Rapid Oxidative Hydrogen Evolution from a Family of Square–Planar Nickel Hydride Complexes

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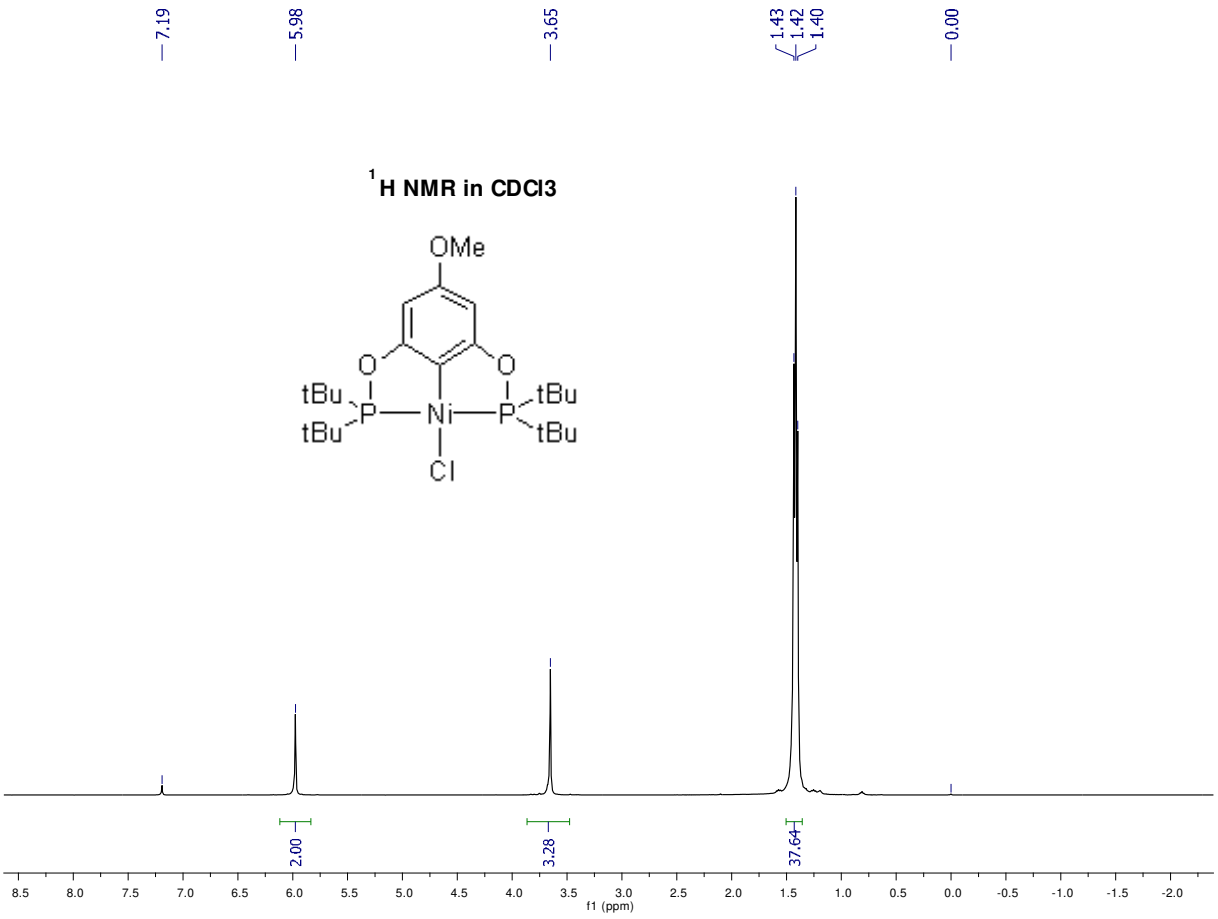
Supporting Information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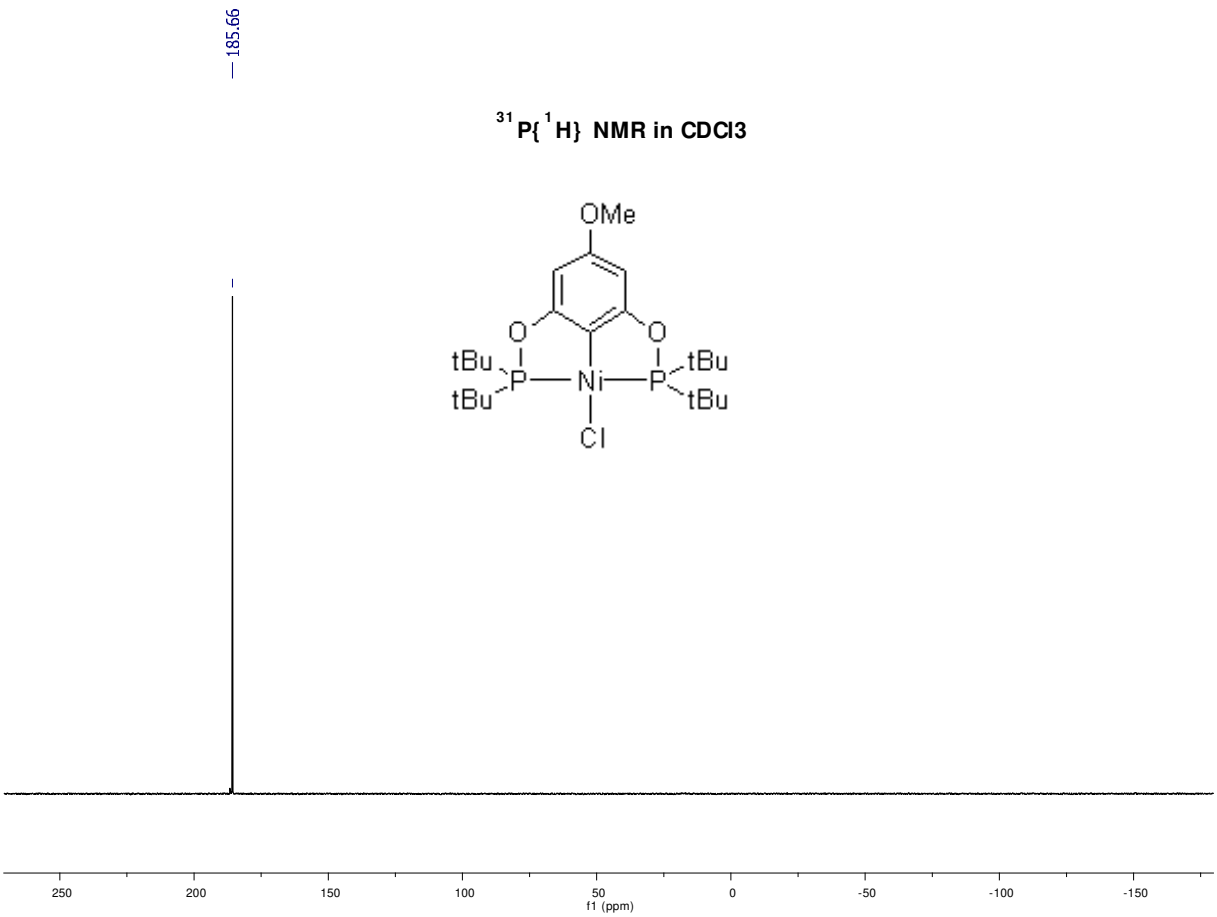
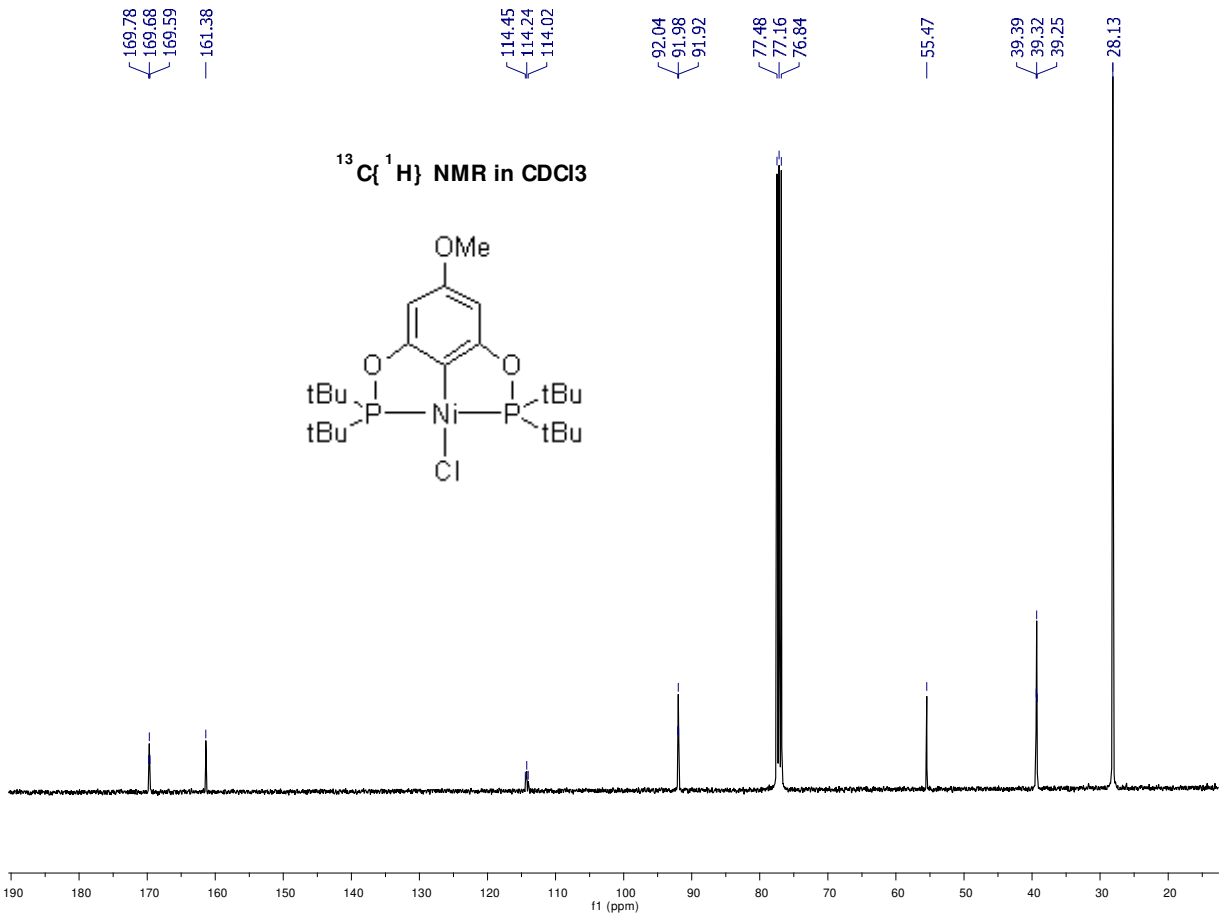
5.98
3.65
1.43
1.42
1.40
0.00
1H NMR in CDCl3
OMe
O
O
tBu
tBu
P
Ni
P
tBu
tBu
Cl
2.00
3.28
37.64
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0 -1.5 -2.0
f1 (ppm)

$^{31}P\{^{1}H\}$ NMR in CDCl3

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169.68
169.59
161.38
114.45
114.24
114.02
92.04
91.98
91.92
77.48
77.16
76.84
55.47
39.39
39.32
39.25
28.13
13C{ 1H} NMR in CDCl3
OMe
O
O
tBu
tBu
P
Ni
P
tBu
tBu
Cl
190
180
170
160
150
140
130
120
110
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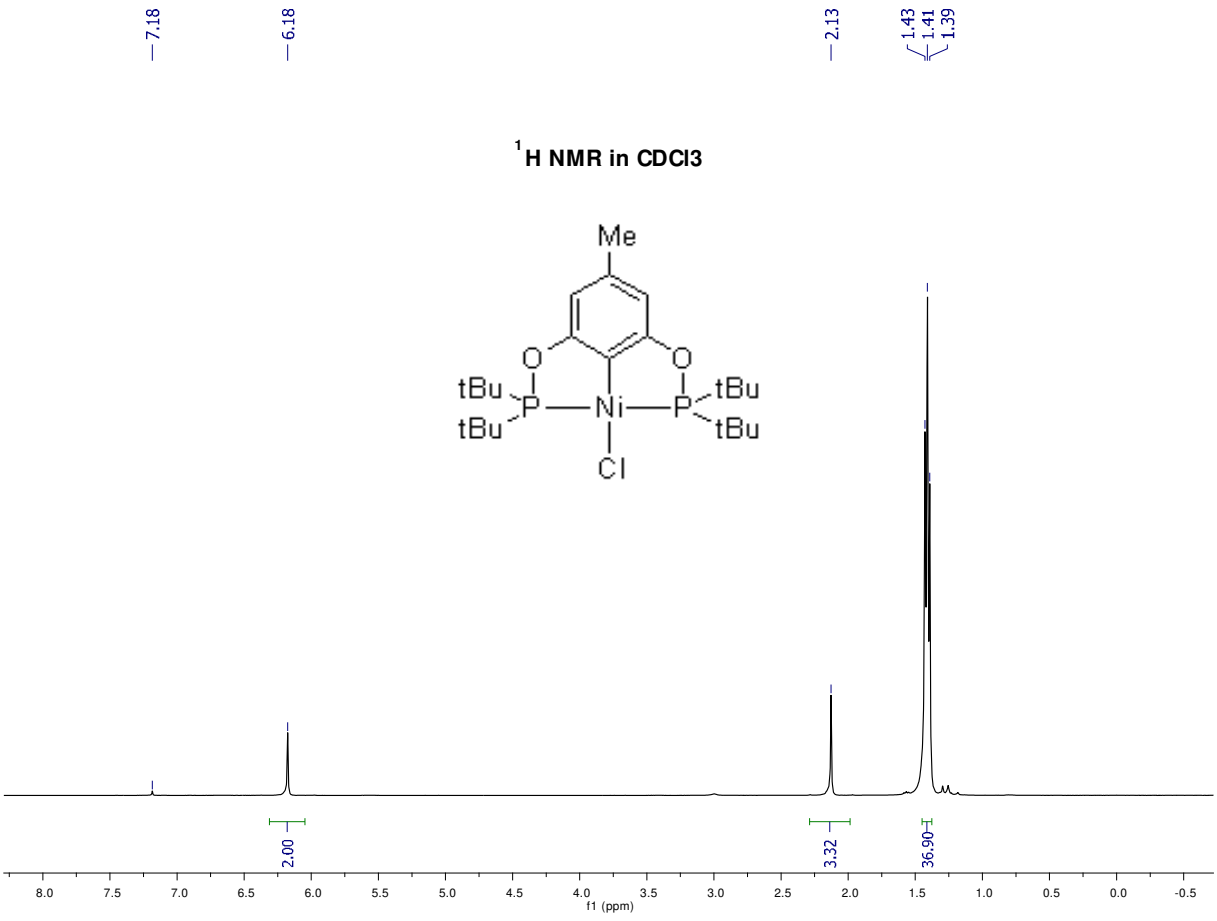
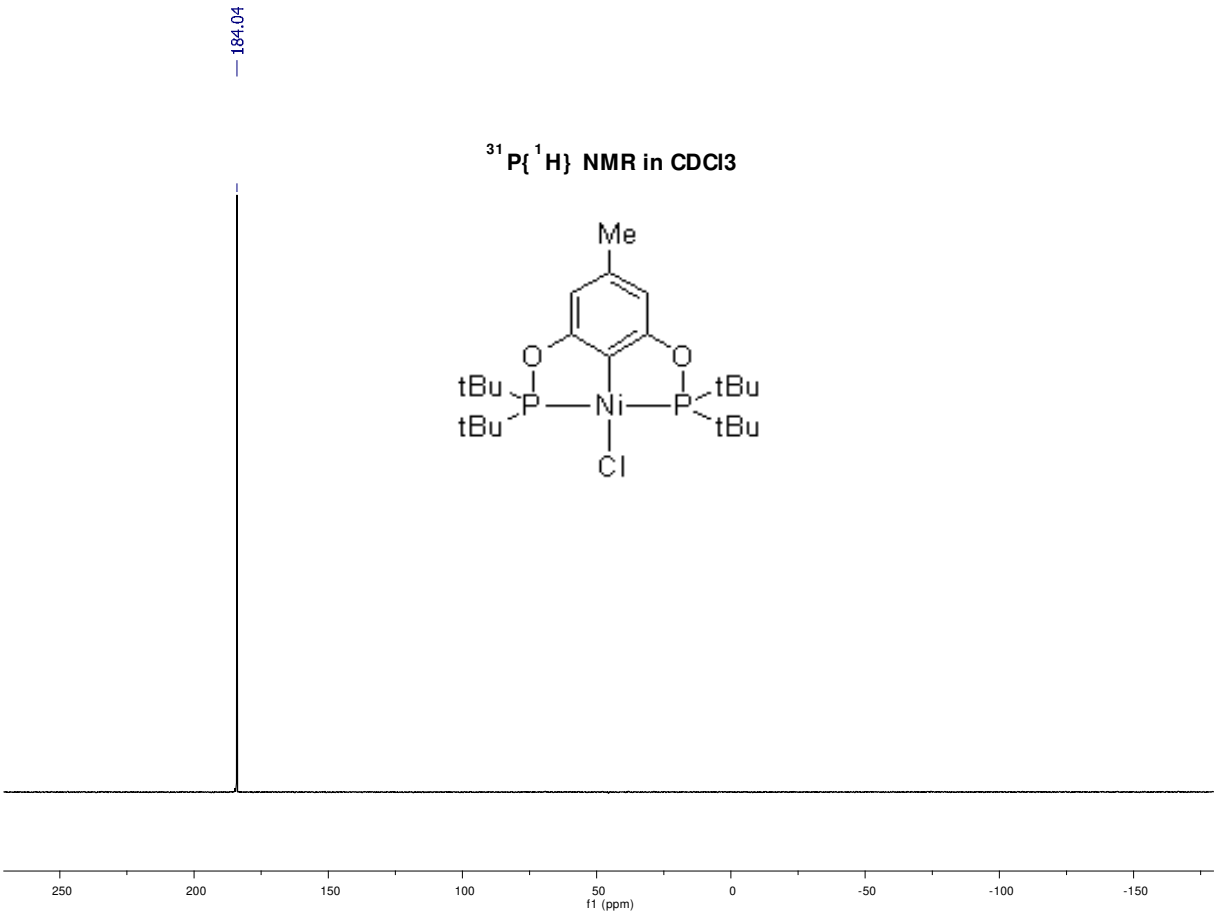
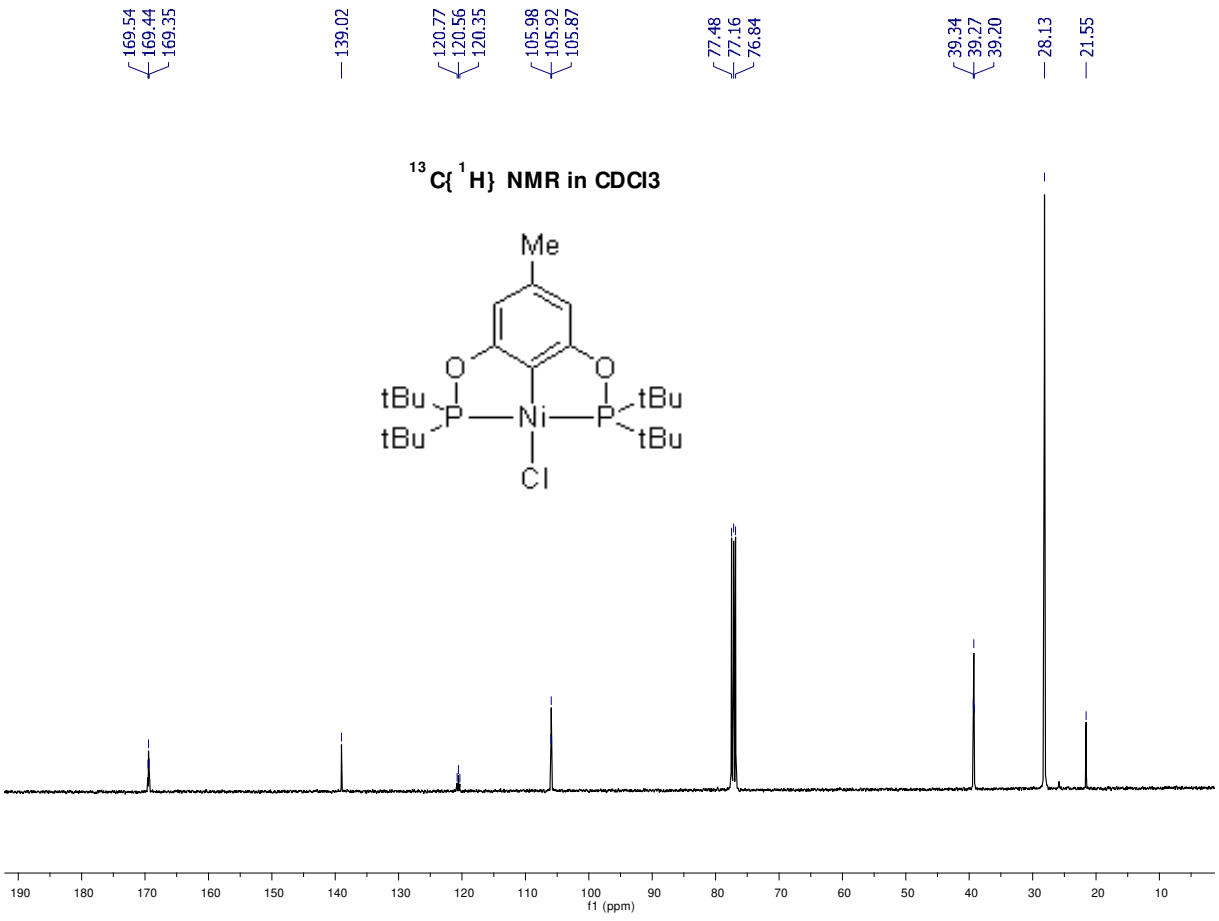
80
70
60
50
40
30
20
f1 (ppm)



^{1}H NMR in CDCl3


184.04
31P{1H} NMR in CDCl3
Me
O
O
tBu
tBu
P
Ni
P
tBu
tBu
Cl
250
200
150
100
50
0
-50
-100
-150
f1 (ppm)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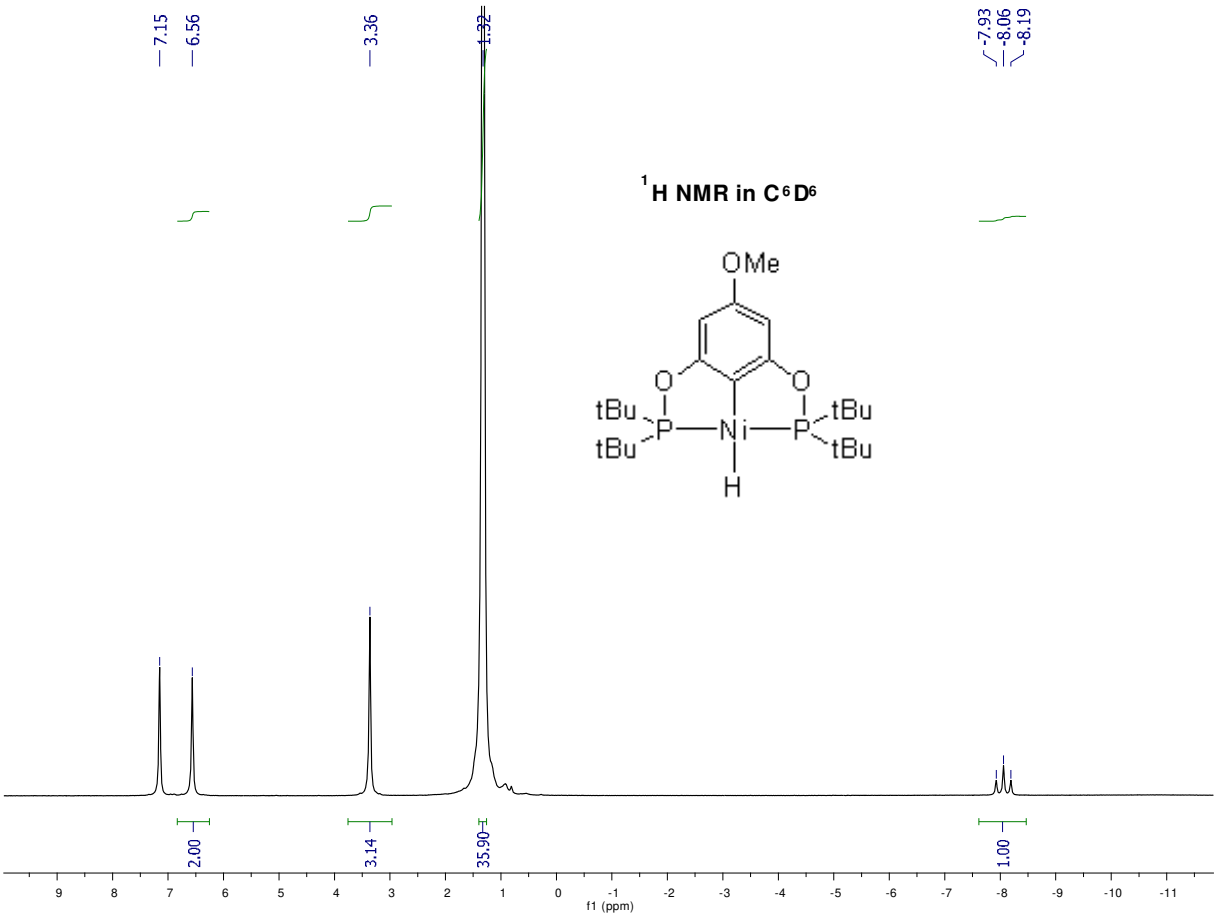
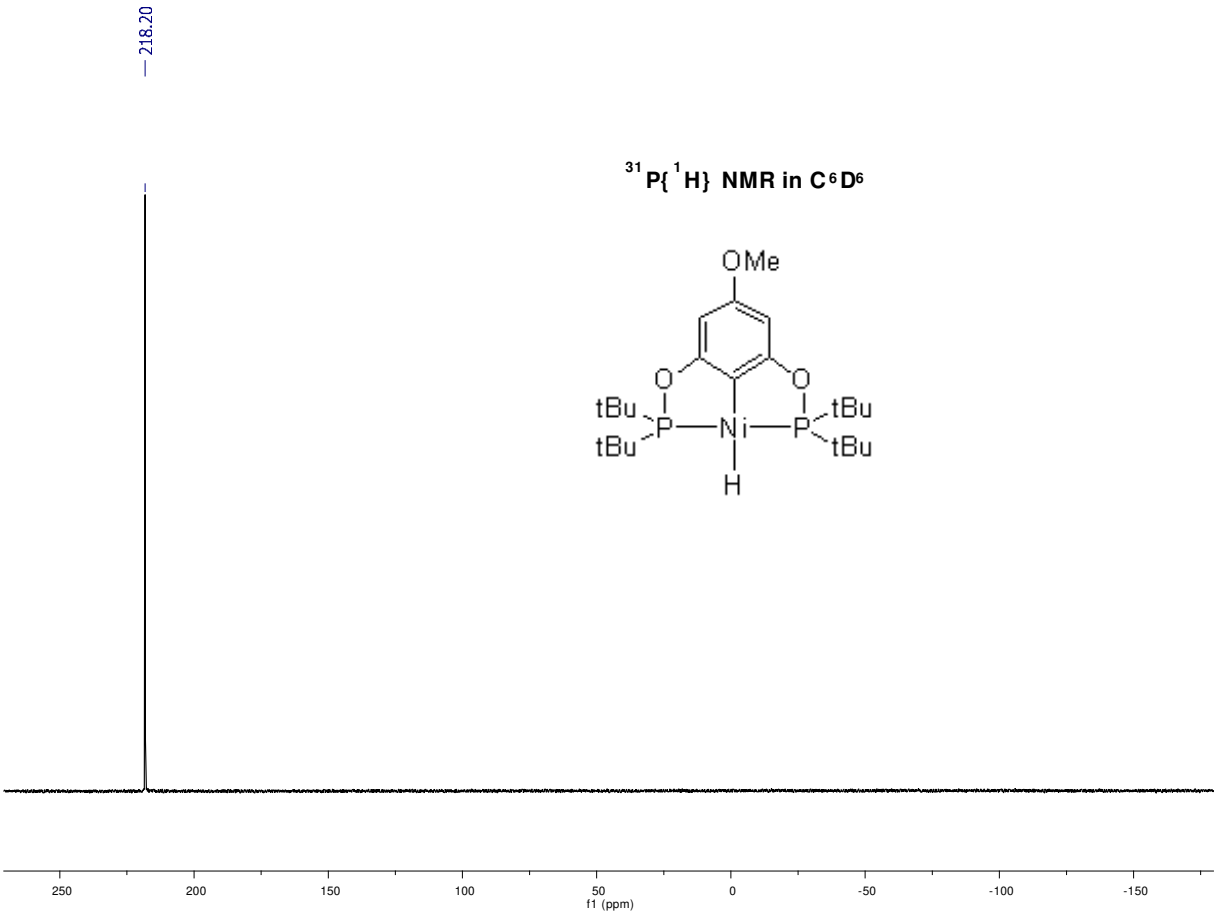
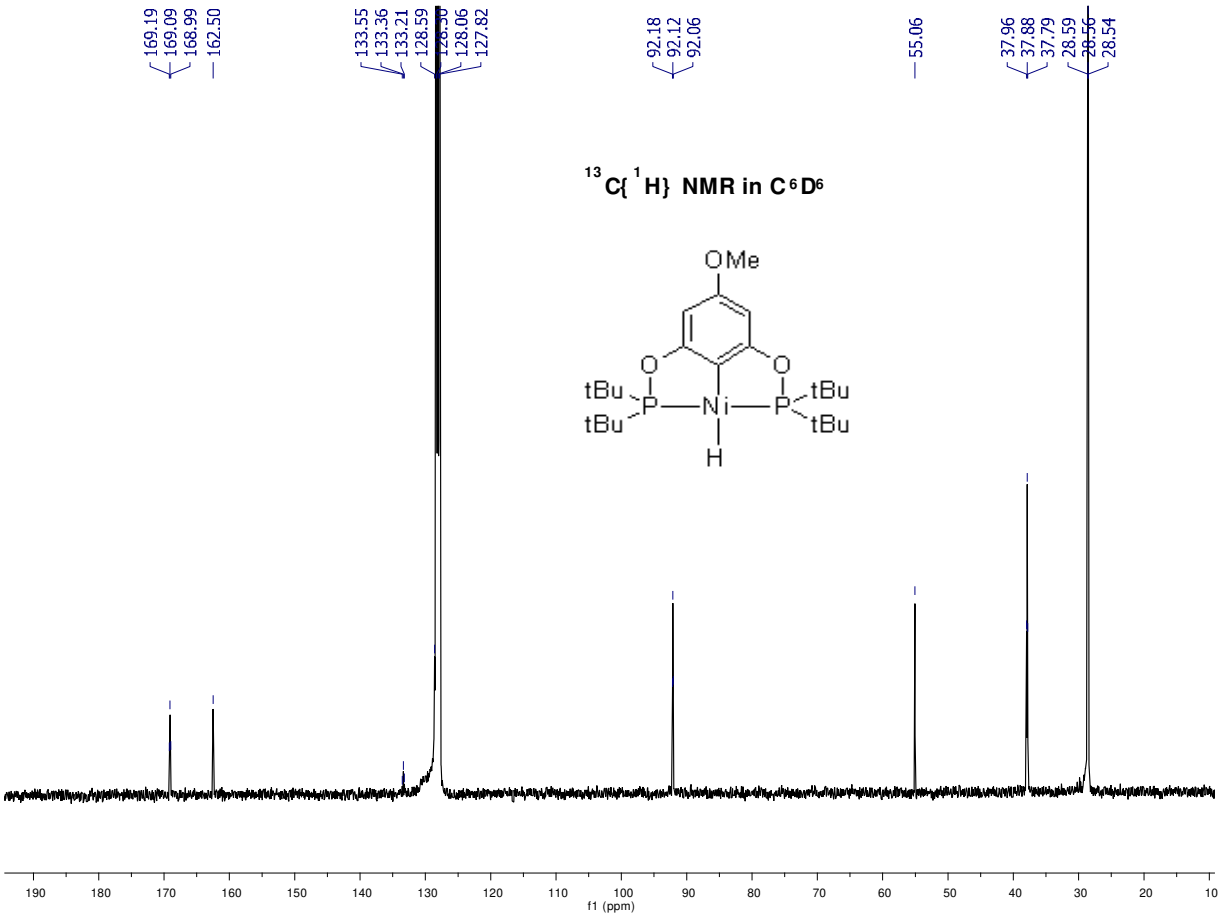
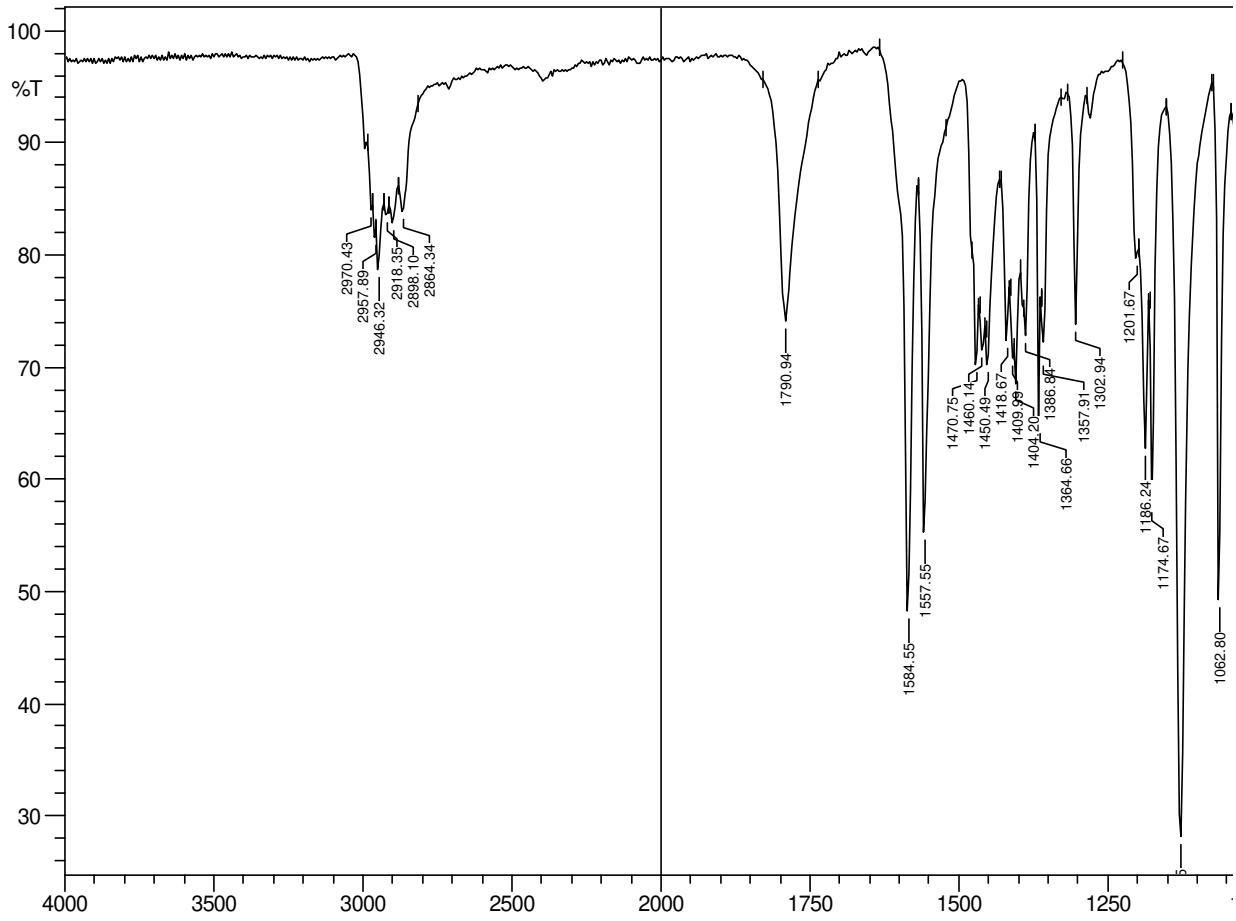
169.44
169.35
139.02
120.77
120.56
120.35
105.98
105.92
105.87
77.48
77.16
76.84
39.34
39.27
39.20
28.13
21.55
13C{ 1H} NMR in CDCl3
Me
O
O
tBu
tBu
P
Ni
P
tBu
tBu
Cl
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

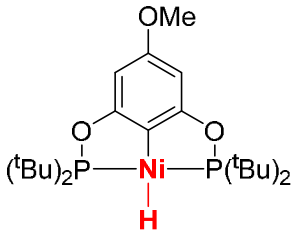


^{1}H NMR in C_6D_6

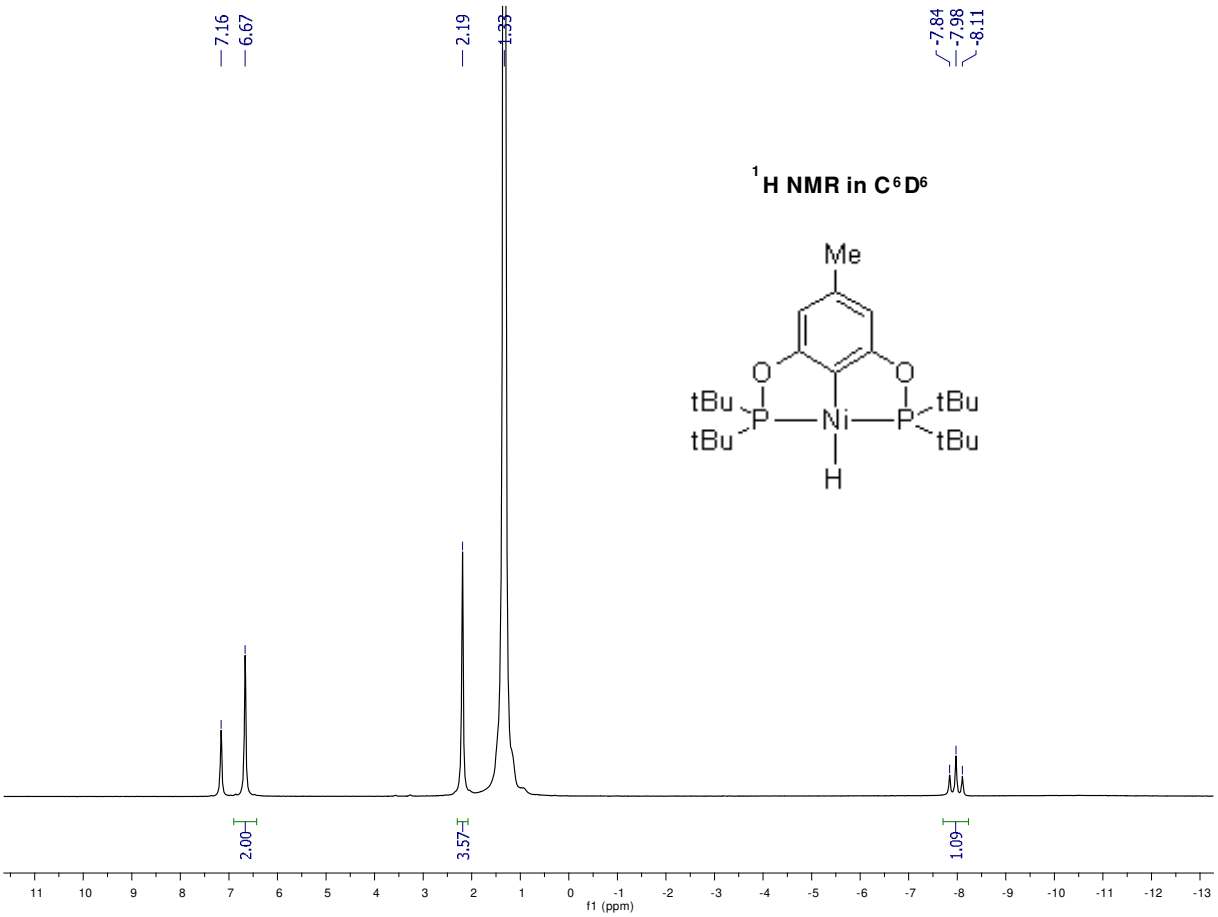

218.20
31P{1H} NMR in C6D6
OMe
O
O
tBu
tBu
P
Ni
P
tBu
tBu
H
250
200
150
100
50
0
-50
-100
-150
f1 (ppm)

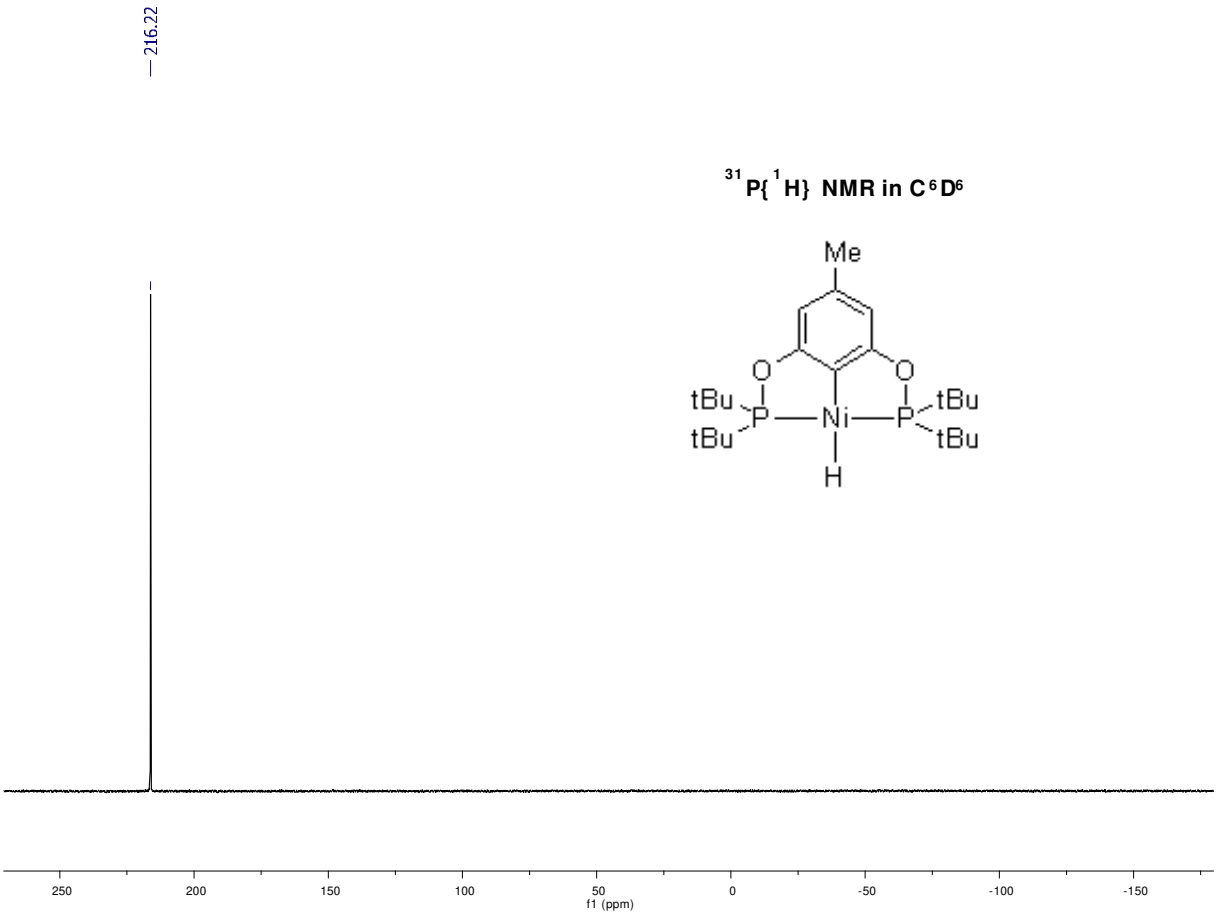

169.19
169.09
168.99
162.50
133.55
133.36
133.21
128.59
128.06
127.82
92.18
92.12
92.06
55.06
37.96
37.88
37.79
28.59
28.56
28.54
13C{1H} NMR in C6D6
OMe
O
O
tBu
tBu
P
Ni
P
tBu
tBu
H
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

