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Synthesis, Mechanism of Formation, and Catalytic Activity of Xantphos Nickel π-Complexes

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Supplementary Information

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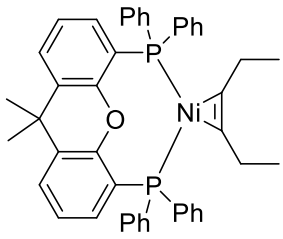
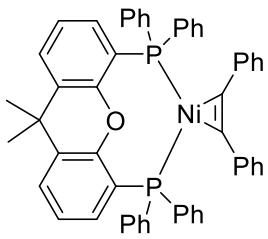
General Considerations

All reactions were carried out under an atmosphere of N_2 using standard schlenk technique or in a N_2 filled glovebox. Benzene, pentane, and THF were dried over neutral alumina under N_2 using a Grubbs type solvent purification system. Xantphos was purchased from Strem and used as received. $Ni(COD)_2$ was purchased from Strem or synthesized according to the literature procedure[1]. 3-Hexyne, benzonitrile, and deuterated benzene were distilled from CaH_2 and degassed by three freeze pump thaw cycles. THF-d_8 was purchased from Cambridge isotope and used as received. 2-Butyne-1,4-diol was recrystallized from toluene prior to use. Diphenylacetylene, *trans*-stilbene, dimethylfumarate, and triphenylphosphine oxide were purchased from Aldrich and used as received.

^{1}H Nuclear Magnetic Resonance spectra were acquired at 300 or 400 MHz. ^{13}C spectra were recorded at 100 MHz. ^{31}P spectra were recorded at 121 MHz. All ^{13}C and ^{31}P NMR spectra were proton decoupled. ^{1}H and ^{13}C spectra were referenced to solvent references (C_6D_6 δ 7.16 and δ 128.6; THF-d_8, δ 3.76 and δ 68.0). ^{31}P NMR shifts were reported with respect to external 85% H_3PO_4 (0 ppm). IR spectra were recorded on a Bruker Tensor 27 FT-IR spectrometer. X-ray crystallography data was collected and analyzed by Dr. Atta Arif at the University of Utah. Elemental analysis was performed by Midwest Microlabs LLC.

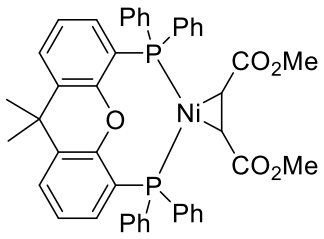


(Xantphos)Ni(3-hexyne) (1). In a drybox $Ni(COD)_2$ (100 mg, 0.363 mmol) and Xantphos (210.4 mg, 0.363 mmol) were weighed into a 20 mL scintillation vial equipped with a Teflon coated stirbar and dissolved in 12 mL of benzene. 3-hexyne (83 μL, 0.726 mmol) followed by PhCN (50 μL, 0.486 mmol) were added to the $Ni(COD)_2$/Xantphos solution and stirred at rt for 3 h. The resultant yellow solution was then reduced in volume to about 5 mL, followed by the addition 15 mL of pentane. This solution was stored at -40 °C overnight, resulting in the formation of a yellow solid, which was removed by filtration, washed with pentane and dried to yield 198.5 mg (76% yield) of **1**. A secondary recrystallization from benzene/pentane afforded another 30 mg (88% total yield) of **1**. Crystals suitable for X-ray crystallographic analysis were prepared by slow diffusion of pentane into a benzene solution of the complex. ^{1}H NMR (300 MHz, C_6D_6): δ 7.81 (br s, 8H), 6.92-7.14 (m, 14H), 6.75-6.82 (m, 4H), 2.15 (q, J = 5.4 Hz, 4H), 1.29 (s, 6H), 1.12 (t, J = 5.4 Hz 6H). $^{13}C\{^1H\}$ NMR (100.6 MHz, C_6D_6): δ 159.0 (t, J_{PC} = 5.6 Hz, *Ar*), 137.9 (m, J_{PC} = 18 Hz, *Ar*), 136.5 (m, *C C*), 134.8 (t, J_{PC} = 18 Hz, *Ar*), 132.5 (s, *Ar*), 129.5 (s, *Ar*), 129.2 (s, *Ar*), 128.8 (t, J_{PC}=4.0, *Ar*) 128.8 (m, *Ar*), 125.9 (s, *Ar*), 124.2 (s, *Ar*), 37.6 (s, $C(CH_3)_2$), 27.3 (s, CH_3), 20.7 (t, J_{PC}=7.1, CH_2), 16.9 (s, CH_3). $^{31}P\{^1H\}$ NMR (121 MHz, C_6D_6): δ 22.0 (s). IR (nujol, NaCl): 1821 (m. ν_{CC}), 1584 (w) 1434 (s) 1403 (s) 1307 (w) 1237 (w) 1219 (s) 1180 (m) 1148 (m) 1118 (m) 1088 (s) 1066 (m) 1026 (w) 998 (w) 872 (w) 793 (w) 776 (m) 757 (w) cm^{-1}.



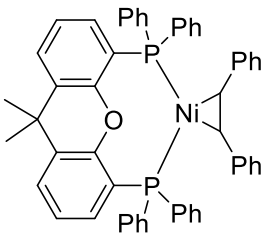
(Xantphos)Ni(diphenylacetylene) (2). In a drybox $Ni(COD)_2$ (100 mg, 0.363 mmol) and Xantphos (210.4 mg, 0.363 mmol) were weighed into a 20 mL scintillation vial equipped with a Teflon coated stirbar and subsequently dissolved in 10 mL of benzene. A solution of diphenylacetylene (129.4 mg, 0.726 mmol) in 2 mL benzene was added followed by PhCN (50 μL, 0.486 mmol). After five minutes the reaction turned from red to yellow and a precipitate began to form. The reaction was stirred for 4 h and 8 mL pentane was added. The reaction was stirred for an additional hour, filtered, washed with several

portions of pentane, and dried under vacuum to yield 276.7 mg (94% yield) of **2** as a yellow solid. Crystals grown from diffusion of pentane into a THF solution of the complex were not suitable for x-ray crystallography. Suitable crystals were grown from a benzene solution containing 0.01 M $Ni(COD)_2$, Xantphos, benzonitrile, and 0.02 M diphenylacetylene. ^1H NMR (400 MHz, THF-d_8): δ 7.67 (d, J = 8.0 Hz, 2 H), 7.44 (m, 13H), 7.21 (t, J = 8.0 Hz, 4H), 7.25 (t, J = 8.0 Hz, 9H), 6.85 (m, 9H), 6.59 (t, J = 8.0 Hz, 2H), 1.76 (s, 6H). $^{13}C\{^1H\}$ NMR (100.8 MHz, THF-d_8): δ 158.6 (t, J_{PC} = 5 Hz, *Ar*), 138.1 (t, J_{PC} = 7 Hz, *Ar*) 136.8 (s, *C C*), 135.8 (m, *Ar*), 135.2 (t, J_{PC} = 7 Hz, *Ar*), 132.3 (s, *Ar*), 129.8 (s, *Ar*), 129.6 (s, *Ar*), 129.0 (t, J_{PC} = 5 Hz, *Ar*), 128.4 (s, *Ar*), 128.2 (s, *Ar*), 126.8 (s, *Ar*), 126.3 (m, *Ar*), 125.0 (s, *Ar*), 124.6 (s, *Ar*), 38.1 (s, $C(CH_3)_2$), 27.6 (s, CH_3).$^{31}P\{^1H\}$ NMR (121 MHz, C_6D_6): δ 22.9 (s). IR (nujol, NaCl): 1792 (s, ν_{CC}) 1589 (s) 1497 (s) 1434 (s) 1409 (s) 1309 (w) 1234 (s) 1184 (m) 1150 (w)) 1120 (w) 1096 (m) 1066 (w) 1026 (w) 1000 (w) 880 (w) 791 (w) 771 (m) cm^{-1}. Anal. Calcd. for $C_{53}H_{42}NiOP_2$: C, 78.05; H, 5.19. Found: C, 77.95; H, 5.42.

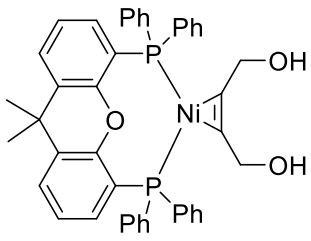


(Xantphos)Ni(dimethylfumarate) (3). In a drybox $Ni(COD)_2$ (100 mg, 0.363 mmol) and Xantphos (210.4 mg, 0.363 mmol) were weighed into a 20 mL scintillation vial equipped with a Teflon coated stirbar and subsequently dissolved in 10 mL of benzene. Dimethylfumarate (104.9 mg, 0.727 mmol) in 2 mL benzene was added followed by PhCN (50 µL, 0.486 mmol). The reaction was stirred for 4 h, at which point it was concentrated to 5 mL. Pentane (15 mL) was added and the solution stored at -40 °C overnight, resulting in the formation of a dark solid, which was removed by filtration, washed with several portions of pentane and dried to yield 252.8 mg (89% yield) of **3** as a strikingly purple solid. Crystals suitable for x-ray crystallography were grown by diffusion of pentane into a benzene solution of the complex. ^1H NMR (400 MHz, C_6D_6): δ 7.84 (t, J = 12 Hz, 4H), 7.44 (t, J = 12 Hz, 4H), 7.15 (t, J = 8.0 Hz, 5H), 7.08 (d, J = 8.0 Hz, 2H), 7.00 (m, 7 H), 6.69 (m, 4H), 3.67 (s, 2H) 3.15 (s, 6H), 1.26 (s, 6H).

$^{13}C\{^1H\}$ (100.6 MHz, C_6D_6): δ 174.4 (s, *C*O_2CH_3), 157.6 (t, J_{PC} = 5 Hz, *Ar*), 135.5 (t, J_{PC} = 2 Hz, *Ar*), 135.0 (d, J_{PC} = 13 Hz, *Ar*), 134.6 (dd, J_{PC} = 6 Hz, 5 Hz), 132.4 (s, *Ar*), 129.8 (d, J_{PC} = 8 Hz, *Ar*), 129.0 (t, J_{PC} = 5 Hz, *Ar*), 126.7 (s, *Ar*), 124.8 (t, J_{PC} = 3 Hz, *Ar*), 124.2 (dd, J_{PC} = 12, 3 Hz, *Ar*), 50.7 (s CO_2CH_3) 50.0 (t, J_{PC} = 8 Hz, *C*=*C*), 36.8 (s, *C*$(CH_3)_2$), 27.4 (s, *C*H_3). $^{31}P\{^1H\}$ NMR (121 MHz, C_6D_6): δ 16.8 (s). IR (nujol, NaCl): 1678 (s ν_{CO}), 1586 (w) 1461 (s) 1434 (m) 1410 (s) 1282 (m) 1259 (m) 1229 (s) 1205 (s) 1156 (w) 1135 (m) 1096 (w) 1036 (s) 999 (w) 881 (w) 846 (w) 793 (w) 776 (w) 754 (m) cm^{-1}. Anal. Calcd. for $C_{45}H_{40}NiO_5P_2$: C, 69.17; H, 5.16. Found: C, 68.80; H, 4.99.



(Xantphos)Ni(*trans*-stilbene) (4) In a drybox $Ni(COD)_2$ (100 mg, 0.363 mmol) and Xantphos (210.4 mg, 0.363 mmol) were weighed into a 20 mL scintillation vial equipped with a Teflon coated stirbar and subsequently dissolved in 10 mL of benzene. *trans*-stilbene (131.3 mg, 0.727 mmol) in 2 mL benzene was added followed by PhCN (50 μL, 0.486 mmol). The reaction was stirred for 4 h, at which point it was concentrated to 5 mL. Pentane (15 mL) was added and the solution stored at -40 °C overnight, resulting in the formation of a dark solid, which was removed by filtration, washed with several portions of pentane and dried to yield 226.6 mg (75.8% yield) of **4** as a red solid. ^1H NMR (400 MHz, C_6D_6): δ 7.63 (t, *J* = 8.0 Hz, 4H) 7.40 (m, 2H), 7.0 (m, 20H), 6.78 (m, 6H), 6.70 (t, *J* = 8.0 Hz, 2H), 6.40 (m, 2H) 3.97 (s, 2H), 1.23 (s, 6H). $^{13}C\{^1H\}$ (100.6 MHz, C_6D_6): δ 156.6 (t, J_{PC} = 6 Hz, Ar), 147.0 (s, Ar), 135.4 (t, J_{PC} = 8 Hz, Ar), 135.2 (dd, J_{PC} = 15, 2 Hz, Ar), 134.7 (t, J_{PC} = 7 Hz, Ar), 133.7 (t, J_{PC} = 6 Hz, Ar), 131.8 (s, Ar), 129.5 (s, Ar), 129.12 (s, Ar), 128.7 (t, J_{PC} = 3 Hz, Ar), 126.7 (s, Ar), 126.4 (s, Ar), 124.7 (t, J_{PC} = 2 Hz, Ar), 123.8 (s, Ar) 65.6 (t, J_{PC} = 8 Hz, *C*=*C*), 36.9 (s, *C*$(CH_3)_2$), 27.7 (s, *C*H_3). $^{31}P\{^1H\}$ NMR (121 MHz, C_6D_6): δ 16.3 (s). IR (nujol, NaCl): 1590 (s) 1435 (s) 1407 (s) 1378 (s) 1305 (w) 1238 (s) 1180 (w) 1149 (w) 1116 (s) 1093 (m) 1068 (w) 1028 (w) 998 (w) 881 (w) 830 (w) 790 (w) 775 (w) cm^{-1}. Anal. Calcd. for $C_{53}H_{44}NiOP_2$: C, 77.86; H, 5.42. Found: C, 77.61; H, 5.43.



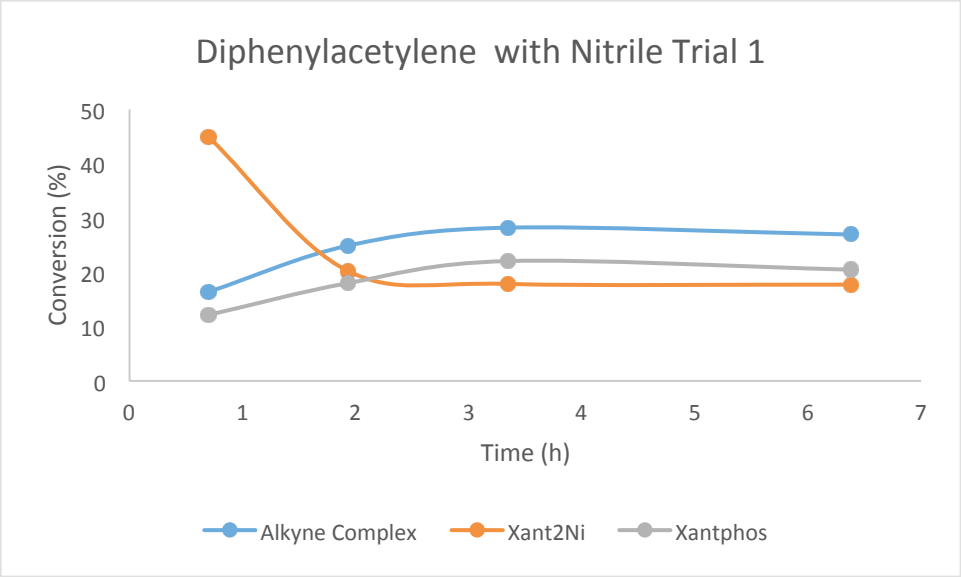
(Xantphos)Ni(2-butyne-1,4-diol) (5). In a drybox $Ni(COD)_2$ (100 mg, 0.363 mmol) and Xantphos (210.4 mg, 0.363 mmol) were weighed into a 20 mL scintillation vial equipped with a Teflon coated stirbar and subsequently dissolved in 10 mL of THF. A solution of 2-butyne-1,4-diol (129.4 mg, 0.726 mmol) in 2 mL THF was added followed by PhCN (50 μL, 0.486 mmol). The reaction was stirred for 4 h and concentrated to dryness. The solids were extracted with benzene and filtered, leaving a small amount of black residue. The filtrate was concentrated to ~10 mL and transferred to a 20 mL scintiallation vial. 10 mL pentane was added and the solution was allowed to sit overnight to afford a yellow solid that was collected by filtration, washed with pentane, and dried under vacuum (116.6 mg, 44%).^{1}H NMR (400 MHz, C_6D_6): δ 7.65 (s, 7H), 7.15 (d, J = 4.0 Hz, 3H), 7.03 (dd, J = 4.0 Hz, J = 8.0 Hz, 2H), 6.97 (br s, 11H), 6.71 (m, 3H), 4.09 (s, 4H), 3.05 (s, 2H), 1.27 (s, 6H).^{13}C{^{1}H} (100.6 MHz, C_6D_6): δ 158.6 (t, J_{PC} = 5Hz, *Ar*), 137.7 (m, *Ar*), 136.3 (s, *C C*), 134.4 (t, J_{PC} = 7 Hz, *Ar*), 132.6 (s, *Ar*), 129.8 (s, *Ar*), 129.2 (s, *Ar*), 129.0 (t, J_{PC} = 5 Hz, *Ar*), 126.4 (s, *Ar*), 125.8 (m, *Ar*) 124.4 (s, *Ar*), 59.4 (t, J_{PC} = 9 Hz, *C*H_2OH), 37.4 (s, *C*$(CH_3)_2$), 27.4 (s, *C*H_3).^{31}P{^{1}H} NMR (121 MHz, C_6D_6): δ 21.4 (s). IR (nujol, NaCl): 3280 (br, OH) 1799 (m, ν_{CC}) 1585 (w) 1478 (m) 1434 (s) 1405 (s) 1306 (w) 1225 (m) 1182 (w) 1150 (w) 1120 (w) 1093 (m) 1067 (w) 999 (m) 876 (w) 794 (w) cm^{-1}. Anal. Calcd. for $C_{43}H_{38}NiO_3P_2$: C, 71.39; H, 5.29. Found: C, 71.30; H, 5.47.

