

Design and Synthesis of α -Carboxy Phosphononucleosides

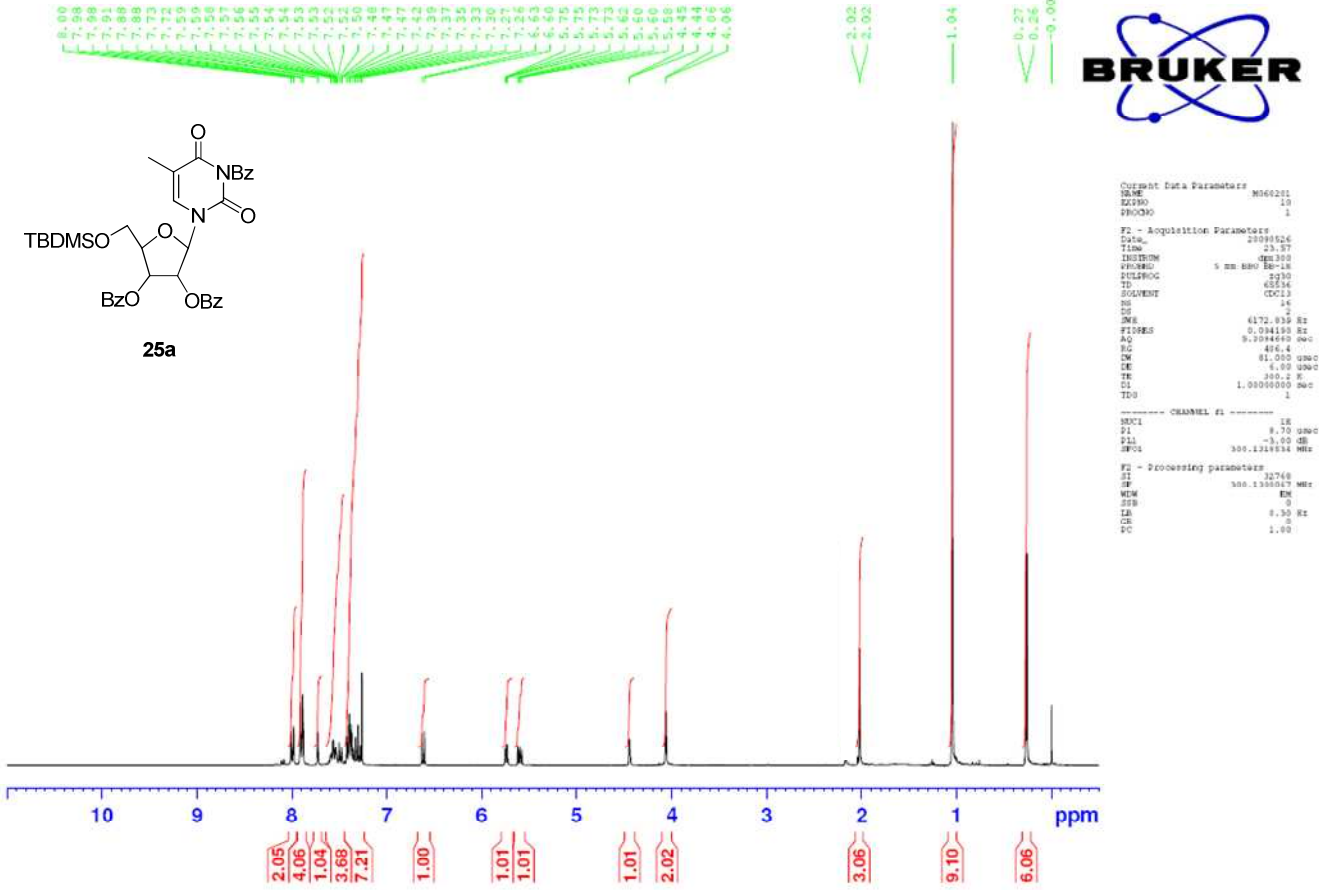
Sebastien Debarge, Jan Balzarini and Anita R. Maguire

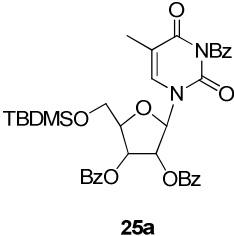
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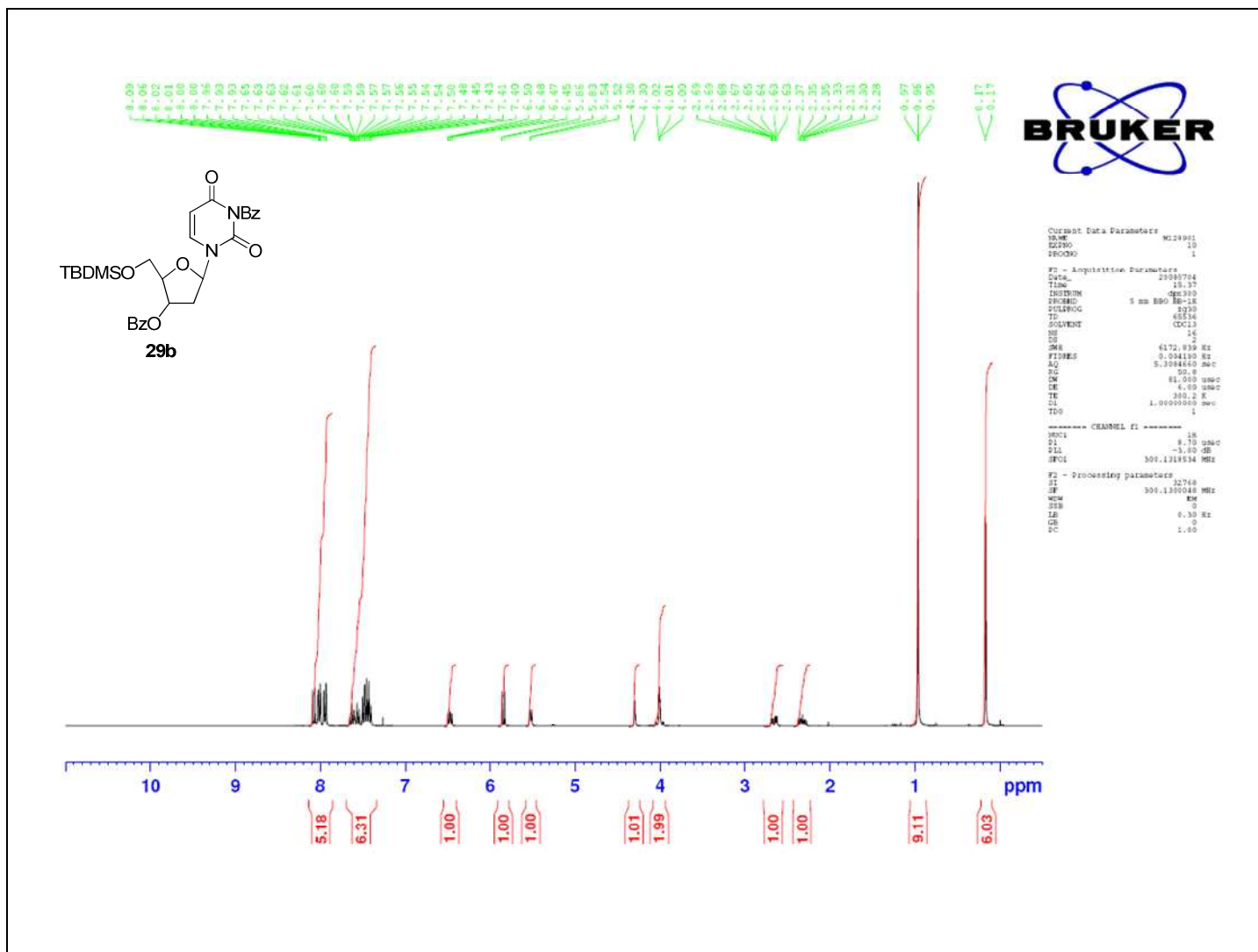
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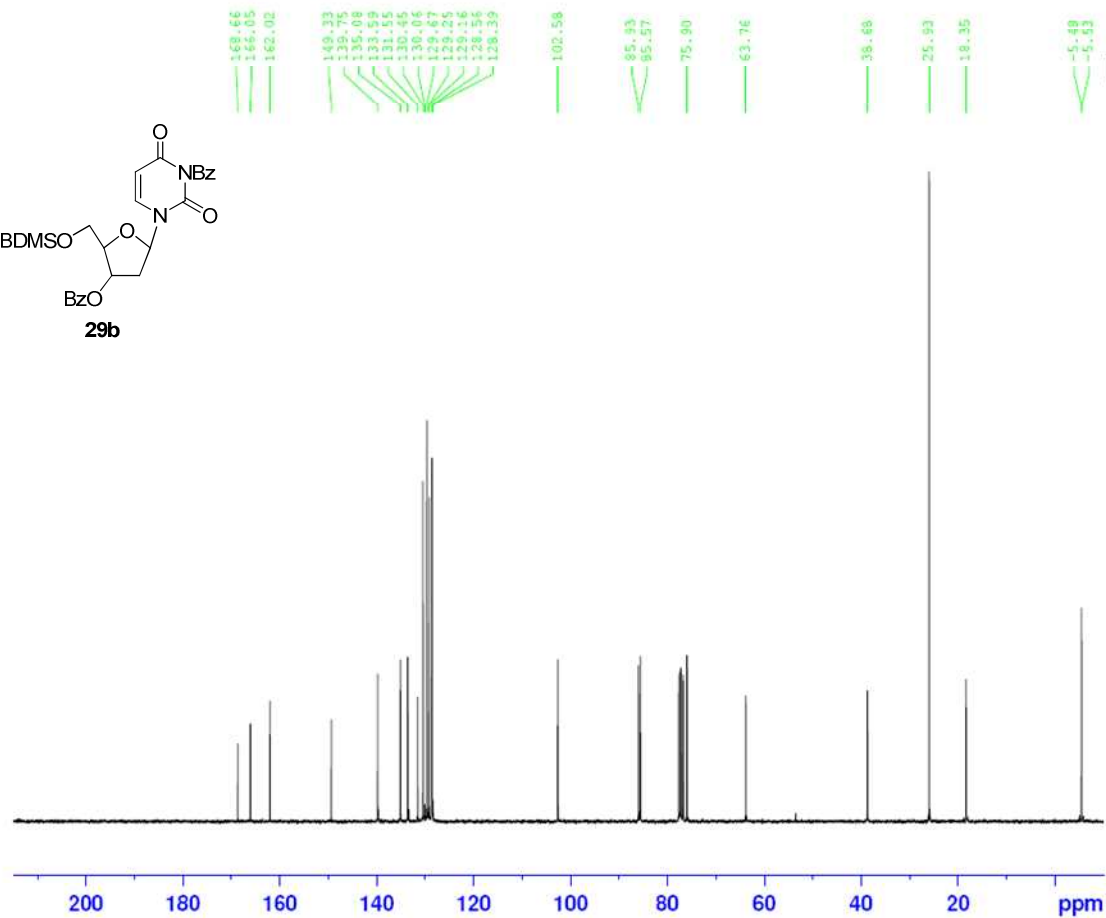
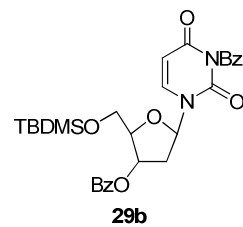
Compound **25a**.





Compound **29b**.





Current Data Parameters
NAME H128902
EXPNO 10
PROCNO 1

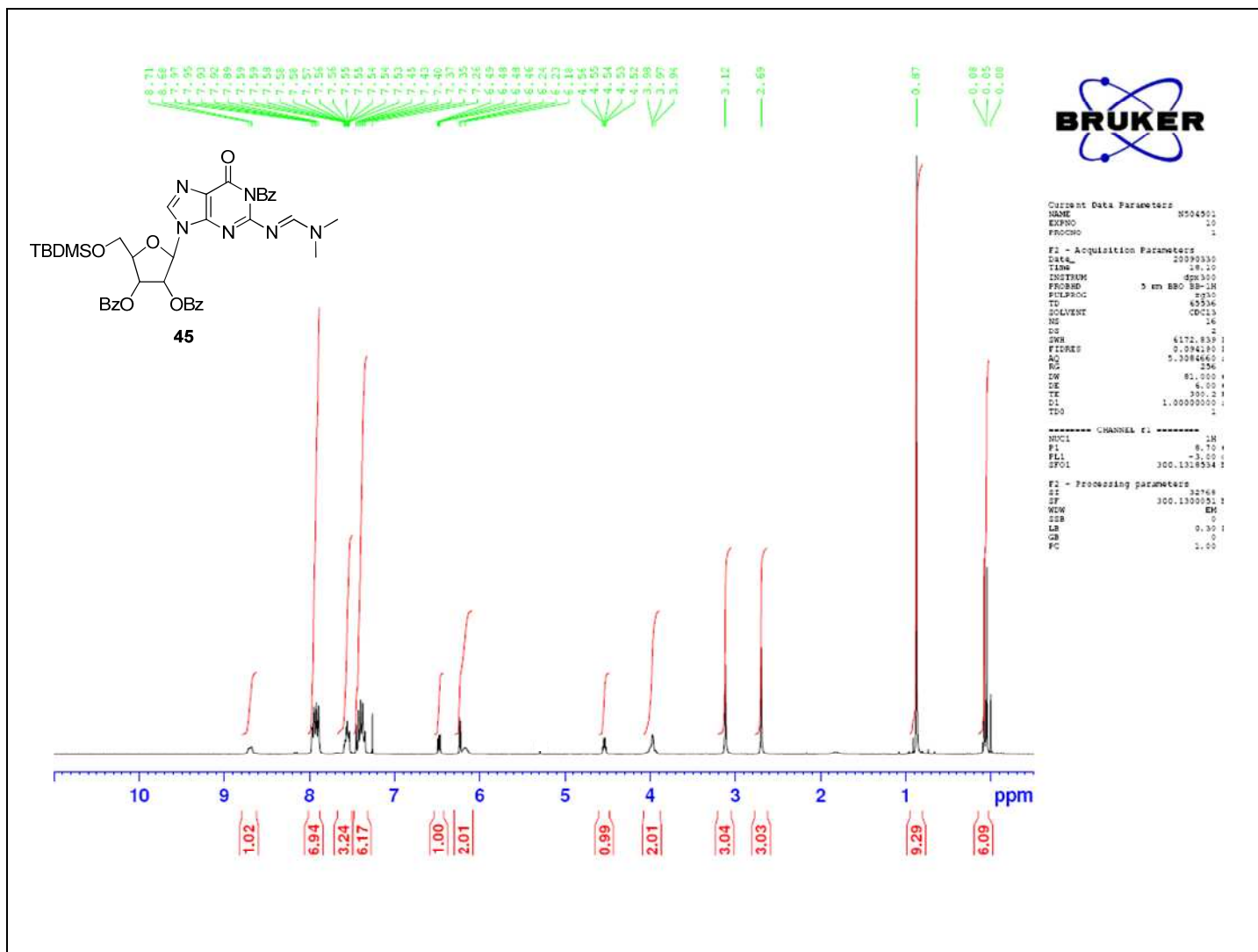
F2 - Acquisition Parameters
Date_ 20090705
Time 3.23
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 17985.411 Hz
FIDRES 0.274439 Hz
AQ 1.9219508 sec
RG 4097.6
RM 27.800 used
DE 6.00 used
TE 300.2 K
d1 0.50000000 sec
d11 0.03000000 sec
DELTA 0.40000002 sec
TD0 1

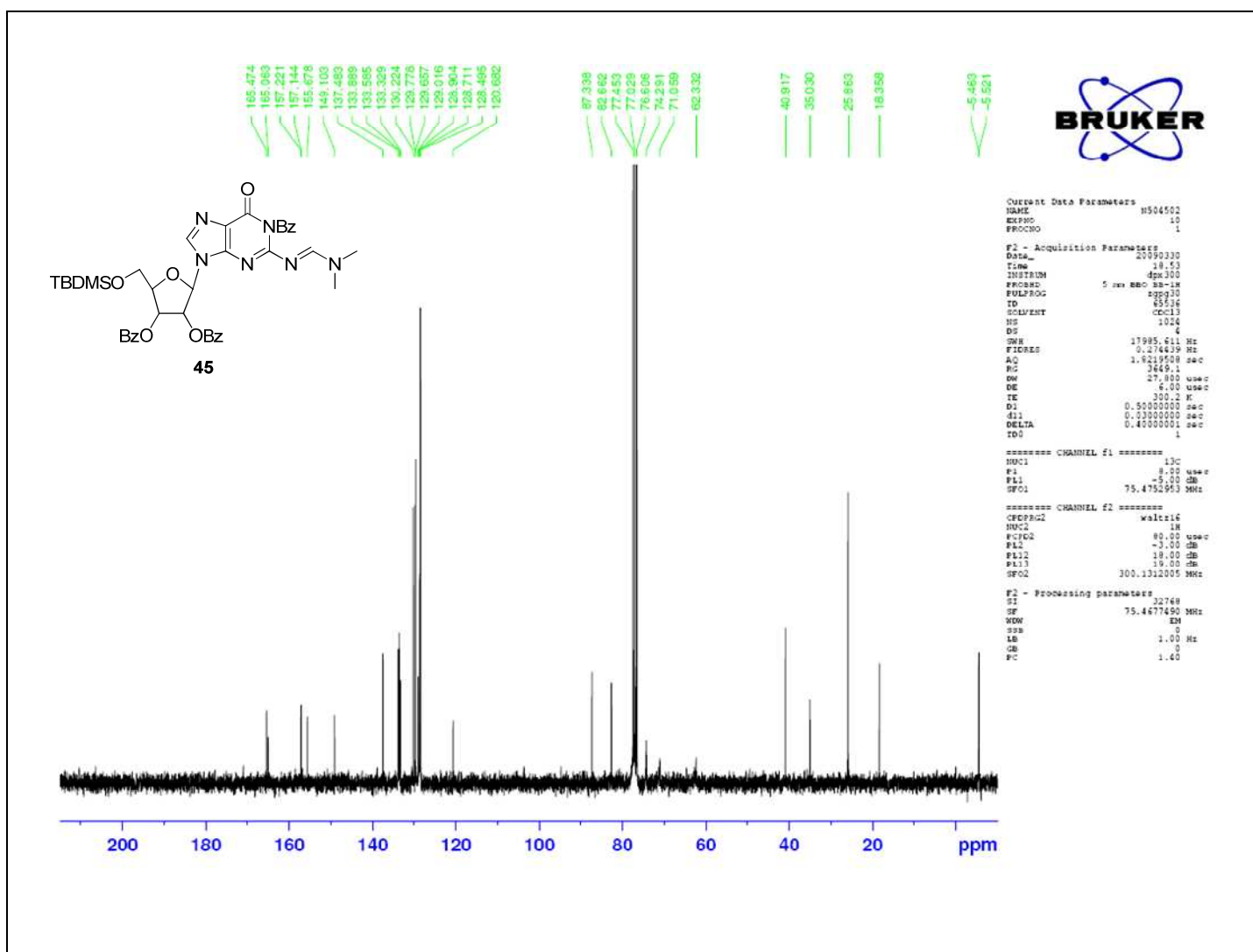
===== CHANNEL f1 =====
NUC1 13C
P1 9.00 used
PL1 -5.00 dB
SFO1 75.4752453 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 used
PL2 -3.00 dB
PL12 19.00 dB
PL13 19.00 dB
SFO2 300.1312015 MHz

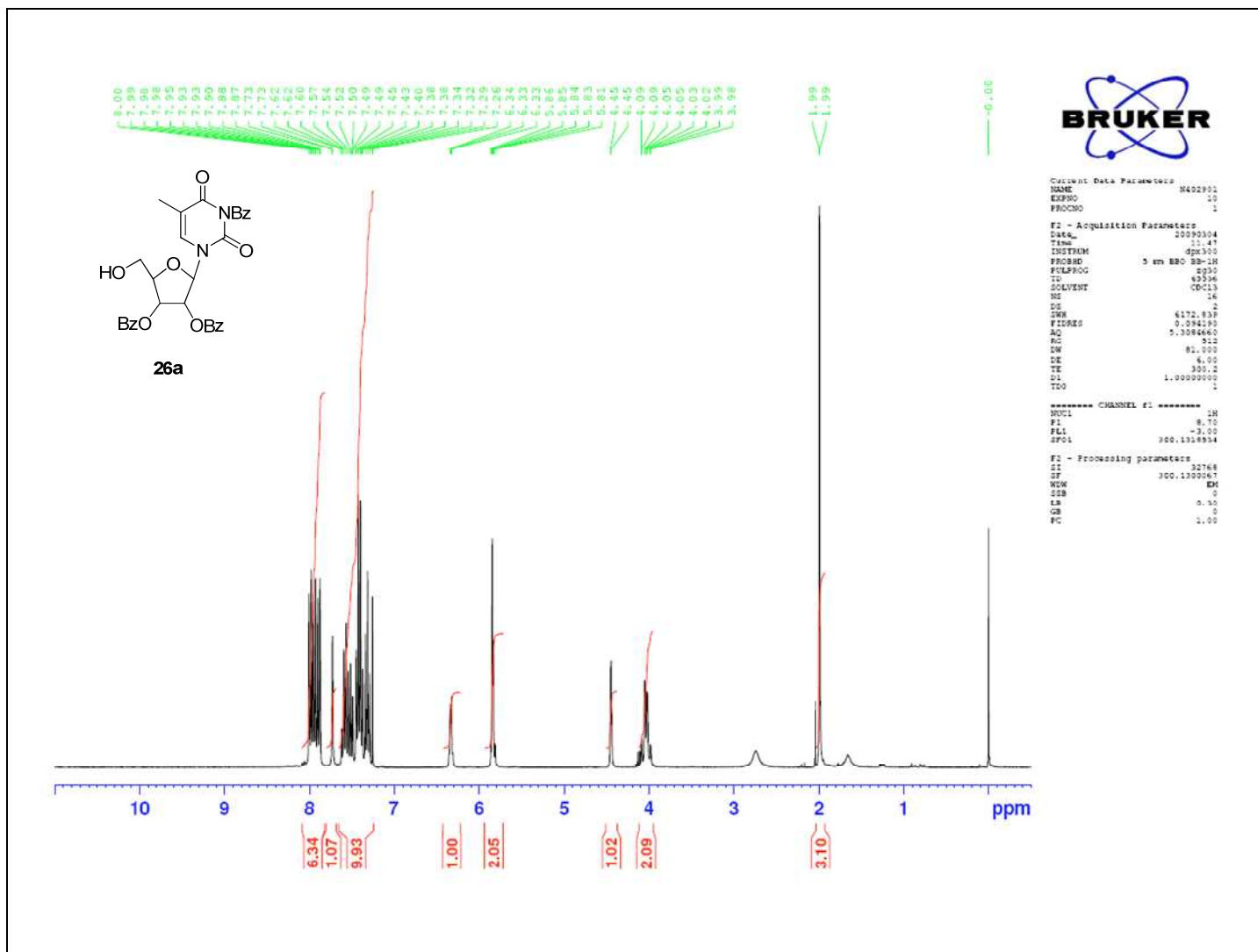
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WDW EN
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

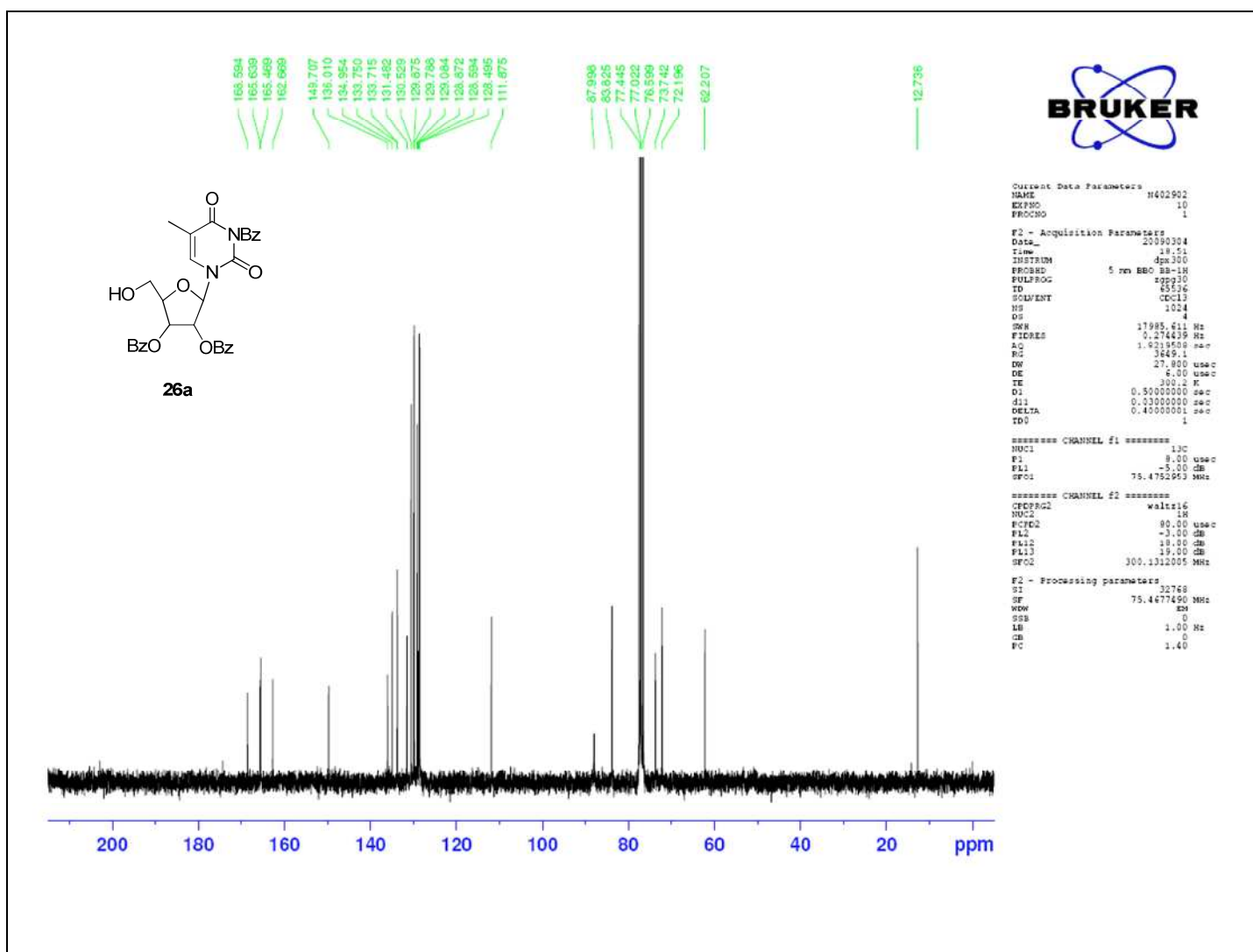
Compound **45**.



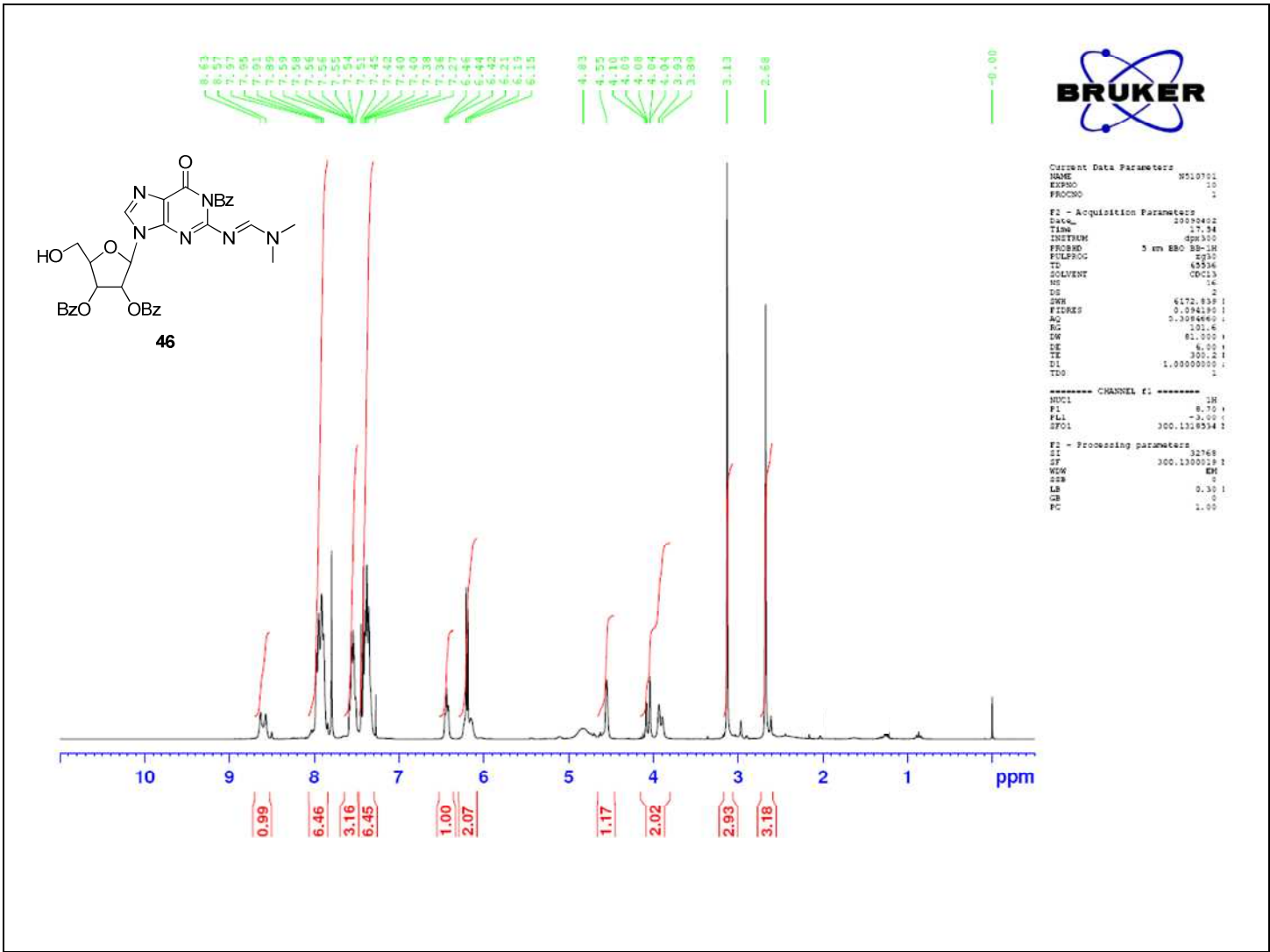


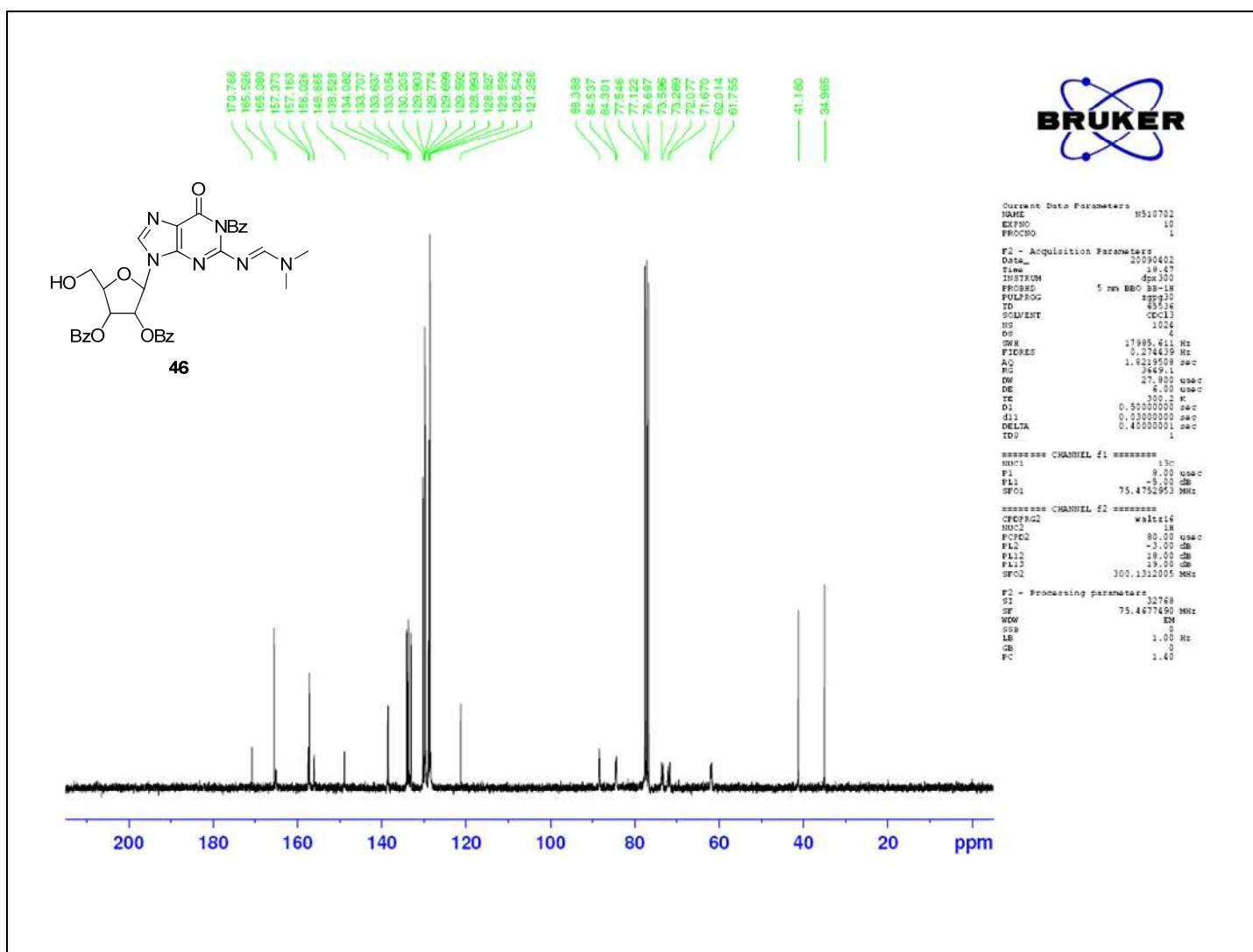
Compound **26a**.



