Diels-Alder Cycloaddition as a New Approach Toward Stable PC(sp^3)P-Metalated Compounds

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General Considerations. All manipulations were performed using standard Schlenk techniques under dry N_2. All reagents were purchased from the usual suppliers and used without further purification. 1,8-bis(diphenylphosphino)anthracene[1] was synthesized according the previously published procedures. All reagents were weighed and handled in air. Flash column chromatography was performed with Merck ultra pure silica gel (230-400 mesh). All catalytic reactions were carried out under air in single use screw-capped tubes. Yields refer to isolated yields of compounds greater than 95% purity as determined proton Nuclear Magnetic Resonance spectroscopy (^{1}H-NMR) analysis. The CAS numbers of the known compound were listed. Spectroscopy data of the known compounds matches with the data reported in the corresponding reference. ^{1}H-NMR and ^{13}C-NMR spectra were recorded on a Bruker 400 MHz instrument with chemical shifts reported in ppm relative to the residual deuterated solvent or the internal standard tetramethylsilane. For the thermogravimetric analysis (TGA), a Mettler TC10A/TC15 TA controller and Mettler M3 thermobalance were used. Samples of 10-20 mg were weighed and heated to 30-500 °C at a rate of 10 °C/min. Diffraction data were collected with a Bruker APEX CCD instrument (MoKα radiation (λ = 0.71073Å)). Crystals were mounted onto glass fibers using epoxy. Single crystal reflection data were collected on a Bruker APEX CCD X-ray diffraction system controlled by a Pentium-based PC running the SMART software package.[2] The integration of data frames and refinement of cell structure were done by the SAINT+ program package.[3] Refinement of the structure **4** on F^2 was carried out by the SHELXTL software package.[4] For compound **5**, solvent molecules were disordered and, therefore, were refined isotropically using SQUEEZE program.[5]

1,8-Bis (diphenylphosphino)-9-anthrylpalladium(II) Chloride [1] **(1)**
(CAS Registry No.: 131276-28-3).
The suspension of 500 mg (0.73 mmol) of 1,8-bis(diphenylphosphino)anthracene and 189.1 mg (0.73 mmol) of $PdCl_2(CH_3CN)_2$ in 20 ml of ethylene glycol monomethyl ether was heated at reflux for 2 hours under nitrogen. After cooling to RT, the green precipitate was filtered off, washed twice with methanol and dried in high vacuum to yield 442 mg (88%) of **1**. ^{1}H NMR, 400 MHz ($CDCl_3$), δ: 7.40-7.50 (12H, m); 7.58 (2H, t, *J*=7.3 Hz); 7.77 (2H, dd, *J*=5.1, *J*=11.1 Hz); 7.96 (8H, m); 8.12 (2H, d, *J*=8.3 Hz); 8.33 (1H, s). ^{31}P NMR, 400 MHz ($CDCl_3$), δ: 42.92 ^{13}C NMR, 400 ($CDCl_3$), δ: 122.6, 125.9 (t, *J*=4.8 Hz), 128.8 (t, *J*=5.2 Hz), 130.7, 131.3 (t, *J*=22.5 Hz), 131.6 (d, *J*=7.3 Hz), 133.8 (t, *J*=7.3 Hz), 138.6, 146.5 (t, *J*=22.4 Hz). Anal. Calcd. For $C_{38}H_{27}ClP_2Pd$: C, 66.39; H, 3.96. Found: C, 66.28; H, 3.81.

1,8-Bis (diphenylphosphino)-9-anthrylnickel(II) Chloride [1] **(2)**
(CAS Registry No.: 131276-26-1).
The suspension of 500 mg (0.73 mmol) of 1,8-bis(diphenylphosphino)anthracene and 160.4 mg (0.73 mmol) of $NiCl_2$(dme) in 20 ml of ethylene glycol monomethyl ether was added 0.12 ml (0.73mmol) of diisopropylethylamine and the mixture heated at reflux for 2 hours under nitrogen. After cooling to room temperature the yellow precipitate was filtered off, washed twice with methanol and dried in high vacuum to yield 280 mg (60%) of **2**. ^{1}H NMR, 400 MHz ($CDCl_3$), δ: 7.40-7.45 (12H, m); 7.54 (2H, t, *J*=10.1 Hz); 7.68 (2H, dd, *J*=5.0, *J*=9.1 Hz); 7.95 (8H, m); 8.07 (2H, d, *J*=9.1 Hz); 8.24 (1H, s). ^{31}P NMR, 400 MHz ($CDCl_3$), δ: 41.87 ^{13}C NMR, 400 ($CDCl_3$), δ: 121.8, 126.1 (t, *J*=3.6 Hz), 128.6 (t, *J*=5.10 Hz), 129.1, 130.5, 131.1 (d, *J*=22.1 Hz), 133.7 (t, *J*=6.4 Hz), 139.0, 148.5. Anal. Calcd. for $C_{38}H_{27}ClP_2Ni$: C, 71.35; H, 4.25. Found: C, 71.29; H, 4.44.

1,8-Bis (diphenylphosphino)-9-anthrylplatinum(II) Chloride (3)
The suspension of 500 mg (0.73 mmol) of 1,8-bis(diphenylphosphino)anthracene and 254 mg (0.73 mmol) of $PtCl_2(CH_3CN)_2$ in 20 ml of ethylene glycol monomethyl ether was added 0.12 ml (0.73mmol) of diisopropylethylamine and the mixture heated at reflux for 2 hours under nitrogen. After cooling to room temperature the green precipitate was filtered off, washed twice with methanol and dried in high vacuum to yield 368 mg (65%) of **3**. ^{1}H NMR, 400 MHz ($CDCl_3$), δ: 7.45 (12H, m); 7.54 (2H, t, J=7.6 Hz); 7.8 (2H, dd, *J*=5.7, *J*=11.8 Hz); 7.96 (8H, m); 8.12 (2H, d, J=8.0 Hz); 8.37 (1H, s). ^{31}P NMR, 400 MHz ($CDCl_3$), δ: 39.65 (t, *J*=1495.3 Hz). ^{13}C NMR, 400 ($CDCl_3$), δ: 120.64, 126.1 (t, *J*=4.9 Hz), 128.7 (t, *J*=5.5 Hz), 130.8, 131.3 (d, *J*=27.4 Hz), 133.0 (t, *J*=7.7 Hz),133.8 (t, *J*=6.9 Hz), 138.6 (t, *J*=6.9 Hz), 145.5 (t, *J*=18.1 Hz). Anal. Calcd. For $C_{38}H_{27}ClP_2Pt$: C, 58.81; H, 3.51. Found: C, 58.64; H, 3.58.

Synthesis of complex 4
To the suspension of 200 mg (0.29 mmol) of **1** in 5 ml of diethylene glycol dimethyl ether (diglyme), 0.053 ml (0.44 mmol) of dimethyl acetylenedicarboxylate (DMAD) was added and the mixture heated at reflux for 24 hours under N_2. After cooling down to RT the solvent was removed by evaporation under reduced pressure. Addition of methanol results in the formation of a dark-green precipitate. The solid was filtered off, washed twice with methanol and dried in high vacuum to yield 154 mg (64%) of **4**. ^{1}H NMR, 400 MHz ($CDCl_3$), δ: 3.20 (3H, s); 3.61 (3H, s); 5.79 (1H, s); 7.23 (4H, m); 7.30 (4H, t, *J*=7.1 Hz); 7.39 (2H, t, *J*=7.8 Hz); 7.47 (6H, m); 7.55-7.61 (6H, m); 7.82 (4H, m). ^{31}P NMR, 400 MHz ($CDCl_3$), δ: 41.88 ^{13}C NMR, 400 ($CDCl_3$), δ: 50.5, 51.9 (d, *J*=45.85 Hz), 126.41,127.1, 128.33 (t, *J*=5.5 Hz),128.6, 128.7 (t, *J*=5.1 Hz),130.3, 130.5, 130.7, 130.9, 133.6 (q, *J*=8.1 Hz), 140.8, 145.4 (t, *J*=10.3Hz),162.4 (d, *J*=20.2 Hz), 164.1, 167.5. Anal. Calcd. for $C_{44}H_{33}ClO_4P_2Pd$: C, 63.71; H, 4.01. Found: C, 63.72; H, 4.22.

Synthesis of complex 5
To the suspension of 200 mg (0.31 mmol) of **2** in 5 ml of diethylene glycol dimethyl ether (diglyme), 0.053 ml (0.47 mmol) of DMAD was added and the mixture heated at reflux for 24 h. After cooling to RT the solvent was removed by evaporation under reduced pressure. Addition of methanol to the mixture results in the formation of a dark-red precipitate. The solid was filtered off, washed twice with methanol and dried in high vacuum to yield 177 mg (73%) of **5**. ^{1}H NMR, 400 MHz ($CDCl_3$), δ: 3.65 (3H, s); 3.71 (3H, s); 5.58 (1H, s); 7.16 (2H, t, *J*=7.3 Hz); 7.23 (6H, t, *J*=7.5

Hz);7.41-7.46 (10H, m); 7.64 (4H, dd, *J*=5.8, *J*=12.6Hz); 7.74 (4H, dd, *J*=6.3, *J*=11.1Hz). ^{31}P NMR, 400 MHz ($CDCl_3$), δ: 32.65 ^{13}C NMR, 400 ($CDCl_3$), δ: 51.9, 52.2 (d, *J*=9.5 Hz), 125.88, 126.87 (t, *J*=2.6 Hz), 127.99, 128.3 (t, *J*=4.8 Hz), 128.6 (t, *J*=4.8 Hz), 130.1 (d, *J*=41.4 Hz), 131.6 (t, *J*=20.1 Hz),132.3 (m), 133.4, 144.1 (t, *J*=8.1 Hz), 146.4, 163.6 (t, *J*=23.1 Hz), 166.7 (d, *J*=76.7 Hz). Anal. Calcd. for $C_{44}H_{33}ClNiO_4P_2$: C, 67.59; H, 4.25 . Found: C, 67.68; H, 4.26.

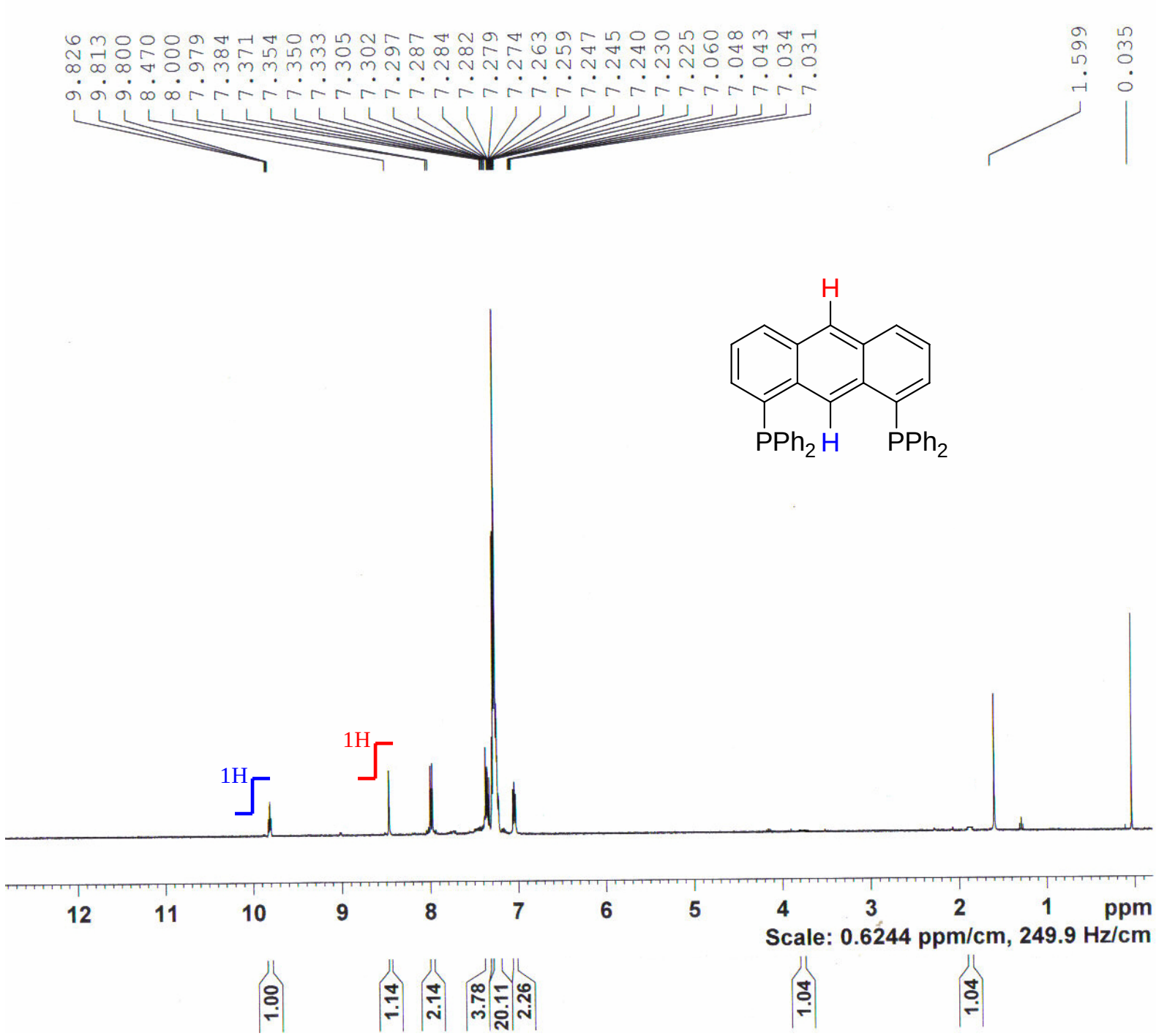
Synthesis of complex 6

To the suspension of 200 mg (0.26 mmol) of **3** in 5 ml of Diethylene glycol dimethyl ether (diglyme), 0.047 ml (0.39 mmol) of DMAD was added and the mixture heated at reflux for 24 h. After cooling to room temp. the solvent was reduced by evaporation under reduced pressure. An addition of methanol to the mixture, results in a light grey precipitate. The solid was filtered off, washed twice with methanol and dried in high vacuum to yield 167 mg (81%) of **6**. ^{1}H NMR, 400 MHz ($CDCl_3$), δ: 3.07 (3H, s); 3.58 (3H, s); 5.82 (1H, s); 7.18-7.33 (8H, m); 7.38 (2H, t, *J*=6.5 Hz); 7.48-7.53 (8H, m); 7.59 (4H, dd, *J*=6.8, *J*=13.3Hz); 7.85 (4H, dd, *J*=6.8, *J*=13.3Hz). ^{31}P NMR, 400 MHz ($CDCl_3$), δ:42.37 (t, *J*=1578.2 Hz). ^{13}C NMR, 400 ($CDCl_3$), δ: 50.3, 51.4 (d, *J*=69.7 Hz), 126.4, 126.9 (t, *J*=3.7 Hz),128.2 (t, *J*=5.1 Hz), 128.7 (t, *J*=5.5 Hz), 130.5 (d, *J*=28.7 Hz), 133.5 (t, *J*=6.6 Hz), 133.8 (t, *J*=7.7 Hz), 140.7, 145.5 (t, *J*=8.1 Hz), 163.3, 164.0, 167.8. Anal. Calcd. for $C_{44}H_{33}ClO_4P_2Pt$: C, 57.55; H, 3.62. Found: C, 57.67; H, 3.81.

References:

1. Haenel, M. W.; Jakubik, D.; Krueger, C.; Betz, P. Chem. Ber. 1991,124, 333.
2. SMART-NT, V. 5.6, Bruker AXS GMBH, Karlsruhe, Germany, 2002.
3. SAINT-NT, V. 5.0, Bruker AXS GMBH, Karlsruhe, Germany, 2002.
4. SHELXTL-NT, V. 6.1, Bruker AXS GMBH, Karlsruhe, Germany, 2002.
5. SQUEEZE - v.d. Sluis, P.; Spek, A. L. *Acta Crystallogr., Sect A* **1990**, *46,* 194.

