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Mono(p-tolyl)platinum(II) and bis(p-tolyl)platinum(II) complexes of diethylsulfide as reagents for organoplatinum synthesis. Structures of $[Pt(p\text{-}Tol)_2(\mu\text{-}SEt_2)]_2$ and $PtCl(p\text{-}Tol)(bpy)$ (bpy = 2,2′-bipyridine)

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Dedicated to Professor Kees Vrieze

Abstract

The complex *trans*-$PtCl(p\text{-}Tol)(SEt_2)_2$ is obtained from the reaction of $[Pt(p\text{-}Tol)_2(SEt_2)]_2$ with $PtCl_2(SEt_2)_2$ and SEt_2 in mole ratio 1:2:2. The mono(p-tolyl)platinum(II) and bis(p-tolyl)platinum(II) complexes of diethylsulfide react with 2,2′-bipyridine to form the complexes $PtX(p\text{-}Tol)(bpy)$ ($X = p$-Tol, Cl) and are useful reagents for organoplatinum chemistry. X-ray crystal structures are presented for square planar $PtCl(p\text{-}Tol)(bpy)$ and the centrosymmetric dimer $[Pt(p\text{-}Tol)_2(\mu\text{-}SEt_2)]_2$. © 2002 Published by Elsevier Science B.V.

Keywords: Organoplatinum; 2,2′-Bipyridine; Diethylsulfide; Crystal structures

1. Introduction

The synthesis of organoplatinum(II) complexes of dialkylsulfide ligands [1–17], including pioneering studies by Kees Vrieze and coworkers [2,3], has been pursued not only as part of the development of fundamental organometallic chemistry, but also as a route to platinum(II) substrates for further synthesis including reactions in which the ancillary dialkylsulfide ligands are readily displaced by other ligands. In recent reports it has been shown that the diethylsulfide bridged complex $[Pt(p\text{-}Tol)_2(SEt_2)]_2$ reacts with the bromoarenes $2,6\text{-}(3,5\text{-}Me_2pzCH_2)_2C_6H_3Br$ ($3,5\text{-}Me_2pz =$ 3,5-dimethylpyrazol-1-yl) and $2,6\text{-}(Me_2NCH_2)_2C_6H_3Br$ to form arylplatinum(II) complexes $PtBr\{2,6\text{-}(pzCH_2)_2\text{-}C_6H_3\text{-}N,C,N\}$ [15] and $PtBr\{2,6\text{-}(Me_2NCH_2)_2C_6H_3\text{-}N,C,N\}$ [16], providing a new and convenient route to intramolecularly coordinated systems. Moreover, this synthetic approach provides a new entrance to mixed metal systems where at least one metal site is platinum [18]. In these studies the p-tolyl group was chosen in preference to the phenyl group in view of its advantages in monitoring reactions and examining products by ^1H NMR spectroscopy, and the new application of this type of diarylplatinum(II) complex has prompted us to examine the synthesis of mono- and di-p-tolylplatinum(II) precursors for organoplatinum synthesis, which we report here together with structural analyses of $[Pt(p\text{-}Tol)_2(SEt_2)]_2$ and $PtCl(p\text{-}Tol)(bpy)$ (bpy = 2,2′-bipyridine).

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2. Experimental

The reagent $PtCl_2(SEt_2)_2$ was prepared as described [19], and other reagents were purchased and used without further purification. Syntheses and NMR experiments were carried out under a nitrogen atmosphere using standard Schlenk techniques. Solvents were purified and dried according to standard procedures, stored under nitrogen and freshly distilled immediately before use. NMR solvents were purchased and used without further purification. NMR spectra were recorded with a Varian Unity Inova 400 WB spectrometer operating at 100.587 (^{13}C) and 399.716 (^{1}H) MHz, at room temperature. Chemical shifts are referenced internally by using residual solvent resonances and are reported in parts per million relative to $SiMe_4$. Microanalyses were performed by the Central Science Laboratory, University of Tasmania.