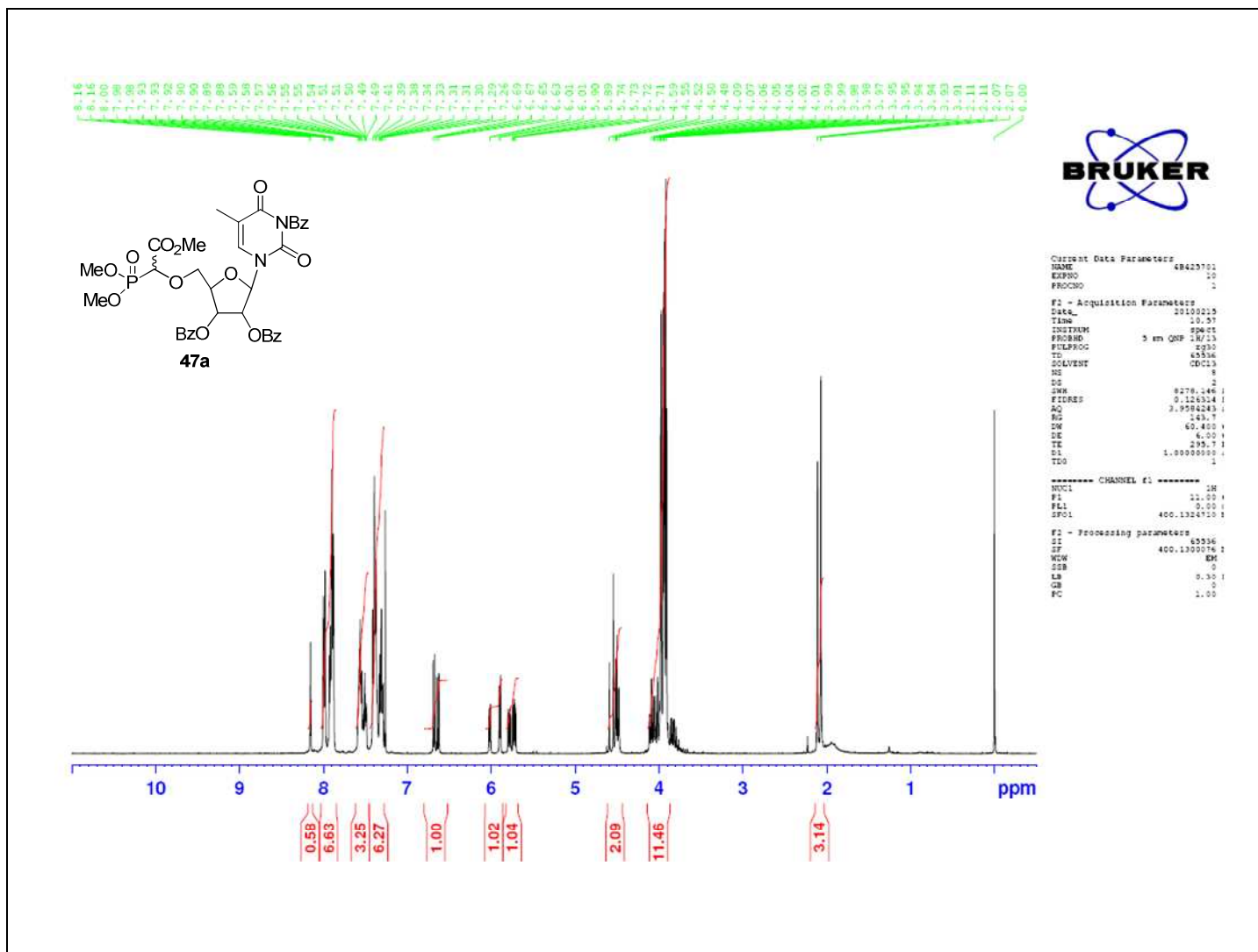


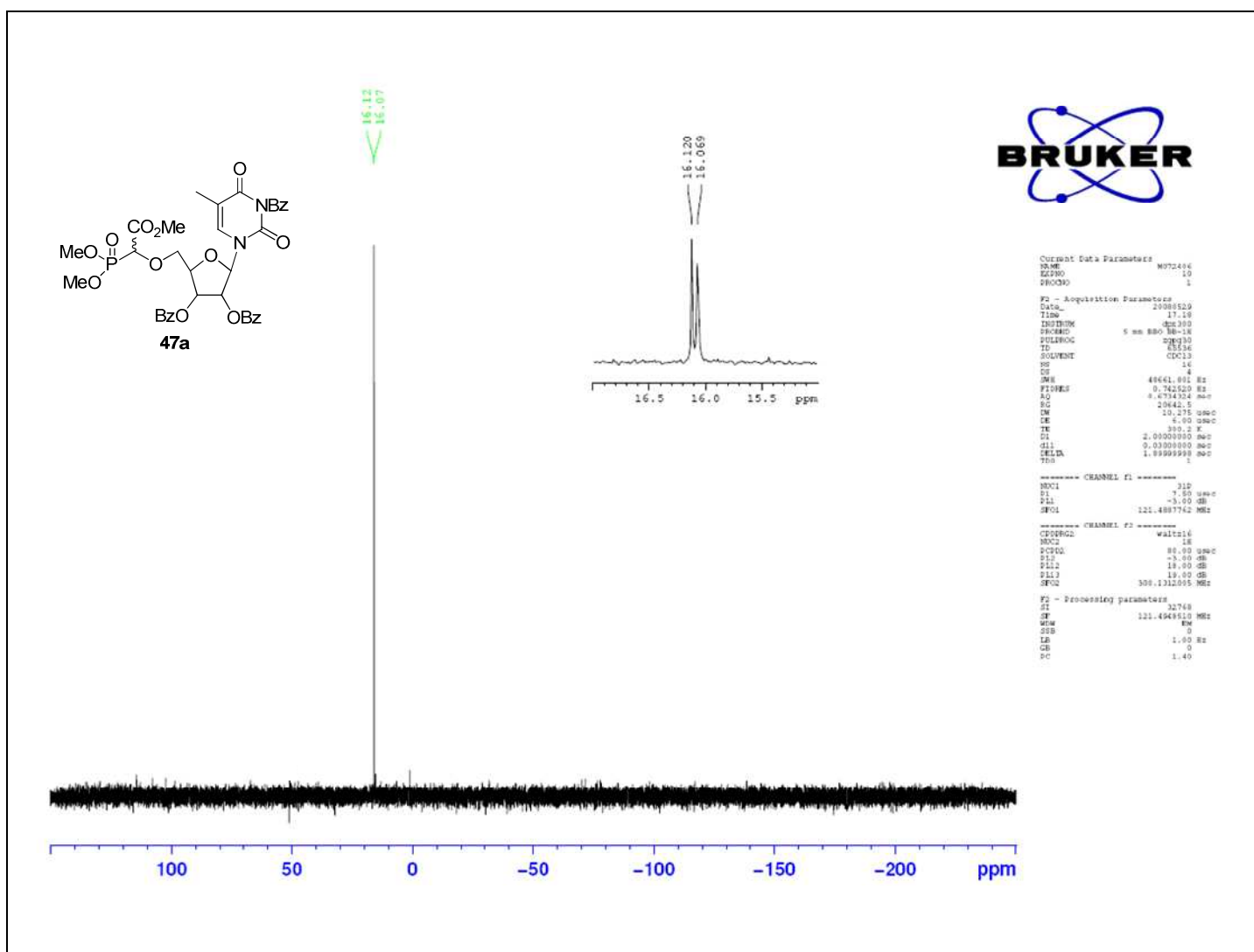
Compound **46**.



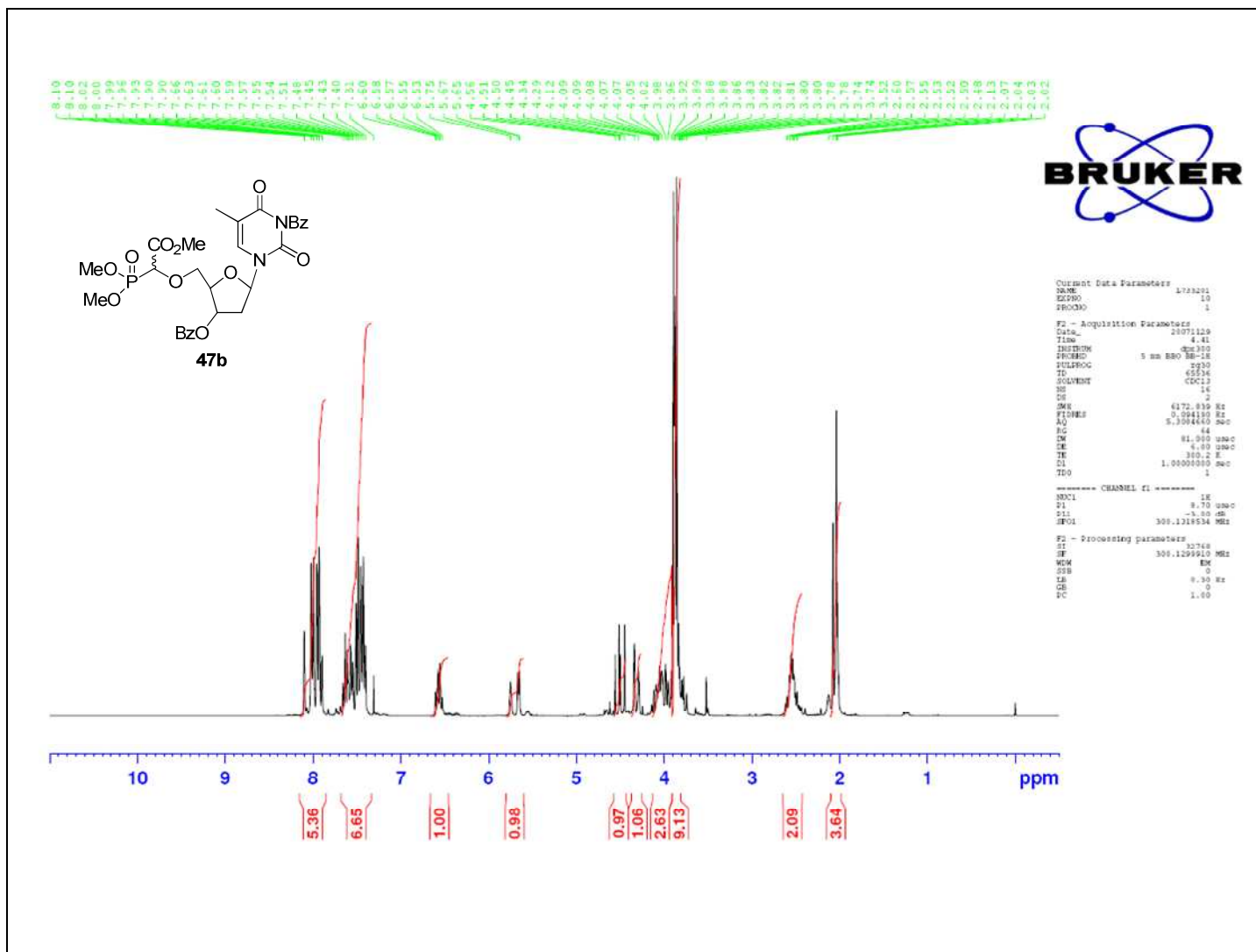


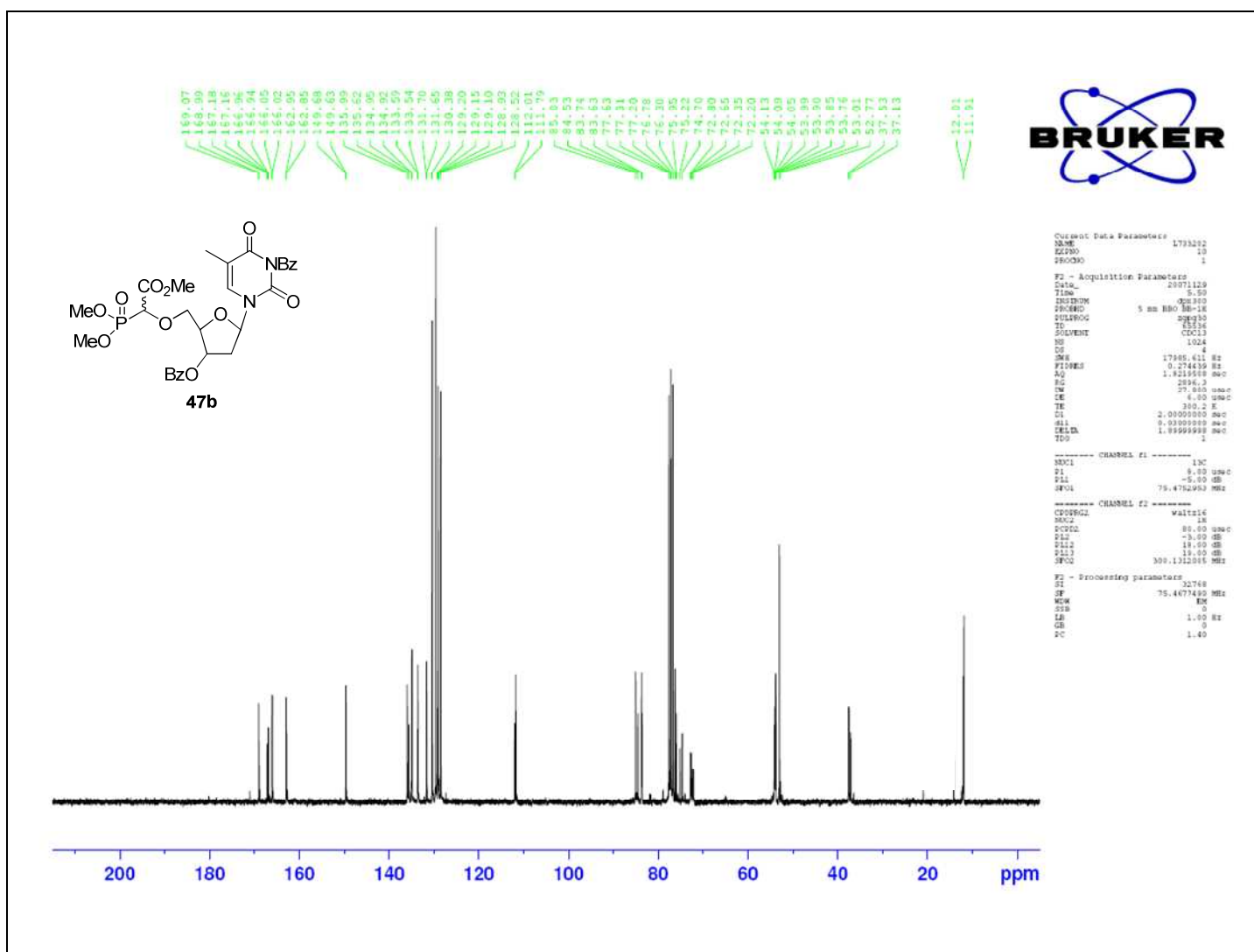
Compound **47a**.

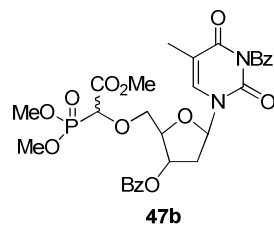




Compound **47b**.







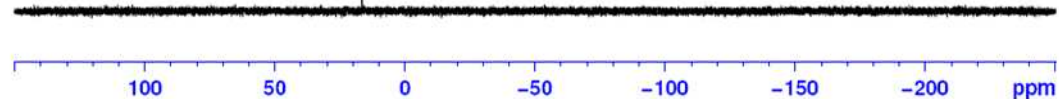
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EXPNO: 10
PROCNO: 1

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Date_: 20090309
Time: 15:14
INSTRUM: dpn300
PROBHD: 5 mm BBO BB-1H
PULPROG: zgpg30
TD: 65536
SOLVENT: CDCl3
NS: 52
DS: 4
SWH: 49641.801 Hz
FIDRES: 0.762520 Hz
AQ: 0.6734324 sec
RG: 40942.5
DW: 10.275 usec
DE: 8.00 usec
TE: 300.2 K
D1: 2.00000000 sec
d11: 0.03000000 sec
DELTA: 1.89999998 sec
TD0: 1

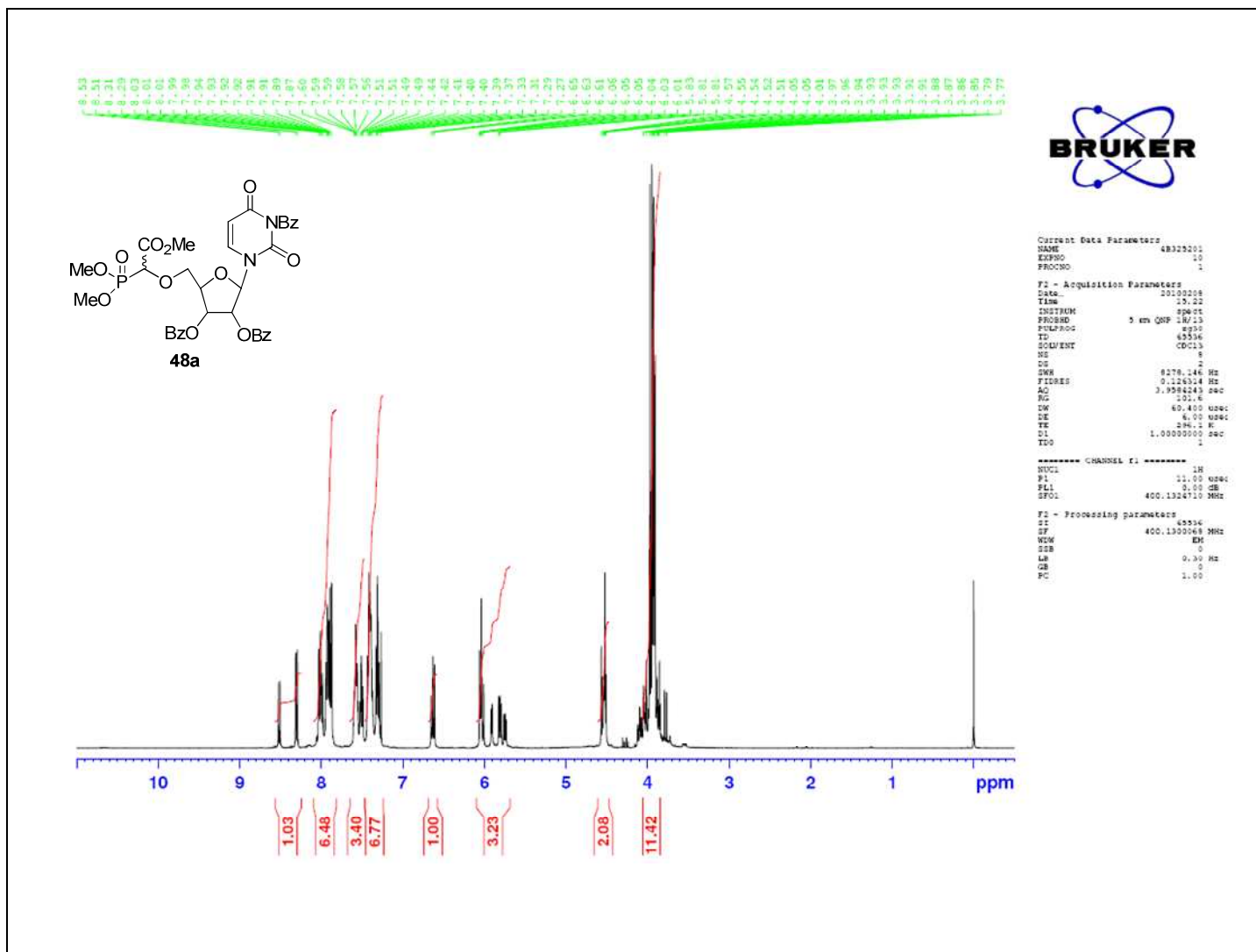
===== CHANNEL f1 =====
NUC1: 31P
P1: 7.50 usec
PL1: -3.00 dB
SFO1: 121.4887762 MHz

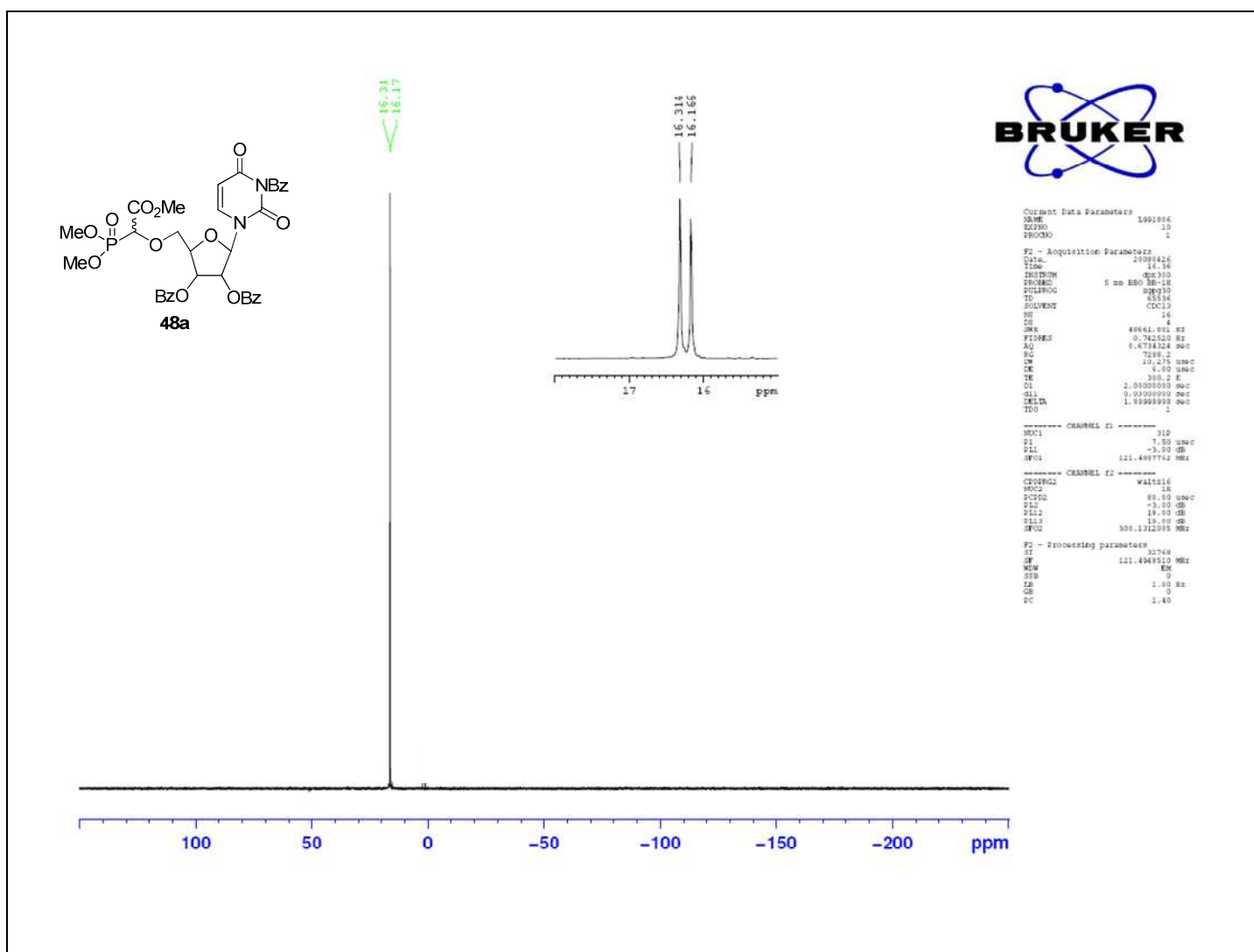
===== CHANNEL f2 =====
CPDPRG2: waltz16
NUC2: 1H
P2: 80.00 usec
PL2: -3.00 dB
PL12: 18.00 dB
PL13: 18.00 dB
SFO2: 300.1312005 MHz

F2 - Processing parameters
SI: 32768
SF: 121.4948510 MHz
WDW: EM
SSB: 0
LB: 1.00 Hz
GB: 0
PC: 1.40

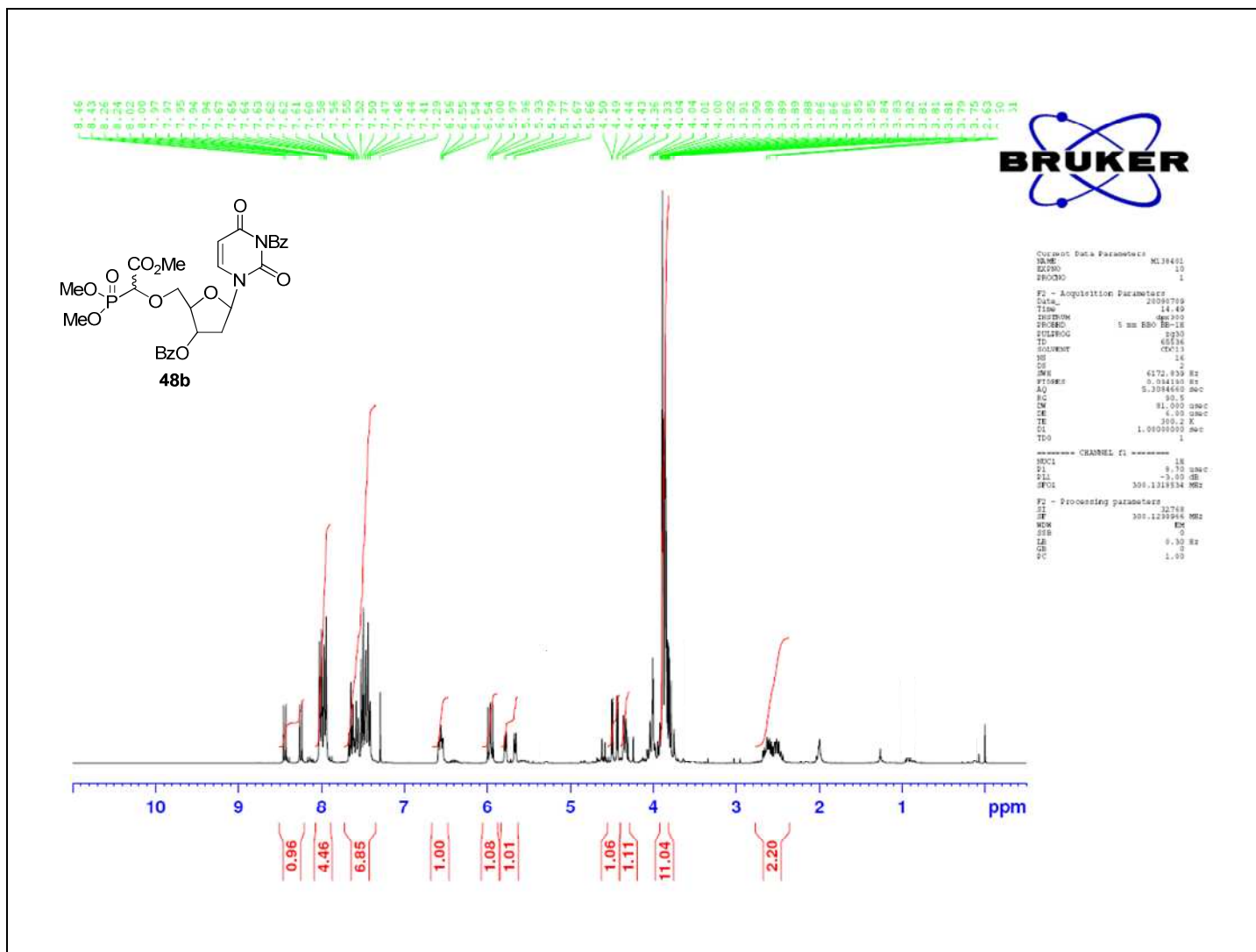


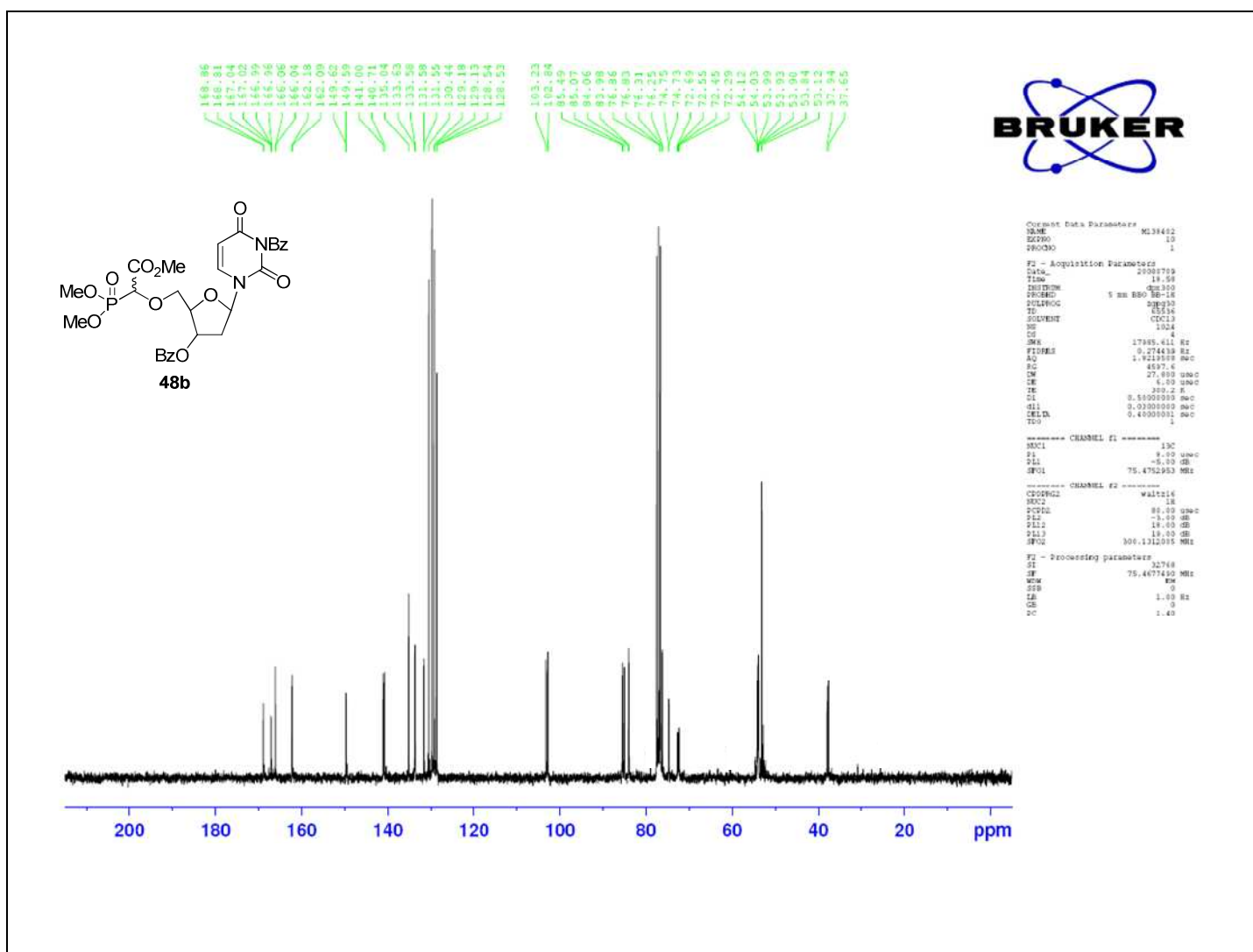
Compound **48a**.

