ORGANOMETALLICS

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Synthesis of 1d,e; To a solution of 2-(9-phenanthrenyl)phenol or 2-(9-anthracenyl)phenol (25 mmol) and 2,6-lutidine (4.6 g, 43 mmol) in toluene (50 mL) under an atmosphere of Ar was slowly added $SnCl_4$ (0.75 mL, 6.4 mmol). The solution was stirred at rt for 20 min. Paraformaldehyde was added (4.3 g, 140 mmol) and the reaction was stirred at 110°C for 12 h. After cooling to rt, the reaction mixture was poured into water (300 mL), acidified to pH 1 with concentrated HCl, extracted with diethyl ether (500 mL), separated, the combined organic layers washed twice with sat. brine, and dried over Na_2SO_4. The solvent was removed by rotary evaporation to yield a yellow oil. The crude product was purified by flash chromatography on silica gel eluting with hexanes:ethyl acetate (9:1) to yield a white solid.

1d (R= 9-phenanthrenyl, 26%): ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.17-7.88 (m, 10H), 8.79 (m, 2H), 10.03 (s, 1H), 11.31 (s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 120.0, 120.6, 122.7, 123.0, 126.6, 126.9, 127.0, 128.5, 128.8, 130.5, 130.8, 131.5, 133.8, 139.1, 159.6, 196.9. Anal. Calcd for $C_{21}H_{14}O_2$: C, 84.54; H, 4.73. Found: C, 84.41; H, 4.59.

1e (R= 9-anthracenyl, 24%): ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.22-8.09 (m, 11H), 8.56 (s, 1H), 10.05 (s, 1H), 11.22 (s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 120.0, 120.9, 125.3, 125.9, 126.1, 127.3, 127.6, 128.8, 130.3, 130.8, 131.5, 134.0, 140.4, 159.9, 196.9. Anal. Calcd for $C_{21}H_{14}O_2$: C, 84.55; H, 4.73. Found: C, 83.93; H, 4.63.

General Procedure for Preparation of Salicylaldimine Ligands (2b-f); To a methanol (15 mL) solution of 2-hydroxy-3-phenyl benzaldehyde (2.4 g, 12 mmol) was added formic acid (0.50 mL) and 2,6-diisopropylaniline (2.8 g, 16 mmol). The resulting mixture was stirred at rt for 10 h. During this time a yellow solid precipitated from the reaction mixture. The solid was filtered, washed with cold methanol and then dried to afford the salicylaldimine ligand **2c** (3.9 g, 92%). The other ligands were prepared by the same procedure with similar yields (ligands **2a** and **2g** were synthesized

according to literature procedure: Chang, S.; Jones, L.; Wang, C.; Henling, L. M.; Grubbs, R. H. *Organometallics* **1998**, *17*, in Press).

2b (R = *t*-Bu, X = H, 90%): ^{1}H NMR (300 MHz, C_6D_6): δ 1.24 (d, 12H, J_{HH} = 6.9 Hz), 1.56 (s, 9H), 3.10 (sept., 2H, J_{HH} = 6.9 Hz), 6.94-7.49 (m, 6H), 8.39 (s, 1H), 13.71 (s, 1H); ^{13}C NMR (75 MHz, C_6D_6): δ 23.5, 28.2, 34.9, 118.3, 118.6, 123.3, 125.4, 130.5, 130.8, 137.6, 139.0, 146.4, 160.7, 167.6. Anal. Calcd for $C_{23}H_{31}NO$: C, 81.85; H, 9.26; N, 4.15. Found: C, 81.98; H, 9.72; N, 4.41.

2c (R = Ph, X = H, 90%): ^{1}H NMR (300 MHz, C_6D_6): δ 1.01 (d, 12H, J_{HH} = 6.9 Hz), 2.96 (sept., 2H, J_{HH} = 6.9 Hz), 7.05-7.74 (m, 11H), 7.92 (s, 1H), 13.90 (s, 1H); ^{13}C NMR (75 MHz, C_6D_6): δ 23.5, 28.5, 119.2, 119.3, 123.5, 125.9, 127.4, 127.7. 129.9, 130.8, 131.9, 134.7, 138.0, 138.9, 146.8, 159.4, 167.6. Anal. Calcd for $C_{25}H_{27}NO$: C, 83.99; H, 7.61; N, 3.92. Found: C, 84.12; H, 7.69; N, 4.05.

2d (R = 9-phenanthrenyl, X = H, 84%): ^{1}H NMR (300 MHz, $CDCl_3$): δ 1.21 (d, 6H, J_{HH} = 6.9 Hz), 1.22 (d, 6H, J_{HH} = 6.9 Hz), 3.07 (sept., 2H, J_{HH} = 6.9 Hz), 7.14-7.97 (m, 13H), 8.46 (s, 1H), 8.80 (m, 2H), 13.45 (s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 23.8, 28.2, 119.0, 122.7, 123.0, 123.4, 125.6, 126.6, 126.8, 127.2, 128.5, 128.9, 129.3, 130.4, 130.6, 131.2, 131.7, 132.2, 135.6, 138.9, 159.3, 166.9. Anal. Calcd for $C_{33}H_{31}NO$: C, 86.62; H, 6.83; N, 3.06. Found: C, 86.69; H, 6.78; N, 2.78.

2e (R = 9-anthracenyl, X = H, 88%): ^{1}H NMR (300 MHz, $CDCl_3$): δ 1.23 (d, 12H, J_{HH} = 6.9 Hz), 3.09 (sept., 2H, J_{HH} = 6.9 Hz), 7.23-8.13 (m, 14H), 8.52 (s, 1H), 8.59 (s, 1H), 13.33 (s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 23.8, 28.2, 118.9, 119.1, 123.4, 125.2, 125.6, 125.7, 126.7, 127.0, 127.3, 128.5, 128.8, 130.5, 131.6, 132.4, 132.5, 136.8, 138.9, 146.3, 159.6, 166.8. Anal. Calcd for $C_{33}H_{31}NO$: C, 86.62; H, 6.83; N, 3.06. Found: C, 86.42; H, 7.05; N, 2.99.

2f (R = H, X = OMe, 72%): ^{1}H NMR (400 MHz, C_6D_6): δ 1.07 (d, 12H, J_{HH} = 8.6 Hz), 2.98 (sept., 2H, J_{HH} = 8.6 Hz), 3.29 (s, 3H), 6.60-7.16 (m, 6H), 7.86 (s, 1H), 12.89 (s, 1H); ^{13}C NMR (75 MHz, C_6D_6): δ 23.5, 28.5, 55.3, 115.8, 118.7, 120.7,

123.5, 125.8, 138.7, 147.1, 152.7, 156.2, 167. Anal. Calcd for $C_{20}H_{25}NO_2$: C, 77.14; H, 8.09; N, 4.50. Found: C, 76.92; H, 8.07; N, 4.52.

General Procedure for Preparation of Sodium Salts (3a-g); A solution of the Schiff base (2.2 mmol) in THF (20 mL) was added to sodium hydride (120 mg, 5 mmol). The resulting mixture was stirred at rt for 1 h, filtered, and evaporated. The solid residue was washed with pentane (20 mL) and dried in vacuum. The salt was immediately used in the next step without further purification.

General Procedure of for Preparation of Salicylaldiminato Substituted Ni Complexes (4a-g); The Na salt of **2a** (0.59 g, 1.5 mmol) and *trans*-[NiCl(Ph)(PPh_3)$_2$] (1.0 g, 1.44 mmol) were in a Schlenk flask dissolved in benzene (20 mL) and stirred at rt for 6 h. After this time, the reaction mixture was filtered by cannula filtration, and the filtrate was concentrated *in vacuo* to ~5 mL. Pentane (30 mL) was added to the reaction. A yellow-orange solid precipitated from solution and was isolated by cannula filtration to yield 0.74 g (76 %) of **4a**. The other nickel complexes **4b-g** were prepared by the same procedure with similar yields.

