

Supporting information for:

Tris(pyrazolyl)phosphine Oxides. Synthesis and Coordination Chemistry with Copper(I)

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Lutz, J. Chris Slootweg,* and Koop Lammertsma*

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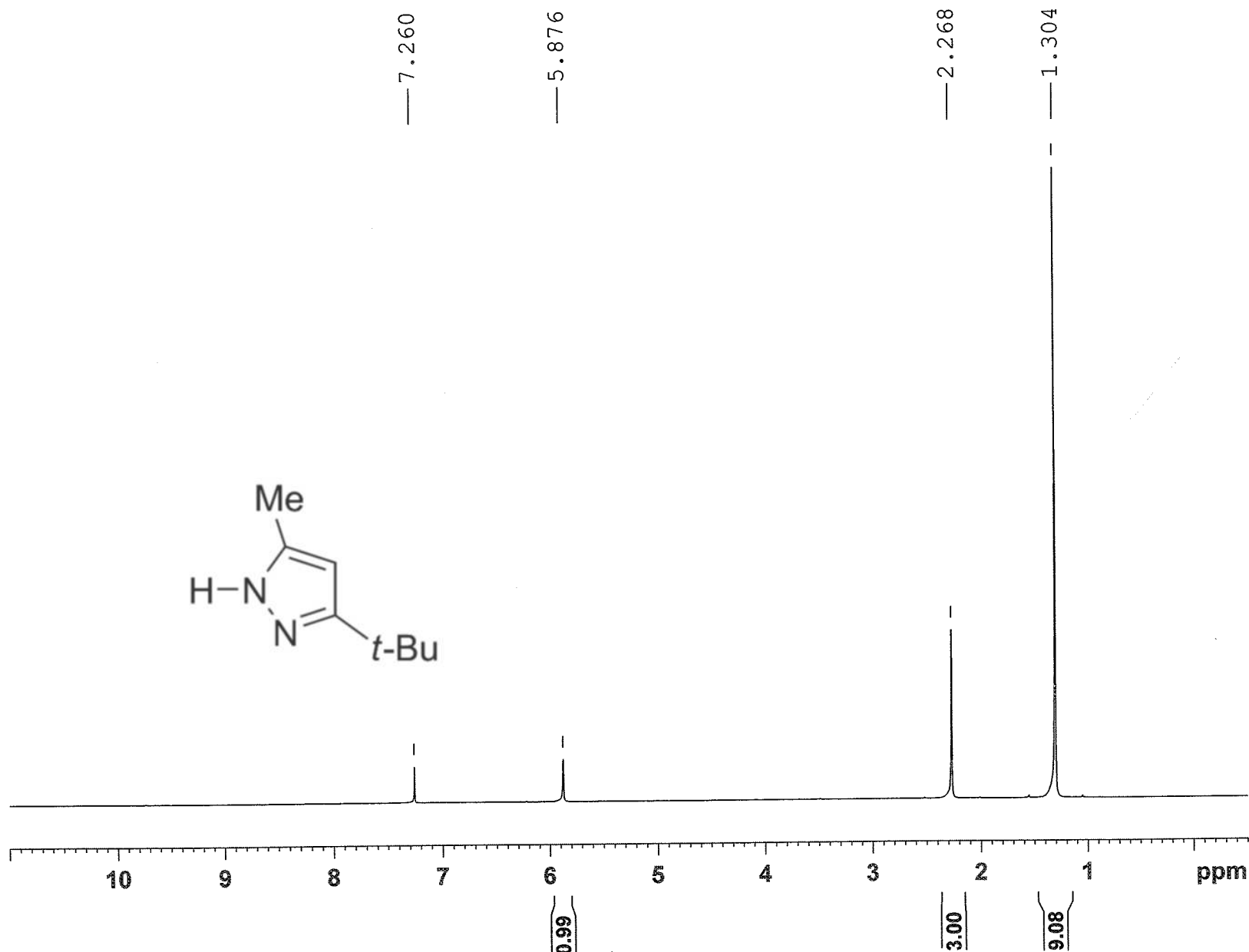
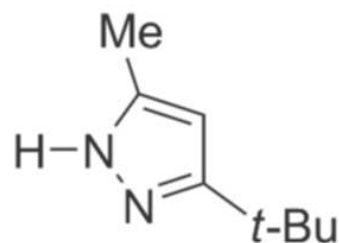
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¹H NMR of 3-*t*-Bu-5-MepzH



NAME LAK74
EXPNO 1
PROCNO 1
Date_ 20090909
Time_ 8.56
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 5175.983 Hz
FIDRES 0.078979 Hz
AQ 6.3308277 sec
RG 1149.4
DW 96.600 usec
DE 6.00 usec
TE 300.2 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 9.50 usec
PL1 -3.00 dB
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SI 65536
SF 250.1300121 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.40



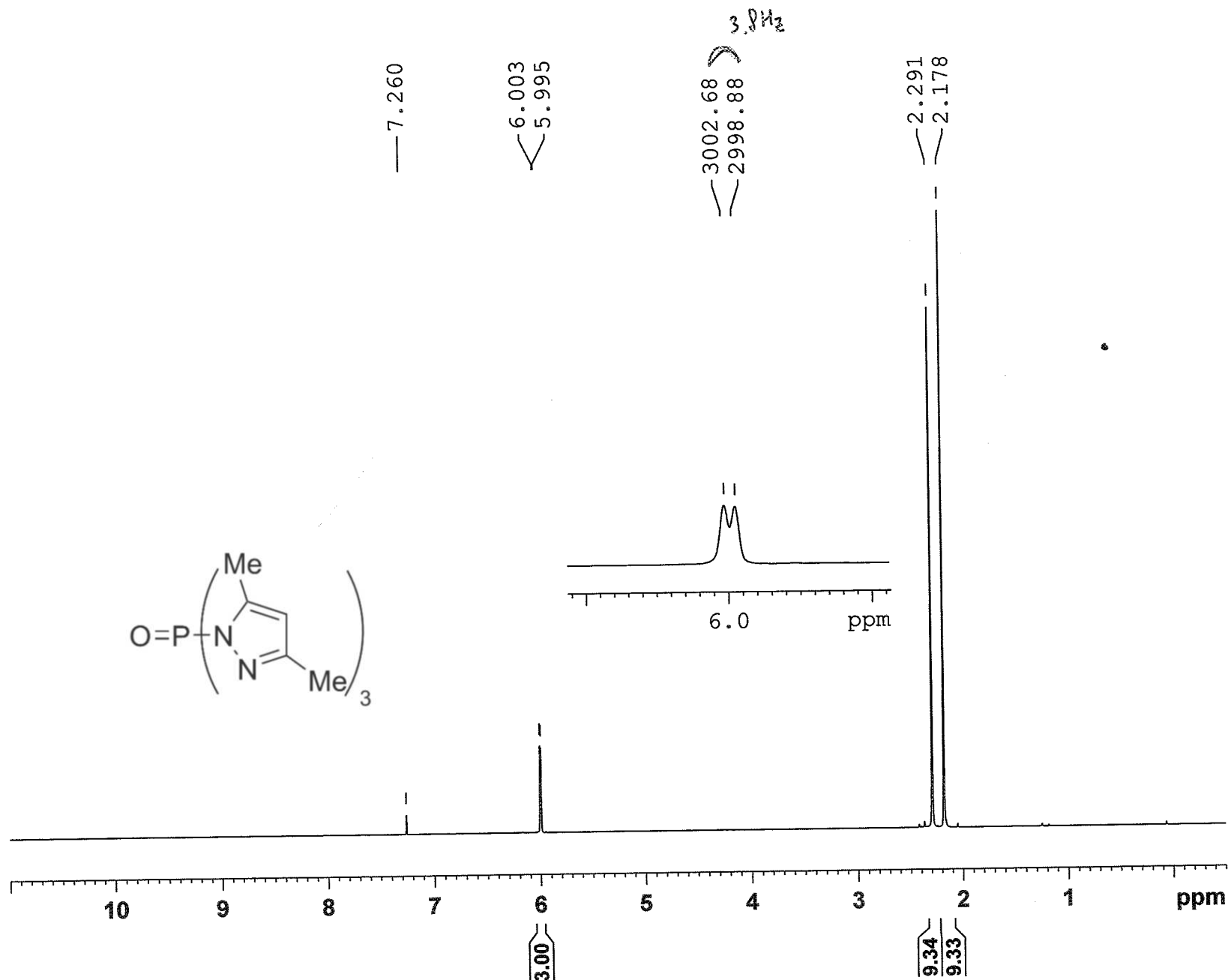
(Me, Me-Pz) 3PO

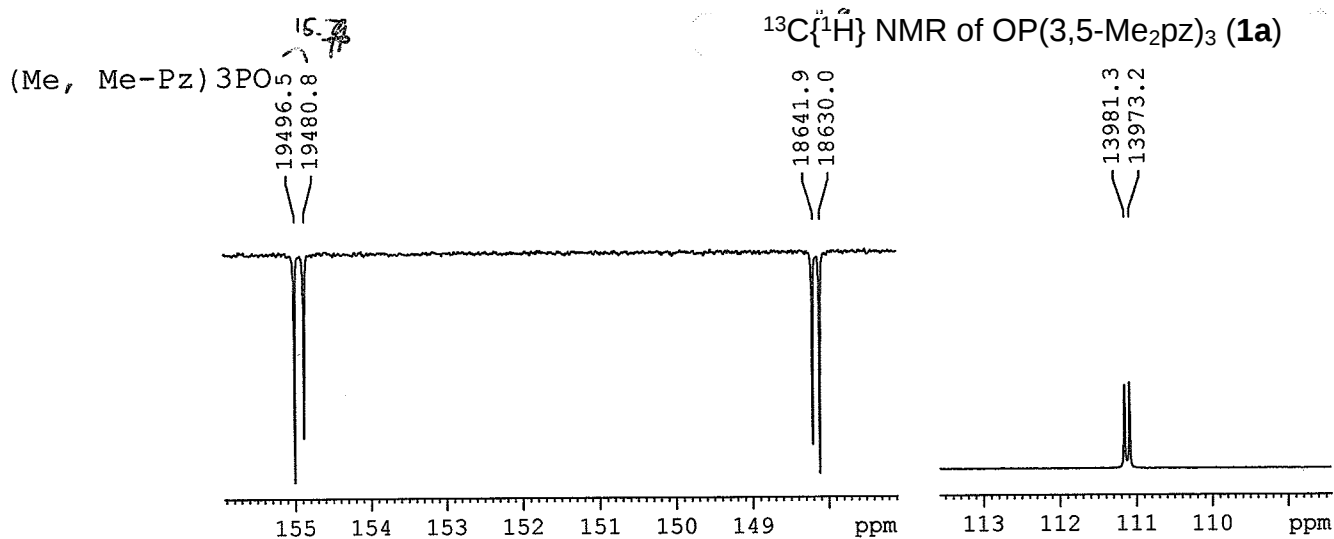
¹H NMR of OP(3,5-Me₂pz)₃ (1a)



NAME VVL 22_500
 EXPNO 2
 PROCNO 1
 Date_ 20100819
 Time_ 14.35
 INSTRUM spect
 PROBHD 5 mm CPTCI 1H-
 PULPROG zg30
 TD 65536
 SOLVENT CDCl₃
 NS 16
 DS 2
 SWH 10330.578 Hz
 FIDRES 0.157632 Hz
 AQ 3.1719923 sec
 RG 22.6
 DW 48.400 usec
 DE 6.50 usec
 TE 296.1 K
 D1 1.00000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 6.70 usec
 PL1 4.00 dB
 PL1W 8.72000027 W
 SFO1 500.2330891 MHz
 SI 32768
 SF 500.2300090 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

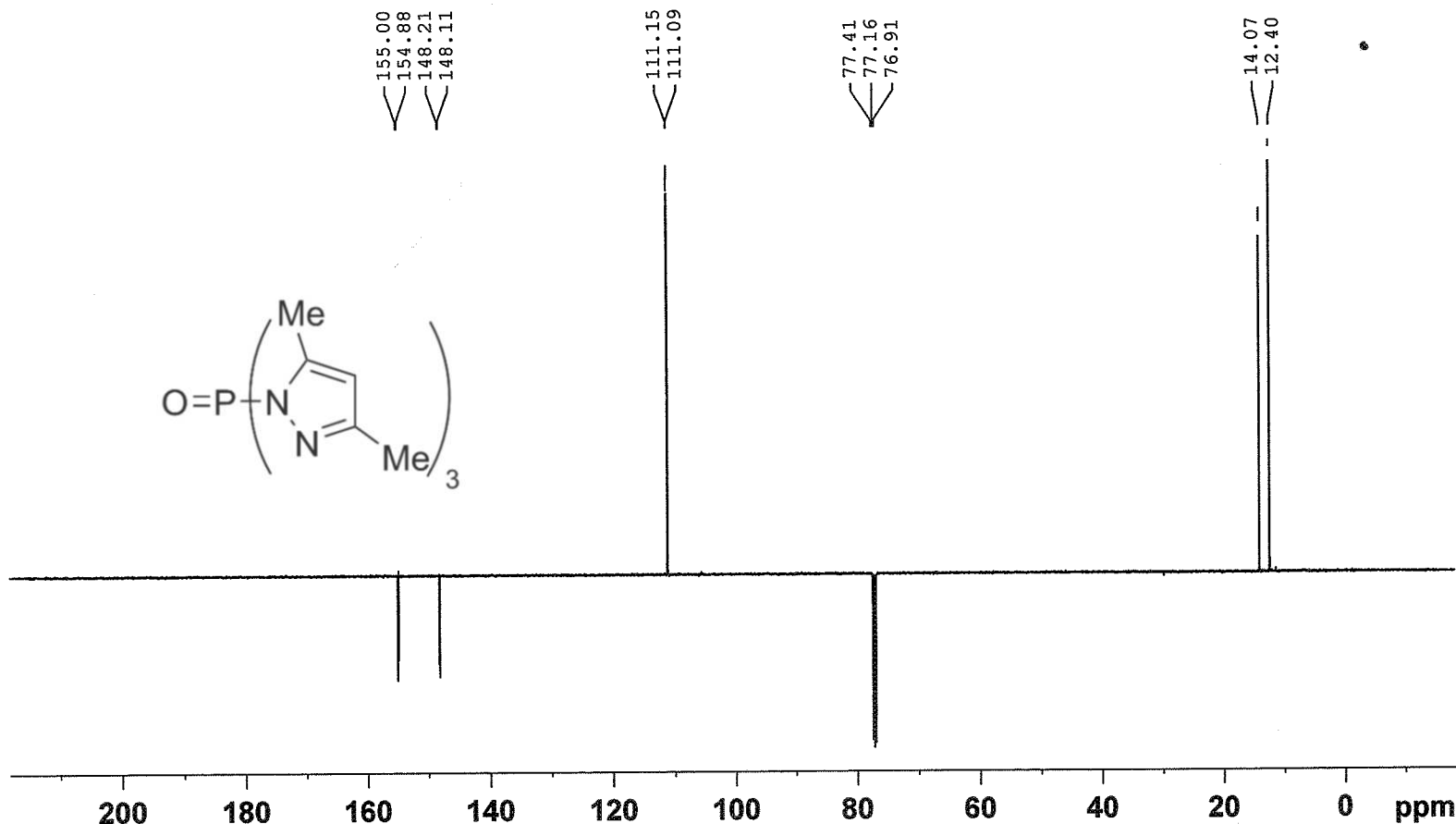
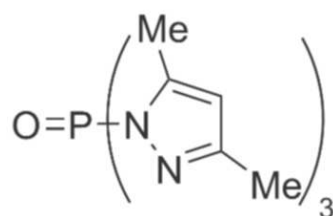




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PROCNO 1
Date_ 20100819
Time 14.33
INSTRUM spect
PROBHD 5 mm CPTCI 1H-
PULPROG jmod
TD 65536
SOLVENT CDC13
NS 256
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 2050
DW 16.800 usec
DE 6.50 usec
TE 295.9 K
CNST2 145.000000
CNST11 1.000000
D1 2.0000000 sec
D20 0.00689655 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 11.20 usec
P2 22.40 usec
PL1 -2.00 dB
PL1W 88.77790070 W
SFO1 125.7955118 MHz

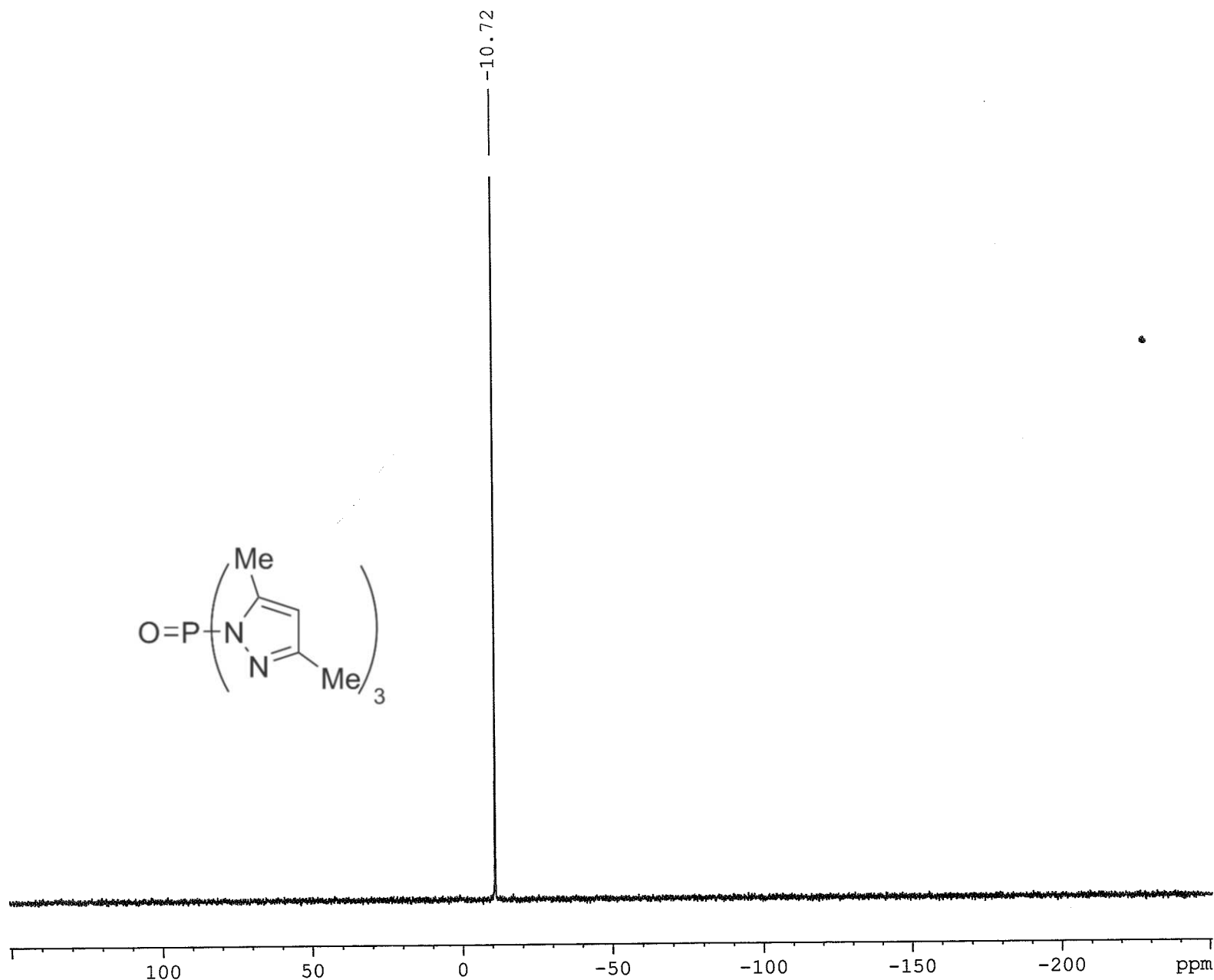
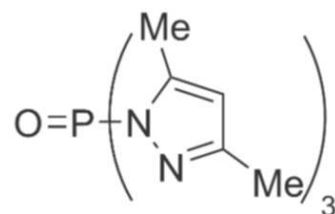
===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 4.00 dB
PL12 25.28 dB
PL2W 8.72000027 W
PL12W 0.06494062 W
SFO2 500.2320009 MHz
SI 32768
SF 125.7829213 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



³¹P{¹H} NMR of OP(3,5-Me₂pz)₃ (**1a**)

(Me,Me-Pz) 3PO

101012 CDC13 {D:\NMRDATA} Volodymyr 60



Current Data Parameters

NAME VVL 441
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters

Date_ 20100819
Time_ 15.39
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 32
DS 4
SWH 40650.406 Hz
FIDRES 0.620276 Hz
AQ 0.8061428 sec
RG 20642.5
DW 12.300 usec
DE 6.00 usec
TE 300.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

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

NUC1 31P
P1 7.00 usec
PL1 0.00 dB
SFO1 101.2494172 MHz

===== CHANNEL f2 =====

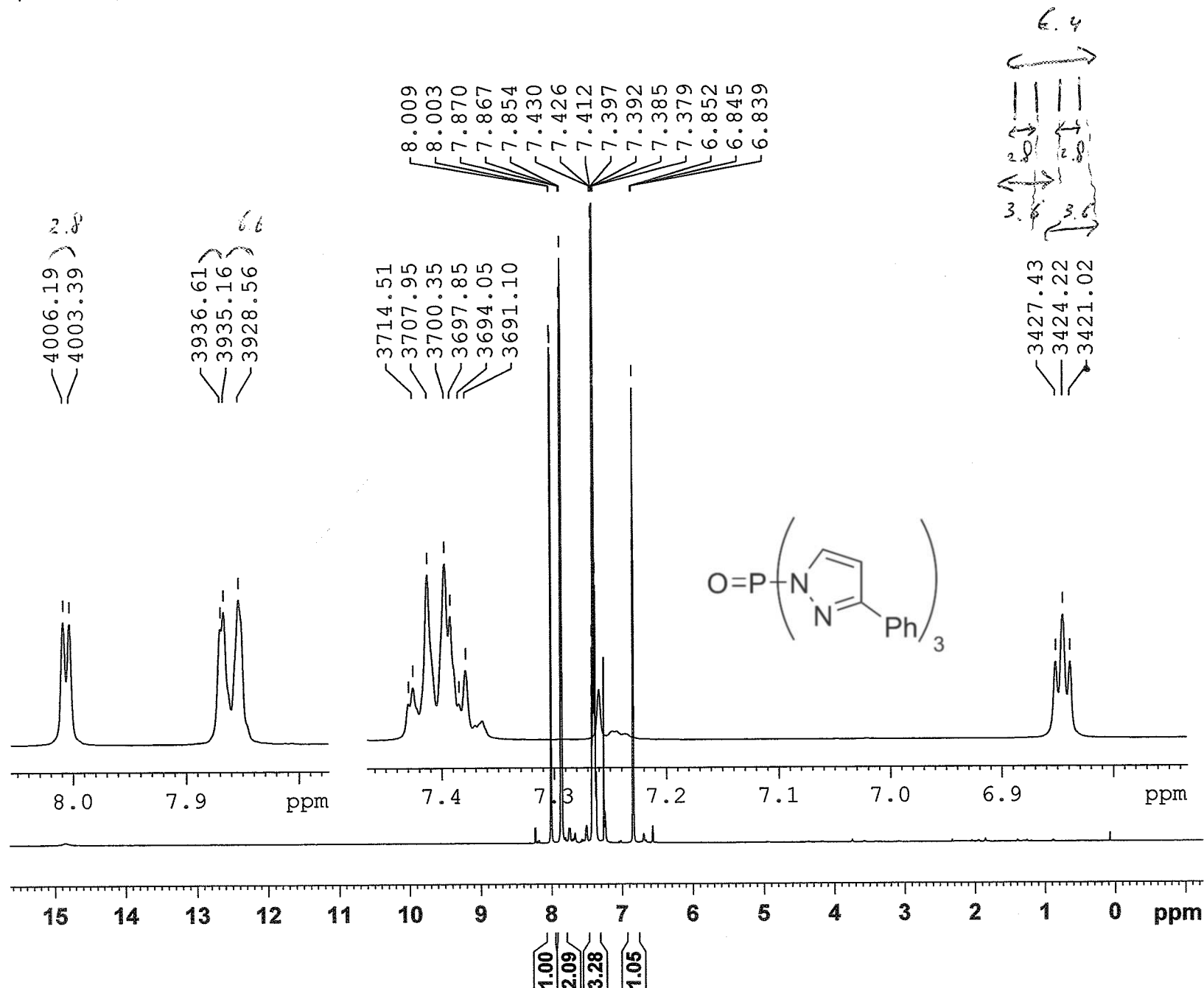
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NUC2 1H
PCPD2 80.00 usec
PL2 -3.00 dB
PL12 20.00 dB
PL13 24.00 dB
SFO2 250.1310005 MHz

F2 - Processing parameters

SI 32768
SF 101.2544800 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40

¹H NMR of OP(3-Phpz)₃ (**1b**)

(H, Ph-Pz) 3PO

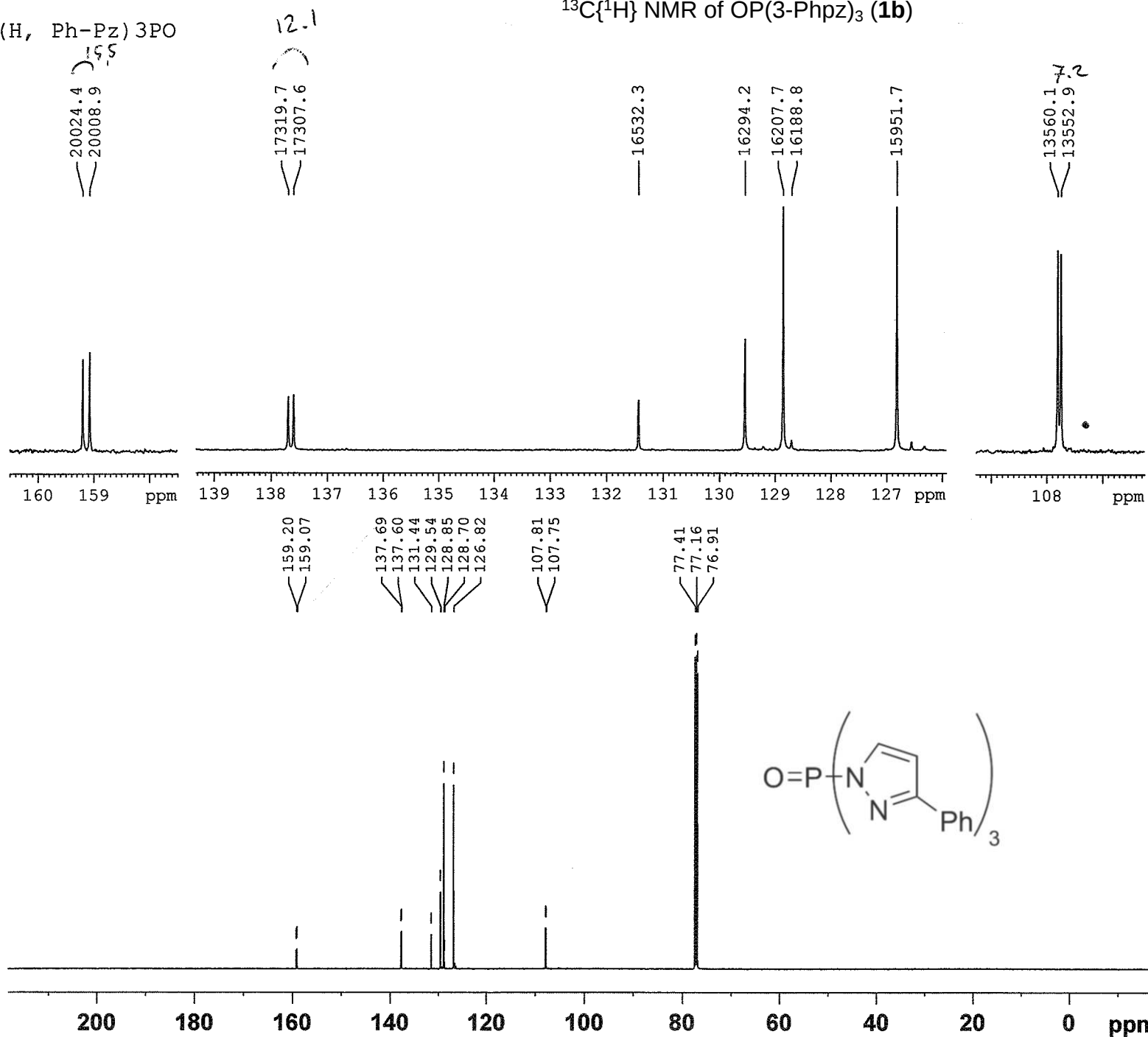


NAME VVL 23_500
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PROCNO 1
Date_ 20100819
Time_ 14.42
INSTRUM spect
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PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719923 sec
RG 14.2
DW 48.400 usec
DE 6.50 usec
TE 296.1 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 6.70 usec
PL1 4.00 dB
PL1W 8.72000027 W
SFO1 500.2330891 MHz
SI 32768
SF 500.2300091 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

(H, Ph-Pz) 3PO

$^{13}\text{C}\{^1\text{H}\}$ NMR of OP(3-Phpz)₃ (**1b**)



NAME VVL 23_500
EXPNO 2
PROCNO 1
Date_ 20100819
Time_ 15.10
INSTRUM spect
PROBHD 5 mm CPTCI 1H-
PULPROG zgpg30
TD 65536
SOLVENT CDCl_3
NS 512
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 2050
DW 16.800 usec
DE 6.50 usec
TE 296.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 ^{13}C
P1 11.20 usec
PL1 -2.00 dB
PL1W 88.77790070 W
SFO1 125.7955118 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 ^1H
PCPD2 80.00 usec
PL2 4.00 dB
PL12 25.28 dB
PL13 28.00 dB
PL2W 8.72000027 W
PL12W 0.06494062 W
PL13W 0.03471494 W
SFO2 500.2320009 MHz
SI 32768
SF 125.7829167 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

³¹P{¹H} NMR of OP(3-Phpz)₃ (**1b**)

11,11,11,11-tetrafluoroethane-d₂ after working with pentane



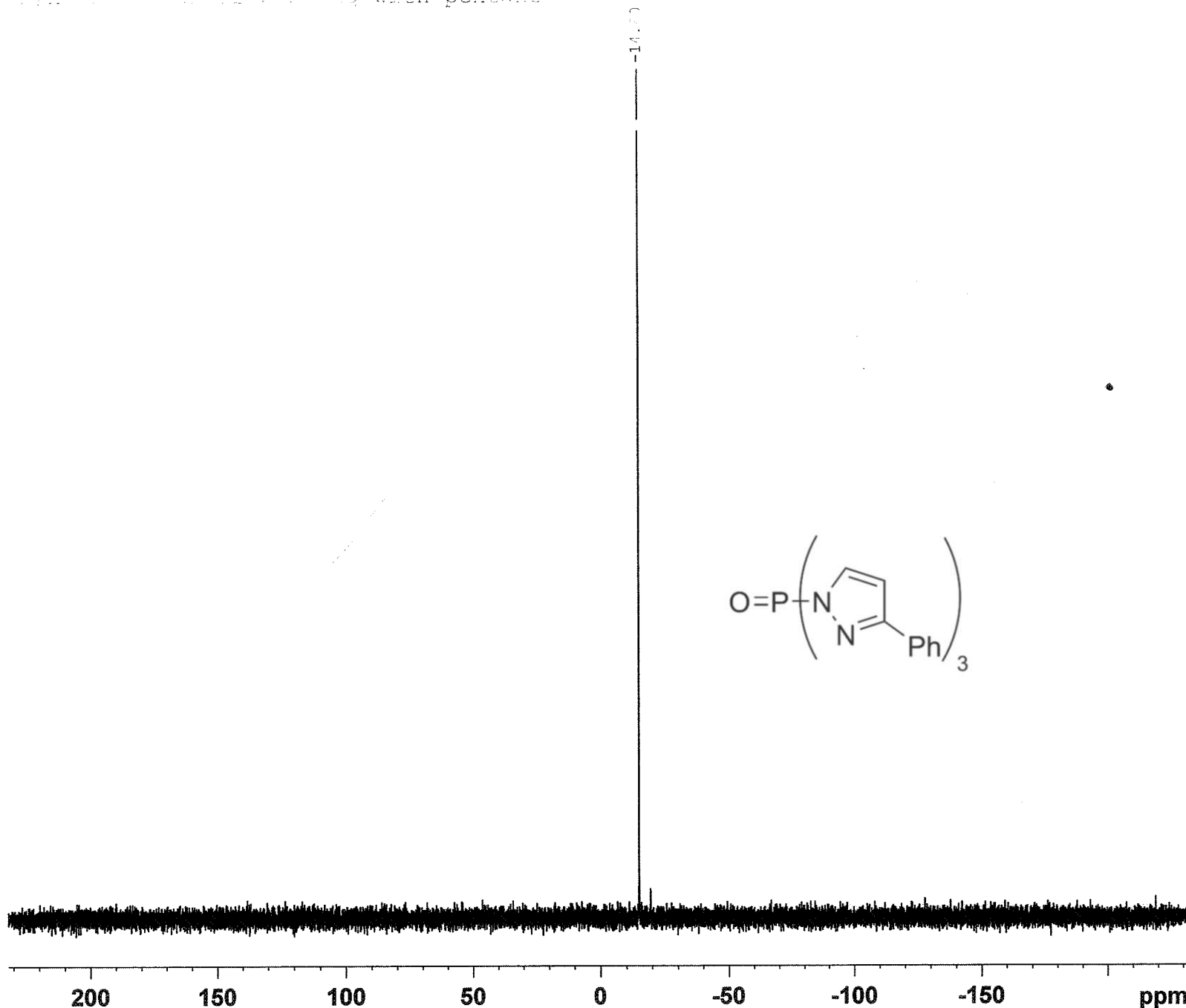
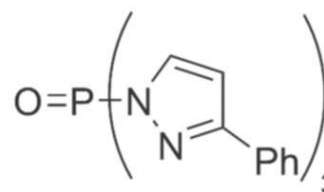
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PROCNO 1