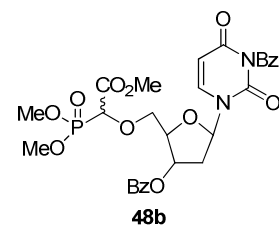




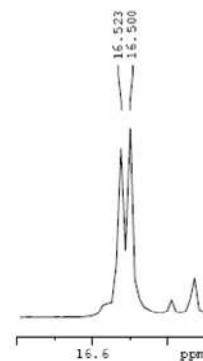
Compound **48b**.







16.523
16.500



Current Data Parameters
NAME M134414
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20080719
Time 14.52
INSTRUM spect
PROBHD 5 mm BBO BB-1X
PULPROG zgpg30
TD 65536
FIDRES 0.000000
SOLVENT CDCl3
NUC1 13C
NUC2 1H
P1 1.00
PL1 0.00
PC 1.00
FIDRES 0.000000
AQ 0.6734324 sec
RG 9192
TD 10.275 sec
DE 4.00 sec
TE 300.2 K
CL 0.0000000 sec
SOL 0.0000000 sec
DELTA 1.8999999 sec
TD0 1

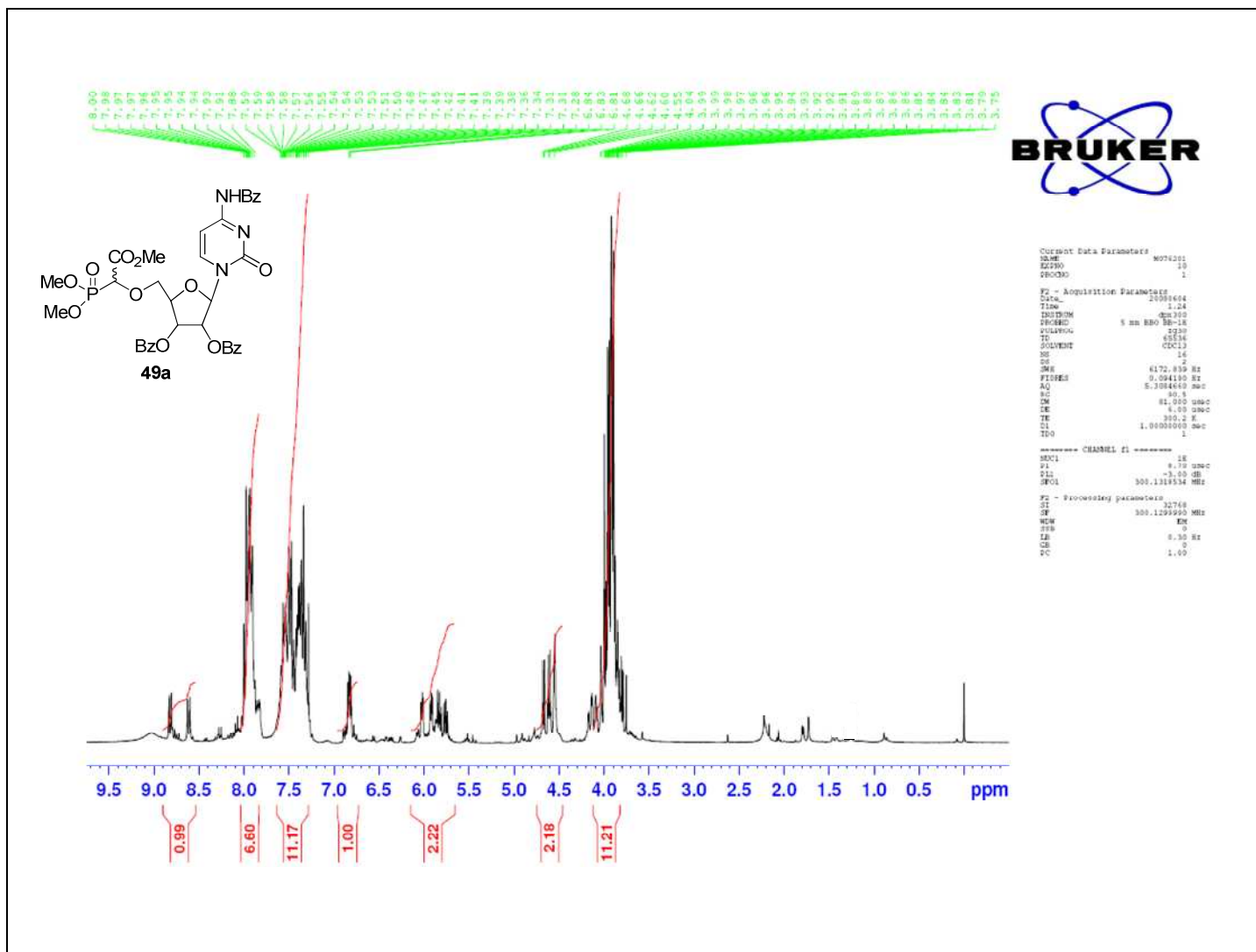
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NUC1 13C
P1 1.00 sec
PL1 -3.00 dB
SFO1 121.4807742 MHz

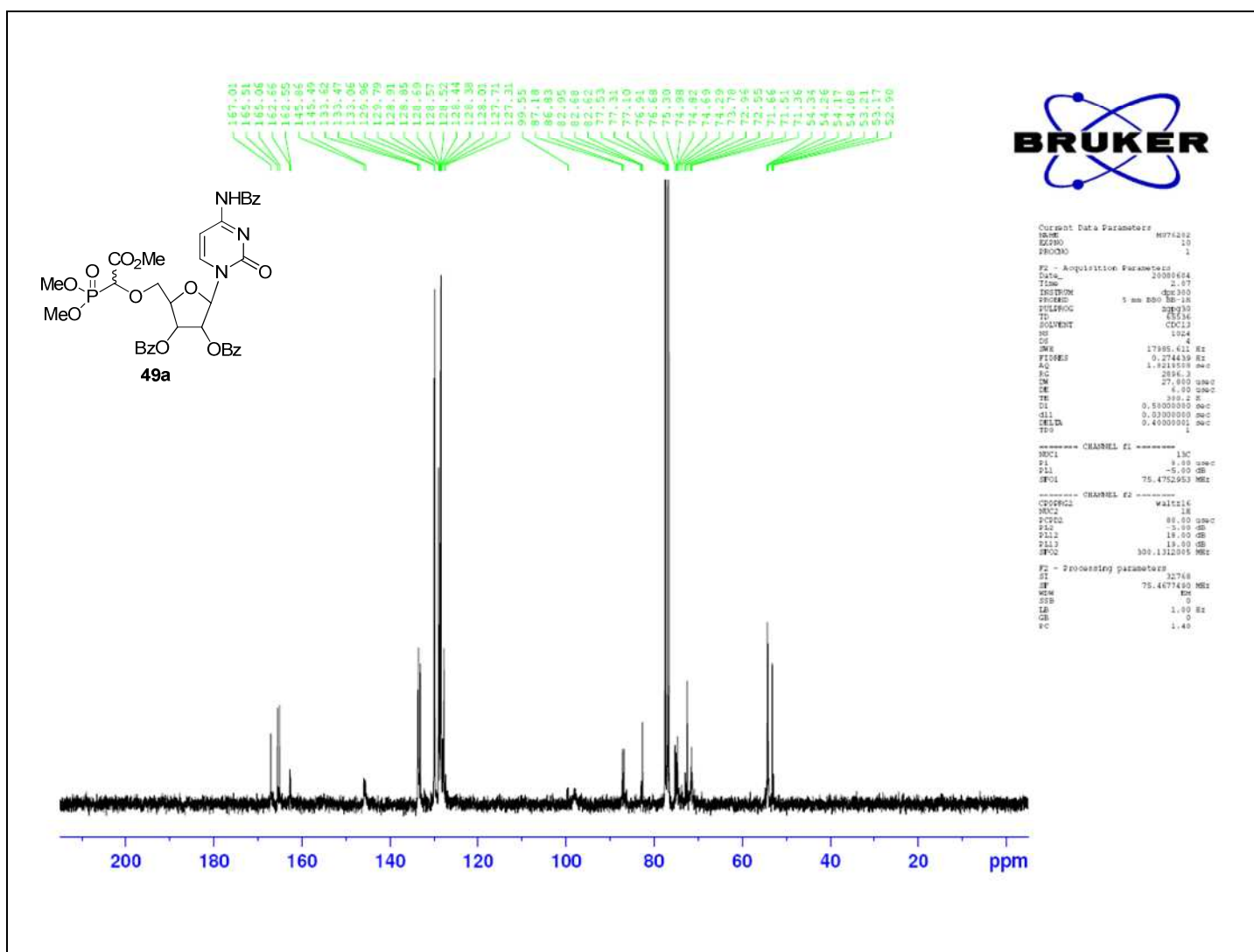
===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
P2 0.00 sec
PL2 -3.00 dB
PL12 19.00 dB
PL13 19.00 dB
SFO2 300.1312088 MHz

F2 - Processing parameters
SI 32768
SF 121.4949510 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

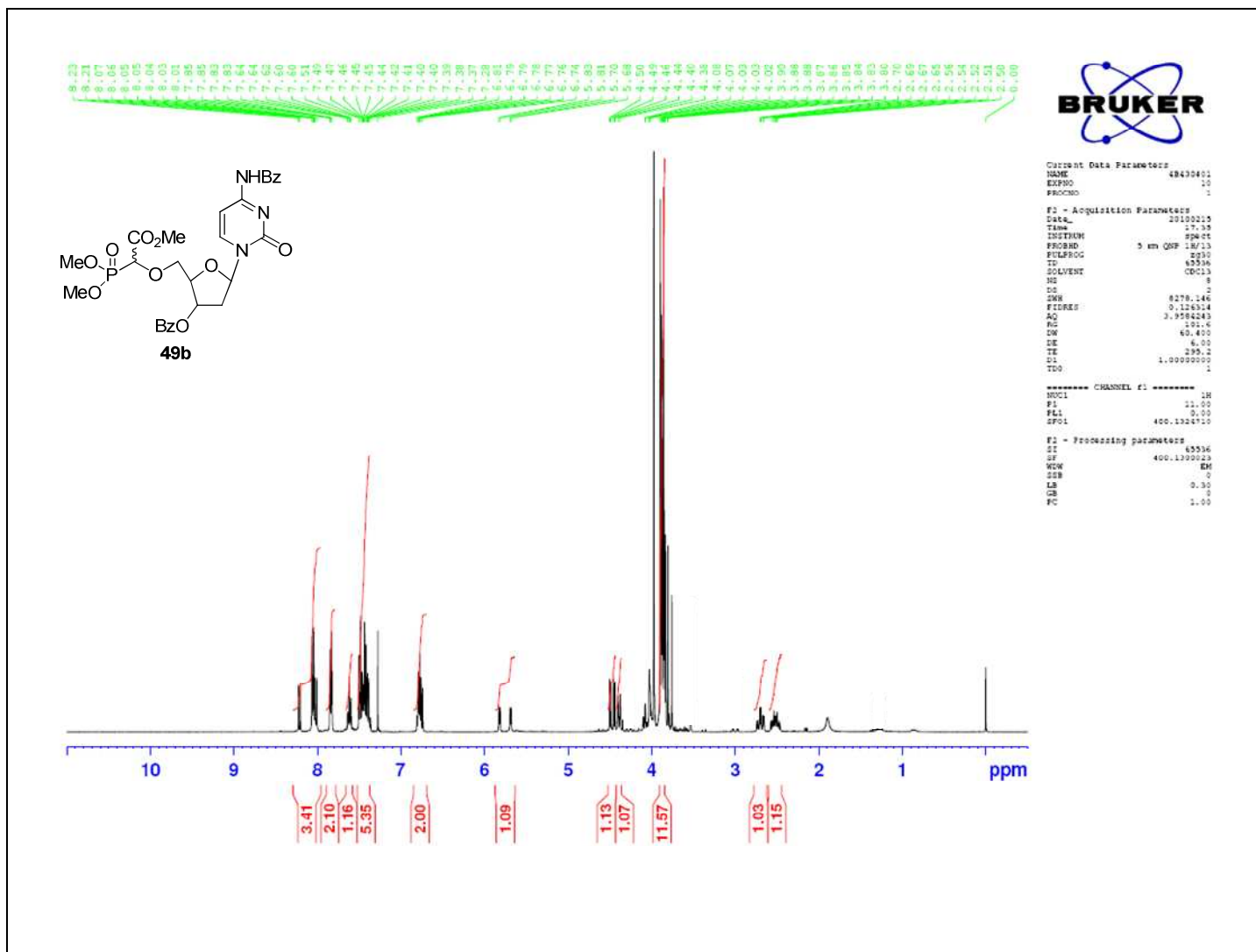


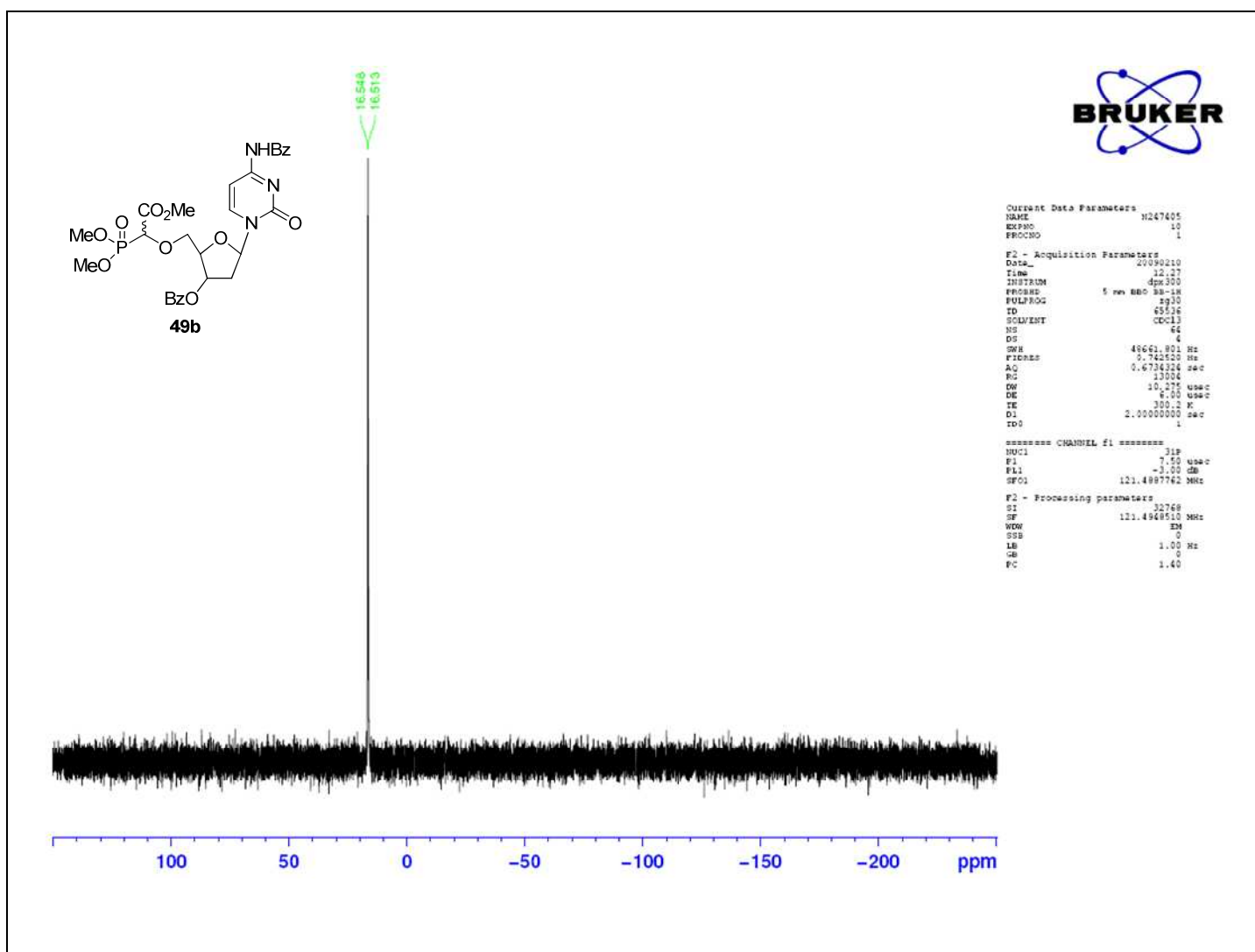
Compound **49a**.



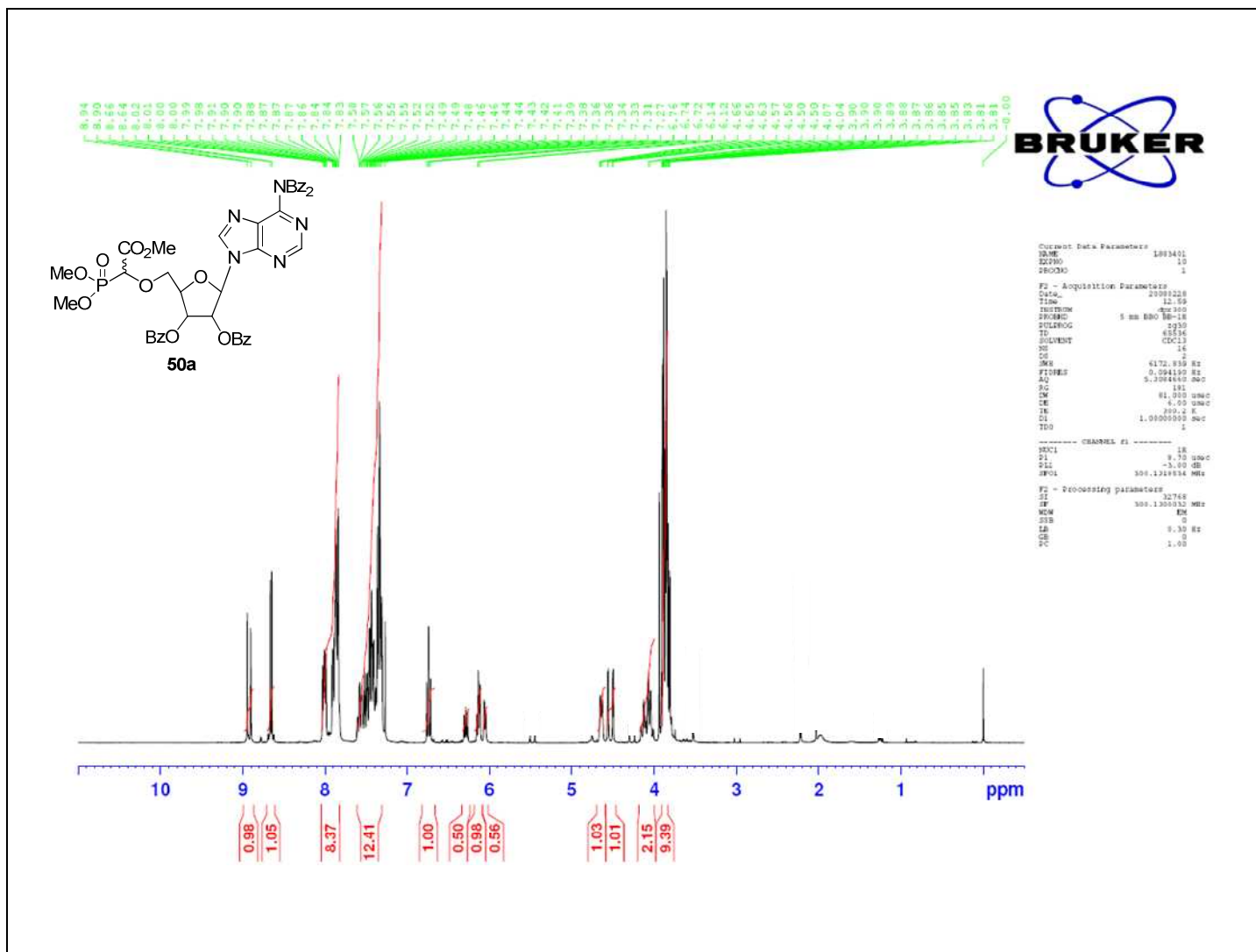


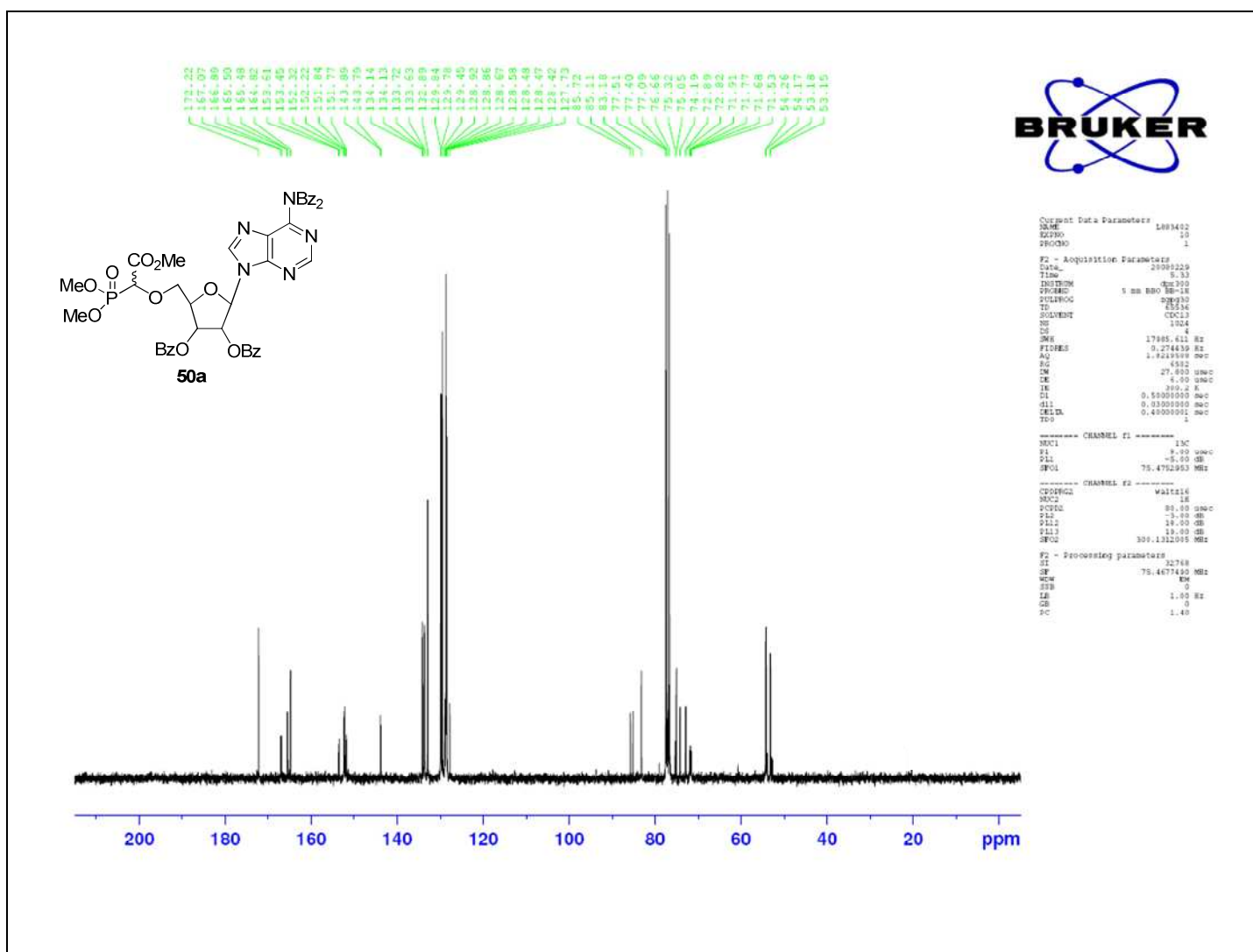
Compound **49b**.

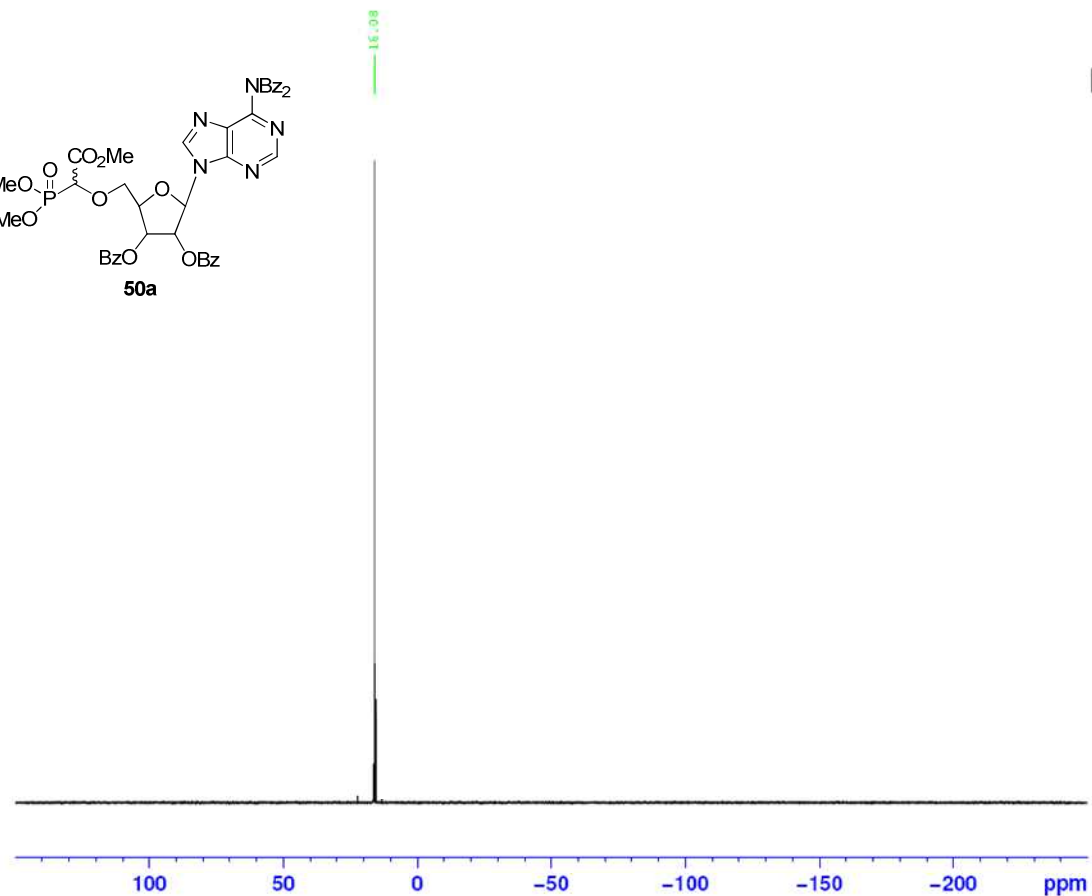
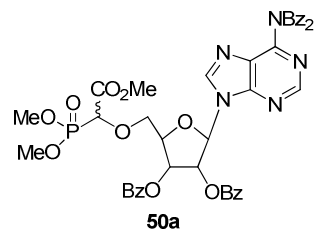