4a (R = H, X = H, 76%): ^{1}H NMR (400 MHz, C_6D_6): δ 1.03 (d, 6H, J_{HH} = 6.8 Hz), 1.29 (d, 6H, J_{HH} = 6.8 Hz), 4.05 (sept., 2H, J_{HH} = 6.8 Hz), 6.31-7.69 (m, 27H), 7.93 (d, 1H, J_{HP} = 8.8 Hz); ^{13}C NMR (100 MHz, C_6D_6): δ 22.6, 25.5, 28.8, 117.4, 120.0, 122.8, 125.3, 126.2, 128.3, 128.6, 129.7, 130.5, 131.0, 131.5, 133.3, 133.8, 134.0, 134.4 (d, J_{CP} = 9.8 Hz), 137.4, 140.1, 149.4, 159.6, 165.2; ^{31}P NMR (162 MHz, C_6D_6): δ 25.9. Anal. Calcd for $C_{43}H_{42}NNiOP$: C, 76.35; H, 6.25; N, 2.07. Found: C, 76.20; H, 6.64; N, 1.89.

4b (R = *t*-Bu, X = H, 83%): ^{1}H NMR (400 MHz, C_6D_6): δ 0.93 (s, 9H), 1.08 (d, 6H, J_{HH} = 5.9 Hz), 1.22 (d, 6H, J_{HH} = 5.9 Hz), 4.28 (sept., 2H, J_{HH} = 5.9 Hz), 6.21-7.83 (m, 26H), 7.97 (d, 1H, J_{HP} = 9.1 Hz); ^{13}C (100 MHz, C_6D_6): δ 22.7, 25.5, 28.9,

29.8, 34.6, 113.9, 120.2, 121.0, 122.8, 125.0, 125.9, 128.3, 128.5, 129.1, 129.7, 131.5, 131.8, 132.2, 133.3, 134.9 (d, J_{CP} = 10.4 Hz), 137.0, 140.8, 141.9, 150.2, 166.1, 166.8; ^{31}P NMR (162 MHz, C_6D_6): δ 23.4. Anal. Calcd for $C_{47}H_{50}NNiOP$: C, 77.06; H, 6.88; N, 1.91. Found: C, 76.93; H, 6.81; N, 1.63.

4c (R = Ph, X = H, 89%): ^{1}H NMR (400 MHz, C_6D_6): δ 1.12 (d, 6H, J_{HH} = 6.6 Hz), 1.21 (d, 6H, J_{HH} = 6.6 Hz), 3.31 (s, 3H), 4.11 (sept., 2H, J_{HH} = 6.6 Hz), 3.29 (s, 3H), 6.18-7.80 (m, 31H), 7.99 (d, 1H, J_{HP} = 9.5 Hz); ^{13}C NMR (100 MHz, C_6D_6): δ 22.6, 25.6, 28.9, 114.4, 119.8, 121.1, 122.7, 125.0, 126.0, 127.4, 128.6, 129.4, 129.6, 131.7, 132.1, 134.0, 134.3, 134.4 (d, J_{CP} = 9.8 Hz), 135.3, 136.8, 137.8, 140.1, 140.7, 150.0, 163.7, 166.5; ^{31}P NMR (162 MHz, C_6D_6): δ 21.9. Anal. Calcd for $C_{49}H_{46}NNiOP$: C, 78.20; H, 6.16; N, 1.86. Found: C, 77.69; H, 6.36; N, 1.42.

4d (R = 9-phenanthrenyl, X = H, 75%): ^{1}H NMR (400 MHz, C_6D_6): δ 1.09 (d, 3H, J_{HH} = 7.0 Hz), 1.19 (d, 3H, J_{HH} = 7.0 Hz), 1.21 (d, 3H, J_{HH} = 7.0 Hz), 1.30 (d, 3H, J_{HH} = 7.0 Hz), 4.16 (sept., 2H, J_{HH} = 7.0 Hz), 6.13-7.85 (m, 32H), 8.13 (d, 1H, J_{HP} = 8.8 Hz), 8.32 (m, 2H); ^{13}C NMR (75 MHz, C_6D_6): δ 22.3, 22.8, 25.5, 25.7, 28.8, 28.9, 113.8, 119.9, 121.0, 122.0, 122.4, 122.6, 122.7, 124.8, 124.9, 125.0, 125.3, 125.4, 125.9 (d, J_{CP} = 11.4 Hz), 127.1, 127.3, 128.3, 128.5, 128.9 (d, J_{CP} = 1.5 Hz), 129.8, 130.7, 131.2 (d, J_{CP} = 43.9 Hz), 131.7, 132.4, 133.1, 133.9 (d, J_{CP} = 10.1 Hz), 134.2, 136.1, 137.1, 137.4, 140.4, 140.5, 146.5, 147.2, 150.0, 164.3, 166.5; ^{31}P NMR (162 MHz, C_6D_6): δ 25.4. Anal. Calcd for $C_{57}H_{50}NNiOP$: C, 80.10; H, 5.90; N, 1.64. Found: C, 80.06; H, 6.14; N, 1.25.

4e (R = 9-anthracenyl, X = H, 87%): ^{1}H NMR (400 MHz, C_6D_6): δ 1.14 (d, 6H, J_{HH} = 6.6 Hz), 1.18 (d, 6H, J_{HH} = 6.6 Hz), 4.17 (sept., 2H, J_{HH} = 6.6 Hz), 6.17-7.21 (m, 30H), 7.61 (m, 2H), 7.81 (m, 2H), 7.83 (s, 1H), 8.15 (d, 1H, J_{HP} = 11.3 Hz); ^{13}C NMR (75 MHz, C_6D_6): δ 22.6, 25.6, 28.9, 114.2, 119.9, 121.2, 122.8, 124.5, 124.7, 124.9, 125.8 (d, J_{CP} = 34.3 Hz), 127.3 (d, J_{CP} = 9.8 Hz), 128.4, 129.0 (d, J_{CP} = 1.6 Hz), 130.6, 130.8, 131.5, 131.8, 133.5 (d, J_{CP} = 10.1 Hz), 134.7, 136.6, 137.4, 138.3,

140.7, 145.2, 146.4, 150.1, 165.2, 166.7; ^{31}P NMR (162 MHz, C_6D_6): δ 22.7. Anal. Calcd for $C_{57}H_{50}NNiOP$: C, 80.10; H, 5.90; N, 1.64. Found: C, 79.77; H, 6.09; N, 1.49.

4f (R = H, X = OMe, 86%): ^{1}H NMR (400 MHz, C_6D_6): δ 1.08 (d, 6H, J_{HH} = 6.8 Hz), 1.30 (d, 6H, J_{HH} = 6.8 Hz), 3.31 (s, 3H), 4.09 (sept., 2H, J_{HH} = 6.8 Hz), 3.29 (s, 3H), 6.32-7.69 (m, 40H), 7.88 (d, 1H, J_{HP} = 9.3 Hz); ^{13}C NMR (100 MHz, C_6D_6): δ 22.6, 25.6, 28.8, 55.4, 113.1, 117.6, 121.2, 122.6, 123.7, 125.0, 125.2, 126.0, 129.41, 131.6, 132.0, 134.5 (d, J_{CP} = 9.8 Hz), 138.2, 140.6, 149.4, 150.4, 162, 165.7; ^{31}P NMR (162 MHz, C_6D_6): δ 24.6. Anal. Calcd for $C_{44}H_{44}NNiO_2P$: C, 74.59; H, 6.26; N, 1.98. Found: C, 74.01; H, 6.20; N, 1.65.