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PULPROG zgpg30
TD 65536
SOLVENT Acetone
NS 17
DS 4
SWH 75187.969 Hz
FIDRES 1.147277 Hz
AQ 0.4358644 sec
RG 20642.5
DW 6.650 usec
DE 6.00 usec
TE 300.4 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

===== CHANNEL f1 =====
NUC1 31P
P1 6.25 usec
PL1 -1.00 dB
SFO1 161.9755930 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 3.00 dB
PL12 18.00 dB
PL13 18.00 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 161.9755930 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



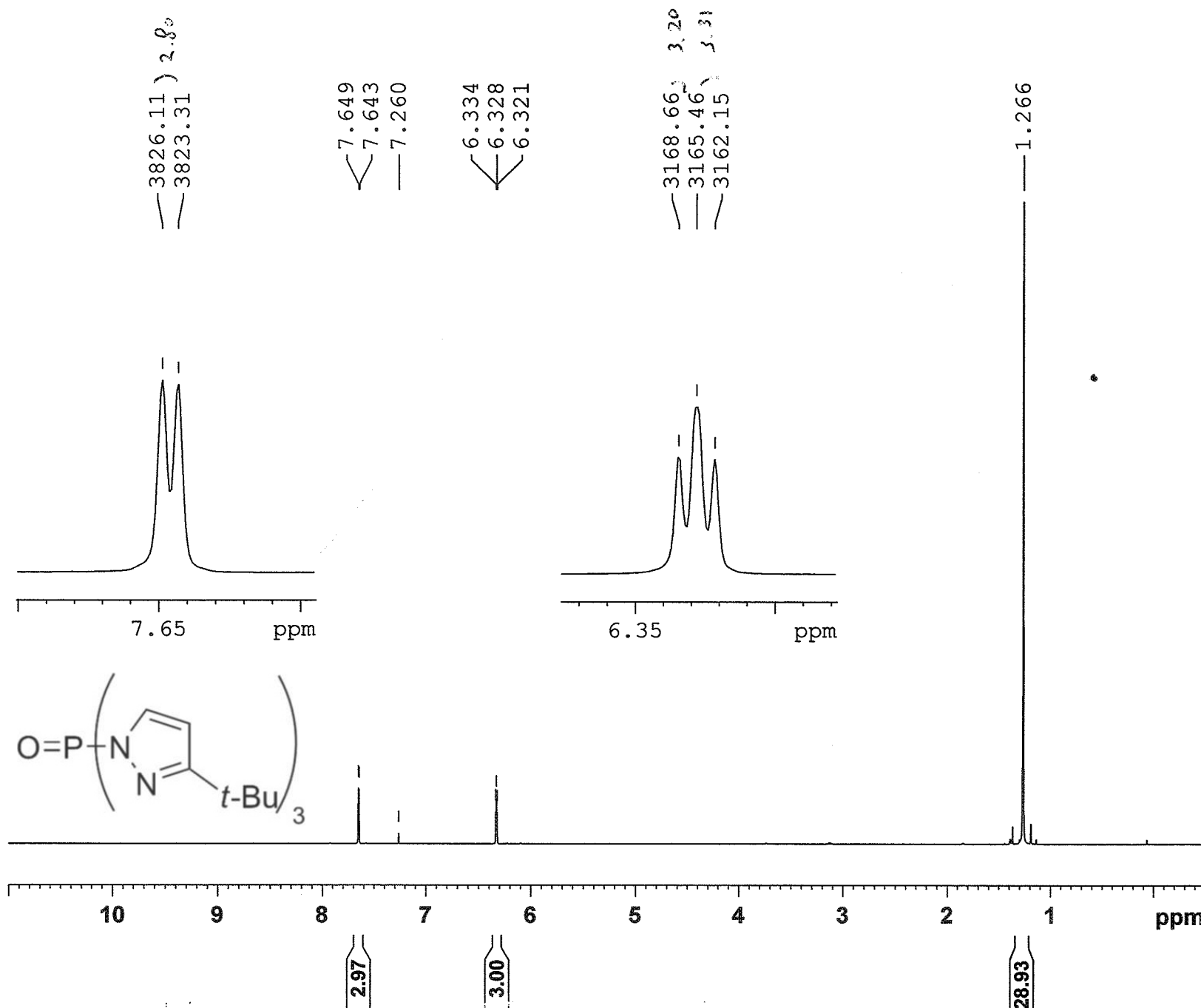
¹H NMR of OP(3-*t*-Bupz)₃ (**1c**)

(H, *t*-Bu-Pz)₃-PO



NAME VVL 20_500
EXPNO 1
PROCNO 1
Date_ 20100819
Time 11.20
INSTRUM spect
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719923 sec
RG 12.7
DW 48.400 usec
DE 6.50 usec
TE 296.0 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 6.70 usec
PL1 4.00 dB
PL1W 8.72000027 W
SFO1 500.2330891 MHz
SI 32768
SF 500.2300091 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



(H, t-Bu-Pz) 3-PO

¹H NMR of OP(3-*t*-Bupz)₃ (**1c**)
after line narrowing

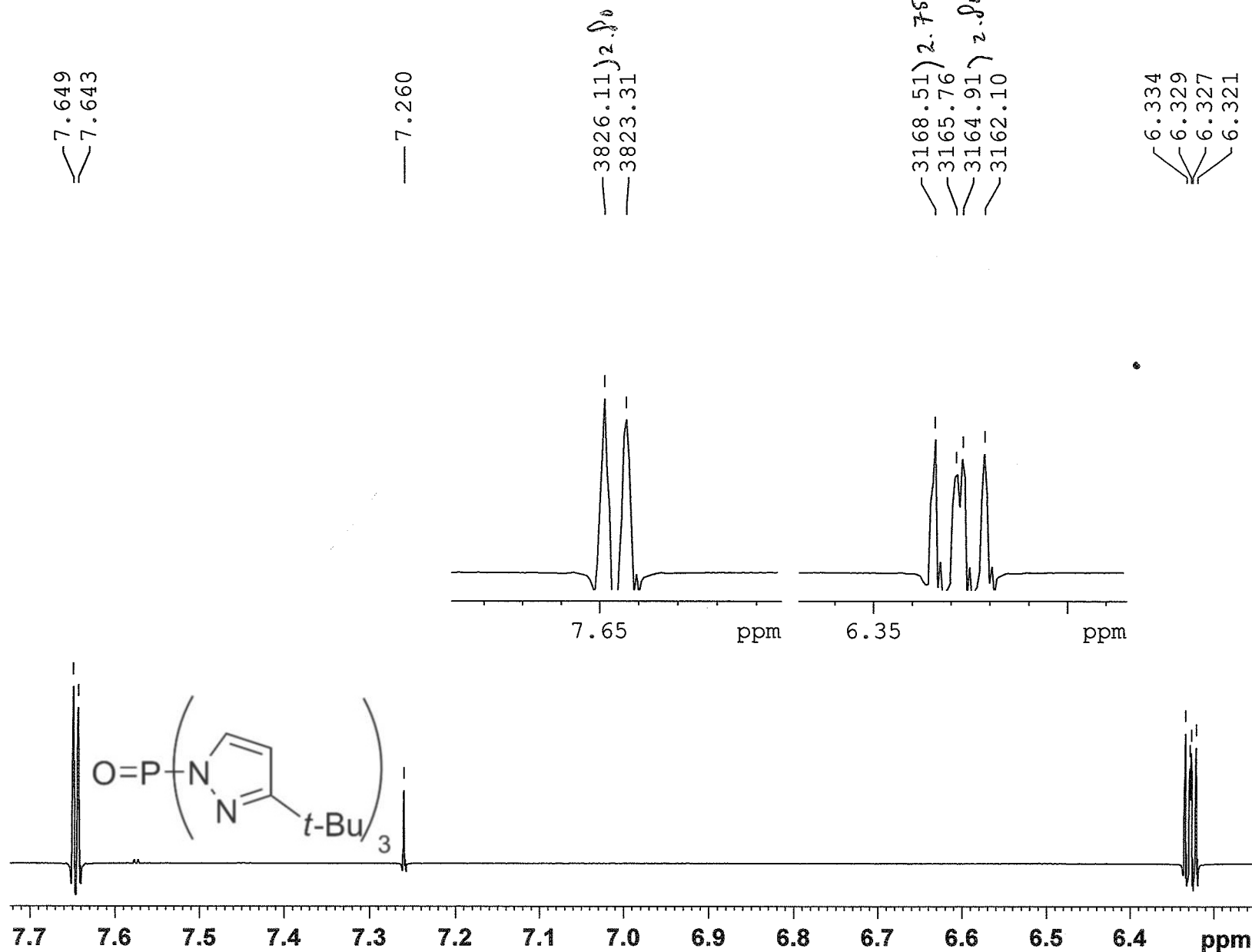


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NAME      VVL 20_500
EXPNO     1
PROCNO    2
Date_     20100819
Time      11.20
INSTRUM   spect
PROBHD    5 mm CPTCI 1H-
PULPROG   zg30
TD        65536
SOLVENT   CDCl3
NS        16
DS        2
SWH       10330.578 Hz
FIDRES    0.157632 Hz
AQ        3.1719923 sec
RG        12.7
DW        48.400 usec
DE        6.50 usec
TE        296.0 K
D1        1.00000000 sec
TD0       1
  
```

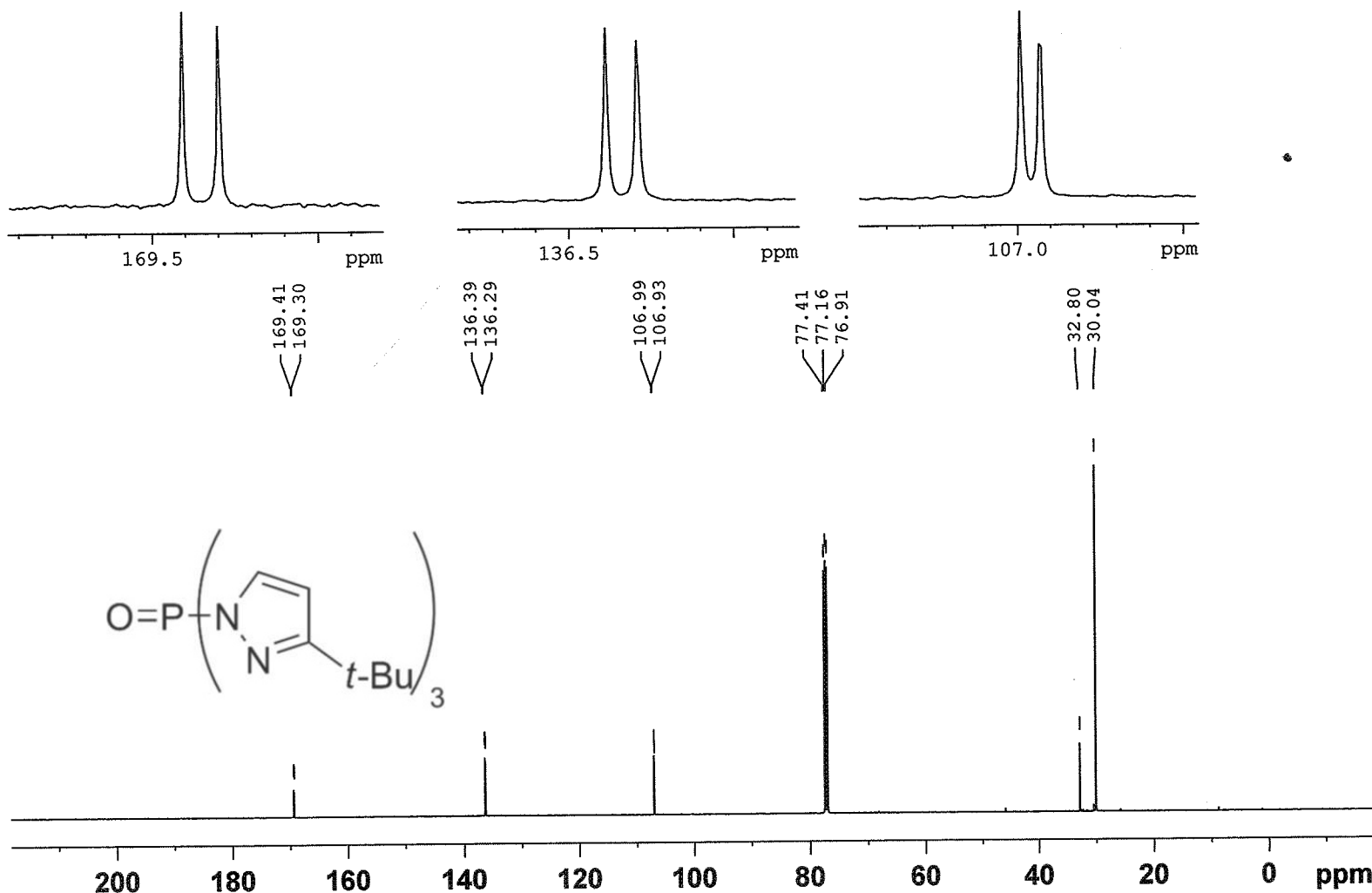
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===== CHANNEL f1 =====
NUC1      1H
P1        6.70 usec
PL1       4.00 dB
PL1W      8.72000027 W
SFO1      500.2330891 MHz
SI        32768
SF        500.2300091 MHz
WDW       GM
SSB       0
LB        -1.40 Hz
GB        0.5
PC        1.00
  
```



(H, t-Bu-Pz)3-PO

¹³C{¹H} NMR of OP(3-*t*-Bupz)₃ (1c)



NAME VVL 20_500
EXPNO 2
PROCNO 1
Date_ 20100819
Time_ 11.48
INSTRUM spect
PROBHD 5 mm CPTCI 1H-
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 512
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 2050
DW 16.800 usec
DE 6.50 usec
TE 296.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TDO 1

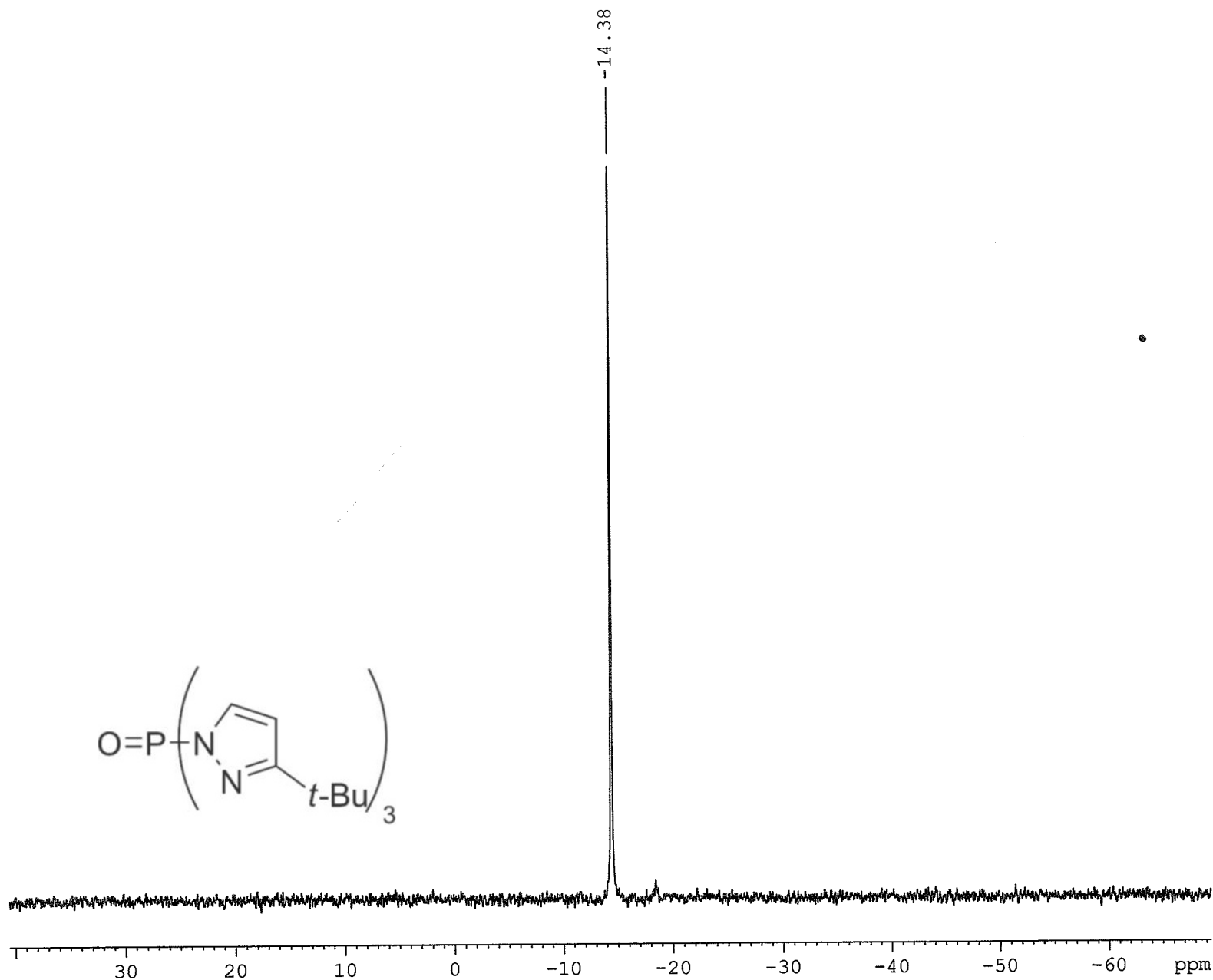
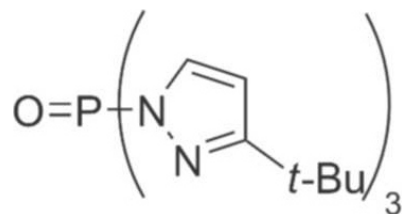
===== CHANNEL f1 =====
NUC1 13C
P1 11.20 usec
PL1 -2.00 dB
PL1W 88.77790070 W
SFO1 125.7955118 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 4.00 dB
PL12 25.28 dB
PL13 28.00 dB
PL2W 8.72000027 W
PL12W 0.06494062 W
PL13W 0.03471494 W
SFO2 500.2320009 MHz
SI 32768
SF 125.7829159 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

³¹P{¹H} NMR of OP(3-*t*-Bupz)₃ (**1c**)

(*t*-Bu, H-Pz) 3PO

P31C7D CDCl₃ {D:\NMRDATA} Volodymyr 58



Current Data Parameters
NAME VVL 439
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
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Time_ 15.24
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PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 65536
SOLVENT CDCl₃
NS 32
DS 4
SWH 40650.406 Hz
FIDRES 0.620276 Hz
AQ 0.8061428 sec
RG 6502
DW 12.300 usec
DE 6.00 usec
TE 300.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

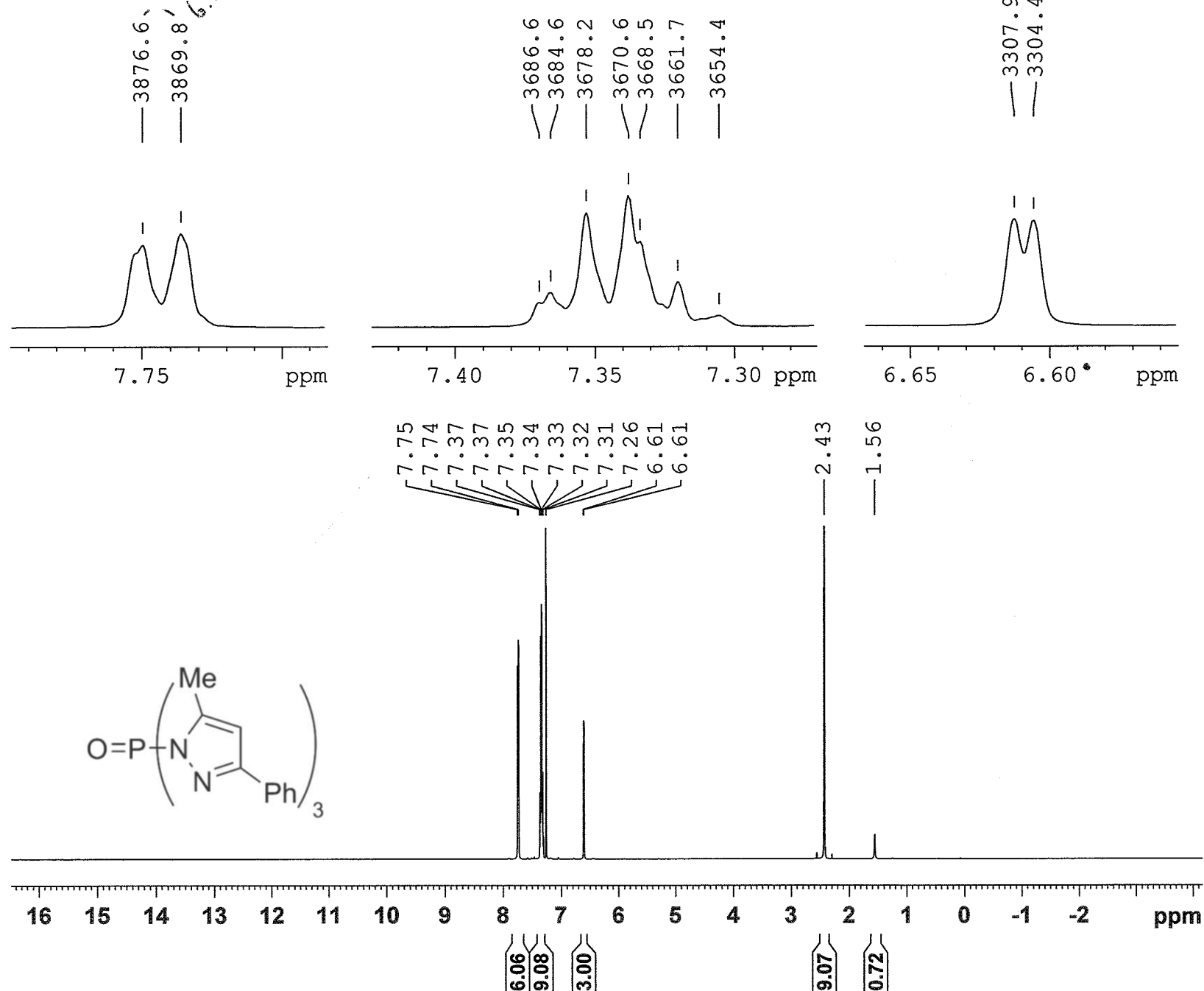
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NUC1 31P
P1 7.00 usec
PL1 0.00 dB
SFO1 101.2494172 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -3.00 dB
PL12 20.00 dB
PL13 24.00 dB
SFO2 250.1310005 MHz

F2 - Processing parameters
SI 32768
SF 101.2544800 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40

¹H NMR of OP(3-Ph-5-Mepz)₃ (**1d**)

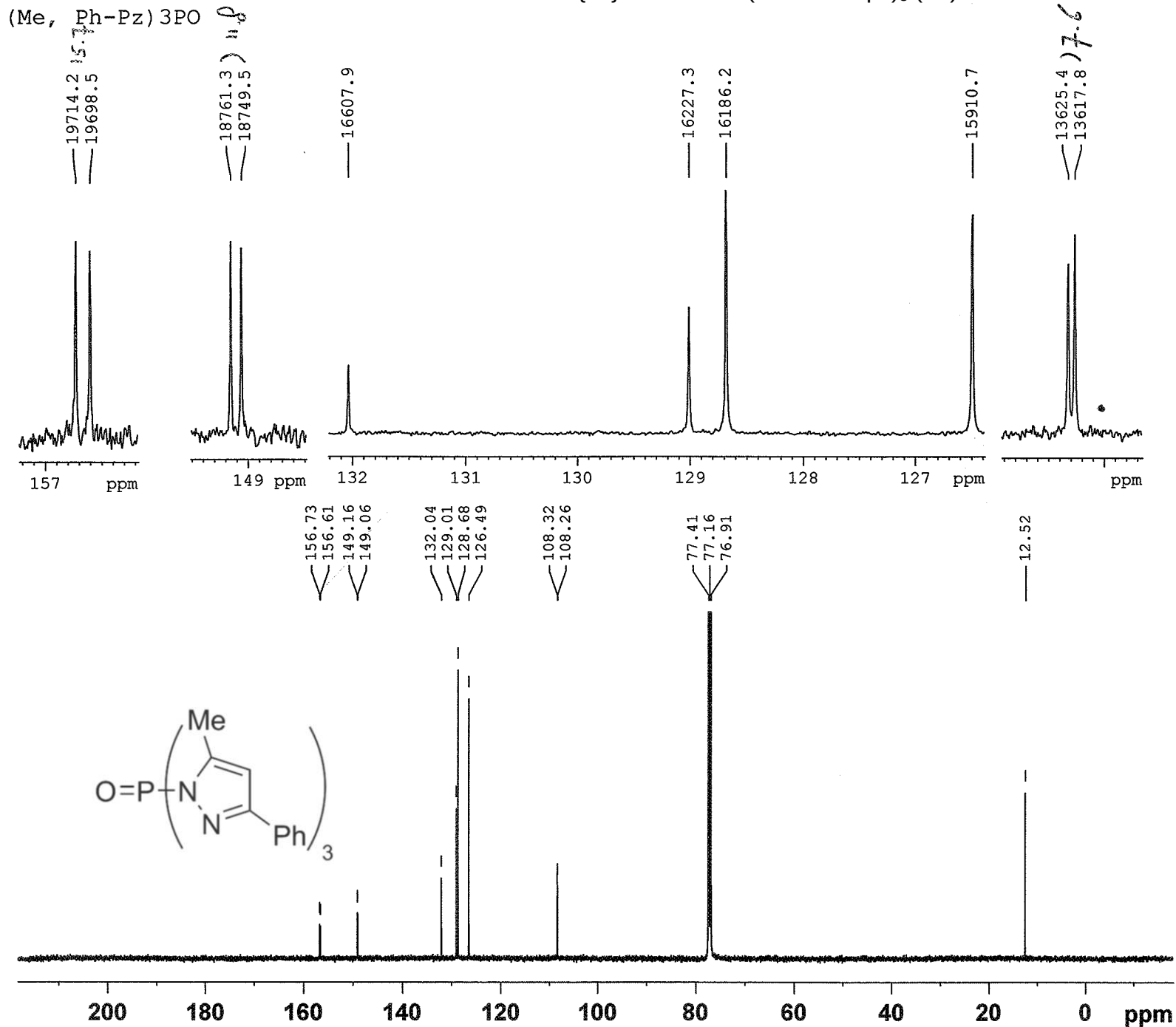
(Me, Ph-Pz) 3PO



NAME VVL 21_500
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PROCNO 1
Date_ 20100819
Time 12.57
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PULPROG zg30
TD 65536
SOLVENT CDC13
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719923 sec
RG 14.2
DW 48.400 usec
DE 6.50 usec
TE 296.1 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 6.70 usec
PL1 4.00 dB
PL1W 8.72000027 W
SFO1 500.2330891 MHz
SI 32768
SF 500.2300091 MHz
WDW EM
SSB 0
LB 0.50 Hz
GB 0
PC 1.00

$^{13}\text{C}\{^1\text{H}\}$ NMR of $\text{OP}(\text{3-Ph-5-Mepz})_3$ (**1d**)



```

NAME      VVL 21_500
EXPNO     2
PROCNO    1
Date_     20100819
Time      13.25
INSTRUM   spect
PROBHD    5 mm CPTCI 1H-
PULPROG   zgpg30
TD        65536
SOLVENT   CDCl3
NS        512
DS        4
SWH       29761.904 Hz
FIDRES    0.454131 Hz
AQ        1.1010548 sec
RG        2050
DW        16.800 usec
DE        6.50 usec
TE        295.9 K
D1        2.0000000 sec
D11       0.03000000 sec
TDO       1
  
```

```

===== CHANNEL f1 =====
NUC1      13C
P1        11.20 usec
PL1       -2.00 dB
PL1W      88.77790070 W
SFO1      125.7955118 MHz
  
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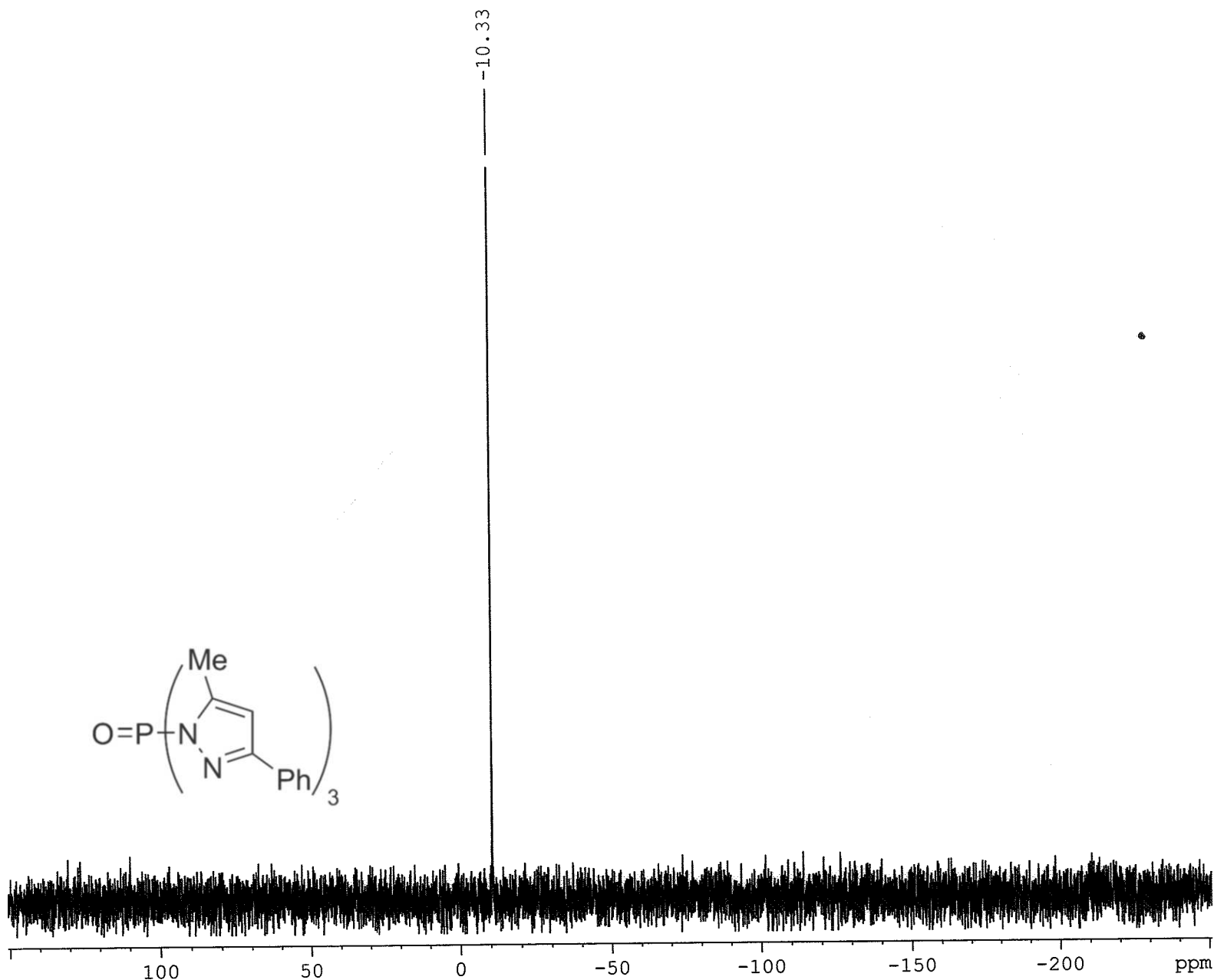
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===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2      1H
PCPD2     80.00 usec
PL2       4.00 dB
PL12      25.28 dB
PL13      28.00 dB
PL2W      8.72000027 W
PL12W     0.06494062 W
PL13W     0.03471494 W
SFO2      500.2320009 MHz
SI        32768
SF        125.7829163 MHz
WDW       EM
SSB       0
LB        1.00 Hz
GB        0
PC        1.40
  
```

³¹P{¹H} NMR of OP(3-Ph-5-Mepz)₃ (**1d**)

(me, Ph-Pz)3PO

101010 CDCl3 {D:\NMRDATA} Volodymyr 59



Current Data Parameters
NAME VVL 440
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20100819
Time_ 15.31
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PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 32
DS 4
SWH 40650.406 Hz
FIDRES 0.620276 Hz
AQ 0.8061428 sec
RG 20642.5
DW 12.300 usec
DE 6.00 usec
TE 300.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

===== CHANNEL f1 =====
NUC1 31P
P1 7.00 usec
PL1 0.00 dB
SFO1 101.2494172 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -3.00 dB
PL12 20.00 dB
PL13 24.00 dB
SFO2 250.1310005 MHz

F2 - Processing parameters
SI 32768
SF 101.2544800 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40