Compound **50a**.







```

Current Data Parameters
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EXPNO     10
PROCNO    1

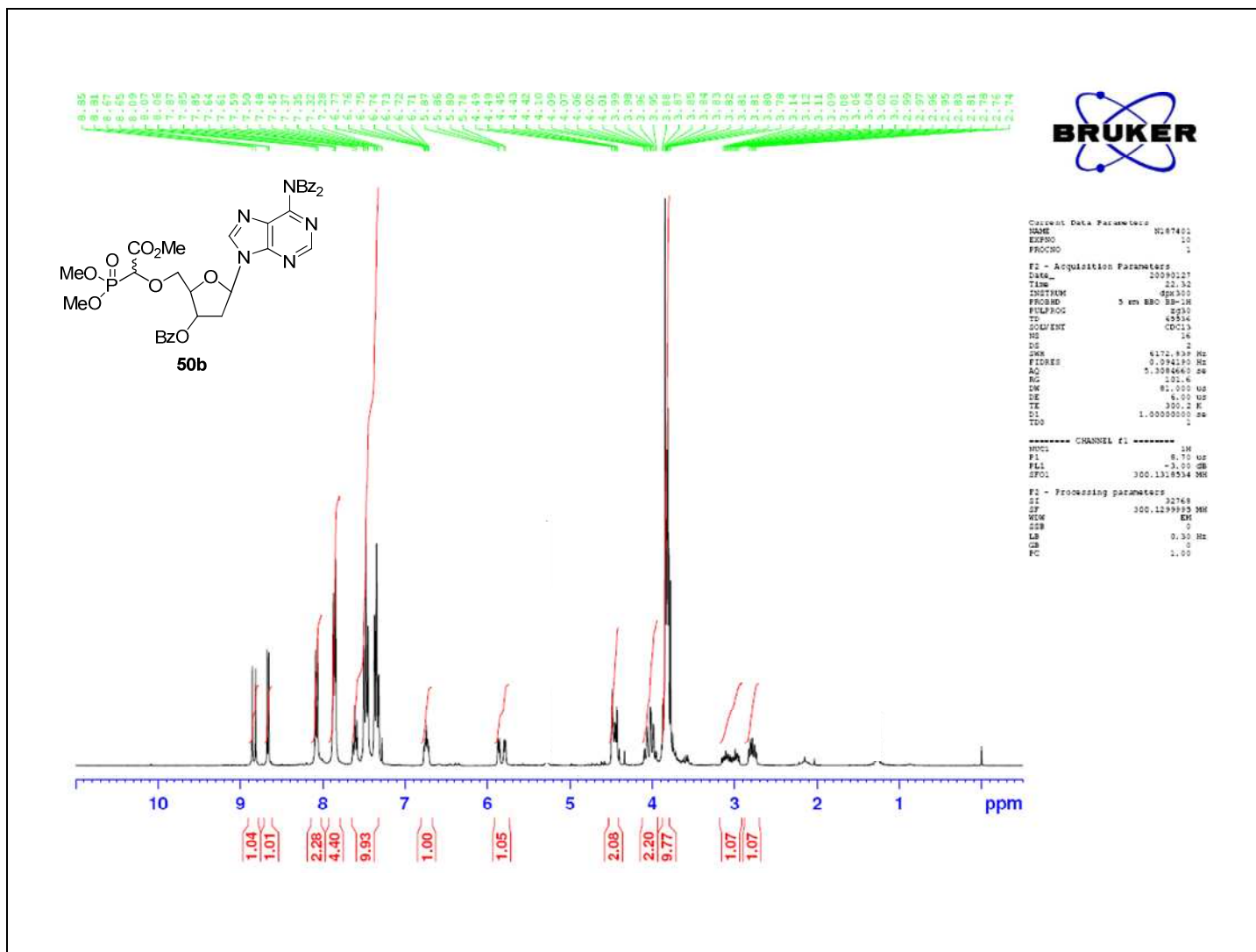
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Time      13.52
INSTRUM   spect
PROBHD    5 mm BBO 1H-1
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         16
DS         4
SWH        40641.801 Hz
FIDRES     0.742350 Hz
AQ         0.6734324 sec
RG         20642.5
SN         10.215 umw
RM         4.40 umw
TE         300.2 K
D1         2.0000000 sec
d11        0.0300000 sec
DELTA      1.8999998 sec
TD0        1

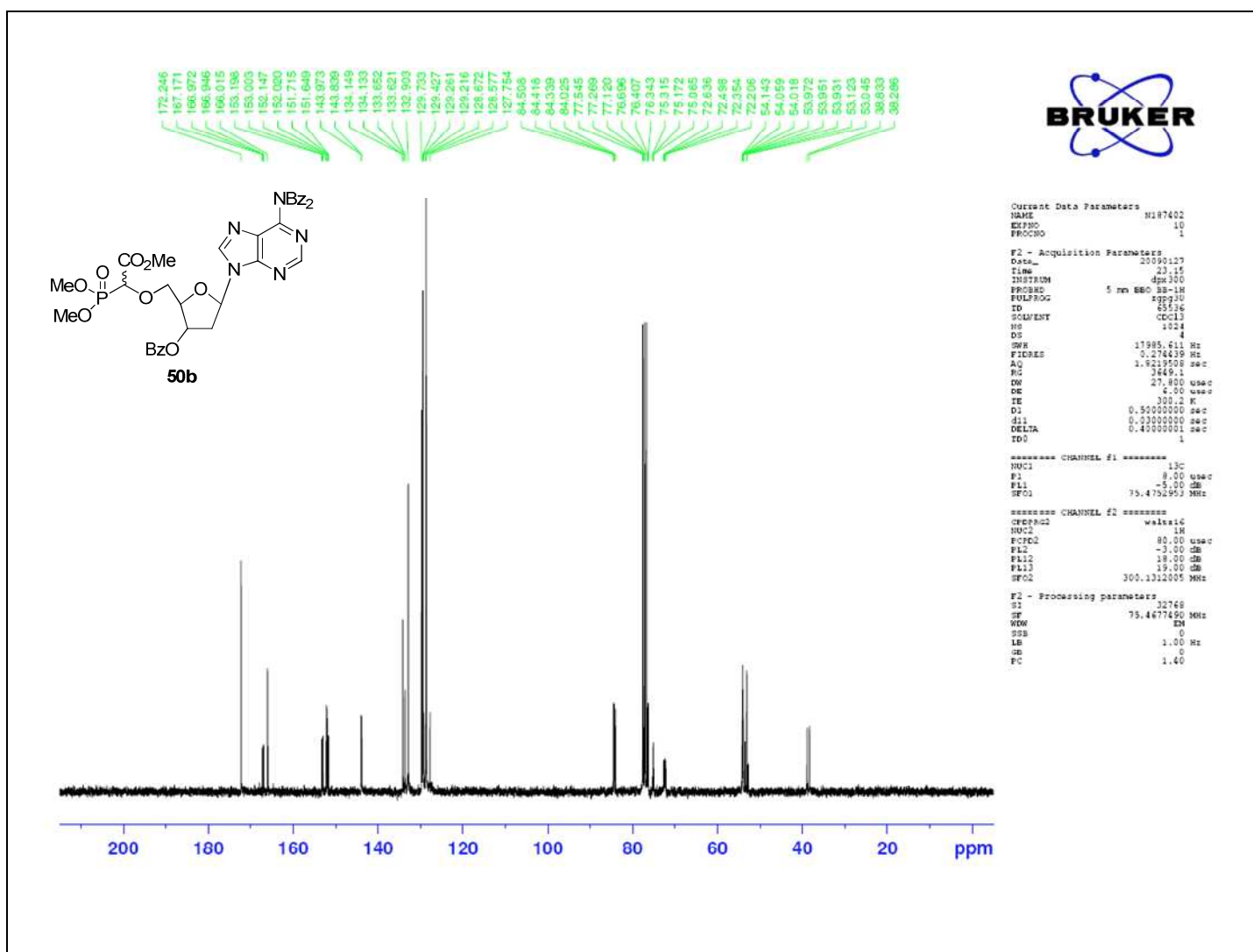
===== CHANNEL f1 =====
NUC1       13C
P1         7.50 umw
PL1        -1.50 dB
RF1        121.487712 MHz

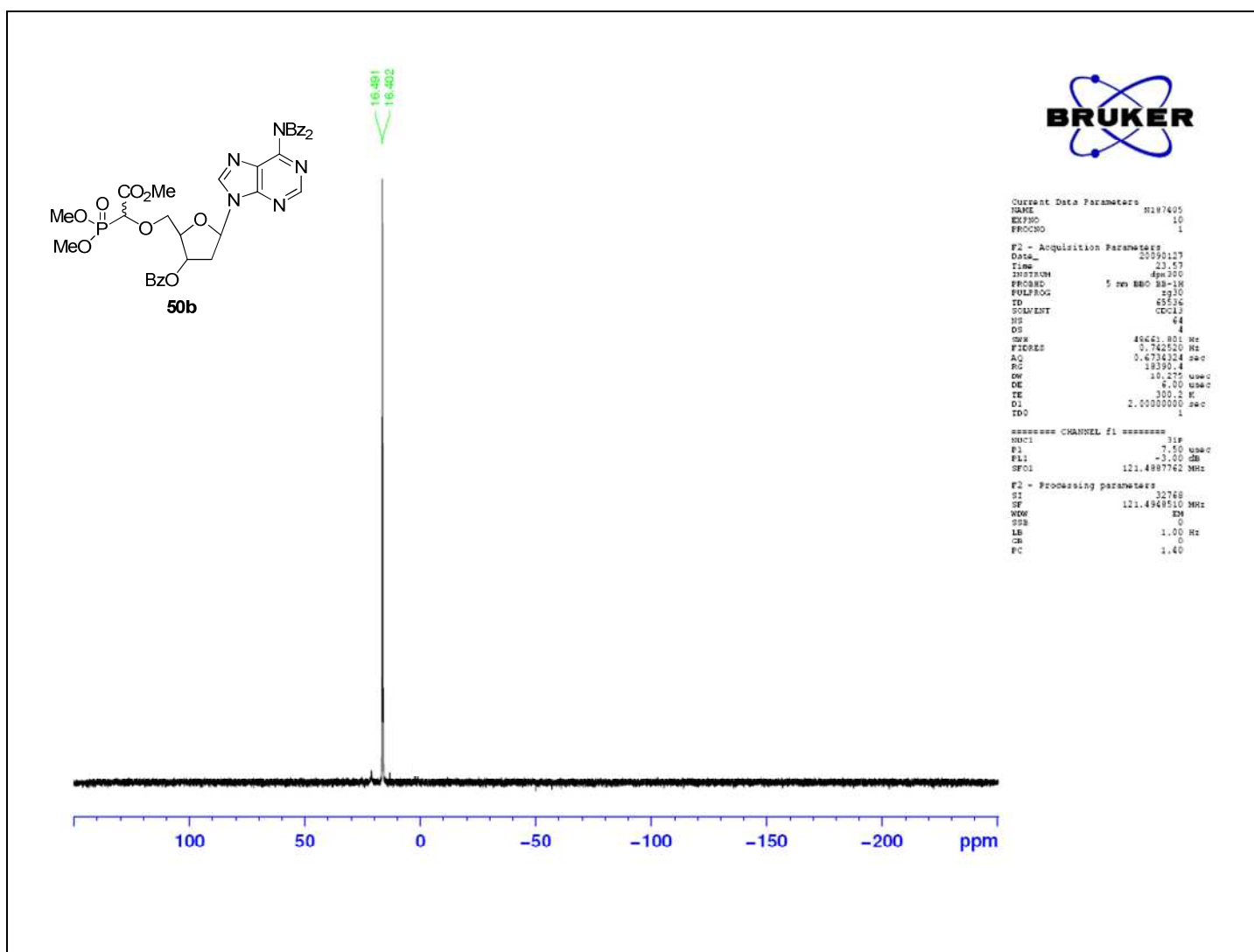
===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2       1H
PCPD2     80.00 umw
PL2        -2.00 dB
PL12       19.00 dB
PL13       19.00 dB
RF2        300.1312005 MHz

F2 - Processing parameters
SI         32768
SF         121.4944910 MHz
WDW        EM
SSB        0
LA         1.00 Hz
GB         0
PC         1.40
  
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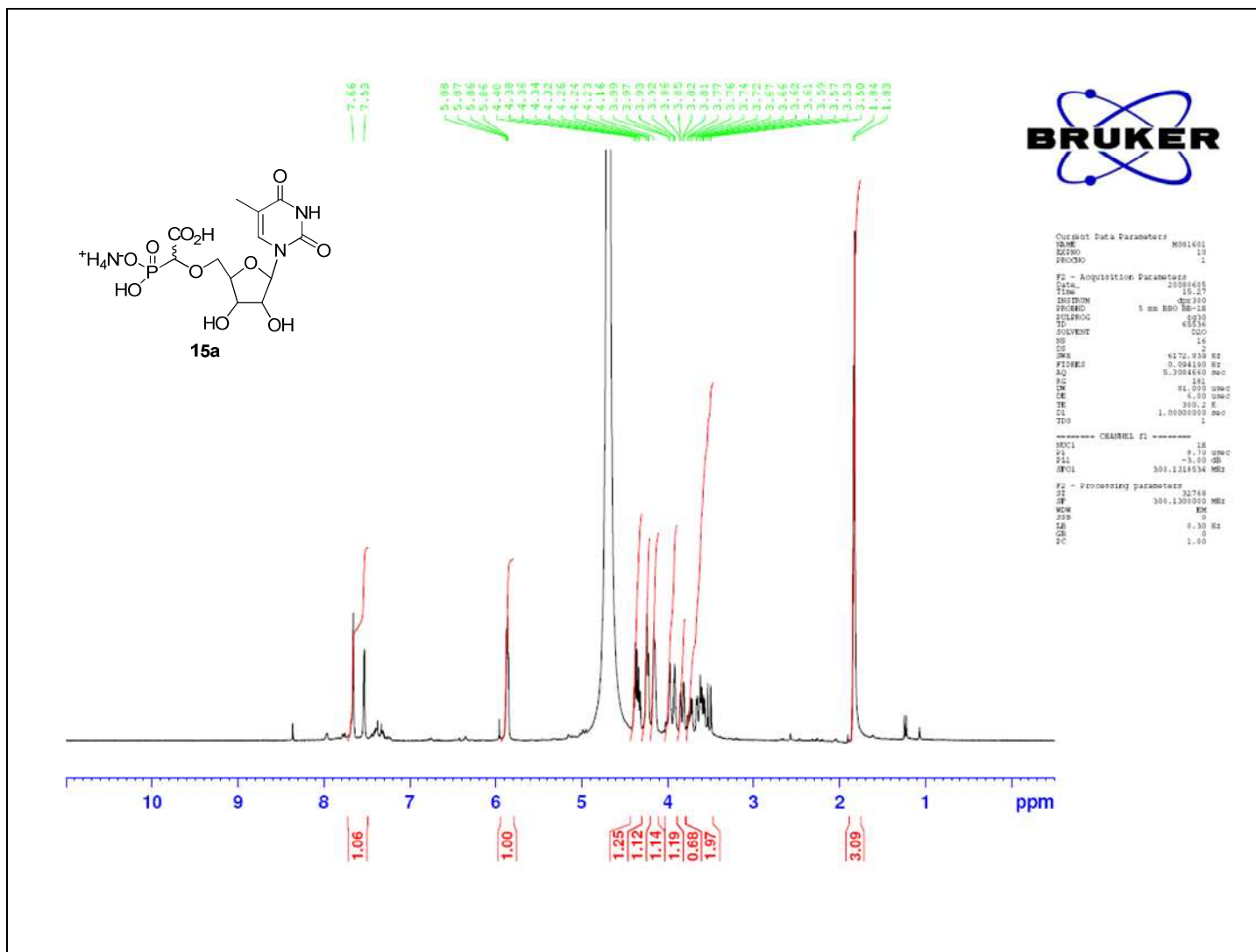