4g (R = H, X = NO_2, 89%): ^{1}H NMR (400 MHz, C_6D_6): δ 0.96 (d, 6H, J_{HH} = 7.0 Hz), 1.22 (d, 6H, J_{HH} = 7.0 Hz), 3.89 (sept., 2H, J_{HH} = 7.0 Hz), 5.91-7.90 (m, 30H), 8.06 (d, 1H, J_{HP} = 2.9 Hz); ^{13}C NMR (100 MHz, CD_2Cl_2): δ 22.2, 25.5, 28.7, 118.4, 121.4, 122.4, 122.6, 123.3, 125.2, 126.1, 128.0, 128.3, 129.9, 130.4, 130.9, 131.7, 134.2 (d, J_{CP} = 9.9 Hz), 137.5, 140.1, 149.0, 165.8, 170.5; ^{31}P NMR (162 MHz, C_6D_6): δ 25.5. Anal. Calcd for $C_{43}H_{41}N_2NiO_3P$: C, 71.39; H, 5.71; N, 3.87. Found: C, 71.13; H, 5.53; N, 3.61.

General Procedure for Polymerization of Ethylene by Ni Complexes 4 a-g; A 6 oz or 12 oz Fisher-Porter glass pressure bottle was charged with the appropriate amount of Ni complex under an atmosphere of nitrogen (for the polymerization at 14 atm an 200 mL autoclave was used). Toluene (80 mL) was then cannula transferred into the reactor, followed by a solution of cocatalyst, $Ni(COD)_2$ or $B(C_6F_5)_3$, in toluene (10 mL). The ethylene pressure was raised to the specified value and maintained for 40 min. Catalysts **4d** and **4e** were stirred for only 15 min since catalyst deactivation was faster for these catalysts. MeOH/HCl workup afforded the polyethylene, which was filtered and dried under vacuum.

X-ray analysis of **4e**

Figure 1. Crystal structure of **4e** (ORTEP diagram displaying 50% probability ellipsoids). Toluenes of crystallization are omitted.

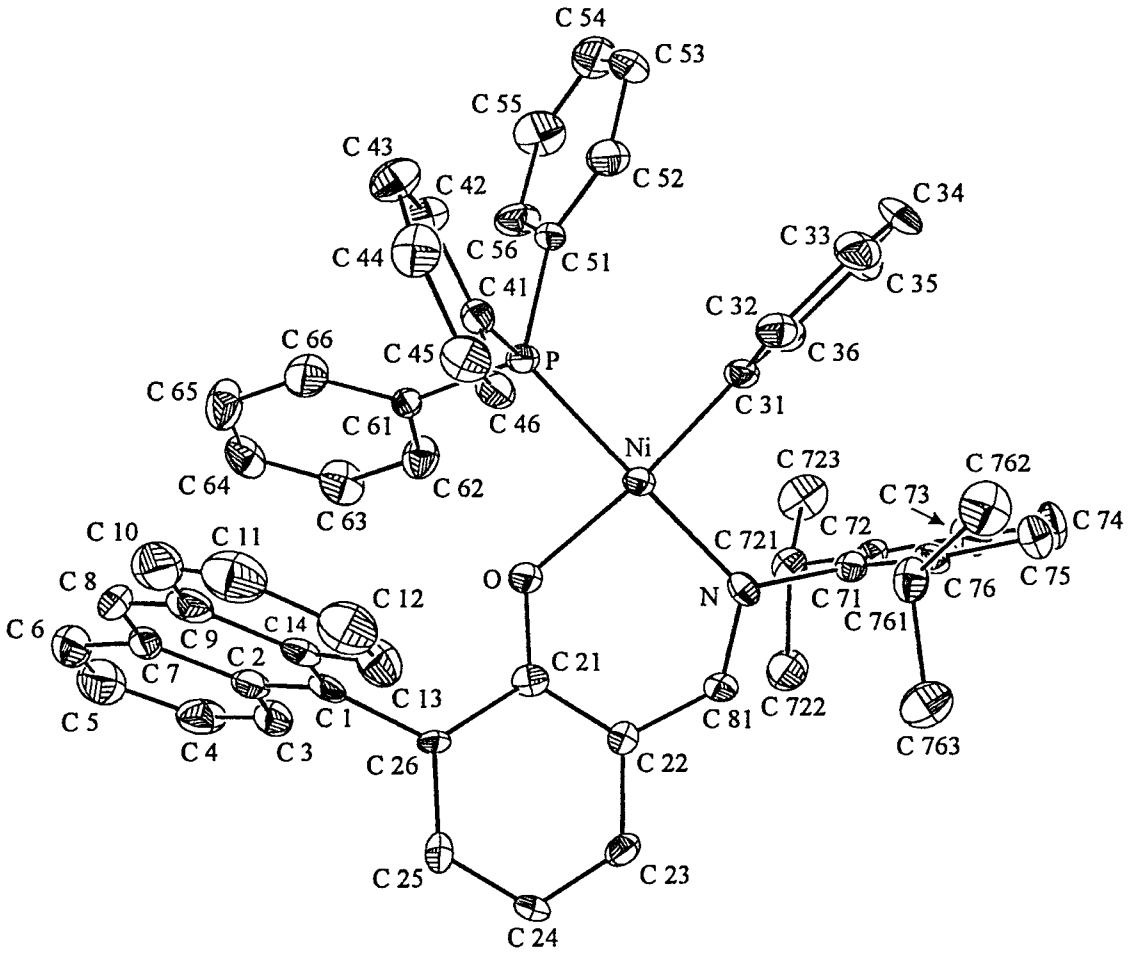

Table 1. Crystal data and structure refinement for **4e**.

Empirical formula	C71 H66 N Ni O P
Formula weight	1038.93
Crystallization solvent	Toluene
Crystal shape	Colunmar
Crystal size	0.48 x 0.10 x 0.07 mm
Crystal color	Yellow
========== Data Collection ==========	
Preliminary photos	None
Type of diffractometer	CAD-4
Wavelength	0.71073- MoK\alpha
Data collection temperature	160 K
Lattice determination from	25 reflections
Theta range for reflections used in lattice determination	8.6 to 10.9-
Unit cell dimensions	a = 20.397(9)- alpha = 90- b = 10.994(5)- beta = 107.85(4)- c = 26.028(13)- gamma = 90-
Volume	5556(5)-^3
Z	4
Crystal system and space group	Monoclinic P2(1)/c
Density (calculated)	1.242 g/cm^3
Absorption coefficient	0.424 mm^-1
F(000)	2200
Theta range for data collection	1.6 to 22.5-
Index ranges	0<=h<=-20, -11<=k<=11, -28<=l<=28
Data collection scan type	Omega scans
Reflections collected	16164
Independent reflections	7238 [R(merge) = 0.073 GOF(merge) = 1.08]
Absorption correction	None
Number of standards	3 reflections measured every 60 min.
Variation of standards	2.0%

Table 1 (cont).