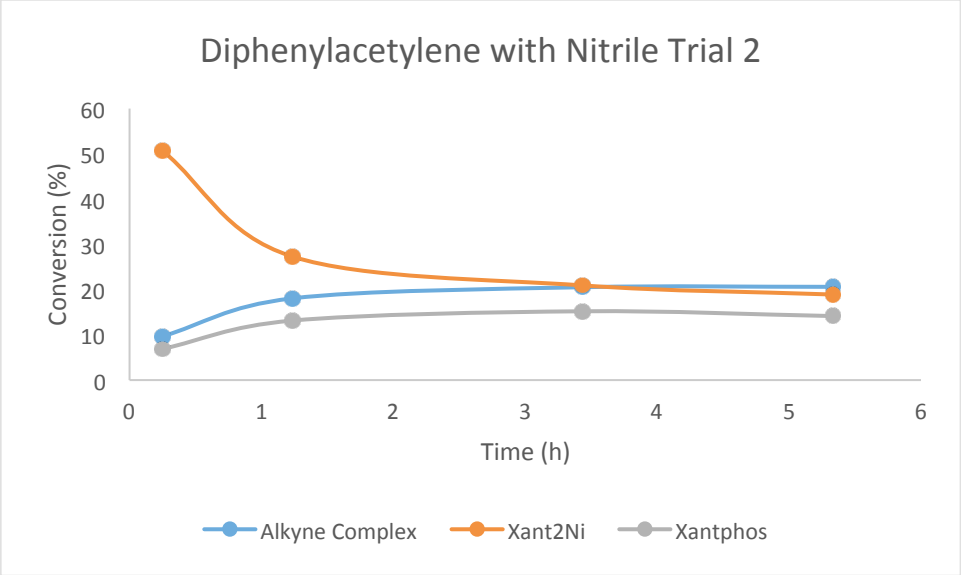
Complex 1 by Reduction: In a drybox (Xant)$NiBr_2$[2] (159.4 mg, 0.2 mmol, 1 eq) was suspended in THF (2 mL). 3-hexyne (45 μL, 0.4 mmol, 2 eq) was added, followed by a suspension of Zn (19.6 mg, 0.3 mmol, 1.5 eq) in THF (2 mL). The green suspension began turning yellow within 20 minutes, and was stirred overnight. Pentane (8 mL) was added and the reaction mixture stirred for an additional hour then filtered through celite. The filtrate was concentrated to dryness, taken up in benzene (4 mL), and pentane (16 mL) was added. The solution was stored at -40 ºC for 24 h. A yellow-brown solid (46.8 mg) was isolated by filtration. This material was silent by ^{31}P NMR. The ^{1}H NMR displayed peaks corresponding to THF and several weak, broad peaks.

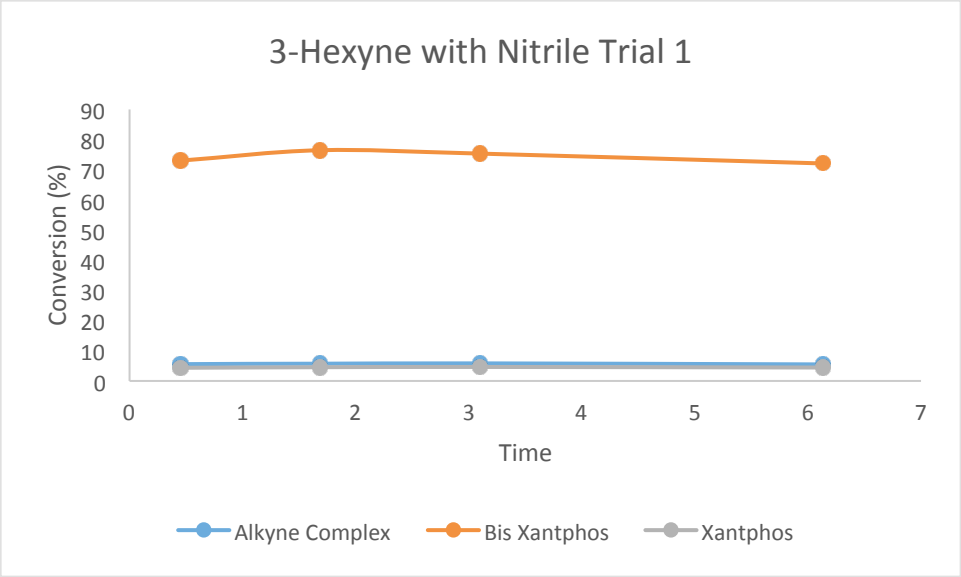
NMR Experiments

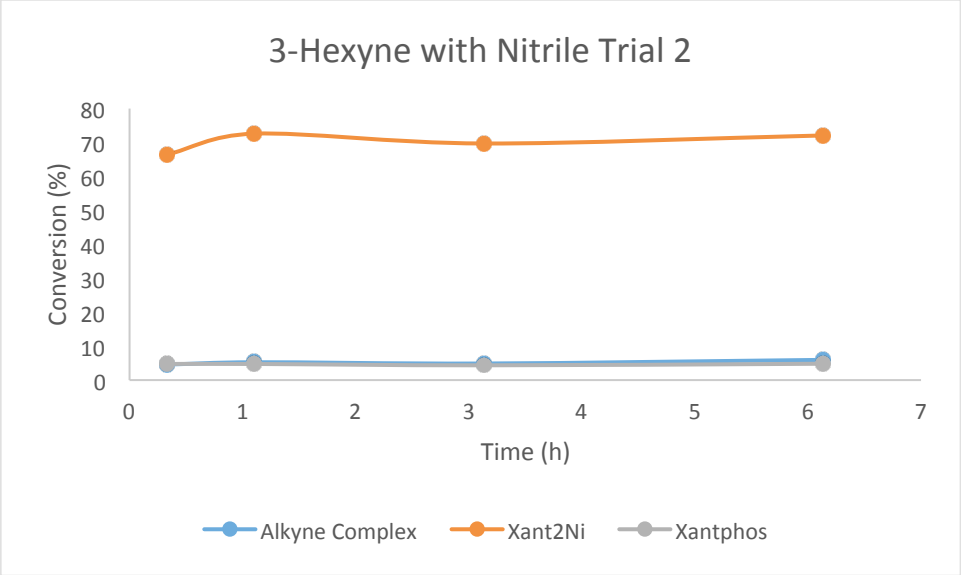
Reaction of $(Xant)_2Ni$ ($(Xant)_2Ni$ (52 mg, 0.0428 mmol), triphenylphosphine oxide (6.0 mg, 0.021 mmol), and benzene (6 mL) were added to a 20 mL vial and stirred for 1 h. The residual solids were allowed to settle and the supernatant was filtered. 3-hexyne, diphenylacetylene, and trans-stilbene (0.032 mmol) were added to three separate vials. Aliquots of 1.5 mL of the $(Xant)_2Ni$/TPPO solution were added to each alkene/alkyne. The resulting solutions were divided into two 0.75 mL portions and benzonitrile (3 μL, 0.03 mmol) was added to one of these portions. The solutions were transferred to NMR tubes, sealed with septa, and monitored by ^{31}P NMR. A spectrum of the $(Xant)_2Ni$/TPPO solution was obtained as a T_0.

$(Xant)_2Ni$ (26 mg, 0.021 mmol) and TPPO (6.2 mg, 0.022 mmol) were weighed out into a vial. Benzene (3 mL) was added and the solution was stirred for 1 h, and filtered. An aliquot (1.5 mL) was added to diphenylacetylene (7.2 mg, 0.04 mmol). Half (0.75 mL) of this solution was added to N-methyl-2- Pyrrolidone (NMP) (3 μL, 0.03 mmol). The solutions were stirred for 3 h, transferred to septum-sealed NMR tubes and analyzed by ^{31}P NMR. Only trace alkyne complex and free Xantphos were observed in the tube containing NMP. The tube was brought back into the glovebox and NMP (25 μL) was added. The tube was allowed to sit overnight and the solution again analysed by ^{31}P NMR, at which point an 8% yield of alkyne complex was observed.


Diphenylacetylene with Nitrile Trial 1
Conversion (%)
50
40
30
20
10
0
0
1
2
3
4
5
6
7
Time (h)
Alkyne Complex
Xant2Ni
Xantphos


Diphenylacetylene with Nitrile Trial 2
Conversion (%)
60
50
40
30
20
10
0
0
1
2
3
4
5
6
Time (h)
Alkyne Complex
Xant2Ni
Xantphos



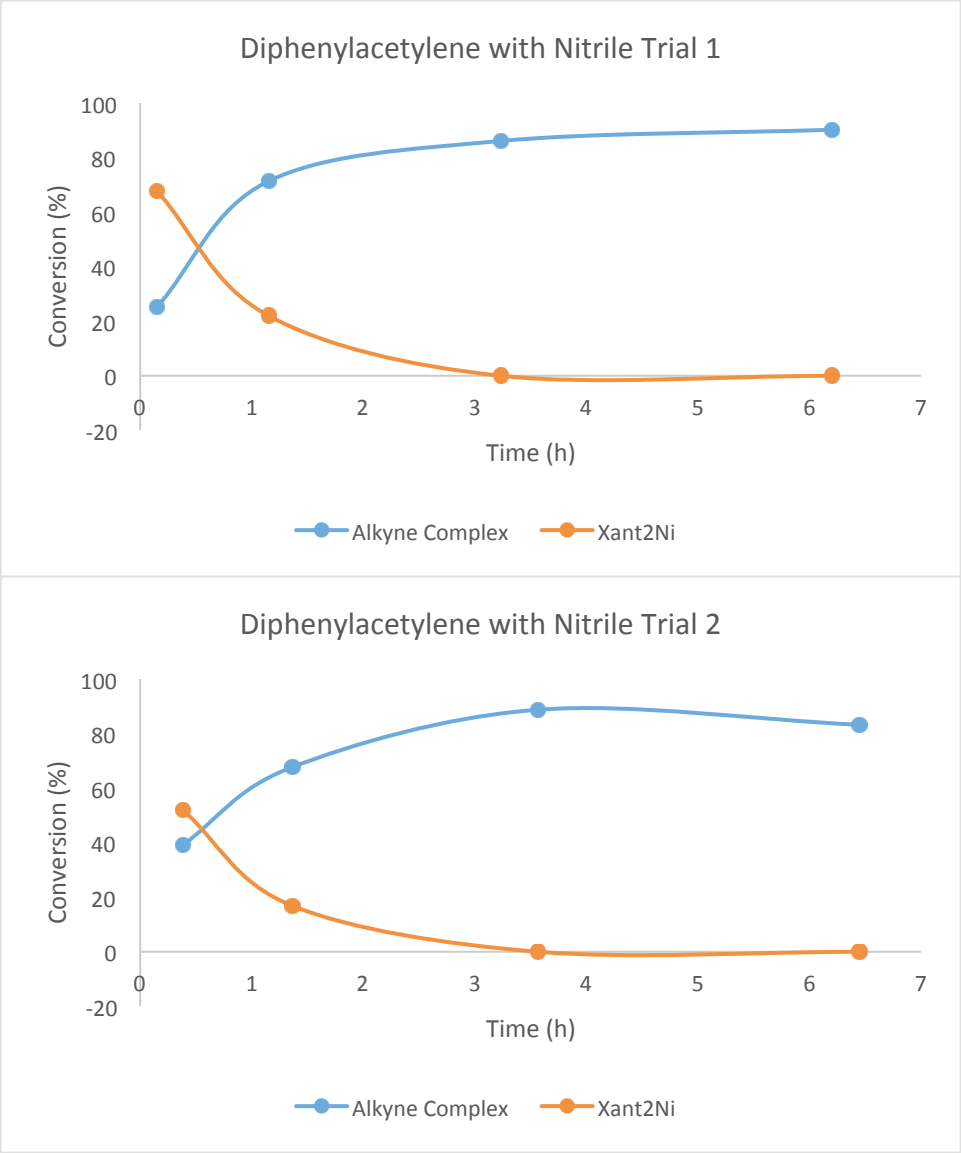


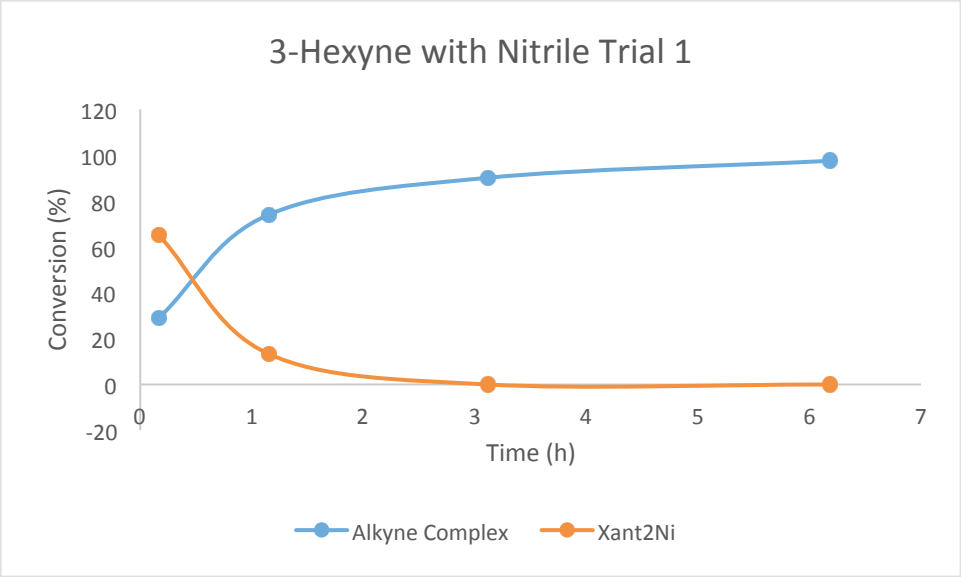
Reactions of $Ni(COD)_2$, Xantphos, Diphenylacetylene, and Benzonitrile: The following stock solutions were prepared: $Ni(COD)_2$ (11.6 mg, 0.042 mmol) in benzene (2 mL); Xantphos (24.3 mg, 0.042 mmol), and triphenylphosphine oxide (5.8 mg, 0.021 mmol) in benzene (2 mL); and diphenylacetylene (37.4 mg, 0.210 mmol) in benzene (2 mL). A ^{31}P NMR of the Xantphos/TPPO solution was taken as a T_0.

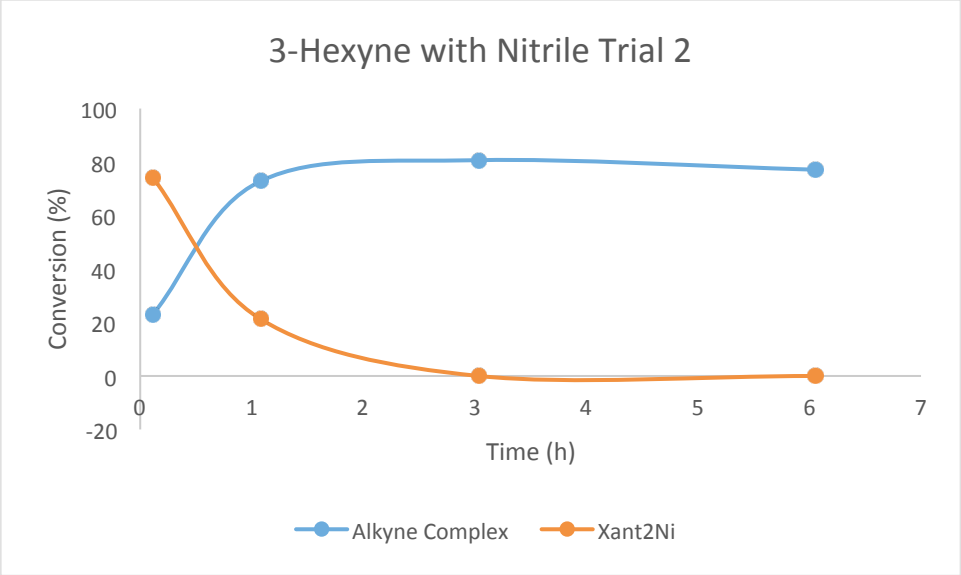
$(Xant)_2Ni$ Correction: A solution of Xantphos (12.2 mg, 0.021 mmol) and triphenylphosphine oxide (2.9 mg, 0.0105 mmol) in 1.5 mL C_6H_6 was prepared. A 0.75 mL aliquot of this solution was added to $Ni(COD)_2$ (1.4 mg, 0.00525 mmol). ^{31}P NMR spectra of each solution were taken to obtain a correction factor of 2.15.

Alkyne Complex Correction: Experiments A, B, and C were run in parallel. The average integration of alkyne complex from experiments in which alkyne complex was produced quantitatively (B, C) was used as a T_0 for alkyne complex.

A) $Ni(COD)_2$, then Xantphos, then Alkyne, then Nitrile: An aliquot of the Xantphos solution (0.5 mL, 1 eq) was added an aliquot of the $Ni(COD)_2$ solution (0.5 mL, 1 eq). The resulting solution was allowed to sit for 2 minutes at which point an aliquot of the alkyne solution (0.5 mL, 5 eq) was added. Half (0.75 mL) of this solution was transferred to a NMR tube and sealed with a septum. Benzonitrile (3 μL, 5 eq) was added to the remainder and it was transferred to a NMR tube and sealed with a septum. The solutions were monitored by ^{31}P NMR.


Diphenylacetylene with Nitrile Trial 1
Conversion (%)
100
80
60
40
20
0
-20
0
1
2
3
4
5
6
7
Time (h)
Alkyne Complex
Xant2Ni
Diphenylacetylene with Nitrile Trial 2
Conversion (%)
100
80
60
40
20
0
-20
0
1
2
3
4
5
6
7
Time (h)
Alkyne Complex
Xant2Ni





B) Ni(COD)$_2$, then Alkyne, then Xantphos, then Nitrile: An aliquot of the alkyne solution (0.5 mL, 5 eq) was added an aliquot of the Ni(COD)$_2$ solution (0.5 mL, 1 eq). The resulting solution was allowed to sit for 5 minutes at which point an aliquot of the Xantphos solution (0.5 mL, 1 eq) was added. Half (0.75 mL) of this solution was transferred to a NMR tube and sealed with a septum. Benzonitrile (3 µL, 5 eq) was added to the remainder and it was transferred to a NMR tube and sealed with a septum. The solutions were monitored by ^{31}P NMR.

C) Xantphos and Alkyne, then $Ni(COD)_2$ and Nitrile: An aliquot of the Xantphos solution (0.5 mL, 1 eq) was added an aliquot of the alkyne solution (0.5 mL, 5 eq). followed by an aliquot of the $Ni(COD)_2$ solution (0.5 mL, 1 eq). Half (0.75 mL) of this solution was transferred to a NMR tube and sealed with a septum. Benzonitrile (3 μL, 5 eq) was added to the remainder and it was transferred to a NMR tube and sealed with a septum. The solutions were monitored by ^{31}P NMR.

