Synthesis of Gold(I) and Gold(II) Complexes with Diphenyl(trimethylsilylmethyl)phosphane

Manuel Bardají[a], Peter G. Jones[b], and Antonio Laguna*[a]

Departamento de Química Inorgánica, Instituto de Ciencia de Materiales de Aragón, Universidad de Zaragoza-CSIC[a],
E-50009 Zaragoza, Spain

Institut für Anorganische und Analytische Chemie, Technische Universität Braunschweig[b],
Postfach 3329, D-38023 Braunschweig, Germany

Received February 16, 1998

Keywords: Gold(I) / Gold(II) / Diphenyl(trimethylsilylmethyl)phosphane / Ligand effects

The reaction of diphenyl(trimethylsilylmethyl)phosphane with [AuCl(tht)] affords the gold(I) complexes [AuCl($PPh_2CH_2SiMe_3$)] (**1**) or [Au($PPh_2CH_2SiMe_3$)$_2$]Cl (**2**). An X-ray study of **1** does not reveal any Au···Au interaction. Treatment of [Au(tht)$_2$](CF_3SO_3) or [Au(tht)(PPh_3)](CF_3SO_3) with diphenyl(trimethylsilylmethyl)phosphane results in the displacement of the thioether ligands to give the ionic complexes [Au($PPh_2CH_2SiMe_3$)$_2$](CF_3SO_3) (**3**) or [Au($PPh_2CH_2SiMe_3$)(PPh_3)](CF_3SO_3) (**4**), the latter mixed with the symmetrical derivatives [Au(PPh_2R)$_2$](CF_3SO_3) (R = Ph, CH_2SiMe_3). The molecular structure of complex **3** has been established by X-ray diffraction and shows an antiperiplanar arrangement of the two PR_2R' rotors in the solid state. The reaction of the phosphane ligand with [Au_2{μ-(CH_2)$_2$-PPh_2}$_2$(tht)$_2$](ClO_4)$_2$ affords the dinuclear gold(II) complex [Au_2{μ-(CH_2)$_2PPh_2$}$_2$($PPh_2CH_2SiMe_3$)$_2$](ClO_4)$_2$ (**5**).

Introduction

The supramolecular aggregates of gold(I) complexes of type L−Au−X (X = halide or pseudohalide) include dimers, trimers, higher oligomers, and polymers. The degree of oligomerization is determined by several factors, the steric requirement of the ligands being the most clear.[1][2][3]

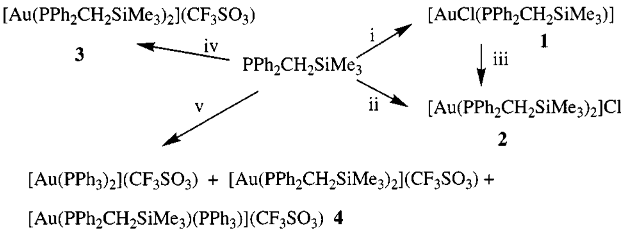
Gold(I) complexes with silanes attracted particular attention after it was found that auration reactions of silanes or silylated alkylgold complexes afford gold(I) clusters with hypercoordinated carbon or nitrogen centers.[4][5][6][7] Gold complexes containing a silicon unit via a bridging O, S, Se, Te, or As donor atom are also known.[8][9][10][11][12] In contrast, no gold complexes with (silylmethyl)phosphanes have been described so far.

In this paper we describe the synthesis of gold(I) and gold(II) complexes with diphenyl(trimethylsilylmethyl)phosphane. The crystal structures of [AuCl($PPh_2CH_2SiMe_3$)] and [Au($PPh_2CH_2SiMe_3$)$_2$](CF_3SO_3) have been solved by X-ray diffraction studies.