1H-NMR



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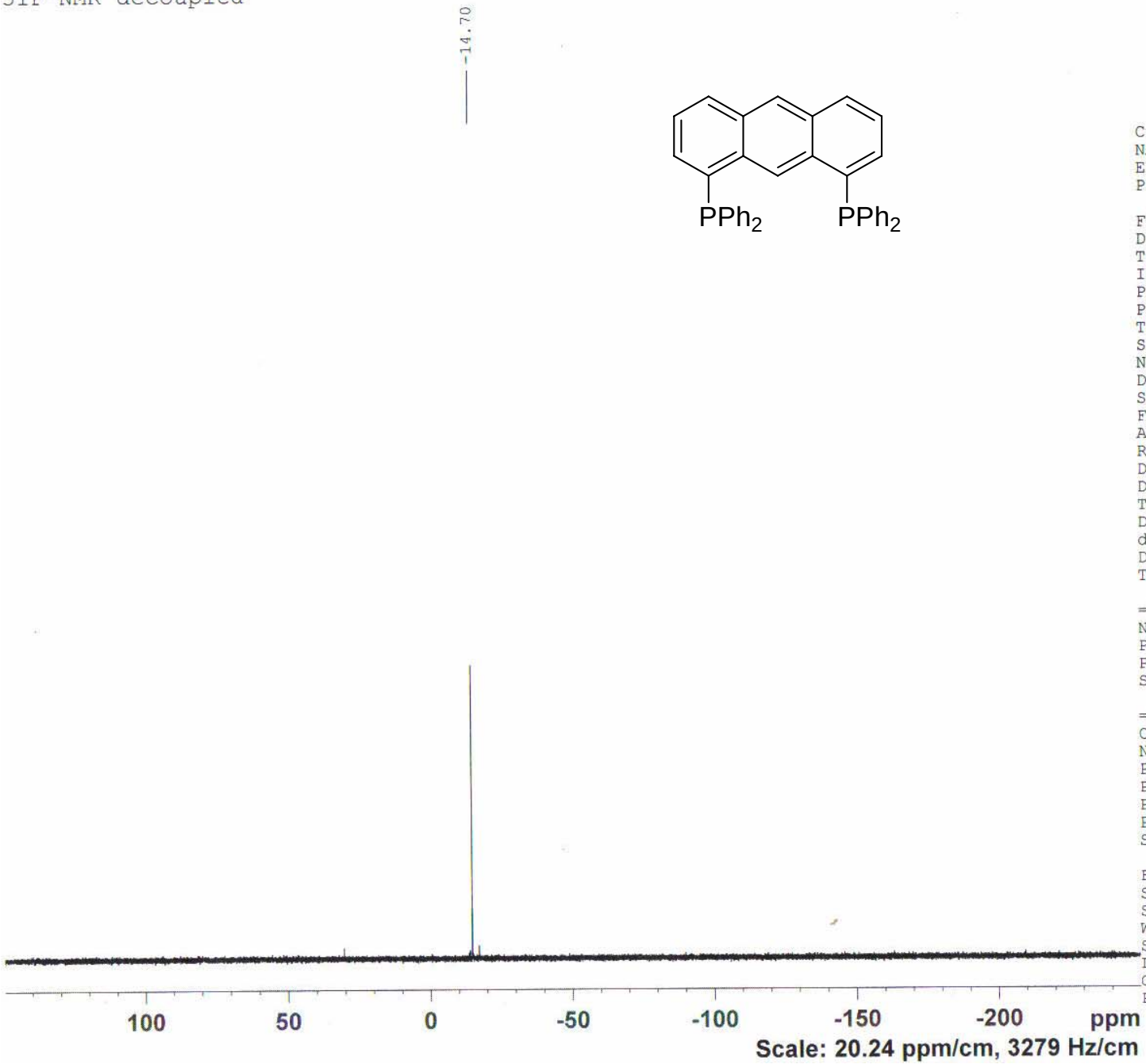
31P-NMR decoupled



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F2 - Processing parameters

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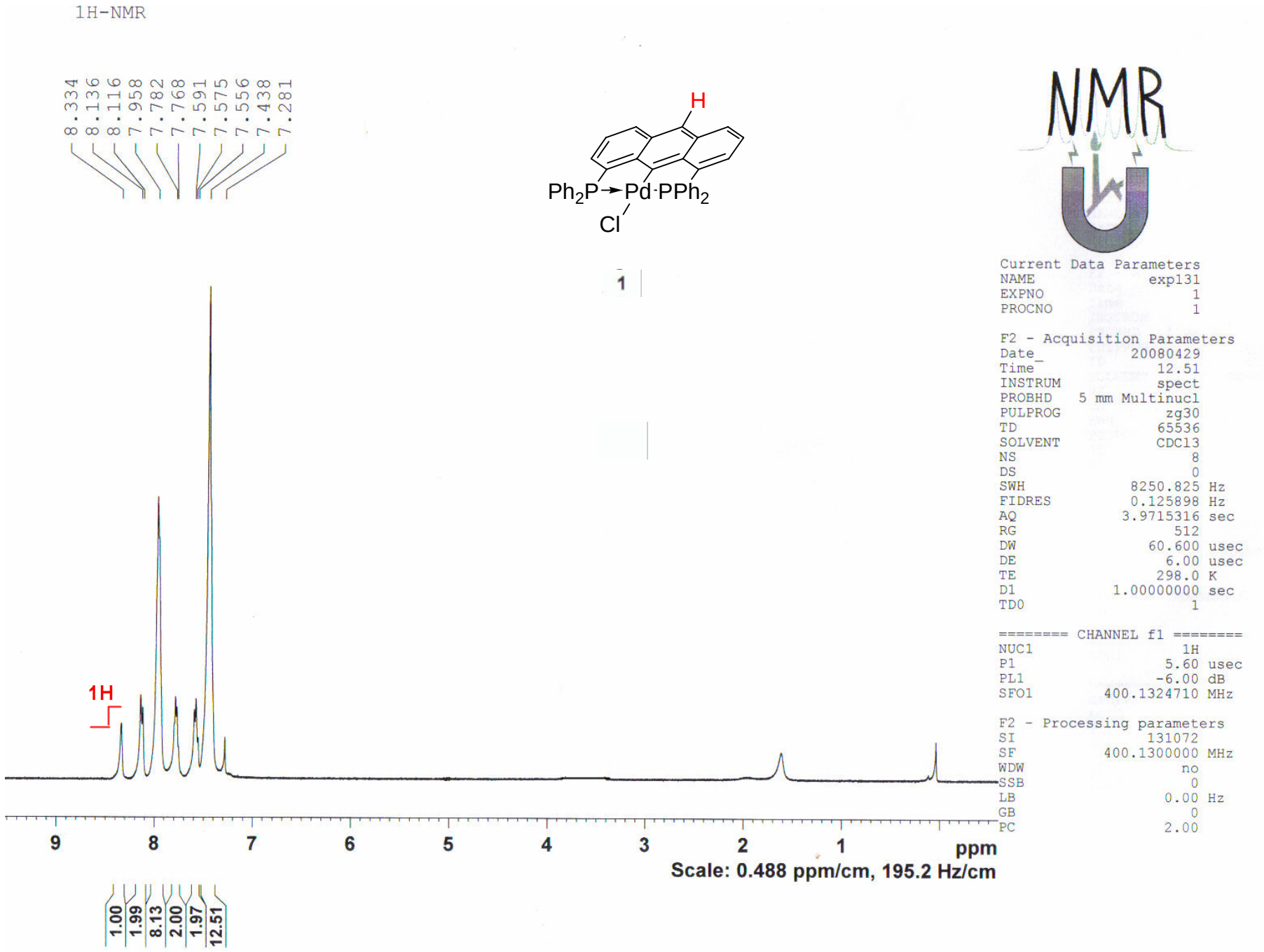
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Cl
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NMR
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SF 400.1300000 MHz
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SSB 0
LB 0.00 Hz
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7
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5
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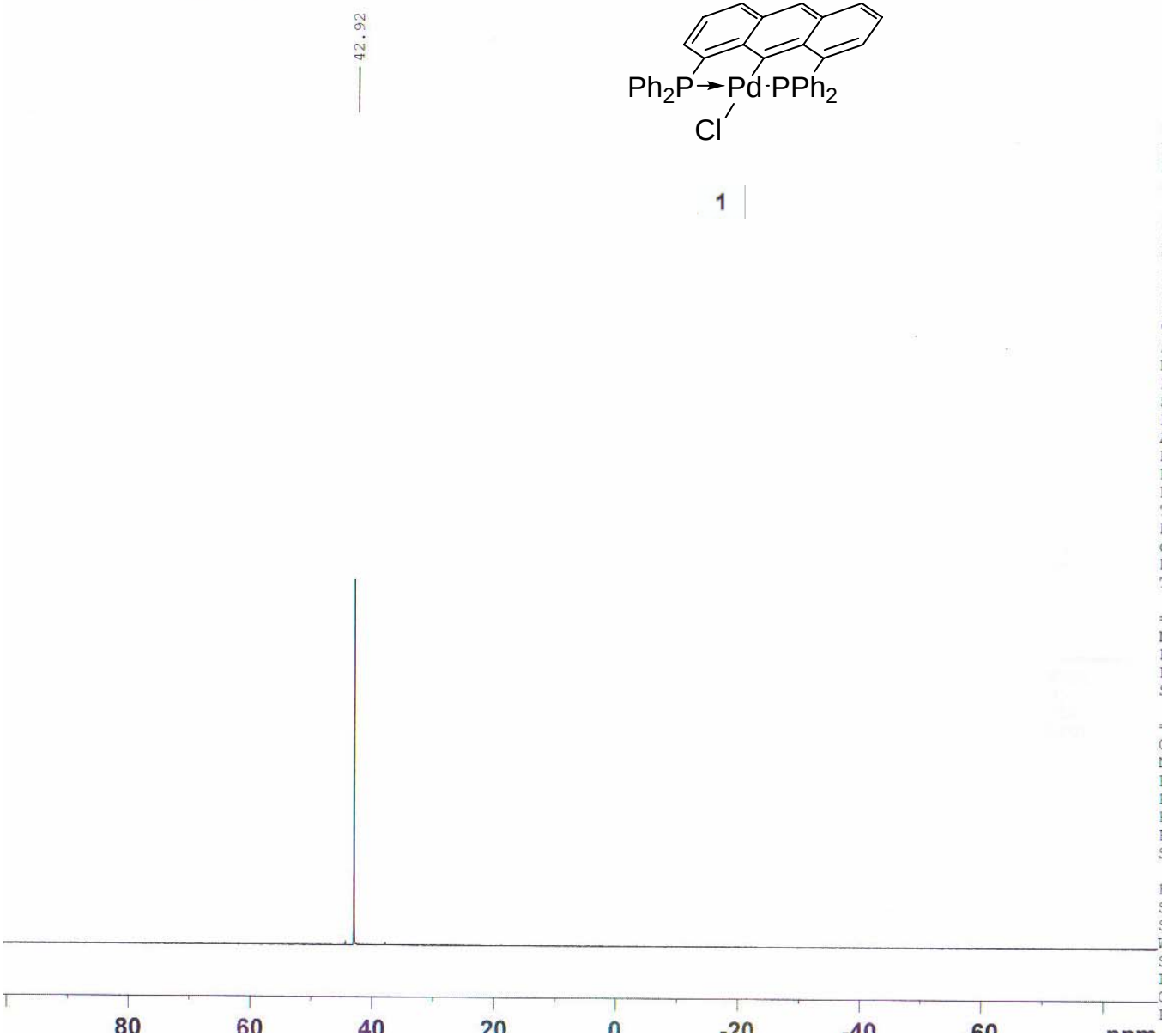
31P-NMR decoupled



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PROCNO                     1

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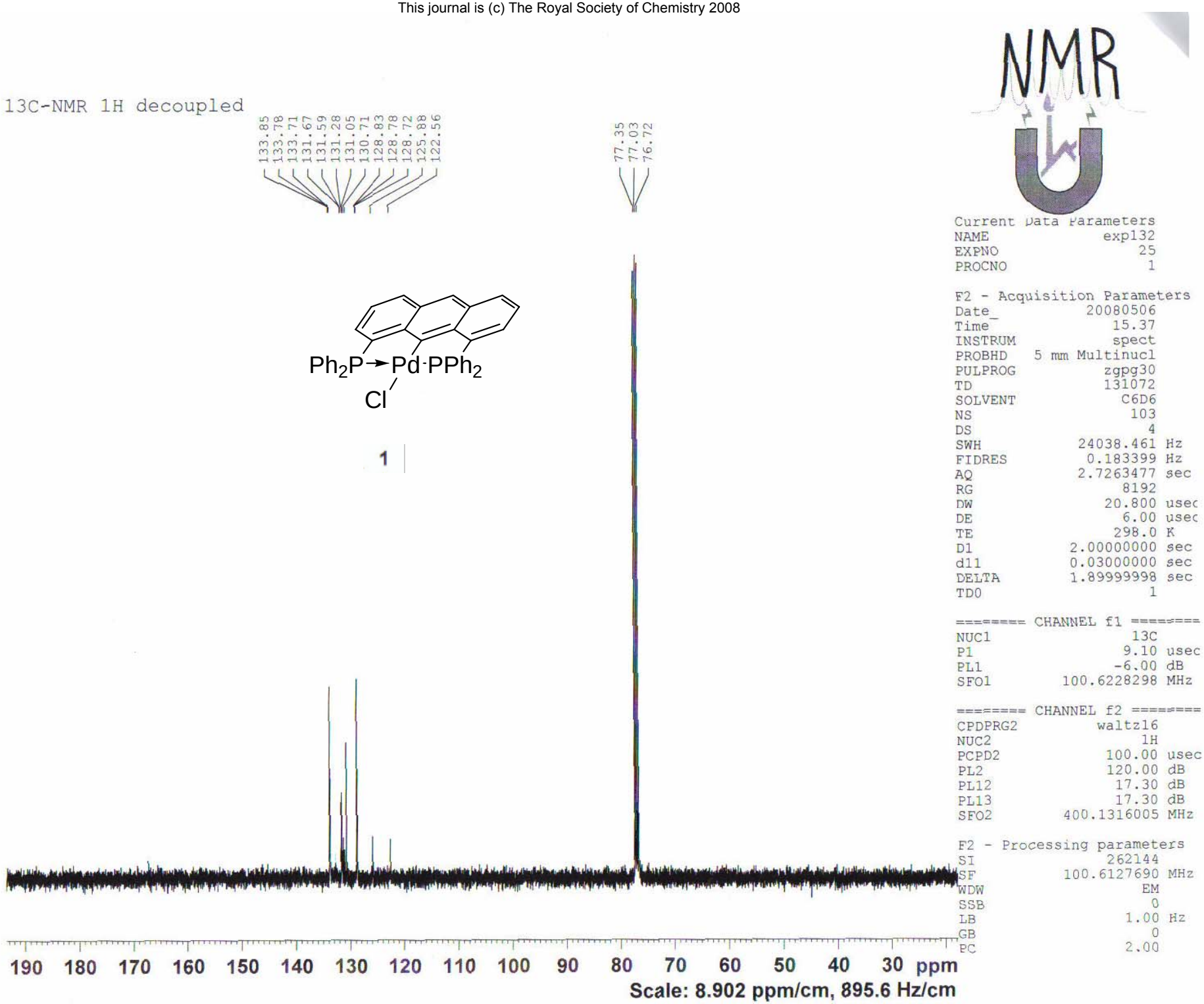
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77.03
76.72
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Cl
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NMR
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PROCNO 1
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TD 131072
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WDW EM
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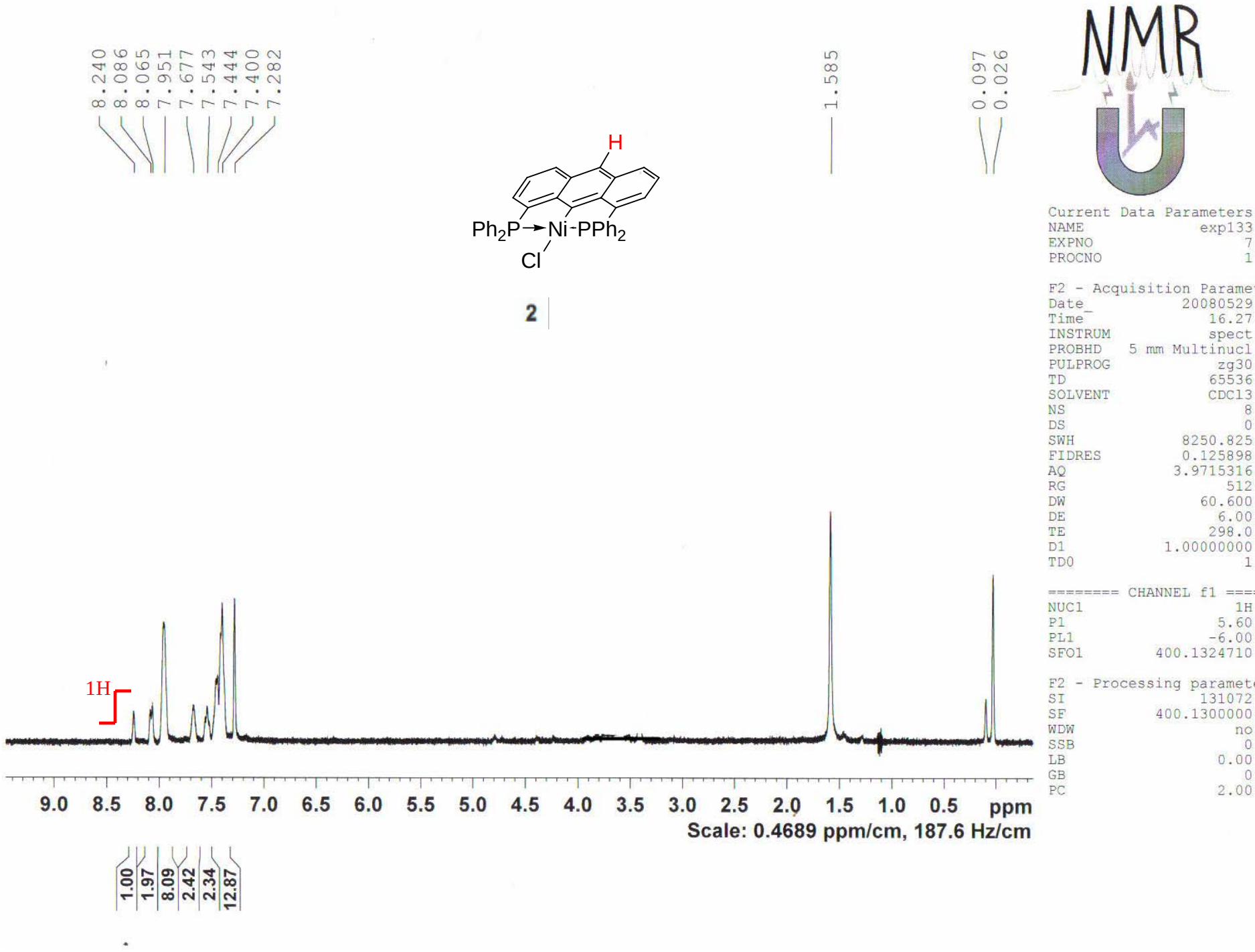
1H-NMR



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PROCNO	1

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F2 - Processing parameters

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31P-NMR decoupled

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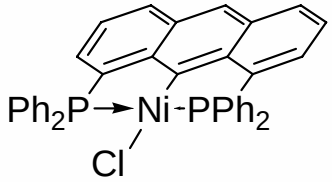

