

Synthesis and crystal structure of nickel(0) dicarbonyl complex with 4-amino-3,5-di-2-pyridyl-4*H*-1,2,4-triazole. [Ni(adpt)(CO)₂]₂•(adpt)₂(thf)

Masahiko Maekawa,^{a,*} Megumu Munakata^{b,*} Takayoshi Kuroda-Sowa,^b
and Yusaku Suenaga^b

^a*Research Institute for Science and Technology, Kinki University,
3-4-1, Kowakae, Higashi-Osaka, Osaka 577, Japan*

^b*Department of Chemistry, Kinki University, 3-4-1, Kowakae,
Higashi-Osaka, Osaka 577, Japan*

(Received, October 18, 1997)

Abstract

A nickel(0) dicarbonyl complex [Ni(adpt)(CO)₂]₂•(adpt)₂(thf) (**1**) (adpt=4-amino-3,5-di-2-pyridyl-4*H*-1,2,4-triazole) has been prepared and has been characterized crystallographically. Complex **1** has a triclinic, space group *P*1, *a*=11.473(6), *b*=15.779(3), *c*=8.179(1)Å, α =101.29(1), β =94.70(3), γ =102.87(3)°, *V*=1403.3(9)Å³, *Z*=1, *R*=0.036 and *R*_w=0.037. There includes two crystallographically different dicarbonyl nickel(0) complexes [Ni(adpt)(CO)₂] (**1a** and **1b**), two free adpt ligands and one solvated thf in a unit cell. On both complexes each Ni atom is coordinated by N atom from one side of two pyridyl groups in the coordinating adpt ligand, one N atom of triazole group and two terminal coordinating CO to provide the four-coordinate mononuclear structure in the distorted tetrahedral geometry. It was found that the Ni-CO bond is strengthened (shorter) and the C≡O bond is correspondingly weakened (longer), depending on the σ donation from N donor atom in the *trans* position.

Key words: Nickel complexes, Carbonyl complexes, Crystal structures, Metal-CO bond

1 Introduction

We have prepared lots of nickel(0)-alkene¹ and alkyne² complexes and have investigated the crystal structures and properties to understand the Ni(0)-alkene and alkyne bonds. It has been reported that the larger π back donation from Ni(0) to alkenes and alkynes is contributed to the Ni(0)-alkene and alkyne bond. A large number of Ni(0) carbonyl complexes also have prepared by the substitution of the carbonyl groups of [Ni(CO)₄] for phosphine,

isocyanide and pyridine-type ligands.³⁻⁶ In particular, there are considerable interests in the Ni(0) carbonyl complexes with the phosphine ligand because they have been found to be efficient catalysts of oligomerization of alkanes. On the other hand the Ni(0) carbonyl complexes with the amine and pyridine-type ligand generally lead to the unstable species. It was not still enough to investigate their crystal structures and properties.

The metal-CO bond consists of σ donation bond and π back-donation bond³ similar to metal-alkene and alkyne bond. The most conclusive indications of the bond order have been provided by structural and spectroscopic study. In $[\text{Mo}(\text{L})_3(\text{CO})_3]$ (L= phosphines, pyridines) series, Cotton^{3,7} has suggested that the M-C bond should become stronger (shorter) and the $\text{C}\equiv\text{O}$ bond weaker (longer), with increasing the π back donation. The M-CO bond in the *trans* position should be strengthened and the $\text{C}\equiv\text{O}$ bond weakened by replacing a CO group with a weaker π -acceptor ligand. On the other hand Sakaki et al⁸ has

theoretically explained in the series of $[\text{Ni}(\text{PR}_3)_2(\text{L})]$ (L= H_2CO , C_2H_4 , C_2H_2 , CO_2 and $(\text{CO})_2$) that the CO ligand is coordinated to the Ni atom in the end-on coordination mode and the $[\text{Ni}(\text{PR}_3)_2(\text{CO})_2]$ complex should be pseudotetrahedral geometry. The strengthening of π back-donation should be the driving force which stabilizes the pseudotetrahedral geometry.