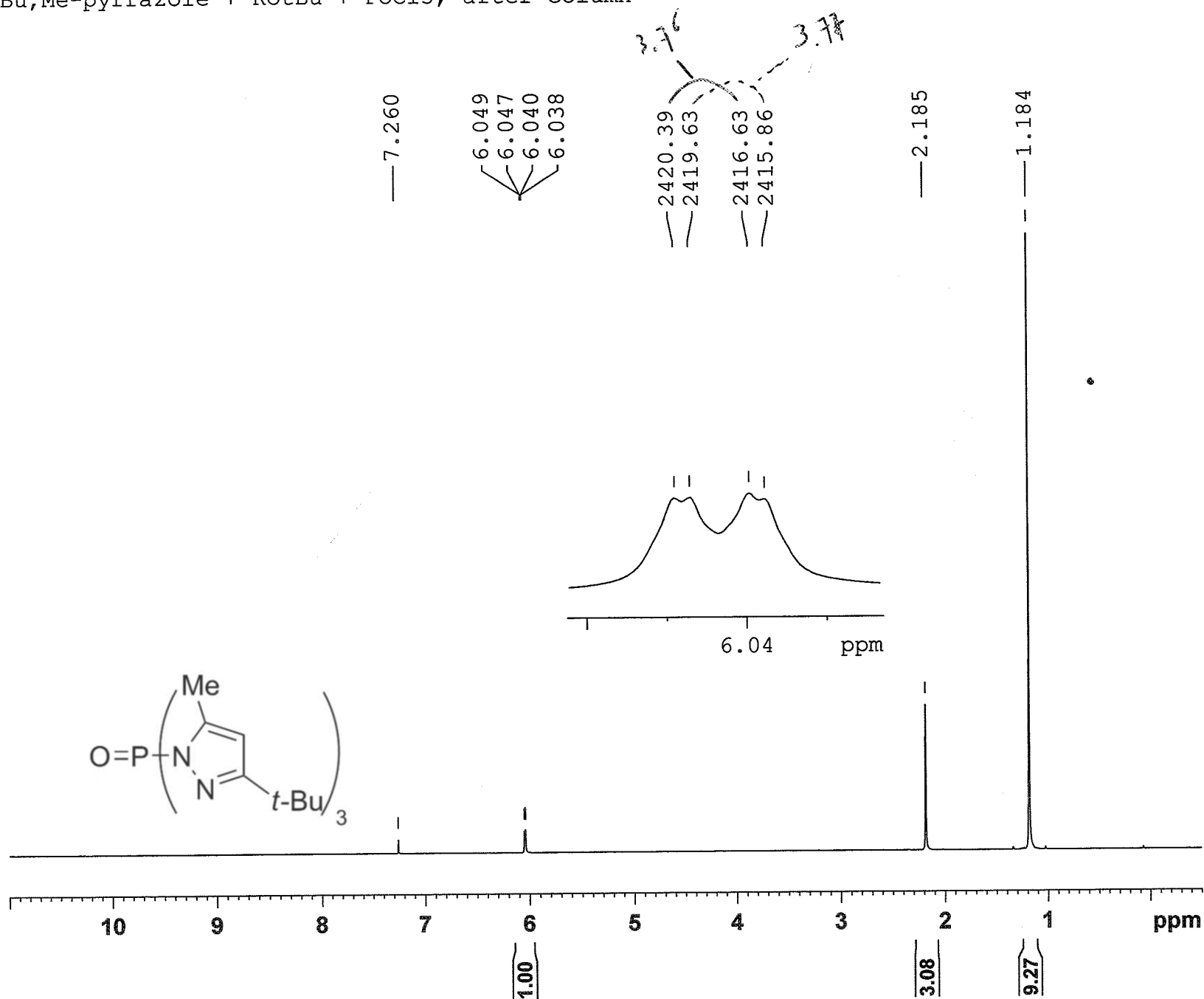
¹H NMR of OP(3-*t*-Bu-5-Mepz)₃ (**1e**)

*t*Bu,Me-pyrrazole + KO^tBu + POCl₃, after column

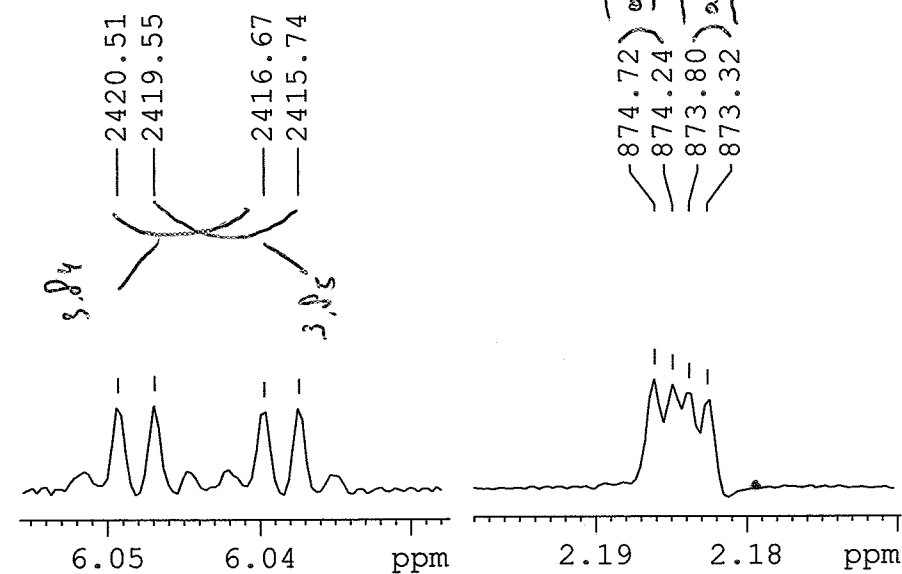


NAME VVL 315_400
EXPNO 1
PROCNO 1
Date_ 20100812
Time_ 16.03
INSTRUM spect
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PULPROG zg30
TD 65536
SOLVENT CDCl₃
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 143.7
DW 62.400 usec
DE 6.00 usec
TE 300.4 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 13.00 usec
PL1 3.00 dB
SFO1 400.1330010 MHz
SI 65536
SF 400.1300091 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

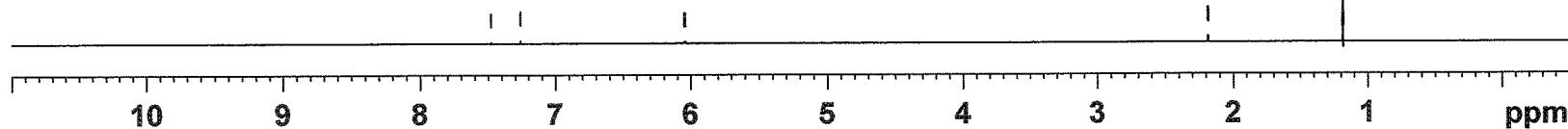
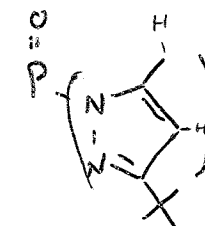
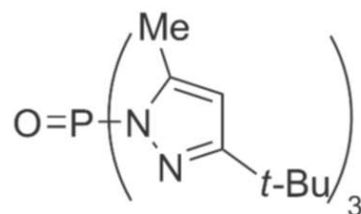


tBu,Me-pyrrazole + KOtBu + POCl₃, after ----- with line narrowing



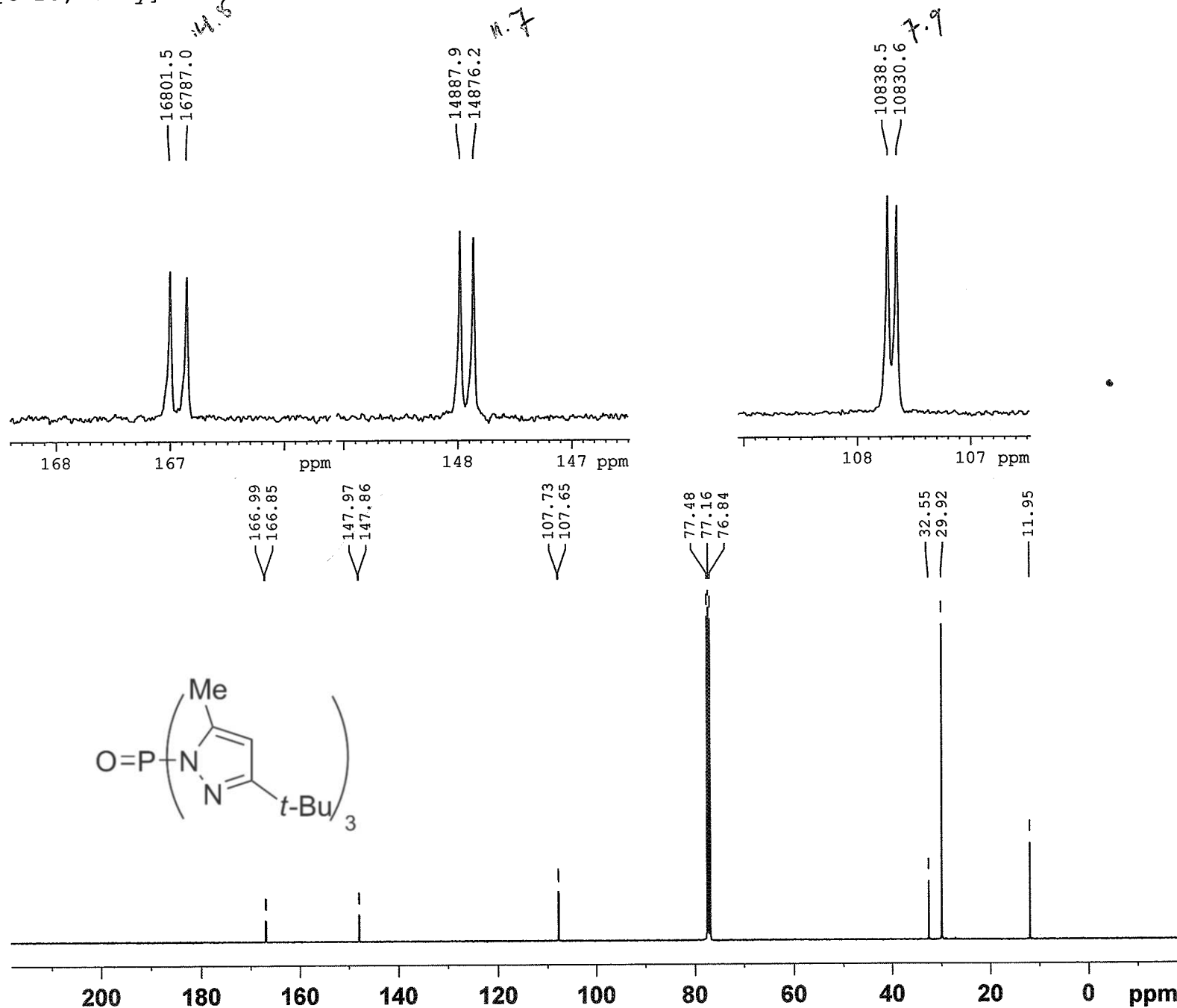
NAME VVL 315_400
 EXPNO 1
 PROCNO 2
 Date_ 20100812
 Time_ 16.03
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CDCl₃
 NS 16
 DS 2
 SWH 8012.820 Hz
 FIDRES 0.122266 Hz
 AQ 4.0894966 sec
 RG 143.7
 DW 62.400 usec
 DE 6.00 usec
 TE 300.4 K
 D1 1.00000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 13.00 usec
 PL1 3.00 dB
 SFO1 400.1330010 MHz
 SI 65536
 SF 400.1300091 MHz
 WDW GM
 SSB 0
 LB -1.40 Hz
 GB 0.6
 PC 1.00



¹³C{¹H} NMR of OP(3-*t*-Bu-5-Mepz)₃ (**1e**)

[*t*-Bu, Me-Py] 3PO



NAME VVL 315_400
 EXPNO 5
 PROCNO 1
 Date_ 20100813
 Time_ 2.55
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDC13
 NS 10240
 DS 4
 SWH 23980.814 Hz
 FIDRES 0.365918 Hz
 AQ 1.3664756 sec
 RG 812.7
 DW 20.850 usec
 DE 6.00 usec
 TE 300.4 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 DELTA 1.89999998 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 13C
 P1 8.80 usec
 PL1 -1.00 dB
 SFO1 100.6228298 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 3.00 dB
 PL12 18.00 dB
 PL13 18.00 dB
 SFO2 400.1316005 MHz
 SI 32768
 SF 100.6127528 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

³¹P{¹H} NMR of OP(3-*t*-Bu-5-Mepz)₃ (**1e**)

[*t*-Bu, Me-Py] 3PO



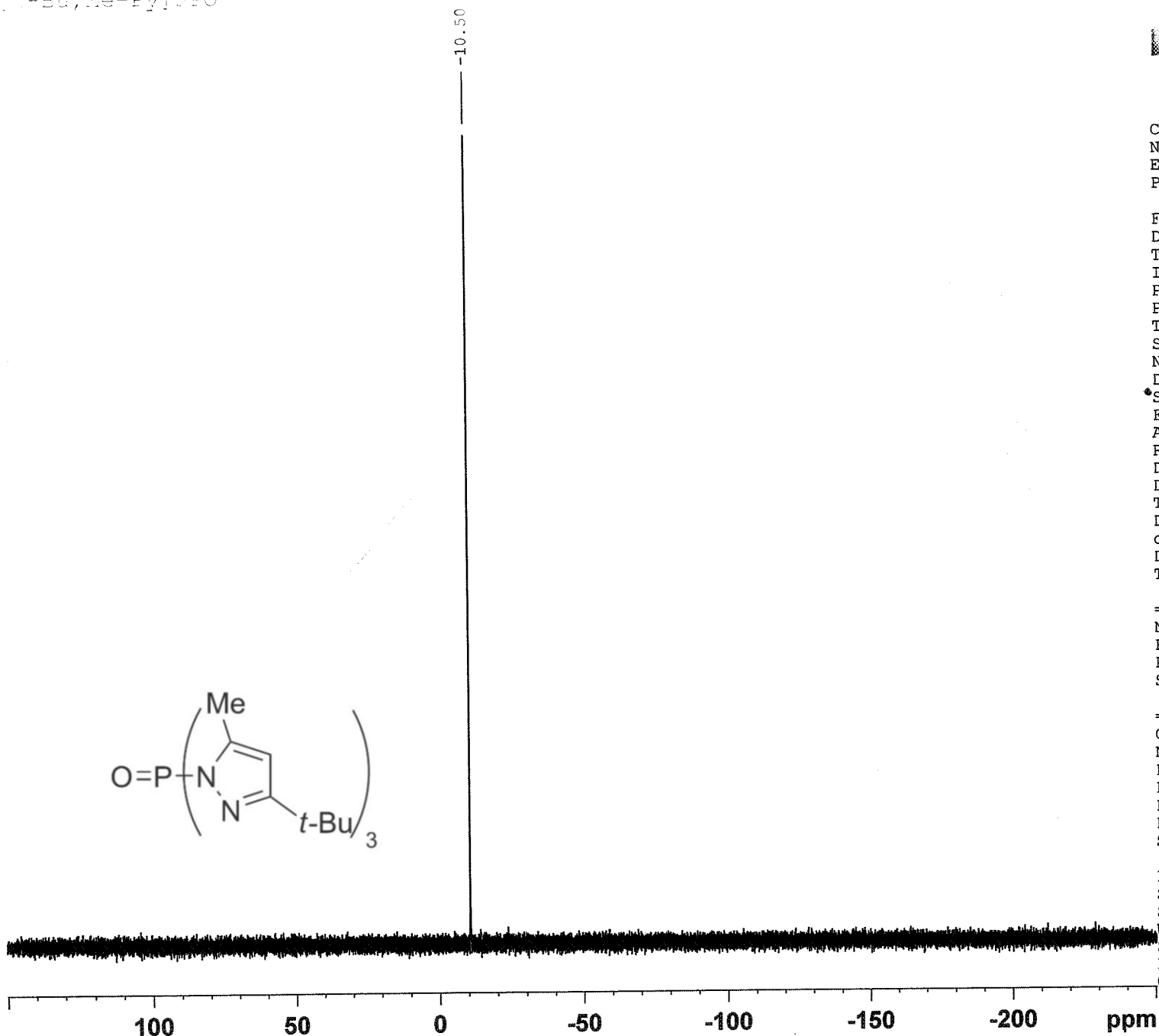
Current Data Parameters
NAME VVL 315_400
EXPNO 8
PROCNO 1

F2 - Acquisition Parameters
Date_ 20100813
Time_ 7.03
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 16
DS 4
*SWH 64935.066 Hz
FIDRES 0.990830 Hz
AQ 0.5046772 sec
RG 20642.5
DW 7.700 usec
DE 6.00 usec
TE 300.4 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

===== CHANNEL f1 =====
NUC1 31P
P1 6.25 usec
PL1 -1.00 dB
SFO1 161.9674942 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 3.00 dB
PL12 18.00 dB
PL13 18.00 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 65536
SF 161.9755930 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

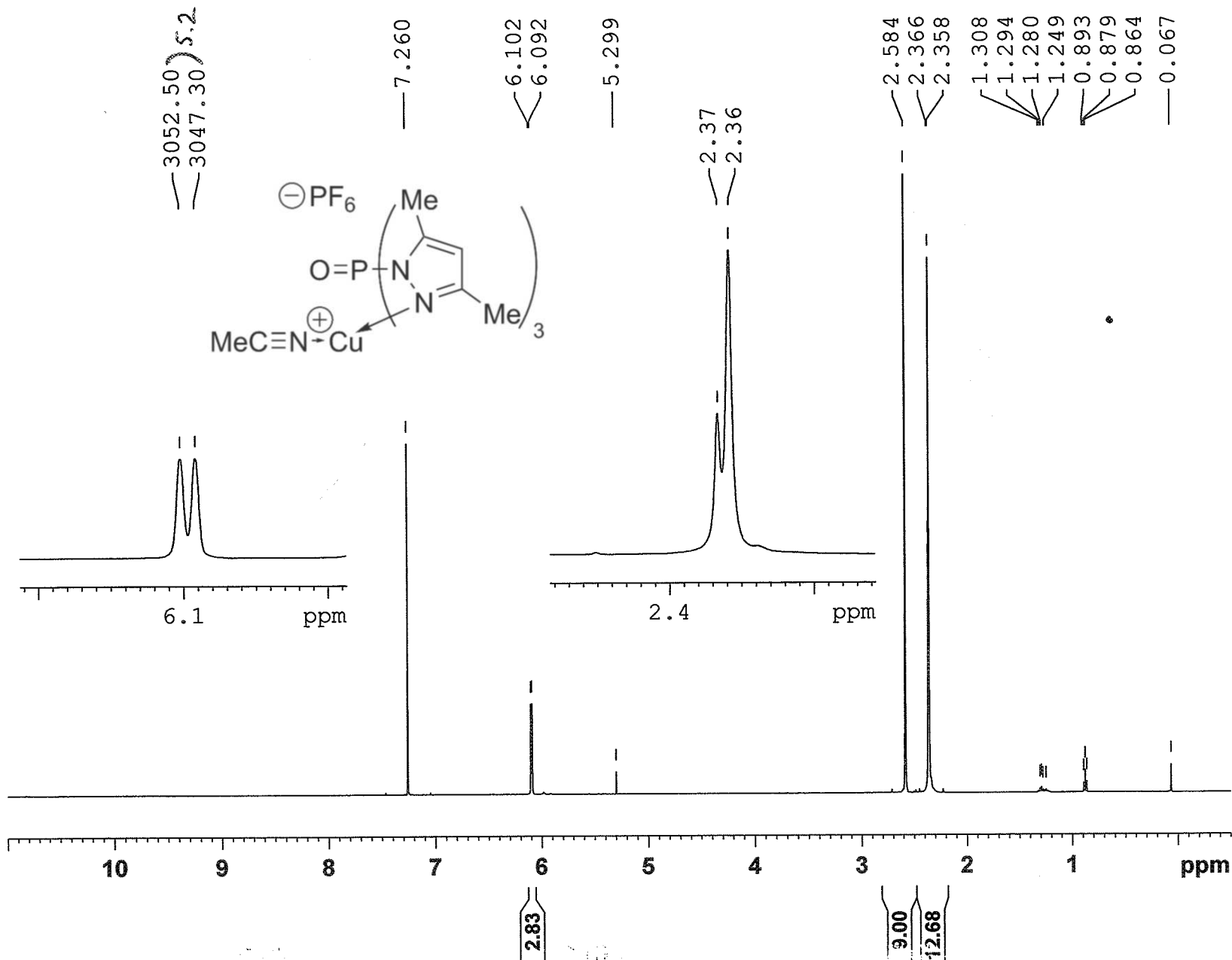


¹H NMR of [OP(3,5-Me₂pz)₃Cu(NCMe)][PF₆] (2a)

¹H (CDCl₃ GB)

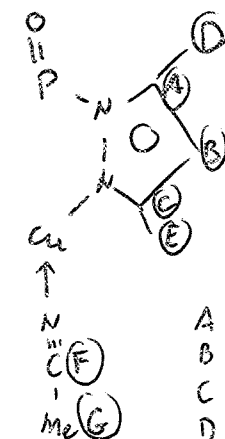
OP(pzMe₂)₃CuNCMe: Compound w. 1eq. MeCN

-Sample (schlenk w+), after night in GB. Colorless susp.-



NAME CGJT-10-23_500
 EXPNO 9
 PROCNO 1
 Date_ 20110520
 Time_ 10.35
 INSTRUM spect
 PROBHD 5 mm CPTCI 1H-
 PULPROG zg30
 TD 65536
 SOLVENT CDCl₃
 NS 8
 DS 2
 SWH 10000.000 Hz
 FIDRES 0.152588 Hz
 AQ 3.2768500 sec
 RG 18
 DW 50.000 usec
 DE 6.50 usec
 TE 296.0 K
 D1 1.70000005 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 6.70 usec
 PL1 4.00 dB
 PL1W 8.72000027 W
 SFO1 500.2335016 MHz
 SI 65536
 SF 500.2300092 MHz
 WDW EM
 SSB 0
 LB 0.10 Hz
 GB 0
 PC 1.00



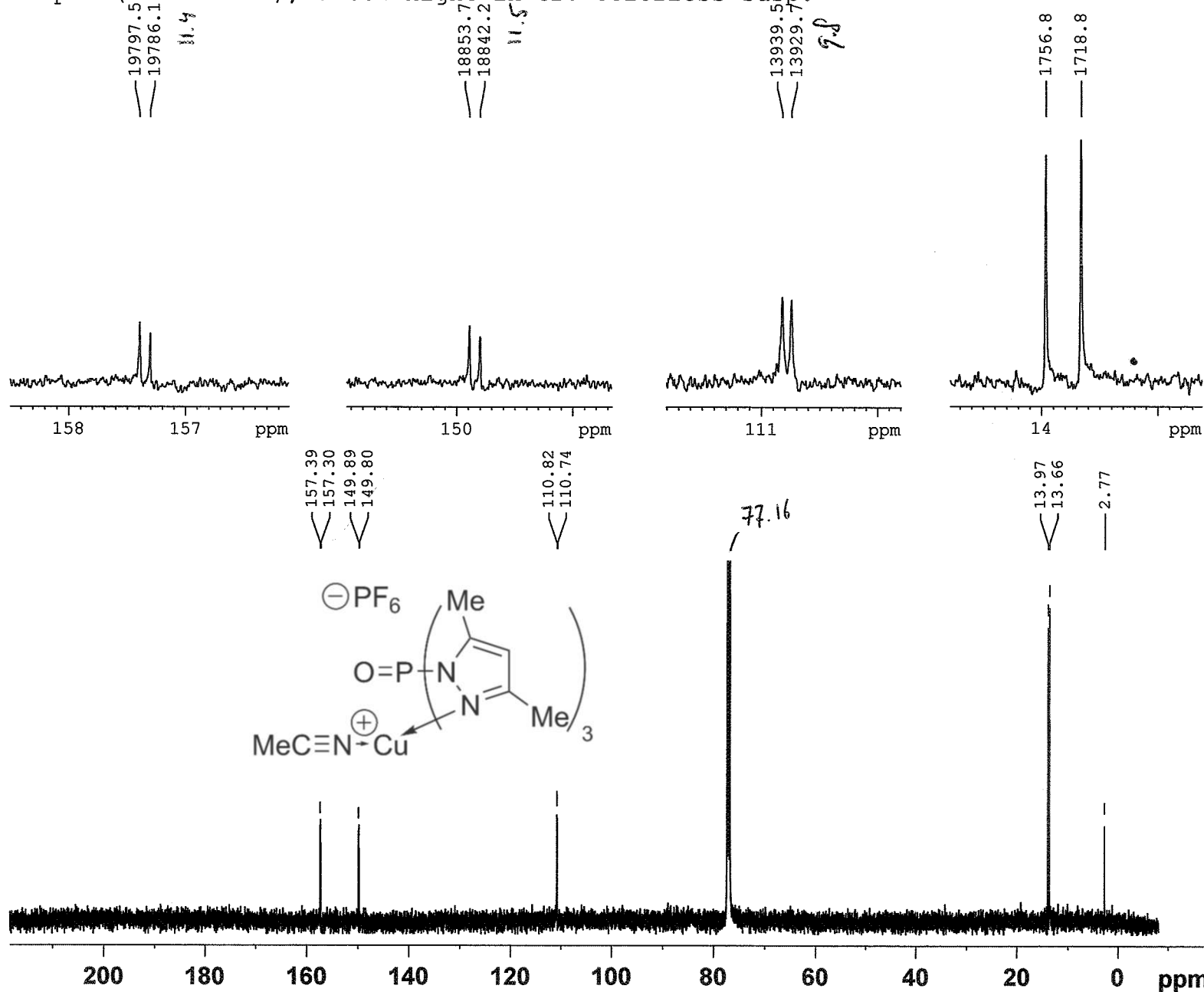
	C	H
A	149.8	→ Me ₂ O
B	110.8	6.10
C	157.3	→ Me ₂ E
D	137.7	2.58
E	137.0	2.36
F	117.3	← From H ₂ O
G	2.77	2.37

$^{13}\text{C}\{^1\text{H}\}$ NMR of $[\text{OP}(3,5\text{-Me}_2\text{pz})_3\text{Cu}(\text{NCMe})][\text{PF}_6]$ (**2a**)

$^{13}\text{C}\{^1\text{H}\}$ (CDCl₃ GB)

OP(pzMe₂)₃CuNCMe: Compound w. 1eq. MeCN

-Sample (schlenk w+), after night in GB. Colorless susp.-

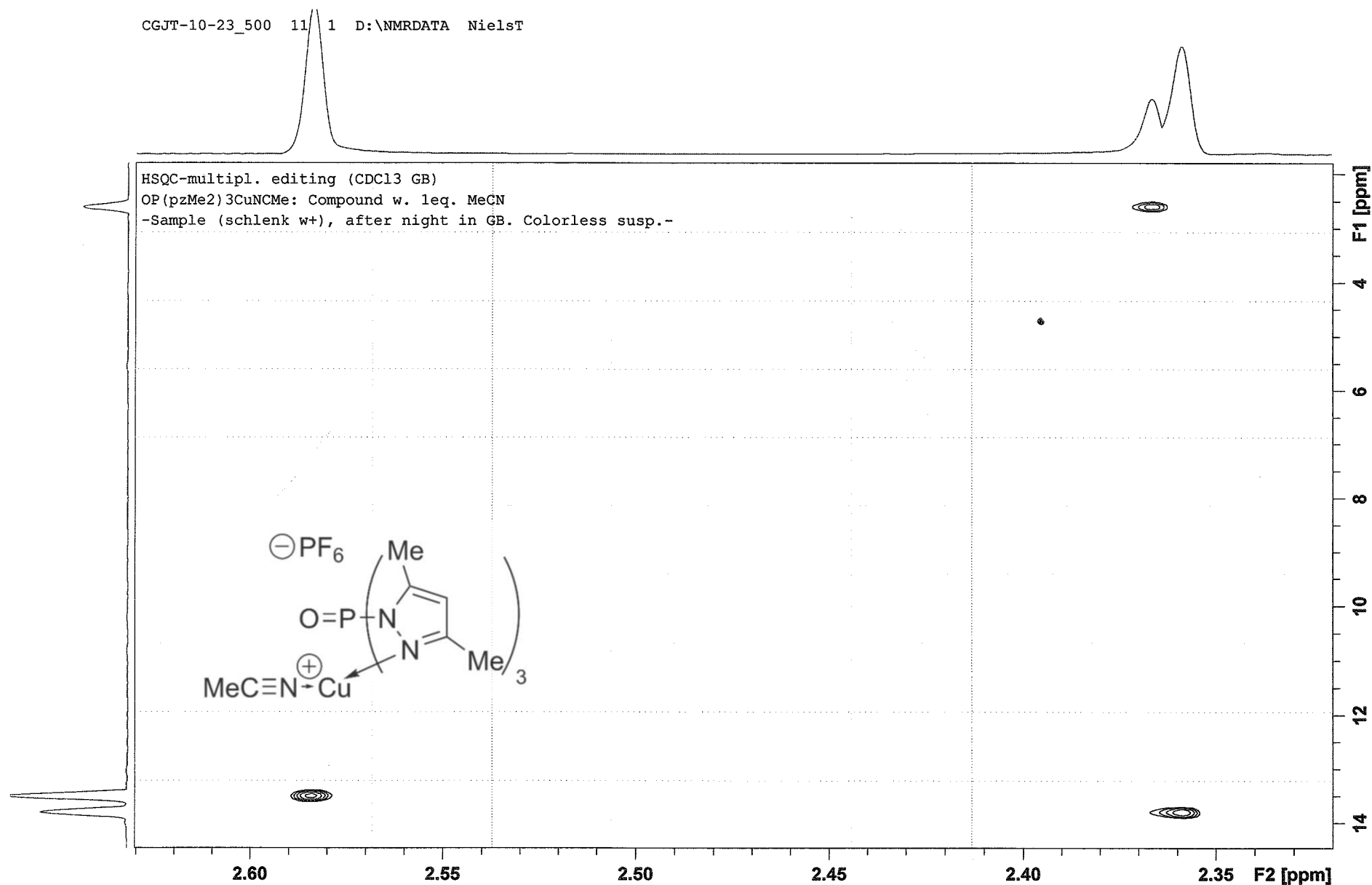


NAME CGJT-10-23_500
EXPNO 10
PROCNO 1
Date_ 20110520
Time_ 10.38
INSTRUM spect
PROBHD 5 mm CPTCI 1H-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 234
DS 2
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 2050
DW 16.800 usec
DE 6.50 usec
TE 296.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TDO 1

===== CHANNEL f1 =====
NUC1 13C
P1 11.20 usec
PL1 -2.00 dB
PL1W 88.77790070 W
SFO1 125.7967701 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 4.00 dB
PL12 25.28 dB
PL13 28.00 dB
PL2W 8.72000027 W
PL12W 0.06494062 W
PL13W 0.03471494 W
SFO2 500.2320009 MHz
SI 65536
SF 125.7829148 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

^1H - ^{13}C HSQC NMR of $[\text{OP}(3,5\text{-Me}_2\text{pz})_3\text{Cu}(\text{NCMe})][\text{PF}_6]$ (**2a**)



³¹P{¹H} NMR of [OP(3,5-Me₂pz)₃Cu(NCMe)][PF₆] (2a)

³¹P{¹H} (CDCl₃ GB)

exp A: OP(pz-Me,Me)₃CuNCMe PF₆, solid from schlenk W+

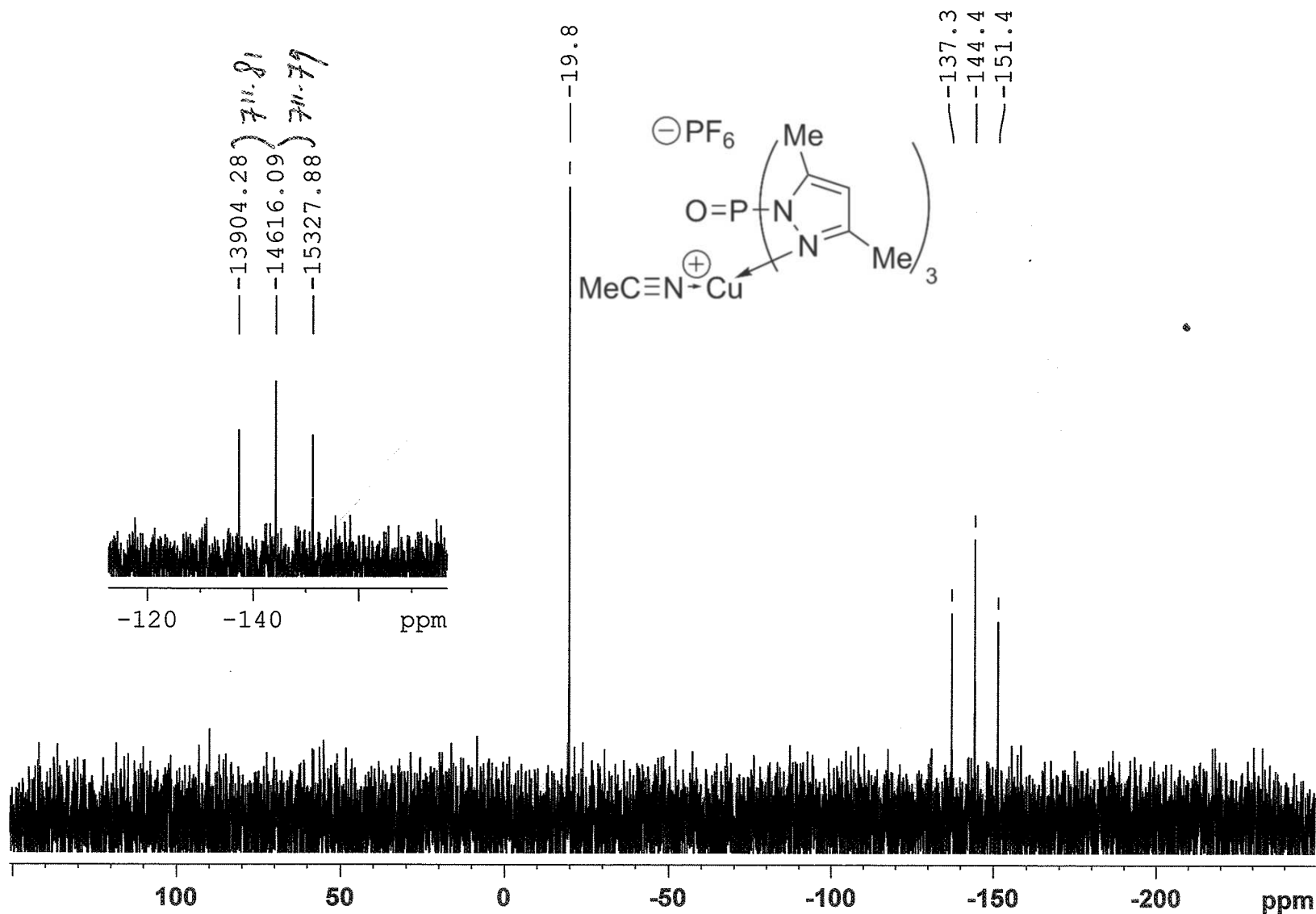
31PCPD_NO_processing CDCl₃ {D:\NMRDATA} Nielst 56