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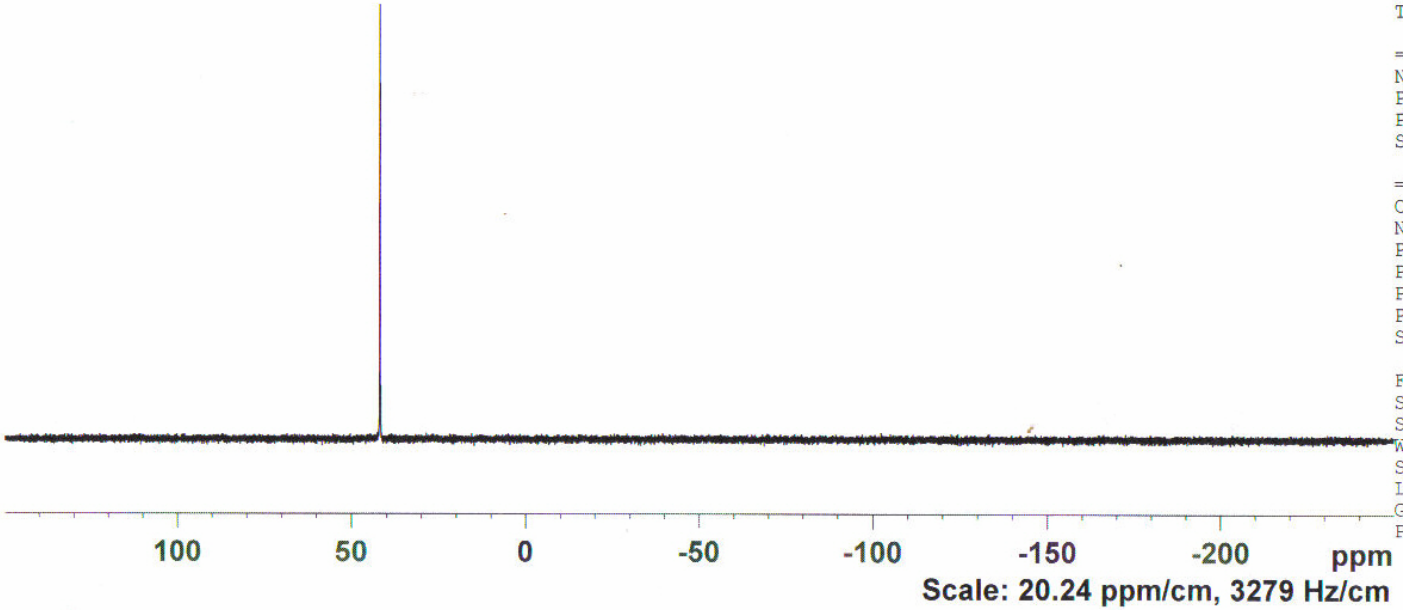


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PROCNO                     1

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RG                    9195.2
DW                     7.725 usec
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TE                    298.0 K
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d11               0.03000000 sec
DELTA             1.89999998 sec
TD0                        1

======== CHANNEL f1 ========
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SFO1            161.9674940 MHz

======== CHANNEL f2 ========
CPDPRG2              waltz16
NUC2                     1H
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PL12                  17.30 dB
PL13                  17.30 dB
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F2 - Processing parameters
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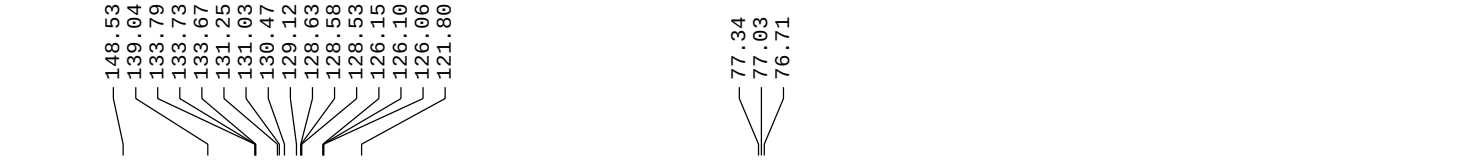
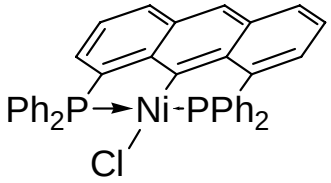
13C-NMR 1H decoupled





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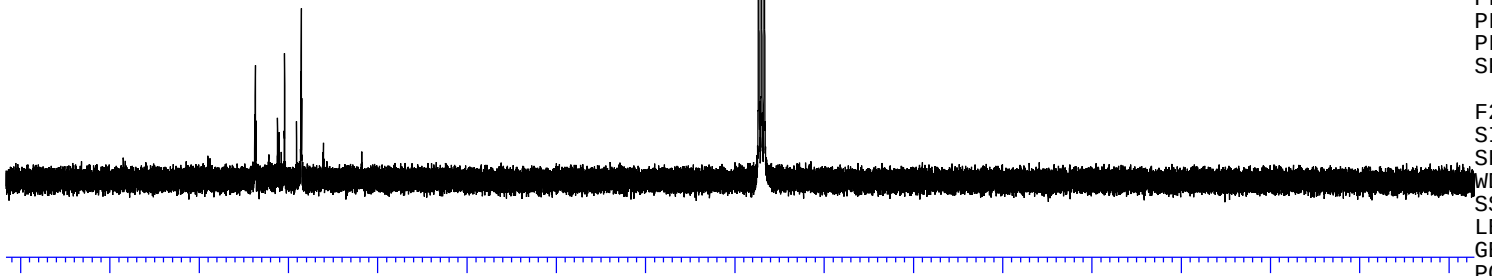

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DE 6.00 usec
TE 298.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
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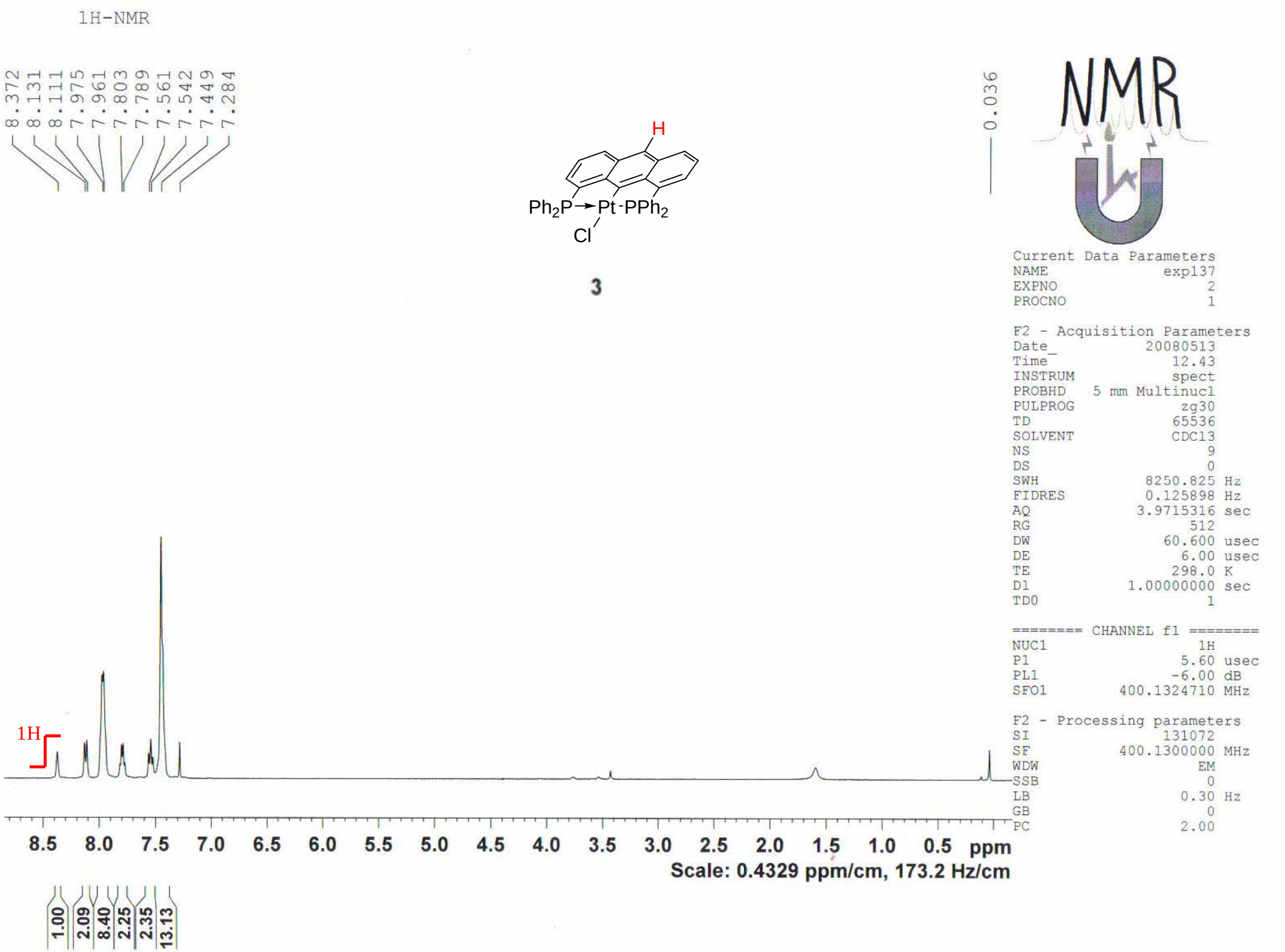
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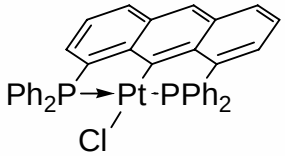
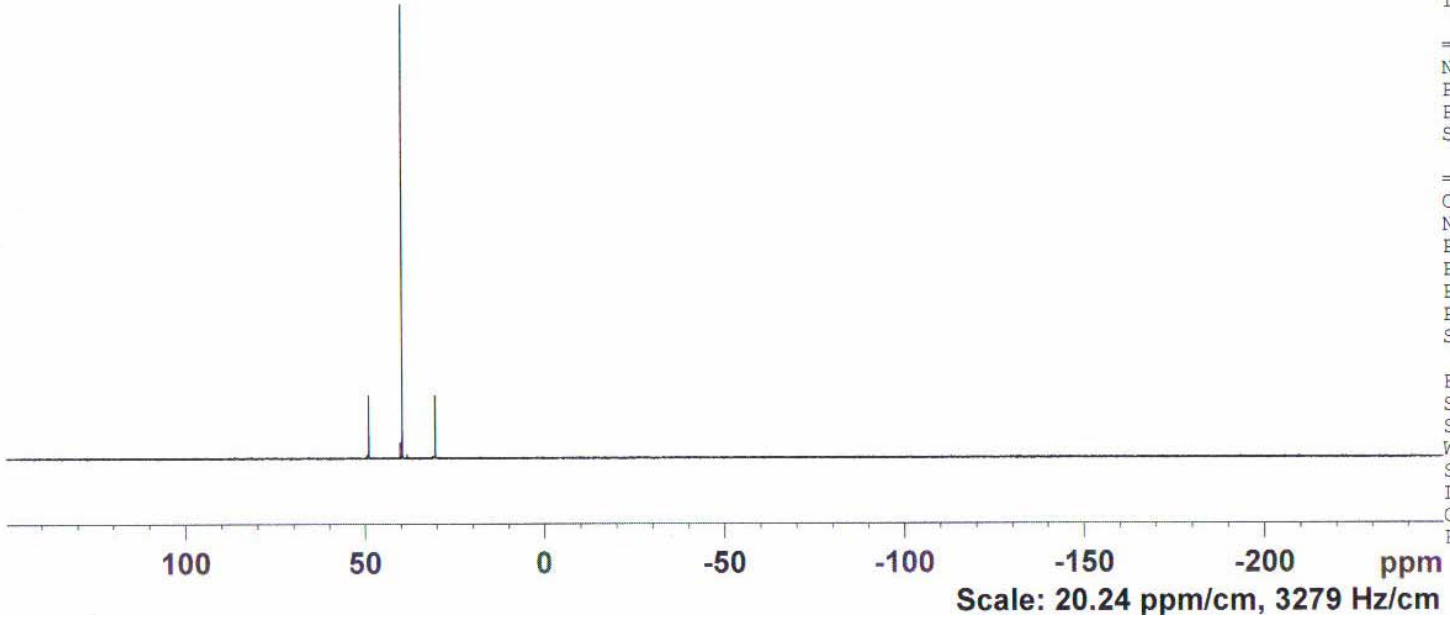
7.542
7.449
7.284
H
Ph2P→Pt-PPh2
Cl
3
0.036
NMR
Current Data Parameters
NAME exp137
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080513
Time 12.43
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 9
DS 0
SWH 8250.825 Hz
FIDRES 0.125898 Hz
AQ 3.9715316 sec
RG 512
DW 60.600 usec
DE 6.00 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 5.60 usec
PL1 -6.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 131072
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 2.00
1H
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
Scale: 0.4329 ppm/cm, 173.2 Hz/cm
1.00
2.09
8.40
2.25
2.35
13.13

31P-NMR decoupled

48.89
39.65
30.42



3



Current Data Parameters

NAME	exp137
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20080513
Time	12.38
INSTRUM	spect
PROBHD	5 mm Multinucl
PULPROG	zgpg30
TD	65536
SOLVENT	Acetone
NS	67
DS	4
SWH	64724.918 Hz
FIDRES	0.987624 Hz
AQ	0.5063156 sec
RG	9195.2
DW	7.725 usec
DE	6.00 usec
TE	298.0 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
TD0	1

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

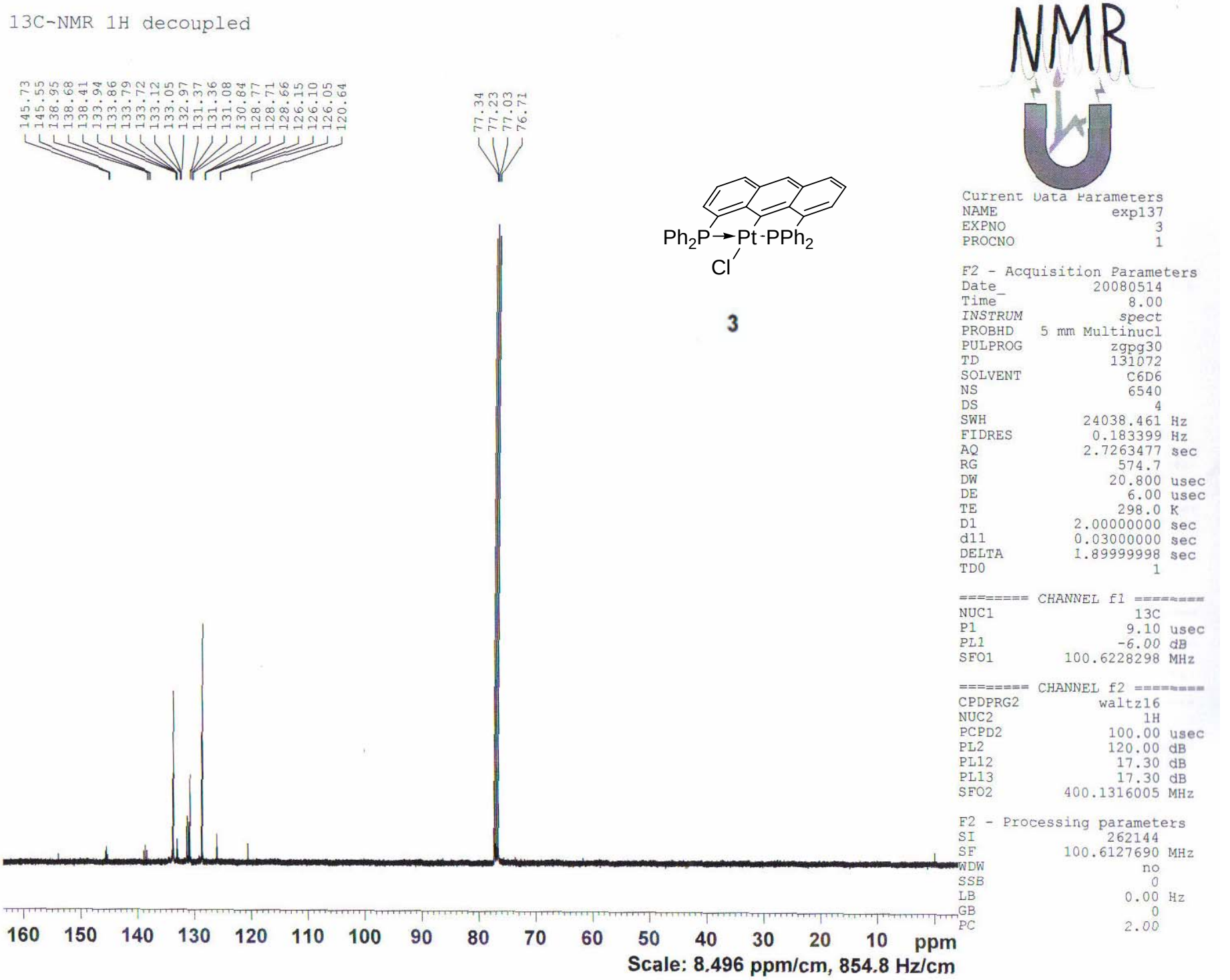
NUC1	31P
P1	6.92 usec
PL1	-6.00 dB
SFO1	161.9674940 MHz

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

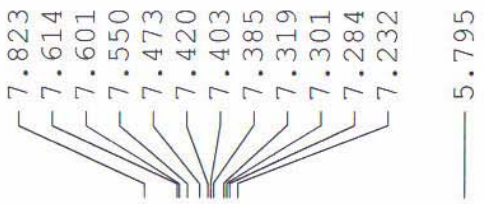
CPDPRG2	waltz16
NUC2	1H
PCPD2	100.00 usec
PL2	120.00 dB
PL12	17.30 dB
PL13	17.30 dB
SFO2	400.1324710 MHz

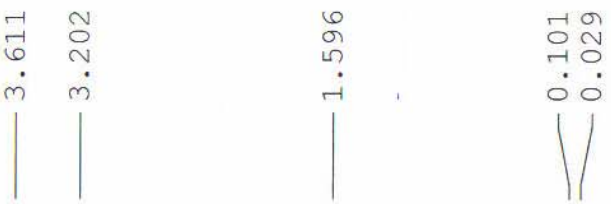
F2 - Processing parameters

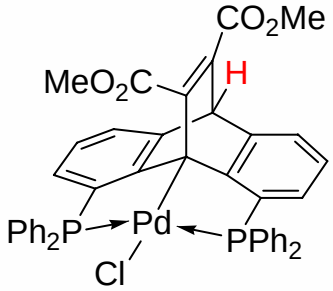
SI	32768
SF	161.9755956 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	2.00


13C-NMR 1H decoupled
145.73
145.55
138.95
138.68
138.41
133.94
133.86
133.79
133.72
133.12
133.05
132.97
131.37
131.36
131.08
130.84
128.77
128.71
128.66
126.15
126.10
126.05
120.64
77.34
77.23
77.03
76.71
Ph2P→Pt-PPh2
Cl
3
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm
Scale: 8.496 ppm/cm, 854.8 Hz/cm
NMR
Current Data Parameters
NAME exp137
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080514
Time 8.00
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zgpg30
TD 131072
SOLVENT C6D6
NS 6540
DS 4
SWH 24038.461 Hz
FIDRES 0.183399 Hz
AQ 2.7263477 sec
RG 574.7
DW 20.800 usec
DE 6.00 usec
TE 298.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.10 usec
PL1 -6.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 17.30 dB
PL13 17.30 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 262144
SF 100.6127690 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 2.00