Compound **50b**.

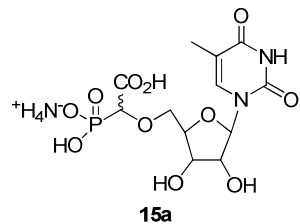






Compound **15a**.





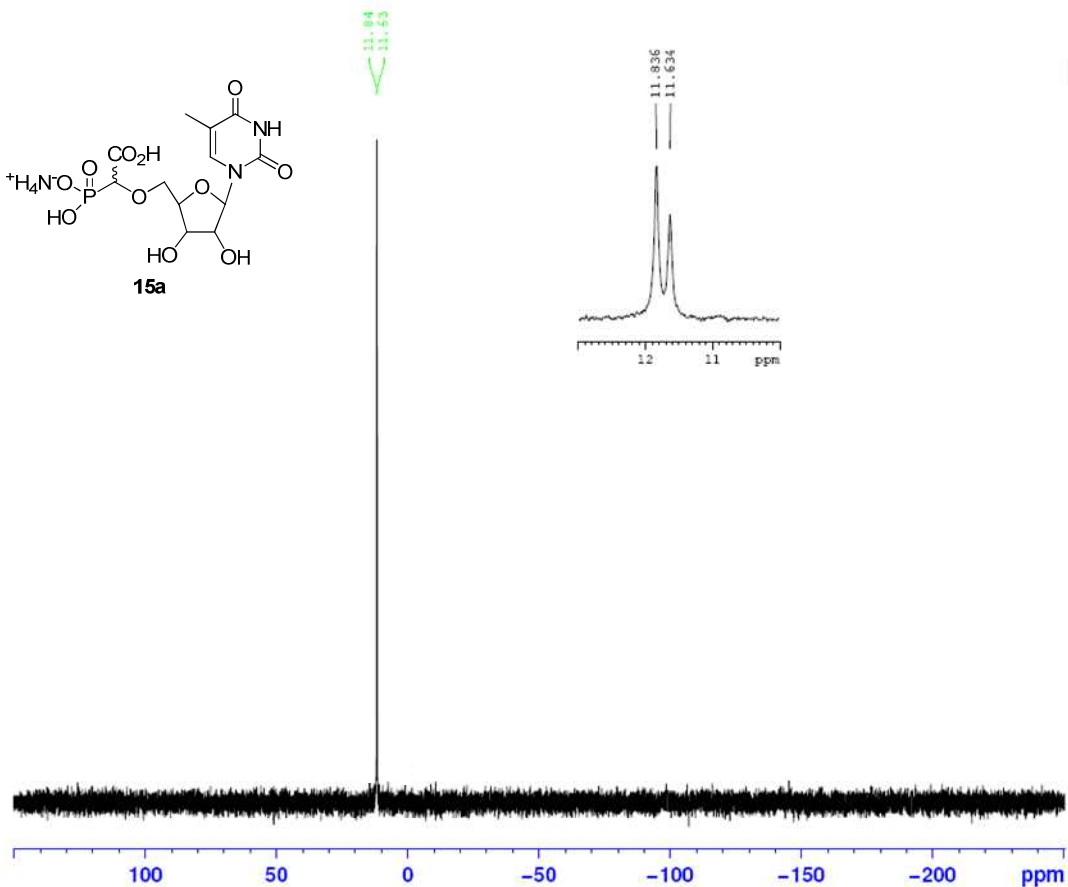
Current Data Parameters
NAME 0001001
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20080604
Time 22:51
INSTRUM spect
PROBHD 5 mm BBO 50-1A
PULPROG zgpg30
TD 65536
SOLVENT D2O
NS 4
DS 4
SWH 40661.001 Hz
FIDRES 0.742520 Hz
AQ 0.6714934 sec
RG 20640.5
CW 10.275 usec
DE 4.00 usec
TE 300.2 K
CL 2.0000000 sec
d11 0.0000000 sec
DELTA 1.9999999 sec
TD0 1

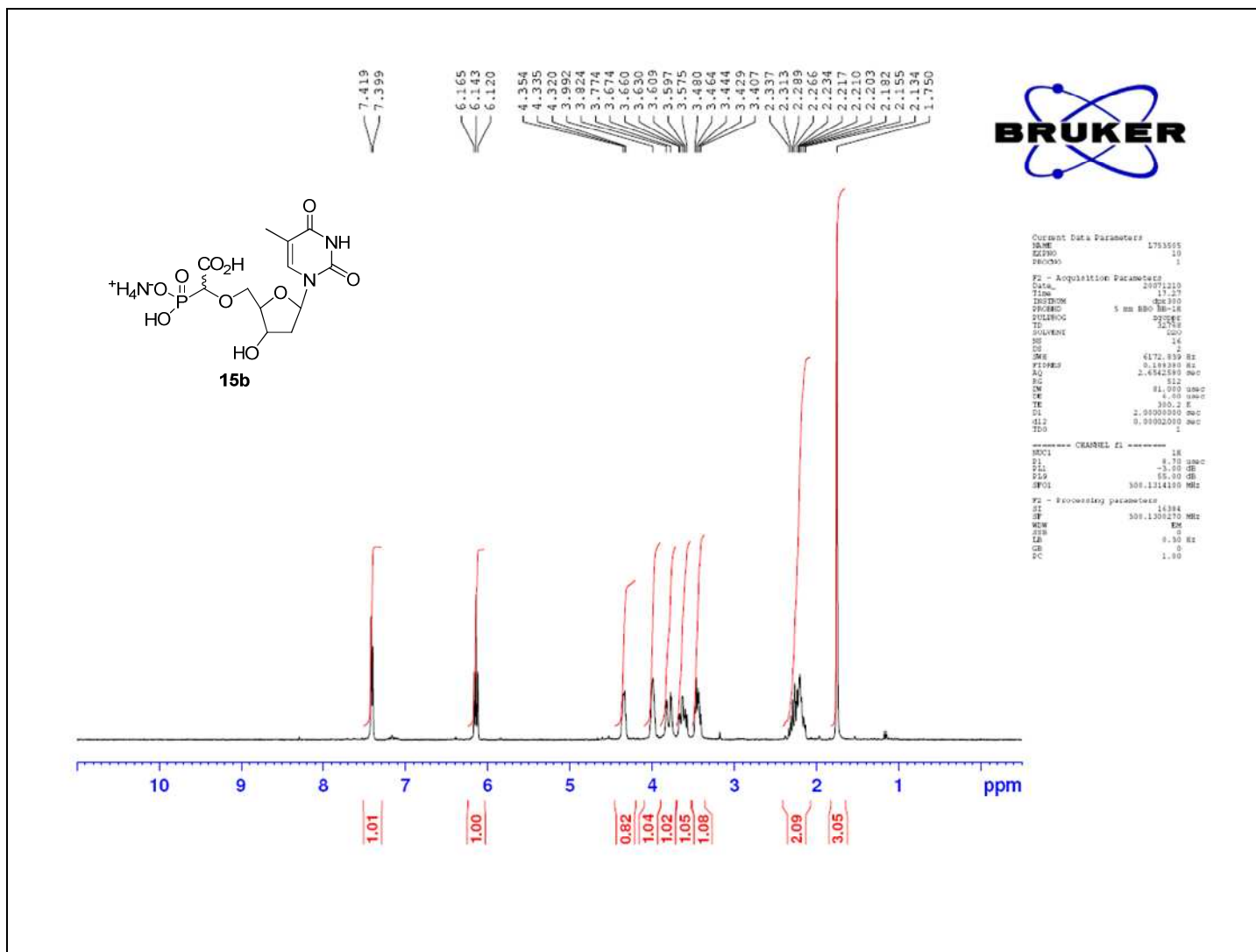
===== CHANNEL F1 =====
NUC1 31P
P1 1.00 usec
PL -3.00 dB
SFO1 121.4987742 MHz

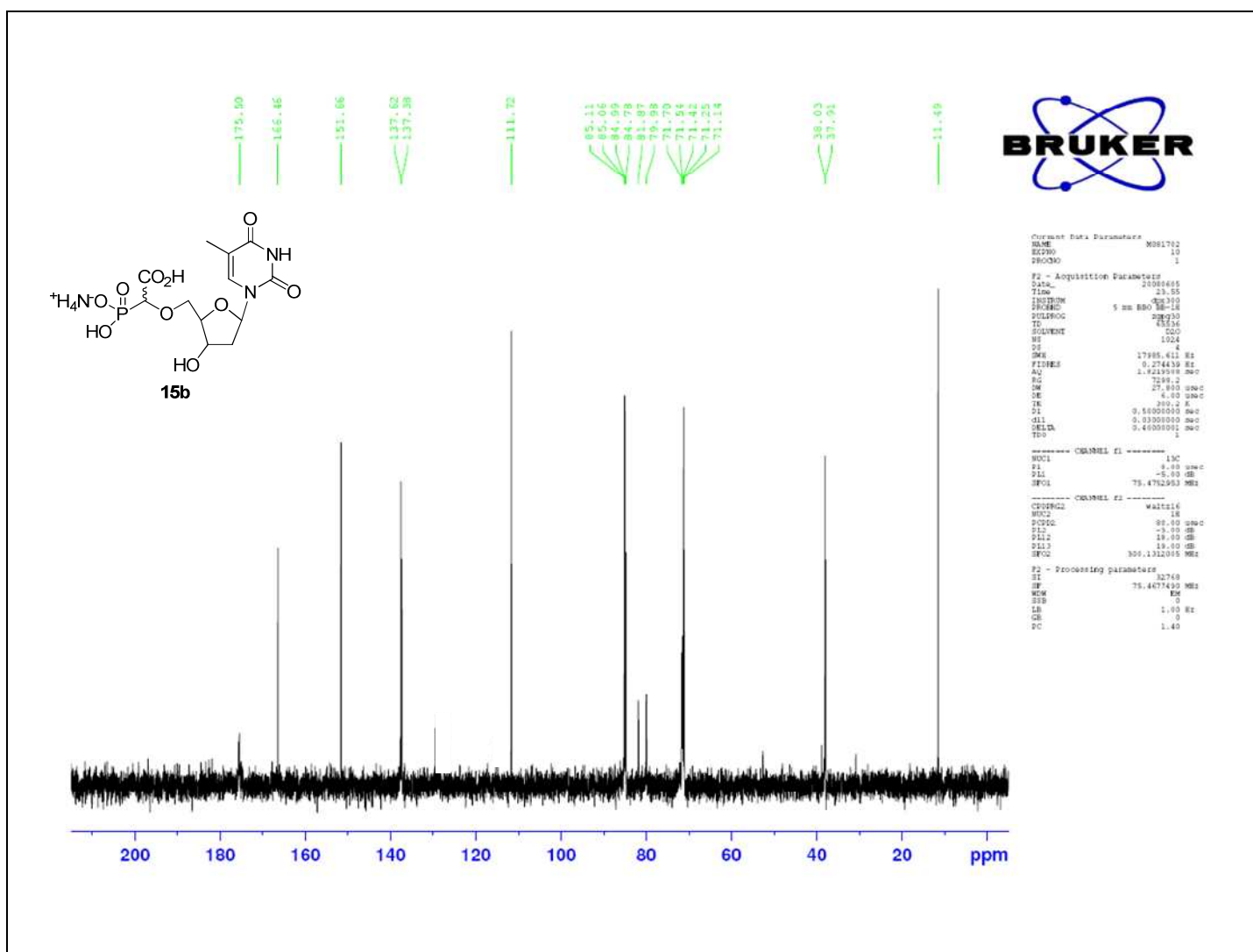
===== CHANNEL F2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -3.00 dB
PL12 19.00 dB
PL13 19.00 dB
SFO2 300.1312015 MHz

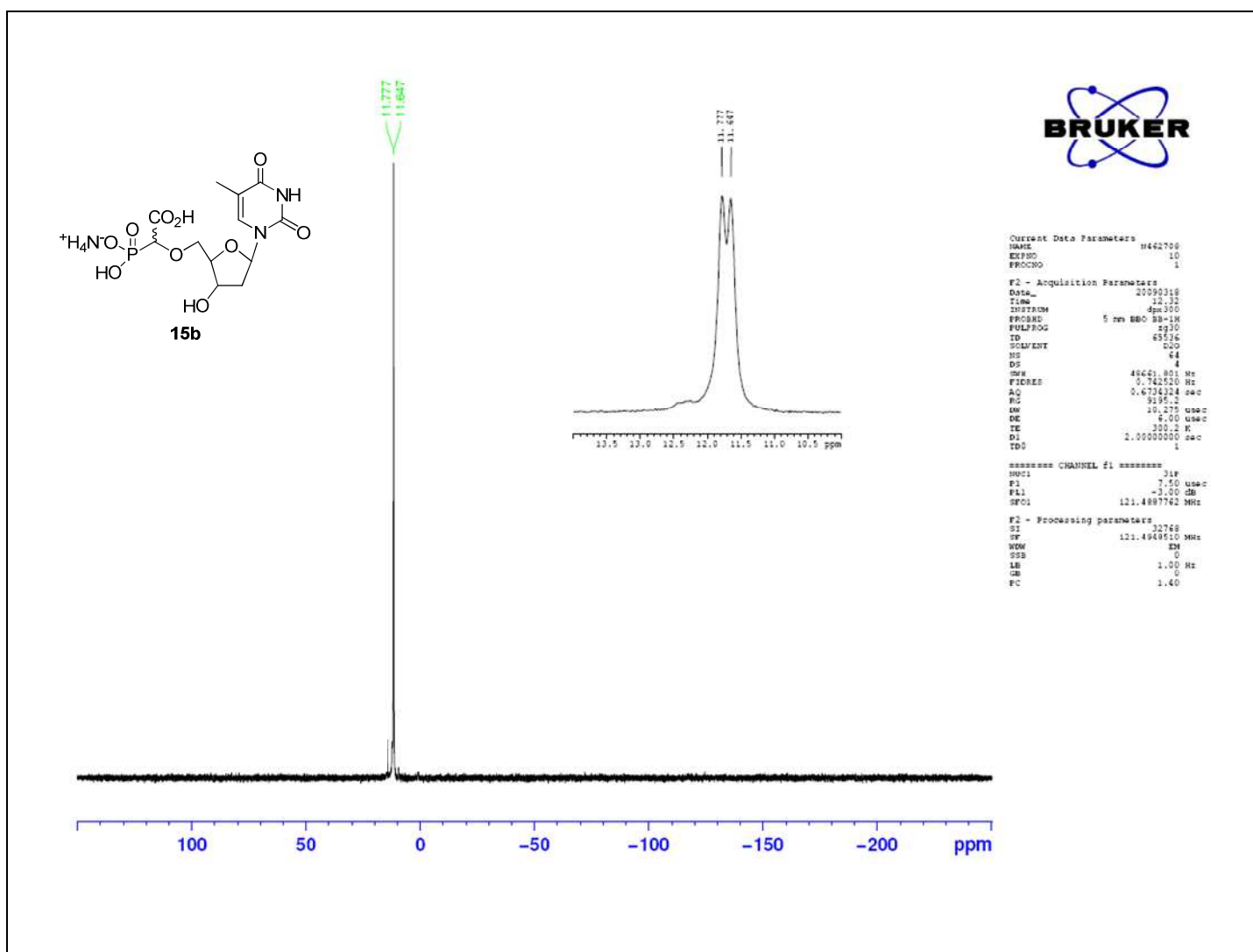
F2 - Processing parameters
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SF 121.4949510 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



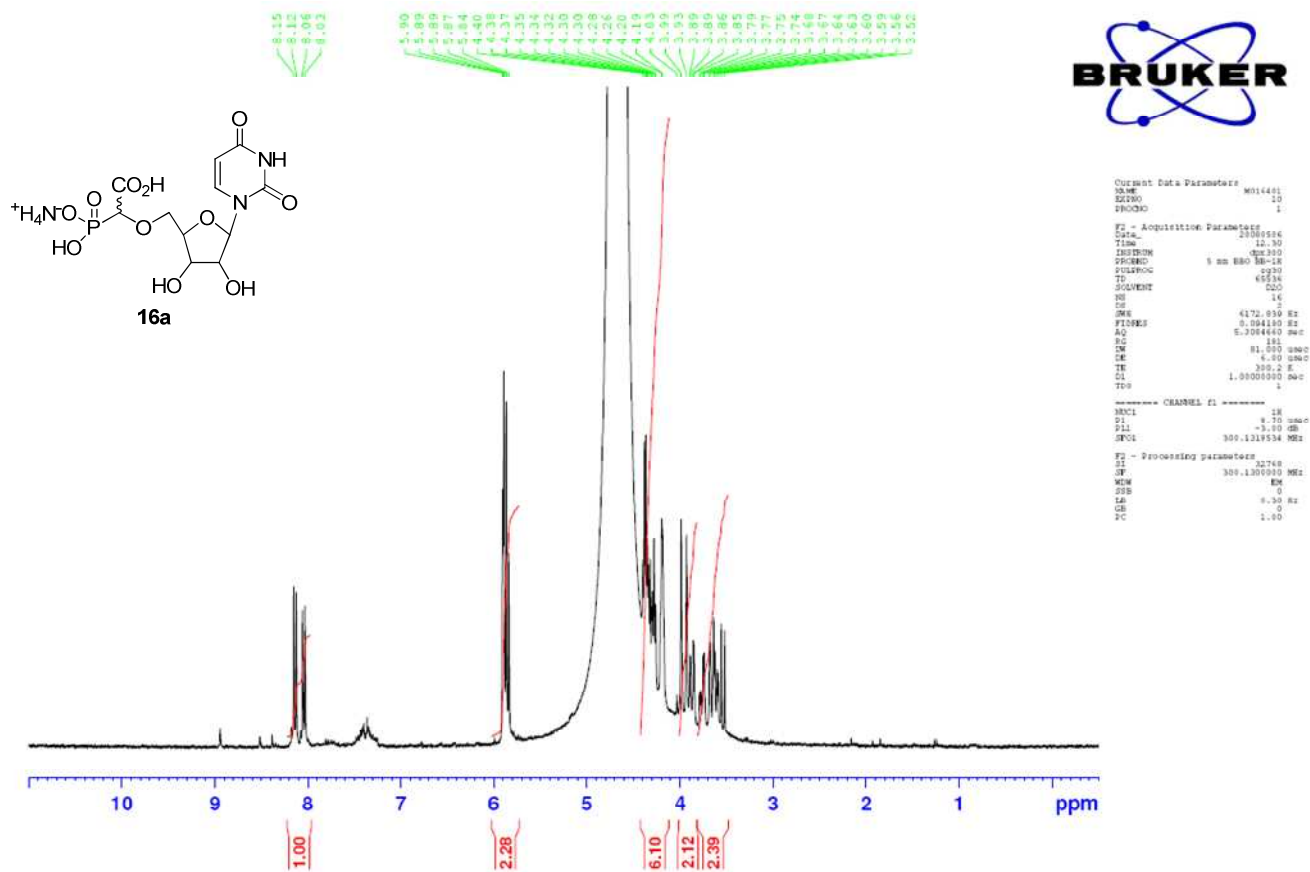
Compound **15b**.

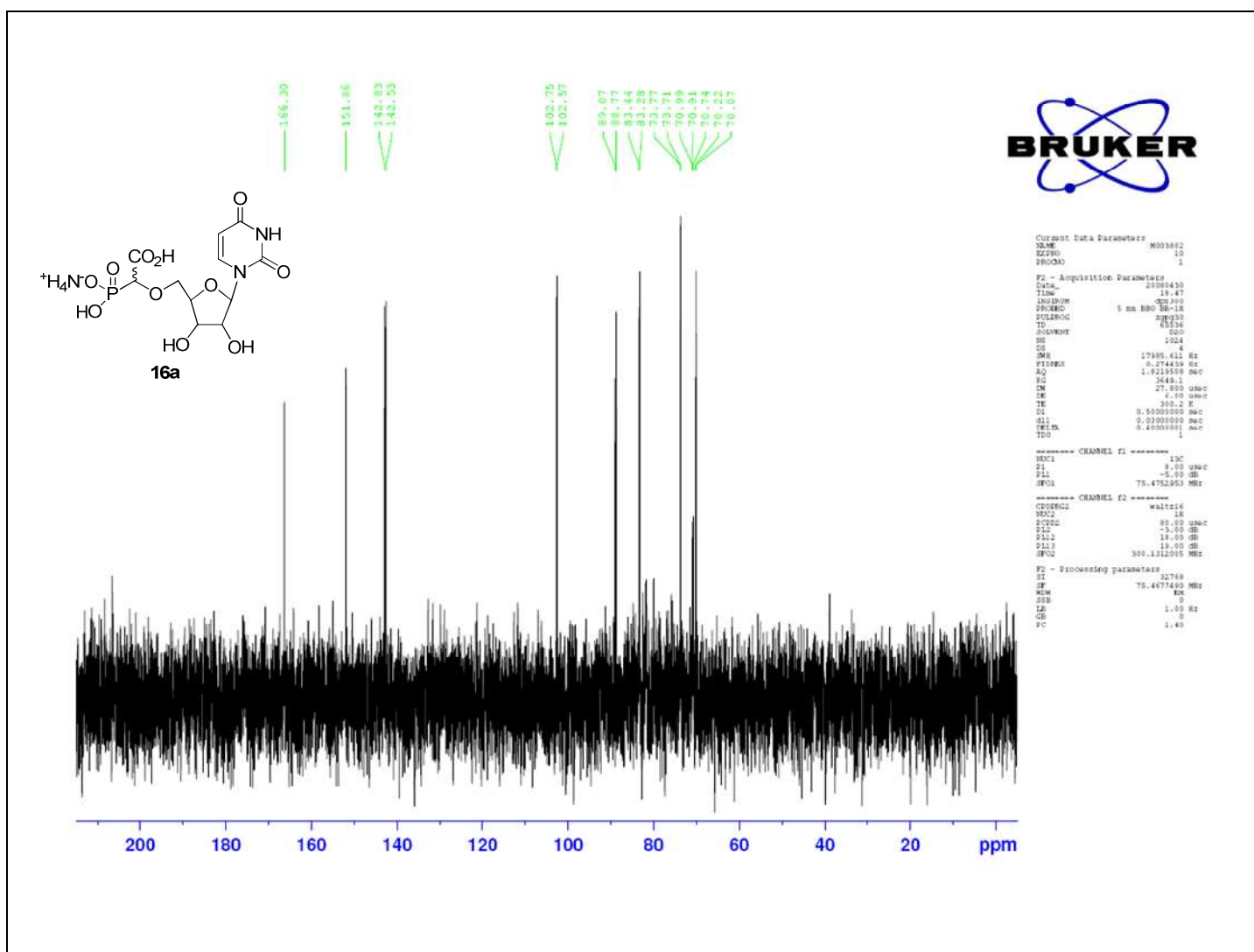


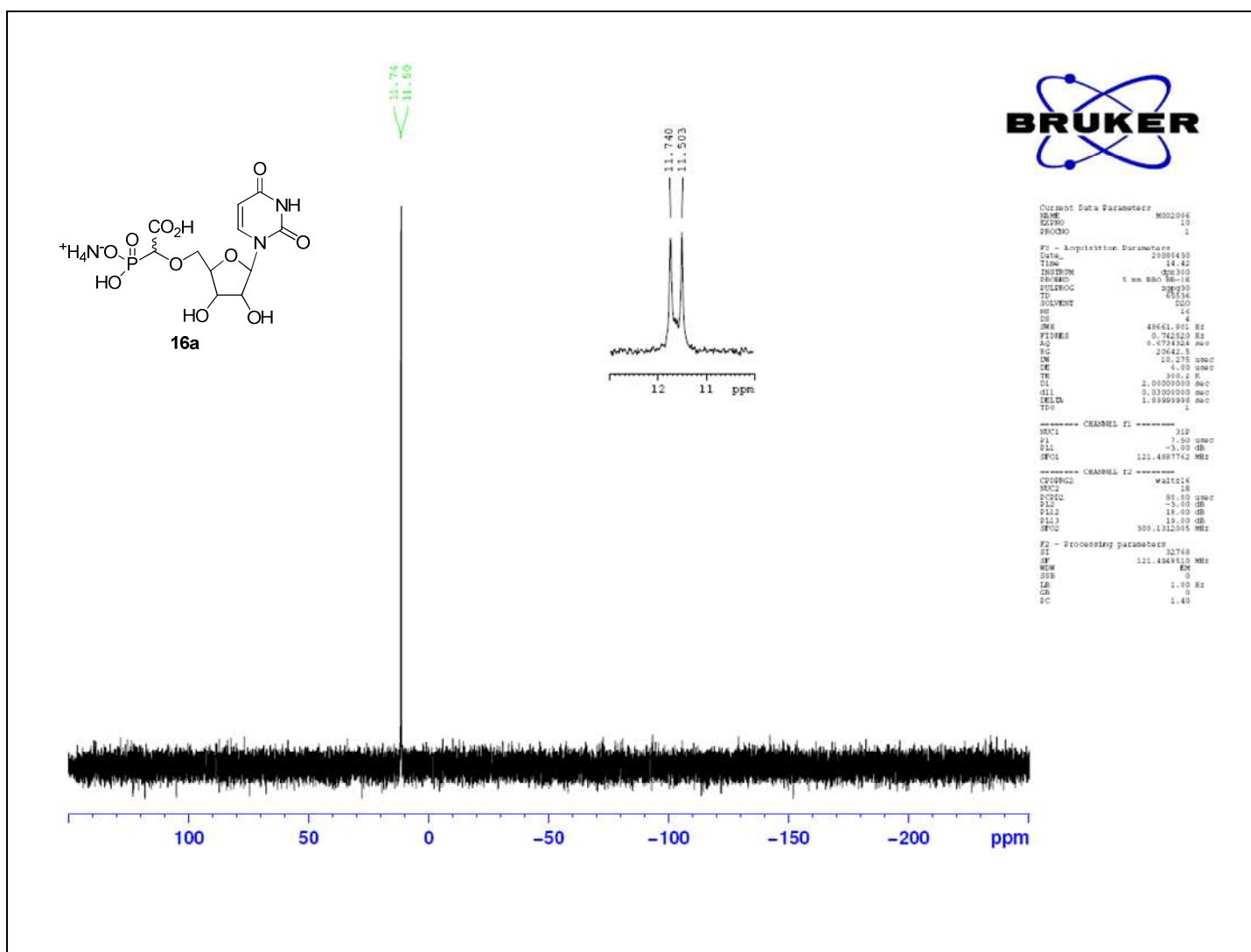




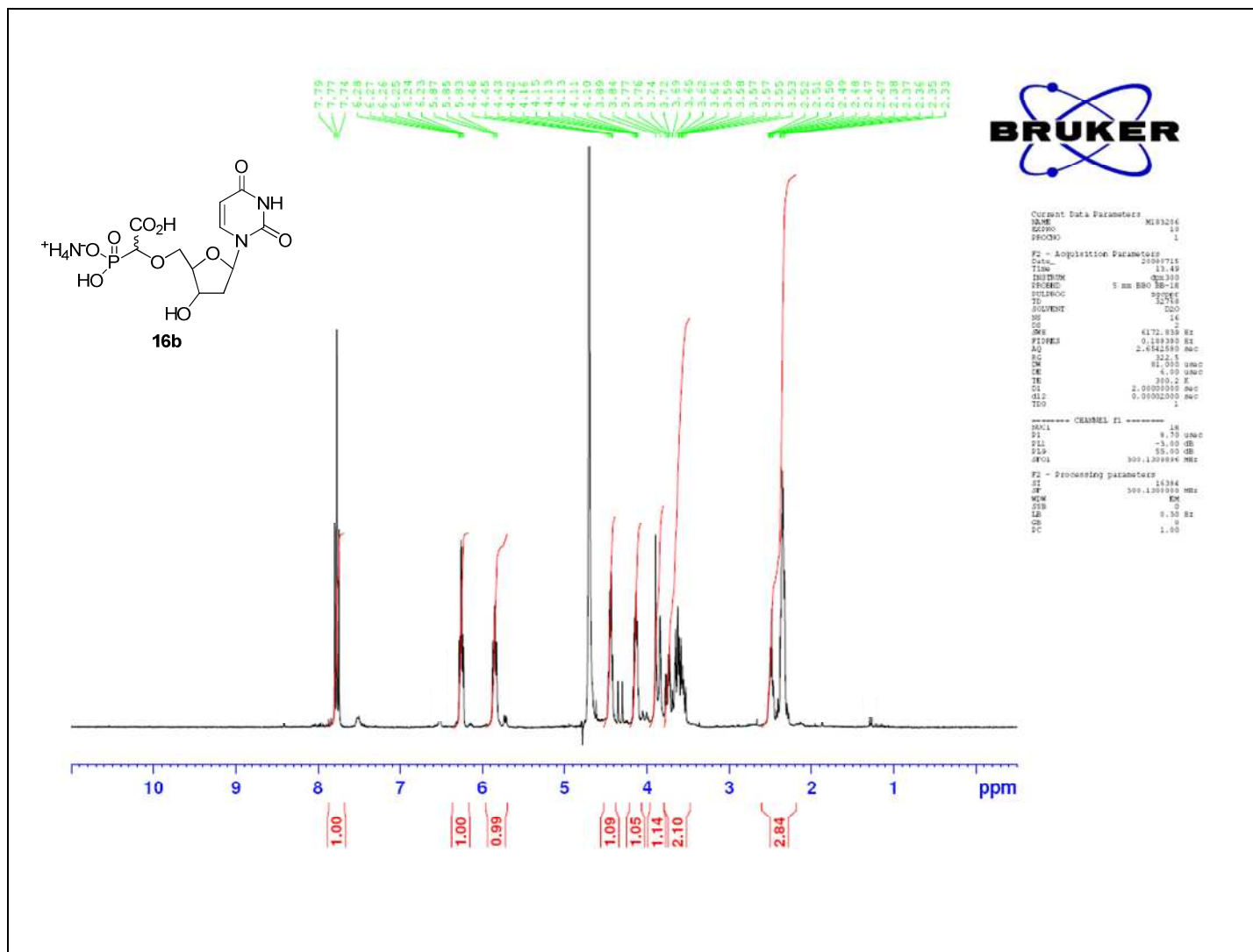
Compound **16a**.