In this study we prepared the nickel(0) dicarbonyl complex with 4-amino-3,5-di-2-pyridyl-4*H*-1,2,4-triazole as a N donor ligand and the nickel-CO bond was characterized by a single X-ray crystal structure analysis.

2 Experimental

2.1 General

All operations were performed using a usual Schlenk techniques. $[\text{Ni}(\text{cod})_2]$ (cod=1,5-cyclo-octadiene) and 4-amino-3,5-di-2-pyridyl-4*H*-1,2,4-triazole (adpt) were commercially purchased from Stream Chemicals and Aldrich Co. The carbon monoxide gas (>99%) was available from Nippon Sanso Co. These chemicals were used without further purification. All organic solvents were dried and distilled by a standard method before use.

2.2 Synthesis of $[\text{Ni}(\text{adpt})(\text{CO})_2]_2 \cdot (\text{adpt})_2(\text{thf})$ (1)

$[\text{Ni}(\text{cod})_2]$ (137.5mg, 5.0×10^{-4} mol) and adpt (59.6mg, 5.0×10^{-4} mol) were stirred in thf (10ml) at -50°C for 20 mins under an argon. The CO gas was bubbled into the yellow suspension for 5 mins. The resultant orange solution was quickly filtered and the filtrate was sealed in 5mm diameter glass tube under CO. The glass tube was allowed to stand for 6 months at -30°C and the red brick crystals were collected. Yields (8mg, 9%).

2.3 Crystallography of $[\text{Ni}(\text{adpt})(\text{CO})_2]_2 \cdot (\text{adpt})_2(\text{thf})$ (1)

Complex 1 is quite unstable in air, although it is thermally stable. A red crystal was quickly taken out from the sealed glass tube and was sealed in the capillary for the X-ray measurement together with

the mother liquid. The red crystal was mounted on a Rigaku AFC-5R automated diffractometer with

Table 1 Crystal data and the measurement conditions for $[\text{Ni}(\text{adpt})(\text{CO})_2]_2 \cdot (\text{adpt})_2(\text{thf})$ (1)

Formula	$\text{Ni}_2\text{O}_5\text{N}_{24}\text{C}_{56}\text{H}_{48}$
Formula weight	1254.55
Crystal system	triclinic
Space group	<i>P</i> 1
<i>a</i> (Å)	11.473(6)
<i>b</i> (Å)	15.779(3)
<i>c</i> (Å)	8.179(1)
α (°)	101.29(1)
β (°)	94.70(3)
γ (°)	102.87(3)
<i>V</i> (Å ³)	1403.3(9)
<i>Z</i>	1
<i>D</i> _{calc} (g/cm ³)	1.484
<i>F</i> (000)	648
λ (Mo K α) (Å)	0.71069
μ (Mo K α) (cm ⁻¹)	7.43
Scan type	ω -2 θ
Scan rate (°/min)	8.0
Scan width (°)	1.47+0.30tan θ
2 θ _{max} (°)	55.0
No. of reflections measured	6760 (total), 6437 (unique)
No. of observed reflections	3257. (<i>I</i> >3 σ (<i>I</i>))
^a <i>R</i>	0.036
^b <i>R</i> _w	0.037

$$^a R = \frac{\sum ||F_o| - |F_c||}{\sum |F_o|}$$

$$^b R_w = \left[\frac{\sum w(|F_o| - |F_c|)^2}{\sum w F_o^2} \right]^{1/2}, \text{ where } w = 4F_o^2 / \sigma^2(F_o^2).$$

graphite mono-chromated Mo-K α radiation ($\lambda=0.71069\text{\AA}$). The crystal data and measurement conditions are revealed in Table 1. Intensity data were measured by ω -2 θ scans at 23°C and were corrected for Lorentz and polarization effects. Empirical absorption corrections were carried out. A total of 6760 independent reflections having $I=3\sigma(I)$ was collected.

