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Journal of Organometallic Chemistry 676 (2003) 89–93

Journal of Organo metallic Chemistry

www.elsevier.com/locate/jorganchem

Nickel(II) and nickel(0) derivatives of bis(diphenylphosphino)amine: $[N(PPh_2)_2]_2Ni$, $(Ph_3P)_2Ni[(Ph_2P)_2NH]$. Synthesis, characterization, and some properties

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Received 14 March 2003; received in revised form 2 April 2003; accepted 17 April 2003

Abstract

Disproportionation of nickel(I) bis(triphenylphosphino)bis(trimethylsilyl)amide, $(Ph_3P)_2Ni{-}N(SiMe_3)_2$, in the presence of bis(diphenylphosphino)amine, $(Ph_2P)_2NH$, yields Ni(II) and Ni(0) phosphinoamide complexes: $[N(Ph_2P)_2]_2Ni$ (**1**), $(Ph_3P)_2Ni[(Ph_2P)_2NH]$ (**2**). Ether solution, containing **2** and Ph_3P (1:2) reacts with dioxygen (one equivalent) to form triphenylphosphinoxide adduct $(Ph_3P)_2Ni[(Ph_2P)_2NH\cdots OPPh_3]$ (**3**) in high yield. The crystal structures of compounds **1** and **3** have been determined by X-ray diffraction method.

Keywords: Transition metals; Nickel; Phosphazanes; Phosphinoamides; X-ray diffraction

1. Introduction

bis(Diphenylphosphino)amine (dppa) is one of the most used and intriguing phosphorus–nitrogen ligands in transition metal chemistry [1]. It demonstrates coordinative versatility both in its neutral form and as an anion $[Ph_2PNPPh_2]^-$ [1,2]. However, one of the problems arising is the easy implication of dppa into redox processes. Particularly, it is specific to the bis(diphenylphosphino)amide anion $[Ph_2PNPPh_2]^-$. So, lithium salt of dppa reacts with some inorganic halides to form unpredictable products of phosphazene type. This is the situation, for example, in the reaction of $LiN(PPh_2)_2$ with PCl_3, leading to the mixture of cyclic $\{N(Ph_2P{=}P{-}PPh_2)_2N\}$, and linear $\{(Ph_2P{-}N{=}PPh_2{-})_2\}$ phosphazenes [3]. Reactions of nickel and cobalt chlorides with $LiN(PPh_2)_2$ represent a combination of redox and substitution processes. Prolonged heating of $LiN(Ph_2P)_2$ with cobalt(II) or nickel(II) chlorides in toluene yields phosphazene complexes $[(Ph_2P{-}N)_2PPh_2]_2M$ (M = Co [4], Ni [5]):

$$3MCl_2 + 6LiN(PPh_2)_2 \rightarrow [(Ph_2PN)_2PPh_2]_2M + 2(Ph_2P)_3N + 2M + 6LiCl \quad (1)$$

It remains unclear, however, whether homoleptic complexes $[N(Ph_2P)_2]_2M$ were formed in the beginning of the reaction or not. The same reaction (M = Ni) in the presence of trimethylphosphine ligands yields dimer **4**.

$$2NiCl_2 + 4Me_3P + 2LiN(Ph_2P)_2 \rightarrow \underset{\mathbf{4}}{\{[N(Ph_2P)_2]Ni(\mu-Cl)\}_2} + 2LiCl \quad (2)$$

Further substitution of the remaining chlorine atom for dppa is difficult apparently due to considerable steric

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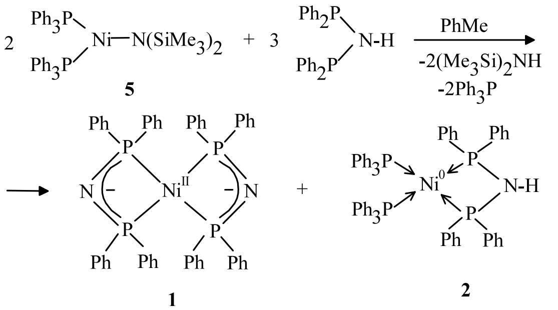
doi:10.1016/S0022-328X(03)00314-0

hindrances and insolubility of **4**. As a part of our studies on Ni(I) compounds, we investigated the interaction of nickel(I) bis(triphenylphosphino)bis(trimethylsilyl)amide with dppa.