========== Structure solution and Refinement ==========

Structure solution program	SHELXS-86 (Sheldrick, 1990)
Primary solution method	Direct methods
Secondary solution method	Difference Fourier map
Hydrogen placement	Geometric calculated sites
Structure refinement program	SHELXL-93 (Sheldrick, 1993)
Refinement method	Full matrix least-squares on F^2
Data / restraints / parameters	7234 / 0 / 731
Treatment of hydrogen atoms	Geometric calculated sites
Goodness-of-fit on F^2	1.149
Final R indices [I>2sigma(I)]	R1 = 0.0620, wR2 = 0.0896
R indices (all data)	R1 = 0.1126, wR2 = 0.1067
Max shift/error	1.623
Average shift/error	0.015
Extinction coefficient	0.00009(6)
Largest diff. peak and hole	0.330 and -0.298 e.-^-3

========== Special Notes ==========

The coordination geometry around the Nickel is very nearly square planar with the Nickel displaced approximately 0.03 Ang. from the plane of its ligands. The RMS deviation from the plane for the four ligand atoms (O, N, P and C31) is 0.16 Ang. The plane defined by the atoms of the anthracene moiety forms an approximately 80 deg. angle with plane defined by the atoms of salicylideneiminato fragment. The triphenyl phosphine ligand lies trans- to N and cis- to the anthracene fragment while the phenyl ligand lies trans- to O and cis- to the bis-isopropylphenyl group.

Refinement on F^2^ for ALL reflections except for 4 with very negative F^2^ or flagged by the user for potential systematic errors. Weighted R-factors wR and all goodnesses of fit S are based on F^2^, conventional R-factors R are based on F, with F set to zero for negative F^2^. The observed criterion of F^2^ > 2sigma(F^2^) is used only for calculating _R_factor_obs etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2^ are statistically about twice as large as those based on F, and R-factors based on ALL data will be even larger.

All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (-^2 x 10^3) for **4e**. U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
Ni	7098(1)	2298(1)	9321(1)	17(1)
P	8163(1)	2382(1)	9321(1)	18(1)
O	7439(2)	2346(3)	10091(1)	21(1)
N	6141(2)	2454(4)	9290(1)	17(1)
C(1)	8210(3)	2075(5)	11214(2)	21(1)
C(2)	8628(3)	3066(5)	11447(2)	22(1)
C(3)	8364(3)	4275(5)	11447(2)	28(1)
C(4)	8778(3)	5204(6)	11695(2)	36(2)
C(5)	9485(3)	5007(6)	11958(2)	44(2)
C(6)	9763(3)	3892(6)	11960(2)	34(2)
C(7)	9353(3)	2887(5)	11698(2)	25(1)
C(8)	9635(3)	1738(5)	11695(2)	30(2)
C(9)	9221(3)	738(5)	11470(2)	28(1)
C(10)	9502(3)	-461(6)	11493(2)	39(2)
C(11)	9082(3)	-1430(6)	11306(2)	42(2)
C(12)	8373(3)	-1266(6)	11087(2)	39(2)
C(13)	8080(3)	-150(5)	11039(2)	30(2)
C(14)	8491(3)	908(5)	11231(2)	21(1)
C(21)	7087(2)	2359(4)	10428(2)	16(1)
C(22)	6361(2)	2504(4)	10268(2)	14(1)
C(23)	6009(3)	2545(5)	10655(2)	22(1)
C(24)	6356(2)	2444(5)	11195(2)	26(1)
C(25)	7065(2)	2299(5)	11355(2)	25(1)
C(26)	7444(2)	2248(5)	11002(2)	18(1)
C(31)	6883(3)	1899(4)	8580(2)	19(1)
C(32)	6904(3)	676(5)	8437(2)	23(1)
C(33)	6727(3)	313(5)	7905(2)	30(2)
C(34)	6528(3)	1172(5)	7496(2)	36(2)
C(35)	6509(3)	2386(6)	7631(2)	30(2)
C(36)	6688(2)	2740(5)	8164(2)	23(1)
C(41)	8583(3)	903(4)	9475(2)	19(1)
C(42)	9173(3)	583(5)	9343(2)	26(1)
C(43)	9446(3)	-568(5)	9450(2)	30(2)
C(44)	9126(3)	-1433(5)	9686(2)	31(2)
C(45)	8556(3)	-1113(5)	9831(2)	30(2)
C(46)	8281(3)	44(5)	9733(2)	24(1)
C(51)	8392(2)	2861(5)	8725(2)	20(1)
C(52)	8333(3)	2046(5)	8303(2)	29(2)
C(53)	8502(3)	2396(6)	7847(2)	36(2)
C(54)	8718(3)	3570(6)	7803(2)	43(2)
C(55)	8777(3)	4379(6)	8216(2)	43(2)
C(56)	8620(3)	4023(5)	8675(2)	30(2)
C(61)	8663(3)	3428(4)	9843(2)	16(1)
C(62)	8377(3)	4530(5)	9915(2)	29(2)
C(63)	8746(3)	5379(5)	10272(2)	34(2)
C(64)	9416(3)	5139(5)	10572(2)	32(2)
C(65)	9709(3)	4046(5)	10516(2)	39(2)
C(66)	9328(3)	3208(5)	10145(2)	33(2)
C(71)	5578(2)	2527(4)	8790(2)	17(1)
C(72)	5383(3)	3655(4)	8543(2)	19(1)
C(721)	5729(3)	4823(4)	8800(2)	24(1)
C(722)	5335(3)	5409(5)	9155(2)	35(2)
C(723)	5797(3)	5763(5)	8380(2)	38(2)
C(74)	4506(3)	2621(5)	7836(2)	31(1)
C(75)	4711(3)	1521(5)	8086(2)	29(2)

C(76)	5249(3)	1445(4)	8567(2)	19(1)
C(73)	4835(3)	3677(5)	8068(2)	29(2)
C(761)	5442(3)	236(4)	8855(2)	24(1)
C(762)	5373(3)	-847(5)	8474(2)	36(2)
C(763)	5019(3)	6(5)	9243(2)	34(2)
C(81)	5950(2)	2580(4)	9716(2)	15(1)
C(101)	3457(4)	4096(7)	8850(3)	75(2)
C(102)	3381(3)	3351(6)	9314(3)	46(2)
C(103)	3584(3)	2151(7)	9359(3)	61(2)
C(104)	3514(4)	1450(7)	9780(4)	73(3)
C(105)	3240(4)	1924(7)	10156(3)	59(2)
C(106)	3031(3)	3110(6)	10110(3)	47(2)
C(107)	3104(3)	3821(6)	9692(3)	44(2)
C(201)	2822(4)	503(7)	7859(3)	75(2)
C(202)	2493(3)	1461(6)	7465(3)	46(2)
C(203)	2540(4)	1466(7)	6949(3)	58(2)
C(204)	2231(4)	2336(7)	6576(3)	66(2)
C(205)	1861(4)	3255(6)	6725(3)	60(2)
C(206)	1798(4)	3273(6)	7236(3)	58(2)
C(207)	2120(3)	2391(7)	7599(3)	55(2)

Table 3. Selected bond lengths [Å] and angles [deg] for **4e**.

Ni-C(31)	1.895(5)
Ni-O	1.910(3)
Ni-N	1.937(4)
Ni-P	2.172(2)
C(31)-Ni-O	166.2(2)
C(31)-Ni-N	93.5(2)
O-Ni-N	94.5(2)
C(31)-Ni-P	86.0(2)
O-Ni-P	87.51(11)
N-Ni-P	172.16(12)

Symmetry transformations used to generate equivalent atoms:

Table 4. Bond lengths [Å] and angles [deg] for **4e**.