The structure was solved by a direct method (MITHRIL)⁹ and refined by full-matrix least squares with anisotropic thermal parameters. Isotropic hydrogen atoms were located by Fourier difference

Table 2 Atomic positional parameters for non-hydrogen atoms and B_{eq} for [Ni(adpt)(CO)₂]₂•(adpt)₂(thf) (1)

Atoms	x	y	z	$B_{eq}(\text{\AA}^2)$
Ni(1)	0.3280	0.3612	0.6993	3.92(8)
Ni(2)	0.8044(1)	0.98297(6)	0.3338(1)	4.15(9)
O(1)	0.249(1)	0.296(1)	0.994(2)	6.9(6)
O(2)	0.521(1)	0.277(1)	0.609(2)	8.8(6)
O(3)	0.619(1)	1.065(1)	0.443(2)	9.0(7)
O(4)	0.888(1)	1.051(1)	0.053(2)	7.8(7)
O(5)	0.487(2)	0.0887(9)	-0.103(2)	14.1(8)
N(1)	0.205(1)	0.353(1)	0.514(2)	4.0(5)
N(2)	0.360(1)	0.4896(7)	0.685(1)	3.3(4)
N(3)	0.446(1)	0.5657(7)	0.757(1)	3.0(4)
N(4)	0.327(1)	0.5934(7)	0.554(1)	3.7(4)
N(5)	0.275(1)	0.6268(6)	0.430(1)	2.8(3)
N(6)	0.481(1)	0.761(1)	0.589(2)	3.9(5)
N(7)	0.942(1)	0.9884(9)	0.531(2)	3.1(4)
N(8)	0.778(1)	0.8507(8)	0.340(1)	3.6(4)
N(9)	0.691(1)	0.7723(8)	0.276(2)	4.5(5)
N(10)	0.808(1)	0.7551(8)	0.482(1)	2.6(4)
N(11)	0.856(1)	0.7088(9)	0.601(2)	5.7(5)
N(12)	0.646(1)	0.573(1)	0.447(2)	3.8(5)
N(13)	0.298(1)	0.709(1)	1.070(1)	4.1(5)
N(14)	0.077(1)	0.6431(9)	0.700(2)	4.8(5)
N(15)	-0.001(1)	0.562(1)	0.636(2)	5.2(5)
N(16)	0.107(1)	0.5621(8)	0.872(1)	3.1(4)
N(17)	0.145(1)	0.5360(7)	1.014(1)	4.2(4)
N(18)	-0.000(1)	0.372(1)	0.822(2)	6.3(6)
N(19)	-0.163(1)	0.634(1)	-0.019(2)	5.0(5)
N(20)	0.066(1)	0.702(1)	0.346(1)	5.2(5)
N(21)	0.138(1)	0.7863(8)	0.390(2)	5.2(5)
N(22)	0.023(1)	0.7853(8)	0.166(1)	4.4(4)
N(23)	-0.017(1)	0.811(1)	0.018(2)	6.4(5)
N(24)	0.128(1)	0.974(1)	0.204(2)	4.2(5)
C(1)	0.279(2)	0.321(1)	0.882(2)	5.5(6)
C(2)	0.447(1)	0.310(1)	0.634(2)	4.9(6)
C(3)	0.120(1)	0.2750(9)	0.431(2)	3.9(4)
C(4)	0.028(2)	0.286(1)	0.309(2)	4.5(5)
C(5)	0.029(2)	0.365(1)	0.257(2)	4.9(5)
C(6)	0.108(1)	0.441(1)	0.342(2)	3.5(5)