2. Results and discussion

2.1. Reactivity

Reaction of nickel silylamide (**5**) with dppa in molar ratio 2:3 yields Ni(II)– and Ni(0)–dppa complexes: $[N(Ph_2P)_2]_2Ni$ (**1**), $(Ph_3P)_2Ni[(Ph_2P)_2NH]$ (**2**) as depicted in Eq. (3):



(3)

It seems nickel silylamide **5** may react with dppa in several different ways. One of the possible initial stages is the substitution of $(Me_3Si)_2N-$ group for $(Ph_2P)_2N-$ since dppa has a reactive hydrogen:

$$\underset{\mathbf{5}}{(Ph_3P)_2Ni\text{–}N(SiMe_3)_2} + H\text{–}N(PPh_2)_2 \rightarrow \underset{\mathbf{6}}{(Ph_3P)_2Ni[(PPh_2)_2N]} + (Me_3Si)_2NH \qquad (4)$$

The another version of the possible initial stage may be the substitution of two triphenylphosphine ligands for bidentate dppa ligand:

$$\underset{\mathbf{5}}{(Ph_3P)_2Ni\text{–}N(SiMe_3)_2} + H\text{–}N(PPh_2)_2 \rightarrow \underset{\mathbf{7}}{[HN(Ph_2P)_2Ni\text{–}N(SiMe_3)_2]} + 2Ph_3P \qquad (5)$$

In both reactions we should have obtained new compounds (**6**, **7**) of monovalent nickel. However, an ESR signal of these complexes was not detected in the course of the reaction while the starting signal of **5** is merely diminished until disappearance. These results are consistent with known data indicating that small chelate angle at nickel atom is unfavorable for existence and formation of Ni(I) complexes [6]. So, the electrochemical reduction of the similar nickel(II) complex containing the ligand of small bite size, $[CH_2(Ph_2P)_2]_2Ni[BF_4]_2$, does not give any defined products [6].

This circumstance leads us to the conjecture of another possible and most probable reaction path including disproportionation of starting nickel silylamide (Eq. (6)) follows by the substitution of triphenylphosphine (Eq. (7)) or silylamide groups (Eq. (8)):

$$2\underset{\mathbf{5}}{(Ph_3P)_2Ni\text{–}N(SiMe_3)_2} \rightleftarrows \underset{\mathbf{8}}{(Ph_3P)_4Ni} + \underset{\mathbf{9}}{\{Ni[N(SiMe_3)_2]_2\}} \qquad (6)$$

$$\underset{\mathbf{8}}{(Ph_3P)_4Ni} + H\text{–}N(PPh_2)_2 \rightleftarrows \underset{\mathbf{2}}{(Ph_3P)_2Ni(Ph_2P)_2NH} + 2Ph_3P \qquad (7)$$

$$\underset{\mathbf{9}}{\{Ni[N(SiMe_3)_2]_2\}} + 2H\text{–}N(PPh_2)_2 \rightarrow \underset{\mathbf{1}}{[N(Ph_2P)_2]_2Ni} + 2(Me_3Si)_2NH \qquad (8)$$

The ^{31}P-NMR spectrum of the reaction mixture reveals the presence of Ph_3P (−4.04 ppm in C_6D_6) along with two triplets of the Ni(0) complex **2**. The formation of **2** according to Eq. (7) was confirmed by ^{31}P-NMR spectroscopy. Tetrakis(trimethylphosphine)nickel and dppa to be mixed in C_6D_6 gave the same signals, inherent to **2** and Ph_3P. Moreover, our attempts to obtain homoleptic complex, $[HN(PPh_2)_2]_2Ni$ from dppa and bis(1,5-cyclooctadiene)nickel were unsuccessful. The color of their mixture in THF changed steadily in time from orange–red to black–brown. No defined products were isolated. It was also found that **2** is slowly decomposed in benzene solution (in sealed NMR tube) for 2 months.