Ni-C(31)	1.895(5)
Ni-O	1.910(3)
Ni-N	1.937(4)
Ni-P	2.172(2)
P-C(41)	1.824(5)
P-C(61)	1.832(5)
P-C(51)	1.830(5)
O-C(21)	1.293(5)
N-C(81)	1.291(5)
N-C(71)	1.451(5)
C(1)-C(2)	1.401(7)
C(1)-C(14)	1.401(7)
C(1)-C(26)	1.501(6)
C(2)-C(7)	1.436(7)
C(2)-C(3)	1.434(7)
C(3)-C(4)	1.357(7)
C(3)-H(3A)	0.94(5)
C(4)-C(5)	1.409(8)
C(4)-H(4A)	0.90(5)

C(5)-C(6)	1.350(8)
C(5)-H(5A)	0.91(5)
C(6)-C(7)	1.427(7)
C(6)-H(6A)	0.98(5)
C(7)-C(8)	1.389(7)
C(8)-C(9)	1.401(7)
C(8)-H(8A)	0.98(5)
C(9)-C(10)	1.431(8)
C(9)-C(14)	1.437(7)
C(10)-C(11)	1.361(8)
C(10)-H(10A)	1.04(5)
C(11)-C(12)	1.394(8)
C(11)-H(11A)	0.96(5)
C(12)-C(13)	1.353(7)
C(12)-H(12A)	0.92(5)
C(13)-C(14)	1.431(7)
C(13)-H(13A)	0.97(5)
C(21)-C(22)	1.418(6)
C(21)-C(26)	1.454(6)
C(22)-C(23)	1.405(6)
C(22)-C(81)	1.427(6)
C(23)-C(24)	1.370(6)
C(23)-H(23A)	0.99(4)
C(24)-C(25)	1.387(6)
C(24)-H(24A)	0.92(4)
C(25)-C(26)	1.370(6)
C(25)-H(25A)	0.97(4)
C(31)-C(36)	1.386(6)
C(31)-C(32)	1.399(7)
C(32)-C(33)	1.380(7)
C(32)-H(32A)	0.95(4)
C(33)-C(34)	1.387(7)
C(33)-H(33A)	1.00(5)
C(34)-C(35)	1.384(7)
C(34)-H(34A)	0.94(5)
C(35)-C(36)	1.376(6)
C(35)-H(35A)	0.96(5)
C(36)-H(36A)	0.93(5)
C(41)-C(42)	1.395(7)
C(41)-C(46)	1.404(7)
C(42)-C(43)	1.376(7)
C(42)-H(42A)	0.99(5)
C(43)-C(44)	1.396(7)
C(43)-H(43A)	0.98(5)
C(44)-C(45)	1.374(7)
C(44)-H(44A)	0.97(5)
C(45)-C(46)	1.382(7)
C(45)-H(45A)	0.93(5)
C(46)-H(46A)	1.07(5)
C(51)-C(56)	1.380(7)
C(51)-C(52)	1.395(6)
C(52)-C(53)	1.389(7)
C(52)-H(52A)	0.88(5)
C(53)-C(54)	1.379(8)
C(53)-H(53A)	0.88(5)
C(54)-C(55)	1.372(8)
C(54)-H(54A)	0.96(5)
C(55)-C(56)	1.384(7)
C(55)-H(55A)	0.94(5)
C(56)-H(56A)	1.04(5)
C(61)-C(66)	1.366(7)
C(61)-C(62)	1.381(7)
C(62)-C(63)	1.369(7)
C(62)-H(62A)	0.87(5)

C(63)-C(64)	1.377(7)
C(63)-H(63A)	0.83(5)
C(64)-C(65)	1.369(7)
C(64)-H(64A)	0.94(5)
C(65)-C(66)	1.388(7)
C(65)-H(65A)	0.97(5)
C(66)-H(66A)	0.90(5)
C(71)-C(72)	1.397(7)
C(71)-C(76)	1.401(7)
C(72)-C(73)	1.390(7)
C(72)-C(721)	1.518(7)
C(721)-C(722)	1.539(7)
C(721)-C(723)	1.541(7)
C(721)-H(72A)	1.03(5)
C(722)-H(72B)	0.96
C(722)-H(72C)	0.96
C(722)-H(72D)	0.96
C(723)-H(72E)	0.96
C(723)-H(72F)	0.96
C(723)-H(72G)	0.96
C(74)-C(73)	1.383(7)
C(74)-C(75)	1.376(7)
C(74)-H(74A)	0.97(4)
C(75)-C(76)	1.390(7)
C(75)-H(75A)	0.97(5)
C(76)-C(761)	1.519(7)
C(73)-H(73A)	0.86(5)
C(761)-C(762)	1.528(7)
C(761)-C(763)	1.537(7)
C(761)-H(76A)	0.99(5)
C(762)-H(76B)	0.96
C(762)-H(76C)	0.96
C(762)-H(76D)	0.96
C(763)-H(76E)	0.96
C(763)-H(76F)	0.96
C(763)-H(76G)	0.96
C(81)-H(81A)	0.97(4)
C(101)-C(102)	1.507(8)
C(101)-H(10B)	0.96
C(101)-H(10C)	0.96
C(101)-H(10D)	0.96
C(102)-C(107)	1.377(8)
C(102)-C(103)	1.376(8)
C(103)-C(104)	1.382(10)
C(103)-H(10E)	0.91(6)
C(104)-C(105)	1.370(10)
C(104)-H(10F)	0.85(6)
C(105)-C(106)	1.366(9)
C(105)-H(10G)	1.02(6)
C(106)-C(107)	1.386(8)
C(106)-H(10H)	0.92(5)
C(107)-H(10I)	0.90(5)
C(201)-C(202)	1.480(8)
C(201)-H(20A)	0.96
C(201)-H(20B)	0.96
C(201)-H(20C)	0.96
C(202)-C(207)	1.383(8)
C(202)-C(203)	1.374(8)
C(203)-C(204)	1.371(9)
C(203)-H(20D)	0.93(6)
C(204)-C(205)	1.385(9)
C(204)-H(20E)	1.01(6)
C(205)-C(206)	1.372(9)
C(205)-H(20F)	0.87(6)

C(206)-C(207)	1.373(8)
C(206)-H(20G)	1.08(6)
C(207)-H(20H)	0.89(5)
C(31)-Ni-O	166.2(2)
C(31)-Ni-N	93.5(2)
O-Ni-N	94.5(2)
C(31)-Ni-P	86.0(2)
O-Ni-P	87.51(11)
N-Ni-P	172.16(12)
C(41)-P-C(61)	106.2(2)
C(41)-P-C(51)	102.6(2)
C(61)-P-C(51)	102.6(2)
C(41)-P-Ni	111.4(2)
C(61)-P-Ni	110.9(2)
C(51)-P-Ni	121.8(2)
C(21)-O-Ni	127.8(3)
C(81)-N-C(71)	113.7(4)
C(81)-N-Ni	122.6(3)
C(71)-N-Ni	123.6(3)
C(2)-C(1)-C(14)	120.6(5)
C(2)-C(1)-C(26)	119.1(5)
C(14)-C(1)-C(26)	119.9(5)
C(1)-C(2)-C(7)	119.7(5)
C(1)-C(2)-C(3)	122.8(5)
C(7)-C(2)-C(3)	117.5(5)
C(4)-C(3)-C(2)	121.2(6)
C(4)-C(3)-H(3A)	119.4(4)
C(2)-C(3)-H(3A)	119.4(3)
C(3)-C(4)-C(5)	120.7(6)
C(3)-C(4)-H(4A)	119.6(4)
C(5)-C(4)-H(4A)	119.6(4)
C(6)-C(5)-C(4)	120.5(6)
C(6)-C(5)-H(5A)	119.7(4)
C(4)-C(5)-H(5A)	119.7(4)
C(5)-C(6)-C(7)	121.1(6)
C(5)-C(6)-H(6A)	119.5(4)
C(7)-C(6)-H(6A)	119.5(4)
C(8)-C(7)-C(6)	121.6(5)
C(8)-C(7)-C(2)	119.5(5)
C(6)-C(7)-C(2)	118.9(5)
C(7)-C(8)-C(9)	121.2(5)
C(7)-C(8)-H(8A)	119.4(3)
C(9)-C(8)-H(8A)	119.4(3)
C(8)-C(9)-C(10)	121.5(6)
C(8)-C(9)-C(14)	119.4(5)
C(10)-C(9)-C(14)	119.1(6)
C(11)-C(10)-C(9)	120.4(6)
C(11)-C(10)-H(10A)	119.8(4)
C(9)-C(10)-H(10A)	119.8(4)
C(10)-C(11)-C(12)	120.3(6)
C(10)-C(11)-H(11A)	119.8(4)
C(12)-C(11)-H(11A)	119.8(4)
C(13)-C(12)-C(11)	121.9(6)
C(13)-C(12)-H(12A)	119.1(4)
C(11)-C(12)-H(12A)	119.0(4)
C(12)-C(13)-C(14)	120.6(6)
C(12)-C(13)-H(13A)	119.7(4)
C(14)-C(13)-H(13A)	119.7(3)
C(1)-C(14)-C(13)	122.8(5)
C(1)-C(14)-C(9)	119.5(5)
C(13)-C(14)-C(9)	117.6(5)
O-C(21)-C(22)	123.3(4)
O-C(21)-C(26)	119.3(4)