NAME CGJT-10-23
EXPNO 5
PROCNO 1
Date_ 20101223
Time 11.51
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 65536
SOLVENT CDCl₃
NS 64
DS 4
SWH 40650.406 Hz
FIDRES 0.620276 Hz
AQ 0.8061428 sec
RG 6502
DW 12.300 usec
DE 6.00 usec
TE 300.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

===== CHANNEL f1 =====
NUC1 31P
P1 7.00 usec
PL1 0.00 dB
SFO1 101.2494172 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -3.00 dB
PL12 20.00 dB
PL13 24.00 dB
SFO2 250.1310005 MHz
SI 65536
SF 101.2544800 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40



¹⁹F NMR of [OP(3,5-Me₂pz)₃Cu(NCMe)][PF₆] (2a)

¹⁹F (CDC13 GB)

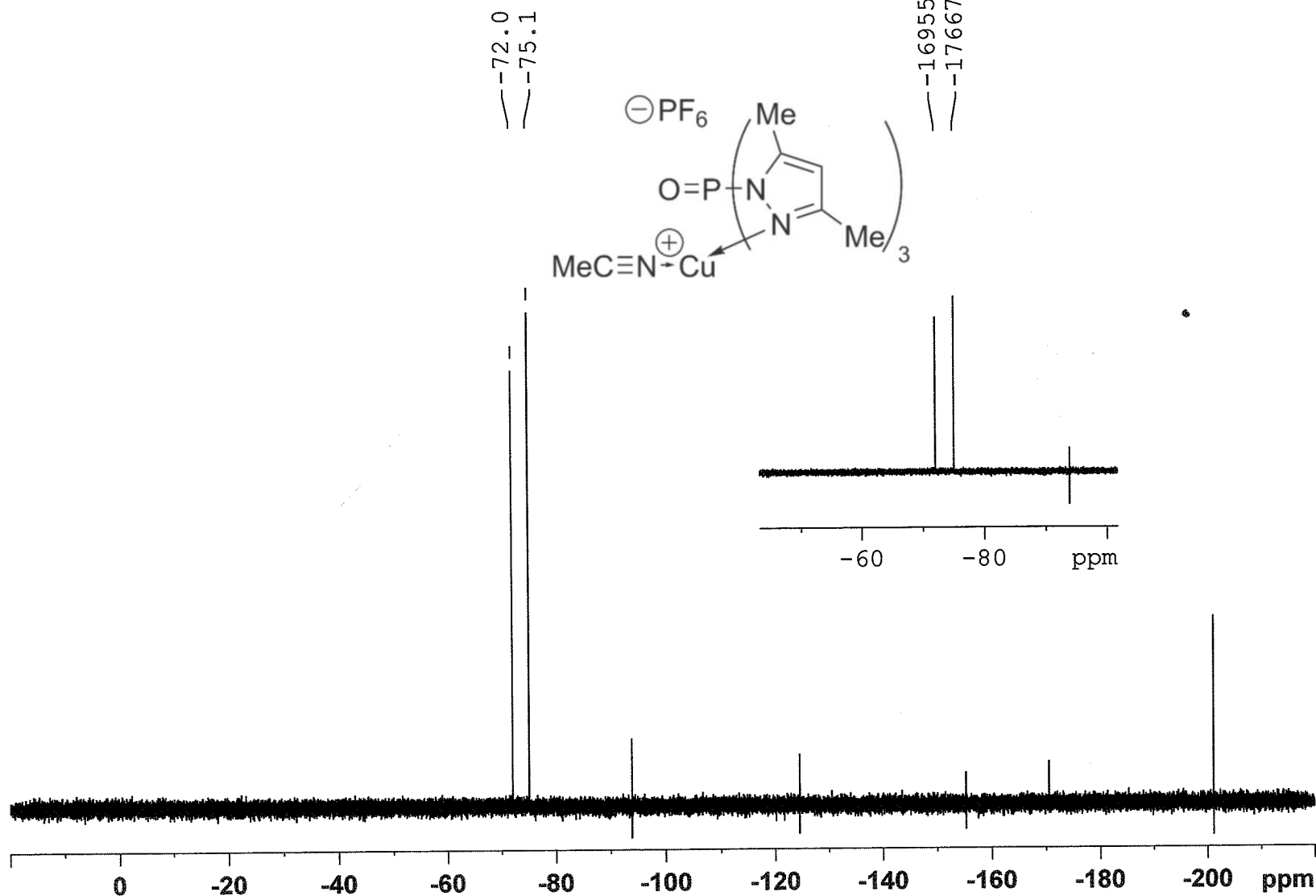
exp A: OP(pz-Me,Me)₃CuNCMe PF₆, solid from schlenk W+

F19 CDC13 {D:\NMRDATA} NielsT 56



NAME CGJT-10-23
EXPNO 6
PROCNO 1
Date_ 20101223
Time_ 11.54
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 131072
SOLVENT CDC13
NS 64
DS 4
SWH 56497.176 Hz
FIDRES 0.431039 Hz
AQ 1.1600372 sec
RG 23170.5
DW 8.850 usec
DE 6.00 usec
TE 300.2 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 19F
P1 8.00 usec
PL1 -3.00 dB
SFO1 235.3338140 MHz
SI 65536
SF 235.3573500 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



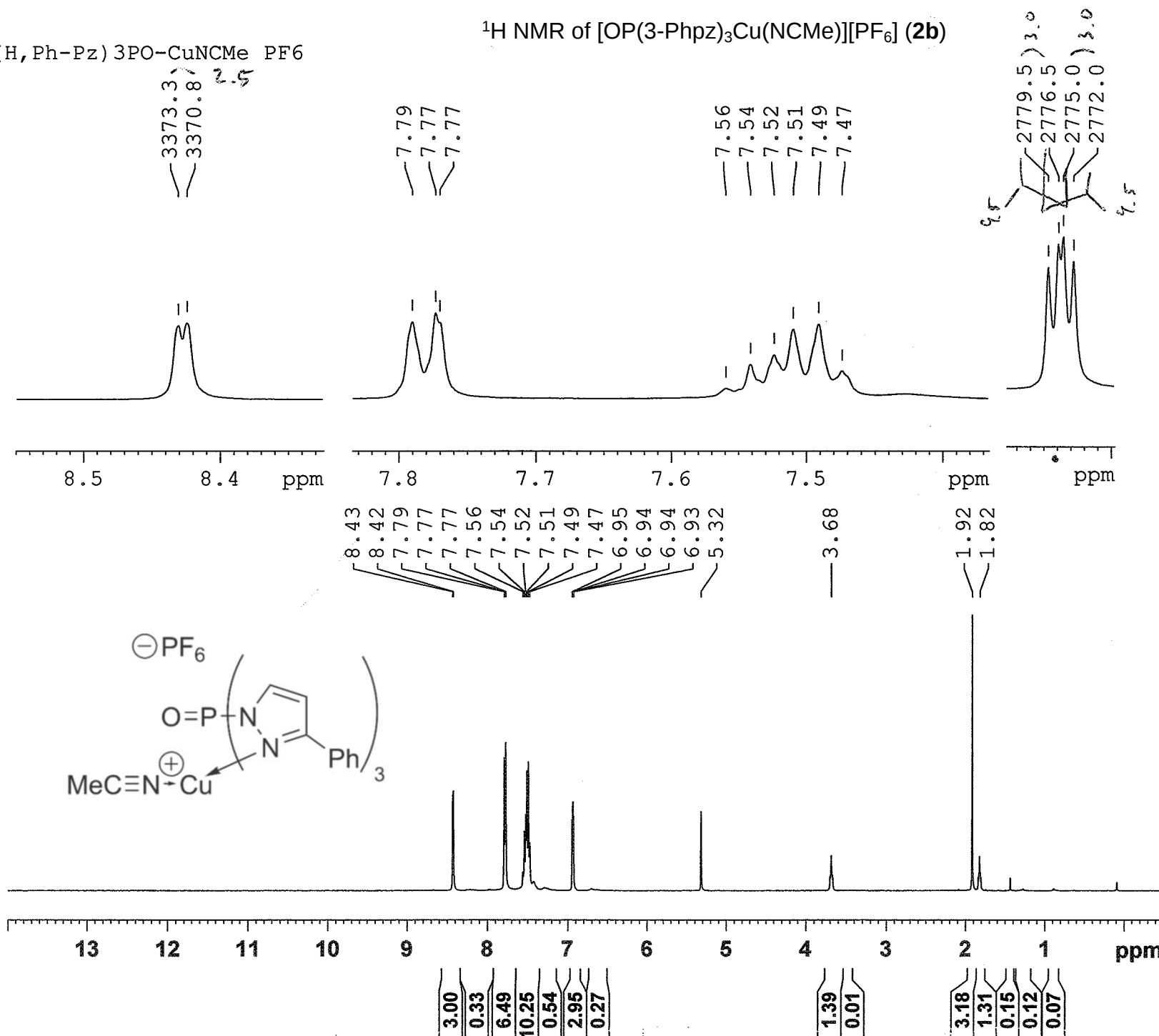
(H, Ph-Pz) 3PO-CuNCMe PF₆

¹H NMR of [OP(3-Phpz)₃Cu(NCMe)][PF₆] (2b)



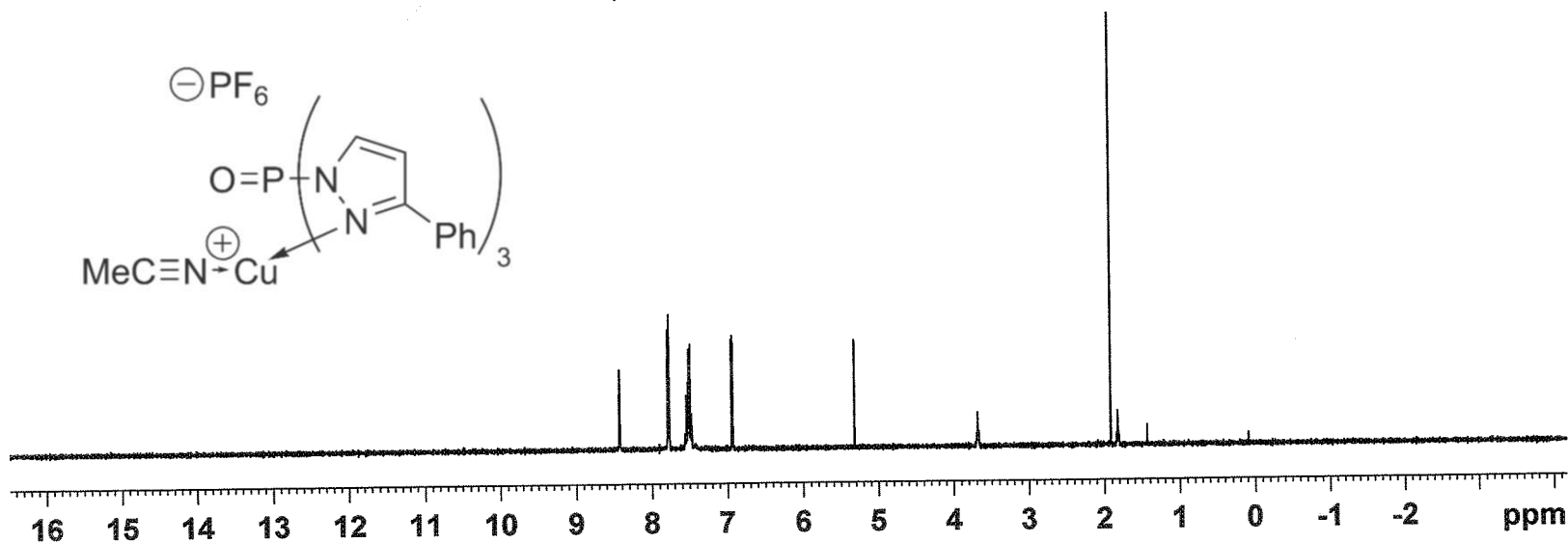
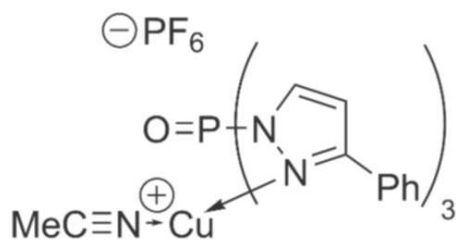
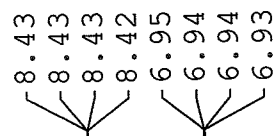
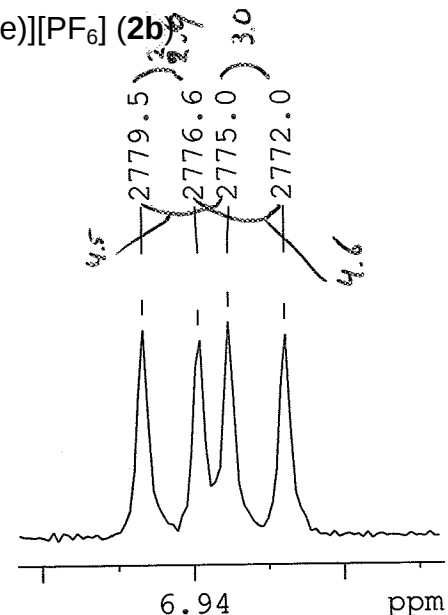
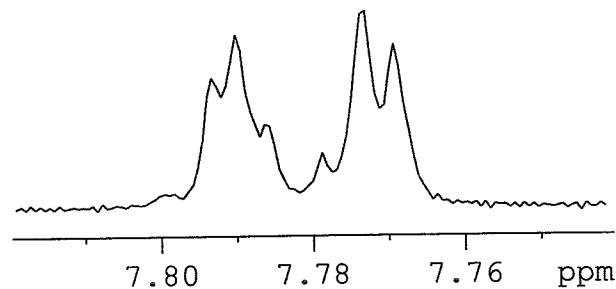
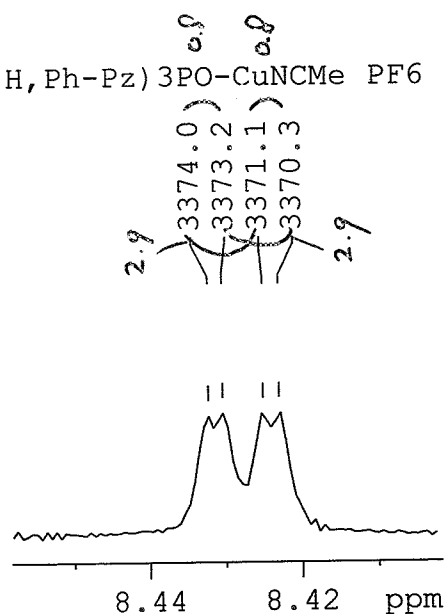
NAME VVL 331_400
 EXPNO 1
 PROCNO 1
 Date_ 20100828
 Time_ 10.36
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CD₂Cl₂
 NS 16
 DS 2
 SWH 8278.146 Hz
 FIDRES 0.126314 Hz
 AQ 3.9584243 sec
 RG 322.5
 DW 60.400 usec
 DE 6.00 usec
 TE 300.4 K
 D1 1.00000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 13.00 usec
 PL1 3.00 dB
 SFO1 400.1324710 MHz
 SI 32768
 SF 400.1300149 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



(H, Ph-Pz) 3PO-CuNCMe PF6

^1H NMR of $[\text{OP}(\text{3-Phpz})_3\text{Cu}(\text{NCMe})][\text{PF}_6]$ (**2b**)
after line narrowing



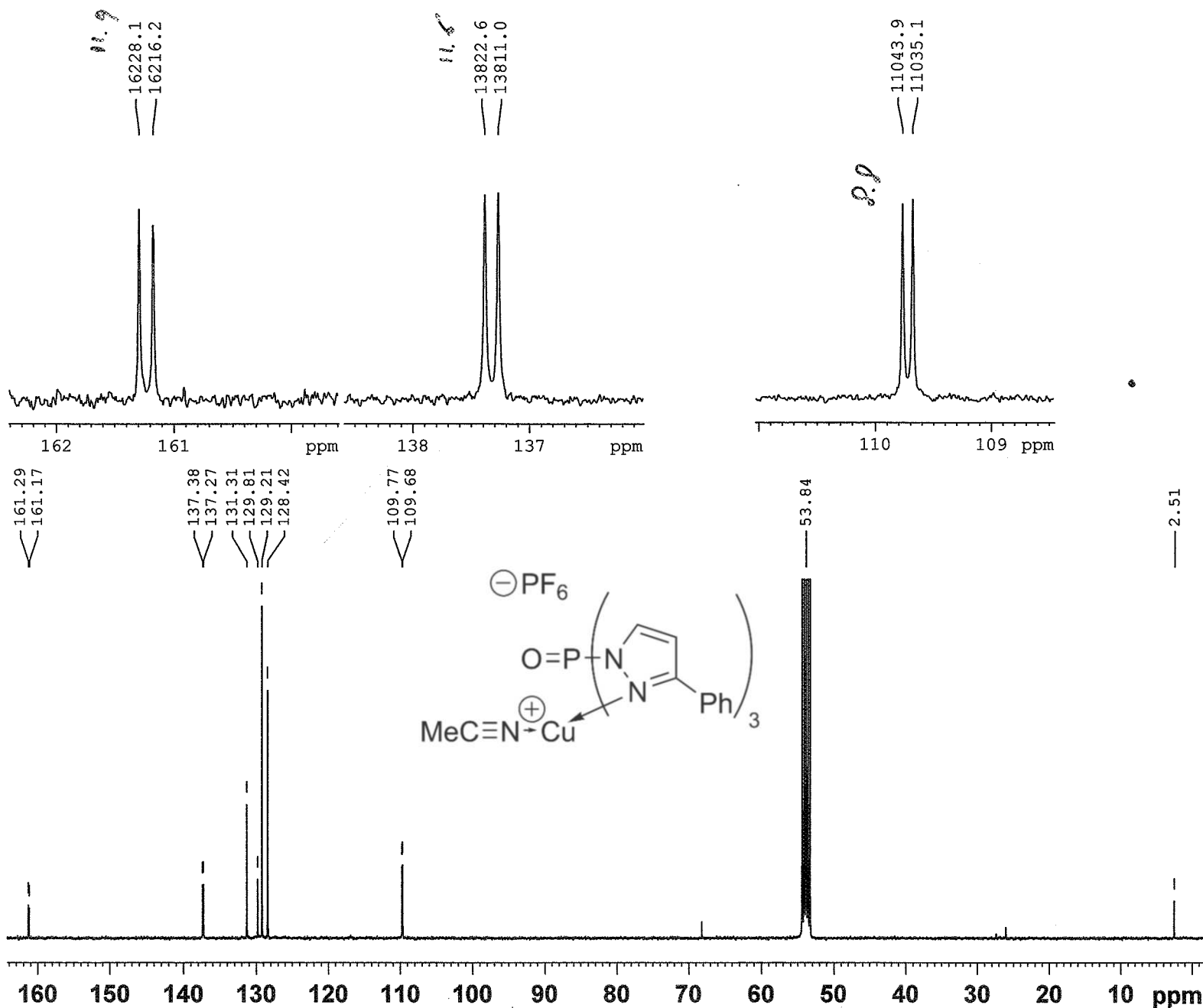
BRUKER

NAME VVL 331_400
EXPNO 1
PROCNO 2
Date 20100828
Time 10.36
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CD2Cl2
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 322.5
DW 60.400 usec
DE 6.00 usec
TE 300.4 K
D1 1.00000000 sec
D10 1

===== CHANNEL f1 =====
NUC1 1H
P1 13.00 usec
PL1 3.00 dB
SFO1 400.1324710 MHz
SI 32768
SF 400.1300149 MHz
WDW GM
SSB 0
LB -1.00 Hz
GB 0.5
PC 1.00

$^{13}\text{C}\{^1\text{H}\}$ NMR of $[\text{OP}(\text{3-Phpz})_3\text{Cu}(\text{NCMe})][\text{PF}_6]$ (**2b**)

(H, Ph-Pz) 3PO-CuNCMe PF6



NAME VVL 331_400
 EXPNO 2
 PROCNO 1
 Date_ 20100829
 Time_ 6.08
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CD2Cl2
 NS 20480
 DS 4
 SWH 23980.814 Hz
 FIDRES 0.365918 Hz
 AQ 1.3664756 sec
 RG 1448.2
 DW 20.850 usec
 DE 6.00 usec
 TE 300.4 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 DELTA 1.89999998 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 13C
 P1 8.80 usec
 PL1 -1.00 dB
 SFO1 100.6228298 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 3.00 dB
 PL12 18.00 dB
 PL13 18.00 dB
 SFO2 400.1316005 MHz
 SI 32768
 SF 100.6127223 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

³¹P{¹H} NMR of [OP(3-Phpz)₃Cu(NCMe)][PF₆] (2b)

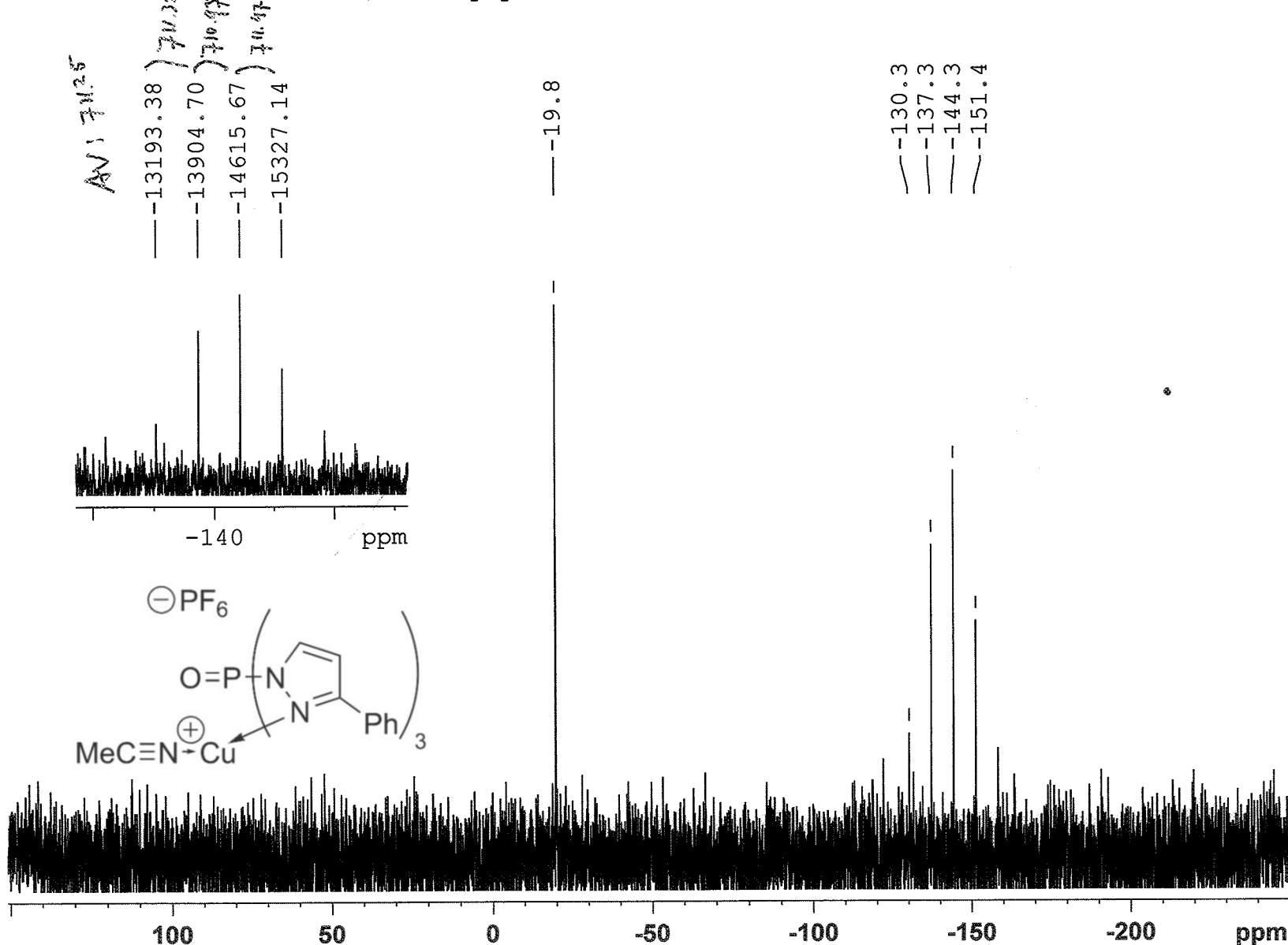
(H, Ph-Pz) 3PO-CuNCMe PF6
P31CPD CD2Cl2 {D:\NMRDATA} Volodymyr 23



NAME VVL 445
EXPNO 2
PROCNO 1
Date 20100823
Time 15.17
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 65536
SOLVENT CD2Cl2
NS 16
DS 4
SWH 40650.406 Hz
FIDRES 0.620276 Hz
AQ 0.8061428 sec
RG 20642.5
DW 12.300 usec
DE 6.00 usec
TE 300.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

===== CHANNEL f1 =====
NUC1 31P
P1 7.00 usec
PL1 0.00 dB
SFO1 101.2494172 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -3.00 dB
PL12 20.00 dB
PL13 24.00 dB
SFO2 250.1310005 MHz
SI 32768
SF 101.2544800 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40



¹⁹F NMR of [OP(3-Phpz)₃Cu(NCMe)][PF₆] (2b)

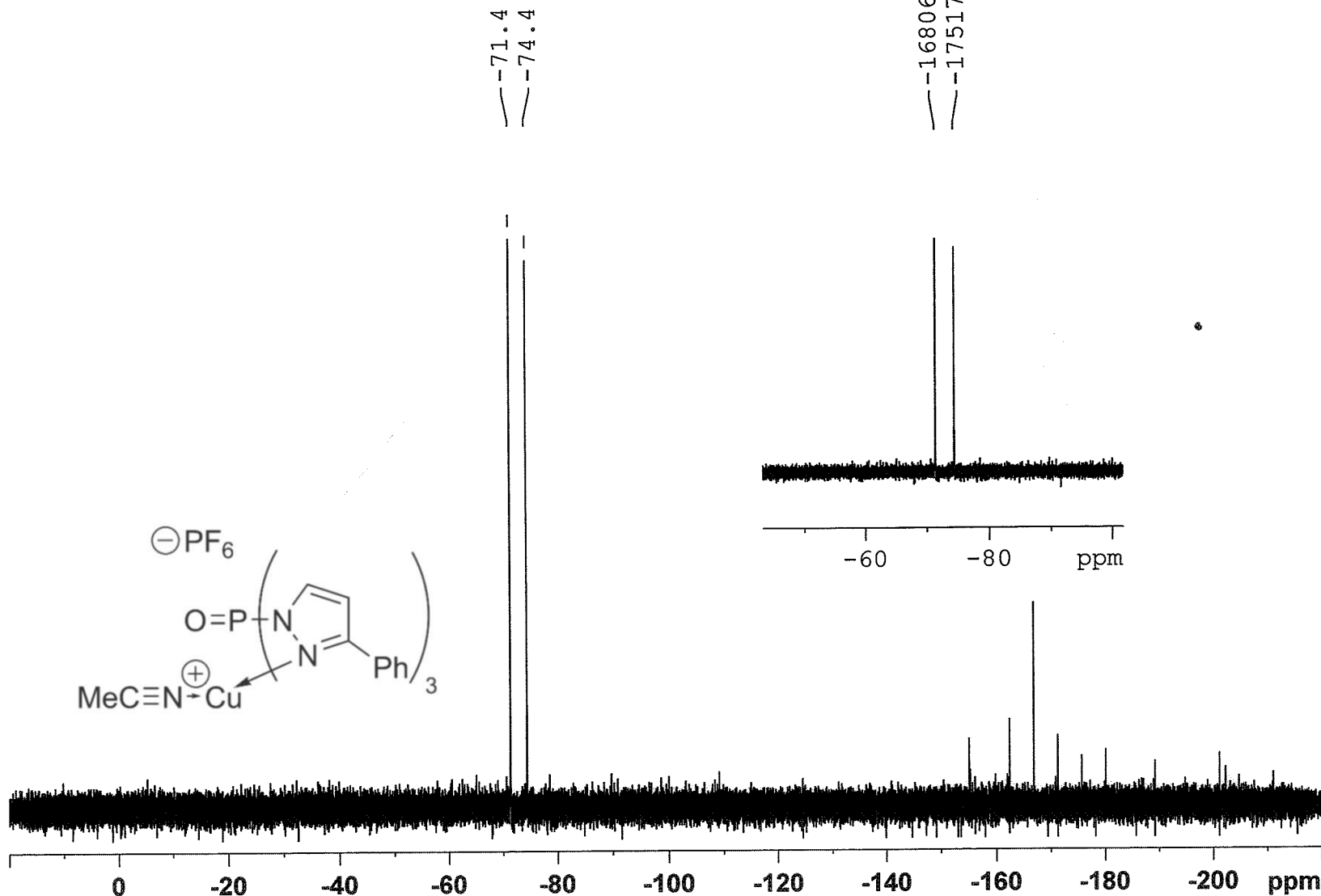
(H, Ph-Pz) 3PO-CuNCMe PF6
F19CPD CD2C12 {D:\NMRDATA} Volodymyr 23



NAME VVL 445
EXPNO 3
PROCNO 1
Date_ 20100823
Time 15.18
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgfhigqn
TD 131072
SOLVENT CD2C12
NS 16
DS 4
SWH 56497.176 Hz
FIDRES 0.431039 Hz
AQ 1.1600372 sec
RG 11585.2
DW 8.850 usec
DE 6.00 usec
TE 300.2 K
D1 1.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 19F
P1 8.00 usec
PL1 -3.00 dB
SFO1 235.3338140 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -3.00 dB
PL12 20.00 dB
SFO2 250.1310005 MHz
SI 65536
SF 235.3573500 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.20

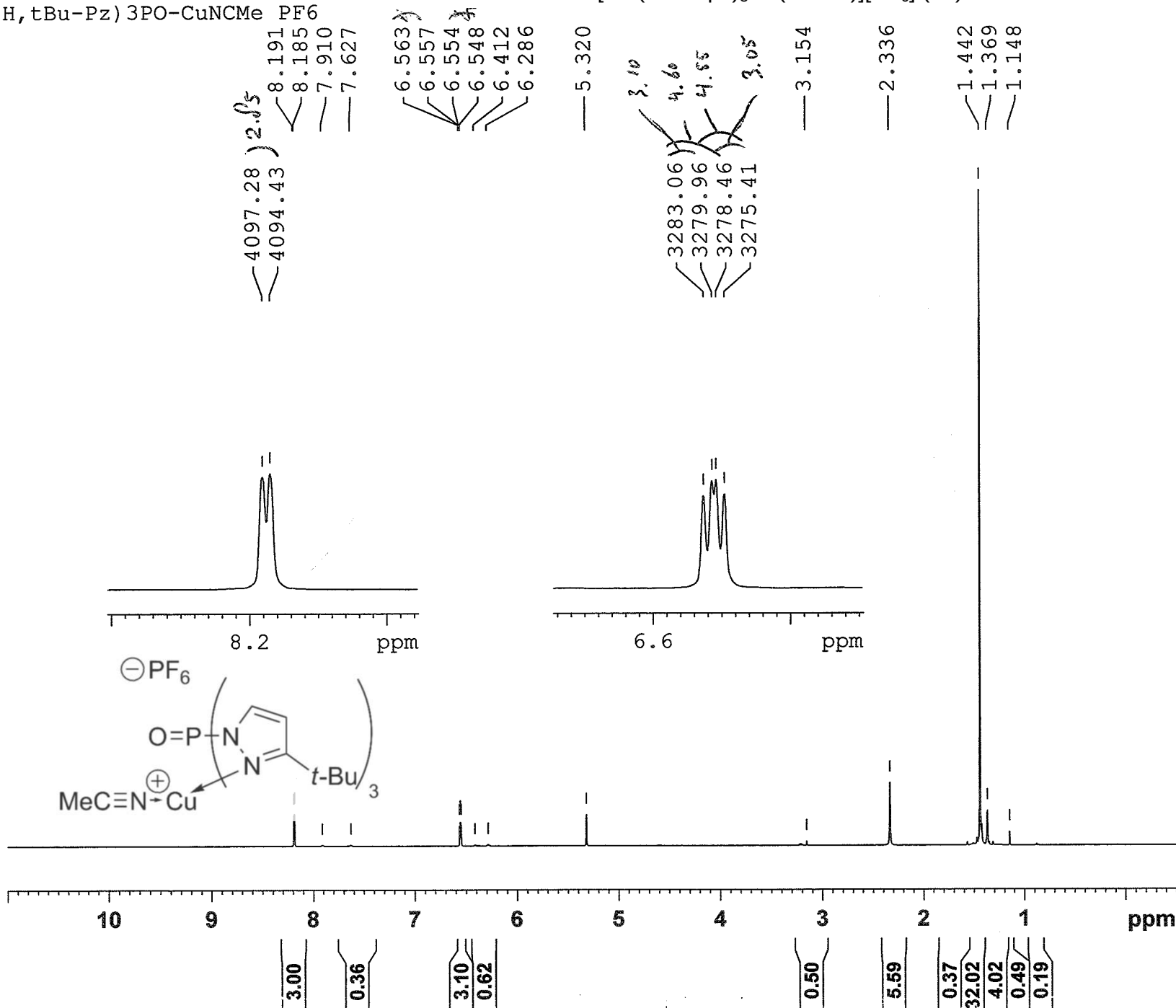


$(H, tBu-Pz)_3PO-CuNCMe PF_6$ 1H NMR of $[OP(3-t-Bupz)_3Cu(NCMe)][PF_6]$ (**2c**)



NAME VVL 25_500
 EXPNO 1
 PROCNO 1
 Date_ 20100826
 Time_ 14.41
 INSTRUM spect
 PROBHD 5 mm CPTCI 1H-
 PULPROG zg30
 TD 65536
 SOLVENT CD2Cl2
 NS 16
 DS 2
 SWH 10330.578 Hz
 FIDRES 0.157632 Hz
 AQ 3.1719923 sec
 RG 14.2
 DW 48.400 usec
 DE 6.50 usec
 TE 296.0 K
 D1 1.00000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 6.70 usec
 PL1 4.00 dB
 PL1W 8.72000027 W
 SFO1 500.2330891 MHz
 SI 32768
 SF 500.2300164 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