It should be noted, that homoleptic nickel(II) silylamide (**9**) does not exist at ambient conditions. The attempts of its preparation from nickel(II) bromide and $(Me_3Si)_2NNa$ in THF gave the mixture of unidentified products. Furthermore, we ascertained that nickel(I) silylamide (**5**) is also labile in solutions.

Freshly prepared concentrated solution of **5** in toluene, diethyl ether or methylene chloride[1] at 5–10 °C has pale-yellow color and absorption at 438 nm in electronic spectra. It was noted that optical density of the solutions rises (!) under dilution at first time. Moreover, after dilution the electronic spectra contained four absorption bands 378, 417, 462 and 543 nm. The same bands occurred after prolonged exposition of more concentrated solutions for a 2 weeks. We cannot separate any defined products from this solution. An ESR signal of **5** disappeared and recovery of **5** was also impossible. These observations lead us to a supposition that disproportionation and dissociation processes may take place in the solutions of nickel(I) silylamide (**5**).

Since the Ni(0) compound **2** contains two different ligands (Ph_3P and $HN(PPh_2)_2$) we decide to clarify which of the ligands being coordinate to Ni atom is more sensitive to oxidation. One equivalent of dioxygen (1/2 mol) per mol of **2** was added to a mixture contain-

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[1] Methylene chloride slowly reacts with nickel silylamide **5** on the sunlight. Full decoloration observed for 1 h.

Table 1
Summary of crystal and refinement data for complexes

	$[N(PPh_2)_2]_2Ni$ (**1**)	$(Ph_3P)_2Ni[(Ph_2P)_2NH\cdots OPPh_3]$ (**3**)
Empirical formula	$C_{48}H_{40}N_2NiP_4$	$C_{78}H_{66}NNiOP_5$
Formula weight	827.41	1246.88
Temperature (K)	150(2)	150(2)
Wavelength (Å)	0.71073	0.71073
Crystal system	monoclinic	triclinic
Space group	*I*2/*a*	$P\bar{1}$
Unit cell dimensions		
a (Å)	17.5853(8)	13.0058(7)
b (Å)	12.3427(6)	13.3297(8)
c (Å)	18.5459(9)	19.5573(11)
α (°)	90	76.8900(10)
β (°)	96.5050(10)	80.1530(10)
γ (°)	90	78.8340(10)
V (Å^3)	3999.5(3)	3210.9(3)
Z	4	2
D_{calc} (Mg m^{-3})	1.374	1.290
Absorption coefficient (mm^{-1})	0.683	0.474
Crystal size (mm^3)	0.25 × 0.20 × 0.10	0.35 × 0.30 × 0.25
Reflections collected	12152	20623
Independent reflections	4590 [$R_{int} = 0.0169$]	14378 [$R_{int} = 0.0190$]
Absorption correction	SADABS	SADABS
Max. and min. transmission	1.000; 0.898	1.000; 0.837
Refinement method	Full-matrix least-squares on F^2	Full-matrix least-squares on F^2
Data/restraints/parameters	4590/0/395	14378/0/779
Final R indices [$I > 2\sigma(I)$]	$R = 0.1260$ [a], $wR = 0.3394$ [b]	$R = 0.0438$ [a], $wR = 0.1053$ [b]
R indices (all data)	$R = 0.1269$ [a], $wR = 0.3397$ [b]	$R_1 = 0.0559$ [a], $wR = 0.1122$ [b]
S [c]	1.154	1.014
Largest difference peak and hole (e Å^{-3})	0.890; −1.630	0.618; −0.608

[a] $R = \Sigma||F_o| - |F_c||/\Sigma|F_o|$.