C(22)-C(21)-C(26)	117.4(4)
C(23)-C(22)-C(21)	120.7(4)
C(23)-C(22)-C(81)	116.6(4)
C(21)-C(22)-C(81)	122.7(4)
C(24)-C(23)-C(22)	121.0(5)
C(24)-C(23)-H(23A)	119.5(3)
C(22)-C(23)-H(23A)	119.5(3)
C(23)-C(24)-C(25)	118.8(5)
C(23)-C(24)-H(24A)	120.6(3)
C(25)-C(24)-H(24A)	120.6(3)
C(26)-C(25)-C(24)	123.6(5)
C(26)-C(25)-H(25A)	118.2(3)
C(24)-C(25)-H(25A)	118.2(3)
C(25)-C(26)-C(21)	118.6(4)
C(25)-C(26)-C(1)	119.8(4)
C(21)-C(26)-C(1)	121.7(4)
C(36)-C(31)-C(32)	117.3(5)
C(36)-C(31)-Ni	124.3(4)
C(32)-C(31)-Ni	118.4(4)
C(33)-C(32)-C(31)	121.6(5)
C(33)-C(32)-H(32A)	119.2(3)
C(31)-C(32)-H(32A)	119.2(3)
C(32)-C(33)-C(34)	119.9(5)
C(32)-C(33)-H(33A)	120.0(3)
C(34)-C(33)-H(33A)	120.1(3)
C(35)-C(34)-C(33)	119.1(5)
C(35)-C(34)-H(34A)	120.5(3)
C(33)-C(34)-H(34A)	120.5(3)
C(36)-C(35)-C(34)	120.6(5)
C(36)-C(35)-H(35A)	119.7(3)
C(34)-C(35)-H(35A)	119.7(3)
C(35)-C(36)-C(31)	121.5(5)
C(35)-C(36)-H(36A)	119.2(3)
C(31)-C(36)-H(36A)	119.2(3)
C(42)-C(41)-C(46)	119.0(5)
C(42)-C(41)-P	123.6(4)
C(46)-C(41)-P	117.4(4)
C(43)-C(42)-C(41)	120.6(5)
C(43)-C(42)-H(42A)	119.7(3)
C(41)-C(42)-H(42A)	119.7(3)
C(42)-C(43)-C(44)	120.0(5)
C(42)-C(43)-H(43A)	120.0(3)
C(44)-C(43)-H(43A)	120.0(3)
C(45)-C(44)-C(43)	119.7(5)
C(45)-C(44)-H(44A)	120.2(3)
C(43)-C(44)-H(44A)	120.1(3)
C(44)-C(45)-C(46)	120.9(5)
C(44)-C(45)-H(45A)	119.5(3)
C(46)-C(45)-H(45A)	119.6(3)
C(45)-C(46)-C(41)	119.8(5)
C(45)-C(46)-H(46A)	120.1(3)
C(41)-C(46)-H(46A)	120.1(3)
C(56)-C(51)-C(52)	118.0(5)
C(56)-C(51)-P	121.9(4)
C(52)-C(51)-P	120.1(4)
C(53)-C(52)-C(51)	120.8(5)
C(53)-C(52)-H(52A)	119.6(4)
C(51)-C(52)-H(52A)	119.6(3)
C(54)-C(53)-C(52)	120.0(6)
C(54)-C(53)-H(53A)	120.0(3)
C(52)-C(53)-H(53A)	120.0(4)
C(55)-C(54)-C(53)	119.7(6)
C(55)-C(54)-H(54A)	120.1(4)
C(53)-C(54)-H(54A)	120.1(3)

C(54)-C(55)-C(56)	120.3(6)
C(54)-C(55)-H(55A)	119.9(4)
C(56)-C(55)-H(55A)	119.9(4)
C(51)-C(56)-C(55)	121.2(6)
C(51)-C(56)-H(56A)	119.4(3)
C(55)-C(56)-H(56A)	119.4(4)
C(66)-C(61)-C(62)	117.6(5)
C(66)-C(61)-P	123.0(4)
C(62)-C(61)-P	119.2(4)
C(63)-C(62)-C(61)	121.5(5)
C(63)-C(62)-H(62A)	119.2(3)
C(61)-C(62)-H(62A)	119.2(3)
C(62)-C(63)-C(64)	119.9(5)
C(62)-C(63)-H(63A)	120.1(4)
C(64)-C(63)-H(63A)	120.1(3)
C(65)-C(64)-C(63)	119.9(5)
C(65)-C(64)-H(64A)	120.1(3)
C(63)-C(64)-H(64A)	120.1(3)
C(64)-C(65)-C(66)	119.1(6)
C(64)-C(65)-H(65A)	120.4(3)
C(66)-C(65)-H(65A)	120.4(3)
C(61)-C(66)-C(65)	121.9(5)
C(61)-C(66)-H(66A)	119.1(3)
C(65)-C(66)-H(66A)	119.1(4)
C(72)-C(71)-C(76)	122.0(4)
C(72)-C(71)-N	119.8(4)
C(76)-C(71)-N	118.2(4)
C(73)-C(72)-C(71)	117.6(5)
C(73)-C(72)-C(721)	121.0(5)
C(71)-C(72)-C(721)	121.4(4)
C(72)-C(721)-C(722)	111.2(4)
C(72)-C(721)-C(723)	112.7(4)
C(722)-C(721)-C(723)	108.8(4)
C(72)-C(721)-H(72A)	108.0(3)
C(722)-C(721)-H(72A)	108.0(3)
C(723)-C(721)-H(72A)	108.0(3)
C(721)-C(722)-H(72B)	109.5(3)
C(721)-C(722)-H(72C)	109.5(3)
H(72B)-C(722)-H(72C)	109.5
C(721)-C(722)-H(72D)	109.5(3)
H(72B)-C(722)-H(72D)	109.5
H(72C)-C(722)-H(72D)	109.5
C(721)-C(723)-H(72E)	109.5(3)
C(721)-C(723)-H(72F)	109.5(3)
H(72E)-C(723)-H(72F)	109.5
C(721)-C(723)-H(72G)	109.5(3)
H(72E)-C(723)-H(72G)	109.5
H(72F)-C(723)-H(72G)	109.5
C(73)-C(74)-C(75)	119.7(5)
C(73)-C(74)-H(74A)	120.1(3)
C(75)-C(74)-H(74A)	120.2(3)
C(74)-C(75)-C(76)	121.3(5)
C(74)-C(75)-H(75A)	119.4(3)
C(76)-C(75)-H(75A)	119.4(3)
C(75)-C(76)-C(71)	117.9(5)
C(75)-C(76)-C(761)	120.7(5)
C(71)-C(76)-C(761)	121.3(4)
C(74)-C(73)-C(72)	121.5(5)
C(74)-C(73)-H(73A)	119.2(3)
C(72)-C(73)-H(73A)	119.2(3)
C(76)-C(761)-C(762)	113.8(4)
C(76)-C(761)-C(763)	110.8(4)
C(762)-C(761)-C(763)	109.9(4)
C(76)-C(761)-H(76A)	107.4(3)

