

Supporting Information for:

**Synthesis of a phosphinate analogue of the anti-tumour phosphate
di-ester perifosine *via* sequential radical processes**

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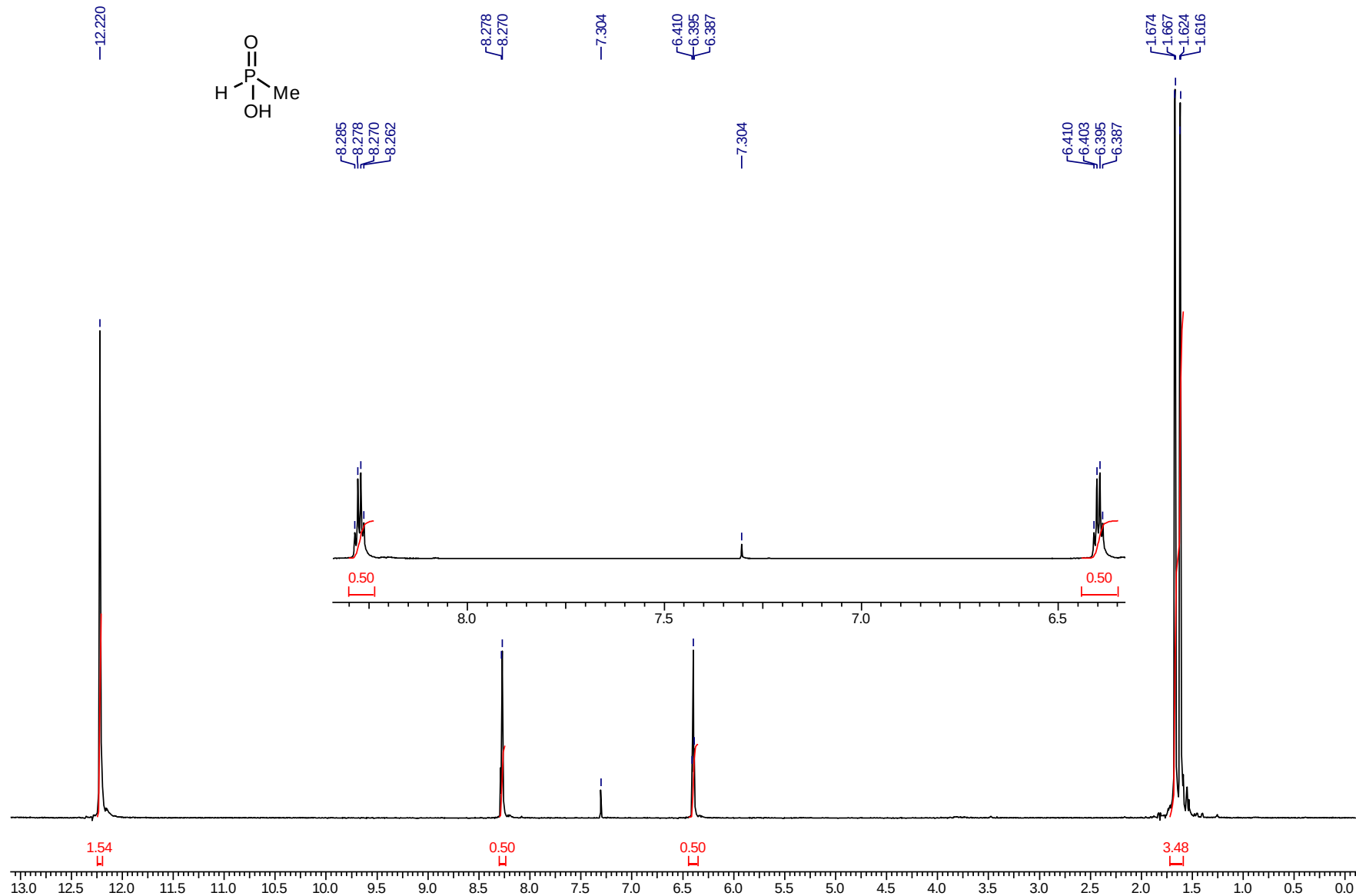
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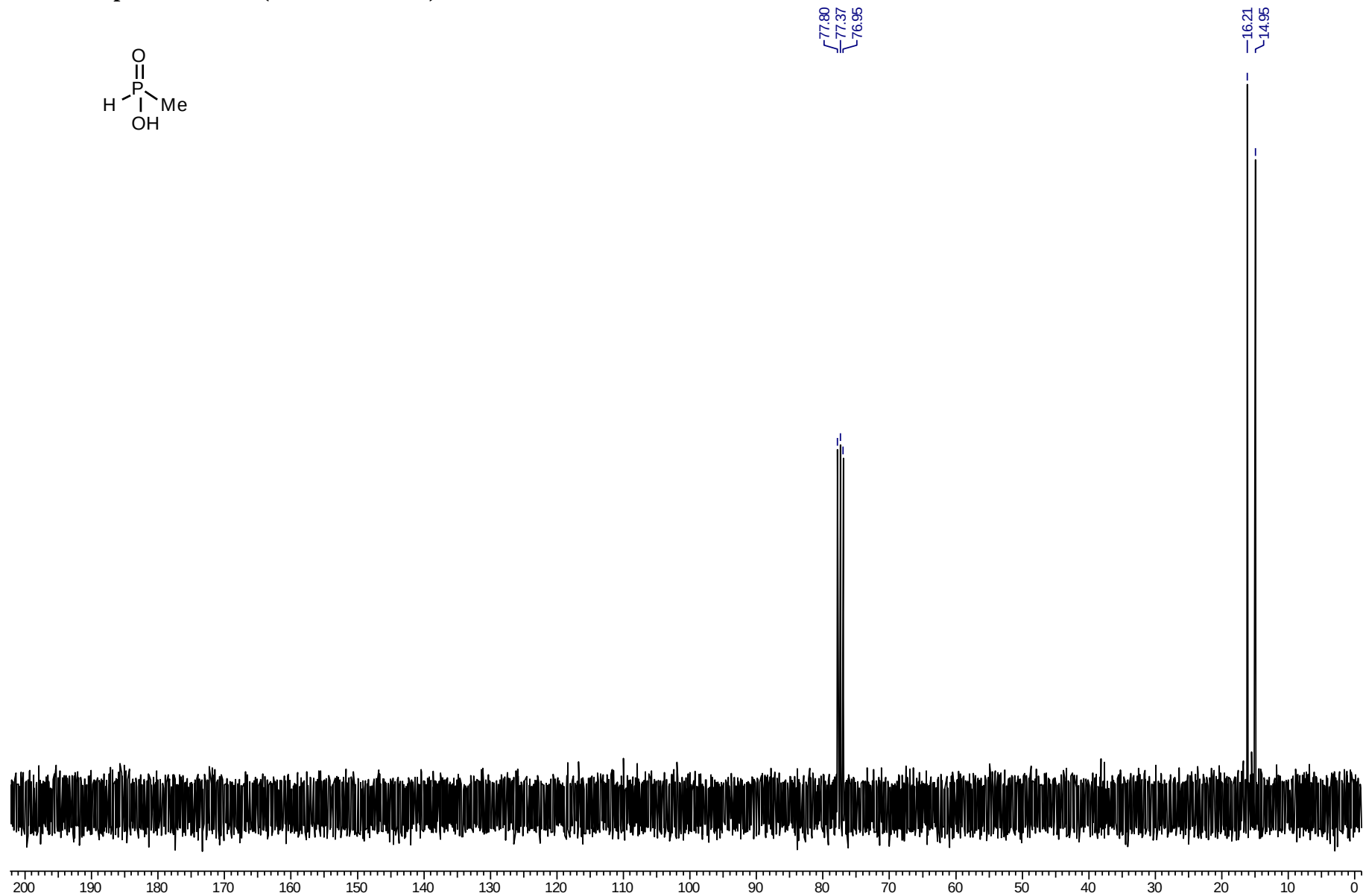
S2

1 ¹H, ¹³C and ³¹P NMR spectra

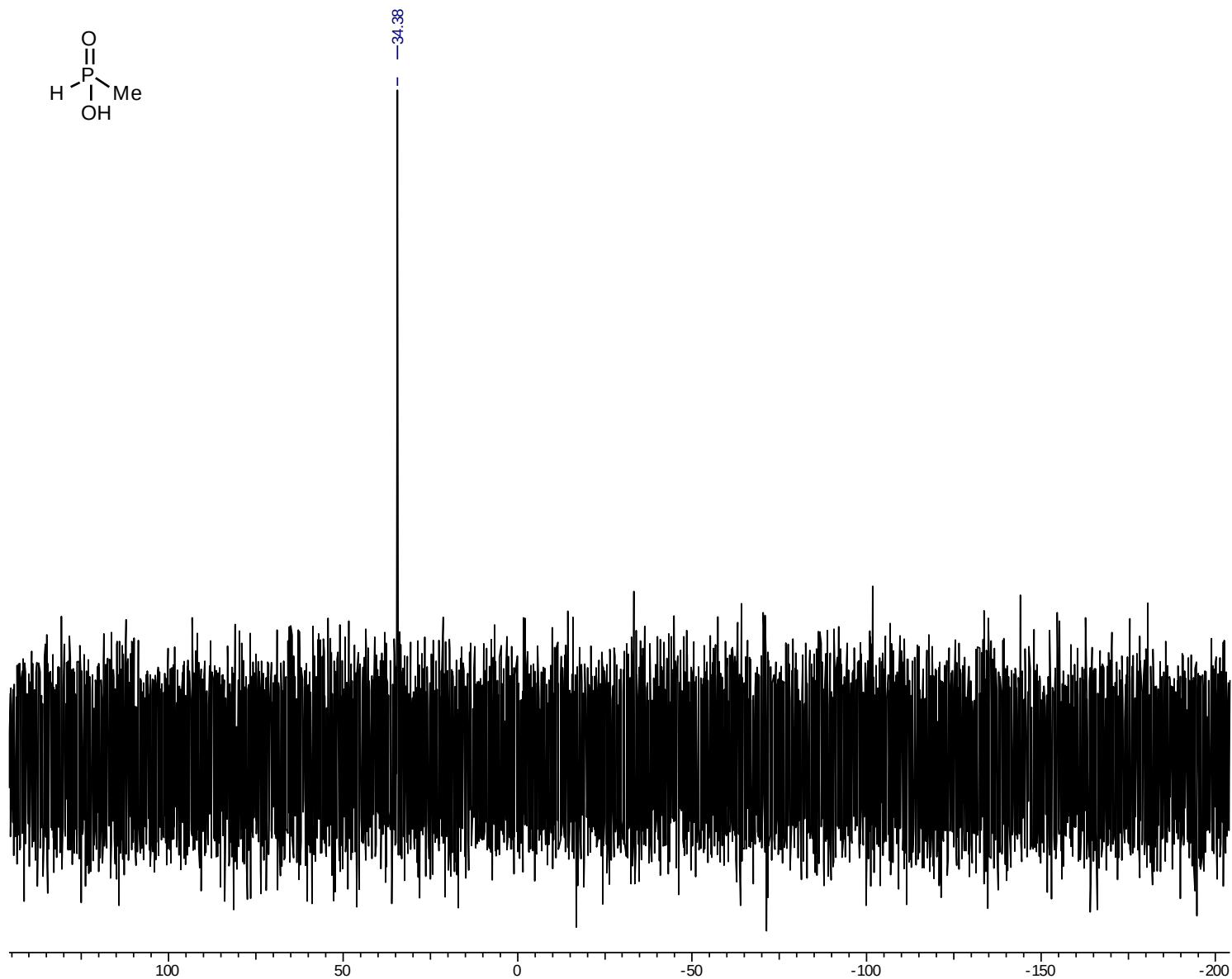
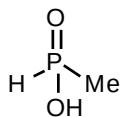
¹H NMR spectrum of 12 (300 MHz, CDCl₃)



¹³C NMR spectrum of 12 (75 MHz, CDCl₃)



^{31}P (proton-decoupled) NMR spectrum of 12 (162 MHz, CDCl_3)

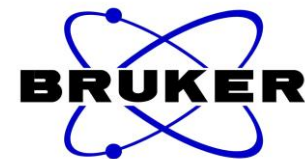
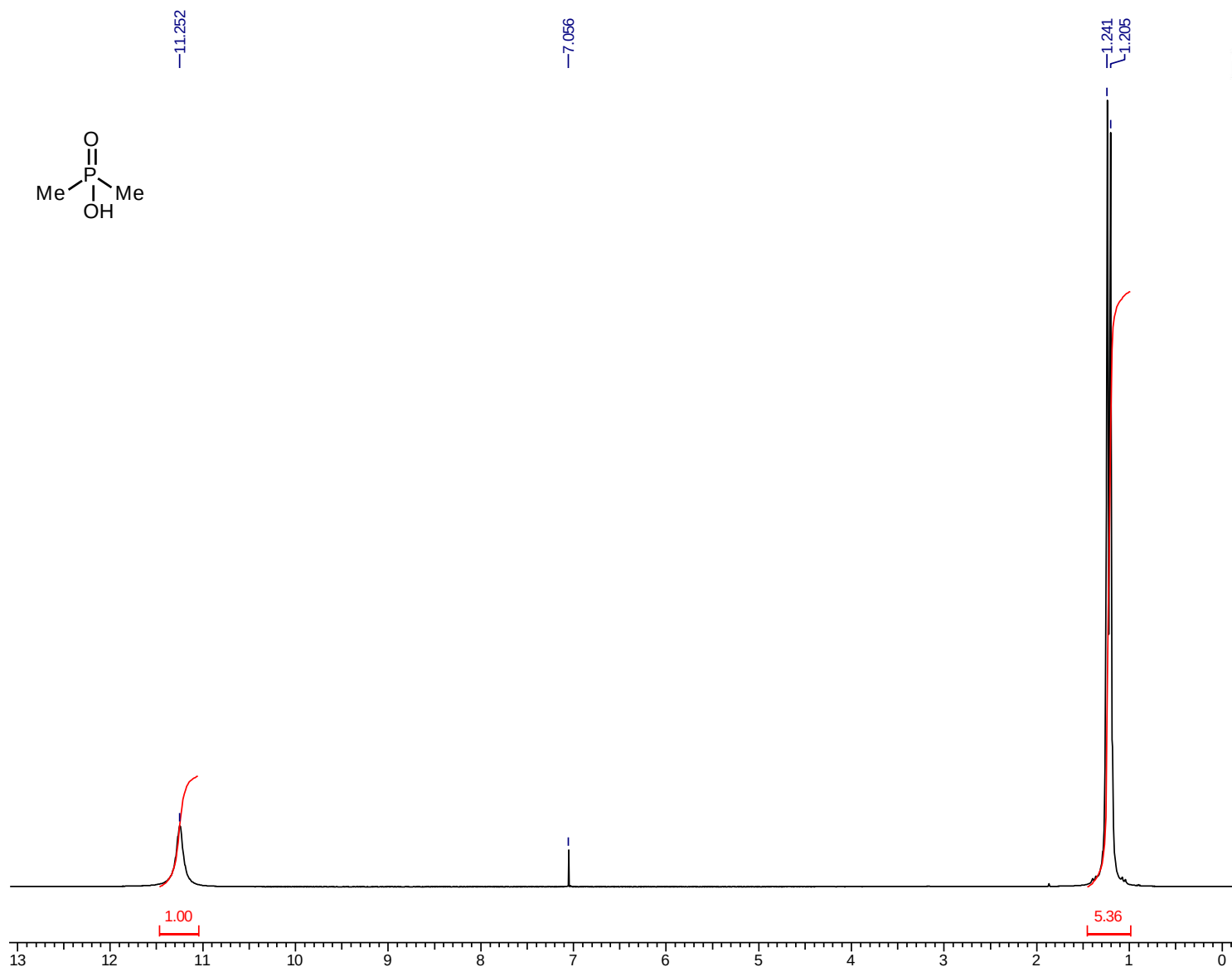


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NAME          acr-42
EXPNO          11
PROCNO         1
INSTRUM        AV400
PROBHD         5 mm PABBO BB-
PULPROG        zgpg30
TD             65536
SOLVENT        CDC13
NS             64
DS             4
SWH            64102.563 Hz
FIDRES         0.978127 Hz
AQ             0.5112308 sec
RG             23100
DW             7.800 usec
DE             6.50 usec
TE             295.4 K
D1             2.0000000 sec
D11            0.03000000 sec
TD0            1
```

```
===== CHANNEL f1 =====
NUC1           31P
P1             9.40 usec
PL1            0.00 dB
PL1W           26.87718391 W
SF01           161.9674942 MHz
```

```
===== CHANNEL f2 =====
CPDPRG2        wa1tz16
NUC2           1H
PCPD2          90.00 usec
PL2            -3.60 dB
PL12           15.31 dB
PL13           18.00 dB
PL2W           17.83863831 W
PL12W          0.22927761 W
PL13W          0.12341322 W
SF02           400.1316005 MHz
SI             32768
SF             161.9755930 MHz
WDW            EM
SSB            0
LB             1.00 Hz
GB             0
PC             1.40
```

¹H NMR spectrum of 13 (400 MHz, CDCl₃)



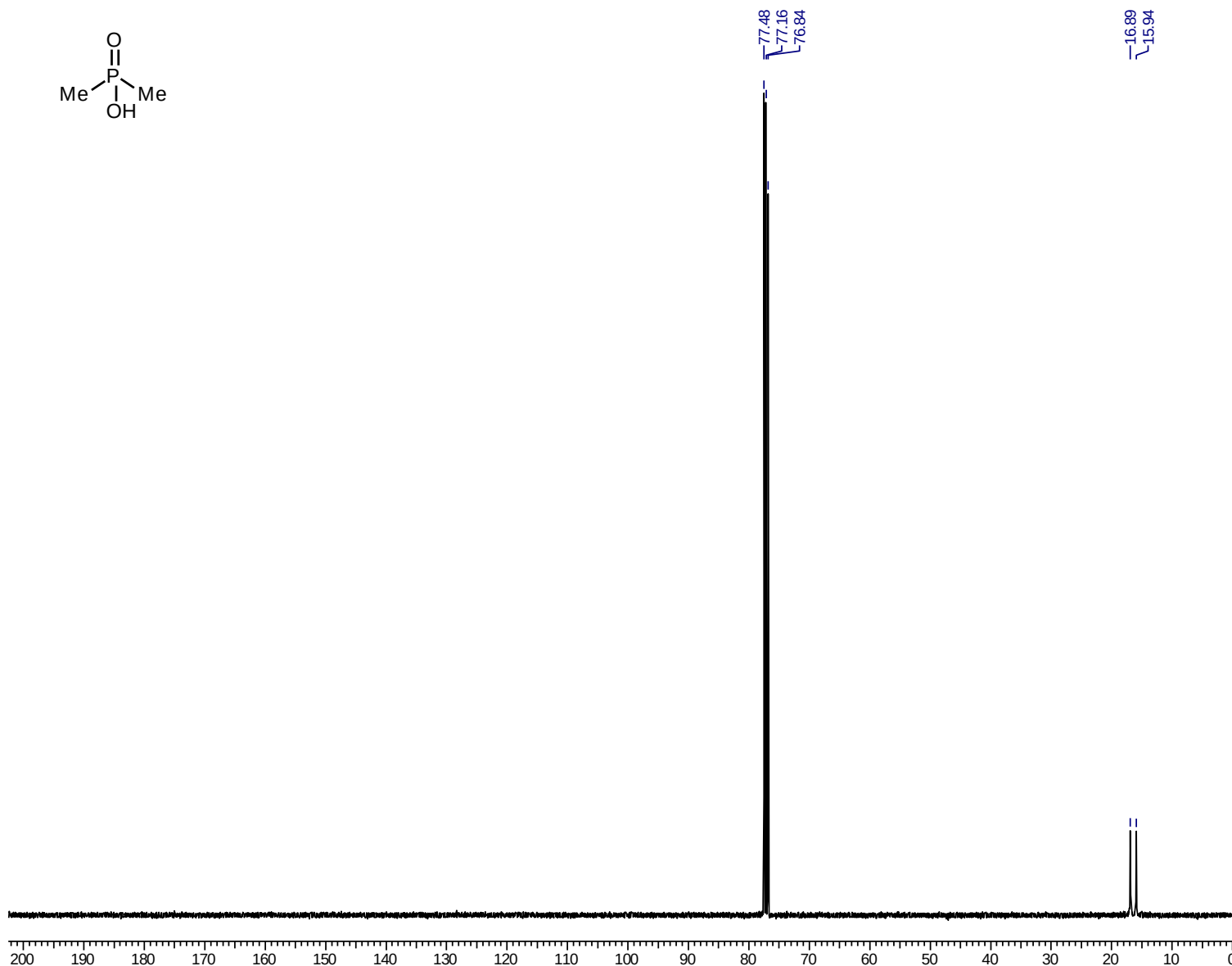
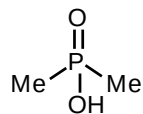
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NAME          acr-49
EXPNO         10
PROCNO        1
INSTRUM       AV400
PROBHD        5 mm PABBO BB-
PULPROG       zg30b
TD            65536
SOLVENT       CDCl3
NS            64
DS            0
SWH           8264.463 Hz
FIDRES        0.126106 Hz
AQ            3.9649780 sec
RG            36
DW            60.500 usec
DE            9.40 usec
TE            294.3 K
D1            1.00000000 sec
TD0           1
  
```

```

===== CHANNEL f1 =====
NUC1          1H
P1            10.00 usec
PL1           -3.60 dB
PL1W          17.83863831 W
SF01          400.1324710 MHz
SI            32768
SF            400.1300983 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
  
```

¹³C NMR spectrum of 13 (100 MHz, CDCl₃)

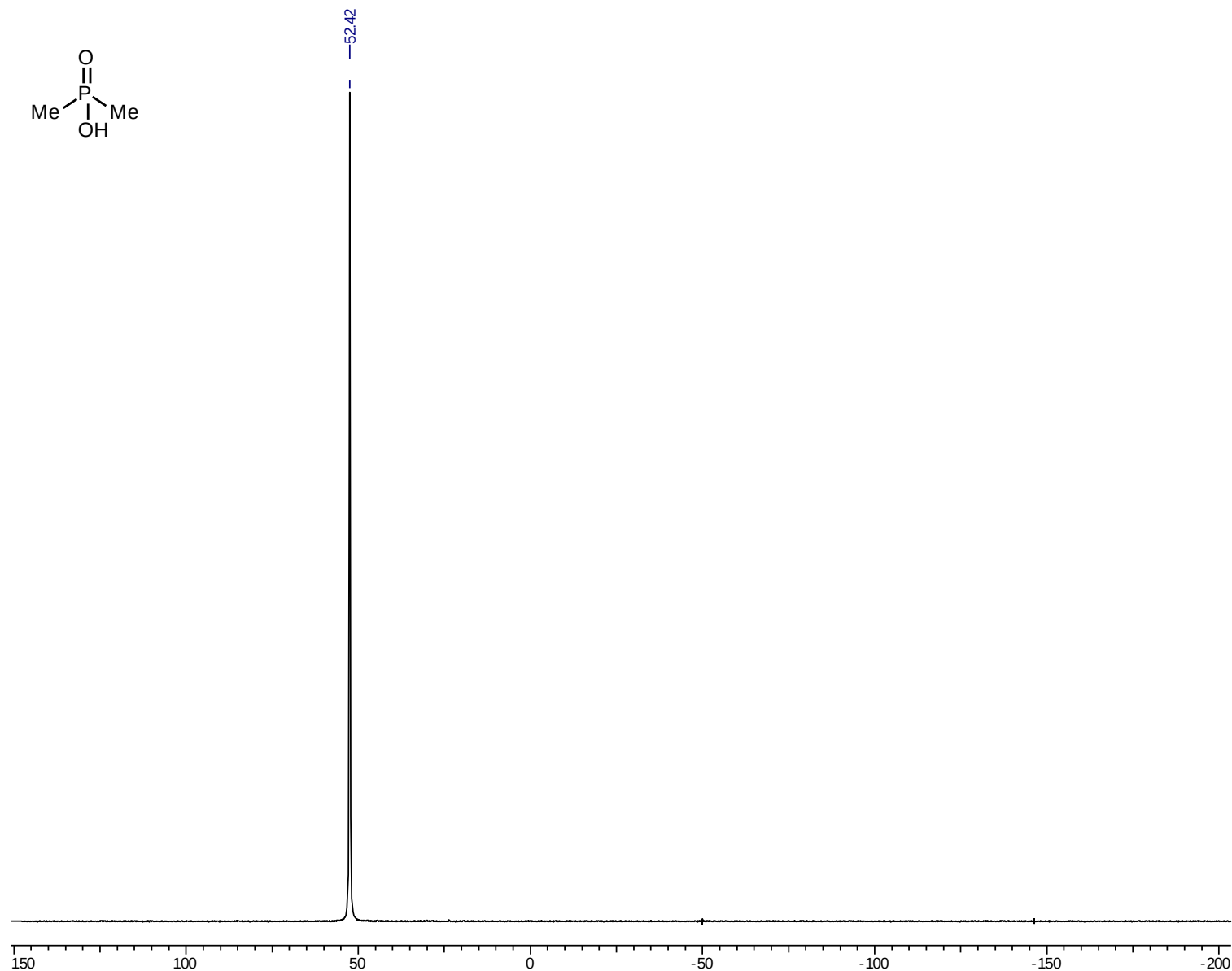
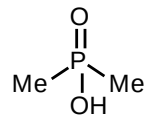


```
NAME          acr-49
EXPNO          11
PROCNO         1
INSTRUM        AV400
PROBHD         5 mm PABBO BB-
PULPROG        zgpg30
TD             65536
SOLVENT        CDCl3
NS             3072
DS             2
SWH            23809.523 Hz
FIDRES         0.363304 Hz
AQ            1.3763061 sec
RG             512
DW            21.000 usec
DE             7.66 usec
TE            295.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0            1
```

```
===== CHANNEL f1 =====
NUC1           13C
P1             8.00 usec
PL1            0.00 dB
PL1W          34.91522217 W
SF01          100.6228294 MHz
```

```
===== CHANNEL f2 =====
CPDPRG2        waltz16
NUC2           1H
PCPD2          90.00 usec
PL2            -3.60 dB
PL12           15.31 dB
PL13           18.00 dB
PL2W          17.83863831 W
PL12W          0.22927761 W
PL13W          0.12341322 W
SF02          400.1316005 MHz
SI            32768
SF            100.6127690 MHz
WDW            EM
SSB            0
LB             1.00 Hz
GB             0
PC             1.40
```

^{31}P (proton-decoupled) NMR spectrum of 13 (162 MHz, CDCl_3)

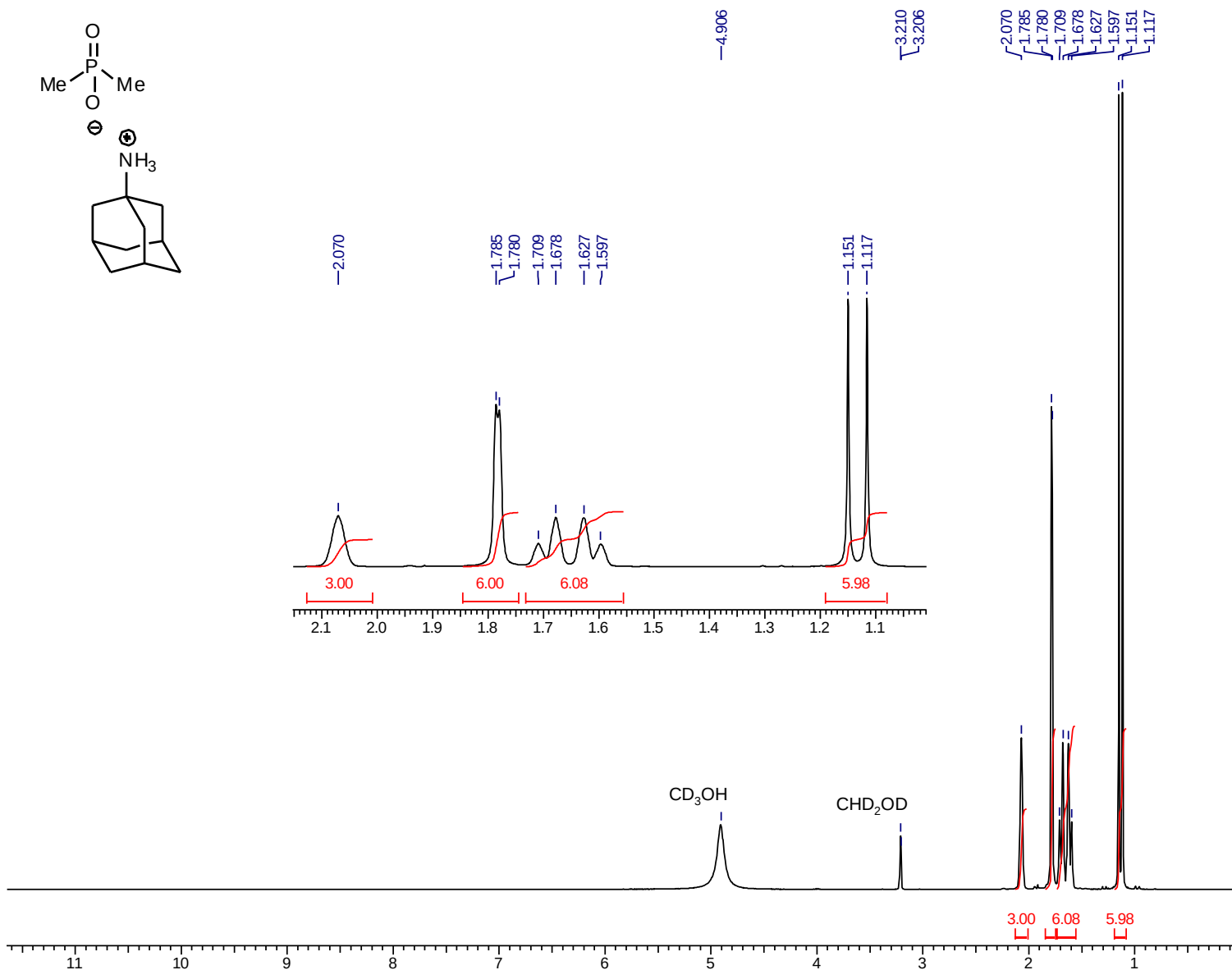


```
NAME          acr-49
EXPNO          13
PROCNO         1
INSTRUM        AV400
PROBHD         5 mm PABBO BB-
PULPROG        zgpg30
TD             65536
SOLVENT        CDCl3
NS             1024
DS             4
SWH            64102.563 Hz
FIDRES         0.978127 Hz
AQ             0.5112308 sec
RG             23100
DW             7.800 usec
DE             6.50 usec
TE             294.9 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0            1
```

```
===== CHANNEL f1 =====
NUC1           31P
P1             9.40 usec
PL1            0.00 dB
PL1W           26.87718391 W
SFO1           161.9674942 MHz
```

```
===== CHANNEL f2 =====
CPDPRG2        waltz16
NUC2           1H
PCPD2          90.00 usec
PL2            -3.60 dB
PL12           15.31 dB
PL13           18.00 dB
PL2W           17.83863831 W
PL12W          0.22927761 W
PL13W          0.12341322 W
SFO2           400.1316005 MHz
SI             32768
SF             161.9755930 MHz
WDW            EM
SSB            0
LB             1.00 Hz
GB             0
PC             1.40
```