[b] $wR = R(wF^2) = \{\Sigma[w(F_o^2 - F_c^2)^2]/\Sigma[w(F_o^2)^2]\}^{1/2}$; $w = 1/[\sigma^2(F_o^2) + (aP)^2 + bP]$, $P = [2F_c^2 + \max(F_o, 0)]/3$.

[c] S = Goodness-of-fit on $F^2 = \{\Sigma[w(F_o^2 - F_c^2)^2]/(n-p)\}^{1/2}$, where n is the number of reflections, and p is the number of refined parameters.

ing **2** and triphenylphosphine (reaction mixture after separation of the complex **1**). To our surprise we succeeded to separate not a free oxidized ligands but in the complex with **2**. The compound **3** contained $Ph_3P{=}O$ molecule associated with hydrogen atom of dppa ligand by hydrogen bond. Since the $Ph_3P{=}O$ molecule was not coordinate to a nickel atom, the oxidation process may conditionally consider as catalytic one.

$$\underset{\mathbf{2}}{(Ph_3P)_2Ni[(Ph_2P)_2NH]} \xrightarrow[Ph_3P]{1/2O_2} \underset{\mathbf{3}}{(Ph_3P)_2Ni[(Ph_2P)_2NH\cdots O{=}PPh_3]} \quad (9)$$

Addition of an excess of dioxygen to the reaction mixture at room temperature instantly affords a lot of colorless crystals of pure triphenylphosphine oxide. No other free phosphine oxides (particularly $Ph_2PNHP(O)Ph_2$, possible products of dppa oxidation) were detected.

2.2. Structures

The structures of **1** and **3** have been determined by X-ray diffraction methods. The crystal data and some details of the data collection and refinement for **1** and **3** are given in Table 1. Selected bond distances and angles for **1** and **3** are in Tables 2 and 3, respectively.

The Ni atom in **1** is on a center of symmetry; i.e. the NP_2NiP_2N fragment is planar (Fig. 1). In **3** the Ni atom has a distorted tetrahedral environment (Fig. 2). The N–P bond lengths in a free $(Ph_2P)_2NH$ molecule are 1.692(2) Å [7]. The close distances [1.6916(18) and 1.6935(18) Å] were observed in Ni(0) complex **3**. So the chelation of dppa to the nickel atom in **3** does not change the length and the order of N–P bond. At the same time the valence PNP angle is contracted significantly from 118.9(2)° in the free ligand [7] to 101.97(10)° in the Ni(0) complex **3**. The average Ni–P distance of 2.22 Å in **1** is within the typical range observed for Ni(II) complexes and slightly longer than those (2.1926(6) and 2.1987(5) Å) founded in Ni(0) complex **3**.

In going from **1** to **3** we observed the shortening of the P–N bonds from 1.6935(18) and 1.6916(18) Å to 1.653(7) and 1.656(9) Å, respectively. This can be rationalized by a higher bond order of the N–P bonds when the metal center is more electropositive, as electron density is transferred from the metal to appropriate N–P π-bonding orbitals. It causes in turn further decreasing of PNP and chelating PNiP angles to 96.5(4) and 67.43(8)°, respectively. To the best of our knowledge, the chelating PNiP angle 67.43(8)° in **1** is the smallest known. For example, in the similar compound $[CH_2(PPh_2)_2]_2Ni[BF_4]_2$ [6] chelating PNiP angle is significantly larger, 73.2(3)°. Interestingly, the complex **1** is paramagnetic at room temperature with magnetic moment 3.1 μ_B, while the other planar phosphine complexes are diamagnetic [6]. Perhaps, this phenomenon is associated with temperature dependent change in geo-

Table 2
Selected bond lengths (Å) and angles (°) for $[N(PPh_2)_2]_2Ni$ (**1**)

