Supporting Information for:

Monomeric Phosphido and Phosphinidene Complexes of Nickel

Rory Melenkivitz, Daniel J. Mindiola, and Gregory L. Hillhouse*

Searle Chemistry Laboratory, Department of Chemistry, The University of Chicago, Chicago, Illinois 60637

Experimental Section

General Considerations. Unless otherwise stated, all operations were performed in a MBraun Lab Master dry box under an atmosphere of purified nitrogen or using high-vacuum and standard Schlenk techniques under an argon atmosphere. Hexane was dried by passage through activated alumina and Q-5 columns. All other solvents were dried and degassed by using standard high-vacuum and Schlenk techniques.[1] C_6D_6, THF-d_8, and CD_2Cl_2 were purchased form Cambridge Isotope Laboratory (CIL), degassed, and dried over CaH_2 or activated 4Å molecular sieves. Celite, alumina, and 4Å molecular sieves were activated under vacuum overnight at a temperature above 180°C. Infrared data (Nujol mulls, KBr plates) were measured using a Nicolet Nexus 670 FT-IR. Elemental analysis were performed by Desert Analytics (Tucson, Az). ^{1}H, ^{13}C, and ^{31}P NMR spectra were recorded on Bruker 500 and 400 MHz NMR spectrometers. ^{1}H and ^{13}C NMR are reported with reference to solvent resonances (residual C_6D_5H in C_6D_6, 7.16 ppm and 128.0 ppm; CD_2Cl_2, 5.32 ppm and 54 ppm; THF-d_8, 1.73 and 3.58 ppm, and 65.6 and 23.5 ppm). ^{31}P NMR spectra were reported with respect to external 85% H_3PO_4 (0 ppm). Solution magnetic susceptibilities were determined by ^{1}H NMR using the method of Evans.[8,9] ^{31}P NMR simulation of [(dtbpe)Ni(P(H)(dmp))][PF_6] was done with gNMR V3.6.5 software package. Cyclic voltammetry measurements were collected with the assistance of a Eco-Chemie Autolab potentiostat (pgstat20) and the GPES 2.0 software from Bioanalytical Systems (BAS). X-ray diffraction data were collected on a Bruker Platform goniometer with a Charged Coupled Device (CCD) detector (Smart

Apex). Structures were solved by direct methods using the SHELXTL (version 5.1) program library (G. Sheldrick, Bruker Analytical X-ray Systems, Madison, WI).[10]

KC_8,[2] dtbpe ($^tBu_2PCH_2CH_2P^tBu_2$),[3] (dtbpe)$NiCl_2$[4], and $[(dtbpe)NiCl]_2$[5] (**1**) were prepared according to the literature. PH_2(dmp) (dmp = 2,6-dimesetylphenyl) was a generous gift from Professor J. D. Protasiewicz, and LiPH(dmp)•Et_2O was prepared according to the literature procedure.[6] $PCl(^tBu)_2$ was purchased from Strem Chemical Company. $PH(^tBu)_2$ was prepared by the literature method[7] and deprotonated with 1 eq. nBuLi in hexane at –35° C. $Cl_2PCH_2CH_2PCl_2$ was purchased from Strem chemicals and used without further purification. All other chemicals were used as received.

Preparation of (dtbpe)Ni($P(^tBu)_2$) (2)

A cold (-35° C) 5 mL Et_2O solution of $LiP(^tBu)_2$ [249 mg, 1.64 mmol] was added dropwise to a cooled 15 mL Et_2O suspension of (**1**) [527 mg, 1.49 mmol]. The solution immediately became turquoise and nearly homogeneous. After 30 min of stirring, the solution was filtered. Pure crystals of the title compound were obtained by cooling a concentrated Et_2O solution [550 mg, 1.05 mmol, 71%].

For **2**: ^{1}H NMR (22° C, 500.1MHz, C_6D_6): δ 22 (br, $\Delta\nu_{1/2}$ = >3000 Hz, $C(CH_3)_3$, 36 H), 7.5 (br, $\Delta\nu_{1/2}$ = 730 Hz, $C(CH_3)_3$, 18 H), -1 (br, $\Delta\nu_{1/2}$ = 118 Hz, CH_2 CH_2, 4 H). IR (Nujol, KBr): 2170 (m), 1179 (s), 1017 (m), 844 (s) 812 (m), 778 (w), 664 (m) 647 (m), 566 (m) 489 (m) cm^{-1}. μ_{eff}: 2.01 μB (C_6D_6, 298 K, Evans's method). Anal. Calcd. for $C_{26}H_{58}NiP_3$: C, 59.78; H, 11.19; Ni, 11.24; P, 17.79. Found: C, 60.01; H, 11.34.

Preparation of [(dtbpe)Ni($P(^tBu)_2$)][PF_6] (3)

To a cold (-35° C) 6 mL Et_2O solution of (dtbpe)Ni($P(^tBu)_2$) [190 mg, 0.364 mmol] was added a cold 2 mL Et_2O suspension of $FcPF_6$ [128 mg, 0.387 mmol] The solution was stirred for 3 h where a bright green precipitate formed. The solution was cooled after addition of 2 mL hexane. The solid was collected by filtration, washed twice each with

Et_2O and hexane, and dried. Pure compound can be obtained by recrystallization from CH_2Cl_2/Et_2O [220 mg, 0.330 mmol, 91%].

For **3**: ^1H NMR (22° C, 500.1 MHz, CD_2Cl_2): δ 1.48 (d, C(CH_3), 18 H), 1.43 (d, C(CH_3), 36 H, $^3J_{PH}$ = 13.3 Hz), 1.38 (m, CH_2 CH_2, 4 H). $^{13}C\{^1H\}$ NMR (22° C, 125.8MHz, CD_2Cl_2): δ 45.5 (d, CH_2CH_2, J = 16 Hz), 35.9 (m, $C(CH_3)$), 31.3 (s, C(CH_3)), 29.3 (s, C(CH_3)), 22.7 (m, $C(CH_3)$). $^{31}P\{^1H\}$ NMR (22° C, 202.4 MHz, CD_2Cl_2): δ 348 (t, $P(^tBu)_2$, $^3J_{PP}$ = 175 Hz, 1 P), 105 (d, $P(^tBu)_2$, $^3J_{PP}$ = 175 Hz, 2 P), -144 (sept., PF_6, $^1J_{PF}$ = 710 Hz, 1 P). IR (Nujol, KBr): 1178 (m), 1060 (w), 1022 (w), 936 (w), 840 (s), 748 (w), 722(w), 686 (w), 557 (m), 504 (w) cm^{-1}. Anal. Calcd. for $C_{26}H_{58}F_6NiP_4$: C, 46.80; H, 8.76; F, 17.08; Ni, 8.80; P, 18.57. Found: C, 46.60; H, 8.60.

Preparation of (dtbpe)Ni(PH(dmp)) (4).

An 8 mL Et_2O suspension of **1** [155 mg, 0.329 mmol] was cooled to -35° C. To the orange suspension, a cold suspension of LiPH(dmp)•Et_2O [138 mg, 0.329] in 2 mL Et_2O was added dropwise. The addition caused a nearly immediate color change to green, and upon warming to ambient temperature, the solution became almost completely homogeneous. After 45 min of stirring, the solution was filtered and concentrated to about 4 mL. Cooling (-35° C) of the concentrated solution overnight yielded pure green crystals of the title compound [220 mg, 0.304 mmol, 92.5%].

For **4**: ^1H NMR (22° C, 500.1MHz, C_6D_6): δ 9.1 (br, $\Delta\nu_{1/2}$ = 70 Hz, C_6H_3 3 H), 8.05 (br, $\Delta\nu_{1/2}$ = 269 Hz, $C(CH_3)_3$, 36 H), 3.6 (br, $\Delta\nu_{1/2}$ = 28.5 Hz, C_6H_2, 4 H), 3.2 (br, $\Delta\nu_{1/2}$ = 124 Hz, CH_3, 18 H), -2.6 (br, $\Delta\nu_{1/2}$ = 421 Hz, CH_2CH_2, 4 H). IR (Nujol, KBr): 1178 (m), 1043 (w), 1018 (w), 843.6 (m), 812 (w), 779 (w), 740 (w), 655 (w), 647 (w), 565 (w), 489 (m) cm^{-1}. μ_{eff}: 1.92 μB (C_6D_6, 298 K, Evans's method). Anal. Calcd. for $C_{42}H_{66}NiP_3$: C, 69.81; H, 9.21; Ni, 8.12; P, 12.86. Found: C, 69.44; H, 9.28.

Preparation of [(dtbpe)Ni(P(H)(dmp))][PF_6] (5)

A 12 mL THF solution of (dtbpe)Ni(PH(dmp)) [200 mg, 0.277 mmol] was cooled to -35° C. Up on dropwise addition of a cold (-35° C) 3 mL THF suspension of tropylium hexafluorophosphate ([C_7H_7][PF_6]) [66 mg, 0.279 mmol], the green solution changed to a brown-yellow color. After 1 h of stirring, the solution was filtered and concentrated to ca. 4 mL. 8 mL of a 1:1 mixture of Et_2O:hexane was added to induce precipitation. Complete precipitation was insured by cooling the reaction mixture to -35° C for 2 h. The slurry was then filtered, and the olive-green powder washed twice each with Et_2O and hexane [150 mg, 0.173 mmol, 62.5%]. Analytically pure crystals of [(dtbpe)Ni(P(H)(dmp))][PF_6] can be obtained from slow cooling (-35° C) of a dilute THF/Et_2O layered solution.

For **5**: ^{1}H NMR (22° C, 500.1 MHz, CD_2Cl_2): δ 8.055 (dt, P-*H*, 1 H, $^1J_{PH}$ = 280 Hz), 8.05 (m, C_6H_3, 1 H), 7.02 (d, C_6H_3 2 H), 6.97 (s, $C_6(CH_3)_3H_2$, 4 H), 2.36 (s, $C_6(CH_3)_3H_2$, 6 H), 1.95 (s, $C_6(CH_3)_3H_2$, 12 H), 1.67 (m, CH_2CH_2, 4 H), 1.21 (d, $C(CH_3)_3$, 18 H, $^3J_{PH}$ = 13.7), 1.18 (d, $C(CH_3)_3$, 18 H, $^3J_{PH}$ = 13.7). $^{13}C\{^1H\}$ NMR (-20° C, 125.8 MHz, CD_2Cl_2): δ 143 (s, P*Ar*), 138.5 (s, P*Ar*), 138 (s, P*Ar*), 135 (s, P*Ar*), 132.5 (br s, P*Ar*), 131 (d, P*Ar*, J_{PC} = 9.7 Hz), 129 (s, P*Ar*), 128.5 (s, P*Ar*), 35.4 (m, P*C*$(CH_3)_3$), 34.5 (m, P*C*$(CH_3)_3$), 31 (s, PC(CH_3)$_3$), 29.6 (s, PC(CH_3)$_3$), 22.5 (br, PCH_2CH_2P), 21 (s, CH_3), 20.5 (s, CH_3). $^{31}P\{^1H\}$ NMR (22° C, 202.4 MHz, CD_2Cl_2): δ 125 (dd, Ni*P*Ar, 1 P, $^2J_{PP}$ = 183 Hz), 114 (dd, *P*CH_2CH_2*P*, 2 P, $^2J_{PP}$ = 183 Hz), -143.5 (sept., *P*F_6, 1 P, $^1J_{PF}$ = 709 Hz). IR (Nujol, KBr): 1601 (w), 1182 (m), 1067 (w), 1022.8 (w), 844.7 (s), 735 (m), 685.2 (w) 558 (m) 503 (m) cm^{-1}. Anal. Calcd. for $C_{42}H_{66}F_6NiP_4$: C, 58.15; H, 7.67; F, 13.14; Ni, 6.77; P, 14.28. Found: C, 58.17 H; 7.92.

Preparation of (dtbpe)Ni(P(dmp)) (6).

A 6 mL THF solution of [(dtbpe)Ni(PH(dmp))][PF_6] [25 mg, 0.0288 mmol] was cooled to -35° C. A cold solution of NaN($Si(CH_3)_3$)$_2$ [5.5 mg, 0.0299 mmol] in 2 mL THF was

added dropwise to the yellow-brown solution of **5**. The color of the solution changed to a dark green rapidly. After 30 min the THF was removed under reduced pressure. The green residual was redissolved in Et_2O, filtered, and cooled to give dark green blocks of (dtbpe)Ni(P(dmp)) [18 mg, 0.025 mmol, 86%].

For **6**: ^{1}H NMR (22° C, 500.1 MHz, C_6D_6): δ 7.18 (s, $C_6(CH_3)_3\mathit{H}_2$ 4 H), 7.03 (s, $C_6\mathit{H}_3$, 1 H), 6.78 (d, $C_6\mathit{H}_3$ 2 H), 2.47 (s, $C_6(C\mathit{H}_3)_3H_2$, 12 H), 2.23 (s, $C_6(C\mathit{H}_3)_3H_2$, 6 H), 1.04 (d, $C(C\mathit{H}_3)_3$, $^3J_{PH}$ = 12 Hz, 36 H), δ 0.90 (m, $C\mathit{H}_2C\mathit{H}_2$, 4 H). $^{13}C\{^1H\}$ NMR (-20° C, 125.8 MHz, THF-d_8) δ 142 (s, P*Ar*), 138.5 (s, P*Ar*), 136 (s, P*Ar*), 135 (s, P*Ar*), 129.3 (t, P*Ar*, J = 7.3 Hz), 129 (s, P*Ar*), 128 (s, P*Ar*), 123.6 (P*Ar*), 33.8 (t, $C\mathit{H}_2C\mathit{H}_2$, J = 6 Hz), 31 (br. s, $PC(C\mathit{H}_3)_3$,), 22.7 (t, $PC(CH_3)$ $^1J_{PC}$ = 16 Hz), 21.3 (s, $C\mathit{H}_3$), 21.1 (s, $C\mathit{H}_3$). $^{31}P\{^1H\}$ NMR (22° C, 202.4 MHz, C_6D_6): δ 969.6 (t, Ni*P*Ar, $^2J_{PP}$ = 134 Hz, 1 P), 107.5 (d, $P\mathit{CH}_2\mathit{CH}_2P$, $^2J_{PP}$ = 134 Hz, 2 P). IR (Nujol, KBr): 1179 (m), 1040 (m), 1021 (m), 849 (s), 814 (w), 792 (m), 782(w), 735 (m), 671 (m), 650 (w), 601 (w), 563 (w), 494 (m) cm^{-1}. Anal. Calcd. for $C_{42}H_{65}NiP_3$: C, 69.91; H, 9.08; Ni, 8.13; P 12.88. Found: C, 69.75; H, 9.06.

Cyclic Voltammetry Experimentals

Cyclic voltammetry was performed in pre-dried solutions of THF (0.3-0.4 M solution at containing pre-dried and recrystallized tetrabutylammonium hexafluorophosphate, TBAH, Aldrich). A platinum disk (2.0 mm diameter, Bioanalytical Systems), a platinum wire and a silver wire were employed as the working electrode, the auxiliary and the reference electrode, respectively. A one cell compartment was used in the CV measurements. The electrochemical response was collected with the assistance of a Eco-Chemie Autolab potentionstat (pgstat20) and the GPES 4.3 software. The IR correction drop was not employed due to no significant resistance in the solution. All the potentials were reported against ferrocenium/ferrocene couple (0 V) measure as an internal standard. All spectra were recorded at a scan rate ranging from100-300 mV/sec under an N_2 atmosphere. For the experiment 10 mg of crystalline (dtbpe)Ni($P(^tBu)_2$) was dissolved in a solution of TBAH in THF at 28ºC.

References

(1) For a general description of the equipment and techniques used in carrying out this chemistry see: Burger, B. J.; Bercaw, J. E. In *Experimental Organometallic Chemistry*; Wayda, A. L., Darensbourg, M. Y., Eds.; ACS Symposium Series 357; American Chemical Society; Washington, DC, 1987; pp 79-98.

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(10) All software and sources of scattering factors are contained in the SHELXTL (version 5.1) program library; G. Sheldrick, Bruker Analytical Systems, Madison, WI.

Crystallographic Experimental Section and Tables

Data Collection for $Ni(P^tBu_2)(^tBu_2PCH_2CH_2P^tBu_2)$

A green block was selected under a stereo-microscope while immersed in Paratone oil to avoid possible reaction with air. The crystal was removed from the oil using a tapered fiber that also served to hold the crystal for data collection. The crystal was mounted and centered on a Bruker SMART APEX system. Rotation and still images showed diffractions to be sharp while frames separated in reciprocal space were obtained and provided an orientation matrix and initial cell parameters. Final cell parameters were obtained from the full data set.