2.1. Synthesis of complexes

*2.1.1. [Pt(p-Tol)$_2$(SEt$_2$)]$_2$ (**1**)*

Solid $PtCl_2(SEt_2)_2$ (1.00 g, 2.24 mmol) was added to a solution of Li(*p*-Tol) (0.66 g, 6.72 mmol) [prepared from equimolar amounts of *p*-TolI and $LiBu^n$ in toluene) in diethyl ether (50 ml)] at −78 °C to give a bright yellow solution. The mixture was stirred for 3 h at this temperature, then for 4 h at ambient temperature to give a white suspension. Water (10 ml) was added and the volatiles removed in vacuum. The mixture was extracted with dichloromethane (3 × 15 ml) and the organic fractions combined and dried over $MgSO_4$, filtered and evaporated by reduced pressure to give a light brown powder. Recrystallization from acetone gave the product as a white crystalline solid (1.57 g, 75%). ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.16 (d, $^{3}J = 7.8$ Hz, $^{3}J_{Pt-H} = 68.8$ Hz, 8, H2,6-$C_6H_4CH_3$), 6.76 (d, $^{3}J = 7.8$ Hz, 8, H3,5-$C_6H_4CH_3$), 2.51 (q, $^{3}J = 7.2$ Hz, 8, S($CH_2CH_3)_2$), 2.13 (s, 12, $C_6H_4CH_3$), 1.36 (t, $^{3}J = 7.2$ Hz, 12, S($CH_2CH_3)_2$). ^{13}C NMR ($CDCl_3$, 100 MHz): δ 144.2, 134.2, 131.7, 128.5, 29.2, 21.1, 12.7.

*2.1.2. trans-PtCl(p-Tol)(SEt$_2$)$_2$ (**2**)*

A solution of [Pt(*p*-Tol)$_2$(SEt$_2$)]$_2$ (0.043 g, 0.043 mmol), $PtCl_2(SEt_2)_2$ (0.038 g, 0.086 mmol) and SEt_2 (9.3 µl, 0.086 mmol) were stirred in dichloromethane (2 ml) for 12 h. The volatile species were removed in vacuum and the product dissolved in diethyl ether (2 ml). The solution was filtered through Celite and the solvent removed in vacuum to give a tan powder (0.074 g, 86%). ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.11 (d, $^{3}J = 8.1$ Hz, $^{3}J_{Pt-H} = 46.4$ Hz, 2, *H*2,6-$C_6H_4CH_3$), 6.79 (d, $^{3}J = 8.1$ Hz, 2, *H*3,5- $C_6H_4CH_3$), 2.83 (s-broad, 8, S($CH_2CH_3)_2$), 2.22 (s, 3, $C_6H_4CH_3$), 1.36 (t, $^{3}J = 7.4$ Hz, 12, S($CH_2CH_3)_2$). ^{13}C NMR ($CDCl_3$, 100 MHz): δ 136.5, 132.5, 129.3 ($J_{Pt-C} = 29.2$), 129.1, 30.2, 21.0, 13.2 ($J_{Pt-C} = 35.3$ Hz). *Anal.* Calc. for $C_{15}H_{27}ClPtS_2$: C, 35.89; H, 5.42; S, 12.77. Found: C, 35.52; H, 5.64; S, 12.96%.

*2.1.3. Pt(p-Tol)$_2$(bpy) (**3**)*

A solution of [Pt(*p*-Tol)$_2$(SEt$_2$)]$_2$ (0.043 g, 0.043 mmol) and 2,2′-bipyridine (0.021 g, 0.13 mmol) in benzene (2 ml) was stirred for 1 h. Diethyl ether (10 ml) was added to the solution resulting in a yellow precipitate which was collected by filtration and dried in vacuum (0.034 g, 71%). ^{1}H NMR ($CDCl_3$, 400 MHz): δ 8.64 (d, $^{3}J = 5.2$ Hz, 2, *H*6-bpy), 8.00–7.94 (overlapping, 4, *H*3,4-bpy), 7.34 (overlapping, 4, *H*5-bpy + *H*2,6-$C_6H_4CH_3$), 6.85 (d, $^{3}J = 8.0$ Hz, 2, *H*3,5-$C_6H_4CH_3$), 2.22 (s, 6, $C_6H_4CH_3$). ^{13}C NMR ($CDCl_3$, 100 MHz): δ 156.2, 150.5 ($^{2}J_{Pt-C} = 39.2$ Hz), 141.0 ($^{1}J_{Pt-C} = 1102.7$ Hz), 138.1 ($^{3}J_{Pt-C} = 33.2$ Hz), 137.6, 130.9, 128.3 ($^{2}J_{Pt-C} = 79.4$ Hz), 127.1, 122.3. *Anal.* Calc. for $C_{24}H_{22}N_2Pt$: C, 54.03; H, 4.16; N, 5.25. Found: C, 53.90; H, 4.08; N, 5.13%.