Results

The reaction of diphenyl(trimethylsilylmethyl)phosphane with [AuCl(tht)] (tht = tetrahydrothiophene), in molar ratio 1:1, proceeds by displacing the tht ligand to give complex **1**, which by reaction with another $PPh_2CH_2SiMe_3$ affords complex **2** (see Scheme 1). The latter can be also synthesised by reaction of $PPh_2CH_2SiMe_3$ and [AuCl(tht)] (molar ratio 2:1). Treatment of [Au(tht)$_2$](CF_3SO_3) with $PPh_2CH_2SiMe_3$ in a 1:2 molar ratio gives complex **3**, containing two phosphane ligands. Treatment of this phosphane with [Au(tht)(PPh_3)](CF_3SO_3) or [Au(thf)(PPh_3)](CF_3SO_3) (thf = tetrahydrofuran) leads to a mixture of **4** and the corresponding symmetric complexes, **3** and [Au(PPh_3)$_2$](CF_3SO_3), as shown in the $^{31}P\{^1H\}$-NMR spectra (Scheme 1).

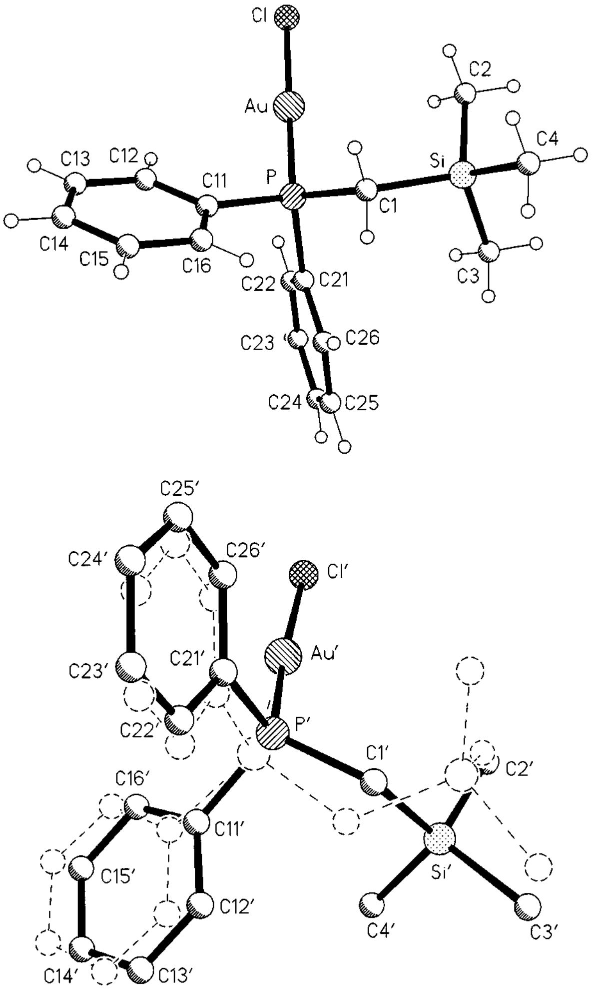
Scheme 1. (i) [AuCl(tht)]; (ii) 2 [AuCl(tht)]; (iii) [AuCl(tht)]; (iv) [Au(tht)$_2$](CF_3SO_3); (v) [Au(tht)(PPh_3)](CF_3SO_3) or [Au(thf)(PPh_3)](CF_3SO_3)



These complexes are air- and moisture-stable white solids at room temperature. They were readily characterised by ^1H and $^{31}P\{^1H\}$ NMR. Their $^{31}P\{^1H\}$-NMR spectra show a singlet at δ = 21.6 (**1**), 27.6 (**2**), and 36.4 (**3**); an AB spin system is observed for **4** (δ_A = 46.7, δ_B = 36.5, $^2J_{AB}$ = 331 Hz), besides two singlets corresponding to symmetric phosphane complexes. In the ^1H-NMR spectra the resonance of the CH_2 is observed at $\delta \approx 2$: a doublet for **1−2** [$^2J_{HP}$ = 15.3 (**1**) and 14.3 (**2**) Hz], a pseudotriplet for **3** (part A of an $A_2XX'A'_2$ system, N = 5.9 Hz) and a doublet of doublets for **4** ($^2J_{HP}$ = 13.7 Hz and $^4J_{HP}$ = 3.5 Hz). Their IR spectra show absorptions at 338 (**1**, medium) and 331 (**2**, very weak) cm^{-1} due to ν(Au−Cl)[13] and to 1264 (br., s) and 636 (m) from triflate[14] (**3**). The LSI-MS mass spectra show peaks at *m/z* (complex, abundance in%) 469 (**1**, 55), 741 (**2**, 100), 741 (**3**, 100), and 731 (**4**, 85) corresponding to $[M - X]^+$ (X = Cl or CF_3SO_3).