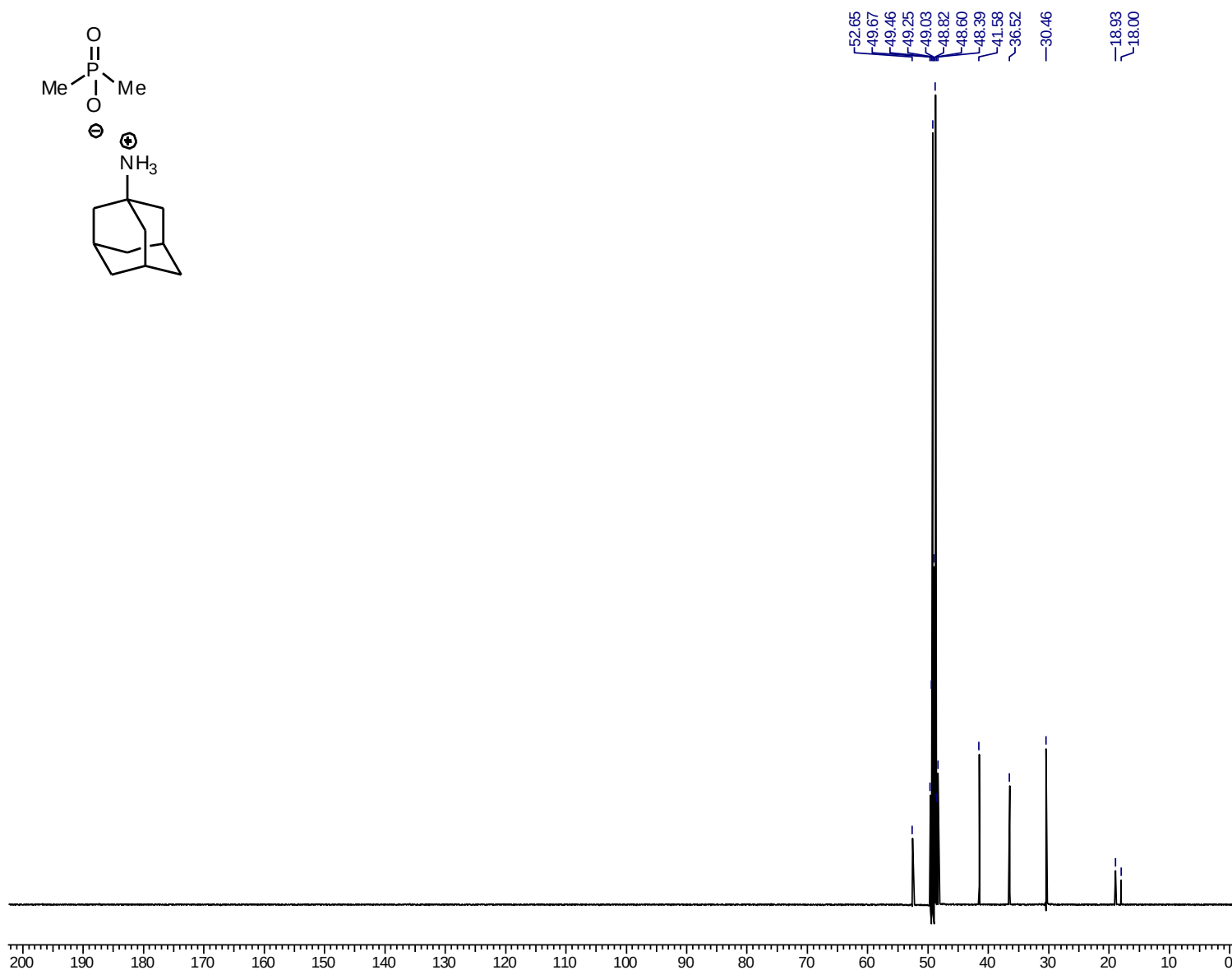
¹H NMR spectrum of the adamantanamine derivative of 13 (400 MHz, CD₃OD)



NAME acr-21
EXPNO 10
PROCNO 1
INSTRUM AV400
PROBHD 5 mm PABBO BB-
PULPROG zg30b
TD 65536
SOLVENT MeOD
NS 16
DS 0
SWH 8264.463 Hz
FIDRES 0.126106 Hz
AQ 3.9649780 sec
RG 80.6
DW 60.500 usec
DE 9.40 usec
TE 294.0 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 10.00 usec
PL1 -3.60 dB
PL1W 17.83863831 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300482 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

¹³C NMR spectrum of the adamantanamine derivative of 13 (100 MHz, CD₃OD)

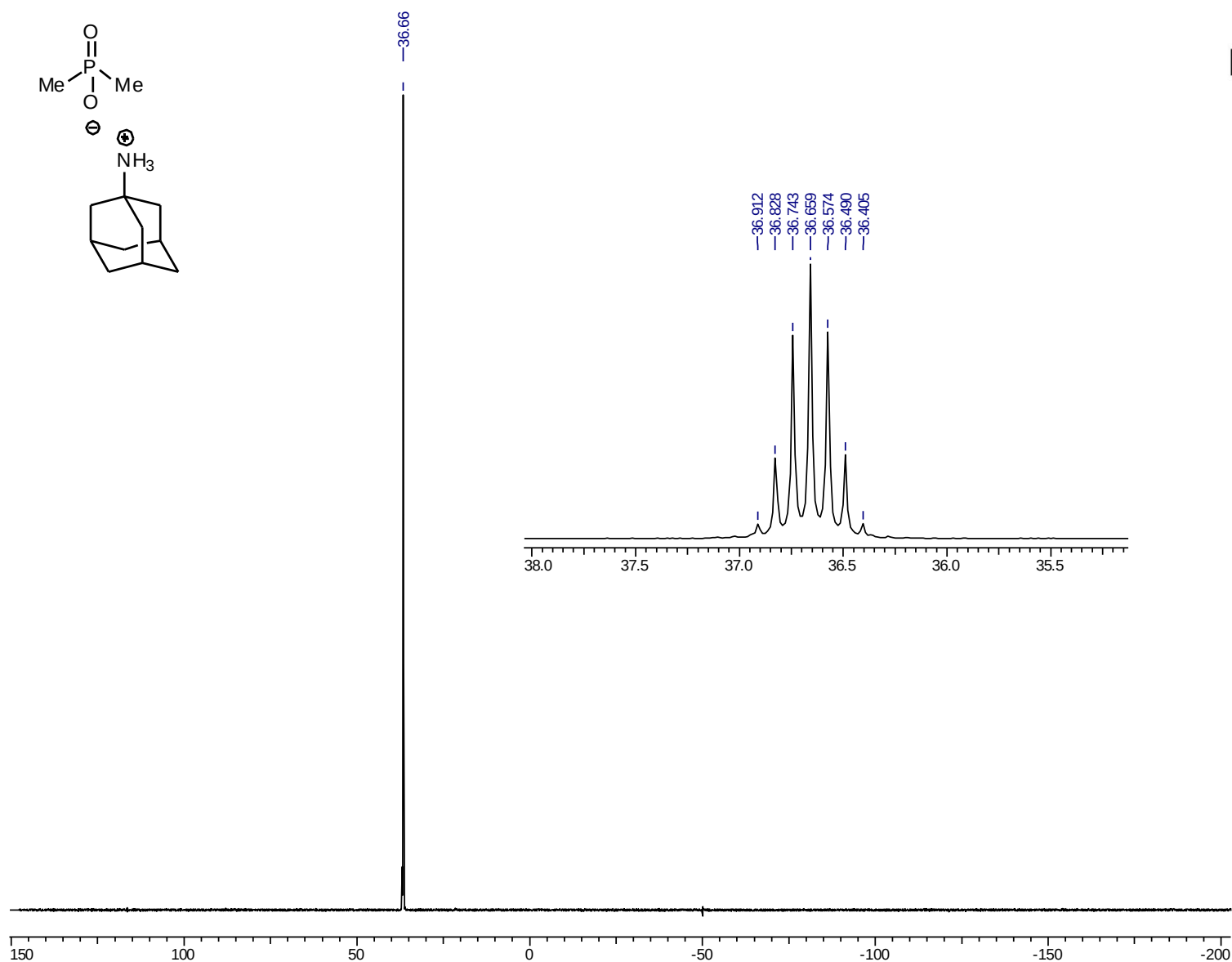


NAME acr-55
EXPNO 11
PROCNO 1
INSTRUM AV400
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT MeOD
NS 3072
DS 2
SWH 23809.523 Hz
FIDRES 0.363304 Hz
AQ 1.3763061 sec
RG 512
DW 21.000 usec
DE 7.66 usec
TE 294.7 K
D1 2.0000000 sec
D11 0.0300000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 8.00 usec
PL1 0.00 dB
PL1W 34.91522217 W
SF01 100.6228294 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -3.60 dB
PL12 15.31 dB
PL13 18.00 dB
PL2W 17.83863831 W
PL12W 0.22927761 W
PL13W 0.12341322 W
SF02 400.1316005 MHz
SI 32768
SF 100.6126261 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

³¹P (proton-coupled) NMR spectrum of the adamantanamine derivative of 13 (162 MHz, CD₃OD)



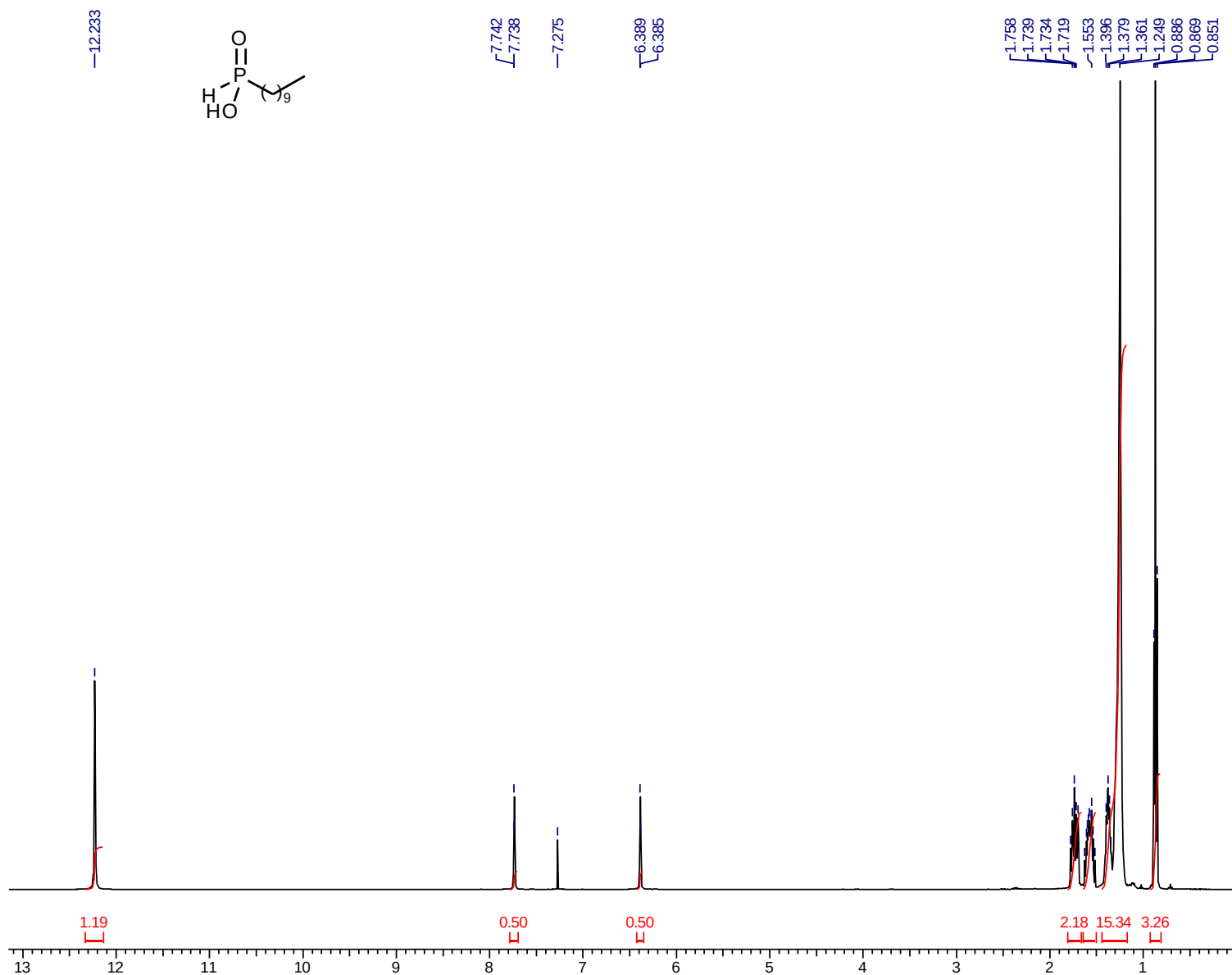
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NAME          acr-55
EXPNO         14
PROCNO        1
INSTRUM       AV400
PROBHD        5 mm PABBO BB-
PULPROG       zg30
TD            65536
SOLVENT       MeOD
NS            1024
DS            4
SWH           64102.563 Hz
FIDRES        0.978127 Hz
AQ            0.5112308 sec
RG            23100
DW            7.800 usec
DE            6.50 usec
TE            293.8 K
D1            2.00000000 sec
TD0           1
    
```

```

===== CHANNEL f1 =====
NUC1          31P
P1            9.40 usec
PL1           0.00 dB
PL1W          26.87718391 W
SF01          161.9674942 MHz
SI            32768
SF            161.9755930 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
    
```

¹H NMR spectrum of 15 (400 MHz, CDCl₃)



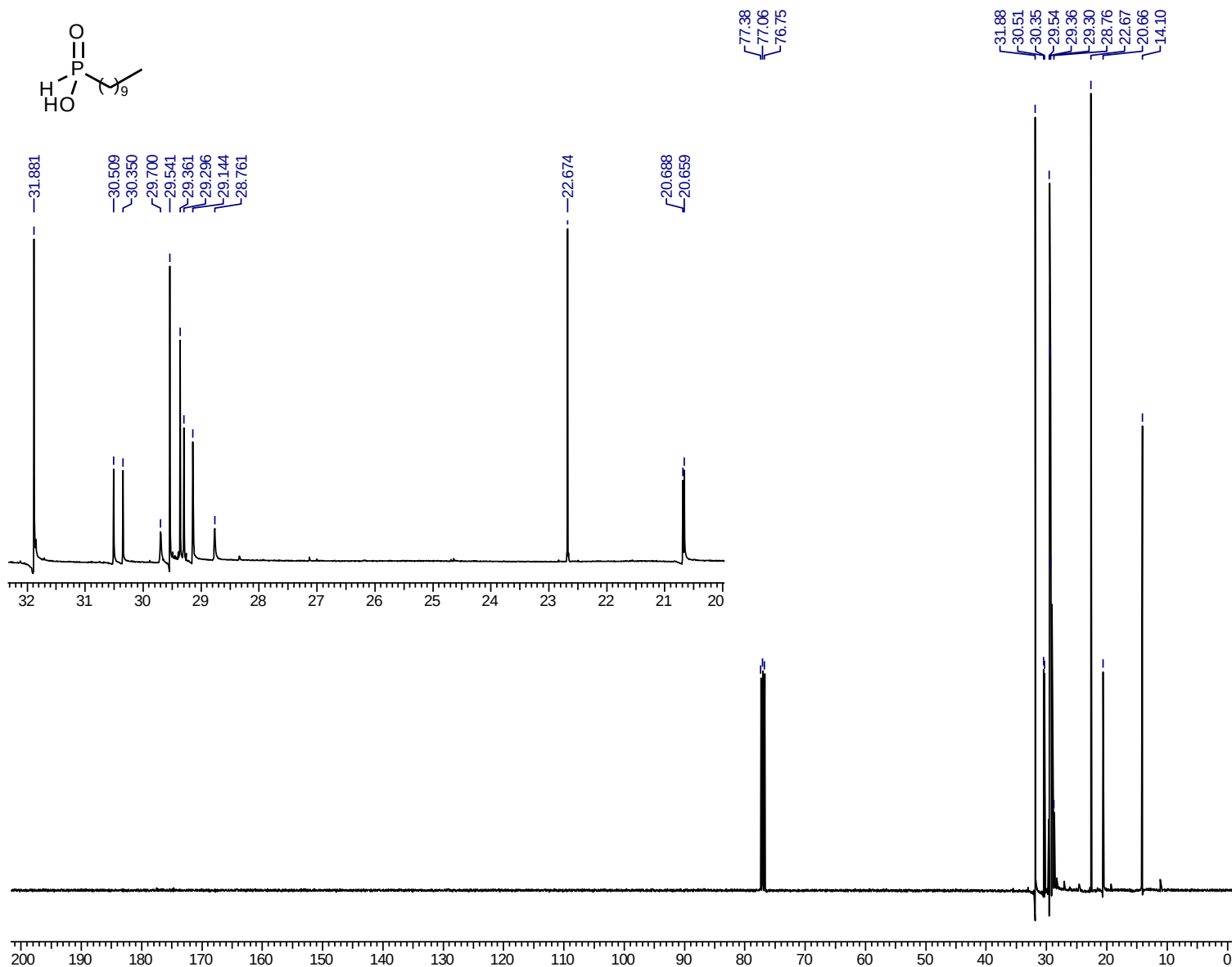
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NAME          acr-53
EXPNO          10
PROCNO         1
INSTRUM        AV400
PROBHD         5 mm PABBO BB-
PULPROG        zg30b
TD             65536
SOLVENT        CDCl3
NS             128
DS             0
SWH            8264.463 Hz
FIDRES         0.126106 Hz
AQ            3.9649780 sec
RG             25.4
DW            60.500 usec
DE             9.40 usec
TE            294.5 K
D1            1.00000000 sec
TD0            1
  
```

```

===== CHANNEL f1 =====
NUC1           1H
P1             10.00 usec
PL1            -3.60 dB
PL1W           17.83863831 W
SF01           400.1324710 MHz
SI             32768
SF            400.1300000 MHz
WDW            EM
SSB            0
LB             0.30 Hz
GB             0
PC             1.00
  
```

¹³C NMR spectrum of 15 (100 MHz, CDCl₃)



```

NAME          acr-53
EXPNO         11
PROCNO        1
INSTRUM       AV400
PROBHD        5 mm PABBO BB-
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            3072
DS            2
SWH           23809.523 Hz
FIDRES        0.363304 Hz
AQ            1.3763061 sec
RG            512
DW            21.000 usec
DE            7.66 usec
TE            296.1 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0           1
  
```

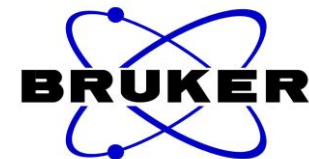
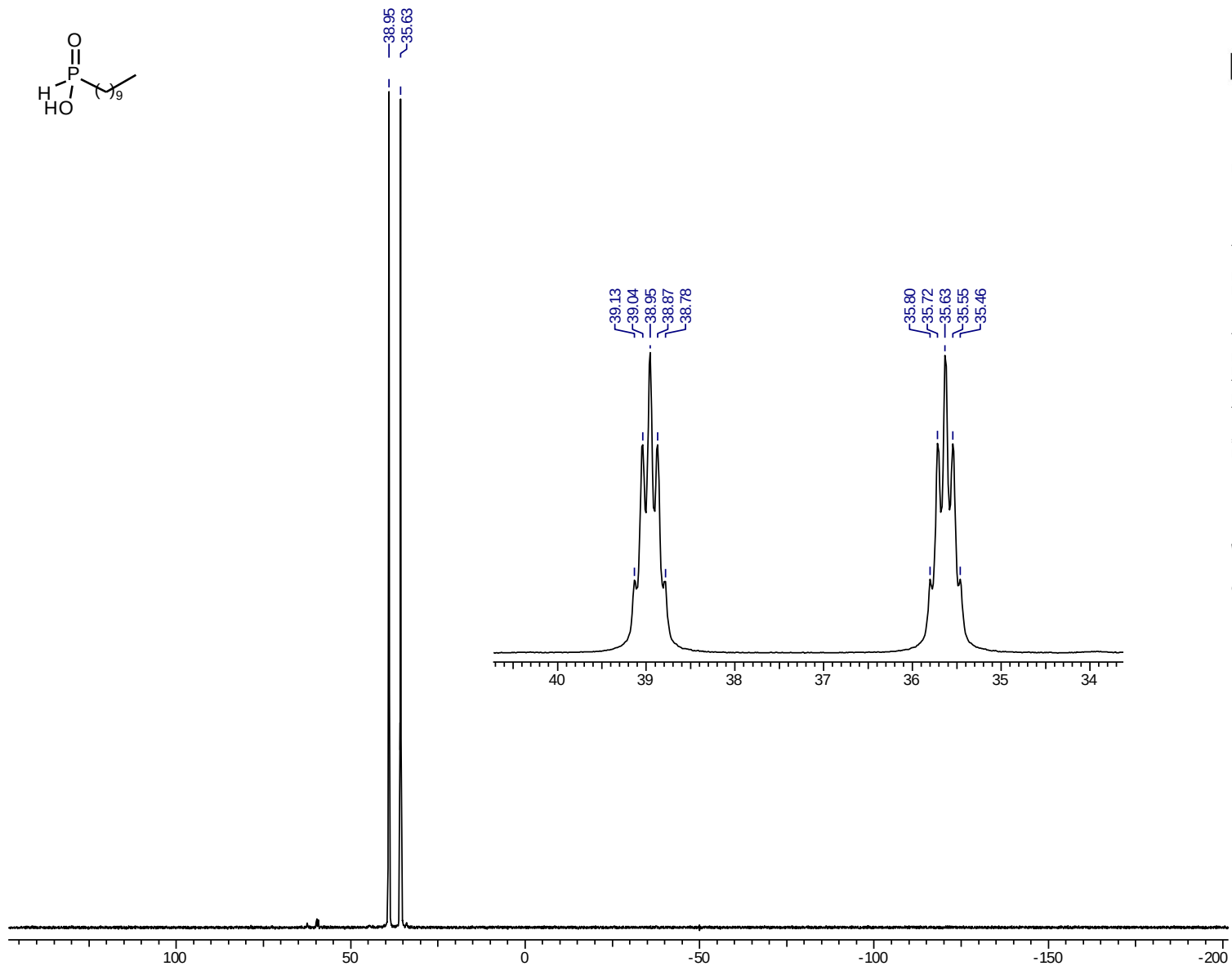
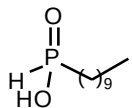
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===== CHANNEL f1 =====
NUC1          13C
P1            8.00 usec
PL1           0.00 dB
PL1W          34.91522217 W
SF01          100.6228294 MHz
  
```

```

===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2          1H
PCPD2         80.00 usec
PL2           -3.60 dB
PLI2          13.80 dB
PLI3          14.00 dB
PL2W          17.83863831 W
PLI2W         0.32460985 W
PLI3W         0.31000000 W
SF02          400.1316005 MHz
SI            32768
SF            100.6127690 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
  
```

³¹P (proton-coupled) NMR spectrum of 15 (162 MHz, CDCl₃)



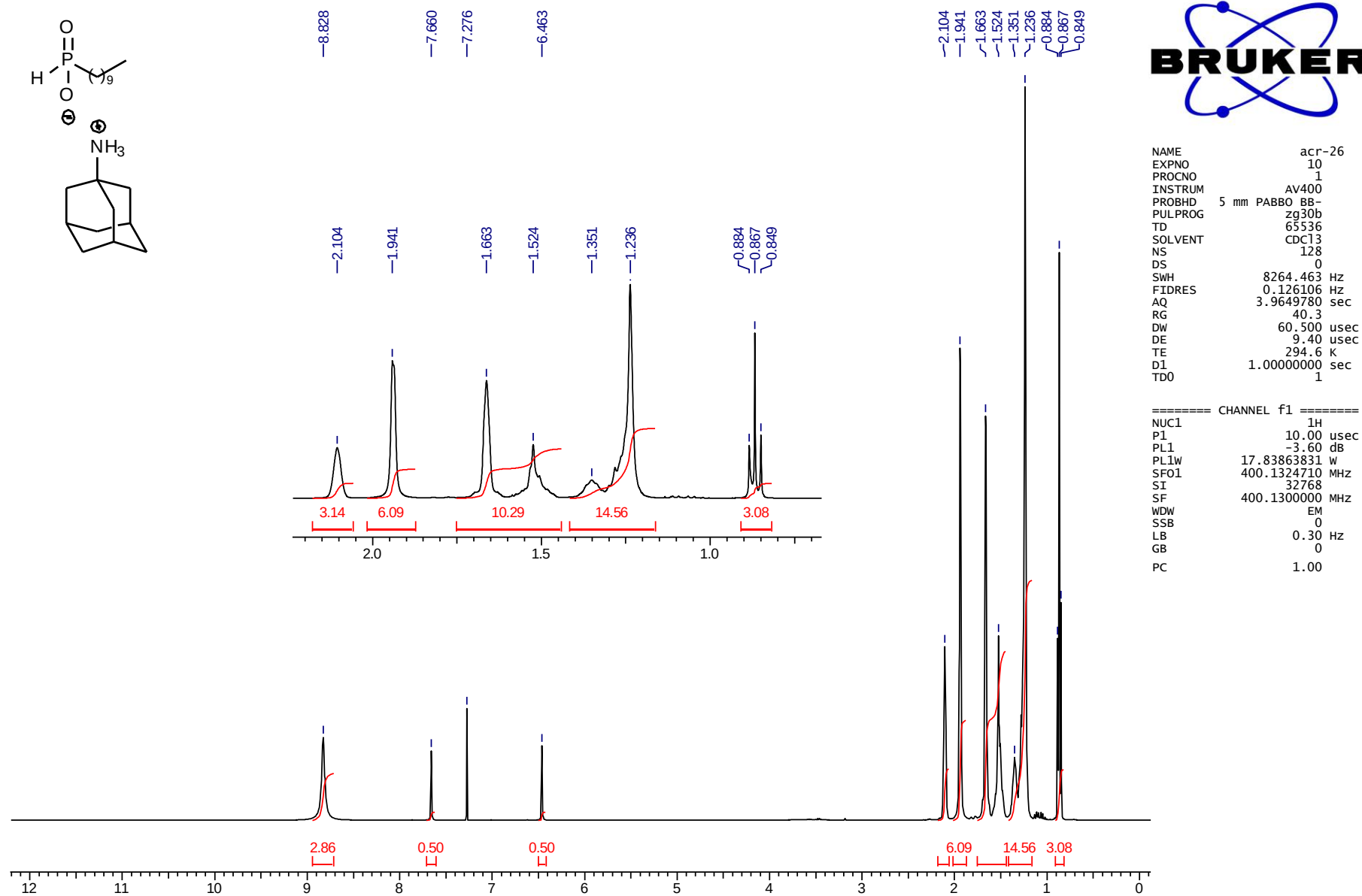
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EXPNO		15
PROCNO		1
INSTRUM		AV400
PROBHD	5 mm	PABBO BB-
PULPROG		zg30
TD		65536
SOLVENT		CDCl3
NS		1024
DS		4
SWH	64102.563	Hz
FIDRES	0.978127	Hz
AQ	0.5112308	sec
RG	23100	
DW	7.800	usec
DE	6.50	usec
TE	294.5	K
D1	2.00000000	sec
TD0		1

```

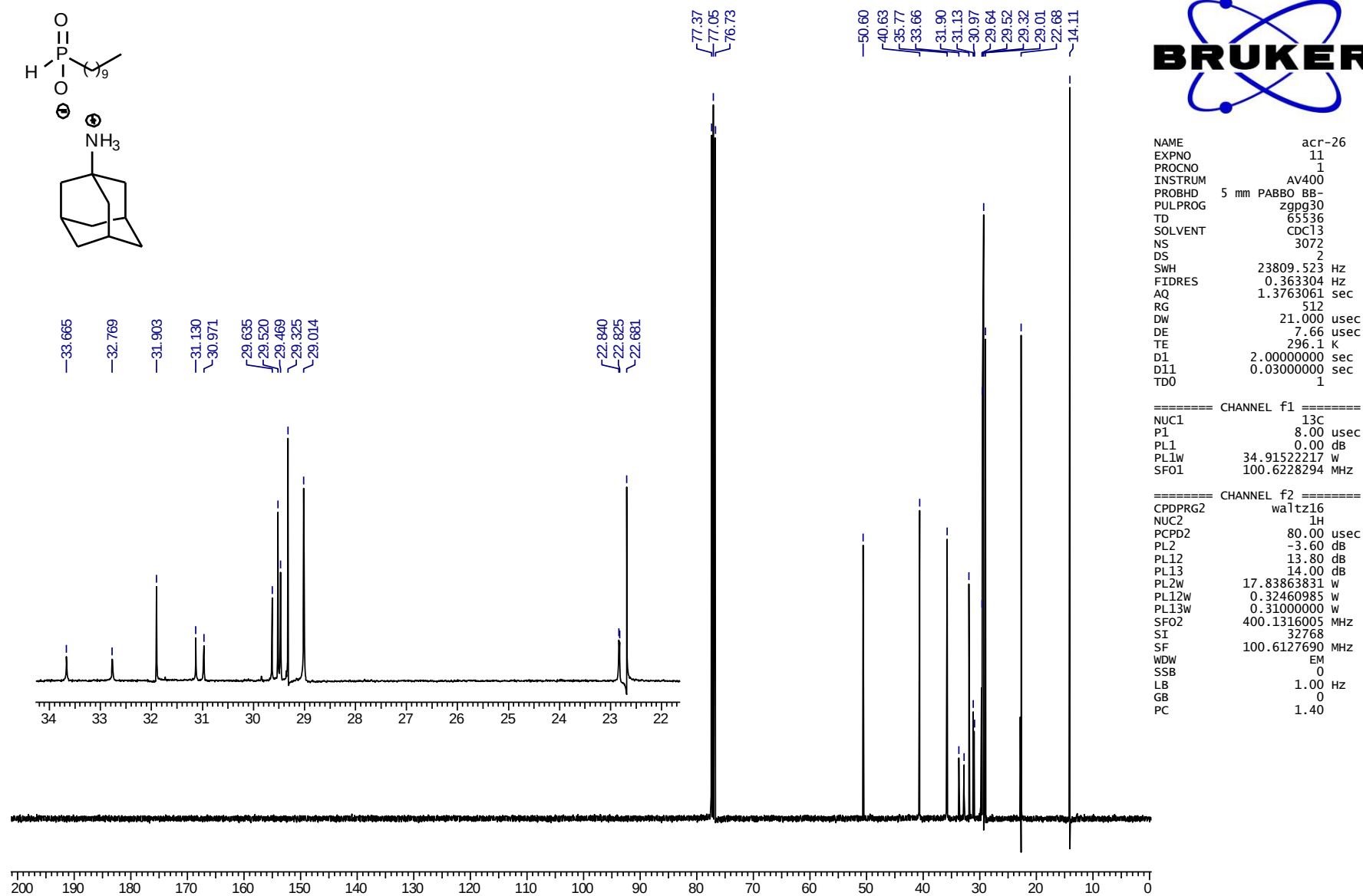
===== CHANNEL f1 =====
NUC1                31P
P1                  9.40 usec
PL1                 0.00 dB
PL1W                26.87718391 W
SF01                161.9674942 MHz
SI                  32768
SF                  161.9755930 MHz
WDW                  EM
SSB                  0
LB                  1.00 Hz
GB                  0
PC                  1.40

```

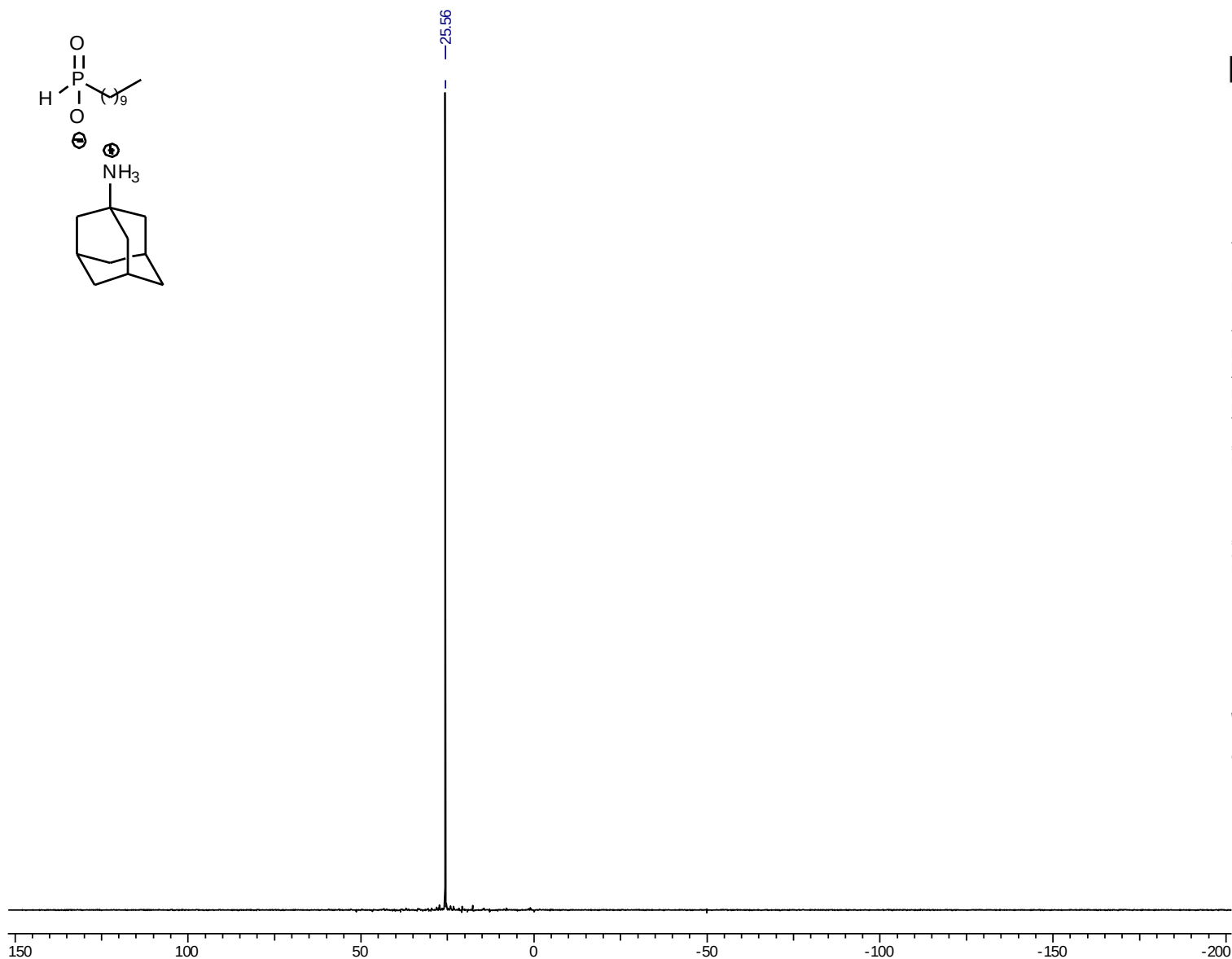
¹H NMR spectrum of the adamantanamine derivative of 15 (400 MHz, CDCl₃)



¹³C NMR spectrum of the adamantanamine derivative of 15 (100 MHz, CDCl₃)



³¹P (proton-decoupled) NMR spectrum of the adamantanamine derivative of 15 (162 MHz, CDCl₃)