1H-NMR

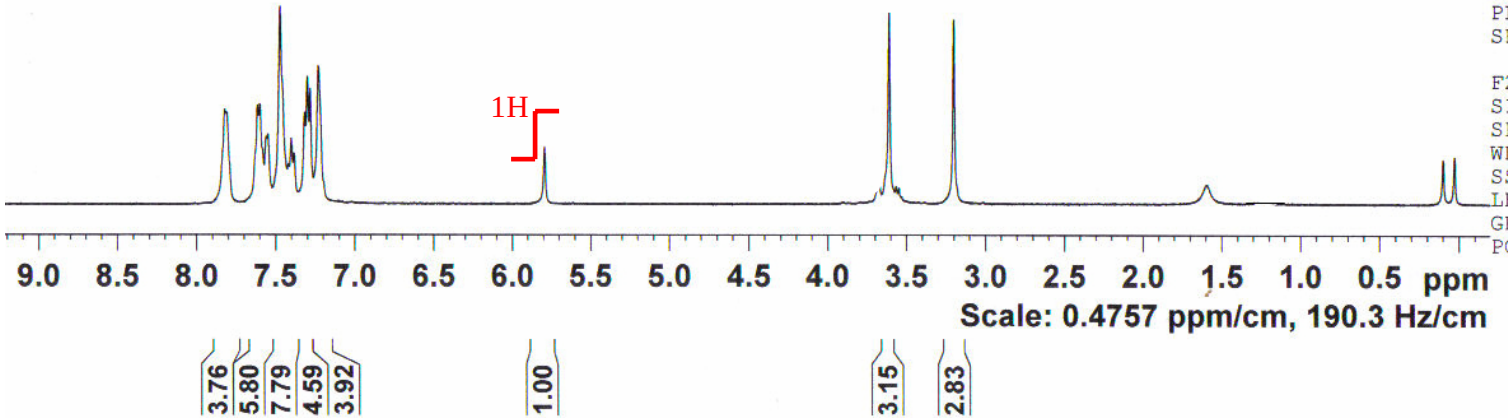






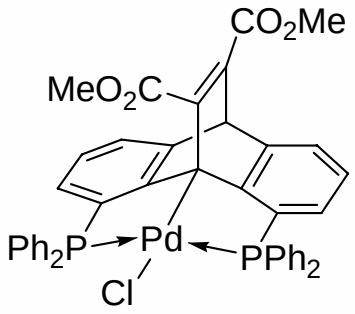
4

```
Current Data Parameters
NAME                  exp132
EXPNO                     29
PROCNO                     1

F2 - Acquisition Parameters
Date_              20080511
Time                  16.19
INSTRUM               spect
PROBHD    5 mm Multinucl
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        0
SWH                8250.825 Hz
FIDRES            0.125898 Hz
AQ                3.9715316 sec
RG                      512
DW                   60.600 usec
DE                    6.00 usec
TE                   298.0 K
D1              1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                      1H
P1                    5.60 usec
PL1                  -6.00 dB
SFO1          400.1324710 MHz

F2 - Processing parameters
SI                    131072
SF          400.1300000 MHz
WDW                       no
SSB                        0
LB                    0.00 Hz
GB                         0
PC                    2.00
```

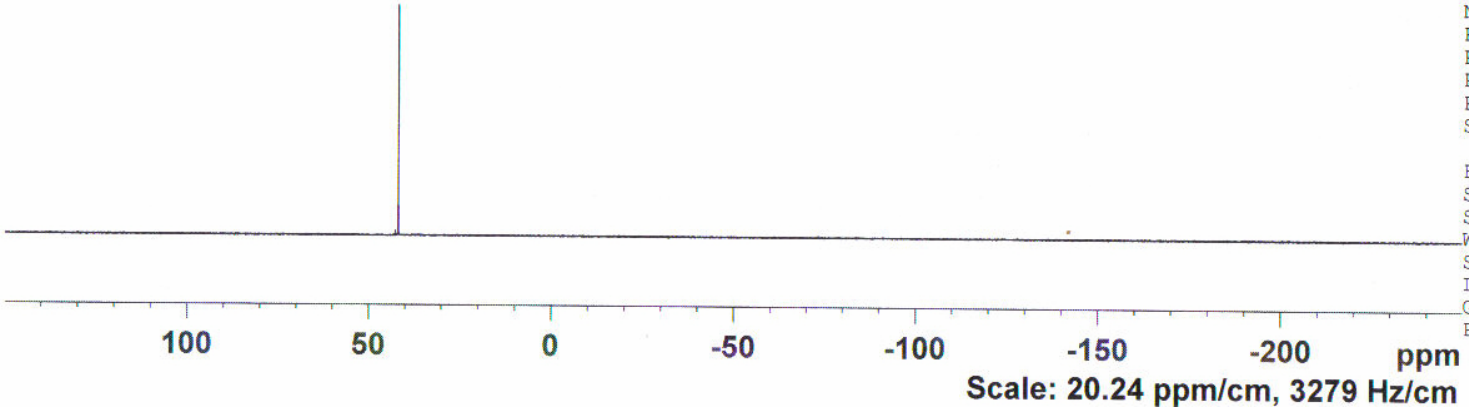


31P-NMR decoupled

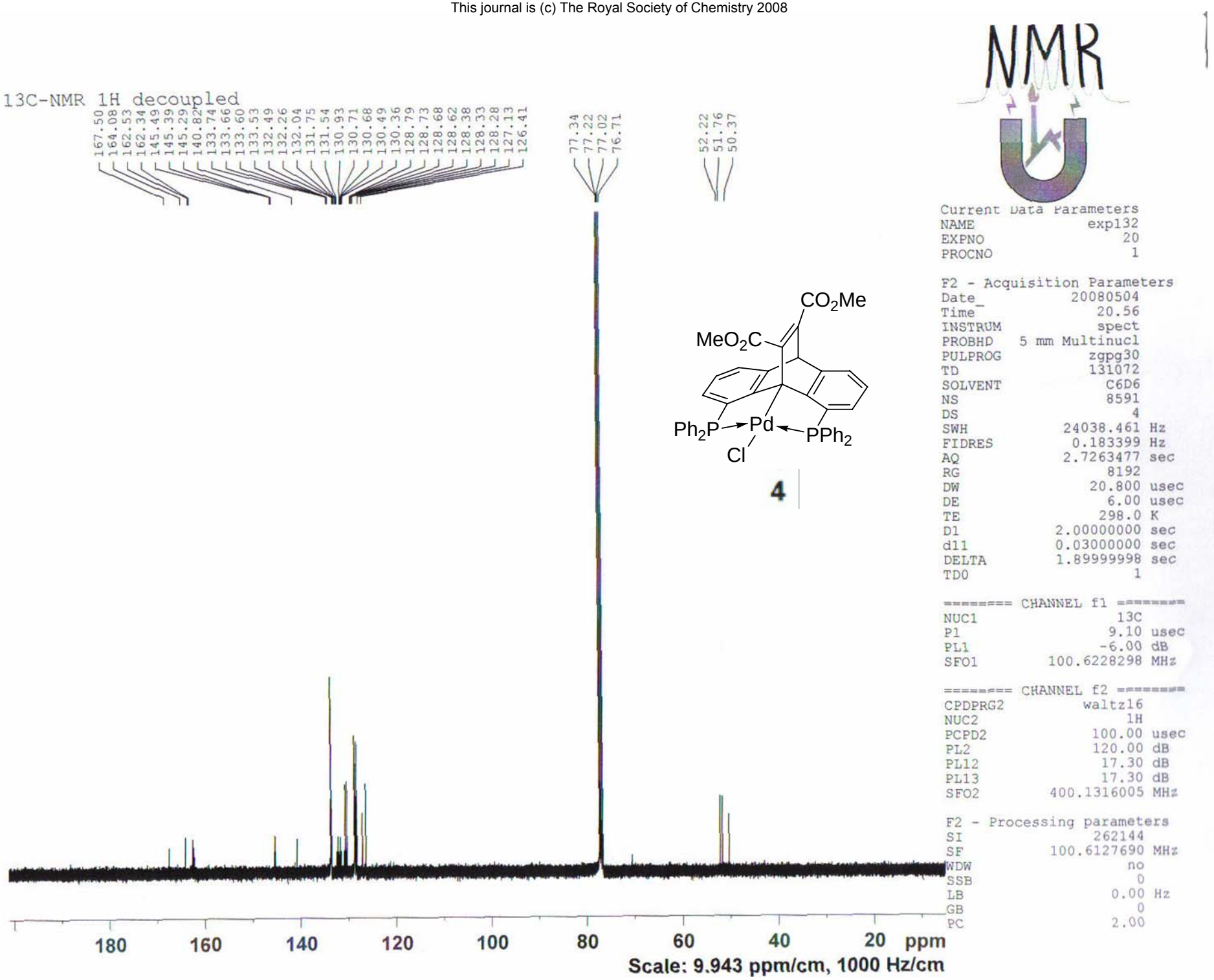
41.88

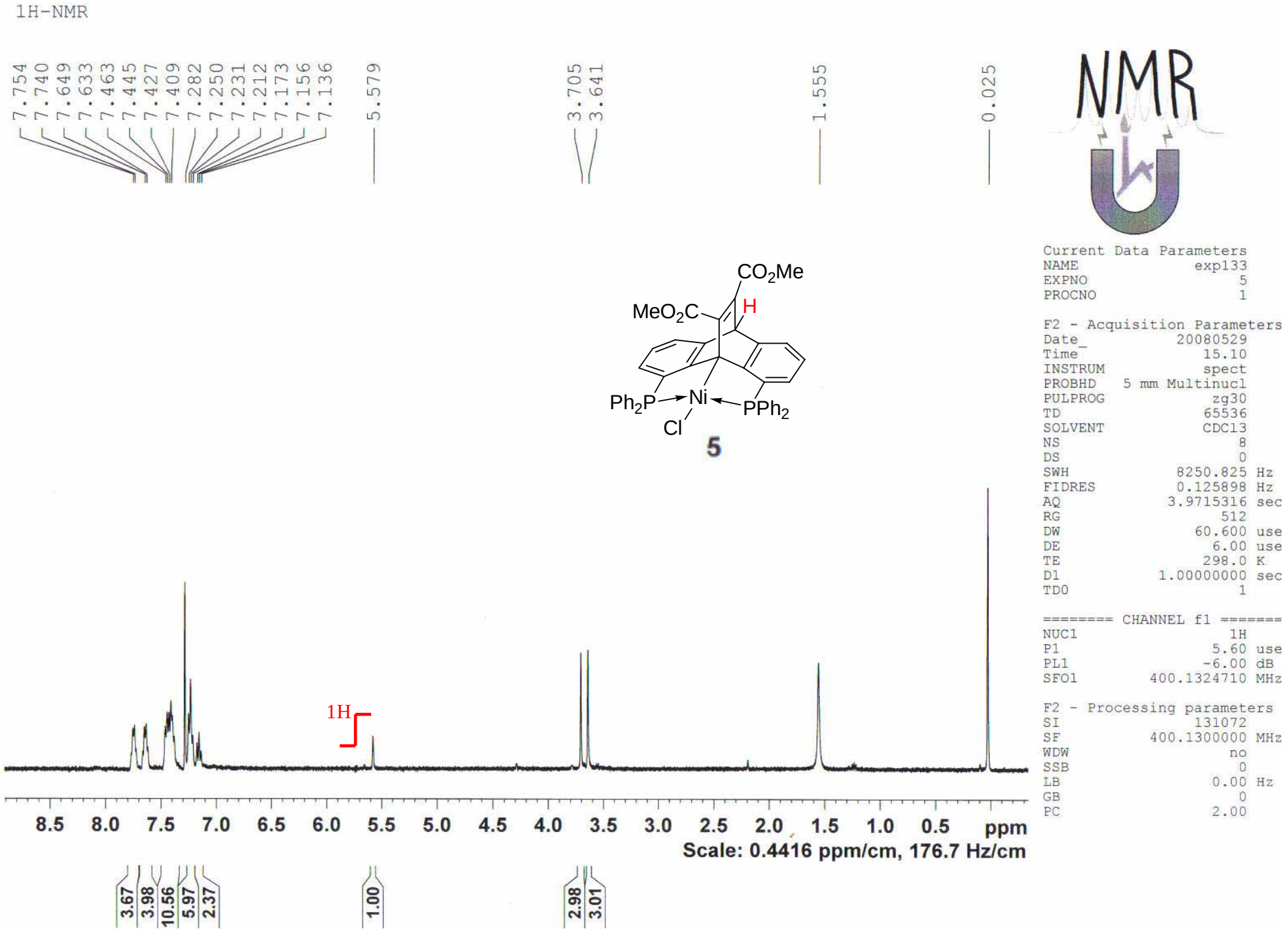


4



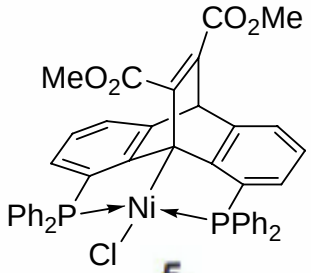

```
Current Data Parameters
NAME                  exp132
EXPNO                     28
PROCNO                     1

F2 - Acquisition Parameters
Date_               20080511
Time                   16.16
INSTRUM                spect
PROBHD    5 mm Multinucl
PULPROG                zgpg30
TD                      65536
SOLVENT              Acetone
NS                         29
DS                          4
SWH                64724.918 Hz
FIDRES             0.987624 Hz
AQ                 0.5063156 sec
RG                    9195.2
DW                    7.725 usec
DE                    6.00 usec
TE                    298.0 K
D1                 2.00000000 sec
d11                0.03000000 sec
DELTA              1.89999998 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      31P
P1                     6.92 usec
PL1                   -6.00 dB
SFO1             161.9674940 MHz

======== CHANNEL f2 ========
CPDPRG2              waltz16
NUC2                      1H
PCPD2                100.00 usec
PL2                  120.00 dB
PL12                  17.30 dB
PL13                  17.30 dB
SFO2             400.1324710 MHz

F2 - Processing parameters
SI                     32768
SF               161.9755956 MHz
WDW                       EM
SSB                        0
LB                    1.00 Hz
GB                         0
PC                     2.00
```


13C-NMR 1H decoupled
167.50
164.08
162.53
162.34
145.49
145.39
145.29
140.82
133.74
133.66
133.60
133.53
132.49
132.26
132.04
131.75
131.54
130.93
130.71
130.68
130.49
130.36
128.79
128.73
128.68
128.62
128.38
128.33
128.28
127.13
126.41
77.34
77.22
77.02
76.71
52.22
51.76
50.37
CO2Me
MeO2C
Ph2P
Pd
PPh2
Cl
4
NMR
Current Data Parameters
NAME exp132
EXPNO 20
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080504
Time 20.56
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zgpg30
TD 131072
SOLVENT C6D6
NS 8591
DS 4
SWH 24038.461 Hz
FIDRES 0.183399 Hz
AQ 2.7263477 sec
RG 8192
DW 20.800 usec
DE 6.00 usec
TE 298.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.10 usec
PL1 -6.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 17.30 dB
PL13 17.30 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 262144
SF 100.6127690 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 2.00
180
160
140
120
100
80
60
40
20 ppm
Scale: 9.943 ppm/cm, 1000 Hz/cm

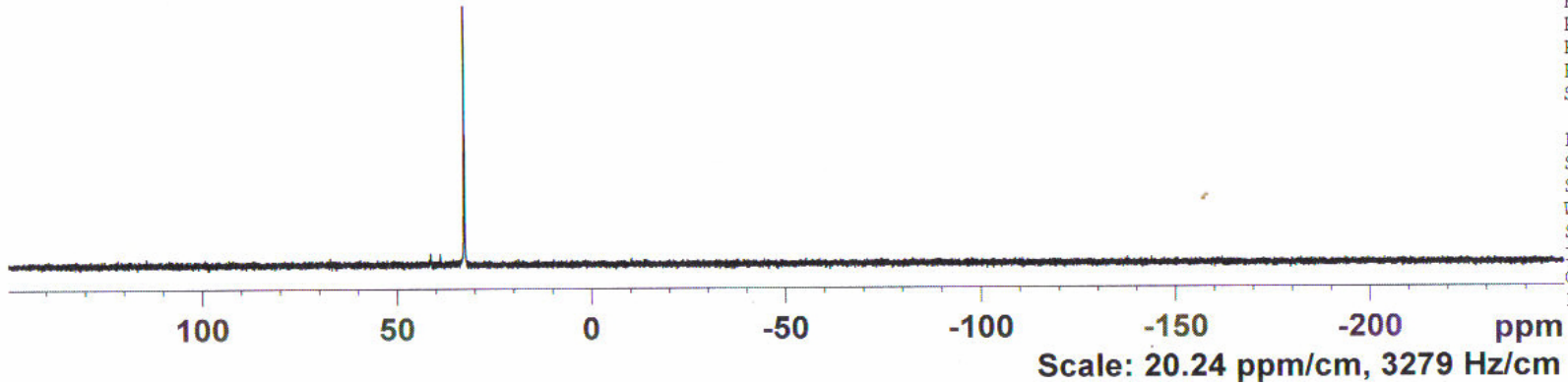

1H-NMR
7.754
7.740
7.649
7.633
7.463
7.445
7.427
7.409
7.282
7.250
7.231
7.212
7.173
7.156
7.136
5.579
3.705
3.641
1.555
0.025
NMR
CO2Me
MeO2C
H
Ph2P
Ni
PPh2
Cl
5
1H
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
Scale: 0.4416 ppm/cm, 176.7 Hz/cm
3.67
3.98
10.56
5.97
2.37
1.00
2.98
3.01
Current Data Parameters
NAME exp133
EXPNO 5
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080529
Time 15.10
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 8250.825 Hz
FIDRES 0.125898 Hz
AQ 3.9715316 sec
RG 512
DW 60.600 use
DE 6.00 use
TE 298.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 =======
NUC1 1H
P1 5.60 use
PL1 -6.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 131072
SF 400.1300000 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 2.00