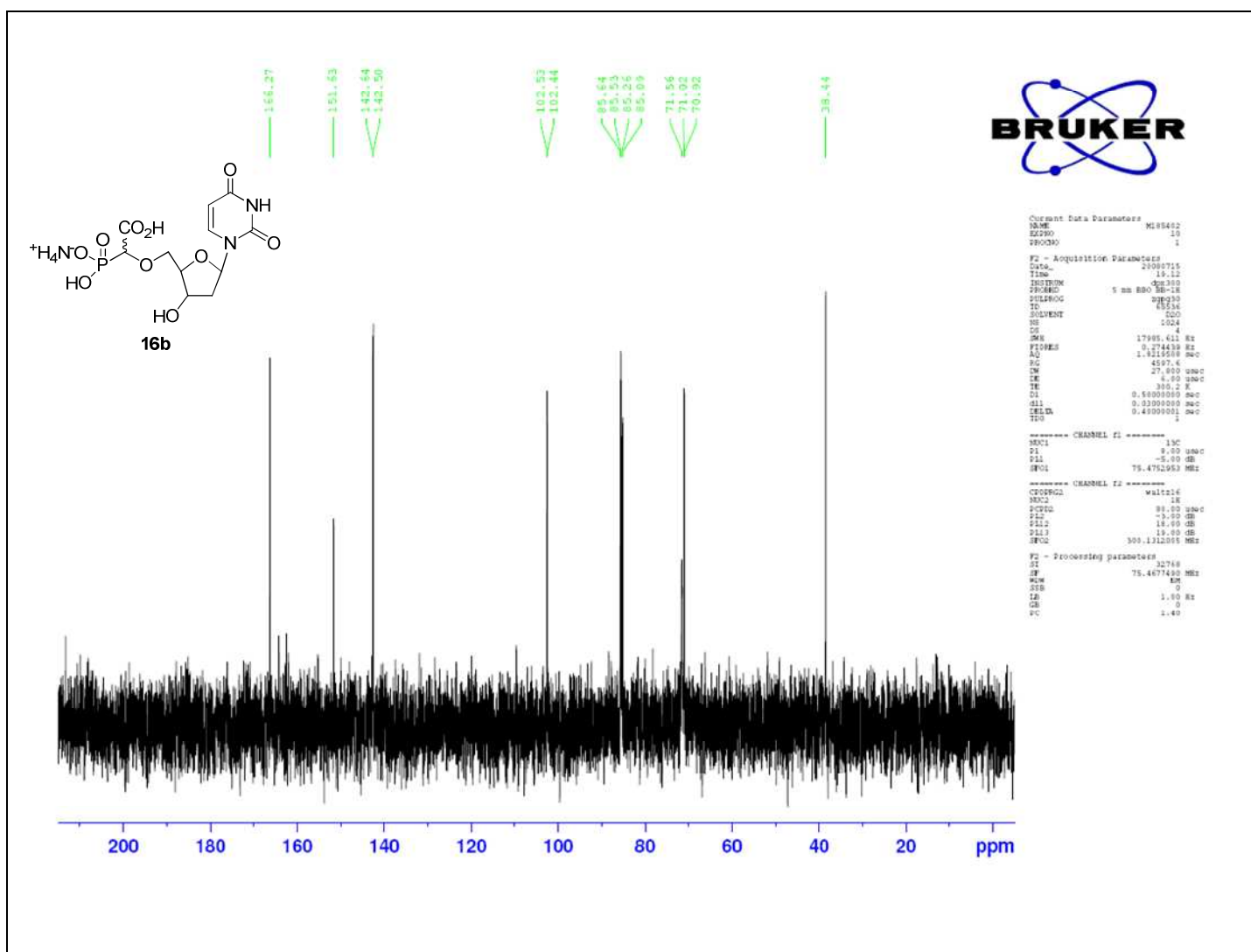


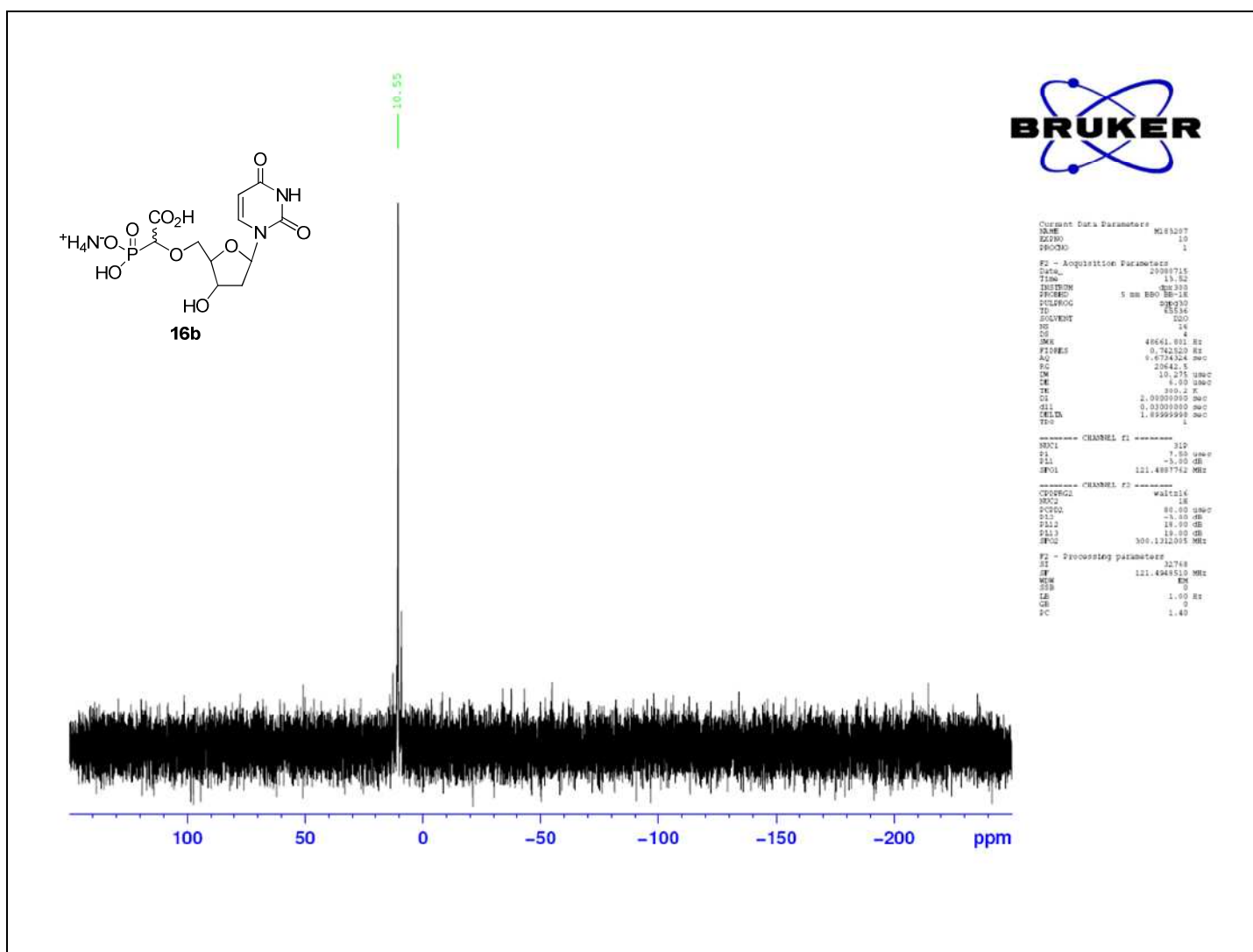




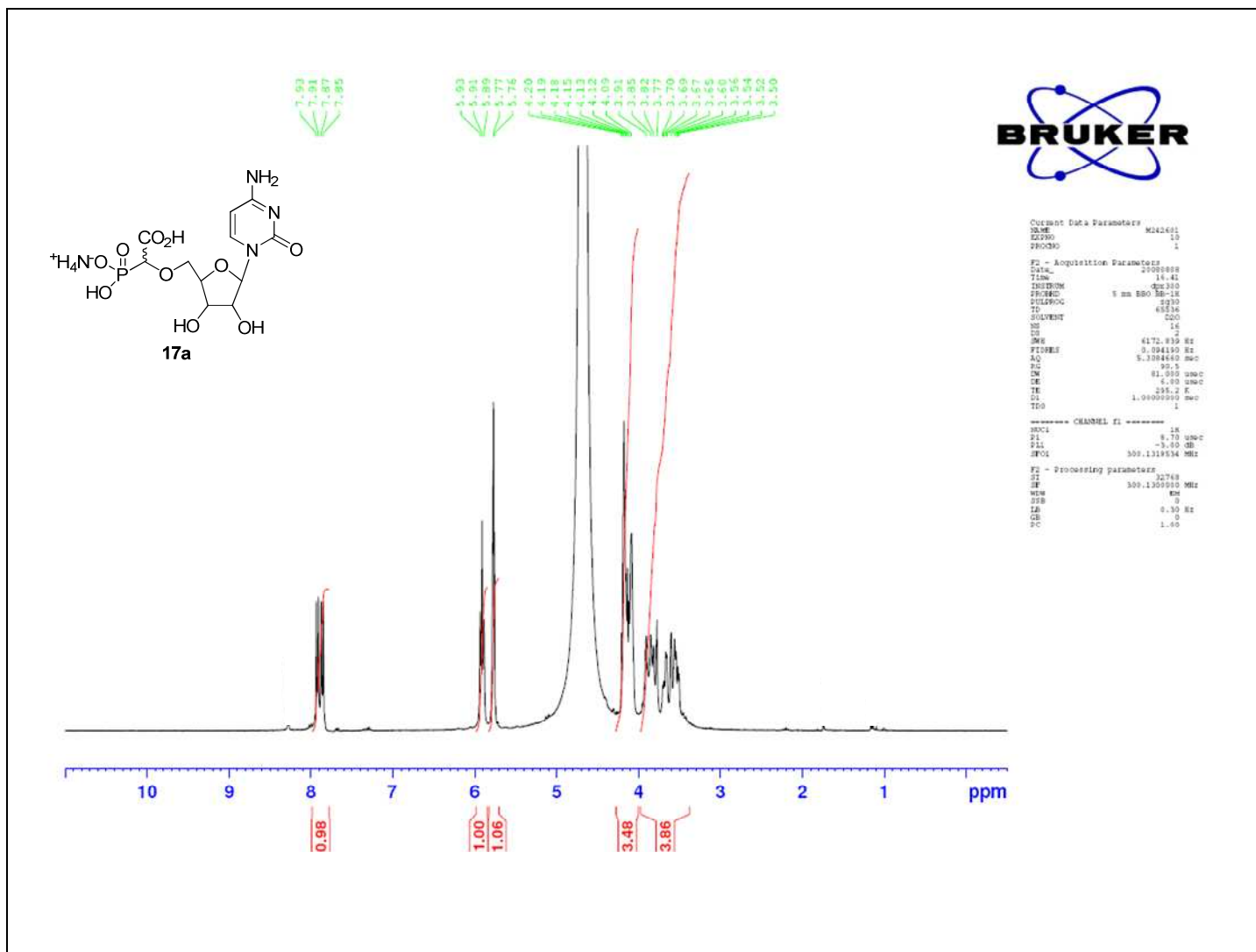
Compound **16b**.

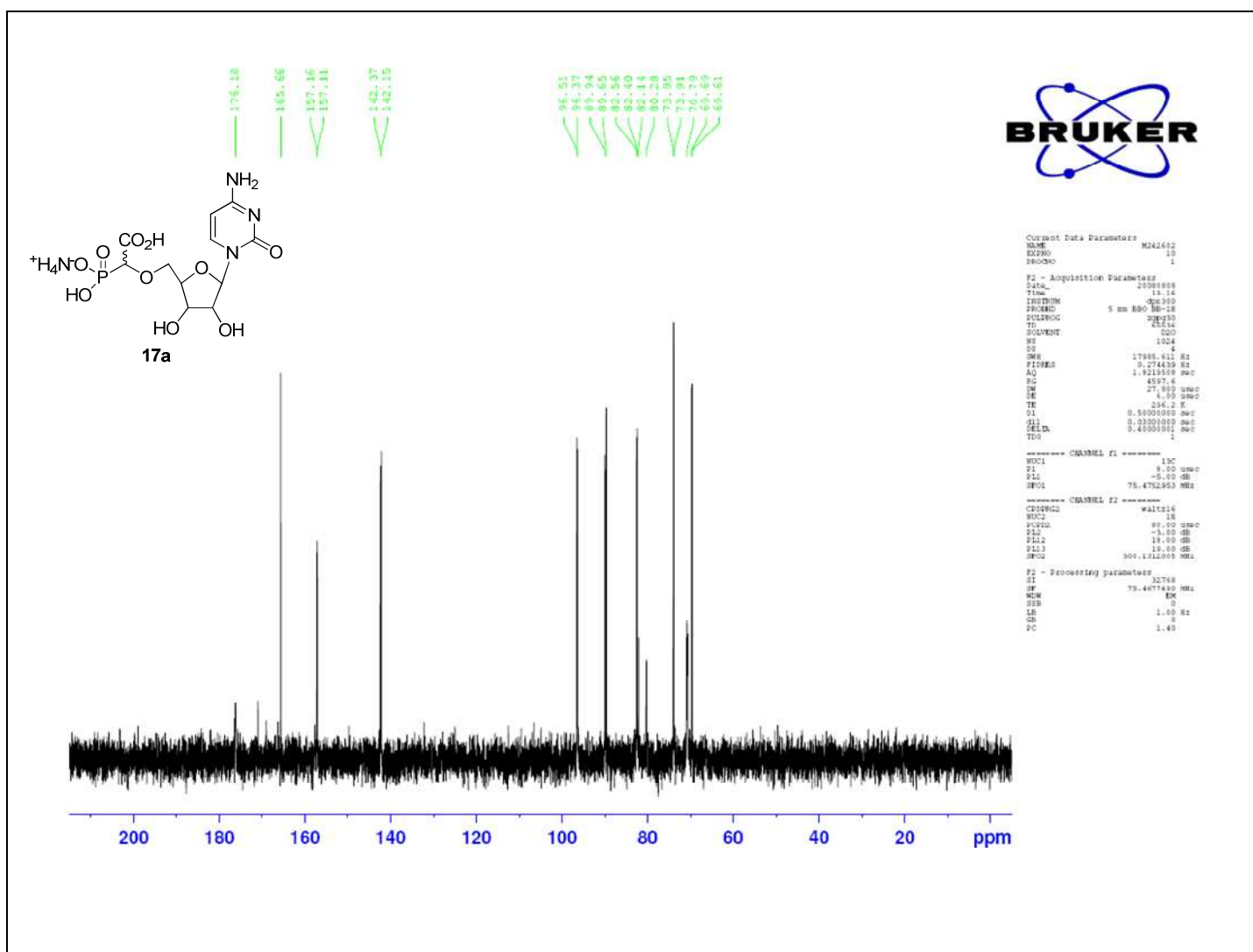


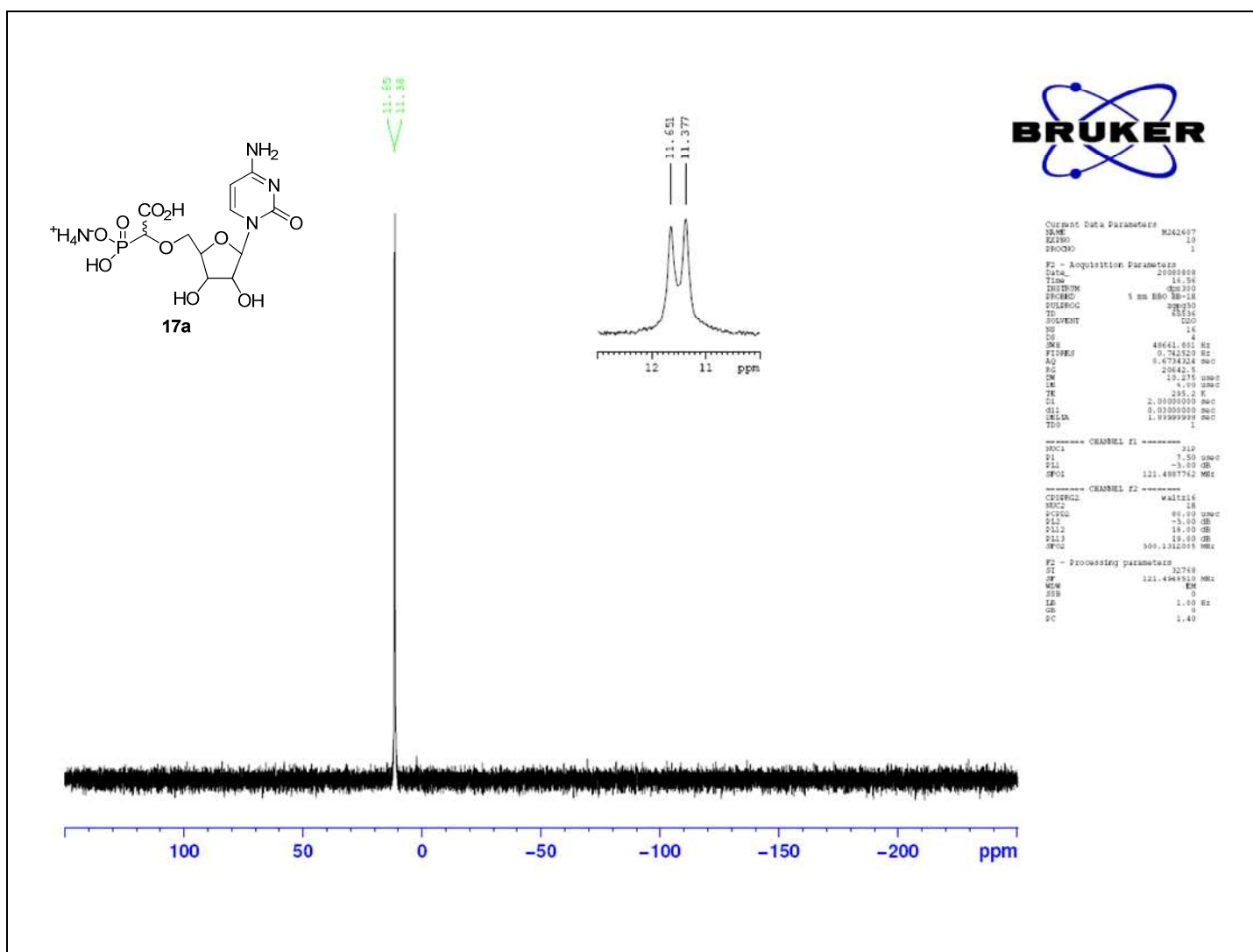




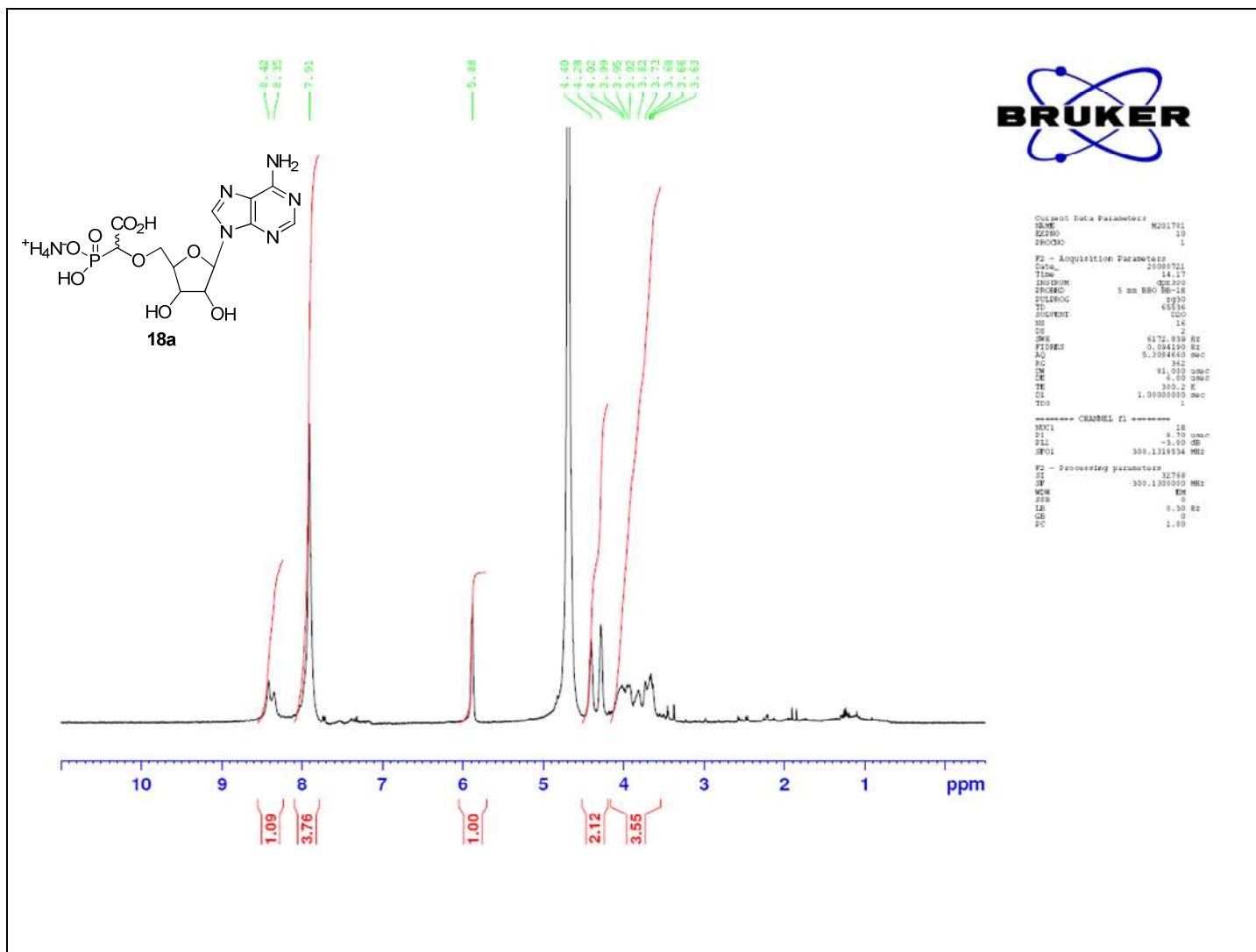
Compound **17a**.

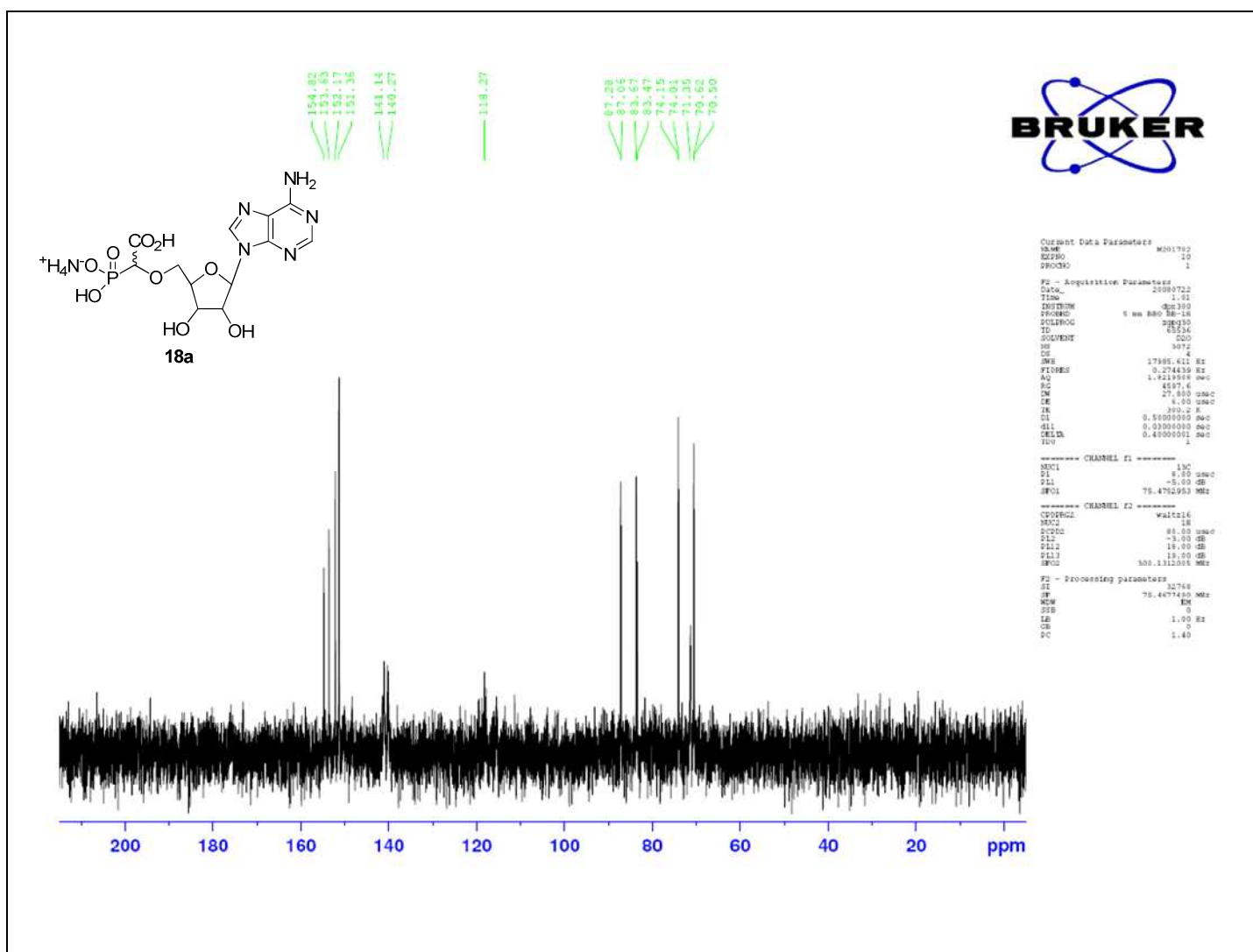


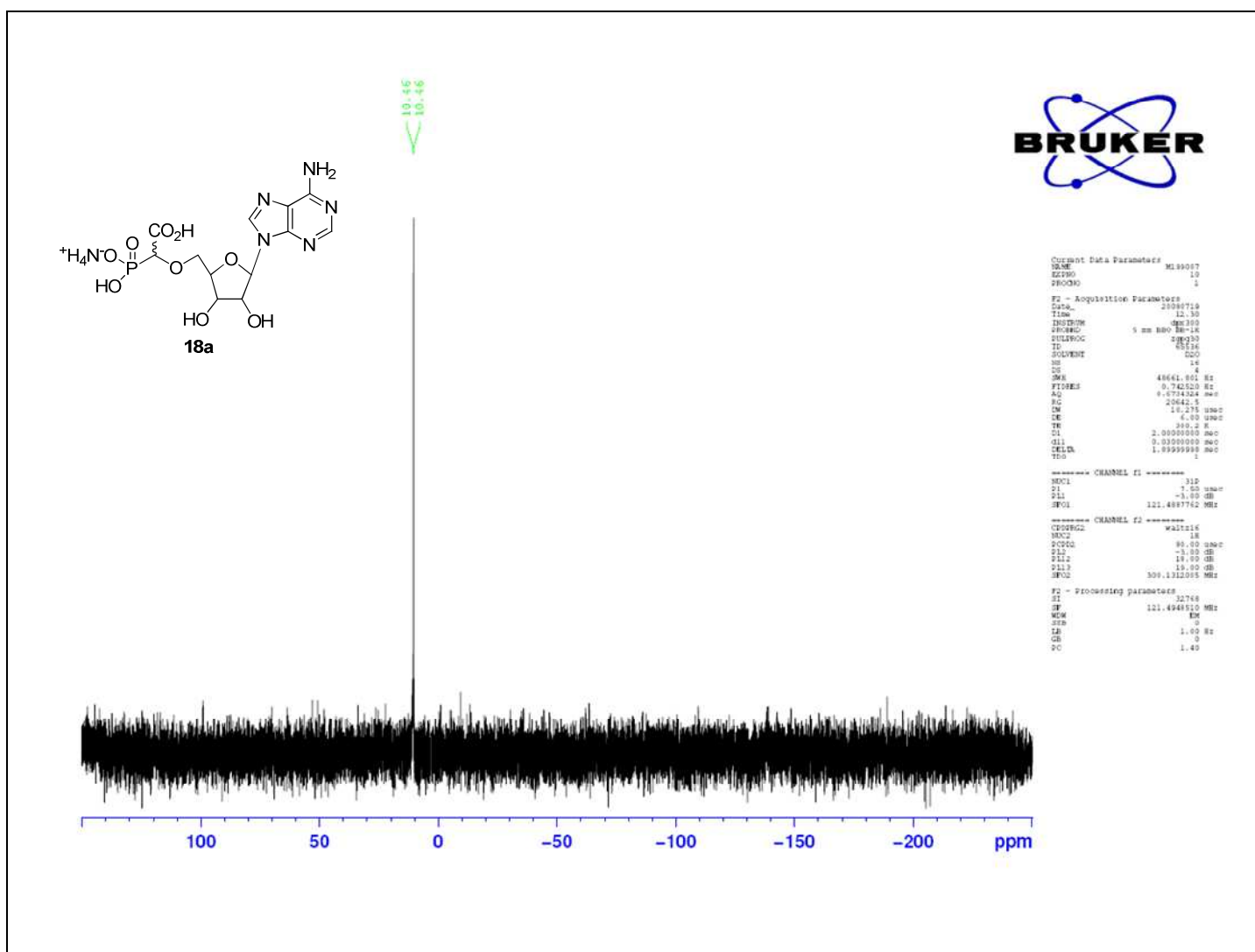




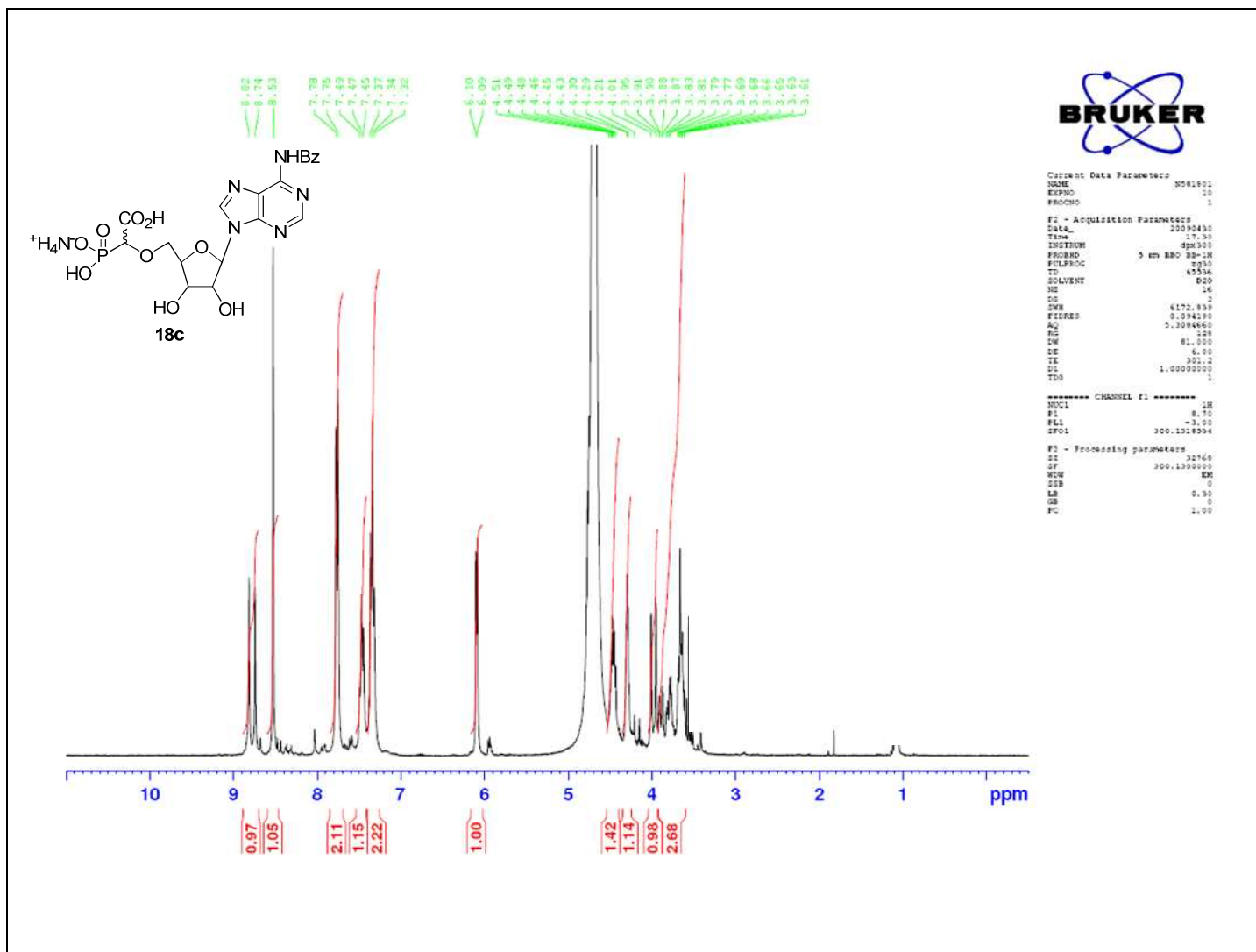
Compound **18a**.

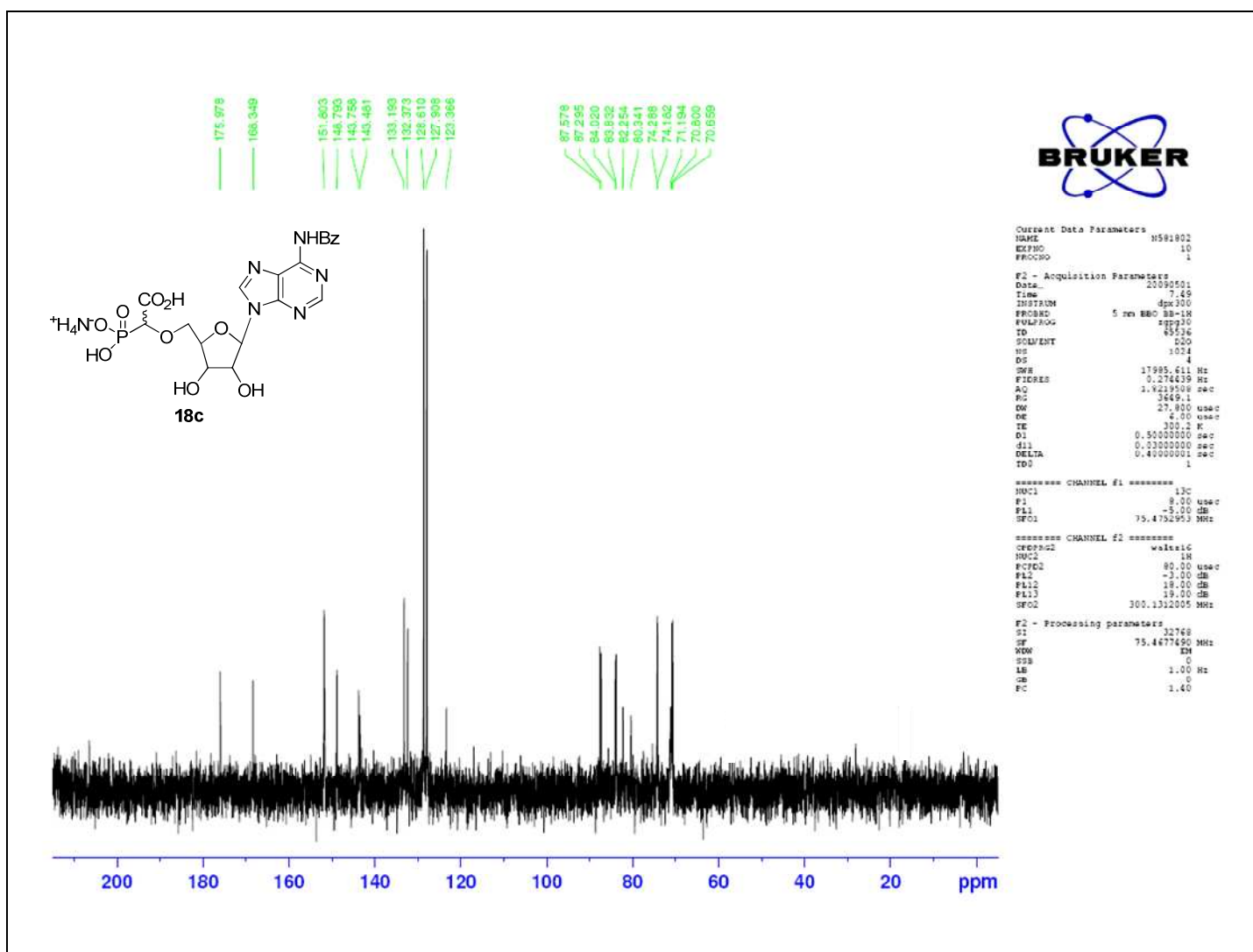


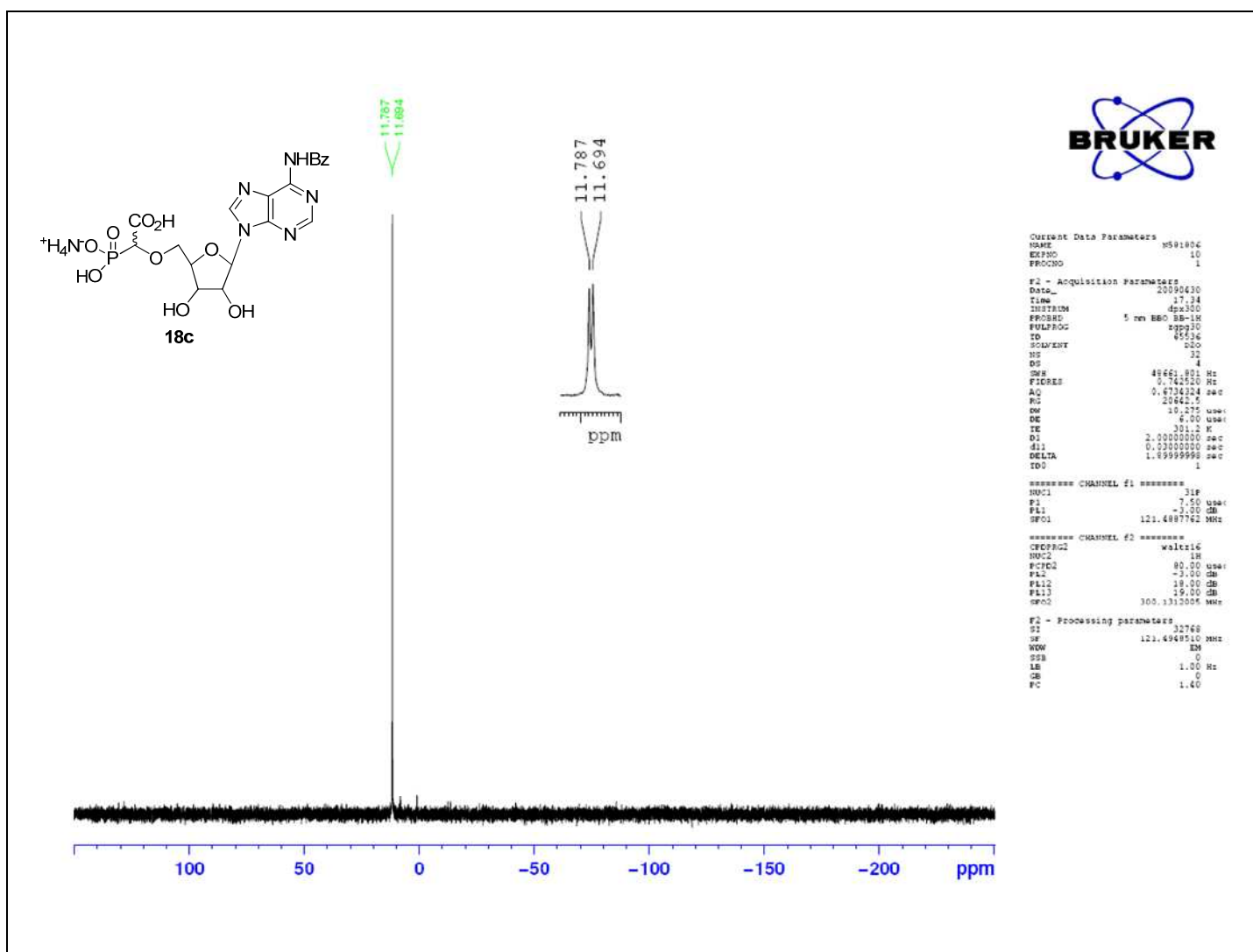




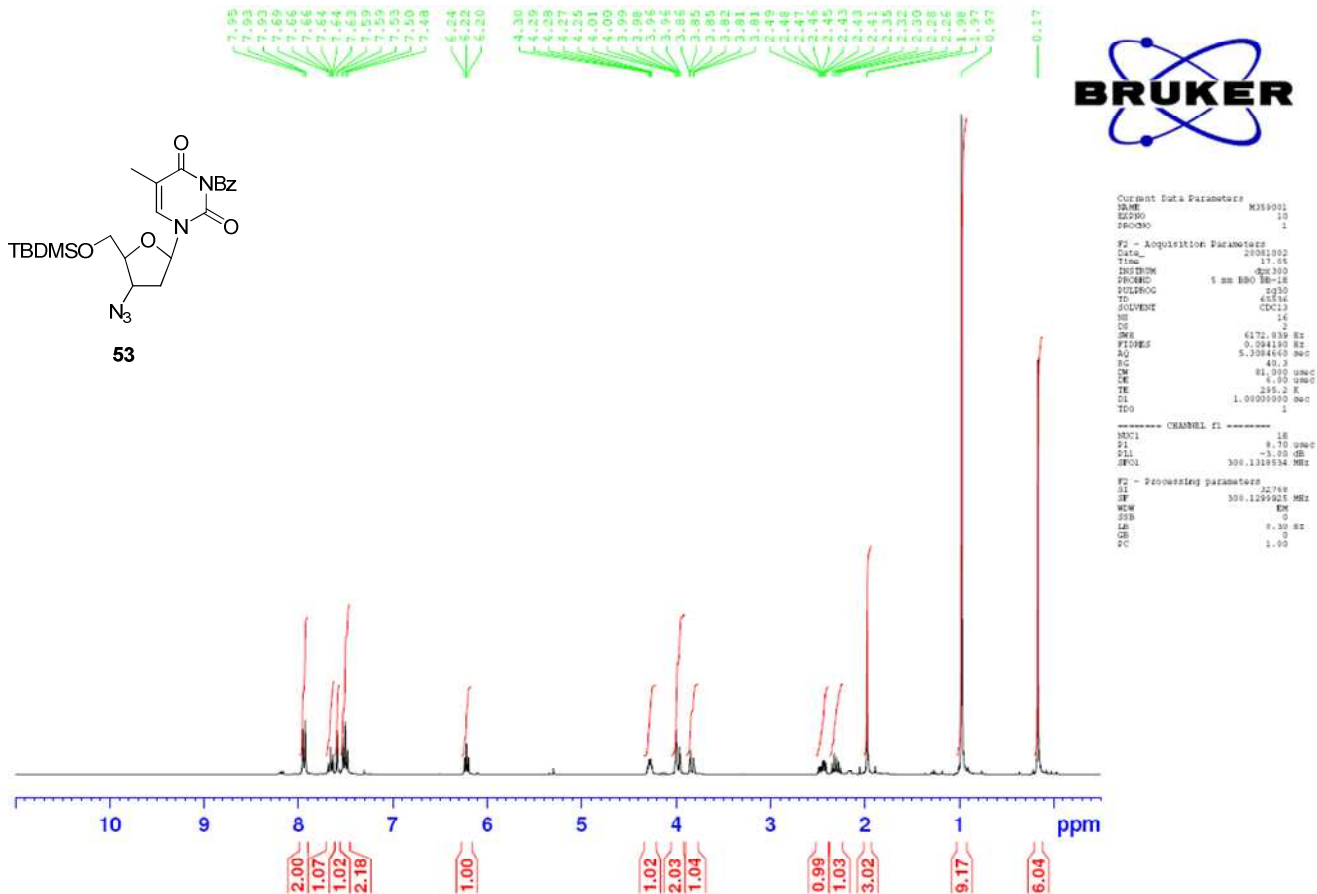
Compound **18c**.



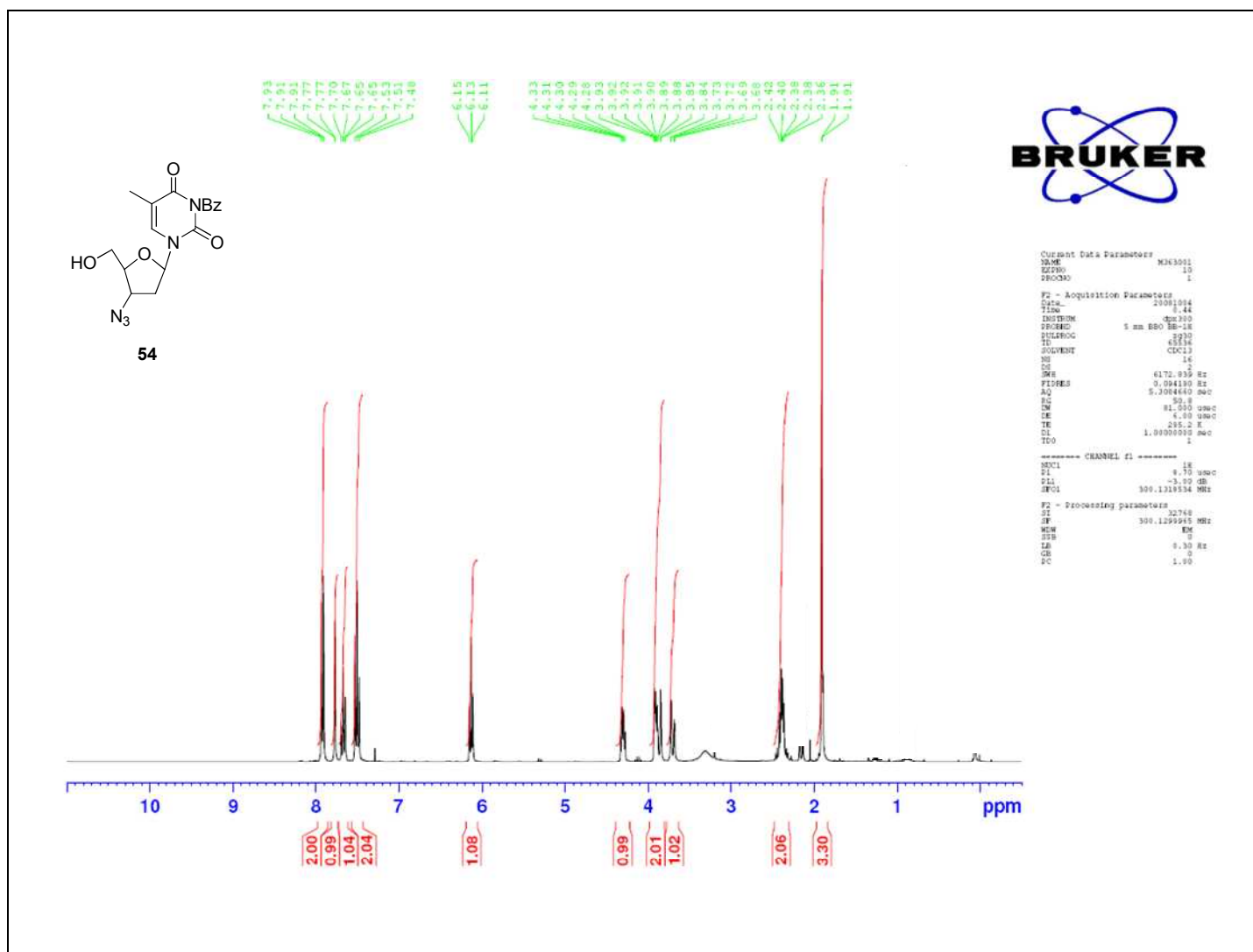


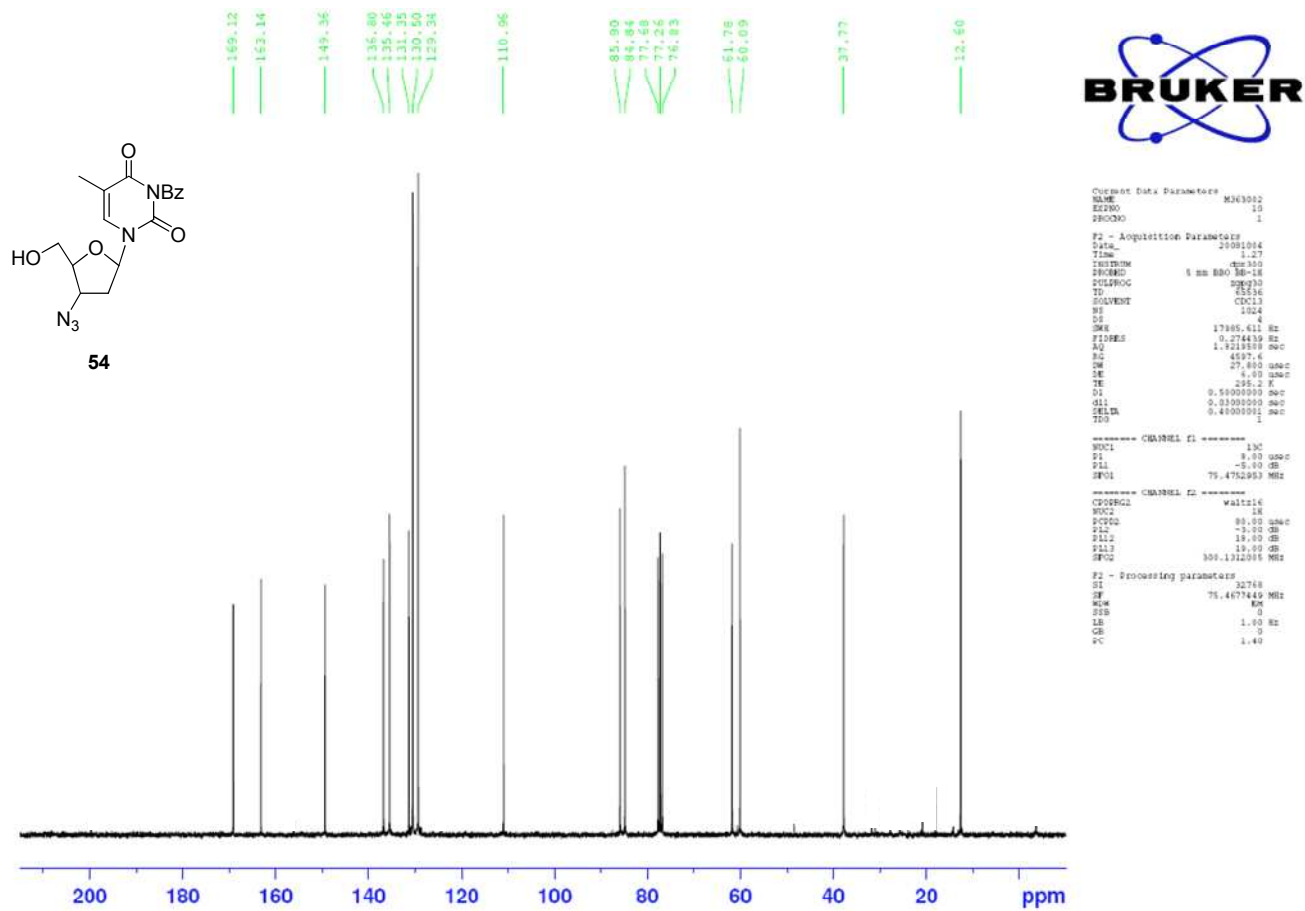


Compound **53**.

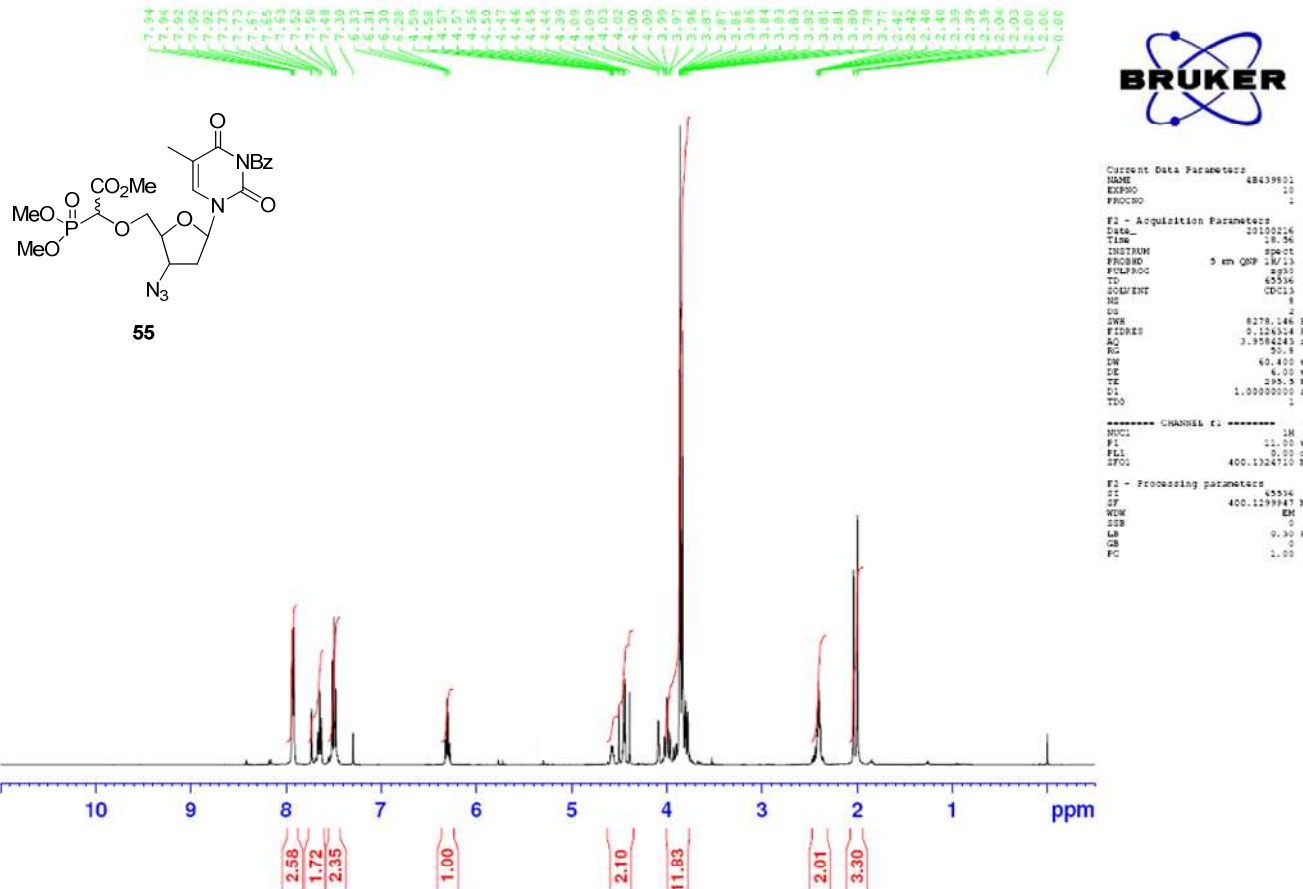


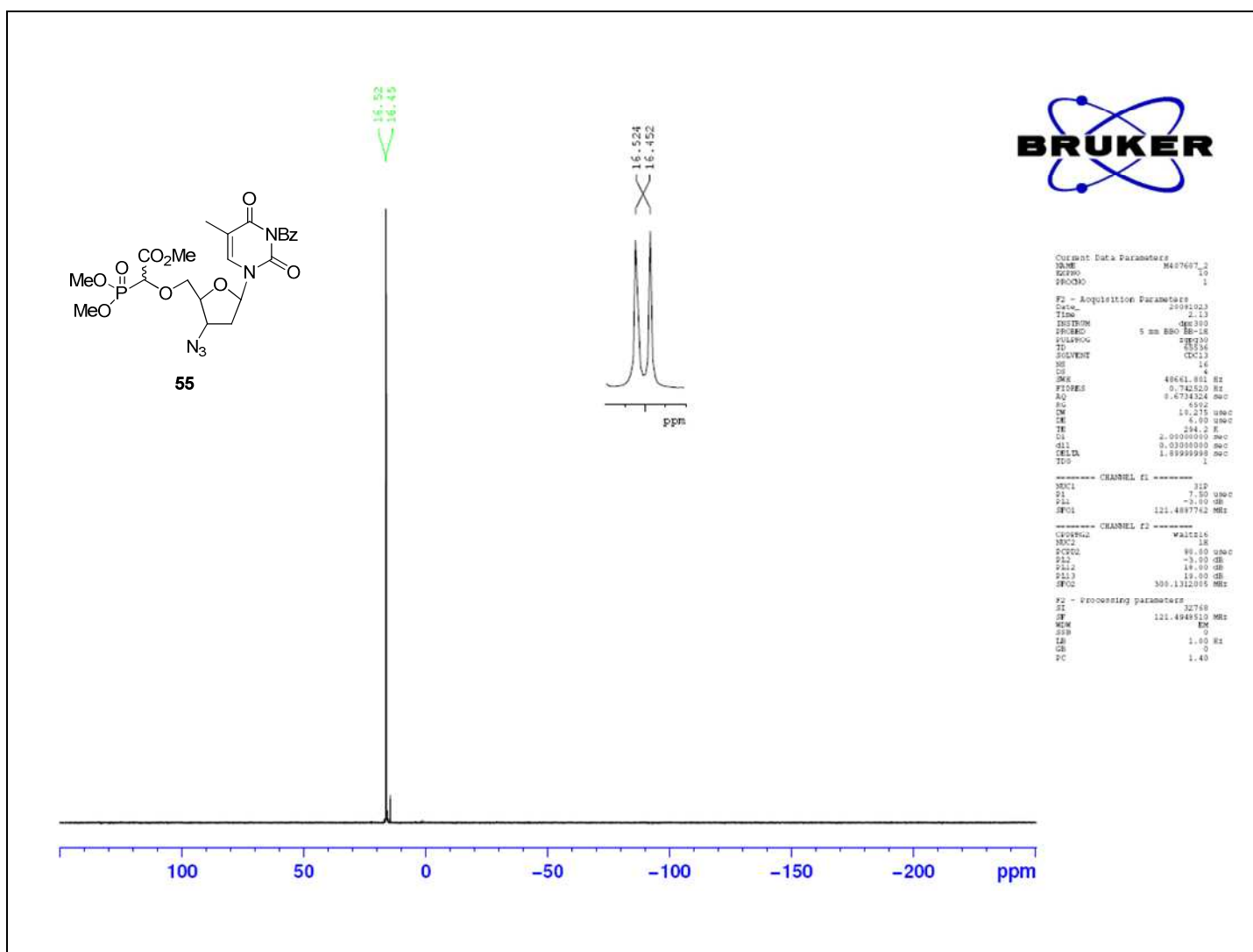
Compound **54**.



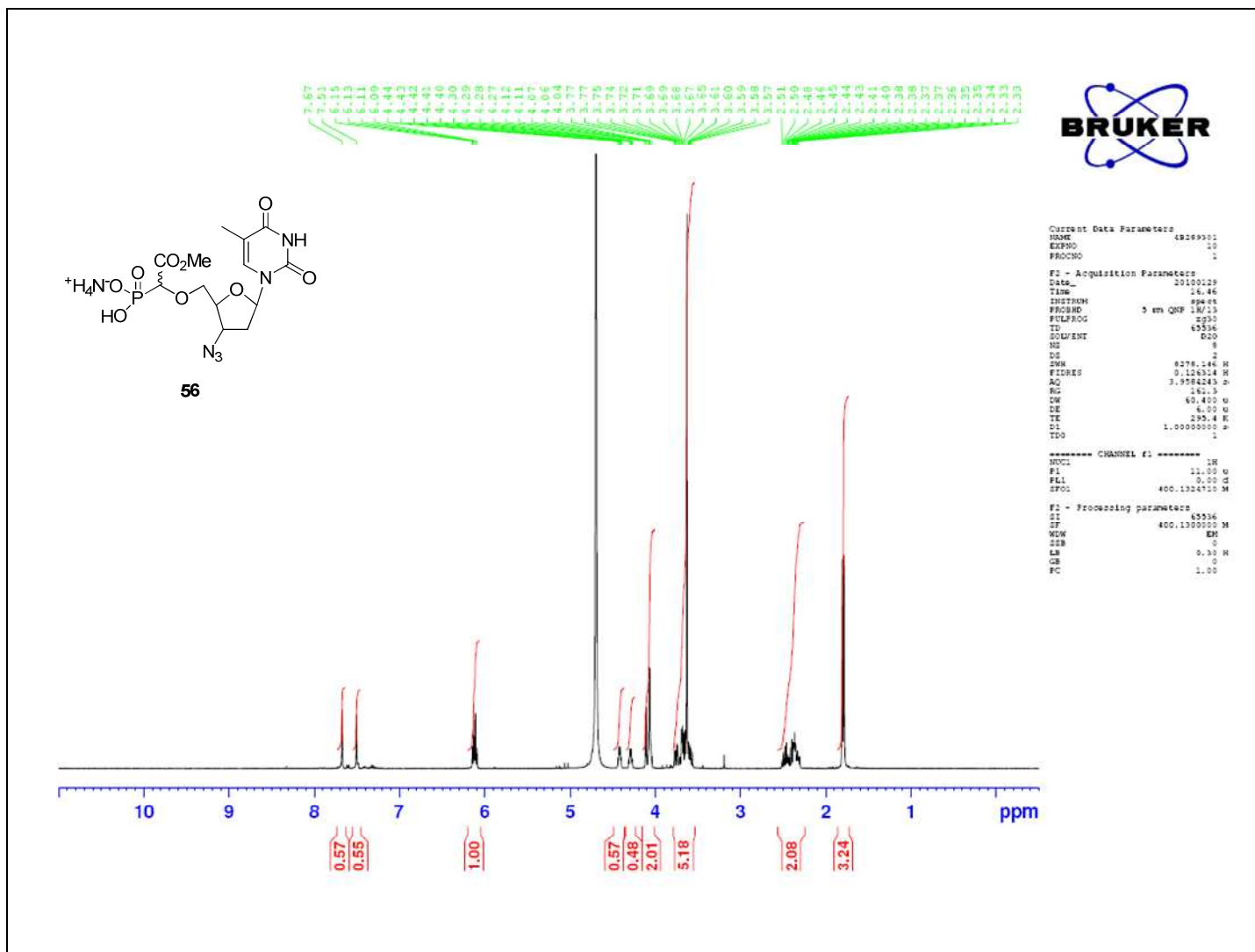


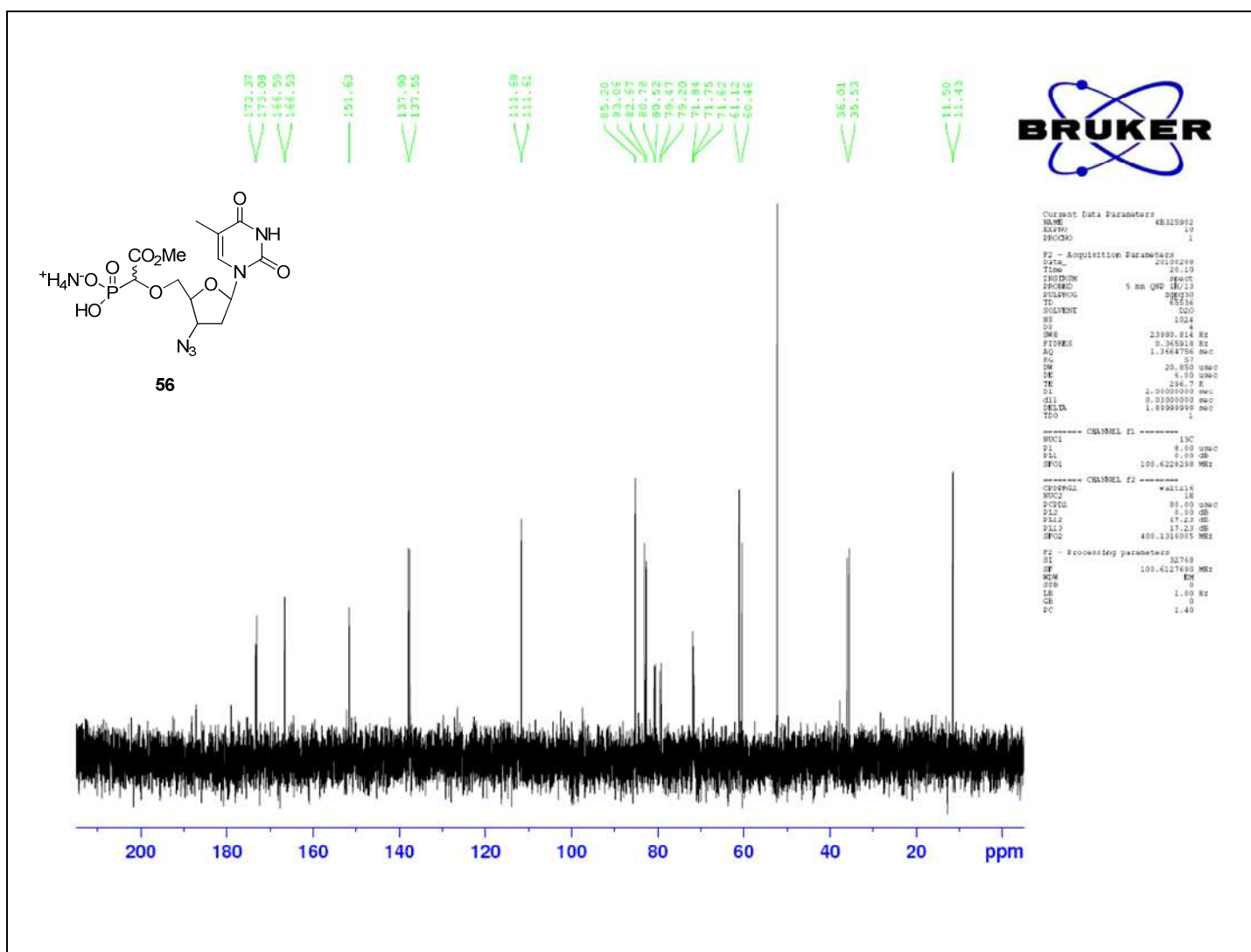
Compound **55**.

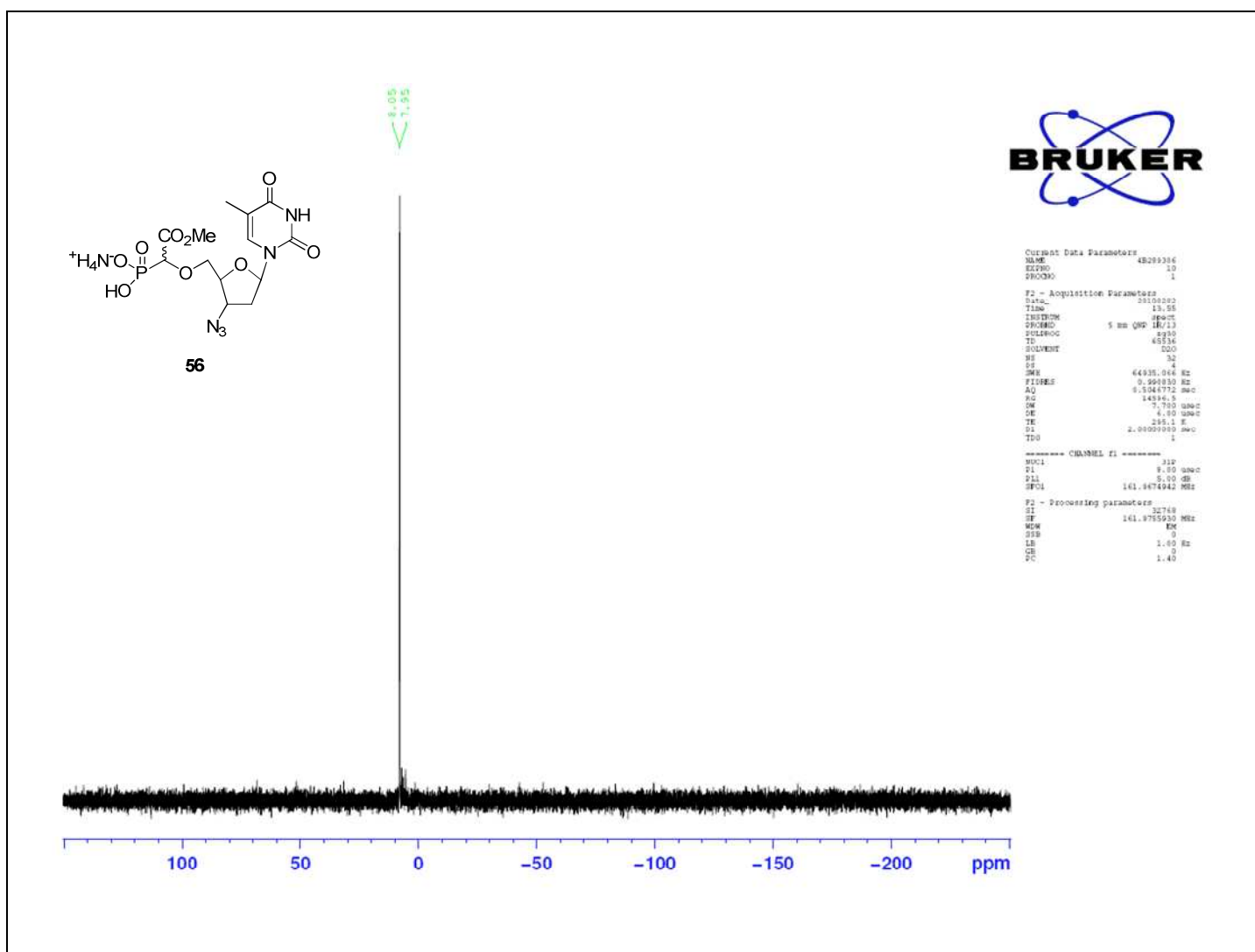




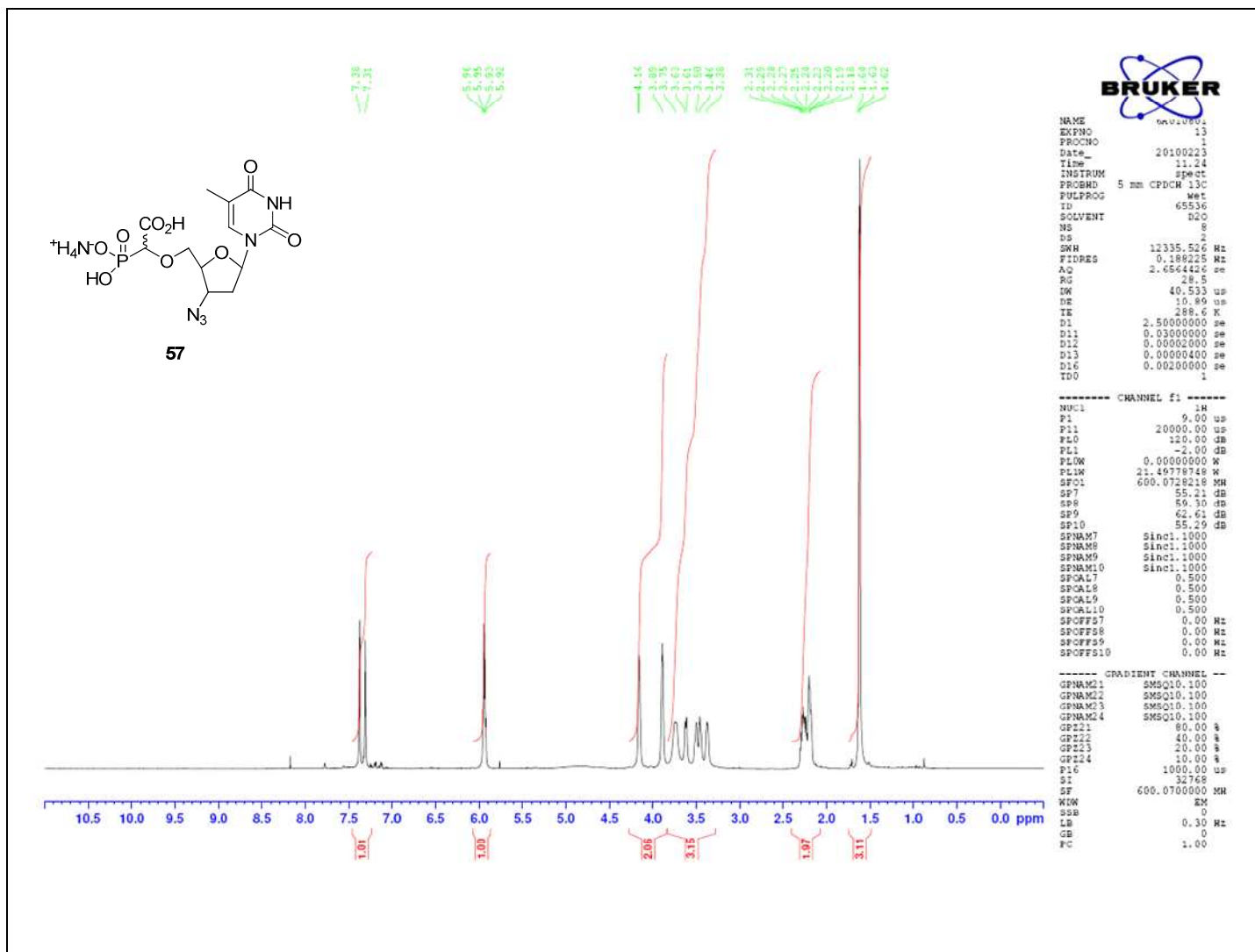
Compound **56**.

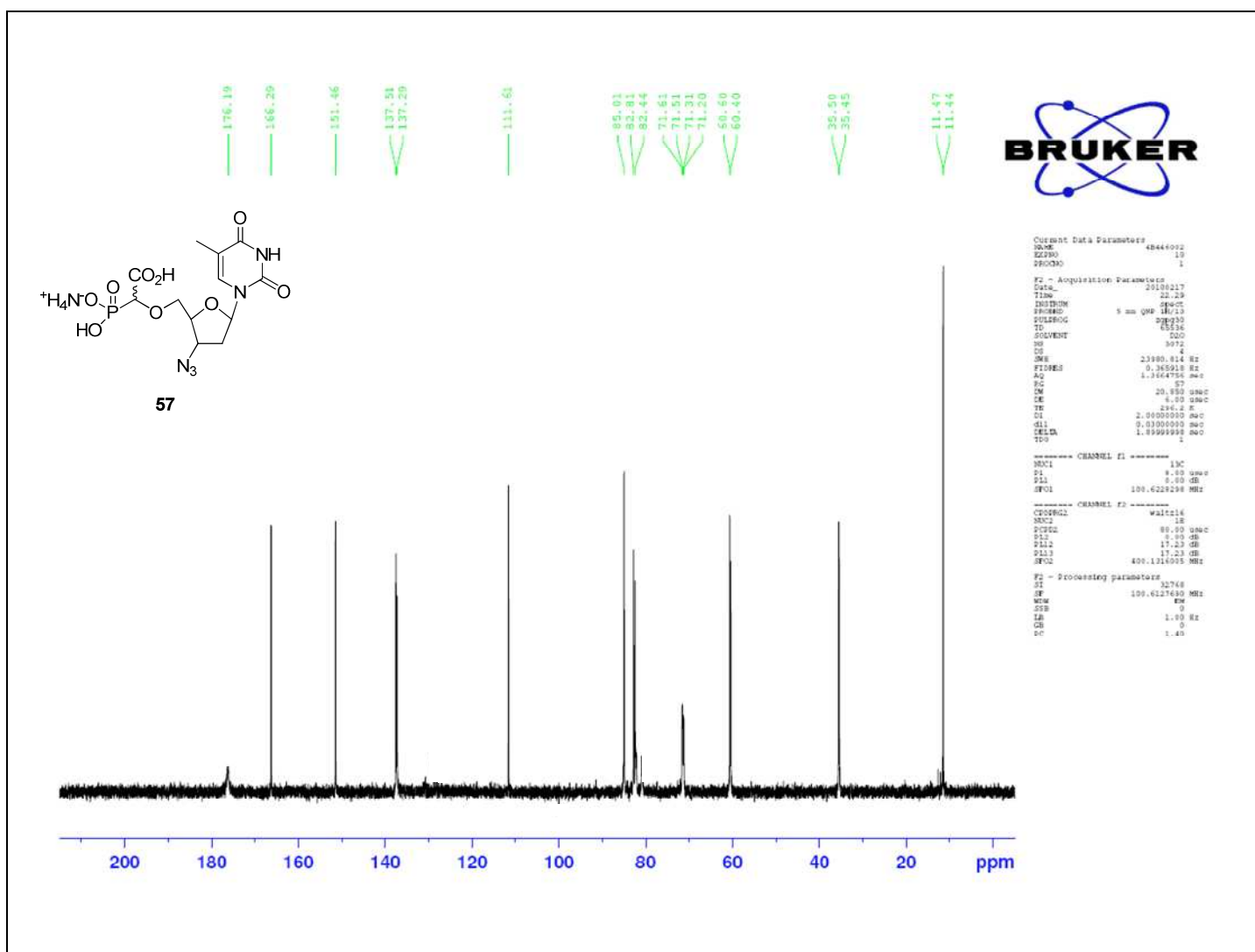


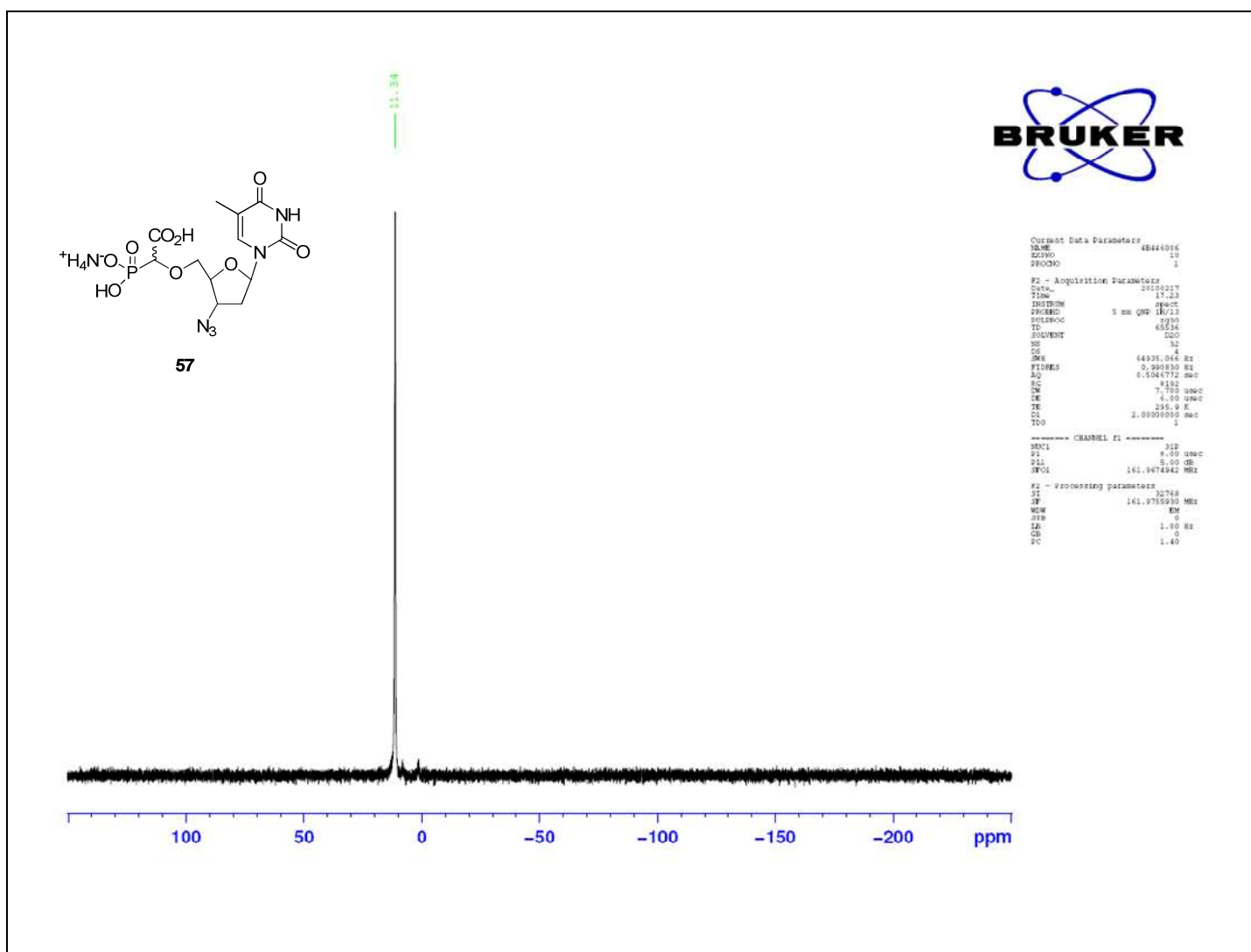




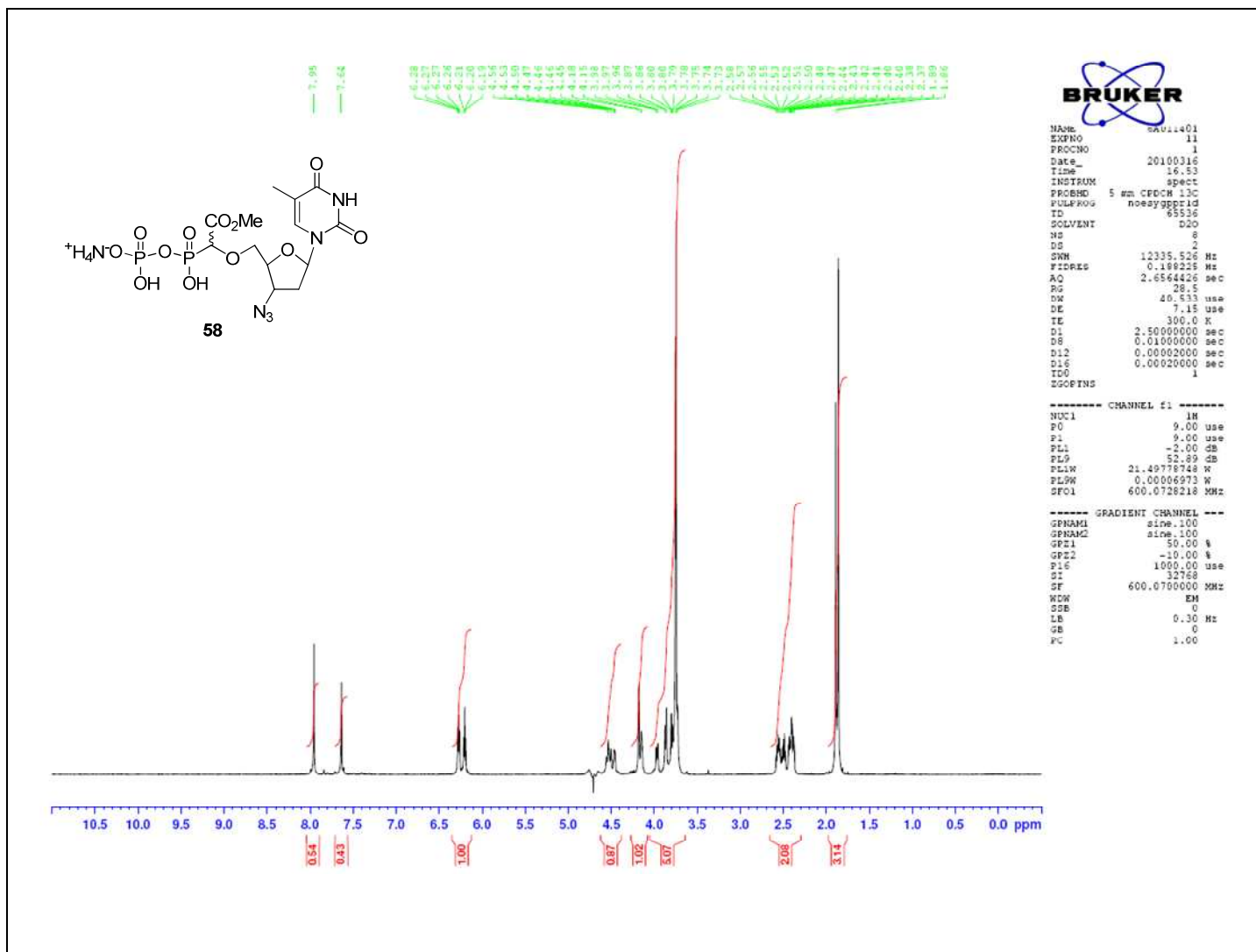
Compound **57**.

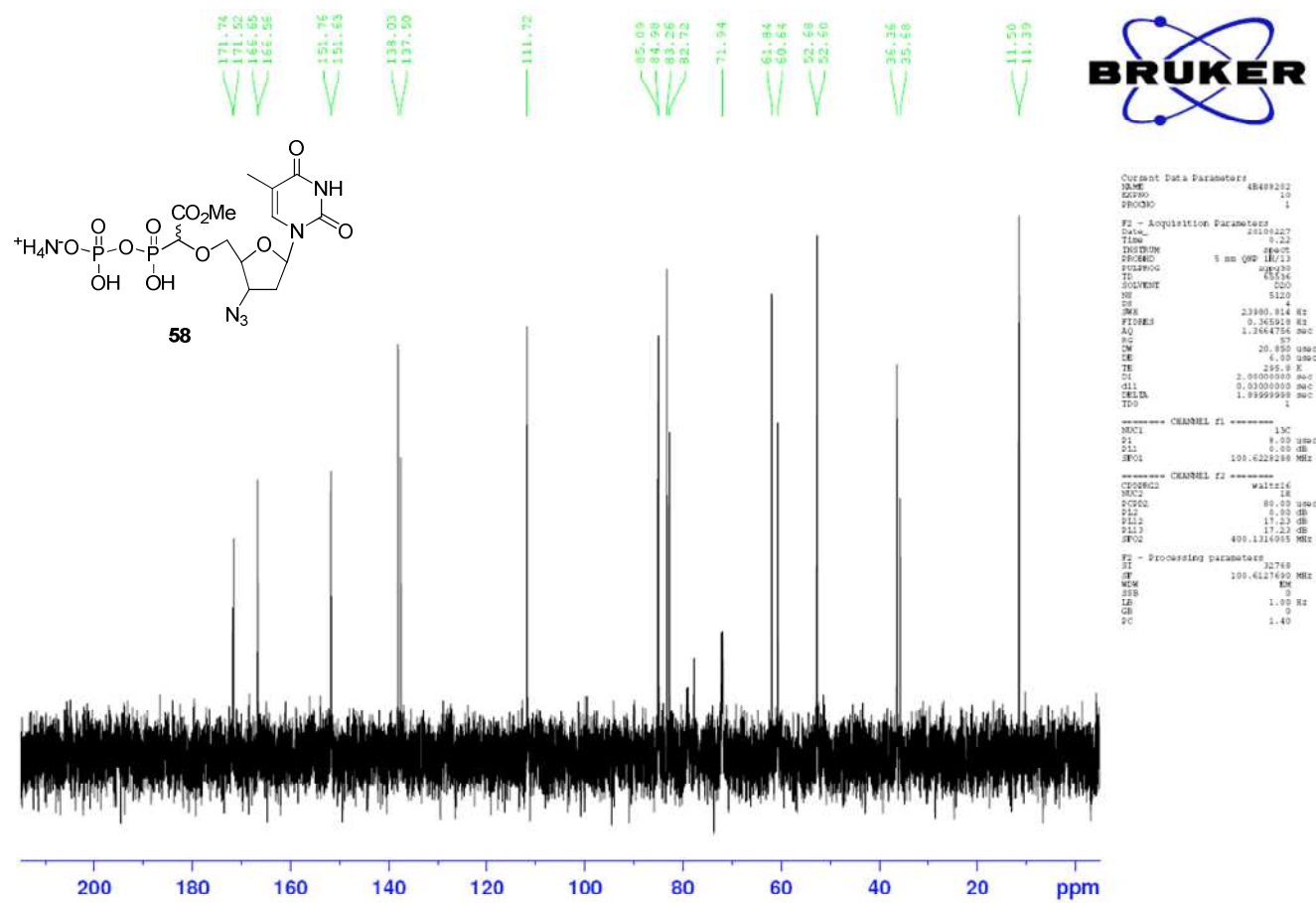






Compound **58**.