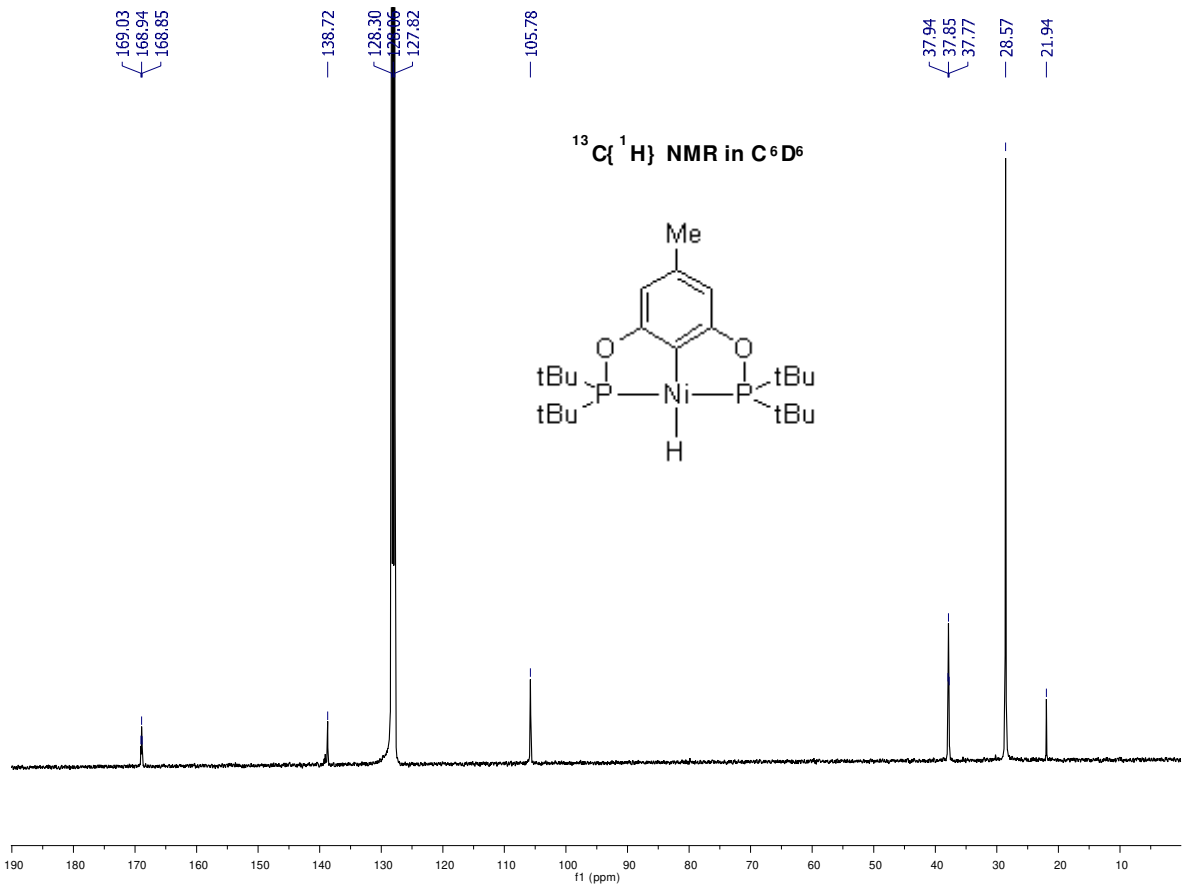


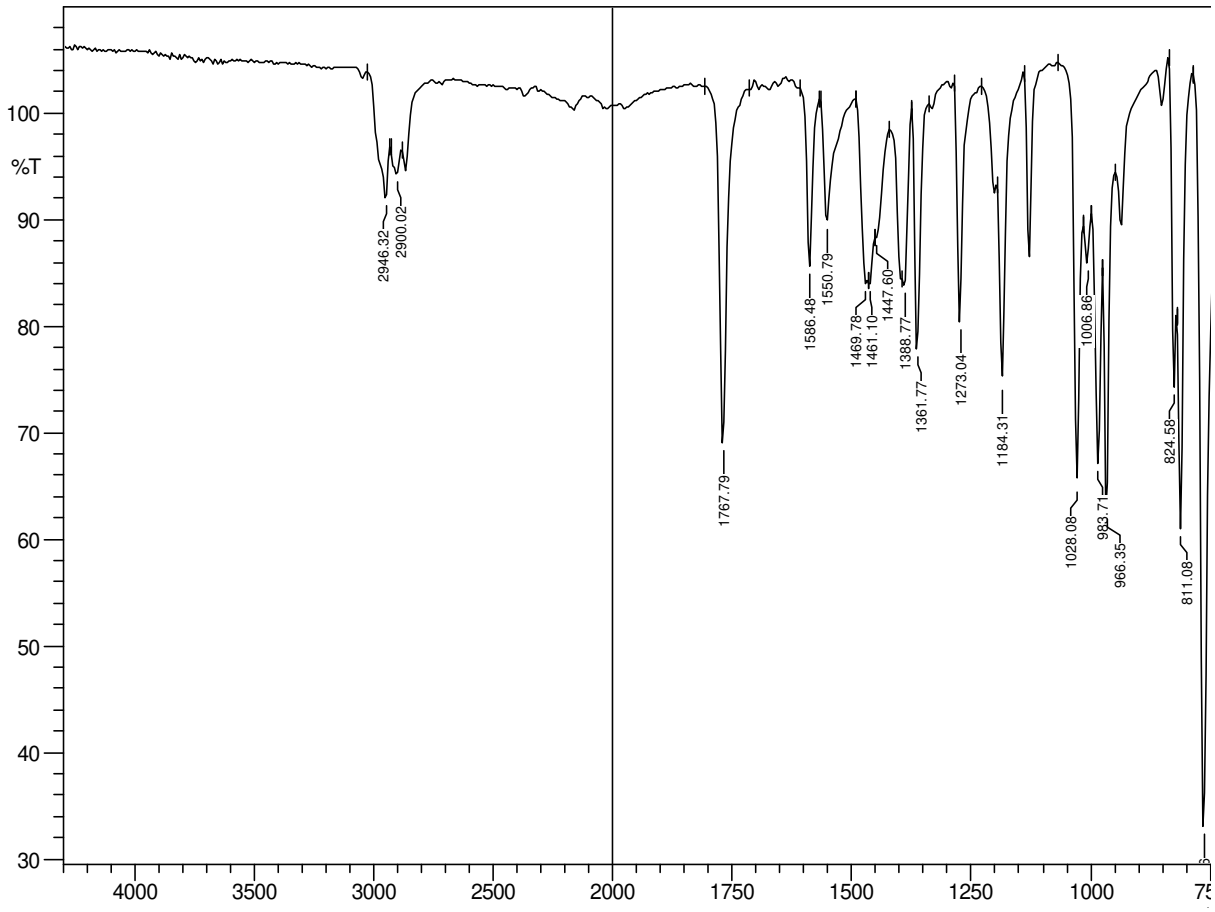


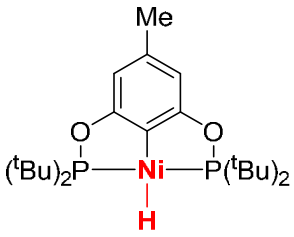
Solid state IR of **3b**


7.16
6.67
2.19
1.33
-7.84
-7.98
-8.11
1H NMR in C6D6
Me
O
O
tBu
tBu
P
Ni
P
tBu
tBu
H
2.00
3.57
1.09
11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3 -4 -5 -6 -7 -8 -9 -10 -11 -12 -13
f1 (ppm)

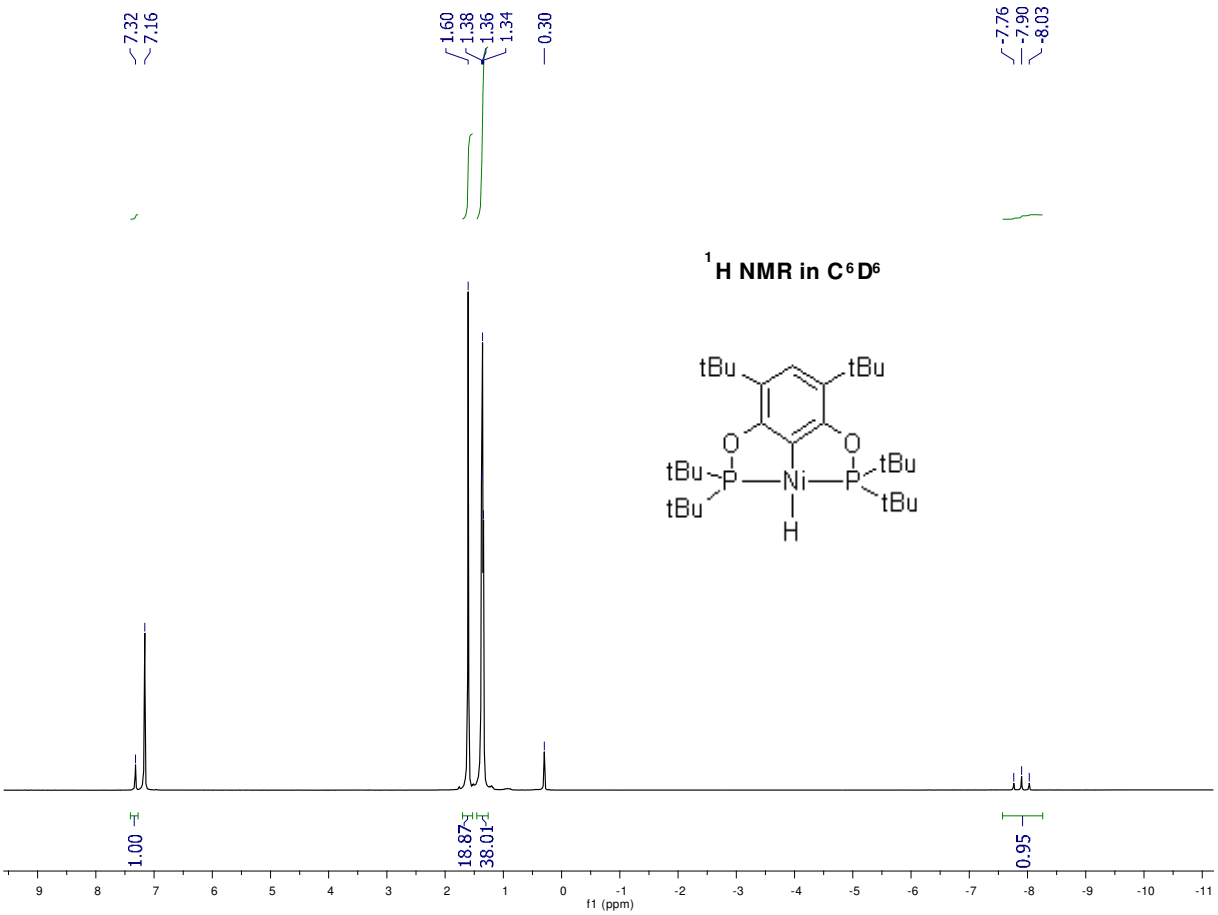

216.22
31P{1H} NMR in C6D6
Me
O
O
tBu
tBu
P
Ni
P
tBu
tBu
H
250
200
150
100
50
0
-50
-100
-150
f1 (ppm)

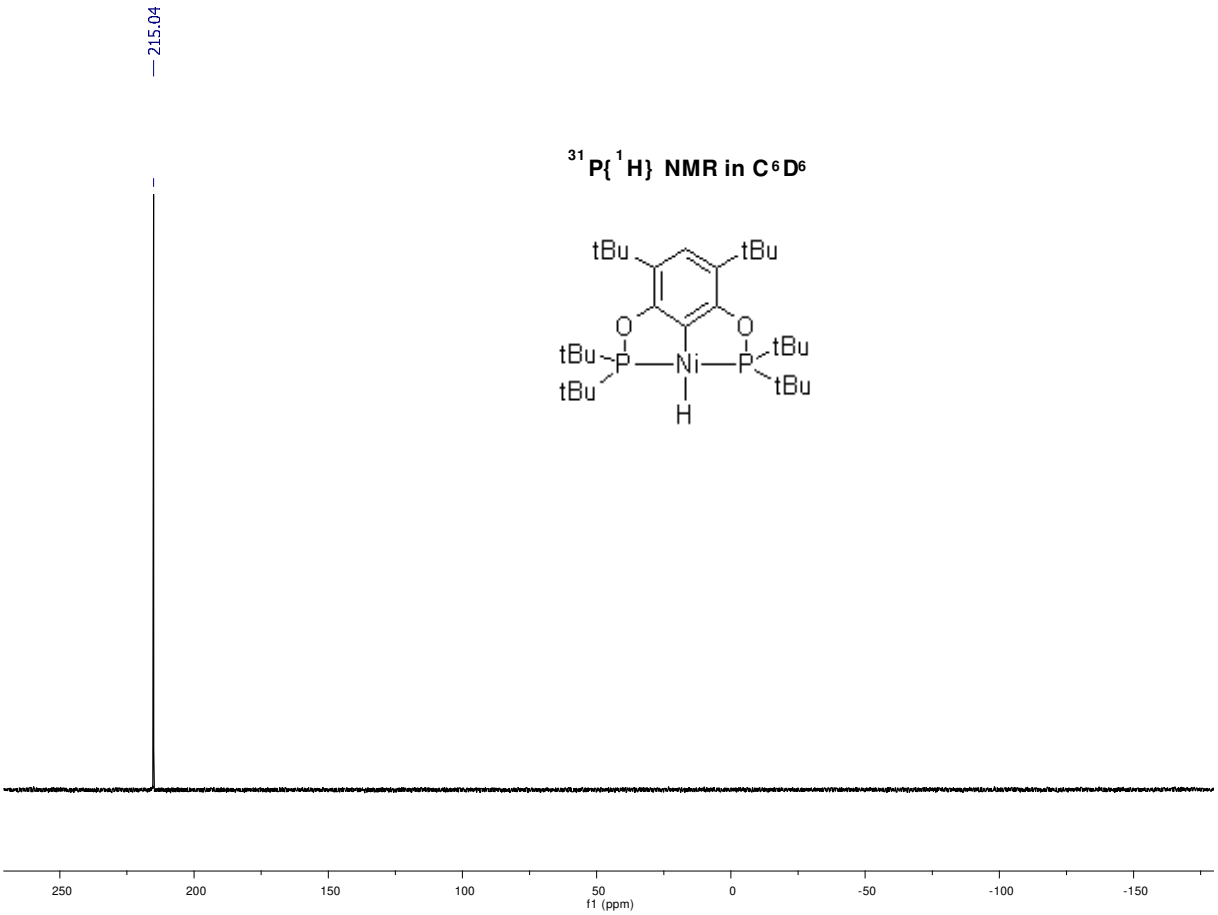

169.03
168.94
168.85
138.72
128.30
127.82
105.78
37.94
37.85
37.77
28.57
21.94
13C{ 1H} NMR in C6D6
Me
O
O
tBu
tBu
P
Ni
P
tBu
tBu
H
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

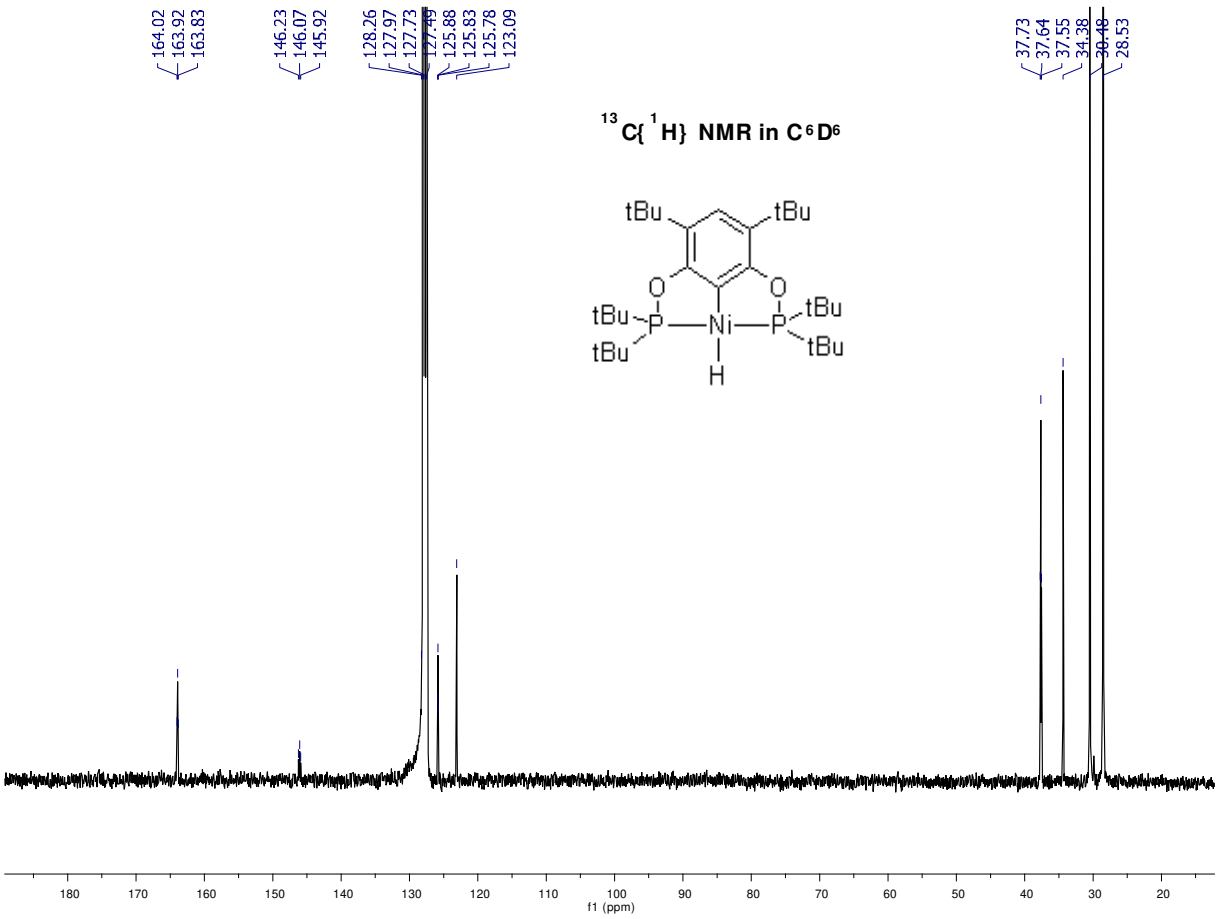


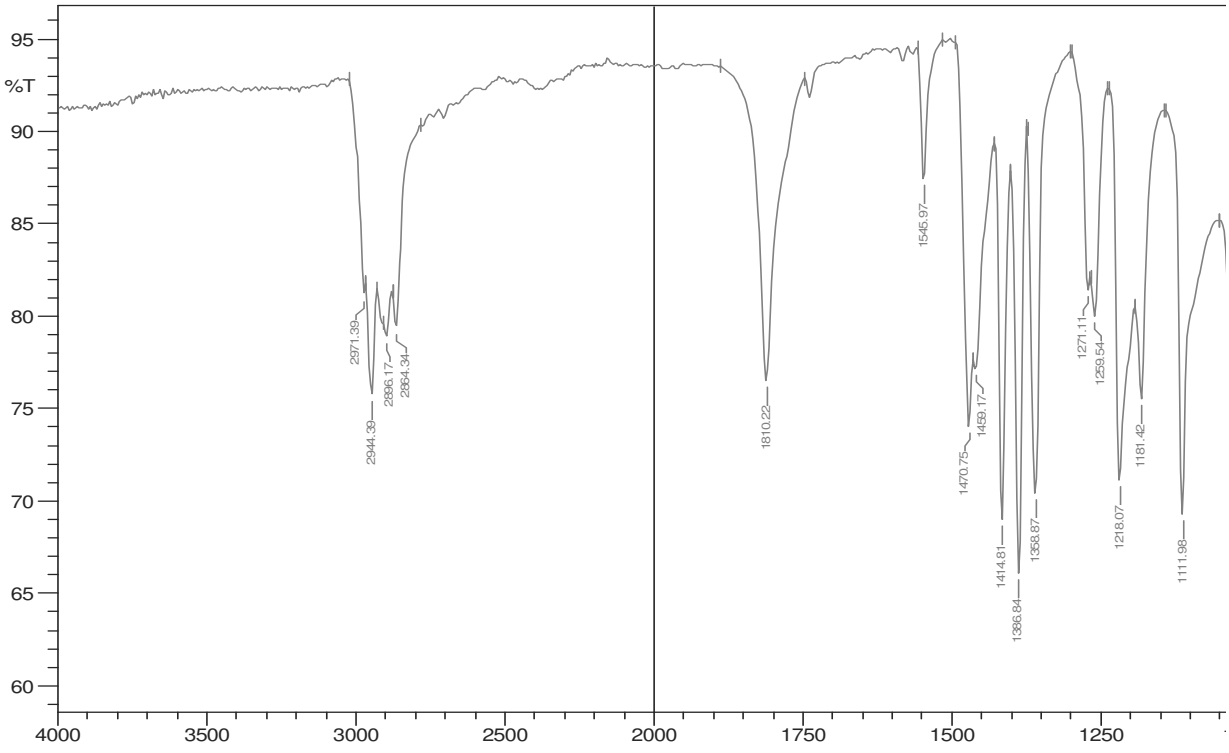


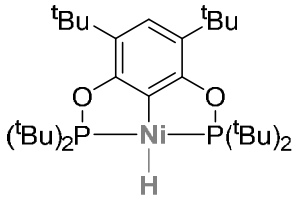
Solid state IR of **3c**


7.32
7.16
1.60
1.38
1.36
1.34
0.30
-7.76
-7.90
-8.03
1H NMR in C6D6
tBu
tBu
O
O
tBu
P
Ni
P
tBu
tBu
tBu
H
1.00
18.87
38.01
0.95
f1 (ppm)

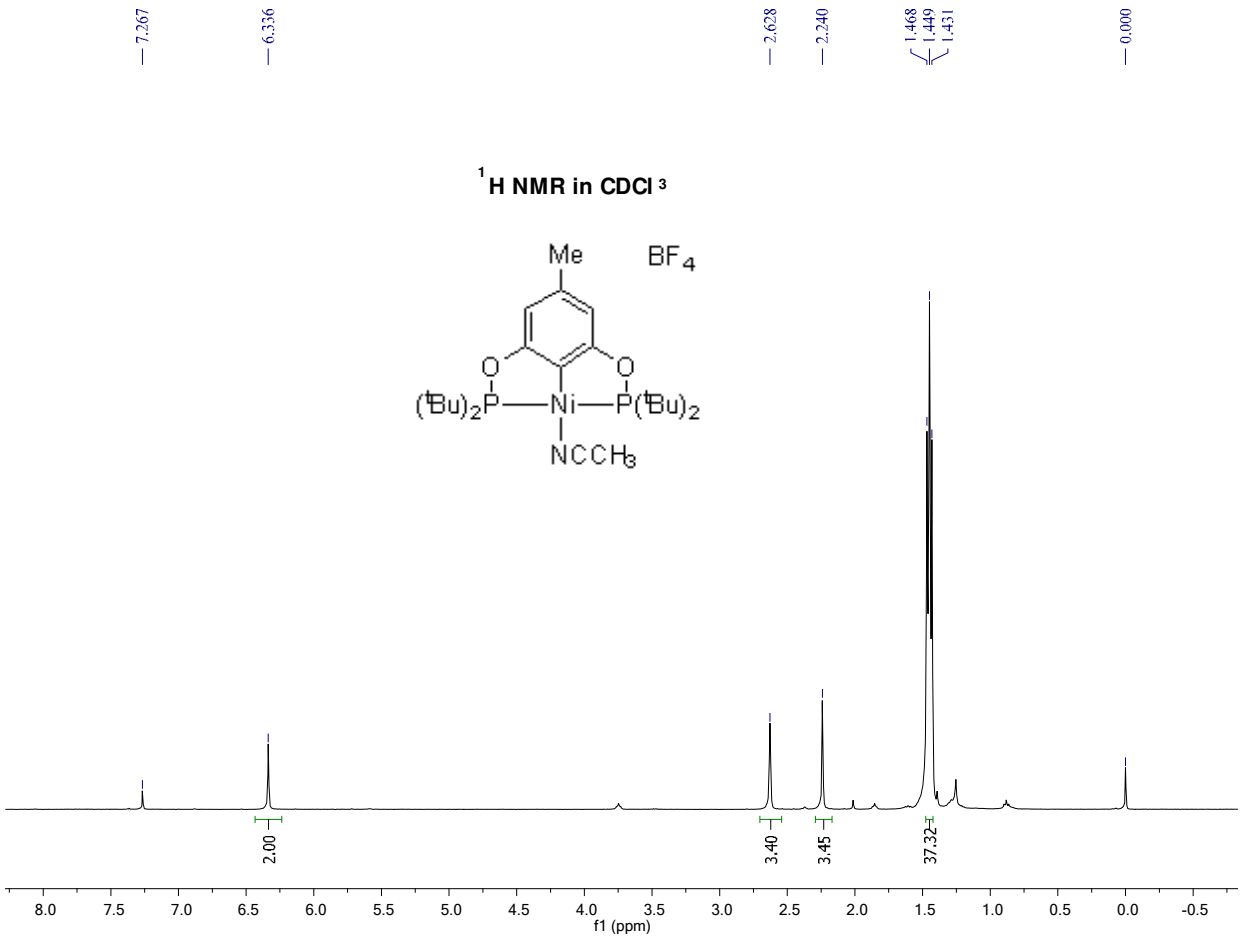

215.04
31P{1H} NMR in C6D6
tBu
tBu
O
O
tBu
P
Ni
P
tBu
tBu
tBu
H
250
200
150
100
50
0
-50
-100
-150
f1 (ppm)


164.02
163.92
163.83
146.23
146.07
145.92
128.26
127.97
127.73
127.49
125.88
125.83
125.78
123.09
37.73
37.64
37.55
34.38
30.48
28.53
13C{1H} NMR in C6D6
tBu
tBu
O
O
tBu
P
Ni
P
tBu
tBu
tBu
H
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
f1 (ppm)

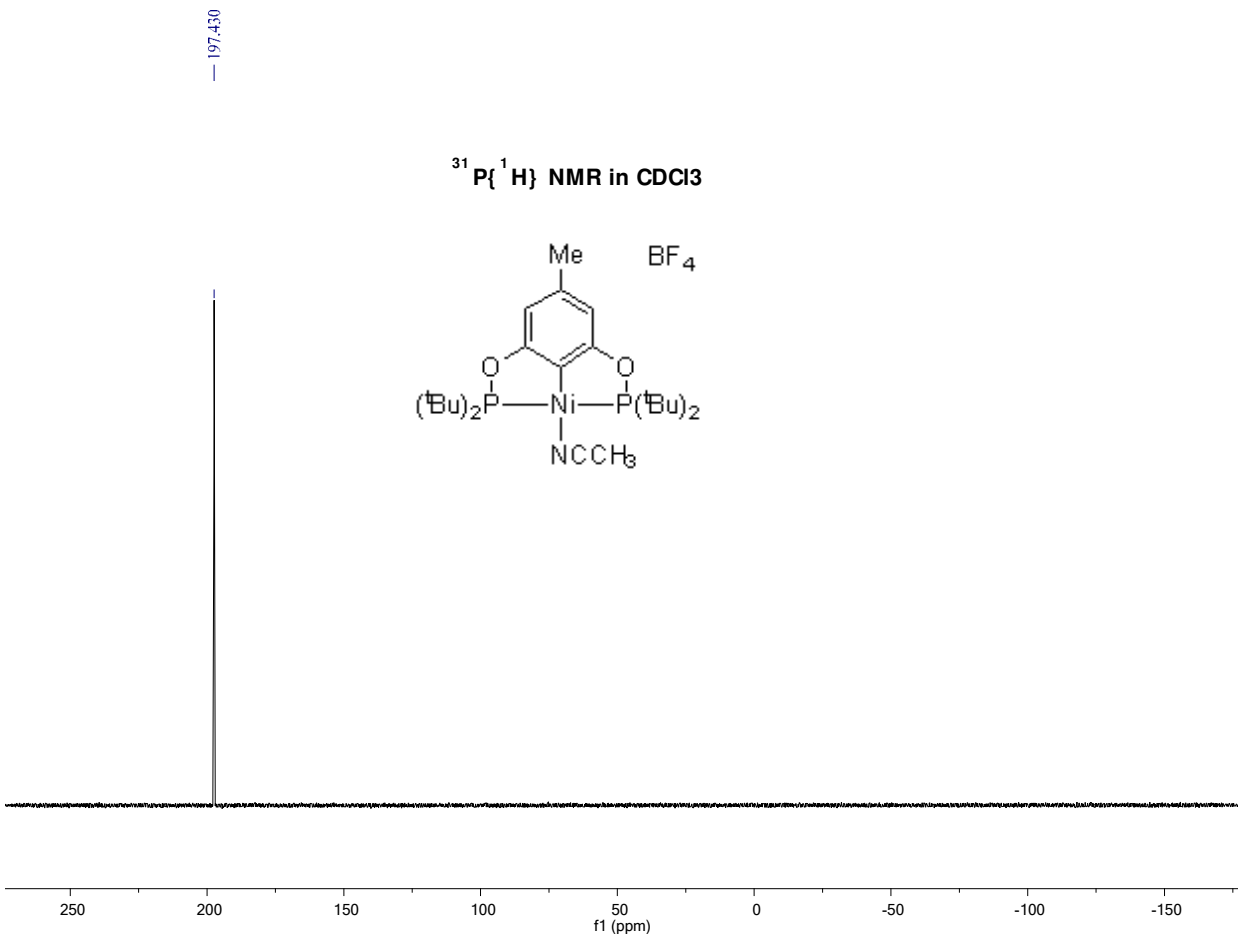


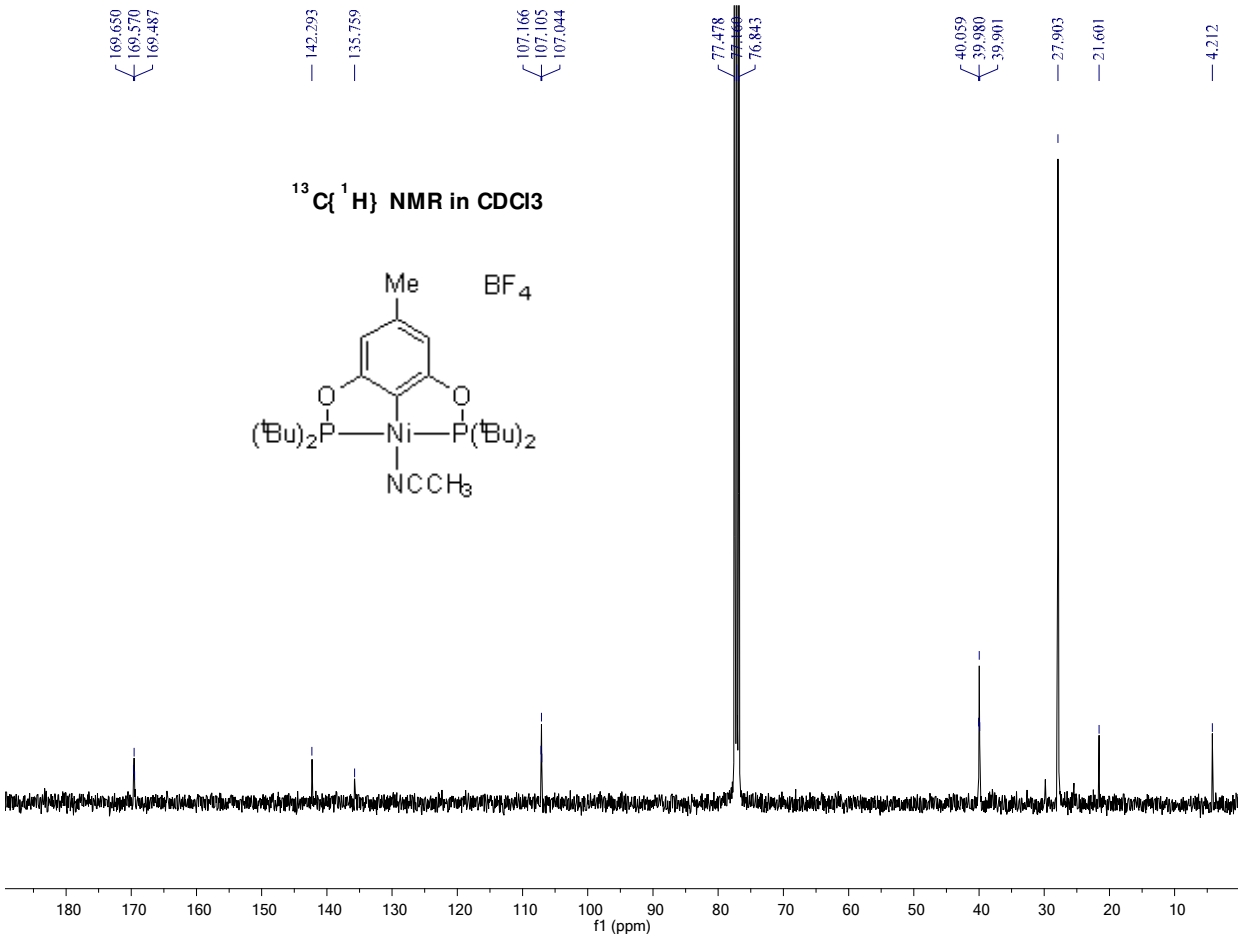


Solid state IR of **3d**


7.267
6.336
2.628
2.240
1.468
1.449
1.431
0.000
1H NMR in CDCl3
Me
BF4
O
O
(tBu)2P—Ni—P(tBu)2
NCCH3
2.00
3.40
3.45
37.32
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
0.0
-0.5
f1 (ppm)

$^{31}P\{^{1}H\}$ NMR in CDCl3

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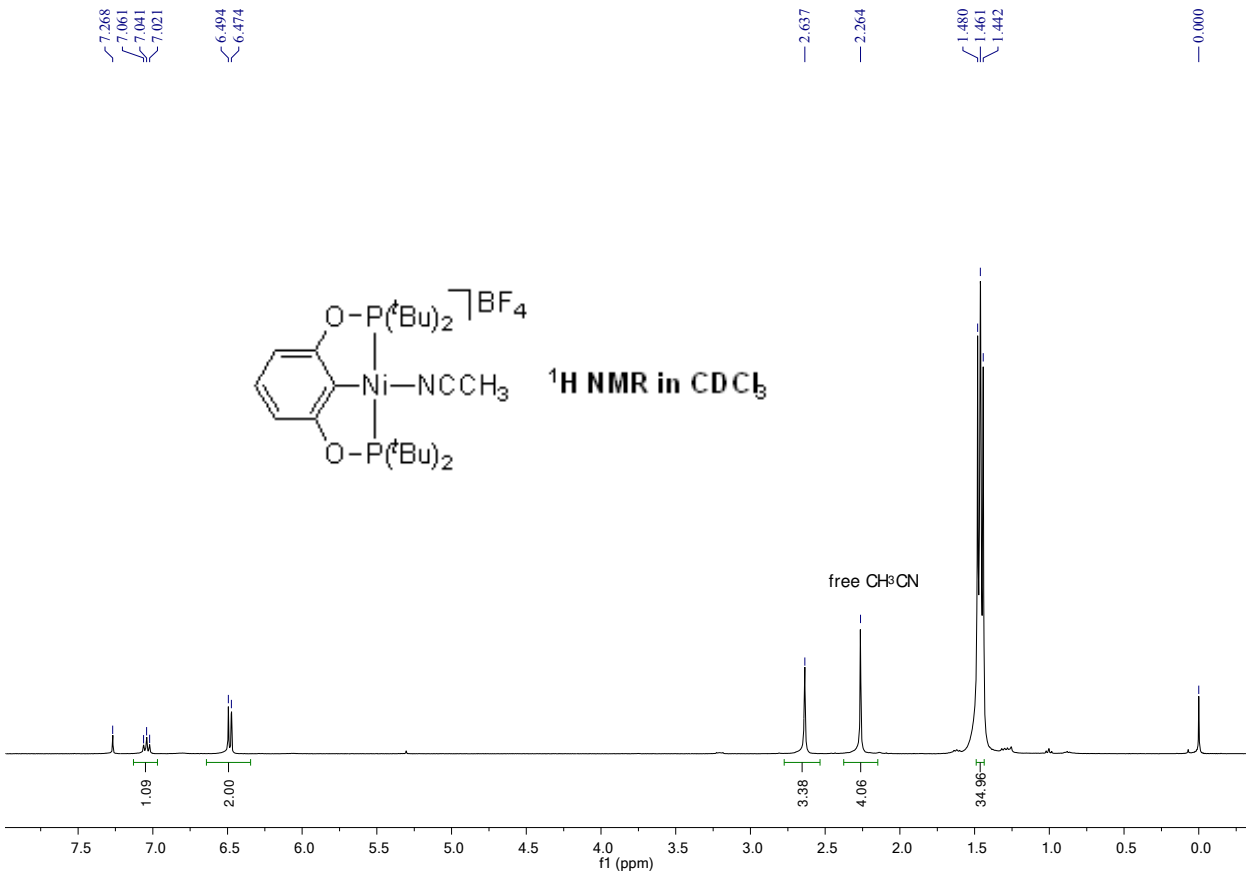
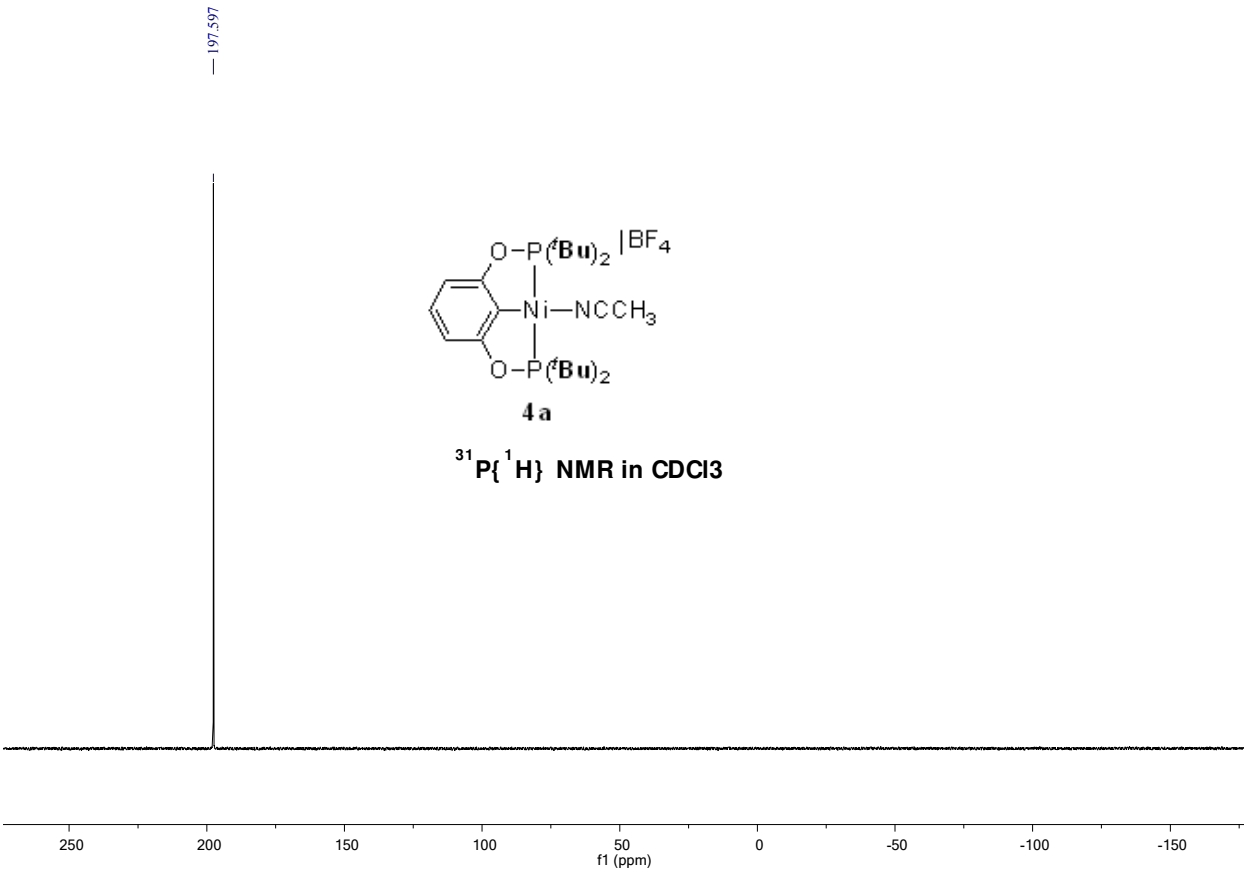
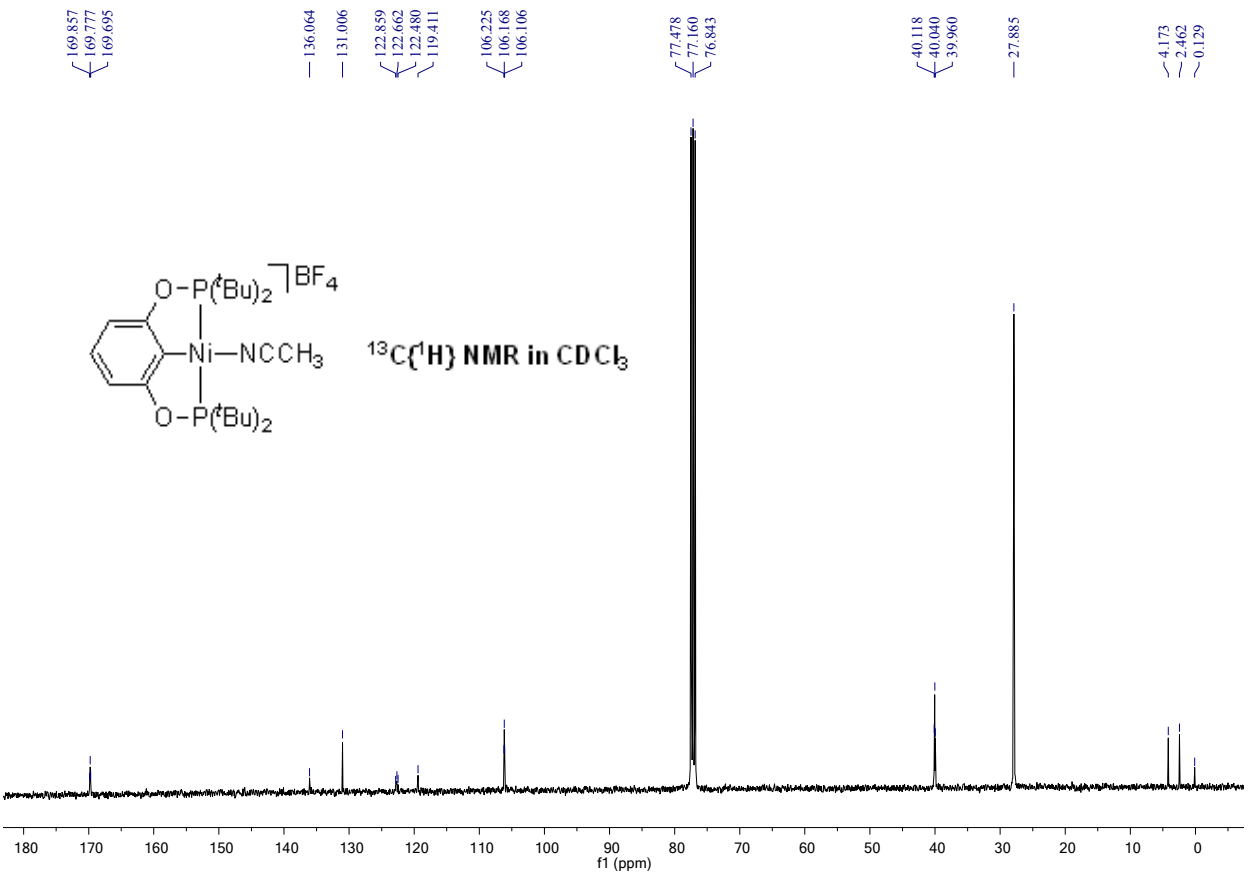
169.570
169.487
142.293
135.759
107.166
107.105
107.044
77.478
76.843
40.059
39.980
39.901
27.903
21.601
4.212
13C{ 1H} NMR in CDCl3
Me
BF4
O
O
(tBu)2P—Ni—P(tBu)2
NCCH3
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)


7.268
7.061
7.041
7.021
6.494
6.474
2.637
2.264
1.480
1.461
1.442
0.000
O-P(tBu)2
BF4
Ni—NCCH3
O-P(tBu)2
1H NMR in CDCl3
free CH3CN
1.09
2.00
3.38
4.06
34.96
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
0.0
f1 (ppm)



4a

$^{31}P\{^{1}H\}$ NMR in CDCl3


169.857
169.777
169.695
136.064
131.006
122.859
122.662
122.480
119.411
106.225
106.168
106.106
77.478
77.160
76.843
40.118
40.040
39.960
27.885
4.173
2.462
0.129
O-P(tBu)2
BF4
Ni—NCCH3
O-P(tBu)2
13C{1H} NMR in CDCl3
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

1. Quantification of H_2 produced by one-electron oxidation of 3a by NMR

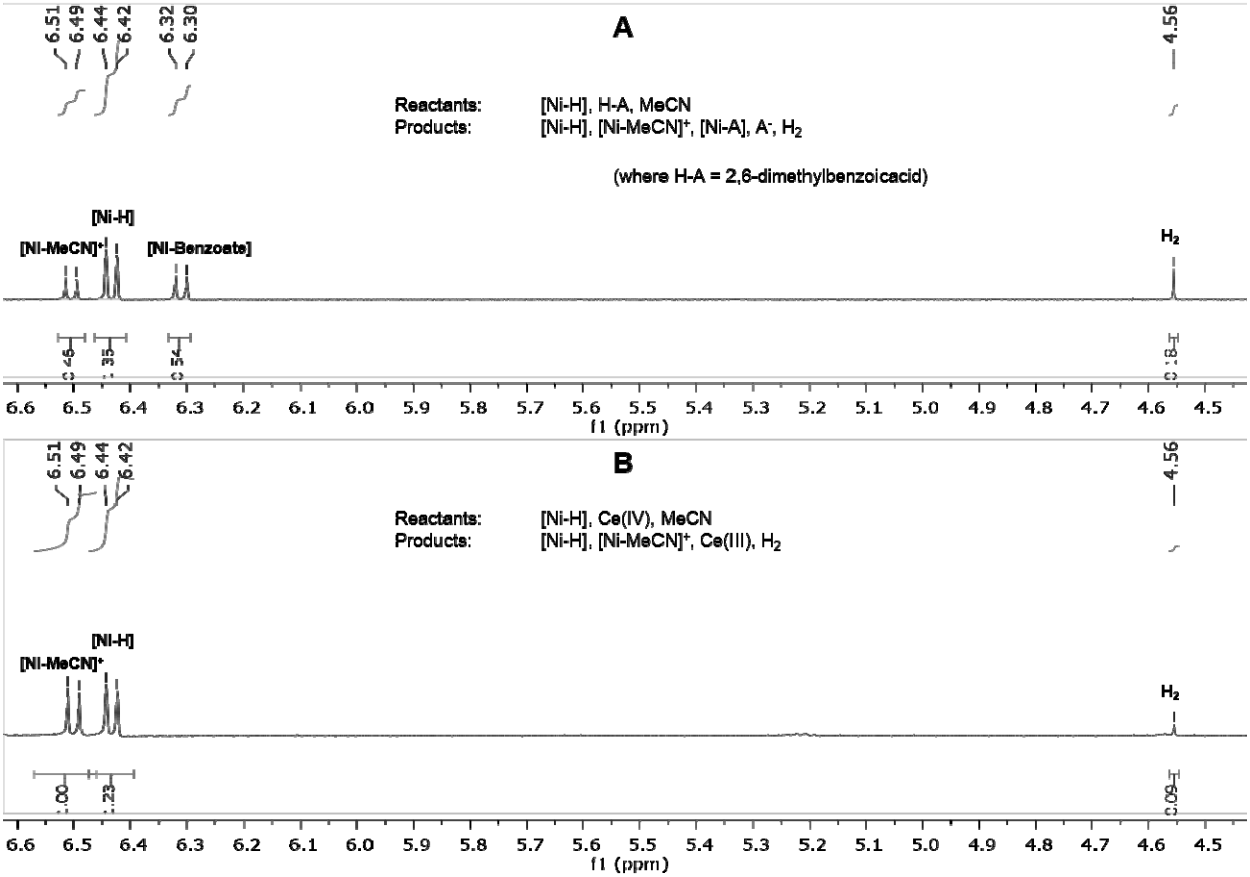


Fig S1. Reaction of **3a** (0.011 mmol) with (A) 2,6-dimethylbenzoicacid, at 42.5% conversion, and (B) $Ce(NBu_4)_2(NO_3)_6$ at 44.8% conversion, based on the ratio of integrations of the doublets corresponding to the reactants and products as shown.

2. High Scan-rate cyclic voltammograms of 3a.

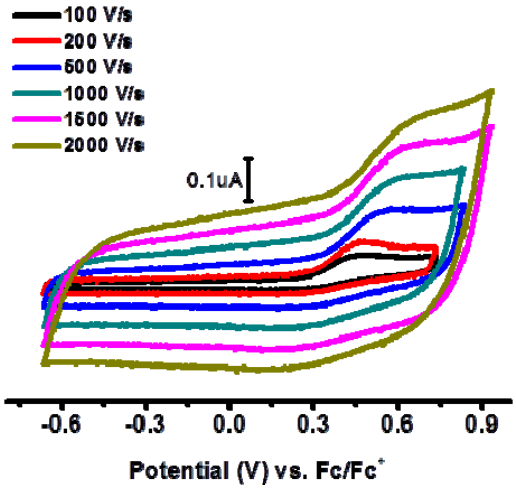


Fig S2. Cyclic voltammograms of a 4 mM solution of **3a** in MeCN-THF (4:1). Experimental Parameters: Carbon Fibre (11 μm dia) working electrode (MF2007 from BASi inc.), $Ag/AgNO_3$(0.01 M), $TBABF_4$(0.1 M)/MeCN reference electrode, Pt wire counter electrode; N_2 atmosphere.