A "hemisphere" data set was obtained which samples approximately 1.2 hemispheres of reciprocal space to a resolution of 0.84 Å using 0.3 degree steps in ω using 30 sec integration times for each frame. Absorption corrections were applied using SADABS [1] or semi-empirical from psi-scans.

Structure solution and refinement

The space group was determined as $P2_1/n$ based on systematic absences and intensity statistics. Direct Methods were used to refine the nickel and phosphorous atoms. All atoms were converted to and refined anisotropically. Hydrogen atoms were refined isotropically and fixed at calculated positions.

$R_{int} = \Sigma \mid F_o^2 - \langle F_o^2 \rangle \mid / \Sigma \mid F_o^2 \mid$

$wR_2 = [\Sigma [w (F_o^2 - F_c^2)^2] / \Sigma [w (F_o^2)^2]]^{1/2}$

$GoF = S = [\Sigma [w (F_o^2 - F_c^2)^2] / (n-p)^{1/2}$

$R_1 = \Sigma \mid \mid F_o \mid - \mid F_c \mid \mid / \Sigma \mid F_o \mid$

where: $w = q / \sigma^2 (F_o^2) + (aP)^2 + bP$;

n = number of independent reflections;

p = number of parameters refined.

References

[1] All software and sources of scattering factors are contained in the SHELXTL (version 5.1) program library (G. Sheldrick, Bruker Analytical X-ray Systems, Madison, WI).

Table 1. Crystal and structure refinement for $Ni(P^tBu_2)(^tBu_2PCH_2CH_2P^tBu_2)$.

Identification Code	rory07m
Empirical formula	C26 H54 Ni
Formula weight	522.34

Temperature	100 K	
Wavelength	0.71073 Å	
Crystal system	monoclinic	
Space Group	$P2_1/n$	
Unit cell dimensions	a = 8.9029(12) Å	α = 90.00 °
	b = 19.092(3) Å	β = 90.524(3) °
	c = 17.7739(24) Å	γ = 90.00 °
Volume	3020.9(7) $Å^3$	
Z	4	
Density (calculated)	1.148 Mg/m^3	
Absorption coefficient	0.821 mm^{-1}	
F(000)	1148.0	
Crystal size	200 x 140 x 40 µm, yellow plate	
Theta range for data collection	1.57 to 25.00	
Index ranges	$-10 \le h \le 10$, $-22 \le k \le 21$ $-21 \le l \le 16$	
Reflections collected	15154	
Independent reflections	5305 (R_{int} = 0.0625)	
Completeness to theta = 25.00°	99.8%	
Absorption correction	Semi-empirical from psi scans	
Max. and min. transmission	0.9628 and 0.8544	
Refinement method	Full-matrix least squares on F^2	
Data / restraints / parameters	5305 / 0 / 271	
Goodness-of-fit on F^2	1.143	
Final R indices [I > 2 sigma(I)]	R_1 = 0.0639, wR_2 = 0.1096	
R indices (all data)	R_1 = 0.0847, wR_2 = 0.1167	
Largest diff. peak and hole	0.609 and –0.415 $eÅ^{-3}$	

Table 2. Atomic coordinates [x 10^4] and equivalent isotropic displacement parameters [$Å^2$ x 10^3] for Ni(P^tBu_2)($^tBu_2PCH_2CH_2P^tBu_2$). U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Ni	8029(1)	923(1)	7731(1)	9(1)
P(2)	7154(1)	720(1)	6579(1)	10(1)

P(3)	8159(1)	-225(1)	7969(1)	12(1)
P(1)	8371(1)	1937(1)	8304(1)	13(1)
C(21)	5128(5)	968(2)	6416(2)	13(1)
C(1)	7220(5)	-242(2)	6436(2)	12(1)
C(11)	10394(5)	2181(2)	8511(3)	15(1)
C(2)	7130(5)	-648(2)	7178(2)	14(1)
C(22)	8343(5)	1066(2)	5784(2)	14(1)
C(31)	7045(5)	-532(2)	8805(2)	17(1)
C(32)	10054(5)	-677(2)	7982(3)	17(1)
C(223)	9967(5)	912(2)	6006(3)	21(1)
C(12)	7137(5)	2148(2)	9134(2)	16(1)
C(213)	4519(5)	837(2)	5621(2)	22(1)
C(313)	5595(5)	-99(2)	8804(3)	23(1)
C(212)	4882(5)	1737(2)	6623(3)	20(1)
C(123)	5530(5)	2121(2)	8822(3)	28(1)
C(222)	8063(5)	732(2)	5011(2)	23(1)
C(312)	6578(6)	-1305(2)	8806(3)	29(1)
C(311)	7921(5)	-362(2)	9530(2)	25(1)
C(113)	11286(5)	1865(2)	7870(3)	22(1)
C(122)	7256(5)	1622(2)	9783(3)	23(1)
C(323)	10684(5)	-637(2)	7187(3)	23(1)
C(322)	10040(5)	-1446(2)	8216(3)	25(1)
C(211)	4192(5)	528(2)	6954(3)	24(1)
C(221)	8168(5)	1858(2)	5732(3)	24(1)
C(321)	11126(5)	-265(2)	8504(3)	26(1)
C(112)	10989(5)	1872(2)	9250(2)	24(1)
C(121)	7394(5)	2884(2)	9451(3)	27(1)
C(111)	10680(5)	2972(2)	8501(3)	22(1)

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

Table 3. Bond lengths [Å] and angles [°] for $Ni(P^tBu_2)(^tBu_2PCH_2CH_2P^tBu_2)$.

Ni-P(1)	2.2077(12)
Ni-P(2)	2.2169(12)
Ni-P(3)	2.2351(12)
P(2)-C(1)	1.856(4)
P(2)-C(21)	1.885(4)
P(2)-C(22)	1.892(4)
P(3)-C(2)	1.856(4)
P(3)-C(31)	1.886(5)
P(3)-C(32)	1.894(4)
P(1)-C(12)	1.891(5)
P(1)-C(11)	1.894(4)
C(21)-C(211)	1.526(6)
C(21)-C(212)	1.529(5)
C(21)-C(213)	1.529(5)

C(1)-C(2)	1.532(5)
C(11)-C(113)	1.520(6)
C(11)-C(112)	1.529(6)
C(11)-C(111)	1.531(5)
C(22)-C(221)	1.522(5)
C(22)-C(223)	1.525(6)
C(22)-C(222)	1.533(6)
C(31)-C(313)	1.532(6)
C(31)-C(311)	1.536(6)
C(31)-C(312)	1.535(6)
C(32)-C(323)	1.526(6)
C(32)-C(322)	1.526(5)
C(32)-C(321)	1.541(6)
C(12)-C(123)	1.531(6)
C(12)-C(122)	1.532(6)
C(12)-C(121)	1.531(5)

P(1)-Ni-P(2)	128.68(5)
P(1)-Ni-P(3)	139.95(5)
P(2)-Ni-P(3)	91.22(4)
C(1)-P(2)-C(21)	105.00(19)
C(1)-P(2)-C(22)	102.99(19)
C(21)-P(2)-C(22)	109.76(19)
C(1)-P(2)-Ni	106.73(13)
C(21)-P(2)-Ni	115.23(14)
C(22)-P(2)-Ni	115.74(14)
C(2)-P(3)-C(31)	101.67(19)
C(2)-P(3)-C(32)	104.16(19)
C(31)-P(3)-C(32)	108.9(2)
C(2)-P(3)-Ni	104.97(13)
C(31)-P(3)-Ni	115.27(14)
C(32)-P(3)-Ni	119.53(14)
C(12)-P(1)-C(11)	110.7(2)
C(12)-P(1)-Ni	117.94(14)
C(11)-P(1)-Ni	115.65(14)
C(211)-C(21)-C(212)	107.3(4)
C(211)-C(21)-C(213)	107.3(3)
C(212)-C(21)-C(213)	109.2(3)
C(211)-C(21)-P(2)	107.0(3)
C(212)-C(21)-P(2)	110.0(3)
C(213)-C(21)-P(2)	115.6(3)
C(2)-C(1)-P(2)	112.4(3)
C(113)-C(11)-C(112)	108.1(4)
C(113)-C(11)-C(111)	107.1(4)
C(112)-C(11)-C(111)	109.6(4)
C(113)-C(11)-P(1)	105.0(3)
C(112)-C(11)-P(1)	113.1(3)
C(111)-C(11)-P(1)	113.5(3)
C(1)-C(2)-P(3)	113.8(3)
C(221)-C(22)-C(223)	107.7(4)
C(221)-C(22)-C(222)	110.1(4)
C(223)-C(22)-C(222)	107.3(4)
C(221)-C(22)-P(2)	109.6(3)

C(223)-C(22)-P(2)	106.0(3)
C(222)-C(22)-P(2)	115.9(3)
C(313)-C(31)-C(311)	107.9(4)
C(313)-C(31)-C(312)	106.9(4)
C(311)-C(31)-C(312)	109.8(4)
C(313)-C(31)-P(3)	106.4(3)
C(311)-C(31)-P(3)	109.2(3)
C(312)-C(31)-P(3)	116.3(3)
C(323)-C(32)-C(322)	107.7(4)
C(323)-C(32)-C(321)	107.5(4)
C(322)-C(32)-C(321)	109.5(3)
C(323)-C(32)-P(3)	107.5(3)
C(322)-C(32)-P(3)	115.6(3)
C(321)-C(32)-P(3)	108.8(3)
C(123)-C(12)-C(122)	108.0(4)
C(123)-C(12)-C(121)	107.5(4)
C(122)-C(12)-C(121)	108.4(4)
C(123)-C(12)-P(1)	104.9(3)
C(122)-C(12)-P(1)	114.3(3)
C(121)-C(12)-P(1)	113.4(3)

Table 4. Anisotropic displacement parameters [$Å^2$ x 10^3] for $Ni(P^tBu_2)(^tBu_2PCH_2CH_2P^tBu_2)$. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U^{11}+ ... + 2hka^*b^*U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Ni	11(1)	8(1)	8(1)	-1(1)	-1(1)	-1(1)
P(2)	13(1)	7(1)	9(1)	-1(1)	-1(1)	1(1)
P(3)	16(1)	11(1)	10(1)	0(1)	-3(1)	-2(1)
P(1)	11(1)	15(1)	12(1)	-4(1)	1(1)	-2(1)
C(21)	12(2)	15(2)	12(2)	-2(2)	-1(2)	4(2)
C(1)	15(2)	9(2)	12(2)	-4(2)	-2(2)	3(2)
C(11)	8(2)	16(2)	22(3)	-3(2)	1(2)	-3(2)
C(2)	15(2)	11(2)	15(2)	-1(2)	-1(2)	0(2)
C(22)	18(3)	16(2)	9(2)	6(2)	4(2)	6(2)
C(31)	21(3)	17(2)	13(2)	0(2)	2(2)	-2(2)
C(32)	15(3)	13(2)	23(3)	3(2)	-6(2)	3(2)
C(223)	18(3)	24(3)	21(3)	5(2)	6(2)	3(2)
C(12)	14(2)	14(2)	20(3)	-7(2)	4(2)	-2(2)
C(213)	18(3)	24(3)	24(3)	-5(2)	-7(2)	3(2)
C(313)	22(3)	32(3)	16(3)	4(2)	3(2)	-3(2)
C(212)	23(3)	15(2)	21(3)	-3(2)	-3(2)	6(2)
C(123)	20(3)	32(3)	32(3)	-10(2)	8(2)	1(2)
C(222)	25(3)	30(3)	13(2)	3(2)	6(2)	6(2)
C(312)	33(3)	27(3)	27(3)	11(2)	5(2)	-9(2)
C(311)	31(3)	33(3)	10(2)	4(2)	-2(2)	3(2)

C(113)	15(3)	25(3)	26(3)	-5(2)	1(2)	-1(2)
C(122)	20(3)	29(3)	21(3)	-4(2)	7(2)	-9(2)
C(323)	17(3)	25(3)	27(3)	7(2)	0(2)	6(2)
C(322)	27(3)	20(3)	27(3)	8(2)	-1(2)	3(2)
C(211)	11(3)	28(3)	35(3)	7(2)	2(2)	3(2)
C(221)	24(3)	15(2)	33(3)	9(2)	12(2)	3(2)
C(321)	20(3)	21(3)	37(3)	5(2)	-6(2)	-3(2)
C(112)	20(3)	32(3)	19(3)	0(2)	-5(2)	-1(2)
C(121)	23(3)	29(3)	30(3)	-11(2)	8(2)	-3(2)
C(111)	18(3)	21(3)	27(3)	-1(2)	3(2)	-6(2)

Table 5. Hydrogen coordinates [x 10^4] and isotropic displacement parameters [$Å^2$ x 10^3] for Ni(P^tBu_2)($^tBu_2PCH_2CH_2P^tBu_2$).

	x	y	z	U(eq)
H(1A)	6390	-381	6111	14
H(1B)	8147	-364	6184	14
H(2A)	7534	-1114	7102	17
H(2B)	6083	-698	7314	17
H(22A)	10624	1079	5620	32
H(22B)	10098	416	6065	32
H(22C)	10203	1143	6472	32
H(21A)	3487	982	5592	33
H(21B)	4588	347	5506	33
H(21C)	5098	1100	5266	33
H(31A)	4987	-237	9223	35
H(31B)	5050	-180	8344	35
H(31C)	5838	389	8847	35
H(21D)	3850	1859	6534	30
H(21E)	5515	2027	6319	30
H(21F)	5127	1806	7144	30
H(12A)	4836	2224	9217	42
H(12B)	5328	1662	8625	42
H(12C)	5418	2461	8427	42
H(22D)	8711	945	4648	34
H(22E)	7034	803	4863	34
H(22F)	8268	239	5039	34
H(31D)	6020	-1405	9253	44
H(31E)	7459	-1595	8794	44
H(31F)	5964	-1400	8371	44
H(31G)	7356	-516	9957	37
H(31H)	8080	135	9563	37
H(31I)	8873	-598	9525	37
H(11A)	12333	1971	7939	33
H(11B)	10941	2059	7401	33
H(11C)	11148	1366	7864	33

H(12D)	6604	1764	10182	35
H(12E)	8274	1608	9966	35
H(12F)	6967	1166	9608	35
H(32A)	11648	-861	7176	34
H(32B)	10787	-155	7042	34
H(32C)	10011	-870	6844	34
H(32D)	11045	-1627	8207	37
H(32E)	9418	-1707	7873	37
H(32F)	9648	-1486	8715	37
H(21G)	3148	640	6887	36
H(21H)	4492	628	7463	36
H(21I)	4348	40	6851	36
H(22G)	8768	2033	5327	35
H(22H)	8493	2069	6196	35
H(22I)	7132	1972	5640	35
H(32G)	12091	-489	8515	39
H(32H)	10724	-253	9003	39
H(32I)	11233	205	8319	39
H(11D)	12014	2012	9325	36
H(11E)	10932	1370	9228	36
H(11F)	10392	2039	9659	36
H(12G)	6741	2960	9870	41
H(12H)	7183	3225	9067	41
H(12I)	8420	2930	9614	41
H(11G)	11721	3061	8609	33
H(11H)	10072	3194	8875	33
H(11I)	10426	3155	8013	33

Table 6. Torsion angles [°] for Ni(P^tBu_2)($^tBu_2PCH_2CH_2P^tBu_2$).