Catalytic Activity

Cycloaddition: A stock solution containing dimethyl 2,2-di(but-2-yn-1-yl)malonate (diyne) (236.3 mg, 1.0 mmol, 1 eq) acetonitrile (78.3 μL, 1.5 mmol, 1.5 eq) and naphthalene (64.1 mg, 0.5 mmol, 0.5 eq) in 10 mL toluene (0.1 M in diyne) was prepared. Complexes **1-5**, (0.003 mmol, 3 mol%) were weighed into separate vials. $(Xant)_2Ni$ (0.003 mmol, 3 mol%) were weighed into two separate vials. Benzonitrile (1 μL, 10 mol%) was added to one of the vials containing $(Xant)_2Ni$. Aliquots (1 mL, 0.1 mmol diyne) of the stock solution were added to each vial. The vials were stirred at room temperature for 3 h, filtered, and subjected to GC analysis. Conversions and yields were calculated based on a GC of the stock solution.

Cross-Coupling: Stock solution 1, containing 1-bromonaphthalene (370.7 μL, 2.66 mmol), and naphthalene (128.0 mg, 1 mmol) in THF (2 mL, 1.33 M in aryl halide) was prepared. Stock solution 2 containing 1.0 M vinyl zinc bromide was prepared according to the literature procedure.[3] Complexes **1-5**, (0.010 mmol, 5 mol%) were weighed into separate vials. $(Xant)_2Ni$ (0.003 mmol, 3 mol%) were weighed into two separate vials. Benzonitrile (1 μL, 10 mol%) was added to one of the vials containing $(Xant)_2Ni$. Stock solution 1 (150 μL, 0.2 mmol, 1 eq aryl halide) was added to each vial followed by stock solution 2 (350 μL, 0.35 mmol, 1.75 eq). The solutions were stirred at 50 ºC for 5 h. The solutions were diluted with 1 mL EtOAc, filtered, and

subjected to GC analysis. Conversions and yields were calculated based on a GC of the stock solution.

Air Stability

Solid: Several samples ~10 mg for complex **1** and ~5 mg for $(Xant)_2Ni$ were weighed into vials. The vials were removed from the glovebox. After ~5 min ~15 min, ~30 min, ~60 min for complex **1** and $(Xant)_2Ni$, as well as 2 h, 4 h, and 18 h for $(Xant)_2Ni$ the vials were flushed with nitrogen through a 16 G needle, capped, and brought back into the glovebox. C_6D_6 (0.5 mL) was added to the complex **1** vials and the solutions analyzed by ^{31}P and ^{1}H NMR. Benzene (0.75 mL) was added to the $(Xant)_2Ni$ vials. After 1 h, the solids were filtered off and the solutions analyzed by ^{31}P NMR.

Solution: A solution containing complex **1** (16.8 mg, 0.023 mmol) and TPPO (8.2 mg, 0.029 mmol) in C_6D_6 (0.75 mL, 0.03 M in **1**) was made and transferred to a septum-sealed vial. A ^{31}P spectrum was obtained as a T_0. The cap was removed, and air introduced by inserting a needle/syringe into the tube and manipulating the plunger, followed by capping the tube and shaking. Spectra were recorded after 5, 10, and 20 minutes. The experiment was repeated using a solution of $(Xant)_2Ni$ and TPPO prepared as described in section 2: NMR Experiments.

Table 1a. Crystal data and structure refinement for (Xant)Ni(3-Hexyne).

Empirical formula	C45 H42 Ni O P2	
Formula weight	719.44	
Temperature	150(1) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	$\boldsymbol{P}\,2_1/n$	
Unit cell dimensions	a = 13.6794(3) Å	α= 90°.
	b = 15.0260(3) Å	β= 93.2001(14)°.
	c = 17.7954(4) Å	γ = 90°.
Volume	3652.08(14) $Å^3$	
Z	4	
Density (calculated)	1.308 Mg/m^3	
Absorption coefficient	0.653 mm^{-1}	
F(000)	1512	
Crystal size	0.23 x 0.23 x 0.15 mm^3	
Theta range for data collection	1.93 to 27.00°.	
Index ranges	-17<=h<=16, -19<=k<=18, -22<=l<=22	
Reflections collected	29344	
Independent reflections	7946 [R(int) = 0.0653]	
Completeness to theta = 27.00°	99.7 %	
Absorption correction	Multi-scan	
Max. and min. transmission	0.9083 and 0.8642	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	7946 / 0 / 442	
Goodness-of-fit on F^2	1.044	
Final R indices [I>2sigma(I)]	R1 = 0.0423, wR2 = 0.0713	
R indices (all data)	R1 = 0.0826, wR2 = 0.0843	
Largest diff. peak and hole	0.500 and -0.405 $e.Å^{-3}$	

Table 1b. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for **1**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Ni(1)	8563(1)	2059(1)	8684(1)	14(1)
P(1)	7746(1)	2003(1)	7600(1)	13(1)
P(2)	7764(1)	2204(1)	9699(1)	15(1)
O(1)	6091(1)	2278(1)	8582(1)	16(1)
C(1)	5774(2)	1810(2)	7938(1)	15(1)
C(2)	6457(2)	1656(2)	7406(1)	15(1)
C(3)	6124(2)	1206(2)	6753(1)	20(1)
C(4)	5160(2)	916(2)	6661(1)	24(1)
C(5)	4515(2)	1049(2)	7225(1)	23(1)
C(6)	4810(2)	1497(2)	7882(1)	19(1)
C(7)	4178(2)	1660(2)	8551(2)	23(1)
C(8)	4852(2)	1550(2)	9261(1)	20(1)
C(9)	5803(2)	1875(2)	9240(1)	16(1)
C(10)	6511(2)	1808(2)	9828(1)	17(1)
C(11)	6215(2)	1410(2)	10493(1)	22(1)
C(12)	5265(2)	1105(2)	10542(1)	27(1)
C(13)	4594(2)	1168(2)	9933(1)	25(1)
C(14)	3796(2)	2628(2)	8536(2)	32(1)
C(15)	3307(2)	1016(2)	8541(2)	32(1)
C(16)	7728(2)	3103(2)	7146(1)	15(1)
C(17)	8493(2)	3692(2)	7323(1)	18(1)
C(18)	8579(2)	4477(2)	6923(1)	19(1)
C(19)	7901(2)	4691(2)	6349(1)	22(1)
C(20)	7114(2)	4127(2)	6188(2)	27(1)
C(21)	7029(2)	3338(2)	6582(1)	23(1)
C(22)	8332(2)	1317(2)	6891(1)	15(1)
C(23)	8429(2)	408(2)	7035(1)	23(1)
C(24)	8874(2)	-149(2)	6537(2)	31(1)
C(25)	9253(2)	198(2)	5899(2)	36(1)
C(26)	9182(2)	1102(2)	5753(2)	37(1)
C(27)	8715(2)	1660(2)	6245(1)	25(1)

C(28)	7683(2)	3340(2)	10073(1)	19(1)
C(29)	8296(2)	3997(2)	9808(1)	24(1)
C(30)	8285(2)	4852(2)	10109(2)	33(1)
C(31)	7661(2)	5060(2)	10668(2)	38(1)
C(32)	7041(2)	4421(2)	10926(2)	36(1)
C(33)	7051(2)	3567(2)	10630(2)	27(1)
C(34)	8416(2)	1593(2)	10468(1)	16(1)
C(35)	8729(2)	1941(2)	11165(1)	25(1)
C(36)	9281(2)	1430(2)	11677(2)	33(1)
C(37)	9514(2)	559(2)	11518(2)	32(1)
C(38)	9201(2)	201(2)	10835(2)	34(1)
C(39)	8670(2)	717(2)	10313(2)	29(1)
C(40)	11504(2)	2563(2)	9706(2)	34(1)
C(41)	10432(2)	2424(2)	9823(1)	28(1)
C(42)	9852(2)	2224(2)	9105(1)	20(1)
C(43)	9890(2)	2041(2)	8414(1)	23(1)
C(44)	10570(2)	1882(2)	7793(2)	40(1)
C(45)	11034(3)	2715(3)	7517(2)	63(1)

Table 1c. Bond lengths [Å] and angles [°] for **1**.

Ni(1)-C(42)	1.894(2)
Ni(1)-C(43)	1.905(2)
Ni(1)-P(2)	2.1732(6)
Ni(1)-P(1)	2.1758(6)
P(1)-C(16)	1.839(2)
P(1)-C(22)	1.847(2)
P(1)-C(2)	1.852(2)
P(2)-C(34)	1.836(2)
P(2)-C(28)	1.838(2)
P(2)-C(10)	1.841(2)
O(1)-C(1)	1.393(3)
O(1)-C(9)	1.395(3)
C(1)-C(2)	1.386(3)
C(1)-C(6)	1.398(3)
C(2)-C(3)	1.399(3)
C(3)-C(4)	1.390(3)
C(3)-H(3)	0.9500
C(4)-C(5)	1.387(4)
C(4)-H(4)	0.9500
C(5)-C(6)	1.390(3)
C(5)-H(5)	0.9500
C(6)-C(7)	1.530(3)
C(7)-C(8)	1.531(3)
C(7)-C(15)	1.535(3)
C(7)-C(14)	1.546(4)
C(8)-C(13)	1.389(3)
C(8)-C(9)	1.392(3)
C(9)-C(10)	1.389(3)
C(10)-C(11)	1.405(3)
C(11)-C(12)	1.385(4)
C(11)-H(11)	0.9500
C(12)-C(13)	1.384(4)
C(12)-H(12)	0.9500
C(13)-H(13)	0.9500

C(14)-H(14A)	0.9800
C(14)-H(14B)	0.9800
C(14)-H(14C)	0.9800
C(15)-H(15A)	0.9800
C(15)-H(15B)	0.9800
C(15)-H(15C)	0.9800
C(16)-C(21)	1.392(3)
C(16)-C(17)	1.393(3)
C(17)-C(18)	1.387(3)
C(17)-H(17)	0.9500
C(18)-C(19)	1.379(3)
C(18)-H(18)	0.9500
C(19)-C(20)	1.386(4)
C(19)-H(19)	0.9500
C(20)-C(21)	1.387(3)
C(20)-H(20)	0.9500
C(21)-H(21)	0.9500
C(22)-C(27)	1.389(3)
C(22)-C(23)	1.394(3)
C(23)-C(24)	1.385(4)
C(23)-H(23)	0.9500
C(24)-C(25)	1.377(4)
C(24)-H(24)	0.9500
C(25)-C(26)	1.384(4)
C(25)-H(25)	0.9500
C(26)-C(27)	1.394(4)
C(26)-H(26)	0.9500
C(27)-H(27)	0.9500
C(28)-C(33)	1.393(3)
C(28)-C(29)	1.395(3)
C(29)-C(30)	1.393(4)
C(29)-H(29)	0.9500
C(30)-C(31)	1.384(4)
C(30)-H(30)	0.9500
C(31)-C(32)	1.377(4)
C(31)-H(31)	0.9500

C(32)-C(33)	1.388(4)
C(32)-H(32)	0.9500
C(33)-H(33)	0.9500
C(34)-C(35)	1.392(3)
C(34)-C(39)	1.393(3)
C(35)-C(36)	1.382(4)
C(35)-H(35)	0.9500
C(36)-C(37)	1.380(4)
C(36)-H(36)	0.9500
C(37)-C(38)	1.375(4)
C(37)-H(37)	0.9500
C(38)-C(39)	1.385(4)
C(38)-H(38)	0.9500
C(39)-H(39)	0.9500
C(40)-C(41)	1.507(4)
C(40)-H(40A)	0.9800
C(40)-H(40B)	0.9800
C(40)-H(40C)	0.9800
C(41)-C(42)	1.497(3)
C(41)-H(41A)	0.9900
C(41)-H(41B)	0.9900
C(42)-C(43)	1.265(3)
C(43)-C(44)	1.502(3)
C(44)-C(45)	1.498(5)
C(44)-H(44A)	0.9900
C(44)-H(44B)	0.9900
C(45)-H(45A)	0.9800
C(45)-H(45B)	0.9800
C(45)-H(45C)	0.9800

C(42)-Ni(1)-C(43)	38.89(10)
C(42)-Ni(1)-P(2)	99.09(7)
C(43)-Ni(1)-P(2)	137.87(7)
C(42)-Ni(1)-P(1)	140.94(8)
C(43)-Ni(1)-P(1)	103.03(7)
P(2)-Ni(1)-P(1)	118.92(3)

C(16)-P(1)-C(22)	101.36(10)
C(16)-P(1)-C(2)	100.52(10)
C(22)-P(1)-C(2)	99.23(10)
C(16)-P(1)-Ni(1)	110.41(8)
C(22)-P(1)-Ni(1)	113.93(7)
C(2)-P(1)-Ni(1)	127.75(7)
C(34)-P(2)-C(28)	103.37(11)
C(34)-P(2)-C(10)	99.36(11)
C(28)-P(2)-C(10)	100.29(11)
C(34)-P(2)-Ni(1)	108.88(8)
C(28)-P(2)-Ni(1)	115.96(8)
C(10)-P(2)-Ni(1)	125.86(8)
C(1)-O(1)-C(9)	112.42(17)
C(2)-C(1)-O(1)	117.1(2)
C(2)-C(1)-C(6)	124.3(2)
O(1)-C(1)-C(6)	118.6(2)
C(1)-C(2)-C(3)	116.6(2)
C(1)-C(2)-P(1)	119.75(17)
C(3)-C(2)-P(1)	123.58(18)
C(4)-C(3)-C(2)	120.7(2)
C(4)-C(3)-H(3)	119.6
C(2)-C(3)-H(3)	119.6
C(5)-C(4)-C(3)	120.6(2)
C(5)-C(4)-H(4)	119.7
C(3)-C(4)-H(4)	119.7
C(4)-C(5)-C(6)	120.7(2)
C(4)-C(5)-H(5)	119.6
C(6)-C(5)-H(5)	119.6
C(5)-C(6)-C(1)	116.9(2)
C(5)-C(6)-C(7)	125.6(2)
C(1)-C(6)-C(7)	117.5(2)
C(6)-C(7)-C(8)	106.54(19)
C(6)-C(7)-C(15)	111.3(2)
C(8)-C(7)-C(15)	111.9(2)
C(6)-C(7)-C(14)	110.0(2)
C(8)-C(7)-C(14)	107.6(2)

C(15)-C(7)-C(14)	109.4(2)
C(13)-C(8)-C(9)	116.8(2)
C(13)-C(8)-C(7)	125.7(2)
C(9)-C(8)-C(7)	117.4(2)
C(10)-C(9)-C(8)	124.6(2)
C(10)-C(9)-O(1)	116.5(2)
C(8)-C(9)-O(1)	118.9(2)
C(9)-C(10)-C(11)	116.3(2)
C(9)-C(10)-P(2)	119.83(17)
C(11)-C(10)-P(2)	123.84(19)
C(12)-C(11)-C(10)	120.6(2)
C(12)-C(11)-H(11)	119.7
C(10)-C(11)-H(11)	119.7
C(13)-C(12)-C(11)	120.8(2)
C(13)-C(12)-H(12)	119.6
C(11)-C(12)-H(12)	119.6
C(12)-C(13)-C(8)	120.8(2)
C(12)-C(13)-H(13)	119.6
C(8)-C(13)-H(13)	119.6
C(7)-C(14)-H(14A)	109.5
C(7)-C(14)-H(14B)	109.5
H(14A)-C(14)-H(14B)	109.5
C(7)-C(14)-H(14C)	109.5
H(14A)-C(14)-H(14C)	109.5
H(14B)-C(14)-H(14C)	109.5
C(7)-C(15)-H(15A)	109.5
C(7)-C(15)-H(15B)	109.5
H(15A)-C(15)-H(15B)	109.5
C(7)-C(15)-H(15C)	109.5
H(15A)-C(15)-H(15C)	109.5
H(15B)-C(15)-H(15C)	109.5
C(21)-C(16)-C(17)	118.6(2)
C(21)-C(16)-P(1)	122.41(18)
C(17)-C(16)-P(1)	118.71(17)
C(18)-C(17)-C(16)	120.6(2)
C(18)-C(17)-H(17)	119.7

C(16)-C(17)-H(17)	119.7
C(19)-C(18)-C(17)	120.4(2)
C(19)-C(18)-H(18)	119.8
C(17)-C(18)-H(18)	119.8
C(18)-C(19)-C(20)	119.6(2)
C(18)-C(19)-H(19)	120.2
C(20)-C(19)-H(19)	120.2
C(19)-C(20)-C(21)	120.3(2)
C(19)-C(20)-H(20)	119.9
C(21)-C(20)-H(20)	119.9
C(20)-C(21)-C(16)	120.5(2)
C(20)-C(21)-H(21)	119.7
C(16)-C(21)-H(21)	119.7
C(27)-C(22)-C(23)	118.7(2)
C(27)-C(22)-P(1)	123.79(19)
C(23)-C(22)-P(1)	117.47(18)
C(24)-C(23)-C(22)	121.0(2)
C(24)-C(23)-H(23)	119.5
C(22)-C(23)-H(23)	119.5
C(25)-C(24)-C(23)	119.9(3)
C(25)-C(24)-H(24)	120.0
C(23)-C(24)-H(24)	120.0
C(24)-C(25)-C(26)	120.0(3)
C(24)-C(25)-H(25)	120.0
C(26)-C(25)-H(25)	120.0
C(25)-C(26)-C(27)	120.2(3)
C(25)-C(26)-H(26)	119.9
C(27)-C(26)-H(26)	119.9
C(22)-C(27)-C(26)	120.2(3)
C(22)-C(27)-H(27)	119.9
C(26)-C(27)-H(27)	119.9
C(33)-C(28)-C(29)	118.4(2)
C(33)-C(28)-P(2)	122.57(19)
C(29)-C(28)-P(2)	119.02(19)
C(30)-C(29)-C(28)	120.3(3)
C(30)-C(29)-H(29)	119.9

C(28)-C(29)-H(29)	119.9
C(31)-C(30)-C(29)	120.3(3)
C(31)-C(30)-H(30)	119.8
C(29)-C(30)-H(30)	119.8
C(32)-C(31)-C(30)	120.0(3)
C(32)-C(31)-H(31)	120.0
C(30)-C(31)-H(31)	120.0
C(31)-C(32)-C(33)	119.9(3)
C(31)-C(32)-H(32)	120.1
C(33)-C(32)-H(32)	120.1
C(32)-C(33)-C(28)	121.2(3)
C(32)-C(33)-H(33)	119.4
C(28)-C(33)-H(33)	119.4
C(35)-C(34)-C(39)	117.7(2)
C(35)-C(34)-P(2)	126.07(19)
C(39)-C(34)-P(2)	116.15(18)
C(36)-C(35)-C(34)	120.5(2)
C(36)-C(35)-H(35)	119.7
C(34)-C(35)-H(35)	119.7
C(37)-C(36)-C(35)	121.0(2)
C(37)-C(36)-H(36)	119.5
C(35)-C(36)-H(36)	119.5
C(38)-C(37)-C(36)	119.2(3)
C(38)-C(37)-H(37)	120.4
C(36)-C(37)-H(37)	120.4
C(37)-C(38)-C(39)	119.9(3)
C(37)-C(38)-H(38)	120.0
C(39)-C(38)-H(38)	120.0
C(38)-C(39)-C(34)	121.6(2)
C(38)-C(39)-H(39)	119.2
C(34)-C(39)-H(39)	119.2
C(41)-C(40)-H(40A)	109.5
C(41)-C(40)-H(40B)	109.5
H(40A)-C(40)-H(40B)	109.5
C(41)-C(40)-H(40C)	109.5
H(40A)-C(40)-H(40C)	109.5