Ni(1)–P(1)	2.226(2)	P(2) [a]1–Ni(1)–P(1)	112.57(8)
Ni(1)–P(2)	2.222(2)	P(2)–Ni(1)–P(1)	67.43(8)
P(1)–N(1)	1.653(7)	N(1)–P(1)–Ni(1)	98.0(3)
P(1)–C(1)	1.822(9)	N(1)–P(2)–Ni(1)	98.0(3)
P(1)–C(7)	1.832(9)	P(1)–N(1)–P(2)	96.5(4)
P(1)···P(2)	2.469(3)		
P(2)–N(1)	1.656(9)		

[a] Symmetry equivalent atom.

Table 3
Selected bond lengths (Å) and angles (°) for $(Ph_3P)_2Ni[(Ph_2P)_2NH\cdots OPPh_3]$ (**3**)

Ni(1)–P(1)	2.1736(6)	P(1)–Ni(1)–P(2)	113.77(2)
Ni(1)–P(2)	2.1741(6)	P(1)–Ni(1)–P(3)	112.92(2)
Ni(1)–P(3)	2.1926(6)	P(2)–Ni(1)–P(3)	118.35(2)
Ni(1)–P(4)	2.1987(5)	P(1)–Ni(1)–P(4)	116.25(2)
		P(2)–Ni(1)–P(4)	116.26(2)
P(3)–N(1)	1.6935(18)	P(3)–Ni(1)–P(4)	73.59(2)
P(3)···P(4)	2.6301(7)	N(1)–P(4)–Ni(1)	92.13(6)
P(4)–N(1)	1.6916(18)	N(1)–P(3)–Ni(1)	92.29(6)
P(5)–O(1)	1.444(2)	P(4)–N(1)–P(3)	101.97(10)
P(5)–C(73)	1.808(2)	P(4)–N(1)–H(1N)	128.8(16)
N(1)–H(1N)	0.83(2)	P(3)–N(1)–H(1N)	128.9(16)
O(1)···H(1N)	1.99(2)	N(1)–H(1N)···O(1)	170(2)
N(1)···O(1)	2.820(3)		

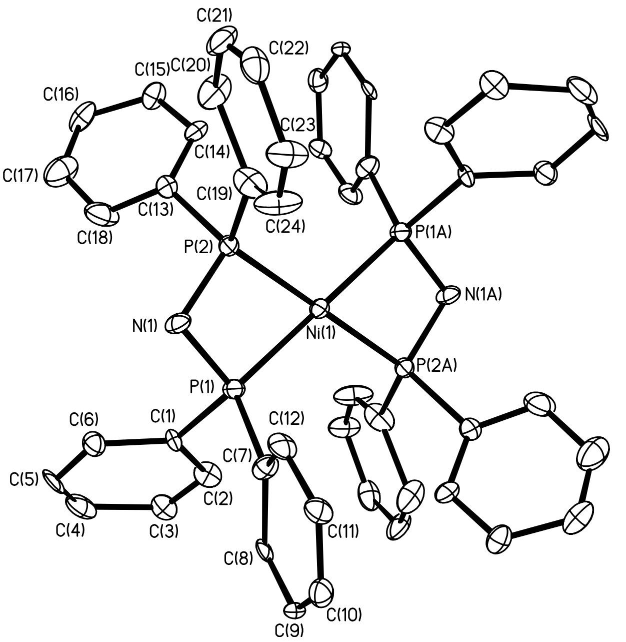

Fig. 1. An ORTEP view of **1** with 30% probability ellipsoids. Only one position of the disordered Ph-rings is shown.

metry of **1** from tetrahedral (at room temperature) to plane-square at 150 K. This study now is in progress.

3. Experimental

3.1. General considerations

Solvents were purified following standard methods [8]. Toluene was thoroughly dried and distilled over P_2O_5 prior to use. Ether was dried and distilled over Na/benzophenone; the compounds $(Ph_3P)_2Ni{-}N(SiMe_3)_2$ [9], $(Ph_2P)_2NH$ [10], were prepared according to known methods. All manipulations were performed with rigorous exclusion of oxygen and moisture, in vacuum or under an argon atmosphere using standard Schlenk techniques.