P(1)-Ni-P(2)-C(1)	177.89(15)
P(3)-Ni-P(2)-C(1)	-5.81(15)
P(1)-Ni-P(2)-C(21)	-65.97(16)
P(3)-Ni-P(2)-C(21)	110.33(15)
P(1)-Ni-P(2)-C(22)	63.97(16)
P(3)-Ni-P(2)-C(22)	-119.73(15)
P(1)-Ni-P(3)-C(2)	164.34(16)
P(2)-Ni-P(3)-C(2)	-11.17(15)
P(1)-Ni-P(3)-C(31)	53.38(18)
P(2)-Ni-P(3)-C(31)	-122.13(16)
P(1)-Ni-P(3)-C(32)	-79.40(18)
P(2)-Ni-P(3)-C(32)	105.10(17)
P(2)-Ni-P(1)-C(12)	112.40(16)
P(3)-Ni-P(1)-C(12)	-61.85(18)
P(2)-Ni-P(1)-C(11)	-113.23(16)
P(3)-Ni-P(1)-C(11)	72.52(18)
C(1)-P(2)-C(21)-C(211)	56.0(3)
C(22)-P(2)-C(21)-C(211)	166.1(3)
Ni-P(2)-C(21)-C(211)	-61.1(3)

C(1)-P(2)-C(21)-C(212)	172.3(3)
C(22)-P(2)-C(21)-C(212)	-77.6(3)
Ni-P(2)-C(21)-C(212)	55.1(3)
C(1)-P(2)-C(21)-C(213)	-63.4(3)
C(22)-P(2)-C(21)-C(213)	46.6(4)
Ni-P(2)-C(21)-C(213)	179.4(3)
C(21)-P(2)-C(1)-C(2)	-96.7(3)
C(22)-P(2)-C(1)-C(2)	148.4(3)
Ni-P(2)-C(1)-C(2)	26.1(3)
C(12)-P(1)-C(11)-C(113)	169.9(3)
Ni-P(1)-C(11)-C(113)	32.4(3)
C(12)-P(1)-C(11)-C(112)	52.2(4)
Ni-P(1)-C(11)-C(112)	-85.3(3)
C(12)-P(1)-C(11)-C(111)	-73.5(4)
Ni-P(1)-C(11)-C(111)	149.0(3)
P(2)-C(1)-C(2)-P(3)	-37.5(4)
C(31)-P(3)-C(2)-C(1)	151.1(3)
C(32)-P(3)-C(2)-C(1)	-95.7(3)
Ni-P(3)-C(2)-C(1)	30.7(3)
C(1)-P(2)-C(22)-C(221)	169.5(3)
C(21)-P(2)-C(22)-C(221)	58.1(3)
Ni-P(2)-C(22)-C(221)	-74.5(3)
C(1)-P(2)-C(22)-C(223)	-74.6(3)
C(21)-P(2)-C(22)-C(223)	174.0(3)
Ni-P(2)-C(22)-C(223)	41.5(3)
C(1)-P(2)-C(22)-C(222)	44.3(3)
C(21)-P(2)-C(22)-C(222)	-67.2(3)
Ni-P(2)-C(22)-C(222)	160.3(3)
C(2)-P(3)-C(31)-C(313)	-75.6(3)
C(32)-P(3)-C(31)-C(313)	174.8(3)
Ni-P(3)-C(31)-C(313)	37.3(3)
C(2)-P(3)-C(31)-C(311)	168.1(3)
C(32)-P(3)-C(31)-C(311)	58.6(3)
Ni-P(3)-C(31)-C(311)	-79.0(3)
C(2)-P(3)-C(31)-C(312)	43.2(4)
C(32)-P(3)-C(31)-C(312)	-66.3(4)
Ni-P(3)-C(31)-C(312)	156.1(3)
C(2)-P(3)-C(32)-C(323)	51.9(3)
C(31)-P(3)-C(32)-C(323)	159.8(3)
Ni-P(3)-C(32)-C(323)	-64.8(3)
C(2)-P(3)-C(32)-C(322)	-68.4(4)
C(31)-P(3)-C(32)-C(322)	39.5(4)
Ni-P(3)-C(32)-C(322)	175.0(3)
C(2)-P(3)-C(32)-C(321)	168.1(3)
C(31)-P(3)-C(32)-C(321)	-84.1(3)
Ni-P(3)-C(32)-C(321)	51.4(4)
C(11)-P(1)-C(12)-C(123)	164.7(3)
Ni-P(1)-C(12)-C(123)	-58.8(3)
C(11)-P(1)-C(12)-C(122)	-77.1(3)
Ni-P(1)-C(12)-C(122)	59.3(3)
C(11)-P(1)-C(12)-C(121)	47.7(4)
Ni-P(1)-C(12)-C(121)	-175.8(3)

Data Collection for $[Ni(P^tBu_2)(^tBu_2PCH_2CH_2P^tBu_2)][PF_6]\cdot CH_2Cl_2$

A green block was selected under a stereo-microscope while immersed in mineral oil to avoid possible reaction with air. The crystal was removed from the oil using a tapered fiber that also served to hold the crystal for data collection. The crystal was mounted and centered on a Bruker SMART APEX system. Rotation and still images showed diffractions to be sharp while frames separated in reciprocal space were obtained and provided an orientation matrix and initial cell parameters. Final cell parameters were obtained from the full data set.

A "hemisphere" data set was obtained which samples approximately 1.2 hemispheres of reciprocal space to a resolution of 0.84 Å using 0.3 degree steps in ω using 30 sec integration times for each frame. Absorption corrections were applied using SADABS [1] or semi-empirical from psi-scans.

Structure solution and refinement

The space group was determined as P*bca* based on systematic absences and intensity statistics. Direct Methods were used to refine the nickel and phosphorous atoms. All atoms were converted to and refined anisotropically. Hydrogen atoms were refined isotropically and fixed at calculated positions. One methylene chloride molecule was confined in the asymmetric unit cell containing configurational disorder at the chlorine atoms. The large residual electron density and peak hole was confined proximal to the methylene chloride chlorine atoms.

$R_{int} = \Sigma \mid F_o^2 - \langle F_o^2 \rangle \mid / \Sigma \mid F_o^2 \mid$

$wR_2 = [\Sigma [w (F_o^2 - F_c^2)^2] / \Sigma [w (F_o^2)^2]]^{1/2}$

$GoF = S = [\Sigma [w (F_o^2 - F_c^2)^2] / (n-p)^{1/2}$

$R_1 = \Sigma \mid \mid F_o \mid - \mid F_c \mid \mid / \Sigma \mid F_o \mid$

where: $w = q / \sigma^2 (F_o^2) + (aP)^2 + bP$;

n = number of independent reflections;

p = number of parameters refined.

References

[1] All software and sources of scattering factors are contained in the SHELXTL (version 5.1) program library (G. Sheldrick, Bruker Analytical X-ray Systems, Madison, WI).

Table 1. Crystal and structure refinement for $[Ni(P^tBu_2)(^tBu_2PCH_2CH_2P^tBu_2)][PF_6]\cdot CH_2Cl_2$

Identification Code	rory06m
Empirical formula	C27 H60 Cl2 F6 Ni P4
Formula weight	752.24

Temperature	100 K
Wavelength	0.71073 Å
Crystal system	orthorhombic
Space Group	P*bca*
Unit cell dimensions	a = 16.8718(15) Å α = 90.00 °
	b = 19.8422(18) Å β = 90.00 °
	c = 22.241(2) Å γ = 90.00 °
Volume	7445.6(12) Å ^{3}
Z	8
Density (calculated)	1.342 Mg/mm^3
Absorption coefficient	0.8840 mm^{-1}
F(000)	3184.0
Crystal size	320 x 200 x 120 μm, green block
Theta range for data collection	1.83 to 22.00
Index ranges	$-17 \le h \le 17$, $-19 \le k \le 20$ $-23 \le l \le 22$
Reflections collected	27437
Independent reflections	4569 (R_{int} = 0.0345)
Completeness to theta = 22.00°	100%
Absorption correction	Semi-empirical from psi scans
Max. and min. transmission	0.9404 and 0.3693
Refinement method	Full-matrix least squares on F^2
Data / restraints / parameters	4569 / 0 / 361
Goodness-of-fit on F^2	1.068
Final R indices [I > 2 sigma(I)]	R_1 = 0.0791, wR_2 = 0.2157
R indices (all data)	R_1 = 0.0842, wR_2 = 0.2212
Largest diff. peak and hole	2.953 and –1.379 eÅ $^{-3}$

Table 2. Atomic coordinates [x 10^4] and equivalent isotropic displacement parameters [$Å^2$ x 10^3] for [Ni(P^tBu_2)($^tBu_2PCH_2CH_2P^tBu_2$)] [PF_6]·CH_2Cl_2. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Ni	3899(1)	2356(1)	3641(1)	24(1)
P(1)	4575(1)	2977(1)	3075(1)	25(1)
P(3)	3647(1)	1259(1)	3648(1)	28(1)
P(4)	2396(1)	5089(1)	5922(1)	31(1)
P(2)	3167(1)	2514(1)	4459(1)	27(1)
F(1)	2353(3)	5233(2)	6621(2)	58(1)
F(4)	3294(3)	4854(3)	5996(2)	53(1)
F(3)	2446(3)	4942(3)	5217(2)	70(2)
C(11)	5669(4)	3116(4)	3175(3)	31(2)
F(5)	2672(3)	5840(2)	5810(2)	63(2)
C(12)	4181(4)	3434(4)	2400(3)	31(2)
F(6)	2127(3)	4333(2)	6031(3)	59(1)
F(2)	1500(3)	5320(3)	5836(3)	64(2)
C(22)	2228(4)	2999(4)	4310(3)	39(2)
C(32)	4531(4)	721(4)	3863(4)	37(2)
C(111)	5951(5)	3817(4)	3033(4)	53(2)
C(112)	6101(4)	2596(4)	2770(4)	45(2)
C(121)	4668(4)	3319(4)	1832(3)	40(2)
C(113)	5852(5)	2948(5)	3829(4)	50(2)
C(122)	3342(4)	3157(4)	2285(3)	40(2)
C(21)	3689(5)	2874(4)	5125(3)	41(2)
C(31)	3140(4)	910(4)	2957(3)	33(2)
C(2)	2872(5)	1665(4)	4720(4)	45(2)
C(321)	5215(5)	888(5)	3468(5)	61(3)
C(123)	4118(5)	4191(4)	2548(4)	48(2)
C(1)	2893(5)	1126(4)	4245(3)	41(2)
C(211)	4501(5)	2526(5)	5185(4)	58(3)
C(221)	2397(6)	3635(5)	3978(5)	70(3)
C(212)	3865(6)	3635(5)	4999(5)	68(3)
C(311)	3756(5)	840(6)	2444(4)	63(3)
C(222)	1750(6)	2539(6)	3890(5)	82(4)
C(322)	4731(5)	915(4)	4521(4)	55(2)
C(223)	1714(4)	3125(4)	4856(4)	45(2)
C(312)	2761(5)	213(4)	3054(4)	52(2)
C(323)	4389(5)	-20(4)	3873(4)	50(2)
C(213)	3241(5)	2805(5)	5723(4)	52(2)
C(313)	2501(5)	1392(4)	2768(4)	50(2)
Cl(1)	409(2)	951(4)	3517(2)	173(3)
Cl(2)	877(2)	443(3)	4624(3)	164(2)
C(1S)	809(11)	1207(12)	4171(11)	190(10)

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

Table 3. Bond lengths [Å] and angles [°] for $[Ni(P^tBu_2)(^tBu_2PCH_2CH_2P^tBu_2)][PF_6]\cdot CH_2Cl_2$.

Ni-P(1)	2.0980(19)
Ni-P(3)	2.218(2)
Ni-P(2)	2.222(2)
P(1)-C(12)	1.876(7)
P(1)-C(11)	1.879(7)
P(3)-C(1)	1.857(8)
P(3)-C(31)	1.891(7)
P(3)-C(32)	1.895(7)
P(4)-F(5)	1.583(5)
P(4)-F(1)	1.583(5)
P(4)-F(6)	1.584(5)
P(4)-F(2)	1.591(5)
P(4)-F(4)	1.595(5)
P(4)-F(3)	1.596(5)
P(2)-C(2)	1.850(8)
P(2)-C(21)	1.866(8)
P(2)-C(22)	1.882(7)
C(11)-C(111)	1.503(11)
C(11)-C(113)	1.523(11)
C(11)-C(112)	1.553(11)
C(12)-C(121)	1.523(10)
C(12)-C(122)	1.539(10)
C(12)-C(123)	1.541(11)
C(22)-C(221)	1.490(12)
C(22)-C(223)	1.512(11)
C(22)-C(222)	1.536(13)
C(32)-C(321)	1.488(12)
C(32)-C(323)	1.491(11)
C(32)-C(322)	1.551(12)
C(21)-C(213)	1.535(11)
C(21)-C(211)	1.540(11)
C(21)-C(212)	1.564(13)
C(31)-C(313)	1.501(11)
C(31)-C(312)	1.539(11)
C(31)-C(311)	1.550(11)
C(2)-C(1)	1.504(11)
Cl(1)-C(1S)	1.68(2)
Cl(2)-C(1S)	1.82(2)

P(1)-Ni-P(3)	133.16(8)
P(1)-Ni-P(2)	135.34(8)
P(3)-Ni-P(2)	91.49(7)

C(12)-P(1)-C(11)	111.8(3)
C(12)-P(1)-Ni	124.8(2)
C(11)-P(1)-Ni	123.3(2)
C(1)-P(3)-C(31)	102.7(3)
C(1)-P(3)-C(32)	106.2(4)
C(31)-P(3)-C(32)	110.8(3)
C(1)-P(3)-Ni	106.0(2)
C(31)-P(3)-Ni	116.1(2)
C(32)-P(3)-Ni	113.8(2)
F(5)-P(4)-F(1)	89.8(3)
F(5)-P(4)-F(6)	179.4(3)
F(1)-P(4)-F(6)	90.5(3)
F(5)-P(4)-F(2)	89.4(3)
F(1)-P(4)-F(2)	91.3(3)
F(6)-P(4)-F(2)	91.1(3)
F(5)-P(4)-F(4)	90.6(3)
F(1)-P(4)-F(4)	89.6(3)
F(6)-P(4)-F(4)	88.9(3)
F(2)-P(4)-F(4)	179.1(3)
F(5)-P(4)-F(3)	90.0(3)
F(1)-P(4)-F(3)	179.6(3)
F(6)-P(4)-F(3)	89.6(3)
F(2)-P(4)-F(3)	89.1(3)
F(4)-P(4)-F(3)	89.9(3)
C(2)-P(2)-C(21)	103.1(4)
C(2)-P(2)-C(22)	107.1(4)
C(21)-P(2)-C(22)	110.0(3)
C(2)-P(2)-Ni	106.1(3)
C(21)-P(2)-Ni	116.2(3)
C(22)-P(2)-Ni	113.3(2)
C(111)-C(11)-C(113)	109.9(7)
C(111)-C(11)-C(112)	110.1(7)
C(113)-C(11)-C(112)	108.2(7)
C(111)-C(11)-P(1)	115.0(5)
C(113)-C(11)-P(1)	106.3(5)
C(112)-C(11)-P(1)	107.1(5)
C(121)-C(12)-C(122)	107.8(6)
C(121)-C(12)-C(123)	111.1(6)
C(122)-C(12)-C(123)	108.7(6)
C(121)-C(12)-P(1)	113.5(5)
C(122)-C(12)-P(1)	106.6(5)
C(123)-C(12)-P(1)	108.9(5)
C(221)-C(22)-C(223)	111.5(7)
C(221)-C(22)-C(222)	107.6(8)
C(223)-C(22)-C(222)	106.6(7)
C(221)-C(22)-P(2)	111.1(6)
C(223)-C(22)-P(2)	115.2(5)
C(222)-C(22)-P(2)	104.2(6)
C(321)-C(32)-C(323)	110.6(7)
C(321)-C(32)-C(322)	109.5(7)
C(323)-C(32)-C(322)	105.4(7)
C(321)-C(32)-P(3)	109.7(6)
C(323)-C(32)-P(3)	115.7(5)
C(322)-C(32)-P(3)	105.6(5)

C(213)-C(21)-C(211)	108.9(7)
C(213)-C(21)-C(212)	109.5(7)
C(211)-C(21)-C(212)	106.3(8)
C(213)-C(21)-P(2)	114.8(6)
C(211)-C(21)-P(2)	108.5(5)
C(212)-C(21)-P(2)	108.4(6)
C(313)-C(31)-C(312)	108.2(6)
C(313)-C(31)-C(311)	109.5(7)
C(312)-C(31)-C(311)	107.5(7)
C(313)-C(31)-P(3)	108.6(5)
C(312)-C(31)-P(3)	113.8(5)
C(311)-C(31)-P(3)	109.1(5)
C(1)-C(2)-P(2)	114.9(5)
C(2)-C(1)-P(3)	114.7(5)
Cl(1)-C(1S)-Cl(2)	104.6(13)

Table 4. Anisotropic displacement parameters [$Å^2$ x 10^3] for [Ni(P^tBu_2)($^tBu_2PCH_2CH_2P^tBu_2$)][PF_6]·CH_2Cl_2. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U^{11}+ ... + 2hka^*b^*U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Ni	17(1)	30(1)	25(1)	4(1)	0(1)	0(1)
P(1)	17(1)	34(1)	24(1)	-2(1)	0(1)	1(1)
P(3)	25(1)	28(1)	30(1)	-1(1)	-2(1)	3(1)
P(4)	27(1)	34(1)	33(1)	0(1)	-2(1)	3(1)
P(2)	27(1)	30(1)	24(1)	0(1)	1(1)	5(1)
F(1)	79(4)	57(3)	39(3)	-1(2)	8(3)	24(3)
F(4)	33(3)	79(4)	48(3)	8(3)	0(2)	12(2)
F(3)	82(4)	89(4)	37(3)	-2(3)	-11(3)	30(3)
C(11)	15(3)	42(4)	37(4)	7(3)	-1(3)	-3(3)
F(5)	66(3)	50(3)	74(4)	25(3)	-21(3)	-17(3)
C(12)	21(4)	42(4)	30(4)	7(3)	-3(3)	-4(3)
F(6)	56(3)	37(3)	84(4)	4(3)	-11(3)	-6(2)
F(2)	33(3)	58(3)	102(4)	7(3)	-10(3)	9(2)
C(22)	30(4)	57(5)	30(4)	1(4)	-2(3)	16(4)
C(32)	27(4)	26(4)	60(5)	-6(4)	-12(4)	5(3)
C(111)	33(4)	48(5)	77(7)	6(5)	-16(4)	-11(4)
C(112)	27(4)	53(5)	56(6)	0(4)	2(4)	3(4)
C(121)	29(4)	59(5)	31(4)	5(4)	2(3)	-6(4)
C(113)	30(4)	73(6)	46(5)	12(5)	-11(4)	-7(4)
C(122)	26(4)	65(6)	29(4)	13(4)	-2(3)	-5(4)
C(21)	39(4)	51(5)	32(4)	-8(4)	-6(4)	15(4)
C(31)	24(4)	43(5)	32(4)	-6(3)	-2(3)	-6(3)
C(2)	44(5)	44(5)	48(5)	5(4)	19(4)	-4(4)
C(321)	35(5)	49(6)	98(8)	17(5)	4(5)	8(4)