```

NAME          acr-26
EXPNO          14
PROCNO         1
INSTRUM        AV400
PROBHD         5 mm PABBO BB-
PULPROG        zgpg30
TD             65536
SOLVENT        CDCl3
NS             1024
DS             4
SWH            64102.563 Hz
FIDRES         0.978127 Hz
AQ             0.5112308 sec
RG             23100
DW             7.800 usec
DE             6.50 usec
TE             296.1 K
D1             2.0000000 sec
D11            0.03000000 sec
TD0            1
  
```

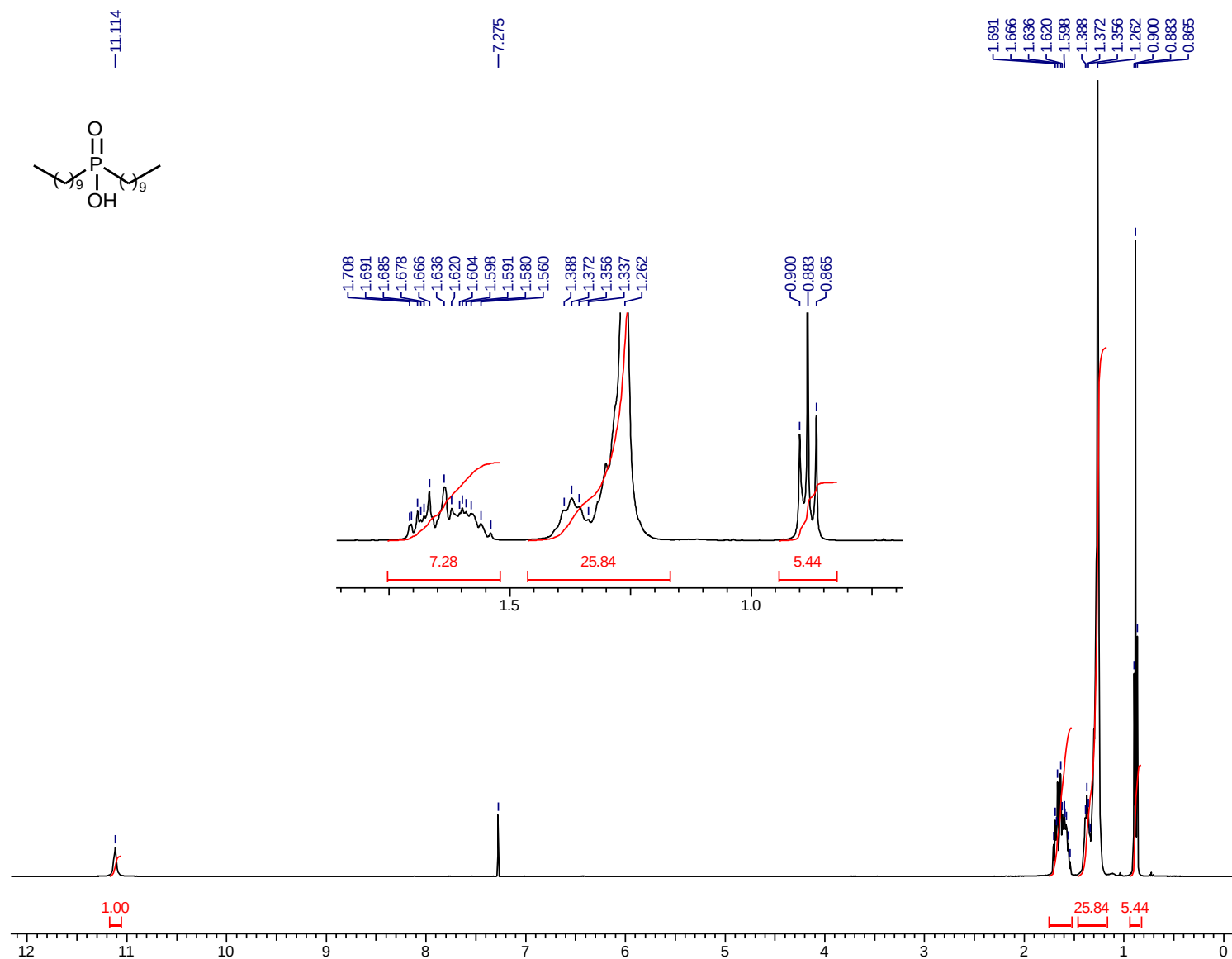
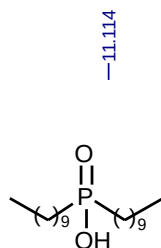
```

===== CHANNEL f1 =====
NUC1           31P
P1             9.40 usec
PL1            0.00 dB
PL1W           26.87718391 W
SF01           161.9674942 MHz
  
```

```

===== CHANNEL f2 =====
CPDPRG2        waltz16
NUC2           1H
PCPD2          80.00 usec
PL2            -3.60 dB
PL12           13.80 dB
PL13           14.00 dB
PL2W           17.83863831 W
PL12W          0.32460985 W
PL13W          0.31000000 W
SF02           400.1316005 MHz
SI             32768
SF             161.9755930 MHz
WDW            EM
SSB            0
LB             1.00 Hz
GB             0
PC             1.40
  
```


¹H NMR spectrum of 16 (400 MHz, CDCl₃)



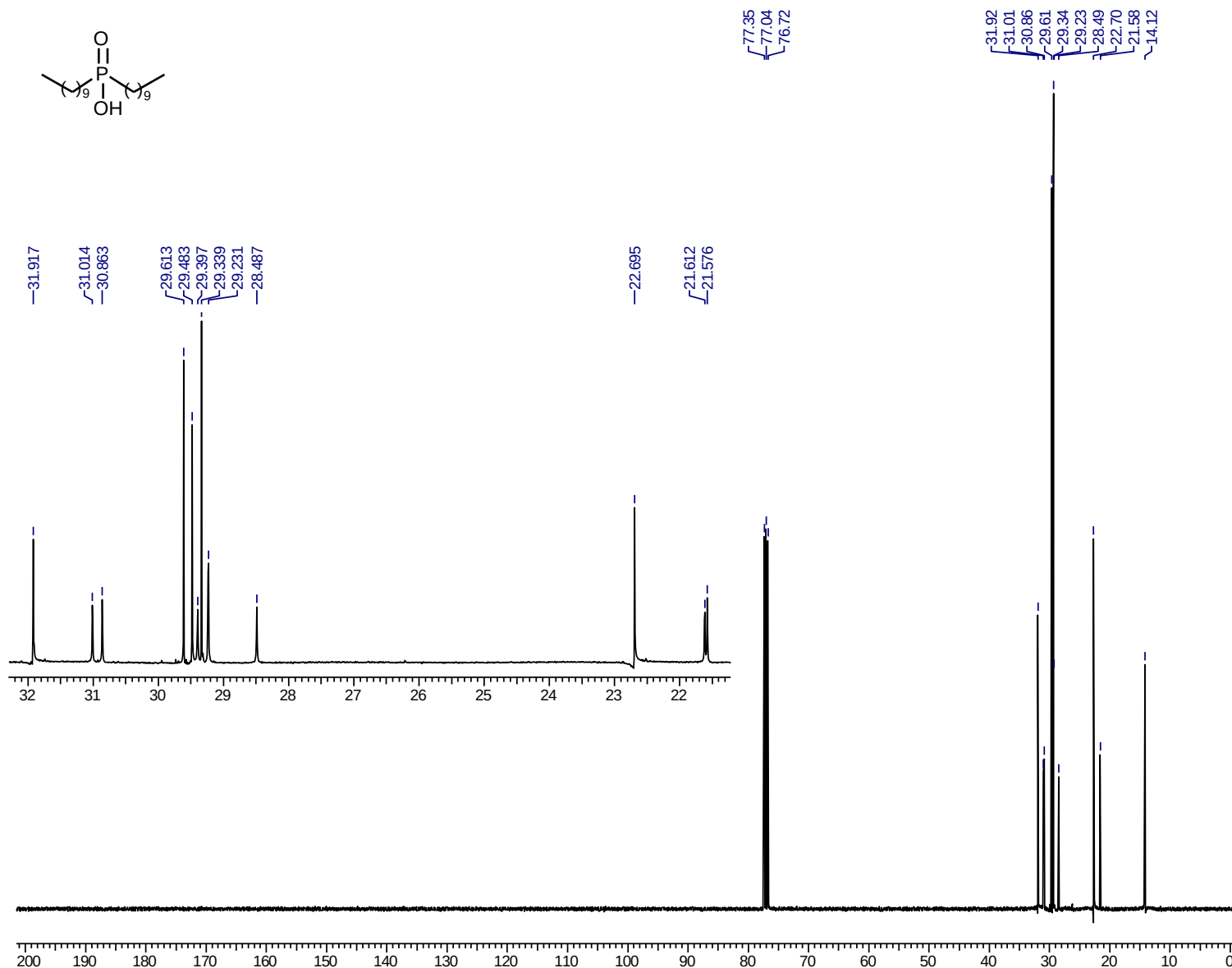
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EXPNO                               10
PROCNO                               1
INSTRUM                             AV400
PROBHD      5 mm PABBO BB-
PULPROG                             zg30b
TD                                 65536
SOLVENT                             CDC13
NS                                 128
DS                                  0
SWH                                8264.463  Hz
FIDRES                             0.126106  Hz
AQ                                3.9649780  sec
RG                                  36
DW                                60.500  usec
DE                                 9.40  usec
TE                                 294.4  K
D1                                1.00000000  sec
TD0                                 1
```

```

===== CHANNEL f1 =====
NUC1                1H
P1                  10.00 usec
PL1                 -3.60 dB
PLW                 17.83863831 W
SF01                400.1324710 MHz
SI                  32768
SF                 400.1300000 MHz
WDW                 EM
SSB                  0
LB                   0.30 Hz
GB                   0
PC                   1.00

```

¹³C NMR spectrum of 16 (100 MHz, CDCl₃)



```

NAME          acr-54
EXPNO         11
PROCNO        1
INSTRUM       AV400
PROBHD        5 mm PABBO BB-
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            3072
DS            2
SWH           23809.523 Hz
FIDRES        0.363304 Hz
AQ            1.3763061 sec
RG            512
DW            21.000 usec
DE            7.66 usec
TE            296.1 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0           1
  
```

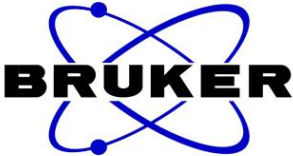
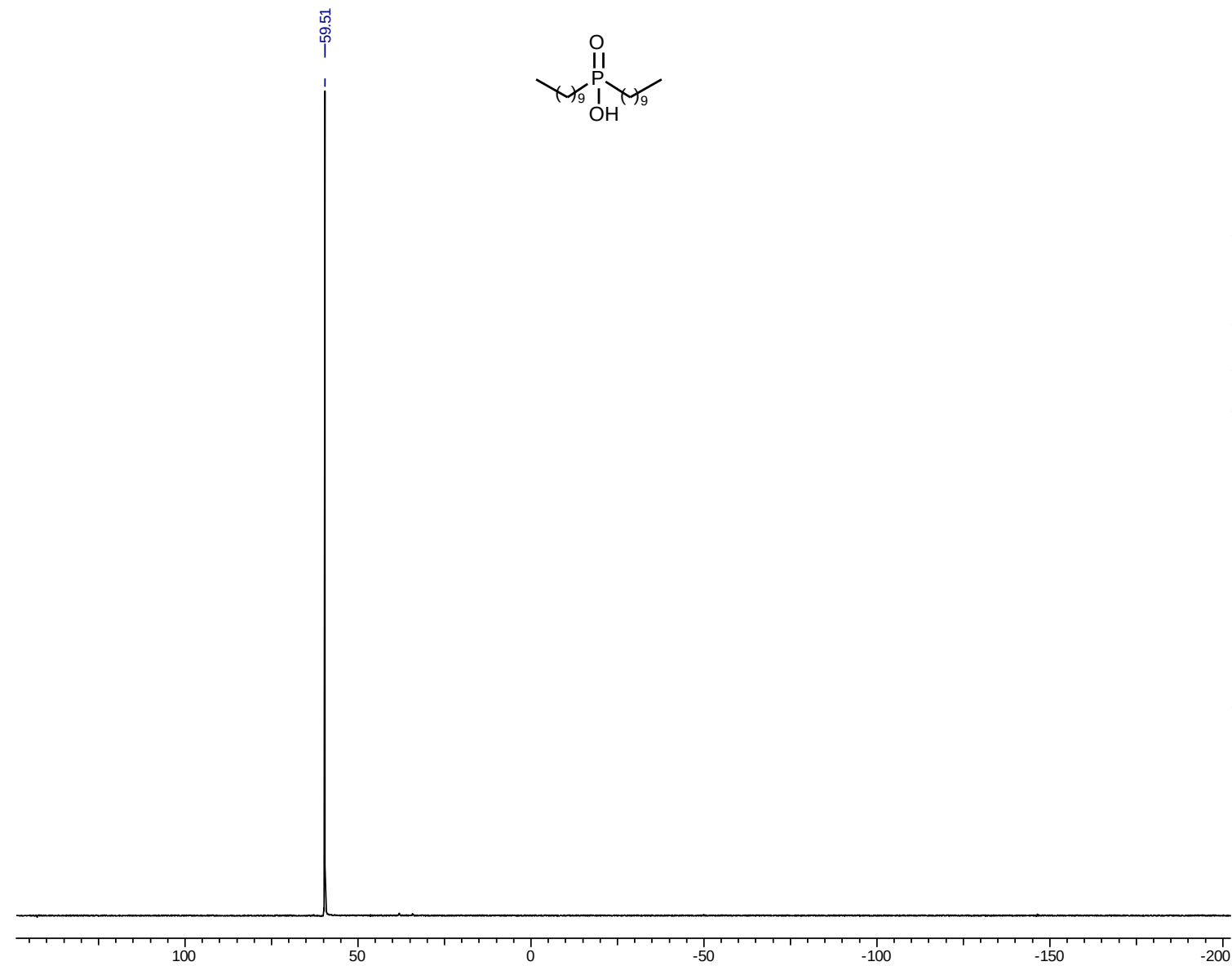
```

===== CHANNEL f1 =====
NUC1          13C
P1            8.00 usec
PL1           0.00 dB
PL1W          34.91522217 W
SF01          100.6228294 MHz
  
```

```

===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2          1H
PCPD2         80.00 usec
PL2           -3.60 dB
PL12          13.80 dB
PL13          14.00 dB
PL2W          17.83863831 W
PL12W         0.32460985 W
PL13W         0.31000000 W
SF02          400.1316005 MHz
SI            32768
SF            100.6127690 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
  
```

³¹P (proton-decoupled) NMR spectrum of 16 (162 MHz, CDCl₃)

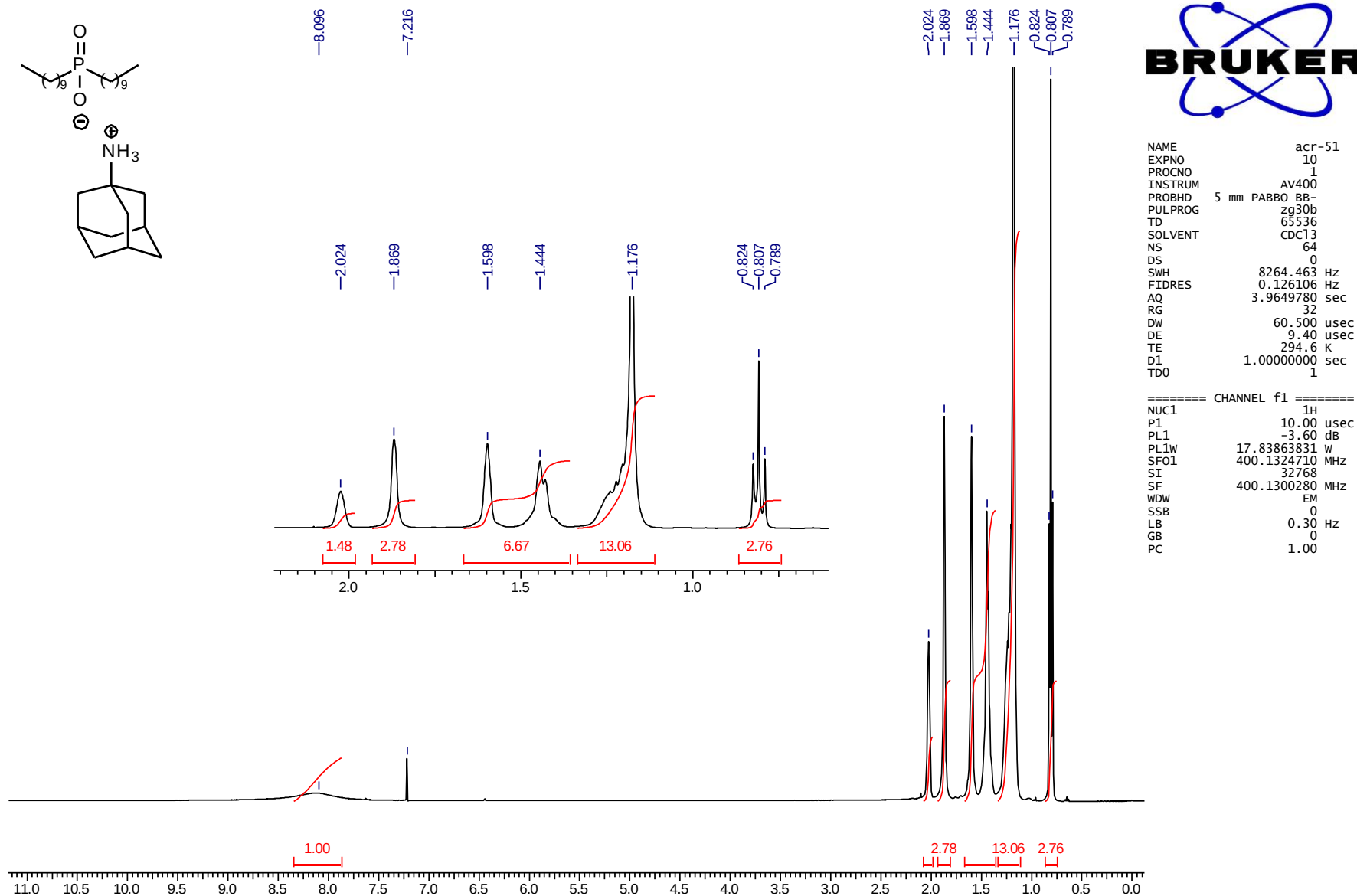


NAME acr-54
EXPNO 13
PROCNO 1
INSTRUM AV400
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 1024
DS 4
SWH 64102.563 Hz
FIDRES 0.978127 Hz
AQ 0.5112308 sec
RG 23100
DW 7.800 usec
DE 6.50 usec
TE 296.0 K
D1 2.0000000 sec
D11 0.0300000 sec
TD0 1

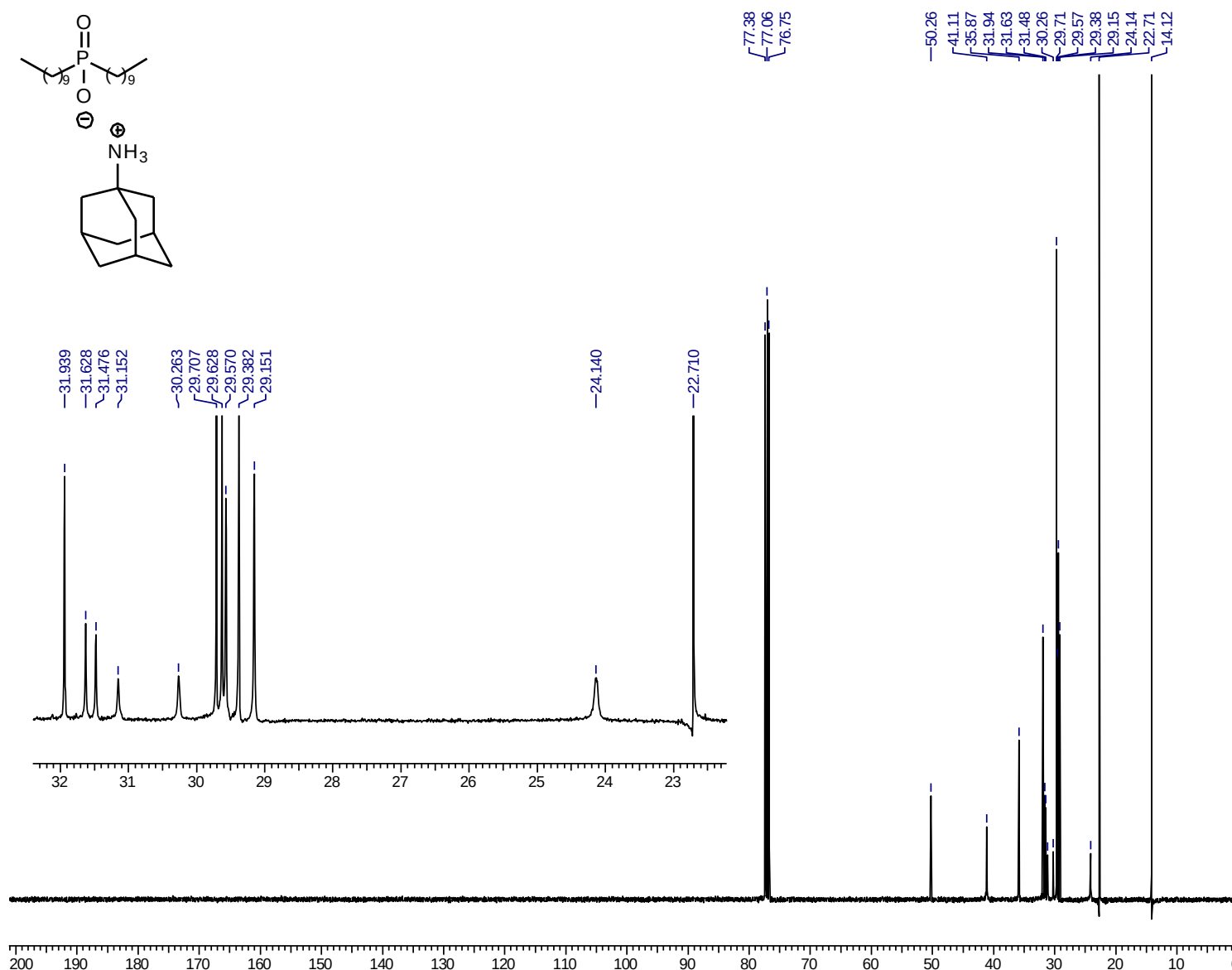
===== CHANNEL f1 =====
NUC1 31P
P1 9.40 usec
PL1 0.00 dB
PL1W 26.87718391 W
SF01 161.9674942 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -3.60 dB
PL12 13.80 dB
PL13 14.00 dB
PL2W 17.83863831 W
PL12W 0.32460985 W
PL13W 0.31000000 W
SF02 400.1316005 MHz
SI 32768
SF 161.9755930 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

¹H NMR spectrum of the adamantanamine derivative of 16 (400 MHz, CDCl₃)



¹³C NMR spectrum of the adamantanamine derivative of 16 (100 MHz, CDCl₃)



```

NAME          acr-51
EXPNO         11
PROCNO        1
INSTRUM       AV400
PROBHD        5 mm PABBO BB-
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            3072
DS            2
SWH           23809.523 Hz
FIDRES        0.363304 Hz
AQ            1.3763061 sec
RG            512
DW            21.000 usec
DE            7.66 usec
TE            295.3 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0           1
  
```

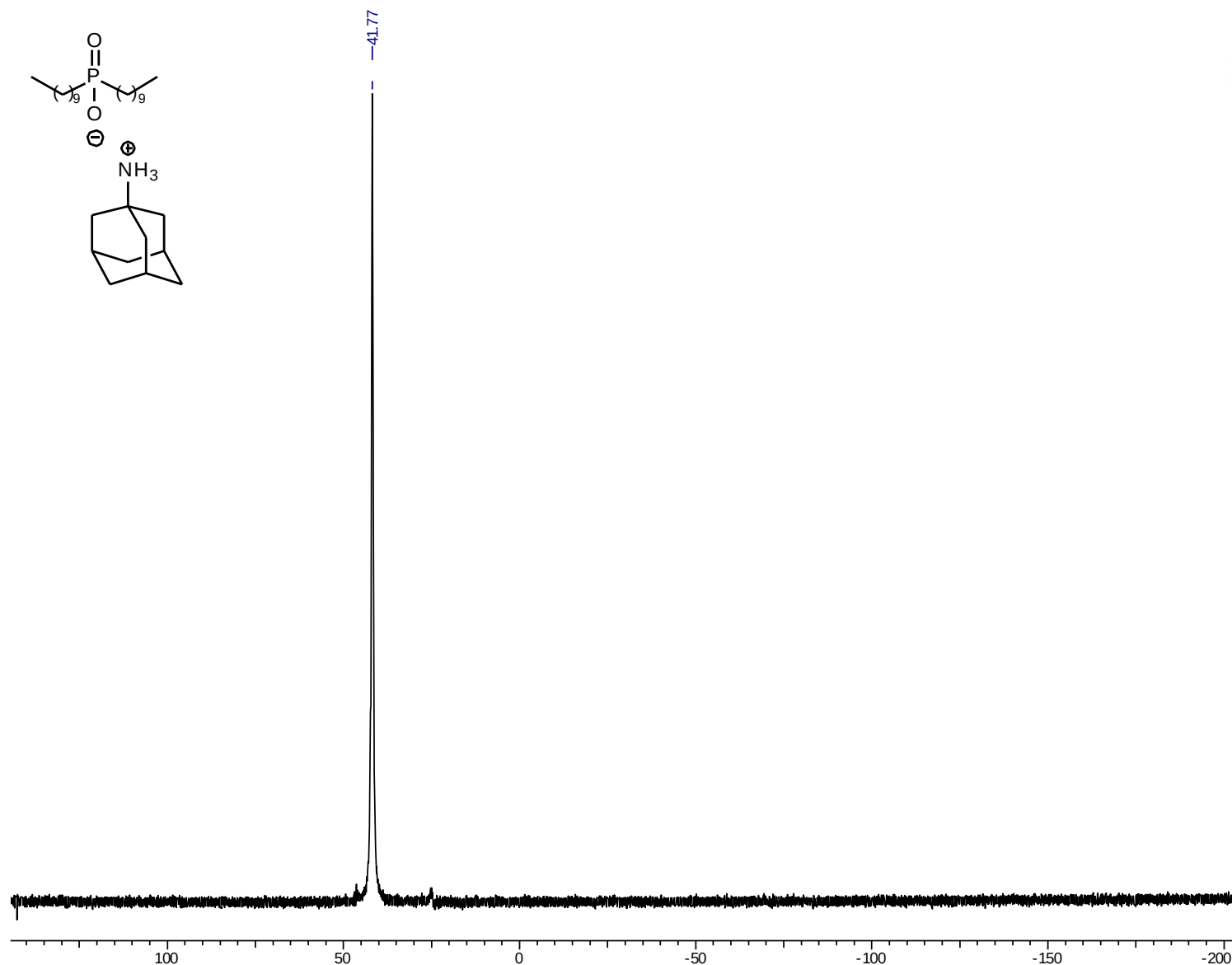
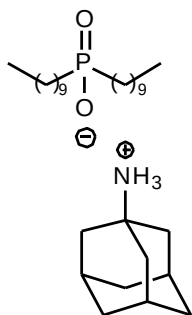
```

===== CHANNEL f1 =====
NUC1          13C
P1            8.00 usec
PL1           0.00 dB
PL1W          34.91522217 W
SFO1          100.6228294 MHz
  
```

```

===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2          1H
PCPD2         90.00 usec
PL2           -3.60 dB
PL12          15.31 dB
PL13          18.00 dB
PL2W          17.83863831 W
PL12W         0.22927761 W
PL13W         0.12341322 W
SFO2          400.1316005 MHz
SI            32768
SF            100.6127690 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
  
```

³¹P (proton-decoupled) NMR spectrum of the adamantanamine derivative of 16 (162 MHz, CDCl₃)



NAME	acr-51	
EXPNO	15	
PROCNO	1	
INSTRUM	AV400	
PROBHD	5 mm	PABBO BB-
PULPROG	zgpg30	
TD	65536	
SOLVENT	CDCl3	
NS	1024	
DS	4	
SWH	64102.563	Hz
FDRES	0.978127	Hz
RG	0.5112308	sec
AQ	23100	
DW	7.800 usec	
DE	6.50 usec	
TE	295.3 K	
D1	2.00000000	sec
D11	0.03000000	sec
TD0	1	