31P-NMR decoupled

32.65



5



Current Data Parameters

NAME	expl35
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

Date_	20080507
Time	12.15
INSTRUM	spect
PROBHD	5 mm Multinucl
PULPROG	zgpg30
TD	65536
SOLVENT	Acetone
NS	58
DS	4
SWH	64724.918 Hz
FIDRES	0.987624 Hz
AQ	0.5063156 sec
RG	9195.2
DW	7.725 usec
DE	6.00 usec
TE	298.0 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
TD0	1

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

NUC1	31P
P1	6.92 usec
PL1	-6.00 dB
SFO1	161.9674940 MHz

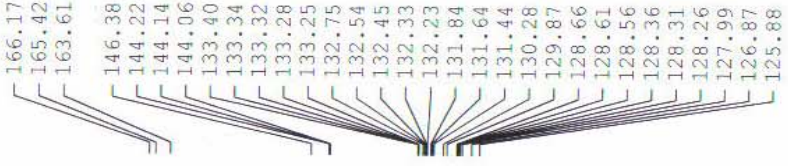
======== CHANNEL f2 ========

CPDPRG2	waltz16
NUC2	1H
PCPD2	100.00 usec
PL2	120.00 dB
PL12	17.30 dB
PL13	17.30 dB
SFO2	400.1324710 MHz

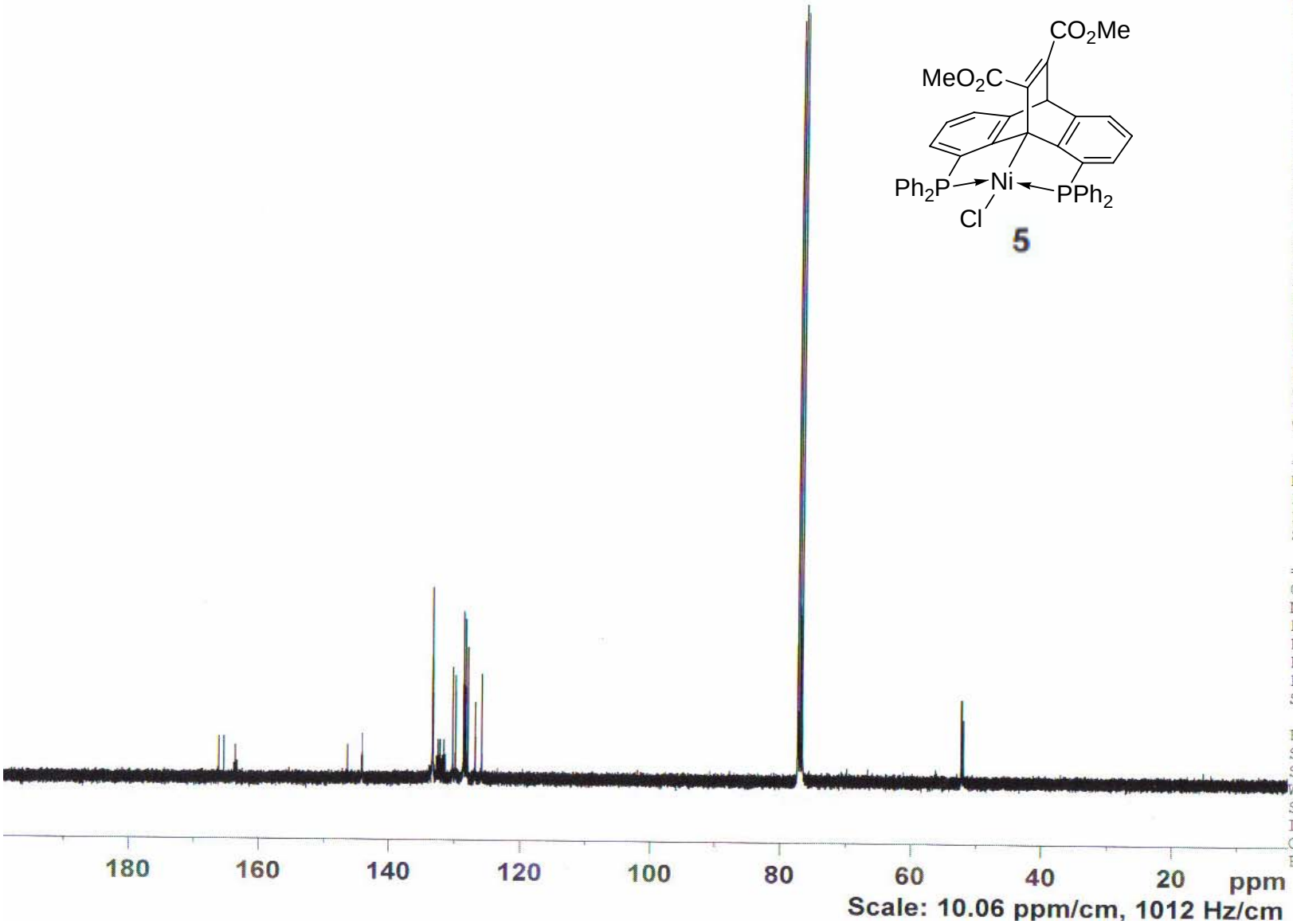
F2 - Processing parameters

SI	32768
SF	161.9755956 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	2.00

13C-NMR 1H decoupled



5



Current Data Parameters

NAME	exp135
EXPNO	3
PROCNO	1

F2 - Acquisition Parameters

Date_	20080528
Time	7.23
INSTRUM	spect
PROBHD	5 mm Multinucl
PULPROG	zgpg30
TD	131072
SOLVENT	C6D6
NS	6500
DS	4
SWH	24038.461 Hz
FIDRES	0.183399 Hz
AQ	2.7263477 sec
RG	1290.2
DW	20.800 use
DE	6.00 use
TE	298.0 K
D1	2.00000000 sec
d11	0.03000000 sec
DELTA	1.89999998 sec
TD0	1

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

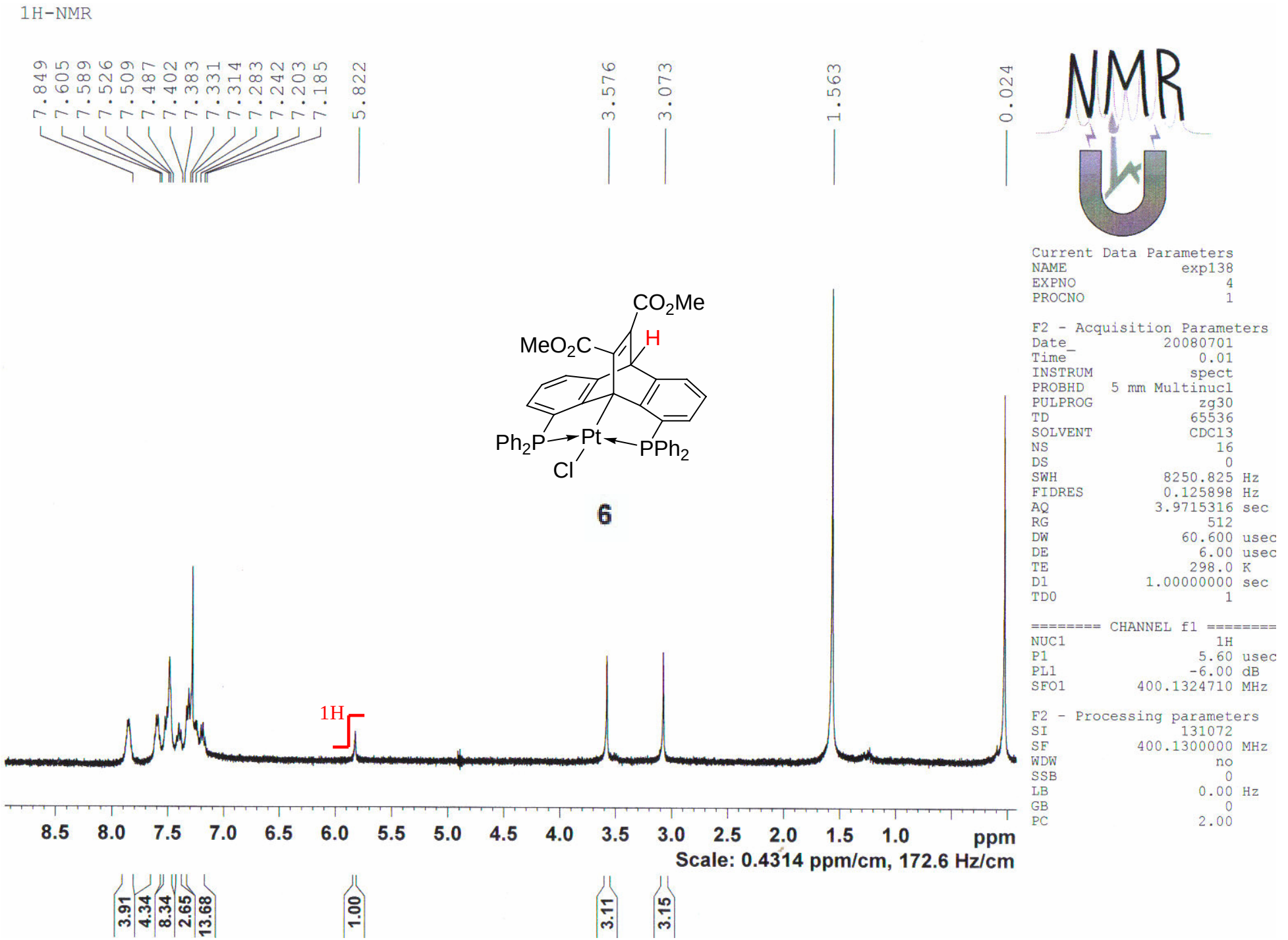
NUC1	13C
P1	9.10 use
PL1	-6.00 dB
SFO1	100.6228298 MHz

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

CPDPRG2	waltz16
NUC2	1H
PCPD2	100.00 use
PL2	120.00 dB
PL12	17.30 dB
PL13	17.30 dB
SFO2	400.1316005 MHz

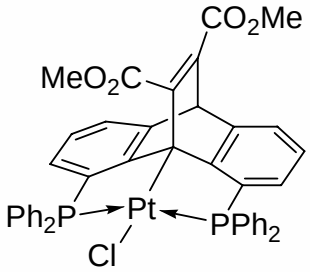
F2 - Processing parameters

SI	262144
SF	100.6127690 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	2.00

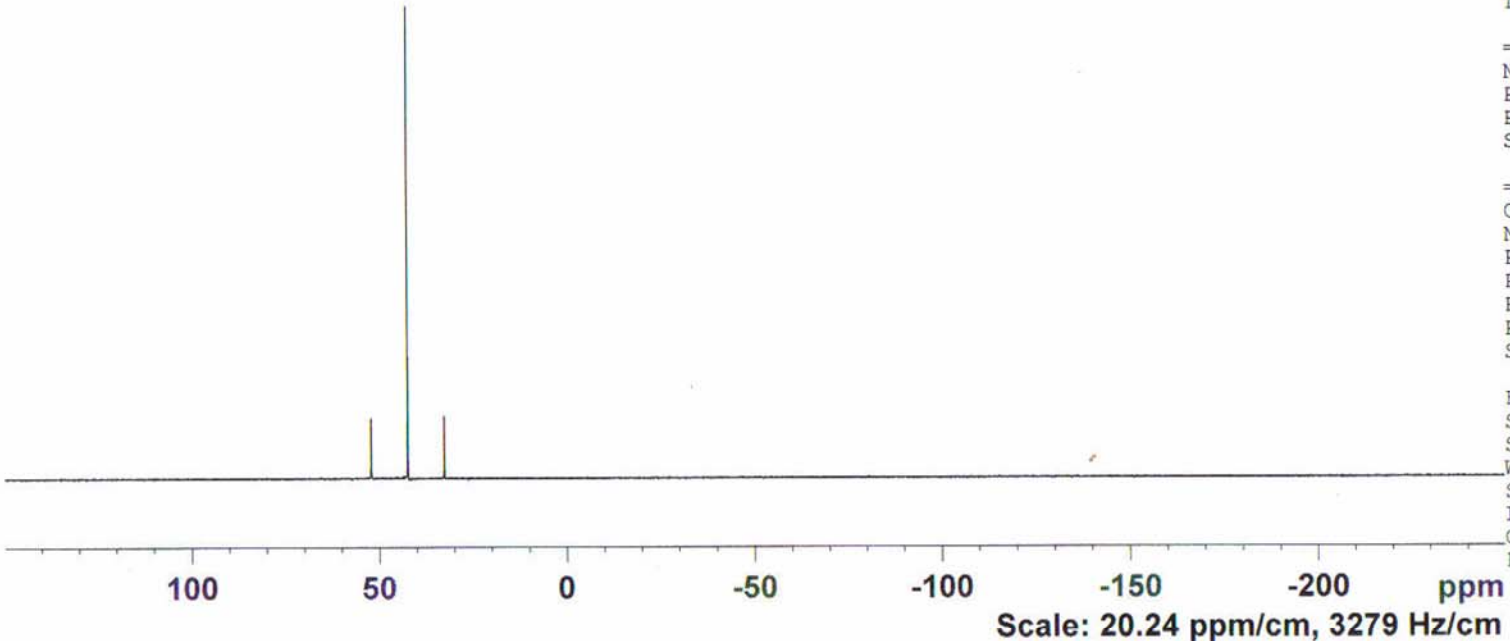

1H-NMR
7.849
7.605
7.589
7.526
7.509
7.487
7.402
7.383
7.331
7.314
7.283
7.242
7.203
7.185
5.822
3.576
3.073
1.563
0.024
NMR
CO2Me
MeO2C
H
Ph2P
Pt
PPh2
Cl
6
1H
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
Scale: 0.4314 ppm/cm, 172.6 Hz/cm
3.91
4.34
8.34
2.65
13.68
1.00
3.11
3.15
Current Data Parameters
NAME exp138
EXPNO 4
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080701
Time 0.01
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 8250.825 Hz
FIDRES 0.125898 Hz
AQ 3.9715316 sec
RG 512
DW 60.600 usec
DE 6.00 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 5.60 usec
PL1 -6.00 dB
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 131072
SF 400.1300000 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 2.00

31P-NMR decoupled

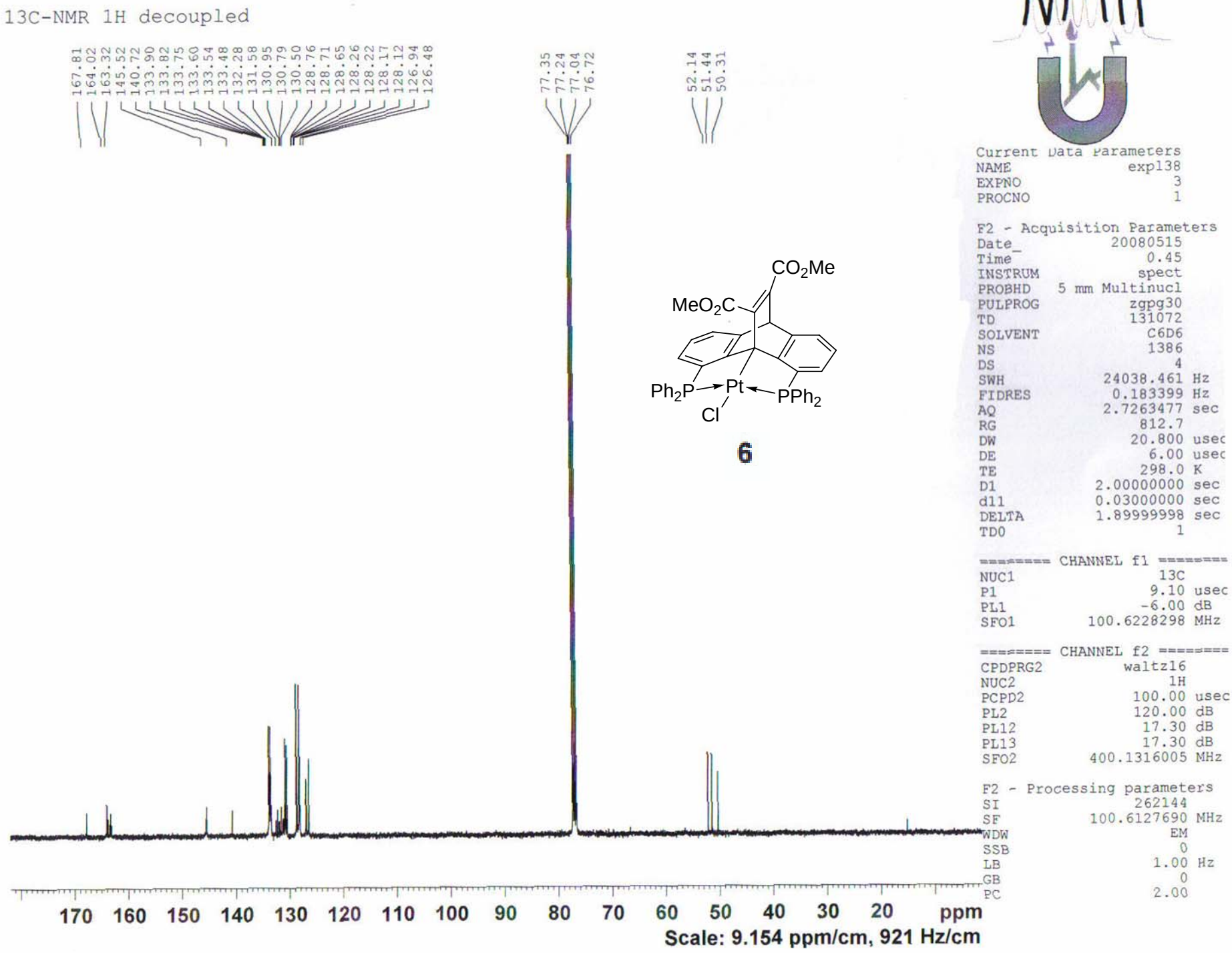
52.12
42.37
32.64



6




```
Current Data Parameters
NAME                  exp138
EXPNO                      2
PROCNO                     1

F2 - Acquisition Parameters
Date_               20080514
Time                   13.51
INSTRUM                spect
PROBHD   5 mm Multinucl
PULPROG               zgpg30
TD                     65536
SOLVENT              Acetone
NS                         155
DS                           4
SWH                64724.918 Hz
FIDRES             0.987624 Hz
AQ                 0.5063156 sec
RG                    9195.2
DW                    7.725 usec
DE                    6.00 usec
TE                    298.0 K
D1                 2.00000000 sec
d11                0.03000000 sec
DELTA              1.89999998 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                     31P
P1                    6.92 usec
PL1                  -6.00 dB
SFO1           161.9674940 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                     1H
PCPD2               100.00 usec
PL2                 120.00 dB
PL12                 17.30 dB
PL13                 17.30 dB
SFO2           400.1324710 MHz

F2 - Processing parameters
SI                       32768
SF             161.9755956 MHz
WDW                         EM
SSB                          0
LB                    1.00 Hz
GB                           0
PC                        2.00
```


13C-NMR 1H decoupled
167.81
164.02
163.32
145.52
140.72
133.90
133.82
133.75
133.60
133.54
133.48
132.28
131.58
130.95
130.79
130.50
128.76
128.71
128.65
128.26
128.22
128.17
128.12
126.94
126.48
77.35
77.24
77.04
76.72
52.14
51.44
50.31
CO2Me
MeO2C
Ph2P
Pt
PPh2
Cl
6
Current Data Parameters
NAME expl38
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20080515
Time 0.45
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zgpg30
TD 131072
SOLVENT C6D6
NS 1386
DS 4
SWH 24038.461 Hz
FIDRES 0.183399 Hz
AQ 2.7263477 sec
RG 812.7
DW 20.800 usec
DE 6.00 usec
TE 298.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.10 usec
PL1 -6.00 dB
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 17.30 dB
PL13 17.30 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 262144
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm
Scale: 9.154 ppm/cm, 921 Hz/cm

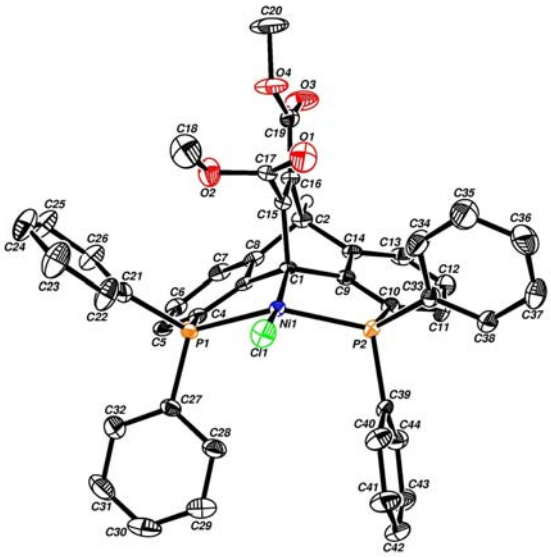


Table 1. Crystal data and structure refinement for Klarit8.