*2.1.4. PtCl(p-Tol)(bpy) (**4**)*

A mixture of *trans*-PtCl(*p*-Tol)(SEt$_2$)$_2$ (0.060 g, 0.12 mmol) and 2,2′-bipyridine (0.021 g, 0.13 mmol) in dichloromethane (2 ml) was stirred for 12 h, then the volatile species was removed in vacuum. The residue was dissolved in dichloromethane (2 ml) and stirred for a further 12 h. This process was repeated three times. Diethyl ether (10 ml) was added to the final solution resulting in a yellow precipitate (0.045 g, 85%). ^{1}H NMR ($CDCl_3$, 400 MHz): δ 9.63 (d, $^{3}J = 6.0$ Hz, 1, *H*6-bpy *trans* to *p*-tolyl), 8.75 (d, $^{3}J = 5.2$ Hz, $^{3}J_{Pt-H} = 58.4$ Hz, 1, *H*6-bpy *trans* to Cl), 8.13–8.00 (m, 4, *H*3,4-bpy), 7.66 (ddd, $^{3}J = 5.2$, 5.6 Hz, $^{4}J = 1.6$ Hz, 1, *H*5-bpy), 7.33 (d, $^{3}J = 8.0$ Hz, $^{3}J_{Pt-H} = 35.2$ Hz, 2, *H*2,6-$C_6H_4CH_3$), 7.29 (ddd, $^{3}J = 5.2$, 5.6 Hz, $^{4}J = 1.6$ Hz, 1, *H*5-bpy), 6.94 (d, $^{3}J = 7.6$ Hz, 2, *H*3,5-$C_6H_4CH_3$), 2.32 (s, 3, $C_6H_4CH_3$). ^{13}C NMR ($CDCl_3$, 100 MHz): δ 157.2, 154.9, 151.7, 148.9, 138.8, 137.6, 137.1, 133.9, 132.6, 128.6, 127.6, 127.1, 123.0, 121.9, 21.1. *Anal.* Calc. for $C_{17}H_{15}ClN_2Pt$: C, 42.73; H, 3.16; N, 5.86. Found: C, 42.68; H, 3.21; N, 5.74%.

*2.2. X-ray structure determinations for **1** and **4***

X-ray intensities were measured in a Nonius Kappa CCD diffractometer with rotating anode (Mo Kα, $\lambda = 0.71073$ Å) at a temperature of 150 K. The structures were solved with automated Patterson methods with the program DIRDIF-97 [20] and refined with the program SHELX-97 [19] against F^2 of all reflections. The drawings, structure calculations, and checking for higher symmetry was performed with the program PLATON [22]. Further details of the structure determinations are given in Table 1.

2.2.1. Structure **1**

The unit cell determination and the integration of intensities were performed with the DENZO package [23]. Reflections (38 588) were measured, of which 3940 were unique ($R_{\text{int}} = 0.054$). An absorption correction was applied with the program PLATON (routine MULABS, 0.35–0.42 transmission).