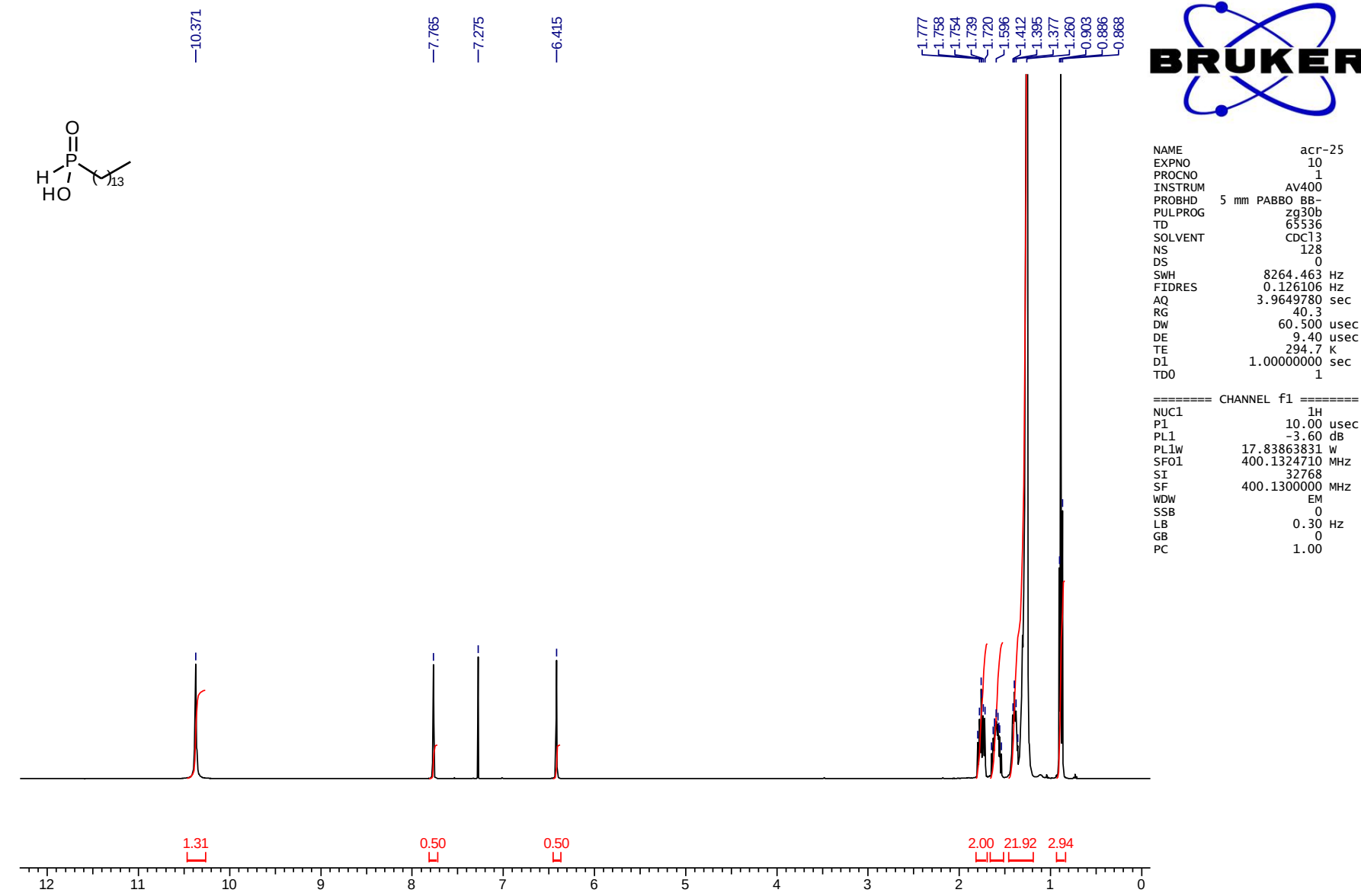
```
===== CHANNEL f1 =====
NUC1          31P
P1             9.40 usec
PL1            0.00 dB
PL1W          26.87718391 W
SF01          161.9674942 MHz
```

```

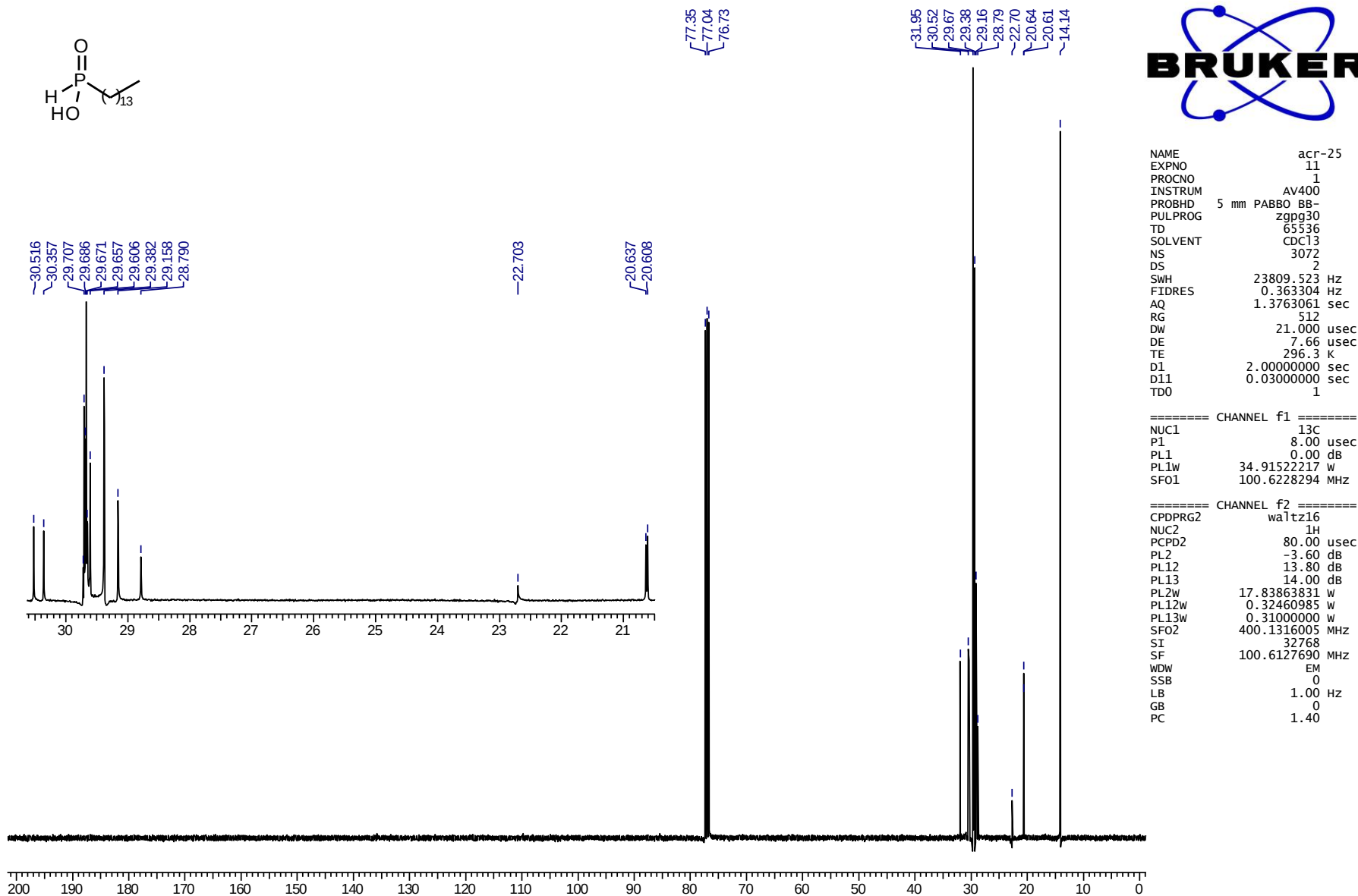
===== CHANNEL f2 =====
CDPRG2      waitz16
NUC2        1H
PCPD2       90.00 usec
PL2         -3.60 dB
PL12        15.31 dB
PL13        18.00 dB
PL2W        17.8386381  W
PL12W       0.22927761  W
PL13W       0.12341322  W
SF02        400.1316005 MHz
SI           32768
SF          161.9755930 MHz
WDW          EM
SSB          0
LB           1.00 Hz
GB           0
PC           1.40

```

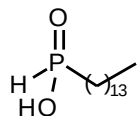
¹H NMR spectrum of 17 (400 MHz, CDCl₃)



¹³C NMR spectrum of 17 (100 MHz, CDCl₃)



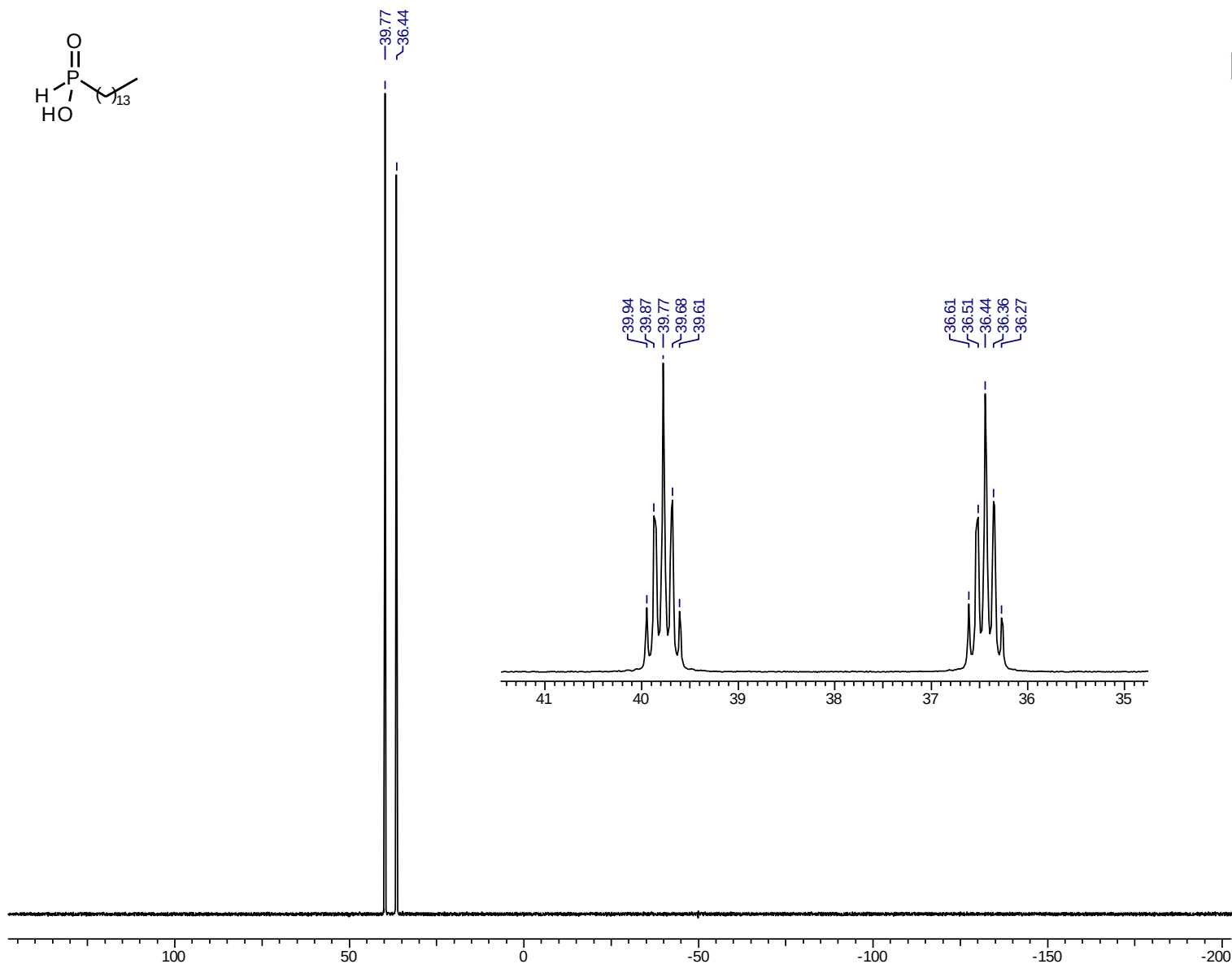
^{31}P (proton-coupled) NMR spectrum of 17 (162 MHz, CDCl_3)



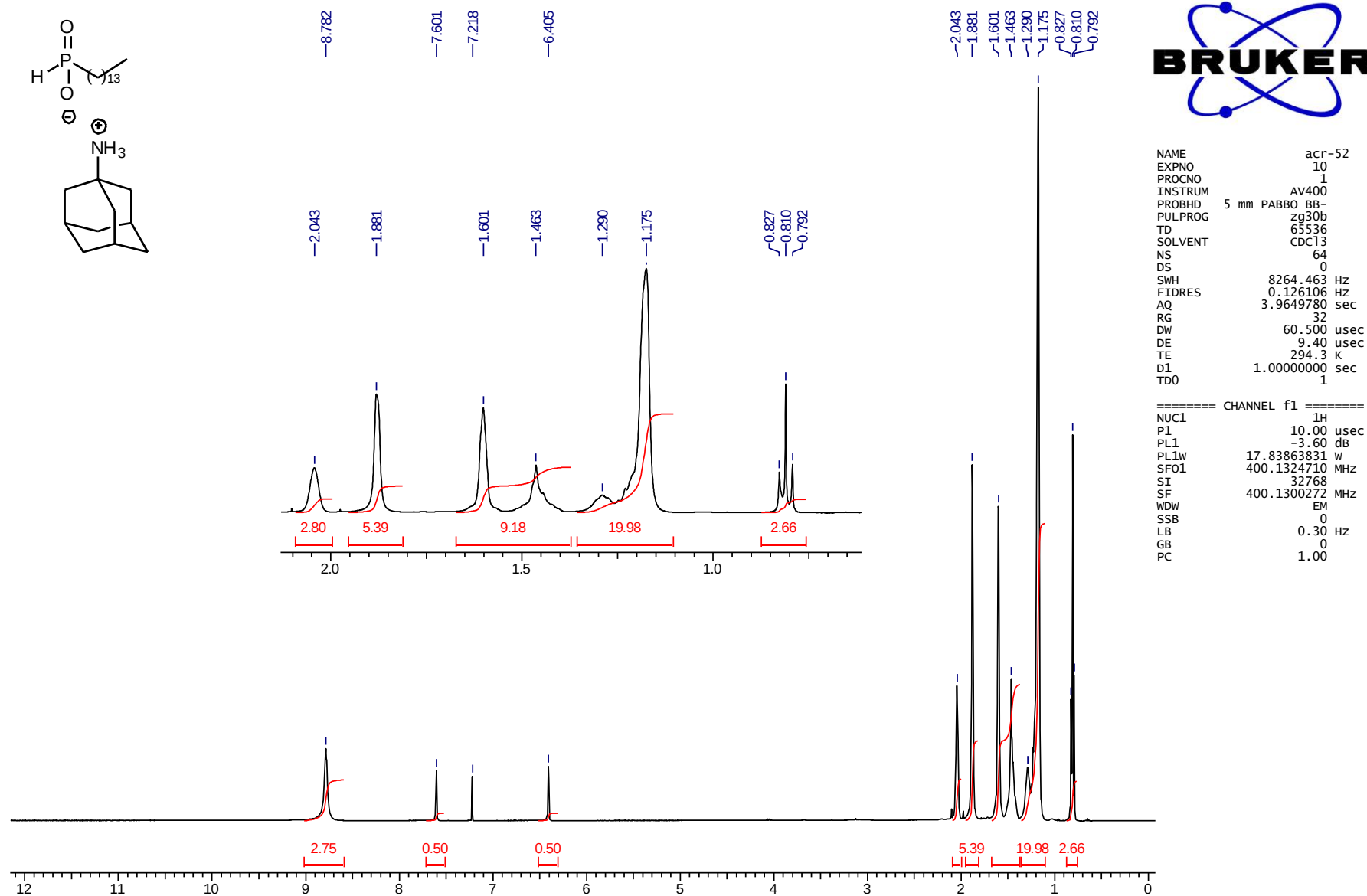
```

NAME          acr-25
EXPNO         15
PROCNO        1
INSTRUM       AV400
PROBHD        5 mm PABBO BB-
PULPROG       zg30
TD            65536
SOLVENT       CDC13
NS            1024
DS            4
SWH           64102.563 Hz
FIDRES        0.978127 Hz
AQ            0.5112308 sec
RG            23100
DW            7.800 usec
DE            6.50 usec
TE            294.6 K
D1            2.00000000 sec
TD0           1

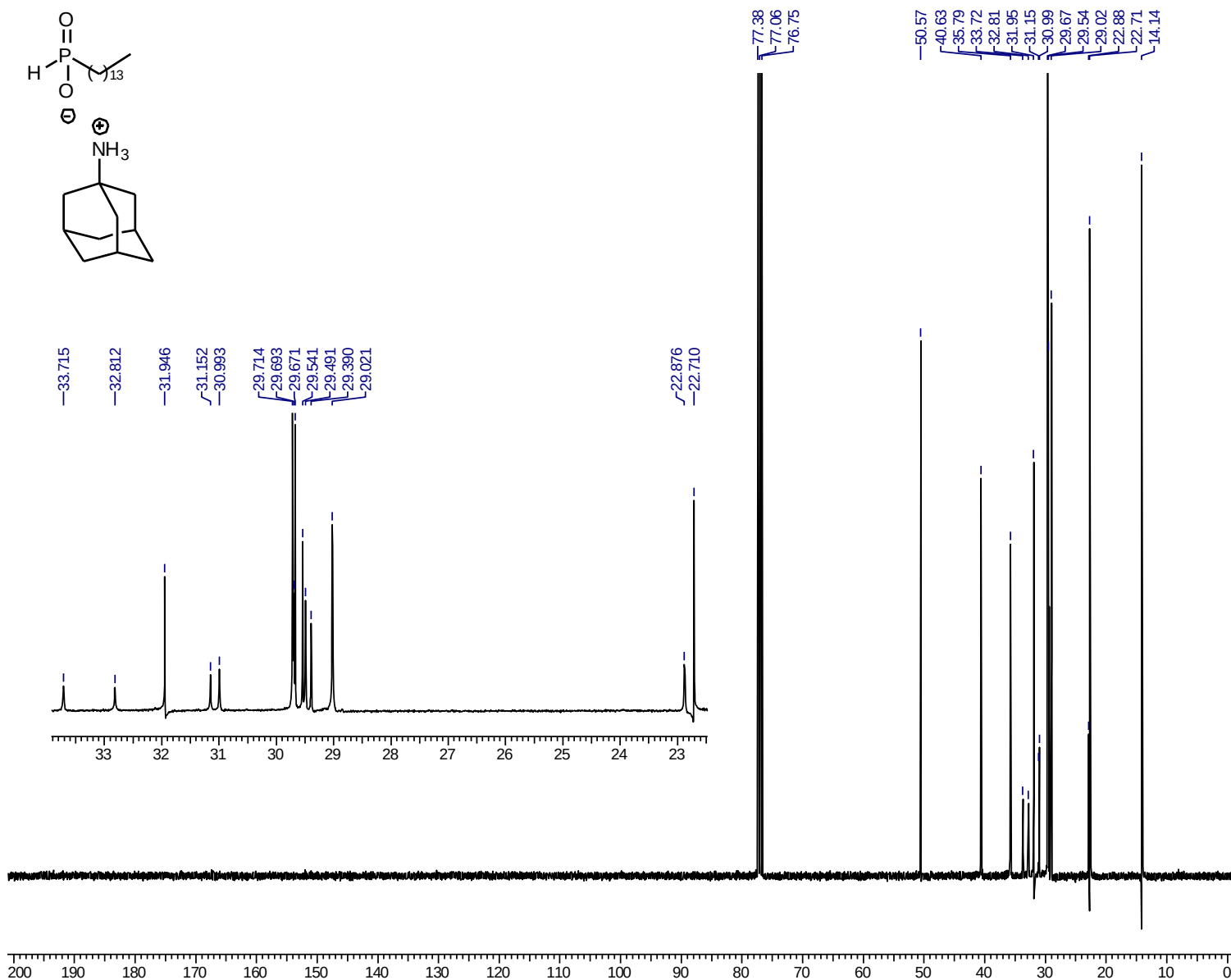
===== CHANNEL f1 =====
NUC1          31P
P1            9.40 usec
PL1           0.00 dB
PL1W          26.87718391 W
SF01          161.9674942 MHz
SI            32768
SF            161.9755930 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
    
```



¹H NMR spectrum of the adamantanamine derivative of 17 (400 MHz, CDCl₃)



¹³C NMR spectrum of the adamantanamine derivative of 17 (100 MHz, CDCl₃)

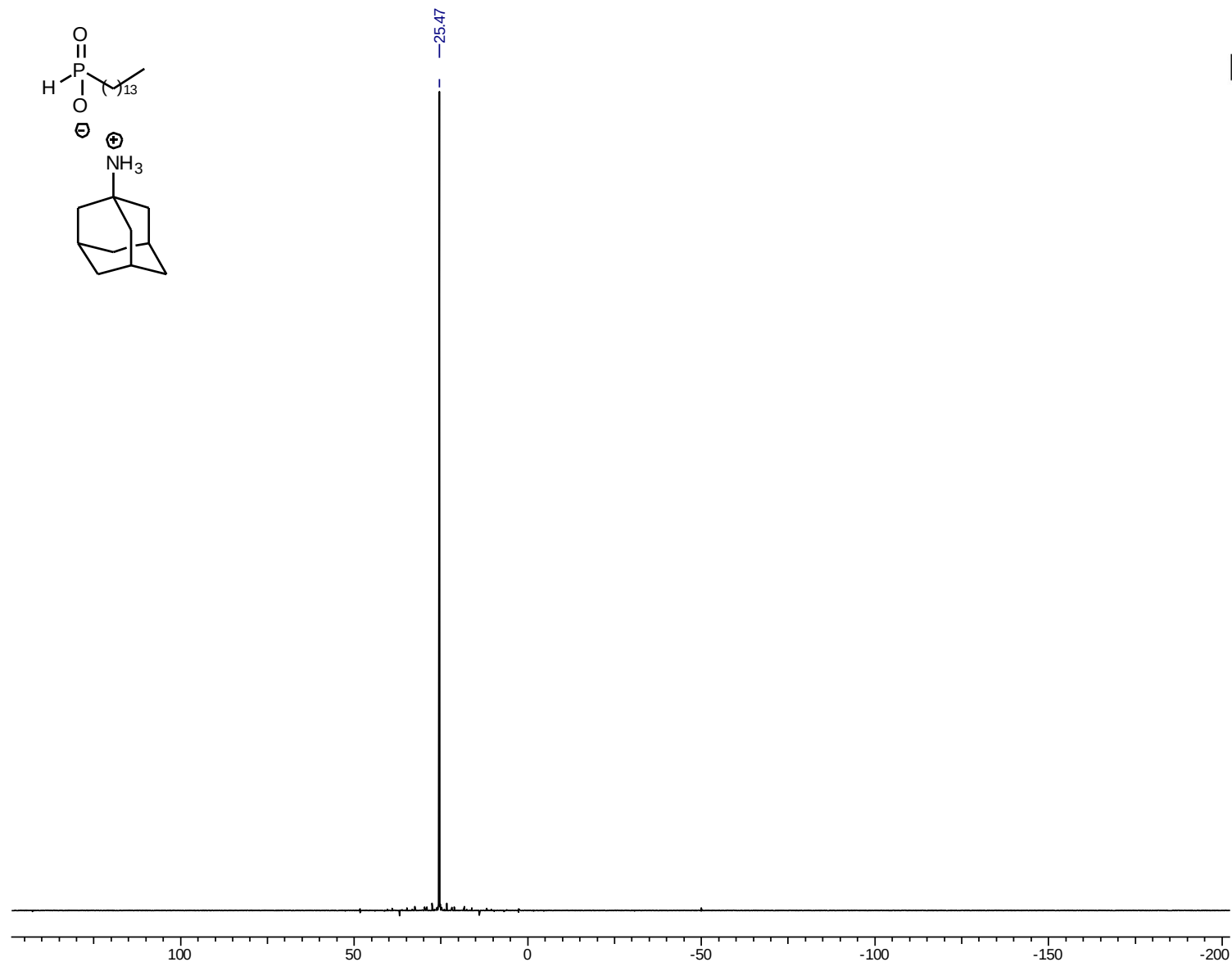
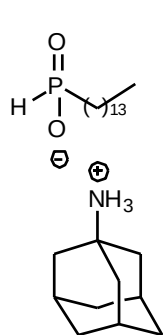


NAME acr-52
EXPNO 11
PROCNO 1
INSTRUM AV400
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl₃
NS 3072
DS 2
SWH 23809.523 Hz
FIDRES 0.363304 Hz
AQ 1.3763061 sec
RG 512
DW 21.000 usec
DE 7.66 usec
TE 295.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 8.00 usec
PL1 0.00 dB
PL1W 34.91522217 W
SFO1 100.6228294 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -3.60 dB
PL12 15.31 dB
PL13 18.00 dB
PL2W 17.83863831 W
PL12W 0.22927761 W
PL13W 0.12341322 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

³¹P (proton-decoupled) NMR spectrum of the adamantanamine derivative of 17 (162 MHz, CDCl₃)

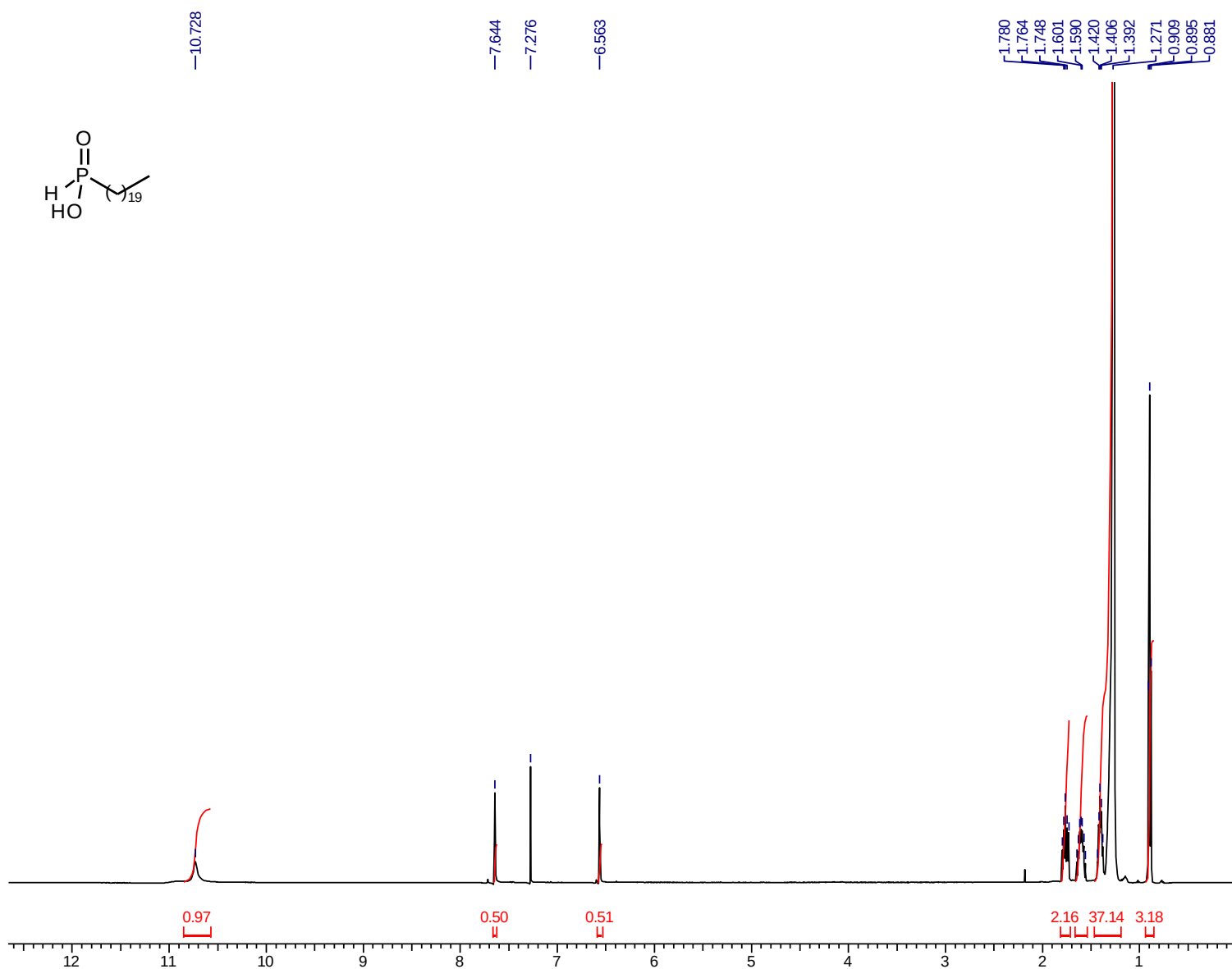


NAME	acr-52
EXPNO	16
PROCNO	1
INSTRUM	AV400
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1024
DS	4
SWH	64102.563 Hz
FIDRES	0.978127 Hz
AQ	0.5112308 sec
RG	23100
DW	7.800 usec
DE	6.50 usec
TE	294.9 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1

```
===== CHANNEL f1 =====
NUC1                31P
P1                  9.40 usec
PL1                 0.00 dB
PL1w                26.87718391 w
SF01                161.9674942 MHz
```

```
===== CHANNEL f2 =====
CPDPRG2      waltz16
NUC2          1H
PCPD2         90.00 usec
PL2           -3.60 dB
PL12          15.31 dB
PL13          18.00 dB
PL2W          17.83863831 W
PL12W         0.22927761 W
PL13W         0.12341322 W
SF02          400.1316005 MHz
SI            32768
SF            161.9755930 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
```

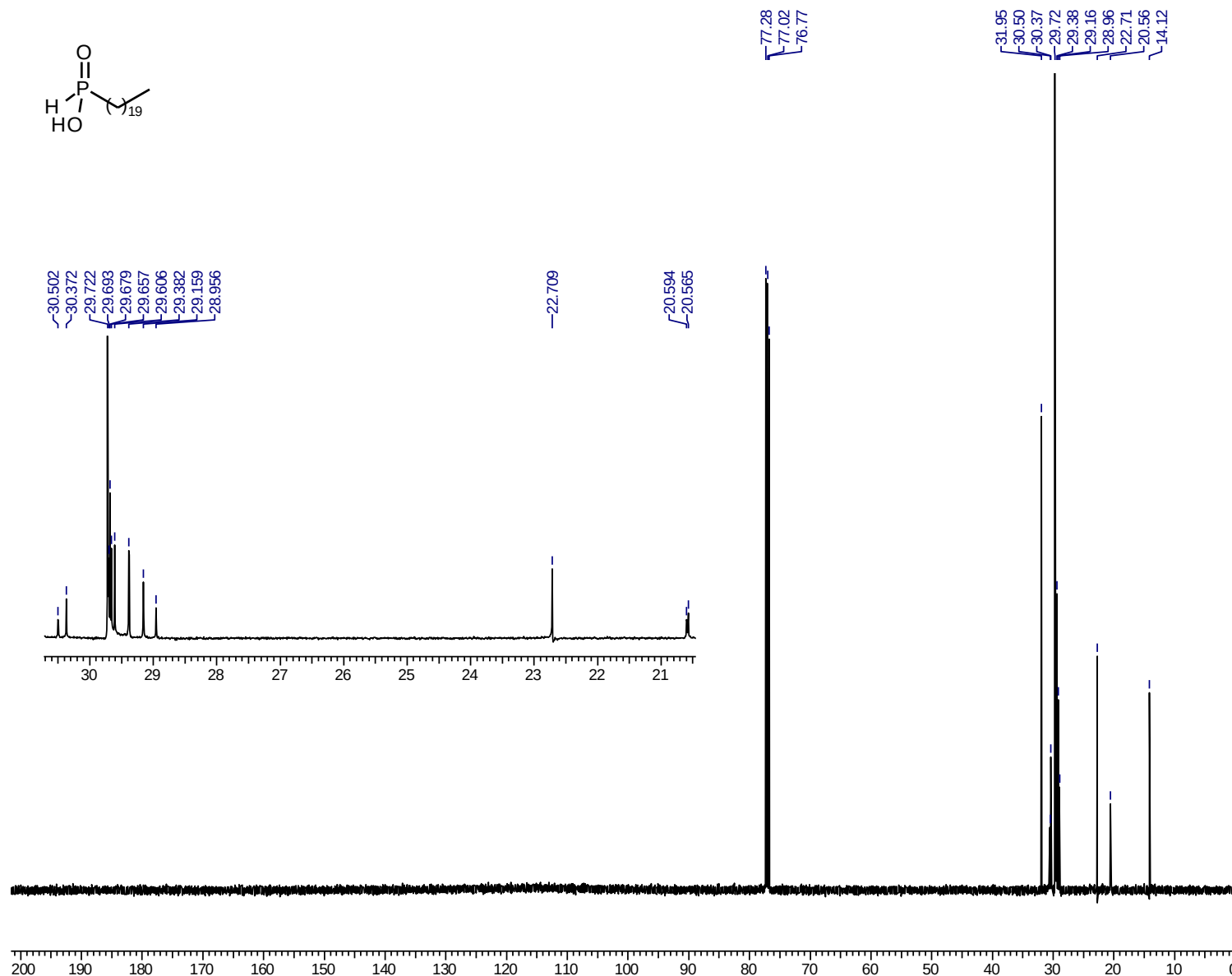
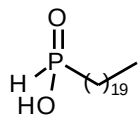
¹H NMR spectrum of 19 (500 MHz, CDCl₃)



```
NAME      acr-49
EXPNO     10
PROCNO    1
INSTRUM   spect
PROBHD    5 mm TXI 1H/D-
PULPROG   zg30b
TD        65536
SOLVENT   CDCl3
NS        64
DS        0
SWH       10330.578 Hz
FIDRES    0.157632 Hz
AQ        3.1719923 sec
RG        45.2
DW        48.400 usec
DE        13.46 usec
TE        300.0 K
D1        1.00000000 sec
TD0       1
```

```
===== CHANNEL f1 =====
NUC1      1H
P1        7.80 usec
PL1       3.25 dB
PL1W      12.12272263 W
SF01      500.1330885 MHz
SI        32768
SF        500.1300000 MHz
WDW       EM
SSB       0
LB        0.30 Hz
GB        0
PC        1.00
```

¹³C NMR spectrum of 19 (125 MHz, CDCl₃)



```

NAME          acr-49
EXPNO         11
PROCNO        1
INSTRUM       spect
PROBHD        5 mm TXI 1H/D-
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            2048
DS            2
SWH           29761.904 Hz
FIDRES        0.454131 Hz
AQ            1.1010548 sec
RG            512
DW            16.800 usec
DE            32.63 usec
TE            300.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0           1
  