(H, tBu-Pz) 3PO-CuNCMe PF₆

¹H NMR of [OP(3-*t*-Bupz)₃Cu(NCMe)][PF₆] (2c)

after line narrowing

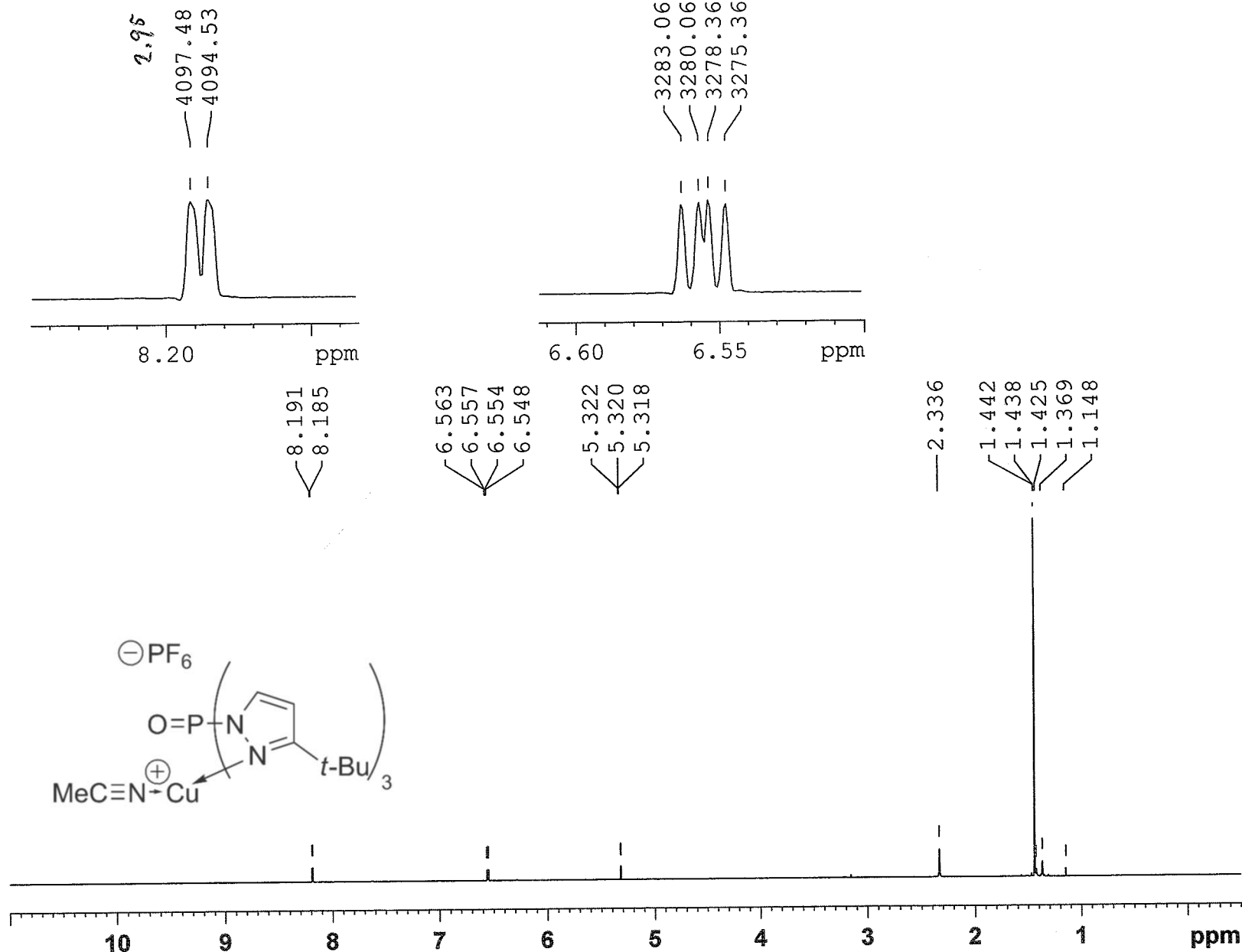


```

NAME          VVL 25_500
EXPNO         1
PROCNO        2
Date_         20100826
Time_         14.41
INSTRUM       spect
PROBHD        5 mm CPTCI 1H-
PULPROG       zg30
TD            65536
SOLVENT       CD2C12
NS            16
DS            2
SWH           10330.578 Hz
FIDRES        0.157632 Hz
AQ            3.1719923 sec
RG            14.2
DW            48.400 usec
DE            6.50 usec
TE            296.0 K
D1            1.00000000 sec
TD0           1
  
```

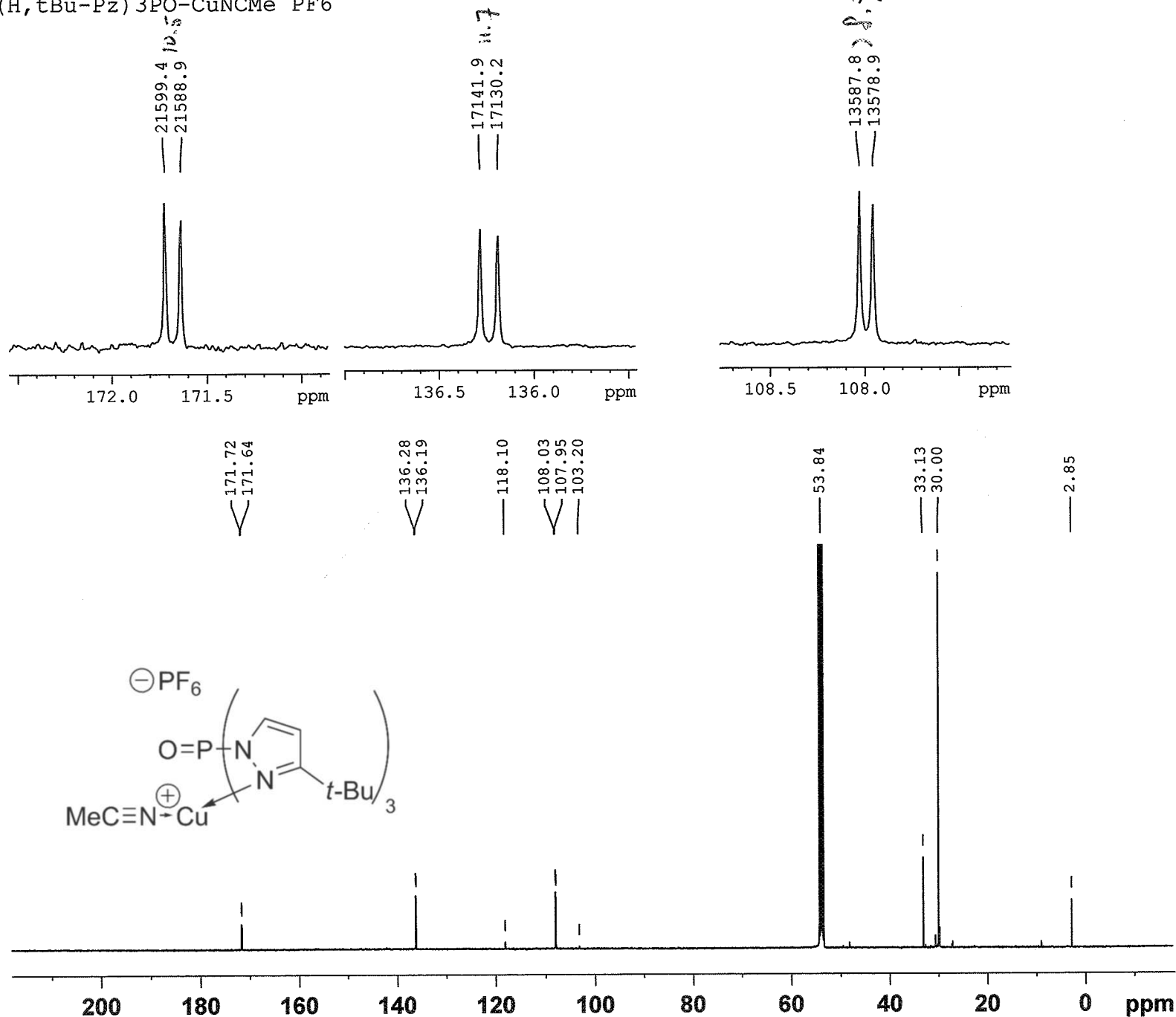
```

===== CHANNEL f1 =====
NUC1          1H
P1            6.70 usec
PL1           4.00 dB
PL1W          8.72000027 W
SFO1          500.2330891 MHz
SI            32768
SF            500.2300164 MHz
WDW           GM
SSB           0
LB            -0.60 Hz
GB            0.3
PC            1.00
  
```



¹³C{¹H} NMR of [OP(3-*t*-Bupz)₃Cu(NCMe)][PF₆] (2c)

(H, *t*Bu-Pz) 3PO-CuNCMe PF₆



NAME VVL 25_500
 EXPNO 2
 PROCNO 1
 Date_ 20100826
 Time_ 16.29
 INSTRUM spect
 PROBHD 5 mm CPTCI 1H-
 PULPROG zgpg30
 TD 65536
 SOLVENT CD2Cl2
 NS 2048
 DS 4
 SWH 29761.904 Hz
 FIDRES 0.454131 Hz
 AQ 1.1010548 sec
 RG 2050
 DW 16.800 usec
 DE 6.50 usec
 TE 295.8 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 13C
 P1 11.20 usec
 PL1 -2.00 dB
 PL1W 88.77790070 W
 SFO1 125.7955118 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 4.00 dB
 PL12 25.28 dB
 PL13 28.00 dB
 PL2W 8.72000027 W
 PL12W 0.06494062 W
 PL13W 0.03471494 W
 SFO2 500.2320009 MHz
 SI 32768
 SF 125.7828819 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

³¹P{¹H} NMR of [OP(3-*t*-Bupz)₃Cu(NCMe)][PF₆] (2c)

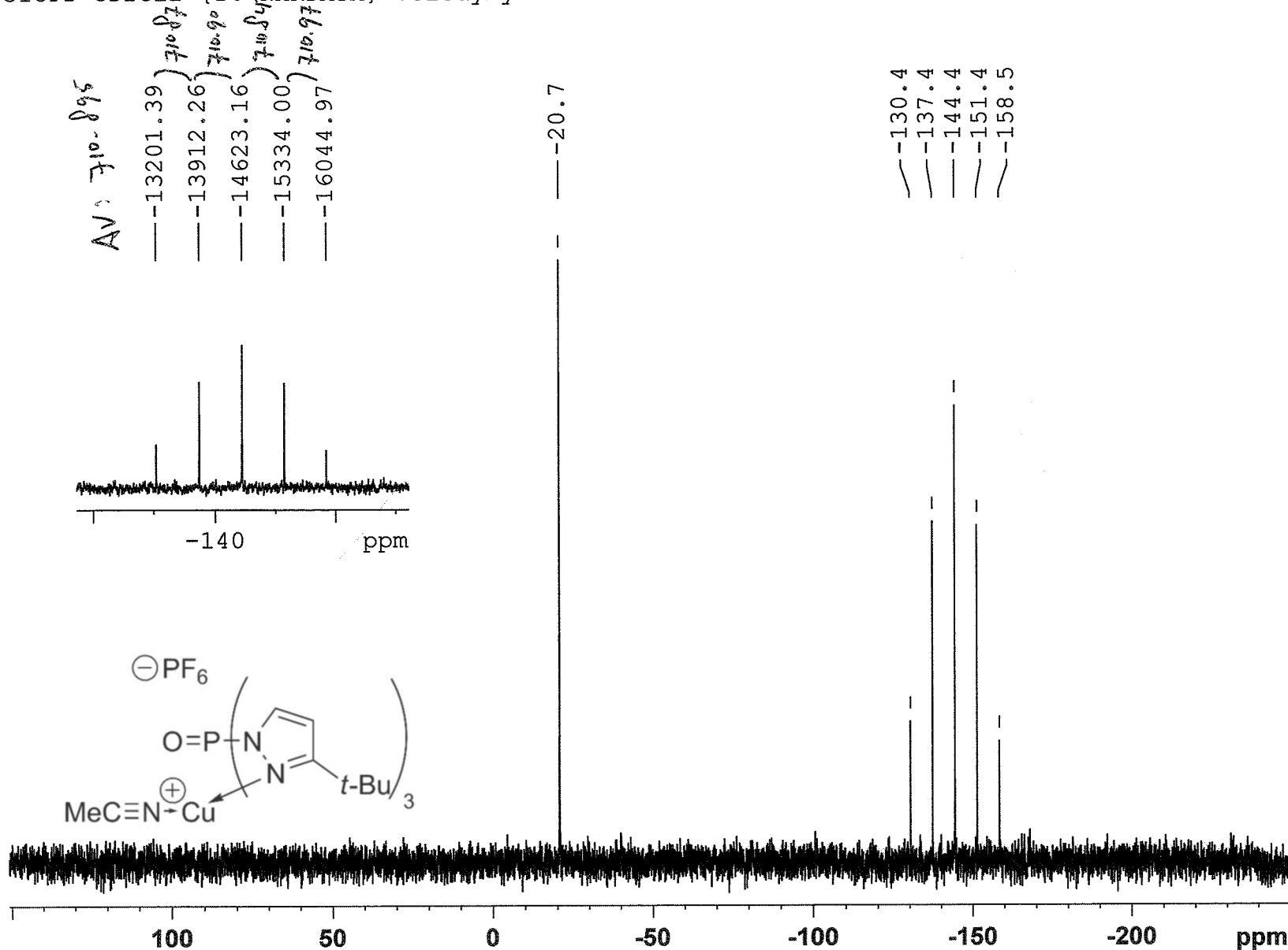
(H,*t*-Bu-Pz)3PO-CuNCMe PF₆
P31CPD CD₂Cl₂ {D:\NMRDATA} Volodymyr 15



NAME VVL 453
EXPNO 1
PROCNO 1
Date 20100826
Time 11.46
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 65536
SOLVENT CD₂Cl₂
NS 64
DS 4
SWH 40650.406 Hz
FIDRES 0.620276 Hz
AQ 0.8061428 sec
RG 20642.5
DW 12.300 usec
DE 6.00 usec
TE 300.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

===== CHANNEL f1 =====
NUC1 31P
P1 7.00 usec
PL1 0.00 dB
SFO1 101.2494172 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -3.00 dB
PL12 20.00 dB
PL13 24.00 dB
SFO2 250.1310005 MHz
SI 32768
SF 101.2544800 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40



¹⁹F NMR of [OP(3-*t*-Bupz)₃Cu(NCMe)][PF₆] (2c)

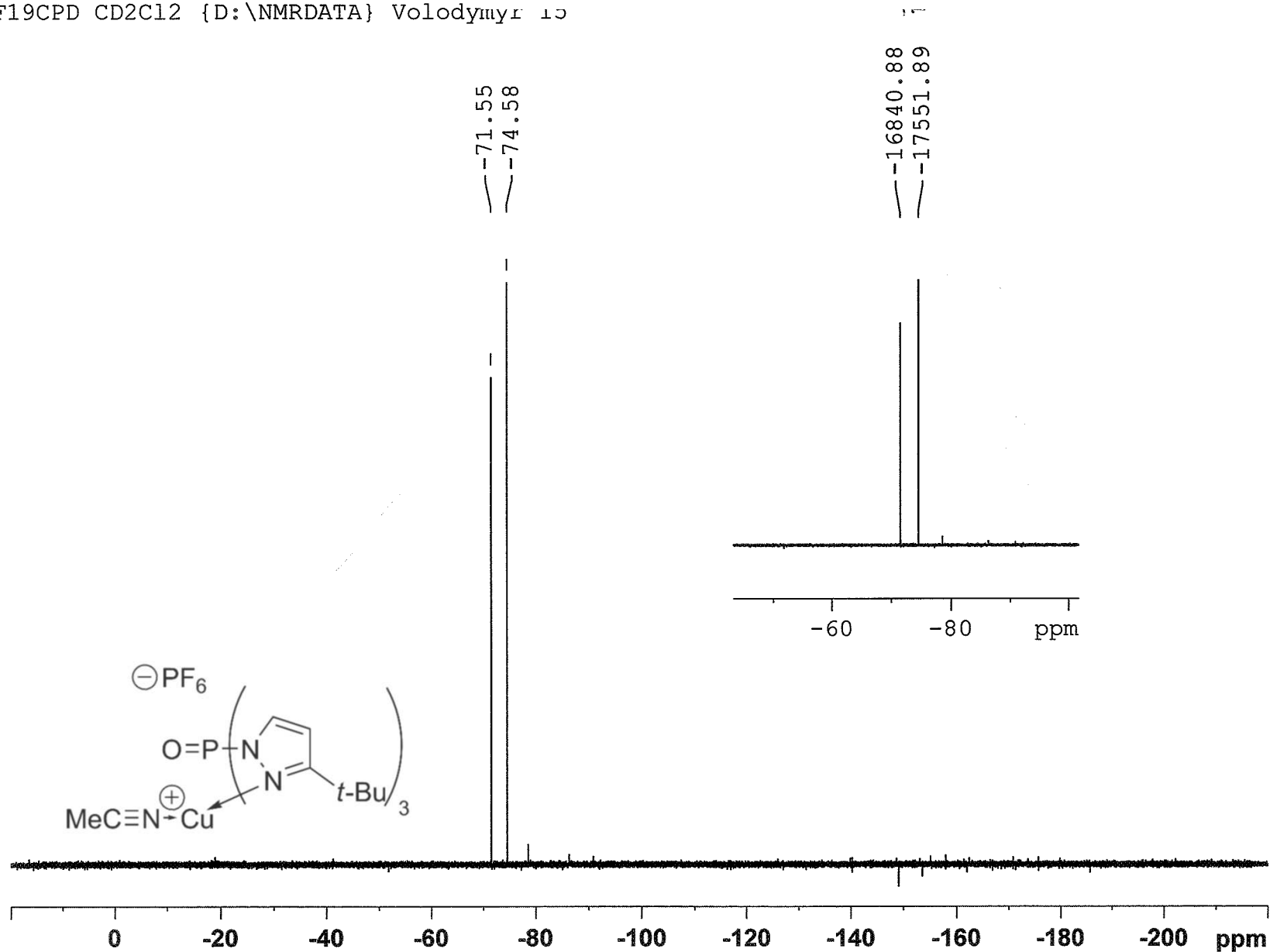
(H, *t*-Bu-Pz) 3PO-CuNCMe PF₆
F19CPD CD₂Cl₂ {D:\NMRDATA} Volodymyr 15



NAME VVL 453
EXPNO 2
PROCNO 1
Date_ 20100826
Time 11.48
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgfhigqn
TD 131072
SOLVENT CD₂Cl₂
NS 32
DS 4
SWH 56497.176 Hz
FIDRES 0.431039 Hz
AQ 1.1600372 sec
RG 11585.2
DW 8.850 usec
DE 6.00 usec
TE 300.2 K
D1 1.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 19F
P1 8.00 usec
PL1 -3.00 dB
SFO1 235.3338140 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -3.00 dB
PL12 20.00 dB
SFO2 250.1310005 MHz
SI 65536
SF 235.3573500 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.20



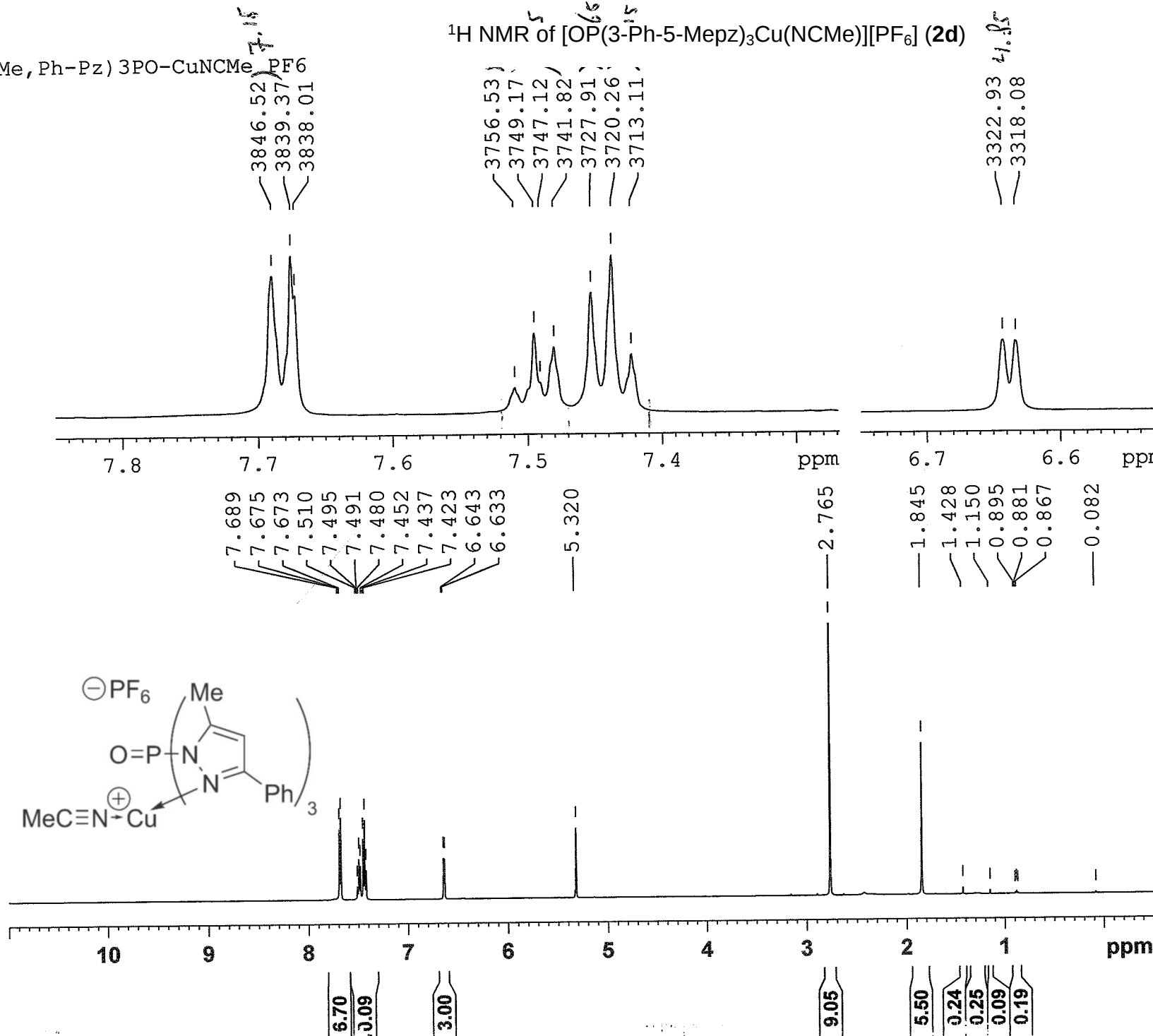
(Me, Ph-Pz) 3PO-CuNCMe PF₆

¹H NMR of [OP(3-Ph-5-Mepz)₃Cu(NCMe)][PF₆] (2d)



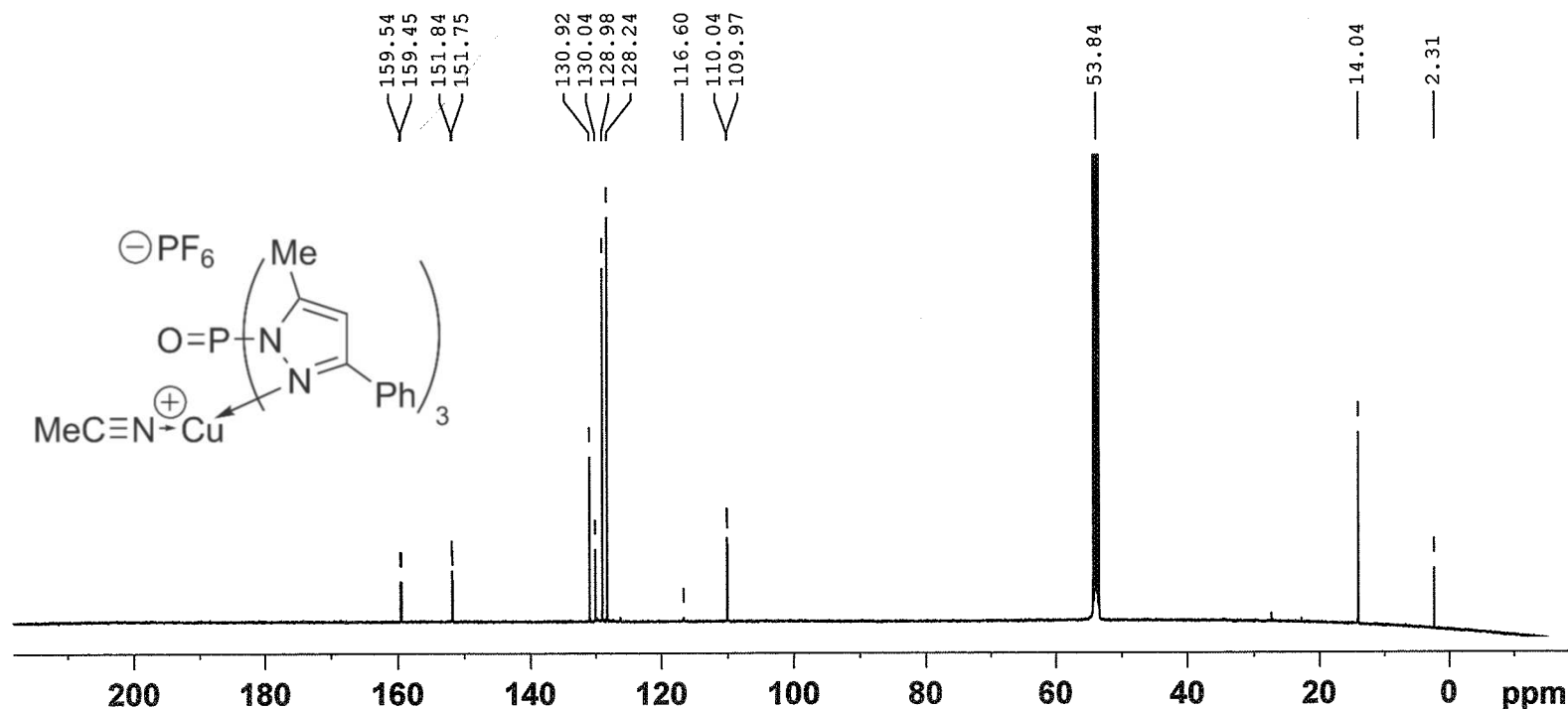
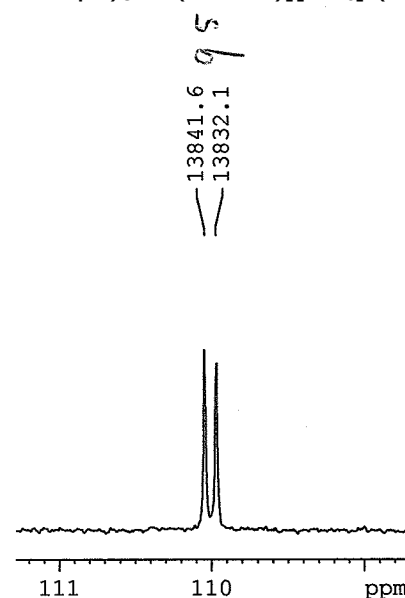
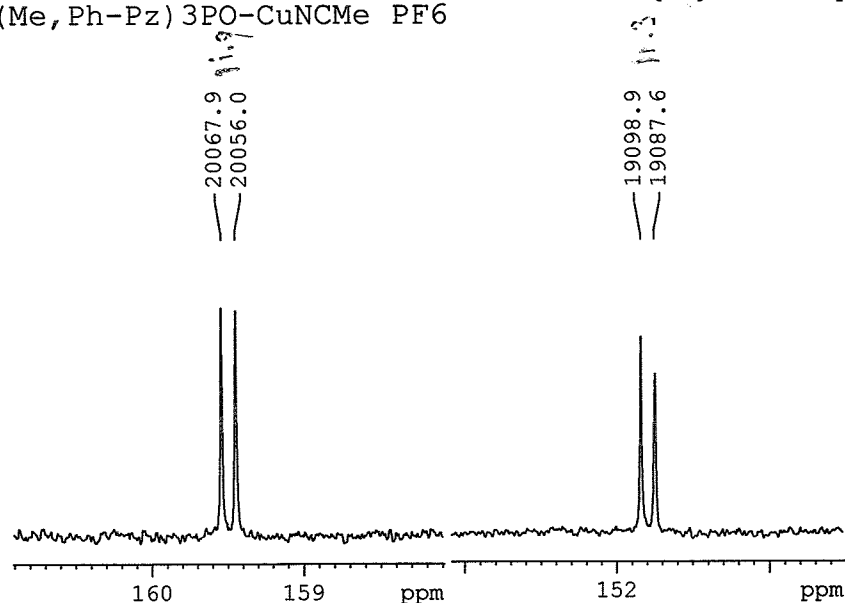
NAME VVL 24_500
EXPNO 1
PROCNO 1
Date_ 20100826
Time_ 10.36
INSTRUM spect
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 65536
SOLVENT CD2Cl2
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719923 sec
RG 12.7
DW 48.400 usec
DE 6.50 usec
TE 296.1 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 6.70 usec
PL1 4.00 dB
PL1W 8.72000027 W
SFO1 500.2330891 MHz
SI 32768
SF 500.2300163 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



(Me, Ph-Pz) 3PO-CuNCMe PF₆

¹³C{¹H} NMR of [OP(3-Ph-5-Mepz)₃Cu(NCMe)][PF₆] (2d)



NAME VVL 24_500
EXPNO 2
PROCNO 1
Date_ 20100826
Time 12.26
INSTRUM spect
PROBHD 5 mm CPTCI 1H-
PULPROG zgpg30
TD 65536
SOLVENT CD2Cl2
NS 2048
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 2050
DW 16.800 usec
DE 6.50 usec
TE 295.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 11.20 usec
PL1 -2.00 dB
PL1W 88.77790070 W
SFO1 125.7955118 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 4.00 dB
PL12 25.28 dB
PL13 28.00 dB
PL2W 8.72000027 W
PL12W 0.06494062 W
PL13W 0.03471494 W
SFO2 500.2320009 MHz
SI 32768
SF 125.7828823 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

³¹P{¹H} NMR of [OP(3-Ph-5-Mepz)₃Cu(NCMe)][PF₆] (**2d**)

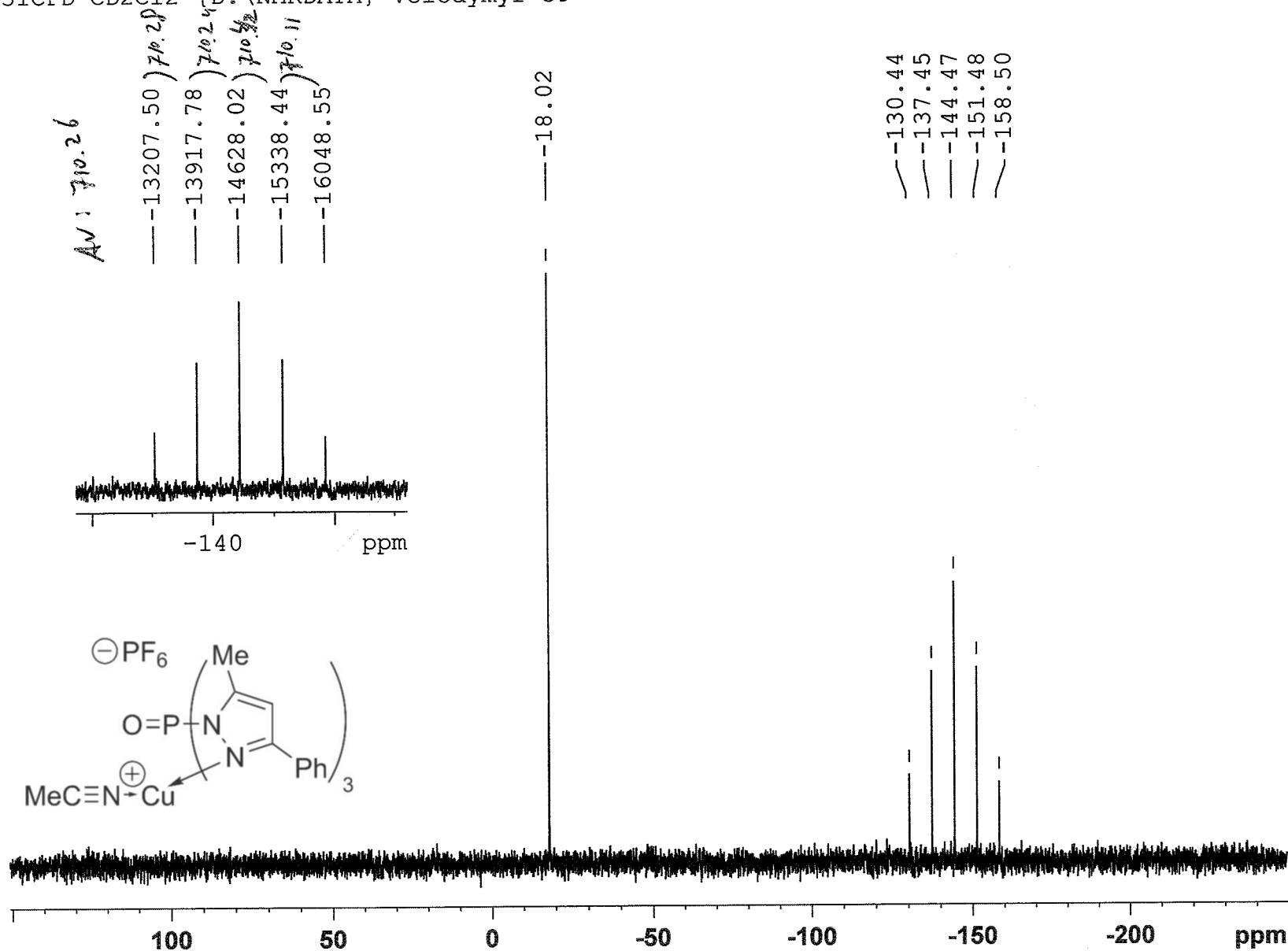
(Me,Ph-Pz)3PO-CuNCMe PF₆, after washing with MTBE
P31CPD CD2C12 {D:\NMRDATA} Volodymyr 39