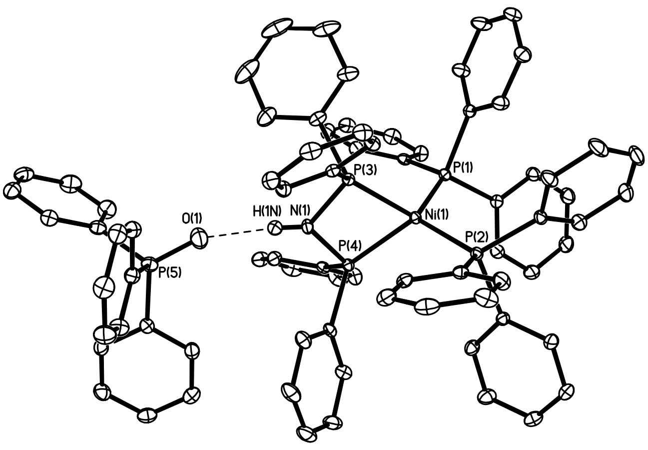

Fig. 2. An ORTEP view of **3** with 30% probability ellipsoids.

Spectrophotometric determination of nickel with dimethylglioxime was provided by the method [11];

Infrared spectra were recorded on a Perkin–Elmer 577 spectrometer from 4000 to 400 cm^{-1} in nujol.
Room-temperature magnetic moments were measured by the Faraday method.
NMR spectra were recorded in $CDCl_3$ or C_6D_6 solutions using 'Bruker DPX-200' instrument, with Me_4Si as internal standard.

3.2. Reaction of $(Ph_3P)Ni{-}N(SiMe_3)_2$ with bis(diphenylphosphino)amide

A mixture of toluene solutions of $(Ph_2P)_2NH$ (0.29 g, 0.75 mmol, 5 ml) and $(Ph_3P)_2Ni{-}N(SiMe_3)_2$ (0.37 g, 0,50 mmol, 5 ml) was maintained at 20 °C. The yellow mixture turned dark-red. Red crystalline precipitate of **1** was formed after 3 h. It was filtered, washed with toluene and dried in vacuum. Yield 0.15 g (73%). Anal. Calc. for $C_{48}H_{40}P_4N_2Ni$ (**1**): C, 69.67, H, 4.87, Ni, 7.09. Found: C, 70.00; H, 5.04, Ni, 6.93%. IR (cm^{-1}): 1430 s, 1300 w, 1220 w, 1180 vw, 1100 s, 1020 w, 1000 w, 920 vs, 870 s, 730 s, 700 s, 550 s, 510 s, 490 s. $\mu_{eff} = 3.1\ \mu_B$.

Toluene was pumped in vacuum from filtrate and changed for ether (8 ml). Dark-brown crystals of **2** formed overnight were filtered, washed with cold ether and dried in vacuum. Yield 0.18 g (75%) of $(Ph_3P)_2Ni[(Ph_2P)_2NH]$. Anal. Calc. For $C_{60}H_{51}P_4NNi$: C, 74.40; H, 5.31; Ni, 6.06. Found: C, 74.56; H, 5.43; Ni, 5.93%. IR (cm^{-1}): 3050 w, 1580 w, 1430 m, 1300 m, 1200 m, 1180 m, 1120 m, 1090 s, 1070 w, 1020 m, 790 m, 730 s, 700 vs, 550 m, 510 vs. ^{31}P-NMR (C_6D_6), δ ppm: 61,9 (t, $^2J_{PP}$ 20.3, Ph_2P), 33.5 (t, $^2J_{PP}$ 20.3, Ph_3P), ^{1}H-NMR: 2.2 (s, 1H, NH), 6.3–8.1 (m, 50H).