C(7)	0.197(1)	0.4346(9)	0.467(2)	3.2(4)
C(8)	0.283(1)	0.505(1)	0.567(2)	4.1(5)
C(9)	0.431(1)	0.633(1)	0.681(2)	3.6(5)
C(10)	0.511(1)	0.7217(8)	0.711(2)	3.6(5)
C(11)	0.595(1)	0.749(1)	0.836(2)	3.4(5)
C(12)	0.671(1)	0.832(1)	0.861(2)	4.7(5)
C(13)	0.650(2)	0.879(1)	0.737(3)	4.9(7)
C(14)	0.551(2)	0.845(1)	0.612(2)	5.1(6)
C(15)	0.692(2)	1.026(1)	0.397(2)	5.2(6)
C(16)	0.862(2)	1.017(1)	0.165(2)	4.3(6)
C(17)	1.022(2)	1.057(1)	0.621(2)	4.3(5)
C(18)	1.102(2)	1.065(1)	0.754(2)	5.1(5)
C(19)	1.105(2)	0.985(1)	0.788(2)	5.4(6)
C(20)	1.017(2)	0.908(1)	0.702(2)	5.0(6)
C(21)	0.942(1)	0.915(1)	0.574(2)	3.9(5)
C(22)	0.841(1)	0.8378(7)	0.470(2)	2.1(4)
C(23)	0.716(1)	0.7183(7)	0.369(2)	2.5(4)
C(24)	0.637(1)	0.629(1)	0.339(2)	2.7(4)
C(25)	0.543(2)	0.595(1)	0.196(2)	5.5(6)
C(26)	0.473(2)	0.510(1)	0.181(2)	5.3(7)
C(27)	0.486(2)	0.456(1)	0.284(2)	4.3(5)
C(28)	0.571(1)	0.4933(9)	0.419(2)	4.2(5)
C(29)	0.393(2)	0.779(1)	1.146(2)	5.0(6)
C(30)	0.418(2)	0.856(1)	1.097(2)	6.0(7)
C(31)	0.356(2)	0.868(1)	0.955(3)	6.4(8)
C(32)	0.264(2)	0.799(1)	0.863(3)	6.4(8)
C(33)	0.233(2)	0.716(1)	0.928(2)	4.1(5)
C(34)	0.141(2)	0.646(1)	0.836(2)	3.3(5)
C(35)	0.020(1)	0.512(1)	0.753(2)	3.6(6)
C(36)	-0.034(2)	0.415(1)	0.730(2)	5.3(7)
C(37)	-0.142(2)	0.375(2)	0.624(3)	7(1)
C(38)	-0.197(2)	0.284(1)	0.595(3)	6.1(7)
C(39)	-0.145(2)	0.238(1)	0.690(2)	6.5(8)
C(40)	-0.055(2)	0.284(1)	0.790(2)	5.4(7)
C(41)	-0.245(2)	0.563(1)	-0.105(2)	5.9(6)
C(42)	-0.283(1)	0.484(1)	-0.047(2)	5.3(7)
C(43)	-0.217(2)	0.480(1)	0.094(2)	5.4(7)
C(44)	-0.126(2)	0.551(1)	0.177(2)	4.2(5)
C(45)	-0.104(2)	0.623(1)	0.118(2)	3.7(5)
C(46)	-0.005(2)	0.706(1)	0.204(2)	4.4(5)
C(47)	0.119(1)	0.834(1)	0.290(1)	4.4(5)
C(48)	0.182(1)	0.925(1)	0.306(2)	3.5(5)
C(49)	0.277(1)	0.960(1)	0.438(2)	5.0(5)
C(50)	0.327(2)	1.046(2)	0.451(2)	8.0(8)
C(51)	0.287(2)	1.099(1)	0.352(3)	8(1)
C(52)	0.178(2)	1.060(2)	0.215(3)	6.9(7)
C(53)	0.464(3)	0.145(3)	0.011(4)	14(2)
C(54)	0.564(4)	0.242(2)	0.037(5)	20(2)
C(55)	0.652(3)	0.187(1)	0.100(4)	16(2)
C(56)	0.609(3)	0.119(2)	-0.069(5)	13(1)

$$B_{eq}=(4/3)\sum_i\sum_j\beta_{ij}(a_i\cdot a_j).$$

synthesis. Reliability factors are defined as $R = \sum(|F_o| - |F_c|) / \sum |F_o|$ and $R_w = \{ \sum w(|F_o| - |F_c|)^2 / \sum w |F_o|^2 \}^{1/2}$, where $w = 4F_o^2 / \sigma(F_o^2)$. Atomic scattering factors and anomalous dispersion terms were taken from the International Tables for X-ray Crystallography, Vol. IV.¹⁰ All calculations were performed using the