Identification code	klarit8m	
Empirical formula	C48 H41 Cl Ni O5 P2	
Formula weight	853.91	
Temperature	173(1) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P-1	
Unit cell dimensions	a = 10.870(2) Å	α= 75.338(2)°.
	b = 14.914(2) Å	β= 72.595(2)°.
	c = 17.181(3) Å	γ = 77.101(3)°.
Volume	2538.0(7) $Å^3$	
Z	2	
Density (calculated)	1.117 Mg/m^3	
Absorption coefficient	0.536 mm^{-1}	
F(000)	888	
Crystal size	0.45 x 0.30 x 0.08 mm^3	
Theta range for data collection	2.07 to 26.00°.	
Index ranges	-12<=h<=13, -17<=k<=18, 0<=l<=21	
Reflections collected	26275	

Independent reflections	9920 [R(int) = 0.0676]
Completeness to theta = 26.00°	99.2 %
Absorption correction	None
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	9920 / 10 / 511
Goodness-of-fit on F^2	1.158
Final R indices [I>2sigma(I)]	R1 = 0.0802, wR2 = 0.2254
R indices (all data)	R1 = 0.0898, wR2 = 0.2347
Largest diff. peak and hole	2.018 and -0.654 e.$Å^{-3}$

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for Klarit8. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
C(1)	4170(4)	3227(3)	3224(2)	14(1)
C(2)	3084(4)	1784(3)	4177(3)	20(1)
C(3)	2743(4)	3470(3)	3687(2)	16(1)
C(4)	2038(4)	4368(3)	3534(3)	17(1)
C(5)	742(4)	4534(3)	4009(3)	22(1)
C(6)	195(4)	3806(4)	4585(3)	24(1)
C(7)	892(4)	2888(3)	4691(3)	22(1)
C(8)	2168(4)	2722(3)	4221(3)	19(1)
C(9)	4864(4)	2641(3)	3886(2)	16(1)
C(10)	6039(4)	2805(3)	3929(3)	18(1)
C(11)	6657(5)	2205(3)	4522(3)	27(1)
C(12)	6059(5)	1463(3)	5059(3)	28(1)
C(13)	4888(5)	1293(3)	4997(3)	26(1)
C(14)	4286(4)	1884(3)	4405(3)	20(1)
C(15)	4096(4)	2496(3)	2754(3)	17(1)
C(16)	3543(4)	1758(3)	3244(3)	18(1)
C(17)	4593(4)	2702(3)	1827(3)	19(1)
C(18)	4040(7)	3590(5)	601(3)	52(2)
C(19)	3384(5)	942(3)	2967(3)	24(1)
C(20)	3503(9)	289(5)	1834(4)	62(2)
C(21)	2187(4)	5368(3)	1823(3)	22(1)
C(22)	2770(5)	5894(4)	1065(3)	35(1)
C(23)	2271(6)	6059(5)	378(4)	45(2)
C(24)	1168(6)	5687(5)	453(4)	43(1)
C(25)	581(6)	5169(5)	1203(4)	43(1)
C(26)	1085(5)	5001(4)	1887(3)	31(1)
C(27)	2803(5)	6226(3)	2946(3)	21(1)
C(28)	3787(5)	6431(3)	3206(3)	27(1)
C(29)	3639(6)	7268(4)	3459(3)	37(1)
C(30)	2521(6)	7919(4)	3438(3)	40(1)
C(31)	1522(6)	7724(4)	3193(4)	41(1)

C(32)	1669(5)	6882(4)	2935(3)	32(1)
C(33)	8251(4)	3236(3)	2547(3)	20(1)
C(34)	8392(5)	2962(4)	1800(3)	35(1)
C(35)	9594(6)	2541(5)	1393(4)	46(2)
C(36)	10676(6)	2395(4)	1719(4)	44(1)
C(37)	10522(5)	2663(4)	2460(4)	39(1)
C(38)	9302(5)	3090(4)	2869(3)	27(1)
C(39)	6949(4)	4611(3)	3552(3)	19(1)
C(40)	7532(5)	5363(4)	3020(3)	32(1)
C(41)	7737(6)	6062(4)	3356(4)	37(1)
C(42)	7352(6)	6015(4)	4198(4)	38(1)
C(43)	6772(5)	5282(4)	4727(3)	36(1)
C(44)	6573(5)	4576(4)	4401(3)	27(1)
Cl(1)	6001(1)	5320(1)	1489(1)	28(1)
Ni(1)	4986(1)	4307(1)	2524(1)	14(1)
O(1)	5707(4)	2489(3)	1459(2)	39(1)
O(2)	3658(4)	3227(3)	1486(2)	35(1)
O(3)	3012(5)	248(3)	3434(2)	44(1)
O(4)	3654(4)	1064(3)	2146(2)	41(1)
P(1)	2997(1)	5103(1)	2661(1)	16(1)
P(2)	6629(1)	3742(1)	3087(1)	16(1)
O(5)	5387(12)	7958(10)	1167(8)	96(4)
C(45)	5828(13)	8551(12)	361(9)	84(5)
C(46)	7265(14)	8157(14)	143(11)	85(5)
C(47)	7626(18)	7543(19)	928(10)	143(10)
C(48)	6401(12)	7726(11)	1607(9)	63(4)
O(6)	2894(11)	-551(7)	7144(8)	83(3)
C(55)	1478(11)	-400(9)	7392(13)	90(6)
C(56)	1032(11)	640(9)	7140(10)	67(4)
C(57)	2237(9)	1097(7)	6974(7)	38(2)
C(58)	3235(11)	290(6)	7247(9)	53(3)

Table 3. Bond lengths [Å] and angles [°] for Klarit8.

C(1)-C(3)	1.525(6)
C(1)-C(9)	1.531(6)
C(1)-C(15)	1.540(5)
C(1)-Ni(1)	1.961(4)
C(2)-C(14)	1.519(6)
C(2)-C(8)	1.530(6)
C(2)-C(16)	1.538(6)
C(2)-H(2)	1.0000
C(3)-C(8)	1.388(6)
C(3)-C(4)	1.391(6)
C(4)-C(5)	1.403(6)
C(4)-P(1)	1.812(4)
C(5)-C(6)	1.379(7)
C(5)-H(5)	0.9500
C(6)-C(7)	1.405(7)
C(6)-H(6)	0.9500
C(7)-C(8)	1.384(6)
C(7)-H(7)	0.9500
C(9)-C(10)	1.379(6)
C(9)-C(14)	1.391(6)
C(10)-C(11)	1.403(6)
C(10)-P(2)	1.811(4)
C(11)-C(12)	1.395(7)
C(11)-H(11)	0.9500
C(12)-C(13)	1.391(7)
C(12)-H(12)	0.9500
C(13)-C(14)	1.388(6)
C(13)-H(13)	0.9500
C(15)-C(16)	1.343(6)
C(15)-C(17)	1.495(6)
C(16)-C(19)	1.474(6)
C(17)-O(1)	1.199(6)
C(17)-O(2)	1.325(6)
C(18)-O(2)	1.444(6)

C(18)-H(18A)	0.9800
C(18)-H(18B)	0.9800
C(18)-H(18C)	0.9800
C(19)-O(3)	1.204(6)
C(19)-O(4)	1.324(6)
C(20)-O(4)	1.451(6)
C(20)-H(20A)	0.9800
C(20)-H(20B)	0.9800
C(20)-H(20C)	0.9800
C(21)-C(22)	1.388(7)
C(21)-C(26)	1.389(7)
C(21)-P(1)	1.821(4)
C(22)-C(23)	1.388(7)
C(22)-H(22)	0.9500
C(23)-C(24)	1.389(9)
C(23)-H(23)	0.9500
C(24)-C(25)	1.374(8)
C(24)-H(24)	0.9500
C(25)-C(26)	1.386(7)
C(25)-H(25)	0.9500
C(26)-H(26)	0.9500
C(27)-C(28)	1.392(7)
C(27)-C(32)	1.394(7)
C(27)-P(1)	1.812(4)
C(28)-C(29)	1.385(7)
C(28)-H(28)	0.9500
C(29)-C(30)	1.379(9)
C(29)-H(29)	0.9500
C(30)-C(31)	1.385(9)
C(30)-H(30)	0.9500
C(31)-C(32)	1.397(7)
C(31)-H(31)	0.9500
C(32)-H(32)	0.9500
C(33)-C(38)	1.367(6)
C(33)-C(34)	1.398(7)
C(33)-P(2)	1.822(4)

C(34)-C(35)	1.379(8)
C(34)-H(34)	0.9500
C(35)-C(36)	1.401(9)
C(35)-H(35)	0.9500
C(36)-C(37)	1.380(8)
C(36)-H(36)	0.9500
C(37)-C(38)	1.396(7)
C(37)-H(37)	0.9500
C(38)-H(38)	0.9500
C(39)-C(44)	1.382(7)
C(39)-C(40)	1.395(7)
C(39)-P(2)	1.825(4)
C(40)-C(41)	1.398(7)
C(40)-H(40)	0.9500
C(41)-C(42)	1.369(8)
C(41)-H(41)	0.9500
C(42)-C(43)	1.372(8)
C(42)-H(42)	0.9500
C(43)-C(44)	1.393(7)
C(43)-H(43)	0.9500
C(44)-H(44)	0.9500
Cl(1)-Ni(1)	2.2069(12)
Ni(1)-P(2)	2.1872(12)
Ni(1)-P(1)	2.1977(12)
O(5)-C(45)	1.4499(10)
O(5)-C(48)	1.4500(10)
C(45)-C(46)	1.5096(11)
C(45)-H(45A)	0.9900
C(45)-H(45B)	0.9900
C(46)-C(47)	1.5298(10)
C(46)-H(46A)	0.9900
C(46)-H(46B)	0.9900
C(47)-C(48)	1.5100(10)
C(47)-H(47A)	0.9900
C(47)-H(47B)	0.9900
C(48)-H(48A)	0.9900

C(48)-H(48B)	0.9900
O(6)-C(55)	1.4497(11)
O(6)-C(58)	1.4500(10)
C(55)-C(56)	1.5096(11)
C(55)-H(55A)	0.9900
C(55)-H(55B)	0.9900
C(56)-C(57)	1.5295(10)
C(56)-H(56A)	0.9900
C(56)-H(56B)	0.9900
C(57)-C(58)	1.5099(10)
C(57)-H(57A)	0.9900
C(57)-H(57B)	0.9900
C(58)-H(58A)	0.9900
C(58)-H(58B)	0.9900

C(3)-C(1)-C(9)	105.8(3)
C(3)-C(1)-C(15)	101.8(3)
C(9)-C(1)-C(15)	103.2(3)
C(3)-C(1)-Ni(1)	114.7(3)
C(9)-C(1)-Ni(1)	114.6(3)
C(15)-C(1)-Ni(1)	115.2(3)
C(14)-C(2)-C(8)	105.8(3)
C(14)-C(2)-C(16)	105.1(3)
C(8)-C(2)-C(16)	104.5(3)
C(14)-C(2)-H(2)	113.5
C(8)-C(2)-H(2)	113.5
C(16)-C(2)-H(2)	113.5
C(8)-C(3)-C(4)	122.2(4)
C(8)-C(3)-C(1)	115.6(4)
C(4)-C(3)-C(1)	122.0(4)
C(3)-C(4)-C(5)	118.0(4)
C(3)-C(4)-P(1)	110.1(3)
C(5)-C(4)-P(1)	131.7(4)
C(6)-C(5)-C(4)	119.8(4)
C(6)-C(5)-H(5)	120.1
C(4)-C(5)-H(5)	120.1

C(5)-C(6)-C(7)	121.5(4)
C(5)-C(6)-H(6)	119.2
C(7)-C(6)-H(6)	119.2
C(8)-C(7)-C(6)	118.8(4)
C(8)-C(7)-H(7)	120.6
C(6)-C(7)-H(7)	120.6
C(7)-C(8)-C(3)	119.3(4)
C(7)-C(8)-C(2)	128.5(4)
C(3)-C(8)-C(2)	112.1(4)
C(10)-C(9)-C(14)	121.7(4)
C(10)-C(9)-C(1)	122.4(4)
C(14)-C(9)-C(1)	115.7(4)
C(9)-C(10)-C(11)	119.3(4)
C(9)-C(10)-P(2)	110.8(3)
C(11)-C(10)-P(2)	129.6(4)
C(12)-C(11)-C(10)	119.0(4)
C(12)-C(11)-H(11)	120.5
C(10)-C(11)-H(11)	120.5
C(13)-C(12)-C(11)	121.1(4)
C(13)-C(12)-H(12)	119.4
C(11)-C(12)-H(12)	119.4
C(14)-C(13)-C(12)	119.6(4)
C(14)-C(13)-H(13)	120.2
C(12)-C(13)-H(13)	120.2
C(13)-C(14)-C(9)	119.3(4)
C(13)-C(14)-C(2)	128.5(4)
C(9)-C(14)-C(2)	112.1(4)
C(16)-C(15)-C(17)	128.4(4)
C(16)-C(15)-C(1)	114.4(4)
C(17)-C(15)-C(1)	117.2(4)
C(15)-C(16)-C(19)	126.0(4)
C(15)-C(16)-C(2)	114.9(4)
C(19)-C(16)-C(2)	119.1(4)
O(1)-C(17)-O(2)	125.6(4)
O(1)-C(17)-C(15)	124.1(4)
O(2)-C(17)-C(15)	110.0(4)

O(2)-C(18)-H(18A)	109.5
O(2)-C(18)-H(18B)	109.5
H(18A)-C(18)-H(18B)	109.5
O(2)-C(18)-H(18C)	109.5
H(18A)-C(18)-H(18C)	109.5
H(18B)-C(18)-H(18C)	109.5
O(3)-C(19)-O(4)	123.3(4)
O(3)-C(19)-C(16)	123.9(4)
O(4)-C(19)-C(16)	112.8(4)
O(4)-C(20)-H(20A)	109.5
O(4)-C(20)-H(20B)	109.5
H(20A)-C(20)-H(20B)	109.5
O(4)-C(20)-H(20C)	109.5
H(20A)-C(20)-H(20C)	109.5
H(20B)-C(20)-H(20C)	109.5
C(22)-C(21)-C(26)	118.8(4)
C(22)-C(21)-P(1)	117.9(4)
C(26)-C(21)-P(1)	123.1(4)
C(21)-C(22)-C(23)	121.2(5)
C(21)-C(22)-H(22)	119.4
C(23)-C(22)-H(22)	119.4
C(22)-C(23)-C(24)	119.1(5)
C(22)-C(23)-H(23)	120.4
C(24)-C(23)-H(23)	120.4
C(25)-C(24)-C(23)	120.0(5)
C(25)-C(24)-H(24)	120.0
C(23)-C(24)-H(24)	120.0
C(24)-C(25)-C(26)	120.8(5)
C(24)-C(25)-H(25)	119.6
C(26)-C(25)-H(25)	119.6
C(25)-C(26)-C(21)	120.1(5)
C(25)-C(26)-H(26)	120.0
C(21)-C(26)-H(26)	120.0
C(28)-C(27)-C(32)	119.0(4)
C(28)-C(27)-P(1)	119.6(4)
C(32)-C(27)-P(1)	121.3(4)