```

Current Data Parameters
NAME          48489202
SAMP          10
PROCNO       1

F2 - Acquisition Parameters
Date_         21080227
Time_         15.17
INSTRUM       SMC05
FREQ          5 mm (GPS 10.717)
P1            63.56
P2            63.56
PC            63.56
SOLVENT       NO
NUC1           13
NUC2           13
MW            239.00 134 Kr
AQ            0.365919 sec
ACQ            1.23844765 sec
RG            2
PG            2
PC            20.8500 HMG
DE            6.00 40000
TE            295.0 K
NUC1          C13
NUC2          C13
d11            2.00000000 sec
d12            0.00000000 sec
d13            1.93999998 sec
TOL0
TOL1

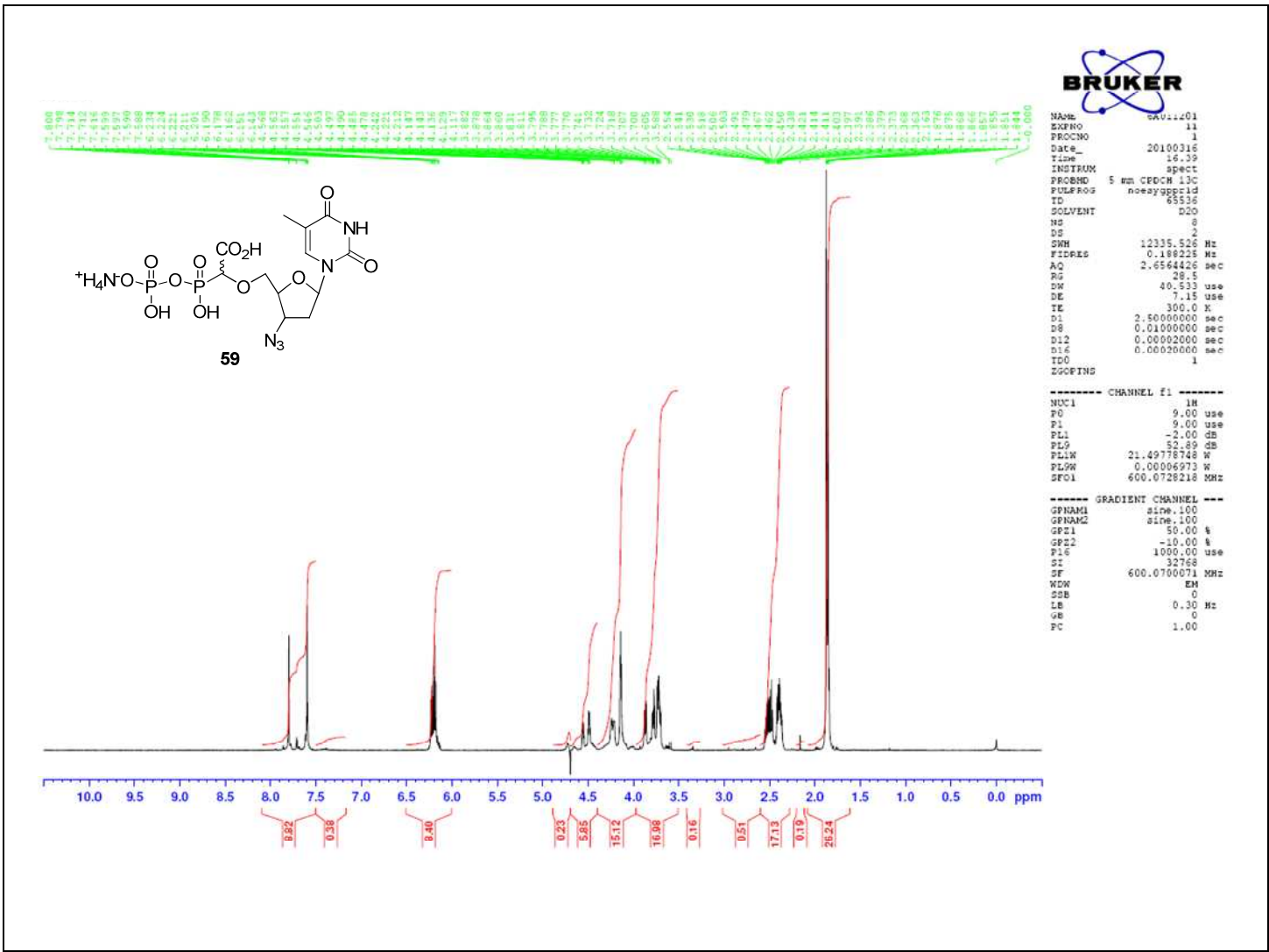
===== CHANNEL f1 =====
NUC1          13C
P1            63.56
P2            63.56
PC            63.56
SFO1          100.6222490 MHz