```

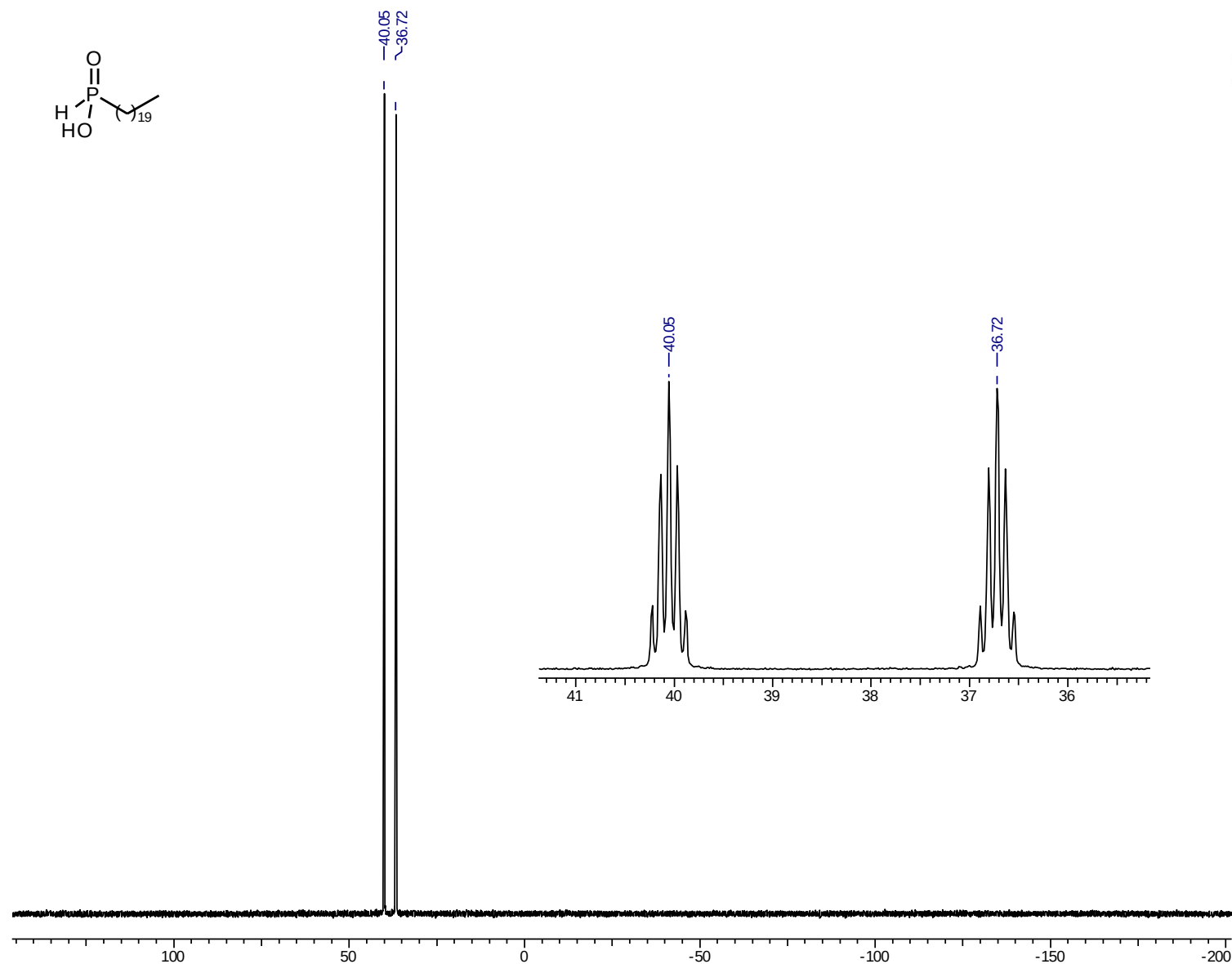
```

===== CHANNEL f1 =====
NUC1          13C
P1            9.50 usec
PL1           -4.20 dB
PL1W          218.02882385 W
SF01          125.7703643 MHz
  
```

```

===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2          1H
PCPD2         80.00 usec
PL2           4.20 dB
PL12          23.47 dB
PL13          23.00 dB
PL2W          9.74092484 W
PL12W         0.11523920 W
PL13W         0.12841040 W
SF02          500.1320005 MHz
SI            32768
SF            125.7577890 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
  
```

^{31}P (proton-coupled) NMR spectrum of 19 (162 MHz, CDCl_3)



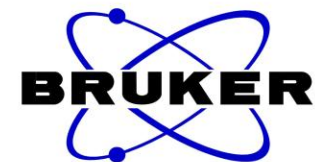
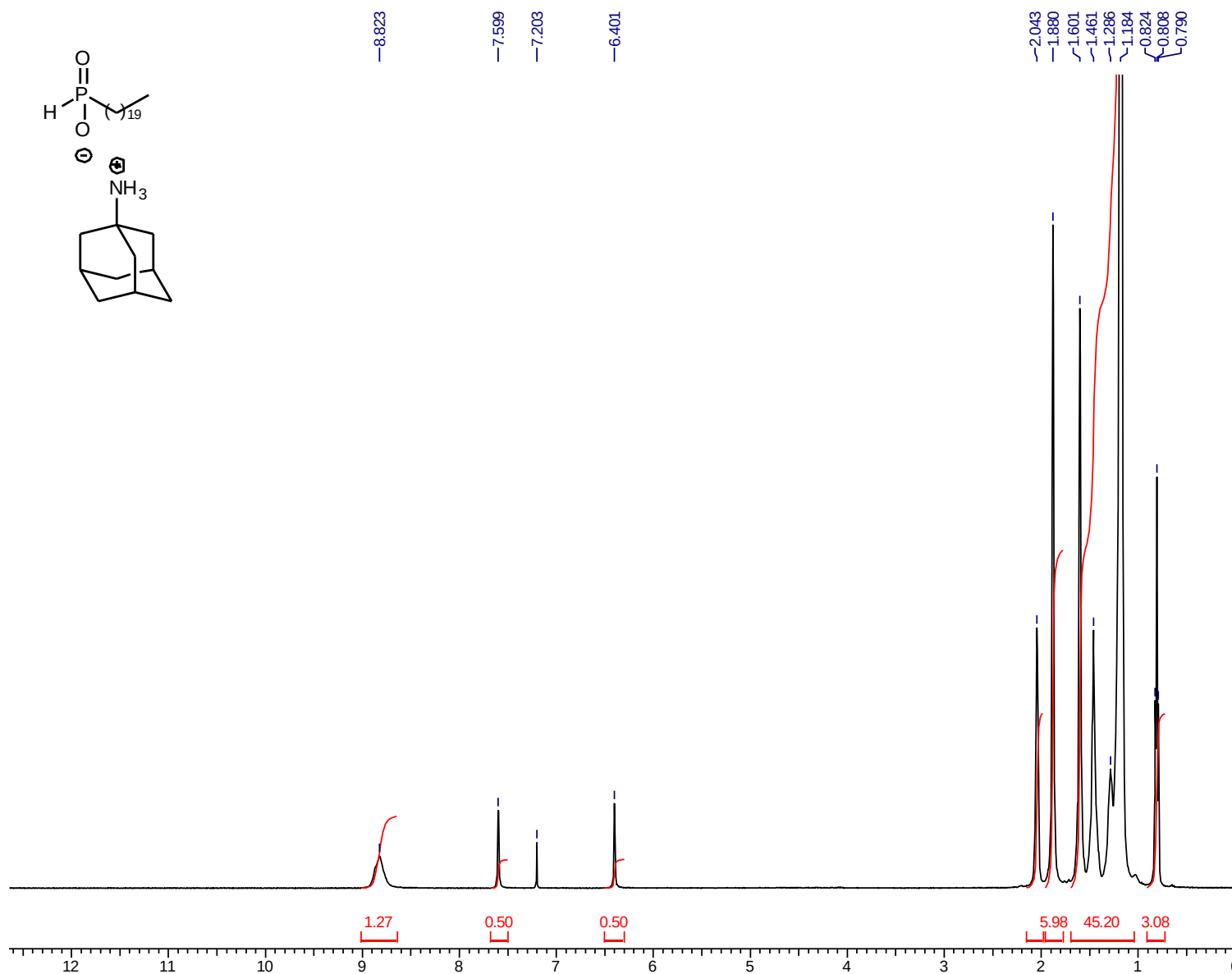
```

NAME          acr-26
EXPNO         11
PROCNO        1
INSTRUM       AV400
PROBHD        5 mm PABBO BB-
PULPROG       zg30
TD            65536
SOLVENT       CDCl3
NS            1024
DS            4
SWH           64102.563 Hz
FIDRES        0.978127 Hz
AQ            0.5112308 sec
RG            23100
DW            7.800 usec
DE            6.50 usec
TE            292.8 K
D1            2.00000000 sec
TD0           1
  
```

```

===== CHANNEL f1 =====
NUC1          31P
P1            9.40 usec
PL1           0.00 dB
PL1W          26.87718391 W
SF01          161.9674942 MHz
SI            32768
SF            161.9755930 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
  
```

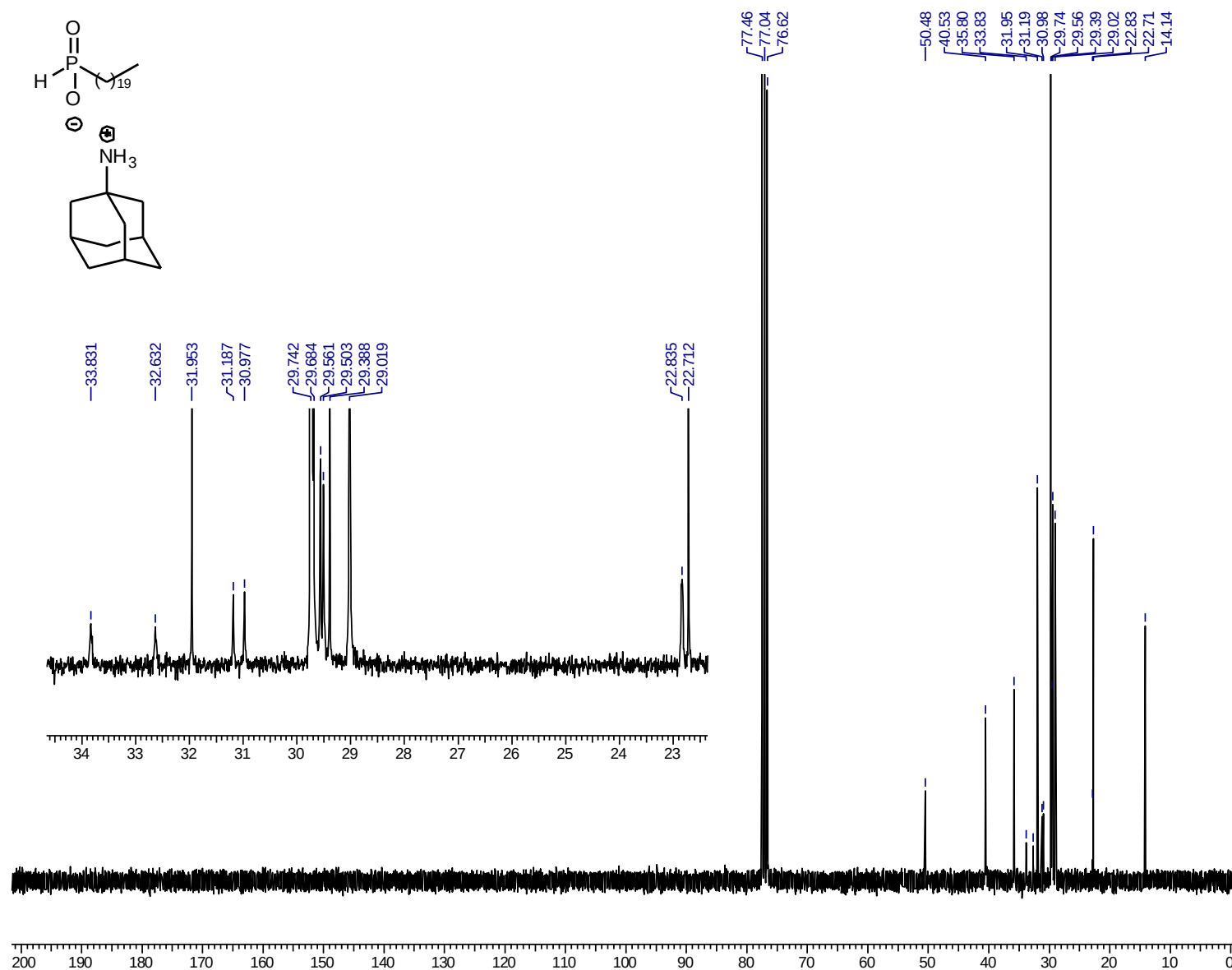
¹H NMR spectrum of the adamantanamine derivative of 19 (400 MHz, CDCl₃)



NAME acr-60
EXPNO 10
PROCNO 1
INSTRUM AV400
PROBHD 5 mm PABBO BB-
PULPROG zg30b
TD 65536
SOLVENT CDCl₃
NS 32
DS 0
SWH 8264.463 Hz
FIDRES 0.126106 Hz
AQ 3.9649780 sec
RG 45.2
DW 60.500 usec
DE 9.40 usec
TE 294.2 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 10.00 usec
PL1 -3.60 dB
PL1W 17.83863831 W
SF01 400.1324710 MHz
SI 32768
SF 400.1300338 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

¹³C NMR spectrum of the adamantanamine derivative of 19 (75 MHz, CDCl₃)



NAME acr-33
EXPNO 11
PROCNO 1

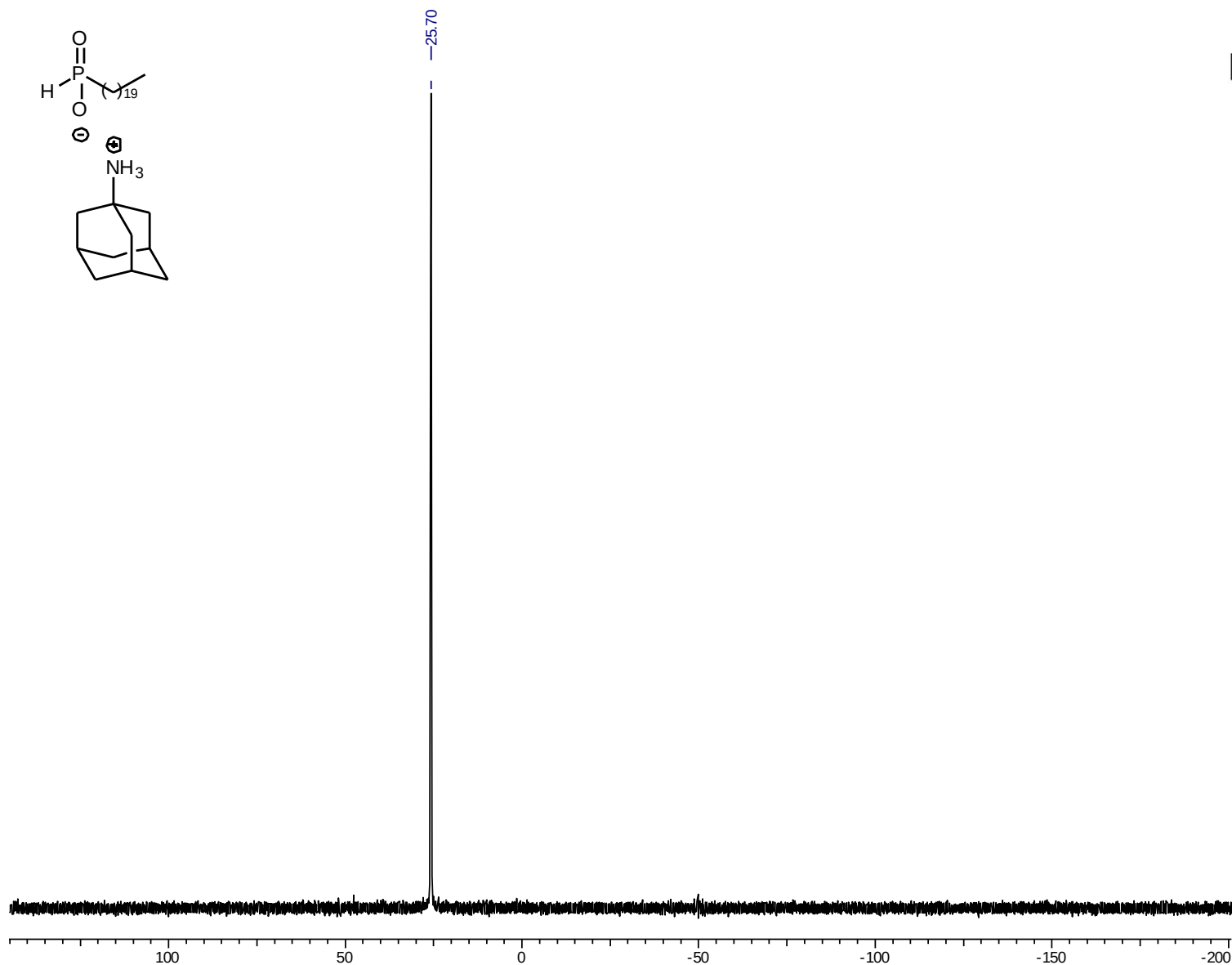
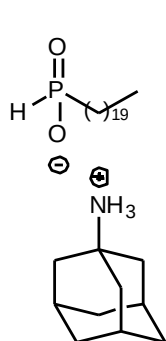
F2 - Acquisition Parameters
INSTRUM DPX300
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 2048
DS 2
SWH 17857.143 Hz
FIDRES 0.272478 Hz
AQ 1.8350580 sec
RG 9195.2
DW 28.000 usec
DE 6.00 usec
TE 333.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 10.00 usec
PL1 -1.20 dB
SF01 75.4300300 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.60 dB
PL12 14.50 dB
PL13 15.00 dB
SF02 299.9512000 MHz

F2 - Processing parameters
SI 32768
SF 75.4224870 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

³¹P (proton-decoupled) NMR spectrum of the adamantanamine derivative of 19 (162 MHz, CDCl₃)



NAME	acr-60
EXPNO	11
PROCNO	1
INSTRUM	AV400
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDC13
NS	128
DS	4
SWH	64102.563 Hz
FIDRES	0.978127 Hz
AQ	0.5112308 sec
RG	23100
DW	7.800 usec
DE	6.50 usec
TE	294.8 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1

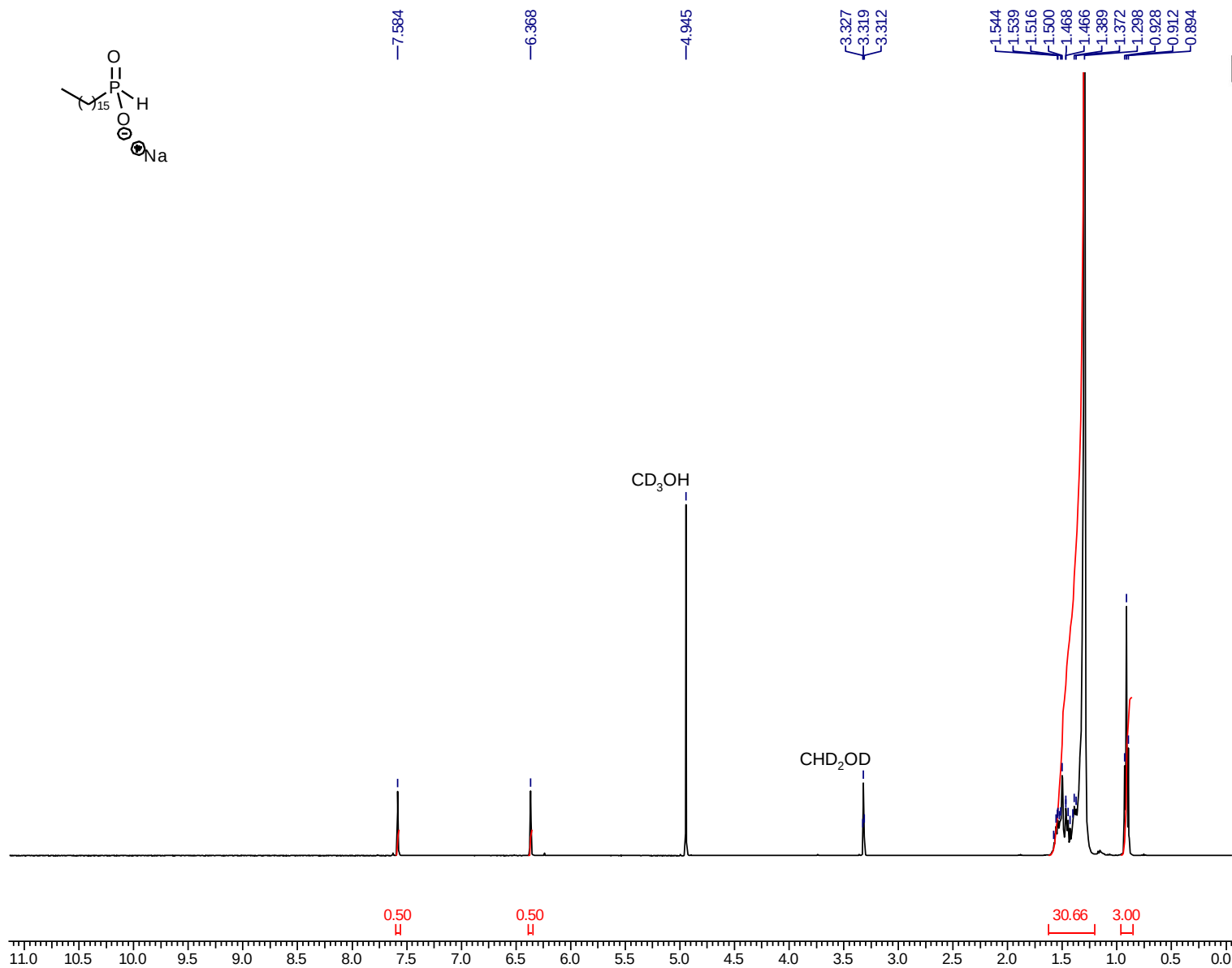
```
===== CHANNEL f1 =====
NUC1          31P
P1             9.40 usec
PL1           0.00 dB
PL1W          26.87718391 W
SF01          161.9674942 MHz
```

```

===== CHANNEL f2 =====
CPDPRG2          waltz16
NUC2              1H
PCPD2            90.00 usec
PL2              -3.60 dB
PL12             15.31 dB
PL13             18.00 dB
PL2W             17.83863831 W
PL12W            0.22927761 W
PL13W            0.12341322 W
SFO2             400.1316005 MHz
SI               32768
SF               161.9755930 MHz
WDW              EM
SSB              0
LB               1.00 Hz
GB               0
PC               1.40

```

¹H NMR spectrum of 20 (400 MHz, CD₃OD)



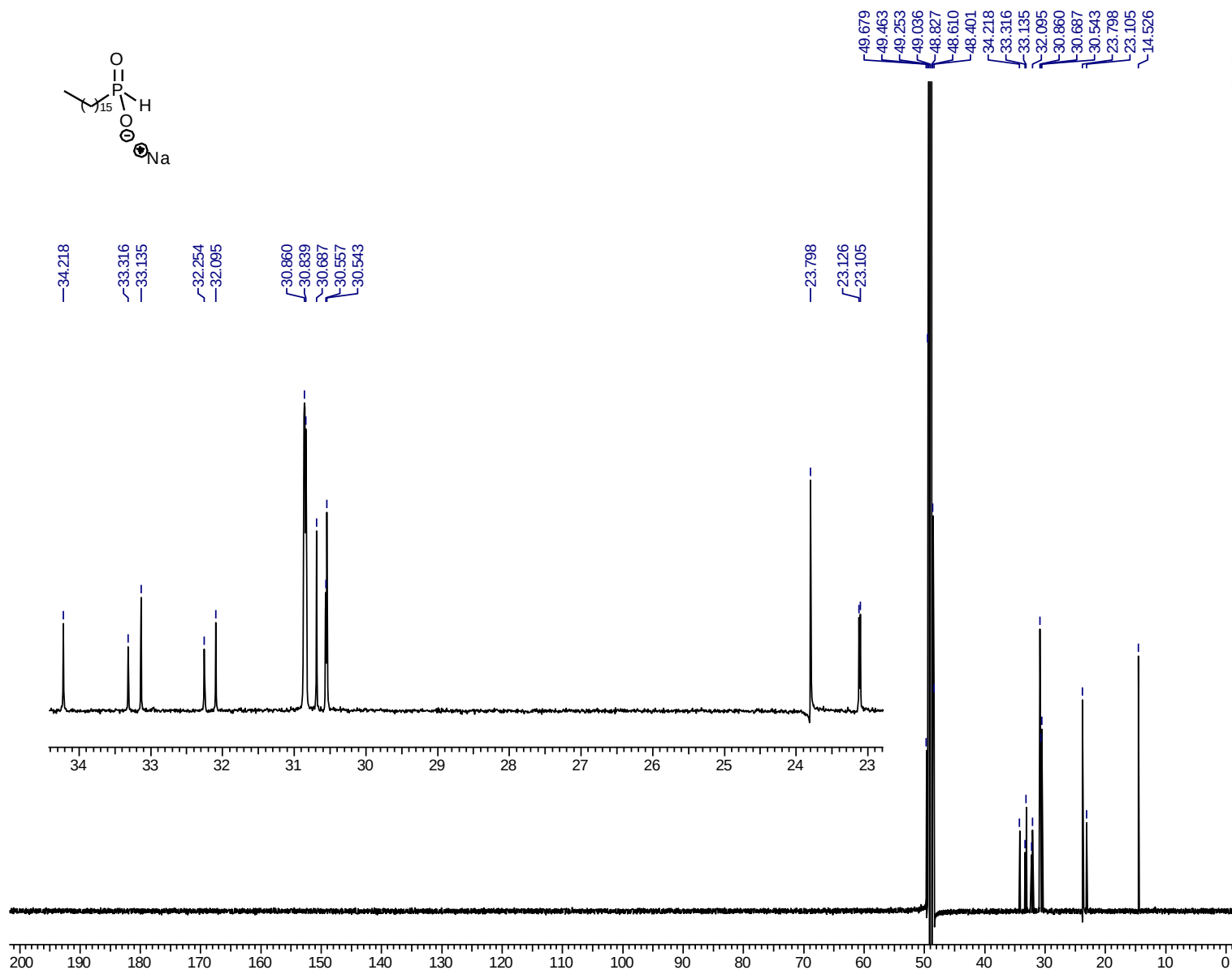
```

NAME          acr-60
EXPNO         10
PROCNO        1
INSTRUM       AV400
PROBHD        5 mm PABBO BB-
PULPROG       zg30b
TD            65536
SOLVENT       MeOD
NS            128
DS            0
SWH           8264.463 Hz
FIDRES        0.126106 Hz
AQ            3.9649780 sec
RG            90.5
DW            60.500 usec
DE            9.40 usec
TE            293.4 K
D1            1.00000000 sec
TD0           1
    
```

```

===== CHANNEL f1 =====
NUC1          1H
P1            10.00 usec
PL1           -3.60 dB
PL1W          17.83863831 W
SF01          400.1324710 MHz
SI            32768
SF            400.1300000 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
    
```

¹³C NMR spectrum of 20 (100 MHz, CD₃OD)

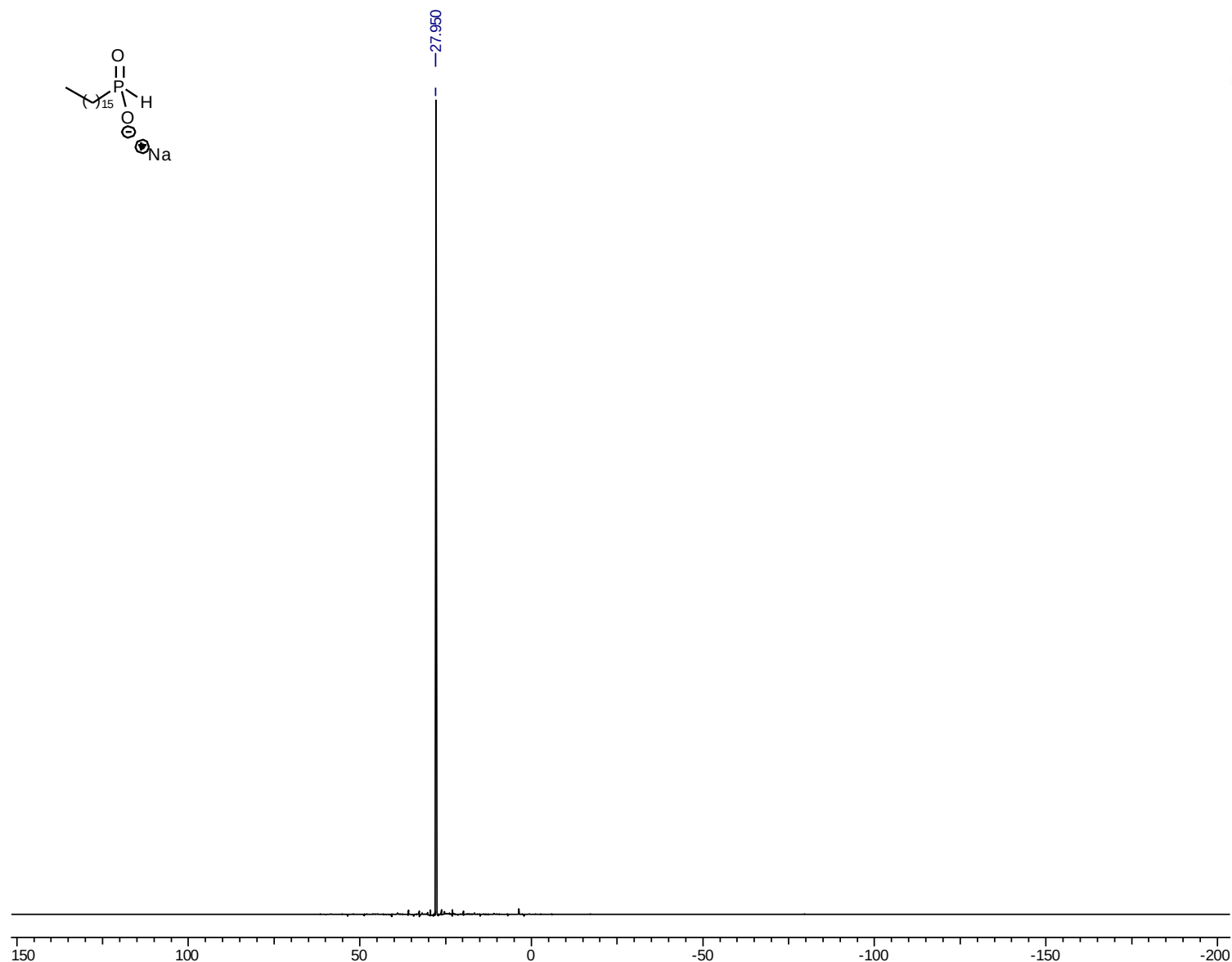


NAME acr-60
EXPNO 11
PROCNO 1
INSTRUM AV400
PROBHD 5 mm PABBO B8-
PULPROG zgpg30
TD 65536
SOLVENT MeOD
NS 2048
DS 2
SWH 23809.523 Hz
FIDRES 0.363304 Hz
AQ 1.3763061 sec
RG 512
DW 21.000 usec
DE 7.66 usec
TE 294.8 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 8.00 usec
PL1 0.00 dB
PL1W 34.91522217 W
SFO1 100.6228294 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -3.60 dB
PL12 13.80 dB
PL13 14.00 dB
PL2W 17.83863831 W
PL12W 0.32460985 W
PL13W 0.31000000 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6126261 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

^{31}P (proton-decoupled) NMR spectrum of 20 (162 MHz, CD_3OD)