NAME VVL 446
EXPNO 2
PROCNO 1
Date_ 20100824
Time 16.16
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 65536
SOLVENT CD2C12
NS 32
DS 4
SWH 40650.406 Hz
FIDRES 0.620276 Hz
AQ 0.8061428 sec
RG 20642.5
DW 12.300 usec
DE 6.00 usec
TE 300.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

===== CHANNEL f1 =====
NUC1 31P
P1 7.00 usec
PL1 0.00 dB
SFO1 101.2494172 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -3.00 dB
PL12 20.00 dB
PL13 24.00 dB
SFO2 250.1310005 MHz
SI 32768
SF 101.2544800 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40



¹⁹F NMR of [OP(3-Ph-5-Mepz)₃Cu(NCMe)][PF₆] (2d)

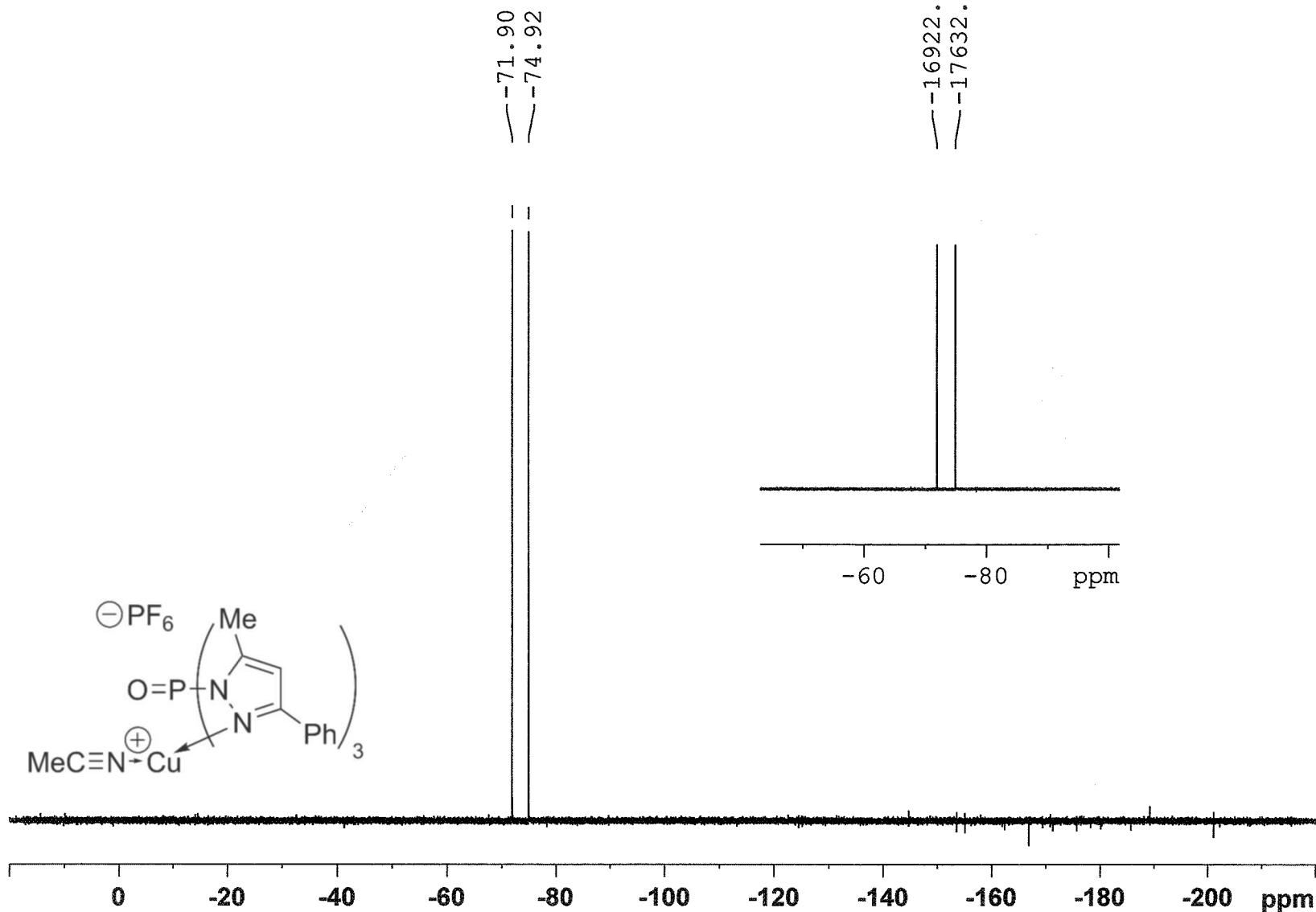
(Me,Ph-Pz)₃PO-CuNCMe PF₆, after washing with MTBE
F19CPD CD₂Cl₂ {D:\NMRDATA} Volodymyr 39



NAME VVL 446
EXPNO 3
PROCNO 1
Date_ 20100824
Time 16.17
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgfhigqn
TD 131072
SOLVENT CD₂Cl₂
NS 16
DS 4
SWH 56497.176 Hz
FIDRES 0.431039 Hz
AQ 1.1600372 sec
RG 10321.3
DW 8.850 usec
DE 6.00 usec
TE 300.2 K
D1 1.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 19F
P1 8.00 usec
PL1 -3.00 dB
SFO1 235.3338140 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -3.00 dB
PL12 20.00 dB
SFO2 250.1310005 MHz
SI 65536
SF 235.3573500 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.20



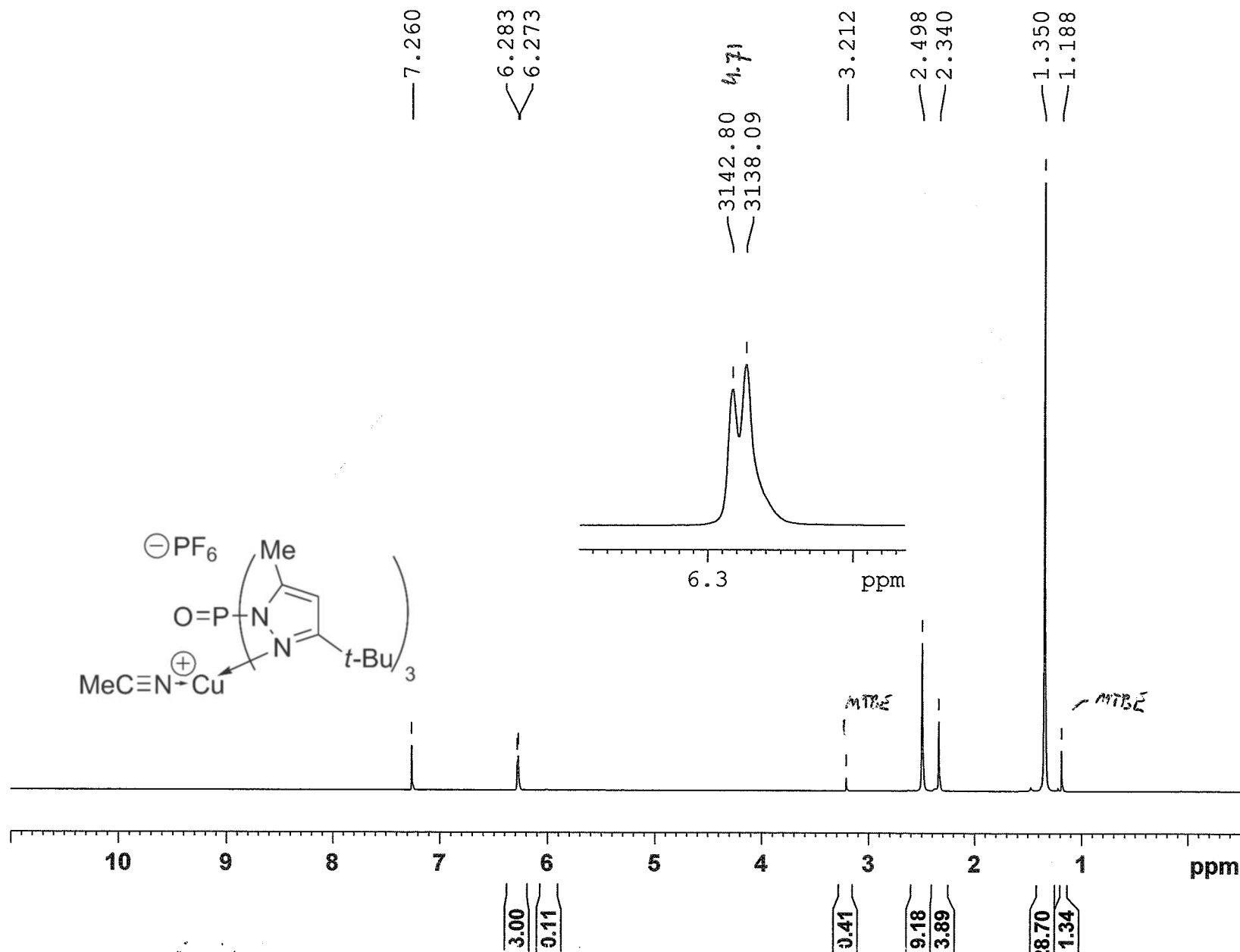
¹H NMR of [OP(3-*t*-Bu-5-Mepz)₃Cu(NCMe)][PF₆] (2e)

(Me, *t*-Bu-Pz)₃-PO-CuNCMe PF₆

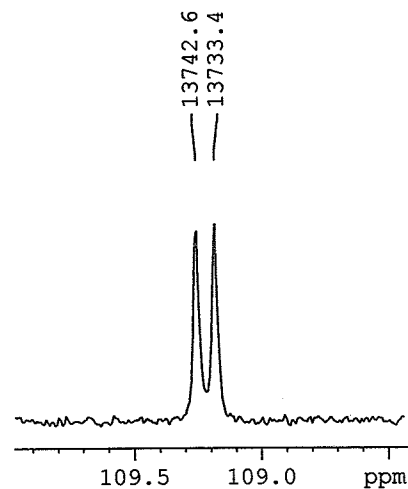
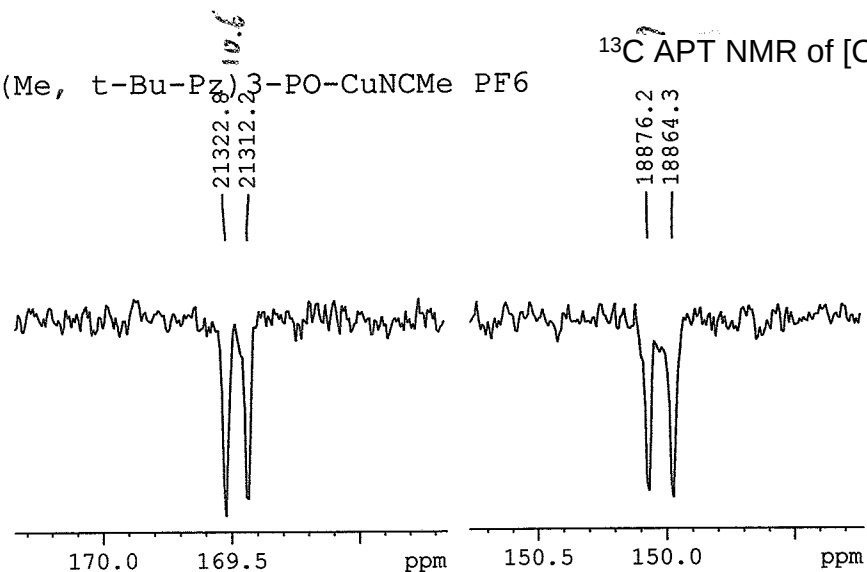


NAME VVL 19_500
EXPNO 1
PROCNO 1
Date_ 20100819
Time_ 10.45
INSTRUM spect
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 65536
SOLVENT CDCl₃
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1719923 sec
RG 12.7
DW 48.400 usec
DE 6.50 usec
TE 296.2 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 6.70 usec
PL1 4.00 dB
PL1W 8.72000027 W
SFO1 500.2330891 MHz
SI 32768
SF 500.2300094 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

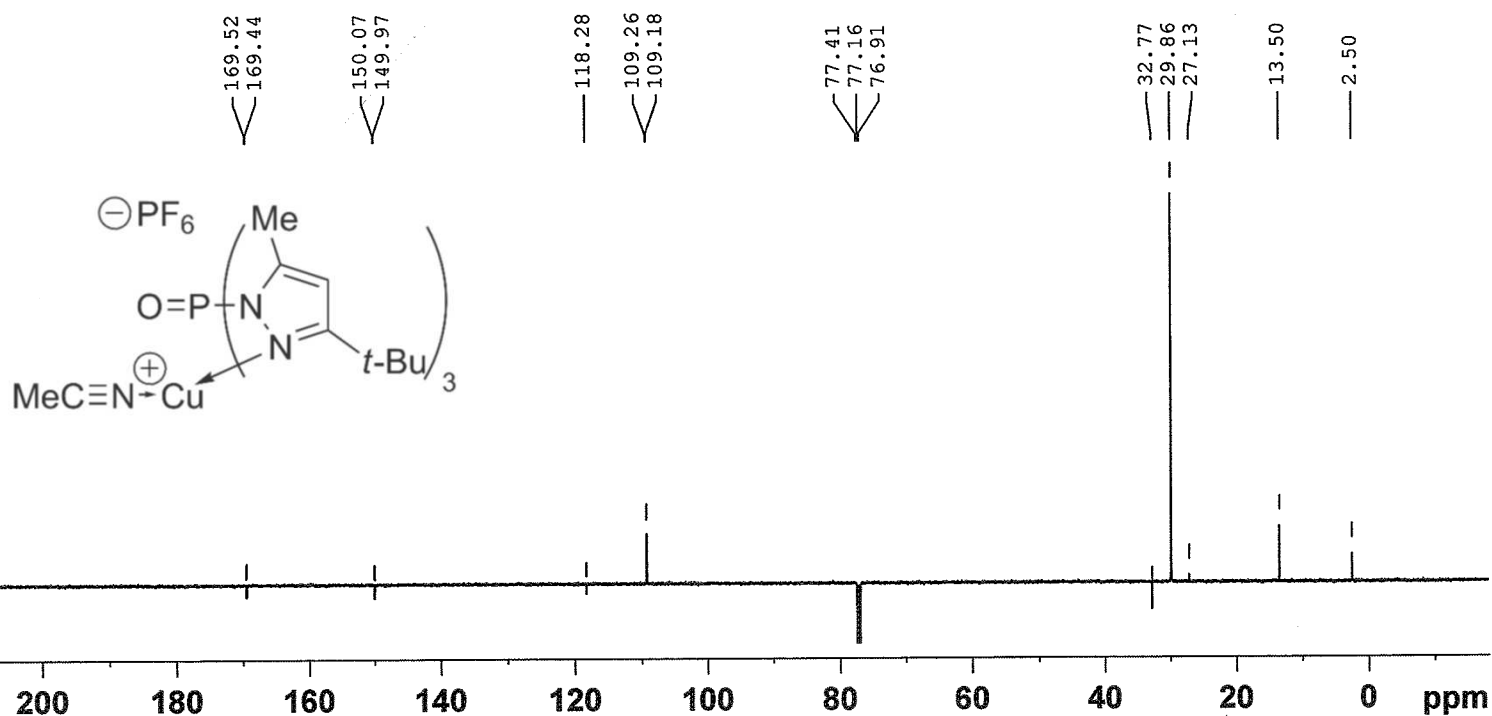


(Me, t-Bu-Pz)₃-PO-CuNCMe PF₆ ¹³C APT NMR of [OP(3-*t*-Bu-5-Mepz)₃Cu(NCMe)][PF₆] (2e)



```

NAME          VVL 19_500
EXPNO         2
PROCNO        1
Date_         20100819
Time_         11.13
INSTRUM       spect
PROBHD        5 mm CPTCI 1H-
PULPROG       jmod
TD            65536
SOLVENT       CDC13
NS            512
DS            4
SWH           29761.904 Hz
FIDRES        0.454131 Hz
AQ            1.1010548 sec
RG            2050
DW            16.800 usec
DE            6.50 usec
TE            296.1 K
CNST2         145.0000000
CNST11        1.0000000
D1            2.00000000 sec
D20           0.00689655 sec
TD0           1
  
```



```

===== CHANNEL f1 =====
NUC1          13C
P1            11.20 usec
P2            22.40 usec
PL1           -2.00 dB
PL1W          88.77790070 W
SFO1          125.7955118 MHz
  
```

```

===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2          1H
PCPD2         80.00 usec
PL2           4.00 dB
PL12          25.28 dB
PL2W          8.72000027 W
PL12W         0.06494062 W
SFO2          500.2320009 MHz
SI            32768
SF            125.7829143 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
  
```

³¹P{¹H} NMR of [OP(3-*t*-Bu-5-Mepz)₃Cu(NCMe)][PF₆] (2e)

(Me, *t*Bu-Pz) 3PO-CuNCMe PF₆

P31CPD CDC13 {D:\NMRDATA} Volodymyr 52



```

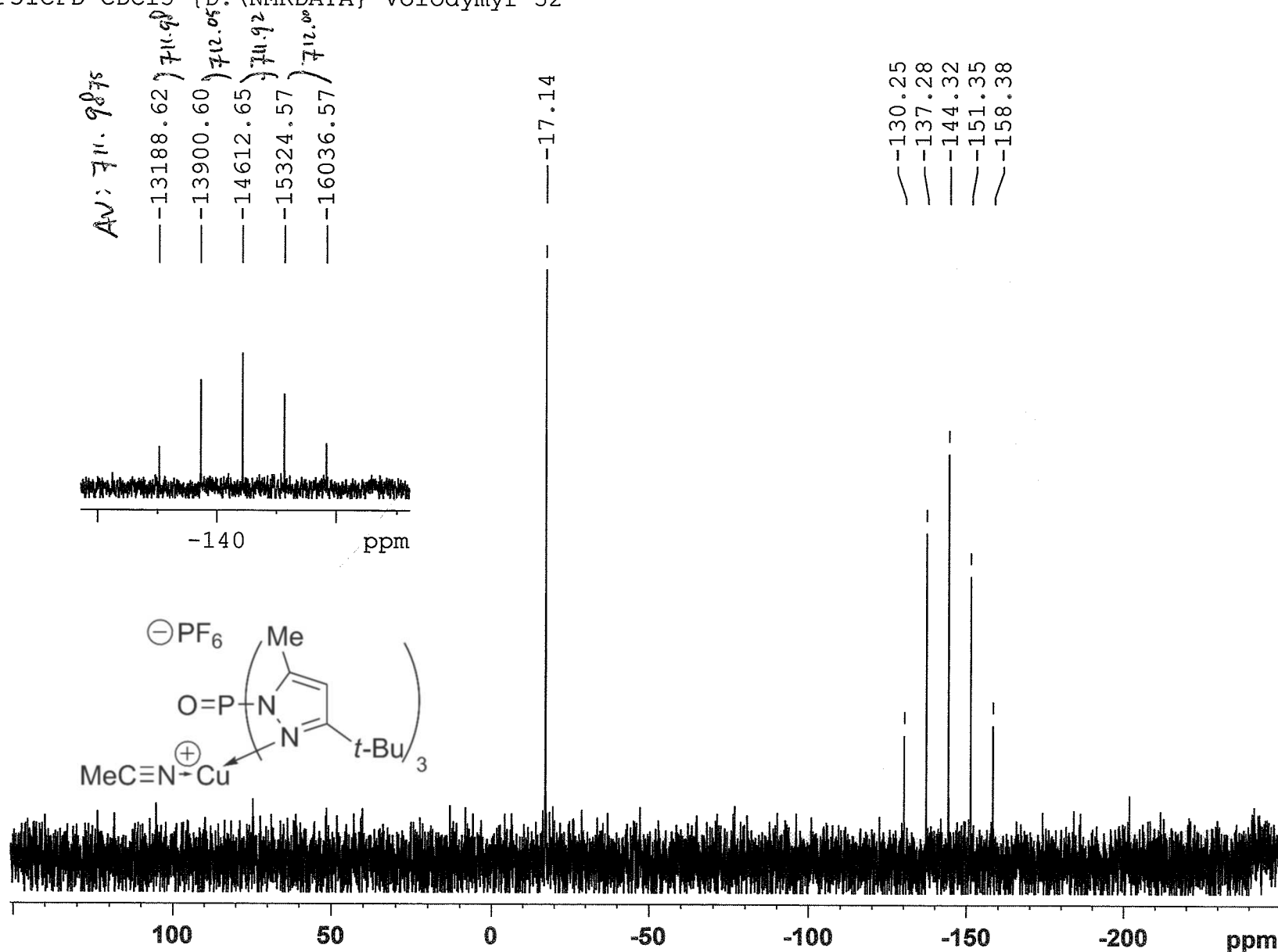
NAME          VVL 438
EXPNO         1
PROCNO        1
Date_         20100819
Time          13.09
INSTRUM       spect
PROBHD        5 mm QNP 1H/1
PULPROG       zgpg30
TD            65536
SOLVENT       CDC13
NS            16
DS            4
SWH           40650.406 Hz
FIDRES        0.620276 Hz
AQ            0.8061428 sec
RG            9195.2
DW            12.300 usec
DE            6.00 usec
TE            300.2 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
TD0           1
  
```

```

===== CHANNEL f1 =====
NUC1          31P
P1            7.00 usec
PL1           0.00 dB
SFO1          101.2494172 MHz
  
```

```

===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2          1H
PCPD2         80.00 usec
PL2           -3.00 dB
PL12          20.00 dB
PL13          24.00 dB
SFO2          250.1310005 MHz
SI            32768
SF            101.2544800 MHz
WDW           EM
SSB           0
LB            3.00 Hz
GB            0
PC            1.40
  
```



¹⁹F NMR of [OP(3-*t*-Bu-5-Mepz)₃Cu(NCMe)][PF₆] (2e)

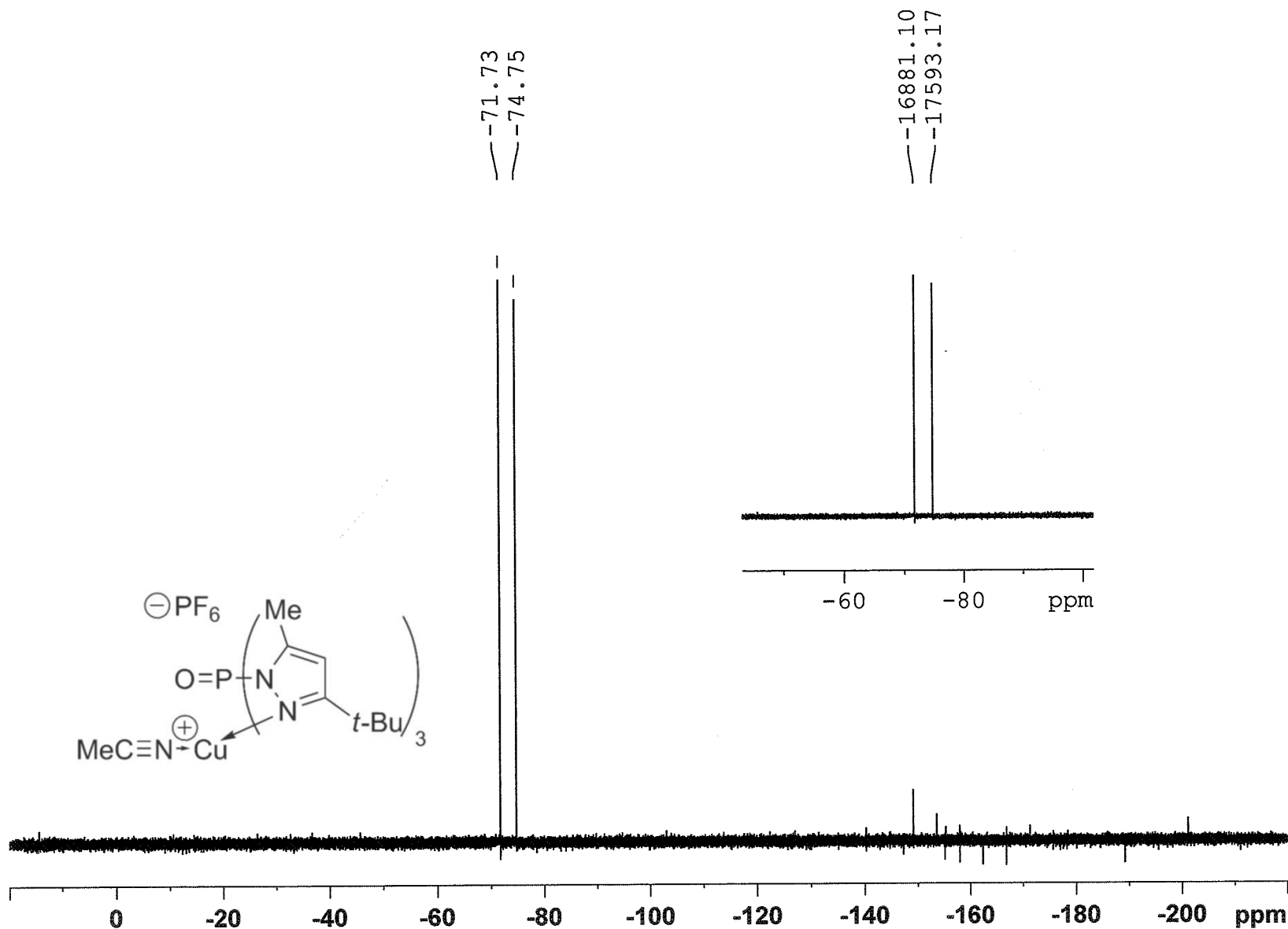
(Me, *t*Bu-Pz) 3PO-CuNCMe PF₆
F19CPD CDC13 {D:\NMRDATA} Volodymyr 52



NAME VVL 438
EXPNO 2
PROCNO 1
Date 20100819
Time 13.11
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgfhigqn
TD 131072
SOLVENT CDC13
NS 16
DS 4
SWH 56497.176 Hz
FIDRES 0.431039 Hz
AQ 1.1600372 sec
RG 10321.3
DW 8.850 usec
DE 6.00 usec
TE 300.2 K
D1 1.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 19F
P1 8.00 usec
PL1 -3.00 dB
SFO1 235.3338140 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -3.00 dB
PL12 20.00 dB
SFO2 250.1310005 MHz
SI 65536
SF 235.3573500 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.20



¹H NMR of [OP(3,5-Me₂pz)₃Cu(PPh₃)] [PF₆] (**3a**)

1H (CDCl₃ GB)

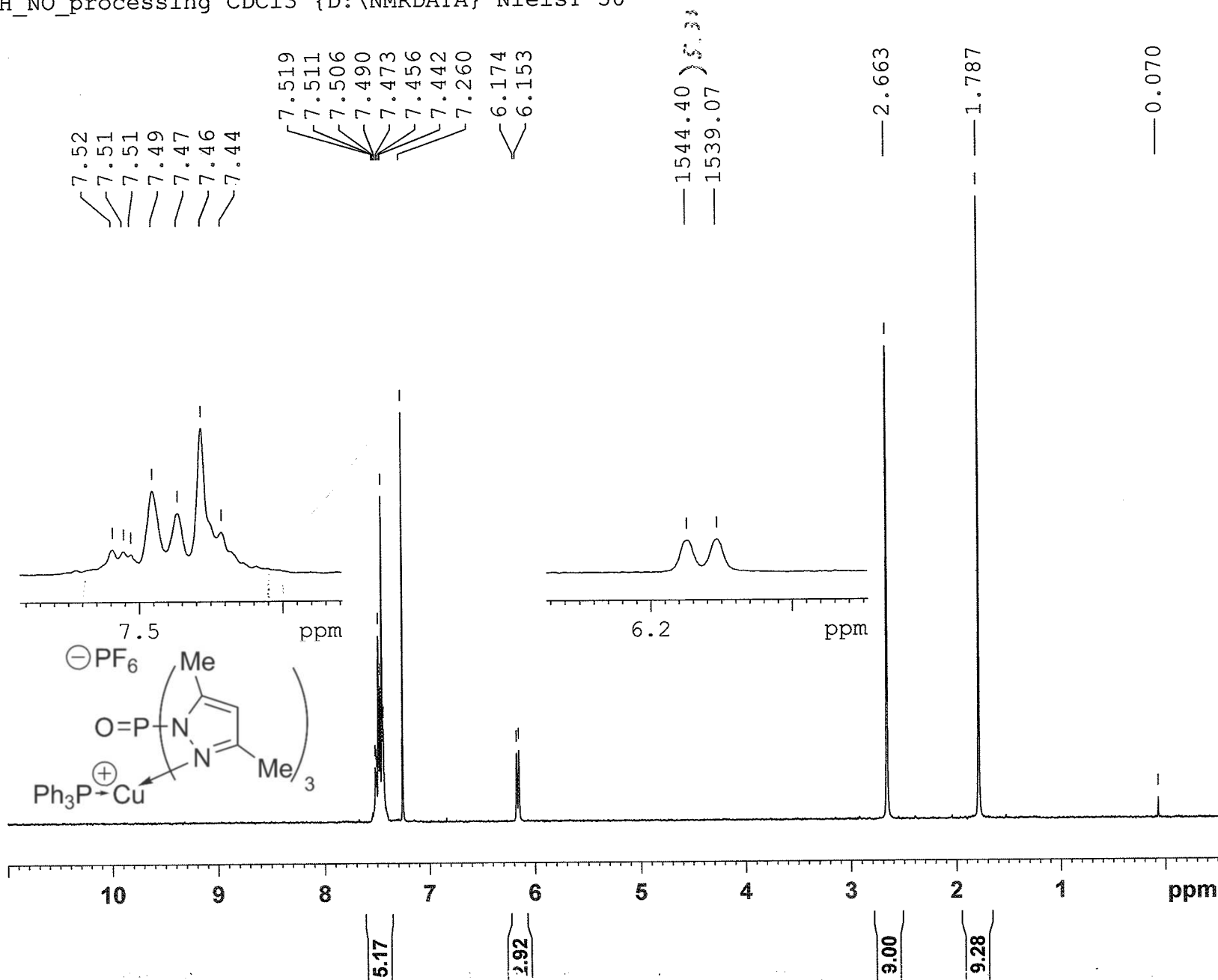
OP(Me₂pz)₃CuPPh₃ PF₆ after 5h @ 65C and vac

1H_NO_processing CDCl₃ {D:\NMRDATA} NielsT 50



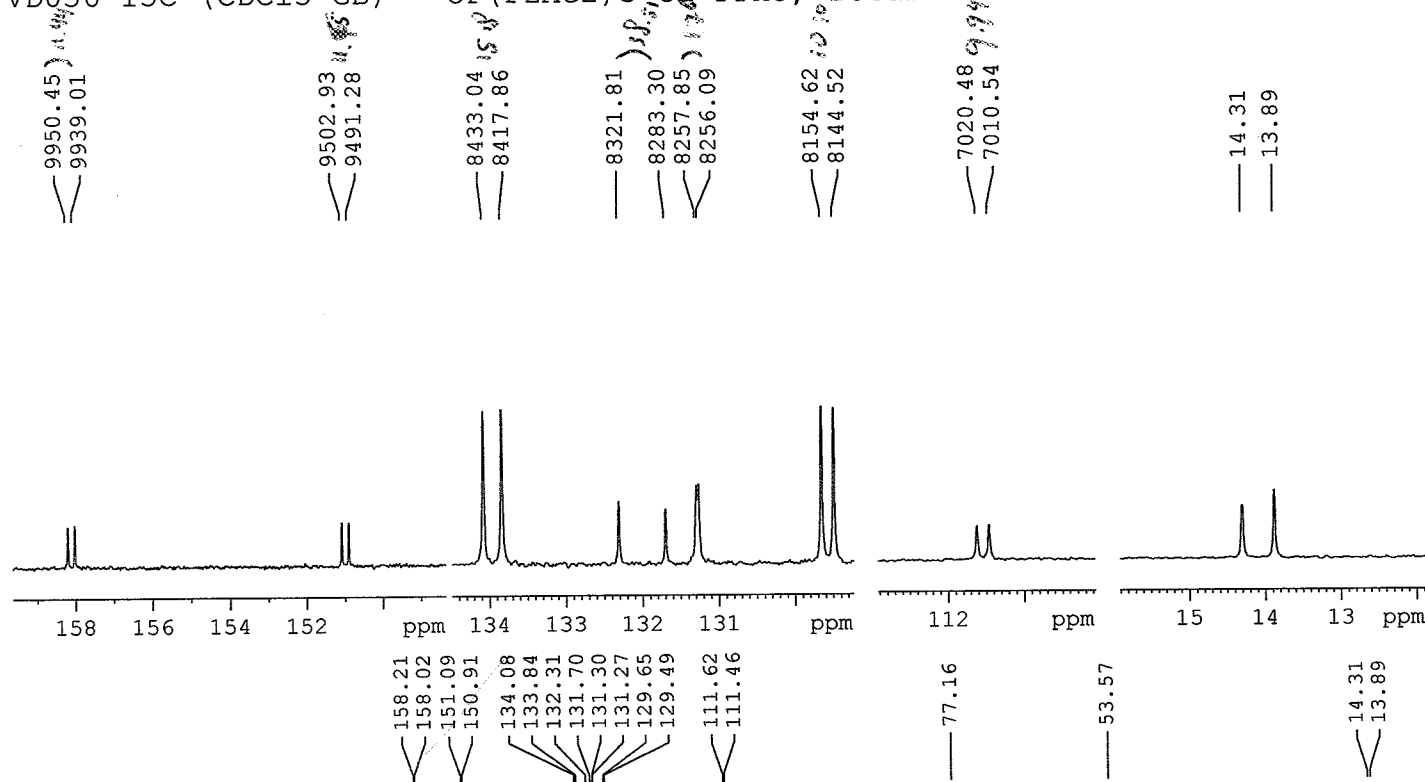
NAME CGJT-11-03
EXPNO 4
PROCNO 1
Date_ 20110614
Time_ 17.41
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg30
TD 65536
SOLVENT CDCl₃
NS 16
DS 2
SWH 5175.983 Hz
FIDRES 0.078979 Hz
AQ 6.3308277 sec
RG 2298.8
DW 96.600 usec
DE 6.00 usec
TE 300.2 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 9.50 usec
PL1 -3.00 dB
SFO1 250.1315447 MHz
SI 65536
SF 250.1300122 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.40