C(123)	39(5)	43(5)	61(6)	8(4)	-14(4)	11(4)
C(1)	51(5)	32(4)	38(5)	9(4)	8(4)	2(4)
C(211)	44(5)	84(7)	45(5)	-15(5)	-11(4)	21(5)
C(221)	60(6)	86(8)	65(6)	34(6)	21(5)	41(6)
C(212)	74(7)	61(6)	69(7)	-21(5)	-9(5)	-11(5)
C(311)	47(5)	90(7)	53(6)	-25(5)	0(4)	-13(5)
C(222)	53(6)	126(10)	66(7)	-37(7)	-22(5)	46(6)
C(322)	56(6)	45(5)	63(6)	-3(4)	-18(5)	17(4)
C(223)	31(4)	59(5)	43(5)	-1(4)	5(4)	13(4)
C(312)	50(5)	41(5)	65(6)	-10(4)	-15(5)	-1(4)
C(323)	42(5)	40(5)	67(6)	12(4)	-6(4)	11(4)
C(213)	58(6)	70(6)	27(4)	-7(4)	-4(4)	21(5)
C(313)	45(5)	42(5)	64(6)	0(4)	-20(5)	-6(4)
Cl(1)	70(2)	352(9)	97(3)	-15(4)	-20(2)	-24(4)
Cl(2)	66(2)	170(4)	256(6)	84(4)	7(3)	-5(3)
C(1S)	96(13)	230(30)	240(30)	10(20)	-39(15)	8(15)

Table 5. Hydrogen coordinates [x 10^4] and isotropic displacement parameters [Å^2 x 10^3] for [Ni(P^tBu_2)($^tBu_2PCH_2CH_2P^tBu_2$)][PF_6]·CH_2Cl_2.

	x	y	z	U(eq)
H(11A)	6513	3844	3096	79
H(11B)	5688	4134	3290	79
H(11C)	5833	3921	2621	79
H(11D)	6663	2652	2810	68
H(11E)	5950	2666	2358	68
H(11F)	5956	2148	2890	68
H(12A)	4436	3564	1504	60
H(12B)	4674	2847	1738	60
H(12C)	5200	3473	1897	60
H(11G)	6408	3011	3903	75
H(11H)	5712	2487	3908	75
H(11I)	5552	3239	4088	75
H(12D)	3115	3379	1942	60
H(12E)	3017	3238	2632	60
H(12F)	3371	2681	2211	60
H(2A)	3221	1532	5046	54
H(2B)	2339	1690	4881	54
H(32A)	5662	616	3579	91
H(32B)	5348	1355	3514	91
H(32C)	5077	800	3057	91
H(12G)	3915	4429	2206	72
H(12H)	4634	4362	2648	72
H(12I)	3768	4253	2884	72
H(1A)	2374	1100	4058	49

H(1B)	2995	696	4437	49
H(21A)	4776	2704	5528	87
H(21B)	4808	2606	4829	87
H(21C)	4425	2050	5236	87
H(22A)	1909	3872	3907	106
H(22B)	2644	3531	3601	106
H(22C)	2745	3913	4213	106
H(21D)	4130	3829	5340	102
H(21E)	3376	3869	4930	102
H(21F)	4197	3674	4650	102
H(31A)	3501	664	2091	95
H(31B)	4170	538	2568	95
H(31C)	3979	1274	2355	95
H(22D)	1256	2752	3791	122
H(22E)	1649	2117	4087	122
H(22F)	2046	2460	3528	122
H(32D)	5179	658	4655	82
H(32E)	4283	820	4774	82
H(32F)	4854	1387	4541	82
H(22G)	1250	3373	4738	67
H(22H)	2007	3381	5147	67
H(22I)	1558	2702	5029	67
H(31D)	2514	67	2687	78
H(31E)	2370	243	3366	78
H(31F)	3163	-104	3168	78
H(32G)	4868	-248	3986	74
H(32H)	4229	-168	3480	74
H(32I)	3979	-122	4158	74
H(21G)	3548	3004	6040	78
H(21H)	3153	2337	5808	78
H(21I)	2740	3033	5693	78
H(31G)	2238	1221	2417	75
H(31H)	2732	1823	2678	75
H(31I)	2124	1441	3088	75
H(1SA)	474	1540	4364	229
H(1SB)	1330	1402	4106	229

Table 6. Torsion angles [°] for $[Ni(P^tBu_2)(^tBu_2PCH_2CH_2P^tBu_2)][PF_6]\cdot CH_2Cl_2$.

P(3)-Ni-P(1)-C(12)	92.1(3)
P(2)-Ni-P(1)-C(12)	-88.4(3)
P(3)-Ni-P(1)-C(11)	-85.9(3)
P(2)-Ni-P(1)-C(11)	93.6(3)
P(1)-Ni-P(3)-C(1)	-173.1(3)
P(2)-Ni-P(3)-C(1)	7.2(3)
P(1)-Ni-P(3)-C(31)	-59.9(3)
P(2)-Ni-P(3)-C(31)	120.5(3)
P(1)-Ni-P(3)-C(32)	70.5(3)
P(2)-Ni-P(3)-C(32)	-109.1(3)

P(1)-Ni-P(2)-C(2)	-173.5(3)
P(3)-Ni-P(2)-C(2)	6.1(3)
P(1)-Ni-P(2)-C(21)	-59.6(3)
P(3)-Ni-P(2)-C(21)	120.0(3)
P(1)-Ni-P(2)-C(22)	69.2(3)
P(3)-Ni-P(2)-C(22)	-111.2(3)
C(12)-P(1)-C(11)-C(111)	38.2(7)
Ni-P(1)-C(11)-C(111)	-143.5(5)
C(12)-P(1)-C(11)-C(113)	160.0(5)
Ni-P(1)-C(11)-C(113)	-21.7(7)
C(12)-P(1)-C(11)-C(112)	-84.5(6)
Ni-P(1)-C(11)-C(112)	93.8(5)
C(11)-P(1)-C(12)-C(121)	49.1(6)
Ni-P(1)-C(12)-C(121)	-129.1(5)
C(11)-P(1)-C(12)-C(122)	167.6(5)
Ni-P(1)-C(12)-C(122)	-10.6(6)
C(11)-P(1)-C(12)-C(123)	-75.3(6)
Ni-P(1)-C(12)-C(123)	106.5(5)
C(2)-P(2)-C(22)-C(221)	-168.1(7)
C(21)-P(2)-C(22)-C(221)	80.6(7)
Ni-P(2)-C(22)-C(221)	-51.4(7)
C(2)-P(2)-C(22)-C(223)	63.9(7)
C(21)-P(2)-C(22)-C(223)	-47.5(7)
Ni-P(2)-C(22)-C(223)	-179.4(5)
C(2)-P(2)-C(22)-C(222)	-52.5(7)
C(21)-P(2)-C(22)-C(222)	-163.9(7)
Ni-P(2)-C(22)-C(222)	64.2(7)
C(1)-P(3)-C(32)-C(321)	-170.2(6)
C(31)-P(3)-C(32)-C(321)	79.1(7)
Ni-P(3)-C(32)-C(321)	-53.9(6)
C(1)-P(3)-C(32)-C(323)	63.8(7)
C(31)-P(3)-C(32)-C(323)	-46.9(7)
Ni-P(3)-C(32)-C(323)	-179.9(6)
C(1)-P(3)-C(32)-C(322)	-52.3(6)
C(31)-P(3)-C(32)-C(322)	-163.0(5)
Ni-P(3)-C(32)-C(322)	64.0(6)
C(2)-P(2)-C(21)-C(213)	-49.0(7)
C(22)-P(2)-C(21)-C(213)	65.0(7)
Ni-P(2)-C(21)-C(213)	-164.6(5)
C(2)-P(2)-C(21)-C(211)	73.1(7)
C(22)-P(2)-C(21)-C(211)	-172.9(6)
Ni-P(2)-C(21)-C(211)	-42.5(7)
C(2)-P(2)-C(21)-C(212)	-171.9(6)
C(22)-P(2)-C(21)-C(212)	-57.9(7)
Ni-P(2)-C(21)-C(212)	72.5(6)
C(1)-P(3)-C(31)-C(313)	72.9(6)
C(32)-P(3)-C(31)-C(313)	-174.1(5)
Ni-P(3)-C(31)-C(313)	-42.2(6)
C(1)-P(3)-C(31)-C(312)	-47.7(6)
C(32)-P(3)-C(31)-C(312)	65.3(6)
Ni-P(3)-C(31)-C(312)	-162.9(5)
C(1)-P(3)-C(31)-C(311)	-167.8(6)
C(32)-P(3)-C(31)-C(311)	-54.8(7)
Ni-P(3)-C(31)-C(311)	77.1(6)

C(21)-P(2)-C(2)-C(1)	-145.0(6)
C(22)-P(2)-C(2)-C(1)	99.0(7)
Ni-P(2)-C(2)-C(1)	-22.3(7)
P(2)-C(2)-C(1)-P(3)	30.1(8)
C(31)-P(3)-C(1)-C(2)	-145.4(6)
C(32)-P(3)-C(1)-C(2)	98.3(6)
Ni-P(3)-C(1)-C(2)	-23.1(7)

Data Collection for [Ni(P(H)(dmp))(^{t}Bu$_2$PCH$_2$CH$_2$P^{t}Bu$_2$)][PF$_6$]·THF

A green block was selected under a stereo-microscope while immersed in mineral oil to avoid possible reaction with air. The crystal was removed from the oil using a tapered fiber that also served to hold the crystal for data collection. The crystal was mounted and centered on a Bruker SMART APEX system. Rotation and still images showed diffractions to be sharp while frames separated in reciprocal space were obtained and provided an orientation matrix and initial cell parameters. Final cell parameters were obtained from the full data set.

A "hemisphere" data set was obtained which samples approximately 1.2 hemispheres of reciprocal space to a resolution of 0.84 Å using 0.3 degree steps in ω using 10 sec integration times for each frame. Absorption corrections were applied using SADABS [1] or semi-empirical from psi-scans.

Structure solution and refinement

The space group was determined as $P2_1/c$ based on systematic absences and intensity statistics. Direct Methods were used to refine the nickel and phosphorous atoms. All atoms were converted to and refined anisotropically. The phosphido hydrogen (H1) was located from the Fourier Difference Map and refined isotropically. All other hydrogen atoms were refined isotropically and fixed at calculated positions.

$R_{int} = \Sigma \mid F_o^2 - \langle F_o^2 \rangle \mid / \Sigma \mid F_o^2 \mid$

$wR_2 = [\Sigma [w (F_o^2 - F_c^2)^2] / \Sigma [w (F_o^2)^2]]^{1/2}$

$GoF = S = [\Sigma [w (F_o^2 - F_c^2)^2] / (n-p)^{1/2}$

$R_1 = \Sigma \mid \mid F_o \mid - \mid F_c \mid \mid / \Sigma \mid F_o \mid$

where: $w = q / \sigma^2 (F_o^2) + (aP)^2 + bP$;

n = number of independent reflections;

p = number of parameters refined.

References

[1] All software and sources of scattering factors are contained in the SHELXTL (version 5.1) program library (G. Sheldrick, Bruker Analytical X-ray Systems, Madison, WI).

Table 1. Crystal and structure refinement for [Ni(P(H)(dmp))(^{t}Bu$_2$PCH$_2$CH$_2$P^{t}Bu$_2$)][PF$_6$]·THF.

Identification Code	rory09t
Empirical formula	C46 H74 F6 Ni O P4

Formula weight	939.64	
Temperature	100 K	
Wavelength	0.71073 Å	
Crystal system	monoclinic	
Space Group	$P2_1/c$	
Unit cell dimensions	a = 21.3459(16) Å	α = 90.00 °
	b = 10.9757(8) Å	β = 112.812(2) °
	c = 22.2867(18) Å	γ = 90.00 °
Volume	4813.1(6) $Å^3$	
Z	4	
Density (calculated)	1.297 Mg/m^3	
Absorption coefficient	0.592 mm^{-1}	
F(000)	2000.0	
Crystal size	70 x 100 x 120 μm, dark green block	
Theta range for data collection	1.85 to 28.29	
Index ranges	$-27 \le h \le 25$, $-14 \le k \le 14$, $-29 \le l \le 28$	
Reflections collected	28830	
Independent reflections	11322 (R_{int} = 0.0601)	
Completeness to theta = 28.29°	94.7%	
Absorption correction	Semi-empirical from psi scans	
Max. and min. transmission	0.4231 and 0.3843	
Refinement method	Full-matrix least squares on F^2	
Data / restraints / parameters	11322 / 0 / 527	
Goodness-of-fit on F^2	1.136	
Final R indices [I > 2 sigma(I)]	R_1 = 0.0751, wR_2 = 0.1532	
R indices (all data)	R_1 = 0.1021, wR_2 = 0.1654	
Largest diff. peak and hole	0.634 and –0.578 e$Å^{-3}$	

Table 2. Atomic coordinates [x 10^4] and equivalent isotropic displacement parameters [$Å^2$ x 10^3] for [Ni(P(H)(dmp))(^{t}Bu$_2$PCH$_2$CH$_2$P^{t}Bu$_2$)][PF_6]·THF. . U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

x	y	z	U(eq)

Ni	2102(1)	6985(1)	4677(1)	12(1)
P(2)	1291(1)	5618(1)	4270(1)	13(1)
P(1)	2761(1)	7787(1)	4327(1)	14(1)
P(4)	1917(1)	2994(1)	1755(1)	19(1)
P(3)	2339(1)	6379(1)	5676(1)	13(1)
F(6)	2420(1)	3460(2)	2453(1)	34(1)
F(5)	1413(1)	2519(2)	1053(1)	41(1)
C(14)	3270(2)	11024(4)	3318(2)	20(1)
C(12)	3641(2)	9190(3)	3952(2)	15(1)
F(4)	2341(2)	3687(3)	1417(1)	56(1)
C(122)	4539(2)	8437(4)	4987(2)	17(1)
C(22)	1444(2)	4660(3)	3632(2)	17(1)
C(21)	378(2)	6156(4)	3968(2)	17(1)
F(3)	2378(2)	1826(3)	1842(2)	57(1)
C(166)	1909(2)	10688(3)	4472(2)	17(1)
C(123)	5084(2)	7669(4)	5314(2)	20(1)
C(2)	1406(2)	4587(4)	4963(2)	18(1)
C(16)	2534(2)	10090(3)	3770(2)	13(1)
C(164)	679(2)	10549(4)	4081(2)	22(1)
C(32)	3172(2)	5553(4)	6075(2)	17(1)
C(163)	670(2)	10022(3)	3515(2)	18(1)
F(2)	1494(2)	2316(4)	2100(2)	71(1)
C(11)	3007(2)	9130(3)	4015(2)	13(1)
C(15)	2670(2)	11013(3)	3414(2)	17(1)
C(126)	4421(2)	7443(4)	3977(2)	19(1)
C(162)	1266(2)	9819(3)	3406(2)	16(1)
C(213)	153(2)	6703(4)	3281(2)	25(1)
C(31)	2259(2)	7642(4)	6214(2)	20(1)
C(222)	1435(2)	5452(4)	3065(2)	23(1)
C(161)	1890(2)	10164(3)	3894(2)	13(1)
C(3)	1666(2)	5274(3)	5616(2)	15(1)
C(121)	4204(2)	8324(4)	4310(2)	16(1)
C(323)	3742(2)	6245(4)	5961(2)	23(1)
F(1)	1447(2)	4158(3)	1662(1)	65(1)
C(212)	351(2)	7174(4)	4429(2)	26(1)
C(127)	4327(2)	9382(4)	5366(2)	21(1)
C(13)	3755(2)	10135(4)	3594(2)	19(1)
C(312)	2897(2)	8441(4)	6443(2)	25(1)
C(128)	5910(2)	6005(4)	5372(2)	32(1)
C(221)	2168(2)	4144(4)	3955(2)	24(1)
C(322)	3371(2)	5375(4)	6807(2)	25(1)
C(167)	1245(2)	9265(4)	2784(2)	23(1)
C(165)	1303(2)	10865(4)	4556(2)	20(1)
C(211)	-125(2)	5156(4)	3967(2)	24(1)
C(125)	4972(2)	6695(4)	4326(2)	23(1)
C(223)	942(2)	3603(4)	3380(2)	25(1)
C(124)	5313(2)	6793(4)	4994(2)	24(1)
C(321)	3102(2)	4290(4)	5761(2)	23(1)
C(311)	1660(2)	8422(4)	5778(2)	27(1)
C(168)	27(2)	10808(4)	4175(2)	33(1)
C(169)	2576(2)	11043(4)	5005(2)	23(1)
C(313)	2110(2)	7205(4)	6798(2)	32(1)
C(129)	4062(2)	7269(4)	3254(2)	29(1)

O(1S)	4714(2)	4750(3)	2743(2)	35(1)
C(14S)	4623(2)	4840(4)	2078(2)	31(1)
C(11S)	4287(3)	3803(5)	2800(3)	42(1)
C(13S)	4169(2)	3802(4)	1714(2)	36(1)
C(12S)	4191(3)	2928(5)	2248(3)	42(1)

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

Table 3. Bond lengths [Å] and angles [°] for [Ni(P(H)(dmp))(^{t}Bu$_2$PCH$_2$CH$_2$P^{t}Bu$_2$)][PF$_6$]·THF.