C(762)-C(761)-H(76A)	107.4(3)
C(763)-C(761)-H(76A)	107.4(3)
C(761)-C(762)-H(76B)	109.5(3)
C(761)-C(762)-H(76C)	109.5(3)
H(76B)-C(762)-H(76C)	109.5
C(761)-C(762)-H(76D)	109.5(3)
H(76B)-C(762)-H(76D)	109.5
H(76C)-C(762)-H(76D)	109.5
C(761)-C(763)-H(76E)	109.5(3)
C(761)-C(763)-H(76F)	109.5(3)
H(76E)-C(763)-H(76F)	109.5
C(761)-C(763)-H(76G)	109.5(3)
H(76E)-C(763)-H(76G)	109.5
H(76F)-C(763)-H(76G)	109.5
N-C(81)-C(22)	128.4(4)
N-C(81)-H(81A)	115.8(3)
C(22)-C(81)-H(81A)	115.8(3)
C(102)-C(101)-H(10B)	109.5(4)
C(102)-C(101)-H(10C)	109.5(4)
H(10B)-C(101)-H(10C)	109.5
C(102)-C(101)-H(10D)	109.5(4)
H(10B)-C(101)-H(10D)	109.47(6)
H(10C)-C(101)-H(10D)	109.5
C(107)-C(102)-C(103)	118.5(7)
C(107)-C(102)-C(101)	122.3(6)
C(103)-C(102)-C(101)	119.2(7)
C(104)-C(103)-C(102)	120.1(8)
C(104)-C(103)-H(10E)	120.0(5)
C(102)-C(103)-H(10E)	120.0(5)
C(105)-C(104)-C(103)	121.2(7)
C(105)-C(104)-H(10F)	119.4(5)
C(103)-C(104)-H(10F)	119.4(5)
C(104)-C(105)-C(106)	119.1(8)
C(104)-C(105)-H(10G)	120.5(5)
C(106)-C(105)-H(10G)	120.5(5)
C(105)-C(106)-C(107)	120.1(7)
C(105)-C(106)-H(10H)	119.9(5)
C(107)-C(106)-H(10H)	120.0(4)
C(102)-C(107)-C(106)	121.1(6)
C(102)-C(107)-H(10I)	119.5(4)
C(106)-C(107)-H(10I)	119.5(4)
C(202)-C(201)-H(20A)	109.5(4)
C(202)-C(201)-H(20B)	109.5(4)
H(20A)-C(201)-H(20B)	109.4
C(202)-C(201)-H(20C)	109.5(4)
H(20A)-C(201)-H(20C)	109.4
H(20B)-C(201)-H(20C)	109.4
C(207)-C(202)-C(203)	116.9(6)
C(207)-C(202)-C(201)	121.2(6)
C(203)-C(202)-C(201)	121.9(6)
C(202)-C(203)-C(204)	122.6(7)
C(202)-C(203)-H(20D)	118.7(4)
C(204)-C(203)-H(20D)	118.7(5)
C(205)-C(204)-C(203)	118.9(7)
C(205)-C(204)-H(20E)	120.6(4)
C(203)-C(204)-H(20E)	120.6(5)
C(206)-C(205)-C(204)	120.1(7)
C(206)-C(205)-H(20F)	119.9(4)
C(204)-C(205)-H(20F)	119.9(4)
C(205)-C(206)-C(207)	119.3(7)
C(205)-C(206)-H(20G)	120.3(4)
C(207)-C(206)-H(20G)	120.3(4)
C(206)-C(207)-C(202)	122.2(6)
C(206)-C(207)-H(20H)	118.9(4)

C(202)-C(207)-H(20H)	118.9(4)

Symmetry transformations used to generate equivalent atoms:

Table 5. Anisotropic displacement parameters (-^2 x 10^3) for **4e**. The anisotropic displacement factor exponent takes the form: -2 pi^2 [h^2 a*^2 U11 + ... + 2 h k a* b* U12]

	U11	U22	U33	U23	U13	U12
Ni	17(1)	21(1)	14(1)	1(1)	6(1)	2(1)
P	17(1)	21(1)	17(1)	1(1)	7(1)	2(1)
O	17(2)	31(2)	16(2)	2(2)	6(2)	3(2)
N	22(2)	13(2)	14(2)	-1(2)	3(2)	0(2)
C(1)	26(3)	30(4)	9(3)	4(2)	8(2)	-3(3)
C(2)	26(3)	28(4)	15(3)	1(2)	10(3)	2(3)
C(3)	29(4)	34(4)	20(3)	4(3)	8(3)	-4(3)
C(4)	43(4)	37(4)	35(4)	-13(3)	22(3)	-6(3)
C(5)	46(5)	52(5)	36(4)	-12(3)	14(4)	-21(4)
C(6)	28(4)	47(4)	27(4)	-1(3)	7(3)	-10(3)
C(7)	19(3)	41(4)	15(3)	0(3)	6(3)	-12(3)
C(8)	14(3)	55(5)	23(3)	5(3)	6(3)	3(3)
C(9)	35(4)	36(4)	17(3)	10(3)	13(3)	11(3)
C(10)	37(4)	51(5)	30(4)	14(3)	11(3)	13(4)
C(11)	57(5)	37(4)	38(4)	5(3)	22(4)	6(4)
C(12)	49(5)	33(4)	35(4)	2(3)	16(3)	1(3)
C(13)	35(4)	29(4)	24(3)	2(3)	6(3)	4(3)
C(14)	26(3)	28(4)	12(3)	3(2)	10(3)	0(3)
C(21)	21(3)	10(3)	19(3)	6(2)	9(2)	-1(2)
C(22)	15(3)	9(3)	19(3)	-2(2)	4(2)	1(2)
C(23)	16(3)	26(3)	26(3)	-7(3)	10(2)	-2(3)
C(24)	22(3)	47(4)	13(3)	-3(3)	9(2)	-1(3)
C(25)	16(3)	37(4)	19(3)	0(3)	-1(2)	-3(3)
C(26)	15(3)	27(3)	14(3)	4(2)	9(2)	-1(3)
C(31)	18(3)	25(3)	15(3)	3(2)	6(2)	5(2)
C(32)	25(3)	23(3)	24(3)	0(3)	11(3)	-1(3)
C(33)	36(4)	26(4)	33(4)	-13(3)	17(3)	-6(3)
C(34)	42(4)	47(4)	19(3)	-16(3)	11(3)	-5(3)
C(35)	29(3)	44(4)	13(3)	10(3)	2(2)	4(3)
C(36)	20(3)	25(3)	23(3)	2(3)	8(2)	6(3)
C(41)	20(3)	19(3)	17(3)	-1(2)	4(3)	2(2)
C(42)	24(3)	31(4)	25(3)	-1(3)	10(3)	2(3)
C(43)	26(4)	26(4)	41(4)	0(3)	15(3)	5(3)
C(44)	32(4)	21(3)	38(4)	0(3)	7(3)	4(3)
C(45)	37(4)	26(4)	28(3)	6(3)	12(3)	0(3)
C(46)	26(3)	28(4)	17(3)	-1(3)	6(3)	5(3)
C(51)	17(3)	29(3)	15(3)	-3(2)	7(2)	-4(3)
C(52)	29(3)	32(4)	28(4)	-1(3)	13(3)	1(3)
C(53)	31(3)	63(5)	16(3)	-1(3)	10(3)	2(4)
C(54)	34(4)	71(5)	26(4)	13(4)	14(3)	-3(4)
C(55)	50(4)	49(4)	39(4)	13(3)	25(4)	-19(4)
C(56)	32(4)	32(4)	33(4)	1(3)	21(3)	-6(3)
C(61)	16(3)	18(3)	16(3)	1(2)	5(2)	-4(2)
C(62)	24(4)	26(4)	33(4)	-1(3)	4(3)	5(3)
C(63)	42(4)	20(3)	37(4)	-15(3)	10(3)	-3(3)
C(64)	40(4)	28(4)	25(4)	-10(3)	7(3)	-12(3)
C(65)	27(4)	39(4)	44(4)	-19(3)	-1(3)	6(3)
C(66)	30(4)	29(4)	38(4)	-10(3)	10(3)	6(3)
C(71)	15(3)	19(3)	17(3)	2(2)	5(2)	4(3)