H(40B)-C(40)-H(40C)	109.5
C(42)-C(41)-C(40)	112.5(2)
C(42)-C(41)-H(41A)	109.1
C(40)-C(41)-H(41A)	109.1
C(42)-C(41)-H(41B)	109.1
C(40)-C(41)-H(41B)	109.1
H(41A)-C(41)-H(41B)	107.8
C(43)-C(42)-C(41)	145.6(2)
C(43)-C(42)-Ni(1)	71.04(15)
C(41)-C(42)-Ni(1)	143.32(18)
C(42)-C(43)-C(44)	144.2(2)
C(42)-C(43)-Ni(1)	70.07(15)
C(44)-C(43)-Ni(1)	145.69(19)
C(45)-C(44)-C(43)	113.5(3)
C(45)-C(44)-H(44A)	108.9
C(43)-C(44)-H(44A)	108.9
C(45)-C(44)-H(44B)	108.9
C(43)-C(44)-H(44B)	108.9
H(44A)-C(44)-H(44B)	107.7
C(44)-C(45)-H(45A)	109.5
C(44)-C(45)-H(45B)	109.5
H(45A)-C(45)-H(45B)	109.5
C(44)-C(45)-H(45C)	109.5
H(45A)-C(45)-H(45C)	109.5
H(45B)-C(45)-H(45C)	109.5

Symmetry transformations used to generate equivalent atoms:

Table 1d. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for **1**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2 a^{*2} U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Ni(1)	12(1)	20(1)	11(1)	1(1)	1(1)	1(1)
P(1)	12(1)	16(1)	11(1)	1(1)	1(1)	0(1)
P(2)	15(1)	16(1)	12(1)	-1(1)	2(1)	-1(1)
O(1)	14(1)	17(1)	16(1)	2(1)	4(1)	-2(1)
C(1)	15(1)	14(1)	16(1)	4(1)	-4(1)	2(1)
C(2)	16(1)	14(1)	16(1)	5(1)	-1(1)	-1(1)
C(3)	22(1)	23(1)	15(1)	3(1)	0(1)	-1(1)
C(4)	25(1)	25(1)	20(1)	0(1)	-9(1)	-4(1)
C(5)	15(1)	25(1)	28(1)	6(1)	-6(1)	-3(1)
C(6)	14(1)	16(1)	27(1)	7(1)	-1(1)	1(1)
C(7)	13(1)	24(1)	32(1)	3(1)	4(1)	0(1)
C(8)	15(1)	19(1)	26(1)	-1(1)	6(1)	2(1)
C(9)	18(1)	13(1)	19(1)	0(1)	8(1)	2(1)
C(10)	18(1)	15(1)	18(1)	-2(1)	6(1)	1(1)
C(11)	24(1)	26(1)	17(1)	0(1)	5(1)	-2(1)
C(12)	31(2)	30(2)	21(1)	2(1)	11(1)	-6(1)
C(13)	18(1)	28(1)	31(2)	1(1)	13(1)	-3(1)
C(14)	21(1)	32(2)	42(2)	7(1)	6(1)	7(1)
C(15)	14(1)	42(2)	41(2)	5(1)	2(1)	-4(1)
C(16)	16(1)	16(1)	14(1)	2(1)	4(1)	1(1)
C(17)	14(1)	19(1)	20(1)	-1(1)	3(1)	1(1)
C(18)	17(1)	16(1)	24(1)	-4(1)	7(1)	-4(1)
C(19)	31(2)	12(1)	24(1)	4(1)	7(1)	1(1)
C(20)	30(2)	23(1)	28(1)	9(1)	-8(1)	0(1)
C(21)	23(1)	20(1)	26(1)	3(1)	-5(1)	-6(1)
C(22)	12(1)	21(1)	12(1)	-1(1)	-1(1)	-1(1)
C(23)	23(1)	26(1)	22(1)	2(1)	5(1)	2(1)
C(24)	32(2)	24(2)	38(2)	-4(1)	5(1)	7(1)
C(25)	38(2)	42(2)	29(2)	-13(1)	10(1)	12(1)
C(26)	43(2)	49(2)	20(1)	2(1)	14(1)	8(2)
C(27)	26(2)	31(2)	19(1)	2(1)	6(1)	1(1)

C(28)	21(1)	16(1)	19(1)	1(1)	-1(1)	3(1)
C(29)	27(2)	20(1)	25(1)	2(1)	-2(1)	0(1)
C(30)	40(2)	18(1)	41(2)	5(1)	-4(1)	-5(1)
C(31)	50(2)	20(2)	42(2)	-9(1)	-5(2)	7(1)
C(32)	40(2)	28(2)	39(2)	-7(1)	5(1)	9(1)
C(33)	31(2)	20(1)	29(2)	-3(1)	7(1)	2(1)
C(34)	16(1)	18(1)	15(1)	1(1)	3(1)	-3(1)
C(35)	30(1)	24(1)	19(1)	-5(1)	-1(1)	2(1)
C(36)	39(2)	43(2)	17(1)	-3(1)	-7(1)	2(1)
C(37)	33(2)	36(2)	26(2)	11(1)	-8(1)	3(1)
C(38)	43(2)	19(1)	39(2)	2(1)	-11(1)	5(1)
C(39)	40(2)	22(1)	24(1)	-6(1)	-12(1)	4(1)
C(40)	23(2)	50(2)	29(2)	-2(1)	-1(1)	-11(1)
C(41)	20(1)	43(2)	20(1)	-3(1)	-2(1)	0(1)
C(42)	14(1)	26(1)	20(1)	2(1)	0(1)	2(1)
C(43)	11(1)	35(2)	21(1)	0(1)	1(1)	2(1)
C(44)	15(1)	80(3)	26(1)	-10(2)	5(1)	-1(2)
C(45)	44(2)	100(3)	48(2)	34(2)	28(2)	21(2)

Table 1e. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for **1**.

	x	y	z	U(eq)
H(3)	6562	1098	6367	24
H(4)	4941	625	6209	29
H(5)	3863	832	7161	28
H(11)	6670	1349	10913	27
H(12)	5072	850	10999	32
H(13)	3948	947	9974	30
H(14A)	3360	2718	8087	38
H(14B)	4350	3040	8526	38
H(14C)	3435	2740	8987	38
H(15A)	2894	1106	8080	39
H(15B)	2922	1128	8980	39
H(15C)	3548	401	8558	39
H(17)	8959	3555	7722	21
H(18)	9109	4870	7044	23
H(19)	7972	5220	6065	27
H(20)	6632	4283	5806	33
H(21)	6490	2954	6467	28
H(23)	8186	168	7482	28
H(24)	8917	-769	6635	37
H(25)	9564	-182	5559	43
H(26)	9453	1341	5317	45
H(27)	8658	2278	6138	30
H(29)	8723	3860	9421	29
H(30)	8709	5296	9928	40
H(31)	7660	5644	10874	45
H(32)	6606	4565	11307	43
H(33)	6621	3130	10810	32
H(35)	8563	2535	11290	29
H(36)	9504	1683	12145	40
H(37)	9885	211	11876	38

H(38)	9350	-400	10721	41
H(39)	8473	468	9838	35
H(40A)	11852	2689	10190	40
H(40B)	11582	3065	9363	40
H(40C)	11775	2024	9487	40
H(41A)	10359	1926	10179	33
H(41B)	10165	2966	10053	33
H(44A)	11093	1467	7976	48
H(44B)	10200	1592	7366	48
H(45A)	11468	2566	7116	76
H(45B)	11414	2999	7934	76
H(45C)	10522	3123	7321	76

Table 1f. Torsion angles [°] for **1**.

C(42)-Ni(1)-P(1)-C(16)	-67.50(15)
C(43)-Ni(1)-P(1)-C(16)	-78.12(12)
P(2)-Ni(1)-P(1)-C(16)	97.82(8)
C(42)-Ni(1)-P(1)-C(22)	45.75(16)
C(43)-Ni(1)-P(1)-C(22)	35.14(12)
P(2)-Ni(1)-P(1)-C(22)	-148.92(9)
C(42)-Ni(1)-P(1)-C(2)	170.19(16)
C(43)-Ni(1)-P(1)-C(2)	159.57(13)
P(2)-Ni(1)-P(1)-C(2)	-24.49(10)
C(42)-Ni(1)-P(2)-C(34)	-43.04(11)
C(43)-Ni(1)-P(2)-C(34)	-39.64(15)
P(1)-Ni(1)-P(2)-C(34)	146.26(8)
C(42)-Ni(1)-P(2)-C(28)	72.96(12)
C(43)-Ni(1)-P(2)-C(28)	76.36(16)
P(1)-Ni(1)-P(2)-C(28)	-97.74(9)
C(42)-Ni(1)-P(2)-C(10)	-160.38(12)
C(43)-Ni(1)-P(2)-C(10)	-156.98(15)
P(1)-Ni(1)-P(2)-C(10)	28.92(10)
C(9)-O(1)-C(1)-C(2)	-132.8(2)
C(9)-O(1)-C(1)-C(6)	44.7(3)
O(1)-C(1)-C(2)-C(3)	-178.3(2)
C(6)-C(1)-C(2)-C(3)	4.4(3)
O(1)-C(1)-C(2)-P(1)	4.0(3)
C(6)-C(1)-C(2)-P(1)	-173.33(18)
C(16)-P(1)-C(2)-C(1)	-94.45(19)
C(22)-P(1)-C(2)-C(1)	162.08(19)
Ni(1)-P(1)-C(2)-C(1)	31.9(2)
C(16)-P(1)-C(2)-C(3)	88.0(2)
C(22)-P(1)-C(2)-C(3)	-15.5(2)
Ni(1)-P(1)-C(2)-C(3)	-145.68(17)
C(1)-C(2)-C(3)-C(4)	-1.5(3)
P(1)-C(2)-C(3)-C(4)	176.10(19)
C(2)-C(3)-C(4)-C(5)	-1.5(4)
C(3)-C(4)-C(5)-C(6)	1.9(4)

C(4)-C(5)-C(6)-C(1)	0.7(4)
C(4)-C(5)-C(6)-C(7)	-178.1(2)
C(2)-C(1)-C(6)-C(5)	-4.0(3)
O(1)-C(1)-C(6)-C(5)	178.7(2)
C(2)-C(1)-C(6)-C(7)	174.9(2)
O(1)-C(1)-C(6)-C(7)	-2.4(3)
C(5)-C(6)-C(7)-C(8)	141.5(2)
C(1)-C(6)-C(7)-C(8)	-37.2(3)
C(5)-C(6)-C(7)-C(15)	19.3(3)
C(1)-C(6)-C(7)-C(15)	-159.5(2)
C(5)-C(6)-C(7)-C(14)	-102.1(3)
C(1)-C(6)-C(7)-C(14)	79.1(3)
C(6)-C(7)-C(8)-C(13)	-142.7(2)
C(15)-C(7)-C(8)-C(13)	-20.9(4)
C(14)-C(7)-C(8)-C(13)	99.3(3)
C(6)-C(7)-C(8)-C(9)	38.5(3)
C(15)-C(7)-C(8)-C(9)	160.3(2)
C(14)-C(7)-C(8)-C(9)	-79.5(3)
C(13)-C(8)-C(9)-C(10)	2.8(4)
C(7)-C(8)-C(9)-C(10)	-178.3(2)
C(13)-C(8)-C(9)-O(1)	-178.9(2)
C(7)-C(8)-C(9)-O(1)	0.0(3)
C(1)-O(1)-C(9)-C(10)	135.0(2)
C(1)-O(1)-C(9)-C(8)	-43.5(3)
C(8)-C(9)-C(10)-C(11)	-2.3(4)
O(1)-C(9)-C(10)-C(11)	179.3(2)
C(8)-C(9)-C(10)-P(2)	176.05(19)
O(1)-C(9)-C(10)-P(2)	-2.3(3)
C(34)-P(2)-C(10)-C(9)	-160.22(19)
C(28)-P(2)-C(10)-C(9)	94.2(2)
Ni(1)-P(2)-C(10)-C(9)	-38.6(2)
C(34)-P(2)-C(10)-C(11)	18.0(2)
C(28)-P(2)-C(10)-C(11)	-87.6(2)
Ni(1)-P(2)-C(10)-C(11)	139.58(18)
C(9)-C(10)-C(11)-C(12)	0.1(4)
P(2)-C(10)-C(11)-C(12)	-178.2(2)

C(10)-C(11)-C(12)-C(13)	1.5(4)
C(11)-C(12)-C(13)-C(8)	-1.0(4)
C(9)-C(8)-C(13)-C(12)	-1.0(4)
C(7)-C(8)-C(13)-C(12)	-179.9(2)
C(22)-P(1)-C(16)-C(21)	79.6(2)
C(2)-P(1)-C(16)-C(21)	-22.2(2)
Ni(1)-P(1)-C(16)-C(21)	-159.35(18)
C(22)-P(1)-C(16)-C(17)	-94.33(19)
C(2)-P(1)-C(16)-C(17)	163.92(18)
Ni(1)-P(1)-C(16)-C(17)	26.7(2)
C(21)-C(16)-C(17)-C(18)	-2.8(4)
P(1)-C(16)-C(17)-C(18)	171.31(18)
C(16)-C(17)-C(18)-C(19)	0.8(4)
C(17)-C(18)-C(19)-C(20)	1.8(4)
C(18)-C(19)-C(20)-C(21)	-2.4(4)
C(19)-C(20)-C(21)-C(16)	0.4(4)
C(17)-C(16)-C(21)-C(20)	2.3(4)
P(1)-C(16)-C(21)-C(20)	-171.7(2)
C(16)-P(1)-C(22)-C(27)	5.7(2)
C(2)-P(1)-C(22)-C(27)	108.5(2)
Ni(1)-P(1)-C(22)-C(27)	-112.9(2)
C(16)-P(1)-C(22)-C(23)	-177.10(19)
C(2)-P(1)-C(22)-C(23)	-74.3(2)
Ni(1)-P(1)-C(22)-C(23)	64.3(2)
C(27)-C(22)-C(23)-C(24)	-1.7(4)
P(1)-C(22)-C(23)-C(24)	-179.0(2)
C(22)-C(23)-C(24)-C(25)	1.9(4)
C(23)-C(24)-C(25)-C(26)	-0.6(4)
C(24)-C(25)-C(26)-C(27)	-0.9(5)
C(23)-C(22)-C(27)-C(26)	0.2(4)
P(1)-C(22)-C(27)-C(26)	177.4(2)
C(25)-C(26)-C(27)-C(22)	1.1(4)
C(34)-P(2)-C(28)-C(33)	-73.2(2)
C(10)-P(2)-C(28)-C(33)	29.1(2)
Ni(1)-P(2)-C(28)-C(33)	167.77(18)
C(34)-P(2)-C(28)-C(29)	104.8(2)

C(10)-P(2)-C(28)-C(29)	-152.9(2)
Ni(1)-P(2)-C(28)-C(29)	-14.3(2)
C(33)-C(28)-C(29)-C(30)	1.3(4)
P(2)-C(28)-C(29)-C(30)	-176.7(2)
C(28)-C(29)-C(30)-C(31)	-0.5(4)
C(29)-C(30)-C(31)-C(32)	-0.5(4)
C(30)-C(31)-C(32)-C(33)	0.7(5)
C(31)-C(32)-C(33)-C(28)	0.1(4)
C(29)-C(28)-C(33)-C(32)	-1.1(4)
P(2)-C(28)-C(33)-C(32)	176.9(2)
C(28)-P(2)-C(34)-C(35)	1.7(2)
C(10)-P(2)-C(34)-C(35)	-101.3(2)
Ni(1)-P(2)-C(34)-C(35)	125.5(2)
C(28)-P(2)-C(34)-C(39)	-174.0(2)
C(10)-P(2)-C(34)-C(39)	83.0(2)
Ni(1)-P(2)-C(34)-C(39)	-50.2(2)
C(39)-C(34)-C(35)-C(36)	0.7(4)
P(2)-C(34)-C(35)-C(36)	-174.9(2)
C(34)-C(35)-C(36)-C(37)	-1.6(4)
C(35)-C(36)-C(37)-C(38)	0.9(5)
C(36)-C(37)-C(38)-C(39)	0.7(5)
C(37)-C(38)-C(39)-C(34)	-1.7(5)
C(35)-C(34)-C(39)-C(38)	1.0(4)
P(2)-C(34)-C(39)-C(38)	177.0(2)
C(40)-C(41)-C(42)-C(43)	9.3(6)
C(40)-C(41)-C(42)-Ni(1)	-173.2(3)
P(2)-Ni(1)-C(42)-C(43)	176.36(16)
P(1)-Ni(1)-C(42)-C(43)	-16.6(2)
C(43)-Ni(1)-C(42)-C(41)	-178.5(4)
P(2)-Ni(1)-C(42)-C(41)	-2.1(3)
P(1)-Ni(1)-C(42)-C(41)	164.9(3)
C(41)-C(42)-C(43)-C(44)	0.5(8)
Ni(1)-C(42)-C(43)-C(44)	-177.9(5)
C(41)-C(42)-C(43)-Ni(1)	178.4(5)
P(2)-Ni(1)-C(43)-C(42)	-5.4(2)
P(1)-Ni(1)-C(43)-C(42)	169.34(15)

C(42)-Ni(1)-C(43)-C(44)	177.9(5)
P(2)-Ni(1)-C(43)-C(44)	172.5(3)
P(1)-Ni(1)-C(43)-C(44)	-12.8(4)
C(42)-C(43)-C(44)-C(45)	-74.4(5)
Ni(1)-C(43)-C(44)-C(45)	109.1(4)

Symmetry transformations used to generate equivalent atoms:

Table 2a. Crystal data and structure refinement for (Xantphos)Ni(diphenylacetylene).