Table 1
Crystal data and structure refinement parameters for $[Pt(p\text{-}Tol)_2(SEt_2)]_2$ (**1**) and PtCl(*p*-Tol)(bpy) (**4**)

	1	**4**
Empirical formula	$C_{36}H_{48}Pt_2S_2$	$C_{17}H_{15}ClN_2Pt$
Formula weight	935.04	477.85
Space group	$P2_1/c$	$P2_1/c$
a (Å)	9.2276(1)	20.144(3)
b (Å)	13.0792(1)	10.1233(8)
c (Å)	14.4199(2)	15.4134(19)
β (°)	98.8018(6)	107.972(9)
V (Å^3)	1719.84(3)	2989.9(6)
Z	2	8
D_{calc} (g cm^{-3})	1.806	2.123
F(000)	904	1808
Crystal size (mm)	$0.09 \times 0.12 \times 0.12$	$0.06 \times 0.18 \times 0.30$
μ (mm^{-1})	8.269	9.558
N	3940	8804
N_O [a]	3570	6642
R, [b] wR_2, [c] S	0.0177, 0.0392, 1.04	0.0502, 0.1498, 1.17
Min., max. residual (e Å^{-3})	−0.77, 0.90	−2.30, 1.71

[a] For $I > 2\sigma(I)$.
[b] $R = (\Sigma|F_o| - |F_c|)/\Sigma|F_o|$ for $I > 2\sigma(I)$.
[c] $wR_2 = [[(\Sigma w(F_o^2 - F_c^2)^2]/\Sigma w(F_o^2)^2]^{1/2}$ for all reflections.

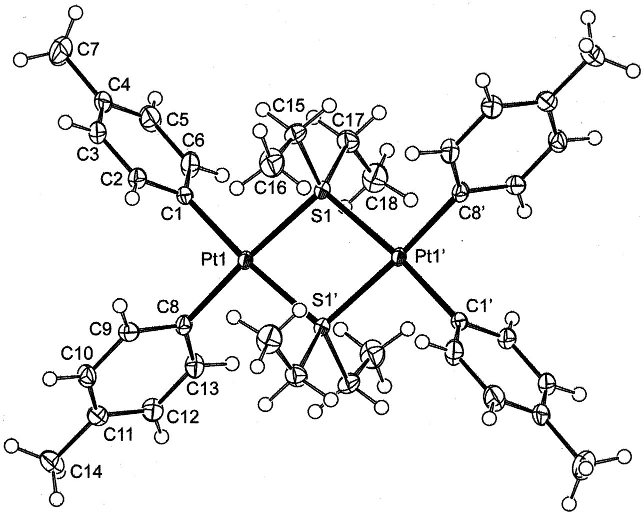

Fig. 1. Projection of a molecule of $[Pt(p\text{-}Tol)_2(SEt_2)]_2$ (**1**) showing 50% displacement ellipsoids for the non-hydrogen atoms, hydrogen atoms having an arbitrary radius. Symmetry operation: $1-x$, $1-y$, $1-z$.

2.2.2. Structure **4**

The plate-shaped crystals are non-merohedrically twinned with a twofold rotation about the crystallographic *c* axis as twin operation. This twin law is also reflected in the crystal structure: the asymmetric unit contains two crystallographically independent molecules, which are also related to each other by a twofold rotation around *c*. The cell parameters and the twin law were determined with the program DIRAX [24]. The intensities were obtained for both twin domains and the overlapping sections using EVAL-14 [25]. An analytical absorption correction was applied with the program PLATON (routine ABST, 0.18–0.81 transmission). Reflections, equivalent with respect to the twin situation, were merged. The refinement used the HKLF option [26] of SHELX-97 [21].

3. Results and discussion

3.1. Synthesis and characterisation of complexes

The parent complex $[Pt(p\text{-}Tol)_2(SEt_2)]_2$ (**1**) was prepared in 75% yield following the procedure of Steele and Vrieze [3] with minor modifications, and the complex formed crystals suitable for X-ray diffraction on slow diffusion of hexanes into a concentrated solution of the complex in dichloromethane. The new diethylsulfide reagent *trans*-$PtCl(p\text{-}Tol)(SEt_2)_2$ (**2**) was obtained readily from $[Pt(p\text{-}Tol)_2(SEt_2)]_2$ in 86% yield when a mixture of $[Pt(p\text{-}Tol)_2(SEt_2)]_2$, $PtCl_2(SEt_2)_2$ and SEt_2 in dichloromethane with an overall mole ratio Pt/Cl/*p*-Tol/SEt_2 = 1:1:1:2 was stirred for 12 h. The diethylsulfide reagents **1** and **2** react with 2,2′-bipyridine at ambient temperature to form the complexes $Pd(p\text{-}Tol)_2(bpy)$ (**3**) and PdCl(*p*-Tol)(bpy) (**4**) in yields of 71 and 85%, respectively.