C(72)	16(3)	19(3)	23(3)	-2(3)	9(3)	0(2)
C(721)	20(3)	23(3)	26(3)	2(3)	5(3)	8(3)
C(722)	32(4)	35(4)	41(4)	-13(3)	14(3)	-3(3)
C(723)	41(4)	24(3)	51(4)	10(3)	17(3)	3(3)
C(74)	26(3)	40(4)	20(3)	-1(3)	-6(3)	-2(3)
C(75)	28(4)	32(4)	23(3)	-6(3)	2(3)	-4(3)
C(76)	24(3)	22(3)	12(3)	1(2)	7(3)	-2(3)
C(73)	30(4)	31(4)	22(3)	9(3)	4(3)	10(3)
C(761)	21(3)	20(3)	28(3)	-3(3)	2(3)	-1(3)
C(762)	41(4)	24(3)	43(4)	-1(3)	12(3)	-2(3)
C(763)	36(4)	27(3)	42(4)	7(3)	17(3)	-5(3)
C(81)	17(3)	12(3)	18(3)	-2(2)	8(2)	5(2)
C(101)	55(5)	80(6)	99(7)	-4(5)	37(5)	-12(4)
C(102)	24(4)	40(4)	69(5)	-4(4)	8(4)	-7(3)
C(103)	40(4)	57(6)	72(6)	-13(5)	-7(4)	14(4)
C(104)	47(5)	39(5)	102(7)	-1(5)	-22(5)	13(4)
C(105)	37(5)	51(6)	73(6)	16(4)	-9(4)	-7(4)
C(106)	29(4)	63(5)	47(5)	8(4)	8(3)	0(3)
C(107)	34(4)	33(4)	60(5)	-5(4)	5(4)	-1(3)
C(201)	61(6)	85(6)	79(6)	26(5)	19(5)	11(5)
C(202)	36(4)	46(4)	57(5)	11(4)	16(4)	5(3)
C(203)	66(5)	54(5)	65(6)	-6(4)	39(5)	-1(4)
C(204)	95(6)	53(5)	51(5)	1(4)	25(4)	-12(5)
C(205)	68(6)	39(5)	58(6)	16(4)	-1(4)	6(4)
C(206)	56(5)	54(5)	63(6)	-5(4)	15(4)	16(4)
C(207)	55(5)	65(5)	42(4)	-6(4)	12(4)	9(4)

Table 6. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (-^2 x 10^3) for **4e**.

	x	y	z	U(eq)
H(3A)	7893(25)	4427(9)	11270(9)	30
H(4A)	8601(11)	5951(44)	11692(2)	40
H(5A)	9756(16)	5637(38)	12127(10)	49
H(6A)	10256(26)	3770(9)	12145(10)	38
H(8A)	10134(26)	1624(8)	11854(8)	33
H(10A)	10033(27)	-592(8)	11655(8)	43
H(11A)	9276(11)	-2234(45)	11324(3)	46
H(12A)	8098(16)	-1932(37)	10972(7)	42
H(13A)	7588(25)	-67(7)	10873(9)	33
H(23A)	5504(22)	2647(6)	10536(6)	24
H(24A)	6122(12)	2473(5)	11446(12)	29
H(25A)	7307(11)	2230(6)	11738(18)	28
H(32A)	7043(7)	81(29)	8714(13)	25
H(33A)	6743(3)	-568(43)	7813(5)	33
H(34A)	6407(7)	933(14)	7131(20)	39
H(35A)	6370(7)	2987(31)	7352(14)	33
H(36A)	6679(2)	3559(42)	8246(5)	25
H(42A)	9398(11)	1192(30)	9170(9)	29
H(43A)	9866(22)	-781(12)	9361(5)	33
H(44A)	9306(9)	-2257(42)	9746(4)	34
H(45A)	8352(11)	-1684(30)	9999(9)	33
H(46A)	7839(19)	284(11)	9852(5)	26
H(52A)	8188(9)	1304(42)	8326(2)	32
H(53A)	8471(3)	1871(31)	7586(15)	39
H(54A)	8827(7)	3820(15)	7484(18)	47
H(55A)	8926(9)	5182(46)	8187(3)	48
H(56A)	8676(4)	4645(29)	8988(15)	33
H(62A)	7952(25)	4687(10)	9729(11)	32

H(63A)	8566(12)	6036(41)	10307(3)	37
H(64A)	9672(14)	5724(31)	10816(13)	35
H(65A)	10179(25)	3860(11)	10734(12)	43
H(66A)	9525(11)	2498(41)	10103(3)	36
H(72A)	6218(23)	4609(11)	9045(11)	26
H(72B)	5329(13)	4861(12)	9441(7)	39
H(72C)	4871(5)	5580(25)	8939(3)	39
H(72D)	5559(9)	6153(14)	9306(10)	39
H(72E)	5970(15)	5369(7)	8119(7)	42
H(72F)	6110(12)	6393(15)	8558(3)	42
H(72G)	5354(4)	6111(20)	8202(9)	42
H(74A)	4134(18)	2656(5)	7501(16)	34
H(75A)	4476(12)	787(38)	7924(8)	32
H(73A)	4698(8)	4362(40)	7914(9)	32
H(76A)	5933(23)	289(5)	9076(11)	27
H(76B)	5609(13)	-673(12)	8215(8)	40
H(76C)	4895(3)	-993(18)	8289(9)	40
H(76D)	5571(14)	-1556(8)	8678(3)	40
H(76E)	5082(12)	671(14)	9492(8)	37
H(76F)	5170(11)	-735(15)	9438(9)	37
H(76G)	4540(3)	-61(27)	9040(2)	37
H(81A)	5464(22)	2749(8)	9653(3)	17
H(10B)	3553(21)	4926(10)	8962(6)	82
H(10C)	3037(8)	4058(31)	8554(7)	82
H(10D)	3829(14)	3778(24)	8736(11)	82
H(10E)	3764(12)	1819(22)	9111(17)	68
H(10F)	3643(11)	710(58)	9806(4)	80
H(10G)	3193(5)	1396(31)	10467(18)	65
H(10H)	2840(12)	3438(21)	10359(15)	52
H(10I)	2968(9)	4603(48)	9667(3)	49
H(20A)	2532(10)	316(27)	8078(11)	83
H(20B)	3260(10)	787(15)	8086(11)	83
H(20C)	2887(19)	-214(14)	7671(3)	83
H(20D)	2791(17)	856(40)	6849(7)	63
H(20E)	2272(5)	2308(7)	6200(22)	73
H(20F)	1667(14)	3821(40)	6495(16)	66
H(20G)	1501(17)	3972(39)	7350(7)	64
H(20H)	2086(4)	2419(7)	7933(20)	60