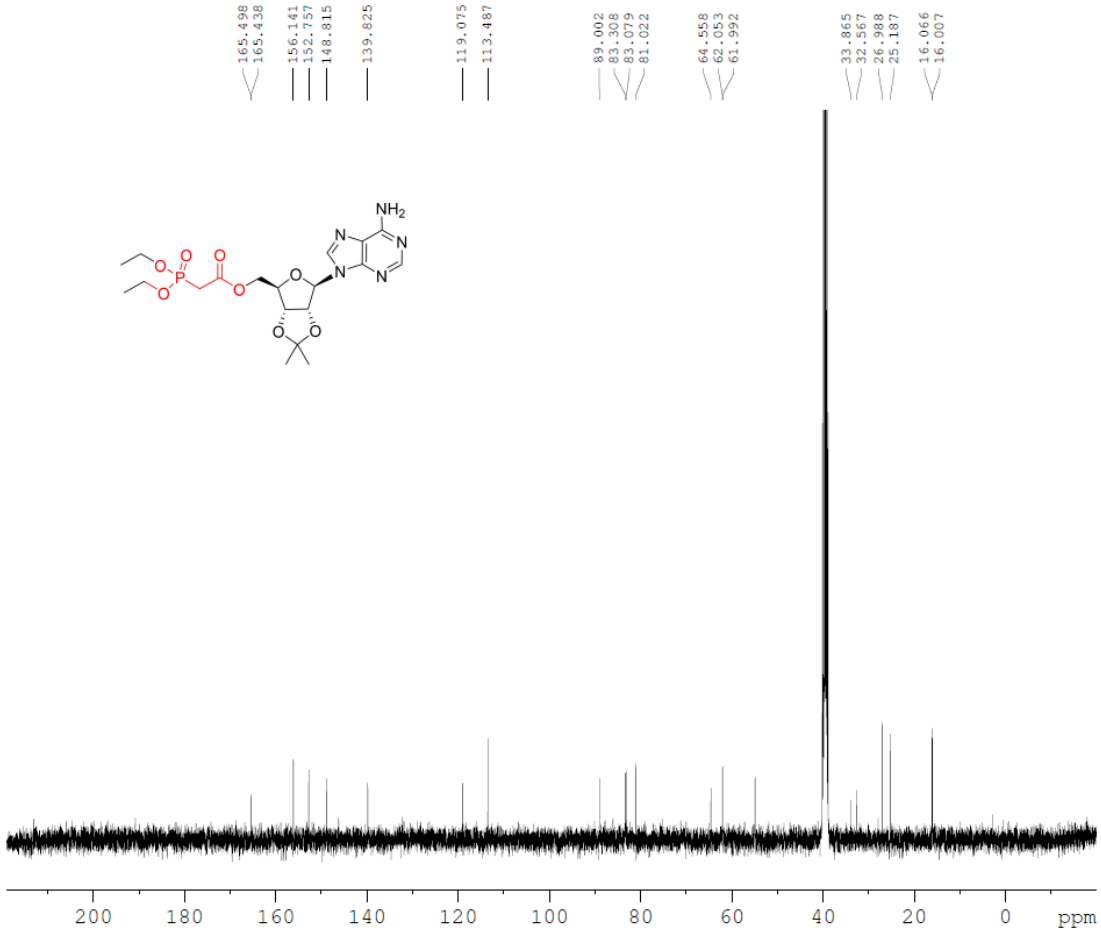
compound-5 ^{1}HNMR




```
NAME        Aug17-2015-ob6263
EXPNO                     10
PROCNO                     1
Date_               20150817
Time                    8.45
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
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 ^{13}CNMR

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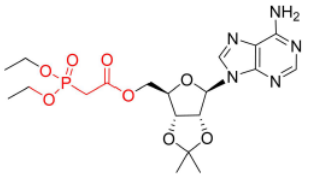
EXPNO                     14
PROCNO                     1
Date_               20150820
Time                    6.09
INSTRUM              AVIII400
PROBHD   5 mm PABBO BB-
PULPROG              zgpg30
TD                    65536
SOLVENT                DMSO
NS                       512
DS                         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              2.00000000 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             100.5901865 MHz
WDW                       EM
SSB                        0
LB                      1.00 Hz
GB                         0
PC                      1.40
```

compound-5 ^{31}PNMR

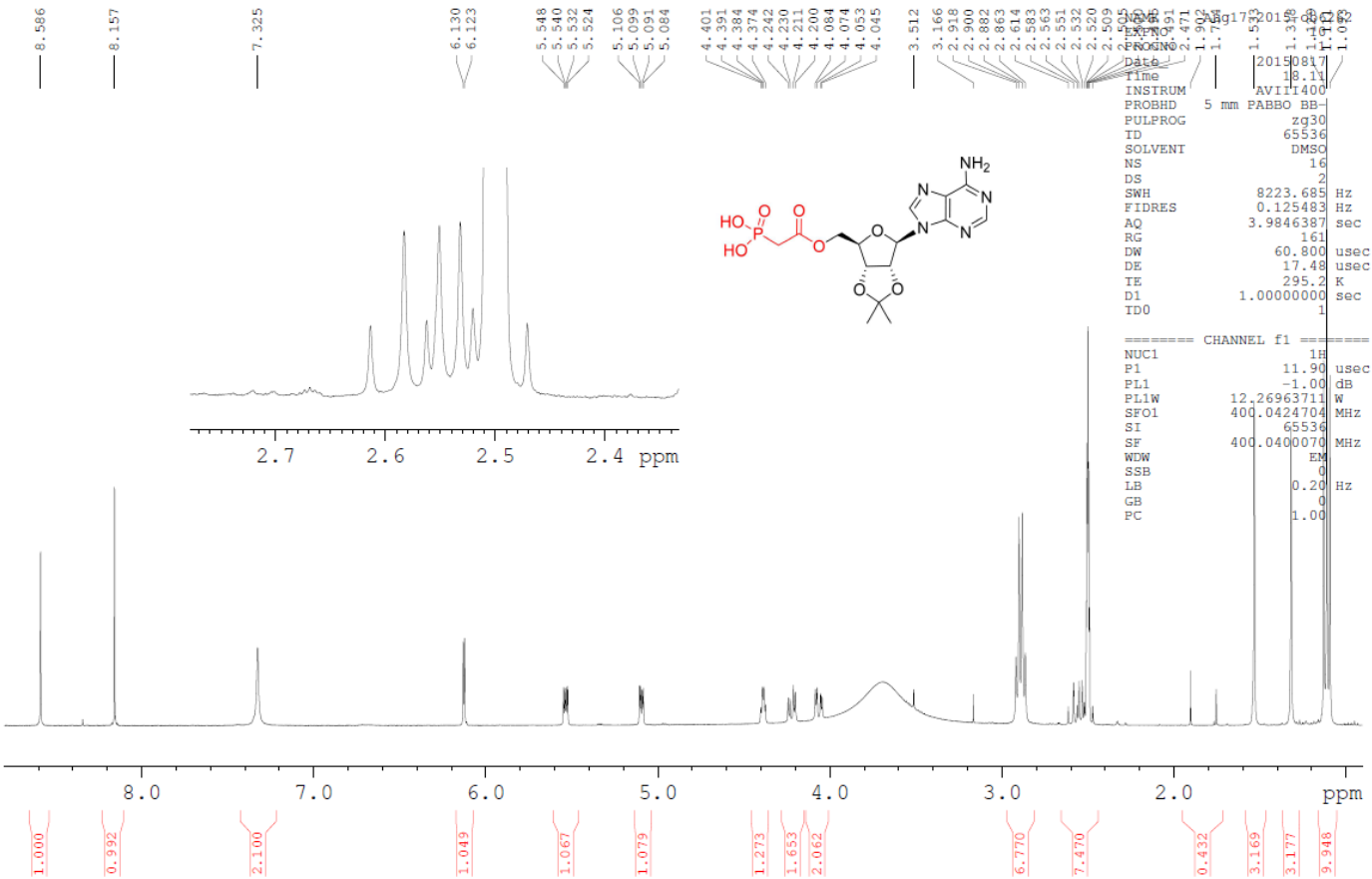



```
NAME          Aug17-2015-ob6263
EXPNO                       11
PROCNO                       1
Date_              20150817
Time                    18.23
INSTRUM              AVIII400
PROBHD     5 mm PABBO BB-
PULPROG                 zgpg30
TD                       65536
SOLVENT                  DMSO
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
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
```