The new complexes **2**–**4** are stable solids with simple NMR spectra showing resonances with appropriate intensities, and coupling involving ^{195}Pt, e.g. $^3J_{\text{Pt–H}} =$

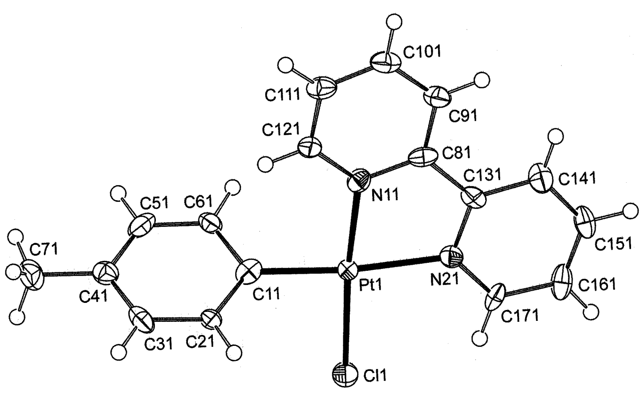

Fig. 2. Projection of molecule 1 for PtCl(*p*-Tol)(bpy) (**4**) showing 50% displacement ellipsoids for the non-hydrogen atoms, hydrogen atoms having an arbitrary radius.

Table 2
Selected bond distances (Å) and bond angles (°) for [Pt(*p*-Tol)$_2$(SEt$_2$)]$_2$ (**1**) [a]

Bond distances [b]			
Pt(1)–C(1)	2.027(3)	Pt(1)–S(1)	2.3637(6)
Pt(1)–C(8)	2.020(3)	Pt(1)–S(1′)	2.3612(6)
Bond angles			
C(1)–Pt(1)–C(8)	89.68(10)	Pt(1)–C(1)–C(6)	121.1(2)
C(1)–Pt(1)–S(1)	94.53(7)	Pt(1)–C(8)–C(9)	123.83(19)
C(1)–Pt(1)–S(1′)	173.86(7)	Pt(1)–C(8)–C(13)	120.30(19)
C(8)–Pt(1)–S(1)	175.77(7)	Pt(1)–S(1)–C(15)	111.93(10)
C(8)–Pt(1)–S(1′)	96.23(7)	Pt(1)–S(1)–C(17)	115.26(10)
S(1)–Pt(1)–S(1′)	79.55(2)	Pt(1′)–S(1)–C(15)	117.50(10)
Pt(1)–S(1)–Pt(1′)	100.45(2)	Pt(1′)–S(1)–C(17)	112.16(10)
Pt(1)–C(1)–C(2)	122.1(2)	C(15)–S(1)–C(17)	100.31(14)

[a] Distances of atoms from the Pt$_2$S$_2$ plane: C(1) −0.058(3), C(8) −0.011(3) Å; distances of the Pt atom from the C$_6$ ring mean planes: −0.066(1) for C(1–6) and −0.070(1) Å for C(8–13). The mean planes of C$_6$ rings of the *p*-tolyl groups form angles of 78.85(10) [C(1–6)] and 65.56(9)° [C(8–13)] with the Pt$_2$S$_2$ plane.

[b] Pt(1)···Pt(1′) 3.6315(1) Å.

46.4 Hz for H2,6-$C_6H_4CH_3$ and one SEt$_2$ environment in **2**, $^{1}J_{\text{Pt–C}} = 1102.7$ Hz in **3**, two pyridine ring environments and $^{3}J_{\text{Pt–H}} = 35.2$ Hz for H2,6-$C_6H_4CH_3$ in **4**.