Ni-P(1)	2.0520(11)
Ni-P(3)	2.1881(11)
Ni-P(2)	2.2034(11)
P(2)-C(2)	1.853(4)
P(2)-C(21)	1.894(4)
P(2)-C(22)	1.894(4)
P(1)-C(11)	1.793(4)
P(4)-F(2)	1.580(3)
P(4)-F(4)	1.580(3)
P(4)-F(3)	1.582(3)
P(4)-F(1)	1.587(3)
P(4)-F(6)	1.595(3)
P(4)-F(5)	1.603(3)
P(3)-C(3)	1.845(4)
P(3)-C(31)	1.882(4)
P(3)-C(32)	1.884(4)
C(14)-C(15)	1.380(5)
C(14)-C(13)	1.382(5)
C(12)-C(13)	1.385(5)
C(12)-C(11)	1.414(5)
C(12)-C(121)	1.498(5)
C(122)-C(123)	1.392(5)
C(122)-C(121)	1.402(5)
C(122)-C(127)	1.514(5)
C(22)-C(222)	1.527(5)
C(22)-C(223)	1.531(5)
C(22)-C(221)	1.537(5)
C(21)-C(211)	1.534(5)
C(21)-C(212)	1.533(5)
C(21)-C(213)	1.538(5)
C(166)-C(165)	1.390(5)
C(166)-C(161)	1.399(5)
C(166)-C(169)	1.510(5)
C(123)-C(124)	1.394(6)
C(2)-C(3)	1.539(5)
C(16)-C(15)	1.385(5)
C(16)-C(11)	1.415(5)

C(16)-C(161)	1.506(5)
C(164)-C(163)	1.381(5)
C(164)-C(165)	1.387(6)
C(164)-C(168)	1.513(5)
C(32)-C(322)	1.530(5)
C(32)-C(321)	1.534(5)
C(32)-C(323)	1.537(5)
C(163)-C(162)	1.402(5)
C(126)-C(125)	1.397(5)
C(126)-C(121)	1.402(5)
C(126)-C(129)	1.505(5)
C(162)-C(161)	1.405(5)
C(162)-C(167)	1.498(5)
C(31)-C(312)	1.532(5)
C(31)-C(311)	1.532(6)
C(31)-C(313)	1.532(6)
C(128)-C(124)	1.500(5)
C(125)-C(124)	1.386(6)
O(1S)-C(14S)	1.420(5)
O(1S)-C(11S)	1.421(5)
C(14S)-C(13S)	1.512(6)
C(11S)-C(12S)	1.511(7)
C(13S)-C(12S)	1.515(7)

P(1)-Ni-P(3)	126.52(4)
P(1)-Ni-P(2)	132.88(4)
P(3)-Ni-P(2)	92.70(4)
C(2)-P(2)-C(21)	105.79(17)
C(2)-P(2)-C(22)	106.32(18)
C(21)-P(2)-C(22)	111.12(17)
C(2)-P(2)-Ni	104.09(13)
C(21)-P(2)-Ni	118.02(13)
C(22)-P(2)-Ni	110.50(13)
C(11)-P(1)-Ni	147.90(13)
F(2)-P(4)-F(4)	179.2(2)
F(2)-P(4)-F(3)	90.3(2)
F(4)-P(4)-F(3)	90.2(2)
F(2)-P(4)-F(1)	89.5(2)
F(4)-P(4)-F(1)	90.0(2)
F(3)-P(4)-F(1)	179.4(2)
F(2)-P(4)-F(6)	88.40(17)
F(4)-P(4)-F(6)	90.97(15)
F(3)-P(4)-F(6)	89.34(15)
F(1)-P(4)-F(6)	91.21(16)
F(2)-P(4)-F(5)	91.59(17)
F(4)-P(4)-F(5)	89.06(16)
F(3)-P(4)-F(5)	90.38(15)
F(1)-P(4)-F(5)	89.06(16)
F(6)-P(4)-F(5)	179.72(17)
C(3)-P(3)-C(31)	105.69(17)
C(3)-P(3)-C(32)	106.44(17)
C(31)-P(3)-C(32)	110.88(18)
C(3)-P(3)-Ni	105.02(12)
C(31)-P(3)-Ni	112.45(14)

C(32)-P(3)-Ni	115.49(12)
C(15)-C(14)-C(13)	120.3(4)
C(13)-C(12)-C(11)	118.7(3)
C(13)-C(12)-C(121)	119.8(3)
C(11)-C(12)-C(121)	121.2(3)
C(123)-C(122)-C(121)	119.0(4)
C(123)-C(122)-C(127)	119.5(4)
C(121)-C(122)-C(127)	121.6(3)
C(222)-C(22)-C(223)	110.2(3)
C(222)-C(22)-C(221)	106.5(3)
C(223)-C(22)-C(221)	109.0(3)
C(222)-C(22)-P(2)	110.8(3)
C(223)-C(22)-P(2)	113.3(3)
C(221)-C(22)-P(2)	106.7(3)
C(211)-C(21)-C(212)	108.3(3)
C(211)-C(21)-C(213)	109.2(3)
C(212)-C(21)-C(213)	107.8(3)
C(211)-C(21)-P(2)	113.6(3)
C(212)-C(21)-P(2)	106.8(3)
C(213)-C(21)-P(2)	110.9(3)
C(165)-C(166)-C(161)	119.0(4)
C(165)-C(166)-C(169)	120.0(4)
C(161)-C(166)-C(169)	120.9(3)
C(122)-C(123)-C(124)	122.5(4)
C(3)-C(2)-P(2)	111.8(3)
C(15)-C(16)-C(11)	119.3(3)
C(15)-C(16)-C(161)	118.3(3)
C(11)-C(16)-C(161)	122.4(3)
C(163)-C(164)-C(165)	118.2(4)
C(163)-C(164)-C(168)	121.2(4)
C(165)-C(164)-C(168)	120.6(4)
C(322)-C(32)-C(321)	108.0(3)
C(322)-C(32)-C(323)	109.2(3)
C(321)-C(32)-C(323)	108.3(3)
C(322)-C(32)-P(3)	112.7(3)
C(321)-C(32)-P(3)	108.3(3)
C(323)-C(32)-P(3)	110.2(3)
C(164)-C(163)-C(162)	122.1(4)
C(16)-C(11)-C(12)	119.6(3)
C(16)-C(11)-P(1)	119.2(3)
C(12)-C(11)-P(1)	120.8(3)
C(14)-C(15)-C(16)	120.6(4)
C(125)-C(126)-C(121)	119.4(4)
C(125)-C(126)-C(129)	119.4(4)
C(121)-C(126)-C(129)	121.2(4)
C(163)-C(162)-C(161)	118.3(3)
C(163)-C(162)-C(167)	121.4(3)
C(161)-C(162)-C(167)	120.3(3)
C(312)-C(31)-C(311)	107.8(3)
C(312)-C(31)-C(313)	110.3(3)
C(311)-C(31)-C(313)	108.5(4)
C(312)-C(31)-P(3)	110.2(3)
C(311)-C(31)-P(3)	105.5(3)
C(313)-C(31)-P(3)	114.2(3)

C(166)-C(161)-C(162)	120.3(3)
C(166)-C(161)-C(16)	119.3(3)
C(162)-C(161)-C(16)	120.1(3)
C(2)-C(3)-P(3)	112.1(2)
C(126)-C(121)-C(122)	119.7(4)
C(126)-C(121)-C(12)	121.2(3)
C(122)-C(121)-C(12)	119.1(3)
C(14)-C(13)-C(12)	121.1(3)
C(164)-C(165)-C(166)	122.0(4)
C(124)-C(125)-C(126)	122.0(4)
C(125)-C(124)-C(123)	117.5(4)
C(125)-C(124)-C(128)	122.5(4)
C(123)-C(124)-C(128)	120.1(4)
C(14S)-O(1S)-C(11S)	108.1(3)
O(1S)-C(14S)-C(13S)	108.0(4)
O(1S)-C(11S)-C(12S)	106.3(4)
C(14S)-C(13S)-C(12S)	103.7(4)
C(11S)-C(12S)-C(13S)	101.0(4)

Table 4. Anisotropic displacement parameters [$Å^2$ x 10^3] for [Ni(P(H)(dmp))(^{t}Bu$_2$PCH$_2$CH$_2$P^{t}Bu$_2$)][PF_6]·THF. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U^{11}+ ... + 2hka^*b^*U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Ni	11(1)	13(1)	12(1)	1(1)	5(1)	-1(1)
P(2)	12(1)	14(1)	11(1)	1(1)	4(1)	0(1)
P(1)	12(1)	15(1)	15(1)	3(1)	6(1)	1(1)
P(4)	20(1)	18(1)	20(1)	3(1)	9(1)	2(1)
P(3)	12(1)	16(1)	10(1)	0(1)	4(1)	-1(1)
F(6)	45(2)	31(1)	21(1)	-9(1)	9(1)	-1(1)
F(5)	36(2)	39(2)	31(2)	1(1)	-6(1)	-2(1)
C(14)	26(2)	19(2)	17(2)	4(2)	12(2)	-4(2)
C(12)	12(2)	16(2)	16(2)	-1(2)	4(2)	2(2)
F(4)	55(2)	85(2)	35(2)	0(2)	24(2)	-33(2)
C(122)	10(2)	24(2)	16(2)	0(2)	5(2)	-3(2)
C(22)	21(2)	15(2)	14(2)	-5(2)	4(2)	2(2)
C(21)	14(2)	20(2)	16(2)	1(2)	5(2)	-1(2)
F(3)	66(2)	41(2)	41(2)	-14(1)	-6(2)	38(2)
C(166)	18(2)	17(2)	16(2)	4(2)	5(2)	6(2)
C(123)	15(2)	32(2)	14(2)	2(2)	5(2)	-3(2)
C(2)	16(2)	20(2)	13(2)	2(2)	-1(2)	-3(2)
C(16)	11(2)	18(2)	9(2)	-3(1)	1(1)	1(2)
C(164)	21(2)	22(2)	26(2)	7(2)	14(2)	5(2)
C(32)	16(2)	19(2)	14(2)	3(2)	3(2)	0(2)
C(163)	15(2)	19(2)	20(2)	2(2)	6(2)	2(2)

F(2)	30(2)	118(3)	56(2)	50(2)	7(2)	-18(2)
C(11)	13(2)	17(2)	8(2)	0(1)	3(1)	1(2)
C(15)	21(2)	18(2)	12(2)	2(2)	6(2)	4(2)
C(126)	17(2)	25(2)	18(2)	2(2)	9(2)	1(2)
C(162)	17(2)	18(2)	15(2)	3(2)	7(2)	3(2)
C(213)	20(2)	33(2)	21(2)	8(2)	7(2)	6(2)
C(31)	20(2)	19(2)	22(2)	-9(2)	8(2)	-3(2)
C(222)	27(2)	27(2)	13(2)	-3(2)	8(2)	4(2)
C(161)	17(2)	11(2)	13(2)	3(1)	6(2)	3(2)
C(3)	16(2)	19(2)	12(2)	2(2)	8(2)	-2(2)
C(121)	9(2)	24(2)	15(2)	2(2)	6(2)	1(2)
C(323)	12(2)	29(2)	28(2)	8(2)	7(2)	3(2)
F(1)	99(3)	66(2)	28(2)	11(2)	21(2)	57(2)
C(212)	22(2)	29(2)	27(2)	0(2)	11(2)	2(2)
C(127)	17(2)	32(2)	10(2)	1(2)	3(2)	1(2)
C(13)	14(2)	27(2)	18(2)	2(2)	8(2)	-2(2)
C(312)	28(2)	21(2)	23(2)	-8(2)	7(2)	-1(2)
C(128)	22(2)	41(3)	31(3)	7(2)	9(2)	14(2)
C(221)	25(2)	22(2)	23(2)	-2(2)	7(2)	6(2)
C(322)	25(2)	28(2)	17(2)	4(2)	4(2)	1(2)
C(167)	18(2)	32(2)	15(2)	0(2)	5(2)	0(2)
C(165)	28(2)	20(2)	17(2)	1(2)	14(2)	7(2)
C(211)	17(2)	31(2)	21(2)	1(2)	6(2)	-3(2)
C(125)	21(2)	27(2)	22(2)	2(2)	11(2)	8(2)
C(223)	27(2)	21(2)	21(2)	-10(2)	2(2)	-6(2)
C(124)	17(2)	30(2)	24(2)	7(2)	8(2)	4(2)
C(321)	19(2)	27(2)	18(2)	1(2)	2(2)	4(2)
C(311)	27(2)	23(2)	30(3)	-11(2)	10(2)	0(2)
C(168)	24(2)	37(3)	48(3)	4(2)	24(2)	9(2)
C(169)	27(2)	27(2)	13(2)	-4(2)	6(2)	0(2)
C(313)	37(3)	40(3)	24(2)	-11(2)	17(2)	-7(2)
C(129)	35(3)	34(3)	16(2)	-3(2)	9(2)	12(2)
O(1S)	39(2)	40(2)	27(2)	-4(2)	14(2)	-9(2)
C(14S)	27(2)	37(3)	29(3)	3(2)	11(2)	1(2)
C(11S)	42(3)	53(3)	35(3)	1(3)	19(2)	-12(3)
C(13S)	38(3)	44(3)	27(3)	-6(2)	12(2)	-8(2)
C(12S)	41(3)	33(3)	51(3)	1(3)	16(3)	-4(2)

Table 5. Hydrogen coordinates [x 10^4] and isotropic displacement parameters [$Å^2$ x 10^3] for [Ni(P(H)(dmp))(^{t}Bu$_2$PCH$_2$CH$_2$P^{t}Bu$_2$)][PF_6]·THF.

	x	y	z	U(eq)
H(14A)	3349	11632	3065	23
H(12A)	5304	7743	5763	24
H(2A)	976	4200	4897	22

H(2B)	1728	3952	4977	22
H(16A)	255	9793	3195	22
H(15A)	2353	11632	3239	20
H(21A)	-311	6968	3137	37
H(21B)	191	6096	2987	37
H(21C)	438	7385	3289	37
H(22A)	1514	4951	2748	34
H(22B)	1785	6058	3223	34
H(22C)	1001	5843	2868	34
H(3A)	1842	4691	5969	18
H(3B)	1290	5700	5663	18
H(32A)	4163	5815	6166	35
H(32B)	3783	7048	6144	35
H(32C)	3634	6305	5502	35
H(21D)	-105	7473	4293	38
H(21E)	647	7826	4420	38
H(21F)	497	6859	4864	38
H(12B)	4614	9320	5820	31
H(12C)	3863	9244	5309	31
H(12D)	4370	10181	5211	31
H(13A)	4165	10171	3540	23
H(31A)	2850	9081	6717	37
H(31B)	2953	8790	6072	37
H(31C)	3287	7953	6684	37
H(12E)	5999	5449	5081	47
H(12F)	5812	5553	5694	47
H(12G)	6303	6507	5583	47
H(22D)	2267	3651	3647	36
H(22E)	2201	3655	4323	36
H(22F)	2487	4803	4096	36
H(32D)	3796	4950	6987	37
H(32E)	3026	4909	6880	37
H(32F)	3415	6156	7014	37
H(16B)	1699	9198	2798	34
H(16C)	980	9772	2424	34
H(16D)	1044	8470	2731	34
H(16E)	1317	11206	4943	24
H(21G)	-576	5486	3810	35
H(21H)	-6	4856	4402	35
H(21I)	-106	4501	3689	35
H(12H)	5115	6114	4102	27
H(22G)	1045	3153	3060	38
H(22H)	488	3918	3187	38
H(22I)	979	3075	3736	38
H(32G)	3522	3853	5960	34
H(32H)	2996	4379	5304	34
H(32I)	2745	3846	5824	34
H(31D)	1594	9088	6027	41
H(31E)	1256	7933	5614	41
H(31F)	1756	8736	5420	41
H(16F)	-353	10536	3800	50
H(16G)	-11	11668	4231	50
H(16H)	33	10385	4554	50
H(16I)	2492	11390	5363	34

H(16J)	2802	11632	4841	34
H(16K)	2858	10334	5152	34
H(31G)	2077	7896	7049	48
H(31H)	2471	6682	7065	48
H(31I)	1689	6764	6647	48
H(12I)	3700	7851	3085	43
H(12J)	3878	6460	3166	43
H(12K)	4378	7386	3048	43
H(14B)	5058	4789	2038	37
H(14C)	4415	5615	1900	37
H(11A)	3852	4130	2766	51
H(11B)	4497	3393	3216	51
H(13B)	4342	3421	1416	43
H(13C)	3709	4085	1470	43
H(12N)	3771	2473	2129	51
H(12O)	4569	2364	2355	51
H(1)	3222(19)	7130(30)	4287(19)	18(11)

Table 6. Torsion angles [°] for [Ni(P(H)(dmp))(^{t}Bu$_2$PCH$_2$CH$_2$P^{t}Bu$_2$)][PF$_6$]·THF.