C(29)-C(28)-C(27)	120.7(5)
C(29)-C(28)-H(28)	119.7
C(27)-C(28)-H(28)	119.7
C(30)-C(29)-C(28)	120.1(5)
C(30)-C(29)-H(29)	120.0
C(28)-C(29)-H(29)	120.0
C(29)-C(30)-C(31)	120.2(5)
C(29)-C(30)-H(30)	119.9
C(31)-C(30)-H(30)	119.9
C(30)-C(31)-C(32)	119.9(5)
C(30)-C(31)-H(31)	120.1
C(32)-C(31)-H(31)	120.1
C(27)-C(32)-C(31)	120.1(5)
C(27)-C(32)-H(32)	119.9
C(31)-C(32)-H(32)	119.9
C(38)-C(33)-C(34)	120.1(4)
C(38)-C(33)-P(2)	121.6(4)
C(34)-C(33)-P(2)	118.2(3)
C(35)-C(34)-C(33)	119.8(5)
C(35)-C(34)-H(34)	120.1
C(33)-C(34)-H(34)	120.1
C(34)-C(35)-C(36)	120.3(5)
C(34)-C(35)-H(35)	119.9
C(36)-C(35)-H(35)	119.9
C(37)-C(36)-C(35)	119.4(5)
C(37)-C(36)-H(36)	120.3
C(35)-C(36)-H(36)	120.3
C(36)-C(37)-C(38)	120.0(5)
C(36)-C(37)-H(37)	120.0
C(38)-C(37)-H(37)	120.0
C(33)-C(38)-C(37)	120.4(5)
C(33)-C(38)-H(38)	119.8
C(37)-C(38)-H(38)	119.8
C(44)-C(39)-C(40)	119.4(4)
C(44)-C(39)-P(2)	122.5(3)
C(40)-C(39)-P(2)	118.0(4)

C(39)-C(40)-C(41)	119.4(5)
C(39)-C(40)-H(40)	120.3
C(41)-C(40)-H(40)	120.3
C(42)-C(41)-C(40)	120.3(5)
C(42)-C(41)-H(41)	119.9
C(40)-C(41)-H(41)	119.9
C(41)-C(42)-C(43)	120.8(5)
C(41)-C(42)-H(42)	119.6
C(43)-C(42)-H(42)	119.6
C(42)-C(43)-C(44)	119.5(5)
C(42)-C(43)-H(43)	120.2
C(44)-C(43)-H(43)	120.2
C(39)-C(44)-C(43)	120.6(5)
C(39)-C(44)-H(44)	119.7
C(43)-C(44)-H(44)	119.7
C(1)-Ni(1)-P(2)	87.39(12)
C(1)-Ni(1)-P(1)	86.60(12)
P(2)-Ni(1)-P(1)	146.64(5)
C(1)-Ni(1)-Cl(1)	165.57(12)
P(2)-Ni(1)-Cl(1)	96.04(5)
P(1)-Ni(1)-Cl(1)	97.86(5)
C(17)-O(2)-C(18)	116.3(4)
C(19)-O(4)-C(20)	115.5(4)
C(4)-P(1)-C(27)	107.1(2)
C(4)-P(1)-C(21)	106.7(2)
C(27)-P(1)-C(21)	104.1(2)
C(4)-P(1)-Ni(1)	103.07(15)
C(27)-P(1)-Ni(1)	113.83(16)
C(21)-P(1)-Ni(1)	121.16(15)
C(10)-P(2)-C(33)	105.2(2)
C(10)-P(2)-C(39)	107.3(2)
C(33)-P(2)-C(39)	103.7(2)
C(10)-P(2)-Ni(1)	103.60(14)
C(33)-P(2)-Ni(1)	124.92(15)
C(39)-P(2)-Ni(1)	110.97(14)
C(45)-O(5)-C(48)	109.5(12)

O(5)-C(45)-C(46)	100.9(12)
O(5)-C(45)-H(45A)	111.6
C(46)-C(45)-H(45A)	111.6
O(5)-C(45)-H(45B)	111.6
C(46)-C(45)-H(45B)	111.6
H(45A)-C(45)-H(45B)	109.4
C(45)-C(46)-C(47)	108.9(14)
C(45)-C(46)-H(46A)	109.9
C(47)-C(46)-H(46A)	109.9
C(45)-C(46)-H(46B)	109.9
C(47)-C(46)-H(46B)	109.9
H(46A)-C(46)-H(46B)	108.3
C(48)-C(47)-C(46)	103.0(14)
C(48)-C(47)-H(47A)	111.2
C(46)-C(47)-H(47A)	111.2
C(48)-C(47)-H(47B)	111.2
C(46)-C(47)-H(47B)	111.2
H(47A)-C(47)-H(47B)	109.1
O(5)-C(48)-C(47)	102.5(13)
O(5)-C(48)-H(48A)	111.3
C(47)-C(48)-H(48A)	111.3
O(5)-C(48)-H(48B)	111.3
C(47)-C(48)-H(48B)	111.3
H(48A)-C(48)-H(48B)	109.2
C(55)-O(6)-C(58)	104.1(11)
O(6)-C(55)-C(56)	106.9(11)
O(6)-C(55)-H(55A)	110.3
C(56)-C(55)-H(55A)	110.3
O(6)-C(55)-H(55B)	110.3
C(56)-C(55)-H(55B)	110.3
H(55A)-C(55)-H(55B)	108.6
C(55)-C(56)-C(57)	105.3(10)
C(55)-C(56)-H(56A)	110.7
C(57)-C(56)-H(56A)	110.7
C(55)-C(56)-H(56B)	110.7
C(57)-C(56)-H(56B)	110.7

H(56A)-C(56)-H(56B)	108.8
C(58)-C(57)-C(56)	103.5(9)
C(58)-C(57)-H(57A)	111.1
C(56)-C(57)-H(57A)	111.1
C(58)-C(57)-H(57B)	111.1
C(56)-C(57)-H(57B)	111.1
H(57A)-C(57)-H(57B)	109.0
O(6)-C(58)-C(57)	106.4(9)
O(6)-C(58)-H(58A)	110.5
C(57)-C(58)-H(58A)	110.5
O(6)-C(58)-H(58B)	110.5
C(57)-C(58)-H(58B)	110.5
H(58A)-C(58)-H(58B)	108.6

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for Klarit8. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
C(1)	17(2)	14(2)	12(2)	-6(2)	-3(2)	-3(2)
C(2)	25(2)	18(2)	17(2)	-3(2)	-2(2)	-8(2)
C(3)	19(2)	22(2)	12(2)	-7(2)	-5(2)	-7(2)
C(4)	22(2)	19(2)	15(2)	-3(2)	-7(2)	-8(2)
C(5)	18(2)	26(2)	25(2)	-17(2)	-4(2)	-1(2)
C(6)	16(2)	37(3)	22(2)	-14(2)	0(2)	-7(2)
C(7)	22(2)	31(2)	16(2)	-7(2)	1(2)	-16(2)
C(8)	23(2)	24(2)	14(2)	-7(2)	-3(2)	-9(2)
C(9)	22(2)	14(2)	11(2)	-6(2)	-4(2)	-1(2)
C(10)	18(2)	19(2)	16(2)	-3(2)	-5(2)	-4(2)
C(11)	29(3)	30(3)	26(2)	-4(2)	-17(2)	-2(2)
C(12)	43(3)	20(2)	23(2)	0(2)	-19(2)	0(2)
C(13)	43(3)	17(2)	18(2)	0(2)	-8(2)	-8(2)
C(14)	27(2)	17(2)	16(2)	-7(2)	-5(2)	-3(2)
C(15)	18(2)	16(2)	18(2)	-6(2)	-6(2)	-1(2)
C(16)	20(2)	16(2)	19(2)	-7(2)	-4(2)	-5(2)
C(17)	22(2)	18(2)	19(2)	-8(2)	-4(2)	-2(2)
C(18)	72(5)	56(4)	19(3)	-2(3)	-13(3)	4(3)
C(19)	31(2)	17(2)	28(2)	-5(2)	-15(2)	-3(2)
C(20)	112(6)	41(4)	53(4)	-28(3)	-36(4)	-11(4)
C(21)	26(2)	20(2)	20(2)	-5(2)	-11(2)	1(2)
C(22)	34(3)	48(3)	24(3)	6(2)	-13(2)	-17(2)
C(23)	45(3)	64(4)	24(3)	7(3)	-14(2)	-15(3)
C(24)	45(3)	59(4)	30(3)	-3(3)	-23(3)	-7(3)
C(25)	40(3)	61(4)	38(3)	-9(3)	-18(3)	-21(3)
C(26)	32(3)	43(3)	23(2)	-3(2)	-10(2)	-16(2)
C(27)	28(2)	15(2)	17(2)	-3(2)	-5(2)	-3(2)
C(28)	32(3)	24(2)	26(2)	-10(2)	-6(2)	-3(2)
C(29)	46(3)	32(3)	38(3)	-14(2)	-11(3)	-10(2)
C(30)	64(4)	28(3)	31(3)	-15(2)	-3(3)	-13(3)
C(31)	48(3)	25(3)	44(3)	-11(2)	-13(3)	8(2)

C(32)	32(3)	30(3)	37(3)	-10(2)	-14(2)	2(2)
C(33)	16(2)	24(2)	22(2)	-5(2)	-3(2)	-6(2)
C(34)	27(3)	53(3)	28(3)	-17(2)	-11(2)	-2(2)
C(35)	34(3)	67(4)	40(3)	-29(3)	-6(3)	2(3)
C(36)	27(3)	49(4)	49(4)	-21(3)	1(3)	4(2)
C(37)	20(2)	50(3)	50(3)	-14(3)	-13(2)	-4(2)
C(38)	21(2)	32(3)	32(3)	-11(2)	-10(2)	-4(2)
C(39)	16(2)	21(2)	23(2)	-6(2)	-8(2)	-3(2)
C(40)	32(3)	34(3)	37(3)	-9(2)	-11(2)	-13(2)
C(41)	44(3)	30(3)	47(3)	-10(2)	-14(3)	-18(2)
C(42)	39(3)	35(3)	54(4)	-24(3)	-23(3)	-3(2)
C(43)	40(3)	44(3)	32(3)	-19(2)	-13(2)	-4(2)
C(44)	26(2)	30(3)	28(3)	-10(2)	-5(2)	-8(2)
Cl(1)	30(1)	30(1)	23(1)	7(1)	-5(1)	-13(1)
Ni(1)	14(1)	14(1)	13(1)	-2(1)	-4(1)	-4(1)
O(1)	31(2)	55(3)	24(2)	-10(2)	0(2)	-3(2)
O(2)	33(2)	42(2)	22(2)	-5(2)	-9(2)	6(2)
O(3)	80(3)	22(2)	37(2)	-2(2)	-18(2)	-23(2)
O(4)	74(3)	28(2)	30(2)	-13(2)	-15(2)	-17(2)
P(1)	19(1)	15(1)	17(1)	-4(1)	-6(1)	-3(1)
P(2)	15(1)	19(1)	16(1)	-4(1)	-5(1)	-4(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for Klarit8.

	x	y	z	U(eq)
H(2)	2670	1231	4529	24
H(5)	244	5145	3935	26
H(6)	-672	3929	4917	29
H(7)	494	2391	5078	26
H(11)	7471	2304	4557	32
H(12)	6458	1067	5473	34
H(13)	4502	775	5358	31
H(18A)	4662	4023	483	78
H(18B)	3267	3925	409	78
H(18C)	4451	3069	310	78
H(20A)	4103	-273	2006	93
H(20B)	3703	450	1224	93
H(20C)	2602	166	2059	93
H(22)	3526	6147	1015	42
H(23)	2678	6422	-138	54
H(24)	820	5790	-14	52
H(25)	-180	4923	1253	51
H(26)	677	4635	2400	38
H(28)	4568	5992	3209	32
H(29)	4309	7394	3647	44
H(30)	2436	8502	3593	48
H(31)	738	8162	3201	49
H(32)	995	6755	2751	39
H(34)	7662	3066	1574	41
H(35)	9690	2350	888	55
H(36)	11508	2115	1434	52
H(37)	11246	2556	2691	46
H(38)	9203	3281	3374	33
H(40)	7788	5399	2435	39
H(41)	8144	6571	2998	45

H(42)	7489	6497	4420	46
H(43)	6507	5255	5310	43
H(44)	6175	4065	4765	32
H(45A)	5679	9219	401	101
H(45B)	5393	8485	-48	101
H(46A)	7785	8675	-80	102
H(46B)	7459	7778	-289	102
H(47A)	7840	6872	891	172
H(47B)	8378	7732	1022	172
H(48A)	6276	7162	2055	76
H(48B)	6424	8256	1850	76
H(55A)	1158	-610	8002	109
H(55B)	1137	-757	7111	109
H(56A)	319	874	7591	80
H(56B)	715	778	6632	80
H(57A)	2541	1377	6377	46
H(57B)	2051	1590	7305	46
H(58A)	4124	378	6899	64
H(58B)	3210	246	7836	64

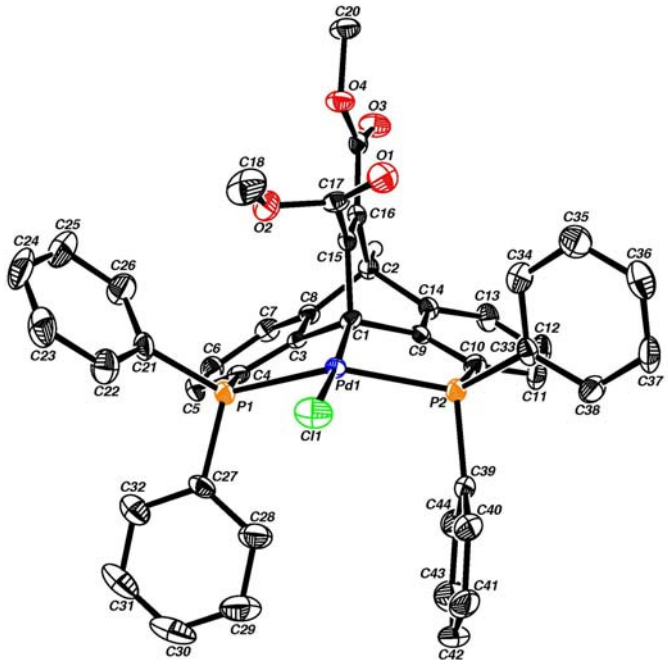


Table 1. Crystal data and structure refinement for Alina2.

Identification code	elena21m	
Empirical formula	C44 H33 Cl O4 P2 Pd	
Formula weight	829.49	
Temperature	173(1) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	P2(1)2(1)2(1)	
Unit cell dimensions	a = 8.3159(5) Å	α= 90°.
	b = 11.2104(7) Å	β= 90°.
	c = 38.813(2) Å	γ = 90°.
Volume	3618.4(4) $Å^3$	
Z	4	
Density (calculated)	1.523 Mg/m^3	
Absorption coefficient	0.720 mm^{-1}	
F(000)	1688	
Crystal size	0.19 x 0.16 x 0.11 mm^3	

Theta range for data collection	2.10 to 27.00°.
Index ranges	-10<=h<=10, -14<=k<=14, -49<=l<=49
Reflections collected	40630
Independent reflections	7909 [R(int) = 0.0515]
Completeness to theta = 27.00°	100.0 %
Absorption correction	None
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	7909 / 0 / 471
Goodness-of-fit on F^2	1.161
Final R indices [I>2sigma(I)]	R1 = 0.0376, wR2 = 0.0818
R indices (all data)	R1 = 0.0395, wR2 = 0.0826
Absolute structure parameter	-0.02(2)
Largest diff. peak and hole	0.926 and -0.767 e.$Å^{-3}$

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for Alina2. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
C(1)	8317(4)	9156(3)	9064(1)	15(1)
C(2)	9196(4)	10339(3)	9617(1)	18(1)
C(3)	9275(4)	10287(3)	8992(1)	17(1)
C(4)	9479(4)	10754(3)	8669(1)	18(1)
C(5)	10399(4)	11795(3)	8626(1)	24(1)
C(6)	11061(4)	12340(3)	8913(1)	25(1)
C(7)	10740(4)	11914(3)	9247(1)	22(1)
C(8)	9820(4)	10895(3)	9284(1)	18(1)
C(9)	9261(4)	8421(3)	9327(1)	17(1)
C(10)	9472(4)	7207(3)	9302(1)	20(1)
C(11)	10325(5)	6603(3)	9560(1)	27(1)
C(12)	10916(5)	7231(3)	9839(1)	30(1)
C(13)	10636(4)	8457(3)	9872(1)	25(1)
C(14)	9780(4)	9045(3)	9617(1)	20(1)
C(15)	6906(4)	9638(3)	9283(1)	17(1)
C(16)	7362(4)	10241(3)	9567(1)	18(1)
C(17)	5248(4)	9339(3)	9160(1)	20(1)
C(18)	3513(5)	9691(4)	8690(1)	46(1)
C(19)	6282(4)	10768(3)	9828(1)	18(1)
C(20)	3574(5)	11128(3)	9981(1)	29(1)
C(21)	6929(4)	10962(3)	8159(1)	24(1)
C(22)	6255(5)	10714(4)	7838(1)	33(1)
C(23)	5076(5)	11457(4)	7708(1)	40(1)
C(24)	4549(6)	12426(4)	7894(1)	47(1)
C(25)	5180(6)	12658(4)	8217(1)	49(1)
C(26)	6387(5)	11932(4)	8343(1)	35(1)
C(27)	9829(4)	9674(3)	7992(1)	24(1)
C(28)	10654(5)	8596(4)	7982(1)	36(1)
C(29)	11830(5)	8407(4)	7736(1)	47(1)
C(30)	12183(6)	9284(5)	7501(1)	53(1)
C(31)	11374(6)	10359(5)	7510(1)	47(1)

C(32)	10199(5)	10552(4)	7756(1)	32(1)
C(33)	7215(4)	5368(3)	9073(1)	20(1)
C(34)	5662(5)	5665(3)	9186(1)	32(1)
C(35)	4631(5)	4796(4)	9309(1)	39(1)
C(36)	5133(5)	3624(4)	9323(1)	36(1)
C(37)	6656(5)	3311(3)	9214(1)	32(1)
C(38)	7696(5)	4183(3)	9091(1)	27(1)
C(39)	10116(4)	5915(3)	8671(1)	23(1)
C(40)	9774(5)	5138(3)	8402(1)	30(1)
C(41)	10994(6)	4784(4)	8179(1)	41(1)
C(42)	12512(6)	5242(4)	8216(1)	43(1)
C(43)	12875(6)	6003(4)	8482(1)	47(1)
C(44)	11670(5)	6348(4)	8709(1)	34(1)
Cl(1)	6671(1)	7216(1)	8138(1)	33(1)
O(1)	4474(3)	8503(2)	9256(1)	32(1)
O(2)	4869(3)	10057(2)	8898(1)	29(1)
O(3)	6733(3)	11165(2)	10098(1)	30(1)
O(4)	4747(3)	10719(2)	9732(1)	26(1)
P(1)	8394(1)	9933(1)	8338(1)	18(1)
P(2)	8501(1)	6569(1)	8925(1)	18(1)
Pd(1)	7702(1)	8226(1)	8627(1)	15(1)

Table 3. Bond lengths [Å] and angles [°] for Alina2.