3. Concentration dependent cyclic voltammograms of 3a.

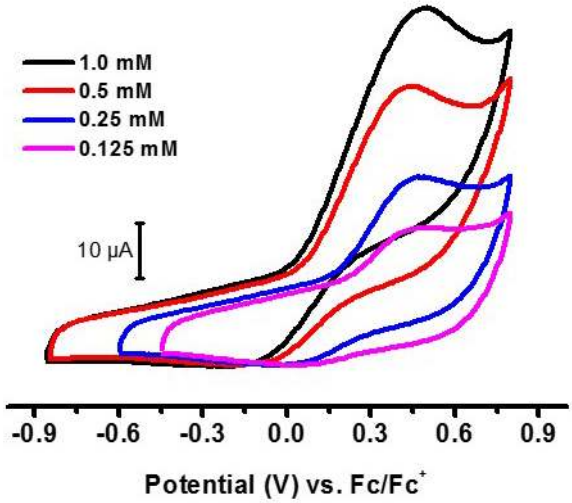


Fig S3. Cyclic voltammograms of a 4 mM solution of **3a** in MeCN-THF (4:1). Experimental Parameters: Glassy carbon (3 mm dia) working electrode, $Ag/AgNO_3$(0.01 M), $TBABF_4$(0.1 M)/MeCN reference electrode, Pt wire counter electrode, N_2 atmosphere.

4. Electrochemical Simulation of 2a with DigiElch™

4a. Sensitivity Analysis at 4 V/s

Fixed Parameters: C_{dl}=9 μF, Area of electrode A = 0.070685 cm^2; Radius of electrode r_0 = 1.5 mm; $E_{1/2}$ = 0.72 V vs. Fc/Fc^+; α = 0.5; $D_{Ox} = D_{Red}$ = 1x10^{-5} cm^2/s; [**2a**] = 4 mM. Solution conductivity ρ = 119 Ω cm(Kadish, Ding, & Malinski, 1984); diffusion model: "planar 1D semi-infinite".

Varied Parameters: k_s, R_u

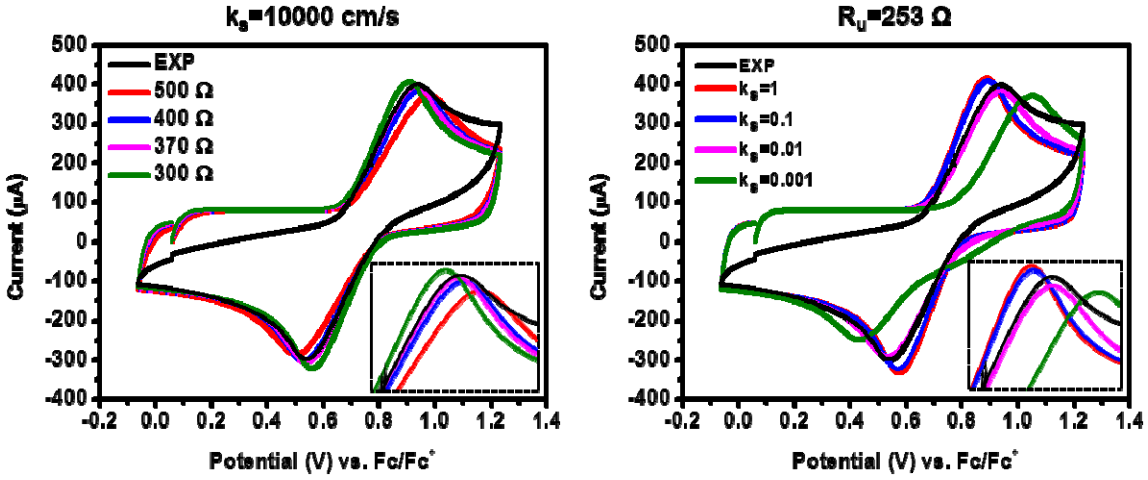


Fig S4a. Experimental (in black) and simulated (in color) voltammograms of **2a** at 4 V/s. The lower limit of R_u i.e $R_{u(min)}$ at a distance of L=r_0 was obtained from R = ρL/A = 253 Ω.

Best fits to the experimental data were obtained for R_u=370 Ω at k_s=10000 cm/s (infinite) and at k_s=0.01 cm/s at R_u=$R_{u(min)}$. Insets show magnification of the data near E_{pa}.

4b. Sensitivity Analysis at 400 V/s

Fixed Parameters: C_{dl}=100 pF, Area of electrode A = $9.5x10^{-7}$ cm^2; Radius of electrode r_0 = 5.5 µm; $E_{1/2}$ = 0.72 V vs. Fc/Fc^+; α = 0.5; D_{Ox} = D_{Red} = $1x10^{-5}$ cm^2/s; [**2a**] = 4 mM. Solution conductivity ρ = 119 Ω cm

Varied Parameters: k_s, R_u

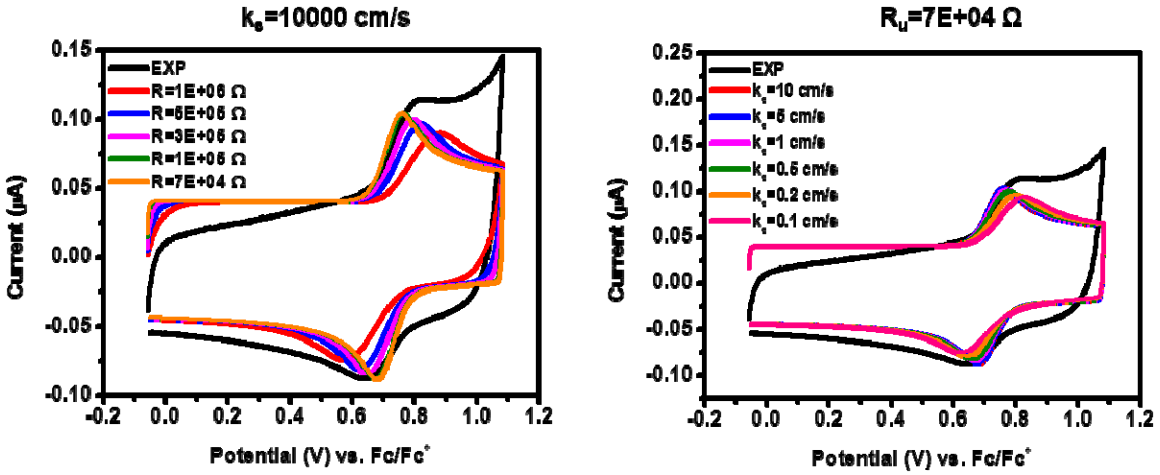


Fig S4b. Experimental (in black) and simulated (in color) voltammograms of **2a** at 400 V/s. The lower limit of R_u i.e $R_{u(min)}$ at a distance of L=r_0 was obtained from R = (ρ x L)/A.

Best fits to the experimental data was obtained for R_u=$3x10^5$ Ω at k_s=10000 cm/s (infinite) and k_s=0.02 cm/s at R_u=$R_{u(min)}$=$7x10^4$ Ω.

5. Electrochemical Simulation of 3a with DigiElch™

5a. Sensitivity Analysis at 4 V/s to obtain limits on k_s and R_u

Fixed Parameters: C_{dl}=9 μF, Area of electrode A = 0.070685 cm^2; Radius of electrode r_0 = 1.5 mm; K = 5.66 x 10^{53}; α = 0.5; $D_{Ox} = D_{Red}$ = 1x10^{-5} cm^2/s; [**3a**] = 4 mM. Solution conductivity, ρ = 119 Ω cm; k_f = 1x10^6 $M^{-1}s^{-1}$; Diffusion model: planar 1D semi-infinite.

Varied Parameters: k_s (cm/s), R_{unc} (Ω)

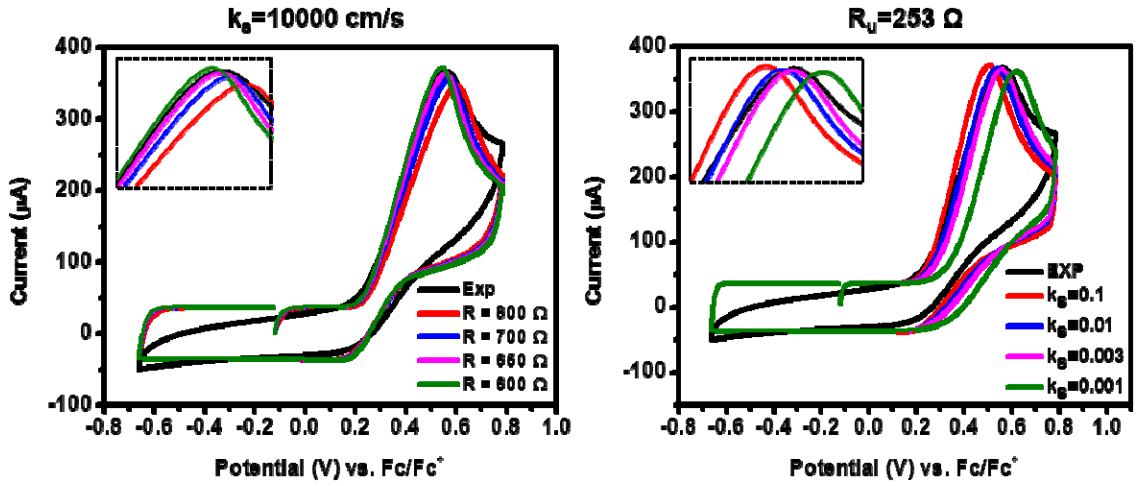


Fig S5a. Experimental (in black) and simulated (in color) voltammograms of **3a** at 4 V/s. The lower limit of R_u i.e $R_{u(min)}$ at a distance of L=r_0 was obtained from R = (ρ x L)/A. $R_{u(min)}$ =253 Ω Best fits to the experimental data was obtained for R_u=650 Ω at k_s=10000 cm/s (infinite) and k_s=0.003 cm/s at R_u=$R_{u(min)}$.

5b. Sensitivity Analysis at 4 V/s to obtain a lower limit on k_f and E°

Fixed Parameters: C_{dl}=9 μF, Area of electrode A = 0.070685 cm^2; Radius of electrode r_0 = 1.5 mm; K = 5.66 x 10^{53}; α = 0.5; $D_{Ox} = D_{Red}$ = 1x10^{-5} cm^2/s; [**3a**] = 4 mM. Solution conductivity, ρ = 119 Ω cm; Diffusion model: planar 1D semi-infinite.

Varied Parameters: k_f, E°

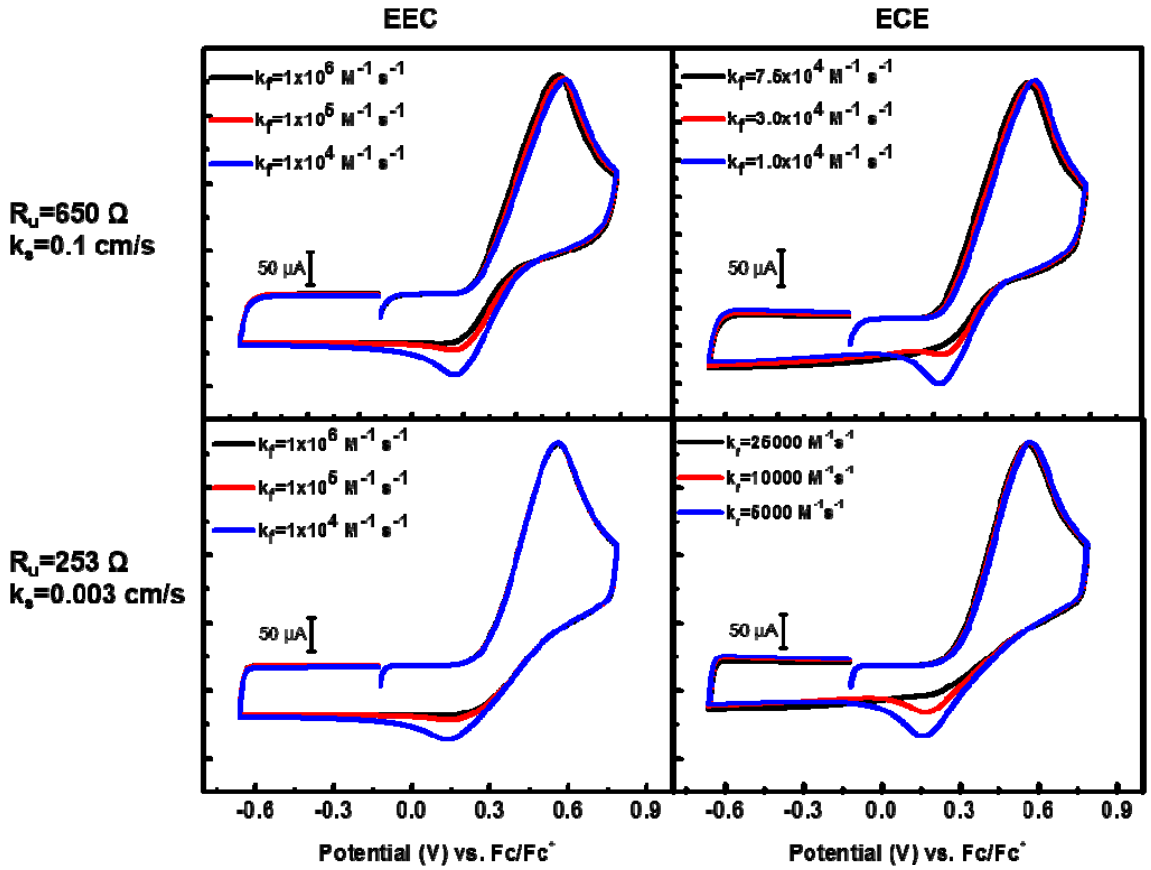


Fig S5b. Simulated voltammograms of **3a** at 4 V/s at the two limiting k_s, R_u values for both mechanisms, EEC and ECE.

6. Cyclic voltammograms of 2b-d.

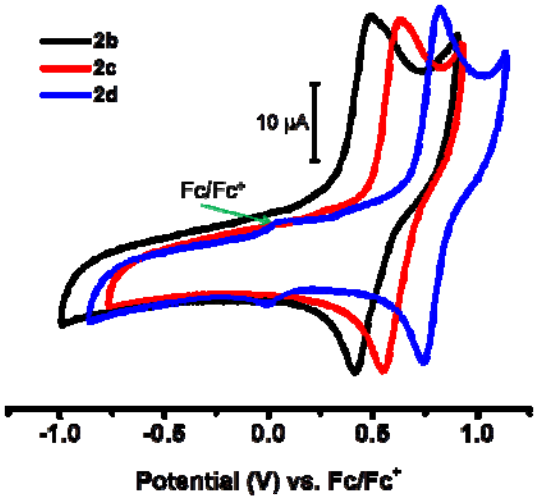


Fig S6. Cyclic voltammograms of a 2 mM solution of **2b-d** in MeCN. Experimental Parameters: Glassy carbon (3 mm dia) working electrode, $Ag/AgNO_3$ (0.01 M), $TBABF_4$(0.1 M)/MeCN reference electrode, Pt wire counter electrode, N_2 atmosphere; Scan-rate: 100 mV/s.

7. Cyclic voltammograms of 4a.

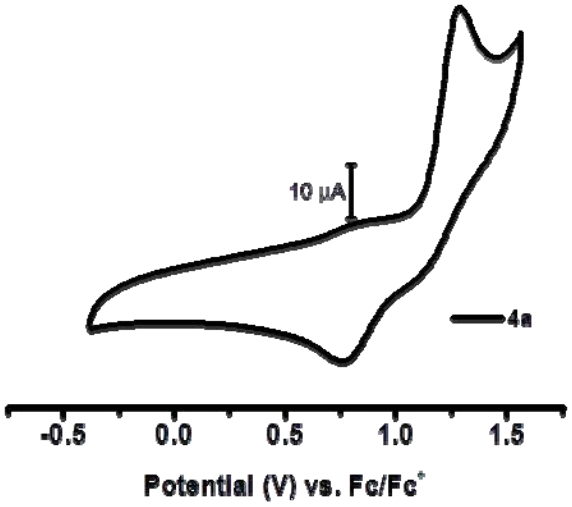


Fig S7. Cyclic voltammograms of a 4 mM solution of **4a** in MeCN. Experimental Parameters: Glassy carbon (3 mm dia) working electrode, $Ag/AgNO_3$(0.01 M), $TBABF_4$(0.1 M)/MeCN reference electrode, Pt wire counter electrode, N_2 atmosphere. Scan-rate: 100 mV/s.

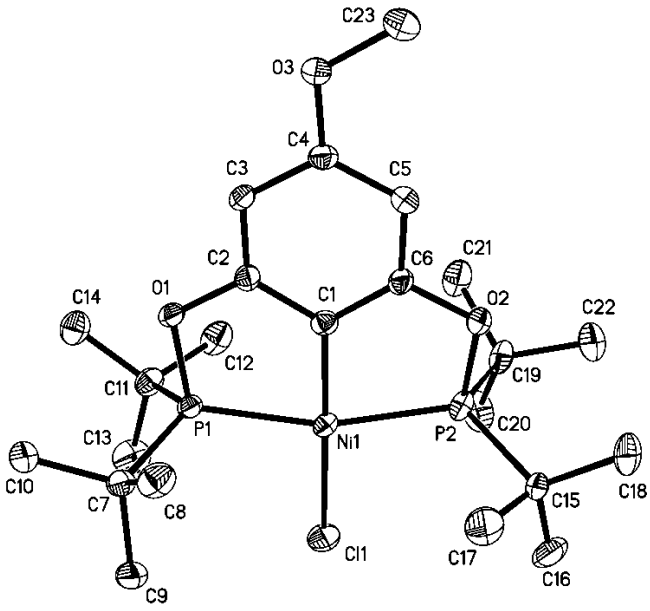
CRYSTAL STRUCTURE REPORT

$C_{23}H_{41}ClNiO_3P_2$ (**2b**)

Report prepared for:

Dr. S. Chakraborty, Prof. W. D. Jones

September 24, 2013



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Data collection

A crystal (0.20 x 0.20 x 0.12 mm^3) was placed onto the tip of a 0.1 mm diameter glass capillary tube and mounted on a Bruker SMART APEX II CCD platform diffractometer for a data collection at 100.0(5) K.[1] A preliminary set of cell constants and an orientation matrix were calculated from reflections harvested from three orthogonal wedges of reciprocal space. The full data collection was carried out using MoKα radiation (graphite monochromator) with a frame time of 60 seconds and a detector distance of 4.01 cm. A randomly oriented region of reciprocal space was surveyed: four major sections of frames were collected with 0.50º steps in ω at four different ϕ settings and a detector position of -38º in 2θ. The intensity data were corrected for absorption.[2] Final cell constants were calculated from the xyz centroids of 4051 strong reflections from the actual data collection after integration.[3] See Table 1 for additional crystal and refinement information.

Structure solution and refinement

The structure was solved using SIR2011[4] and refined using SHELXL-2013.[5] The space group $P2_1/c$ was determined based on systematic absences. A direct-methods solution was calculated which provided most non-hydrogen atoms from the E-map. Full-matrix least squares / difference Fourier cycles were performed which located the remaining non-hydrogen atoms. All non-hydrogen atoms were refined with anisotropic displacement parameters. All hydrogen atoms were placed in ideal positions and refined as riding atoms with relative isotropic displacement parameters. The final full matrix least squares refinement converged to $R1 = 0.0498$ (F^2, $I > 2\sigma(I)$) and $wR2 = 0.1087$ (F^2, all data).

Structure description

The structure is the one suggested. The asymmetric unit contains one molecule in a general position. Two *tert*-butyl groups (bonded to P2) are modeled as disordered over two positions (63:37).

Unless noted otherwise all structural diagrams containing thermal displacement ellipsoids are drawn at the 50 % probability level.

Data collection, structure solution, and structure refinement were conducted at the X-ray Crystallographic Facility, B51 Hutchison Hall, Department of Chemistry, University of Rochester. All publications arising from this report MUST either 1) include William W. Brennessel as a coauthor or 2) acknowledge William W. Brennessel and the X-ray Crystallographic Facility of the Department of Chemistry at the University of Rochester.

[1] *APEX2*, version 2013.2-0; Bruker AXS: Madison, WI, 2013.

[2] Sheldrick, G. M. *SADABS*, version 2012/1; University of Göttingen: Göttingen, Germany, 2012.

[3] *SAINT*, version 8.27B; Bruker AXS: Madison, WI, 2013.

[4] Burla, M. C.; Caliandro, R.; Camalli, M.; Carrozzini, B.; Cascarano, G. L.; Giacovazzo, C.; Mallamo, M.; Mazzone, A.; Polidori, G.; Spagna, R. *SIR2011: a new package for crystal structure determination and refinement*, version 1.0; Istituto di Cristallografia: Bari, Italy, 2012.

[5] Sheldrick, G. M. *SHELXL-2013/4*; University of Göttingen: Göttingen, Germany, 2013.

Some equations of interest:

$$R_{int} = \Sigma\, |F_o^2 - < F_o^2 >| \,/\, \Sigma |F_o{}^2|$$

$$R1 = \Sigma ||F_o| - |F_c|| \,/\, \Sigma |F_o|$$

$$wR2 = [\Sigma [w(F_o^2 - F_c{}^2)^2] \,/\, \Sigma\, [w(F_o^2)^2]]^{1/2}$$

where $w = 1 \,/\, [\sigma^2 (F_o^2) + (aP)^2 + bP]$ and

$$P = 1/3 \max (0, F_o^2) + 2/3\, F_c^2$$

$$\text{GOF} = S = [\Sigma [w(F_o^2 - F_c{}^2)^2] \,/\, (m-n)]^{1/2}$$

where m = number of reflections and n = number of parameters

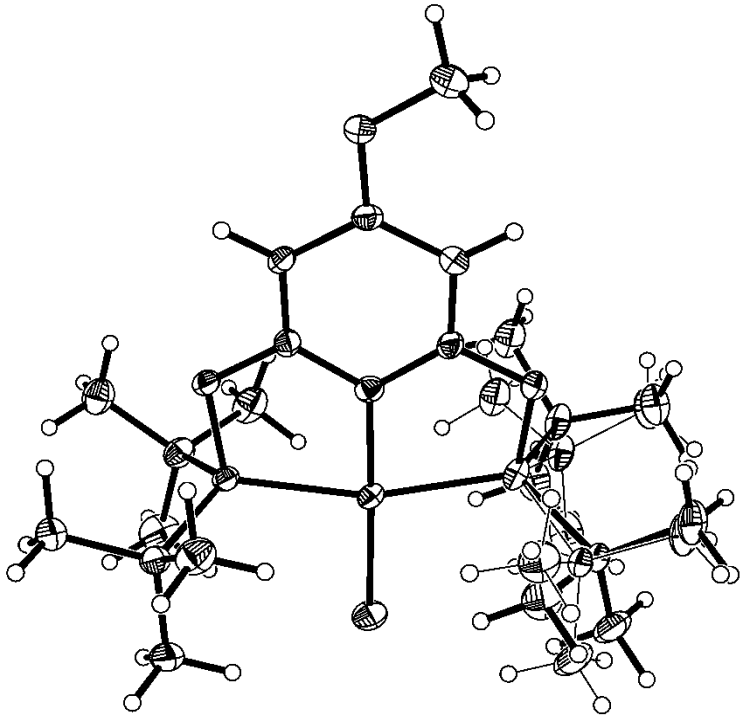


Table 1. Crystal data and structure refinement for **2b**.

Empirical formula	$C_{23}H_{41}ClNiO_3P_2$	
Formula weight	521.66	
Temperature	100.0(5) K	
Wavelength	0.71073 Å	
Crystal system	monoclinic	
Space group	$P2_1/c$	
Unit cell dimensions	a = 14.485(4) Å	α = 90°
	b = 7.985(2) Å	β = 106.418(6)°
	c = 23.646(7) Å	γ = 90°
Volume	2623.3(13) Å^3	
Z	4	
Density (calculated)	1.321 Mg/m^3	
Absorption coefficient	0.984 mm^{-1}	
F(000)	1112	
Crystal color, morphology	yellow, needle	
Crystal size	0.20 x 0.20 x 0.12 mm^3	
Theta range for data collection	1.796 to 32.181°	
Index ranges	$-21 \le h \le 21$, $-11 \le k \le 11$, $-35 \le l \le 35$	
Reflections collected	45634	
Independent reflections	9223 [R(int) = 0.0967]	
Observed reflections	5960	
Completeness to theta = 32.030°	100.0%	
Absorption correction	Multi-scan	
Max. and min. transmission	0.7463 and 0.6714	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	9223 / 24 / 315	
Goodness-of-fit on F^2	1.024	
Final R indices [I>2sigma(I)]	R1 = 0.0498, wR2 = 0.0939	
R indices (all data)	R1 = 0.0934, wR2 = 0.1087	
Largest diff. peak and hole	0.711 and -0.586 e.Å^{-3}	

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

	x	y	z	U_{eq}
Ni1	7320(1)	950(1)	8684(1)	17(1)
Cl1	7063(1)	-430(1)	7847(1)	28(1)
P1	8700(1)	2023(1)	8688(1)	16(1)
P2	6056(1)	150(1)	8946(1)	19(1)
O1	9067(1)	3100(2)	9307(1)	19(1)
O2	6079(1)	1243(2)	9541(1)	22(1)
C1	7556(2)	2154(3)	9392(1)	17(1)
C2	8390(2)	3104(3)	9623(1)	16(1)
C3	8578(2)	4025(3)	10134(1)	17(1)
C4	7899(2)	3995(3)	10450(1)	18(1)
C5	7059(2)	3071(3)	10255(1)	21(1)
C6	6916(2)	2184(3)	9733(1)	18(1)
C7	8696(2)	3692(3)	8141(1)	21(1)
C8	7989(2)	5008(3)	8249(1)	29(1)
C9	8315(2)	3036(3)	7509(1)	27(1)
C10	9680(2)	4516(3)	8234(1)	28(1)
C11	9696(2)	489(3)	8789(1)	22(1)
C12	9449(2)	-901(3)	9169(1)	31(1)
C13	9726(2)	-247(4)	8199(1)	35(1)
C14	10682(2)	1220(4)	9112(1)	28(1)
C15	4820(4)	614(9)	8486(3)	26(1)
C16	4553(3)	-755(8)	8009(2)	42(1)
C17	4951(5)	2315(9)	8219(2)	48(2)
C18	4031(12)	769(18)	8796(8)	30(2)
C19	6181(4)	-1957(5)	9315(2)	22(1)
C20	6382(4)	-3255(6)	8880(2)	35(1)
C21	7070(3)	-1890(6)	9850(2)	31(1)
C22	5316(12)	-2500(40)	9513(8)	31(1)
C15'	4915(7)	989(16)	8448(5)	26(1)
C16'	4658(6)	415(13)	7804(3)	42(1)
C17'	5046(9)	2904(15)	8465(4)	48(2)

C18'	4090(20)	560(30)	8713(14)	30(2)
C19'	6031(7)	-2112(8)	9101(3)	22(1)
C20'	5755(6)	-3136(10)	8533(4)	35(1)
C21'	7059(5)	-2451(10)	9433(3)	31(1)
C22'	5370(20)	-2490(70)	9481(14)	31(1)
O3	8116(1)	4930(2)	10952(1)	23(1)
C23	7431(2)	4986(4)	11281(1)	29(1)

Table 3. Bond lengths [Å] and angles [°] for **2b**.

Ni(1)-C(1)	1.876(2)	C(10)-H(10C)	0.9800
Ni(1)-P(1)	2.1740(9)	C(11)-C(13)	1.527(4)
Ni(1)-P(2)	2.1863(9)	C(11)-C(14)	1.531(3)
Ni(1)-Cl(1)	2.2043(8)	C(11)-C(12)	1.533(4)
P(1)-O(1)	1.6523(17)	C(12)-H(12A)	0.9800
P(1)-C(7)	1.855(2)	C(12)-H(12B)	0.9800
P(1)-C(11)	1.856(2)	C(12)-H(12C)	0.9800
P(2)-O(2)	1.6469(18)	C(13)-H(13A)	0.9800
P(2)-C(19')	1.845(6)	C(13)-H(13B)	0.9800
P(2)-C(15)	1.851(4)	C(13)-H(13C)	0.9800
P(2)-C(15')	1.863(7)	C(14)-H(14A)	0.9800
P(2)-C(19)	1.879(4)	C(14)-H(14B)	0.9800
O(1)-C(2)	1.390(3)	C(14)-H(14C)	0.9800
O(2)-C(6)	1.390(3)	C(15)-C(18)	1.526(8)
C(1)-C(6)	1.390(3)	C(15)-C(17)	1.532(7)
C(1)-C(2)	1.400(3)	C(15)-C(16)	1.540(8)
C(2)-C(3)	1.373(3)	C(16)-H(16A)	0.9800
C(3)-C(4)	1.394(3)	C(16)-H(16B)	0.9800
C(3)-H(3)	0.9500	C(16)-H(16C)	0.9800
C(4)-O(3)	1.361(3)	C(17)-H(17A)	0.9800
C(4)-C(5)	1.387(3)	C(17)-H(17B)	0.9800
C(5)-C(6)	1.386(3)	C(17)-H(17C)	0.9800
C(5)-H(5)	0.9500	C(18)-H(18A)	0.9800
C(7)-C(10)	1.528(3)	C(18)-H(18B)	0.9800
C(7)-C(9)	1.533(3)	C(18)-H(18C)	0.9800
C(7)-C(8)	1.538(4)	C(19)-C(22)	1.521(9)
C(8)-H(8A)	0.9800	C(19)-C(21)	1.531(6)
C(8)-H(8B)	0.9800	C(19)-C(20)	1.544(6)
C(8)-H(8C)	0.9800	C(20)-H(20A)	0.9800
C(9)-H(9A)	0.9800	C(20)-H(20B)	0.9800
C(9)-H(9B)	0.9800	C(20)-H(20C)	0.9800
C(9)-H(9C)	0.9800	C(21)-H(21A)	0.9800
C(10)-H(10A)	0.9800	C(21)-H(21B)	0.9800
C(10)-H(10B)	0.9800	C(21)-H(21C)	0.9800

C(22)-H(22A)	0.9800	P(2)-Ni(1)-Cl(1)	99.59(3)
C(22)-H(22B)	0.9800	O(1)-P(1)-C(7)	100.41(10)
C(22)-H(22C)	0.9800	O(1)-P(1)-C(11)	100.68(10)
C(15')-C(16')	1.532(12)	C(7)-P(1)-C(11)	114.44(11)
C(15')-C(18')	1.535(13)	O(1)-P(1)-Ni(1)	105.82(6)
C(15')-C(17')	1.540(13)	C(7)-P(1)-Ni(1)	117.34(8)
C(16')-H(16D)	0.9800	C(11)-P(1)-Ni(1)	114.90(9)
C(16')-H(16E)	0.9800	O(2)-P(2)-C(19')	110.2(3)
C(16')-H(16F)	0.9800	O(2)-P(2)-C(15)	100.3(2)
C(17')-H(17D)	0.9800	O(2)-P(2)-C(15')	98.6(4)
C(17')-H(17E)	0.9800	C(19')-P(2)-C(15')	114.4(5)
C(17')-H(17F)	0.9800	O(2)-P(2)-C(19)	95.89(16)
C(18')-H(18D)	0.9800	C(15)-P(2)-C(19)	113.7(3)
C(18')-H(18E)	0.9800	O(2)-P(2)-Ni(1)	105.73(6)
C(18')-H(18F)	0.9800	C(19')-P(2)-Ni(1)	114.0(3)
C(19')-C(21')	1.499(10)	C(15)-P(2)-Ni(1)	121.6(2)
C(19')-C(22')	1.522(13)	C(15')-P(2)-Ni(1)	112.3(4)
C(19')-C(20')	1.526(9)	C(19)-P(2)-Ni(1)	114.48(17)
C(20')-H(20D)	0.9800	C(2)-O(1)-P(1)	112.31(14)
C(20')-H(20E)	0.9800	C(6)-O(2)-P(2)	111.73(14)
C(20')-H(20F)	0.9800	C(6)-C(1)-C(2)	114.0(2)
C(21')-H(21D)	0.9800	C(6)-C(1)-Ni(1)	122.88(17)
C(21')-H(21E)	0.9800	C(2)-C(1)-Ni(1)	123.08(17)
C(21')-H(21F)	0.9800	C(3)-C(2)-O(1)	118.62(19)
C(22')-H(22D)	0.9800	C(3)-C(2)-C(1)	124.6(2)
C(22')-H(22E)	0.9800	O(1)-C(2)-C(1)	116.78(19)
C(22')-H(22F)	0.9800	C(2)-C(3)-C(4)	117.9(2)
O(3)-C(23)	1.424(3)	C(2)-C(3)-H(3)	121.0
C(23)-H(23A)	0.9800	C(4)-C(3)-H(3)	121.0
C(23)-H(23B)	0.9800	O(3)-C(4)-C(5)	123.1(2)
C(23)-H(23C)	0.9800	O(3)-C(4)-C(3)	115.8(2)
C(1)-Ni(1)-P(1)	81.89(7)	C(5)-C(4)-C(3)	121.1(2)
C(1)-Ni(1)-P(2)	81.56(7)	C(6)-C(5)-C(4)	117.7(2)
P(1)-Ni(1)-P(2)	163.12(3)	C(6)-C(5)-H(5)	121.2
C(1)-Ni(1)-Cl(1)	178.78(8)	C(4)-C(5)-H(5)	121.2
P(1)-Ni(1)-Cl(1)	96.98(3)	C(5)-C(6)-C(1)	124.7(2)

C(5)-C(6)-O(2)	117.6(2)	H(12A)-C(12)-H(12C)	109.5
C(1)-C(6)-O(2)	117.7(2)	H(12B)-C(12)-H(12C)	109.5
C(10)-C(7)-C(9)	110.5(2)	C(11)-C(13)-H(13A)	109.5
C(10)-C(7)-C(8)	108.7(2)	C(11)-C(13)-H(13B)	109.5
C(9)-C(7)-C(8)	108.8(2)	H(13A)-C(13)-H(13B)	109.5
C(10)-C(7)-P(1)	113.00(16)	C(11)-C(13)-H(13C)	109.5
C(9)-C(7)-P(1)	111.27(17)	H(13A)-C(13)-H(13C)	109.5
C(8)-C(7)-P(1)	104.26(17)	H(13B)-C(13)-H(13C)	109.5
C(7)-C(8)-H(8A)	109.5	C(11)-C(14)-H(14A)	109.5
C(7)-C(8)-H(8B)	109.5	C(11)-C(14)-H(14B)	109.5
H(8A)-C(8)-H(8B)	109.5	H(14A)-C(14)-H(14B)	109.5
C(7)-C(8)-H(8C)	109.5	C(11)-C(14)-H(14C)	109.5
H(8A)-C(8)-H(8C)	109.5	H(14A)-C(14)-H(14C)	109.5
H(8B)-C(8)-H(8C)	109.5	H(14B)-C(14)-H(14C)	109.5
C(7)-C(9)-H(9A)	109.5	C(18)-C(15)-C(17)	108.8(7)
C(7)-C(9)-H(9B)	109.5	C(18)-C(15)-C(16)	110.0(6)
H(9A)-C(9)-H(9B)	109.5	C(17)-C(15)-C(16)	111.2(5)
C(7)-C(9)-H(9C)	109.5	C(18)-C(15)-P(2)	117.8(9)
H(9A)-C(9)-H(9C)	109.5	C(17)-C(15)-P(2)	102.0(4)
H(9B)-C(9)-H(9C)	109.5	C(16)-C(15)-P(2)	106.9(4)
C(7)-C(10)-H(10A)	109.5	C(15)-C(16)-H(16A)	109.5
C(7)-C(10)-H(10B)	109.5	C(15)-C(16)-H(16B)	109.5
H(10A)-C(10)-H(10B)	109.5	H(16A)-C(16)-H(16B)	109.5
C(7)-C(10)-H(10C)	109.5	C(15)-C(16)-H(16C)	109.5
H(10A)-C(10)-H(10C)	109.5	H(16A)-C(16)-H(16C)	109.5
H(10B)-C(10)-H(10C)	109.5	H(16B)-C(16)-H(16C)	109.5
C(13)-C(11)-C(14)	109.9(2)	C(15)-C(17)-H(17A)	109.5
C(13)-C(11)-C(12)	109.2(2)	C(15)-C(17)-H(17B)	109.5
C(14)-C(11)-C(12)	108.8(2)	H(17A)-C(17)-H(17B)	109.5
C(13)-C(11)-P(1)	110.92(17)	C(15)-C(17)-H(17C)	109.5
C(14)-C(11)-P(1)	113.21(18)	H(17A)-C(17)-H(17C)	109.5
C(12)-C(11)-P(1)	104.62(17)	H(17B)-C(17)-H(17C)	109.5
C(11)-C(12)-H(12A)	109.5	C(15)-C(18)-H(18A)	109.5
C(11)-C(12)-H(12B)	109.5	C(15)-C(18)-H(18B)	109.5
H(12A)-C(12)-H(12B)	109.5	H(18A)-C(18)-H(18B)	109.5
C(11)-C(12)-H(12C)	109.5	C(15)-C(18)-H(18C)	109.5

H(18A)-C(18)-H(18C)	109.5	H(16D)-C(16')-H(16F)	109.5
H(18B)-C(18)-H(18C)	109.5	H(16E)-C(16')-H(16F)	109.5
C(22)-C(19)-C(21)	109.1(9)	C(15')-C(17')-H(17D)	109.5
C(22)-C(19)-C(20)	109.9(10)	C(15')-C(17')-H(17E)	109.5
C(21)-C(19)-C(20)	107.5(4)	H(17D)-C(17')-H(17E)	109.5
C(22)-C(19)-P(2)	114.9(13)	C(15')-C(17')-H(17F)	109.5
C(21)-C(19)-P(2)	107.5(3)	H(17D)-C(17')-H(17F)	109.5
C(20)-C(19)-P(2)	107.7(3)	H(17E)-C(17')-H(17F)	109.5
C(19)-C(20)-H(20A)	109.5	C(15')-C(18')-H(18D)	109.5
C(19)-C(20)-H(20B)	109.5	C(15')-C(18')-H(18E)	109.5
H(20A)-C(20)-H(20B)	109.5	H(18D)-C(18')-H(18E)	109.5
C(19)-C(20)-H(20C)	109.5	C(15')-C(18')-H(18F)	109.5
H(20A)-C(20)-H(20C)	109.5	H(18D)-C(18')-H(18F)	109.5
H(20B)-C(20)-H(20C)	109.5	H(18E)-C(18')-H(18F)	109.5
C(19)-C(21)-H(21A)	109.5	C(21')-C(19')-C(22')	110.8(16)
C(19)-C(21)-H(21B)	109.5	C(21')-C(19')-C(20')	109.8(7)
H(21A)-C(21)-H(21B)	109.5	C(22')-C(19')-C(20')	111.2(15)
C(19)-C(21)-H(21C)	109.5	C(21')-C(19')-P(2)	102.0(5)
H(21A)-C(21)-H(21C)	109.5	C(22')-C(19')-P(2)	111(2)
H(21B)-C(21)-H(21C)	109.5	C(20')-C(19')-P(2)	111.4(5)
C(19)-C(22)-H(22A)	109.5	C(19')-C(20')-H(20D)	109.5
C(19)-C(22)-H(22B)	109.5	C(19')-C(20')-H(20E)	109.5
H(22A)-C(22)-H(22B)	109.5	H(20D)-C(20')-H(20E)	109.5
C(19)-C(22)-H(22C)	109.5	C(19')-C(20')-H(20F)	109.5
H(22A)-C(22)-H(22C)	109.5	H(20D)-C(20')-H(20F)	109.5
H(22B)-C(22)-H(22C)	109.5	H(20E)-C(20')-H(20F)	109.5
C(16')-C(15')-C(18')	109.9(13)	C(19')-C(21')-H(21D)	109.5
C(16')-C(15')-C(17')	108.5(9)	C(19')-C(21')-H(21E)	109.5
C(18')-C(15')-C(17')	108.4(12)	H(21D)-C(21')-H(21E)	109.5
C(16')-C(15')-P(2)	116.3(8)	C(19')-C(21')-H(21F)	109.5
C(18')-C(15')-P(2)	108.4(16)	H(21D)-C(21')-H(21F)	109.5
C(17')-C(15')-P(2)	105.1(7)	H(21E)-C(21')-H(21F)	109.5
C(15')-C(16')-H(16D)	109.5	C(19')-C(22')-H(22D)	109.5
C(15')-C(16')-H(16E)	109.5	C(19')-C(22')-H(22E)	109.5
H(16D)-C(16')-H(16E)	109.5	H(22D)-C(22')-H(22E)	109.5
C(15')-C(16')-H(16F)	109.5	C(19')-C(22')-H(22F)	109.5

H(22D)-C(22')-H(22F)	109.5	H(23A)-C(23)-H(23B)	109.5
H(22E)-C(22')-H(22F)	109.5	O(3)-C(23)-H(23C)	109.5
C(4)-O(3)-C(23)	117.94(19)	H(23A)-C(23)-H(23C)	109.5
O(3)-C(23)-H(23A)	109.5	H(23B)-C(23)-H(23C)	109.5
O(3)-C(23)-H(23B)	109.5		

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for **2b**. 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}
Ni1	15(1)	17(1)	16(1)	-2(1)	2(1)	-2(1)
Cl1	31(1)	30(1)	21(1)	-9(1)	4(1)	-8(1)
P1	15(1)	18(1)	15(1)	-2(1)	3(1)	-1(1)
P2	13(1)	17(1)	25(1)	-3(1)	3(1)	-1(1)
O1	15(1)	26(1)	15(1)	-5(1)	4(1)	-3(1)
O2	20(1)	29(1)	19(1)	-3(1)	5(1)	-10(1)
C1	18(1)	16(1)	16(1)	1(1)	3(1)	0(1)
C2	14(1)	18(1)	17(1)	4(1)	3(1)	2(1)
C3	14(1)	20(1)	16(1)	-3(1)	2(1)	-2(1)
C4	18(1)	20(1)	14(1)	0(1)	3(1)	1(1)
C5	20(1)	27(1)	16(1)	0(1)	7(1)	-2(1)
C6	17(1)	17(1)	17(1)	2(1)	2(1)	-6(1)
C7	21(1)	21(1)	18(1)	1(1)	3(1)	-3(1)
C8	28(1)	25(1)	30(1)	2(1)	2(1)	2(1)
C9	30(1)	31(2)	19(1)	0(1)	3(1)	-7(1)
C10	28(1)	32(2)	24(1)	3(1)	6(1)	-9(1)
C11	18(1)	23(1)	25(1)	-2(1)	4(1)	3(1)
C12	25(1)	23(1)	38(1)	6(1)	1(1)	2(1)
C13	35(2)	35(2)	35(2)	-10(1)	11(1)	9(1)
C14	19(1)	31(2)	34(1)	1(1)	6(1)	4(1)
C15	11(1)	47(4)	19(1)	1(2)	4(1)	-1(2)
C16	19(2)	79(4)	24(2)	-16(2)	-1(2)	3(2)
C17	25(2)	72(5)	41(4)	37(3)	0(3)	11(3)
C18	17(2)	33(4)	43(5)	1(3)	14(2)	9(3)
C19	18(2)	14(2)	34(3)	-10(2)	8(2)	-5(1)
C20	40(2)	21(2)	51(3)	-5(2)	24(2)	-2(2)
C21	21(2)	29(2)	39(2)	18(2)	2(2)	1(2)
C22	28(2)	29(2)	42(2)	3(2)	17(1)	-3(2)
C15'	11(1)	47(4)	19(1)	1(2)	4(1)	-1(2)
C16'	19(2)	79(4)	24(2)	-16(2)	-1(2)	3(2)
C17'	25(2)	72(5)	41(4)	37(3)	0(3)	11(3)

C18'	17(2)	33(4)	43(5)	1(3)	14(2)	9(3)
C19'	18(2)	14(2)	34(3)	-10(2)	8(2)	-5(1)
C20'	40(2)	21(2)	51(3)	-5(2)	24(2)	-2(2)
C21'	21(2)	29(2)	39(2)	18(2)	2(2)	1(2)
C22'	28(2)	29(2)	42(2)	3(2)	17(1)	-3(2)
O3	22(1)	31(1)	17(1)	-6(1)	7(1)	-6(1)
C23	31(1)	37(2)	23(1)	-7(1)	11(1)	-3(1)

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

	x	y	z	U(eq)
H3	9152	4663	10267	20
H5	6596	3047	10470	25
H8A	7925	5922	7964	43
H8B	8233	5453	8650	43
H8C	7359	4487	8202	43
H9A	8264	3965	7231	41
H9B	7679	2531	7455	41
H9C	8759	2190	7436	41
H10A	9609	5537	7995	42
H10B	10114	3738	8114	42
H10C	9949	4803	8651	42
H12A	9974	-1722	9270	46
H12B	8853	-1457	8949	46
H12C	9364	-415	9531	46
H13A	10169	-1201	8268	52
H13B	9948	610	7970	52
H13C	9080	-621	7979	52
H14A	11166	329	9189	43
H14B	10655	1710	9487	43
H14C	10855	2089	8868	43
H16A	4467	-1825	8191	63
H16B	5068	-862	7818	63
H16C	3952	-449	7714	63
H17A	5461	2232	8023	72
H17B	5127	3158	8532	72
H17C	4347	2643	7931	72
H18A	3428	1111	8509	45
H18B	4218	1611	9109	45
H18C	3939	-314	8967	45
H20A	6642	-4280	9095	52

H20B	6851	-2794	8694	52
H20C	5782	-3515	8577	52
H21A	6956	-1099	10140	47
H21B	7627	-1523	9725	47
H21C	7195	-3006	10028	47
H22A	5464	-3560	9731	47
H22B	4760	-2663	9167	47
H22C	5167	-1642	9769	47
H16D	4108	1063	7570	63
H16E	4492	-778	7781	63
H16F	5211	591	7650	63
H17D	4477	3429	8198	72
H17E	5617	3192	8341	72
H17F	5127	3309	8868	72
H18D	4277	863	9132	45
H18E	3957	-644	8671	45
H18F	3513	1186	8505	45
H20D	6095	-2699	8260	52
H20E	5059	-3057	8351	52
H20F	5934	-4310	8623	52
H21D	7259	-1680	9768	47
H21E	7468	-2287	9171	47
H21F	7122	-3608	9577	47
H22D	5493	-3626	9643	47
H22E	4695	-2413	9240	47
H22F	5481	-1681	9805	47
H23A	7690	5648	11640	44
H23B	7295	3845	11388	44
H23C	6835	5503	11041	44

Table 6. Torsion angles [°] for **2b**.