The crystal structures of **1** and **3** have been determined by means of X-ray diffraction studies. Complex **1** crystallises with two independent molecules (Figure 1), of which one is disordered over two positions for all atoms except Au and Cl; a suitably restrained refinement proved successful, but the molecular dimensions of the disordered molecule should be interpreted with caution. Here we discuss only the ordered molecule. The gold(I) center displays an almost linear coordination with Cl−Au−P 176.86(6)°. The Au−P distance is 2.236(2) A, similar to that found in other chlorophosphanegold(I) complexes, 2.235(3) A in [AuCl(PPh$_3$)],[15] 2.236(6) A in [AuCl(PPhMe$_2$)],[16] 2.232(9) A in [AuCl(PEt$_3$)],[17] but somewhat longer than in [AuCl(PPh$_2$CH$_2$CONHMe)] [2.221(1) A].[18] The Au−Cl distance is 2.292(2) A, similar to that found in [AuCl(PEt$_3$)] [2.305(8) A], longer than in [AuCl(PPh$_2$CH$_2$CONHMe)] [2.275(2) A] or in [AuCl(PPh$_3$)] [2.279(3) A]; and marginally shorter than in [AuCl(PPhMe$_2$)] [2.316(6) A].

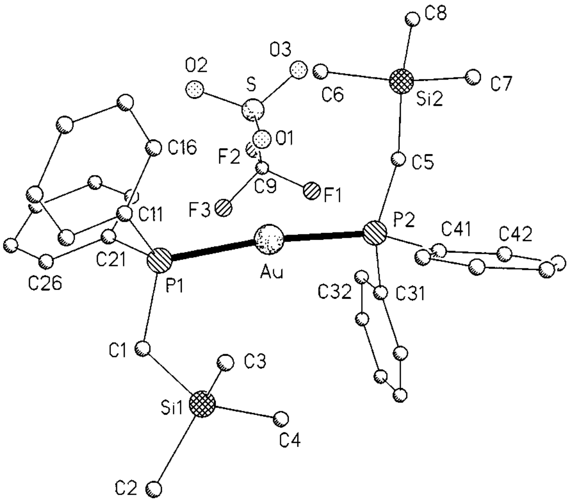
Figure 1. The two independent molecules of compound **1** in the crystal: (a) the ordered molecule; (b) the disordered molecule showing both alternative sites (see text); H atoms omitted for clarity



There are no short gold−gold contacts, the shortest Au−Au distance being 6.6 A. This long distance can be rationalised in terms of the steric bulk of the ligand, but it contrasts with other cases, as the 2.881 (1) A found in [Au(PPhMe$_2$)$_2$][Au(GeCl$_3$)$_2$][3], ca. 3.1 A in [AuX(PPhMe$_2$)] [X = Cl, 3.091(2) and 3.230(2) A; Br, 3.119(2) A; and I 3.104(2) Å] or even the 5.916(1) Å found in [AuCl(AsPh$_3$)][19]. There are no other contacts that could compete with gold−gold interactions, such as the Au···Cl found in [Au{P(*o*-Tol)$_3$}(GeCl$_3$)][3] or the hydrogen bonds in [AuCl(PPh$_2$CH$_2$CONHMe)].