3.2. X-ray structural studies

The complexes [Pt(*p*-Tol)$_2$(SEt$_2$)]$_2$ (**1**) and PdCl(*p*-Tol)(bpy) (**4**) exhibit square planar geometry as expected for platinum(II) (Figs. 1 and 2, Tables 2 and 3), with angles at Pt in the range 79.55(2)–96.23(7)° for **1**, and 79.6(3)–96.1(4) and 80.0(3)–96.2(4)° for the two independent, but essentially identical molecules in crystals of **4**. Complex **1** is a centrosymmetric dimer with a geometry similar to that of centrosymmetric [PtMe$_2$(SEt$_2$)]$_2$ [7]. Thus, the diethylsulfide ligands are symmetrically coordinated to Pt(1) and Pt(1′) [Pt–S = 2.3637(6) and 2.3612(6) Å], compared to 2.356(3) and 2.353(6) Å in the Pt(II)Me$_2$ complex, and the Pt(1)···Pt(1′) separation is 3.6315(1) Å compared to 3.610(1) Å in the Pt(II)Me$_2$ complex. The mean planes of the *p*-tolyl groups form dihedral angles of 78.85(10) and 65.56(9)° with the Pt$_2$S$_2$ plane in **1**, and 63.1(5) and 58.3(5)° with the mean coordination plane in **4**.

4. Supplementary material

Crystallographic data for the structural analysis have been deposited with the Cambridge Crystallographic Data Centre, CCDC Nos. 156358 and 156359 for compounds **1** and **4**, respectively. Copies of this information may be obtained free of charge from The Director, CCDC, 12 Union Road, Cambridge, CB2 1EZ, UK (fax: +44-1223-336-033; e-mail: deposit@ccdc.cam.ac.uk or www: http://www.ccdc.cam.ac.uk).

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Table 3
Selected bond distances (Å) and bond angles (°) for PtCl(*p*-Tol)(bpy) (**4**), molecules 1 and 2 [a]

	$n = 1$	$n = 2$		$n = 1$	$n = 2$
Bond distances					
Pt(*n*)–C(1*n*)	1.979(9)	1.982(9)	Pt(*n*)–N(1*n*)	2.022(8)	2.019(7)
Pt(*n*)–Cl(*n*)	2.293(3)	2.291(3)	Pt(*n*)–N(2*n*)	2.094(7)	2.089(7)
Bond angles					
C(1*n*)–Pt(*n*)–Cl(*n*)	88.8(3)	88.8(3)	Pt(*n*)–C(1*n*)–C(2*n*)	124.4(7)	122.5(7)
C(1*n*)–Pt(*n*)–N(1*n*)	96.1(4)	96.2(4)	Pt(*n*)–C(1*n*)–C(6*n*)	121.2(7)	124.1(7)
C(1*n*)–Pt(*n*)–N(2*n*)	173.2(4)	175.5(4)	Pt(*n*)–N(1*n*)–C(8*n*)	115.5(6)	114.8(6)
Cl(*n*)–Pt(*n*)–N(1*n*)	174.2(3)	174.8(2)	Pt(*n*)–N(1*n*)–C(12*n*)	124.8(7)	124.9(7)
Cl(*n*)–Pt(*n*)–N(2*n*)	95.8(2)	95.1(2)	Pt(*n*)–N(2*n*)–C(13*n*)	113.6(6)	113.4(6)
N(1*n*)–Pt(*n*)–N(2*n*)	79.6(3)	80.0(3)	Pt(*n*)–N(2*n*)–C(17*n*)	127.8(7)	128.2(7)

[a] Distance of atoms from the ‘Pt(*n*),C(1*n*),N(1*n*),N(2*n*),Cl(*n*)’ coordination mean plane [χ^2 467.8 ($n = 1$), 87.7 ($n = 2$)]: Pt(1, 2) 0.011(1), −0.006(1); C(11, 12) 0.078(10), 0.035(10); N(11, 12) −0.082(9), −0.036(9); N(21, 22) 0.081(10), 0.036(10); Cl (1, 2) −0.066(3), −0.029(4) Å; and the platinum atoms from the *p*-tolyl, pyridine [N(1*n*)], and pyridine [N(2*n*)] mean planes: Pt(1) 0.069(1), −0.103(1), 0.114(1) and Pt(2) −0.013(1), −0.019(1), 0.260(1) Å. Dihedral angles between the coordination mean plane and the *p*-tolyl, pyridine [N(1*n*)] and pyridine [N(2*n*)] mean planes: 63.1(5), 6.8(5), 5.8(5)° for $n = 1$, and 58.3(5), 8.3(5), 9.1(5)° ($n = 2$).