P(1)-Ni-P(2)-C(2)	137.52(14)
P(3)-Ni-P(2)-C(2)	-11.45(14)
P(1)-Ni-P(2)-C(21)	-105.66(14)
P(3)-Ni-P(2)-C(21)	105.38(14)
P(1)-Ni-P(2)-C(22)	23.74(15)
P(3)-Ni-P(2)-C(22)	-125.22(13)
P(3)-Ni-P(1)-C(11)	-111.0(2)
P(2)-Ni-P(1)-C(11)	108.8(2)
P(1)-Ni-P(3)-C(3)	-159.91(13)
P(2)-Ni-P(3)-C(3)	-7.95(13)
P(1)-Ni-P(3)-C(31)	85.63(14)
P(2)-Ni-P(3)-C(31)	-122.41(14)
P(1)-Ni-P(3)-C(32)	-43.00(15)
P(2)-Ni-P(3)-C(32)	108.96(14)
C(2)-P(2)-C(22)-C(222)	-174.1(3)
C(21)-P(2)-C(22)-C(222)	71.3(3)
Ni-P(2)-C(22)-C(222)	-61.7(3)
C(2)-P(2)-C(22)-C(223)	61.5(3)
C(21)-P(2)-C(22)-C(223)	-53.2(3)
Ni-P(2)-C(22)-C(223)	173.8(2)
C(2)-P(2)-C(22)-C(221)	-58.5(3)
C(21)-P(2)-C(22)-C(221)	-173.1(3)
Ni-P(2)-C(22)-C(221)	53.9(3)
C(2)-P(2)-C(21)-C(211)	-39.8(3)
C(22)-P(2)-C(21)-C(211)	75.2(3)
Ni-P(2)-C(21)-C(211)	-155.7(2)
C(2)-P(2)-C(21)-C(212)	79.6(3)
C(22)-P(2)-C(21)-C(212)	-165.4(3)

Ni-P(2)-C(21)-C(212)	-36.3(3)
C(2)-P(2)-C(21)-C(213)	-163.2(3)
C(22)-P(2)-C(21)-C(213)	-48.2(3)
Ni-P(2)-C(21)-C(213)	80.9(3)
C(121)-C(122)-C(123)-C(124)	0.3(6)
C(127)-C(122)-C(123)-C(124)	-178.9(4)
C(21)-P(2)-C(2)-C(3)	-91.6(3)
C(22)-P(2)-C(2)-C(3)	150.1(3)
Ni-P(2)-C(2)-C(3)	33.4(3)
C(3)-P(3)-C(32)-C(322)	-75.1(3)
C(31)-P(3)-C(32)-C(322)	39.4(3)
Ni-P(3)-C(32)-C(322)	168.8(2)
C(3)-P(3)-C(32)-C(321)	44.3(3)
C(31)-P(3)-C(32)-C(321)	158.8(3)
Ni-P(3)-C(32)-C(321)	-71.8(3)
C(3)-P(3)-C(32)-C(323)	162.7(3)
C(31)-P(3)-C(32)-C(323)	-82.8(3)
Ni-P(3)-C(32)-C(323)	46.6(3)
C(165)-C(164)-C(163)-C(162)	-1.0(6)
C(168)-C(164)-C(163)-C(162)	177.3(4)
C(15)-C(16)-C(11)-C(12)	6.4(5)
C(161)-C(16)-C(11)-C(12)	-172.1(3)
C(15)-C(16)-C(11)-P(1)	-167.3(3)
C(161)-C(16)-C(11)-P(1)	14.2(5)
C(13)-C(12)-C(11)-C(16)	-6.1(5)
C(121)-C(12)-C(11)-C(16)	168.4(3)
C(13)-C(12)-C(11)-P(1)	167.4(3)
C(121)-C(12)-C(11)-P(1)	-18.0(5)
Ni-P(1)-C(11)-C(16)	-18.4(4)
Ni-P(1)-C(11)-C(12)	168.0(2)
C(13)-C(14)-C(15)-C(16)	-2.3(6)
C(11)-C(16)-C(15)-C(14)	-2.2(6)
C(161)-C(16)-C(15)-C(14)	176.4(3)
C(164)-C(163)-C(162)-C(161)	0.3(6)
C(164)-C(163)-C(162)-C(167)	-179.0(4)
C(3)-P(3)-C(31)-C(312)	165.3(3)
C(32)-P(3)-C(31)-C(312)	50.4(3)
Ni-P(3)-C(31)-C(312)	-80.6(3)
C(3)-P(3)-C(31)-C(311)	-78.5(3)
C(32)-P(3)-C(31)-C(311)	166.6(3)
Ni-P(3)-C(31)-C(311)	35.6(3)
C(3)-P(3)-C(31)-C(313)	40.6(4)
C(32)-P(3)-C(31)-C(313)	-74.4(3)
Ni-P(3)-C(31)-C(313)	154.6(3)
C(165)-C(166)-C(161)-C(162)	-0.2(6)
C(169)-C(166)-C(161)-C(162)	179.0(4)
C(165)-C(166)-C(161)-C(16)	173.7(3)
C(169)-C(166)-C(161)-C(16)	-7.2(5)
C(163)-C(162)-C(161)-C(166)	0.3(5)
C(167)-C(162)-C(161)-C(166)	179.6(4)
C(163)-C(162)-C(161)-C(16)	-173.4(3)
C(167)-C(162)-C(161)-C(16)	5.8(5)
C(15)-C(16)-C(161)-C(166)	-94.3(4)
C(11)-C(16)-C(161)-C(166)	84.3(5)

C(15)-C(16)-C(161)-C(162)	79.6(5)
C(11)-C(16)-C(161)-C(162)	-101.9(4)
P(2)-C(2)-C(3)-P(3)	-42.6(3)
C(31)-P(3)-C(3)-C(2)	150.0(3)
C(32)-P(3)-C(3)-C(2)	-92.1(3)
Ni-P(3)-C(3)-C(2)	30.9(3)
C(125)-C(126)-C(121)-C(122)	-0.6(6)
C(129)-C(126)-C(121)-C(122)	178.2(4)
C(125)-C(126)-C(121)-C(12)	176.6(4)
C(129)-C(126)-C(121)-C(12)	-4.6(6)
C(123)-C(122)-C(121)-C(126)	0.3(6)
C(127)-C(122)-C(121)-C(126)	179.5(3)
C(123)-C(122)-C(121)-C(12)	-176.9(3)
C(127)-C(122)-C(121)-C(12)	2.2(5)
C(13)-C(12)-C(121)-C(126)	-73.4(5)
C(11)-C(12)-C(121)-C(126)	112.2(4)
C(13)-C(12)-C(121)-C(122)	103.8(4)
C(11)-C(12)-C(121)-C(122)	-70.6(5)
C(15)-C(14)-C(13)-C(12)	2.5(6)
C(11)-C(12)-C(13)-C(14)	1.7(6)
C(121)-C(12)-C(13)-C(14)	-172.9(4)
C(163)-C(164)-C(165)-C(166)	1.2(6)
C(168)-C(164)-C(165)-C(166)	-177.1(4)
C(161)-C(166)-C(165)-C(164)	-0.6(6)
C(169)-C(166)-C(165)-C(164)	-179.8(4)
C(121)-C(126)-C(125)-C(124)	0.3(6)
C(129)-C(126)-C(125)-C(124)	-178.5(4)
C(126)-C(125)-C(124)-C(123)	0.3(6)
C(126)-C(125)-C(124)-C(128)	-179.4(4)
C(122)-C(123)-C(124)-C(125)	-0.6(6)
C(122)-C(123)-C(124)-C(128)	179.1(4)
C(11S)-O(1S)-C(14S)-C(13S)	-6.3(5)
C(14S)-O(1S)-C(11S)-C(12S)	27.0(5)
O(1S)-C(14S)-C(13S)-C(12S)	-16.6(5)
O(1S)-C(11S)-C(12S)-C(13S)	-36.1(5)
C(14S)-C(13S)-C(12S)-C(11S)	31.0(5)

Data Collection for Ni(P(dmp))(^{t}Bu$_2$PCH$_2$CH$_2$P^{t}Bu$_2$)

A green block was selected under a stereo-microscope while immersed in mineral oil to avoid possible reaction with air. The crystal was removed from the oil using a tapered fiber that also served to hold the crystal for data collection. The crystal was mounted and centered on a Bruker SMART APEX system. Rotation and still images showed diffractions to be sharp while frames separated in reciprocal space were obtained and provided an orientation matrix and initial cell parameters. Final cell parameters were obtained from the full data set.

A "hemisphere" data set was obtained which samples approximately 1.2 hemispheres of reciprocal space to a resolution of 0.84 Å using 0.3 degree steps in ω using 10 sec integration times for each frame. Absorption corrections were applied using SADABS [1] or semi-empirical from psi-scans.

Structure solution and refinement

The space group was determined as $P2_12_12_1$ based on systematic absences and intensity statistics. Direct Methods were used to refine the nickel and phosphorous atoms. All atoms were converted to and refined anisotropically. Hydrogen atoms were refined isotropically and fixed at calculated positions. When the structure was inverted and refined the wrong absolute structure was obtained (Flack x parameter ca 1).

$R_{int} = \Sigma \mid F_o^2 - \langle F_o^2 \rangle \mid / \Sigma \mid F_o^2 \mid$

$wR_2 = [\Sigma [w (F_o^2 - F_c^2)^2] / \Sigma [w (F_o^2)^2]]^{1/2}$

$GoF = S = [\Sigma [w (F_o^2 - F_c^2)^2] / (n-p)^{1/2}$

$R_1 = \Sigma \mid \mid F_o \mid - \mid F_c \mid \mid / \Sigma \mid F_o \mid$

where: $w = q / \sigma^2 (F_o^2) + (aP)^2 + bP$;

n = number of independent reflections;

p = number of parameters refined.

References

[1] All software and sources of scattering factors are contained in the SHELXTL (version 5.1) program library (G. Sheldrick, Bruker Analytical X-ray Systems, Madison, WI).

Table 1. Crystal and structure refinement for Ni(P(dmp))(^{t}Bu$_2$PCH$_2$CH$_2$P^{t}Bu$_2$).

Identification Code	rory08m
Empirical formula	C42 H65 Ni P3
Formula weight	721.56

Temperature	100 K
Wavelength	0.71073 Å
Crystal system	orthorhombic
Space Group	$P2_12_12_1$
Unit cell dimensions	a = 11.8184(10) Å α = 90.00 °
	b = 16.2135(14) Å β = 90.00 °
	c = 21.4755(18)Å γ = 90.00 °
Volume	4115.1(6) $Å^3$
Z	4
Density (calculated)	1.165 Mg/m^3
Absorption coefficient	0.615 mm^{-1}
F(000)	1560.0
Crystal size	90 x 100 x 220 μm, green block
Theta range for data collection	1.57 to 28.31
Index ranges	$-15 \le h \le 15$, $-19 \le k \le 21$ $-28 \le l \le 22$
Reflections collected	25512
Independent reflections	9745 (R_{int} = 0.0666)
Completeness to theta = 28.31°	97.3%
Absorption correction	Semi-empirical from psi scans
Max. and min. transmission	0.2666 and 0.2300
Refinement method	Full-matrix least squares on F^2
Data / restraints / parameters	9745 / 0 / 416
Goodness-of-fit on F^2	0.856
Final R indices [I > 2 sigma(I)]	R_1 = 0.0442, wR_2 = 0.0724
R indices (all data)	R_1 = 0.0652, wR_2 = 0.0753
Largest diff. peak and hole	0.675 and –0.455 $eÅ^{-3}$
Flack x parameter	-0.009 0.012 esd

Table 2. Atomic coordinates [x 10^4] and equivalent isotropic displacement parameters [$Å^2$ x 10^3] for Ni(P(dmp))($^tBu_2PCH_2CH_2P^tBu_2$). U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Ni	9158(1)	4313(1)	728(1)	18(1)
P(1)	10892(1)	4520(1)	736(1)	20(1)
P(3)	7984(1)	4556(1)	-38(1)	18(1)
P(2)	8062(1)	3334(1)	1101(1)	18(1)
C(11)	11740(3)	5259(2)	1172(2)	19(1)
C(3)	6968(3)	3681(2)	-46(2)	19(1)
C(121)	12081(3)	4172(2)	2000(2)	17(1)
C(163)	11869(3)	6436(2)	-802(2)	21(1)
C(12)	12241(3)	5014(2)	1749(2)	20(1)
C(313)	7825(3)	4343(2)	-1369(1)	27(1)
C(161)	11647(3)	6334(2)	309(2)	18(1)
C(165)	10419(3)	7213(2)	-299(2)	20(1)
C(166)	10755(3)	6907(2)	280(2)	19(1)
C(312)	9485(3)	3757(2)	-821(2)	24(1)
C(162)	12214(3)	6110(2)	-238(2)	19(1)
C(126)	12772(3)	3538(2)	1785(2)	17(1)
C(2)	6763(2)	3334(2)	613(2)	20(1)
C(32)	7076(3)	5515(2)	14(2)	23(1)
C(323)	6227(3)	5387(2)	541(2)	30(1)
C(164)	10968(3)	6980(2)	-844(2)	23(1)
C(16)	12046(3)	6036(2)	931(2)	18(1)
C(13)	12941(3)	5553(2)	2062(2)	24(1)
C(31)	8656(3)	4492(2)	-832(2)	21(1)
C(123)	11273(3)	3240(2)	2757(2)	25(1)
C(124)	11937(3)	2602(2)	2538(2)	24(1)
C(223)	6353(3)	3037(2)	2038(2)	34(1)
C(122)	11336(3)	4024(2)	2494(2)	21(1)
C(168)	10570(3)	7301(2)	-1465(2)	30(1)
C(322)	6401(3)	5730(2)	-574(2)	30(1)
C(169)	10149(3)	7170(2)	865(1)	24(1)
C(15)	12760(3)	6549(2)	1262(2)	25(1)
C(14)	13211(3)	6319(2)	1830(2)	29(1)
C(127)	10602(3)	4706(2)	2742(2)	30(1)
C(213)	8766(3)	2086(2)	327(2)	29(1)
C(129)	13600(3)	3708(2)	1260(2)	26(1)
C(167)	13196(3)	5521(2)	-213(2)	28(1)
C(222)	8370(3)	3198(2)	2402(2)	30(1)
C(212)	9829(3)	2206(2)	1306(2)	25(1)
C(21)	8644(3)	2254(2)	1023(2)	20(1)
C(211)	7888(3)	1578(2)	1296(2)	27(1)
C(321)	7850(3)	6241(2)	190(2)	35(1)
C(311)	9362(3)	5272(2)	-959(2)	28(1)
C(125)	12694(3)	2770(2)	2052(2)	23(1)
C(128)	11874(3)	1756(2)	2828(2)	37(1)
C(221)	7288(3)	4406(2)	2008(2)	41(1)

C(22)	7491(3)	3474(2)	1914(2)	22(1)

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

Table 3. Bond lengths [Å] and angles [°] for Ni(P(dmp))(^{t}Bu$_2$PCH$_2$CH$_2$P^{t}Bu$_2$).