The crystal structure of cation of **3** is shown in Figure 2. The geometry around the gold atom is distorted linear, P(1)−Au−P(2) = 172.12(4)°. The Au−P bond lengths are 2.3080(10) and 2.3149(10) A, similar to that found in other [Au(PR$_2$R′)$_2$]$^+$ complexes, 2.309(3) in [Au(PPh$_2$Me)$_2$][3,4-S$_2$C$_6$H$_3$CH$_3$)$_2$][20], and 2.316(4) A in [Au(PPh$_2$Me)$_2$]PF$_6$,[21] but longer than in [AuCl(PPh$_2$CH$_2$CONHMe)] [2.221(1) A][18] and in **1** [2.236(2) A] probably as a consequence of the lesser *trans* influence of Cl. The PR$_2$R′ ligands adopt an approximately antiperiplanar arrangement, with the torsion angle C1−P1···P2−C5 166°.

Figure 2. The cation of compound **3** in the crystal; H atoms omitted for clarity



As in the similar complexes [Au(PPh$_2$Me)$_2$]$^+$,[20][21][22] there are no short gold−gold contacts, the shortest Au−Au distance being 9.1 A.

The presence of a pseudotriplet (which can be attributed to two superposed doublets) in the ^{1}H-NMR spectrum instead of the expected doublet could indicate a case of propeller isomerism. Schmidbaur and coworkers[23] reported recently that *ortho*-substituted triarylphosphanes can induce this kind of isomerism in complexes with a coordination number of two, even at room temperature, by hindered rotation of the aryl groups about the P−C$_{ipso}$ axes. The variable-temperature ^{1}H-NMR spectrum of **3** shows a triplet for the CH$_2$ resonance and the ^{31}P{^{1}H} spectrum a singlet from −60 to +20°C in CDCl$_3$ solution; when a ^{1}H{^{31}P}-NMR experiment is carried out at −60°C only one singlet (rather than two) is found, which confirms that there is no propeller isomerism but only two magnetically inequivalent CH$_2$ units (as the parts A of an A$_2$XX′A′$_2$ spin system).

We have also prepared gold(II) complexes by displacement of tht from [Au$_2${μ-(CH$_2$)$_2$PPh$_2$}$_2$(tht)$_2$](ClO$_4$)$_2$ [see Eq. (1)]. Complex **5** is a green solid, air- and moisture-stable at room temperature. We were unable to obtain single crystals of **5** but expect its structure to be similar to that of $[Au_2(CH_2PPh_2CH_2)(PPh_3)_2](ClO_4)_2$[24]. The IR spectrum shows absorptions at 1092 (br., s) and 623 (m) cm^{-1} from the perchlorate anion[25] and at 568 (m) from ν(Au−C_{ylide})[26]. The $^{31}P\{^1H\}$-NMR spectrum shows two triplets at δ = 46.7 and 23.6 ($^3J_{PP}$ = 38.8 Hz) as expected for an A_2X_2 spin system. In the ^1H-NMR spectra the resonance of the CH_2-Si unit appears as a pseudotriplet (*N* = 5.4 Hz) at δ = 1.98 whilst a doublet at δ = 1.61 ($^2J_{HP}$ = 11.5 Hz) is observed for CH_2-Au; again when a $^1H\{^{31}P\}$-NMR experiment is carried out only a singlet is found for the CH_2-Si unit. The LSI-MS mass spectrum show peaks at *m/z* (abundance in%) 1463 (25) from $[M - ClO_4]^+$ whilst the base peak appears at 741, corresponding to $[Au(PPh_2CH_2SiMe_3)_2]^+$.

$$[Au_2\{\mu\text{-}(CH_2)_2PPh_2\}_2(tht)_2](ClO_4)_2 + 2\ PPh_2CH_2SiMe_3 \rightarrow$$
$$\underset{\mathbf{5}}{[Au_2\{\mu\text{-}(CH_2)_2PPh_2\}_2(PPh_2CH_2SiMe_3)_2](ClO_4)_2} + 2\ tht \quad (1)$$

We thank the *Direccion General de Investigacion Cientifica y Tecnica* (Project PB94-0079) and the *Fonds der Chemischen Industrie* for financial support.