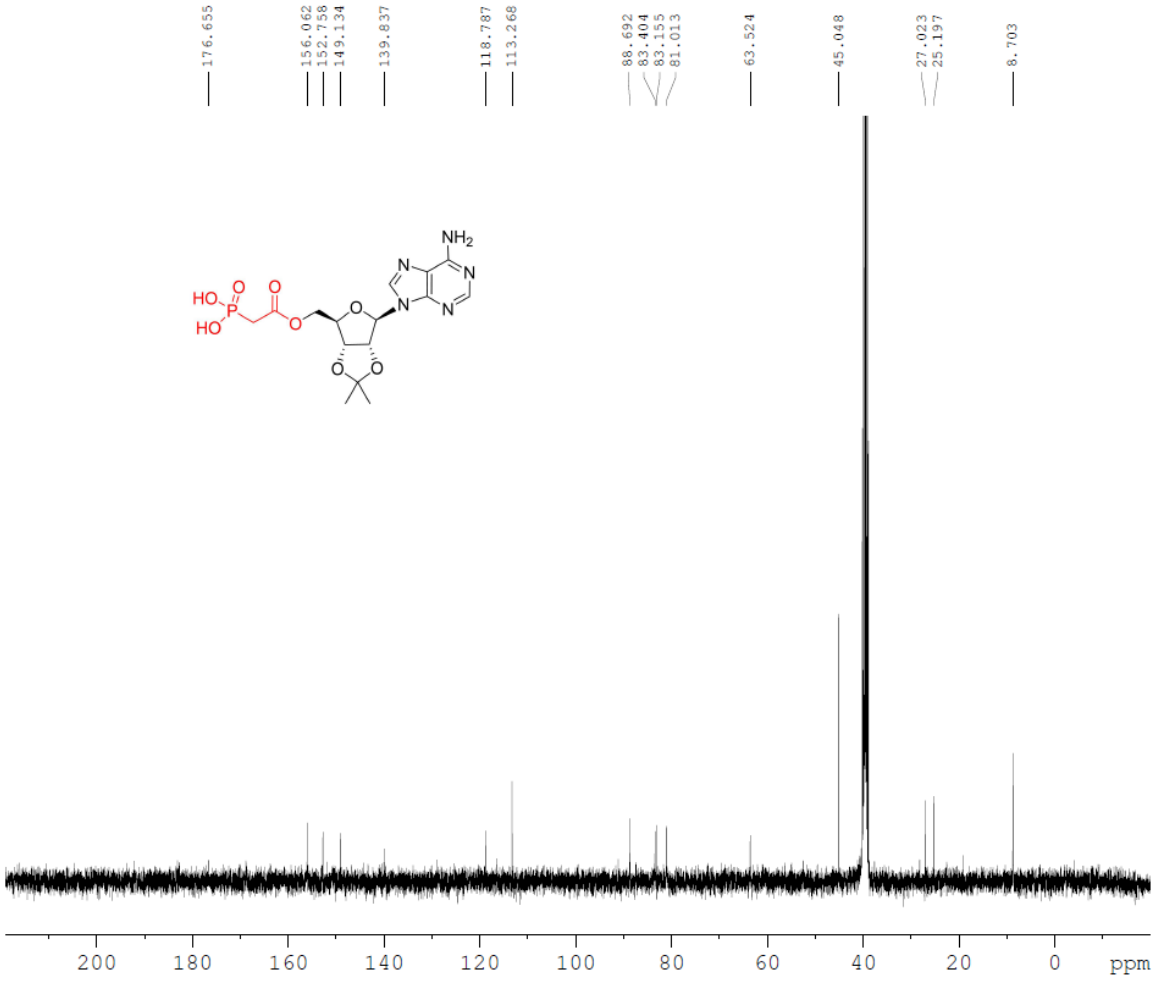
compound-6 ^{1}HNMR




```
NAME          Aug17-2015-ob6263
EXPNO                       12
PROCNO                       1
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
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-6 ^{13}CNMR

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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                    1.363198 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
PL2W                12.26963711 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
```
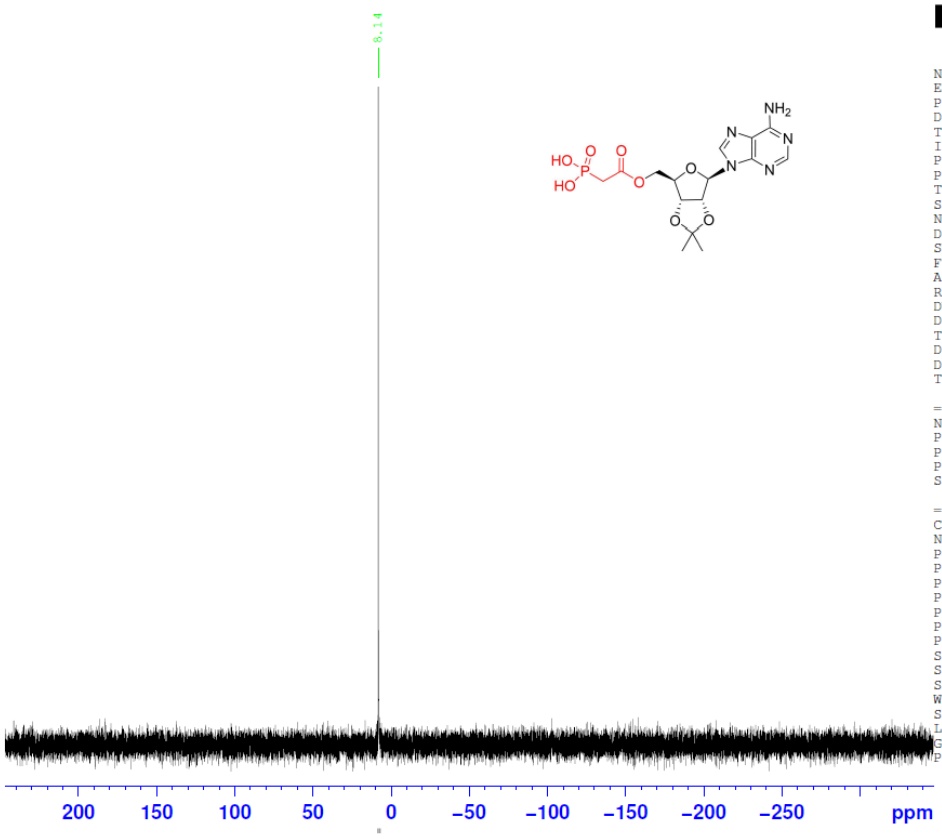
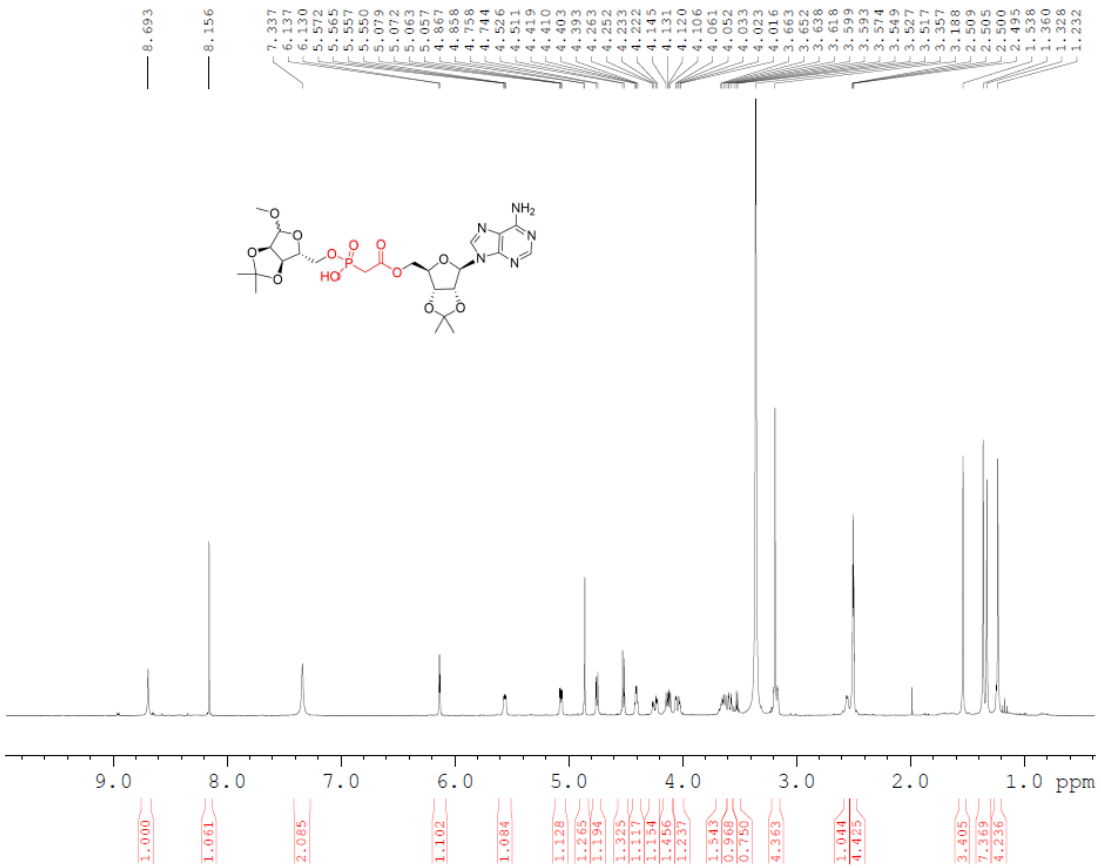

compound-6 ^{31}PNMR



BRUKER

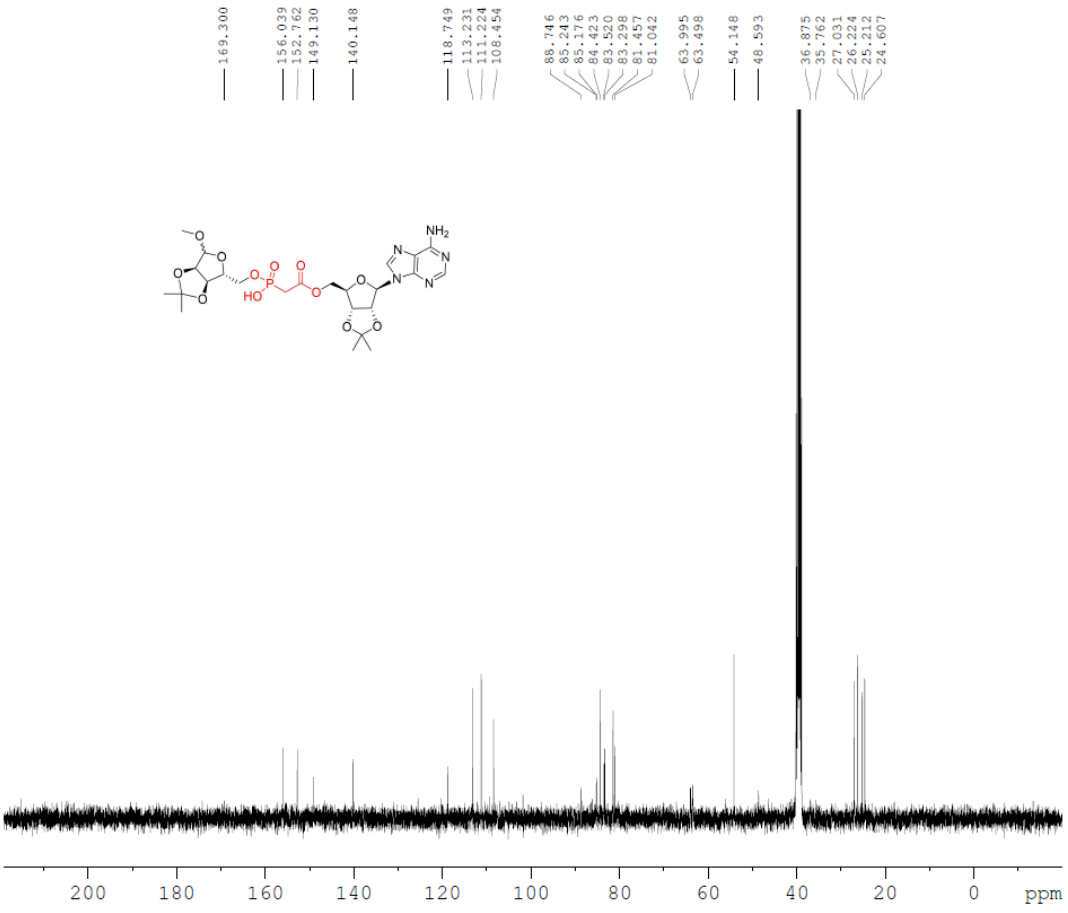
```
NAME          Aug17-2015-ob6262
EXPNO                       11
PROCNO                       1
Date_                 20150817
Time                     18.15
INSTRUM                AVIII400
PROBHD    5 mm PABBO BB-
PULPROG                 zgpg30
TD                       65536
SOLVENT                   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                  2.00000000 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-7 ^{1}HNMR



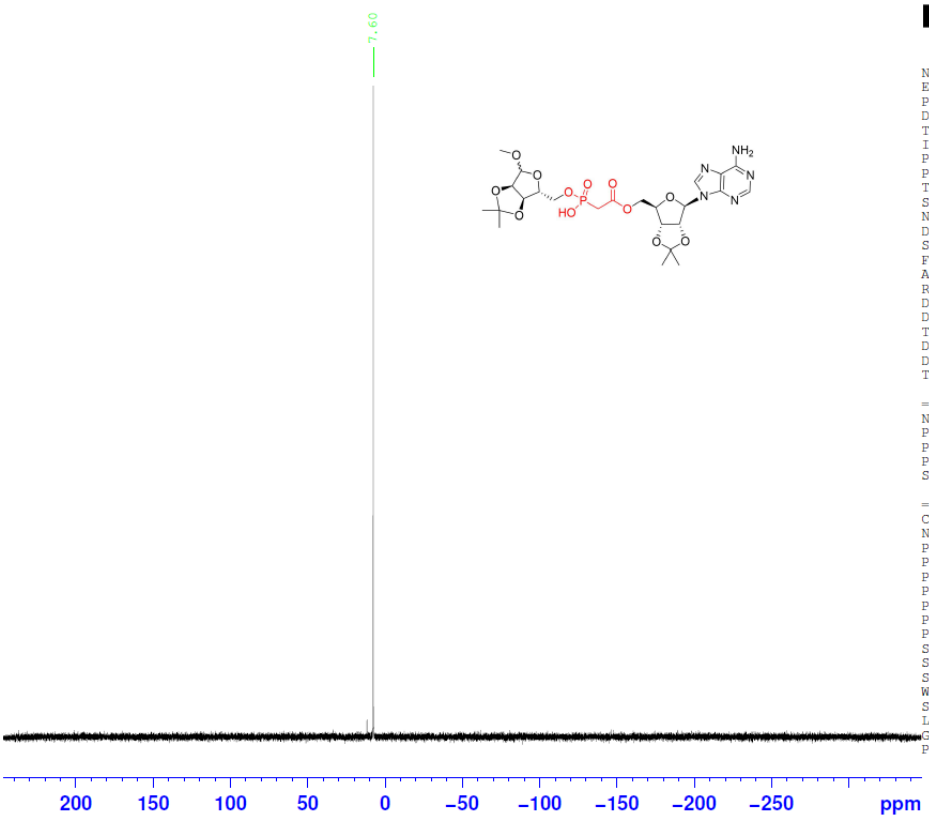