C7-P1-O1-C2	118.93(16)	Ni1-P1-C7-C10	172.44(15)
C11-P1-O1-C2	-123.50(16)	O1-P1-C7-C9	-176.52(17)
Ni1-P1-O1-C2	-3.58(16)	C11-P1-C7-C9	76.6(2)
C19'-P2-O2-C6	117.4(4)	Ni1-P1-C7-C9	-62.51(19)
C15-P2-O2-C6	-133.4(3)	O1-P1-C7-C8	-59.41(17)
C15'-P2-O2-C6	-122.5(5)	C11-P1-C7-C8	-166.30(15)
C19-P2-O2-C6	111.2(2)	Ni1-P1-C7-C8	54.61(17)
Ni1-P2-O2-C6	-6.28(16)	O1-P1-C11-C13	-159.65(19)
P1-Ni1-C1-C6	179.0(2)	C7-P1-C11-C13	-52.9(2)
P2-Ni1-C1-C6	-4.37(18)	Ni1-P1-C11-C13	87.18(19)
P1-Ni1-C1-C2	-0.93(18)	O1-P1-C11-C14	-35.6(2)
P2-Ni1-C1-C2	175.7(2)	C7-P1-C11-C14	71.1(2)
P1-O1-C2-C3	-176.65(17)	Ni1-P1-C11-C14	-148.78(16)
P1-O1-C2-C1	3.3(2)	O1-P1-C11-C12	82.75(17)
C6-C1-C2-C3	-1.2(3)	C7-P1-C11-C12	-170.53(16)
Ni1-C1-C2-C3	178.71(18)	Ni1-P1-C11-C12	-30.42(18)
C6-C1-C2-O1	178.88(19)	O2-P2-C15-C18	-36.7(7)
Ni1-C1-C2-O1	-1.2(3)	C19'-P2-C15-C18	77.4(8)
O1-C2-C3-C4	-179.0(2)	C15'-P2-C15-C18	-119(4)
C1-C2-C3-C4	1.1(4)	C19-P2-C15-C18	64.5(7)
C2-C3-C4-O3	-179.9(2)	Ni1-P2-C15-C18	-152.4(6)
C2-C3-C4-C5	-0.4(4)	O2-P2-C15-C17	82.3(4)
O3-C4-C5-C6	179.3(2)	C19'-P2-C15-C17	-163.7(5)
C3-C4-C5-C6	-0.1(4)	C15'-P2-C15-C17	0(3)
C4-C5-C6-C1	0.0(4)	C19-P2-C15-C17	-176.6(4)
C4-C5-C6-O2	179.2(2)	Ni1-P2-C15-C17	-33.5(5)
C2-C1-C6-C5	0.6(3)	O2-P2-C15-C16	-161.0(4)
Ni1-C1-C6-C5	-179.27(19)	C19'-P2-C15-C16	-46.9(5)
C2-C1-C6-O2	-178.62(19)	C15'-P2-C15-C16	117(4)
Ni1-C1-C6-O2	1.5(3)	C19-P2-C15-C16	-59.8(5)
P2-O2-C6-C5	-175.51(18)	Ni1-P2-C15-C16	83.3(4)
P2-O2-C6-C1	3.8(3)	O2-P2-C19-C22	68.2(9)
O1-P1-C7-C10	58.42(19)	C19'-P2-C19-C22	-89.7(18)
C11-P1-C7-C10	-48.5(2)	C15-P2-C19-C22	-35.8(9)

C15'-P2-C19-C22	-36.5(10)	Ni1-P2-C19'-C22'	159.6(14)
Ni1-P2-C19-C22	178.4(9)	O2-P2-C19'-C20'	165.6(5)
O2-P2-C19-C21	-53.5(3)	C15-P2-C19'-C20'	58.9(7)
C19'-P2-C19-C21	148.7(19)	C15'-P2-C19'-C20'	55.6(9)
C15-P2-C19-C21	-157.4(4)	C19-P2-C19'-C20'	-171(2)
C15'-P2-C19-C21	-158.1(6)	Ni1-P2-C19'-C20'	-75.7(6)
Ni1-P2-C19-C21	56.8(3)	C5-C4-O3-C23	-1.6(3)
O2-P2-C19-C20	-169.0(3)	C3-C4-O3-C23	177.9(2)
C19'-P2-C19-C20	33.1(15)		
C15-P2-C19-C20	87.0(4)		
C15'-P2-C19-C20	86.3(6)		
Ni1-P2-C19-C20	-58.8(3)		
O2-P2-C15'-C16'	174.1(8)		
C19'-P2-C15'-C16'	-69.0(10)		
C15-P2-C15'-C16'	-86(3)		
C19-P2-C15'-C16'	-82.6(10)		
Ni1-P2-C15'-C16'	63.1(10)		
O2-P2-C15'-C18'	-61.6(12)		
C19'-P2-C15'-C18'	55.4(13)		
C15-P2-C15'-C18'	38(3)		
C19-P2-C15'-C18'	41.7(13)		
Ni1-P2-C15'-C18'	-172.6(11)		
O2-P2-C15'-C17'	54.1(8)		
C19'-P2-C15'-C17'	171.0(7)		
C15-P2-C15'-C17'	154(4)		
C19-P2-C15'-C17'	157.4(6)		
Ni1-P2-C15'-C17'	-56.9(9)		
O2-P2-C19'-C21'	-77.4(6)		
C15-P2-C19'-C21'	175.9(5)		
C15'-P2-C19'-C21'	172.6(6)		
C19-P2-C19'-C21'	-53.8(14)		
Ni1-P2-C19'-C21'	41.3(6)		
O2-P2-C19'-C22'	40.9(15)		
C15-P2-C19'-C22'	-65.9(15)		
C15'-P2-C19'-C22'	-69.1(15)		
C19-P2-C19'-C22'	64(2)		

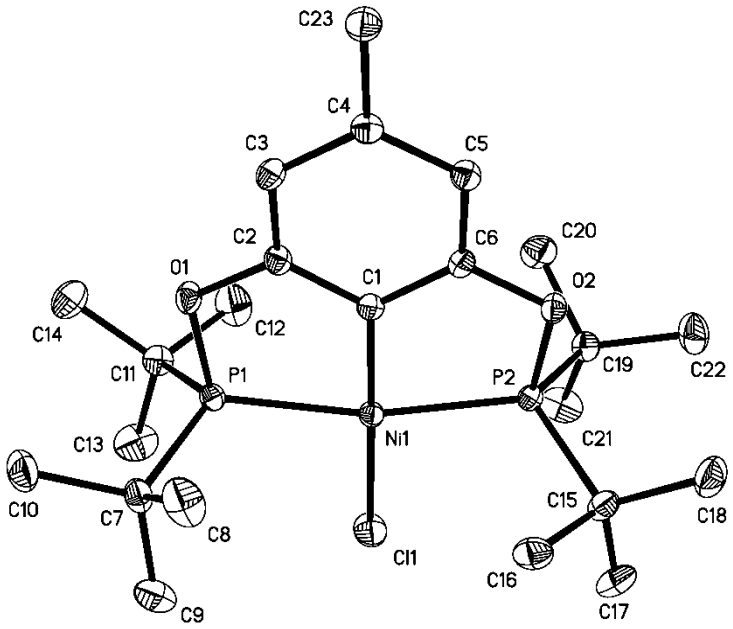
CRYSTAL STRUCTURE REPORT

$C_{23}H_{41}ClNiO_2P_2$ (**2c**)

Report prepared for:

Dr. S. Chakraborty, Prof. W. D. Jones

October 07, 2013



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Data collection

A crystal (0.25 x 0.12 x 0.08 mm^3) was placed onto the tip of a 0.1 mm diameter glass capillary tube and mounted on a Bruker SMART APEX II CCD platform diffractometer for a data collection at 100.0(5) K.[1] A preliminary set of cell constants and an orientation matrix were calculated from reflections harvested from three orthogonal wedges of reciprocal space. The full data collection was carried out using MoKα radiation (graphite monochromator) with a frame time of 60 seconds and a detector distance of 4.01 cm. A randomly oriented region of reciprocal space was surveyed: five major sections of frames were collected with 0.50º steps in ω at five different ϕ settings and a detector position of -38º in 2θ. The intensity data were corrected for absorption.[2] Final cell constants were calculated from the xyz centroids of 4087 strong reflections from the actual data collection after integration.[3] See Table 1 for additional crystal and refinement information.

Structure solution and refinement

The structure was solved using SIR2011[4] and refined using SHELXL-2013.[5] The space group $P2_1/n$ was determined based on systematic absences. A direct-methods solution was calculated which provided most non-hydrogen atoms from the E-map. Full-matrix least squares / difference Fourier cycles were performed which located the remaining non-hydrogen atoms. All non-hydrogen atoms were refined with anisotropic displacement parameters. All hydrogen atoms were placed in ideal positions and refined as riding atoms with relative isotropic displacement parameters. The final full matrix least squares refinement converged to $R1 = 0.0485$ (F^2, $I > 2\sigma(I)$) and $wR2 = 0.1123$ (F^2, all data).

Structure description

The structure is the one suggested. The asymmetric unit contains one molecule in a general position.

Unless noted otherwise all structural diagrams containing thermal displacement ellipsoids are drawn at the 50 % probability level.

Data collection, structure solution, and structure refinement were conducted at the X-ray Crystallographic Facility, B51 Hutchison Hall, Department of Chemistry, University of Rochester. All publications arising from this report MUST either 1) include William W. Brennessel as a coauthor or 2) acknowledge William W. Brennessel and the X-ray Crystallographic Facility of the Department of Chemistry at the University of Rochester.

[1] *APEX2*, version 2013.2-0; Bruker AXS: Madison, WI, 2013.

[2] Sheldrick, G. M. *SADABS*, version 2012/1; University of Göttingen: Göttingen, Germany, 2012.

[3] *SAINT*, version 8.27B; Bruker AXS: Madison, WI, 2013.

[4] Burla, M. C.; Caliandro, R.; Camalli, M.; Carrozzini, B.; Cascarano, G. L.; Giacovazzo, C.; Mallamo, M.; Mazzone, A.; Polidori, G.; Spagna, R. *SIR2011: a new package for crystal structure determination and refinement*, version 1.0; Istituto di Cristallografia: Bari, Italy, 2012.

[5] Sheldrick, G. M. *SHELXL-2013/4*; University of Göttingen: Göttingen, Germany, 2013.

Some equations of interest:

$$R_{int} = \Sigma\, |F_o^2 - < F_o^2 >| \,/\, \Sigma |F_o{}^2|$$

$$R1 = \Sigma ||F_o| - |F_c|| \,/\, \Sigma |F_o|$$

$$wR2 = [\Sigma [w(F_o^2 - F_c{}^2)^2] \,/\, \Sigma\, [w(F_o^2)^2]]^{1/2}$$

where $w = 1 \,/\, [\sigma^2 (F_o^2) + (aP)^2 + bP]$ and

$$P = 1/3 \max (0, F_o^2) + 2/3\, F_c^2$$

$$\text{GOF} = S = [\Sigma [w(F_o^2 - F_c{}^2)^2] \,/\, (m-n)]^{1/2}$$

where m = number of reflections and n = number of parameters

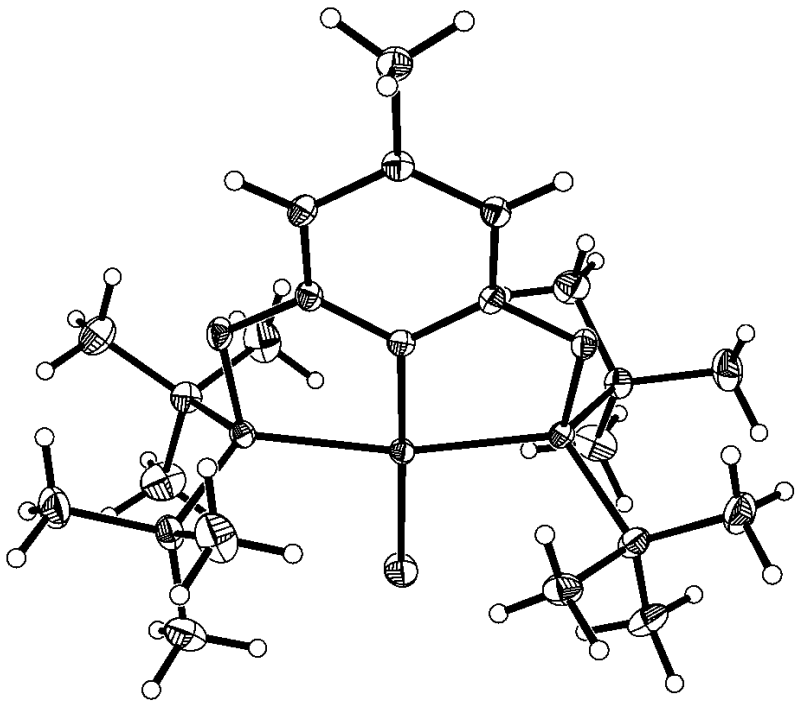


Table 1. Crystal data and structure refinement for **2c.**

Empirical formula	$C_{23}H_{41}ClNiO_2P_2$	
Formula weight	505.66	
Temperature	100.0(5) K	
Wavelength	0.71073 Å	
Crystal system	monoclinic	
Space group	$P2_1/n$	
Unit cell dimensions	a = 9.8433(17) Å	α = 90°
	b = 19.618(3) Å	β = 105.052(4)°
	c = 13.982(2) Å	γ = 90°
Volume	2607.5(8) Å^3	
Z	4	
Density (calculated)	1.288 Mg/m^3	
Absorption coefficient	0.985 mm^{-1}	
F(000)	1080	
Crystal color, morphology	yellow, needle	
Crystal size	0.25 x 0.12 x 0.08 mm^3	
Theta range for data collection	1.831 to 36.359°	
Index ranges	$-16 \le h \le 16$, $-32 \le k \le 32$, $-23 \le l \le 23$	
Reflections collected	69051	
Independent reflections	12656 [R(int) = 0.0951]	
Observed reflections	8216	
Completeness to theta = 36.320°	99.9%	
Absorption correction	Multi-scan	
Max. and min. transmission	0.7471 and 0.5872	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	12656 / 0 / 275	
Goodness-of-fit on F^2	1.022	
Final R indices [I>2sigma(I)]	R1 = 0.0485, wR2 = 0.0973	
R indices (all data)	R1 = 0.0928, wR2 = 0.1123	
Largest diff. peak and hole	0.709 and -0.890 e.Å^{-3}	

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

	x	y	z	U_{eq}
Ni1	4946(1)	2527(1)	7578(1)	13(1)
Cl1	5808(1)	1899(1)	6574(1)	25(1)
P1	6131(1)	3446(1)	7480(1)	13(1)
P2	3526(1)	1786(1)	7949(1)	13(1)
O1	5721(1)	3995(1)	8256(1)	17(1)
O2	2669(1)	2191(1)	8652(1)	14(1)
C1	4210(2)	3078(1)	8421(1)	13(1)
C2	4658(2)	3748(1)	8647(1)	14(1)
C3	4094(2)	4177(1)	9232(1)	15(1)
C4	3050(2)	3931(1)	9656(1)	14(1)
C5	2591(2)	3256(1)	9476(1)	14(1)
C6	3158(2)	2856(1)	8859(1)	13(1)
C7	5554(2)	3932(1)	6305(1)	19(1)
C8	3976(2)	4062(1)	6178(2)	31(1)
C9	5726(3)	3507(1)	5426(2)	32(1)
C10	6315(2)	4616(1)	6347(2)	30(1)
C11	8073(2)	3396(1)	7981(1)	18(1)
C12	8293(2)	2862(1)	8810(2)	33(1)
C13	8791(2)	3149(1)	7194(2)	33(1)
C14	8731(2)	4070(1)	8422(2)	27(1)
C15	2040(2)	1498(1)	6914(1)	18(1)
C16	1646(2)	2137(1)	6254(1)	21(1)
C17	2527(2)	937(1)	6314(2)	26(1)
C18	741(2)	1269(1)	7246(2)	26(1)
C19	4367(2)	1086(1)	8789(1)	17(1)
C20	5216(2)	1442(1)	9735(1)	25(1)
C21	5395(2)	689(1)	8339(2)	28(1)
C22	3315(2)	596(1)	9051(2)	26(1)
C23	2421(2)	4394(1)	10290(1)	19(1)

Table 3. Bond lengths [Å] and angles [°] for **2c**.

Ni(1)-C(1)	1.8772(16)	C(11)-C(13)	1.533(3)
Ni(1)-P(1)	2.1709(5)	C(11)-C(12)	1.536(3)
Ni(1)-P(2)	2.1720(5)	C(12)-H(12A)	0.9800
Ni(1)-Cl(1)	2.1970(5)	C(12)-H(12B)	0.9800
P(1)-O(1)	1.6521(12)	C(12)-H(12C)	0.9800
P(1)-C(7)	1.8566(18)	C(13)-H(13A)	0.9800
P(1)-C(11)	1.8602(18)	C(13)-H(13B)	0.9800
P(2)-O(2)	1.6547(12)	C(13)-H(13C)	0.9800
P(2)-C(19)	1.8555(17)	C(14)-H(14A)	0.9800
P(2)-C(15)	1.8578(18)	C(14)-H(14B)	0.9800
O(1)-C(2)	1.3873(19)	C(14)-H(14C)	0.9800
O(2)-C(6)	1.3951(19)	C(15)-C(17)	1.534(3)
C(1)-C(2)	1.398(2)	C(15)-C(18)	1.537(3)
C(1)-C(6)	1.402(2)	C(15)-C(16)	1.544(2)
C(2)-C(3)	1.387(2)	C(16)-H(16A)	0.9800
C(3)-C(4)	1.399(2)	C(16)-H(16B)	0.9800
C(3)-H(3)	0.9500	C(16)-H(16C)	0.9800
C(4)-C(5)	1.401(2)	C(17)-H(17A)	0.9800
C(4)-C(23)	1.511(2)	C(17)-H(17B)	0.9800
C(5)-C(6)	1.385(2)	C(17)-H(17C)	0.9800
C(5)-H(5)	0.9500	C(18)-H(18A)	0.9800
C(7)-C(9)	1.531(3)	C(18)-H(18B)	0.9800
C(7)-C(10)	1.531(3)	C(18)-H(18C)	0.9800
C(7)-C(8)	1.537(3)	C(19)-C(22)	1.526(2)
C(8)-H(8A)	0.9800	C(19)-C(21)	1.535(2)
C(8)-H(8B)	0.9800	C(19)-C(20)	1.537(3)
C(8)-H(8C)	0.9800	C(20)-H(20A)	0.9800
C(9)-H(9A)	0.9800	C(20)-H(20B)	0.9800
C(9)-H(9B)	0.9800	C(20)-H(20C)	0.9800
C(9)-H(9C)	0.9800	C(21)-H(21A)	0.9800
C(10)-H(10A)	0.9800	C(21)-H(21B)	0.9800
C(10)-H(10B)	0.9800	C(21)-H(21C)	0.9800
C(10)-H(10C)	0.9800	C(22)-H(22A)	0.9800
C(11)-C(14)	1.530(3)	C(22)-H(22B)	0.9800

C(22)-H(22C)	0.9800	C(6)-C(5)-C(4)	118.91(15)
C(23)-H(23A)	0.9800	C(6)-C(5)-H(5)	120.5
C(23)-H(23B)	0.9800	C(4)-C(5)-H(5)	120.5
C(23)-H(23C)	0.9800	C(5)-C(6)-O(2)	119.07(14)
C(1)-Ni(1)-P(1)	82.16(5)	C(5)-C(6)-C(1)	123.99(15)
C(1)-Ni(1)-P(2)	81.84(5)	O(2)-C(6)-C(1)	116.94(14)
P(1)-Ni(1)-P(2)	163.999(19)	C(9)-C(7)-C(10)	110.70(16)
C(1)-Ni(1)-Cl(1)	178.96(5)	C(9)-C(7)-C(8)	108.49(17)
P(1)-Ni(1)-Cl(1)	97.01(2)	C(10)-C(7)-C(8)	109.19(17)
P(2)-Ni(1)-Cl(1)	98.99(2)	C(9)-C(7)-P(1)	110.99(13)
O(1)-P(1)-C(7)	100.11(8)	C(10)-C(7)-P(1)	112.44(13)
O(1)-P(1)-C(11)	100.91(7)	C(8)-C(7)-P(1)	104.78(12)
C(7)-P(1)-C(11)	114.14(8)	C(7)-C(8)-H(8A)	109.5
O(1)-P(1)-Ni(1)	105.84(5)	C(7)-C(8)-H(8B)	109.5
C(7)-P(1)-Ni(1)	115.90(6)	H(8A)-C(8)-H(8B)	109.5
C(11)-P(1)-Ni(1)	116.68(6)	C(7)-C(8)-H(8C)	109.5
O(2)-P(2)-C(19)	101.11(7)	H(8A)-C(8)-H(8C)	109.5
O(2)-P(2)-C(15)	100.89(7)	H(8B)-C(8)-H(8C)	109.5
C(19)-P(2)-C(15)	113.58(8)	C(7)-C(9)-H(9A)	109.5
O(2)-P(2)-Ni(1)	106.18(4)	C(7)-C(9)-H(9B)	109.5
C(19)-P(2)-Ni(1)	115.96(6)	H(9A)-C(9)-H(9B)	109.5
C(15)-P(2)-Ni(1)	116.23(6)	C(7)-C(9)-H(9C)	109.5
C(2)-O(1)-P(1)	111.65(10)	H(9A)-C(9)-H(9C)	109.5
C(6)-O(2)-P(2)	111.82(10)	H(9B)-C(9)-H(9C)	109.5
C(2)-C(1)-C(6)	114.78(14)	C(7)-C(10)-H(10A)	109.5
C(2)-C(1)-Ni(1)	122.17(12)	C(7)-C(10)-H(10B)	109.5
C(6)-C(1)-Ni(1)	123.05(12)	H(10A)-C(10)-H(10B)	109.5
O(1)-C(2)-C(3)	118.64(14)	C(7)-C(10)-H(10C)	109.5
O(1)-C(2)-C(1)	117.78(14)	H(10A)-C(10)-H(10C)	109.5
C(3)-C(2)-C(1)	123.57(15)	H(10B)-C(10)-H(10C)	109.5
C(2)-C(3)-C(4)	119.39(15)	C(14)-C(11)-C(13)	109.73(16)
C(2)-C(3)-H(3)	120.3	C(14)-C(11)-C(12)	108.85(16)
C(4)-C(3)-H(3)	120.3	C(13)-C(11)-C(12)	108.97(17)
C(3)-C(4)-C(5)	119.29(15)	C(14)-C(11)-P(1)	113.12(13)
C(3)-C(4)-C(23)	119.88(15)	C(13)-C(11)-P(1)	111.49(13)
C(5)-C(4)-C(23)	120.83(15)	C(12)-C(11)-P(1)	104.47(12)

C(11)-C(12)-H(12A)	109.5	C(15)-C(18)-H(18A)	109.5
C(11)-C(12)-H(12B)	109.5	C(15)-C(18)-H(18B)	109.5
H(12A)-C(12)-H(12B)	109.5	H(18A)-C(18)-H(18B)	109.5
C(11)-C(12)-H(12C)	109.5	C(15)-C(18)-H(18C)	109.5
H(12A)-C(12)-H(12C)	109.5	H(18A)-C(18)-H(18C)	109.5
H(12B)-C(12)-H(12C)	109.5	H(18B)-C(18)-H(18C)	109.5
C(11)-C(13)-H(13A)	109.5	C(22)-C(19)-C(21)	109.85(16)
C(11)-C(13)-H(13B)	109.5	C(22)-C(19)-C(20)	109.29(15)
H(13A)-C(13)-H(13B)	109.5	C(21)-C(19)-C(20)	108.27(16)
C(11)-C(13)-H(13C)	109.5	C(22)-C(19)-P(2)	113.55(13)
H(13A)-C(13)-H(13C)	109.5	C(21)-C(19)-P(2)	110.49(12)
H(13B)-C(13)-H(13C)	109.5	C(20)-C(19)-P(2)	105.19(12)
C(11)-C(14)-H(14A)	109.5	C(19)-C(20)-H(20A)	109.5
C(11)-C(14)-H(14B)	109.5	C(19)-C(20)-H(20B)	109.5
H(14A)-C(14)-H(14B)	109.5	H(20A)-C(20)-H(20B)	109.5
C(11)-C(14)-H(14C)	109.5	C(19)-C(20)-H(20C)	109.5
H(14A)-C(14)-H(14C)	109.5	H(20A)-C(20)-H(20C)	109.5
H(14B)-C(14)-H(14C)	109.5	H(20B)-C(20)-H(20C)	109.5
C(17)-C(15)-C(18)	110.92(15)	C(19)-C(21)-H(21A)	109.5
C(17)-C(15)-C(16)	108.72(15)	C(19)-C(21)-H(21B)	109.5
C(18)-C(15)-C(16)	108.58(15)	H(21A)-C(21)-H(21B)	109.5
C(17)-C(15)-P(2)	110.43(13)	C(19)-C(21)-H(21C)	109.5
C(18)-C(15)-P(2)	113.69(13)	H(21A)-C(21)-H(21C)	109.5
C(16)-C(15)-P(2)	104.17(12)	H(21B)-C(21)-H(21C)	109.5
C(15)-C(16)-H(16A)	109.5	C(19)-C(22)-H(22A)	109.5
C(15)-C(16)-H(16B)	109.5	C(19)-C(22)-H(22B)	109.5
H(16A)-C(16)-H(16B)	109.5	H(22A)-C(22)-H(22B)	109.5
C(15)-C(16)-H(16C)	109.5	C(19)-C(22)-H(22C)	109.5
H(16A)-C(16)-H(16C)	109.5	H(22A)-C(22)-H(22C)	109.5
H(16B)-C(16)-H(16C)	109.5	H(22B)-C(22)-H(22C)	109.5
C(15)-C(17)-H(17A)	109.5	C(4)-C(23)-H(23A)	109.5
C(15)-C(17)-H(17B)	109.5	C(4)-C(23)-H(23B)	109.5
H(17A)-C(17)-H(17B)	109.5	H(23A)-C(23)-H(23B)	109.5
C(15)-C(17)-H(17C)	109.5	C(4)-C(23)-H(23C)	109.5
H(17A)-C(17)-H(17C)	109.5	H(23A)-C(23)-H(23C)	109.5
H(17B)-C(17)-H(17C)	109.5	H(23B)-C(23)-H(23C)	109.5

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for **2c**. 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}
Ni1	14(1)	12(1)	14(1)	-1(1)	6(1)	0(1)
Cl1	34(1)	21(1)	28(1)	-7(1)	20(1)	-1(1)
P1	12(1)	14(1)	14(1)	1(1)	4(1)	0(1)
P2	13(1)	11(1)	14(1)	-1(1)	3(1)	0(1)
O1	16(1)	14(1)	23(1)	-3(1)	11(1)	-3(1)
O2	13(1)	12(1)	18(1)	-2(1)	6(1)	-2(1)
C1	14(1)	14(1)	12(1)	0(1)	4(1)	0(1)
C2	10(1)	17(1)	16(1)	0(1)	4(1)	-1(1)
C3	14(1)	12(1)	18(1)	-2(1)	3(1)	0(1)
C4	13(1)	15(1)	15(1)	0(1)	4(1)	2(1)
C5	13(1)	15(1)	16(1)	0(1)	5(1)	1(1)
C6	12(1)	12(1)	14(1)	0(1)	2(1)	-1(1)
C7	17(1)	19(1)	20(1)	5(1)	3(1)	-2(1)
C8	18(1)	39(1)	34(1)	16(1)	1(1)	4(1)
C9	42(1)	35(1)	18(1)	3(1)	7(1)	-1(1)
C10	35(1)	24(1)	28(1)	10(1)	3(1)	-6(1)
C11	13(1)	21(1)	20(1)	0(1)	5(1)	2(1)
C12	23(1)	37(1)	33(1)	13(1)	-1(1)	0(1)
C13	20(1)	45(1)	37(1)	-10(1)	13(1)	4(1)
C14	18(1)	28(1)	32(1)	-4(1)	1(1)	-3(1)
C15	18(1)	18(1)	17(1)	-2(1)	1(1)	-2(1)
C16	22(1)	22(1)	17(1)	2(1)	1(1)	4(1)
C17	28(1)	23(1)	23(1)	-8(1)	-1(1)	-1(1)
C18	18(1)	28(1)	29(1)	0(1)	1(1)	-7(1)
C19	17(1)	15(1)	18(1)	2(1)	4(1)	2(1)
C20	26(1)	26(1)	20(1)	3(1)	-1(1)	1(1)
C21	29(1)	27(1)	28(1)	4(1)	9(1)	15(1)
C22	26(1)	21(1)	31(1)	11(1)	5(1)	-1(1)
C23	20(1)	18(1)	22(1)	-4(1)	9(1)	0(1)

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

	x	y	z	U(eq)
H3	4414	4635	9345	18
H5	1902	3075	9772	17
H8A	3590	4312	5560	47
H8B	3848	4333	6738	47
H8C	3486	3626	6157	47
H9A	5323	3755	4809	48
H9B	5237	3071	5414	48
H9C	6728	3424	5493	48
H10A	5871	4886	5759	44
H10B	7304	4536	6366	44
H10C	6257	4865	6943	44
H12A	9299	2825	9140	49
H12B	7939	2420	8527	49
H12C	7782	3002	9293	49
H13A	9778	3041	7509	49
H13B	8743	3507	6698	49
H13C	8311	2740	6871	49
H14A	9746	4008	8699	40
H14B	8302	4215	8947	40
H14C	8568	4419	7902	40
H16A	824	2036	5705	32
H16B	2439	2265	5989	32
H16C	1426	2513	6648	32
H17A	1796	856	5700	39
H17B	2700	517	6705	39
H17C	3397	1081	6155	39
H18A	-52	1202	6663	39
H18B	497	1619	7673	39
H18C	949	839	7613	39
H20A	5724	1099	10203	38

H20B	4575	1695	10036	38
H20C	5891	1758	9568	38
H21A	5895	350	8817	41
H21B	6075	1005	8180	41
H21C	4872	458	7734	41
H22A	3812	284	9574	40
H22B	2842	333	8463	40
H22C	2614	855	9286	40
H23A	2081	4120	10766	29
H23B	1634	4649	9868	29
H23C	3140	4714	10647	29

Table 6. Torsion angles [°] for **2c**.

C7-P1-O1-C2	114.13(12)	C11-P1-C7-C10	-47.51(17)
C11-P1-O1-C2	-128.65(12)	Ni1-P1-C7-C10	172.62(12)
Ni1-P1-O1-C2	-6.65(12)	O1-P1-C7-C8	-59.10(14)
C19-P2-O2-C6	117.18(11)	C11-P1-C7-C8	-165.99(13)
C15-P2-O2-C6	-125.89(11)	Ni1-P1-C7-C8	54.15(14)
Ni1-P2-O2-C6	-4.26(11)	O1-P1-C11-C14	-33.54(15)
P1-Ni1-C1-C2	-1.56(13)	C7-P1-C11-C14	72.85(15)
P2-Ni1-C1-C2	178.39(14)	Ni1-P1-C11-C14	-147.61(12)
P1-Ni1-C1-C6	177.31(14)	O1-P1-C11-C13	-157.78(14)
P2-Ni1-C1-C6	-2.74(13)	C7-P1-C11-C13	-51.38(16)
P1-O1-C2-C3	-173.45(13)	Ni1-P1-C11-C13	88.15(14)
P1-O1-C2-C1	6.23(19)	O1-P1-C11-C12	84.68(14)
C6-C1-C2-O1	178.55(14)	C7-P1-C11-C12	-168.92(13)
Ni1-C1-C2-O1	-2.5(2)	Ni1-P1-C11-C12	-29.39(15)
C6-C1-C2-C3	-1.8(2)	O2-P2-C15-C17	-163.65(13)
Ni1-C1-C2-C3	177.17(13)	C19-P2-C15-C17	-56.31(15)
O1-C2-C3-C4	-178.18(15)	Ni1-P2-C15-C17	82.08(13)
C1-C2-C3-C4	2.2(3)	O2-P2-C15-C18	-38.23(14)
C2-C3-C4-C5	-0.2(2)	C19-P2-C15-C18	69.11(15)
C2-C3-C4-C23	-179.63(16)	Ni1-P2-C15-C18	-152.50(11)
C3-C4-C5-C6	-1.8(2)	O2-P2-C15-C16	79.78(12)
C23-C4-C5-C6	177.55(16)	C19-P2-C15-C16	-172.87(11)
C4-C5-C6-O2	-177.86(14)	Ni1-P2-C15-C16	-34.49(13)
C4-C5-C6-C1	2.2(3)	O2-P2-C19-C22	63.65(14)
P2-O2-C6-C5	-177.21(13)	C15-P2-C19-C22	-43.55(16)
P2-O2-C6-C1	2.71(18)	Ni1-P2-C19-C22	177.95(11)
C2-C1-C6-C5	-0.5(2)	O2-P2-C19-C21	-172.42(13)
Ni1-C1-C6-C5	-179.39(13)	C15-P2-C19-C21	80.38(15)
C2-C1-C6-O2	179.64(14)	Ni1-P2-C19-C21	-58.12(14)
Ni1-C1-C6-O2	0.7(2)	O2-P2-C19-C20	-55.80(13)
O1-P1-C7-C9	-176.01(14)	C15-P2-C19-C20	-163.00(12)
C11-P1-C7-C9	77.10(15)	Ni1-P2-C19-C20	58.50(13)
Ni1-P1-C7-C9	-62.76(15)		
O1-P1-C7-C10	59.38(15)		

Table 1. Crystal Data and Refinement Parameters for **2b** and **2c**.

	2b	**2c**
empirical formula	$C_{23}H_{41}O_3P_2ClNi$	$C_{23}H_{41}O_2P_2ClNi$
crystal system	monoclinic	monoclinic
space group	$P2_1/c$	$P2_1/n$
a, Å	14.485(4)	9.8433(17)
b, Å	7.985(2)	19.618(3)
c, Å	23.646(7)	13.982(2)
α, deg	90	90
β, deg	106.418(6)	105.052(4)
γ, deg	90	90
Volume, Å^3	2623.3(13)	2607.5(8)
Z	4	4
no. of data collected	45634	69051
no. of unique data, R_{int}	9223, 0.0967	12656, 0.0951
R1, wR2 (I > 2σ(I))	0.0498, 0.0939	0.0485, 0.0973
R1, wR2 (all data)	0.0934, 0.1087	0.0928, 0.1123

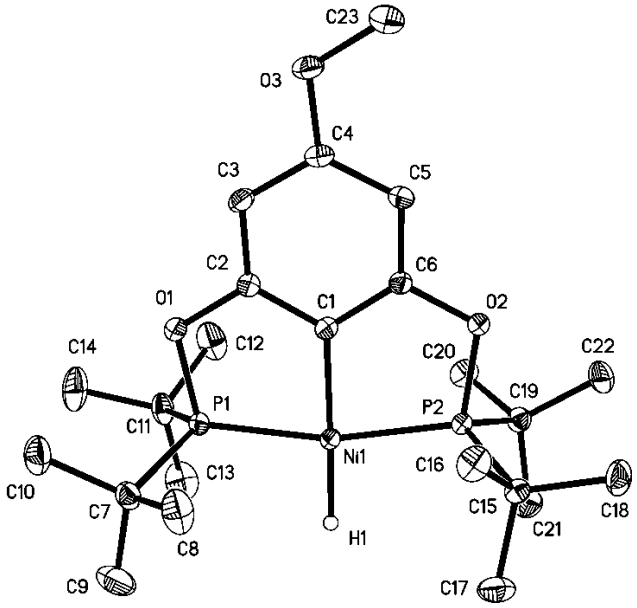
CRYSTAL STRUCTURE REPORT

$C_{23}H_{42}NiO_3P_2$ (**3b**)

Report prepared for:

Dr. S. Chakraborty, Prof. W. D. Jones

September 30, 2013



William W. Brennessel

X-ray Crystallographic Facility

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Data collection

A crystal (0.20 x 0.18 x 0.12 mm^3) was placed onto the tip of a 0.1 mm diameter glass capillary tube and mounted on a Bruker SMART APEX II CCD platform diffractometer for a data collection at 100.0(5) K.[1] A preliminary set of cell constants and an orientation matrix were calculated from reflections harvested from three orthogonal wedges of reciprocal space. The full data collection was carried out using MoKα radiation (graphite monochromator) with a frame time of 60 seconds and a detector distance of 4.02 cm. A randomly oriented region of reciprocal space was surveyed: four major sections of frames were collected with 0.50º steps in ω at four different ϕ settings and a detector position of -38º in 2θ. The intensity data were corrected for absorption.[2] Final cell constants were calculated from the xyz centroids of 4047 strong reflections from the actual data collection after integration.[3] See Table 1 for additional crystal and refinement information.

Structure solution and refinement

The structure was solved using SIR2011[4] and refined using SHELXL-2013.[5] The space group $P2_1/n$ was determined based on systematic absences. A direct-methods solution was calculated which provided most non-hydrogen atoms from the E-map. Full-matrix least squares / difference Fourier cycles were performed which located the remaining non-hydrogen atoms. All non-hydrogen atoms were refined with anisotropic displacement parameters. The hydride ligand was found from the difference Fourier map; its position was refined freely and its isotropic displacement parameter was refined relative to that of the nickel atom: U_{iso}[H] = 2*U_{eq}[Ni1]. All other hydrogen atoms were placed in ideal positions and refined as riding atoms with relative isotropic displacement parameters. The final full matrix least squares refinement converged to $R1 = 0.0493$ (F^2, $I > 2\sigma(I)$) and $wR2 = 0.1152$ (F^2, all data).

Structure description

The structure is the one suggested. The asymmetric unit contains one molecule in a general position.

Unless noted otherwise all structural diagrams containing thermal displacement ellipsoids are drawn at the 50 % probability level.

Data collection, structure solution, and structure refinement were conducted at the X-ray Crystallographic Facility, B51 Hutchison Hall, Department of Chemistry, University of Rochester. All publications arising from this report MUST either 1) include William W. Brennessel as a coauthor or 2) acknowledge William W. Brennessel and the X-ray Crystallographic Facility of the Department of Chemistry at the University of Rochester.

[1] *APEX2*, version 2013.2-0; Bruker AXS: Madison, WI, 2013.

[2] Sheldrick, G. M. *SADABS*, version 2012/1; University of Göttingen: Göttingen, Germany, 2012.

[3] *SAINT*, version 8.27B; Bruker AXS: Madison, WI, 2013.

[4] Burla, M. C.; Caliandro, R.; Camalli, M.; Carrozzini, B.; Cascarano, G. L.; Giacovazzo, C.; Mallamo, M.; Mazzone, A.; Polidori, G.; Spagna, R. *SIR2011: a new package for crystal structure determination and refinement*, version 1.0; Istituto di Cristallografia: Bari, Italy, 2012.

[5] Sheldrick, G. M. *SHELXL-2013/4*; University of Göttingen: Göttingen, Germany, 2013.

Some equations of interest:

$$R_{int} = \Sigma\, |F_o^2 - < F_o^2 >| \,/\, \Sigma |F_o{}^2|$$

$$R1 = \Sigma ||F_o| - |F_c|| \,/\, \Sigma |F_o|$$

$$wR2 = [\Sigma [w(F_o^2 - F_c{}^2)^2] \,/\, \Sigma\, [w(F_o^2)^2]]^{1/2}$$

where $w = 1 \,/\, [\sigma^2 (F_o^2) + (aP)^2 + bP]$ and

$$P = 1/3 \max (0, F_o^2) + 2/3\, F_c^2$$

$$\text{GOF} = S = [\Sigma [w(F_o^2 - F_c{}^2)^2] \,/\, (m-n)]^{1/2}$$

where m = number of reflections and n = number of parameters

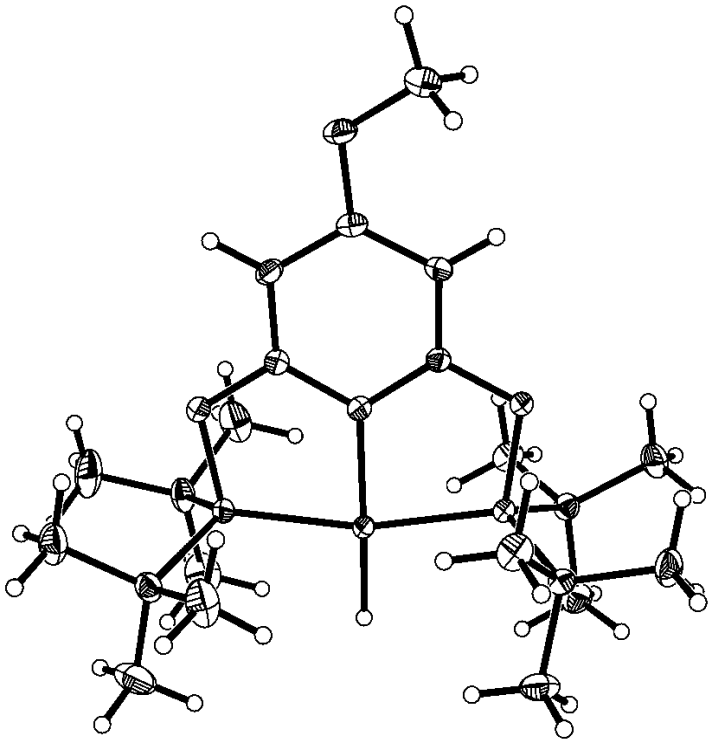


Table 1. Crystal data and structure refinement for **3b**.

Empirical formula	$C_{23}H_{42}NiO_3P_2$	
Formula weight	487.21	
Temperature	100.0(5) K	
Wavelength	0.71073 Å	
Crystal system	monoclinic	
Space group	$P2_1/n$	
Unit cell dimensions	a = 9.756(3) Å	α = 90°
	b = 19.408(6) Å	β = 104.528(6)°
	c = 14.026(4) Å	γ = 90°
Volume	2570.9(14) Å^3	
Z	4	
Density (calculated)	1.259 Mg/m^3	
Absorption coefficient	0.899 mm^{-1}	
F(000)	1048	
Crystal color, morphology	yellow, needle	
Crystal size	0.20 x 0.18 x 0.12 mm^3	
Theta range for data collection	1.830 to 38.710°	
Index ranges	$-16 \le h \le 17$, $-33 \le k \le 33$, $-24 \le l \le 24$	
Reflections collected	64153	
Independent reflections	14241 [R(int) = 0.0784]	
Observed reflections	9065	
Completeness to theta = 37.785°	99.7%	
Absorption correction	Multi-scan	
Max. and min. transmission	0.7476 and 0.6603	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	14241 / 0 / 278	
Goodness-of-fit on F^2	1.029	
Final R indices [I>2sigma(I)]	$R1$ = 0.0493, $wR2$ = 0.0990	
R indices (all data)	$R1$ = 0.0949, $wR2$ = 0.1152	
Largest diff. peak and hole	0.637 and -0.722 e.Å^{-3}	

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

	x	y	z	U_{eq}
Ni1	9874(1)	2500(1)	2502(1)	13(1)
P1	11056(1)	1602(1)	2397(1)	13(1)
P2	8489(1)	3240(1)	2860(1)	12(1)
O1	10591(1)	1014(1)	3121(1)	18(1)
O2	7578(1)	2840(1)	3555(1)	15(1)
C1	9098(2)	1935(1)	3330(1)	14(1)
C2	9550(2)	1260(1)	3548(1)	15(1)
C3	9029(2)	835(1)	4164(1)	16(1)
C4	8010(2)	1107(1)	4603(1)	16(1)
C5	7516(2)	1779(1)	4422(1)	16(1)
C6	8069(1)	2170(1)	3776(1)	14(1)
C7	12990(2)	1636(1)	2955(1)	18(1)
C8	13142(2)	2096(1)	3864(1)	32(1)
C9	13768(2)	1958(1)	2253(2)	35(1)
C10	13645(2)	933(1)	3313(1)	28(1)
C11	10567(2)	1147(1)	1195(1)	21(1)
C12	8969(2)	1028(1)	999(2)	33(1)
C13	10838(2)	1620(1)	386(1)	34(1)
C14	11325(2)	461(1)	1200(2)	34(1)
C15	9358(2)	3931(1)	3720(1)	16(1)
C16	10149(2)	3559(1)	4664(1)	24(1)
C17	10454(2)	4301(1)	3286(1)	26(1)
C18	8330(2)	4451(1)	3968(1)	25(1)
C19	7029(2)	3551(1)	1833(1)	17(1)
C20	6550(2)	2907(1)	1199(1)	21(1)
C21	7608(2)	4079(1)	1218(1)	23(1)
C22	5754(2)	3846(1)	2146(1)	24(1)
O3	7562(1)	663(1)	5223(1)	22(1)
C23	6719(2)	943(1)	5822(1)	27(1)

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

Ni(1)-C(1)	1.8876(14)	C(11)-C(12)	1.531(2)
Ni(1)-P(1)	2.1155(6)	C(11)-C(13)	1.533(3)
Ni(1)-P(2)	2.1161(6)	C(12)-H(12A)	0.9800
Ni(1)-H(1)	1.47(2)	C(12)-H(12B)	0.9800
P(1)-O(1)	1.6624(11)	C(12)-H(12C)	0.9800
P(1)-C(7)	1.8546(16)	C(13)-H(13A)	0.9800
P(1)-C(11)	1.8559(16)	C(13)-H(13B)	0.9800
P(2)-O(2)	1.6668(11)	C(13)-H(13C)	0.9800
P(2)-C(19)	1.8540(16)	C(14)-H(14A)	0.9800
P(2)-C(15)	1.8602(15)	C(14)-H(14B)	0.9800
O(1)-C(2)	1.3873(17)	C(14)-H(14C)	0.9800
O(2)-C(6)	1.3932(17)	C(15)-C(18)	1.523(2)
C(1)-C(6)	1.3860(19)	C(15)-C(17)	1.534(2)
C(1)-C(2)	1.392(2)	C(15)-C(16)	1.536(2)
C(2)-C(3)	1.380(2)	C(16)-H(16A)	0.9800
C(3)-C(4)	1.396(2)	C(16)-H(16B)	0.9800
C(3)-H(3)	0.9500	C(16)-H(16C)	0.9800
C(4)-O(3)	1.3713(18)	C(17)-H(17A)	0.9800
C(4)-C(5)	1.391(2)	C(17)-H(17B)	0.9800
C(5)-C(6)	1.3909(19)	C(17)-H(17C)	0.9800
C(5)-H(5)	0.9500	C(18)-H(18A)	0.9800
C(7)-C(9)	1.520(2)	C(18)-H(18B)	0.9800
C(7)-C(8)	1.533(2)	C(18)-H(18C)	0.9800
C(7)-C(10)	1.535(2)	C(19)-C(22)	1.530(2)
C(8)-H(8A)	0.9800	C(19)-C(21)	1.535(2)
C(8)-H(8B)	0.9800	C(19)-C(20)	1.538(2)
C(8)-H(8C)	0.9800	C(20)-H(20A)	0.9800
C(9)-H(9A)	0.9800	C(20)-H(20B)	0.9800
C(9)-H(9B)	0.9800	C(20)-H(20C)	0.9800
C(9)-H(9C)	0.9800	C(21)-H(21A)	0.9800
C(10)-H(10A)	0.9800	C(21)-H(21B)	0.9800
C(10)-H(10B)	0.9800	C(21)-H(21C)	0.9800
C(10)-H(10C)	0.9800	C(22)-H(22A)	0.9800
C(11)-C(14)	1.522(2)	C(22)-H(22B)	0.9800