Ni-P(1)	2.0772(9)
Ni-P(3)	2.1869(10)
Ni-P(2)	2.1987(10)
P(1)-C(11)	1.822(3)
P(3)-C(3)	1.858(3)
P(3)-C(31)	1.885(3)
P(3)-C(32)	1.892(3)
P(2)-C(2)	1.859(3)
P(2)-C(22)	1.887(3)
P(2)-C(21)	1.889(3)
C(11)-C(16)	1.409(4)
C(11)-C(12)	1.430(4)
C(3)-C(2)	1.541(4)
C(121)-C(126)	1.392(4)
C(121)-C(122)	1.399(4)
C(121)-C(12)	1.481(4)
C(163)-C(162)	1.383(4)
C(163)-C(164)	1.386(4)
C(12)-C(13)	1.378(4)
C(313)-C(31)	1.534(4)
C(161)-C(162)	1.400(4)
C(161)-C(166)	1.407(4)
C(161)-C(16)	1.497(4)
C(165)-C(164)	1.391(4)
C(165)-C(166)	1.397(4)
C(166)-C(169)	1.508(4)
C(312)-C(31)	1.543(4)
C(162)-C(167)	1.504(4)
C(126)-C(125)	1.374(4)
C(126)-C(129)	1.518(4)
C(32)-C(323)	1.527(4)
C(32)-C(322)	1.534(4)
C(32)-C(321)	1.538(4)
C(164)-C(168)	1.507(4)
C(16)-C(15)	1.383(4)
C(13)-C(14)	1.375(4)
C(31)-C(311)	1.540(4)
C(123)-C(124)	1.381(4)
C(123)-C(122)	1.391(4)
C(124)-C(125)	1.402(4)
C(124)-C(128)	1.509(4)
C(223)-C(22)	1.544(4)
C(122)-C(127)	1.503(4)
C(15)-C(14)	1.382(5)
C(213)-C(21)	1.527(4)
C(222)-C(22)	1.542(4)

C(212)-C(21)	1.529(4)
C(21)-C(211)	1.531(4)
C(221)-C(22)	1.544(4)

P(1)-Ni-P(3)	127.04(4)
P(1)-Ni-P(2)	134.03(4)
P(3)-Ni-P(2)	91.72(4)
C(11)-P(1)-Ni	130.78(11)
C(3)-P(3)-C(31)	102.81(14)
C(3)-P(3)-C(32)	105.13(14)
C(31)-P(3)-C(32)	109.67(14)
C(3)-P(3)-Ni	106.20(10)
C(31)-P(3)-Ni	113.83(11)
C(32)-P(3)-Ni	117.65(11)
C(2)-P(2)-C(22)	103.11(15)
C(2)-P(2)-C(21)	104.53(15)
C(22)-P(2)-C(21)	108.86(15)
C(2)-P(2)-Ni	106.38(10)
C(22)-P(2)-Ni	117.43(11)
C(21)-P(2)-Ni	114.97(11)
C(16)-C(11)-C(12)	117.4(3)
C(16)-C(11)-P(1)	122.7(3)
C(12)-C(11)-P(1)	119.4(2)
C(2)-C(3)-P(3)	111.9(2)
C(126)-C(121)-C(122)	119.6(3)
C(126)-C(121)-C(12)	119.0(3)
C(122)-C(121)-C(12)	121.0(3)
C(162)-C(163)-C(164)	121.8(3)
C(13)-C(12)-C(11)	119.7(3)
C(13)-C(12)-C(121)	118.9(3)
C(11)-C(12)-C(121)	121.3(3)
C(162)-C(161)-C(166)	119.5(3)
C(162)-C(161)-C(16)	120.9(3)
C(166)-C(161)-C(16)	119.3(3)
C(164)-C(165)-C(166)	121.3(3)
C(165)-C(166)-C(161)	119.2(3)
C(165)-C(166)-C(169)	120.4(3)
C(161)-C(166)-C(169)	120.4(3)
C(163)-C(162)-C(161)	119.6(3)
C(163)-C(162)-C(167)	120.1(3)
C(161)-C(162)-C(167)	120.3(3)
C(125)-C(126)-C(121)	119.4(3)
C(125)-C(126)-C(129)	121.2(3)
C(121)-C(126)-C(129)	119.4(3)
C(3)-C(2)-P(2)	112.7(2)
C(323)-C(32)-C(322)	107.4(3)
C(323)-C(32)-C(321)	108.2(3)
C(322)-C(32)-C(321)	109.7(3)
C(323)-C(32)-P(3)	107.7(2)
C(322)-C(32)-P(3)	115.8(2)
C(321)-C(32)-P(3)	107.8(2)
C(163)-C(164)-C(165)	118.5(3)
C(163)-C(164)-C(168)	121.2(3)
C(165)-C(164)-C(168)	120.3(3)

C(15)-C(16)-C(11)	120.4(3)
C(15)-C(16)-C(161)	117.2(3)
C(11)-C(16)-C(161)	122.4(3)
C(14)-C(13)-C(12)	122.3(3)
C(313)-C(31)-C(311)	110.1(3)
C(313)-C(31)-C(312)	107.2(3)
C(311)-C(31)-C(312)	107.0(2)
C(313)-C(31)-P(3)	114.9(2)
C(311)-C(31)-P(3)	110.1(2)
C(312)-C(31)-P(3)	107.2(2)
C(124)-C(123)-C(122)	121.1(3)
C(123)-C(124)-C(125)	118.1(3)
C(123)-C(124)-C(128)	120.9(3)
C(125)-C(124)-C(128)	121.0(3)
C(123)-C(122)-C(121)	119.8(3)
C(123)-C(122)-C(127)	119.9(3)
C(121)-C(122)-C(127)	120.3(3)
C(14)-C(15)-C(16)	121.8(3)
C(13)-C(14)-C(15)	118.3(3)
C(213)-C(21)-C(212)	107.1(3)
C(213)-C(21)-C(211)	107.5(3)
C(212)-C(21)-C(211)	110.3(3)
C(213)-C(21)-P(2)	106.7(2)
C(212)-C(21)-P(2)	110.3(2)
C(211)-C(21)-P(2)	114.7(2)
C(126)-C(125)-C(124)	122.0(3)
C(222)-C(22)-C(223)	109.7(3)
C(222)-C(22)-C(221)	107.5(3)
C(223)-C(22)-C(221)	106.9(3)
C(222)-C(22)-P(2)	110.7(2)
C(223)-C(22)-P(2)	114.6(2)
C(221)-C(22)-P(2)	107.1(2)

Table 4. Anisotropic displacement parameters [$Å^2$ x 10^3] for Ni(P(dmp))($^tBu_2PCH_2CH_2P^tBu_2$). The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U^{11}+ ... + 2hka^*b^*U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Ni	16(1)	18(1)	20(1)	-1(1)	-2(1)	-1(1)
P(1)	18(1)	19(1)	22(1)	0(1)	-2(1)	-2(1)
P(3)	16(1)	17(1)	23(1)	0(1)	-1(1)	-1(1)
P(2)	16(1)	20(1)	19(1)	0(1)	-1(1)	1(1)
C(11)	13(2)	23(2)	20(2)	0(2)	2(2)	2(1)
C(3)	16(2)	19(2)	21(2)	0(2)	-7(2)	-1(2)
C(121)	18(2)	20(2)	15(2)	3(2)	-6(2)	-5(2)
C(163)	20(2)	20(2)	21(2)	-2(2)	7(2)	-5(2)
C(12)	15(2)	24(2)	20(2)	-3(2)	1(2)	1(2)
C(313)	29(2)	26(2)	26(2)	0(2)	-2(2)	0(2)
C(161)	16(2)	15(2)	22(2)	-1(2)	-1(2)	-5(1)
C(165)	19(2)	14(2)	28(2)	3(2)	1(2)	-2(1)
C(166)	21(2)	16(2)	22(2)	1(2)	3(2)	-8(2)
C(312)	27(2)	28(2)	18(2)	-4(2)	0(2)	-2(2)
C(162)	13(2)	18(2)	28(2)	-4(2)	3(2)	-4(2)
C(126)	15(2)	20(2)	16(2)	1(2)	-5(2)	-4(2)
C(2)	16(2)	16(2)	27(2)	5(2)	-4(2)	0(1)
C(32)	18(2)	20(2)	31(2)	-1(2)	0(2)	0(2)
C(323)	27(2)	27(2)	37(3)	5(2)	6(2)	12(2)
C(164)	23(2)	22(2)	24(2)	2(2)	0(2)	-10(2)
C(16)	16(2)	17(2)	22(2)	-3(2)	2(2)	2(2)
C(13)	28(2)	25(2)	19(2)	1(2)	-5(2)	-5(2)
C(31)	22(2)	22(2)	18(2)	1(2)	2(2)	-2(2)
C(123)	29(2)	29(2)	16(2)	-1(2)	-2(2)	-10(2)
C(124)	24(2)	29(2)	20(2)	7(2)	-8(2)	-4(2)
C(223)	25(2)	53(3)	25(2)	12(2)	7(2)	8(2)
C(122)	21(2)	23(2)	20(2)	-2(2)	-5(2)	1(2)
C(168)	25(2)	31(2)	35(2)	5(2)	1(2)	-2(2)
C(322)	25(2)	27(2)	39(2)	4(2)	-2(2)	3(2)
C(169)	28(2)	17(2)	27(2)	-1(2)	4(2)	0(2)
C(15)	30(2)	19(2)	26(2)	3(2)	-2(2)	-2(2)
C(14)	27(2)	31(2)	29(2)	-9(2)	-3(2)	-5(2)
C(127)	33(2)	29(2)	28(2)	-3(2)	5(2)	-6(2)
C(213)	30(2)	26(2)	31(2)	-5(2)	-4(2)	13(2)
C(129)	22(2)	28(2)	27(2)	-1(2)	1(2)	4(2)
C(167)	24(2)	33(2)	27(2)	-5(2)	2(2)	1(2)
C(222)	38(2)	37(2)	16(2)	-6(2)	-1(2)	4(2)
C(212)	27(2)	24(2)	24(2)	-3(2)	-4(2)	7(2)
C(21)	18(2)	19(2)	23(2)	0(2)	-4(2)	0(2)
C(211)	34(2)	19(2)	27(2)	1(2)	-6(2)	-2(2)
C(321)	36(3)	23(2)	45(3)	-9(2)	-4(2)	4(2)
C(311)	29(2)	27(2)	27(2)	1(2)	5(2)	-3(2)
C(125)	21(2)	26(2)	24(2)	-6(2)	-4(2)	3(2)
C(128)	44(3)	31(2)	37(3)	7(2)	-1(2)	0(2)
C(221)	60(3)	38(2)	25(2)	-5(2)	6(2)	10(2)

C(22)	25(2)	22(2)	18(2)	0(2)	1(2)	6(2)

Table 5. Hydrogen coordinates [x 10^4] and isotropic displacement parameters [$Å^2$ x 10^3] for Ni(P(dmp))($^tBu_2PCH_2CH_2P^tBu_2$)

	x	y	z	U(eq)
H(3A)	6255	3866	-220	23
H(3B)	7261	3247	-311	23
H(16A)	12252	6285	-1163	25
H(31A)	8235	4314	-1755	40
H(31B)	7290	4787	-1387	40
H(31C)	7431	3833	-1302	40
H(16B)	9815	7580	-321	24
H(31D)	9844	3706	-1220	36
H(31E)	9077	3260	-729	36
H(31F)	10049	3849	-507	36
H(2A)	6482	2773	578	23
H(2B)	6185	3661	817	23
H(32A)	5750	5864	575	45
H(32B)	6626	5305	925	45
H(32C)	5771	4911	454	45
H(13A)	13242	5393	2443	29
H(12A)	10776	3145	3085	30
H(22A)	6116	3145	2458	52
H(22B)	6442	2454	1980	52
H(22C)	5792	3242	1754	52
H(16C)	9947	7673	-1403	45
H(16D)	10330	6848	-1721	45
H(16E)	11178	7588	-1667	45
H(32D)	5951	6213	-498	45
H(32E)	5916	5277	-681	45
H(32F)	6914	5836	-911	45
H(16F)	9561	7555	761	36
H(16G)	10677	7427	1144	36
H(16H)	9822	6695	1063	36
H(15A)	12942	7064	1099	30
H(14A)	13685	6672	2049	35
H(12B)	10161	4504	3084	45
H(12C)	10107	4897	2418	45
H(12D)	11071	5154	2880	45
H(21A)	9060	1540	266	43
H(21B)	9275	2481	147	43
H(21C)	8039	2131	130	43
H(12E)	14022	3216	1170	39
H(12F)	14111	4139	1382	39
H(12G)	13191	3876	895	39
H(16I)	13484	5435	-626	43
H(16J)	12950	5004	-43	43
H(16K)	13782	5748	45	43
H(22D)	8069	3282	2813	45
H(22E)	9049	3516	2353	45
H(22F)	8539	2624	2344	45
H(21D)	10114	1654	1266	38

H(21E)	9795	2354	1738	38
H(21F)	10323	2580	1091	38
H(21G)	8244	1051	1239	40
H(21H)	7170	1582	1086	40
H(21I)	7776	1678	1732	40
H(32G)	7406	6734	228	52
H(32H)	8412	6316	-128	52
H(32I)	8215	6126	580	52
H(31G)	9703	5233	-1364	41
H(31H)	9944	5321	-649	41
H(31I)	8881	5749	-944	41
H(12H)	13157	2349	1905	28
H(12I)	12406	1396	2626	56
H(12J)	11124	1539	2779	56
H(12K)	12053	1793	3263	56
H(22G)	6998	4501	2420	61
H(22H)	6750	4599	1707	61
H(22I)	7988	4698	1957	61

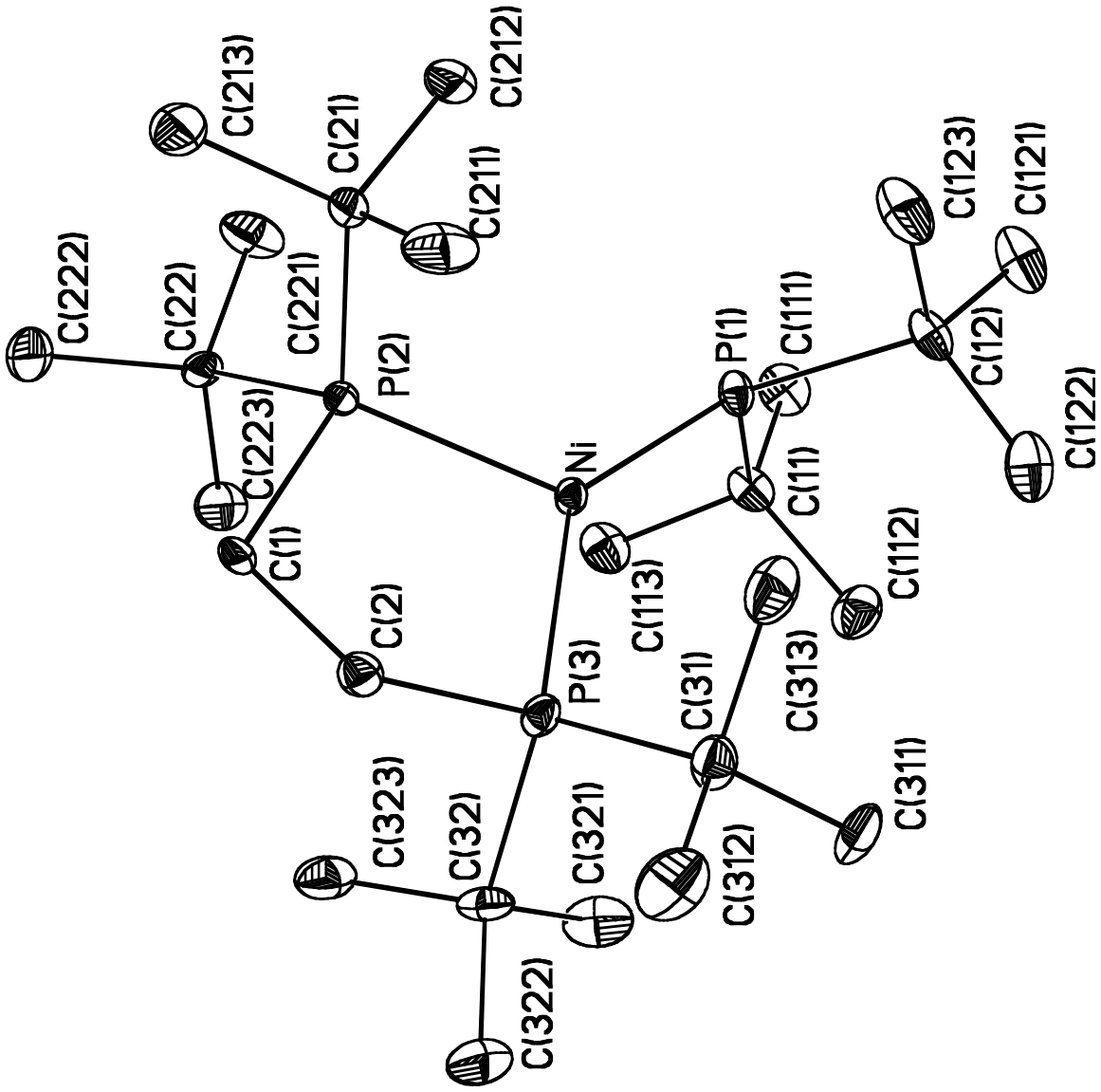
Table 6. Torsion angles [°] for Ni(Pdmp)(^{t}Bu$_2$PCH$_2$CH$_2$P^{t}Bu$_2$).