¹³C{¹H} NMR of [OP(3,5-Me₂pz)₃Cu(PPh₃)] [PF₆] (3a)

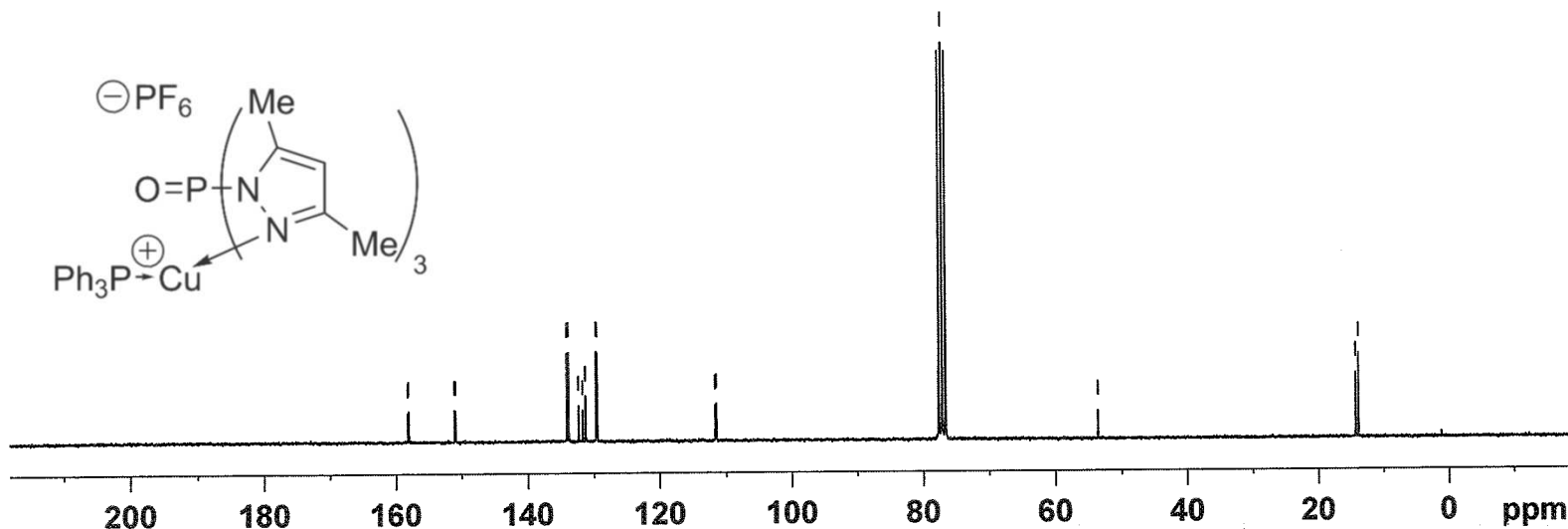
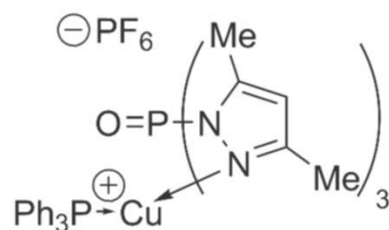
TVD036 13C (CDC13-GB) - OP(PzMe₂)₃-Cu-PPh₃, returned from Utrecht



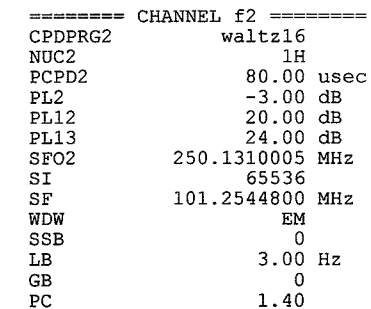
NAME TVD036
 EXPNO 1
 PROCNO 1
 Date_ 20080618
 Time_ 2.28
 INSTRUM spect
 PROBHD 5 mm QNP 1H/1
 PULPROG zgpg30 1001
 TD 65536
 SOLVENT CDC13
 NS 5664
 DS 4
 SWH 15060.241 Hz
 FIDRES 0.229801 Hz
 AQ 2.1758451 sec
 RG 1448.2
 DW 33.200 usec
 DE 6.00 usec
 TE 300.2 K
 D1 2.3199993 sec
 d11 0.03000000 sec
 DELTA 2.22000003 sec
 L4 8
 TDO 1

===== CHANNEL f1 =====
 NUC1 13C
 P1 6.75 usec
 PL1 -3.00 dB
 SFO1 62.9015280 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 -3.00 dB
 PL12 20.00 dB
 PL13 24.00 dB
 SFO2 250.1310005 MHz
 SI 65536
 SF 62.8952313 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40



31PCPD NO processing CDC13 {D:\NMRDATA} Nielst 7



¹⁹F NMR of [OP(3,5-Me₂pz)₃Cu(PPh₃)]⁺[PF₆]⁻ (**3a**)

¹⁹F (CDCl₃ GB)

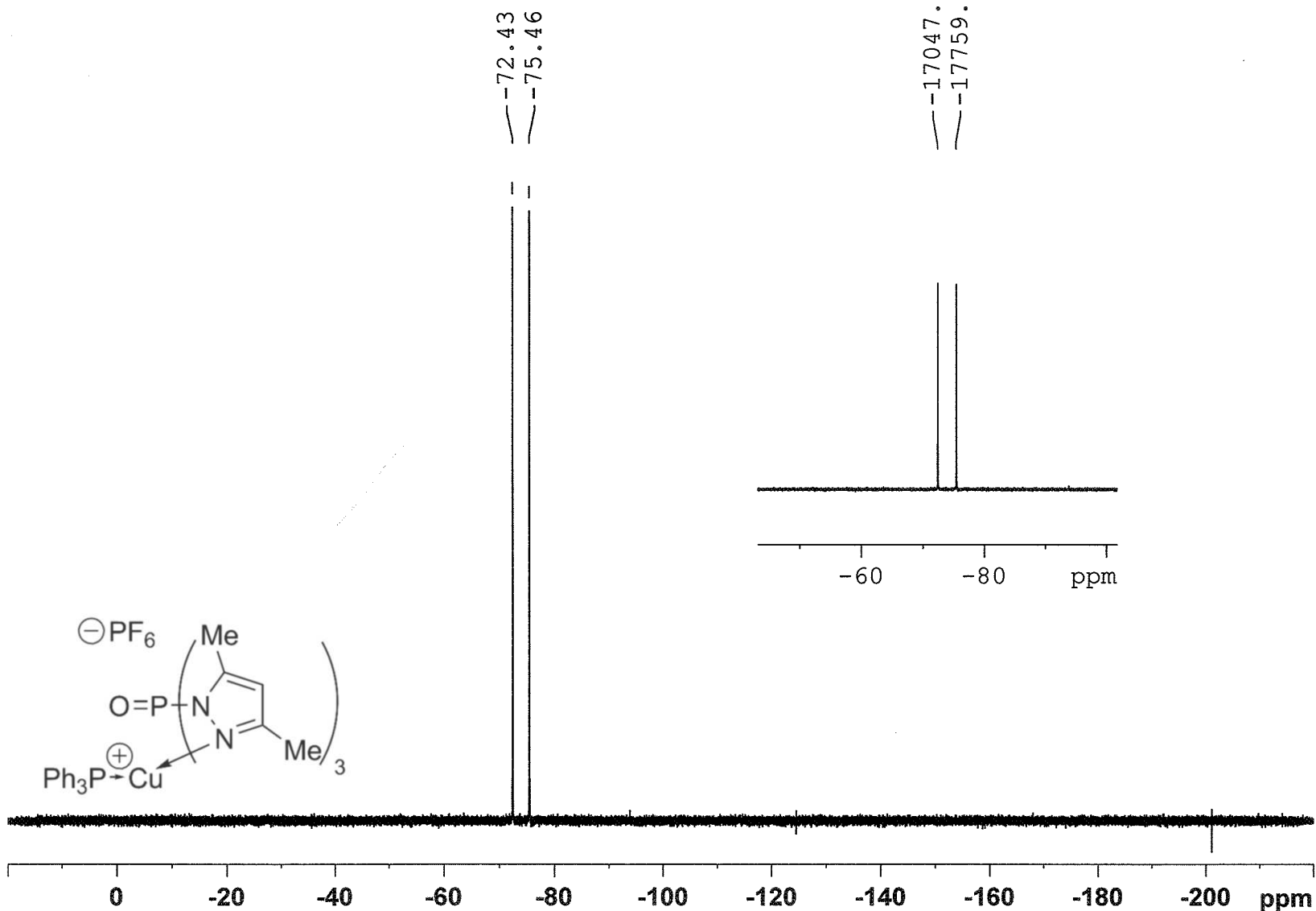
OP(Me₂pz)₃CuPPh₃ PF₆ after Et₂O wash

F19_NO_processing CDCl₃ {D:\NMRDATA} NielsT 7



NAME CGJT-11-03
 EXPNO 3
 PROCNO 1
 Date_ 20110614
 Time_ 10.31
 INSTRUM spect
 PROBHD 5 mm QNP 1H/1
 PULPROG zgpg30
 TD 131072
 SOLVENT CDCl₃
 NS 64
 DS 4
 SWH 56497.176 Hz
 FIDRES 0.431039 Hz
 AQ 1.1600372 sec
 RG 18390.4
 DW 8.850 usec
 DE 6.00 usec
 TE 300.2 K
 D1 1.00000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 19F
 P1 8.00 usec
 PL1 -3.00 dB
 SFO1 235.3338140 MHz
 SI 65536
 SF 235.3573500 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



¹H NMR of [OP(3,5-Me₂pz)₃Cu(CO)][PF₆] (4a)

¹H (CDCl₃ GB)

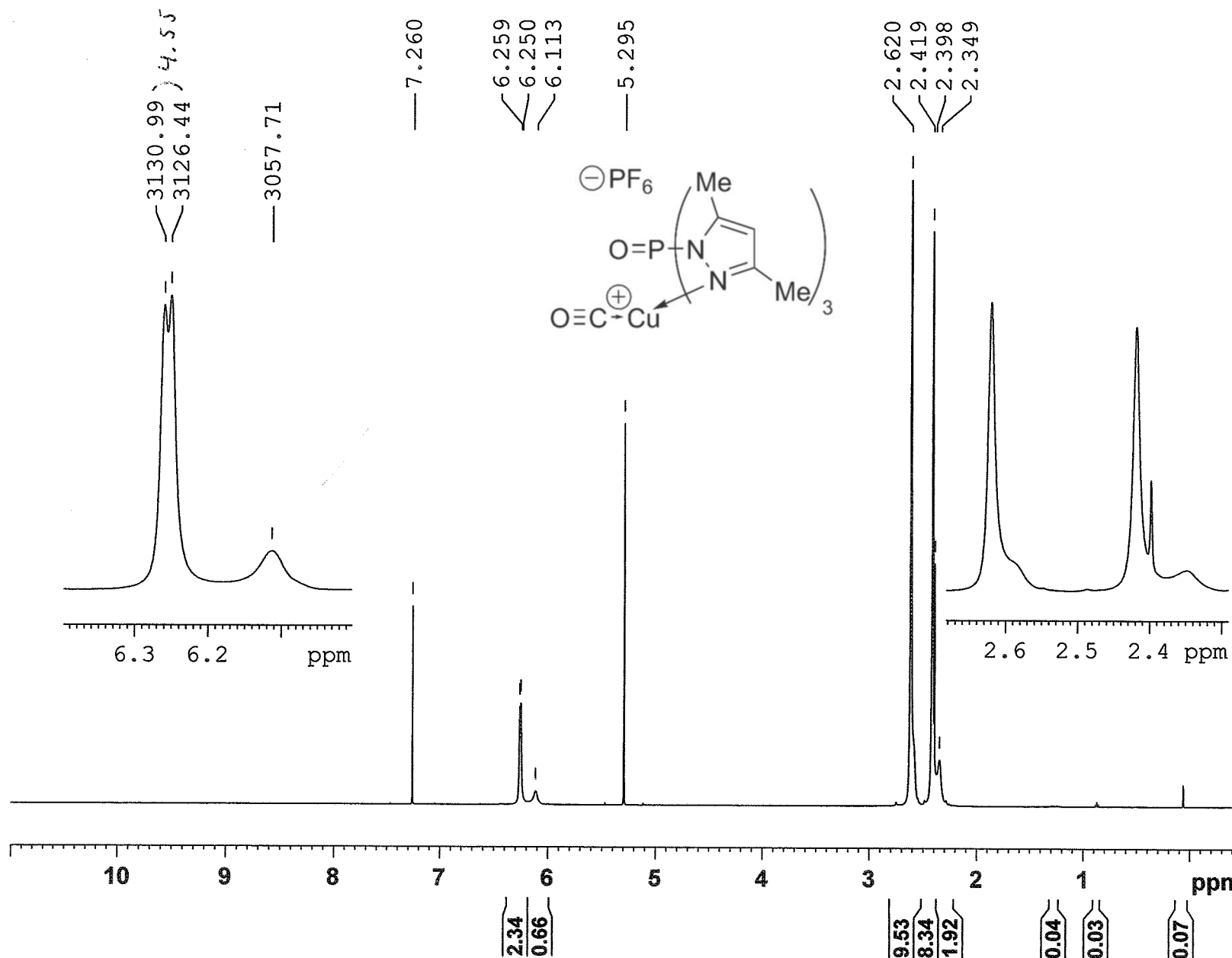
OP(pzMe₂)₃CuNCMe + CO: After ~1 month stirring

-Fresh sample (also 250exp7-9) -



NAME CGJT-10-28_500
EXPNO 4
PROCNO 1
Date 20110614
Time 13.26
INSTRUM spect
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 65536
SOLVENT CDCl₃
NS 8
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 18
DW 50.000 usec
DE 6.50 usec
TE 296.0 K
D1 1.70000005 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 6.70 usec
PL1 4.00 dB
PL1W 8.72000027 W
SFO1 500.2335016 MHz
SI 65536
SF 500.2300097 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

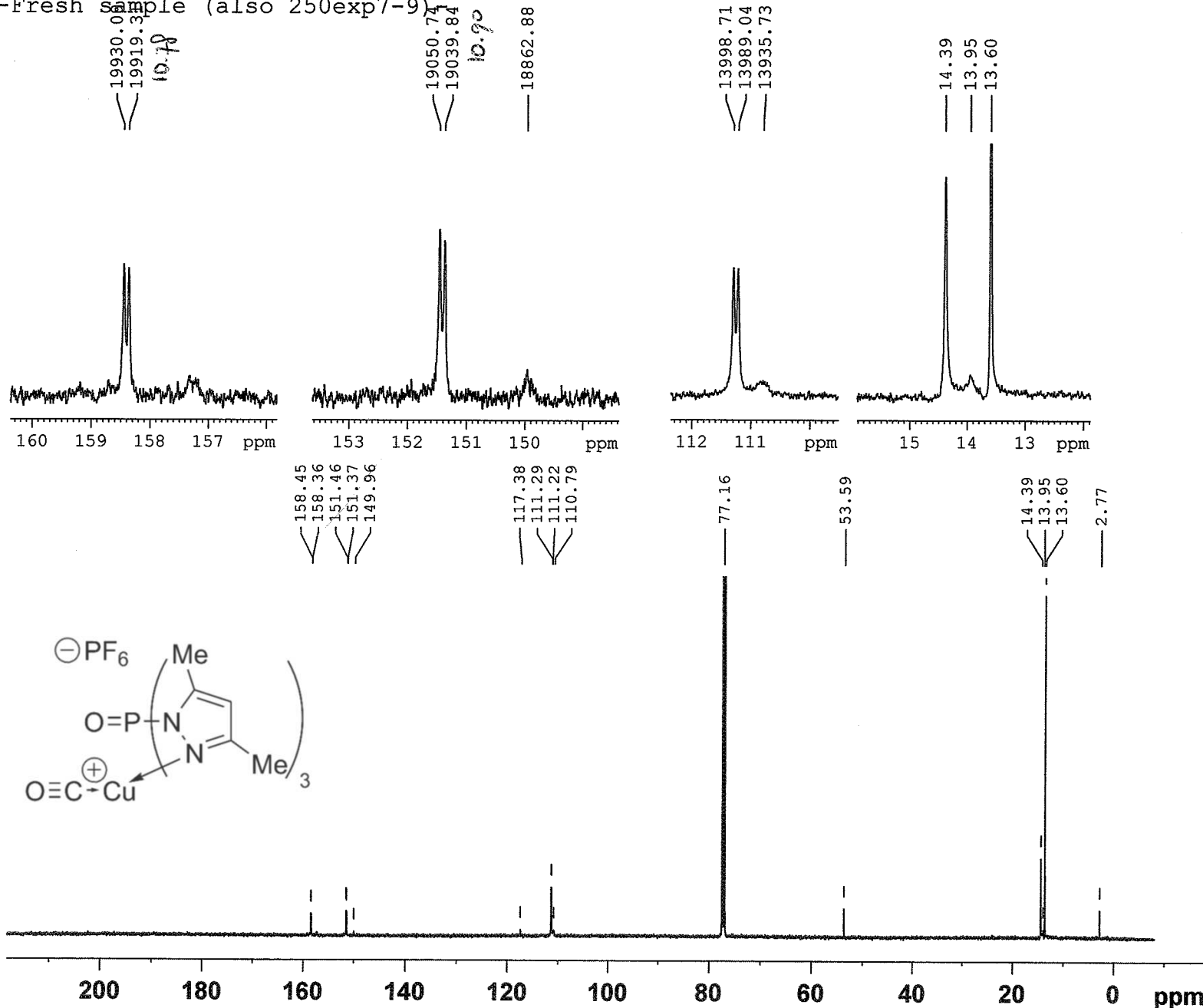


$^{13}\text{C}\{^1\text{H}\}$ NMR of $[\text{OP}(3,5\text{-Me}_2\text{pz})_3\text{Cu}(\text{CO})][\text{PF}_6]$ (**4a**)

$^{13}\text{C}\{^1\text{H}\}$ (CDCl₃ GB)

OP(pzMe₂)₃CuNCMe + CO: After ~1 month stirring

-Fresh sample (also 250exp7-9)



NAME CGJT-10-28_500
EXPNO 5
PROCNO 1
Date_ 20110614
Time_ 13.32
INSTRUM spect
PROBHD 5 mm CPTCI 1H-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 416
DS 2
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010548 sec
RG 2050
DW 16.800 usec
DE 6.50 usec
TE 296.0 K
D1 2.0000000 sec
D11 0.0300000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 ¹³C
P1 11.20 usec
PL1 -2.00 dB
PL1W 88.77790070 W
SFO1 125.7967701 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 ¹H
PCPD2 80.00 usec
PL2 4.00 dB
PL12 25.28 dB
PL13 28.00 dB
PL2W 8.72000027 W
PL12W 0.06494062 W
PL13W 0.03471494 W
SFO2 500.2320009 MHz
SI 65536
SF 125.7829179 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

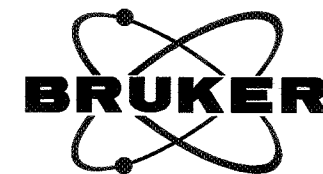
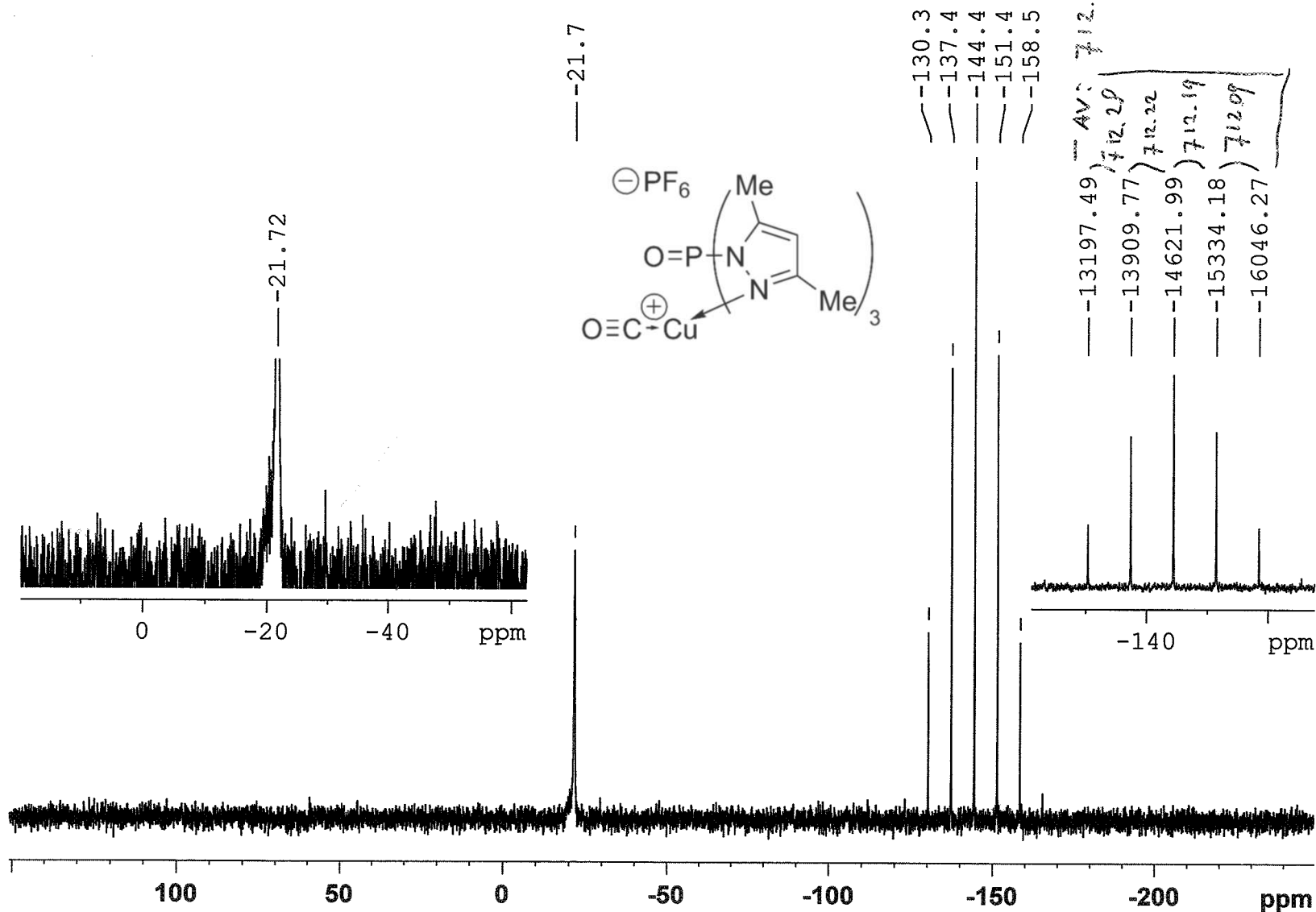
³¹P{¹H} NMR of [OP(3,5-Me₂pz)₃Cu(CO)][PF₆] (4a)

³¹P{¹H} (CDCl₃ GB)

OP(Me₂-pz)₃Cu-NCMe + CO after 1 month exposure

-Sample of exp7-9 and _500exp1-4 after some days in GB-

31PCPD_NO_processing CDCl₃ {D:\NMRDATA} Nielst 34



NAME CGJT-10-28
 EXPNO 13
 PROCNO 1
 Date 20110617
 Time 10.56
 INSTRUM spect
 PROBHD 5 mm QNP 1H/1
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl₃
 NS 64
 DS 4
 SWH 40650.406 Hz
 FIDRES 0.620276 Hz
 AQ 0.8061428 sec
 RG 20642.5
 DW 12.300 usec
 DE 6.00 usec
 TE 300.2 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 DELTA 1.89999998 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 31P
 P1 7.00 usec
 PL1 0.00 dB
 SFO1 101.2494172 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 -3.00 dB
 PL12 20.00 dB
 PL13 24.00 dB
 SFO2 250.1310005 MHz
 SI 65536
 SF 101.2544800 MHz
 WDW EM
 SSB 0
 LB 3.00 Hz
 GB 0
 PC 1.40

¹⁹F NMR of [OP(3,5-Me₂pz)₃Cu(CO)][PF₆] (4a)

¹⁹F (CDCl₃ GB)

OP(Me₂-pz)₃Cu-NCMe + CO after 1 month exposure

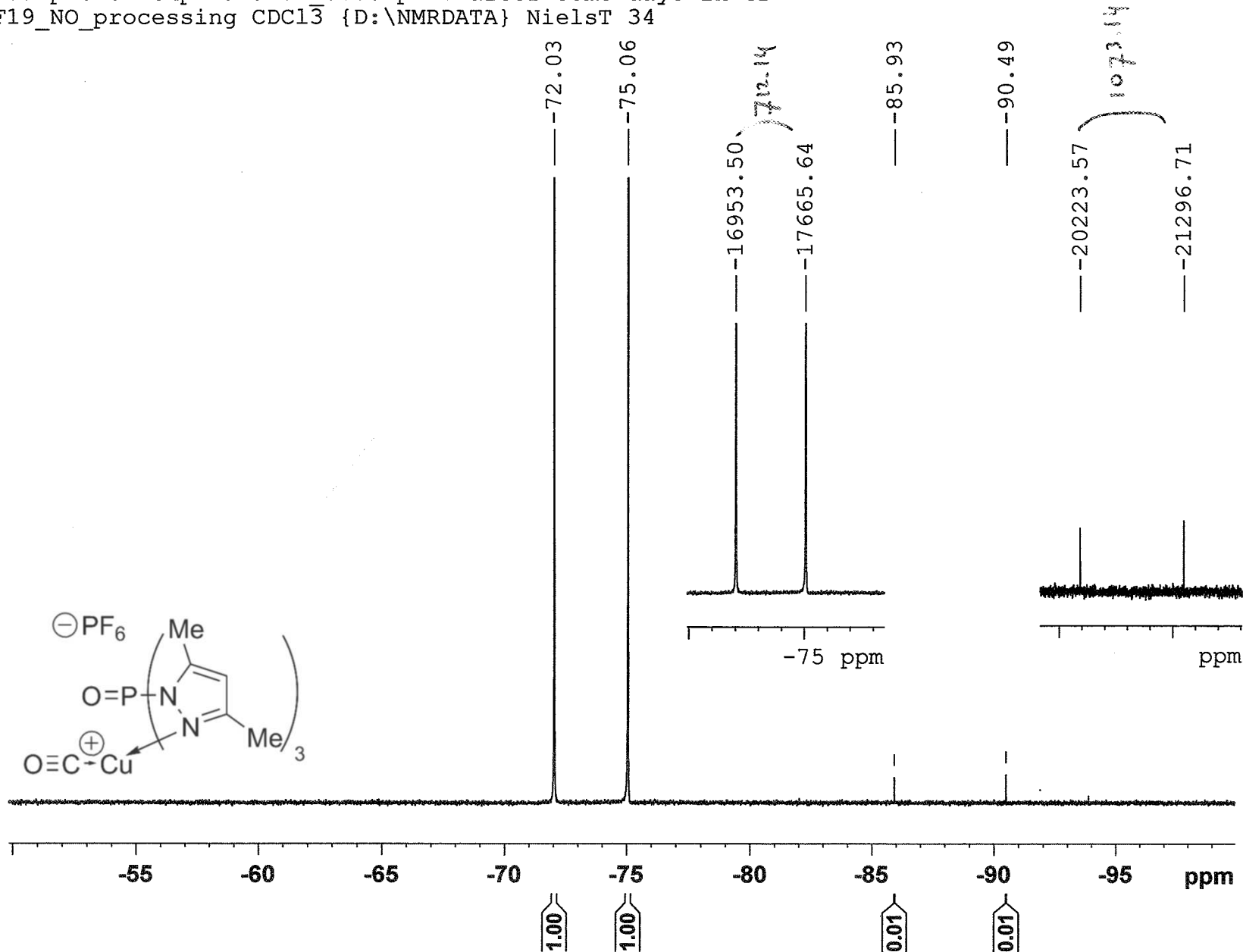
-Sample of exp7-9 and _500exp1-4 after some days in GB-

F19_NO_processing CDCl₃ {D:\NMRDATA} NielsT 34



NAME CGJT-10-28
EXPNO 11
PROCNO 1
Date_ 20110617
Time_ 10.49
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgflgn
TD 131072
SOLVENT CDCl₃
NS 64
DS 4
SWH 56497.176 Hz
FIDRES 0.431039 Hz
AQ 1.1600372 sec
RG 9195.2
DW 8.850 usec
DE 6.00 usec
TE 300.2 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 19F
P1 8.00 usec
PL1 -3.00 dB
SFO1 235.3338140 MHz
SI 65536
SF 235.3573500 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



¹H NMR of [OP(3-*t*-Bupz)₃Cu(CO)][PF₆] (**4c**)

¹H (CDCl₃ GB)

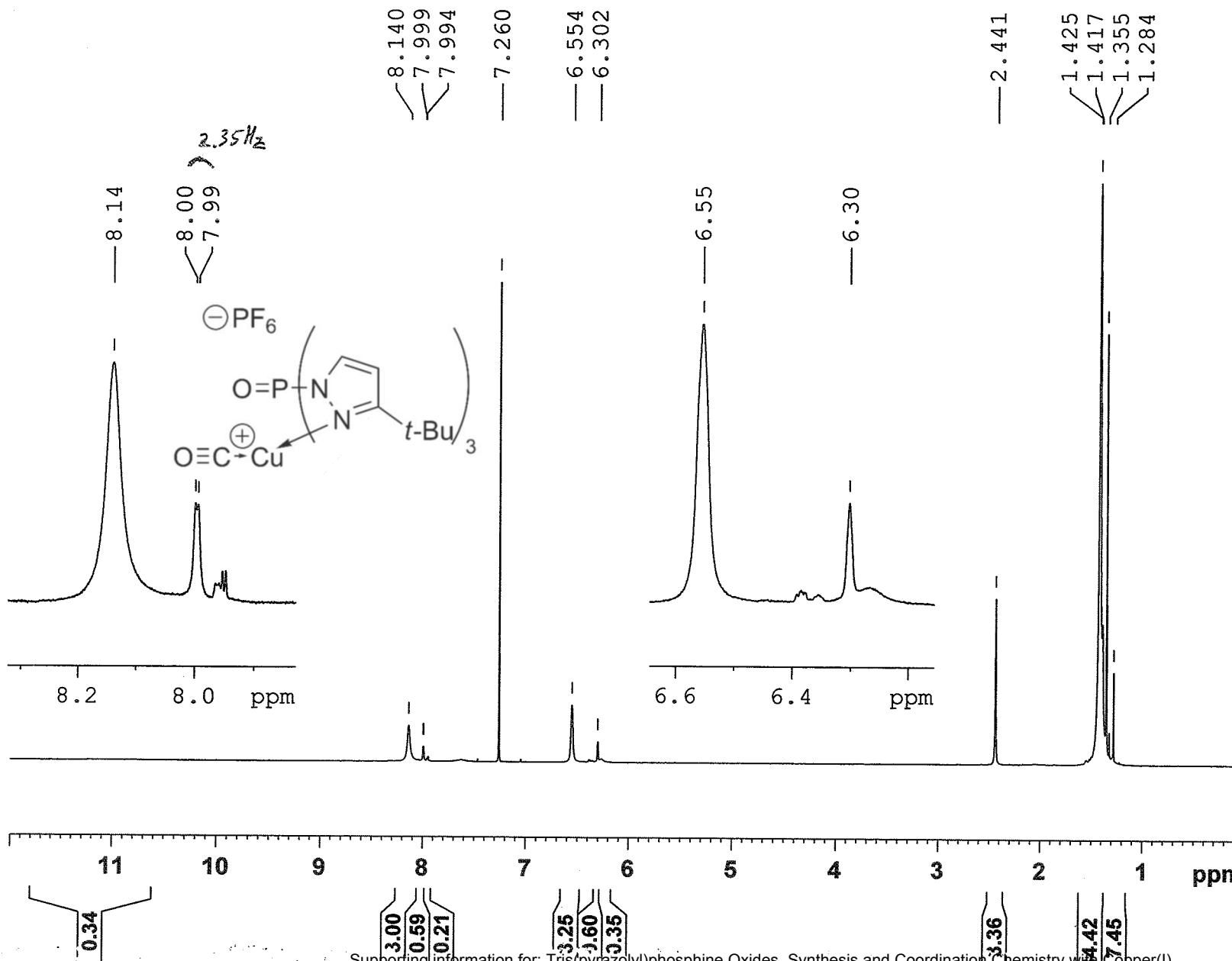
OP(*t*Bu-pz)₃CuNCMe + CO: After ~1 month stirring

-Fresh sample (6 mg), clear sln-



NAME CGJT-10-26_500
EXPNO 1
PROCNO 1
Date 20110615
Time 17.25
INSTRUM spect
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 65536
SOLVENT CDCl₃
NS 8
DS 2
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 18
DW 50.000 usec
DE 6.50 usec
TE 296.0 K
D1 1.70000005 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 6.70 usec
PL1 4.00 dB
PL1W 8.72000027 W
SFO1 500.2335016 MHz
SI 65536
SF 500.2300096 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