References

[1] S. Sergi, V. Marsala, R. Pietropaolo, F. Faraone, J. Organomet. Chem. 23 (1970) 281.

[2] J. Kuyper, R. van der Laan, F. Jeanneaus, K. Vrieze, Transition Met. Chem. 1 (1976) 199.

[3] B.R. Steele, K. Vrieze, Transition Met. Chem. 2 (1977) 140.

[4] R.J. Puddephatt, M.A. Thomson, J. Organomet. Chem. 238 (1982) 231.

[5] J.D. Scott, R.J. Puddephatt, Organometallics 2 (1983) 1643.

[6] M. Lashanizadehgan, M. Rashidi, J.E. Hux, R.J. Puddephatt, S.S.M. Ling, J. Organomet. Chem. 269 (1984) 317.

[7] D.P. Bancroft, F.A. Cotton, L.R. Falvello, W. Schwotzer, Inorg. Chem. 25 (1986) 764.

[8] H. Tobita, H. Habazaki, H. Ogino, Bull. Chem. Soc. Jpn. 60 (1987) 797.

[9] N. Hadj-Bagheri, R.J. Puddephatt, Polyhedron 7 (1988) 2695.

[10] G. Alibrandi, D. Minniti, L. Monsù Scolari, R. Romeo, Inorg. Chem. 28 (1989) 1939.

[11] M. Rashidi, Z. Fakhroeian, R.J. Puddephatt, J. Organomet. Chem. 406 (1990) 261.

[12] K.C. Lövqvist, O.F. Wendt, J.G. Leipoldt, Acta Chem. Scand. 50 (1996) 1069.

[13] P. Kapoor, V.Yu. Kukushkin, K. Lövqvist, A. Oskarsson, J. Organomet. Chem. 517 (1996) 71.

[14] M. Rashidi, M. Hashemi, M. Khorasani-Motlagh, R.J. Puddephatt, Organometallics 19 (2000) 2751.

[15] A.J. Canty, J. Patel, B.W. Skelton, A.H. White, J. Organomet. Chem. 599 (2000) 195.

[16] M. Albrecht, G. Rodriguez, J. Schoenmaker, G. van Koten, Org. Lett. 22 (2000) 3461.

[17] L. Johansson, M. Tilset, J.A. Labinger, J.E. Bercaw, J. Am. Chem. Soc. 122 (2000) 10846.

[18] M. Albrecht, H.P. Dijkstra, G. van Koten, submitted for publication.

[19] G.B. Kauffman, D.O. Cowan, Inorg. Synth. 6 (1960) 211.

[20] P.T. Beurskens, G. Admiraal, G. Beurskens, W.P. Bosman, S. Garcia-Granda, R.O. Gould, J.M.M. Smits, C. Smykalla, The DIRDIF-97 Program System, Technical Report of the Crystallography Laboratory, University of Nijmegen, The Netherlands, 1997.

[21] G.M. Sheldrick, SHELX-97: Program for Crystal Structure Refinement, University of Göttingen, Göttingen, Germany, 1997.

[22] A.L. Spek, PLATON: A Multipurpose Crystallographic Tool, Utrecht University, The Netherlands, 2000.

[23] Z. Otwinowski, W. Minor, in: C.W. Carter, R.M. Sweet (Eds.), Methods in Enzymology, Academic Press, New York, 1997, p. 307.

[24] A.J.M. Duisenberg, J. Appl. Crystallogr. 25 (1992) 92.

[25] A.J.M. Duisenberg, Thesis, Utrecht University, The Netherlands, 1998.

[26] R. Herbst-Irmer, G.M. Sheldrick, Acta Crystallogr., Sect B 54 (1998) 443.