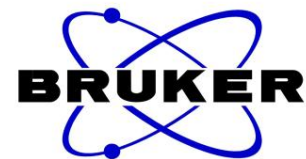
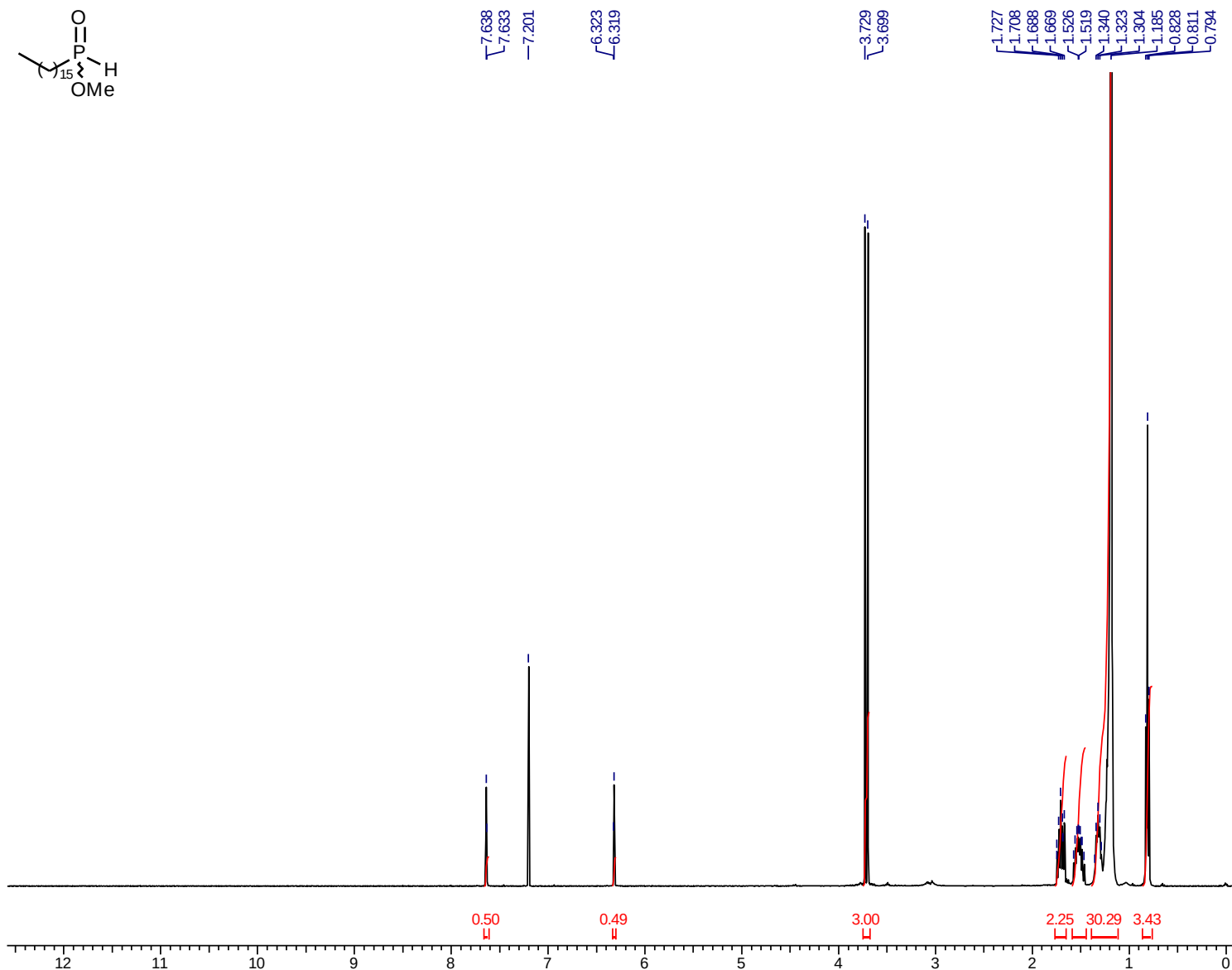


```
NAME          acr-60
EXPNO          15
PROCNO         1
INSTRUM        AV400
PROBHD         5 mm PABBO BB-
PULPROG        zgpg30
TD             65536
SOLVENT        MeOD
NS             1024
DS             4
SWH            64102.563 Hz
FIDRES         0.978127 Hz
AQ             0.5112308 sec
RG             23100
DW             7.800 usec
DE             6.50 usec
TE             294.4 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0            1
```

```
===== CHANNEL f1 =====
NUC1           31P
P1             9.40 usec
PL1            0.00 dB
PL1W           26.87718391 W
SF01           161.9674942 MHz
```

```
===== CHANNEL f2 =====
CPDPRG2        waltz16
NUC2           1H
PCPD2          80.00 usec
PL2            -3.60 dB
PL12           13.80 dB
PL13           14.00 dB
PL2W           17.83863831 W
PL12W          0.32460985 W
PL13W          0.31000000 W
SF02           400.1316005 MHz
SI             32768
SF             161.9755930 MHz
WDW            EM
SSB            0
LB             1.00 Hz
GB             0
PC             1.40
```

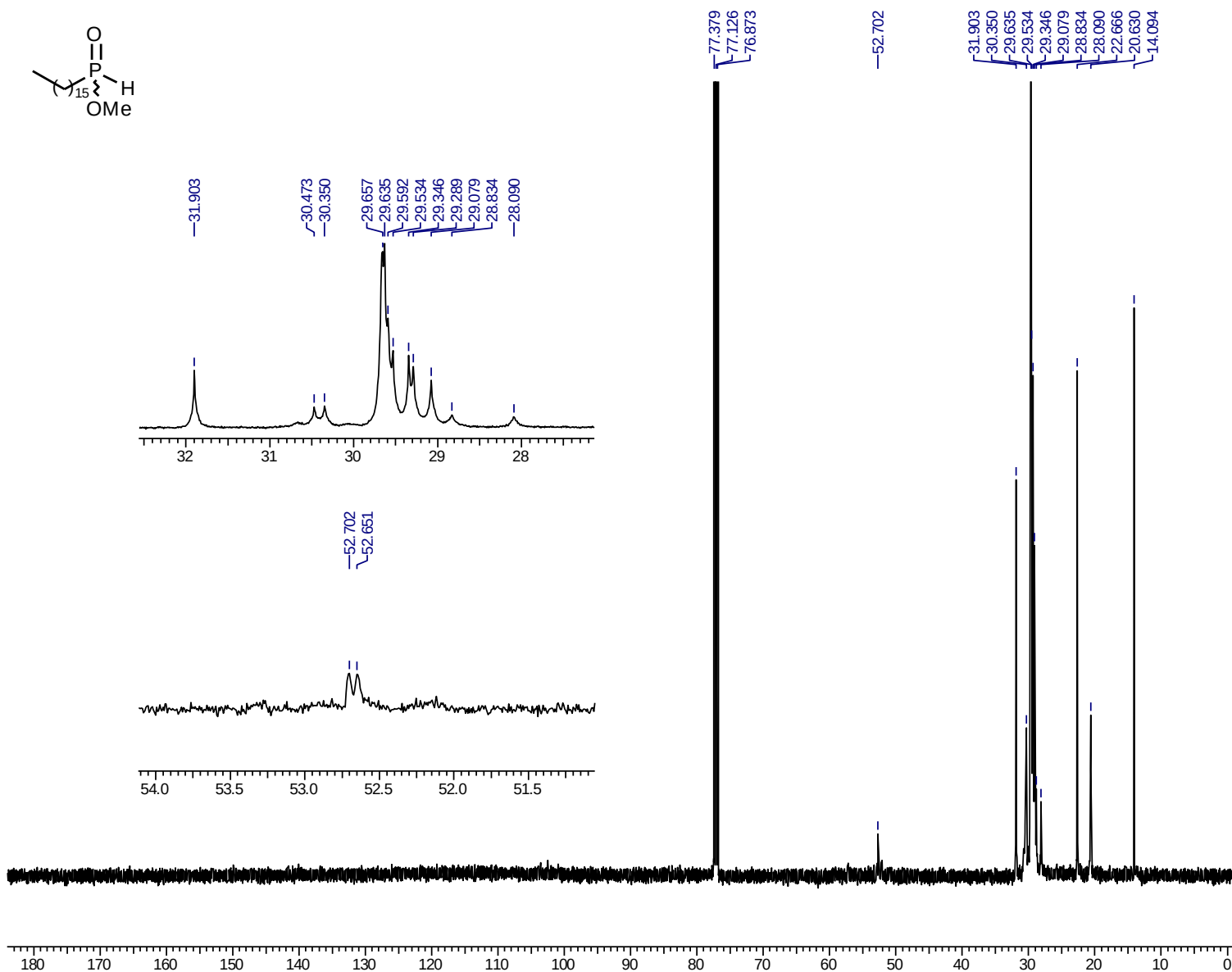
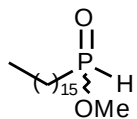
¹H NMR spectrum of 5 (400 MHz, CDCl₃)



NAME acr-6
EXPNO 10
PROCNO 1
INSTRUM AV400
PROBHD 5 mm PABBO BB-
PULPROG zg30b
TD 65536
SOLVENT CDCl3
NS 32
DS 0
SWH 8264.463 Hz
FIDRES 0.126106 Hz
AQ 3.9649780 sec
RG 128
DW 60.500 usec
DE 9.40 usec
TE 294.4 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 10.00 usec
PL1 -3.60 dB
PL1W 17.83863831 W
SF01 400.1324710 MHz
SI 32768
SF 400.1300293 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

¹³C NMR spectrum of 5 (125 MHz, CDCl₃)



```

NAME          acr-58
EXPNO         12
PROCNO        1
INSTRUM       spect
PROBHD        5 mm TXI 1H/D-
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            2048
DS            2
SWH           29761.904 Hz
FIDRES        0.454131 Hz
AQ            1.1010548 sec
RG            512
DW            16.800 usec
DE            32.21 usec
TE            291.4 K
D1            2.0000000 sec
D11           0.0300000 sec
TD0           1
  
```

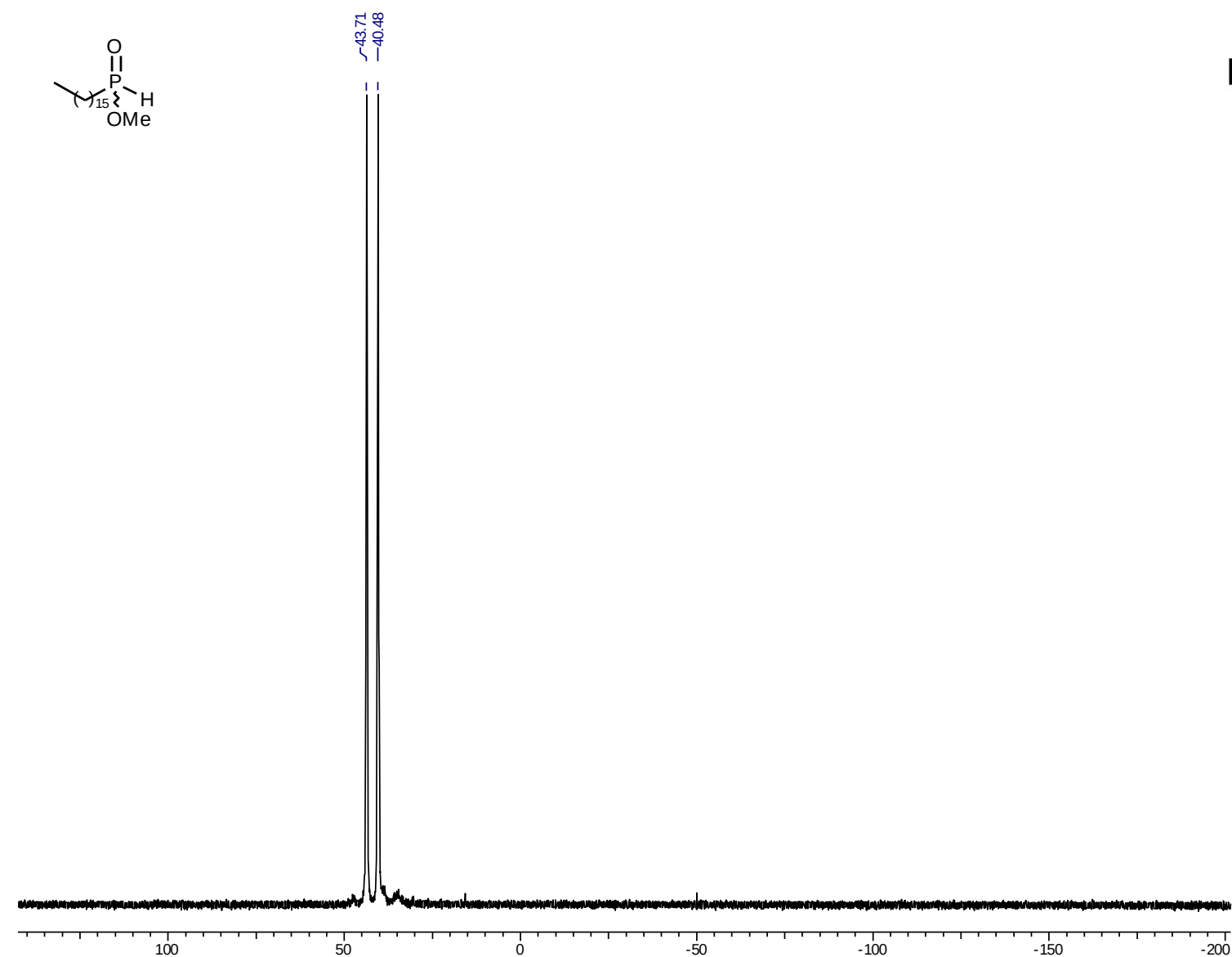
```

===== CHANNEL f1 =====
NUC1          13C
P1            11.50 usec
PL1           -4.20 dB
PL1W          218.02882385 W
SF01          125.7703643 MHz
  
```

```

===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2          1H
PCPD2         80.00 usec
PL2           4.20 dB
PL12          24.42 dB
PL13          23.00 dB
PL2W          9.74092484 W
PL12W         0.09259769 W
PL13W         0.12841040 W
SF02          500.1320005 MHz
SI            32768
SF            125.7577890 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
  
```

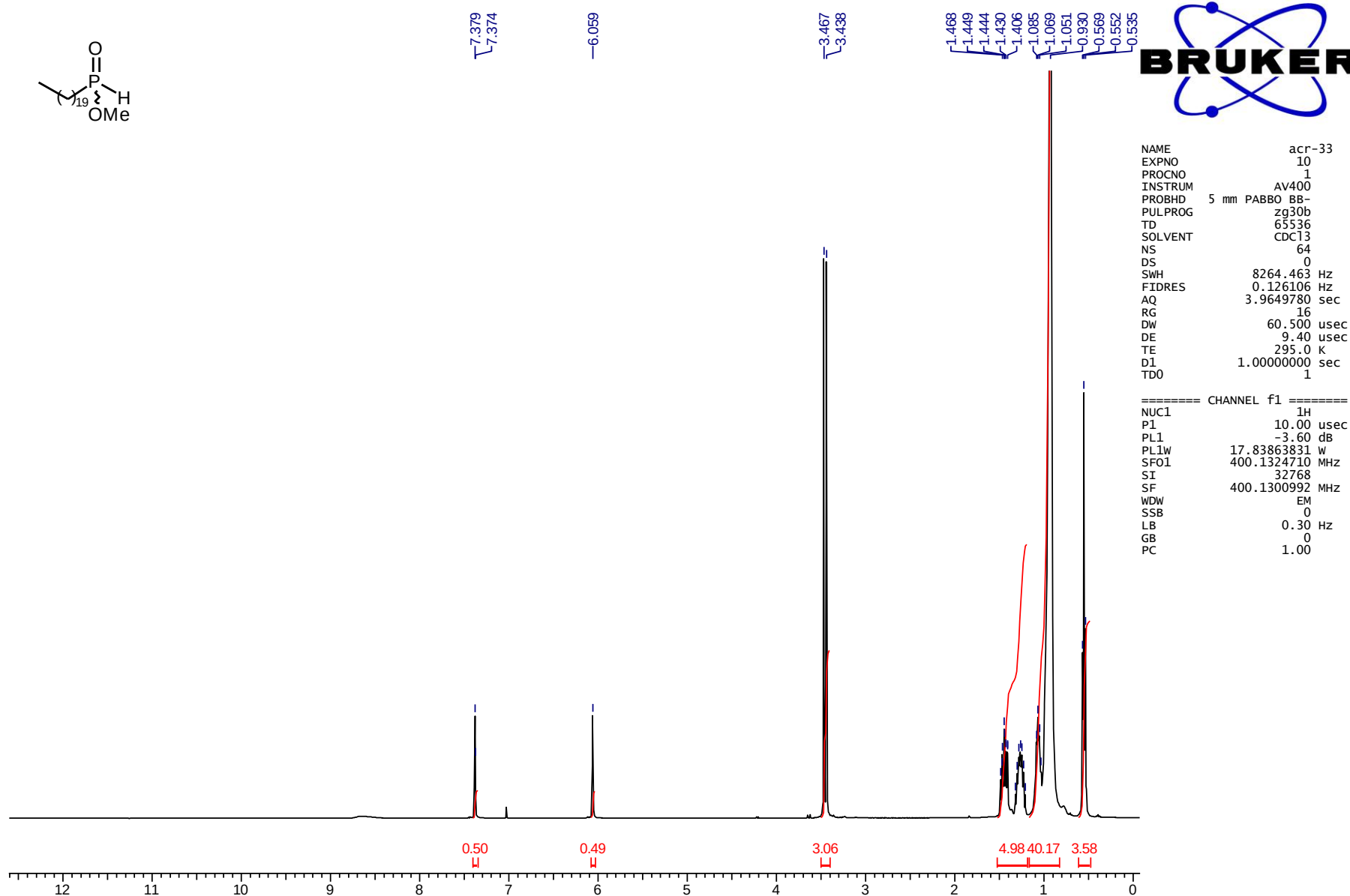
³¹P (proton-coupled) NMR spectrum of 5 (162 MHz, CDCl₃)



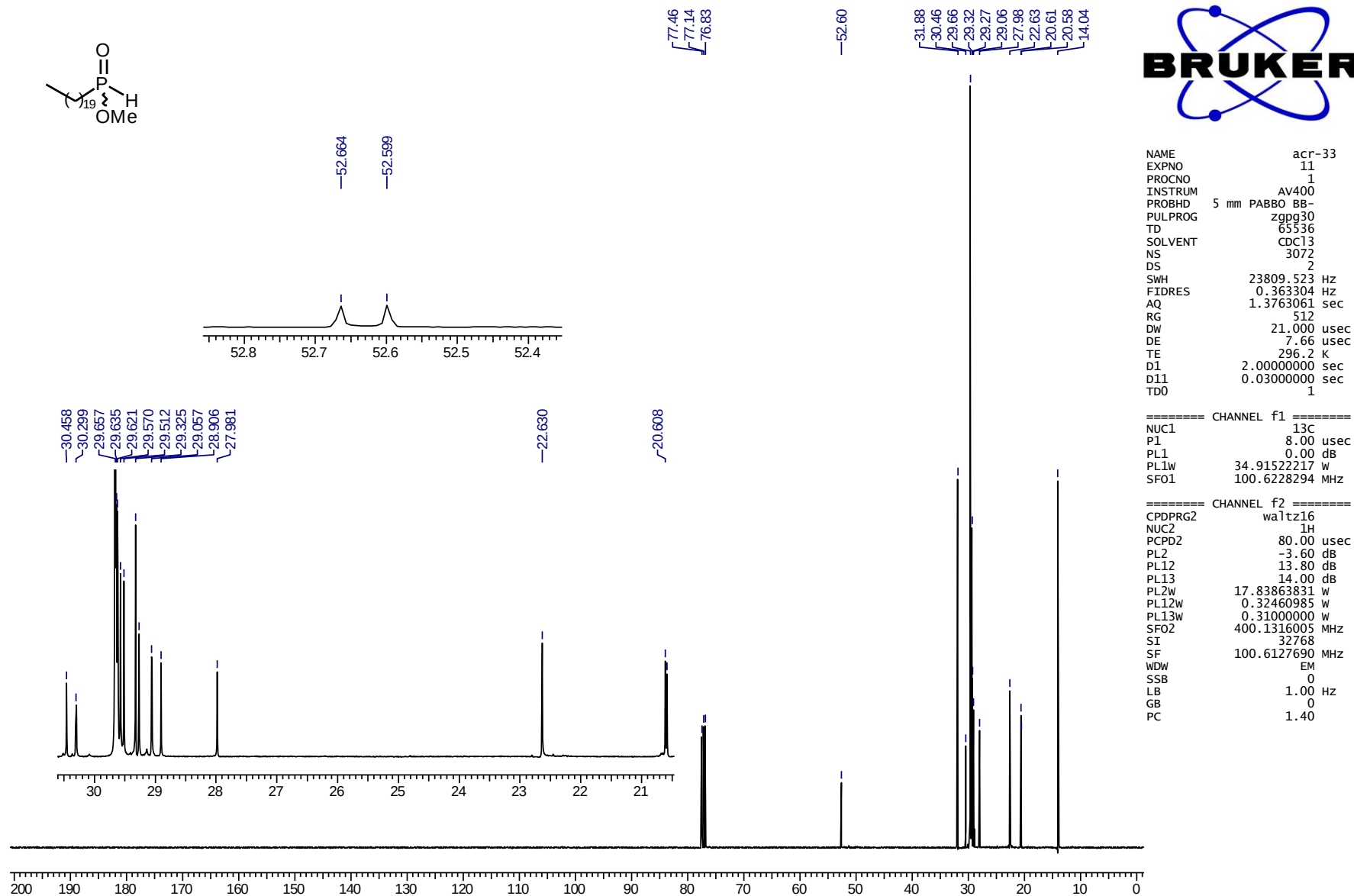
NAME acr-55
EXPNO 11
PROCNO 1
INSTRUM AV400
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl₃
NS 1024
DS 4
SWH 64102.563 Hz
FIDRES 0.978127 Hz
AQ 0.5112308 sec
RG 23100
DW 7.800 usec
DE 6.50 usec
TE 294.9 K
D1 2.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 31P
P1 9.40 usec
PL1 0.00 dB
PL1w 26.87718391 w
SF01 161.9674942 MHz
SI 32768
SF 161.9755930 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

¹H NMR spectrum of 21 (400 MHz, CDCl₃)



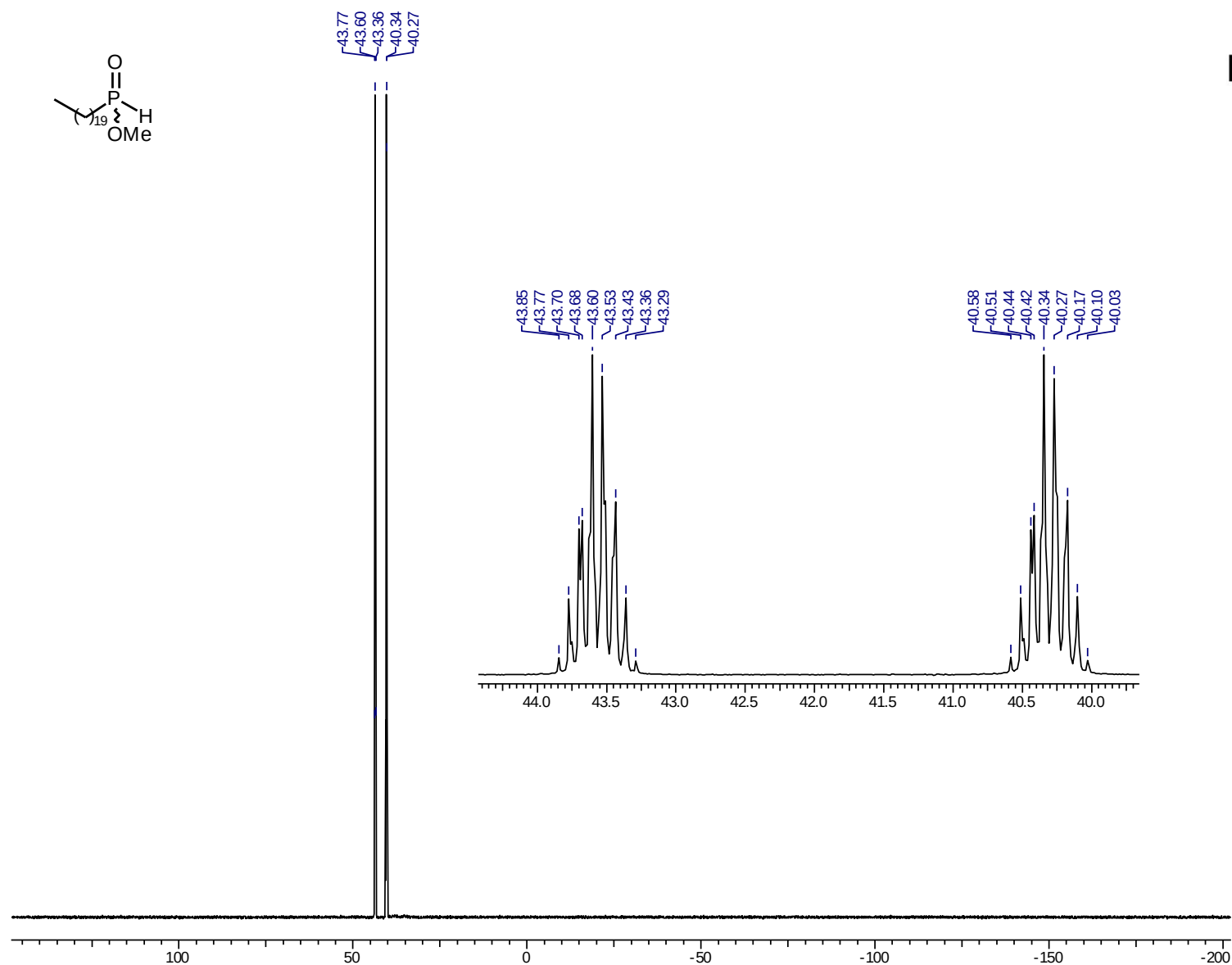
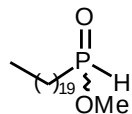
¹³C NMR spectrum of 21 (100 MHz, CDCl₃)



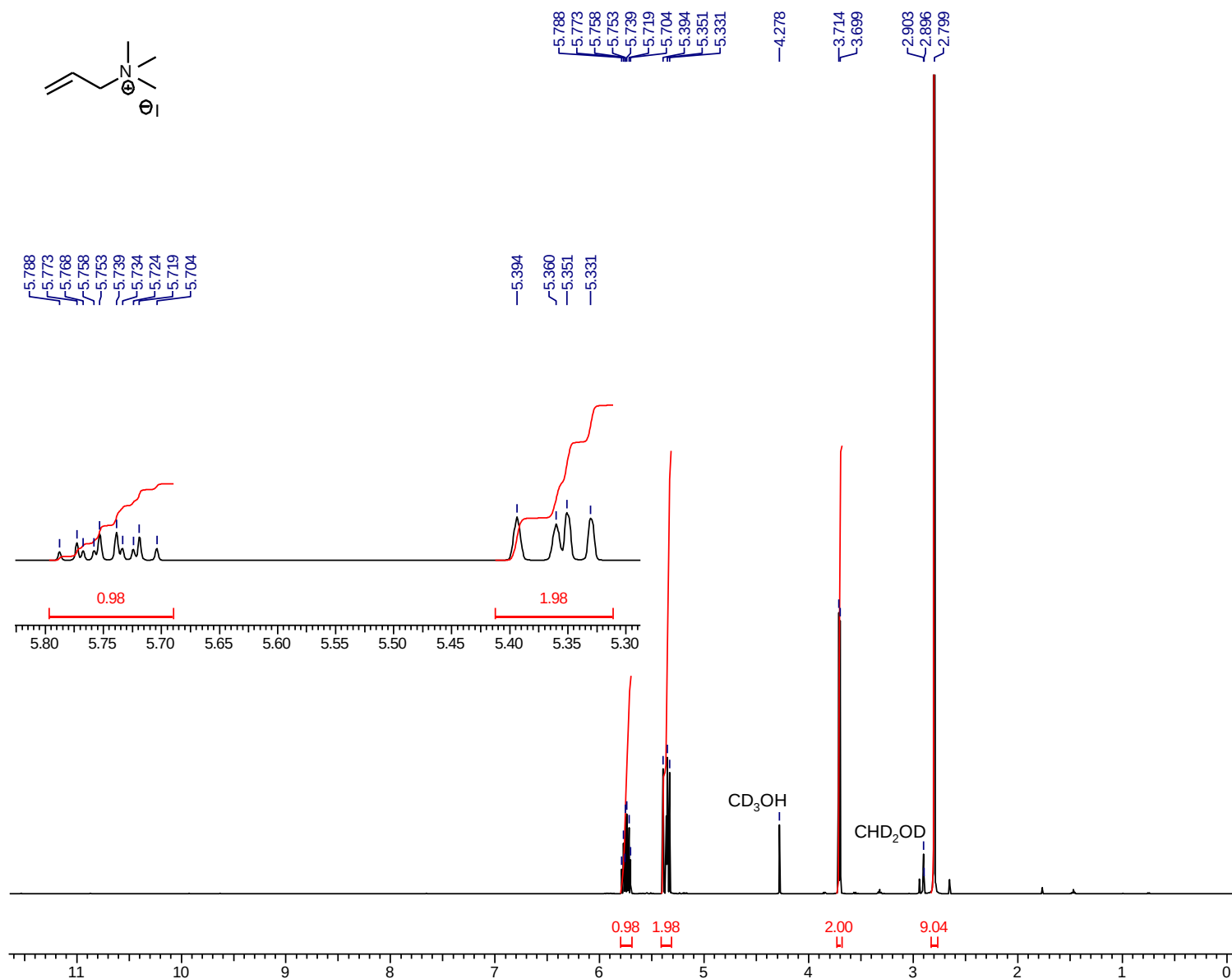
```
NAME                                acr-33
EXPNO                               16
PROCNO                              1
INSTRUM                             AV400
PROBHD      5 mm PABBO BB-
PULPRG          zg30
TD              65536
SOLVENT         CDC13
NS              512
DS               4
SWH            64102.563 Hz
FIDRES        0.978127 Hz
AQ           0.5112308 sec
RG             23100
DW             7.800    usec
DE             6.50    usec
TE             294.6 K
D1           2.00000000 sec
TD0            1
```

===== CHANNEL f1 =====

```
NUC1                31P
P1                  9.40 usec
PL1                 0.00 dB
PL1W       26.87718391 W
SF01     161.9674942 MHz
SI                   32768
SF       161.9755930 MHz
WDW                      EM
SSB                       0
LB                    1.00 Hz
GB                       0
PC                     1.40
```



¹H NMR spectrum of 23 (500 MHz, CD₃OD)



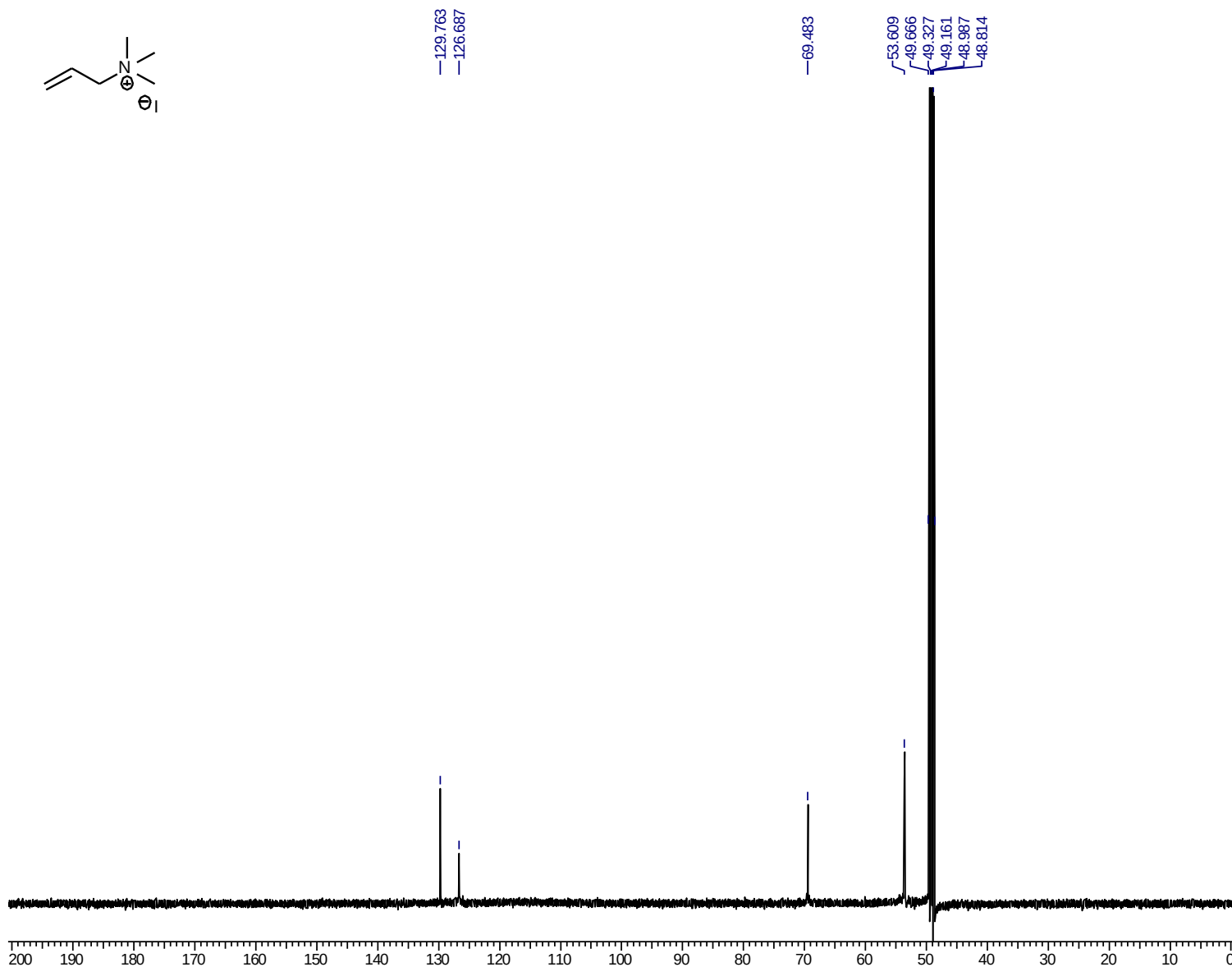
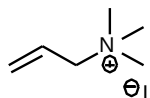
```

NAME          acr-42
EXPNO         10
PROCNO        1
INSTRUM       spect
PROBHD        5 mm TXI
PULPROG       zg30b
TD            65536
SOLVENT       MeOD
NS            64
DS            0
SWH           10330.578 Hz
FIDRES        0.157632 Hz
AQ            3.1719923 sec
RG            161
DW            48.400 usec
DE            13.22 usec
TE            300.0 K
D1            1.00000000 sec
TD0           1
  
```

```

===== CHANNEL f1 =====
NUC1           1H
P1             37.76 usec
PL1            3.25 dB
PL1w           12.12272263 w
SFO1           500.1330885 MHz
SI             32768
SF             500.1302123 MHz
WDW            EM
SSB            0
LB             0.30 Hz
GB             0
PC             1.00
  