```
NAME        Aug17-2015-ob6261
EXPNO                      10
PROCNO                      1
Date_                20150817
Time                    18.03
INSTRUM              AVIII400
PROBHD    5 mm PABBO BB-
PULPROG                  zg30
TD                      65536
SOLVENT                  DMSO
NS                         16
DS                          2
SWH                  8223.685 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             400.0400070 MHz
WDW                        EM
SSB                         0
LB                   0.20 Hz
GB                          0
PC                       1.00
```

compound-7 ^{13}CNMR



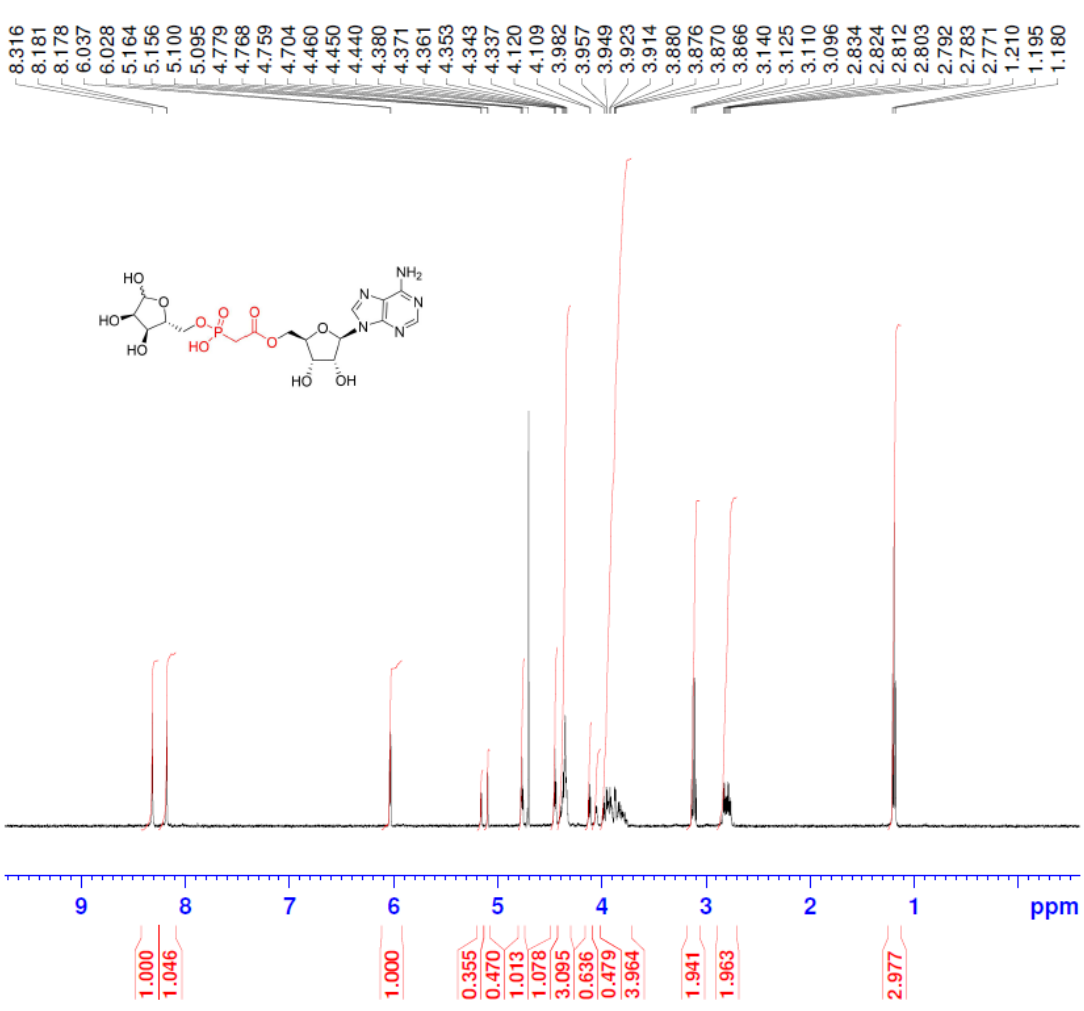

```
NAME        Aug17-2015-ob6261
EXPNO                      13
PROCNO                      1
Date_                20150817
Time                    21.31
INSTRUM              AVIII400
PROBHD    5 mm PABBO BB-
PULPROG                zgpg30
TD                      65536
SOLVENT                  DMSO
NS                        512
DS                          4
SWH                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                2.00000000 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             100.5901857 MHz
WDW                        EM
SSB                         0
LB                    1.00 Hz
GB                          0
PC                       1.40
```

compound-7 ^{31}PNMR



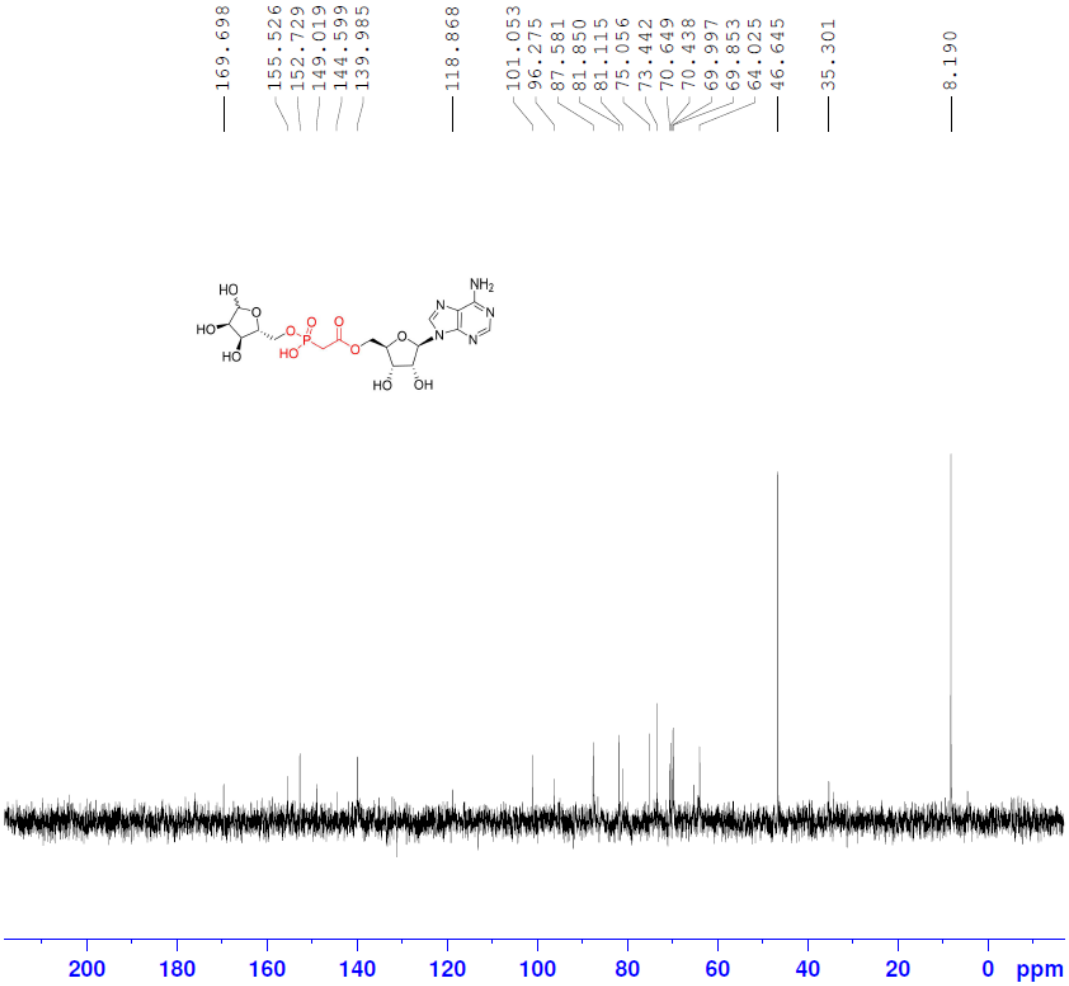