3 Results and discussion

The molecular structure of **1** is shown in Figure 1, together with the atom labeling scheme. There are two crystallographically different dicarbonyl Ni(0) complexes [Ni(adpt)(CO)₂], two free adpt ligands and one solvated thf in a unit cell. As mentioned later, the remarkable feature on this complex is that there includes two Ni(0) dicarbonyl complexes having the slightly different coordination environments. The Ni(0) dicarbonyl complex including the Ni(1) and Ni(2) atom is abbreviated as complex **1a** and **1b**, respectively. On both Ni(0) carbonyl complexes the Ni atom is coordinated by the N atom from one side of two pyridyl groups in the coordinating adpt ligand, the N atom of triazole group and two terminal coordinating CO. These Ni(0) dicarbonyl complex are therefore mononuclear complexes. The scheme of the coordination sphere

TEXSAN¹¹ crystallographic software package. The final R and R_w values were 0.036 and 0.037, respectively. Atomic positional parameters for non-hydrogen atoms are listed in Table 2.

around each nickel atom is shown in Figure 2. Selected bond distances and bond angles are listed in Table 3. Each Ni atom is four-coordinate by two N atoms from the adpt ligand and two terminal coordinating CO to provide a distorted tetrahedral geometry with the Ni-CO linkage essentially linear: The N(1)-Ni(1)-N(2) and C(1)-Ni(1)-C(2) angles are 79.9(6) and 107.6(8)° for **1a**, and the N(7)-

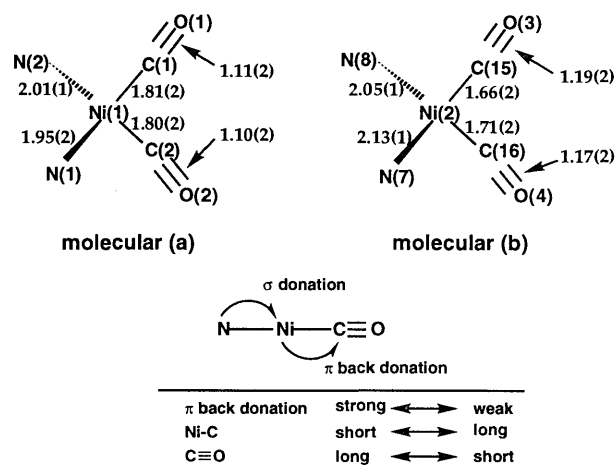


Figure 2. The scheme of the coordination sphere around each Ni atom with bond distances and bond angles.

Ni(2)-N(8) and C(15)-Ni(2)-C(16) angles are 78.8(5) and 114.7(8)° for **1b**. The average Ni-C≡O angle of 176(2) and 171(2)° for **1a** and **1b** are close to those (173-178.7°) of the other Ni dicarbonyl complexes.¹²⁻¹⁹ The average Ni-N distance of 1.98(2) and 2.09(1) Å for **1a** and **1b** are in the range (1.936-2.03 Å) of the similar four-coordinate Ni(0) complexes.^{12-14,20} The average Ni-C distance is 1.80(2) and 1.68(2) Å for **1a** and **1b**. Interestingly the shorter Ni-C distance (1.66(2) Å) of **1b** is shorter than other Ni(0) carbonyl complexes (1.70(2)-1.86(6) Å),¹²⁻¹⁹ indicative of the larger contribution of π back donation. The average C≡O distance of 1.10(2) and 1.18(2) Å for **1a** and **1b** are close to those (1.06(7)-1.17(2) Å) of other Ni(0) carbonyl complexes.¹²⁻¹⁹