C(22)-H(22C)	0.9800	C(5)-C(4)-C(3)	121.70(13)
O(3)-C(23)	1.424(2)	C(6)-C(5)-C(4)	117.26(13)
C(23)-H(23A)	0.9800	C(6)-C(5)-H(5)	121.4
C(23)-H(23B)	0.9800	C(4)-C(5)-H(5)	121.4
C(23)-H(23C)	0.9800	C(1)-C(6)-C(5)	123.82(13)
C(1)-Ni(1)-P(1)	82.77(5)	C(1)-C(6)-O(2)	117.06(12)
C(1)-Ni(1)-P(2)	82.53(5)	C(5)-C(6)-O(2)	119.12(12)
P(1)-Ni(1)-P(2)	165.288(17)	C(9)-C(7)-C(8)	109.76(15)
C(1)-Ni(1)-H(1)	177.9(8)	C(9)-C(7)-C(10)	110.08(14)
P(1)-Ni(1)-H(1)	95.7(8)	C(8)-C(7)-C(10)	107.49(14)
P(2)-Ni(1)-H(1)	99.0(8)	C(9)-C(7)-P(1)	111.20(12)
O(1)-P(1)-C(7)	99.86(7)	C(8)-C(7)-P(1)	104.33(11)
O(1)-P(1)-C(11)	100.45(7)	C(10)-C(7)-P(1)	113.72(11)
C(7)-P(1)-C(11)	113.75(7)	C(7)-C(8)-H(8A)	109.5
O(1)-P(1)-Ni(1)	106.59(4)	C(7)-C(8)-H(8B)	109.5
C(7)-P(1)-Ni(1)	117.13(5)	H(8A)-C(8)-H(8B)	109.5
C(11)-P(1)-Ni(1)	115.77(5)	C(7)-C(8)-H(8C)	109.5
O(2)-P(2)-C(19)	100.81(7)	H(8A)-C(8)-H(8C)	109.5
O(2)-P(2)-C(15)	100.32(6)	H(8B)-C(8)-H(8C)	109.5
C(19)-P(2)-C(15)	113.84(7)	C(7)-C(9)-H(9A)	109.5
O(2)-P(2)-Ni(1)	106.78(4)	C(7)-C(9)-H(9B)	109.5
C(19)-P(2)-Ni(1)	116.71(5)	H(9A)-C(9)-H(9B)	109.5
C(15)-P(2)-Ni(1)	115.40(5)	C(7)-C(9)-H(9C)	109.5
C(2)-O(1)-P(1)	111.69(9)	H(9A)-C(9)-H(9C)	109.5
C(6)-O(2)-P(2)	111.09(8)	H(9B)-C(9)-H(9C)	109.5
C(6)-C(1)-C(2)	115.89(13)	C(7)-C(10)-H(10A)	109.5
C(6)-C(1)-Ni(1)	122.24(11)	C(7)-C(10)-H(10B)	109.5
C(2)-C(1)-Ni(1)	121.86(10)	H(10A)-C(10)-H(10B)	109.5
C(3)-C(2)-O(1)	119.36(13)	C(7)-C(10)-H(10C)	109.5
C(3)-C(2)-C(1)	123.60(13)	H(10A)-C(10)-H(10C)	109.5
O(1)-C(2)-C(1)	117.04(12)	H(10B)-C(10)-H(10C)	109.5
C(2)-C(3)-C(4)	117.71(13)	C(14)-C(11)-C(12)	109.90(15)
C(2)-C(3)-H(3)	121.1	C(14)-C(11)-C(13)	110.64(15)
C(4)-C(3)-H(3)	121.1	C(12)-C(11)-C(13)	108.35(15)
O(3)-C(4)-C(5)	123.64(13)	C(14)-C(11)-P(1)	113.44(12)
O(3)-C(4)-C(3)	114.66(13)	C(12)-C(11)-P(1)	104.66(11)

C(13)-C(11)-P(1)	109.58(12)	H(17B)-C(17)-H(17C)	109.5
C(11)-C(12)-H(12A)	109.5	C(15)-C(18)-H(18A)	109.5
C(11)-C(12)-H(12B)	109.5	C(15)-C(18)-H(18B)	109.5
H(12A)-C(12)-H(12B)	109.5	H(18A)-C(18)-H(18B)	109.5
C(11)-C(12)-H(12C)	109.5	C(15)-C(18)-H(18C)	109.5
H(12A)-C(12)-H(12C)	109.5	H(18A)-C(18)-H(18C)	109.5
H(12B)-C(12)-H(12C)	109.5	H(18B)-C(18)-H(18C)	109.5
C(11)-C(13)-H(13A)	109.5	C(22)-C(19)-C(21)	110.57(13)
C(11)-C(13)-H(13B)	109.5	C(22)-C(19)-C(20)	108.62(13)
H(13A)-C(13)-H(13B)	109.5	C(21)-C(19)-C(20)	108.55(13)
C(11)-C(13)-H(13C)	109.5	C(22)-C(19)-P(2)	114.90(11)
H(13A)-C(13)-H(13C)	109.5	C(21)-C(19)-P(2)	109.58(11)
H(13B)-C(13)-H(13C)	109.5	C(20)-C(19)-P(2)	104.28(10)
C(11)-C(14)-H(14A)	109.5	C(19)-C(20)-H(20A)	109.5
C(11)-C(14)-H(14B)	109.5	C(19)-C(20)-H(20B)	109.5
H(14A)-C(14)-H(14B)	109.5	H(20A)-C(20)-H(20B)	109.5
C(11)-C(14)-H(14C)	109.5	C(19)-C(20)-H(20C)	109.5
H(14A)-C(14)-H(14C)	109.5	H(20A)-C(20)-H(20C)	109.5
H(14B)-C(14)-H(14C)	109.5	H(20B)-C(20)-H(20C)	109.5
C(18)-C(15)-C(17)	110.17(14)	C(19)-C(21)-H(21A)	109.5
C(18)-C(15)-C(16)	109.35(13)	C(19)-C(21)-H(21B)	109.5
C(17)-C(15)-C(16)	108.20(13)	H(21A)-C(21)-H(21B)	109.5
C(18)-C(15)-P(2)	114.01(11)	C(19)-C(21)-H(21C)	109.5
C(17)-C(15)-P(2)	109.30(11)	H(21A)-C(21)-H(21C)	109.5
C(16)-C(15)-P(2)	105.58(10)	H(21B)-C(21)-H(21C)	109.5
C(15)-C(16)-H(16A)	109.5	C(19)-C(22)-H(22A)	109.5
C(15)-C(16)-H(16B)	109.5	C(19)-C(22)-H(22B)	109.5
H(16A)-C(16)-H(16B)	109.5	H(22A)-C(22)-H(22B)	109.5
C(15)-C(16)-H(16C)	109.5	C(19)-C(22)-H(22C)	109.5
H(16A)-C(16)-H(16C)	109.5	H(22A)-C(22)-H(22C)	109.5
H(16B)-C(16)-H(16C)	109.5	H(22B)-C(22)-H(22C)	109.5
C(15)-C(17)-H(17A)	109.5	C(4)-O(3)-C(23)	117.24(13)
C(15)-C(17)-H(17B)	109.5	O(3)-C(23)-H(23A)	109.5
H(17A)-C(17)-H(17B)	109.5	O(3)-C(23)-H(23B)	109.5
C(15)-C(17)-H(17C)	109.5	H(23A)-C(23)-H(23B)	109.5
H(17A)-C(17)-H(17C)	109.5	O(3)-C(23)-H(23C)	109.5

H(23A)-C(23)-H(23C)	109.5	H(23B)-C(23)-H(23C)	109.5

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for **3b**. 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}
Ni1	14(1)	11(1)	15(1)	1(1)	6(1)	1(1)
P1	13(1)	12(1)	16(1)	-1(1)	5(1)	1(1)
P2	13(1)	10(1)	14(1)	0(1)	4(1)	0(1)
O1	20(1)	13(1)	26(1)	3(1)	12(1)	2(1)
O2	15(1)	12(1)	18(1)	2(1)	7(1)	1(1)
C1	14(1)	12(1)	16(1)	1(1)	4(1)	0(1)
C2	14(1)	13(1)	18(1)	1(1)	4(1)	1(1)
C3	18(1)	12(1)	19(1)	3(1)	4(1)	-1(1)
C4	17(1)	16(1)	16(1)	2(1)	4(1)	-3(1)
C5	14(1)	16(1)	18(1)	0(1)	7(1)	-2(1)
C6	12(1)	12(1)	16(1)	0(1)	3(1)	-1(1)
C7	14(1)	19(1)	22(1)	-3(1)	3(1)	0(1)
C8	24(1)	35(1)	32(1)	-16(1)	-2(1)	4(1)
C9	21(1)	45(1)	40(1)	8(1)	10(1)	-6(1)
C10	21(1)	27(1)	32(1)	-2(1)	-1(1)	7(1)
C11	18(1)	23(1)	20(1)	-7(1)	2(1)	4(1)
C12	20(1)	38(1)	38(1)	-20(1)	-1(1)	-2(1)
C13	38(1)	44(1)	18(1)	-2(1)	6(1)	7(1)
C14	36(1)	28(1)	34(1)	-17(1)	-3(1)	11(1)
C15	16(1)	14(1)	19(1)	-2(1)	5(1)	-2(1)
C16	25(1)	23(1)	19(1)	-1(1)	0(1)	0(1)
C17	27(1)	26(1)	28(1)	-3(1)	10(1)	-11(1)
C18	24(1)	20(1)	31(1)	-11(1)	5(1)	1(1)
C19	16(1)	16(1)	17(1)	1(1)	3(1)	1(1)
C20	23(1)	21(1)	17(1)	-1(1)	1(1)	-2(1)
C21	24(1)	19(1)	24(1)	8(1)	4(1)	2(1)
C22	16(1)	27(1)	26(1)	-2(1)	3(1)	5(1)
O3	29(1)	17(1)	24(1)	5(1)	14(1)	-2(1)
C23	30(1)	27(1)	29(1)	5(1)	16(1)	0(1)

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

	x	y	z	U(eq)
H1	10530(20)	2929(10)	1874(14)	26
H3	9351	374	4286	20
H5	6830	1962	4727	19
H8A	14142	2123	4222	48
H8B	12592	1901	4295	48
H8C	12790	2559	3655	48
H9A	14759	2037	2601	52
H9B	13321	2398	2011	52
H9C	13723	1647	1696	52
H10A	14649	993	3644	42
H10B	13553	624	2748	42
H10C	13151	734	3776	42
H12A	8624	800	360	50
H12B	8487	1471	993	50
H12C	8774	735	1519	50
H13A	10431	1412	-260	50
H13B	11861	1680	478	50
H13C	10398	2069	424	50
H14A	10938	221	576	52
H14B	11187	177	1746	52
H14C	12339	543	1282	52
H16A	10683	3896	5134	35
H16B	10803	3221	4505	35
H16C	9465	3323	4957	35
H17A	10999	4624	3774	40
H17B	9967	4555	2694	40
H17C	11095	3961	3115	40
H18A	8842	4758	4491	38
H18B	7589	4206	4192	38
H18C	7897	4722	3380	38

H20A	5789	3032	623	32
H20B	6204	2560	1589	32
H20C	7353	2719	980	32
H21A	6907	4155	591	35
H21B	8486	3904	1090	35
H21C	7798	4515	1580	35
H22A	4985	3937	1560	36
H22B	6027	4277	2508	36
H22C	5435	3514	2571	36
H23A	6571	592	6289	41
H23B	5802	1089	5405	41
H23C	7204	1341	6186	41

Table 6. Torsion angles [°] for **3b**.

C7-P1-O1-C2	124.62(10)	C11-P1-C7-C8	173.87(12)
C11-P1-O1-C2	-118.79(10)	Ni1-P1-C7-C8	34.48(13)
Ni1-P1-O1-C2	2.29(10)	O1-P1-C7-C10	36.79(13)
C19-P2-O2-C6	127.95(10)	C11-P1-C7-C10	-69.32(14)
C15-P2-O2-C6	-115.11(10)	Ni1-P1-C7-C10	151.29(10)
Ni1-P2-O2-C6	5.58(10)	O1-P1-C11-C14	-58.78(14)
P1-Ni1-C1-C6	-179.72(12)	C7-P1-C11-C14	46.96(16)
P2-Ni1-C1-C6	0.97(11)	Ni1-P1-C11-C14	-173.07(12)
P1-Ni1-C1-C2	1.72(12)	O1-P1-C11-C12	61.02(13)
P2-Ni1-C1-C2	-177.60(13)	C7-P1-C11-C12	166.77(12)
P1-O1-C2-C3	179.30(11)	Ni1-P1-C11-C12	-53.27(13)
P1-O1-C2-C1	-1.25(17)	O1-P1-C11-C13	177.02(11)
C6-C1-C2-C3	0.1(2)	C7-P1-C11-C13	-77.23(13)
Ni1-C1-C2-C3	178.75(12)	Ni1-P1-C11-C13	62.73(12)
C6-C1-C2-O1	-179.33(12)	O2-P2-C15-C18	-65.18(12)
Ni1-C1-C2-O1	-0.68(19)	C19-P2-C15-C18	41.61(14)
O1-C2-C3-C4	178.25(13)	Ni1-P2-C15-C18	-179.47(10)
C1-C2-C3-C4	-1.2(2)	O2-P2-C15-C17	171.05(11)
C2-C3-C4-O3	-178.59(13)	C19-P2-C15-C17	-82.16(12)
C2-C3-C4-C5	0.8(2)	Ni1-P2-C15-C17	56.76(12)
O3-C4-C5-C6	179.93(14)	O2-P2-C15-C16	54.88(11)
C3-C4-C5-C6	0.6(2)	C19-P2-C15-C16	161.67(10)
C2-C1-C6-C5	1.4(2)	Ni1-P2-C15-C16	-59.42(11)
Ni1-C1-C6-C5	-177.21(12)	O2-P2-C19-C22	41.82(12)
C2-C1-C6-O2	-178.81(12)	C15-P2-C19-C22	-64.67(13)
Ni1-C1-C6-O2	2.54(18)	Ni1-P2-C19-C22	156.98(10)
C4-C5-C6-C1	-1.8(2)	O2-P2-C19-C21	167.01(10)
C4-C5-C6-O2	178.47(13)	C15-P2-C19-C21	60.52(12)
P2-O2-C6-C1	-5.37(15)	Ni1-P2-C19-C21	-77.84(11)
P2-O2-C6-C5	174.39(11)	O2-P2-C19-C20	-76.96(10)
O1-P1-C7-C9	161.73(12)	C15-P2-C19-C20	176.56(9)
C11-P1-C7-C9	55.61(15)	Ni1-P2-C19-C20	38.20(11)
Ni1-P1-C7-C9	-83.77(13)	C5-C4-O3-C23	-10.1(2)
O1-P1-C7-C8	-80.02(13)	C3-C4-O3-C23	169.25(14)

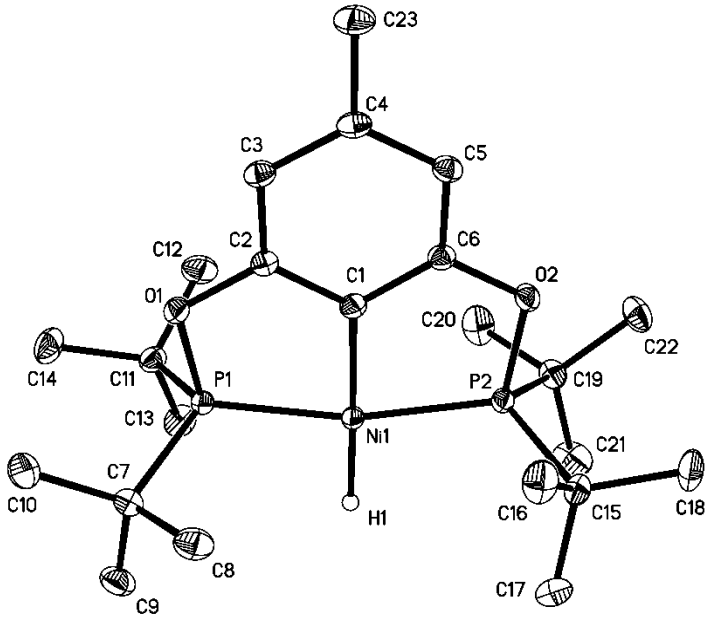
CRYSTAL STRUCTURE REPORT

$C_{23}H_{42}NiO_2P_2$ (**3c**)

Report prepared for:

Dr. S. Chakraborty, Prof. W. D. Jones

September 28, 2013



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Data collection

A crystal (0.32 x 0.14 x 0.10 mm^3) was placed onto the tip of a 0.1 mm diameter glass capillary tube and mounted on a Bruker SMART APEX II CCD platform diffractometer for a data collection at 100.0(5) K.[1] A preliminary set of cell constants and an orientation matrix were calculated from reflections harvested from three orthogonal wedges of reciprocal space. The full data collection was carried out using MoKα radiation (graphite monochromator) with a frame time of 45 seconds and a detector distance of 4.02 cm. A randomly oriented region of reciprocal space was surveyed: six major sections of frames were collected with 0.50º steps in ω at six different ϕ settings and a detector position of -38º in 2θ. The intensity data were corrected for absorption.[2] Final cell constants were calculated from the xyz centroids of 4012 strong reflections from the actual data collection after integration.[3] See Table 1 for additional crystal and refinement information.

Structure solution and refinement

The structure was solved using SIR2011[4] and refined using SHELXL-2013.[5] The space group $P2_1/n$ was determined based on systematic absences. A direct-methods solution was calculated which provided most non-hydrogen atoms from the E-map. Full-matrix least squares / difference Fourier cycles were performed which located the remaining non-hydrogen atoms. All non-hydrogen atoms were refined with anisotropic displacement parameters. The hydride ligand was found from the difference Fourier map; its position was refined freely and its isotropic displacement parameter was refined relative to that of the nickel atom: U_{iso}[H] = 2*U_{eq}[Ni1]. All other hydrogen atoms were placed in ideal positions and refined as riding atoms with relative isotropic displacement parameters. The final full matrix least squares refinement converged to $R1 = 0.0571$ (F^2, $I > 2\sigma(I)$) and $wR2 = 0.1264$ (F^2, all data).

Structure description

The structure is the one suggested. There is one molecule in the asymmetric unit in a general position.

Unless noted otherwise all structural diagrams containing thermal displacement ellipsoids are drawn at the 50 % probability level.

Data collection, structure solution, and structure refinement were conducted at the X-ray Crystallographic Facility, B51 Hutchison Hall, Department of Chemistry, University of Rochester. All publications arising from this report MUST either 1) include William W. Brennessel as a coauthor or 2) acknowledge William W. Brennessel and the X-ray Crystallographic Facility of the Department of Chemistry at the University of Rochester.

[1] *APEX2*, version 2013.2-0; Bruker AXS: Madison, WI, 2013.

[2] Sheldrick, G. M. *SADABS*, version 2012/1; University of Göttingen: Göttingen, Germany, 2012.

[3] *SAINT*, version 8.27B; Bruker AXS: Madison, WI, 2013.

[4] Burla, M. C.; Caliandro, R.; Camalli, M.; Carrozzini, B.; Cascarano, G. L.; Giacovazzo, C.; Mallamo, M.; Mazzone, A.; Polidori, G.; Spagna, R. *SIR2011: a new package for crystal structure determination and refinement*, version 1.0; Istituto di Cristallografia: Bari, Italy, 2012.

[5] Sheldrick, G. M. *SHELXL-2013/4*; University of Göttingen: Göttingen, Germany, 2013.

Some equations of interest:

$$R_{int} = \Sigma\, |F_o^2 - < F_o^2 >| \,/\, \Sigma |F_o^2|$$

$$R1 = \Sigma ||F_o| - |F_c|| \,/\, \Sigma |F_o|$$

$$wR2 = [\Sigma [w(F_o^2 - F_c^2)^2] \,/\, \Sigma\, [w(F_o^2)^2]]^{1/2}$$

where $w = 1 \,/\, [\sigma^2 (F_o^2) + (aP)^2 + bP]$ and

$$P = 1/3 \max (0, F_o^2) + 2/3\, F_c^2$$

$$\text{GOF} = S = [\Sigma [w(F_o^2 - F_c^2)^2] \,/\, (m-n)]^{1/2}$$

where m = number of reflections and n = number of parameters

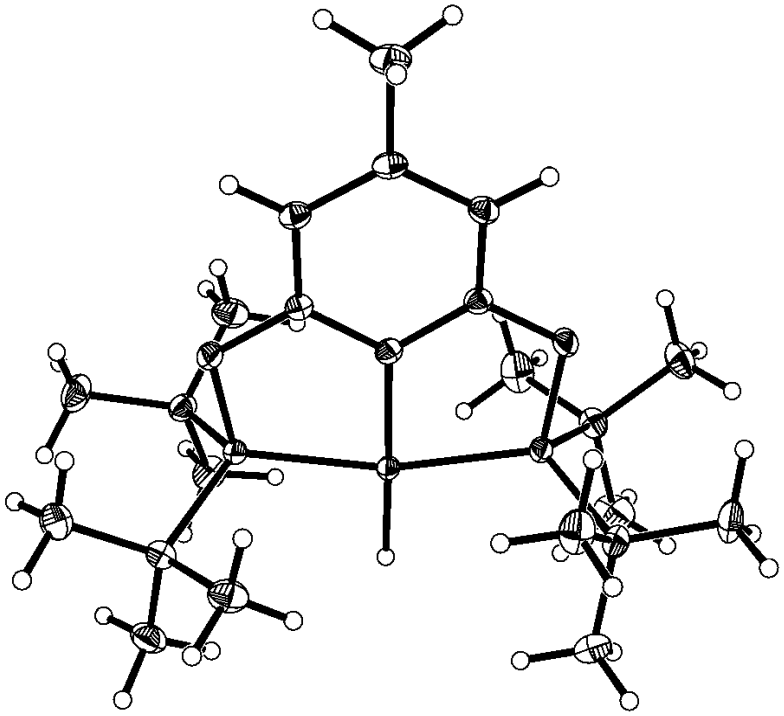


Table 1. Crystal data and structure refinement for **3c**.

Empirical formula	$C_{23}H_{42}NiO_2P_2$	
Formula weight	471.21	
Temperature	100.0(5) K	
Wavelength	0.71073 Å	
Crystal system	monoclinic	
Space group	$P2_1/n$	
Unit cell dimensions	a = 9.528(3) Å	α = 90°
	b = 20.201(7) Å	β = 100.908(7)°
	c = 13.434(5) Å	γ = 90°
Volume	2539.1(16) Å^3	
Z	4	
Density (calculated)	1.233 Mg/m^3	
Absorption coefficient	0.905 mm^{-1}	
F(000)	1016	
Crystal color, morphology	yellow, needle	
Crystal size	0.32 x 0.14 x 0.10 mm^3	
Theta range for data collection	1.844 to 38.629°	
Index ranges	$-16 \le h \le 16$, $-35 \le k \le 35$, $-23 \le l \le 23$	
Reflections collected	80520	
Independent reflections	13995 [R(int) = 0.0707]	
Observed reflections	10209	
Completeness to theta = 37.785°	99.7%	
Absorption correction	Multi-scan	
Max. and min. transmission	0.7476 and 0.6748	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	13995 / 0 / 269	
Goodness-of-fit on F^2	1.134	
Final R indices [I>2sigma(I)]	$R1$ = 0.0571, $wR2$ = 0.1174	
R indices (all data)	$R1$ = 0.0868, $wR2$ = 0.1264	
Largest diff. peak and hole	2.316 and -0.759 e.Å^{-3}	

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

	x	y	z	U_{eq}
Ni1	3824(1)	2410(1)	9504(1)	12(1)
P1	5117(1)	3185(1)	9120(1)	12(1)
P2	2716(1)	1510(1)	9585(1)	13(1)
O1	6072(1)	2867(1)	8314(1)	15(1)
O2	3148(1)	1014(1)	8696(1)	16(1)
C1	4618(2)	1940(1)	8528(1)	13(1)
C2	5666(2)	2216(1)	8056(1)	13(1)
C3	6295(2)	1876(1)	7356(1)	15(1)
C4	5883(2)	1221(1)	7120(1)	15(1)
C5	4827(2)	926(1)	7568(1)	15(1)
C6	4217(2)	1293(1)	8250(1)	14(1)
C7	6583(2)	3488(1)	10128(1)	17(1)
C8	7091(2)	2866(1)	10751(2)	22(1)
C9	5993(2)	3982(1)	10812(2)	23(1)
C10	7855(2)	3784(1)	9732(2)	28(1)
C11	4131(2)	3857(1)	8335(1)	16(1)
C12	3337(2)	3508(1)	7375(2)	24(1)
C13	3008(2)	4158(1)	8886(2)	23(1)
C14	5088(2)	4398(1)	8032(2)	26(1)
C15	3328(2)	996(1)	10734(1)	18(1)
C16	4920(2)	861(1)	10735(2)	26(1)
C17	3228(3)	1398(1)	11687(2)	29(1)
C18	2536(2)	335(1)	10730(2)	25(1)
C19	743(2)	1543(1)	9177(1)	18(1)
C20	480(2)	2096(1)	8375(2)	29(1)
C21	39(2)	1739(1)	10067(2)	31(1)
C22	84(2)	903(1)	8691(2)	25(1)
C23	6594(2)	832(1)	6394(2)	22(1)

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

Ni(1)-C(1)	1.8885(16)	C(11)-C(13)	1.537(3)
Ni(1)-P(1)	2.1155(7)	C(11)-C(12)	1.538(3)
Ni(1)-P(2)	2.1155(7)	C(12)-H(12A)	0.9800
Ni(1)-H(1)	1.45(3)	C(12)-H(12B)	0.9800
P(1)-O(1)	1.6689(13)	C(12)-H(12C)	0.9800
P(1)-C(7)	1.8563(19)	C(13)-H(13A)	0.9800
P(1)-C(11)	1.8614(19)	C(13)-H(13B)	0.9800
P(2)-O(2)	1.6693(13)	C(13)-H(13C)	0.9800
P(2)-C(19)	1.858(2)	C(14)-H(14A)	0.9800
P(2)-C(15)	1.8597(19)	C(14)-H(14B)	0.9800
O(1)-C(2)	1.396(2)	C(14)-H(14C)	0.9800
O(2)-C(6)	1.394(2)	C(15)-C(18)	1.533(3)
C(1)-C(6)	1.393(2)	C(15)-C(17)	1.534(3)
C(1)-C(2)	1.395(2)	C(15)-C(16)	1.541(3)
C(2)-C(3)	1.388(2)	C(16)-H(16A)	0.9800
C(3)-C(4)	1.400(3)	C(16)-H(16B)	0.9800
C(3)-H(3)	0.9500	C(16)-H(16C)	0.9800
C(4)-C(5)	1.400(2)	C(17)-H(17A)	0.9800
C(4)-C(23)	1.509(2)	C(17)-H(17B)	0.9800
C(5)-C(6)	1.389(2)	C(17)-H(17C)	0.9800
C(5)-H(5)	0.9500	C(18)-H(18A)	0.9800
C(7)-C(10)	1.534(3)	C(18)-H(18B)	0.9800
C(7)-C(9)	1.534(3)	C(18)-H(18C)	0.9800
C(7)-C(8)	1.537(3)	C(19)-C(21)	1.529(3)
C(8)-H(8A)	0.9800	C(19)-C(22)	1.529(3)
C(8)-H(8B)	0.9800	C(19)-C(20)	1.540(3)
C(8)-H(8C)	0.9800	C(20)-H(20A)	0.9800
C(9)-H(9A)	0.9800	C(20)-H(20B)	0.9800
C(9)-H(9B)	0.9800	C(20)-H(20C)	0.9800
C(9)-H(9C)	0.9800	C(21)-H(21A)	0.9800
C(10)-H(10A)	0.9800	C(21)-H(21B)	0.9800
C(10)-H(10B)	0.9800	C(21)-H(21C)	0.9800
C(10)-H(10C)	0.9800	C(22)-H(22A)	0.9800
C(11)-C(14)	1.526(3)	C(22)-H(22B)	0.9800

C(22)-H(22C)	0.9800	C(6)-C(5)-C(4)	118.77(16)
C(23)-H(23A)	0.9800	C(6)-C(5)-H(5)	120.6
C(23)-H(23B)	0.9800	C(4)-C(5)-H(5)	120.6
C(23)-H(23C)	0.9800	C(5)-C(6)-C(1)	123.29(15)
C(1)-Ni(1)-P(1)	82.77(6)	C(5)-C(6)-O(2)	119.94(15)
C(1)-Ni(1)-P(2)	82.75(6)	C(1)-C(6)-O(2)	116.76(14)
P(1)-Ni(1)-P(2)	165.46(2)	C(10)-C(7)-C(9)	110.95(17)
C(1)-Ni(1)-H(1)	175.9(11)	C(10)-C(7)-C(8)	108.65(17)
P(1)-Ni(1)-H(1)	95.9(11)	C(9)-C(7)-C(8)	108.64(15)
P(2)-Ni(1)-H(1)	98.6(11)	C(10)-C(7)-P(1)	114.23(13)
O(1)-P(1)-C(7)	99.97(8)	C(9)-C(7)-P(1)	109.95(13)
O(1)-P(1)-C(11)	101.04(8)	C(8)-C(7)-P(1)	104.07(13)
C(7)-P(1)-C(11)	113.68(9)	C(7)-C(8)-H(8A)	109.5
O(1)-P(1)-Ni(1)	106.72(5)	C(7)-C(8)-H(8B)	109.5
C(7)-P(1)-Ni(1)	117.33(6)	H(8A)-C(8)-H(8B)	109.5
C(11)-P(1)-Ni(1)	115.06(6)	C(7)-C(8)-H(8C)	109.5
O(2)-P(2)-C(19)	100.56(8)	H(8A)-C(8)-H(8C)	109.5
O(2)-P(2)-C(15)	100.27(8)	H(8B)-C(8)-H(8C)	109.5
C(19)-P(2)-C(15)	113.95(9)	C(7)-C(9)-H(9A)	109.5
O(2)-P(2)-Ni(1)	106.60(5)	C(7)-C(9)-H(9B)	109.5
C(19)-P(2)-Ni(1)	116.15(6)	H(9A)-C(9)-H(9B)	109.5
C(15)-P(2)-Ni(1)	116.20(6)	C(7)-C(9)-H(9C)	109.5
C(2)-O(1)-P(1)	111.32(10)	H(9A)-C(9)-H(9C)	109.5
C(6)-O(2)-P(2)	111.32(11)	H(9B)-C(9)-H(9C)	109.5
C(6)-C(1)-C(2)	115.90(15)	C(7)-C(10)-H(10A)	109.5
C(6)-C(1)-Ni(1)	122.10(12)	C(7)-C(10)-H(10B)	109.5
C(2)-C(1)-Ni(1)	121.99(13)	H(10A)-C(10)-H(10B)	109.5
C(3)-C(2)-C(1)	123.26(16)	C(7)-C(10)-H(10C)	109.5
C(3)-C(2)-O(1)	119.88(14)	H(10A)-C(10)-H(10C)	109.5
C(1)-C(2)-O(1)	116.87(14)	H(10B)-C(10)-H(10C)	109.5
C(2)-C(3)-C(4)	118.81(15)	C(14)-C(11)-C(13)	110.59(16)
C(2)-C(3)-H(3)	120.6	C(14)-C(11)-C(12)	109.21(16)
C(4)-C(3)-H(3)	120.6	C(13)-C(11)-C(12)	107.95(16)
C(5)-C(4)-C(3)	119.94(15)	C(14)-C(11)-P(1)	114.22(14)
C(5)-C(4)-C(23)	120.19(16)	C(13)-C(11)-P(1)	109.75(12)
C(3)-C(4)-C(23)	119.87(16)	C(12)-C(11)-P(1)	104.81(13)

C(11)-C(12)-H(12A)	109.5	C(15)-C(18)-H(18A)	109.5
C(11)-C(12)-H(12B)	109.5	C(15)-C(18)-H(18B)	109.5
H(12A)-C(12)-H(12B)	109.5	H(18A)-C(18)-H(18B)	109.5
C(11)-C(12)-H(12C)	109.5	C(15)-C(18)-H(18C)	109.5
H(12A)-C(12)-H(12C)	109.5	H(18A)-C(18)-H(18C)	109.5
H(12B)-C(12)-H(12C)	109.5	H(18B)-C(18)-H(18C)	109.5
C(11)-C(13)-H(13A)	109.5	C(21)-C(19)-C(22)	110.34(17)
C(11)-C(13)-H(13B)	109.5	C(21)-C(19)-C(20)	108.82(18)
H(13A)-C(13)-H(13B)	109.5	C(22)-C(19)-C(20)	108.33(17)
C(11)-C(13)-H(13C)	109.5	C(21)-C(19)-P(2)	110.37(14)
H(13A)-C(13)-H(13C)	109.5	C(22)-C(19)-P(2)	113.96(13)
H(13B)-C(13)-H(13C)	109.5	C(20)-C(19)-P(2)	104.75(13)
C(11)-C(14)-H(14A)	109.5	C(19)-C(20)-H(20A)	109.5
C(11)-C(14)-H(14B)	109.5	C(19)-C(20)-H(20B)	109.5
H(14A)-C(14)-H(14B)	109.5	H(20A)-C(20)-H(20B)	109.5
C(11)-C(14)-H(14C)	109.5	C(19)-C(20)-H(20C)	109.5
H(14A)-C(14)-H(14C)	109.5	H(20A)-C(20)-H(20C)	109.5
H(14B)-C(14)-H(14C)	109.5	H(20B)-C(20)-H(20C)	109.5
C(18)-C(15)-C(17)	110.86(16)	C(19)-C(21)-H(21A)	109.5
C(18)-C(15)-C(16)	109.23(17)	C(19)-C(21)-H(21B)	109.5
C(17)-C(15)-C(16)	108.06(18)	H(21A)-C(21)-H(21B)	109.5
C(18)-C(15)-P(2)	113.95(14)	C(19)-C(21)-H(21C)	109.5
C(17)-C(15)-P(2)	109.75(14)	H(21A)-C(21)-H(21C)	109.5
C(16)-C(15)-P(2)	104.66(13)	H(21B)-C(21)-H(21C)	109.5
C(15)-C(16)-H(16A)	109.5	C(19)-C(22)-H(22A)	109.5
C(15)-C(16)-H(16B)	109.5	C(19)-C(22)-H(22B)	109.5
H(16A)-C(16)-H(16B)	109.5	H(22A)-C(22)-H(22B)	109.5
C(15)-C(16)-H(16C)	109.5	C(19)-C(22)-H(22C)	109.5
H(16A)-C(16)-H(16C)	109.5	H(22A)-C(22)-H(22C)	109.5
H(16B)-C(16)-H(16C)	109.5	H(22B)-C(22)-H(22C)	109.5
C(15)-C(17)-H(17A)	109.5	C(4)-C(23)-H(23A)	109.5
C(15)-C(17)-H(17B)	109.5	C(4)-C(23)-H(23B)	109.5
H(17A)-C(17)-H(17B)	109.5	H(23A)-C(23)-H(23B)	109.5
C(15)-C(17)-H(17C)	109.5	C(4)-C(23)-H(23C)	109.5
H(17A)-C(17)-H(17C)	109.5	H(23A)-C(23)-H(23C)	109.5
H(17B)-C(17)-H(17C)	109.5	H(23B)-C(23)-H(23C)	109.5

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for **3c**. 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}
Ni1	15(1)	10(1)	12(1)	-1(1)	5(1)	-1(1)
P1	14(1)	11(1)	12(1)	0(1)	3(1)	-1(1)
P2	14(1)	12(1)	14(1)	-1(1)	5(1)	-1(1)
O1	17(1)	14(1)	15(1)	-1(1)	7(1)	-2(1)
O2	18(1)	13(1)	18(1)	-4(1)	8(1)	-3(1)
C1	13(1)	14(1)	12(1)	0(1)	4(1)	1(1)
C2	14(1)	14(1)	12(1)	0(1)	3(1)	1(1)
C3	14(1)	18(1)	14(1)	-1(1)	4(1)	1(1)
C4	15(1)	18(1)	13(1)	-1(1)	3(1)	4(1)
C5	17(1)	13(1)	14(1)	-2(1)	3(1)	2(1)
C6	14(1)	14(1)	13(1)	-1(1)	4(1)	1(1)
C7	17(1)	18(1)	15(1)	-2(1)	2(1)	-2(1)
C8	23(1)	23(1)	18(1)	0(1)	-1(1)	4(1)
C9	30(1)	19(1)	20(1)	-6(1)	2(1)	-1(1)
C10	21(1)	39(1)	24(1)	-3(1)	3(1)	-13(1)
C11	21(1)	15(1)	15(1)	2(1)	5(1)	1(1)
C12	26(1)	27(1)	17(1)	1(1)	-2(1)	3(1)
C13	24(1)	20(1)	25(1)	3(1)	8(1)	6(1)
C14	34(1)	17(1)	28(1)	7(1)	11(1)	-1(1)
C15	22(1)	16(1)	18(1)	4(1)	4(1)	-3(1)
C16	20(1)	26(1)	30(1)	8(1)	-1(1)	0(1)
C17	42(1)	30(1)	15(1)	1(1)	6(1)	-5(1)
C18	27(1)	18(1)	31(1)	8(1)	6(1)	-6(1)
C19	16(1)	17(1)	22(1)	-2(1)	6(1)	-1(1)
C20	22(1)	26(1)	35(1)	6(1)	-4(1)	0(1)
C21	22(1)	40(1)	35(1)	-11(1)	14(1)	0(1)
C22	20(1)	22(1)	32(1)	-6(1)	4(1)	-7(1)
C23	24(1)	24(1)	20(1)	-5(1)	10(1)	4(1)

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

	x	y	z	U(eq)
H1	3310(30)	2768(13)	10300(20)	24
H3	6994	2085	7044	18
H5	4533	483	7407	18
H8A	7885	2982	11301	33
H8B	7413	2534	10311	33
H8C	6301	2685	11038	33
H9A	6710	4061	11427	35
H9B	5124	3801	10999	35
H9C	5766	4401	10448	35
H10A	8640	3869	10302	42
H10B	7563	4201	9378	42
H10C	8172	3472	9262	42
H12A	2801	3835	6916	36
H12B	2675	3180	7562	36
H12C	4030	3285	7035	36
H13A	2462	4497	8457	34
H13B	3487	4358	9525	34
H13C	2358	3809	9028	34
H14A	4514	4697	7544	38
H14B	5837	4195	7723	38
H14C	5530	4648	8636	38
H16A	5339	617	11350	39
H16B	5424	1282	10717	39
H16C	5008	597	10137	39
H17A	3619	1138	12292	43
H17B	2226	1505	11689	43
H17C	3777	1809	11690	43
H18A	3007	63	11300	38
H18B	2550	102	10092	38
H18C	1544	418	10795	38

H20A	-550	2143	8121	43
H20B	957	1983	7812	43
H20C	864	2514	8681	43
H21A	-976	1832	9818	47
H21B	509	2134	10396	47
H21C	133	1374	10557	47
H22A	-952	959	8482	37
H22B	280	540	9182	37
H22C	501	799	8096	37
H23A	6898	1135	5906	33
H23B	5916	509	6032	33
H23C	7429	599	6773	33

Table 6. Torsion angles [°] for **3c**.

C7-P1-O1-C2	127.91(12)	C11-P1-C7-C9	56.47(15)
C11-P1-O1-C2	-115.36(12)	Ni1-P1-C7-C9	-81.89(14)
Ni1-P1-O1-C2	5.26(12)	O1-P1-C7-C8	-80.51(13)
C19-P2-O2-C6	128.74(12)	C11-P1-C7-C8	172.67(12)
C15-P2-O2-C6	-114.30(12)	Ni1-P1-C7-C8	34.31(14)
Ni1-P2-O2-C6	7.18(12)	O1-P1-C11-C14	-63.25(15)
P1-Ni1-C1-C6	-177.02(14)	C7-P1-C11-C14	42.90(16)
P2-Ni1-C1-C6	1.63(13)	Ni1-P1-C11-C14	-177.76(12)
P1-Ni1-C1-C2	4.40(13)	O1-P1-C11-C13	171.92(13)
P2-Ni1-C1-C2	-176.95(14)	C7-P1-C11-C13	-81.93(15)
C6-C1-C2-C3	-0.4(3)	Ni1-P1-C11-C13	57.41(14)
Ni1-C1-C2-C3	178.25(13)	O1-P1-C11-C12	56.22(13)
C6-C1-C2-O1	179.21(15)	C7-P1-C11-C12	162.37(12)
Ni1-C1-C2-O1	-2.1(2)	Ni1-P1-C11-C12	-58.29(13)
P1-O1-C2-C3	177.11(13)	O2-P2-C15-C18	-62.87(15)
P1-O1-C2-C1	-2.53(19)	C19-P2-C15-C18	43.64(17)
C1-C2-C3-C4	-1.1(3)	Ni1-P2-C15-C18	-177.25(12)
O1-C2-C3-C4	179.27(15)	O2-P2-C15-C17	172.14(14)
C2-C3-C4-C5	1.6(3)	C19-P2-C15-C17	-81.35(16)
C2-C3-C4-C23	-177.50(17)	Ni1-P2-C15-C17	57.76(15)
C3-C4-C5-C6	-0.6(3)	O2-P2-C15-C16	56.39(14)
C23-C4-C5-C6	178.53(17)	C19-P2-C15-C16	162.90(13)
C4-C5-C6-C1	-1.1(3)	Ni1-P2-C15-C16	-57.99(14)
C4-C5-C6-O2	179.05(15)	O2-P2-C19-C21	158.04(15)
C2-C1-C6-C5	1.5(3)	C15-P2-C19-C21	51.71(17)
Ni1-C1-C6-C5	-177.14(13)	Ni1-P2-C19-C21	-87.42(15)
C2-C1-C6-O2	-178.58(15)	O2-P2-C19-C22	33.24(16)
Ni1-C1-C6-O2	2.8(2)	C15-P2-C19-C22	-73.09(16)
P2-O2-C6-C5	173.28(13)	Ni1-P2-C19-C22	147.78(13)
P2-O2-C6-C1	-6.62(19)	O2-P2-C19-C20	-84.98(14)
O1-P1-C7-C10	37.81(16)	C15-P2-C19-C20	168.69(13)
C11-P1-C7-C10	-69.01(17)	Ni1-P2-C19-C20	29.55(15)
Ni1-P1-C7-C10	152.62(13)		
O1-P1-C7-C9	163.29(13)		

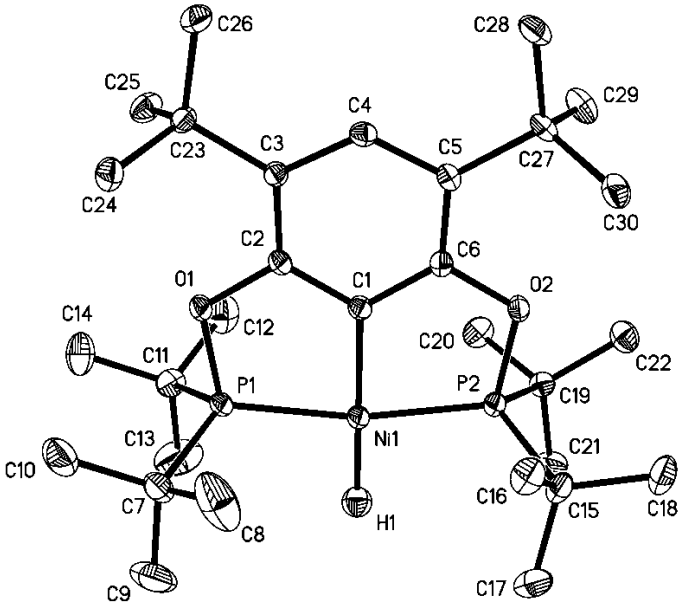
CRYSTAL STRUCTURE REPORT

$C_{30}H_{56}NiO_2P_2$ (**3d**)

Report prepared for:

Dr. S. Chakraborty, Prof. W. D. Jones

September 26, 2013



William W. Brennessel

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Data collection

A crystal (0.24 x 0.14 x 0.14 mm^3) was placed onto the tip of a 0.1 mm diameter glass capillary tube and mounted on a Bruker SMART APEX II CCD platform diffractometer for a data collection at 100.0(5) K.[1] A preliminary set of cell constants and an orientation matrix were calculated from reflections harvested from three orthogonal wedges of reciprocal space. The full data collection was carried out using MoKα radiation (graphite monochromator) with a frame time of 60 seconds and a detector distance of 4.01 cm. A randomly oriented region of reciprocal space was surveyed: four major sections of frames were collected with 0.50º steps in ω at four different ϕ settings and a detector position of -38º in 2θ. The intensity data were corrected for absorption.[2] Final cell constants were calculated from the xyz centroids of 3765 strong reflections from the actual data collection after integration.[3] See Table 1 for additional crystal and refinement information.

Structure solution and refinement

The structure was solved using SIR2011[4] and refined using SHELXL-2013.[5] The space group $P2_1/n$ was determined based on systematic absences. A direct-methods solution was calculated which provided most non-hydrogen atoms from the E-map. Full-matrix least squares / difference Fourier cycles were performed which located the remaining non-hydrogen atoms. All non-hydrogen atoms were refined with anisotropic displacement parameters. The hydride ligand was found from the difference Fourier map and refined freely. All other hydrogen atoms were placed in ideal positions and refined as riding atoms with relative isotropic displacement parameters. The final full matrix least squares refinement converged to $R1 = 0.0527$ (F^2, $I > 2\sigma(I)$) and $wR2 = 0.1271$ (F^2, all data).

Structure description

The structure is the one suggested. There is one molecule in the asymmetric unit in a general position.

Unless noted otherwise all structural diagrams containing thermal displacement ellipsoids are drawn at the 50 % probability level.

Data collection, structure solution, and structure refinement were conducted at the X-ray Crystallographic Facility, B51 Hutchison Hall, Department of Chemistry, University of Rochester. All publications arising from this report MUST either 1) include William W. Brennessel as a coauthor or 2) acknowledge William W. Brennessel and the X-ray Crystallographic Facility of the Department of Chemistry at the University of Rochester.

[1] *APEX2*, version 2013.2-0; Bruker AXS: Madison, WI, 2013.

[2] Sheldrick, G. M. *SADABS*, version 2012/1; University of Göttingen: Göttingen, Germany, 2012.

[3] *SAINT*, version 8.27B; Bruker AXS: Madison, WI, 2013.

[4] Burla, M. C.; Caliandro, R.; Camalli, M.; Carrozzini, B.; Cascarano, G. L.; Giacovazzo, C.; Mallamo, M.; Mazzone, A.; Polidori, G.; Spagna, R. *SIR2011: a new package for crystal structure determination and refinement*, version 1.0; Istituto di Cristallografia: Bari, Italy, 2012.

[5] Sheldrick, G. M. *SHELXL-2013/4* University of Göttingen: Göttingen, Germany, 2013.

Some equations of interest:

$$R_{int} = \Sigma\, |F_o^2 - < F_o^2 >| \,/\, \Sigma |F_o{}^2|$$

$$R1 = \Sigma ||F_o| - |F_c|| \,/\, \Sigma |F_o|$$

$$wR2 = [\Sigma [w(F_o^2 - F_c{}^2)^2] \,/\, \Sigma\, [w(F_o^2)^2]]^{1/2}$$

where $w = 1 \,/\, [\sigma^2 (F_o^2) + (aP)^2 + bP]$ and

$$P = 1/3 \max (0, F_o^2) + 2/3\, F_c^2$$

$$\text{GOF} = S = [\Sigma [w(F_o^2 - F_c{}^2)^2] \,/\, (m-n)]^{1/2}$$

where m = number of reflections and n = number of parameters

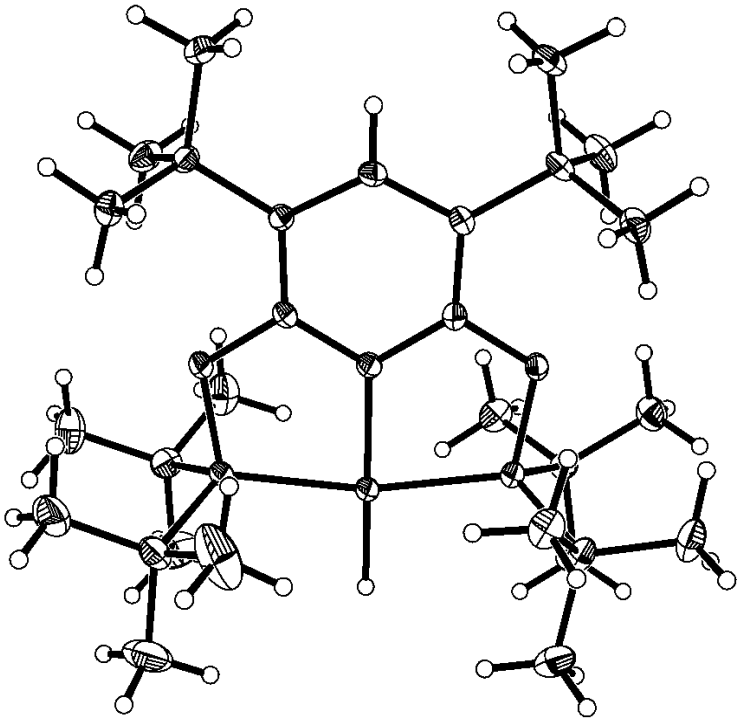


Table 1. Crystal data and structure refinement for **3d**.