Empirical formula	C59 H48 Ni O P2	
Formula weight	893.62	
Temperature	150(1) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	$\boldsymbol{P}\,2_1/n$	
Unit cell dimensions	a = 12.8793(3) Å	α= 90°.
	b = 18.2497(3) Å	β= 98.8516(10)°.
	c = 19.5980(5) Å	γ = 90°.
Volume	4551.52(17) $Å^3$	
Z	4	
Density (calculated)	1.304 Mg/m^3	
Absorption coefficient	0.539 mm^{-1}	
F(000)	1872	
Crystal size	0.250 x 0.230 x 0.130 mm^3	
Theta range for data collection	2.096 to 27.001°.	
Index ranges	-16<=h<=16, -23<=k<=22, -25<=l<=25	
Reflections collected	60117	
Independent reflections	9871 [R(int) = 0.1244]	
Completeness to theta = 25.242°	99.7 %	
Absorption correction	Multi-scan	
Max. and min. transmission	0.933 and 0.877	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	9871 / 0 / 568	
Goodness-of-fit on F^2	1.084	
Final R indices [I>2sigma(I)]	R1 = 0.0516, wR2 = 0.0755	
R indices (all data)	R1 = 0.1186, wR2 = 0.0950	
Extinction coefficient	n/a	
Largest diff. peak and hole	0.366 and -0.433 $e.Å^{-3}$	

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

	x	y	z	U(eq)
Ni(1)	412(1)	484(1)	3347(1)	15(1)
P(1)	1263(1)	1443(1)	3025(1)	15(1)
P(2)	1215(1)	-522(1)	3086(1)	14(1)
O(1)	2981(1)	433(1)	3066(1)	15(1)
C(1)	3361(2)	1067(2)	3405(2)	16(1)
C(2)	2643(2)	1634(2)	3393(2)	16(1)
C(3)	3015(2)	2288(2)	3712(2)	21(1)
C(4)	4050(2)	2339(2)	4039(2)	25(1)
C(5)	4731(3)	1749(2)	4054(2)	24(1)
C(6)	4402(2)	1094(2)	3727(2)	19(1)
C(7)	5091(2)	417(2)	3683(1)	19(1)
C(8)	4389(2)	-250(2)	3723(2)	16(1)
C(9)	3340(2)	-200(2)	3414(1)	15(1)
C(10)	2595(2)	-746(2)	3430(1)	15(1)
C(11)	2946(2)	-1406(2)	3748(1)	18(1)
C(12)	3991(2)	-1485(2)	4043(2)	20(1)
C(13)	4699(2)	-911(2)	4043(2)	21(1)
C(14)	5488(2)	434(2)	2976(2)	25(1)
C(15)	6041(2)	401(2)	4255(2)	27(1)
C(16)	1264(2)	1469(2)	2089(2)	18(1)
C(17)	2167(3)	1519(2)	1786(2)	26(1)
C(18)	2098(3)	1529(2)	1071(2)	33(1)
C(19)	1124(3)	1504(2)	659(2)	34(1)
C(20)	227(3)	1463(2)	954(2)	35(1)
C(21)	296(3)	1433(2)	1664(2)	28(1)
C(22)	706(2)	2344(2)	3178(2)	16(1)
C(23)	575(2)	2894(2)	2682(2)	24(1)
C(24)	208(3)	3578(2)	2835(2)	30(1)
C(25)	-23(3)	3725(2)	3485(2)	30(1)
C(26)	108(2)	3181(2)	3988(2)	26(1)
C(27)	463(2)	2495(2)	3834(2)	18(1)

C(28)	1196(2)	-553(2)	2153(1)	20(1)
C(29)	2068(3)	-702(2)	1843(2)	28(1)
C(30)	1984(3)	-713(2)	1130(2)	42(1)
C(31)	1040(4)	-581(2)	721(2)	47(1)
C(32)	163(3)	-443(2)	1023(2)	47(1)
C(33)	238(3)	-421(2)	1736(2)	35(1)
C(34)	626(2)	-1412(2)	3244(2)	17(1)
C(35)	416(2)	-1556(2)	3905(2)	22(1)
C(36)	76(3)	-2246(2)	4070(2)	33(1)
C(37)	-68(3)	-2794(2)	3579(2)	37(1)
C(38)	123(3)	-2650(2)	2915(2)	34(1)
C(39)	478(2)	-1964(2)	2747(2)	26(1)
C(40)	-1917(2)	1951(2)	3089(2)	24(1)
C(41)	-2567(3)	2538(2)	3168(2)	33(1)
C(42)	-2804(3)	2708(2)	3814(2)	36(1)
C(43)	-2398(3)	2286(2)	4379(2)	33(1)
C(44)	-1768(2)	1686(2)	4294(2)	25(1)
C(45)	-1516(2)	1509(2)	3647(2)	18(1)
C(46)	-864(2)	865(2)	3574(2)	18(1)
C(47)	-834(2)	171(2)	3663(2)	18(1)
C(48)	-1429(2)	-443(2)	3889(1)	19(1)
C(49)	-1441(2)	-564(2)	4594(2)	26(1)
C(50)	-2010(3)	-1134(2)	4814(2)	35(1)
C(51)	-2574(3)	-1599(2)	4344(2)	37(1)
C(52)	-2576(3)	-1490(2)	3646(2)	35(1)
C(53)	-2000(2)	-920(2)	3418(2)	24(1)
C(54)	6874(3)	1013(3)	535(2)	57(1)
C(55)	7348(3)	1082(2)	1211(2)	47(1)
C(56)	7569(3)	472(2)	1615(2)	46(1)
C(57)	7336(3)	-208(2)	1342(2)	55(1)
C(58)	6862(3)	-296(3)	671(2)	57(1)
C(59)	6625(3)	322(3)	269(2)	60(1)

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

Ni(1)-C(47)	1.895(3)
Ni(1)-C(46)	1.899(3)
Ni(1)-P(2)	2.2054(8)
Ni(1)-P(1)	2.2088(8)
P(1)-C(16)	1.835(3)
P(1)-C(22)	1.836(3)
P(1)-C(2)	1.846(3)
P(2)-C(28)	1.826(3)
P(2)-C(34)	1.838(3)
P(2)-C(10)	1.847(3)
O(1)-C(9)	1.385(3)
O(1)-C(1)	1.386(3)
C(1)-C(2)	1.385(4)
C(1)-C(6)	1.391(4)
C(2)-C(3)	1.397(4)
C(3)-C(4)	1.390(4)
C(3)-H(3)	0.9500
C(4)-C(5)	1.387(4)
C(4)-H(4)	0.9500
C(40)-C(41)	1.384(4)
C(40)-C(45)	1.392(4)
C(40)-H(40)	0.9500
C(54)-C(55)	1.376(5)
C(54)-C(59)	1.382(6)
C(54)-H(54)	0.9500
C(5)-C(6)	1.392(4)
C(5)-H(5)	0.9500
C(6)-C(7)	1.531(4)
C(7)-C(8)	1.527(4)
C(7)-C(15)	1.527(4)
C(7)-C(14)	1.549(4)
C(8)-C(13)	1.389(4)
C(8)-C(9)	1.396(4)
C(9)-C(10)	1.388(4)

C(10)-C(11)	1.399(4)
C(11)-C(12)	1.388(4)
C(11)-H(11)	0.9500
C(12)-C(13)	1.389(4)
C(12)-H(12)	0.9500
C(13)-H(13)	0.9500
C(14)-H(14A)	0.9800
C(14)-H(14B)	0.9800
C(14)-H(14C)	0.9800
C(15)-H(15A)	0.9800
C(15)-H(15B)	0.9800
C(15)-H(15C)	0.9800
C(16)-C(17)	1.387(4)
C(16)-C(21)	1.391(4)
C(17)-C(18)	1.391(4)
C(17)-H(17)	0.9500
C(18)-C(19)	1.385(5)
C(18)-H(18)	0.9500
C(19)-C(20)	1.371(5)
C(19)-H(19)	0.9500
C(20)-C(21)	1.381(4)
C(20)-H(20)	0.9500
C(21)-H(21)	0.9500
C(22)-C(23)	1.390(4)
C(22)-C(27)	1.397(4)
C(23)-C(24)	1.383(4)
C(23)-H(23)	0.9500
C(24)-C(25)	1.379(5)
C(24)-H(24)	0.9500
C(25)-C(26)	1.391(4)
C(25)-H(25)	0.9500
C(26)-C(27)	1.382(4)
C(26)-H(26)	0.9500
C(27)-H(27)	0.9500
C(28)-C(29)	1.385(4)
C(28)-C(33)	1.392(4)

C(29)-C(30)	1.385(4)
C(29)-H(29)	0.9500
C(30)-C(31)	1.371(5)
C(30)-H(30)	0.9500
C(31)-C(32)	1.377(6)
C(31)-H(31)	0.9500
C(32)-C(33)	1.387(5)
C(32)-H(32)	0.9500
C(33)-H(33)	0.9500
C(34)-C(35)	1.389(4)
C(34)-C(39)	1.393(4)
C(35)-C(36)	1.388(4)
C(35)-H(35)	0.9500
C(36)-C(37)	1.381(5)
C(36)-H(36)	0.9500
C(37)-C(38)	1.385(5)
C(37)-H(37)	0.9500
C(38)-C(39)	1.389(4)
C(38)-H(38)	0.9500
C(39)-H(39)	0.9500
C(41)-C(42)	1.382(5)
C(41)-H(41)	0.9500
C(42)-C(43)	1.383(5)
C(42)-H(42)	0.9500
C(43)-C(44)	1.388(4)
C(43)-H(43)	0.9500
C(44)-C(45)	1.395(4)
C(44)-H(44)	0.9500
C(45)-C(46)	1.464(4)
C(46)-C(47)	1.278(4)
C(47)-C(48)	1.464(4)
C(48)-C(53)	1.393(4)
C(48)-C(49)	1.402(4)
C(49)-C(50)	1.379(4)
C(49)-H(49)	0.9500
C(50)-C(51)	1.375(5)

C(50)-H(50)	0.9500
C(51)-C(52)	1.382(5)
C(51)-H(51)	0.9500
C(52)-C(53)	1.391(4)
C(52)-H(52)	0.9500
C(53)-H(53)	0.9500
C(55)-C(56)	1.371(5)
C(55)-H(55)	0.9500
C(56)-C(57)	1.367(6)
C(56)-H(56)	0.9500
C(57)-C(58)	1.372(5)
C(57)-H(57)	0.9500
C(58)-C(59)	1.382(6)
C(58)-H(58)	0.9500
C(59)-H(59)	0.9500

C(47)-Ni(1)-C(46)	39.36(12)
C(47)-Ni(1)-P(2)	106.03(9)
C(46)-Ni(1)-P(2)	144.36(9)
C(47)-Ni(1)-P(1)	144.54(9)
C(46)-Ni(1)-P(1)	105.17(9)
P(2)-Ni(1)-P(1)	108.83(3)
C(16)-P(1)-C(22)	101.55(13)
C(16)-P(1)-C(2)	103.74(13)
C(22)-P(1)-C(2)	98.29(13)
C(16)-P(1)-Ni(1)	112.52(10)
C(22)-P(1)-Ni(1)	116.03(9)
C(2)-P(1)-Ni(1)	121.90(9)
C(28)-P(2)-C(34)	101.47(13)
C(28)-P(2)-C(10)	102.87(13)
C(34)-P(2)-C(10)	98.21(13)
C(28)-P(2)-Ni(1)	108.80(10)
C(34)-P(2)-Ni(1)	118.44(9)
C(10)-P(2)-Ni(1)	123.96(9)
C(9)-O(1)-C(1)	113.15(19)
C(2)-C(1)-O(1)	115.4(2)

C(2)-C(1)-C(6)	125.2(3)
O(1)-C(1)-C(6)	119.4(2)
C(1)-C(2)-C(3)	116.6(3)
C(1)-C(2)-P(1)	117.7(2)
C(3)-C(2)-P(1)	125.4(2)
C(4)-C(3)-C(2)	120.1(3)
C(4)-C(3)-H(3)	119.9
C(2)-C(3)-H(3)	119.9
C(5)-C(4)-C(3)	121.1(3)
C(5)-C(4)-H(4)	119.4
C(3)-C(4)-H(4)	119.4
C(41)-C(40)-C(45)	121.2(3)
C(41)-C(40)-H(40)	119.4
C(45)-C(40)-H(40)	119.4
C(55)-C(54)-C(59)	119.3(4)
C(55)-C(54)-H(54)	120.4
C(59)-C(54)-H(54)	120.4
C(4)-C(5)-C(6)	120.6(3)
C(4)-C(5)-H(5)	119.7
C(6)-C(5)-H(5)	119.7
C(1)-C(6)-C(5)	116.3(3)
C(1)-C(6)-C(7)	118.2(3)
C(5)-C(6)-C(7)	125.5(3)
C(8)-C(7)-C(15)	111.2(2)
C(8)-C(7)-C(6)	106.7(2)
C(15)-C(7)-C(6)	112.3(2)
C(8)-C(7)-C(14)	110.0(2)
C(15)-C(7)-C(14)	108.6(2)
C(6)-C(7)-C(14)	108.0(2)
C(13)-C(8)-C(9)	116.4(3)
C(13)-C(8)-C(7)	125.8(3)
C(9)-C(8)-C(7)	117.8(2)
O(1)-C(9)-C(10)	115.5(2)
O(1)-C(9)-C(8)	119.8(2)
C(10)-C(9)-C(8)	124.7(3)
C(9)-C(10)-C(11)	116.8(3)

C(9)-C(10)-P(2)	117.6(2)
C(11)-C(10)-P(2)	125.4(2)
C(12)-C(11)-C(10)	120.1(3)
C(12)-C(11)-H(11)	120.0
C(10)-C(11)-H(11)	120.0
C(11)-C(12)-C(13)	121.1(3)
C(11)-C(12)-H(12)	119.5
C(13)-C(12)-H(12)	119.5
C(8)-C(13)-C(12)	120.8(3)
C(8)-C(13)-H(13)	119.6
C(12)-C(13)-H(13)	119.6
C(7)-C(14)-H(14A)	109.5
C(7)-C(14)-H(14B)	109.5
H(14A)-C(14)-H(14B)	109.5
C(7)-C(14)-H(14C)	109.5
H(14A)-C(14)-H(14C)	109.5
H(14B)-C(14)-H(14C)	109.5
C(7)-C(15)-H(15A)	109.5
C(7)-C(15)-H(15B)	109.5
H(15A)-C(15)-H(15B)	109.5
C(7)-C(15)-H(15C)	109.5
H(15A)-C(15)-H(15C)	109.5
H(15B)-C(15)-H(15C)	109.5
C(17)-C(16)-C(21)	118.7(3)
C(17)-C(16)-P(1)	124.0(2)
C(21)-C(16)-P(1)	117.3(2)
C(16)-C(17)-C(18)	120.3(3)
C(16)-C(17)-H(17)	119.9
C(18)-C(17)-H(17)	119.9
C(19)-C(18)-C(17)	119.9(3)
C(19)-C(18)-H(18)	120.0
C(17)-C(18)-H(18)	120.0
C(20)-C(19)-C(18)	120.1(3)
C(20)-C(19)-H(19)	119.9
C(18)-C(19)-H(19)	119.9
C(19)-C(20)-C(21)	120.0(3)

C(19)-C(20)-H(20)	120.0
C(21)-C(20)-H(20)	120.0
C(20)-C(21)-C(16)	120.9(3)
C(20)-C(21)-H(21)	119.5
C(16)-C(21)-H(21)	119.5
C(23)-C(22)-C(27)	118.7(3)
C(23)-C(22)-P(1)	122.8(2)
C(27)-C(22)-P(1)	118.4(2)
C(24)-C(23)-C(22)	120.7(3)
C(24)-C(23)-H(23)	119.7
C(22)-C(23)-H(23)	119.7
C(25)-C(24)-C(23)	120.3(3)
C(25)-C(24)-H(24)	119.8
C(23)-C(24)-H(24)	119.8
C(24)-C(25)-C(26)	119.8(3)
C(24)-C(25)-H(25)	120.1
C(26)-C(25)-H(25)	120.1
C(27)-C(26)-C(25)	120.0(3)
C(27)-C(26)-H(26)	120.0
C(25)-C(26)-H(26)	120.0
C(26)-C(27)-C(22)	120.6(3)
C(26)-C(27)-H(27)	119.7
C(22)-C(27)-H(27)	119.7
C(29)-C(28)-C(33)	118.8(3)
C(29)-C(28)-P(2)	124.0(2)
C(33)-C(28)-P(2)	117.2(2)
C(30)-C(29)-C(28)	120.2(3)
C(30)-C(29)-H(29)	119.9
C(28)-C(29)-H(29)	119.9
C(31)-C(30)-C(29)	120.8(4)
C(31)-C(30)-H(30)	119.6
C(29)-C(30)-H(30)	119.6
C(30)-C(31)-C(32)	119.5(3)
C(30)-C(31)-H(31)	120.2
C(32)-C(31)-H(31)	120.2
C(31)-C(32)-C(33)	120.3(3)

C(31)-C(32)-H(32)	119.9
C(33)-C(32)-H(32)	119.9
C(32)-C(33)-C(28)	120.3(3)
C(32)-C(33)-H(33)	119.8
C(28)-C(33)-H(33)	119.8
C(35)-C(34)-C(39)	119.3(3)
C(35)-C(34)-P(2)	117.9(2)
C(39)-C(34)-P(2)	122.5(2)
C(36)-C(35)-C(34)	120.3(3)
C(36)-C(35)-H(35)	119.9
C(34)-C(35)-H(35)	119.9
C(37)-C(36)-C(35)	120.5(3)
C(37)-C(36)-H(36)	119.8
C(35)-C(36)-H(36)	119.8
C(36)-C(37)-C(38)	119.6(3)
C(36)-C(37)-H(37)	120.2
C(38)-C(37)-H(37)	120.2
C(37)-C(38)-C(39)	120.4(3)
C(37)-C(38)-H(38)	119.8
C(39)-C(38)-H(38)	119.8
C(38)-C(39)-C(34)	120.0(3)
C(38)-C(39)-H(39)	120.0
C(34)-C(39)-H(39)	120.0
C(42)-C(41)-C(40)	120.0(3)
C(42)-C(41)-H(41)	120.0
C(40)-C(41)-H(41)	120.0
C(41)-C(42)-C(43)	119.9(3)
C(41)-C(42)-H(42)	120.1
C(43)-C(42)-H(42)	120.1
C(42)-C(43)-C(44)	120.0(3)
C(42)-C(43)-H(43)	120.0
C(44)-C(43)-H(43)	120.0
C(43)-C(44)-C(45)	120.9(3)
C(43)-C(44)-H(44)	119.5
C(45)-C(44)-H(44)	119.5
C(40)-C(45)-C(44)	118.0(3)