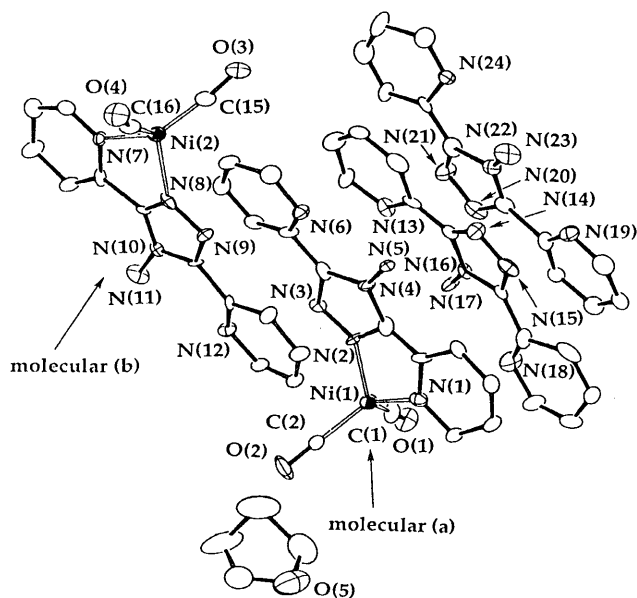


Figure 1. The molecular structure of complex **1**. Only Ni(1), Ni(2), N(1), N(2), N(7), N(8), C(1), C(2), C(15) and C(16) in the coordination sphere, and the hetero atoms N(3), N(4), N(5), N(6), N(9), N(10), N(11), N(12), N(13), N(14), N(15), N(16), N(17), N(18), N(19), N(20), N(21), N(22), N(23), N(24), O(1), O(2), O(3), O(4) and O(5) are labeled.

Table 3 Selected bond distances (Å) and angles (°) for [Ni(adpt)(CO)₂]₂•(adpt)₂(thf) (1)

Molecular (a)			
Ni(1)-N(1)	1.95(2)	Ni(1)-N(2)	2.01(1)
Ni(1)-C(1)	1.81(2)	Ni(1)-C(2)	1.80(2)
C(1)≡O(1)	1.11(2)	C(2)≡O(2)	1.10(2)
Ni(1)-C(1)≡O(1)	180(2)	Ni(1)-C(2)≡O(2)	173(2)
N(1)-Ni(1)-N(2)	79.9(6)	N(1)-Ni(1)-C(2)	113.2(7)
N(2)-Ni(1)-C(2)	112.8(6)	N(1)-Ni(1)-C(1)	117.8(8)
N(2)-Ni(1)-C(1)	123.6(7)	C(1)-Ni(1)-C(2)	107.6(8)
Molecular (b)			
Ni(2)-N(7)	2.13(1)	Ni(2)-N(8)	2.05(1)
Ni(2)-C(15)	1.66(2)	Ni(2)-C(16)	1.71(2)
C(15)≡O(3)	1.19(2)	C(16)≡O(4)	1.17(2)
Ni(2)-C(15)≡O(3)	174(2)	Ni(1)-C(16)≡O(4)	168(2)
N(7)-Ni(2)-N(8)	78.8(5)	N(7)-Ni(2)-C(16)	112.6(8)
N(8)-Ni(2)-C(16)	118.1(6)	N(7)-Ni(2)-C(15)	114.5(7)
N(8)-Ni(2)-C(15)	113.2(7)	C(15)-Ni(2)-C(16)	114.7(8)

It has been so far known that the M-CO bond should be strengthened (shorter) and the C≡O bond weakened (longer) in the [Mo(L)₃(CO)₃]series,^{3,7} with increasing π back donation. On complex **1b**,

the Ni(2)-N(7), Ni(2)-C(15) and C(15)-O(3) distance in the *trans* position is 2.13(1), 1.66(2) and 1.19(2) Å, respectively. On the other hand the Ni(2)-N(8), Ni(2)-C(16) and C(16)-O(4) distance is 2.05(1), 1.71(2) and 1.17(2) Å, respectively. These facts indicate that the M-C distance is stronger (shorter) and the C≡O distance is correspondingly weaker (longer), depending on the σ-donation from the N donor ligand in the *trans* position (vide Figure 2). The similar relationship could be found in complex **1a**: The Ni(1)-N(1), Ni(1)-C(1) and C(1)-O(1) distance is 1.95(2), 1.81(2) and 1.11(2) Å, and the Ni(1)-N(2), Ni(1)-C(2) and C(2)-O(2) distance is 2.01(1), 1.80(2) and 1.10(2) Å, respectively.

In a unit cell two coordinating adpt and one non-coordinating adpt ligand are overlapping each other along *ac* vector. However these most close separations are apart more than 3.4 Å. Consequently the special intermolecular interaction could not be found. Investigations by IR and NMR also did not give the further beneficial informations owing to their unstability.

Acknowledgment

We thank Mr. Koji Hachiya for his assistance. This work was partially supported by a Grant-in Aid for Science Research from the Ministry of Education, Science and Culture in Japan.