```
NAME          Aug17-2015-ob6261
EXPNO                        11
PROCNO                        1
Date_                  20150817
Time                      18.07
INSTRUM                AVIII400
PROBHD    5 mm PABBO BB-
PULPROG                 zgpg30
TD                       65536
SOLVENT                   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
SI                        32768
SF                  161.9391600 MHz
WDW                         EM
SSB                          0
LB                        1.00 Hz
GB                           0
PC                        1.40
```

compound-1 ^{1}HNMR (DOSY)



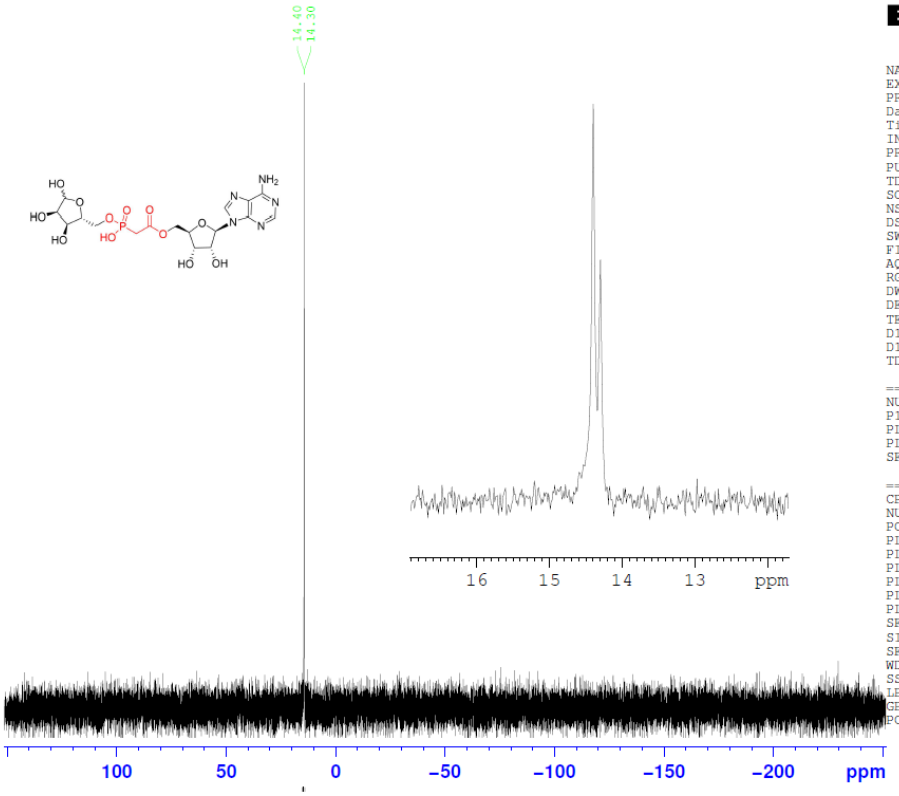

```
NAME                  OBB44
EXPNO                    15
PROCNO                    1
Date_              20151119
Time                  17.29
INSTRUM                spect
PROBHD    5 mm PABBO BB-
PULPROG     ledbpgp2s1d
TD                   65536
SOLVENT                D2O
NS                       16
DS                        2
SWH              10330.578 Hz
FIDRES            0.157632 Hz
AQ                3.1719923 sec
RG                      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

======== 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 ^{13}CNMR



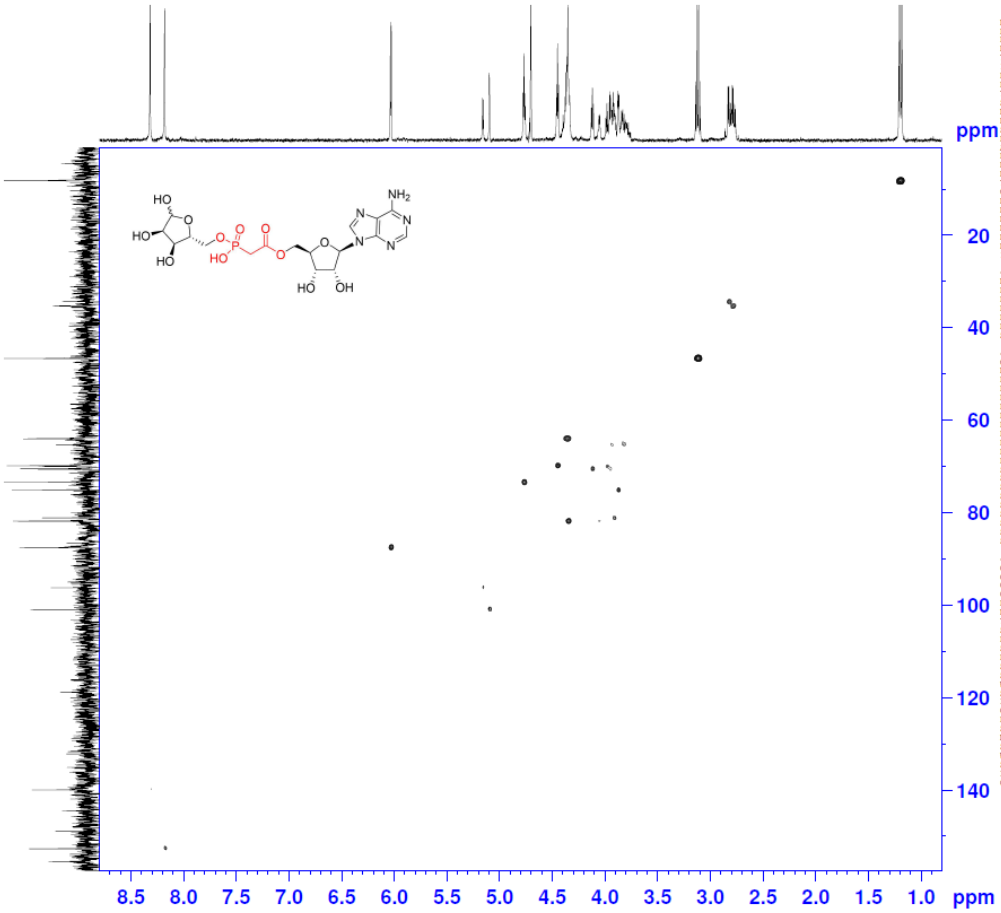

```
NAME          OBB44_more
EXPNO                 12
PROCNO                 1
Date_           20151119
Time               23.22
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT             D2O
NS                 4096
DS                    4
SWH           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            2.00000000 sec
D11           0.03000000 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 ^{31}PNMR



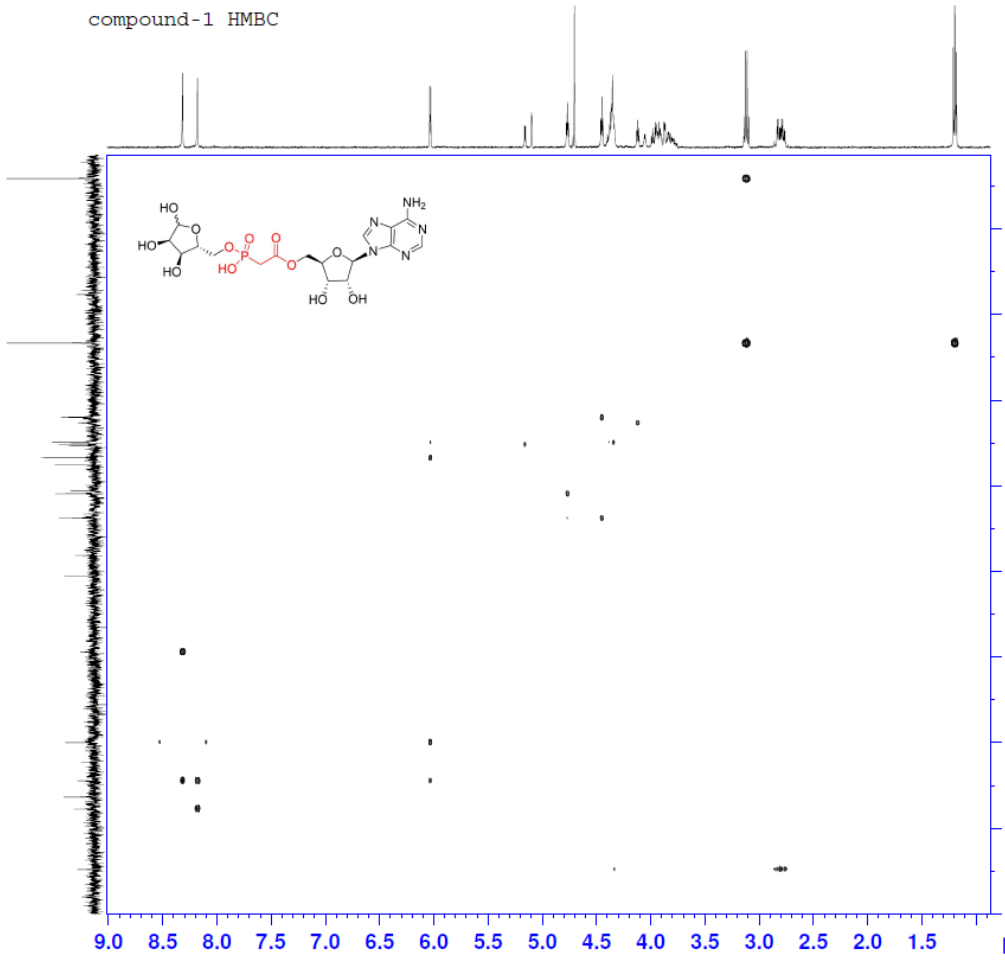

```
NAME      Nov18-2015-OBB7300
EXPNO                  15
PROCNO                  1
Date_            20151119
Time                 6.55
INSTRUM             spect
PROBHD    5 mm PABBO BB-
PULPROG           zgpg30
TD                 65536
SOLVENT              D2O
NS                   256
DS                     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            2.00000000 sec
D11           0.03000000 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

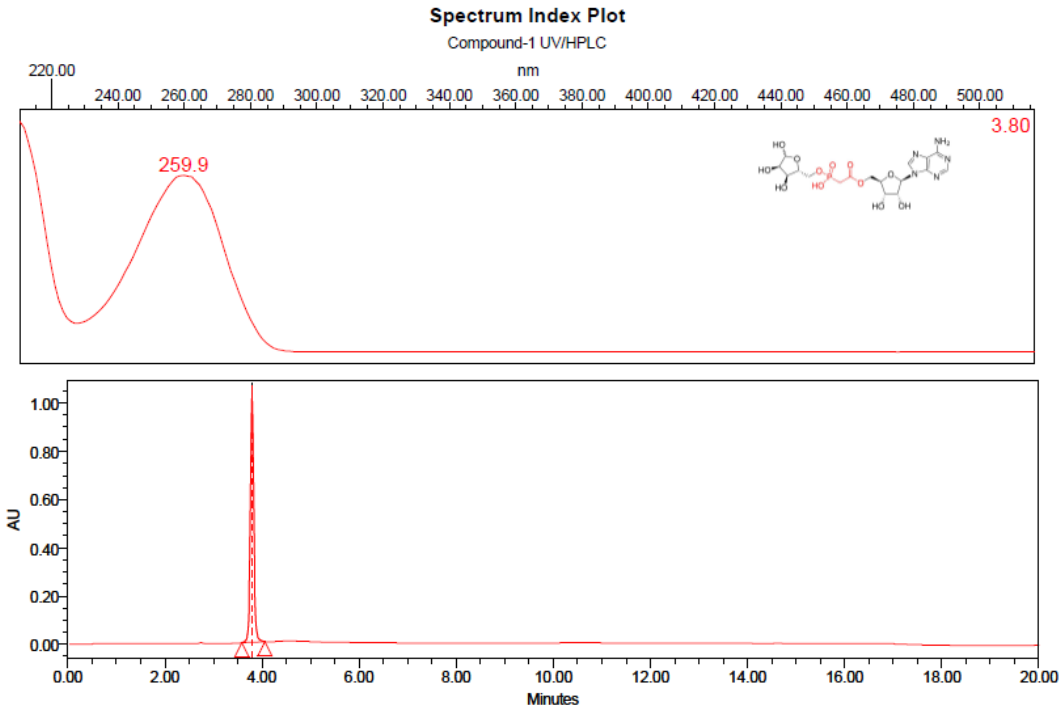



```
NAME          Nov18-2015-OBB7300
EXPNO                 13
PROCNO                 1
Date_           20151119
Time                5.55
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG   hsqcadatgpsp.3
TD                  1024
SOLVENT              D2O
NS                     2
DS                    16
SWH           5980.861 Hz
FIDRES           5.840685 Hz
AQ            0.0856564 sec
RG                  2050
DW                83.600 usec
DE                 6.50 usec
TE               298.3 K
CNST2       145.0000000
D0            0.00000300 sec
D1            1.50000000 sec
D4            0.00172414 sec
D11           0.03000000 sec
D16           0.00020000 sec
D21           0.00360000 sec
IN0           0.00001805 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                 10.00 usec
P2                 20.00 usec
P28                0.00 usec
PL1               -0.12 dB
PL1W        19.35150909 W
SFO1        500.1323521 MHz

======== CHANNEL f2 ========
CPDPRG2            garp4
NUC2                  13C
P3                 9.40 usec
P14              500.00 usec
P31             1900.00 usec
PCPD2             74.00 usec
PL0              120.00 dB
PL2               -0.51 dB
PL12              17.41 dB
PL0W          0.00000000 W
PL1W         99.92730713 W
PL12W         1.61318505 W
SFO2        125.7665916 MHz
SP3                8.19 dB
SP18              14.39 dB
SPNAM3   Crp60,0.5,20.1
SPNAM18   Crp60_srfit.2
SPOAL3             0.500
SPOAL18            0.500
SPOFFS3            0.00 Hz
SPOFFS18           0.00 Hz

====== GRADIENT CHANNEL ======
GPNAM1       SMSQ10.100
GPNAM2       SMSQ10.100
GPZ1              80.00 %
GPZ2              20.10 %
P16             1000.00 usec
ND0                    2
TD                   256
SFO1        125.7666 MHz
FIDRES      108.080666 Hz
SW          220.000 ppm
FnMODE    Echo-Antiecho
SI                  1024
SF          500.1300000 MHz
WDW                QSINE
SSB                    2
LB                 0.00 Hz
GB                     0
PC                 1.40
SI                  1024
MC2       echo-antiecho
SF          125.7577890 MHz
WDW                QSINE
SSB                    2
LB                 0.00 Hz
GB                     0
```