C(1)-C(3)	1.523(5)
C(1)-C(9)	1.528(4)
C(1)-C(15)	1.546(4)
C(1)-Pd(1)	2.057(3)
C(2)-C(8)	1.525(5)
C(2)-C(14)	1.530(5)
C(2)-C(16)	1.541(5)
C(2)-H(2)	1.0000
C(3)-C(4)	1.371(5)
C(3)-C(8)	1.398(5)
C(4)-C(5)	1.405(5)
C(4)-P(1)	1.819(3)
C(5)-C(6)	1.384(5)
C(5)-H(5)	0.9500
C(6)-C(7)	1.408(5)
C(6)-H(6)	0.9500
C(7)-C(8)	1.382(5)
C(7)-H(7)	0.9500
C(9)-C(10)	1.376(5)
C(9)-C(14)	1.393(5)
C(10)-C(11)	1.401(5)
C(10)-P(2)	1.821(4)
C(11)-C(12)	1.381(5)
C(11)-H(11)	0.9500
C(12)-C(13)	1.400(5)
C(12)-H(12)	0.9500
C(13)-C(14)	1.386(5)
C(13)-H(13)	0.9500
C(15)-C(16)	1.347(4)
C(15)-C(17)	1.498(5)
C(16)-C(19)	1.476(4)
C(17)-O(1)	1.196(4)
C(17)-O(2)	1.335(4)
C(18)-O(2)	1.446(5)

C(18)-H(18A)	0.9800
C(18)-H(18B)	0.9800
C(18)-H(18C)	0.9800
C(19)-O(3)	1.198(4)
C(19)-O(4)	1.332(4)
C(20)-O(4)	1.448(4)
C(20)-H(20A)	0.9800
C(20)-H(20B)	0.9800
C(20)-H(20C)	0.9800
C(21)-C(26)	1.376(5)
C(21)-C(22)	1.394(5)
C(21)-P(1)	1.815(3)
C(22)-C(23)	1.382(5)
C(22)-H(22)	0.9500
C(23)-C(24)	1.376(6)
C(23)-H(23)	0.9500
C(24)-C(25)	1.383(7)
C(24)-H(24)	0.9500
C(25)-C(26)	1.381(6)
C(25)-H(25)	0.9500
C(26)-H(26)	0.9500
C(27)-C(32)	1.381(5)
C(27)-C(28)	1.390(5)
C(27)-P(1)	1.820(4)
C(28)-C(29)	1.384(6)
C(28)-H(28)	0.9500
C(29)-C(30)	1.371(6)
C(29)-H(29)	0.9500
C(30)-C(31)	1.380(7)
C(30)-H(30)	0.9500
C(31)-C(32)	1.383(6)
C(31)-H(31)	0.9500
C(32)-H(32)	0.9500
C(33)-C(38)	1.389(4)
C(33)-C(34)	1.404(5)
C(33)-P(2)	1.813(3)

C(34)-C(35)	1.383(6)
C(34)-H(34)	0.9500
C(35)-C(36)	1.380(6)
C(35)-H(35)	0.9500
C(36)-C(37)	1.380(6)
C(36)-H(36)	0.9500
C(37)-C(38)	1.390(5)
C(37)-H(37)	0.9500
C(38)-H(38)	0.9500
C(39)-C(44)	1.388(6)
C(39)-C(40)	1.391(5)
C(39)-P(2)	1.818(4)
C(40)-C(41)	1.390(6)
C(40)-H(40)	0.9500
C(41)-C(42)	1.370(7)
C(41)-H(41)	0.9500
C(42)-C(43)	1.374(6)
C(42)-H(42)	0.9500
C(43)-C(44)	1.389(6)
C(43)-H(43)	0.9500
C(44)-H(44)	0.9500
Cl(1)-Pd(1)	2.3694(9)
P(1)-Pd(1)	2.2908(9)
P(2)-Pd(1)	2.2874(9)

C(3)-C(1)-C(9)	107.6(3)
C(3)-C(1)-C(15)	102.0(2)
C(9)-C(1)-C(15)	102.2(2)
C(3)-C(1)-Pd(1)	113.6(2)
C(9)-C(1)-Pd(1)	113.9(2)
C(15)-C(1)-Pd(1)	116.3(2)
C(8)-C(2)-C(14)	106.2(3)
C(8)-C(2)-C(16)	105.1(3)
C(14)-C(2)-C(16)	104.2(3)
C(8)-C(2)-H(2)	113.5
C(14)-C(2)-H(2)	113.5

C(16)-C(2)-H(2)	113.5
C(4)-C(3)-C(8)	121.0(3)
C(4)-C(3)-C(1)	123.5(3)
C(8)-C(3)-C(1)	115.3(3)
C(3)-C(4)-C(5)	119.5(3)
C(3)-C(4)-P(1)	113.0(3)
C(5)-C(4)-P(1)	127.4(3)
C(6)-C(5)-C(4)	119.3(3)
C(6)-C(5)-H(5)	120.3
C(4)-C(5)-H(5)	120.3
C(5)-C(6)-C(7)	121.0(3)
C(5)-C(6)-H(6)	119.5
C(7)-C(6)-H(6)	119.5
C(8)-C(7)-C(6)	118.7(3)
C(8)-C(7)-H(7)	120.6
C(6)-C(7)-H(7)	120.6
C(7)-C(8)-C(3)	119.9(3)
C(7)-C(8)-C(2)	127.9(3)
C(3)-C(8)-C(2)	112.1(3)
C(10)-C(9)-C(14)	120.8(3)
C(10)-C(9)-C(1)	123.6(3)
C(14)-C(9)-C(1)	115.3(3)
C(9)-C(10)-C(11)	119.6(3)
C(9)-C(10)-P(2)	112.8(3)
C(11)-C(10)-P(2)	127.5(3)
C(12)-C(11)-C(10)	119.5(3)
C(12)-C(11)-H(11)	120.3
C(10)-C(11)-H(11)	120.3
C(11)-C(12)-C(13)	120.9(3)
C(11)-C(12)-H(12)	119.5
C(13)-C(12)-H(12)	119.6
C(14)-C(13)-C(12)	119.1(3)
C(14)-C(13)-H(13)	120.5
C(12)-C(13)-H(13)	120.5
C(13)-C(14)-C(9)	119.9(3)
C(13)-C(14)-C(2)	127.8(3)

C(9)-C(14)-C(2)	112.2(3)
C(16)-C(15)-C(17)	129.3(3)
C(16)-C(15)-C(1)	114.3(3)
C(17)-C(15)-C(1)	116.4(3)
C(15)-C(16)-C(19)	126.2(3)
C(15)-C(16)-C(2)	114.6(3)
C(19)-C(16)-C(2)	119.2(3)
O(1)-C(17)-O(2)	125.7(3)
O(1)-C(17)-C(15)	124.8(3)
O(2)-C(17)-C(15)	109.0(3)
O(2)-C(18)-H(18A)	109.5
O(2)-C(18)-H(18B)	109.5
H(18A)-C(18)-H(18B)	109.5
O(2)-C(18)-H(18C)	109.5
H(18A)-C(18)-H(18C)	109.5
H(18B)-C(18)-H(18C)	109.5
O(3)-C(19)-O(4)	124.1(3)
O(3)-C(19)-C(16)	123.9(3)
O(4)-C(19)-C(16)	111.9(3)
O(4)-C(20)-H(20A)	109.5
O(4)-C(20)-H(20B)	109.5
H(20A)-C(20)-H(20B)	109.5
O(4)-C(20)-H(20C)	109.5
H(20A)-C(20)-H(20C)	109.5
H(20B)-C(20)-H(20C)	109.5
C(26)-C(21)-C(22)	119.2(3)
C(26)-C(21)-P(1)	121.6(3)
C(22)-C(21)-P(1)	119.0(3)
C(23)-C(22)-C(21)	119.5(4)
C(23)-C(22)-H(22)	120.3
C(21)-C(22)-H(22)	120.3
C(24)-C(23)-C(22)	120.7(4)
C(24)-C(23)-H(23)	119.7
C(22)-C(23)-H(23)	119.7
C(23)-C(24)-C(25)	120.2(4)
C(23)-C(24)-H(24)	119.9

C(25)-C(24)-H(24)	119.9
C(26)-C(25)-C(24)	119.0(4)
C(26)-C(25)-H(25)	120.5
C(24)-C(25)-H(25)	120.5
C(21)-C(26)-C(25)	121.4(4)
C(21)-C(26)-H(26)	119.3
C(25)-C(26)-H(26)	119.3
C(32)-C(27)-C(28)	119.4(4)
C(32)-C(27)-P(1)	121.5(3)
C(28)-C(27)-P(1)	118.9(3)
C(29)-C(28)-C(27)	120.1(4)
C(29)-C(28)-H(28)	119.9
C(27)-C(28)-H(28)	119.9
C(30)-C(29)-C(28)	119.9(4)
C(30)-C(29)-H(29)	120.0
C(28)-C(29)-H(29)	120.0
C(29)-C(30)-C(31)	120.4(4)
C(29)-C(30)-H(30)	119.8
C(31)-C(30)-H(30)	119.8
C(30)-C(31)-C(32)	119.9(4)
C(30)-C(31)-H(31)	120.1
C(32)-C(31)-H(31)	120.1
C(27)-C(32)-C(31)	120.3(4)
C(27)-C(32)-H(32)	119.9
C(31)-C(32)-H(32)	119.9
C(38)-C(33)-C(34)	118.5(3)
C(38)-C(33)-P(2)	123.8(3)
C(34)-C(33)-P(2)	117.7(3)
C(35)-C(34)-C(33)	120.7(4)
C(35)-C(34)-H(34)	119.6
C(33)-C(34)-H(34)	119.6
C(36)-C(35)-C(34)	119.8(4)
C(36)-C(35)-H(35)	120.1
C(34)-C(35)-H(35)	120.1
C(35)-C(36)-C(37)	120.5(4)
C(35)-C(36)-H(36)	119.8

C(37)-C(36)-H(36)	119.8
C(36)-C(37)-C(38)	119.9(4)
C(36)-C(37)-H(37)	120.1
C(38)-C(37)-H(37)	120.1
C(33)-C(38)-C(37)	120.7(4)
C(33)-C(38)-H(38)	119.7
C(37)-C(38)-H(38)	119.7
C(44)-C(39)-C(40)	119.2(3)
C(44)-C(39)-P(2)	119.4(3)
C(40)-C(39)-P(2)	120.5(3)
C(41)-C(40)-C(39)	119.8(4)
C(41)-C(40)-H(40)	120.1
C(39)-C(40)-H(40)	120.1
C(42)-C(41)-C(40)	120.1(4)
C(42)-C(41)-H(41)	119.9
C(40)-C(41)-H(41)	119.9
C(41)-C(42)-C(43)	120.8(4)
C(41)-C(42)-H(42)	119.6
C(43)-C(42)-H(42)	119.6
C(42)-C(43)-C(44)	119.5(4)
C(42)-C(43)-H(43)	120.3
C(44)-C(43)-H(43)	120.3
C(39)-C(44)-C(43)	120.5(4)
C(39)-C(44)-H(44)	119.7
C(43)-C(44)-H(44)	119.7
C(17)-O(2)-C(18)	116.0(3)
C(19)-O(4)-C(20)	116.4(3)
C(21)-P(1)-C(4)	106.34(16)
C(21)-P(1)-C(27)	105.05(16)
C(4)-P(1)-C(27)	106.04(16)
C(21)-P(1)-Pd(1)	123.29(12)
C(4)-P(1)-Pd(1)	101.67(11)
C(27)-P(1)-Pd(1)	113.07(13)
C(33)-P(2)-C(39)	108.00(16)
C(33)-P(2)-C(10)	107.25(15)
C(39)-P(2)-C(10)	105.43(17)

C(33)-P(2)-Pd(1)	126.36(12)
C(39)-P(2)-Pd(1)	105.58(11)
C(10)-P(2)-Pd(1)	102.50(11)
C(1)-Pd(1)-P(2)	85.55(9)
C(1)-Pd(1)-P(1)	85.29(9)
P(2)-Pd(1)-P(1)	148.53(3)
C(1)-Pd(1)-Cl(1)	173.14(10)
P(2)-Pd(1)-Cl(1)	96.96(3)
P(1)-Pd(1)-Cl(1)	95.68(3)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for Alina2. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
C(1)	18(2)	15(2)	13(2)	-5(1)	-3(1)	3(1)
C(2)	22(2)	17(2)	16(2)	-1(1)	-1(1)	1(1)
C(3)	13(2)	16(2)	21(2)	2(1)	4(1)	4(1)
C(4)	17(2)	18(2)	17(2)	-3(1)	2(1)	3(1)
C(5)	31(2)	21(2)	20(2)	7(2)	7(2)	2(2)
C(6)	24(2)	19(2)	31(2)	2(2)	2(2)	-2(2)
C(7)	23(2)	21(2)	21(2)	-7(1)	-4(1)	1(2)
C(8)	19(2)	18(2)	17(2)	-2(1)	1(1)	2(1)
C(9)	15(2)	22(2)	13(2)	4(1)	3(1)	2(1)
C(10)	20(2)	20(2)	20(2)	0(1)	-1(1)	1(1)
C(11)	37(2)	16(2)	28(2)	6(2)	-2(2)	2(2)
C(12)	37(2)	28(2)	26(2)	9(2)	-12(2)	3(2)
C(13)	31(2)	25(2)	19(2)	2(1)	-6(1)	-3(2)
C(14)	22(2)	22(2)	15(2)	3(1)	0(1)	1(1)
C(15)	20(2)	13(2)	17(2)	2(1)	2(1)	0(1)
C(16)	21(2)	16(1)	18(2)	3(1)	3(1)	1(1)
C(17)	20(2)	23(2)	16(2)	-8(1)	0(1)	4(1)
C(18)	30(2)	64(3)	44(3)	-4(2)	-16(2)	9(2)
C(19)	24(2)	10(2)	20(2)	1(1)	2(1)	-1(1)
C(20)	25(2)	34(2)	29(2)	-5(2)	6(2)	7(2)
C(21)	24(2)	28(2)	19(2)	9(1)	-4(1)	2(2)
C(22)	37(2)	33(2)	30(2)	3(2)	-8(2)	1(2)
C(23)	35(2)	51(3)	34(2)	14(2)	-15(2)	-3(2)
C(24)	39(3)	42(3)	61(3)	20(2)	-15(2)	12(2)
C(25)	57(3)	35(2)	57(3)	8(2)	0(2)	21(2)
C(26)	44(2)	34(2)	27(2)	-5(2)	-5(2)	11(2)
C(27)	26(2)	32(2)	12(2)	-1(1)	3(1)	0(2)
C(28)	38(2)	36(2)	33(2)	-5(2)	12(2)	3(2)
C(29)	47(3)	49(3)	46(3)	-14(2)	17(2)	5(2)
C(30)	38(3)	84(4)	36(2)	-10(2)	19(2)	-9(3)
C(31)	47(3)	66(3)	28(2)	8(2)	11(2)	-18(3)

C(32)	29(2)	42(2)	26(2)	4(2)	1(2)	-8(2)
C(33)	29(2)	17(2)	14(1)	2(1)	-2(1)	-4(2)
C(34)	37(2)	22(2)	36(2)	3(2)	3(2)	4(2)
C(35)	29(2)	38(2)	50(3)	9(2)	6(2)	-2(2)
C(36)	41(2)	31(2)	35(2)	8(2)	-2(2)	-12(2)
C(37)	49(2)	18(2)	29(2)	8(2)	-1(2)	-1(2)
C(38)	28(2)	24(2)	28(2)	1(1)	0(2)	7(2)
C(39)	29(2)	17(2)	22(2)	4(1)	6(2)	6(1)
C(40)	36(2)	24(2)	29(2)	-2(2)	4(2)	6(2)
C(41)	66(3)	33(2)	24(2)	-4(2)	6(2)	23(2)
C(42)	43(3)	49(2)	36(2)	8(2)	19(2)	24(2)
C(43)	34(2)	57(3)	51(3)	10(2)	9(2)	8(2)
C(44)	30(2)	38(2)	34(2)	1(2)	1(2)	5(2)
Cl(1)	35(1)	40(1)	22(1)	-15(1)	-4(1)	-3(1)
O(1)	27(1)	26(2)	43(2)	0(1)	3(1)	-7(1)
O(2)	24(1)	35(2)	29(1)	4(1)	-7(1)	4(1)
O(3)	32(2)	36(2)	23(1)	-11(1)	5(1)	0(1)
O(4)	23(1)	34(2)	20(1)	-4(1)	3(1)	4(1)
P(1)	23(1)	19(1)	14(1)	2(1)	0(1)	2(1)
P(2)	24(1)	13(1)	19(1)	0(1)	2(1)	1(1)
Pd(1)	18(1)	15(1)	13(1)	-1(1)	0(1)	1(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for Alina2.

	x	y	z	U(eq)
H(2)	9512	10792	9828	22
H(5)	10566	12120	8403	29
H(6)	11743	13013	8884	30
H(7)	11147	12320	9444	26
H(11)	10497	5767	9543	33
H(12)	11521	6826	10010	36
H(13)	11027	8881	10066	30
H(18A)	3728	8904	8591	69
H(18B)	3346	10271	8505	69
H(18C)	2547	9652	8834	69
H(20A)	3694	10674	10195	44
H(20B)	2489	11010	9889	44
H(20C)	3745	11978	10027	44
H(22)	6602	10040	7710	40
H(23)	4625	11298	7488	48
H(24)	3749	12937	7801	57
H(25)	4789	13306	8350	59
H(26)	6854	12107	8560	42
H(28)	10410	7989	8144	43
H(29)	12393	7669	7729	56
H(30)	12988	9152	7332	63
H(31)	11624	10964	7347	57
H(32)	9644	11292	7762	39
H(34)	5315	6472	9178	38
H(35)	3581	5006	9384	47
H(36)	4425	3027	9408	43
H(37)	6994	2503	9224	38
H(38)	8745	3966	9017	32
H(40)	8710	4850	8370	36
H(41)	10776	4224	8002	49

H(42)	13324	5031	8055	52
H(43)	13941	6291	8511	57
H(44)	11911	6884	8891	41