```

¹³C NMR spectrum of 23 (125 MHz, CD₃OD)



```

NAME          acr-42
EXPNO         12
PROCNO        1
INSTRUM       spect
PROBHD        5 mm TXI 1H/D-
PULPROG       zgpg30
TD            65536
SOLVENT       MeOD
NS            1024
DS            2
SWH           29761.904 Hz
FIDRES        0.454131 Hz
AQ            1.1010548 sec
RG            512
DW            16.800 usec
DE            32.63 usec
TE            300.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0           1
  
```

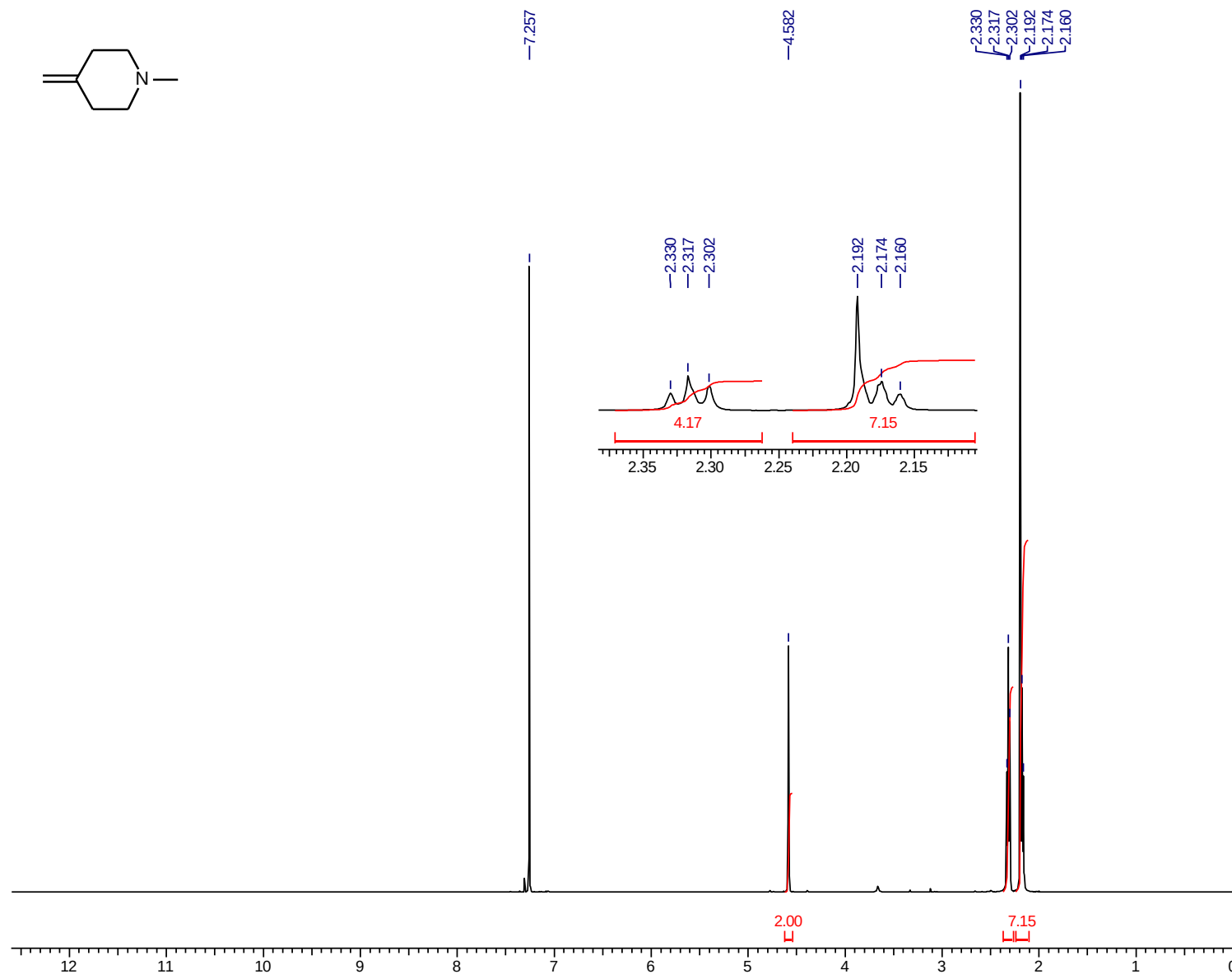
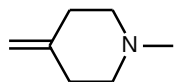
```

===== CHANNEL f1 =====
NUC1          13C
P1            9.50 usec
PL1           -4.20 dB
PL1W          218.02882385 w
SF01          125.7703643 MHz
  
```

```

===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2          1H
PCPD2         80.00 usec
PL2           4.20 dB
PL12          10.67 dB
PL13          23.00 dB
PL2W          9.74092484 w
PL12W         2.19583726 w
PL13W         0.12841040 w
SF02          500.1320005 MHz
SI            32768
SF            125.7576104 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
  
```

¹H NMR spectrum of 6 (400 MHz, CDCl₃)



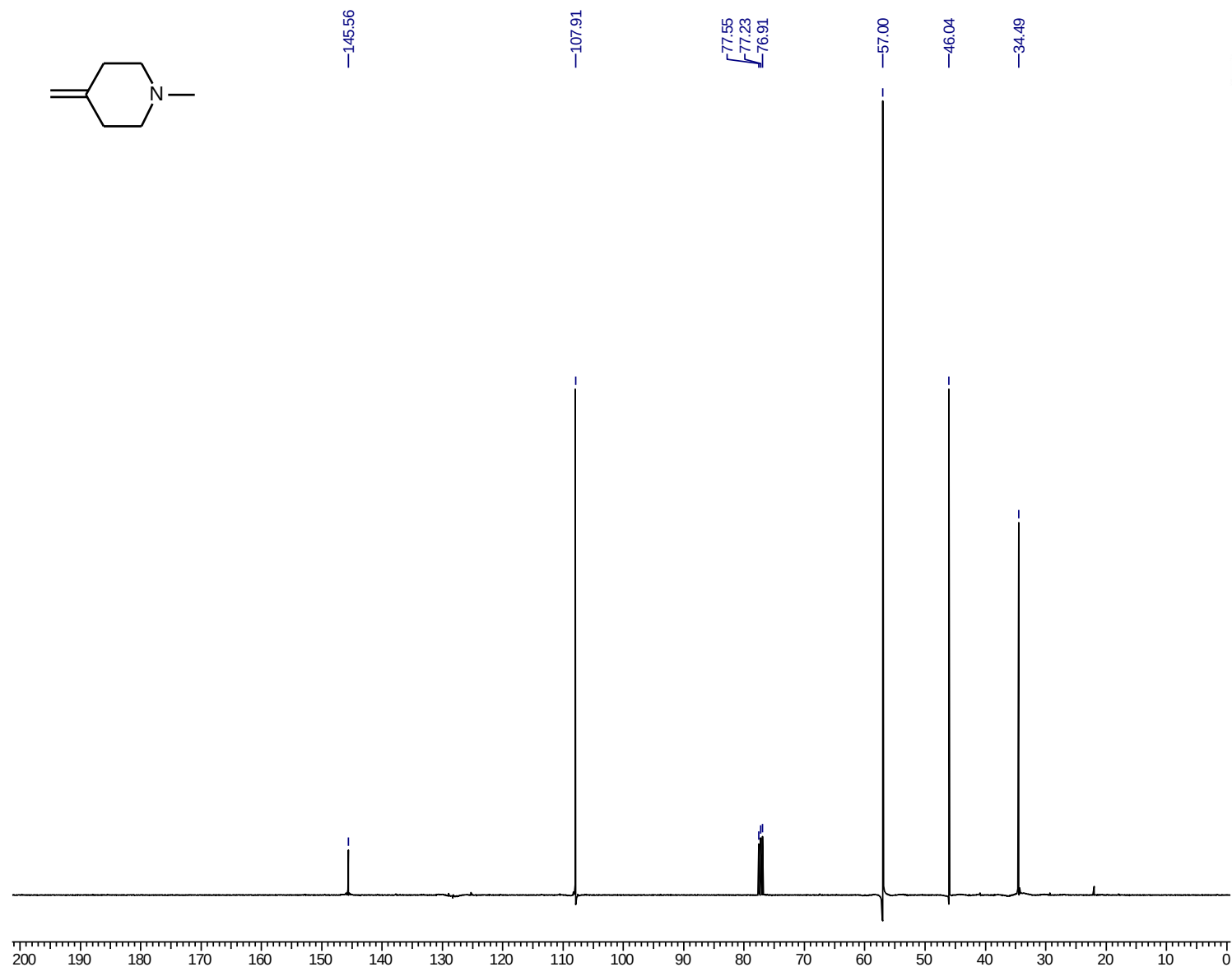
```

NAME          acr-27
EXPNO          10
PROCNO         1
INSTRUM        AV400
PROBHD         5 mm PABBO BB-
PULPROG        zg30b
TD             65536
SOLVENT        CDCl3
NS             64
DS             0
SWH            8264.463 Hz
FIDRES         0.126106 Hz
AQ            3.9649780 sec
RG            18
DW            60.500 usec
DE            9.40 usec
TE            294.0 K
D1            1.00000000 sec
TD0           1
  
```

```

===== CHANNEL f1 =====
NUC1           1H
P1            10.00 usec
PL1           -3.60 dB
PL1W          17.83863831 W
SF01          400.1324710 MHz
SI            32768
SF            400.1299938 MHz
WDW            EM
SSB            0
LB            0.30 Hz
GB            0
PC            1.00
  
```

¹³C NMR spectrum of 6 (100 MHz, CDCl₃)



```

NAME          acr-27
EXPNO         12
PROCNO        1
INSTRUM       AV400
PROBHD        5 mm PABBO BB-
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            3072
DS            2
SWH           23809.523 Hz
FIDRES        0.363304 Hz
AQ            1.3763061 sec
RG            512
DW            21.000 usec
DE            7.66 usec
TE            294.8 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0           1
  
```

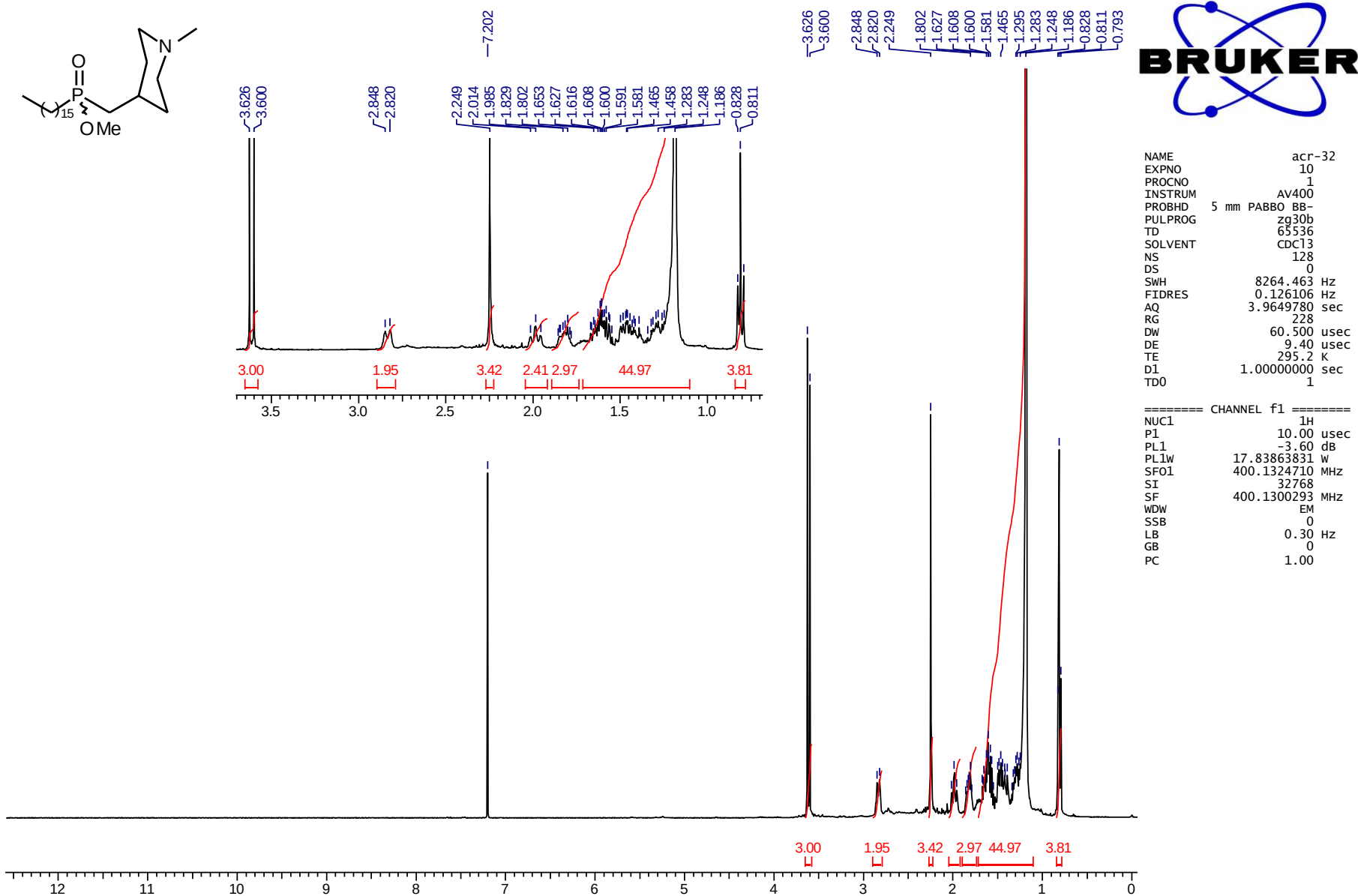
```

===== CHANNEL f1 =====
NUC1          13C
P1            8.00 usec
PL1           0.00 dB
PL1W          34.91522217 w
SF01          100.6228294 MHz
  
```

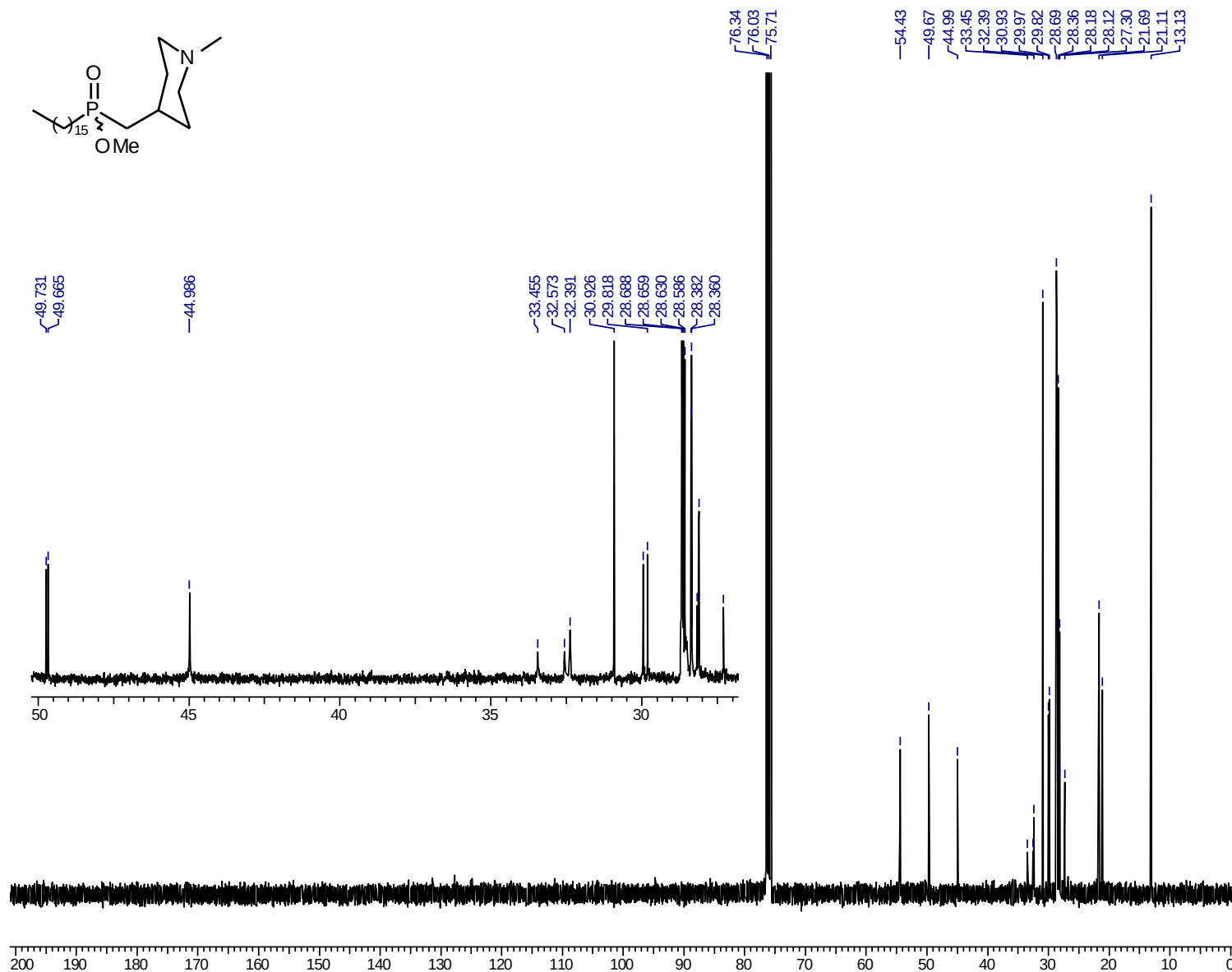
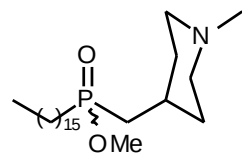
```

===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2          1H
PCPD2         90.00 usec
PL2           -3.60 dB
PL12          15.31 dB
PL13          18.00 dB
PL2W          17.83863831 w
PL12W         0.22927761 w
PL13W         0.12341322 w
SF02          400.1316005 MHz
SI            32768
SF            100.6127690 MHz
WDW            EM
SSB            0
LB            1.00 Hz
GB            0
PC            1.40
  
```

¹H NMR spectrum of 26a (400 MHz, CDCl₃)



¹³C NMR spectrum of 26a (100 MHz, CDCl₃)

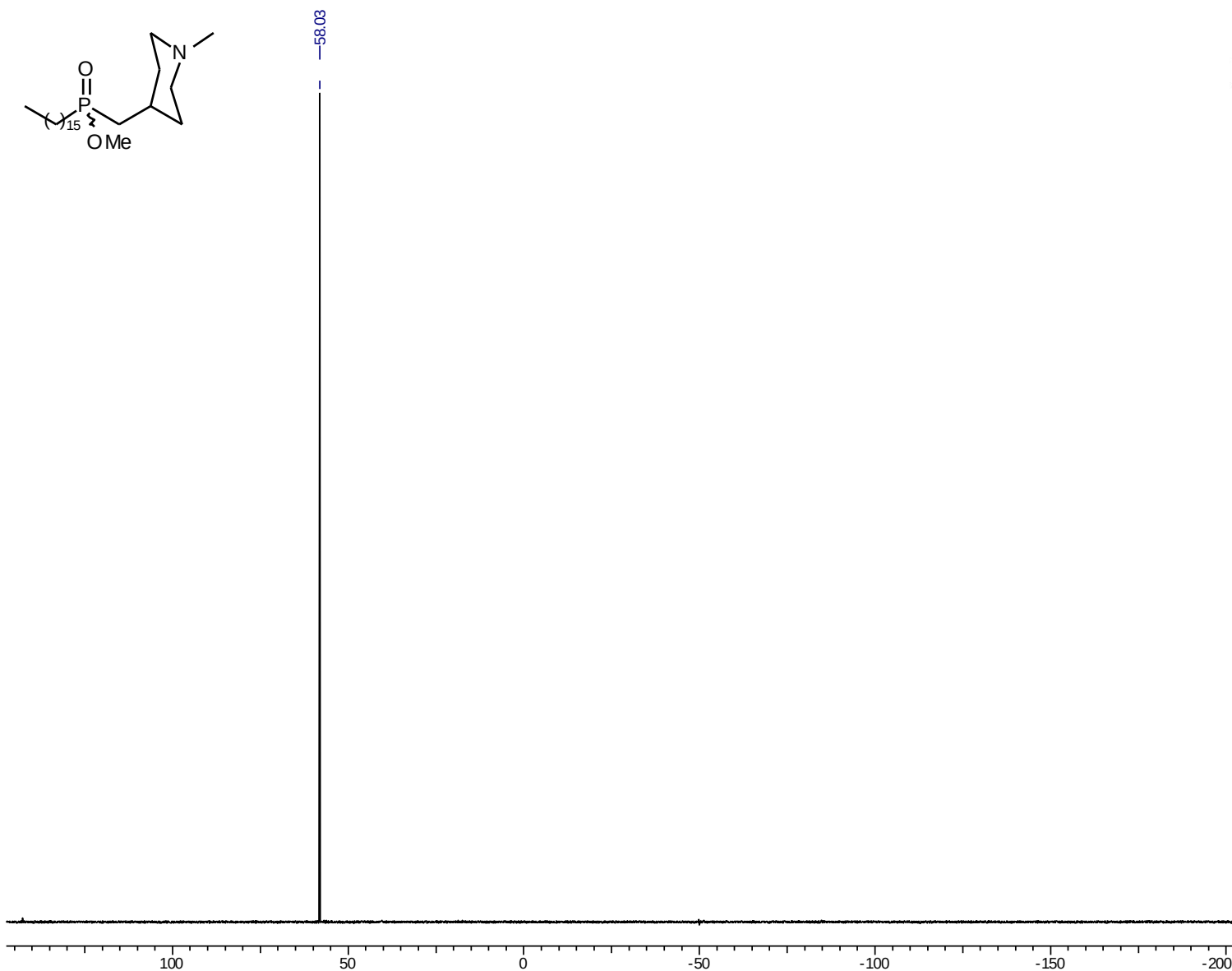


NAME Administrator-57
EXPNO 10
PROCNO 1
INSTRUM AV400_S
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 16384
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 406
DW 20.800 usec
DE 6.50 usec
TE 295.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 8.00 usec
PL1 0.00 dB
PL1W 33.91046524 W
SF01 100.6479773 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -3.60 dB
PL12 13.80 dB
PL13 14.00 dB
PL2W 18.98951721 W
PL12W 0.34555241 W
PL13W 0.33000001 W
SF02 400.2316009 MHz
SI 32768
SF 100.6380168 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

^{31}P (proton-decoupled) NMR spectrum of 26a (162 MHz, CDCl_3)

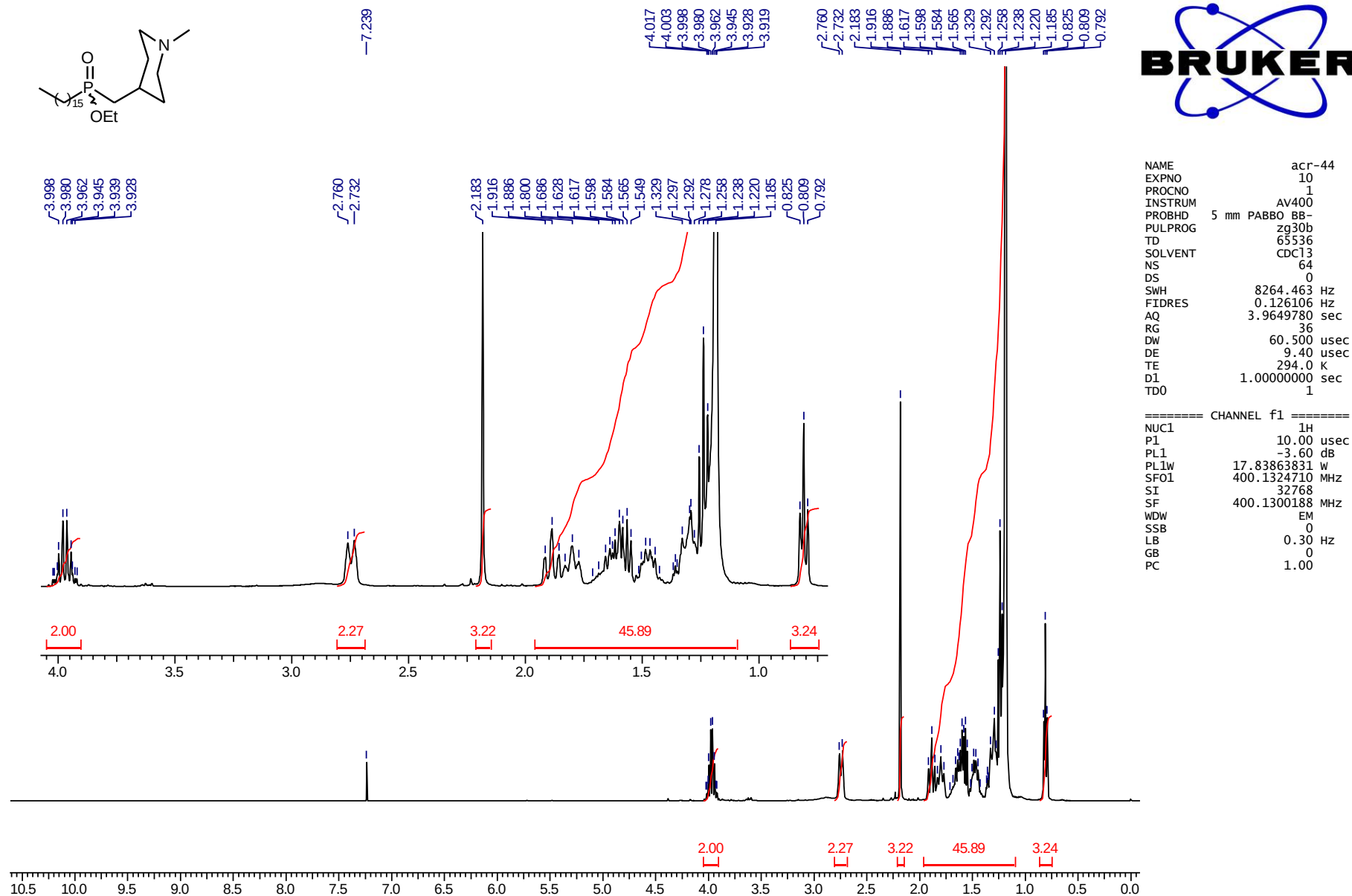


```
NAME          acr-32
EXPNO          16
PROCNO         1
INSTRUM        AV400
PROBHD         5 mm PABBO BB-
PULPROG        zgpg30
TD             65536
SOLVENT        CDCl3
NS             1024
DS             4
SWH            64102.563 Hz
FIDRES         0.978127 Hz
AQ             0.5112308 sec
RG             23100
DW             7.800 usec
DE             6.50 usec
TE             296.6 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0            1
```

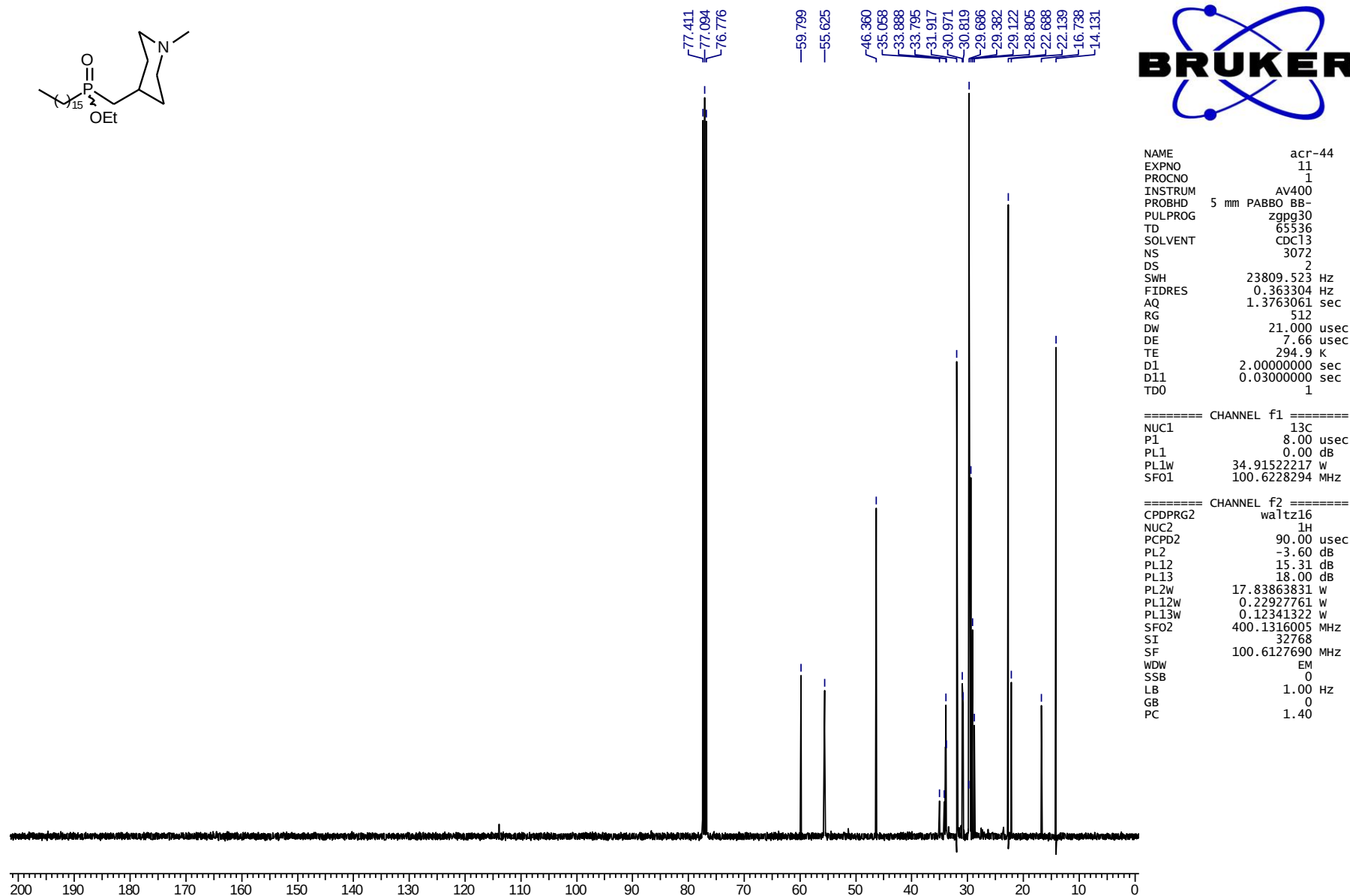
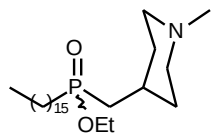
```
===== CHANNEL f1 =====
NUC1           31P
P1             9.40 usec
PL1            0.00 dB
PL1W           26.87718391 W
SFO1           161.9674942 MHz
```

```
===== CHANNEL f2 =====
CPDPRG2        wa1tz16
NUC2           1H
PCPD2          80.00 usec
PL2            -3.60 dB
PL12           13.80 dB
PL13           14.00 dB
PL2W           17.83863831 W
PL12W          0.32460985 W
PL13W          0.31000000 W
SFO2           400.1316005 MHz
SI             32768
SF             161.9755930 MHz
WDW            EM
SSB            0
LB             1.00 Hz
GB             0
PC             1.40
```