compound-1 HMBC




```
NAME          OBB44_more
EXPNO                 11
PROCNO                 1
Date_           20151119
Time               17.36
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG    hmbcatgpl3nd
TD                  4096
SOLVENT              D2O
NS                    16
DS                    16
SWH           6510.417 Hz
FIDRES           1.589457 Hz
AQ            0.3146228 sec
RG                  2050
DW                76.800 usec
DE                 6.50 usec
TE               298.2 K
CNST6       120.0000000
CNST7       170.0000000
CNST13        8.0000000
CNST30        0.5981126
D0            0.00000300 sec
D1            1.50000000 sec
D6            0.06250000 sec
D16           0.00020000 sec
IN0           0.00001655 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                 10.00 usec
P2                 20.00 usec
PL1               -0.12 dB
PL1W        19.35150909 W
SFO1        500.1331508 MHz

======== CHANNEL f2 ========
NUC2                  13C
P3                 9.40 usec
P24              2000.00 usec
PL2               -0.51 dB
PL2W         99.92730713 W
SFO2        125.7716219 MHz
SP7                8.19 dB
SPNAM7    Crp60comp.4
SPOAL7             0.500
SPOFFS7            0.00 Hz

====== GRADIENT CHANNEL ======
GPNAM1       SMSQ10.100
GPNAM3       SMSQ10.100
GPNAM4       SMSQ10.100
GPNAM5       SMSQ10.100
GPNAM6       SMSQ10.100
GPZ1              80.00 %
GPZ3              14.00 %
GPZ4              -8.00 %
GPZ5              -4.00 %
GPZ6              -2.00 %
P16             1000.00 usec
ND0                    2
TD                   256
SFO1        125.7716 MHz
FIDRES      117.910896 Hz
SW          240.000 ppm
FnMODE    Echo-Antiecho
SI                  2048
SF          500.1300000 MHz
WDW                QSINE
SSB                    2
LB                 0.00 Hz
GB                     0
PC                 1.40
SI                  1024
MC2       echo-antiecho
SF          125.7577890 MHz
WDW                QSINE
SSB                    2
LB                 0.00 Hz
GB                     0
```



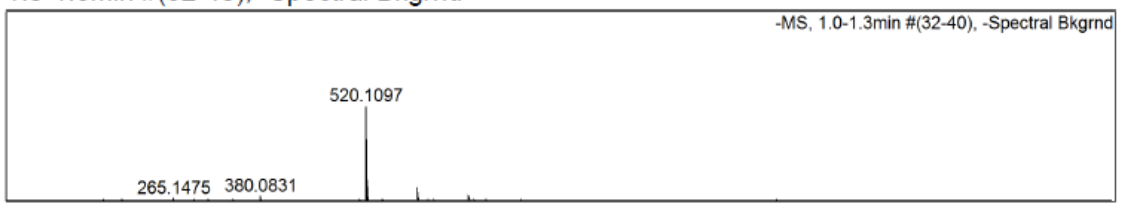
PDA Result Table

	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

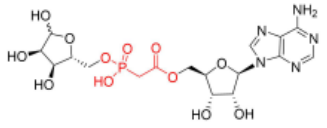
Confirmation of Expected Formula

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



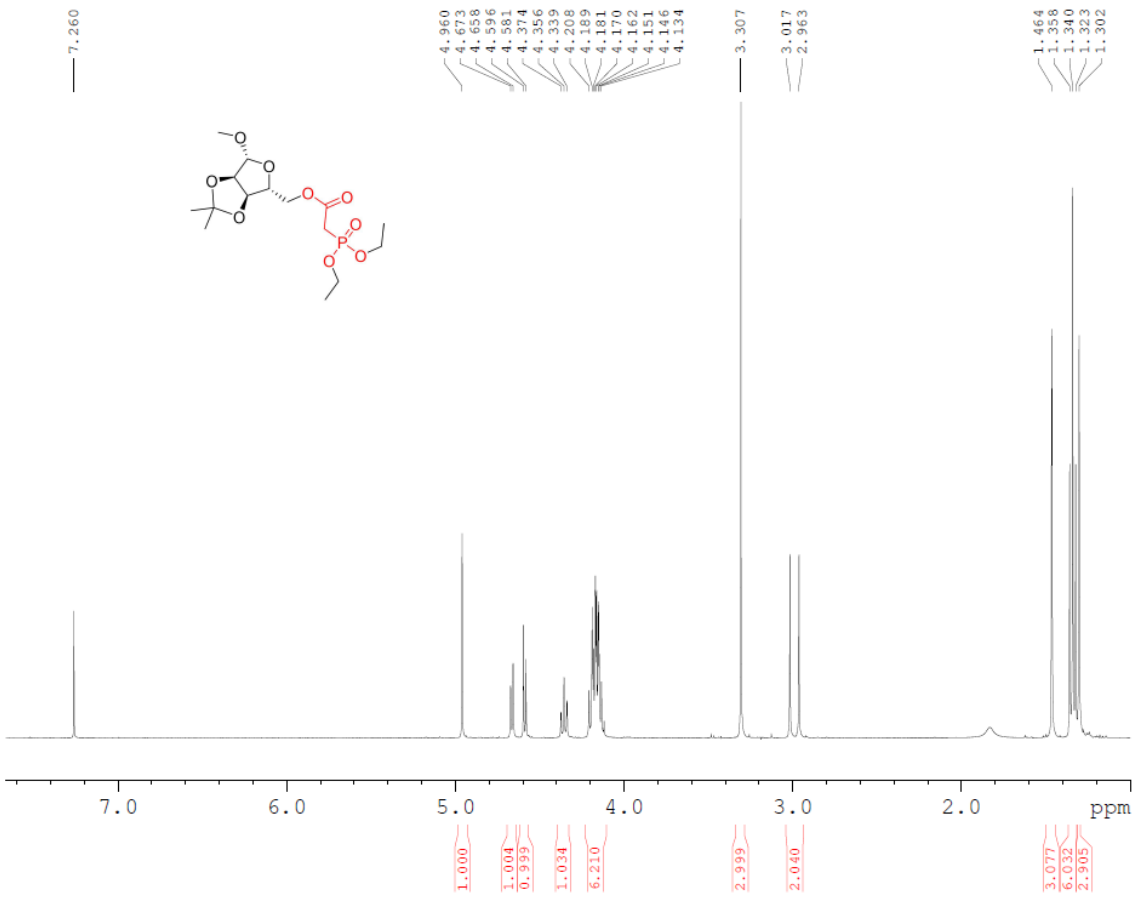
Generate Molecular Formula Parameters

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

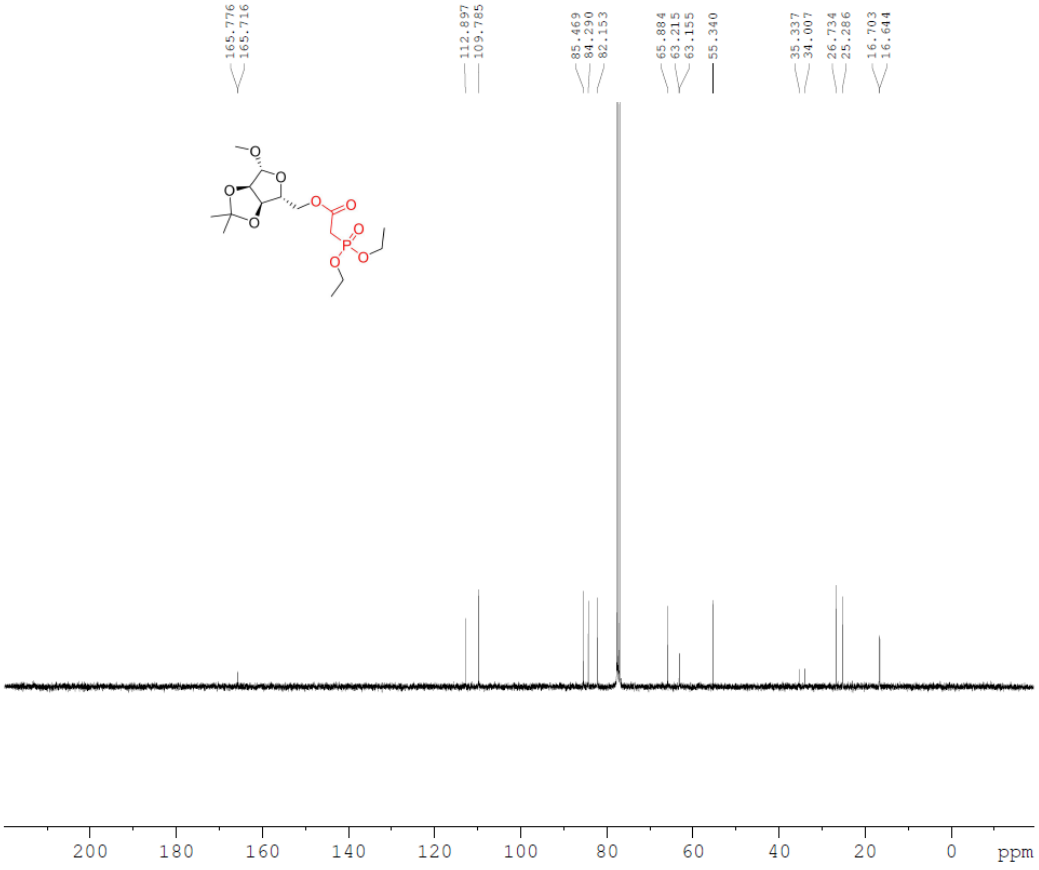
#	meas. m/z	theo. m/z	Err[ppm]	Sigma	Formula
1	520.1097	520.108083	-2.10	0.0078	C 17 H 23 N 5 O 12 P 1