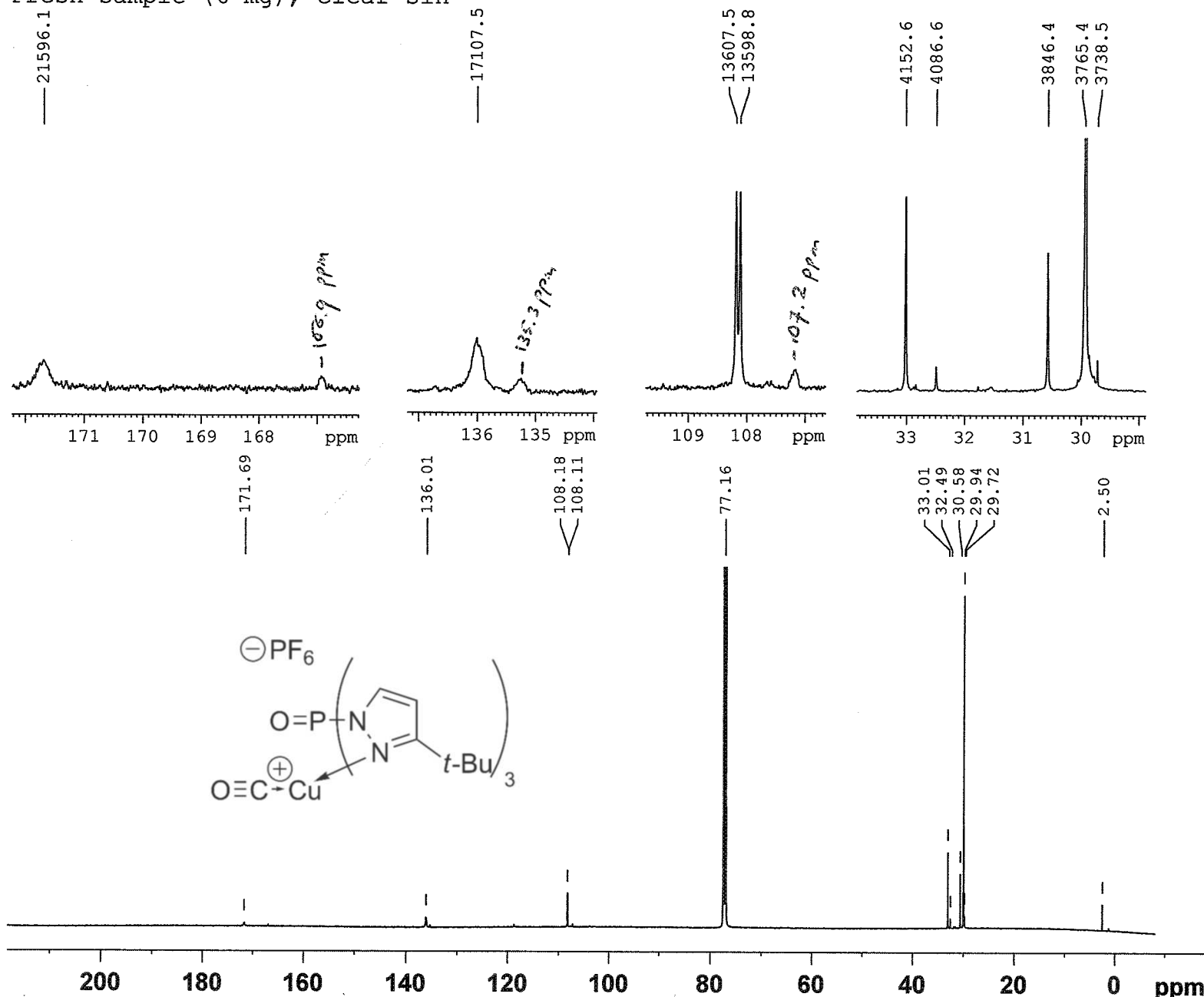


$^{13}\text{C}\{^1\text{H}\}$ NMR of $[\text{OP}(3\text{-}t\text{-Bupz})_3\text{Cu}(\text{CO})][\text{PF}_6]$ (**4c**)

$^{13}\text{C}\{^1\text{H}\}$ (CDCl₃ GB)

exp C: OP(tBu-pz)₃CuNCMe + CO: After ~1 month stirring

-Fresh sample (6 mg), clear sln-



NAME CGJT-10-26_500
 EXPNO 2
 PROCNO 1
 Date_ 20110615
 Time 17.34
 INSTRUM spect
 PROBHD 5 mm CPTCI 1H-
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 16384
 DS 2
 SWH 29761.904 Hz
 FIDRES 0.454131 Hz
 AQ 1.1010548 sec
 RG 2050
 DW 16.800 usec
 DE 6.50 usec
 TE 296.0 K
 D1 2.0000000 sec
 D11 0.03000000 sec
 TDO 1

===== CHANNEL f1 =====
 NUC1 13C
 P1 11.20 usec
 PL1 -2.00 dB
 PL1W 88.77790070 W
 SFO1 125.7967701 MHz

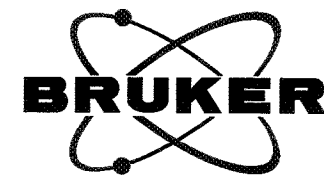
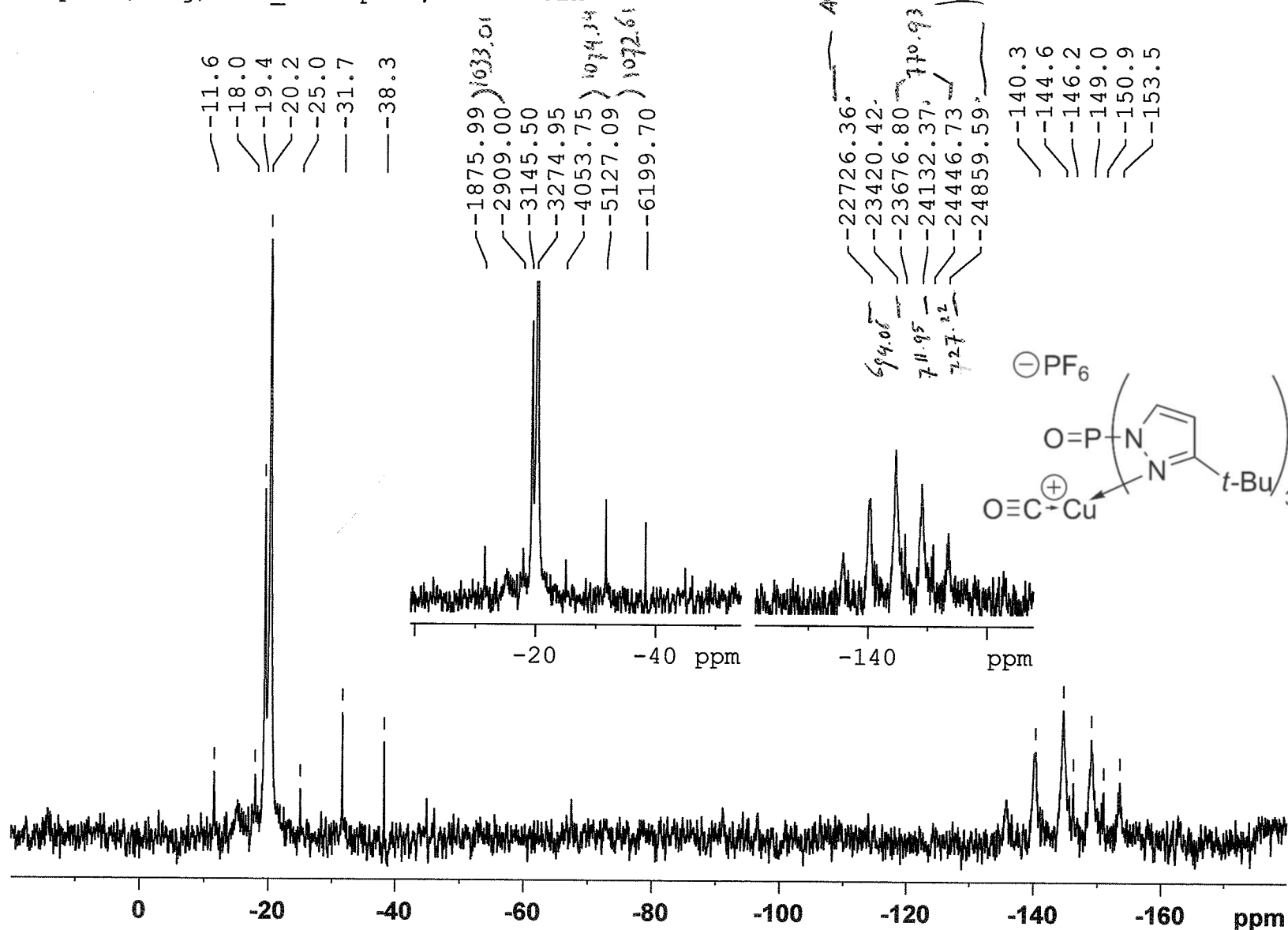
===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 4.00 dB
 PL12 25.28 dB
 PL13 28.00 dB
 PL2W 8.72000027 W
 PL12W 0.06494062 W
 PL13W 0.03471494 W
 SFO2 500.2320009 MHz
 SI 65536
 SF 125.7829150 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

³¹P{¹H} NMR of [OP(3-*t*-Bupz)₃Cu(CO)][PF₆] (4c)

³¹P {¹H} (CDC13 GB)

OP(*t*Bu-pz)₃CuNCMe + CO: After ~1 month stirring

-Sample (6 mg) of _500expl-4, clear sln-



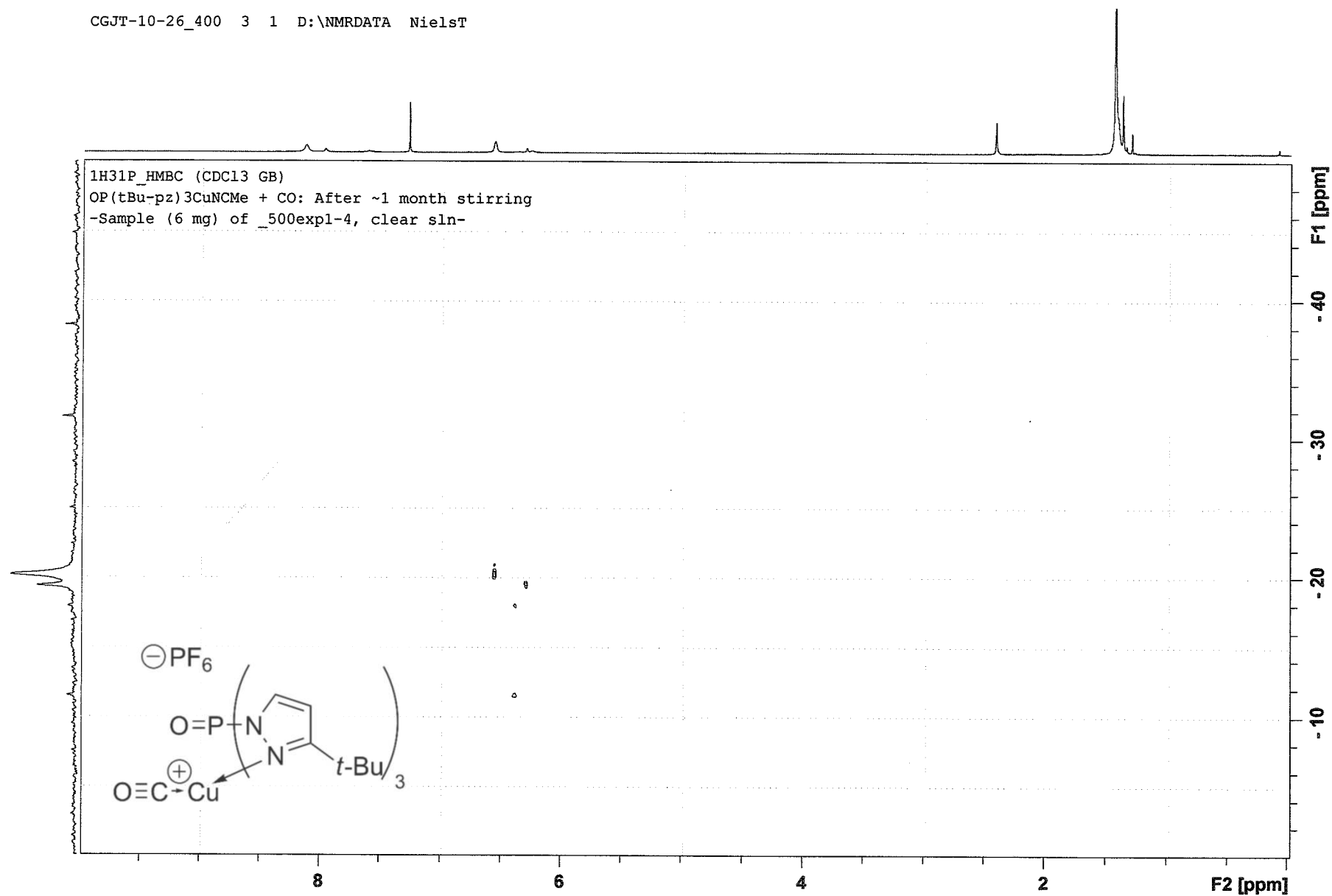
NAME CGJT-10-26_400
 EXPNO 2
 PROCNO 1
 Date 20110616
 Time 12.30
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 512
 DS 4
 SWH 32467.533 Hz
 FIDRES 0.495415 Hz
 AQ 1.0093044 sec
 RG 2580.3
 DW 15.400 usec
 DE 6.00 usec
 TE 300.4 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 DELTA 1.89999998 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 ³¹P
 P1 6.25 usec
 PL1 -1.00 dB
 SFO1 161.9626350 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 ¹H
 PCPD2 80.00 usec
 PL2 3.00 dB
 PL12 18.00 dB
 PL13 18.00 dB
 SFO2 400.1316005 MHz
 SI 32768
 SF 161.9755930 MHz
 WDW EM
 SSB 0
 LB 10.00 Hz
 GB 0
 PC 1.40

^1H - ^{31}P HMBC NMR of $[\text{OP}(3\text{-}t\text{-Bupz})_3\text{Cu}(\text{CO})][\text{PF}_6]$ (**4c**)

CGJT-10-26_400 3 1 D:\NMRDATA Nielst



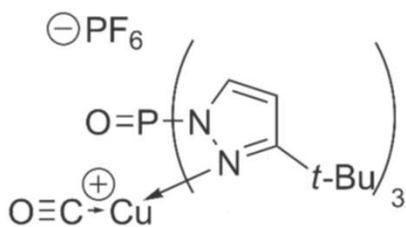
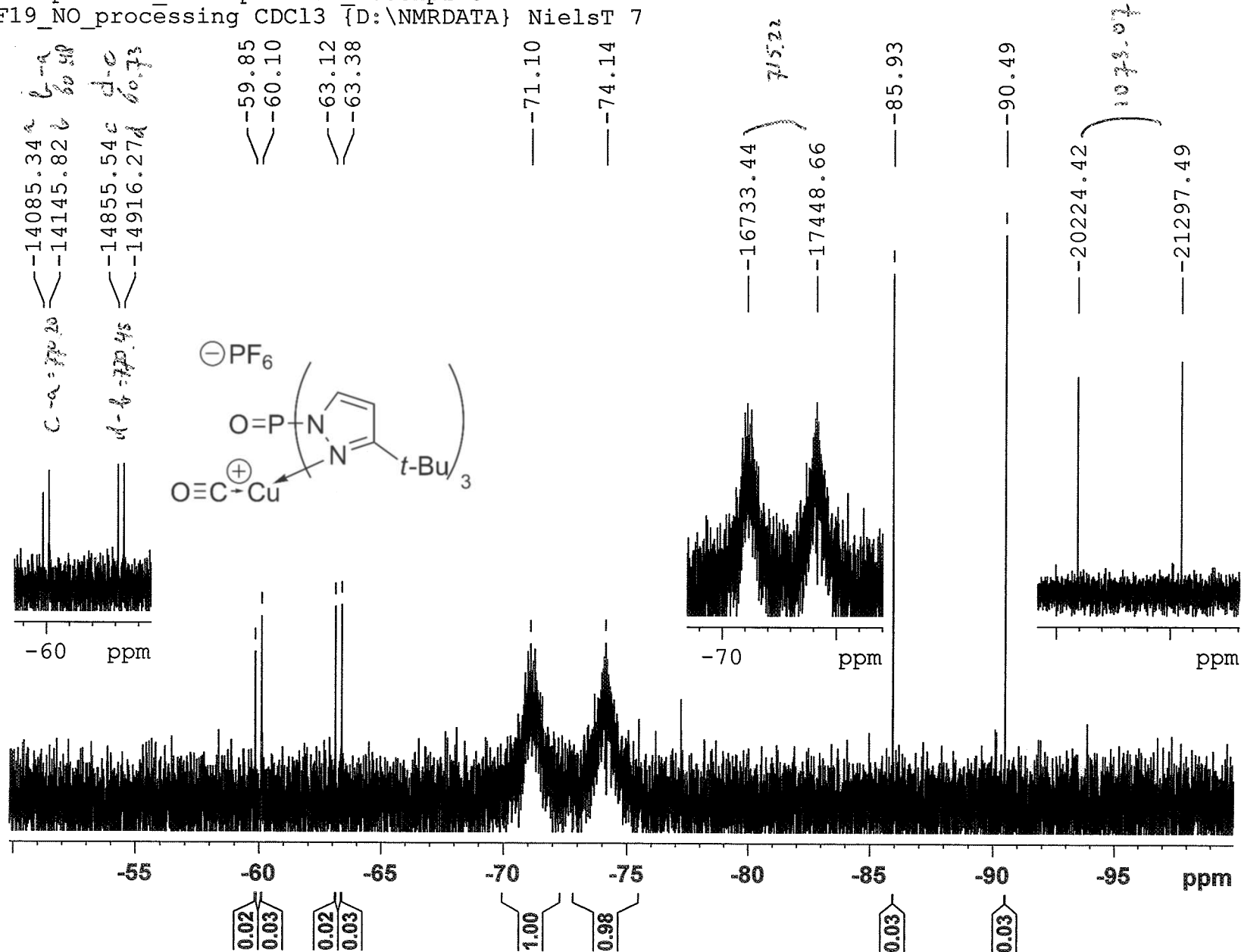
$$^{19}\text{F} \text{ NMR of } [\text{OP}(3\text{-}t\text{-Bupz})_3\text{Cu}(\text{CO})][\text{PF}_6] \text{ (4c)}$$

19F (CDC13 GB)

OP(tBu-pz)₃Cu-NCMe PF₆ + CO. Exp C.

-Sample of 500exp1-4 & 400exp1-3-

F19 NO processing CDC13 {D:\NMRDATA} NielsT 7



NAME	CGJT-10-26	
EXPNO	15	
PROCNO	1	
Date_	20110616	
Time	13.09	
INSTRUM	spect	
PROBHD	5 mm QNP	1H/1
PULPROG	zgpg1qn	
TD	131072	
SOLVENT	CDCl3	
NS	64	
DS	4	
SWH	56497.176	Hz
FIDRES	0.431039	Hz
AQ	1.1600372	sec
RG	10321.3	
DW	8.850	usec
DE	6.00	usec
TE	300.2	K
D1	1.00000000	sec
TD0	1	

```

===== CHANNEL f1 =====
NUC1                19F
P1                   8.00 usec
PL1                  -3.00 dB
SF01                235.3338140 MHz
SI                   65536
SF                  235.3573500 MHz
WDW                  EM
SSB                   0
LB                   0.30 Hz
GB                   0
PC                   1.00

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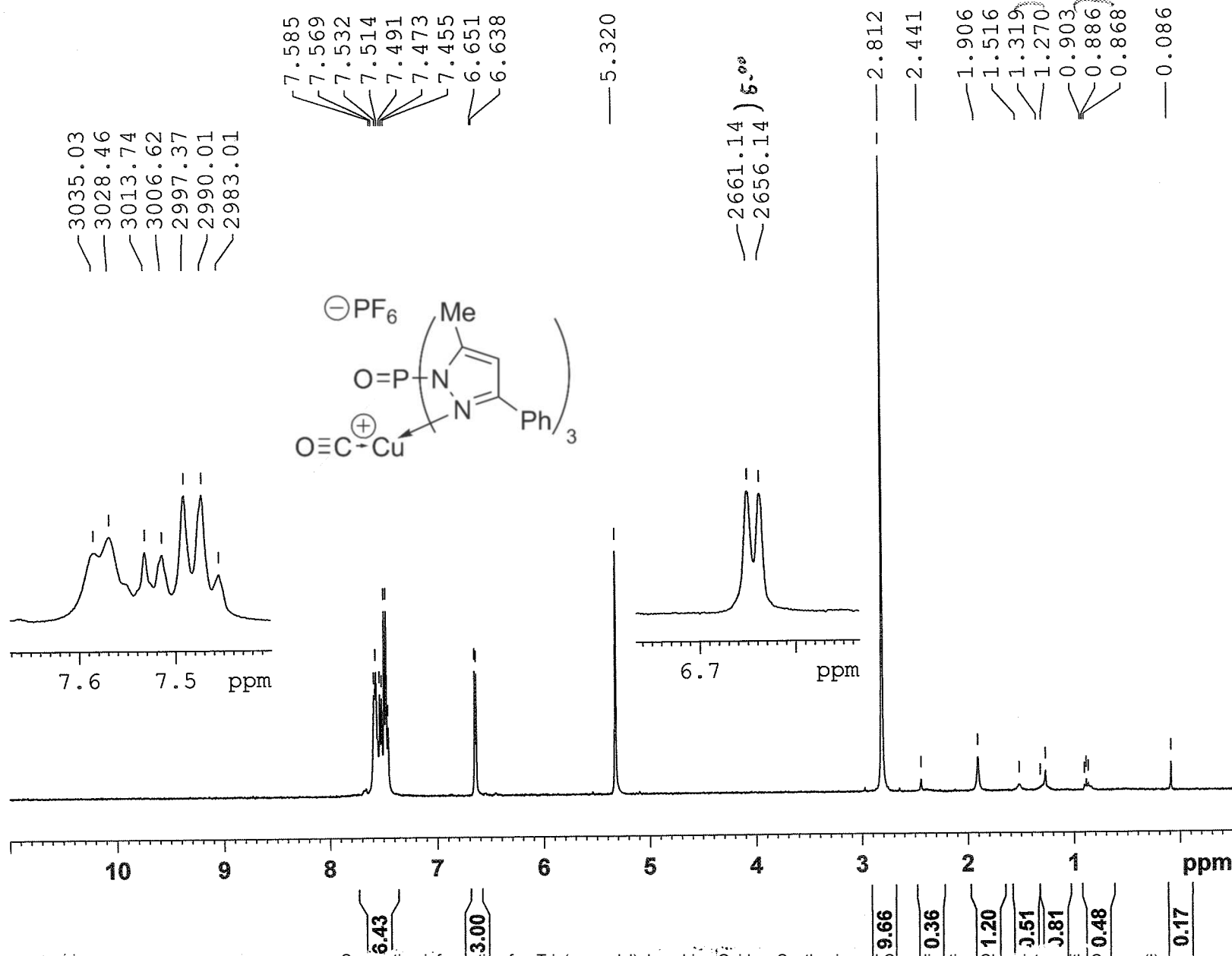
¹H NMR of [OP(3-Ph-5-Mepz)₃Cu(CO)][PF₆] (4d)

(Me, Ph-Pz) 3PO-Cu(CO) PF₆



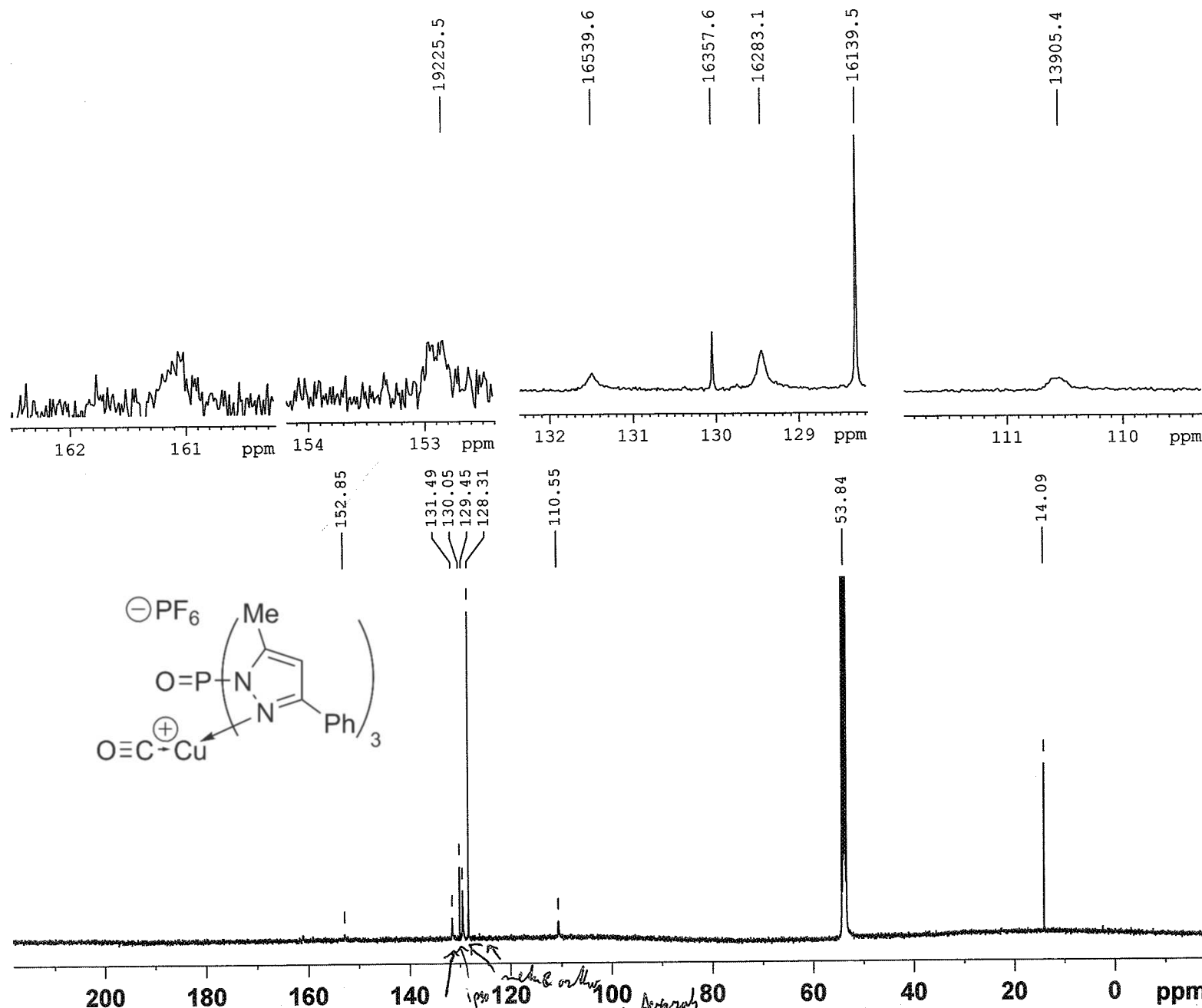
NAME VVL 349_400
 EXPNO 1
 PROCNO 1
 Date_ 20100915
 Time_ 17.04
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zg30
 TD 65536
 SOLVENT CD2C12
 NS 16
 DS 2
 SWH 8278.146 Hz
 FIDRES 0.126314 Hz
 AQ 3.9584243 sec
 RG 512
 DW 60.400 usec
 DE 6.00 usec
 TE 300.4 K
 D1 1.00000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 13.00 usec
 PL1 3.00 dB
 SFO1 400.1324710 MHz
 SI 32768
 SF 400.1300149 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



$^{13}\text{C}\{^1\text{H}\}$ NMR of $[\text{OP}(\text{3-Ph-5-Mepz})_3\text{Cu}(\text{CO})][\text{PF}_6]$ (**4d**)

(Me, Ph-Pz) 3-PO-CuNCMe PF6 + CO



NAME VVL 28_500
 EXPNO 1
 PROCNO 1
 Date_ 20100915
 Time_ 15.46
 INSTRUM spect
 PROBHD 5 mm CPTCI 1H-
 PULPROG zgpg30
 TD 65536
 SOLVENT CD2Cl2
 NS 4410
 DS 4
 SWH 50000.000 Hz
 FIDRES 0.762939 Hz
 AQ 0.6554100 sec
 RG 2050
 DW 10.000 usec
 DE 6.50 usec
 TE 295.9 K
 D1 2.00000000 sec
 D11 0.03000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 13C
 P1 11.20 usec
 PL1 -2.00 dB
 PL1W 88.77790070 W
 SFO1 125.7955118 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 80.00 usec
 PL2 4.00 dB
 PL12 25.28 dB
 PL13 28.00 dB
 PL2W 8.72000027 W
 PL12W 0.06494062 W
 PL13W 0.03471494 W
 SFO2 500.2320009 MHz
 SI 32768
 SF 125.7828811 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

$^{31}\text{P}\{^1\text{H}\}$ NMR of $[\text{OP}(3\text{-Ph-5-Mepz})_3\text{Cu}(\text{CO})][\text{PF}_6]$ (**4d**)

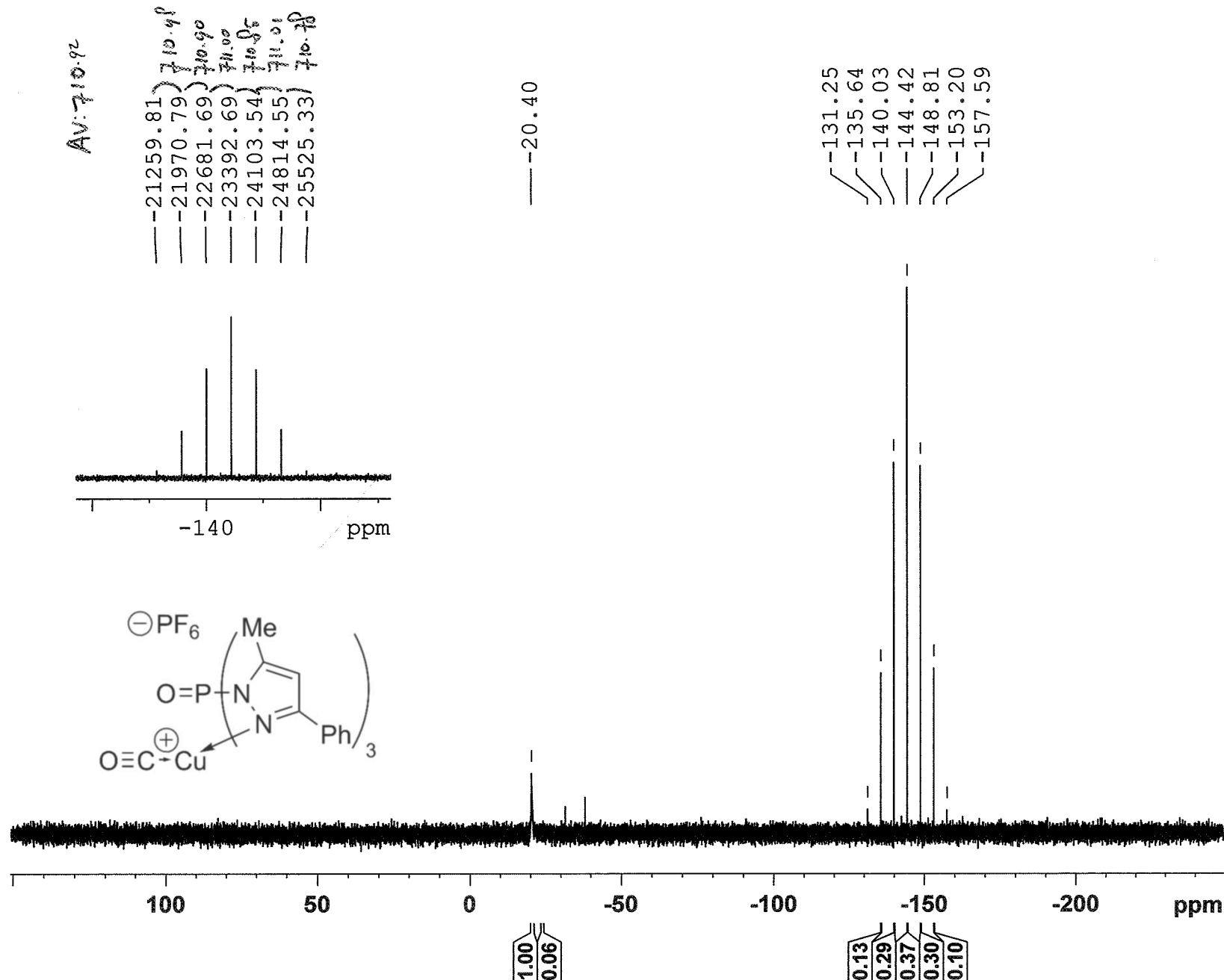
(Me, Ph-Pz) $3\text{PO-Cu}(\text{CO}) \text{ PF}_6$



NAME VVL 349_400
 EXPNO 3
 PROCNO 1
 Date_ 20100916
 Time 3.37
 INSTRUM spect
 PROBHD 5 mm PABBO BB/
 PULPROG zgpg30
 TD 65536
 SOLVENT CD2Cl2
 NS 1024
 DS 4
 SWH 64935.066 Hz
 FIDRES 0.990830 Hz
 AQ 0.5046772 sec
 RG 14596.5
 DW 7.700 usec
 DE 6.00 usec
 TE 300.4 K
 D1 2.00000000 sec
 d11 0.03000000 sec
 DELTA 1.89999998 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 ^{31}P
 P1 6.25 usec
 PL1 -1.00 dB
 SFO1 161.9674942 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 ^1H
 PCPD2 80.00 usec
 PL2 3.00 dB
 PL12 18.00 dB
 PL13 18.00 dB
 SFO2 400.1316005 MHz
 SI 65536
 SF 161.9755930 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40



¹⁹F NMR of [OP(3-Ph-5-Mepz)₃Cu(CO)][PF₆] (4d)

(Me, Ph-Pz) 3PO-Cu(CO) PF₆

F19CPD CD₂Cl₂ {D:\NMRDATA} Volodymyr 18



NAME VVL 464
EXPNO 2
PROCNO 1
Date_ 20100916
Time 10.08
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgfhigqn
TD 131072
SOLVENT CD₂Cl₂
NS 32
DS 4
SWH 56497.176 Hz
FIDRES 0.431039 Hz
AQ 1.1600372 sec
RG 11585.2
DW 8.850 usec
DE 6.00 usec
TE 300.2 K
D1 1.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 19F
P1 8.00 usec
PL1 -3.00 dB
SFO1 235.3338140 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -3.00 dB
PL12 20.00 dB
SFO2 250.1310005 MHz
SI 65536
SF 235.3573500 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.20