P(3)-Ni-P(1)-C(11)	111.71(15)
P(2)-Ni-P(1)-C(11)	-106.86(15)
P(1)-Ni-P(3)-C(3)	140.65(11)
P(2)-Ni-P(3)-C(3)	-12.70(12)
P(1)-Ni-P(3)-C(31)	28.28(12)
P(2)-Ni-P(3)-C(31)	-125.08(11)
P(1)-Ni-P(3)-C(32)	-102.02(12)
P(2)-Ni-P(3)-C(32)	104.62(12)
P(1)-Ni-P(2)-C(2)	-154.68(11)
P(3)-Ni-P(2)-C(2)	-4.54(11)
P(1)-Ni-P(2)-C(22)	90.55(13)
P(3)-Ni-P(2)-C(22)	-119.31(12)
P(1)-Ni-P(2)-C(21)	-39.51(14)
P(3)-Ni-P(2)-C(21)	110.63(12)
Ni-P(1)-C(11)-C(16)	-93.8(3)
Ni-P(1)-C(11)-C(12)	94.3(3)
C(31)-P(3)-C(3)-C(2)	151.4(2)
C(32)-P(3)-C(3)-C(2)	-93.9(2)
Ni-P(3)-C(3)-C(2)	31.5(2)
C(16)-C(11)-C(12)-C(13)	3.6(5)
P(1)-C(11)-C(12)-C(13)	175.9(2)
C(16)-C(11)-C(12)-C(121)	-172.5(3)
P(1)-C(11)-C(12)-C(121)	-0.2(4)
C(126)-C(121)-C(12)-C(13)	-93.2(4)
C(122)-C(121)-C(12)-C(13)	79.9(4)
C(126)-C(121)-C(12)-C(11)	83.0(4)
C(122)-C(121)-C(12)-C(11)	-104.0(4)
C(164)-C(165)-C(166)-C(161)	-1.7(5)
C(164)-C(165)-C(166)-C(169)	179.8(3)

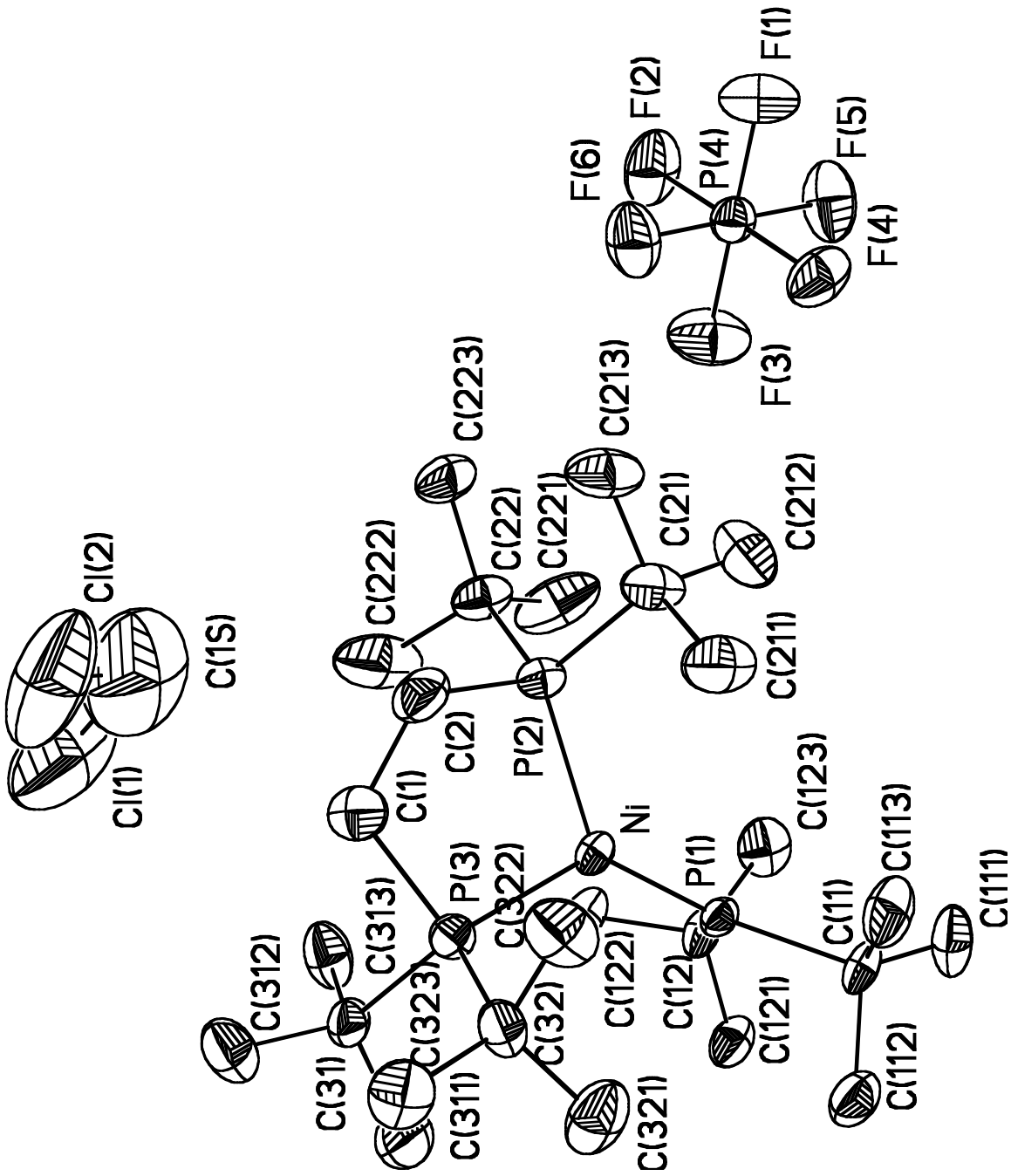
C(162)-C(161)-C(166)-C(165)	2.8(4)
C(16)-C(161)-C(166)-C(165)	177.1(3)
C(162)-C(161)-C(166)-C(169)	-178.8(3)
C(16)-C(161)-C(166)-C(169)	-4.4(4)
C(164)-C(163)-C(162)-C(161)	-0.2(5)
C(164)-C(163)-C(162)-C(167)	180.0(3)
C(166)-C(161)-C(162)-C(163)	-1.9(5)
C(16)-C(161)-C(162)-C(163)	-176.1(3)
C(166)-C(161)-C(162)-C(167)	177.9(3)
C(16)-C(161)-C(162)-C(167)	3.7(5)
C(122)-C(121)-C(126)-C(125)	1.5(5)
C(12)-C(121)-C(126)-C(125)	174.7(3)
C(122)-C(121)-C(126)-C(129)	-178.5(3)
C(12)-C(121)-C(126)-C(129)	-5.4(5)
P(3)-C(3)-C(2)-P(2)	-37.0(3)
C(22)-P(2)-C(2)-C(3)	149.5(2)
C(21)-P(2)-C(2)-C(3)	-96.7(2)
Ni-P(2)-C(2)-C(3)	25.4(2)
C(3)-P(3)-C(32)-C(323)	49.2(3)
C(31)-P(3)-C(32)-C(323)	159.1(2)
Ni-P(3)-C(32)-C(323)	-68.7(2)
C(3)-P(3)-C(32)-C(322)	-71.0(3)
C(31)-P(3)-C(32)-C(322)	38.9(3)
Ni-P(3)-C(32)-C(322)	171.10(19)
C(3)-P(3)-C(32)-C(321)	165.7(2)
C(31)-P(3)-C(32)-C(321)	-84.4(3)
Ni-P(3)-C(32)-C(321)	47.8(3)
C(162)-C(163)-C(164)-C(165)	1.4(5)
C(162)-C(163)-C(164)-C(168)	-177.4(3)
C(166)-C(165)-C(164)-C(163)	-0.4(5)
C(166)-C(165)-C(164)-C(168)	178.4(3)
C(12)-C(11)-C(16)-C(15)	-3.1(5)
P(1)-C(11)-C(16)-C(15)	-175.2(2)
C(12)-C(11)-C(16)-C(161)	175.4(3)
P(1)-C(11)-C(16)-C(161)	3.3(4)
C(162)-C(161)-C(16)-C(15)	94.6(4)
C(166)-C(161)-C(16)-C(15)	-79.6(4)
C(162)-C(161)-C(16)-C(11)	-83.9(4)
C(166)-C(161)-C(16)-C(11)	101.9(4)
C(11)-C(12)-C(13)-C(14)	-2.0(5)
C(121)-C(12)-C(13)-C(14)	174.1(3)
C(3)-P(3)-C(31)-C(313)	44.2(3)
C(32)-P(3)-C(31)-C(313)	-67.2(3)
Ni-P(3)-C(31)-C(313)	158.6(2)
C(3)-P(3)-C(31)-C(311)	169.2(2)
C(32)-P(3)-C(31)-C(311)	57.7(2)
Ni-P(3)-C(31)-C(311)	-76.4(2)
C(3)-P(3)-C(31)-C(312)	-74.8(2)
C(32)-P(3)-C(31)-C(312)	173.8(2)
Ni-P(3)-C(31)-C(312)	39.6(2)
C(122)-C(123)-C(124)-C(125)	1.3(5)
C(122)-C(123)-C(124)-C(128)	179.4(3)
C(124)-C(123)-C(122)-C(121)	0.1(5)
C(124)-C(123)-C(122)-C(127)	179.9(3)

C(126)-C(121)-C(122)-C(123)	-1.5(5)
C(12)-C(121)-C(122)-C(123)	-174.5(3)
C(126)-C(121)-C(122)-C(127)	178.6(3)
C(12)-C(121)-C(122)-C(127)	5.6(5)
C(11)-C(16)-C(15)-C(14)	1.1(5)
C(161)-C(16)-C(15)-C(14)	-177.5(3)
C(12)-C(13)-C(14)-C(15)	-0.1(5)
C(16)-C(15)-C(14)-C(13)	0.5(5)
C(2)-P(2)-C(21)-C(213)	53.9(3)
C(22)-P(2)-C(21)-C(213)	163.6(2)
Ni-P(2)-C(21)-C(213)	-62.3(2)
C(2)-P(2)-C(21)-C(212)	169.9(2)
C(22)-P(2)-C(21)-C(212)	-80.5(3)
Ni-P(2)-C(21)-C(212)	53.6(3)
C(2)-P(2)-C(21)-C(211)	-65.0(3)
C(22)-P(2)-C(21)-C(211)	44.7(3)
Ni-P(2)-C(21)-C(211)	178.8(2)
C(121)-C(126)-C(125)-C(124)	-0.2(5)
C(129)-C(126)-C(125)-C(124)	179.8(3)
C(123)-C(124)-C(125)-C(126)	-1.2(5)
C(128)-C(124)-C(125)-C(126)	-179.4(3)
C(2)-P(2)-C(22)-C(222)	160.6(2)
C(21)-P(2)-C(22)-C(222)	50.0(3)
Ni-P(2)-C(22)-C(222)	-82.9(2)
C(2)-P(2)-C(22)-C(223)	35.9(3)
C(21)-P(2)-C(22)-C(223)	-74.7(3)
Ni-P(2)-C(22)-C(223)	152.45(19)
C(2)-P(2)-C(22)-C(221)	-82.5(2)
C(21)-P(2)-C(22)-C(221)	166.9(2)
Ni-P(2)-C(22)-C(221)	34.0(3)

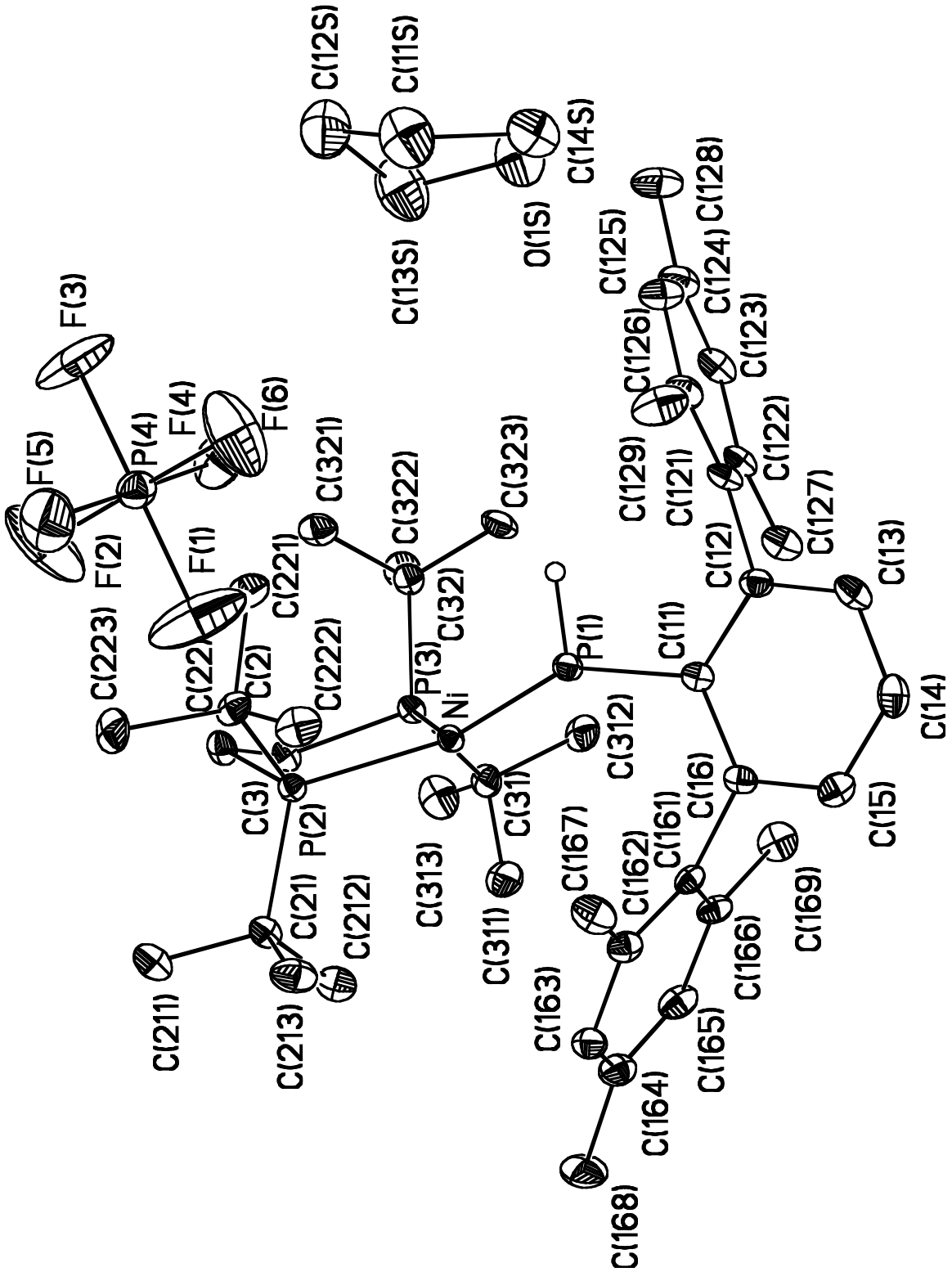
Thermal Ellipsoid Plot of (dtbpe)Ni(P(tBu)$_2$) at the 50% probability level



Thermal Ellipsoid Plot of [(dtbpe)Ni(P(tBu)$_2$)][PF$_6$]•CH$_2$Cl$_2$ at the 50% probability level



Thermal Ellipsoid Plot of [(dtbpe)Ni(P(H)(dmp))][PF_6]•THF at the 50% probability level



Thermal Ellipsoid Plot of (dtbpe)Ni(P(dmp)) at the 50% probability level