compound-8 ^{1}HNMR



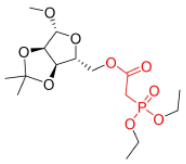

```
NAME          Jul17-2015-OB6088
EXPNO                       10
PROCNO                       1
Date_                 20150717
Time                     16.37
INSTRUM                AVIII400
PROBHD    5 mm PABBO BB-
PULPROG                   zg30
TD                       65536
SOLVENT                  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 ^{13}CNMR



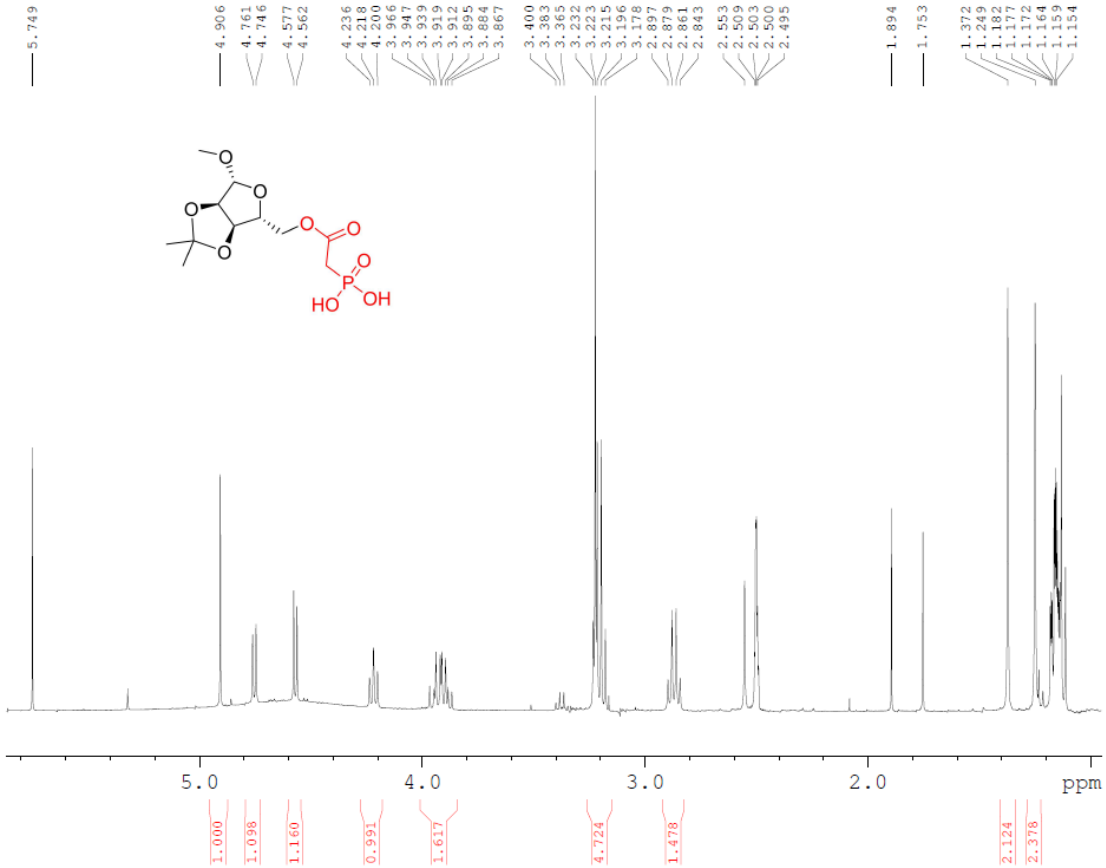

```
NAME          Jul17-2015-OB6088
EXPNO                       13
PROCNO                       1
Date_                 20150718
Time                     2.00
INSTRUM                AVIII400
PROBHD    5 mm PABBO BB-
PULPROG                 zgpg30
TD                       65536
SOLVENT                  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                58.91986084 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                  100.5900987 MHz
WDW                         EM
SSB                          0
LB                      1.00 Hz
GB                           0
PC                        1.40
```

compound-8 ^{31}PNMR



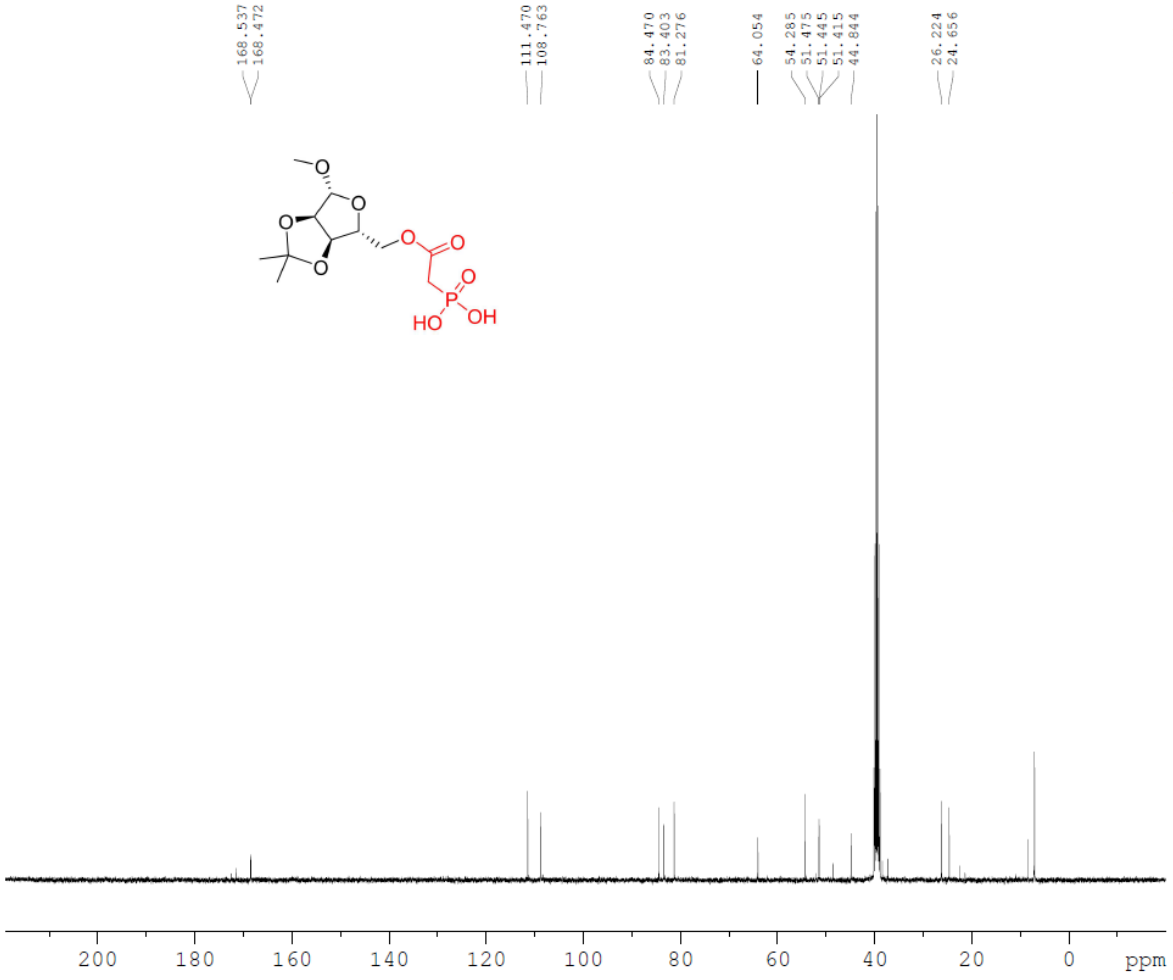

```
NAME          Jul17-2015-OB6088
EXPNO                       11
PROCNO                       1
Date_                 20150717
Time                     16.40
INSTRUM                AVIII400
PROBHD    5 mm PABBO BB-
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDCl3
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-9 ^{1}HNMR