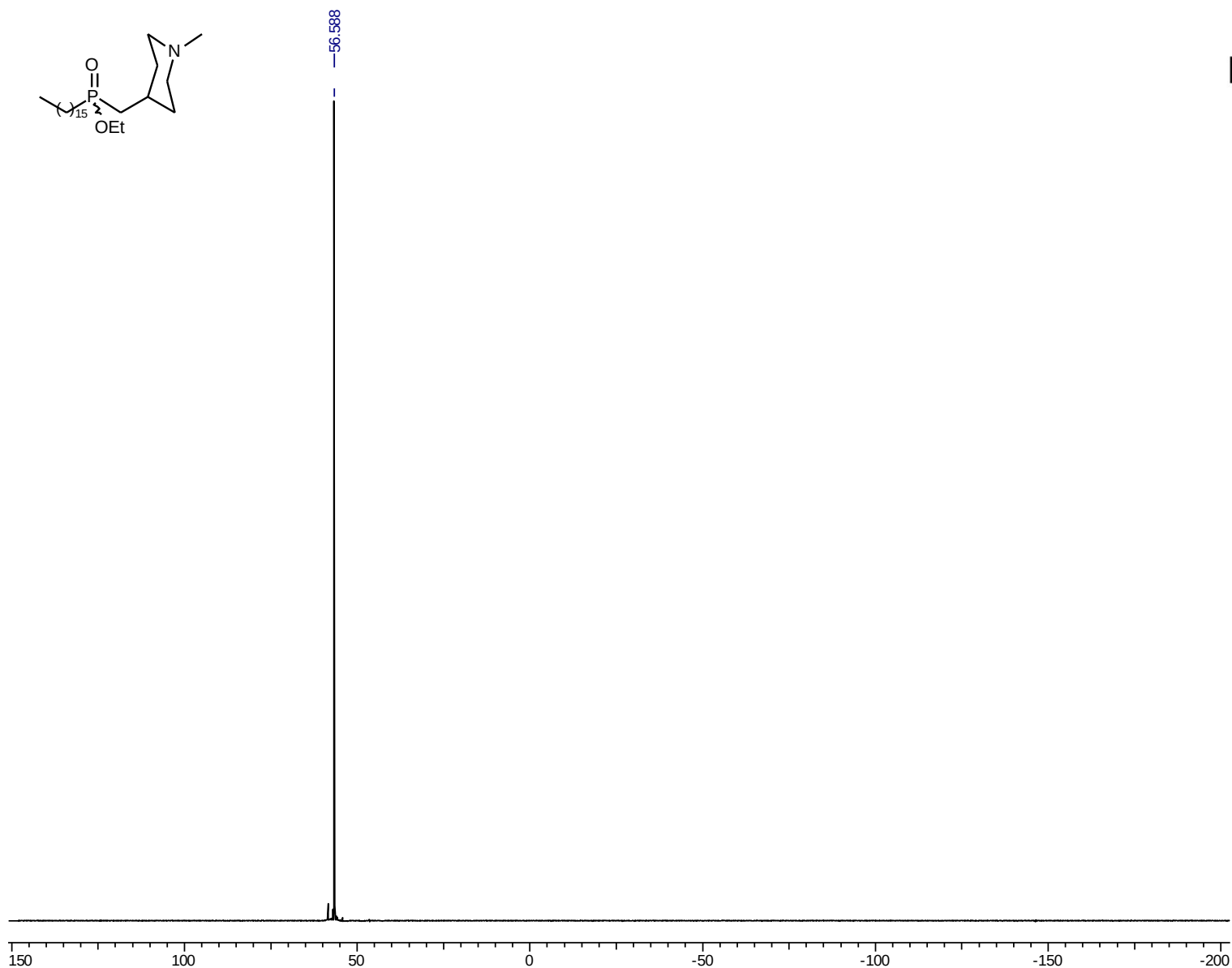
¹H NMR spectrum of 26b (400 MHz, CDCl₃)



¹³C NMR spectrum of 26b (100 MHz, CDCl₃)



^{31}P (proton-decoupled) NMR spectrum of 26b (162 MHz, CDCl_3)



```

NAME          acr-44
EXPNO          18
PROCNO         1
INSTRUM        AV400
PROBHD         5 mm PABBO B8-
PULPROG        zgpg30
TD             65536
SOLVENT        CDCl3
NS             512
DS             4
SWH            64102.563 Hz
FIDRES         0.978127 Hz
AQ            0.5112308 sec
RG            23100
DW            7.800 usec
DE            6.50 usec
TE            294.7 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0           1
  
```

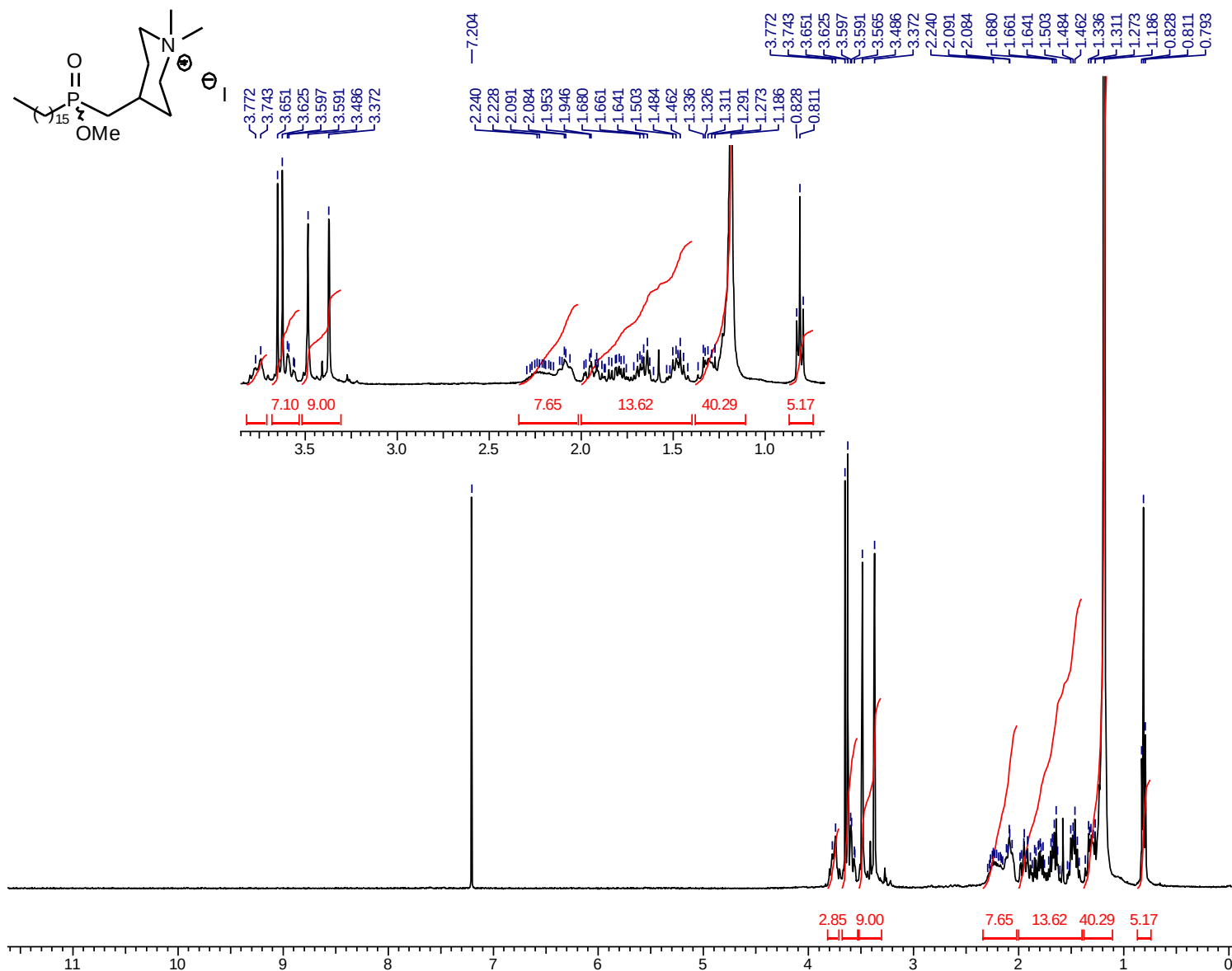
```

===== CHANNEL f1 =====
NUC1          31P
P1            9.40 usec
PL1           0.00 dB
PL1W          26.87718391 W
SF01          161.9674942 MHz
  
```

```

===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2          1H
PCPD2         90.00 usec
PL2           -3.60 dB
PL12          15.31 dB
PL13          18.00 dB
PL2W          17.83863831 W
PL12W         0.22927761 W
PL13W         0.12341322 W
SF02          400.1316005 MHz
SI            32768
SF            161.9755930 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
  
```

¹H NMR spectrum of 28 (400 MHz, CDCl₃)

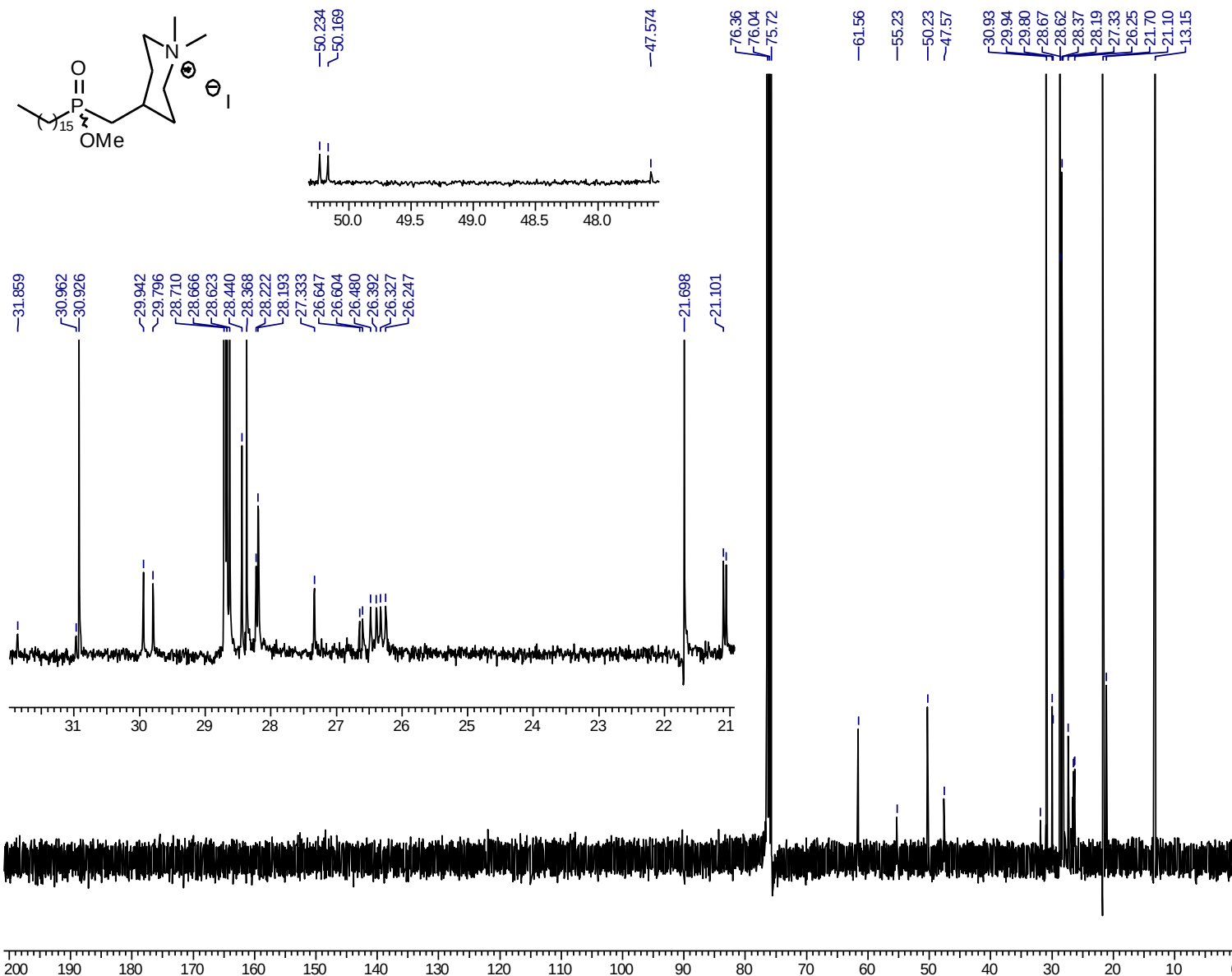


```

NAME          acr-37
EXPNO         10
PROCNO        1
INSTRUM       AV400
PROBHD        5 mm PABBO BB-
PULPROG       zg30b
TD            65536
SOLVENT       CDCl3
NS            32
DS            0
SWH           8264.463 Hz
FIDRES        0.126106 Hz
AQ            3.9649780 sec
RG            256
DW            60.500 usec
DE            9.40 usec
TE            295.6 K
D1            1.00000000 sec
TD0           1

===== CHANNEL f1 =====
NUC1          1H
P1            10.00 usec
PL1           -3.60 dB
PL1w          17.83863831 W
SFO1          400.1324710 MHz
SI            32768
SF            400.1300279 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
    
```

¹³C NMR spectrum of 28 (100 MHz, CDCl₃)

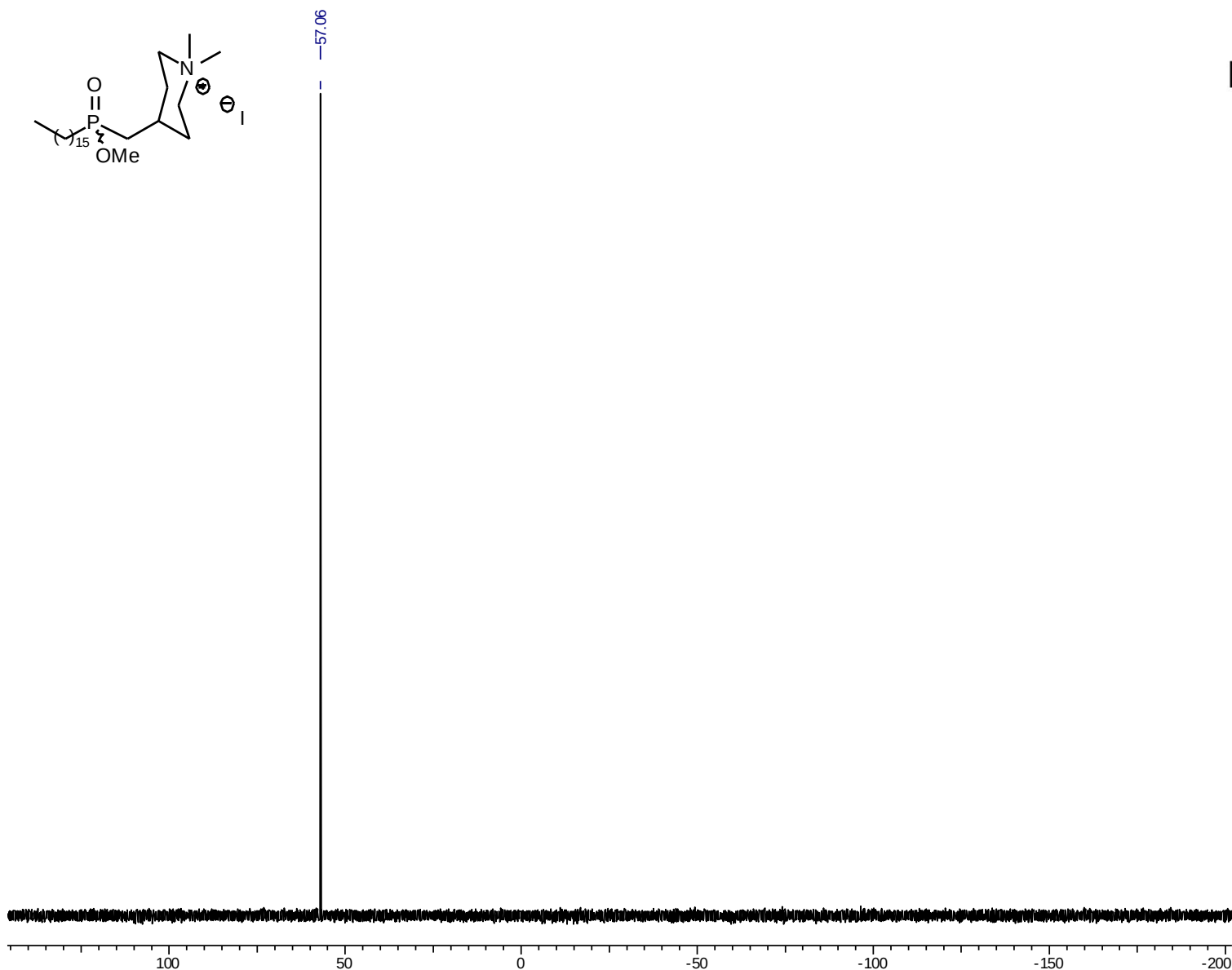


NAME Administrator-55
EXPNO 10
PROCNO 1
INSTRUM AV400_S
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl₃
NS 16384
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 362
DW 20.800 usec
DE 6.50 usec
TE 294.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 ¹³C
P1 8.00 usec
PL1 0.00 dB
PL1W 33.91046524 W
SF01 100.6479773 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 ¹H
PCPD2 90.00 usec
PL2 -3.60 dB
PL12 15.31 dB
PL13 18.00 dB
PL2W 18.98951721 W
PL12W 0.24406971 W
PL13W 0.13137537 W
SF02 400.2316009 MHz
SI 32768
SF 100.6380182 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

³¹P (proton-decoupled) NMR spectrum of 28 (162 MHz, CDCl₃)

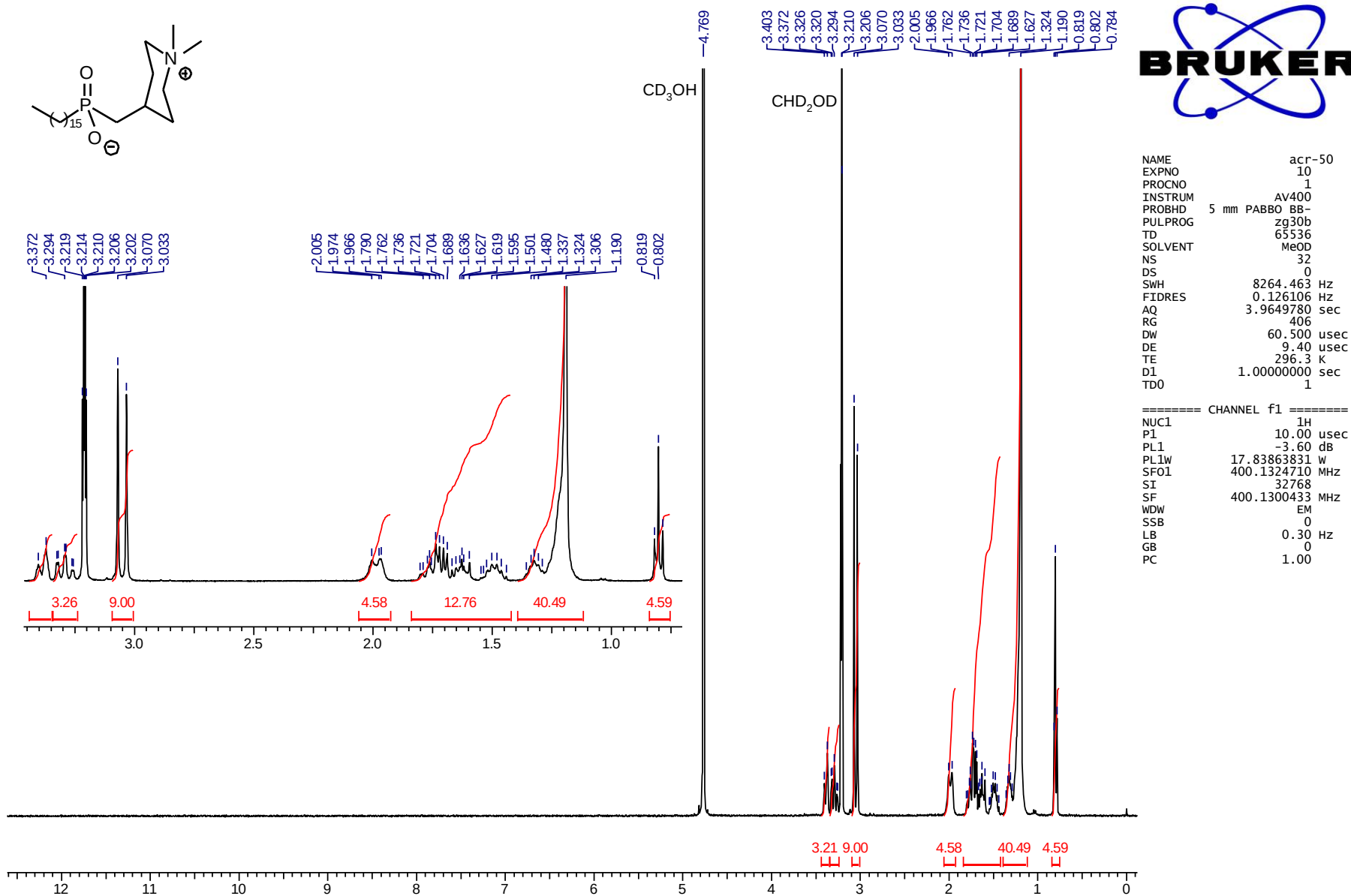


```
NAME          acr-37
EXPNO          11
PROCNO         1
INSTRUM        AV400
PROBHD         5 mm PABBO BB-
PULPROG        zgpg30
TD             65536
SOLVENT        CDCl3
NS             64
DS             4
SWH            64102.563 Hz
FIDRES         0.978127 Hz
AQ            0.5112308 sec
RG            23100
DW            7.800 usec
DE            6.50 usec
TE            296.7 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0           1
```

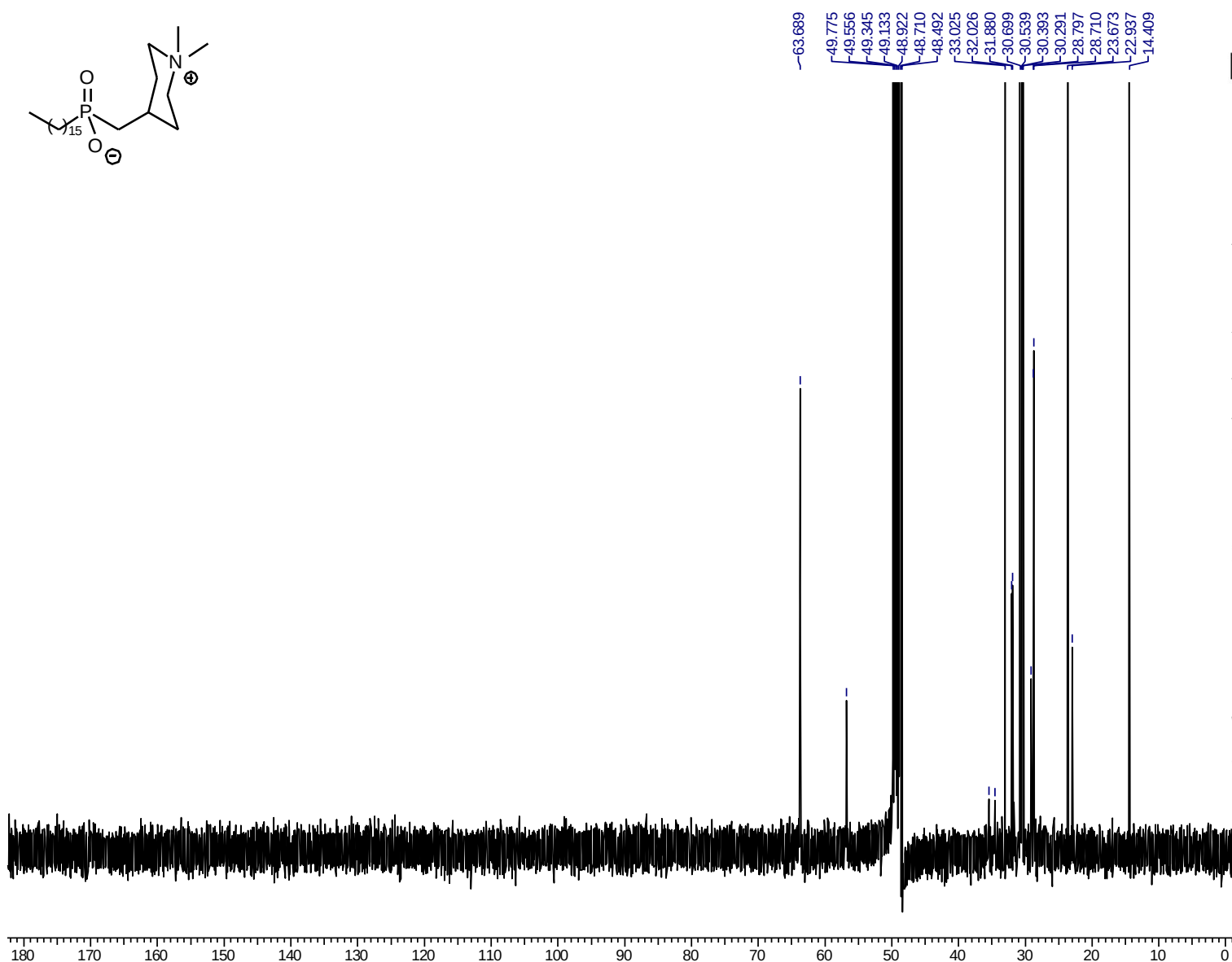
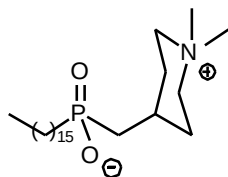
```
===== CHANNEL f1 =====
NUC1           31P
P1            9.40 usec
PL1           0.00 dB
PL1W          26.87718391 W
SFO1          161.9674942 MHz
```

```
===== CHANNEL f2 =====
CPDPRG2        waltz16
NUC2           1H
PCPD2          80.00 usec
PL2           -3.60 dB
PL12          13.80 dB
PL13          14.00 dB
PL2W          17.83863831 W
PL12W         0.32460985 W
PL13W         0.31000000 W
SFO2          400.1316005 MHz
SI            32768
SF            161.9755930 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB           0
PC            1.40
```


¹H NMR spectrum of 4 (400 MHz, CD₃OD)



¹³C NMR spectrum of 4 (100 MHz, 328.1 K, CD₃OD)



```

NAME          acr-1
EXPNO          11
PROCNO         1
INSTRUM        AV400_S
PROBHD         5 mm PABBO BB-
PULPROG        zgpg30
TD             65536
SOLVENT        MeOD
NS             3072
DS             4
SWH            24038.461 Hz
FIDRES         0.366798 Hz
AQ            1.3631988 sec
RG            1030
DW            20.800 usec
DE            6.50 usec
TE            328.1 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0           1
  
```

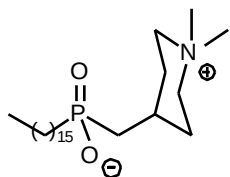
```

===== CHANNEL f1 =====
NUC1           13C
P1             8.00 usec
PL1            0.00 dB
PL1W          33.91046524 W
SF01          100.6479773 MHz
  
```

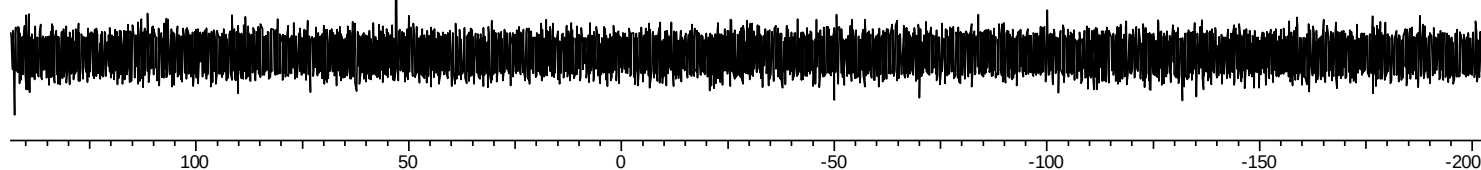
```

===== CHANNEL f2 =====
CPDPRG2        waltz16
NUC2           1H
PCPD2          90.00 usec
PL2            -3.60 dB
PL12          15.31 dB
PL13          18.00 dB
PL2W          18.98951721 W
PL12W         0.24406971 W
PL13W         0.13137537 W
SF02          400.2316009 MHz
SI            32768
SF            100.6377711 MHz
WDW            EM
SSB            0
LB            1.00 Hz
GB            0
PC            1.40
  
```

^{31}P (proton-decoupled) NMR spectrum of 4 (162 MHz, CD_3OD)



—52.86

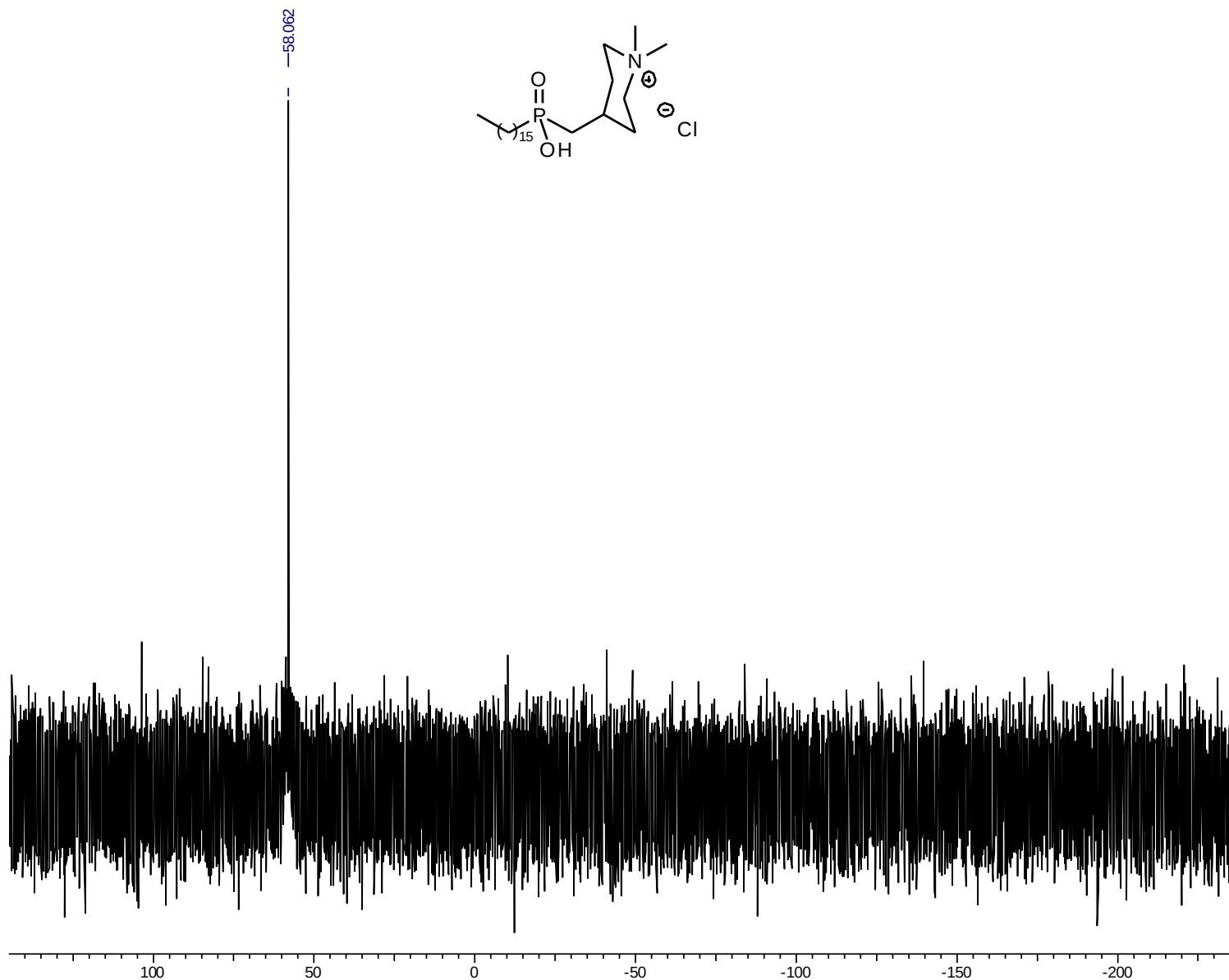


```
NAME      acr-50
EXPNO     11
PROCNO    1
INSTRUM   AV400
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD        65536
SOLVENT   MeOD
NS         64
DS         4
SWH        64102.563 Hz
FIDRES     0.978127 Hz
AQ         0.5112308 sec
RG         23100
DW         7.800 usec
DE         6.50 usec
TE         297.2 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1
```

```
===== CHANNEL f1 =====
NUC1       31P
P1         9.40 usec
PL1        0.00 dB
PL1W       26.87718391 w
SF01       161.9674942 MHz
```

```
===== CHANNEL f2 =====
CPDPRG2    waltz16
NUC2       1H
PCPD2      80.00 usec
PL2        -3.60 dB
PL12       13.80 dB
PL13       14.00 dB
PL2W       17.83863831 w
PL12W      0.32460985 w
PL13W      0.31000000 w
SF02       400.1316005 MHz
SI         32768
SF         161.9755930 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.40
```

^{31}P (proton-decoupled) NMR spectrum of the hydrochloride salt of 4 (162 MHz, CD_3OD)



```

NAME          acr-18
EXPNO         11
PROCNO        1
INSTRUM       AV400
PROBHD        5 mm PABBO BB-
PULPROG       zgpg30
TD            65536
SOLVENT       MeOD
NS            64
DS            4
SWH           64102.563 Hz
FIDRES        0.978127 Hz
AQ            0.5112308 sec
RG            23100
DW            7.800 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          26.87718391 w
SF01          161.9674942 MHz
    
```

```

===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2          1H
PCPD2         90.00 usec
PL2           -3.60 dB
PL12          15.31 dB
PL13          18.00 dB
PL2W          17.83863831 w
PL12W         0.22927761 w
PL13W         0.12341322 w
SF02          400.1316005 MHz
SI            32768
SF            161.9755930 MHz
WDS           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
    
```