C(40)-C(45)-C(46)	122.5(3)
C(44)-C(45)-C(46)	119.5(3)
C(47)-C(46)-C(45)	141.9(3)
C(47)-C(46)-Ni(1)	70.15(19)
C(45)-C(46)-Ni(1)	147.9(2)
C(46)-C(47)-C(48)	142.6(3)
C(46)-C(47)-Ni(1)	70.5(2)
C(48)-C(47)-Ni(1)	146.9(2)
C(53)-C(48)-C(49)	118.0(3)
C(53)-C(48)-C(47)	121.7(3)
C(49)-C(48)-C(47)	120.3(3)
C(50)-C(49)-C(48)	120.9(3)
C(50)-C(49)-H(49)	119.6
C(48)-C(49)-H(49)	119.6
C(51)-C(50)-C(49)	120.6(3)
C(51)-C(50)-H(50)	119.7
C(49)-C(50)-H(50)	119.7
C(50)-C(51)-C(52)	119.6(3)
C(50)-C(51)-H(51)	120.2
C(52)-C(51)-H(51)	120.2
C(51)-C(52)-C(53)	120.3(3)
C(51)-C(52)-H(52)	119.8
C(53)-C(52)-H(52)	119.8
C(52)-C(53)-C(48)	120.6(3)
C(52)-C(53)-H(53)	119.7
C(48)-C(53)-H(53)	119.7
C(56)-C(55)-C(54)	120.3(4)
C(56)-C(55)-H(55)	119.9
C(54)-C(55)-H(55)	119.9
C(57)-C(56)-C(55)	119.9(4)
C(57)-C(56)-H(56)	120.1
C(55)-C(56)-H(56)	120.1
C(56)-C(57)-C(58)	121.2(4)
C(56)-C(57)-H(57)	119.4
C(58)-C(57)-H(57)	119.4
C(57)-C(58)-C(59)	118.6(4)

C(57)-C(58)-H(58)	120.7
C(59)-C(58)-H(58)	120.7
C(54)-C(59)-C(58)	120.7(4)
C(54)-C(59)-H(59)	119.6
C(58)-C(59)-H(59)	119.6

Symmetry transformations used to generate equivalent atoms:

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

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Ni(1)	14(1)	14(1)	17(1)	2(1)	3(1)	1(1)
P(1)	14(1)	14(1)	16(1)	2(1)	3(1)	1(1)
P(2)	13(1)	14(1)	15(1)	0(1)	2(1)	0(1)
O(1)	16(1)	12(1)	18(1)	1(1)	1(1)	1(1)
C(1)	17(2)	16(2)	15(2)	0(1)	5(1)	-3(1)
C(2)	17(2)	16(2)	14(2)	3(1)	3(1)	-2(1)
C(3)	20(2)	17(2)	27(2)	1(1)	5(1)	2(1)
C(4)	25(2)	16(2)	33(2)	-8(1)	2(2)	-4(1)
C(5)	19(2)	23(2)	27(2)	-3(1)	-2(1)	-1(1)
C(6)	17(2)	19(2)	21(2)	0(1)	5(1)	-1(1)
C(7)	14(1)	18(2)	23(2)	-1(1)	2(1)	1(1)
C(8)	12(2)	19(2)	16(2)	-4(1)	1(1)	-1(1)
C(9)	20(2)	14(1)	13(2)	-2(1)	4(1)	4(1)
C(10)	18(2)	14(1)	12(2)	-1(1)	3(1)	2(1)
C(11)	19(2)	16(2)	18(2)	0(1)	4(1)	0(1)
C(12)	22(2)	14(2)	23(2)	2(1)	0(1)	4(1)
C(13)	16(2)	23(2)	22(2)	-1(1)	0(1)	7(1)
C(14)	21(2)	20(2)	37(2)	1(2)	11(1)	1(1)
C(15)	14(2)	26(2)	38(2)	-3(2)	-3(1)	1(1)
C(16)	24(2)	12(2)	19(2)	2(1)	3(1)	-1(1)
C(17)	28(2)	28(2)	24(2)	5(1)	7(2)	5(2)
C(18)	41(2)	33(2)	29(2)	6(2)	18(2)	1(2)
C(19)	60(3)	25(2)	18(2)	-1(1)	9(2)	-4(2)
C(20)	43(2)	31(2)	26(2)	1(2)	-6(2)	-12(2)
C(21)	28(2)	30(2)	24(2)	1(2)	2(2)	-10(2)
C(22)	10(1)	16(2)	24(2)	0(1)	3(1)	-2(1)
C(23)	25(2)	22(2)	26(2)	4(1)	11(1)	3(1)
C(24)	32(2)	21(2)	38(2)	11(2)	11(2)	5(2)
C(25)	28(2)	19(2)	46(2)	-2(2)	11(2)	6(2)
C(26)	26(2)	27(2)	24(2)	-4(1)	8(2)	2(1)
C(27)	17(2)	14(2)	24(2)	1(1)	1(1)	0(1)

C(28)	28(2)	12(2)	18(2)	0(1)	-1(1)	-2(1)
C(29)	35(2)	28(2)	20(2)	-3(1)	7(2)	-8(2)
C(30)	66(3)	36(2)	26(2)	-6(2)	19(2)	-18(2)
C(31)	102(4)	23(2)	13(2)	0(2)	-2(2)	-10(2)
C(32)	73(3)	29(2)	28(2)	-2(2)	-24(2)	10(2)
C(33)	43(2)	27(2)	29(2)	-4(2)	-9(2)	11(2)
C(34)	15(2)	13(1)	23(2)	1(1)	-1(1)	2(1)
C(35)	18(2)	26(2)	23(2)	4(1)	3(1)	5(1)
C(36)	25(2)	30(2)	45(2)	19(2)	14(2)	6(2)
C(37)	24(2)	19(2)	69(3)	12(2)	9(2)	-3(2)
C(38)	30(2)	18(2)	54(3)	-7(2)	4(2)	-6(2)
C(39)	22(2)	21(2)	34(2)	-1(1)	1(2)	-3(1)
C(40)	23(2)	24(2)	25(2)	-1(1)	2(1)	2(1)
C(41)	27(2)	29(2)	42(2)	6(2)	3(2)	7(2)
C(42)	26(2)	24(2)	58(3)	-3(2)	13(2)	7(2)
C(43)	32(2)	29(2)	42(2)	-10(2)	17(2)	-2(2)
C(44)	24(2)	23(2)	29(2)	-2(1)	5(2)	-2(1)
C(45)	12(2)	16(2)	24(2)	-2(1)	3(1)	-1(1)
C(46)	17(2)	21(2)	18(2)	0(1)	3(1)	0(1)
C(47)	14(2)	23(2)	17(2)	1(1)	1(1)	0(1)
C(48)	13(1)	20(2)	25(2)	4(1)	4(1)	5(1)
C(49)	19(2)	32(2)	26(2)	8(2)	5(1)	5(2)
C(50)	30(2)	42(2)	35(2)	22(2)	15(2)	13(2)
C(51)	26(2)	29(2)	61(3)	23(2)	20(2)	4(2)
C(52)	28(2)	24(2)	54(3)	0(2)	9(2)	-5(2)
C(53)	23(2)	22(2)	28(2)	3(1)	7(1)	1(1)
C(54)	52(3)	75(3)	43(3)	11(2)	10(2)	7(2)
C(55)	37(2)	56(3)	47(3)	-3(2)	2(2)	15(2)
C(56)	32(2)	57(2)	45(2)	0(2)	-6(2)	1(2)
C(57)	50(3)	58(3)	52(3)	7(2)	-3(2)	-12(2)
C(58)	56(3)	75(3)	43(3)	-10(2)	16(2)	-30(2)
C(59)	56(3)	101(4)	25(2)	1(2)	14(2)	-22(3)

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

	x	y	z	U(eq)
H(3)	2560	2699	3705	25
H(4)	4294	2786	4255	30
H(40)	-1740	1848	2646	29
H(54)	6719	1435	254	68
H(5)	5430	1792	4291	28
H(11)	2469	-1801	3762	21
H(12)	4226	-1939	4249	24
H(13)	5404	-971	4264	25
H(14A)	5938	864	2954	30
H(14B)	5891	-12	2922	30
H(14C)	4886	461	2604	30
H(15A)	6478	835	4217	32
H(15B)	5801	401	4706	32
H(15C)	6454	-42	4209	32
H(17)	2835	1547	2068	32
H(18)	2719	1552	865	40
H(19)	1076	1515	170	41
H(20)	-441	1455	671	42
H(21)	-327	1388	1865	34
H(23)	739	2800	2234	28
H(24)	114	3948	2490	36
H(25)	-270	4196	3589	36
H(26)	-48	3280	4437	31
H(27)	543	2123	4177	22
H(29)	2726	-798	2120	33
H(30)	2589	-813	922	50
H(31)	991	-585	232	57
H(32)	-496	-362	742	56
H(33)	-368	-317	1941	42
H(35)	507	-1181	4245	27

H(36)	-59	-2342	4525	39
H(37)	-297	-3266	3695	45
H(38)	10	-3023	2573	41
H(39)	620	-1872	2294	31
H(41)	-2851	2824	2779	39
H(42)	-3245	3114	3870	43
H(43)	-2551	2407	4824	40
H(44)	-1504	1393	4682	31
H(49)	-1052	-248	4926	31
H(50)	-2011	-1207	5294	42
H(51)	-2961	-1993	4499	45
H(52)	-2973	-1807	3320	42
H(53)	-1996	-855	2938	29
H(55)	7524	1554	1397	57
H(56)	7882	522	2084	55
H(57)	7505	-629	1623	66
H(58)	6701	-770	486	68
H(59)	6287	272	-195	72

Table 2f. Torsion angles [°] for **2**.

C(9)-O(1)-C(1)-C(2)	-139.8(3)
C(9)-O(1)-C(1)-C(6)	40.1(3)
O(1)-C(1)-C(2)-C(3)	-177.9(2)
C(6)-C(1)-C(2)-C(3)	2.3(4)
O(1)-C(1)-C(2)-P(1)	6.9(3)
C(6)-C(1)-C(2)-P(1)	-172.9(2)
C(16)-P(1)-C(2)-C(1)	-78.1(2)
C(22)-P(1)-C(2)-C(1)	177.8(2)
Ni(1)-P(1)-C(2)-C(1)	50.0(3)
C(16)-P(1)-C(2)-C(3)	107.2(3)
C(22)-P(1)-C(2)-C(3)	3.1(3)
Ni(1)-P(1)-C(2)-C(3)	-124.8(2)
C(1)-C(2)-C(3)-C(4)	-2.0(4)
P(1)-C(2)-C(3)-C(4)	172.7(2)
C(2)-C(3)-C(4)-C(5)	0.3(5)
C(3)-C(4)-C(5)-C(6)	1.5(5)
C(2)-C(1)-C(6)-C(5)	-0.6(5)
O(1)-C(1)-C(6)-C(5)	179.6(2)
C(2)-C(1)-C(6)-C(7)	-179.4(3)
O(1)-C(1)-C(6)-C(7)	0.8(4)
C(4)-C(5)-C(6)-C(1)	-1.4(5)
C(4)-C(5)-C(6)-C(7)	177.3(3)
C(1)-C(6)-C(7)-C(8)	-36.7(3)
C(5)-C(6)-C(7)-C(8)	144.7(3)
C(1)-C(6)-C(7)-C(15)	-158.7(3)
C(5)-C(6)-C(7)-C(15)	22.6(4)
C(1)-C(6)-C(7)-C(14)	81.5(3)
C(5)-C(6)-C(7)-C(14)	-97.2(3)
C(15)-C(7)-C(8)-C(13)	-22.1(4)
C(6)-C(7)-C(8)-C(13)	-144.9(3)
C(14)-C(7)-C(8)-C(13)	98.3(3)
C(15)-C(7)-C(8)-C(9)	157.8(3)
C(6)-C(7)-C(8)-C(9)	35.0(3)
C(14)-C(7)-C(8)-C(9)	-81.8(3)

C(1)-O(1)-C(9)-C(10)	137.8(3)
C(1)-O(1)-C(9)-C(8)	-41.8(3)
C(13)-C(8)-C(9)-O(1)	-177.9(2)
C(7)-C(8)-C(9)-O(1)	2.2(4)
C(13)-C(8)-C(9)-C(10)	2.6(4)
C(7)-C(8)-C(9)-C(10)	-177.3(3)
O(1)-C(9)-C(10)-C(11)	176.8(2)
C(8)-C(9)-C(10)-C(11)	-3.6(4)
O(1)-C(9)-C(10)-P(2)	-7.2(3)
C(8)-C(9)-C(10)-P(2)	172.3(2)
C(28)-P(2)-C(10)-C(9)	76.5(2)
C(34)-P(2)-C(10)-C(9)	-179.6(2)
Ni(1)-P(2)-C(10)-C(9)	-47.0(3)
C(28)-P(2)-C(10)-C(11)	-107.9(3)
C(34)-P(2)-C(10)-C(11)	-4.1(3)
Ni(1)-P(2)-C(10)-C(11)	128.5(2)
C(9)-C(10)-C(11)-C(12)	1.5(4)
P(2)-C(10)-C(11)-C(12)	-174.0(2)
C(10)-C(11)-C(12)-C(13)	1.4(4)
C(9)-C(8)-C(13)-C(12)	0.6(4)
C(7)-C(8)-C(13)-C(12)	-179.6(3)
C(11)-C(12)-C(13)-C(8)	-2.5(5)
C(22)-P(1)-C(16)-C(17)	109.7(3)
C(2)-P(1)-C(16)-C(17)	8.1(3)
Ni(1)-P(1)-C(16)-C(17)	-125.6(2)
C(22)-P(1)-C(16)-C(21)	-70.6(3)
C(2)-P(1)-C(16)-C(21)	-172.2(2)
Ni(1)-P(1)-C(16)-C(21)	54.1(3)
C(21)-C(16)-C(17)-C(18)	-0.3(5)
P(1)-C(16)-C(17)-C(18)	179.4(2)
C(16)-C(17)-C(18)-C(19)	1.3(5)
C(17)-C(18)-C(19)-C(20)	-0.6(5)
C(18)-C(19)-C(20)-C(21)	-1.1(5)
C(19)-C(20)-C(21)-C(16)	2.2(5)
C(17)-C(16)-C(21)-C(20)	-1.4(5)
P(1)-C(16)-C(21)-C(20)	178.8(2)

C(16)-P(1)-C(22)-C(23)	-11.5(3)
C(2)-P(1)-C(22)-C(23)	94.5(3)
Ni(1)-P(1)-C(22)-C(23)	-133.8(2)
C(16)-P(1)-C(22)-C(27)	172.5(2)
C(2)-P(1)-C(22)-C(27)	-81.6(2)
Ni(1)-P(1)-C(22)-C(27)	50.2(3)
C(27)-C(22)-C(23)-C(24)	-0.2(5)
P(1)-C(22)-C(23)-C(24)	-176.2(2)
C(22)-C(23)-C(24)-C(25)	0.7(5)
C(23)-C(24)-C(25)-C(26)	-0.5(5)
C(24)-C(25)-C(26)-C(27)	-0.3(5)
C(25)-C(26)-C(27)-C(22)	0.7(5)
C(23)-C(22)-C(27)-C(26)	-0.5(4)
P(1)-C(22)-C(27)-C(26)	175.7(2)
C(34)-P(2)-C(28)-C(29)	-102.2(3)
C(10)-P(2)-C(28)-C(29)	-0.9(3)
Ni(1)-P(2)-C(28)-C(29)	132.2(2)
C(34)-P(2)-C(28)-C(33)	77.2(3)
C(10)-P(2)-C(28)-C(33)	178.5(2)
Ni(1)-P(2)-C(28)-C(33)	-48.4(3)
C(33)-C(28)-C(29)-C(30)	0.7(5)
P(2)-C(28)-C(29)-C(30)	-179.9(2)
C(28)-C(29)-C(30)-C(31)	-0.5(5)
C(29)-C(30)-C(31)-C(32)	-0.5(5)
C(30)-C(31)-C(32)-C(33)	1.3(6)
C(31)-C(32)-C(33)-C(28)	-1.1(5)
C(29)-C(28)-C(33)-C(32)	0.1(5)
P(2)-C(28)-C(33)-C(32)	-179.4(3)
C(28)-P(2)-C(34)-C(35)	-173.9(2)
C(10)-P(2)-C(34)-C(35)	81.1(2)
Ni(1)-P(2)-C(34)-C(35)	-55.0(3)
C(28)-P(2)-C(34)-C(39)	12.4(3)
C(10)-P(2)-C(34)-C(39)	-92.6(3)
Ni(1)-P(2)-C(34)-C(39)	131.3(2)
C(39)-C(34)-C(35)-C(36)	0.8(4)
P(2)-C(34)-C(35)-C(36)	-173.2(2)

C(34)-C(35)-C(36)-C(37)	-0.6(5)
C(35)-C(36)-C(37)-C(38)	-0.4(5)
C(36)-C(37)-C(38)-C(39)	1.3(5)
C(37)-C(38)-C(39)-C(34)	-1.2(5)
C(35)-C(34)-C(39)-C(38)	0.1(5)
P(2)-C(34)-C(39)-C(38)	173.8(2)
C(45)-C(40)-C(41)-C(42)	2.0(5)
C(40)-C(41)-C(42)-C(43)	-0.6(5)
C(41)-C(42)-C(43)-C(44)	-1.0(5)
C(42)-C(43)-C(44)-C(45)	1.3(5)
C(41)-C(40)-C(45)-C(44)	-1.6(5)
C(41)-C(40)-C(45)-C(46)	177.3(3)
C(43)-C(44)-C(45)-C(40)	0.0(4)
C(43)-C(44)-C(45)-C(46)	-179.0(3)
C(40)-C(45)-C(46)-C(47)	-119.8(5)
C(44)-C(45)-C(46)-C(47)	59.1(6)
C(40)-C(45)-C(46)-Ni(1)	63.9(5)
C(44)-C(45)-C(46)-Ni(1)	-117.2(4)
P(2)-Ni(1)-C(46)-C(47)	17.6(3)
P(1)-Ni(1)-C(46)-C(47)	179.83(17)
C(47)-Ni(1)-C(46)-C(45)	177.6(6)
P(2)-Ni(1)-C(46)-C(45)	-164.8(3)
P(1)-Ni(1)-C(46)-C(45)	-2.6(4)
C(45)-C(46)-C(47)-C(48)	0.7(8)
Ni(1)-C(46)-C(47)-C(48)	178.7(5)
C(45)-C(46)-C(47)-Ni(1)	-177.9(5)
P(2)-Ni(1)-C(47)-C(46)	-169.44(17)
P(1)-Ni(1)-C(47)-C(46)	-0.3(3)
C(46)-Ni(1)-C(47)-C(48)	-178.5(5)
P(2)-Ni(1)-C(47)-C(48)	12.0(4)
P(1)-Ni(1)-C(47)-C(48)	-178.8(3)
C(46)-C(47)-C(48)-C(53)	102.3(5)
Ni(1)-C(47)-C(48)-C(53)	-80.0(5)
C(46)-C(47)-C(48)-C(49)	-77.4(6)
Ni(1)-C(47)-C(48)-C(49)	100.4(4)
C(53)-C(48)-C(49)-C(50)	-0.6(4)

C(47)-C(48)-C(49)-C(50)	179.1(3)
C(48)-C(49)-C(50)-C(51)	0.3(5)
C(49)-C(50)-C(51)-C(52)	-0.4(5)
C(50)-C(51)-C(52)-C(53)	0.8(5)
C(51)-C(52)-C(53)-C(48)	-1.2(5)
C(49)-C(48)-C(53)-C(52)	1.0(4)
C(47)-C(48)-C(53)-C(52)	-178.7(3)
C(59)-C(54)-C(55)-C(56)	-0.1(6)
C(54)-C(55)-C(56)-C(57)	1.3(6)
C(55)-C(56)-C(57)-C(58)	-1.4(6)
C(56)-C(57)-C(58)-C(59)	0.2(6)
C(55)-C(54)-C(59)-C(58)	-1.0(6)
C(57)-C(58)-C(59)-C(54)	1.0(6)

Symmetry transformations used to generate equivalent atoms:

Table 3a. Crystal data and structure refinement for (Xant)Ni(dimethylfumarate).