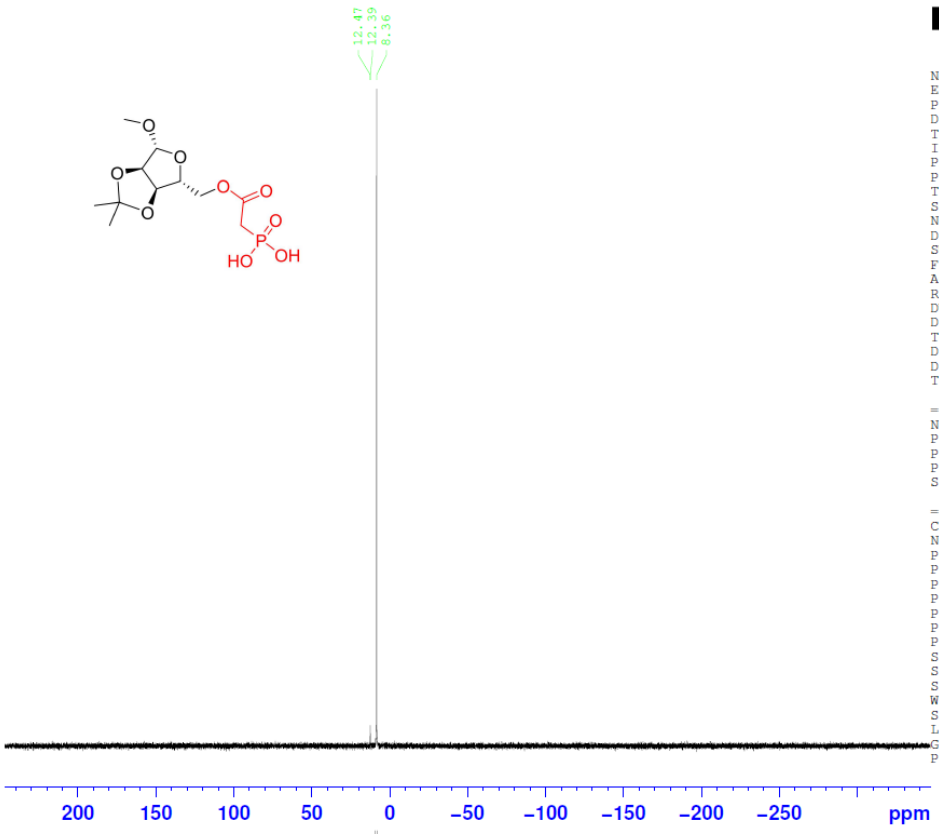

```
NAME          Aug10-2015-OB6081
EXPNO                       10
PROCNO                       1
Date_                 20150810
Time                     11.22
INSTRUM                AVIII400
PROBHD    5 mm PABBO BB-
PULPROG                   zg30
TD                       65536
SOLVENT                   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 ^{13}CNMR



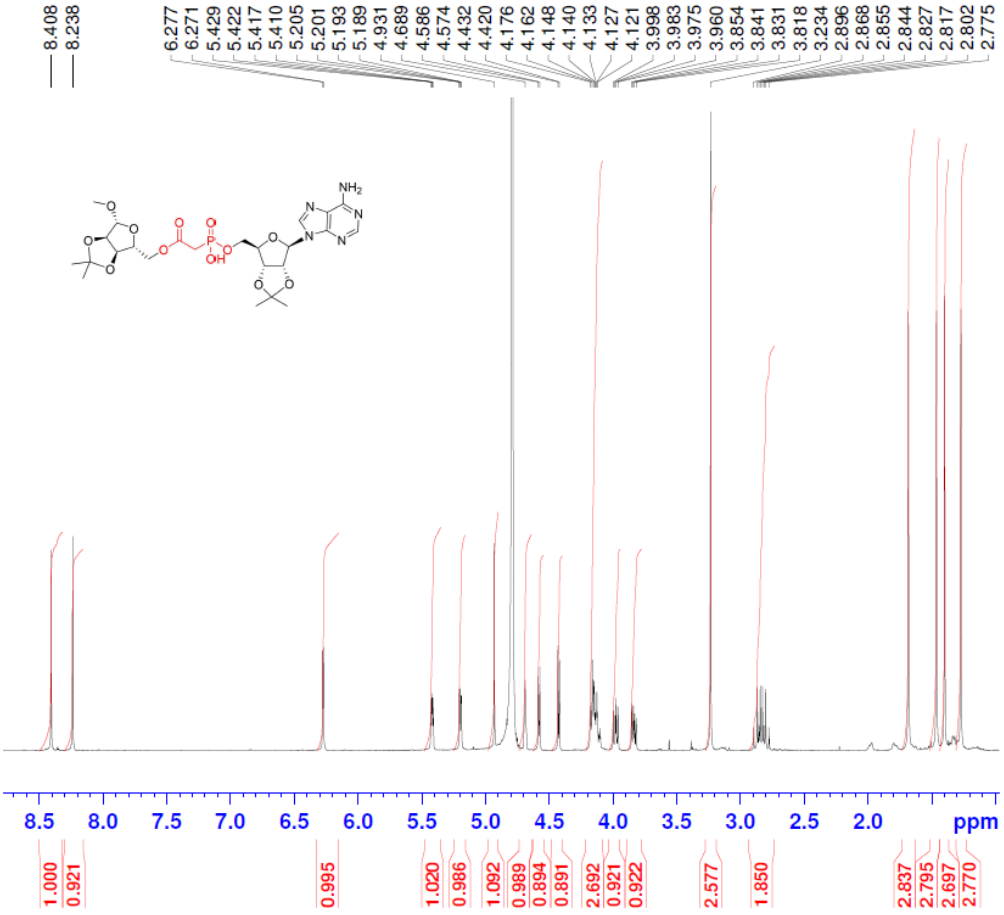

```
NAME          Dec19-2015_OBB14_RE
EXPNO                          12
PROCNO                          1
Date_                    20151219
Time                        18.16
INSTRUM                  AVIII400
PROBHD          5 mm PABBO BB-
PULPROG                    zgpg30
TD                          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 ^{31}PNMR



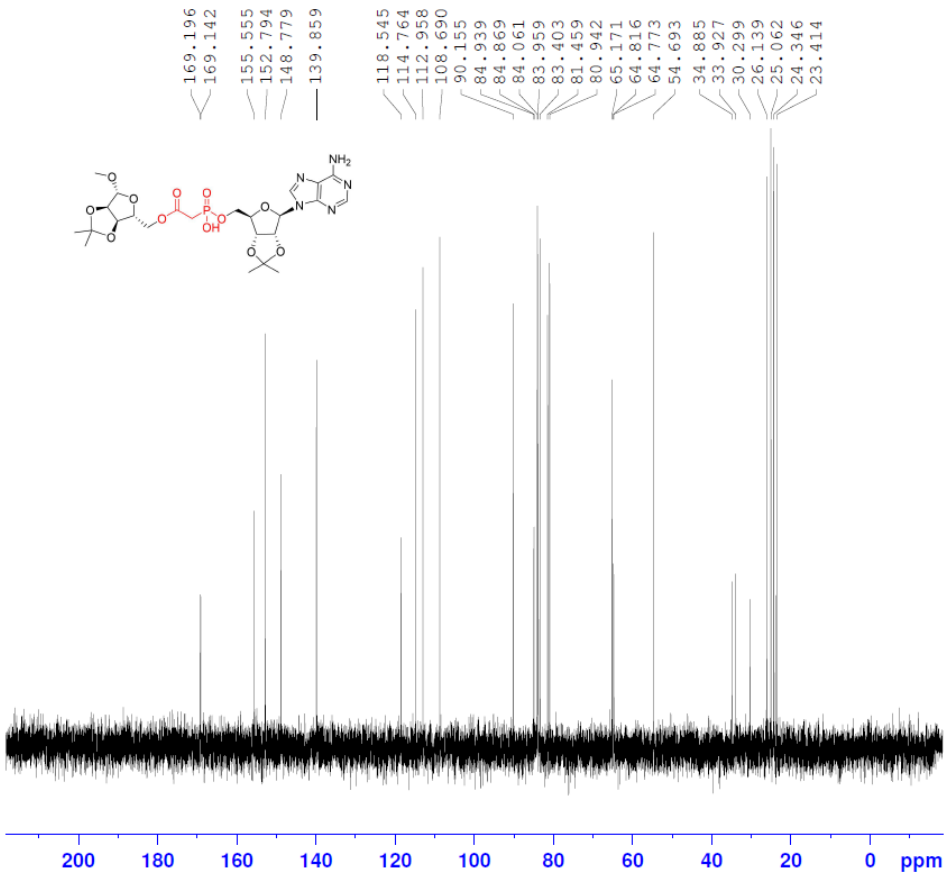

```
NAME          Aug10-2015-OB6081
EXPNO                          11
PROCNO                          1
Date_                    20150810
Time                        11.26
INSTRUM                  AVIII400
PROBHD          5 mm PABBO BB-
PULPROG                    zgpg30
TD                          65536
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 ^{1}HNMR



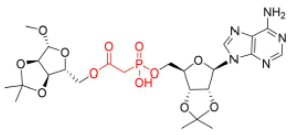

```
NAME                OBB-24
EXPNO                   10
PROCNO                   1
Date_             20151021
Time                 11.51
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG              zg30
TD                  65536
SOLVENT               D2O
NS                       16
DS                        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
```

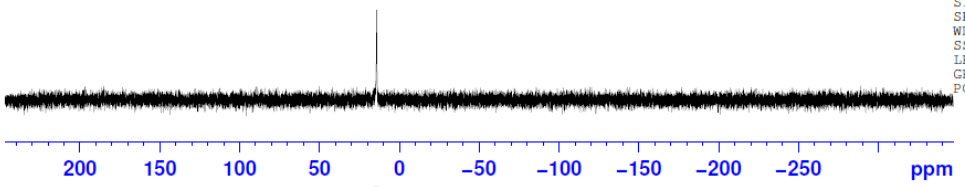
compound-10 ^{13}CNMR




```
NAME                OBB-24
EXPNO                   13
PROCNO                   1
Date_             20151021
Time                 13.04
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                  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 d
PL1W          99.92730713 W
SFO1         125.7703643 M