3.3. Oxidation of **2** in the presence of Ph_3P

The reaction of $(Ph_2P)_2NH$ (0.75 mmol) with $(Ph_3P)_2Ni{-}N(SiMe_3)_2$ (0.50 mmol) was carried out as described above. The toluene filtrate after separation of **1** contained 0.25 mmol of **2** and 0.5 mmol of Ph_3P. Toluene was changed for ether. Dioxygen (2.8 ml, 0.125 mmol) was allowed to react with solution at 20 °C. Large brown–red crystals were grown overnight. The crystals were separated, washed with cold ether and dried in vacuum. Yield 0.24 g (77%) of **3**. Anal. Calc. For $C_{78}H_{66}P_5NONi$: C, 75.13; H, 5.34; Ni, 4.71. Found: C, 74.96; H, 5.40; Ni, 4.75%. IR (cm^{-1}): 1430 m, 1170 s (P=O), 1120 m, 1080 m, 1020 w, 970 w, 820 m, 740 w, 720 m, 700 s, 540 s, 510 s. ^{31}P-NMR (C_6D_6), δ ppm: 61,9 (t, $^2J_{PP}$ 19.7 Hz, Ph_2P), 33.6 (t, $^2J_{PP}$ 19.7 Hz, Ph_3P), 27.3 (s, Ph_3PO). ^{1}H-NMR: 2.3 (s, 1H, NH), 6.3–8.1 (m, 65H).

3.4. X-ray diffraction studies

X-ray data were collected on a Bruker Smart Apex CCD diffractometer at 150(2) K. The crystal data and some details of the data collection and refinement for **1** and **3** are given in Table 1. Both structures were determined using a combination of direct methods and calculations of Fourier maps and refined by full-matrix least-squared procedures based on the structural factors F^2. The positions of the H atoms were calculated using general geometrical conditions and the H atoms were refined in a rigid group model. Selected bond distances and angles in **1** and **3** are given in Tables 2 and 3, respectively. In the crystal structure of **1** there are two possible arrangements of the main molecule with different positions of Ph-rings. The disordered positions of Ph-rings corresponding to two such arrangements were found from the difference F-map and refined without additional restrictions for Ph-rings. Occupation multiplicities of these two arrangements were refined and gave ratio 49/51. The high value of R-factor for this structure seems to be related with existance of such disorder.

SADABS absorption corrections were applied to both structures [12]. All software and sources scattering factors are contained in the SHELXTL (5.10) program package [13].

4. Conclusions

Nickel (I) bis(triphenylphosphino)bis(trimethylsilyl)amide turned out to be a convenient reagent in preparation of novel dppa complexes of nickel, $[N(Ph_2P)_2]_2Ni$ (**1**), $(Ph_3P)_2Ni[(Ph_2P)_2NH]$ (**2**). The molecule of bis[bis(diphenylphosphino)amido]nickel (II) (**1**) shows the smallest of known chelating PNiP angle 67.43(8)° and possess of magnetic moment 3.1 μ_B at 20 °C. The partial oxidation of **2** with dioxygen in the presence of Ph_3P affords hydrogen bonded triphenylphosphine oxide adduct $\{Ph_3PO\cdots HN(PPh_2)_2Ni(PPh_3)_2\}$ (**3**). Oxidation of **2** with an excess of O_2 affords pure triphenylphosphine oxide.

5. Supplementary material

Crystallographic data have been deposited with the Cambridge Crystallographic Data Centre, CCDC 204878 for **1** and CCDC 204879 for **3**. Copies of this information may be obtained free of charge from The Director, CCDC, 12, Union Road, Cambridge CB2 1EZ, UK (Fax: +44-1223-336033); e-mail deposit@ccdc.cam.ac.uk or http://www.ccdc.cam.ac.uk.

Acknowledgements

We are grateful to the Russian Foundation for Basic Research (Grant 03-03-32051 and 1649.2003.3) for financial support of this work.

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