Empirical formula	C48 H43 Ni O5 P2	
Formula weight	820.47	
Temperature	150(1) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P -1	
Unit cell dimensions	a = 11.8270(2) Å	α= 73.8372(9)°.
	b = 12.1552(2) Å	β= 84.9154(12)°.
	c = 14.8937(3) Å	γ = 76.8383(11)°.
Volume	2001.73(6) $Å^3$	
Z	2	
Density (calculated)	1.361 Mg/m^3	
Absorption coefficient	0.613 mm^{-1}	
F(000)	858	
Crystal size	0.280 x 0.250 x 0.100 mm^3	
Theta range for data collection	2.231 to 27.518°.	
Index ranges	-15<=h<=15, -15<=k<=15, -19<=l<=19	
Reflections collected	17370	
Independent reflections	9192 [R(int) = 0.0291]	
Completeness to theta = 25.242°	99.9 %	
Absorption correction	Multi-scan	
Max. and min. transmission	0.941 and 0.847	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	9192 / 0 / 505	
Goodness-of-fit on F^2	1.036	
Final R indices [I>2sigma(I)]	R1 = 0.0414, wR2 = 0.0971	
R indices (all data)	R1 = 0.0665, wR2 = 0.1088	
Extinction coefficient	n/a	
Largest diff. peak and hole	0.539 and -0.650 $e.Å^{-3}$	

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

	x	y	z	U(eq)
Ni(1)	8133(1)	1421(1)	2540(1)	19(1)
P(1)	6976(1)	2660(1)	1447(1)	19(1)
P(2)	8172(1)	-421(1)	2599(1)	19(1)
O(1)	7121(1)	545(1)	814(1)	20(1)
O(2)	8653(1)	-207(1)	5026(1)	28(1)
O(3)	7119(1)	1310(2)	4688(1)	30(1)
O(4)	9063(2)	4298(2)	2814(1)	40(1)
O(5)	10328(2)	2772(2)	2469(1)	41(1)
C(1)	7049(2)	1516(2)	45(2)	21(1)
C(2)	6875(2)	2599(2)	234(2)	22(1)
C(3)	6753(2)	3583(2)	-546(2)	25(1)
C(4)	6865(2)	3449(2)	-1446(2)	28(1)
C(5)	7112(2)	2340(2)	-1599(2)	26(1)
C(6)	7205(2)	1344(2)	-848(2)	23(1)
C(7)	7506(2)	74(2)	-910(2)	26(1)
C(8)	8339(2)	-597(2)	-118(2)	22(1)
C(9)	8064(2)	-353(2)	739(2)	20(1)
C(10)	8678(2)	-927(2)	1551(2)	21(1)
C(11)	9663(2)	-1786(2)	1478(2)	24(1)
C(12)	10000(2)	-2022(2)	621(2)	26(1)
C(13)	9347(2)	-1443(2)	-170(2)	26(1)
C(14)	6387(2)	-418(2)	-740(2)	32(1)
C(15)	8048(2)	-38(2)	-1859(2)	36(1)
C(16)	5478(2)	2764(2)	1905(2)	23(1)
C(17)	5280(2)	2613(2)	2865(2)	29(1)
C(18)	4160(2)	2726(2)	3240(2)	37(1)
C(19)	3217(2)	2985(2)	2667(2)	36(1)
C(20)	3408(2)	3128(2)	1717(2)	34(1)
C(21)	4527(2)	3017(2)	1335(2)	28(1)
C(22)	7157(2)	4183(2)	1197(2)	21(1)
C(23)	8226(2)	4423(2)	808(2)	27(1)

C(24)	8403(2)	5556(2)	572(2)	31(1)
C(25)	7529(2)	6459(2)	740(2)	31(1)
C(26)	6473(2)	6228(2)	1118(2)	31(1)
C(27)	6284(2)	5092(2)	1347(2)	26(1)
C(28)	6754(2)	-819(2)	2877(2)	23(1)
C(29)	6077(2)	-403(2)	3573(2)	29(1)
C(30)	5076(2)	-802(2)	3923(2)	34(1)
C(31)	4733(2)	-1604(2)	3572(2)	39(1)
C(32)	5385(2)	-2008(3)	2864(2)	41(1)
C(33)	6392(2)	-1619(2)	2517(2)	31(1)
C(34)	9057(2)	-1588(2)	3497(2)	24(1)
C(35)	8647(2)	-2570(2)	4024(2)	37(1)
C(36)	9344(3)	-3419(3)	4695(2)	51(1)
C(37)	10448(3)	-3321(2)	4832(2)	46(1)
C(38)	10866(2)	-2366(2)	4309(2)	36(1)
C(39)	10160(2)	-1492(2)	3657(2)	27(1)
C(40)	7962(2)	-811(2)	5763(2)	36(1)
C(41)	8104(2)	845(2)	4508(2)	24(1)
C(42)	8843(2)	1327(2)	3715(2)	23(1)
C(43)	8479(2)	2522(2)	3201(2)	24(1)
C(44)	9387(2)	3168(2)	2780(2)	30(1)
C(45)	9932(3)	4996(3)	2498(2)	60(1)
C(46)	5243(17)	5028(7)	5791(8)	255(8)
C(47)	4029(11)	5173(7)	5455(7)	196(5)
C(48)	3675(14)	4968(13)	4713(15)	357(14)

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

Ni(1)-C(42)	1.972(2)
Ni(1)-C(43)	1.997(2)
Ni(1)-P(2)	2.2056(6)
Ni(1)-P(1)	2.2097(6)
P(1)-C(16)	1.831(2)
P(1)-C(22)	1.841(2)
P(1)-C(2)	1.845(2)
P(2)-C(28)	1.831(2)
P(2)-C(10)	1.837(2)
P(2)-C(34)	1.847(2)
O(1)-C(1)	1.389(2)
O(1)-C(9)	1.393(2)
O(2)-C(41)	1.347(3)
O(2)-C(40)	1.441(3)
O(3)-C(41)	1.216(3)
O(4)-C(44)	1.353(3)
O(4)-C(45)	1.446(3)
O(5)-C(44)	1.213(3)
C(1)-C(2)	1.388(3)
C(1)-C(6)	1.393(3)
C(2)-C(3)	1.406(3)
C(3)-C(4)	1.386(3)
C(3)-H(3)	0.9500
C(4)-C(5)	1.391(3)
C(4)-H(4)	0.9500
C(5)-C(6)	1.392(3)
C(5)-H(5)	0.9500
C(6)-C(7)	1.531(3)
C(7)-C(15)	1.527(3)
C(7)-C(8)	1.527(3)
C(7)-C(14)	1.546(3)
C(8)-C(9)	1.384(3)
C(8)-C(13)	1.398(3)
C(9)-C(10)	1.389(3)

C(10)-C(11)	1.395(3)
C(11)-C(12)	1.390(3)
C(11)-H(11)	0.9500
C(12)-C(13)	1.390(3)
C(12)-H(12)	0.9500
C(13)-H(13)	0.9500
C(14)-H(14A)	0.9800
C(14)-H(14B)	0.9800
C(14)-H(14C)	0.9800
C(15)-H(15A)	0.9800
C(15)-H(15B)	0.9800
C(15)-H(15C)	0.9800
C(16)-C(21)	1.395(3)
C(16)-C(17)	1.396(3)
C(17)-C(18)	1.384(3)
C(17)-H(17)	0.9500
C(18)-C(19)	1.388(4)
C(18)-H(18)	0.9500
C(19)-C(20)	1.382(4)
C(19)-H(19)	0.9500
C(20)-C(21)	1.387(3)
C(20)-H(20)	0.9500
C(21)-H(21)	0.9500
C(22)-C(27)	1.384(3)
C(22)-C(23)	1.402(3)
C(23)-C(24)	1.382(3)
C(23)-H(23)	0.9500
C(24)-C(25)	1.388(3)
C(24)-H(24)	0.9500
C(25)-C(26)	1.380(3)
C(25)-H(25)	0.9500
C(26)-C(27)	1.392(3)
C(26)-H(26)	0.9500
C(27)-H(27)	0.9500
C(28)-C(29)	1.390(3)
C(28)-C(33)	1.391(3)

C(29)-C(30)	1.385(3)
C(29)-H(29)	0.9500
C(30)-C(31)	1.375(4)
C(30)-H(30)	0.9500
C(31)-C(32)	1.385(4)
C(31)-H(31)	0.9500
C(32)-C(33)	1.386(3)
C(32)-H(32)	0.9500
C(33)-H(33)	0.9500
C(34)-C(39)	1.383(3)
C(34)-C(35)	1.396(3)
C(35)-C(36)	1.388(4)
C(35)-H(35)	0.9500
C(36)-C(37)	1.375(4)
C(36)-H(36)	0.9500
C(37)-C(38)	1.375(4)
C(37)-H(37)	0.9500
C(38)-C(39)	1.390(3)
C(38)-H(38)	0.9500
C(39)-H(39)	0.9500
C(40)-H(40A)	0.9800
C(40)-H(40B)	0.9800
C(40)-H(40C)	0.9800
C(41)-C(42)	1.472(3)
C(42)-C(43)	1.427(3)
C(42)-H(42)	0.9500
C(43)-C(44)	1.467(3)
C(43)-H(43)	0.9500
C(45)-H(45A)	0.9800
C(45)-H(45B)	0.9800
C(45)-H(45C)	0.9800
C(46)-C(48)#1	1.42(2)
C(46)-C(47)	1.518(16)
C(46)-C(47)#1	2.028(16)
C(46)-H(46)	0.9500
C(47)-C(48)	1.32(2)

C(47)-C(46)#1	2.028(16)
C(47)-H(47)	0.9500
C(48)-C(46)#1	1.42(2)
C(48)-H(48)	0.9500

C(42)-Ni(1)-C(43)	42.14(9)
C(42)-Ni(1)-P(2)	104.10(7)
C(43)-Ni(1)-P(2)	146.20(7)
C(42)-Ni(1)-P(1)	141.15(7)
C(43)-Ni(1)-P(1)	100.37(7)
P(2)-Ni(1)-P(1)	112.11(2)
C(16)-P(1)-C(22)	102.51(10)
C(16)-P(1)-C(2)	103.78(10)
C(22)-P(1)-C(2)	98.43(10)
C(16)-P(1)-Ni(1)	108.16(7)
C(22)-P(1)-Ni(1)	113.44(7)
C(2)-P(1)-Ni(1)	127.58(7)
C(28)-P(2)-C(10)	103.58(10)
C(28)-P(2)-C(34)	100.09(10)
C(10)-P(2)-C(34)	100.48(10)
C(28)-P(2)-Ni(1)	113.51(7)
C(10)-P(2)-Ni(1)	118.10(7)
C(34)-P(2)-Ni(1)	118.43(7)
C(1)-O(1)-C(9)	112.32(15)
C(41)-O(2)-C(40)	115.76(18)
C(44)-O(4)-C(45)	115.7(2)
C(2)-C(1)-O(1)	116.47(19)
C(2)-C(1)-C(6)	124.5(2)
O(1)-C(1)-C(6)	118.93(19)
C(1)-C(2)-C(3)	116.3(2)
C(1)-C(2)-P(1)	119.25(16)
C(3)-C(2)-P(1)	124.11(17)
C(4)-C(3)-C(2)	120.7(2)
C(4)-C(3)-H(3)	119.7
C(2)-C(3)-H(3)	119.7
C(3)-C(4)-C(5)	120.8(2)

C(3)-C(4)-H(4)	119.6
C(5)-C(4)-H(4)	119.6
C(4)-C(5)-C(6)	120.4(2)
C(4)-C(5)-H(5)	119.8
C(6)-C(5)-H(5)	119.8
C(5)-C(6)-C(1)	117.1(2)
C(5)-C(6)-C(7)	126.0(2)
C(1)-C(6)-C(7)	116.87(19)
C(15)-C(7)-C(8)	111.57(19)
C(15)-C(7)-C(6)	112.47(19)
C(8)-C(7)-C(6)	105.64(18)
C(15)-C(7)-C(14)	108.9(2)
C(8)-C(7)-C(14)	109.16(18)
C(6)-C(7)-C(14)	108.98(19)
C(9)-C(8)-C(13)	116.7(2)
C(9)-C(8)-C(7)	116.92(19)
C(13)-C(8)-C(7)	126.3(2)
C(8)-C(9)-C(10)	124.9(2)
C(8)-C(9)-O(1)	119.20(19)
C(10)-C(9)-O(1)	115.91(18)
C(9)-C(10)-C(11)	116.7(2)
C(9)-C(10)-P(2)	117.06(16)
C(11)-C(10)-P(2)	126.07(17)
C(12)-C(11)-C(10)	120.2(2)
C(12)-C(11)-H(11)	119.9
C(10)-C(11)-H(11)	119.9
C(13)-C(12)-C(11)	121.1(2)
C(13)-C(12)-H(12)	119.4
C(11)-C(12)-H(12)	119.4
C(12)-C(13)-C(8)	120.2(2)
C(12)-C(13)-H(13)	119.9
C(8)-C(13)-H(13)	119.9
C(7)-C(14)-H(14A)	109.5
C(7)-C(14)-H(14B)	109.5
H(14A)-C(14)-H(14B)	109.5
C(7)-C(14)-H(14C)	109.5

H(14A)-C(14)-H(14C)	109.5
H(14B)-C(14)-H(14C)	109.5
C(7)-C(15)-H(15A)	109.5
C(7)-C(15)-H(15B)	109.5
H(15A)-C(15)-H(15B)	109.5
C(7)-C(15)-H(15C)	109.5
H(15A)-C(15)-H(15C)	109.5
H(15B)-C(15)-H(15C)	109.5
C(21)-C(16)-C(17)	118.8(2)
C(21)-C(16)-P(1)	122.95(17)
C(17)-C(16)-P(1)	118.24(17)
C(18)-C(17)-C(16)	120.4(2)
C(18)-C(17)-H(17)	119.8
C(16)-C(17)-H(17)	119.8
C(17)-C(18)-C(19)	120.6(2)
C(17)-C(18)-H(18)	119.7
C(19)-C(18)-H(18)	119.7
C(20)-C(19)-C(18)	119.3(2)
C(20)-C(19)-H(19)	120.3
C(18)-C(19)-H(19)	120.3
C(19)-C(20)-C(21)	120.6(2)
C(19)-C(20)-H(20)	119.7
C(21)-C(20)-H(20)	119.7
C(20)-C(21)-C(16)	120.4(2)
C(20)-C(21)-H(21)	119.8
C(16)-C(21)-H(21)	119.8
C(27)-C(22)-C(23)	119.1(2)
C(27)-C(22)-P(1)	123.32(16)
C(23)-C(22)-P(1)	117.54(16)
C(24)-C(23)-C(22)	120.4(2)
C(24)-C(23)-H(23)	119.8
C(22)-C(23)-H(23)	119.8
C(23)-C(24)-C(25)	120.1(2)
C(23)-C(24)-H(24)	120.0
C(25)-C(24)-H(24)	120.0
C(26)-C(25)-C(24)	119.8(2)

C(26)-C(25)-H(25)	120.1
C(24)-C(25)-H(25)	120.1
C(25)-C(26)-C(27)	120.5(2)
C(25)-C(26)-H(26)	119.8
C(27)-C(26)-H(26)	119.8
C(22)-C(27)-C(26)	120.2(2)
C(22)-C(27)-H(27)	119.9
C(26)-C(27)-H(27)	119.9
C(29)-C(28)-C(33)	118.8(2)
C(29)-C(28)-P(2)	117.06(17)
C(33)-C(28)-P(2)	123.59(17)
C(30)-C(29)-C(28)	120.7(2)
C(30)-C(29)-H(29)	119.6
C(28)-C(29)-H(29)	119.6
C(31)-C(30)-C(29)	120.0(2)
C(31)-C(30)-H(30)	120.0
C(29)-C(30)-H(30)	120.0
C(30)-C(31)-C(32)	120.0(2)
C(30)-C(31)-H(31)	120.0
C(32)-C(31)-H(31)	120.0
C(31)-C(32)-C(33)	120.2(2)
C(31)-C(32)-H(32)	119.9
C(33)-C(32)-H(32)	119.9
C(32)-C(33)-C(28)	120.2(2)
C(32)-C(33)-H(33)	119.9
C(28)-C(33)-H(33)	119.9
C(39)-C(34)-C(35)	118.6(2)
C(39)-C(34)-P(2)	119.33(17)
C(35)-C(34)-P(2)	122.08(18)
C(36)-C(35)-C(34)	119.8(2)
C(36)-C(35)-H(35)	120.1
C(34)-C(35)-H(35)	120.1
C(37)-C(36)-C(35)	120.8(3)
C(37)-C(36)-H(36)	119.6
C(35)-C(36)-H(36)	119.6
C(38)-C(37)-C(36)	119.9(3)