Empirical formula	$C_{30}H_{56}NiO_2P_2$	
Formula weight	569.39	
Temperature	100.0(5) K	
Wavelength	0.71073 Å	
Crystal system	monoclinic	
Space group	$P2_1/n$	
Unit cell dimensions	a = 15.131(3) Å	α = 90°
	b = 12.589(2) Å	β = 106.717(4)°
	c = 17.529(3) Å	γ = 90°
Volume	3197.8(11) Å^3	
Z	4	
Density (calculated)	1.183 Mg/m^3	
Absorption coefficient	0.730 mm^{-1}	
F(000)	1240	
Crystal color, morphology	yellow, needle	
Crystal size	0.24 x 0.14 x 0.14 mm^3	
Theta range for data collection	1.570 to 35.102°	
Index ranges	$-24 \le h \le 24$, $-20 \le k \le 20$, $-28 \le l \le 27$	
Reflections collected	66747	
Independent reflections	14087 [R(int) = 0.0980]	
Observed reflections	9093	
Completeness to theta = 34.970°	99.8%	
Absorption correction	Multi-scan	
Max. and min. transmission	0.7469 and 0.6661	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	14087 / 0 / 338	
Goodness-of-fit on F^2	1.043	
Final R indices [I>2sigma(I)]	R1 = 0.0527, wR2 = 0.1088	
R indices (all data)	R1 = 0.0988, wR2 = 0.1271	
Largest diff. peak and hole	0.636 and -0.599 e.Å^{-3}	

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

	x	y	z	U_{eq}
Ni1	5016(1)	6333(1)	2695(1)	15(1)
P1	5924(1)	5025(1)	2874(1)	15(1)
P2	3924(1)	7423(1)	2305(1)	14(1)
O1	5462(1)	4178(1)	2142(1)	16(1)
O2	3197(1)	6902(1)	1498(1)	15(1)
C1	4311(1)	5521(1)	1820(1)	14(1)
C2	4620(1)	4539(1)	1632(1)	13(1)
C3	4142(1)	3924(1)	980(1)	13(1)
C4	3294(1)	4352(1)	530(1)	14(1)
C5	2923(1)	5321(1)	674(1)	14(1)
C6	3474(1)	5890(1)	1320(1)	14(1)
C7	5984(1)	4196(2)	3764(1)	23(1)
C8	4990(2)	4136(2)	3802(2)	51(1)
C9	6577(2)	4744(2)	4521(1)	45(1)
C10	6346(2)	3070(2)	3720(1)	36(1)
C11	7059(1)	5261(2)	2695(1)	21(1)
C12	6811(2)	5834(2)	1888(1)	32(1)
C13	7646(2)	6007(2)	3328(2)	45(1)
C14	7591(1)	4256(2)	2637(2)	34(1)
C15	3168(1)	7574(1)	2967(1)	20(1)
C16	2772(1)	6461(2)	3016(1)	24(1)
C17	3756(2)	7884(2)	3810(1)	28(1)
C18	2375(2)	8354(2)	2660(1)	28(1)
C19	4191(1)	8698(1)	1891(1)	17(1)
C20	4915(1)	8403(2)	1465(1)	24(1)
C21	4635(2)	9468(2)	2569(1)	25(1)
C22	3367(1)	9214(2)	1284(1)	24(1)
C23	4528(1)	2886(1)	752(1)	15(1)
C24	4731(1)	2088(1)	1443(1)	21(1)
C25	5420(1)	3123(2)	535(1)	22(1)
C26	3845(1)	2349(2)	36(1)	22(1)

C27	1997(1)	5740(1)	140(1)	16(1)
C28	1487(1)	4898(2)	-460(1)	22(1)
C29	2179(1)	6701(2)	-335(1)	25(1)
C30	1352(1)	6065(2)	633(1)	22(1)

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

Ni(1)-C(1)	1.8964(17)	C(11)-C(13)	1.528(3)
Ni(1)-P(2)	2.1045(6)	C(11)-C(12)	1.536(3)
Ni(1)-P(1)	2.1098(6)	C(12)-H(12A)	0.9800
Ni(1)-H(1)	1.54(2)	C(12)-H(12B)	0.9800
P(1)-O(1)	1.6607(13)	C(12)-H(12C)	0.9800
P(1)-C(11)	1.8564(19)	C(13)-H(13A)	0.9800
P(1)-C(7)	1.856(2)	C(13)-H(13B)	0.9800
P(2)-O(2)	1.6573(13)	C(13)-H(13C)	0.9800
P(2)-C(19)	1.8535(18)	C(14)-H(14A)	0.9800
P(2)-C(15)	1.8601(19)	C(14)-H(14B)	0.9800
O(1)-C(2)	1.4050(19)	C(14)-H(14C)	0.9800
O(2)-C(6)	1.404(2)	C(15)-C(18)	1.523(3)
C(1)-C(2)	1.394(2)	C(15)-C(16)	1.536(3)
C(1)-C(6)	1.397(2)	C(15)-C(17)	1.539(3)
C(2)-C(3)	1.397(2)	C(16)-H(16A)	0.9800
C(3)-C(4)	1.406(2)	C(16)-H(16B)	0.9800
C(3)-C(23)	1.531(2)	C(16)-H(16C)	0.9800
C(4)-C(5)	1.397(2)	C(17)-H(17A)	0.9800
C(4)-H(4)	0.9500	C(17)-H(17B)	0.9800
C(5)-C(6)	1.395(2)	C(17)-H(17C)	0.9800
C(5)-C(27)	1.537(2)	C(18)-H(18A)	0.9800
C(7)-C(8)	1.526(3)	C(18)-H(18B)	0.9800
C(7)-C(10)	1.530(3)	C(18)-H(18C)	0.9800
C(7)-C(9)	1.535(3)	C(19)-C(22)	1.532(3)
C(8)-H(8A)	0.9800	C(19)-C(21)	1.531(3)
C(8)-H(8B)	0.9800	C(19)-C(20)	1.537(3)
C(8)-H(8C)	0.9800	C(20)-H(20A)	0.9800
C(9)-H(9A)	0.9800	C(20)-H(20B)	0.9800
C(9)-H(9B)	0.9800	C(20)-H(20C)	0.9800
C(9)-H(9C)	0.9800	C(21)-H(21A)	0.9800
C(10)-H(10A)	0.9800	C(21)-H(21B)	0.9800
C(10)-H(10B)	0.9800	C(21)-H(21C)	0.9800
C(10)-H(10C)	0.9800	C(22)-H(22A)	0.9800
C(11)-C(14)	1.519(3)	C(22)-H(22B)	0.9800

C(22)-H(22C)	0.9800	C(7)-P(1)-Ni(1)	115.89(7)
C(23)-C(26)	1.534(2)	O(2)-P(2)-C(19)	100.08(7)
C(23)-C(25)	1.533(3)	O(2)-P(2)-C(15)	101.32(8)
C(23)-C(24)	1.535(3)	C(19)-P(2)-C(15)	113.50(8)
C(24)-H(24A)	0.9800	O(2)-P(2)-Ni(1)	106.58(5)
C(24)-H(24B)	0.9800	C(19)-P(2)-Ni(1)	116.92(6)
C(24)-H(24C)	0.9800	C(15)-P(2)-Ni(1)	115.48(6)
C(25)-H(25A)	0.9800	C(2)-O(1)-P(1)	112.79(10)
C(25)-H(25B)	0.9800	C(6)-O(2)-P(2)	112.46(10)
C(25)-H(25C)	0.9800	C(2)-C(1)-C(6)	116.90(15)
C(26)-H(26A)	0.9800	C(2)-C(1)-Ni(1)	121.37(12)
C(26)-H(26B)	0.9800	C(6)-C(1)-Ni(1)	121.70(12)
C(26)-H(26C)	0.9800	C(1)-C(2)-C(3)	123.69(15)
C(27)-C(30)	1.534(3)	C(1)-C(2)-O(1)	116.16(14)
C(27)-C(28)	1.536(2)	C(3)-C(2)-O(1)	120.15(14)
C(27)-C(29)	1.537(3)	C(2)-C(3)-C(4)	114.88(15)
C(28)-H(28A)	0.9800	C(2)-C(3)-C(23)	122.69(14)
C(28)-H(28B)	0.9800	C(4)-C(3)-C(23)	122.37(15)
C(28)-H(28C)	0.9800	C(5)-C(4)-C(3)	125.64(16)
C(29)-H(29A)	0.9800	C(5)-C(4)-H(4)	117.2
C(29)-H(29B)	0.9800	C(3)-C(4)-H(4)	117.2
C(29)-H(29C)	0.9800	C(4)-C(5)-C(6)	114.70(15)
C(30)-H(30A)	0.9800	C(4)-C(5)-C(27)	122.21(15)
C(30)-H(30B)	0.9800	C(6)-C(5)-C(27)	123.04(15)
C(30)-H(30C)	0.9800	C(5)-C(6)-C(1)	124.09(15)
C(1)-Ni(1)-P(2)	83.26(5)	C(5)-C(6)-O(2)	119.97(14)
C(1)-Ni(1)-P(1)	83.70(5)	C(1)-C(6)-O(2)	115.94(14)
P(2)-Ni(1)-P(1)	166.94(2)	C(8)-C(7)-C(10)	109.1(2)
C(1)-Ni(1)-H(1)	179.4(8)	C(8)-C(7)-C(9)	108.8(2)
P(2)-Ni(1)-H(1)	96.7(8)	C(10)-C(7)-C(9)	109.36(18)
P(1)-Ni(1)-H(1)	96.3(8)	C(8)-C(7)-P(1)	104.68(14)
O(1)-P(1)-C(11)	100.16(8)	C(10)-C(7)-P(1)	114.26(15)
O(1)-P(1)-C(7)	101.34(8)	C(9)-C(7)-P(1)	110.39(15)
C(11)-P(1)-C(7)	114.08(9)	C(7)-C(8)-H(8A)	109.5
O(1)-P(1)-Ni(1)	105.93(5)	C(7)-C(8)-H(8B)	109.5
C(11)-P(1)-Ni(1)	116.35(6)	H(8A)-C(8)-H(8B)	109.5

C(7)-C(8)-H(8C)	109.5	C(11)-C(14)-H(14C)	109.5
H(8A)-C(8)-H(8C)	109.5	H(14A)-C(14)-H(14C)	109.5
H(8B)-C(8)-H(8C)	109.5	H(14B)-C(14)-H(14C)	109.5
C(7)-C(9)-H(9A)	109.5	C(18)-C(15)-C(16)	109.09(16)
C(7)-C(9)-H(9B)	109.5	C(18)-C(15)-C(17)	110.82(16)
H(9A)-C(9)-H(9B)	109.5	C(16)-C(15)-C(17)	107.79(16)
C(7)-C(9)-H(9C)	109.5	C(18)-C(15)-P(2)	114.07(14)
H(9A)-C(9)-H(9C)	109.5	C(16)-C(15)-P(2)	105.18(13)
H(9B)-C(9)-H(9C)	109.5	C(17)-C(15)-P(2)	109.56(14)
C(7)-C(10)-H(10A)	109.5	C(15)-C(16)-H(16A)	109.5
C(7)-C(10)-H(10B)	109.5	C(15)-C(16)-H(16B)	109.5
H(10A)-C(10)-H(10B)	109.5	H(16A)-C(16)-H(16B)	109.5
C(7)-C(10)-H(10C)	109.5	C(15)-C(16)-H(16C)	109.5
H(10A)-C(10)-H(10C)	109.5	H(16A)-C(16)-H(16C)	109.5
H(10B)-C(10)-H(10C)	109.5	H(16B)-C(16)-H(16C)	109.5
C(14)-C(11)-C(13)	110.61(18)	C(15)-C(17)-H(17A)	109.5
C(14)-C(11)-C(12)	108.82(18)	C(15)-C(17)-H(17B)	109.5
C(13)-C(11)-C(12)	108.49(19)	H(17A)-C(17)-H(17B)	109.5
C(14)-C(11)-P(1)	114.31(14)	C(15)-C(17)-H(17C)	109.5
C(13)-C(11)-P(1)	110.44(15)	H(17A)-C(17)-H(17C)	109.5
C(12)-C(11)-P(1)	103.81(13)	H(17B)-C(17)-H(17C)	109.5
C(11)-C(12)-H(12A)	109.5	C(15)-C(18)-H(18A)	109.5
C(11)-C(12)-H(12B)	109.5	C(15)-C(18)-H(18B)	109.5
H(12A)-C(12)-H(12B)	109.5	H(18A)-C(18)-H(18B)	109.5
C(11)-C(12)-H(12C)	109.5	C(15)-C(18)-H(18C)	109.5
H(12A)-C(12)-H(12C)	109.5	H(18A)-C(18)-H(18C)	109.5
H(12B)-C(12)-H(12C)	109.5	H(18B)-C(18)-H(18C)	109.5
C(11)-C(13)-H(13A)	109.5	C(22)-C(19)-C(21)	111.01(15)
C(11)-C(13)-H(13B)	109.5	C(22)-C(19)-C(20)	108.49(16)
H(13A)-C(13)-H(13B)	109.5	C(21)-C(19)-C(20)	108.42(16)
C(11)-C(13)-H(13C)	109.5	C(22)-C(19)-P(2)	114.41(13)
H(13A)-C(13)-H(13C)	109.5	C(21)-C(19)-P(2)	109.89(13)
H(13B)-C(13)-H(13C)	109.5	C(20)-C(19)-P(2)	104.25(12)
C(11)-C(14)-H(14A)	109.5	C(19)-C(20)-H(20A)	109.5
C(11)-C(14)-H(14B)	109.5	C(19)-C(20)-H(20B)	109.5
H(14A)-C(14)-H(14B)	109.5	H(20A)-C(20)-H(20B)	109.5

C(19)-C(20)-H(20C)	109.5	H(25B)-C(25)-H(25C)	109.5
H(20A)-C(20)-H(20C)	109.5	C(23)-C(26)-H(26A)	109.5
H(20B)-C(20)-H(20C)	109.5	C(23)-C(26)-H(26B)	109.5
C(19)-C(21)-H(21A)	109.5	H(26A)-C(26)-H(26B)	109.5
C(19)-C(21)-H(21B)	109.5	C(23)-C(26)-H(26C)	109.5
H(21A)-C(21)-H(21B)	109.5	H(26A)-C(26)-H(26C)	109.5
C(19)-C(21)-H(21C)	109.5	H(26B)-C(26)-H(26C)	109.5
H(21A)-C(21)-H(21C)	109.5	C(30)-C(27)-C(28)	107.05(15)
H(21B)-C(21)-H(21C)	109.5	C(30)-C(27)-C(29)	109.73(15)
C(19)-C(22)-H(22A)	109.5	C(28)-C(27)-C(29)	107.77(15)
C(19)-C(22)-H(22B)	109.5	C(30)-C(27)-C(5)	111.41(15)
H(22A)-C(22)-H(22B)	109.5	C(28)-C(27)-C(5)	111.78(14)
C(19)-C(22)-H(22C)	109.5	C(29)-C(27)-C(5)	109.02(14)
H(22A)-C(22)-H(22C)	109.5	C(27)-C(28)-H(28A)	109.5
H(22B)-C(22)-H(22C)	109.5	C(27)-C(28)-H(28B)	109.5
C(3)-C(23)-C(26)	111.84(14)	H(28A)-C(28)-H(28B)	109.5
C(3)-C(23)-C(25)	109.17(14)	C(27)-C(28)-H(28C)	109.5
C(26)-C(23)-C(25)	108.47(15)	H(28A)-C(28)-H(28C)	109.5
C(3)-C(23)-C(24)	110.93(15)	H(28B)-C(28)-H(28C)	109.5
C(26)-C(23)-C(24)	106.97(15)	C(27)-C(29)-H(29A)	109.5
C(25)-C(23)-C(24)	109.40(15)	C(27)-C(29)-H(29B)	109.5
C(23)-C(24)-H(24A)	109.5	H(29A)-C(29)-H(29B)	109.5
C(23)-C(24)-H(24B)	109.5	C(27)-C(29)-H(29C)	109.5
H(24A)-C(24)-H(24B)	109.5	H(29A)-C(29)-H(29C)	109.5
C(23)-C(24)-H(24C)	109.5	H(29B)-C(29)-H(29C)	109.5
H(24A)-C(24)-H(24C)	109.5	C(27)-C(30)-H(30A)	109.5
H(24B)-C(24)-H(24C)	109.5	C(27)-C(30)-H(30B)	109.5
C(23)-C(25)-H(25A)	109.5	H(30A)-C(30)-H(30B)	109.5
C(23)-C(25)-H(25B)	109.5	C(27)-C(30)-H(30C)	109.5
H(25A)-C(25)-H(25B)	109.5	H(30A)-C(30)-H(30C)	109.5
C(23)-C(25)-H(25C)	109.5	H(30B)-C(30)-H(30C)	109.5
H(25A)-C(25)-H(25C)	109.5		

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for **3d**. 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}
Ni1	14(1)	15(1)	14(1)	-3(1)	0(1)	2(1)
P1	12(1)	16(1)	14(1)	-1(1)	0(1)	1(1)
P2	14(1)	13(1)	15(1)	-1(1)	4(1)	2(1)
O1	13(1)	16(1)	16(1)	-2(1)	0(1)	2(1)
O2	13(1)	13(1)	17(1)	-2(1)	1(1)	2(1)
C1	12(1)	14(1)	14(1)	-1(1)	2(1)	0(1)
C2	11(1)	15(1)	13(1)	1(1)	2(1)	0(1)
C3	13(1)	12(1)	14(1)	0(1)	3(1)	0(1)
C4	14(1)	16(1)	13(1)	-2(1)	3(1)	-2(1)
C5	12(1)	15(1)	14(1)	3(1)	3(1)	1(1)
C6	13(1)	14(1)	15(1)	1(1)	4(1)	1(1)
C7	23(1)	25(1)	18(1)	4(1)	2(1)	2(1)
C8	32(1)	69(2)	56(2)	39(2)	19(1)	8(1)
C9	71(2)	38(1)	18(1)	1(1)	0(1)	-3(1)
C10	51(1)	27(1)	27(1)	8(1)	2(1)	5(1)
C11	15(1)	25(1)	23(1)	-4(1)	5(1)	-2(1)
C12	29(1)	34(1)	38(1)	7(1)	20(1)	4(1)
C13	28(1)	60(2)	47(2)	-26(1)	10(1)	-21(1)
C14	18(1)	36(1)	48(2)	4(1)	12(1)	9(1)
C15	24(1)	16(1)	22(1)	1(1)	12(1)	4(1)
C16	27(1)	21(1)	29(1)	2(1)	14(1)	-1(1)
C17	40(1)	25(1)	21(1)	-3(1)	14(1)	-2(1)
C18	29(1)	26(1)	35(1)	4(1)	18(1)	10(1)
C19	19(1)	15(1)	17(1)	0(1)	6(1)	-1(1)
C20	24(1)	24(1)	28(1)	-1(1)	12(1)	-3(1)
C21	35(1)	17(1)	21(1)	-3(1)	6(1)	-6(1)
C22	26(1)	19(1)	26(1)	5(1)	5(1)	1(1)
C23	14(1)	15(1)	17(1)	-4(1)	4(1)	1(1)
C24	23(1)	16(1)	23(1)	1(1)	6(1)	3(1)
C25	20(1)	24(1)	25(1)	-6(1)	10(1)	0(1)
C26	21(1)	20(1)	22(1)	-8(1)	2(1)	1(1)

C27	13(1)	21(1)	13(1)	4(1)	0(1)	1(1)
C28	17(1)	27(1)	17(1)	0(1)	-2(1)	0(1)
C29	21(1)	27(1)	23(1)	9(1)	0(1)	1(1)
C30	14(1)	27(1)	23(1)	1(1)	2(1)	2(1)

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

	x	y	z	U(eq)
H1	5583(15)	6989(16)	3411(13)	23(6)
H4	2944	3947	89	17
H8A	4961	3681	4247	76
H8B	4596	3839	3303	76
H8C	4774	4851	3879	76
H9A	6491	4386	4991	67
H9B	6390	5490	4521	67
H9C	7228	4708	4535	67
H10A	6292	2659	4180	55
H10B	6994	3102	3726	55
H10C	5982	2727	3228	55
H12A	7378	6038	1762	47
H12B	6449	6471	1913	47
H12C	6448	5357	1473	47
H13A	8177	6253	3161	68
H13B	7864	5628	3836	68
H13C	7274	6620	3392	68
H14A	8096	4427	2412	51
H14B	7175	3742	2293	51
H14C	7844	3950	3170	51
H16A	2428	6462	3412	36
H16B	3277	5946	3173	36
H16C	2357	6265	2494	36
H17A	3369	7887	4172	42
H17B	4017	8594	3796	42
H17C	4258	7370	3998	42
H18A	1961	8320	2998	42
H18B	2033	8167	2111	42
H18C	2622	9075	2671	42
H20A	5074	9035	1206	36

H20B	4662	7856	1063	36
H20C	5470	8129	1855	36
H21A	4891	10077	2357	38
H21B	5129	9104	2969	38
H21C	4167	9717	2815	38
H22A	3575	9843	1054	36
H22B	2906	9425	1549	36
H22C	3092	8704	859	36
H24A	4165	1954	1594	31
H24B	5204	2379	1900	31
H24C	4952	1420	1276	31
H25A	5287	3615	83	33
H25B	5671	2460	390	33
H25C	5872	3445	993	33
H26A	3260	2221	157	33
H26B	4102	1672	-75	33
H26C	3736	2813	-431	33
H28A	891	5184	-776	33
H28B	1384	4264	-173	33
H28C	1859	4710	-814	33
H29A	2490	7259	35	37
H29B	1591	6972	-677	37
H29C	2572	6483	-665	37
H30A	1630	6649	990	33
H30B	1254	5457	948	33
H30C	758	6296	275	33

Table 6. Torsion angles [°] for **3d**.

C11-P1-O1-C2	-122.38(12)	O1-P1-C7-C8	-74.79(18)
C7-P1-O1-C2	120.31(12)	C11-P1-C7-C8	178.55(17)
Ni1-P1-O1-C2	-1.05(12)	Ni1-P1-C7-C8	39.32(18)
C19-P2-O2-C6	124.76(12)	O1-P1-C7-C10	44.52(17)
C15-P2-O2-C6	-118.58(12)	C11-P1-C7-C10	-62.13(18)
Ni1-P2-O2-C6	2.58(12)	Ni1-P1-C7-C10	158.64(13)
P2-Ni1-C1-C2	178.63(15)	O1-P1-C7-C9	168.26(16)
P1-Ni1-C1-C2	-2.13(14)	C11-P1-C7-C9	61.61(19)
P2-Ni1-C1-C6	0.63(14)	Ni1-P1-C7-C9	-77.62(17)
P1-Ni1-C1-C6	179.87(15)	O1-P1-C11-C14	-52.52(17)
C6-C1-C2-C3	0.1(3)	C7-P1-C11-C14	54.88(18)
Ni1-C1-C2-C3	-177.96(13)	Ni1-P1-C11-C14	-166.09(14)
C6-C1-C2-O1	-179.88(15)	O1-P1-C11-C13	-178.01(16)
Ni1-C1-C2-O1	2.0(2)	C7-P1-C11-C13	-70.61(19)
P1-O1-C2-C1	-0.40(19)	Ni1-P1-C11-C13	68.42(18)
P1-O1-C2-C3	179.59(13)	O1-P1-C11-C12	65.88(14)
C1-C2-C3-C4	-2.0(3)	C7-P1-C11-C12	173.28(13)
O1-C2-C3-C4	178.01(15)	Ni1-P1-C11-C12	-47.69(14)
C1-C2-C3-C23	175.51(16)	O2-P2-C15-C18	-64.26(15)
O1-C2-C3-C23	-4.5(3)	C19-P2-C15-C18	42.11(17)
C2-C3-C4-C5	1.4(3)	Ni1-P2-C15-C18	-178.95(12)
C23-C3-C4-C5	-176.13(16)	O2-P2-C15-C16	55.24(14)
C3-C4-C5-C6	1.1(3)	C19-P2-C15-C16	161.61(12)
C3-C4-C5-C27	178.81(16)	Ni1-P2-C15-C16	-59.44(14)
C4-C5-C6-C1	-3.2(3)	O2-P2-C15-C17	170.87(12)
C27-C5-C6-C1	179.08(16)	C19-P2-C15-C17	-82.77(14)
C4-C5-C6-O2	176.66(15)	Ni1-P2-C15-C17	56.18(14)
C27-C5-C6-O2	-1.1(3)	O2-P2-C19-C22	38.11(15)
C2-C1-C6-C5	2.7(3)	C15-P2-C19-C22	-69.03(16)
Ni1-C1-C6-C5	-179.23(13)	Ni1-P2-C19-C22	152.65(12)
C2-C1-C6-O2	-177.18(14)	O2-P2-C19-C21	163.76(13)
Ni1-C1-C6-O2	0.9(2)	C15-P2-C19-C21	56.62(15)
P2-O2-C6-C5	177.80(13)	Ni1-P2-C19-C21	-81.70(14)
P2-O2-C6-C1	-2.33(19)	O2-P2-C19-C20	-80.22(13)

C15-P2-C19-C20	172.64(12)
Ni1-P2-C19-C20	34.32(14)
C2-C3-C23-C26	177.57(17)
C4-C3-C23-C26	-5.1(2)
C2-C3-C23-C25	-62.4(2)
C4-C3-C23-C25	114.93(18)
C2-C3-C23-C24	58.2(2)
C4-C3-C23-C24	-124.45(18)
C4-C5-C27-C30	128.89(18)
C6-C5-C27-C30	-53.5(2)
C4-C5-C27-C28	9.2(2)
C6-C5-C27-C28	-173.28(16)
C4-C5-C27-C29	-109.87(19)
C6-C5-C27-C29	67.7(2)

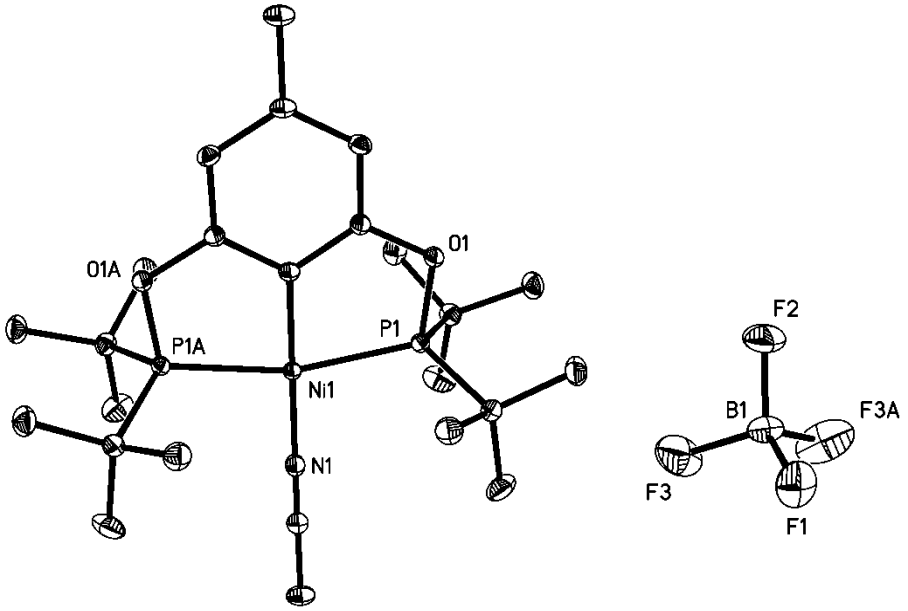
CRYSTAL STRUCTURE REPORT

$C_{25}H_{44}BF_4NNiO_2P_2$ (**4c**)

Report prepared for:

Dr. S. Chakraborty, Prof. W. Jones

February 25, 2014



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Data collection

A crystal (0.22 x 0.14 x 0.12 mm^3) was placed onto the tip of a 0.1 mm diameter glass capillary tube and mounted on a Bruker SMART APEX II CCD platform diffractometer for a data collection at 100.0(5) K.[1] A preliminary set of cell constants and an orientation matrix were calculated from reflections harvested from three orthogonal wedges of reciprocal space. The full data collection was carried out using MoKα radiation (graphite monochromator) with a frame time of 45 seconds and a detector distance of 4.00 cm. A randomly oriented region of reciprocal space was surveyed: five major sections of frames were collected with 0.50º steps in ω at five different ϕ settings and a detector position of -38º in 2θ. The intensity data were corrected for absorption.[2] Final cell constants were calculated from the xyz centroids of 4025 strong reflections from the actual data collection after integration.[3] See Table 1 for additional crystal and refinement information.

Structure solution and refinement

The structure was solved using SIR2011[4] and refined using SHELXL-2014.[5] The space group $P2_1/m$ was determined based on systematic absences and intensity statistics. A direct-methods solution was calculated which provided most non-hydrogen atoms from the E-map. Full-matrix least squares / difference Fourier cycles were performed which located the remaining non-hydrogen atoms. All non-hydrogen atoms were refined with anisotropic displacement parameters. All hydrogen atoms were placed in ideal positions and refined as riding atoms with relative isotropic displacement parameters. The final full matrix least squares refinement converged to $R1 = 0.0389$ (F^2, $I > 2\sigma(I)$) and $wR2 = 0.0896$ (F^2, all data).

Structure description

The structure is the one suggested. The asymmetric unit contains one nickel complex cation and one tetrafluoroborate anion, both located on a crystallographic mirror plane. The plane includes atom Ni1, C1, C4, C13, N1, C14, C15, B1, F1, and F2.

Unless noted otherwise all structural diagrams containing thermal displacement ellipsoids are drawn at the 50 % probability level.

Data collection, structure solution, and structure refinement were conducted at the X-ray Crystallographic Facility, B51 Hutchison Hall, Department of Chemistry, University of Rochester. All publications arising from this report MUST either 1) include William W. Brennessel as a coauthor or 2) acknowledge William W. Brennessel and the X-ray Crystallographic Facility of the Department of Chemistry at the University of Rochester.

[1] *APEX2*, version 2013.10-0; Bruker AXS: Madison, WI, 2013.

[2] Sheldrick, G. M. *SADABS*, version 2012/1; University of Göttingen: Göttingen, Germany, 2012.

[3] *SAINT*, version 8.34A; Bruker AXS: Madison, WI, 2013.

[4] Burla, M. C.; Caliandro, R.; Camalli, M.; Carrozzini, B.; Cascarano, G. L.; Giacovazzo, C.; Mallamo, M.; Mazzone, A.; Polidori, G.; Spagna, R. *SIR2011: a new package for crystal structure determination and refinement*, version 1.0; Istituto di Cristallografia: Bari, Italy, 2012.

[5] Sheldrick, G. M. *SHELXL-2014/1*; University of Göttingen: Göttingen, Germany, 2014.

Some equations of interest:

$$R_{int} = \Sigma\, |F_o^2 - < F_o^2 >| \,/\, \Sigma |F_o^2|$$

$$R1 = \Sigma ||F_o| - |F_c|| \,/\, \Sigma |F_o|$$

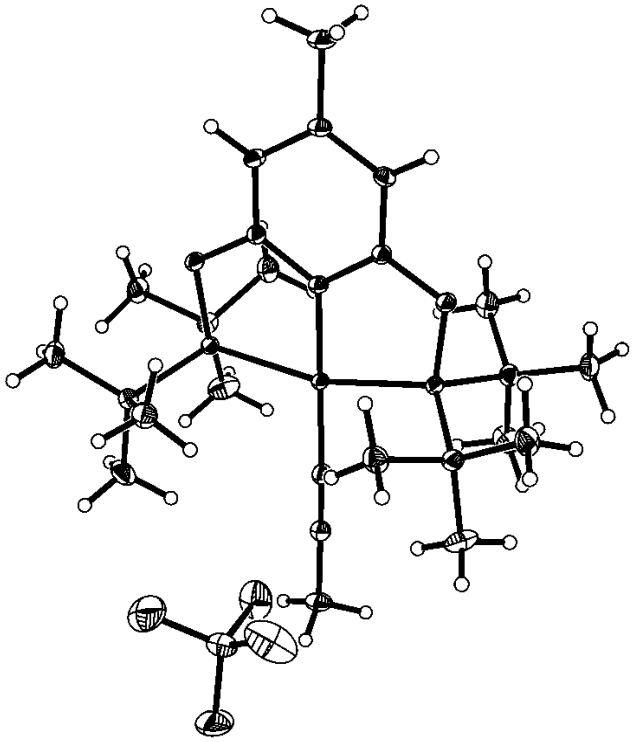
$$wR2 = [\Sigma [w(F_o^2 - F_c^2)^2] \,/\, \Sigma\, [w(F_o^2)^2]]^{1/2}$$

where $w = 1 \,/\, [\sigma^2 (F_o^2) + (aP)^2 + bP]$ and

$$P = 1/3 \max (0, F_o^2) + 2/3\, F_c^2$$

$$\text{GOF} = S = [\Sigma [w(F_o^2 - F_c^2)^2] \,/\, (m-n)]^{1/2}$$

where m = number of reflections and n = number of parameters



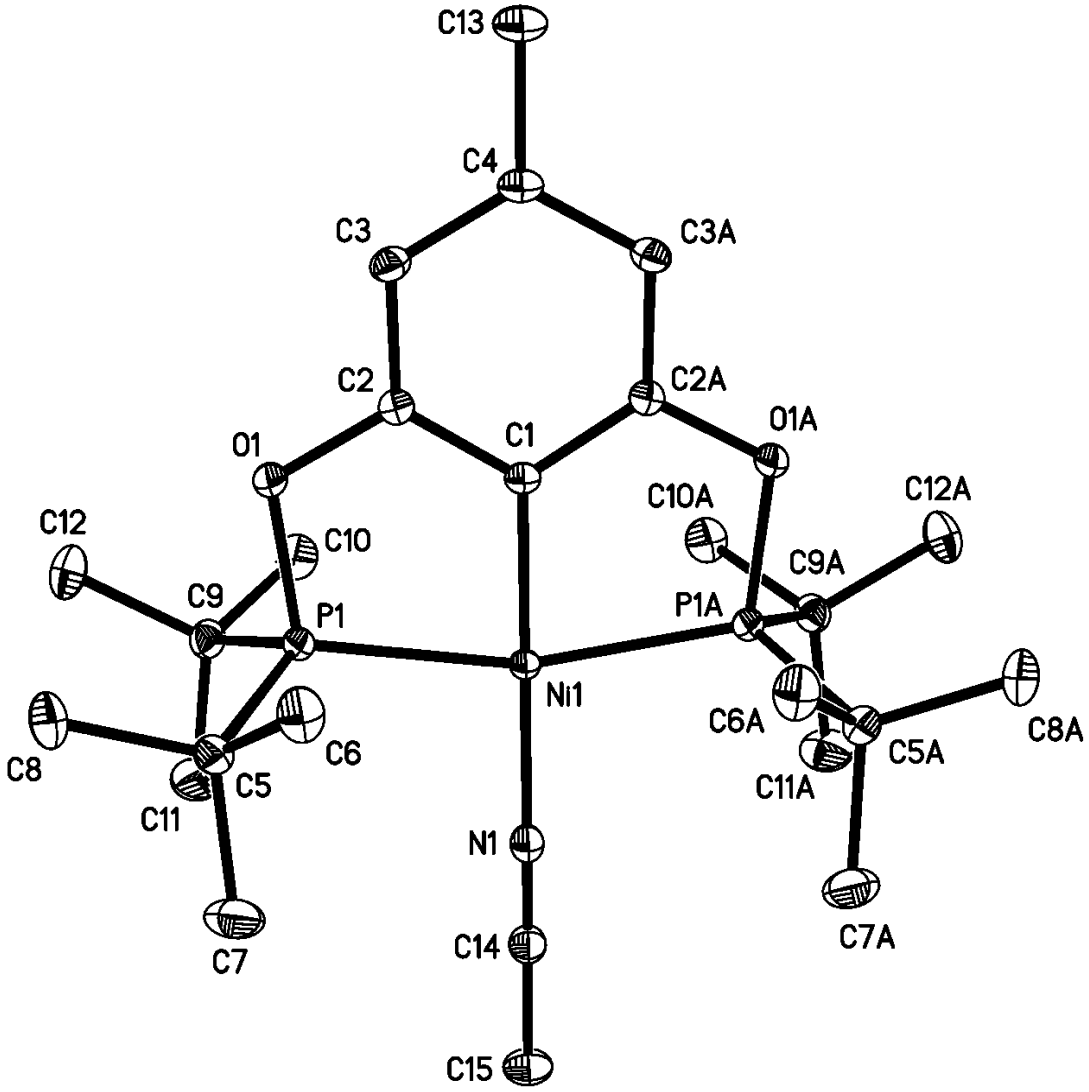

C13
C4
C3
C3A
C2
C2A
O1
C1
O1A
C10A
C12A
C12
C10
C9A
C9
P1
P1A
Ni1
C6A
C8A
C8
C6
C5
C5A
C11
C11A
N1
C7
C14
C7A
C15

Table 1. Crystal data and structure refinement for **4c**.

Empirical formula	$C_{25}H_{44}BF_4NNiO_2P_2$	
Formula weight	598.07	
Temperature	100.0(5) K	
Wavelength	0.71073 Å	
Crystal system	monoclinic	
Space group	$P2_1/m$	
Unit cell dimensions	a = 7.6473(9) Å	α = 90°
	b = 12.5571(15) Å	β = 92.552(2)°
	c = 15.1719(18) Å	γ = 90°
Volume	1455.5(3) Å^3	
Z	2	
Density (calculated)	1.365 Mg/m^3	
Absorption coefficient	0.825 mm^{-1}	
F(000)	632	
Crystal color, morphology	pale yellow, block	
Crystal size	0.22 x 0.14 x 0.12 mm^3	
Theta range for data collection	2.106 to 38.687°	
Index ranges	$-13 \le h \le 13$, $-21 \le k \le 21$, $-26 \le l \le 26$	
Reflections collected	38303	
Independent reflections	8221 [R(int) = 0.0539]	
Observed reflections	6335	
Completeness to theta = 37.785°	98.4%	
Absorption correction	Multi-scan	
Max. and min. transmission	0.7476 and 0.6658	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	8221 / 0 / 186	
Goodness-of-fit on F^2	1.029	
Final R indices [I>2sigma(I)]	$R1$ = 0.0389, $wR2$ = 0.0817	
R indices (all data)	$R1$ = 0.0588, $wR2$ = 0.0896	
Largest diff. peak and hole	0.635 and -0.409 e.Å^{-3}	

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

	x	y	z	U_{eq}
Ni1	734(1)	7500	2427(1)	10(1)
P1	995(1)	5771(1)	2271(1)	11(1)
O1	2453(1)	5603(1)	1510(1)	13(1)
N1	-884(2)	7500	3326(1)	14(1)
C1	2364(2)	7500	1530(1)	12(1)
C2	2986(1)	6561(1)	1160(1)	12(1)
C3	4094(1)	6541(1)	459(1)	13(1)
C4	4644(2)	7500	96(1)	14(1)
C5	-942(1)	5110(1)	1731(1)	15(1)
C6	-1297(2)	5747(1)	872(1)	20(1)
C7	-2543(2)	5210(1)	2298(1)	25(1)
C8	-614(2)	3938(1)	1514(1)	20(1)
C9	2062(1)	5055(1)	3214(1)	15(1)
C10	3554(2)	5798(1)	3541(1)	20(1)
C11	782(2)	4911(1)	3953(1)	25(1)
C12	2829(2)	3978(1)	2950(1)	22(1)
C13	5783(2)	7500	-688(1)	22(1)
C14	-1772(2)	7500	3913(1)	15(1)
C15	-2874(2)	7500	4669(1)	20(1)
B1	2519(3)	7500	5790(1)	22(1)
F1	1210(2)	7500	5124(1)	35(1)
F2	1771(2)	7500	6601(1)	43(1)
F3	3535(1)	6596(1)	5706(1)	51(1)

Table 3. Bond lengths [Å] and angles [°] for **4c**.

Ni(1)-N(1)	1.8824(13)	C(11)-H(11A)	0.9800
Ni(1)-C(1)	1.8861(14)	C(11)-H(11B)	0.9800
Ni(1)-P(1)#1	2.1943(4)	C(11)-H(11C)	0.9800
Ni(1)-P(1)	2.1943(4)	C(12)-H(12A)	0.9800
P(1)-O(1)	1.6531(8)	C(12)-H(12B)	0.9800
P(1)-C(9)	1.8487(11)	C(12)-H(12C)	0.9800
P(1)-C(5)	1.8557(11)	C(13)-H(13A)	0.9800
O(1)-C(2)	1.3836(12)	C(13)-H(13B)	0.9800
N(1)-C(14)	1.144(2)	C(13)-H(13C)	0.9800
C(1)-C(2)#1	1.3990(12)	C(14)-C(15)	1.453(2)
C(1)-C(2)	1.3990(12)	C(15)-H(15A)	0.9800
C(2)-C(3)	1.3893(14)	C(15)-H(15B)	0.9800
C(3)-C(4)	1.3964(12)	C(15)-H(15C)	0.9800
C(3)-H(3)	0.9500	B(1)-F(2)	1.379(2)
C(4)-C(3)#1	1.3965(12)	B(1)-F(3)#1	1.3851(14)
C(4)-C(13)	1.506(2)	B(1)-F(3)	1.3851(14)
C(5)-C(7)	1.5317(15)	B(1)-F(1)	1.390(2)
C(5)-C(8)	1.5322(15)	N(1)-Ni(1)-C(1)	179.69(6)
C(5)-C(6)	1.5437(16)	N(1)-Ni(1)-P(1)#1	98.295(8)
C(6)-H(6A)	0.9800	C(1)-Ni(1)-P(1)#1	81.707(8)
C(6)-H(6B)	0.9800	N(1)-Ni(1)-P(1)	98.294(8)
C(6)-H(6C)	0.9800	C(1)-Ni(1)-P(1)	81.708(8)
C(7)-H(7A)	0.9800	P(1)#1-Ni(1)-P(1)	163.401(16)
C(7)-H(7B)	0.9800	O(1)-P(1)-C(9)	100.96(4)
C(7)-H(7C)	0.9800	O(1)-P(1)-C(5)	100.68(4)
C(8)-H(8A)	0.9800	C(9)-P(1)-C(5)	116.12(5)
C(8)-H(8B)	0.9800	O(1)-P(1)-Ni(1)	105.60(3)
C(8)-H(8C)	0.9800	C(9)-P(1)-Ni(1)	115.88(4)
C(9)-C(11)	1.5317(15)	C(5)-P(1)-Ni(1)	114.53(4)
C(9)-C(12)	1.5348(15)	C(2)-O(1)-P(1)	112.15(6)
C(9)-C(10)	1.5388(16)	C(14)-N(1)-Ni(1)	175.39(13)
C(10)-H(10A)	0.9800	C(2)#1-C(1)-C(2)	114.96(12)
C(10)-H(10B)	0.9800	C(2)#1-C(1)-Ni(1)	122.49(6)
C(10)-H(10C)	0.9800	C(2)-C(1)-Ni(1)	122.49(6)

O(1)-C(2)-C(3)	118.64(8)	C(11)-C(9)-P(1)	110.45(8)
O(1)-C(2)-C(1)	117.84(9)	C(12)-C(9)-P(1)	112.77(8)
C(3)-C(2)-C(1)	123.52(9)	C(10)-C(9)-P(1)	104.51(7)
C(2)-C(3)-C(4)	119.41(10)	C(9)-C(10)-H(10A)	109.5
C(2)-C(3)-H(3)	120.3	C(9)-C(10)-H(10B)	109.5
C(4)-C(3)-H(3)	120.3	H(10A)-C(10)-H(10B)	109.5
C(3)-C(4)-C(3)#1	119.15(13)	C(9)-C(10)-H(10C)	109.5
C(3)-C(4)-C(13)	120.42(7)	H(10A)-C(10)-H(10C)	109.5
C(3)#1-C(4)-C(13)	120.42(7)	H(10B)-C(10)-H(10C)	109.5
C(7)-C(5)-C(8)	110.00(9)	C(9)-C(11)-H(11A)	109.5
C(7)-C(5)-C(6)	108.50(10)	C(9)-C(11)-H(11B)	109.5
C(8)-C(5)-C(6)	109.88(9)	H(11A)-C(11)-H(11B)	109.5
C(7)-C(5)-P(1)	110.98(8)	C(9)-C(11)-H(11C)	109.5
C(8)-C(5)-P(1)	112.91(8)	H(11A)-C(11)-H(11C)	109.5
C(6)-C(5)-P(1)	104.34(7)	H(11B)-C(11)-H(11C)	109.5
C(5)-C(6)-H(6A)	109.5	C(9)-C(12)-H(12A)	109.5
C(5)-C(6)-H(6B)	109.5	C(9)-C(12)-H(12B)	109.5
H(6A)-C(6)-H(6B)	109.5	H(12A)-C(12)-H(12B)	109.5
C(5)-C(6)-H(6C)	109.5	C(9)-C(12)-H(12C)	109.5
H(6A)-C(6)-H(6C)	109.5	H(12A)-C(12)-H(12C)	109.5
H(6B)-C(6)-H(6C)	109.5	H(12B)-C(12)-H(12C)	109.5
C(5)-C(7)-H(7A)	109.5	C(4)-C(13)-H(13A)	109.5
C(5)-C(7)-H(7B)	109.5	C(4)-C(13)-H(13B)	109.5
H(7A)-C(7)-H(7B)	109.5	H(13A)-C(13)-H(13B)	109.5
C(5)-C(7)-H(7C)	109.5	C(4)-C(13)-H(13C)	109.5
H(7A)-C(7)-H(7C)	109.5	H(13A)-C(13)-H(13C)	109.5
H(7B)-C(7)-H(7C)	109.5	H(13B)-C(13)-H(13C)	109.5
C(5)-C(8)-H(8A)	109.5	N(1)-C(14)-C(15)	178.98(16)
C(5)-C(8)-H(8B)	109.5	C(14)-C(15)-H(15A)	109.5
H(8A)-C(8)-H(8B)	109.5	C(14)-C(15)-H(15B)	109.5
C(5)-C(8)-H(8C)	109.5	H(15A)-C(15)-H(15B)	109.5
H(8A)-C(8)-H(8C)	109.5	C(14)-C(15)-H(15C)	109.5
H(8B)-C(8)-H(8C)	109.5	H(15A)-C(15)-H(15C)	109.5
C(11)-C(9)-C(12)	110.56(10)	H(15B)-C(15)-H(15C)	109.5
C(11)-C(9)-C(10)	108.93(10)	F(2)-B(1)-F(3)#1	109.81(11)
C(12)-C(9)-C(10)	109.40(9)	F(2)-B(1)-F(3)	109.81(11)

F(3)#1-B(1)-F(3)	110.15(17)	F(3)#1-B(1)-F(1)	108.77(10)
F(2)-B(1)-F(1)	109.51(16)	F(3)-B(1)-F(1)	108.77(10)

Symmetry transformations used to generate equivalent atoms:

#1 x,-y+3/2,z

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for **4c**. 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}
Ni1	11(1)	9(1)	10(1)	0	4(1)	0
P1	12(1)	10(1)	12(1)	0(1)	3(1)	0(1)
O1	15(1)	11(1)	14(1)	0(1)	6(1)	1(1)
N1	16(1)	11(1)	16(1)	0	3(1)	0
C1	11(1)	12(1)	11(1)	0	3(1)	0
C2	12(1)	11(1)	12(1)	0(1)	1(1)	0(1)
C3	13(1)	14(1)	13(1)	-2(1)	3(1)	1(1)
C4	12(1)	18(1)	12(1)	0	3(1)	0
C5	14(1)	14(1)	17(1)	-1(1)	2(1)	-2(1)
C6	20(1)	18(1)	21(1)	1(1)	-5(1)	-1(1)
C7	16(1)	33(1)	28(1)	-8(1)	6(1)	-6(1)
C8	25(1)	13(1)	22(1)	-2(1)	-2(1)	-3(1)
C9	18(1)	13(1)	14(1)	1(1)	2(1)	1(1)
C10	21(1)	20(1)	19(1)	-1(1)	-3(1)	0(1)
C11	26(1)	28(1)	21(1)	11(1)	9(1)	4(1)
C12	28(1)	14(1)	22(1)	0(1)	-2(1)	5(1)
C13	23(1)	25(1)	18(1)	0	12(1)	0
C14	16(1)	14(1)	14(1)	0	1(1)	0
C15	19(1)	26(1)	14(1)	0	7(1)	0
B1	18(1)	32(1)	16(1)	0	1(1)	0
F1	26(1)	36(1)	40(1)	0	-16(1)	0
F2	68(1)	37(1)	27(1)	0	26(1)	0
F3	45(1)	67(1)	39(1)	-9(1)	-7(1)	35(1)

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

	x	y	z	U(eq)
H3	4475	5881	228	16
H6A	-2280	5425	531	30
H6B	-251	5735	521	30
H6C	-1585	6485	1016	30
H7A	-3584	4953	1963	38
H7B	-2708	5958	2459	38
H7C	-2359	4783	2835	38
H8A	-1547	3679	1104	31
H8B	-604	3516	2058	31
H8C	517	3868	1241	31
H10A	4261	5442	4008	30
H10B	3058	6455	3774	30
H10C	4293	5971	3049	30
H11A	1416	4647	4484	37
H11B	-126	4398	3766	37
H11C	236	5597	4081	37
H12A	3582	3702	3437	32
H12B	3520	4070	2426	32
H12C	1875	3474	2817	32
H13A	6163	8229	-808	33
H13B	5117	7221	-1205	33
H13C	6811	7050	-562	33
H15A	-3735	6924	4606	30
H15B	-3483	8185	4702	30
H15C	-2147	7391	5210	30

Table 6. Torsion angles [°] for **4c**.