References

- [1] (a) M. Maekawa, M. Munakata, T. Kuroda-Sowa and K. Hachiya, *Inorg. Chim. Acta*, **227** (1994) 137.
(b) M. Maekawa, M. Munakata, T. Kuroda-Sowa and K. Hachiya, *Inorg. Chim. Acta*, **230** (1995) 249.
(c) M. Maekawa, M. Munakata, T. Kuroda-Sowa and K. Hachiya, *Inorg. Chim. Acta*, **232** (1995) 231.
- [2] (a) M. Maekawa, M. Munakata, T. Kuroda-Sowa and K. Hachiya, *Inorg. Chim. Acta*, **231** (1995) 213.
(b) M. Maekawa, M. Munakata, T. Kuroda-Sowa and K. Hachiya, *Inorg. Chim. Acta*, **233** (1995) 1.
(c) M. Maekawa, M. Munakata, T. Kuroda-Sowa and K. Hachiya, *Inorg. Chim. Acta*, **236** (1995) 181.
(d) M. Maekawa, M. Munakata, T. Kuroda-Sowa and K. Hachiya, *Polyhedron*, **14** (1995) 2879.
- [3] C. Elschenbroich and A. Salzer, *Organometallics*, 2nd ed., VCH, Weinheim, 1992, p220.
- [4] G. Wilkinson, G. A. Stone and E. W. Abel (editor), *Comprehensive Organometallic Chemistry-I*, Pergamon Press, Oxford, 1982, vol. 6, ch. 37; E. W. Abel, G. A. Stone and G. Wilkinson (editor), *Comprehensive Organometallic Chemistry-II*, Pergamon Press, Oxford, 1995, vol. 9, ch. 1.
- [5] G. Wilkinson, R. D. Gillard and J. A. McCleverty (editor), *Comprehensive Inorganic Chemistry*, Pergamon Press, Oxford, 1987, vol. 5, ch. 50.
- [6] R. J. Cross and D. M. P. Mingos (editor), *Organometallic Compounds of Nickel, Palladium, Platinum, Copper, Silver and Gold*, Chapman and Hall, London, 1985.
- [7] F. A. Cotton, *Inorg. Chem.*, **3** (1963) 702.
- [8] S. Sakaki, K. Kitaura, K. Morokuma and K. Ohkubo, *Inorg. Chem.*, **22** (1983) 104.
- [9] MITHRIL— an integrated direct method computer program, University of Glasgow, Scotland, UK;

- C. J. Gilmore, *J. Appl. Crystallogr.*, **17** (1984) 42.
- [10] International Tables for X-ray Crystallography, Vol. IV, The Kynoch Press, Birmingham, England, 1974.
- [11] TEXSAN—*TEXRAY*, Structure Analysis Package, Molecular Structure Corporation, The Woodlands, TX, USA, 1985.
- [12] V. H. D. Hausen and K. Krogmann, *Z. Anorg. Allg. Chem.*, **389** (1972) 247.
- [13] J. Sieler, N. -N. Thanh, R. Benedix, E. Dinjus and D. Walther, *Z. Anorg. Allg. Chem.*, **522** (1985) 131.
- [14] R. Goddard and Y. -H. Tsay, *Acta Crystallogr.*, **C44** (1988) 810.
- [15] C. Krüger and Y. -H. Tsay, *Cryst. Struct. Comm.*, **3** (1974) 455.
- [16] J. A. J. Jarvis, R. H. B. Mais, P. G. Owston and D. T. Thompson, *J. Chem. Soc.*, (1970) 1867.
- [17] A. D. Pra, G. Zanotti, L. Pandolfo and P. Segala, *Cryst. Struct. Comm.*, **10** (1981) 7.
- [18] M. Baacke, S. Morton, O. Stelzer and W. S. Sheldrick, *Chem. Ber.*, **113** (1980) 1343.
- [19] J. Ladell, B. Post and I. Fankuchen, *Acta Crystallogr.*, **5** (1952) 795.
- [20] E. Dinjus, D. Walther, J. Kaiser, J. Sieler and N. N. Thanh, *J. Organomet. Chem.*, **236** (1982) 123.