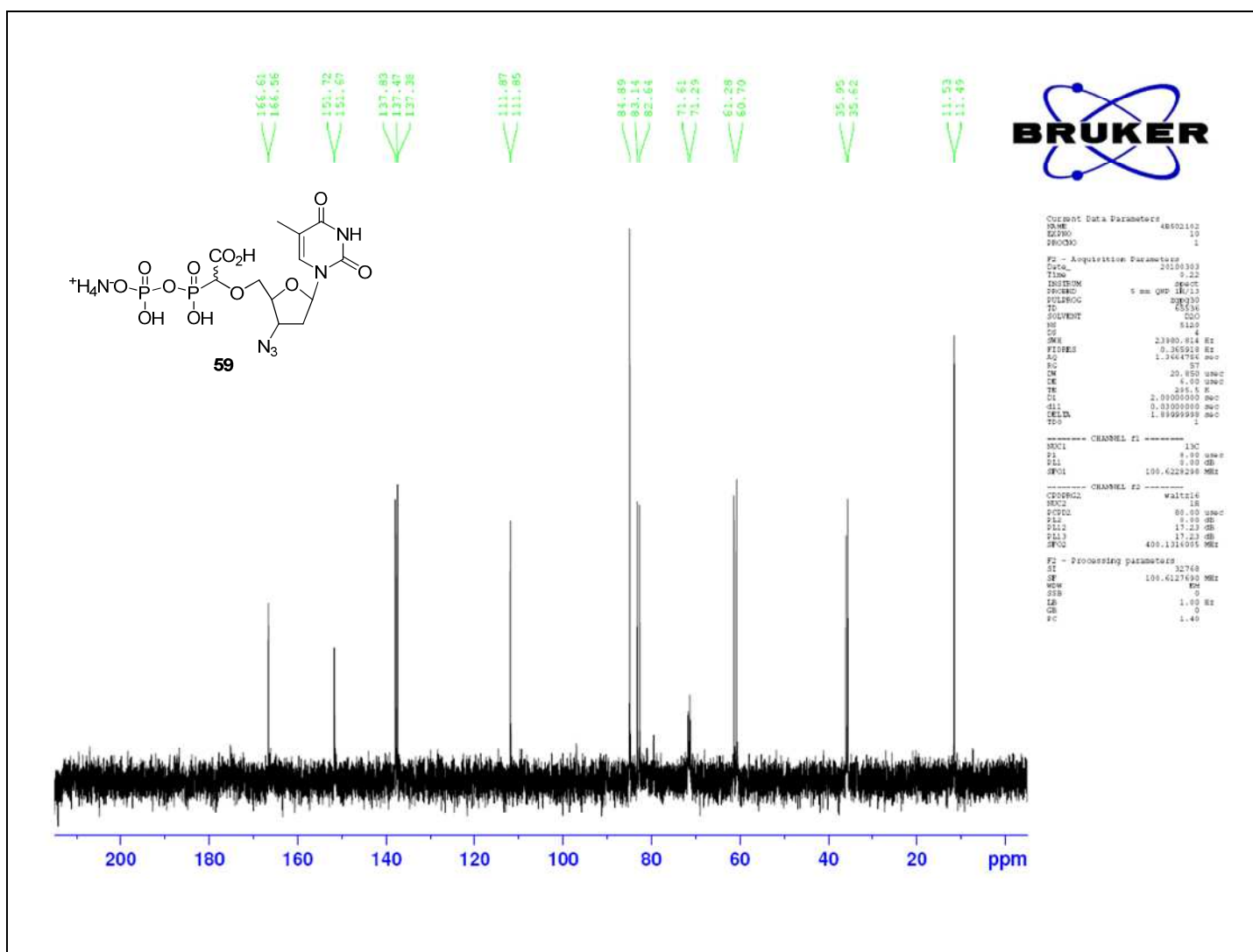
===== CHANNEL f2 =====
NAME          waltz16
NUC1          13C
P1            63.56
P2            63.56
PC            63.56
SFO1          100.6222490 MHz
SFO2          400.1311915 MHz

F2 - Processing parameters
SI            32768
SF            100.6217490 MHz
WDW           RM
SSB            0
LB            0.10 Hz
GB            0
PC            1.40

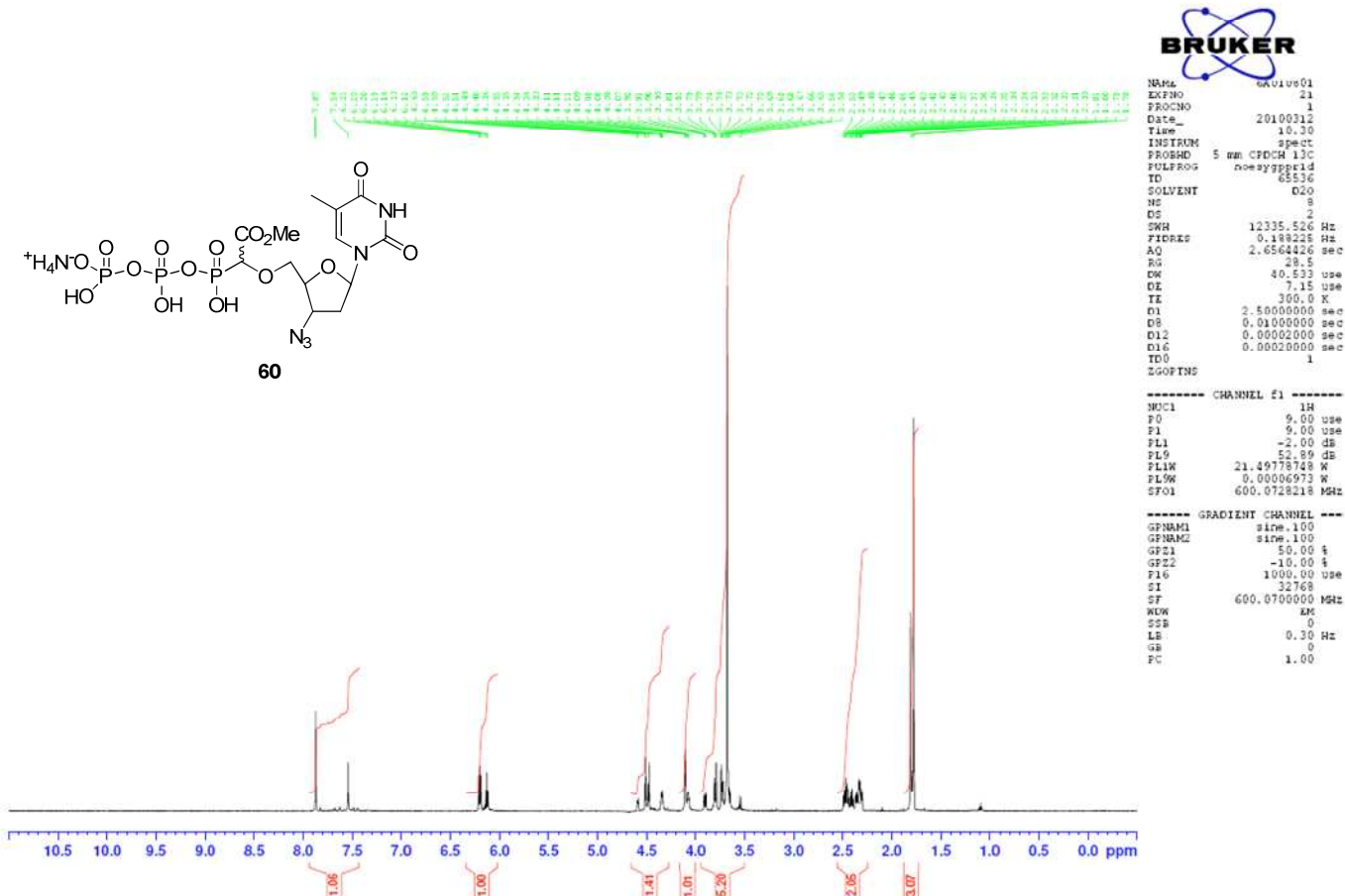
```

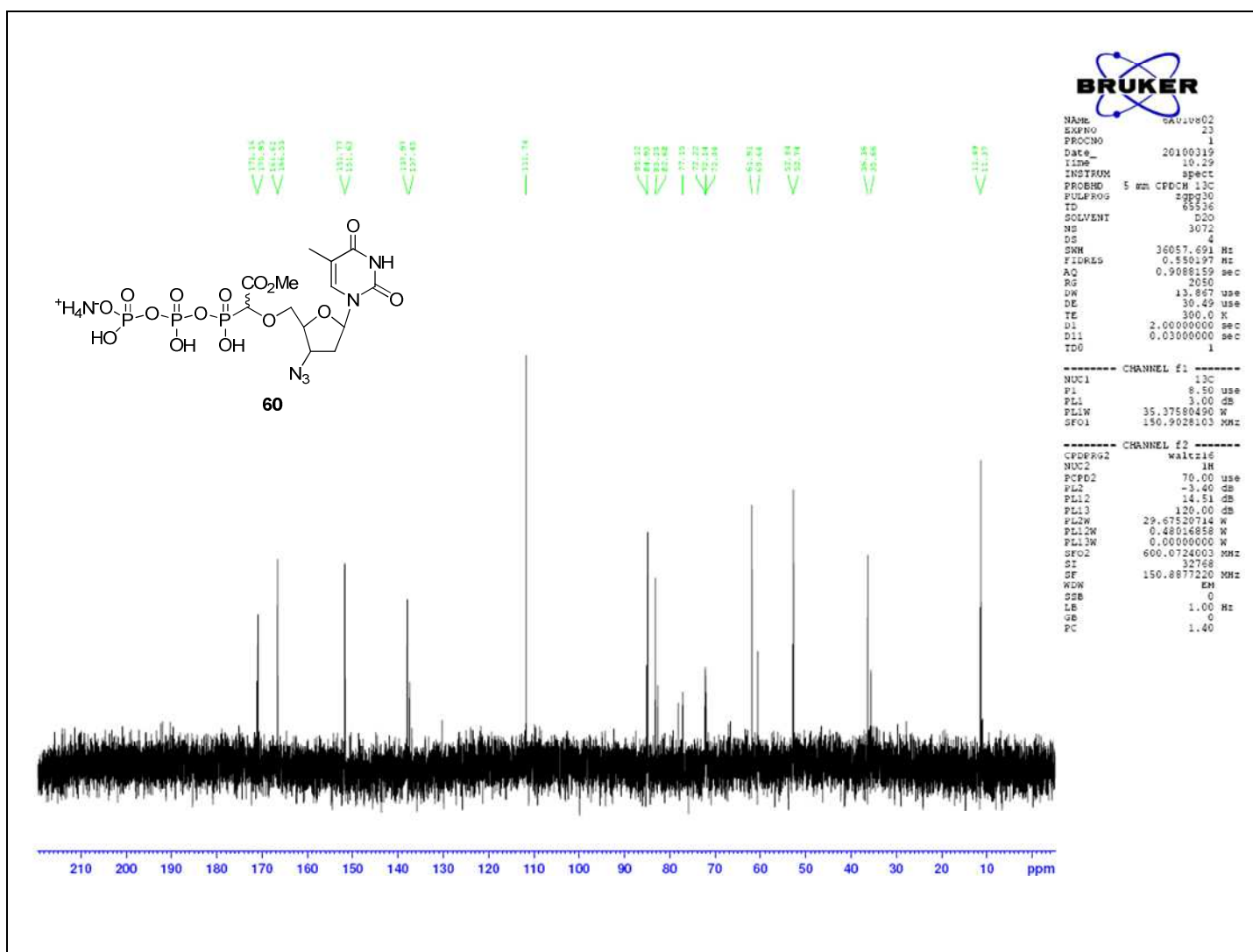

Compound **59**.



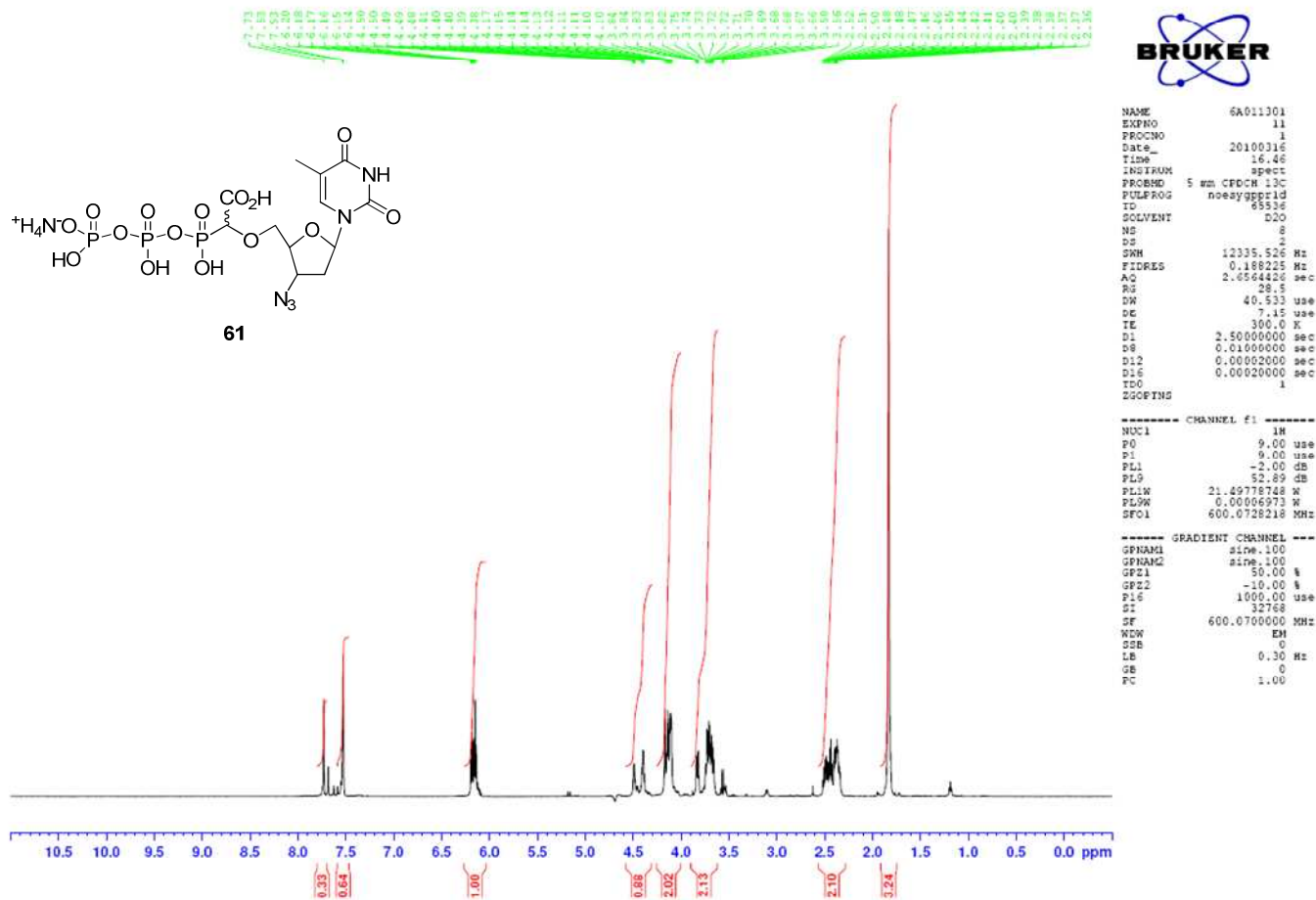


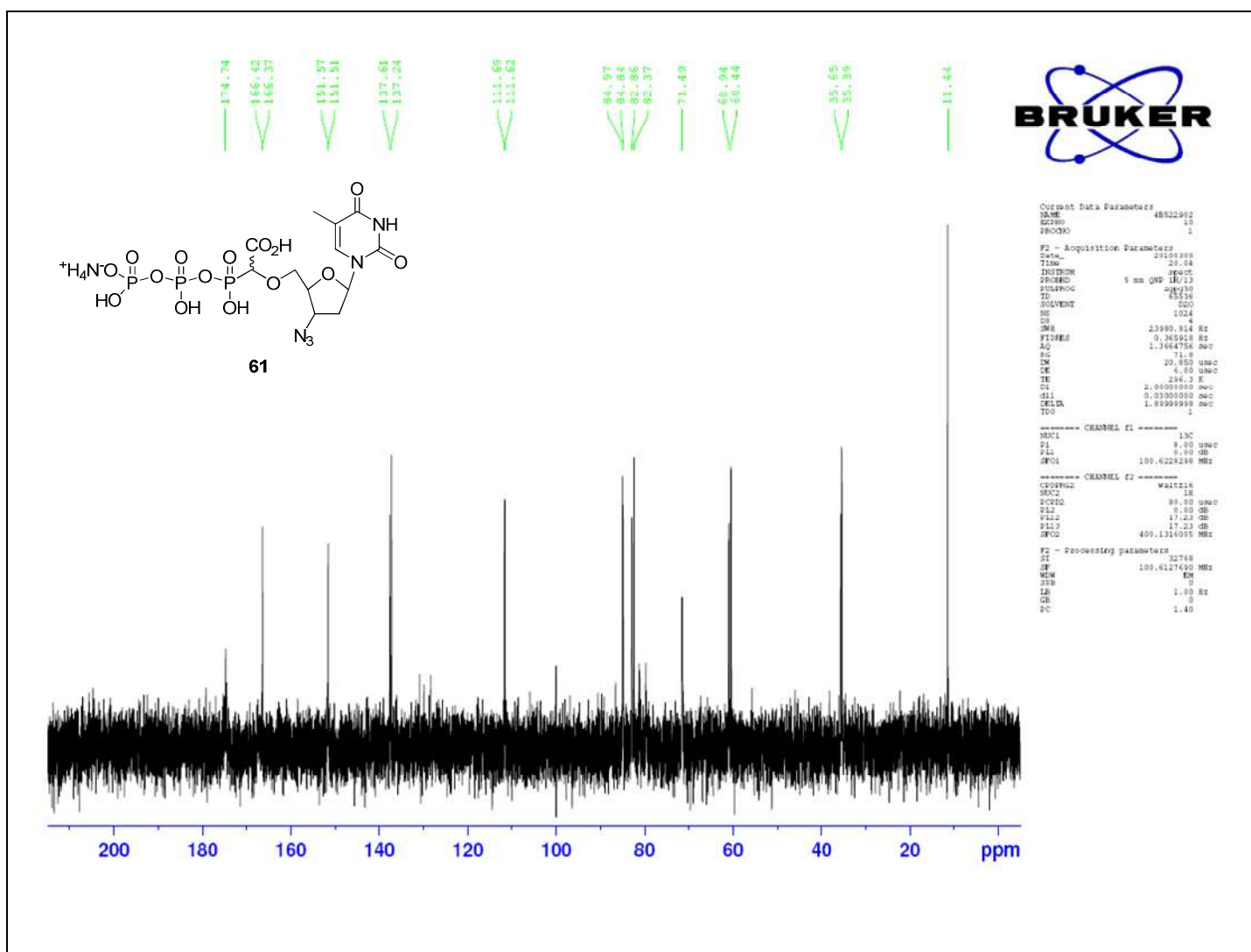
Compound **60**.





Compound **61**.





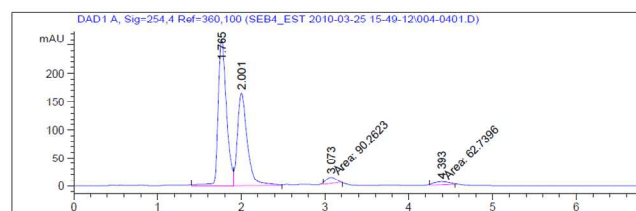
Compound 15a.

Data File C:\CHEM32\1\DATA\SEB4_EST 2010-03-25 15-49-12\004-0401.D
Sample Name: Compound 15 a

```

=====
Acq. Operator   : SB                      Seq. Line :    4
Acq. Instrument : Instrument 1             Location  : Vial 4
Injection Date  : 25/03/2010 16:19:23      Inj       :    1
                                           Inj Volume: 5.000 µl

Acq. Method     : C:\CHEM32\1\DATA\SEB4_EST 2010-03-25 15-49-12\SEB-2.M
Last changed    : 25/03/2010 15:00:42 by SB
Analysis Method : C:\CHEM32\1\METHODS\SHUTDOWN.M
Last changed    : 26/03/2010 13:51:34 by SB
                  (modified after loading)
=====
  
```

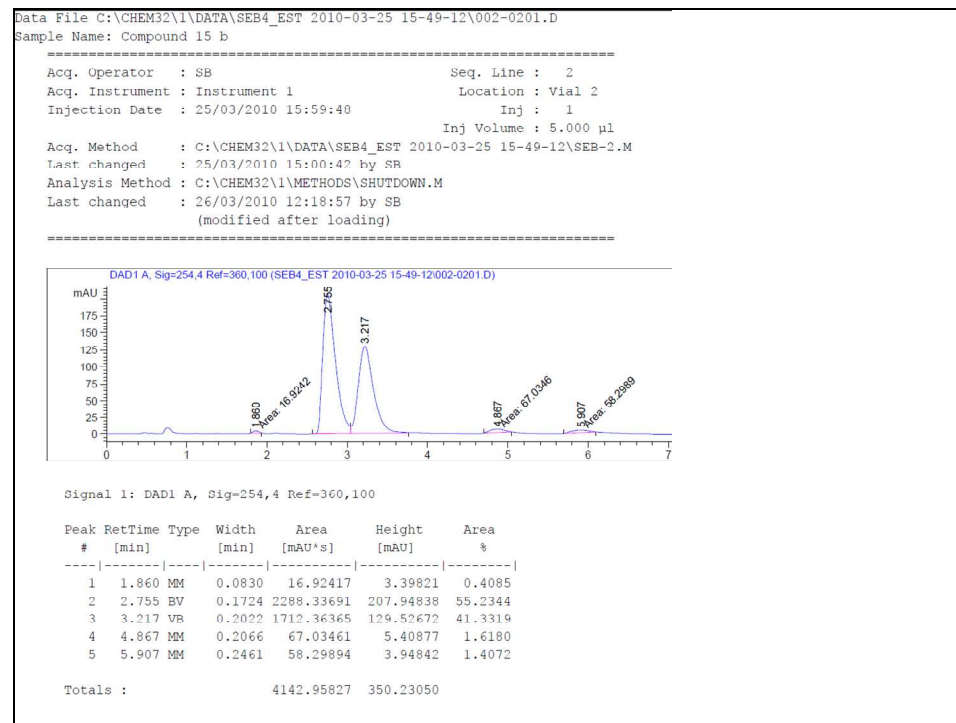


Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	1.765	BV	0.1038	1783.46973	260.64423	54.0226
2	2.001	VB	0.1223	1364.86548	165.36150	41.3428
3	3.073	MM	0.1356	90.26228	11.09455	2.7341
4	4.393	MM	0.2056	62.73962	5.08605	1.9004

Totals : 3301.33711 442.18632

Compound 15b.

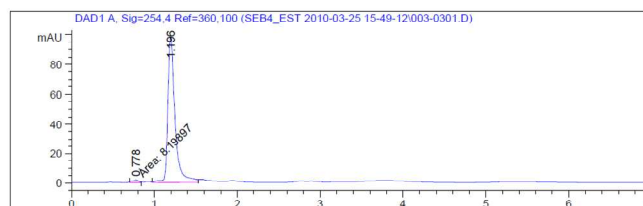


Compound 16a.

Data File C:\CHEM32\1\DATA\SEB4_EST 2010-03-25 15-49-12\003-0301.D
Sample Name: Compound 16 a

```
=====
Acq. Operator   : SB                      Seq. Line :    3
Acq. Instrument : Instrument 1             Location  : Vial 3
Injection Date  : 25/03/2010 16:09:35      Inj       :    1
                                           Inj Volume: 5.000 µl

Acq. Method     : C:\CHEM32\1\DATA\SEB4_EST 2010-03-25 15-49-12\SEB-2.M
Last changed    : 25/03/2010 15:00:42 by SB
Analysis Method : C:\CHEM32\1\METHODS\SHUTDOWN.M
Last changed     : 26/03/2010 13:51:34 by SB
                  (modified after loading)
=====
```



Signal 1: DAD1 A, Sig=254.4 Ref=360.100

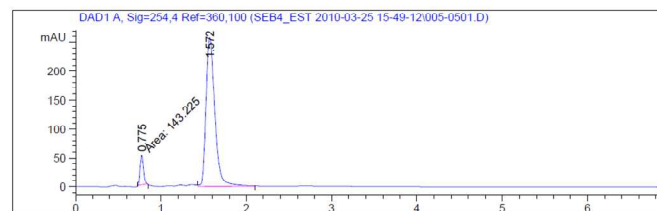
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	0.778	MM	0.0772	8.19897	1.76917	1.3605
2	1.196	VV	0.0906	594.46362	98.20345	98.6395

Totals : 602.66260 99.97261

Compound 16b.

Data File C:\CHEM32\1\DATA\SEB4_EST 2010-03-25 15-49-12\005-0501.D
Sample Name: Compound 16 b

```
=====
Acq. Operator   : SB                      Seq. Line :    5
Acq. Instrument : Instrument 1             Location  : Vial 5
Injection Date  : 25/03/2010 16:29:12      Inj       :    1
                                           Inj Volume: 5.000 µl
Acq. Method     : C:\CHEM32\1\DATA\SEB4_EST 2010-03-25 15-49-12\SEB-2.M
Last changed    : 25/03/2010 15:00:42 by SB
Analysis Method : C:\CHEM32\1\METHODS\SHUTDOWN.M
Last changed    : 26/03/2010 13:51:34 by SB
                  (modified after loading)
=====
```



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	0.775	MM	0.0457	143.22469	52.22586	7.1497
2	1.572	VB	0.1120	1860.01440	258.38666	92.8503

Totals : 2003.23909 310.61251

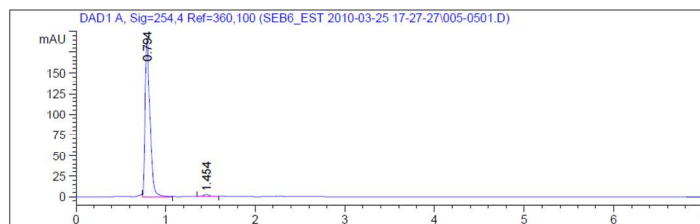
Compound 17a.

Data File C:\CHEM32\1\DATA\SEB6_EST 2010-03-25 17-27-27\005-0501.D

Sample Name: Compound 17a

```
=====
Acq. Operator   : SB                      Seq. Line :    5
Acq. Instrument : Instrument 1             Location  : Vial 5
Injection Date  : 25/03/2010 18:07:22      Inj       :    1
                                           Inj Volume: 5.000 µl

Acq. Method     : C:\CHEM32\1\DATA\SEB6_EST 2010-03-25 17-27-27\SEB-2.M
Last changed    : 25/03/2010 15:00:42 by SB
Analysis Method : C:\CHEM32\1\METHODS\SHUTDOWN.M
Last changed    : 26/03/2010 13:51:34 by SB
                  (modified after loading)
=====
```



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	0.794	VB	0.0580	719.65417	190.81828	98.2454
2	1.454	BB	0.0783	12.85272	2.40690	1.7546

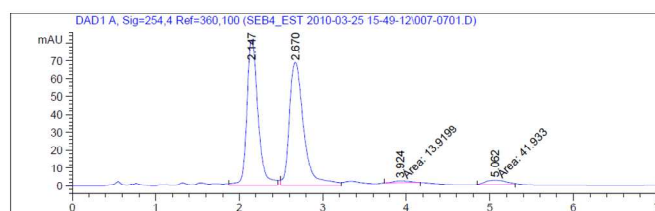
Totals : 732.50690 193.22519

Compound 18a.

Data File C:\CHEM32\1\DATA\SEB4_EST 2010-03-25 15-49-12\007-0701.D
Sample Name: Compound 18 a

```
=====
Acq. Operator   : SB                      Seq. Line :    7
Acq. Instrument : Instrument 1             Location  : Vial 7
Injection Date  : 25/03/2010 16:48:46      Inj       :    1
                                           Inj Volume: 5.000 µl

Acq. Method     : C:\CHEM32\1\DATA\SEB4_EST 2010-03-25 15-49-12\SEB-2.M
Last changed    : 25/03/2010 15:00:42 by SB
Analysis Method : C:\CHEM32\1\METHODS\SHUTDOWN.M
Last changed    : 26/03/2010 13:51:34 by SB
                  (modified after loading)
=====
```



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	2.147	RR	0.1407	762.76422	81.88422	46.2039
2	2.670	BB	0.1841	832.24835	69.28159	50.4129
3	3.924	MM	0.2355	13.91986	9.85146e-1	0.8432
4	5.062	MM	0.2925	41.93305	2.38894	2.5401

Totals : 1650.86548 154.53990

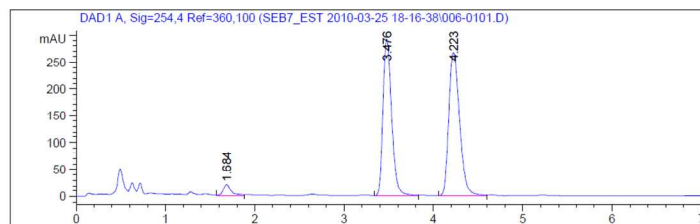
Compound 56.

Data File C:\CHEM32\1\DATA\SEB7_EST 2010-03-25 18-16-38\006-0101.D

Sample Name: Compound 56

```
=====
Acq. Operator   : SB                      Seq. Line :    1
Acq. Instrument : Instrument 1             Location  : Vial 6
Injection Date  : 25/03/2010 18:17:20      Inj       :    1
                                           Inj Volume: 5.000 µl

Acq. Method     : C:\CHEM32\1\DATA\SEB7_EST 2010-03-25 18-16-38\SEB-1.M
Last changed    : 25/03/2010 14:47:02 by SB
Analysis Method : C:\CHEM32\1\METHODS\SHUTDOWN.M
Last changed    : 26/03/2010 13:51:34 by SB
                  (modified after loading)
=====
```



Signal 1: DAD1 A, Sig=254.4 Ref=360,100

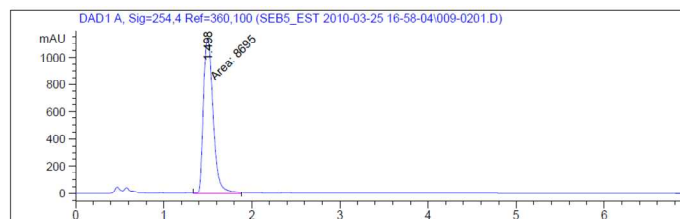
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	1.684	VV	0.0943	135.57188	21.27688	3.1494
2	3.476	VB	0.1037	1946.21606	292.30051	45.2117
3	4.223	BV	0.1314	2222.88403	266.16678	51.6389

Totals : 4304.67198 579.74416

Compound **57**.

Data File C:\CHEM32\1\DATA\SEB5_EST 2010-03-25 16-58-04\009-0201.D
Sample Name: Compound 57

```
=====
Acq. Operator   : SB                      Seq. Line :    2
Acq. Instrument : Instrument 1             Location  : Vial 9
Injection Date  : 25/03/2010 17:08:36      Inj       :    1
                                           Inj Volume: 5.000 µl
Acq. Method     : C:\CHEM32\1\DATA\SEB5_EST 2010-03-25 16-58-04\SEB-1.M
Last changed    : 25/03/2010 14:47:02 by SB
Analysis Method : C:\CHEM32\1\METHODS\SHUTDOWN.M
Last changed    : 26/03/2010 13:51:34 by SB
                  (modified after loading)
=====
```



Signal 1: DAD1 A, Sig=254,4 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	1.498	MM	0.1273	8695.00195	1138.03296	100.0000

Totals : 8695.00195 1138.03296