C9-P1-O1-C2	-124.94(7)	Ni1-P1-C9-C10	-39.26(8)
C5-P1-O1-C2	115.53(7)		
Ni1-P1-O1-C2	-3.89(7)		
P1#1-Ni1-C1-C2#1	-1.06(10)		
P1-Ni1-C1-C2#1	178.26(11)		
P1#1-Ni1-C1-C2	-178.26(11)		
P1-Ni1-C1-C2	1.07(10)		
P1-O1-C2-C3	-174.49(7)		
P1-O1-C2-C1	5.11(12)		
C2#1-C1-C2-O1	178.54(7)		
Ni1-C1-C2-O1	-4.07(16)		
C2#1-C1-C2-C3	-1.9(2)		
Ni1-C1-C2-C3	175.51(8)		
O1-C2-C3-C4	179.93(10)		
C1-C2-C3-C4	0.36(17)		
C2-C3-C4-C3#1	1.3(2)		
C2-C3-C4-C13	-177.39(12)		
O1-P1-C5-C7	-177.01(8)		
C9-P1-C5-C7	75.06(9)		
Ni1-P1-C5-C7	-64.25(9)		
O1-P1-C5-C8	58.95(8)		
C9-P1-C5-C8	-48.99(9)		
Ni1-P1-C5-C8	171.71(7)		
O1-P1-C5-C6	-60.34(8)		
C9-P1-C5-C6	-168.27(7)		
Ni1-P1-C5-C6	52.43(8)		
O1-P1-C9-C11	-168.77(8)		
C5-P1-C9-C11	-61.01(9)		
Ni1-P1-C9-C11	77.75(8)		
O1-P1-C9-C12	-44.51(9)		
C5-P1-C9-C12	63.25(9)		
Ni1-P1-C9-C12	-158.00(7)		
O1-P1-C9-C10	74.22(7)		
C5-P1-C9-C10	-178.01(7)		

Symmetry transformations used to generate equivalent atoms: #1 x,-y+3/2,z

Table 7. Hydrogen bonds and close contacts for **4c** [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
C6-H6B...O1	0.98	2.51	2.9908(14)	110.4
C12-H12A...F3#2	0.98	2.54	3.4507(16)	155.0
C12-H12B...O1	0.98	2.49	2.9937(14)	111.6

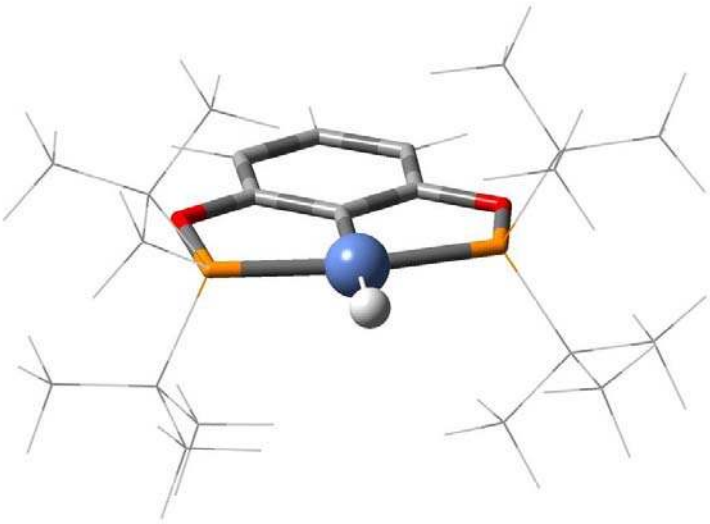
Symmetry transformations used to generate equivalent atoms: #1 x,-y+3/2,z #2 -x+1,-y+1,-z+1

9. Cartesian Coordinates of Intermediates

All reported geometries have been optimized following the procedure described in the Computational Details section. All stationary points were verified to have no imaginary frequencies.

Ni^{II}-H

E_{gas}	-1864.448622 hartree
E_{solv}	-1864.472409 hartree
G_{solv}	-1863.955693 hartree



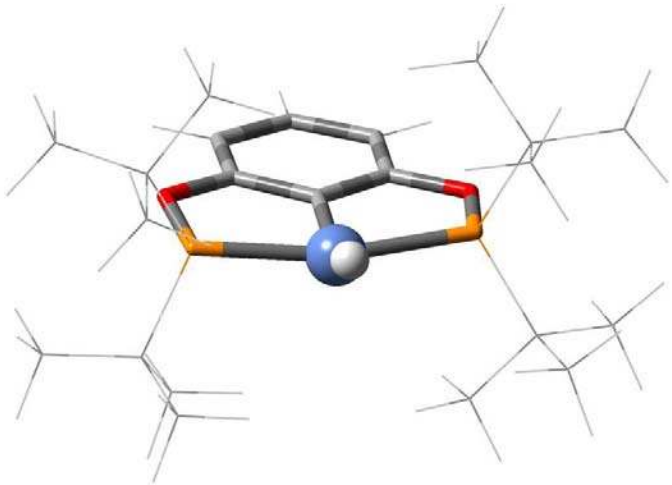
x y z

C	-1.21289300	3.49475700	0.06102900
C	-1.18511600	2.10741600	0.06161900
C	0.00000000	1.38478600	-0.00000300
C	1.18511700	2.10741600	-0.06162200
C	1.21289300	3.49475700	-0.06103100
C	0.00000000	4.17237800	-0.00000100
H	-2.15951500	4.02732100	0.10779700
H	2.15951600	4.02732000	-0.10779800
Ni	0.00000000	-0.53205500	-0.00000500
H	0.00000000	-2.06391600	-0.00000500
O	2.36707700	1.39833800	-0.12088400
O	-2.36707700	1.39833800	0.12088100
P	2.12009900	-0.26241200	-0.01442100
P	-2.12009900	-0.26241200	0.01442000
C	-3.01484100	-0.64569300	-1.57863000
C	-4.50470300	-0.33687300	-1.54241100
H	-4.70419900	0.68617600	-1.20494800
H	-4.92008700	-0.43733300	-2.55470400
H	-5.05903200	-1.02982400	-0.90033600
C	-2.34287200	0.23867100	-2.63076000
H	-2.51929400	1.30351600	-2.44837400
H	-1.25857600	0.07846600	-2.66826100
H	-2.75250700	-0.00708000	-3.61950100
C	-2.77565000	-2.10648300	-1.94831900
H	-3.24452900	-2.80572000	-1.24855700
H	-3.20374300	-2.30126900	-2.94098700
H	-1.70609000	-2.34138600	-1.98541400
C	-3.01934500	-0.82515200	1.54990600
C	-2.02392200	-0.54991000	2.67953600
H	-2.46790500	-0.84859900	3.63885200
H	-1.09610300	-1.11911200	2.54147500
H	-1.76453000	0.51334200	2.74915600
C	-3.28616100	-2.32366100	1.48020700
H	-2.38504600	-2.88991500	1.21532700
H	-3.62166600	-2.67664700	2.46464600
H	-4.07636000	-2.57394700	0.76352400
C	-4.30785800	-0.06014300	1.82594100
H	-5.08961100	-0.26249800	1.08841500
H	-4.69947100	-0.36463100	2.80633300
H	-4.13877300	1.02008500	1.85373300
C	3.01935100	-0.82515400	-1.54990300
C	3.01483500	-0.64569100	1.57863300
C	3.28616200	-2.32366400	-1.48020400
H	2.38504400	-2.88991500	-1.21532900
H	3.62167100	-2.67665000	-2.46464200
H	4.07635700	-2.57395300	-0.76351800

C	4.30786700	-0.06014800	-1.82593300
H	4.69948300	-0.36463700	-2.80632300
H	4.13878500	1.02008000	-1.85372500
H	5.08961700	-0.26250600	-1.08840300
C	2.02393400	-0.54990800	-2.67953700
H	1.09611200	-1.11910700	-2.54148000
H	1.76454500	0.51334400	-2.74915800
H	2.46791900	-0.84859800	-3.63885200
C	2.77564400	-2.10648100	1.94832200
H	3.20373300	-2.30126600	2.94099200
H	1.70608400	-2.34138500	1.98541200
H	3.24452600	-2.80571900	1.24856200
C	4.50469700	-0.33686900	1.54242000
H	4.92007600	-0.43732900	2.55471400
H	5.05902900	-1.02982100	0.90034700
H	4.70419300	0.68617900	1.20495700
C	2.34286100	0.23867200	2.63076000
H	2.51928400	1.30351700	2.44837400
H	1.25856600	0.07846800	2.66825800
H	2.75249300	-0.00707800	3.61950300
H	0.00000000	5.26040200	0.00000000

Ni^{III}-H^{+}

E_{gas}	-1864.199875 hartree
E_{solv}	-1864.277804 hartree
G_{solv}	-1863.763388 hartree

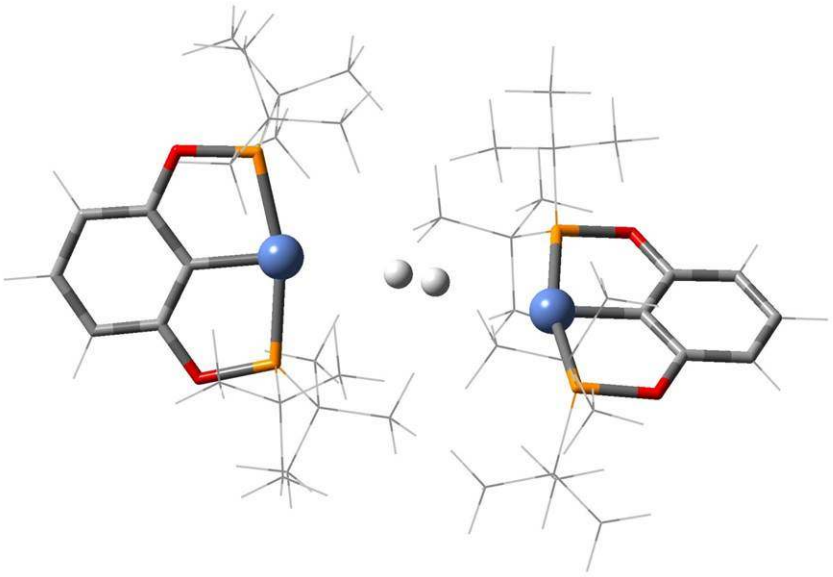


	x	y	z
C	-1.21346700	3.48689100	0.03029400
C	-1.20037200	2.10597900	0.04691900
C	0.00252100	1.38379500	-0.03641100
C	1.21044400	2.10151000	-0.07677900
C	1.22696800	3.48262500	-0.09277300
C	0.00771500	4.15346700	-0.03815700
H	-2.15416500	4.02786300	0.06868600
H	2.16885200	4.02014100	-0.14724400
Ni	-0.00547200	-0.49332700	-0.13606300
H	-0.01917200	-1.90814800	0.44290300
O	2.37665900	1.40095700	-0.11765600
O	-2.36799200	1.41000400	0.12773100
P	2.18333800	-0.25896500	-0.00734900
P	-2.18795400	-0.24990300	0.02064100
C	-3.04692500	-0.67400800	-1.56902500
C	-4.55382700	-0.46112600	-1.51685000
H	-4.81750400	0.55507400	-1.20392600
H	-4.96820200	-0.61074600	-2.52179700
H	-5.05651600	-1.17356400	-0.85559700
C	-2.43606100	0.24419600	-2.62907300
H	-2.67841700	1.29753800	-2.45729400
H	-1.34423600	0.14983500	-2.68979300
H	-2.84124500	-0.03317200	-3.60956800
C	-2.70737200	-2.12436000	-1.90709400
H	-3.12226600	-2.84115800	-1.19211200
H	-3.12505200	-2.36782300	-2.89151400
H	-1.62363900	-2.29293000	-1.96024000
C	-2.99870700	-0.80919500	1.59299700
C	-1.99040400	-0.48285300	2.69606200
H	-2.42085500	-0.76100100	3.66568500
H	-1.05758600	-1.04583100	2.57368900
H	-1.75207600	0.58648400	2.74180100
C	-3.23668400	-2.31412100	1.54735300
H	-2.32901600	-2.87675100	1.29838300
H	-3.56242300	-2.64904300	2.53963600
H	-4.02629400	-2.59433300	0.84254100
C	-4.30140300	-0.06041500	1.86079400
H	-5.08082200	-0.28296400	1.12799400
H	-4.68207000	-0.37067300	2.84181600
H	-4.15598100	1.02283000	1.89096700
C	3.05212600	-0.85388300	-1.53592900
C	2.98922700	-0.63371300	1.62331900
C	3.29947800	-2.35436600	-1.42704300
H	2.39509200	-2.91230000	-1.15179600
H	3.62684800	-2.73177400	-2.40344800

H	4.08919900	-2.59725900	-0.70926500
C	4.34914900	-0.10175900	-1.81118300
H	4.75395600	-0.44552500	-2.77140900
H	4.19086200	0.97772700	-1.88643900
H	5.11588700	-0.28396400	-1.05394700
C	2.05652200	-0.58572800	-2.66754900
H	1.13127900	-1.16772200	-2.54500900
H	1.79335300	0.47521500	-2.75349600
H	2.50311000	-0.89019000	-3.62202000
C	2.75454100	-2.09512100	1.99336300
H	3.19110200	-2.27899000	2.98259700
H	1.68800900	-2.33792000	2.05275100
H	3.22547800	-2.79733000	1.29943700
C	4.47979700	-0.31335900	1.60442000
H	4.87137700	-0.39973600	2.62564300
H	5.04946800	-1.01326400	0.98515000
H	4.68383200	0.70678500	1.26237700
C	2.28236000	0.25966000	2.64387000
H	2.47418400	1.32342800	2.47264100
H	1.19729500	0.10024400	2.65510000
H	2.65853300	0.01419500	3.64419200
H	0.00918100	5.24053000	-0.04741900

$[Ni^{II}$-H---H-$Ni^{III}]^{+}$

E_{gas}	-3728.689051 hartree
E_{solv}	-3728.77787 hartree
G_{solv}	-3727.721189 hartree



	x	y	z
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C	4.91789200	1.96961800	-0.98915400
C	4.63837600	0.76666100	-0.34790400
C	5.72186800	-0.00331100	0.06224800
C	7.03298400	0.38989100	-0.15576000
C	7.26238200	1.59487800	-0.80602400
H	6.38462600	3.34831900	-1.72973100
H	7.85007500	-0.24353600	0.17848500
Ni	2.85059000	0.20256900	-0.02017400
O	5.47888200	-1.19545700	0.69850900
O	3.86256300	2.76104800	-1.37823900
P	3.86392600	-1.56740600	0.85009900
P	2.39838500	2.19949600	-0.82716100
C	1.40561200	2.33032300	-2.40180100
C	1.96065200	3.44997600	0.49021500
C	3.83769000	-3.19824700	-0.06189500
C	3.68076700	-1.70133300	2.69890800
H	8.28506300	1.91655300	-0.98504600
Ni	-2.85056100	-0.20255200	-0.02071900
C	-4.63830600	-0.76655200	-0.34884500
C	-4.91774000	-1.96936600	-0.99039700
C	-5.72185200	0.00333600	0.06132100
C	-6.21217900	-2.39972900	-1.22953900
C	-7.03294000	-0.38980000	-0.15697800
C	-7.26225300	-1.59463900	-0.80754700
H	-6.38437600	-3.34789000	-1.73150000
H	-7.85007500	0.24356200	0.17728500

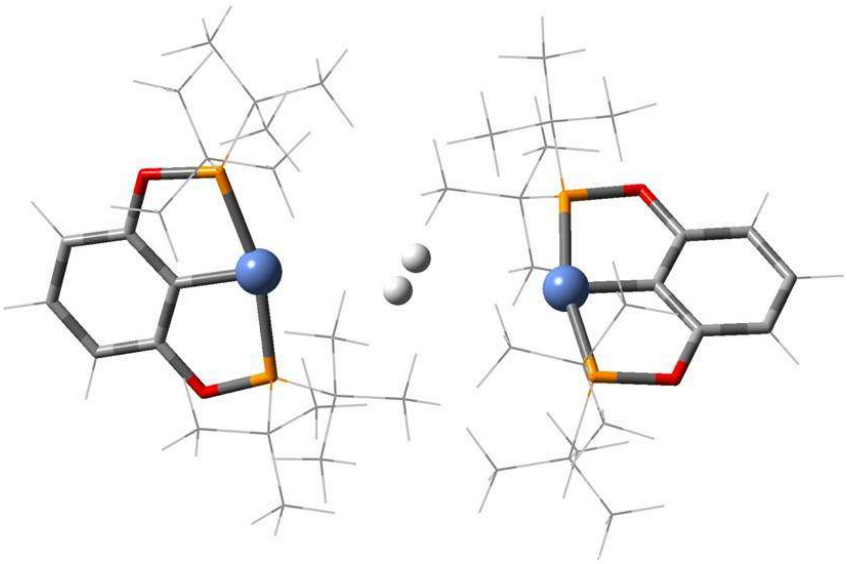
H	-8.28491000	-1.91626100	-0.98679800
O	-5.47894900	1.19532600	0.69790500
O	-3.86236000	-2.76072700	-1.37948500
P	-2.39826400	-2.19933100	-0.82802500
P	-3.86401300	1.56723900	0.84981900
C	-3.68117100	1.70074200	2.69869100
C	-3.83764800	3.19829600	-0.06179400
C	-1.40523800	-2.32987300	-2.40253300
C	-1.96080500	-3.45010200	0.48917000
H	0.39081000	0.02084300	0.34186300
H	-0.39073200	-0.02101200	0.34183000
C	-2.58619900	3.98778200	0.29263500
H	-1.68109400	3.37856300	0.20128600
H	-2.48103000	4.83401500	-0.39970200
H	-2.61845700	4.40256100	1.30600000
C	-5.08400500	4.04154100	0.18221000
H	-5.03700600	4.93035100	-0.46081100
H	-5.99993700	3.49971900	-0.06610500
H	-5.16560600	4.39293000	1.21415000
C	-3.78488300	2.79899600	-1.53861200
H	-2.88884900	2.20957500	-1.77210300
H	-4.66082700	2.21252800	-1.83888100
H	-3.76505700	3.70293400	-2.16133500
C	-4.13184800	0.34360300	3.24125900
H	-3.55245700	-0.48278300	2.80990100
H	-3.97891100	0.32267900	4.32761100
H	-5.19205300	0.15078900	3.04928700
C	-2.20434100	1.89599300	3.03194200
H	-1.81374500	2.85904400	2.68721500
H	-2.07219100	1.86237700	4.12096500
H	-1.57745100	1.10298900	2.59980800
C	-4.52719900	2.80181800	3.32279600
H	-4.47445500	2.72183400	4.41649600
H	-4.17421800	3.80424900	3.05934400
H	-5.58187900	2.71856800	3.03971700
C	3.21688700	3.65099600	1.33807700
H	2.95998200	4.27018700	2.20710400
H	3.62576600	2.70627600	1.71474800
H	4.00971900	4.16362200	0.78517600
C	0.87512600	2.82756700	1.36433600
H	1.24826100	1.94987300	1.90782600
H	0.53828600	3.56168000	2.10881200
H	-0.00502300	2.50521200	0.79707100
C	1.51650200	4.80074400	-0.05517900
H	0.56578200	4.75481500	-0.59670700
H	1.37509100	5.49592300	0.78303100

H	2.27220200	5.24081300	-0.71484600
C	5.08396400	-4.04159900	0.18216100
H	5.03708200	-4.93023300	-0.46111000
H	5.99997000	-3.49973800	-0.06580400
H	5.16532200	-4.39327000	1.21402300
C	2.58613000	-3.98774600	0.29210500
H	1.68108800	-3.37844400	0.20068100
H	2.48105900	-4.83382700	-0.40043300
H	2.61814300	-4.40273700	1.30539100
C	0.08574200	-2.24918100	-2.10004600
H	0.46140500	-3.14917900	-1.60175400
H	0.63906200	-2.15565300	-3.04450800
H	0.34970100	-1.38034000	-1.48132700
C	-0.87540900	-2.82792800	1.36362000
H	-0.53878800	-3.56220300	2.10803800
H	0.00488500	-2.50556500	0.79658700
H	-1.24857300	-1.95029300	1.90718400
C	-1.51659400	-4.80076600	-0.05643100
H	-1.37538500	-5.49613600	0.78165500
H	-2.27216900	-5.24066200	-0.71635500
H	-0.56575400	-4.75474900	-0.59774500
C	-3.21720000	-3.65125900	1.33676100
H	-3.62612400	-2.70660600	1.71355000
H	-4.00994500	-4.16375000	0.78361100
H	-2.96047200	-4.27063700	2.20570900
C	-1.82650600	-1.10075200	-3.21119400
H	-2.90182700	-1.09608600	-3.42484700
H	-1.58047100	-0.16701000	-2.69079100
H	-1.29797500	-1.09760000	-4.17359100
C	-1.71582400	-3.58556600	-3.20986400
H	-1.18960100	-3.52403800	-4.17166100
H	-1.38202900	-4.50240600	-2.71761700
H	-2.78403800	-3.67958500	-3.42165900
C	4.52662500	-2.80260300	3.32290100
H	4.47370600	-2.72286400	4.41661100
H	4.17362700	-3.80495300	3.05916300
H	5.58135600	-2.71935200	3.04001600
C	2.20386600	-1.89658000	3.03185600
H	1.81324800	-2.85950200	2.68679100
H	2.07153900	-1.86326200	4.12086600
H	1.57710400	-1.10340800	2.59984400
C	4.13141900	-0.34434700	3.24187900
H	5.19167100	-0.15155000	3.05016000
H	3.55216100	0.48217500	2.81060100
H	3.97827000	-0.32366700	4.32820600
C	1.82700500	1.10135300	-3.21062500

H	1.58082400	0.16751800	-2.69045700
H	1.29867000	1.09841300	-4.17313000
H	2.90237000	1.09669000	-3.42406100
C	1.71633000	3.58616900	-3.20884200
H	1.19022800	3.52484600	-4.17071800
H	1.38250000	4.50292100	-2.71645600
H	2.78457400	3.68020200	-3.42048200
C	-0.08541700	2.24955900	-2.09956500
H	-0.46118800	3.14945700	-1.60117300
H	-0.63858300	2.15618200	-3.04413200
H	-0.34946000	1.38060000	-1.48104700
C	3.78522500	-2.79860800	-1.53863200
H	2.88925100	-2.20911100	-1.77216100
H	4.66124400	-2.21209800	-1.83860000
H	3.76549200	-3.70240500	-2.16156300

$[Ni^{III}\text{-}H\text{---}H\text{-}Ni^{III}]^{2+}$

E_{gas}	-3728.399775 hartree
E_{solv}	-3728.631869 hartree
G_{solv}	-3727.579169 hartree



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C	4.89233700	0.57963900	-0.48861200
C	5.94995200	-0.28008100	-0.18568300
C	7.24509300	-0.00716100	-0.58902900

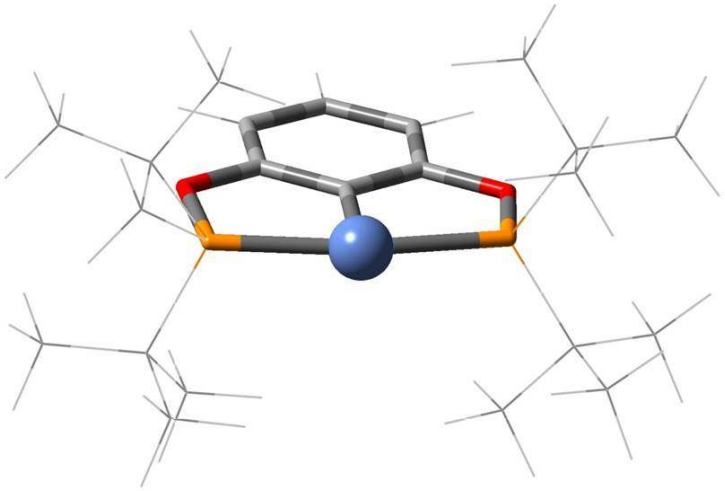
C	7.48865800	1.15205800	-1.31159700
H	6.64806200	2.94606800	-2.19058100
H	8.04033100	-0.70232900	-0.33544500
Ni	3.15059300	0.21038500	0.08942600
O	5.69851400	-1.42710300	0.52264000
O	4.16134400	2.60715200	-1.51662600
P	4.11151800	-1.61776400	0.92770500
P	2.72286400	2.19683400	-0.82507900
C	1.59564800	2.23716900	-2.30665400
C	2.43686200	3.52164900	0.45128800
C	3.70546700	-3.22944300	0.09541400
C	4.11989100	-1.62749500	2.78411700
H	8.50209700	1.37550800	-1.63251300
Ni	-3.15063100	-0.21040400	0.08894600
C	-4.89225100	-0.57955200	-0.48952900
C	-5.17954500	-1.73662300	-1.21555800
C	-5.94994300	0.28007900	-0.18661900
C	-6.46475900	-2.03340500	-1.63163400
C	-7.24500200	0.00721200	-0.59026700
C	-7.48840300	-1.15186400	-1.31312000
H	-6.64759900	-2.94567300	-2.19231600
H	-8.04030500	0.70230800	-0.33668800
H	-8.50177600	-1.37527300	-1.63427200
O	-5.69866400	1.42695400	0.52199800
O	-4.16101500	-2.60683400	-1.51783100
P	-2.72265900	-2.19661100	-0.82597000
P	-4.11175000	1.61754400	0.92741600
C	-4.12049000	1.62689900	2.78382800
C	-3.70553700	3.22938200	0.09551300
C	-1.59521000	-2.23665100	-2.30738300
C	-2.43681000	-3.52166400	0.45018600
H	0.13948000	0.35040800	0.92682400
H	-0.13963900	-0.35041200	0.92642500
C	-2.34037900	3.70852800	0.57063400
H	-1.58454900	2.91509000	0.49006500
H	-2.00661400	4.54245100	-0.06116500
H	-2.34990500	4.07245700	1.60331700
C	-4.77207700	4.29576400	0.31755600
H	-4.52933800	5.16967400	-0.30018700
H	-5.76522300	3.94954500	0.01800800
H	-4.82568200	4.63791700	1.35359700
C	-3.63326900	2.88907900	-1.39535900
H	-2.86026400	2.13975300	-1.61312300
H	-4.58644600	2.51431800	-1.78397900
H	-3.38631300	3.79653200	-1.96070700
C	-4.87328700	0.35820600	3.18913800

H	-4.42870700	-0.54688200	2.75442600
H	-4.83057300	0.25277000	4.27977400
H	-5.92820400	0.39108800	2.90092500
C	-2.67321600	1.51754400	3.26143000
H	-2.06361400	2.38964600	3.00228100
H	-2.66103400	1.42943800	4.35457600
H	-2.17672600	0.62014900	2.86453500
C	-4.80567300	2.84456100	3.38856600
H	-4.90072800	2.70051500	4.47207500
H	-4.23606400	3.76700200	3.23862000
H	-5.81641900	2.98875900	2.99184900
C	3.76212000	3.68980800	1.19517200
H	3.60628600	4.36370000	2.04632700
H	4.14647700	2.74277400	1.59309800
H	4.53751600	4.13009300	0.56165200
C	1.38749700	2.98495300	1.42353100
H	1.74087500	2.08440000	1.94584800
H	1.17816400	3.73969400	2.19209500
H	0.43495700	2.74918400	0.93471000
C	2.01031600	4.86373900	-0.12697700
H	1.01295100	4.83724800	-0.57935300
H	1.97681300	5.60591300	0.68055500
H	2.72258100	5.23339000	-0.87235700
C	4.77199500	-4.29584800	0.31740400
H	4.52935600	-5.16964600	-0.30053600
H	5.76517700	-3.94955600	0.01805900
H	4.82546300	-4.63819500	1.35338800
C	2.34025000	-3.70872000	0.57023500
H	1.58441300	-2.91527900	0.48976300
H	2.00658300	-4.54250000	-0.06180400
H	2.34965200	-4.07289200	1.60283300
C	-0.13578900	-2.18858500	-1.87160000
H	0.17868300	-3.09382000	-1.34154200
H	0.49867700	-2.10423500	-2.76386300
H	0.08678300	-1.31777200	-1.23627600
C	-1.38766500	-2.98506700	1.42272500
H	-1.17832800	-3.73997600	2.19112400
H	-0.43508900	-2.74901600	0.93410900
H	-1.74125300	-2.08470300	1.94522100
C	-2.01005700	-4.86361500	-0.12824900
H	-1.97662000	-5.60592800	0.67915800
H	-2.72217700	-5.23319400	-0.87380400
H	-1.01262400	-4.83696200	-0.58046400
C	-3.76219000	-3.69006300	1.19379500
H	-4.14669100	-2.74313200	1.59183000
H	-4.53743800	-4.13028500	0.56004900

H	-3.60646200	-4.36410300	2.04485200
C	-1.93452500	-0.96315300	-3.08530800
H	-2.98348000	-0.93463600	-3.40093800
H	-1.73126000	-0.05756000	-2.49893800
H	-1.31935700	-0.91702000	-3.99309700
C	-1.84647800	-3.45202900	-3.19533600
H	-1.25478200	-3.34407500	-4.11324600
H	-1.54745000	-4.39274600	-2.72702200
H	-2.89587000	-3.53308300	-3.49078800
C	4.80497200	-2.84527300	3.38873900
H	4.89981200	-2.70145000	4.47229600
H	4.23540700	-3.76769100	3.23848900
H	5.81579900	-2.98937300	2.99219100
C	2.67252000	-1.51826100	3.26145200
H	2.06298200	-2.39031900	3.00200200
H	2.66011600	-1.43037400	4.35461300
H	2.17609800	-0.62079200	2.86463700
C	4.87259000	-0.35887700	3.18983800
H	5.92756500	-0.39168500	2.90182800
H	4.42808500	0.54629500	2.75522500
H	4.82966000	-0.25366600	4.28048800
C	1.93511500	0.96383200	-3.08477600
H	1.73189000	0.05812600	-2.49857000
H	1.32001300	0.91780300	-3.99261400
H	2.98409600	0.93545400	-3.40033300
C	1.84701100	3.45272200	-3.19433800
H	1.25548600	3.34490700	-4.11237400
H	1.54784800	4.39333500	-2.72590400
H	2.89645200	3.53388700	-3.48958700
C	0.13616600	2.18898100	-1.87108400
H	-0.17839900	3.09411600	-1.34091100
H	-0.49817600	2.10476800	-2.76344800
H	-0.08646800	1.31805500	-1.23593400
C	3.63342500	-2.88884000	-1.39540000
H	2.86042800	-2.13949800	-1.61313400
H	4.58665100	-2.51396400	-1.78379000
H	3.38659200	-3.79618500	-1.96097400

Ni^{I}

E_{gas}	-1863.839937 hartree
E_{solv}	-1863.860744 hartree
G_{solv}	-1863.353832 hartree



	x	y	z
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C	-1.18416500	-2.07458200	-0.04061300
C	-1.21560400	-3.46223700	-0.03994800
C	0.00000000	-4.13914100	0.00000000
H	2.16198200	-3.99730700	0.07163000
H	-2.16198200	-3.99730700	-0.07163000
Ni	0.00000000	0.56479900	0.00000100
O	-2.37008700	-1.35994600	-0.08226400
O	2.37008700	-1.35994600	0.08226300
P	-2.18207400	0.30978100	-0.00755400
P	2.18207400	0.30978100	0.00755400
C	3.08464900	0.67241700	-1.59024700
C	4.56304900	0.31506400	-1.58831400
H	4.73207100	-0.71874300	-1.26776500
H	4.96506400	0.41509000	-2.60636400
H	5.15259900	0.97892700	-0.94658400
C	2.36511600	-0.16991800	-2.64521600
H	2.51224400	-1.24247600	-2.48394700
H	1.28467700	0.02164700	-2.65675900
H	2.76073700	0.08125200	-3.63854200
C	2.88697100	2.14632600	-1.93084400
H	3.38594400	2.81818800	-1.22452900
H	3.30327800	2.35179100	-2.92644300
H	1.82266400	2.41422300	-1.95252600
C	3.14739500	0.78165800	1.53880400
C	2.17224600	0.50106000	2.68475700
H	2.64834800	0.75167400	3.64247900

H	1.25919400	1.10370500	2.59336600
H	1.87688900	-0.55413700	2.72460800
C	3.45607100	2.27298600	1.50987600
H	2.56431000	2.87345300	1.28463300
H	3.82361600	2.59071600	2.49492400
H	4.23305600	2.52578500	0.77965600
C	4.41756500	-0.02802600	1.76390400
H	5.19195800	0.18079300	1.02070600
H	4.83612200	0.22429400	2.74830100
H	4.21598700	-1.10291000	1.75103900
C	-3.14739400	0.78165800	-1.53880400
C	-3.08465100	0.67241700	1.59024700
C	-3.45606800	2.27298700	-1.50987700
H	-2.56430700	2.87345300	-1.28463400
H	-3.82361300	2.59071700	-2.49492500
H	-4.23305200	2.52578700	-0.77965700
C	-4.41756500	-0.02802400	-1.76390300
H	-4.83612200	0.22429600	-2.74830000
H	-4.21598800	-1.10290800	-1.75103800
H	-5.19195800	0.18079600	-1.02070400
C	-2.17224500	0.50105900	-2.68475700
H	-1.25919300	1.10370200	-2.59336600
H	-1.87688900	-0.55413900	-2.72460700
H	-2.64834800	0.75167200	-3.64247800
C	-2.88697400	2.14632600	1.93084300
H	-3.30328200	2.35179100	2.92644200
H	-1.82266700	2.41422300	1.95252600
H	-3.38594700	2.81818700	1.22452700
C	-4.56305000	0.31506300	1.58831300
H	-4.96506600	0.41508800	2.60636200
H	-5.15259900	0.97892400	0.94658100
H	-4.73207100	-0.71874500	1.26776400
C	-2.36511700	-0.16991700	2.64521600
H	-2.51224400	-1.24247600	2.48394800
H	-1.28467800	0.02164800	2.65676000
H	-2.76073900	0.08125300	3.63854300
H	0.00000000	-5.22734100	0.00000000

Ni^{II+}

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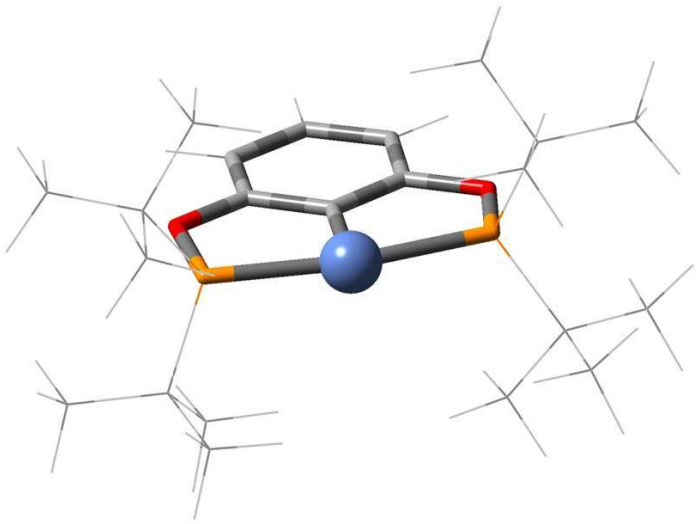
E_{solv}	-1863.724792 hartree
G_{solv}	-1863.217139 hartree



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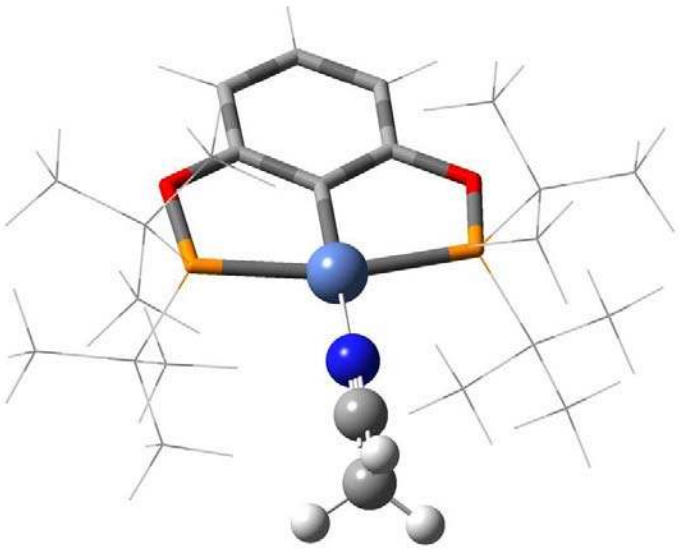
C	0.00000000	-1.36572100	0.00000200
C	-1.19628100	-2.08358400	-0.04459900
C	-1.20978700	-3.46779400	-0.04304900
C	0.00000100	-4.14618800	0.00000700
H	2.16065700	-3.99202500	0.07538600
H	-2.16065600	-3.99202700	-0.07537200
Ni	-0.00000100	0.50125200	-0.00000200
O	-2.38504400	-1.40241400	-0.09077800
O	2.38504400	-1.40241200	0.09078300
P	-2.20381000	0.23943300	-0.00648500
P	2.20380900	0.23943500	0.00648400
C	2.99522300	0.69289700	-1.61042000
C	4.50805000	0.52563800	-1.62365400
H	4.81353800	-0.48065900	-1.31641100
H	4.87558400	0.67909600	-2.64620300
H	5.01872800	1.25598500	-0.98833600
C	2.36812100	-0.23954900	-2.64829600
H	2.65958800	-1.28320600	-2.49587200
H	1.27184700	-0.18994500	-2.65040700
H	2.71234300	0.05987200	-3.64561800
C	2.59953300	2.13335000	-1.92986700
H	3.01363200	2.86208000	-1.22602300
H	2.97394500	2.39854000	-2.92611200
H	1.50815100	2.26432500	-1.95562300
C	3.08336600	0.80216300	1.54119100
C	2.13947700	0.42724400	2.68656400
H	2.60299200	0.70534800	3.64096400

H	1.18060200	0.95807500	2.62127300
H	1.93331700	-0.64865200	2.72416000
C	3.25664100	2.31624700	1.50426800
H	2.31440400	2.84446200	1.30211100
H	3.60920400	2.66125400	2.48385900
H	3.99769500	2.63928200	0.76608600
C	4.42201500	0.10118400	1.74290000
H	5.16596300	0.37712600	0.99181200
H	4.82617800	0.39228500	2.72073800
H	4.31821700	-0.98772700	1.74099300
C	-3.08336700	0.80215600	-1.54119400
C	-2.99522200	0.69290200	1.61041900
C	-3.25665700	2.31623800	-1.50426600
H	-2.31442500	2.84446200	-1.30210600
H	-3.60922100	2.66124500	-2.48385700
H	-3.99771500	2.63926400	-0.76608600
C	-4.42200900	0.10116500	-1.74291300
H	-4.82616800	0.39226200	-2.72075300
H	-4.31820100	-0.98774600	-1.74100500
H	-5.16596500	0.37710000	-0.99183000
C	-2.13947100	0.42725000	-2.68656500
H	-1.18060100	0.95809100	-2.62126800
H	-1.93329800	-0.64864400	-2.72416200
H	-2.60298500	0.70535100	-3.64096500
C	-2.59954700	2.13336400	1.92984600
H	-2.97395900	2.39856200	2.92609000
H	-1.50816700	2.26435200	1.95559600
H	-3.01365800	2.86208000	1.22599400
C	-4.50804600	0.52562600	1.62366600
H	-4.87557500	0.67909100	2.64621500
H	-5.01873900	1.25596000	0.98834400
H	-4.81352500	-0.48067800	1.31643700
C	-2.36810200	-0.23952300	2.64830200
H	-2.65955800	-1.28318500	2.49589300
H	-1.27182800	-0.18990700	2.65040400
H	-2.71232000	0.05990700	3.64562200
H	0.00000100	-5.23267400	0.00000900

Ni^{II}-$MeCN^{+}$

E_{gas}	-1996.393824 hartree
E_{solv}	-1996.475931 hartree
G_{solv}	-1995.923977 hartree



	x	y	z
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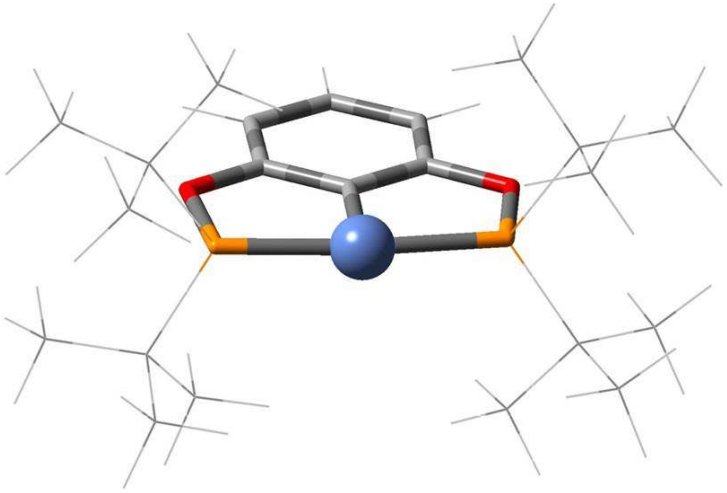
C	0.01554700	1.64472100	-0.01338300
C	1.20239100	2.37571000	-0.09670000
C	1.23467600	3.75955800	-0.11117900
C	0.03262900	4.44943300	-0.04443700
H	-2.12506600	4.30487300	0.08725200
H	2.18836700	4.27572700	-0.17317900
Ni	0.00388400	-0.25821200	0.00598600
O	2.39043200	1.68784300	-0.16205900
O	-2.35893800	1.72002100	0.13216200
P	2.19448800	0.05348500	-0.01087600
P	-2.18394100	0.08039900	0.01709200
C	3.06727900	-0.55237200	-1.54203600
C	3.35909400	-2.04523600	-1.44476500
H	3.72763300	-2.40053200	-2.41522900
H	4.13334900	-2.27629600	-0.70594100
H	2.46434300	-2.63316200	-1.20521600
C	4.35026300	0.22153500	-1.82948700
H	4.17321100	1.29857200	-1.88991500
H	5.13175500	0.04423900	-1.08634400
H	4.74540400	-0.10506000	-2.80001900
C	2.06799300	-0.30069500	-2.67428600
H	2.51862900	-0.59953300	-3.62905300
H	1.14498500	-0.88117500	-2.54607100
H	1.79588100	0.75795900	-2.75835900
C	3.05647300	-0.30393400	1.60048400
C	2.40404800	0.62741500	2.62398300
H	2.61584000	1.68129300	2.41947800

H	1.31502200	0.50148400	2.66735700
H	2.80354300	0.39524400	3.61892700
C	4.55375600	-0.02911500	1.54803500
H	4.96578900	-0.10553600	2.56248200
H	5.09400700	-0.75282600	0.92930500
H	4.77556800	0.97910800	1.18235800
C	2.78492500	-1.74891600	2.01004200
H	1.71270300	-1.93772600	2.13967500
H	3.18107400	-2.48207700	1.29895300
H	3.26885200	-1.94223200	2.97540200
C	-3.05363900	-0.30046500	-1.58529300
C	-4.54701200	-0.00434000	-1.53595500
H	-4.75429200	1.01425400	-1.19117600
H	-4.96222000	-0.09636700	-2.54780900
H	-5.09571400	-0.70739300	-0.90103800
C	-2.39099100	0.60015000	-2.62954800
H	-2.58896900	1.66071500	-2.44704100
H	-1.30369100	0.45959700	-2.67154500
H	-2.79497400	0.35216000	-3.61884700
C	-2.80378600	-1.75737600	-1.96665300
H	-3.20434100	-2.47014400	-1.23702600
H	-3.29832300	-1.96430200	-2.92375400
H	-1.73521300	-1.96226100	-2.10178700
C	-3.06525100	-0.48037000	1.56094100
C	-2.06070800	-0.22219700	2.68703400
H	-2.51491700	-0.49417800	3.64810800
H	-1.14749500	-0.82016700	2.56969800
H	-1.77116600	0.83335000	2.74930100
C	-3.38236800	-1.96984500	1.49528500
H	-2.49758200	-2.57818900	1.27073100
H	-3.75763200	-2.29790100	2.47272300
H	-4.16014200	-2.20376000	0.76097500
C	-4.33488600	0.32062900	1.83319400
H	-5.12050700	0.14021800	1.09515400
H	-4.73367400	0.02160400	2.81108100
H	-4.14012400	1.39564000	1.87003300
N	-0.01615800	-2.15301900	0.01424200
C	-0.05222500	-3.30235900	0.00221800
C	-0.11475200	-4.74126800	-0.02126900
H	0.71785500	-5.14993600	-0.60142700
H	-0.05928500	-5.13988400	0.99564200
H	-1.05380900	-5.07116500	-0.47617200
H	0.03923100	5.53603600	-0.05635000