C(38)-C(37)-H(37)	120.1
C(36)-C(37)-H(37)	120.1
C(37)-C(38)-C(39)	119.7(2)
C(37)-C(38)-H(38)	120.2
C(39)-C(38)-H(38)	120.2
C(34)-C(39)-C(38)	121.2(2)
C(34)-C(39)-H(39)	119.4
C(38)-C(39)-H(39)	119.4
O(2)-C(40)-H(40A)	109.5
O(2)-C(40)-H(40B)	109.5
H(40A)-C(40)-H(40B)	109.5
O(2)-C(40)-H(40C)	109.5
H(40A)-C(40)-H(40C)	109.5
H(40B)-C(40)-H(40C)	109.5
O(3)-C(41)-O(2)	122.8(2)
O(3)-C(41)-C(42)	125.8(2)
O(2)-C(41)-C(42)	111.39(19)
C(43)-C(42)-C(41)	118.2(2)
C(43)-C(42)-Ni(1)	69.86(13)
C(41)-C(42)-Ni(1)	108.87(14)
C(43)-C(42)-H(42)	120.9
C(41)-C(42)-H(42)	120.9
Ni(1)-C(42)-H(42)	91.2
C(42)-C(43)-C(44)	117.5(2)
C(42)-C(43)-Ni(1)	68.00(12)
C(44)-C(43)-Ni(1)	116.91(16)
C(42)-C(43)-H(43)	121.3
C(44)-C(43)-H(43)	121.3
Ni(1)-C(43)-H(43)	85.7
O(5)-C(44)-O(4)	122.6(2)
O(5)-C(44)-C(43)	126.8(2)
O(4)-C(44)-C(43)	110.6(2)
O(4)-C(45)-H(45A)	109.5
O(4)-C(45)-H(45B)	109.5
H(45A)-C(45)-H(45B)	109.5
O(4)-C(45)-H(45C)	109.5

H(45A)-C(45)-H(45C)	109.5
H(45B)-C(45)-H(45C)	109.5
C(48)#1-C(46)-C(47)	130.7(13)
C(48)#1-C(46)-C(47)#1	40.4(10)
C(47)-C(46)-C(47)#1	91.8(7)
C(48)#1-C(46)-H(46)	114.7
C(47)-C(46)-H(46)	114.7
C(47)#1-C(46)-H(46)	151.1
C(48)-C(47)-C(46)	131.0(11)
C(48)-C(47)-C(46)#1	44.4(8)
C(46)-C(47)-C(46)#1	88.2(7)
C(48)-C(47)-H(47)	114.5
C(46)-C(47)-H(47)	114.5
C(46)#1-C(47)-H(47)	154.0
C(47)-C(48)-C(46)#1	95.3(15)
C(47)-C(48)-H(48)	132.4
C(46)#1-C(48)-H(48)	132.4

Symmetry transformations used to generate equivalent atoms:

#1 -x+1,-y+1,-z+1

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

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Ni(1)	21(1)	17(1)	20(1)	-5(1)	-1(1)	-5(1)
P(1)	21(1)	17(1)	21(1)	-6(1)	-1(1)	-4(1)
P(2)	22(1)	18(1)	19(1)	-4(1)	0(1)	-6(1)
O(1)	23(1)	17(1)	19(1)	-4(1)	1(1)	-3(1)
O(2)	28(1)	29(1)	24(1)	-1(1)	0(1)	-8(1)
O(3)	28(1)	35(1)	28(1)	-10(1)	3(1)	-6(1)
O(4)	55(1)	23(1)	46(1)	-3(1)	-13(1)	-17(1)
O(5)	32(1)	38(1)	47(1)	1(1)	-1(1)	-14(1)
C(1)	20(1)	20(1)	22(1)	-2(1)	-2(1)	-5(1)
C(2)	20(1)	21(1)	24(1)	-5(1)	-2(1)	-5(1)
C(3)	28(1)	18(1)	26(1)	-4(1)	-4(1)	-2(1)
C(4)	31(1)	24(1)	25(1)	2(1)	-5(1)	-4(1)
C(5)	30(1)	29(1)	19(1)	-5(1)	0(1)	-8(1)
C(6)	24(1)	23(1)	22(1)	-7(1)	-1(1)	-6(1)
C(7)	33(1)	24(1)	23(1)	-8(1)	-1(1)	-7(1)
C(8)	26(1)	20(1)	24(1)	-7(1)	2(1)	-10(1)
C(9)	20(1)	16(1)	25(1)	-6(1)	3(1)	-6(1)
C(10)	22(1)	18(1)	23(1)	-5(1)	2(1)	-8(1)
C(11)	24(1)	21(1)	29(1)	-6(1)	-1(1)	-7(1)
C(12)	22(1)	24(1)	34(1)	-12(1)	4(1)	-4(1)
C(13)	27(1)	25(1)	27(1)	-11(1)	6(1)	-8(1)
C(14)	35(1)	28(1)	36(1)	-9(1)	-7(1)	-10(1)
C(15)	51(2)	34(1)	24(1)	-11(1)	-2(1)	-4(1)
C(16)	23(1)	18(1)	28(1)	-6(1)	0(1)	-7(1)
C(17)	27(1)	31(1)	28(1)	-5(1)	0(1)	-6(1)
C(18)	35(1)	40(2)	31(1)	-4(1)	8(1)	-8(1)
C(19)	23(1)	34(1)	46(2)	-6(1)	7(1)	-8(1)
C(20)	24(1)	33(1)	45(2)	-9(1)	-5(1)	-7(1)
C(21)	28(1)	26(1)	31(1)	-7(1)	-4(1)	-6(1)
C(22)	24(1)	18(1)	20(1)	-2(1)	-5(1)	-5(1)
C(23)	26(1)	23(1)	31(1)	-7(1)	0(1)	-5(1)

C(24)	32(1)	31(1)	32(1)	-5(1)	1(1)	-14(1)
C(25)	42(1)	22(1)	31(1)	-4(1)	-4(1)	-13(1)
C(26)	36(1)	21(1)	36(1)	-12(1)	-2(1)	-2(1)
C(27)	27(1)	22(1)	28(1)	-9(1)	0(1)	-5(1)
C(28)	24(1)	21(1)	22(1)	-2(1)	0(1)	-7(1)
C(29)	29(1)	30(1)	30(1)	-11(1)	5(1)	-10(1)
C(30)	30(1)	43(2)	34(1)	-15(1)	8(1)	-12(1)
C(31)	31(1)	53(2)	37(1)	-12(1)	8(1)	-22(1)
C(32)	43(2)	54(2)	39(2)	-22(1)	7(1)	-29(1)
C(33)	33(1)	40(1)	29(1)	-16(1)	6(1)	-16(1)
C(34)	31(1)	18(1)	21(1)	-5(1)	-1(1)	-2(1)
C(35)	45(2)	27(1)	37(1)	1(1)	-8(1)	-13(1)
C(36)	65(2)	32(2)	48(2)	11(1)	-16(2)	-15(1)
C(37)	61(2)	33(2)	39(2)	-2(1)	-23(1)	3(1)
C(38)	37(1)	37(2)	36(1)	-16(1)	-12(1)	0(1)
C(39)	29(1)	25(1)	28(1)	-9(1)	-1(1)	-5(1)
C(40)	38(1)	39(2)	27(1)	3(1)	2(1)	-16(1)
C(41)	28(1)	27(1)	21(1)	-9(1)	-2(1)	-10(1)
C(42)	25(1)	25(1)	21(1)	-8(1)	-1(1)	-8(1)
C(43)	28(1)	23(1)	26(1)	-7(1)	-6(1)	-7(1)
C(44)	36(1)	26(1)	30(1)	-3(1)	-10(1)	-11(1)
C(45)	75(2)	36(2)	73(2)	5(2)	-28(2)	-36(2)
C(46)	490(20)	89(5)	217(11)	-6(6)	-233(14)	-73(9)
C(47)	325(15)	89(5)	154(7)	-68(5)	-11(8)	41(7)
C(48)	310(19)	239(14)	560(30)	-263(18)	170(20)	-12(12)

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

	x	y	z	U(eq)
H(3)	6591	4347	-455	30
H(4)	6772	4124	-1965	34
H(5)	7219	2262	-2219	31
H(11)	10104	-2211	2015	29
H(12)	10688	-2589	575	32
H(13)	9587	-1622	-749	31
H(14A)	6034	-350	-132	39
H(14B)	5839	29	-1235	39
H(14C)	6580	-1244	-745	39
H(15A)	7492	402	-2351	43
H(15B)	8754	280	-1976	43
H(15C)	8244	-865	-1860	43
H(17)	5919	2432	3264	35
H(18)	4035	2625	3894	44
H(19)	2449	3063	2927	43
H(20)	2767	3304	1322	40
H(21)	4648	3115	680	34
H(23)	8833	3805	706	32
H(24)	9123	5716	295	37
H(25)	7658	7233	595	37
H(26)	5870	6849	1223	37
H(27)	5554	4941	1606	31
H(29)	6302	162	3810	35
H(30)	4627	-521	4406	41
H(31)	4050	-1882	3815	47
H(32)	5140	-2553	2616	49
H(33)	6837	-1900	2032	37
H(35)	7894	-2657	3924	44
H(36)	9055	-4076	5063	62
H(37)	10921	-3913	5287	56

H(38)	11633	-2302	4392	43
H(39)	10440	-818	3316	33
H(40A)	8437	-1563	6096	43
H(40B)	7679	-331	6199	43
H(40C)	7299	-952	5492	43
H(42)	9544	855	3550	27
H(43)	7679	2886	3134	29
H(45A)	9615	5796	2549	72
H(45B)	10617	4650	2886	72
H(45C)	10152	5018	1844	72
H(46)	5289	4917	6445	306
H(47)	3421	5460	5845	235
H(48)	2951	4856	4566	429

Table 3f. Torsion angles [°] for **3**.

C(9)-O(1)-C(1)-C(2)	-132.51(19)
C(9)-O(1)-C(1)-C(6)	44.7(2)
O(1)-C(1)-C(2)-C(3)	-177.34(18)
C(6)-C(1)-C(2)-C(3)	5.6(3)
O(1)-C(1)-C(2)-P(1)	9.2(3)
C(6)-C(1)-C(2)-P(1)	-167.81(17)
C(16)-P(1)-C(2)-C(1)	-92.46(18)
C(22)-P(1)-C(2)-C(1)	162.36(17)
Ni(1)-P(1)-C(2)-C(1)	33.9(2)
C(16)-P(1)-C(2)-C(3)	94.6(2)
C(22)-P(1)-C(2)-C(3)	-10.6(2)
Ni(1)-P(1)-C(2)-C(3)	-138.97(16)
C(1)-C(2)-C(3)-C(4)	-3.3(3)
P(1)-C(2)-C(3)-C(4)	169.81(17)
C(2)-C(3)-C(4)-C(5)	-0.6(3)
C(3)-C(4)-C(5)-C(6)	2.6(3)
C(4)-C(5)-C(6)-C(1)	-0.5(3)
C(4)-C(5)-C(6)-C(7)	-178.1(2)
C(2)-C(1)-C(6)-C(5)	-3.8(3)
O(1)-C(1)-C(6)-C(5)	179.25(18)
C(2)-C(1)-C(6)-C(7)	174.0(2)
O(1)-C(1)-C(6)-C(7)	-2.9(3)
C(5)-C(6)-C(7)-C(15)	16.8(3)
C(1)-C(6)-C(7)-C(15)	-160.8(2)
C(5)-C(6)-C(7)-C(8)	138.7(2)
C(1)-C(6)-C(7)-C(8)	-38.9(2)
C(5)-C(6)-C(7)-C(14)	-104.1(3)
C(1)-C(6)-C(7)-C(14)	78.3(2)
C(15)-C(7)-C(8)-C(9)	165.65(19)
C(6)-C(7)-C(8)-C(9)	43.1(2)
C(14)-C(7)-C(8)-C(9)	-73.9(2)
C(15)-C(7)-C(8)-C(13)	-14.9(3)
C(6)-C(7)-C(8)-C(13)	-137.4(2)
C(14)-C(7)-C(8)-C(13)	105.5(2)

C(13)-C(8)-C(9)-C(10)	-3.7(3)
C(7)-C(8)-C(9)-C(10)	175.81(19)
C(13)-C(8)-C(9)-O(1)	175.06(17)
C(7)-C(8)-C(9)-O(1)	-5.5(3)
C(1)-O(1)-C(9)-C(8)	-40.3(2)
C(1)-O(1)-C(9)-C(10)	138.56(18)
C(8)-C(9)-C(10)-C(11)	2.1(3)
O(1)-C(9)-C(10)-C(11)	-176.67(17)
C(8)-C(9)-C(10)-P(2)	178.39(16)
O(1)-C(9)-C(10)-P(2)	-0.4(2)
C(28)-P(2)-C(10)-C(9)	66.98(17)
C(34)-P(2)-C(10)-C(9)	170.16(16)
Ni(1)-P(2)-C(10)-C(9)	-59.48(17)
C(28)-P(2)-C(10)-C(11)	-117.13(19)
C(34)-P(2)-C(10)-C(11)	-14.0(2)
Ni(1)-P(2)-C(10)-C(11)	116.41(17)
C(9)-C(10)-C(11)-C(12)	1.0(3)
P(2)-C(10)-C(11)-C(12)	-174.92(16)
C(10)-C(11)-C(12)-C(13)	-2.3(3)
C(11)-C(12)-C(13)-C(8)	0.7(3)
C(9)-C(8)-C(13)-C(12)	2.2(3)
C(7)-C(8)-C(13)-C(12)	-177.2(2)
C(22)-P(1)-C(16)-C(21)	88.9(2)
C(2)-P(1)-C(16)-C(21)	-13.2(2)
Ni(1)-P(1)-C(16)-C(21)	-151.03(17)
C(22)-P(1)-C(16)-C(17)	-89.36(19)
C(2)-P(1)-C(16)-C(17)	168.57(18)
Ni(1)-P(1)-C(16)-C(17)	30.74(19)
C(21)-C(16)-C(17)-C(18)	-0.7(3)
P(1)-C(16)-C(17)-C(18)	177.63(19)
C(16)-C(17)-C(18)-C(19)	0.3(4)
C(17)-C(18)-C(19)-C(20)	0.1(4)
C(18)-C(19)-C(20)-C(21)	-0.1(4)
C(19)-C(20)-C(21)-C(16)	-0.2(4)
C(17)-C(16)-C(21)-C(20)	0.6(3)
P(1)-C(16)-C(21)-C(20)	-177.58(18)

C(16)-P(1)-C(22)-C(27)	-0.6(2)
C(2)-P(1)-C(22)-C(27)	105.6(2)
Ni(1)-P(1)-C(22)-C(27)	-117.00(18)
C(16)-P(1)-C(22)-C(23)	-178.55(18)
C(2)-P(1)-C(22)-C(23)	-72.32(19)
Ni(1)-P(1)-C(22)-C(23)	65.08(19)
C(27)-C(22)-C(23)-C(24)	-0.4(3)
P(1)-C(22)-C(23)-C(24)	177.64(18)
C(22)-C(23)-C(24)-C(25)	1.5(4)
C(23)-C(24)-C(25)-C(26)	-1.8(4)
C(24)-C(25)-C(26)-C(27)	1.1(4)
C(23)-C(22)-C(27)-C(26)	-0.4(3)
P(1)-C(22)-C(27)-C(26)	-178.29(18)
C(25)-C(26)-C(27)-C(22)	0.1(4)
C(10)-P(2)-C(28)-C(29)	-171.47(18)
C(34)-P(2)-C(28)-C(29)	85.05(19)
Ni(1)-P(2)-C(28)-C(29)	-42.2(2)
C(10)-P(2)-C(28)-C(33)	16.7(2)
C(34)-P(2)-C(28)-C(33)	-86.7(2)
Ni(1)-P(2)-C(28)-C(33)	146.04(19)
C(33)-C(28)-C(29)-C(30)	1.9(4)
P(2)-C(28)-C(29)-C(30)	-170.3(2)
C(28)-C(29)-C(30)-C(31)	-1.1(4)
C(29)-C(30)-C(31)-C(32)	-0.4(4)
C(30)-C(31)-C(32)-C(33)	1.0(5)
C(31)-C(32)-C(33)-C(28)	-0.1(4)
C(29)-C(28)-C(33)-C(32)	-1.3(4)
P(2)-C(28)-C(33)-C(32)	170.3(2)
C(28)-P(2)-C(34)-C(39)	-167.65(18)
C(10)-P(2)-C(34)-C(39)	86.36(19)
Ni(1)-P(2)-C(34)-C(39)	-43.8(2)
C(28)-P(2)-C(34)-C(35)	12.3(2)
C(10)-P(2)-C(34)-C(35)	-93.7(2)
Ni(1)-P(2)-C(34)-C(35)	136.20(18)
C(39)-C(34)-C(35)-C(36)	0.3(4)
P(2)-C(34)-C(35)-C(36)	-179.6(2)

C(34)-C(35)-C(36)-C(37)	-1.6(5)
C(35)-C(36)-C(37)-C(38)	0.8(5)
C(36)-C(37)-C(38)-C(39)	1.3(4)
C(35)-C(34)-C(39)-C(38)	1.8(3)
P(2)-C(34)-C(39)-C(38)	-178.26(18)
C(37)-C(38)-C(39)-C(34)	-2.6(4)
C(40)-O(2)-C(41)-O(3)	5.9(3)
C(40)-O(2)-C(41)-C(42)	-173.75(19)
O(3)-C(41)-C(42)-C(43)	11.8(3)
O(2)-C(41)-C(42)-C(43)	-168.54(19)
O(3)-C(41)-C(42)-Ni(1)	-64.9(3)
O(2)-C(41)-C(42)-Ni(1)	114.73(17)
C(41)-C(42)-C(43)-C(44)	148.9(2)
Ni(1)-C(42)-C(43)-C(44)	-109.9(2)
C(41)-C(42)-C(43)-Ni(1)	-101.18(19)
C(45)-O(4)-C(44)-O(5)	-3.0(4)
C(45)-O(4)-C(44)-C(43)	174.5(2)
C(42)-C(43)-C(44)-O(5)	33.3(4)
Ni(1)-C(43)-C(44)-O(5)	-44.6(3)
C(42)-C(43)-C(44)-O(4)	-144.1(2)
Ni(1)-C(43)-C(44)-O(4)	138.02(17)
C(48)#1-C(46)-C(47)-C(48)	25(3)
C(47)#1-C(46)-C(47)-C(48)	13.0(15)
C(48)#1-C(46)-C(47)-C(46)#1	12.0(12)
C(47)#1-C(46)-C(47)-C(46)#1	0.000(2)
C(46)-C(47)-C(48)-C(46)#1	-19(2)

Symmetry transformations used to generate equivalent atoms:

#1 -x+1,-y+1,-z+1

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