======== CHANNEL f2 ======
CPDPRG2            waltz16
NUC2                    1H
PCPD2              80.00 u
PL2                -0.12 d
PL12               17.94 d
PL13               21.00 d
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
```

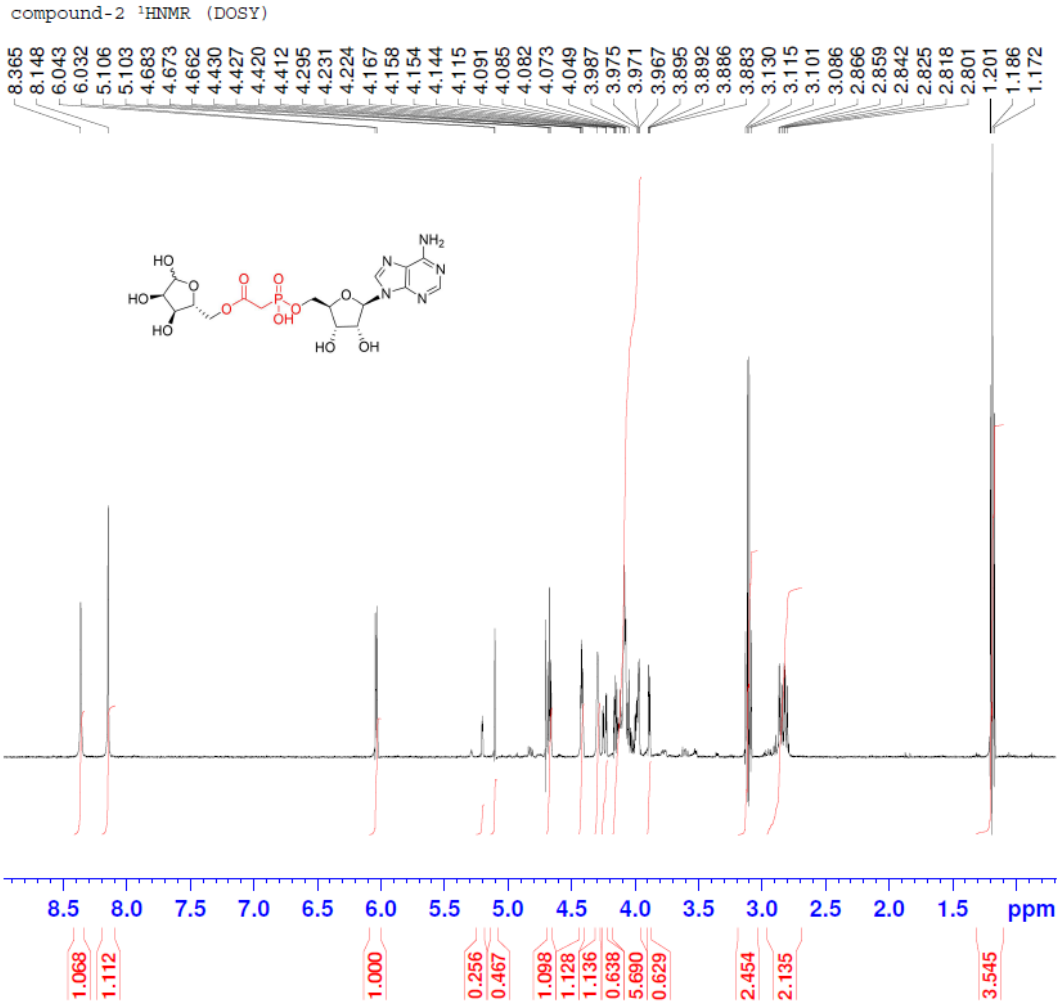
compound-10 ^{31}PNMR





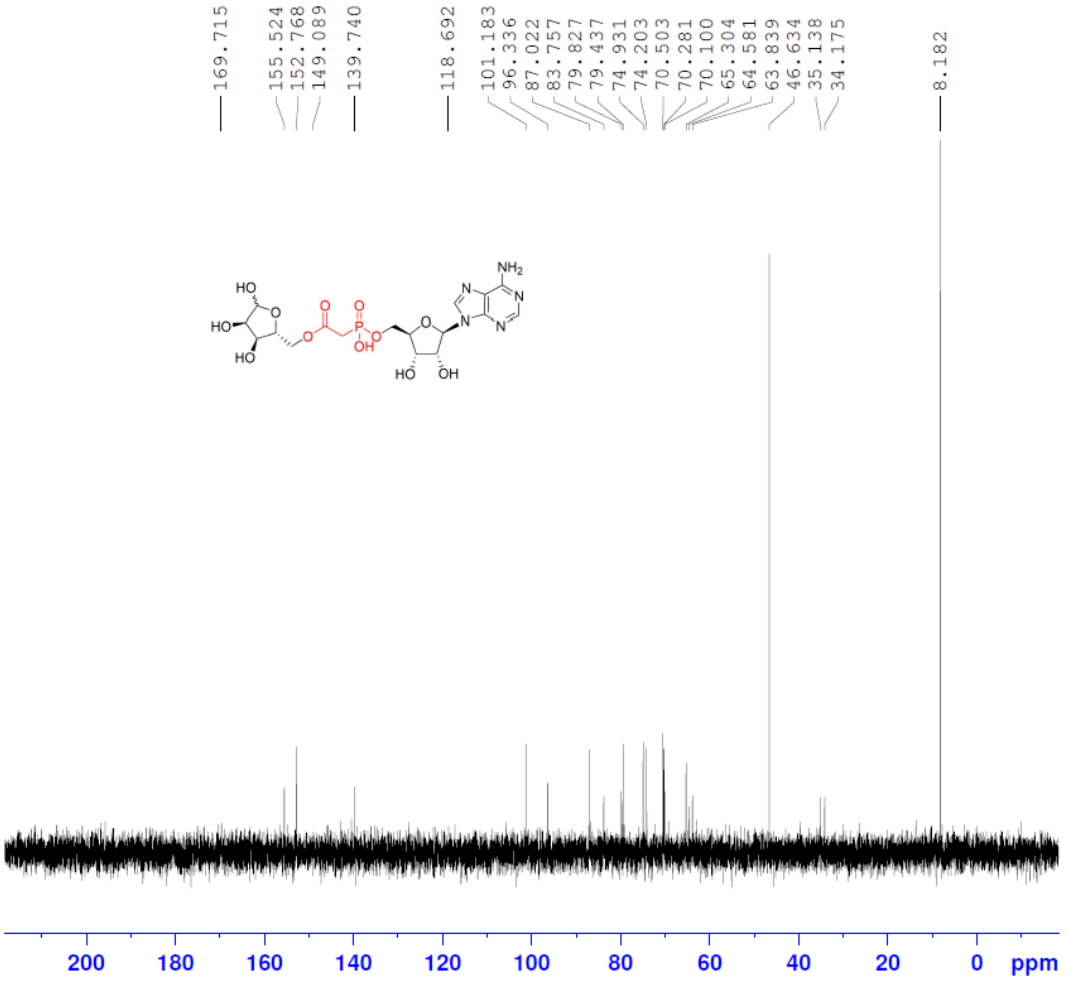

```
NAME            Dec08-2015-OBB24_PUR
EXPNO                             11
PROCNO                             1
Date_                       20151208
Time                           16.43
INSTRUM                     AVIII400
PROBHD   5 mm PABBO BB-
PULPROG                      zgpg30
TD                            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 ^{1}HNMR (DOSY)



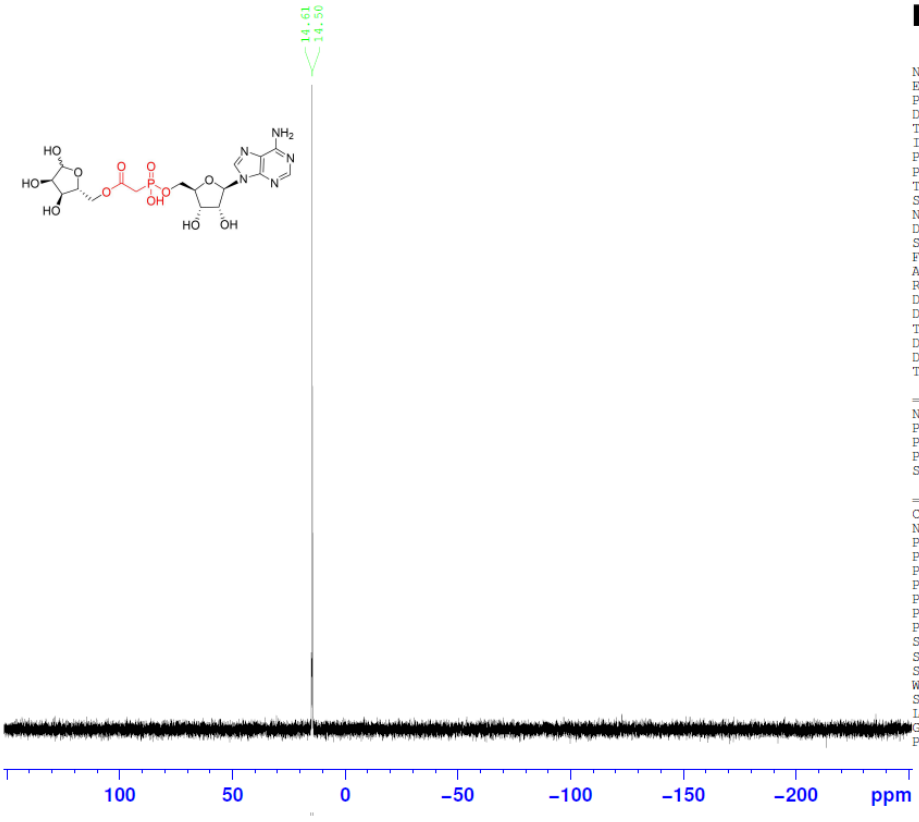

```
NAME                 OBB46
EXPNO                   50
PROCNO                   1
Date_             20151218
Time                 11.50
INSTRUM               spect
PROBHD   5 mm PABBO BB-
PULPROG     ledbpgp2s1d
TD                    65536
SOLVENT                 D2O
NS                       16
DS                        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 =====
GPNAM6              sine.100
GPNAM7              sine.100
```

compound-2 ^{13}CNMR




```
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              2.00000000 sec
D11             0.03000000 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
```

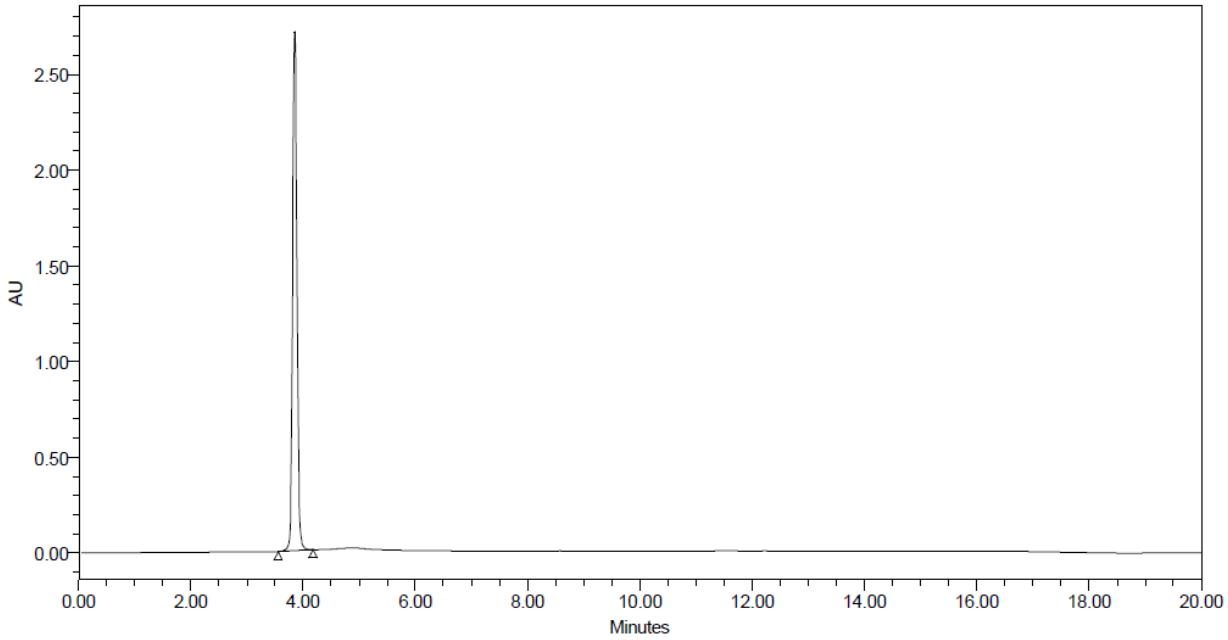
compound-2 ^{31}PNMR



BRUKER

```
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              2.00000000 sec
D11             0.03000000 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

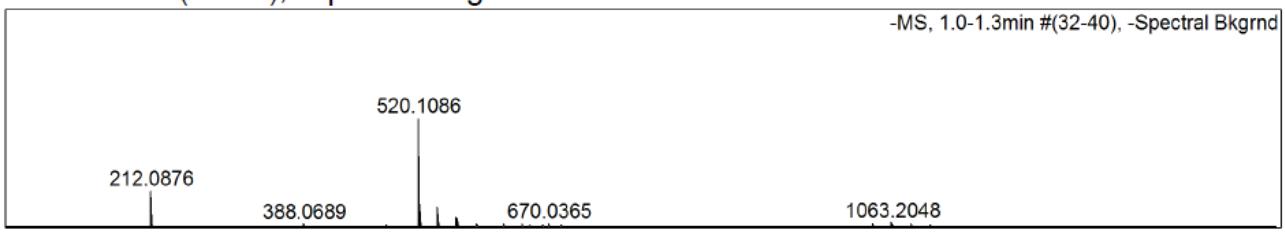


	Peak Name	RT	Area	% Area	Height
1	compound-2	3.854	14751722	100.00	2724648

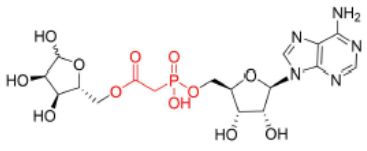
Confirmation of Expected Formula

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