Ni(0)$^{-1}$

E_{gas}	-2627.467572 hartree
E_{solv}	-2627.519236 hartree
G_{solv}	-2626.799587 hartree
$\nu_{imaginary}$	-258.9001 cm^{-1}



	x	y	z
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C	0.00000000	-1.34656400	0.00000000
C	-1.19271500	-2.04770300	-0.02416300
C	-1.21850600	-3.44159700	-0.02208800
C	0.00000100	-4.11817700	-0.00000100
H	2.16297100	-3.98497900	0.03941700
H	-2.16297000	-3.98497900	-0.03941900
Ni	0.00000000	0.55928600	0.00000000
O	-2.36185700	-1.32673900	-0.04836400
O	2.36185800	-1.32673900	0.04836400
P	-2.11162000	0.40690700	-0.00215300
P	2.11162000	0.40690700	0.00215300
C	3.11772600	0.67250200	-1.58263300
C	4.53647200	0.12683100	-1.56583500
H	4.56080900	-0.91855700	-1.24106500
H	4.96720200	0.17606200	-2.57911000
H	5.19707700	0.70908800	-0.91216400
C	2.31839200	-0.05603100	-2.66275900
H	2.31521200	-1.13925100	-2.50351900
H	1.27360000	0.27918100	-2.68193100
H	2.76115100	0.14644600	-3.65047600

C	3.13073900	2.15821400	-1.92221500
H	3.70502900	2.75023400	-1.20008400
H	3.58896100	2.31045900	-2.91216600
H	2.11316500	2.56588600	-1.94605800
C	3.15359300	0.75713100	1.55049200
C	2.15672500	0.54294300	2.68992900
H	2.63290500	0.77800900	3.65463700
H	1.27744400	1.18952900	2.57053900
H	1.80372000	-0.49458200	2.72806600
C	3.59251300	2.21518300	1.55906500
H	2.75559300	2.88904300	1.33555800
H	3.98510200	2.48135300	2.55258600
H	4.39184300	2.41192100	0.83349500
C	4.35451900	-0.14944300	1.78328100
H	5.15129000	0.00607400	1.04880800
H	4.78420600	0.05861500	2.77666700
H	4.06750600	-1.20373200	1.74904000
C	-3.15359400	0.75713200	-1.55049100
C	-3.11772600	0.67250100	1.58263300
C	-3.59250900	2.21518500	-1.55906500
H	-2.75558600	2.88904300	-1.33556100
H	-3.98509900	2.48135400	-2.55258700
H	-4.39183600	2.41192600	-0.83349400
C	-4.35452300	-0.14943800	-1.78327700
H	-4.78421100	0.05861900	-2.77666200
H	-4.06751200	-1.20372900	-1.74903600
H	-5.15129100	0.00608100	-1.04880300
C	-2.15672700	0.54293900	-2.68992900
H	-1.27744400	1.18952200	-2.57054200
H	-1.80372500	-0.49458700	-2.72806500
H	-2.63290700	0.77800500	-3.65463700
C	-3.13074200	2.15821300	1.92221300
H	-3.58896600	2.31045800	2.91216400
H	-2.11317000	2.56588800	1.94605600
H	-3.70503400	2.75023100	1.20008200
C	-4.53647100	0.12682700	1.56583500
H	-4.96720300	0.17605900	2.57911000
H	-5.19707700	0.70908100	0.91216200
H	-4.56080500	-0.91856300	1.24106800
C	-2.31839100	-0.05602900	2.66276000
H	-2.31520900	-1.13924900	2.50352100
H	-1.27359900	0.27918500	2.68193100
H	-2.76115000	0.14644900	3.65047700
H	0.00000100	-5.20892900	-0.00000100

Aniline

E_{gas}	-287.4361761 hartree
E_{solv}	-287.4510835 hartree
G_{solv}	-287.3642095 hartree

	x	y	z
H	-2.95793400	-0.00000100	0.01588300
C	-1.87110400	0.00000000	0.00802000
C	-1.16482200	-1.19513300	0.00348200
H	-1.69958300	-2.14290900	0.00761300
C	0.21975400	-1.20040200	-0.00516900
H	0.76373200	-2.14485000	-0.01481900
C	0.93454000	0.00000000	-0.00882500
N	2.32160800	0.00000300	-0.07399300
H	2.76929200	-0.83749300	0.27076800
H	2.76929300	0.83747800	0.27081600
C	0.21975300	1.20040200	-0.00517200
H	0.76373200	2.14485000	-0.01482800
C	-1.16482300	1.19513300	0.00348200
H	-1.69958400	2.14290900	0.00761300

Aniline-rad

E_{gas}	-286.7866978 hartree
E_{solv}	-286.7995294 hartree
G_{solv}	-286.7261494 hartree

	x	y	z
H	2.88522900	0.05698700	-0.00001700
C	1.79813100	0.03249900	-0.00000600
C	1.07410600	1.22848800	0.00000000
H	1.60349100	2.17877800	0.00000100
C	-0.29991400	1.20693900	0.00000600
H	-0.86825200	2.13693000	0.00001600
C	-1.01729900	-0.02948000	0.00000300
N	-2.33965500	-0.13698800	-0.00000900
H	-2.75255200	0.80193200	-0.00001500
C	-0.24646600	-1.23218900	0.00000700
H	-0.79787300	-2.16931400	0.00001300
C	1.12663300	-1.19393100	-0.00000100
H	1.69640000	-2.12035500	0.00000000

TEMPO-H

E_{gas}	-484.0666437 hartree
E_{solv}	-484.0785718 hartree
G_{solv}	-483.8407558 hartree

	x	y	z
C	-0.04940000	-0.04362200	1.30472800
C	1.46386100	-0.32011800	1.21976000
C	2.10420500	0.31208800	0.00000000
C	1.46386100	-0.32011800	-1.21976000
C	-0.04940000	-0.04362200	-1.30472800
N	-0.51615900	0.46984500	0.00000000
H	1.96531500	1.40175800	0.00000000
H	1.93844200	0.01677200	2.15043000
H	1.93844200	0.01677200	-2.15043000
H	1.63978500	-1.40569500	1.16605300
H	3.18573000	0.12782700	0.00000000
H	1.63978500	-1.40569500	-1.16605300
C	-0.77288700	-1.30470000	1.77900900
H	-0.33957600	-1.64466600	2.72728400
H	-1.83834400	-1.12812500	1.96758500
H	-0.67321700	-2.13141200	1.06550700
C	-0.29948900	1.07836000	2.30755400
H	-1.35152900	1.37583500	2.30673900
H	-0.02678900	0.75871500	3.32127600
H	0.29977000	1.95796700	2.04689200
C	-0.29948900	1.07836000	-2.30755400
H	-0.02678900	0.75871500	-3.32127600
H	-1.35152900	1.37583500	-2.30673900
H	0.29977000	1.95796700	-2.04689200
C	-0.77288700	-1.30470000	-1.77900900
H	-1.83834400	-1.12812500	-1.96758500
H	-0.33957600	-1.64466600	-2.72728400
H	-0.67321700	-2.13141200	-1.06550700
O	-1.90733000	0.63087200	0.00000000
H	-2.30663500	-0.25581700	0.00000000

TEMPO

E_{gas}	-483.4688271 hartree
E_{solv}	-483.4787812 hartree
G_{solv}	-483.2540442 hartree

	x	y	z
C	-0.07225800	-0.02103400	1.31738500
C	1.34542500	-0.58049500	1.23346100
C	2.10298100	-0.13118900	0.00000000
C	1.34542500	-0.58049500	-1.23346100
C	-0.07225800	-0.02103400	-1.31738500
N	-0.75992300	-0.16935600	0.00000000
H	2.24585700	0.95832800	0.00000000
H	1.87586700	-0.30192000	2.15456000
H	1.87586700	-0.30192000	-2.15456000
H	1.28912900	-1.68019300	1.22966800
H	3.11067700	-0.56456700	0.00000000
H	1.28912900	-1.68019300	-1.22966800
C	-0.87073700	-0.81827900	2.33651200
H	-0.35635700	-0.79684700	3.30455200
H	-1.87432000	-0.40698800	2.46298900
H	-0.96842700	-1.86273500	2.02034900
C	-0.07225800	1.45215000	1.72027200
H	-1.08273100	1.86197600	1.62361600
H	0.24095200	1.55619100	2.76604400
H	0.60326200	2.06051400	1.11072600
C	-0.07225800	1.45215000	-1.72027200
H	0.24095200	1.55619100	-2.76604400
H	-1.08273100	1.86197600	-1.62361600
H	0.60326200	2.06051400	-1.11072600
C	-0.87073700	-0.81827900	-2.33651200
H	-1.87432000	-0.40698800	-2.46298900
H	-0.35635700	-0.79684700	-3.30455200
H	-0.96842700	-1.86273500	-2.02034900
O	-2.00897100	0.04134500	0.00000000

4-^{MeO}tempoH

E_{gas}	-598.5506635 hartree
E_{solv}	-598.5647672 hartree
G_{solv}	-598.2989662 hartree

	x	y	z
C	-0.38036600	1.31647300	0.07069500
C	0.95157100	0.80455500	0.62676200
C	1.46742400	-0.37651800	-0.17010400
C	0.53408600	-1.55440800	0.02360900

C	-0.96619600	-1.18449000	0.01684400
N	-1.08470400	0.17340200	-0.55499200
H	1.51792300	-0.10852000	-1.24119700
H	1.67057000	1.63470500	0.62909800
H	0.74866400	-2.30784300	-0.74361700
H	0.85131500	0.48244000	1.67421900
H	0.80063100	-2.01248600	0.98634800
C	-1.16483700	2.01515200	1.18311600
H	-0.52527800	2.76814400	1.65841200
H	-2.04128100	2.54565700	0.79496100
H	-1.49098800	1.32849800	1.97166700
C	-0.12777100	2.32635800	-1.04405700
H	-1.06819700	2.58352900	-1.54036900
H	0.31645600	3.24672500	-0.64416500
H	0.54819600	1.91610900	-1.80163200
C	-1.69998200	-2.13382700	-0.92224300
H	-1.50635300	-3.17125300	-0.62425000
H	-2.78034600	-1.96827700	-0.91263500
H	-1.34579200	-1.99266500	-1.94871200
C	-1.56539100	-1.33733400	1.41990300
H	-2.61562800	-1.01908400	1.45803500
H	-1.55225200	-2.39307500	1.71586000
H	-1.00992400	-0.78382300	2.18353700
O	-2.41809100	0.50869400	-0.81930700
H	-2.87697100	0.60408100	0.03294600
O	2.74531900	-0.79562400	0.25878200
C	3.79335800	-0.01547700	-0.23446600
H	3.75641700	1.02521600	0.12478500
H	4.72781100	-0.46319600	0.11218200
H	3.79876000	0.00384200	-1.33668000

4-^{MeO}tempo

E_{gas}	-597.951023 hartree
E_{solv}	-597.9638451 hartree
G_{solv}	-597.7108941 hartree

	x	y	z
C	-0.41999900	1.35923000	0.03290300
C	0.99472700	0.96965000	0.44990900
C	1.44230400	-0.38174700	-0.07519100
C	0.48361800	-1.43586700	0.42300500
C	-0.96291700	-1.21464100	-0.00032400
N	-1.35012300	0.20873900	0.22641000
H	1.46979000	-0.37224800	-1.18250200

H	1.67173800	1.77114100	0.12376300
H	0.81131800	-2.42635200	0.08072000
H	1.06041400	0.92776000	1.54791700
H	0.55215900	-1.44568700	1.52113200
C	-0.91000200	2.49109000	0.92268700
H	-0.21523700	3.33679500	0.86050900
H	-1.90302300	2.82858300	0.61859000
H	-0.96528000	2.16521700	1.96705700
C	-0.47518900	1.80948000	-1.42635100
H	-1.51762400	1.95211300	-1.72830500
H	0.04979400	2.76474200	-1.54580900
H	-0.01606200	1.09138900	-2.11295600
C	-1.18427600	-1.56776900	-1.47023100
H	-1.08646000	-2.65012900	-1.61522700
H	-2.19368200	-1.27158400	-1.77301400
H	-0.46920600	-1.07927100	-2.13960200
C	-1.87393300	-2.07468700	0.86161700
H	-2.91606100	-1.98068800	0.54934800
H	-1.57374000	-3.12569800	0.77882300
H	-1.80448900	-1.77764200	1.91370700
O	-2.58536200	0.47147700	0.13374500
O	2.71822200	-0.74264700	0.40177600
C	3.77331300	-0.03089900	-0.17520900
H	3.79723100	1.02440000	0.13688400
H	4.70300900	-0.50382700	0.14954700
H	3.72751200	-0.06388000	-1.27649900

NHPI

E_{gas}	-588.0183331 hartree
E_{solv}	-588.0391103 hartree
G_{solv}	-587.9547183 hartree

	x	y	z
C	-0.49761800	0.69461100	0.00278400
C	-0.49761800	-0.69461100	0.00278400
C	-1.67273300	-1.41509300	0.00173900
C	-2.86574800	-0.69601900	-0.00467200
C	-2.86574800	0.69601900	-0.00467300
C	-1.67273300	1.41509300	0.00173900
C	0.90546900	1.18208200	0.01004700
C	0.90546900	-1.18208200	0.01004000
H	-1.65650400	-2.50230800	0.00199000
H	-3.81386000	-1.22808900	-0.01077900
H	-3.81386000	1.22809000	-0.01077800

H	-1.65650400	2.50230900	0.00199200
O	1.33472300	-2.29760000	-0.01576400
O	1.33472400	2.29759900	-0.01576700
O	3.00720900	0.00000000	-0.13961200
H	3.42703400	-0.00000200	0.73172800
N	1.66685700	0.00000000	0.07660700

Nhpi-rad

E_{gas}	-587.3874377 hartree
E_{solv}	-587.4071712 hartree
G_{solv}	-587.3347502 hartree

	x	y	z
C	-0.44731500	0.69627800	0.00000000
C	-0.44731500	-0.69627800	0.00000000
C	-1.62637300	-1.41505700	0.00000000
C	-2.81745900	-0.69700000	0.00000000
C	-2.81745900	0.69700000	0.00000000
C	-1.62637300	1.41505700	0.00000000
C	0.93828200	1.20074600	0.00000000
C	0.93828200	-1.20074600	0.00000000
H	-1.61023800	-2.50206400	0.00000000
H	-3.76560200	-1.22863800	0.00000000
H	-3.76560200	1.22863800	0.00000000
H	-1.61023800	2.50206400	0.00000000
O	1.36904700	-2.31006200	0.00000000
O	1.36904700	2.31006200	0.00000000
O	3.00063900	0.00000000	0.00000000
N	1.75374100	0.00000000	0.00000000

3-F-NHPI

E_{gas}	-687.2528679 hartree
E_{solv}	-687.2737411 hartree
G_{solv}	-687.1983881 hartree

	x	y	z
C	-0.44301400	0.37861600	0.00024700
C	-0.12200600	-0.97645200	0.00240300
C	-1.07901100	-1.96529100	0.00168100

C	-2.41414800	-1.56400100	-0.00523400
C	-2.75810000	-0.21969200	-0.00555700
C	-1.76856600	0.75589100	0.00059800
C	0.80805300	1.18016600	0.00785100
C	1.35834400	-1.12210000	0.01208100
H	-0.79127300	-3.01315200	0.00288600
H	-3.20427300	-2.31025800	-0.01133900
H	-3.79756000	0.09698300	-0.01146800
O	2.02783900	-2.11214700	-0.01520600
O	0.98601900	2.35971000	-0.01985900
O	3.12330700	0.51240500	-0.13886000
H	3.52106700	0.66186200	0.73012500
N	1.82100700	0.19972700	0.07992800
F	-2.11796000	2.03154500	0.00414300

3-F-NHPI-rad

E_{gas}	-686.6213027 hartree
E_{solv}	-686.6410459 hartree
G_{solv}	-686.5778659 hartree

	x	y	z
C	0.40644900	0.37757200	0.00000000
C	0.05051700	-0.97214200	0.00000000
C	0.98748000	-1.98248300	0.00000000
C	2.33038000	-1.61357800	0.00000000
C	2.70817000	-0.27688800	0.00000000
C	1.74373100	0.72128000	0.00000000
C	-0.80110600	1.22408400	-0.00000100
C	-1.42067800	-1.09990000	0.00000000
H	0.67568900	-3.02324900	0.00000000
H	3.10212900	-2.37860400	0.00000000
H	3.75518100	0.01355200	0.00000000
O	-2.11609700	-2.06590800	0.00000000
O	-0.95051100	2.40215100	0.00000100
O	-3.10108800	0.58707600	0.00000000
N	-1.89710700	0.26399400	0.00000000
F	2.11762900	1.98734600	0.00000000

4-Me-NHPI

E_{gas}	-627.3115536 hartree
E_{solv}	-627.3329414 hartree
G_{solv}	-627.2239014 hartree

	x	y	z
C	0.21161400	-0.52558900	0.00469700
C	-0.02793200	0.84349000	0.00233900
C	1.01323200	1.74511800	0.00475700
C	2.30873800	1.23450100	0.00334300
C	2.56486200	-0.13781900	0.00403600
C	1.48939400	-1.03397400	0.00905500
C	-1.08750100	-1.24681800	0.01194900
C	-1.49068300	1.08391000	0.00737700
H	0.82108100	2.81545800	0.00566600
H	3.15279200	1.92238900	0.00146400
H	1.65551400	-2.11065400	0.01301000
O	-2.10820200	2.10762100	-0.01943300
O	-1.31847600	-2.41995300	-0.01178000
O	-3.35977600	-0.44261500	-0.14391100
H	-3.77690500	-0.50080600	0.72672500
N	-2.03938400	-0.21289000	0.07485000
C	3.96732100	-0.65924000	-0.01418700
H	4.12200700	-1.41647400	0.76240600
H	4.19700200	-1.13790800	-0.97378800
H	4.70154100	0.13633600	0.14135900

4-Me-NHPI rad

E_{gas}	-626.6811654 hartree
E_{solv}	-626.7016505 hartree
G_{solv}	-626.6057775 hartree

	x	y	z
C	0.16739000	-0.53259100	0.00000000
C	-0.08623600	0.83754100	0.00000000
C	0.95059300	1.74809500	-0.00000100
C	2.24872700	1.25067800	-0.00000100
C	2.51891600	-0.12122800	-0.00000100
C	1.45317100	-1.02616800	0.00000000
C	-1.10437600	-1.27957600	0.00000000
C	-1.53675300	1.08413100	0.00000000
H	0.74825500	2.81634800	-0.00000100
H	3.08602300	1.94639900	-0.00000100
H	1.62954100	-2.10102600	0.00000000
O	-2.16460600	2.09536800	0.00000100
O	-1.32681600	-2.44912500	0.00000000
O	-3.34891700	-0.47162300	0.00000000
N	-2.12255000	-0.24680200	0.00000000
C	3.92546600	-0.62913700	0.00000100
H	4.12002300	-1.25324200	0.87976500

H	4.12001900	-1.25326100	-0.87975000
H	4.65530100	0.18497200	-0.00000900

4-OMe-NHPI

E_{gas}	-702.5082055 hartree
E_{solv}	-702.5306304 hartree
G_{solv}	-702.4171804 hartree

	x	y	z
C	0.18721900	-0.54897000	-0.00579700
C	0.39072800	0.82904000	-0.00314200
C	-0.67881000	1.69516000	-0.00382000
C	-1.96508000	1.16131300	-0.00101700
C	-2.16264600	-0.22305600	-0.00316200
C	-1.06690200	-1.09960200	-0.00870800
C	1.50780900	-1.23479400	-0.01106500
C	1.84113700	1.10899700	-0.00839000
H	-0.51969300	2.77101300	-0.00278500
H	-2.81540000	1.83577300	0.00397100
H	-1.22966000	-2.17436200	-0.01042600
O	2.43540100	2.14698800	0.01596800
O	1.76888400	-2.40145300	0.01552600
O	3.75367800	-0.36668000	0.14512700
H	4.17462500	-0.39936300	-0.72496100
N	2.42736200	-0.17515700	-0.07455300
O	-3.36973200	-0.81435500	-0.00001400
C	-4.52051400	-0.00419600	0.01162000
H	-4.57334400	0.63263900	-0.88104300
H	-5.37180900	-0.68484000	0.01543900
H	-4.55974500	0.62588400	0.90970300

4-OMe-NHPI-Rad

E_{gas}	-701.878258 hartree
E_{solv}	-701.8999291 hartree
G_{solv}	-701.7983721 hartree

	x	y	z
C	0.23390700	-0.55799500	0.00000300
C	0.44559800	0.82236000	0.00000400
C	-0.62447200	1.69318500	0.00001400
C	-1.91040900	1.16711100	0.00001400

C	-2.11656500	-0.21912400	0.00000300
C	-1.02565500	-1.09989700	0.00000200
C	1.53165800	-1.26328900	0.00000200
C	1.88165000	1.11602100	-0.00000300
H	-0.45953600	2.76795800	0.00002500
H	-2.75755400	1.84547100	0.00002700
H	-1.19366100	-2.17369700	0.00000600
O	2.48122900	2.14484400	-0.00000800
O	1.78986500	-2.42558300	0.00000800
O	3.74530400	-0.38202900	-0.00000900
N	2.51226500	-0.19878300	-0.00000700
O	-3.32376000	-0.80043400	0.00000600
C	-4.47465800	0.01368000	-0.00002000
H	-4.51819000	0.64562600	-0.89601500
H	-5.32611000	-0.66622200	-0.00002600
H	-4.51822100	0.64565800	0.89594900

4-OAc-NHPI

E_{gas}	-815.8334537 hartree
E_{solv}	-815.8579658 hartree
G_{solv}	-815.7396778 hartree

	x	y	z
C	0.79570200	-0.52161200	-0.08813400
C	1.07903100	0.83726600	-0.12162600
C	0.07339700	1.77158500	-0.25243000
C	-1.23810000	1.31719800	-0.34028900
C	-1.50819000	-0.04870000	-0.29823900
C	-0.49338000	-0.99361800	-0.17571800
C	2.06504000	-1.28352600	0.04402400
C	2.54506800	1.03142900	-0.01363100
H	0.30350900	2.83366400	-0.28302200
H	-2.05586100	2.02244400	-0.43490900
H	-0.72328400	-2.05566100	-0.15255400
O	3.19239600	2.03560800	0.02284800
O	2.24924300	-2.46116000	0.13572500
O	4.34101400	-0.54389600	0.35866800
H	4.82781300	-0.65680800	-0.46946000
N	3.05279000	-0.28386400	0.01788400
O	-2.77968600	-0.55605700	-0.44580700
C	-3.84082600	-0.02093400	0.23802200
C	-5.07434900	-0.79012500	-0.10059400
H	-5.22954100	-0.80198000	-1.18319600
H	-4.96473400	-1.83132000	0.21733400
H	-5.93486500	-0.34293000	0.39643700

O	-3.75458300	0.90373800	0.98305000

4-OAc-NHPI-Rad

E_{gas}	-815.2026991 hartree
E_{solv}	-815.2258964 hartree
G_{solv}	-815.1194394 hartree

	x	y	z
C	0.84476600	-0.53669500	-0.07485400
C	1.13221300	0.82477200	-0.11220400
C	0.12360000	1.75984400	-0.24274900
C	-1.18660600	1.30911500	-0.33014300
C	-1.46162400	-0.05876500	-0.28300600
C	-0.44859000	-1.00331300	-0.15689200
C	2.09158600	-1.31366800	0.06184000
C	2.58430500	1.03688300	-0.00401900
H	0.35589500	2.82115400	-0.27736400
H	-2.00151300	2.01646600	-0.43052600
H	-0.68053000	-2.06468600	-0.12992800
O	3.23681800	2.03181200	0.00243300
O	2.27825500	-2.48723300	0.12846100
O	4.34797700	-0.55977000	0.21358800
N	3.13225700	-0.30659300	0.10268600
O	-2.72950400	-0.56423000	-0.42816500
C	-3.80170700	-0.00411100	0.22561200
C	-5.03261300	-0.77442400	-0.11425800
H	-5.17230400	-0.80759600	-1.19854200
H	-4.93211800	-1.80939200	0.22608800
H	-5.89788200	-0.31331300	0.36115700
O	-3.71721100	0.93763200	0.94797900

4-NO_2-Phenol

E_{gas}	-511.7775415 hartree
E_{solv}	-511.7944508 hartree
G_{solv}	-511.7204368 hartree

	x	y	z
C	0.00337600	-1.20515400	0.00000200
C	0.68532900	0.00048100	-0.00000100
C	0.01105400	1.21467600	-0.00000200

C	-1.36691500	1.22174800	0.00000100
C	-2.06789900	0.01551400	0.00000400
C	-1.37798000	-1.19653500	0.00000400
H	0.56450200	-2.13370600	0.00000200
O	-3.41086400	0.08543000	0.00000600
N	2.15174700	-0.00697700	-0.00000400
O	2.71822000	1.06659700	-0.00000600
O	2.70795200	-1.08635800	-0.00000300
H	-1.92891500	-2.13648500	0.00000700
H	-3.79309500	-0.79888300	0.00000800
H	-1.93024800	2.15050100	0.00000000
H	0.58126800	2.13767100	-0.00000400

4-NO_2-phenol-cb

E_{gas}	-511.2511975 hartree
E_{solv}	-511.3261182 hartree
G_{solv}	-511.2649532 hartree

	x	y	z
C	-0.05146800	-1.21458200	0.00000200
C	0.66216000	0.00000800	0.00000000
C	-0.05147500	1.21458300	-0.00000100
C	-1.41463200	1.21834400	0.00000200
C	-2.20629600	0.00000800	0.00000600
C	-1.41462000	-1.21833900	0.00000400
H	0.51814800	-2.14073600	0.00000200
O	-3.44602200	-0.00001000	0.00000500
N	2.07537800	0.00000300	-0.00000300
O	2.67541400	1.07983700	-0.00000800
O	2.67538100	-1.07985000	-0.00000500
H	-1.97206200	-2.15525400	0.00000500
H	-1.97205100	2.15527400	0.00000100
H	0.51812900	2.14074700	-0.00000300

3-5-dinitro-phenol

E_{gas}	-716.2343592 hartree
E_{solv}	-716.2523381 hartree
G_{solv}	-716.1801571 hartree

x	y	z

C	-0.00872300	-0.89560100	0.00000100
C	-1.17763000	-0.15523400	0.00000000
C	-1.19910500	1.22410100	0.00000000
C	0.01041500	1.91459300	0.00000100
C	1.20675800	1.20790000	0.00000100
C	1.16759000	-0.17604200	0.00000100
H	-0.01738900	-1.97875000	0.00000100
N	-2.47228900	-0.87999500	-0.00000200
O	-3.48047400	-0.21074300	0.00000600
O	-2.42602800	-2.08871400	-0.00001200
H	-2.13982900	1.76445500	-0.00000100
H	2.17138300	1.70957800	0.00000000
N	2.45329200	-0.91260800	0.00000100
O	2.39881400	-2.12024300	0.00001800
O	3.46832000	-0.25083400	-0.00001600
O	-0.04961600	3.25745100	0.00000200
H	0.83486200	3.63930000	0.00000400

3-5-dinitro-phenol -cb

E_{gas}	-715.7161853 hartree
E_{solv}	-715.7835892 hartree
G_{solv}	-715.7251112 hartree

	x	y	z
C	0.00000000	-0.87019400	0.00000000
C	-1.16937000	-0.11863000	0.00000000
C	-1.20601200	1.25880500	0.00000000
C	0.00000700	2.05550900	0.00000000
C	1.20601800	1.25880400	0.00000000
C	1.16936500	-0.11863200	0.00000000
H	0.00000000	-1.94983800	0.00000000
N	-2.44992000	-0.85820900	0.00000000
O	-3.48637300	-0.22180200	-0.00000100
O	-2.40900900	-2.07556500	0.00000000
H	-2.15947500	1.77757900	0.00000000
H	2.15948600	1.77756600	0.00000000
N	2.44992000	-0.85821000	0.00000000
O	2.40900400	-2.07556800	0.00000000
O	3.48637000	-0.22180800	-0.00000100
O	0.00000100	3.29669900	0.00000000

3-Cl-4-NO_2-ph

E_{gas}	-971.3577676 hartree

E_{solv}	-971.3758222 hartree
G_{solv}	-971.3121592 hartree

	x	y	z
C	-0.37958300	-1.62510500	-0.00000100
C	0.49463800	-0.54376600	-0.00000100
C	-0.02767000	0.75847000	0.00000000
C	-1.40030900	0.93535000	0.00000300
C	-2.26230600	-0.15446900	0.00000300
C	-1.74436600	-1.44768200	0.00000100
H	0.05370200	-2.61922100	-0.00000200
O	-3.57879400	0.10773400	0.00000500
N	1.93301600	-0.87101000	-0.00000400
O	2.73250600	0.03599300	-0.00001400
O	2.22632400	-2.05042300	0.00000300
H	-2.40915800	-2.31004600	0.00000200
H	-4.08527900	-0.71191900	0.00000500
H	-1.81939700	1.93685700	0.00000400
Cl	0.91801800	2.20695100	0.00000200

3-Cl-4-NO_2-ph -cb

E_{gas}	-970.8404041 hartree
E_{solv}	-970.9126022 hartree
G_{solv}	-970.8648602 hartree

	x	y	z
C	-0.43331000	-1.63449900	0.00000000
C	0.47849000	-0.55358300	-0.00000100
C	-0.08692500	0.74895500	0.00000000
C	-1.43786000	0.93227600	0.00000200
C	-2.39665200	-0.15795100	0.00000200
C	-1.78229300	-1.46824100	0.00000100
H	0.00586300	-2.62765900	-0.00000100
O	-3.61932500	0.03016700	0.00000300
N	1.86707000	-0.86736000	-0.00000400
O	2.70026700	0.03286700	-0.00001000
O	2.19477300	-2.05780100	0.00000200
H	-2.45201700	-2.32757100	0.00000100
H	-1.84716500	1.93984100	0.00000400
Cl	0.88055400	2.22607700	0.00000200

4-Me-benzoicacid

E_{gas}	-895.1507629 hartree
E_{solv}	-895.1709416 hartree
G_{solv}	-895.0664736 hartree

	x	y	z
C	-0.12301800	0.03106300	-0.07602800
C	0.56636500	1.23005600	-0.01723900
C	1.95218500	1.20110600	0.02672700
C	2.64611500	-0.00470500	0.01406200
C	1.92058400	-1.19686300	-0.04670600
C	0.54018500	-1.18944800	-0.09083800
H	0.01932300	2.16868900	-0.01551400
H	2.50601700	2.13716500	0.06971800
H	2.45542700	-2.14549000	-0.06284600
H	-0.02546400	-2.11571600	-0.15328300
S	-1.88806300	0.04644800	-0.12283600
O	-2.37974300	-1.08821300	-0.87151500
O	-2.33902000	1.38207200	-0.40622300
O	-2.19549900	-0.27897600	1.43907000
H	-2.91673800	-0.92170200	1.49839200
C	4.14107000	-0.04012800	0.05077900
H	4.54911600	-0.45696500	-0.87767200
H	4.56985500	0.95737100	0.18236500
H	4.50464700	-0.67207400	0.86902600

4-Me-benzoicacid -cb

E_{gas}	-894.6369678 hartree
E_{solv}	-894.7301385 hartree
G_{solv}	-894.6377895 hartree

	x	y	z
C	0.16662300	0.02420500	0.00000300
C	-0.53781100	1.21482700	-0.00001000
C	-1.92907400	1.20272100	-0.00003200
C	-2.63538500	0.00667000	-0.00003500
C	-1.91035000	-1.18728900	-0.00003200
C	-0.52756600	-1.18157100	-0.00001000
H	0.02712800	2.14442800	-0.00001600
H	-2.47803600	2.14626500	-0.00005300
H	-2.45069200	-2.13639100	-0.00005500
H	0.03802300	-2.11159800	-0.00001900
S	1.97805100	-0.00102300	0.00000700

O	2.31100900	-0.72790400	1.23331200
O	2.36345000	1.41738000	0.00003900
O	2.31101700	-0.72785000	-1.23332700
C	-4.13443400	-0.01851100	0.00004800
H	-4.53293900	-0.53866900	0.88136200
H	-4.55513400	0.99350400	-0.00096500
H	-4.53300100	-0.54049200	-0.88015200

3-CN-benzesnesulfonamide

E_{gas}	-928.1879955 hartree
E_{solv}	-928.2127497 hartree
G_{solv}	-928.1250417 hartree

	x	y	z
C	2.10656700	-0.05647100	0.00613100
C	0.81457300	-0.57923900	-0.06106100
C	-0.25377500	0.29652900	-0.07527500
C	-0.07333000	1.67226300	-0.03247300
C	1.21330300	2.18196900	0.03288300
C	2.30172800	1.32410600	0.05496100
H	0.64757800	-1.65152800	-0.11794000
H	-0.93683700	2.33171300	-0.06631300
H	1.37051900	3.25651700	0.06198200
H	3.31396900	1.71645800	0.10490300
S	-1.91283500	-0.35751800	-0.14728200
O	-1.80389600	-1.76003900	-0.47888400
O	-2.72010300	0.57226400	-0.90403800
N	-2.41582300	-0.21850500	1.43895700
H	-3.30051100	0.27426700	1.49826000
H	-2.43866700	-1.11328400	1.91601300
C	3.23154600	-0.93546400	0.02175000
N	4.14405500	-1.64061000	0.03839800

3-CN-benzesnesulfonamide -cb

E_{gas}	-927.6427326 hartree
E_{solv}	-927.7299218 hartree
G_{solv}	-927.6553848 hartree

	x	y	z
C	2.08715900	-0.06329000	0.00007000
C	0.78923000	-0.58484800	-0.04331800

C	-0.29590500	0.27258900	-0.04927300
C	-0.08975300	1.64874400	-0.01372900
C	1.19474100	2.16705300	0.02314500
C	2.29138600	1.31712700	0.03261100
H	0.60969600	-1.65722900	-0.08078100
H	-0.96047800	2.30190800	-0.01327500
H	1.34820200	3.24466300	0.04652800
H	3.30536300	1.70993600	0.06443200
S	-2.01380100	-0.35913800	-0.02799900
O	-1.81839800	-1.81717800	-0.14328200
O	-2.60022700	0.32988300	-1.18033400
N	-2.64166800	0.14762800	1.30345300
H	-2.40192800	-0.53606100	2.02336000
C	3.21705200	-0.93480800	0.00855400
N	4.14245400	-1.62677900	0.01773200

3,7-dinitro-phenoxazine

E_{gas}	-1001.29174133 hartree
E_{solv}	-1001.31677401 hartree
G_{solv}	-1001.17800601 hartree

	x	y	z
C	3.59294400	-0.14002200	0.00000000
C	2.42949500	-0.89822900	0.00000100
C	1.20519600	-0.25636200	0.00000100
C	1.17958600	1.14550100	0.00000100
C	2.34668800	1.88158700	0.00000000
C	3.57695100	1.24012800	0.00000000
C	-1.17958200	1.14550200	0.00000000
C	-1.20519400	-0.25636100	0.00000100
C	-2.42949400	-0.89822700	0.00000100
H	-2.49810200	-1.98232800	0.00000100
C	-3.59294300	-0.14002000	0.00000000
C	-3.57694800	1.24013100	-0.00000100
C	-2.34668300	1.88158800	0.00000000
H	2.49810300	-1.98233000	0.00000100
H	2.27111900	2.96517100	0.00000000
H	4.50982600	1.79195600	-0.00000100
H	-4.50982100	1.79196000	-0.00000100
H	-2.27111300	2.96517300	-0.00000100
N	0.00000100	-0.93852000	0.00000200
H	0.00000000	-1.94750400	0.00000100
O	0.00000200	1.83706800	0.00000100
N	-4.88538000	-0.84376100	-0.00000100

N	4.88537800	-0.84376400	0.00000000
O	-4.85978200	-2.05722400	0.00000000
O	4.85977100	-2.05722700	-0.00000100
O	-5.89105800	-0.16700200	-0.00000200
O	5.89105400	-0.16700000	-0.00000200

3,7-dinitro-phenoxazine-cb

E_{gas}	-1000.76458981 hartree
E_{solv}	-1000.83204966 hartree
G_{solv}	-1000.70516566 hartree

	x	y	z
C	-3.57212500	0.15547200	0.00000000
C	-2.40144200	0.90497100	0.00000000
C	-1.14274900	0.28186100	0.00000000
C	-1.17474200	-1.13780600	0.00000000
C	-2.34189300	-1.87021200	0.00000000
C	-3.57720900	-1.23009700	0.00000000
C	1.17474400	-1.13780500	0.00000000
C	1.14275000	0.28186200	0.00000000
C	2.40144300	0.90497300	0.00000000
H	2.43860500	1.98887700	0.00000000
C	3.57212600	0.15547400	0.00000000
C	3.57721200	-1.23009400	0.00000000
C	2.34189700	-1.87021000	0.00000000
H	-2.43860500	1.98887500	0.00000000
H	-2.26658600	-2.95589900	0.00000000
H	-4.51245900	-1.77636500	0.00000000
H	4.51246200	-1.77636100	0.00000000
H	2.26659000	-2.95589700	0.00000000
N	0.00000000	1.00488700	0.00000000
O	0.00000200	-1.84660300	0.00000000
N	4.85862700	0.85238500	0.00000000
N	-4.85862800	0.85238300	0.00000000
O	4.85498700	2.07022900	0.00000000
O	-4.85499200	2.07022700	0.00000000
O	5.88006600	0.18237400	-0.00000100
O	-5.88007100	0.18237700	-0.00000100

Fc

E_{gas}	-510.2934577 hartree
E_{solv}	-510.307375 hartree
G_{solv}	-510.173975 hartree

	x	y	z
C	-0.94034400	1.33374200	-1.20997100
C	-1.88081200	0.67074300	-0.37397900
C	-1.52182800	0.92428000	0.97871500
C	-0.35941400	1.74387000	0.97873300
C	0.00000000	1.99686200	-0.37394100
H	-0.91731100	1.30109100	-2.29277200
H	-2.69976500	0.04510500	-0.70856900
H	-2.01949500	0.52564200	1.85476500
H	0.18321500	2.07870400	1.85479100
H	0.86437800	2.55807600	-0.70850200
Fe	0.00000000	0.00000000	0.00022600
C	1.52182800	-0.92428000	0.97871500
C	0.35941400	-1.74387000	0.97873300
C	1.88081200	-0.67074300	-0.37397900
H	2.01949500	-0.52564200	1.85476500
C	0.00000000	-1.99686200	-0.37394100
H	-0.18321500	-2.07870400	1.85479100
C	0.94034400	-1.33374200	-1.20997100
H	2.69976500	-0.04510500	-0.70856900
H	-0.86437800	-2.55807600	-0.70850200
H	0.91731100	-1.30109100	-2.29277200

Fc^+

E_{gas}	-510.0442268 hartree
E_{solv}	-510.1331379 hartree
G_{solv}	-510.0011829 hartree

	x	y	z
C	1.72108600	-1.14692200	-0.37460300
C	1.76503600	-0.00101500	-1.20317400
C	1.72120000	1.14628000	-0.37653300
C	1.65796300	0.71414500	0.97709700
C	1.65790200	-0.71249600	0.97830800
H	1.70420700	-2.17511700	-0.71542600
H	1.76584800	-0.00192400	-2.28678700
H	1.70436900	2.17390400	-0.71907700
H	1.61505200	1.35496000	1.84914400
H	1.61495600	-1.35183000	1.85144100
Fe	0.00000000	0.00000500	0.00108700
C	-1.65796400	0.71413900	0.97710100
C	-1.72120100	1.14628100	-0.37652700
C	-1.65790100	-0.71250200	0.97830400
H	-1.61505300	1.35495000	1.84915100
C	-1.76503600	-0.00100900	-1.20317400

H	-1.70437100	2.17390700	-0.71906500
C	-1.72108400	-1.14692100	-0.37460900
H	-1.61495500	-1.35184100	1.85143300
H	-1.76584800	-0.00191300	-2.28678700
H	-1.70420500	-2.17511400	-0.71543800

MeCN

E_{gas}	-132.6870402 hartree
E_{solv}	-132.6971846 hartree
G_{solv}	-132.6749336 hartree

	x	y	z
C	0.00000000	0.00000000	0.27830500
C	0.00000000	0.00000000	-1.16917000
H	0.00000000	1.02358200	-1.55328500
H	0.88644800	-0.51179100	-1.55328500
H	-0.88644800	-0.51179100	-1.55328500
N	0.00000000	0.00000000	1.42929100

H_2

E_{gas}	-1.16860258 hartree
E_{solv}	-1.16797387 hartree
G_{solv}	-1.16960587 hartree

	x	y	z
H	0.00000000	0.00000000	0.37308800
H	0.00000000	0.00000000	-0.37308800

10. Sample Input Files

a) Optimization + Frequency Calculation

```
%nproc=8
%chk=ni-II-hydride-opt-freq.chk
%mem=15072MB
#p opt=tight freq=noraman M06/genecp int=ultrafine

Ni-II-hydride

0 1
 C          -1.21530200   3.50358000   0.07129200
 C          -1.18669300   2.11280500   0.07125000
 C           0.00000000   1.38150700  -0.00000400
 C           1.18669400   2.11280500  -0.07125500
 C           1.21530300   3.50358000  -0.07129400
 C           0.00000000   4.18278200   0.00000000
 H          -2.15759600   4.03700500   0.12497600
 H           2.15759700   4.03700500  -0.12497600
 Ni          0.00000000  -0.54162500  -0.00000800
 H           0.00000000  -2.06743200  -0.00001300
 O           2.37554100   1.40625600  -0.14219700
 O          -2.37554100   1.40625600   0.14219100
 P           2.12238700  -0.26074500  -0.01800500
 P          -2.12238700  -0.26074600   0.01800100
 C          -3.01534300  -0.63062100  -1.58407300
 C          -4.51621900  -0.36067100  -1.52562700
 H          -4.73511000   0.64791300  -1.16517800
 H          -4.94292200  -0.45318900  -2.53088600
 H          -5.03808800  -1.07851900  -0.88743700
 C          -2.36513800   0.29616500  -2.62002800
 H          -2.56916300   1.34856400  -2.41154300
 H          -1.27859900   0.16464900  -2.65140400
 H          -2.76414800   0.06182600  -3.61262800
 C          -2.73891900  -2.08187100  -1.98392400
 H          -3.18493900  -2.80194100  -1.29474900
 H          -3.16460900  -2.26976900  -2.97575600
 H          -1.66518600  -2.28417800  -2.02653700
 C          -3.02716300  -0.84129700   1.54639600
 C          -2.01825700  -0.60903700   2.68046500
 H          -2.45973800  -0.91804600   3.63447100
 H          -1.10214600  -1.18730400   2.51947200
 H          -1.74118600   0.44633000   2.76458600
 C          -3.32046900  -2.33624600   1.43043600
 H          -2.42946500  -2.90292500   1.14247300
```

H	-3.65934000	-2.71751600	2.39975500
H	-4.11276800	-2.54375800	0.70594500
C	-4.30403000	-0.05557800	1.84179600
H	-5.08903400	-0.23534500	1.10619900
H	-4.69461300	-0.36490800	2.81777900
H	-4.11411700	1.01837200	1.88110700
C	3.02717600	-0.84129900	-1.54639100
C	3.01532900	-0.63061900	1.58407700
C	3.32048200	-2.33624800	-1.43042600
H	2.42947600	-2.90292700	-1.14247000
H	3.65936100	-2.71751900	-2.39974100
H	4.11277400	-2.54375800	-0.70592800
C	4.30404500	-0.05558000	-1.84178200
H	4.69463600	-0.36491200	-2.81776200
H	4.11413100	1.01837000	-1.88109700
H	5.08904300	-0.23534500	-1.10617900
C	2.01827900	-0.60904200	-2.68046800
H	1.10216700	-1.18731100	-2.51948200
H	1.74120700	0.44632500	-2.76459300
H	2.45976800	-0.91805100	-3.63447000
C	2.73889600	-2.08186600	1.98393100
H	3.16458000	-2.26976300	2.97576600
H	1.66516300	-2.28416800	2.02653700
H	3.18491800	-2.80194100	1.29476000
C	4.51620700	-0.36067500	1.52564200
H	4.94290200	-0.45319300	2.53090400
H	5.03807700	-1.07852600	0.88745600
H	4.73510500	0.64790800	1.16519200
C	2.36512200	0.29617400	2.62002400
H	2.56915200	1.34857100	2.41153800
H	1.27858200	0.16466200	2.65139400
H	2.76412400	0.06183600	3.61262900
H	0.00000000	5.26819400	0.00000100

C H O P 0
6-311G*
****
Ni 0
LANL2DZ
****

Ni 0
LANL2DZ

b) Solvation

```
%nproc=4
%chk=ni-II-hydride-smd-thf-solv.chk
%mem=15072MB
#p scrf=(smd,solvent=Acetonitrile) geom=allcheck guess=read M06/genecp int=ultrafine

C H O P 0
6-311G*
****
Ni 0
LANL2DZ
****

Ni 0
LANL2DZ
```

11. Table S1. Computed Bond Dissociation Free Energy (BDFE) for **3a** and **3a**$^{+}$ in MeCN [^{a}TEMPO = (2,2,6,6-tetramethylpiperidin-1-yl)oxidanyl, ^{b}NHPI = *N*-hydroxyphthalimide (See Computational Details)

Reference Compound	**BDFE of Ni(II)-H in SMD(MeCN)**	**BDFE of Ni(III)-H in SMD(MeCN)**
Aniline	71.4	36.5
TEMPO-H[a]	76.0	41.1
4-OMe-TEMPO-H	73.7	38.8
NHPI[b]	73.4	38.5
3-fluoro-NHPI	73.6	38.7
4-methyl-NHPI	74.7	39.8
4-methoxy-NHPI	73.4	38.5
4-Acetyl-NHPI	74.1	39.2

12. Table S2. Computed pKa for **3a** and **3a**$^{+}$ in SMD (MeCN) (See Computational Details)

Reference Compound	**pKa of Ni(II)-H in SMD(MeCN)**	**pKa of Ni(III)-H in SMD(MeCN)**
4-nitrophenol	45.5	-0.4
3,5-dinitrophenol	45.5	-0.4
3-chloro-4-nitro-phenol	48.4	2.5
4-methylbenzenesulfonicacid	45.1	-0.8
$[NiH(PMe_2CH_2CH_2PMe_2)_2]^+$	47.7	1.8
$[NiH(PMe_2CH_2CH_2CH_2PMe_2)_2]^+$	45.3	-0.6
$[NiH(PEt_2CH_2CH_2PEt_2)_2]^+$	47.1	1.2
Average ± Std. Dev.	**46 ± 1**	**1 ± 1**

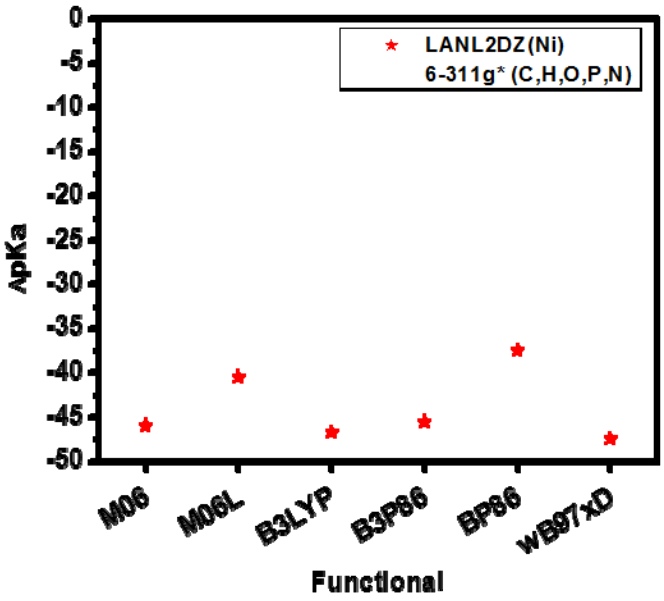

Fig S8. pKa difference between Ni(II)H **(3a)** and Ni(III)H **($3a^+$)** computed using six different functionals. Basis set shown as legend. See Computational Details section for more information.