Experimental Section

General: All the reactions were carried out under argon at room temperature. $[Au(tht)_2](CF_3SO_3)$, $[Au(tht)(PPh_3)](CF_3SO_3)$ and $[Au(thf)(PPh_3)](CF_3SO_3)$ were prepared in situ by reaction of [AuCl(tht)] or $[AuCl(PPh_3)]$ with $[Ag(CF_3SO_3)(tht)]$ or $[Ag(CF_3SO_3)]$ according to a standard procedure: [AuCl(tht)][27], $PPh_2CH_2SiMe_3$[28], and $[Au_2\{\mu\text{-}(CH_2)_2PPh_2\}_2(tht)_2](ClO_4)_2$[24] were prepared according to literature procedures. − IR spectra were recorded on a Perkin-Elmer 883 spectrophotometer, over the range 4000−200 cm^{-1}, by using Nujol mulls between polyethylene sheets. − ^1H- and ^{31}P-NMR spectra were recorded with a Bruker ARX·300 or GEMINI 2000 apparatus in $CDCl_3$ solutions; chemical shifts are quoted relative to $SiMe_4$ (external, ^1H) and H_3PO_4 (external, ^{31}P). − C, H, and N analyses were performed with a Perkin-Elmer 2400 microanalyser. − Melting points were measured with a Büchi apparatus and are uncorrected. − Mass spectra were recorded with a VG Autospec using LSI-MS^+ techniques (with Cs gun) and 3-nitrobenzyl alcohol as matrix.

Caution: Perchlorate salts or derivatives must be manipulated with caution, mainly by avoiding evaporation to dryness.

[AuCl(PPh$_2$CH$_2$SiMe$_3$)] (**1**): To a dichloromethane solution (15 ml) of [AuCl(tht)] (96 mg, 0.3 mmol) was added $PPh_2CH_2SiMe_3$ (82 mg, 0.3 mmol). After stirring for 3 h, the solution was concentrated to ca. 2 ml and hexane (15 ml) was added to obtain **1** as a white solid. **1** was washed with hexane (2 × 5 ml). Yield: 70%, m.p. 118°C. − ^1H NMR: δ = 7.7−7.4 (m, 10 H, Ph), 1.83 (d, 2 H, $^2J_{HP}$ = 15.3 Hz, CH_2-P), 0.08 (s, 9 H, Me). − $C_{16}H_{21}AuClPSi$ (504.82): calcd. C 38.05, H 4.2; found C 37.75, H 3.85.

[Au(PPh$_2$CH$_2$SiMe$_3$)$_2$]Cl (**2**): This product can be synthesised by two different routes: a) To a solution of [AuCl(tht)] (0.064 g, 0.2 mmol) in dichloromethane (10 ml) was added $PPh_2CH_2SiMe_3$ (109 mg, 0.4 mmol). After stirring for 1 h, the solution was concentrated to ca. 2 ml and hexane (15 ml) was added. **2** appeared as a white solid which was washed with hexane (2 × 5 ml). Yield: 70%. b) To a dichloromethane solution (15 ml) of **1** (50 mg, 0.1 mmol) was added $PPh_2CH_2SiMe_3$ (27 mg, 0.1 mmol). After stirring for 1 h, the solution was concentrated to ca. 2 ml and hexane (15 ml) was added. **2** appeared as a white solid which was washed with hexane (2 × 5 ml). Yield: 80%, m.p. 125°C. − ^1H NMR: δ = 7.8−7.3 (m, 10 H, Ph), 2.00 (d, 2 H, $^2J_{HP}$ = 14.3 Hz, CH_2-P), −0.03 (s, 9 H, Me). − $C_{32}H_{42}AuClP_2Si_2$ (777.22) : calcd. C 49.45, H 5.45; found C 49.25, H 5.55.

[Au(PPh$_2$CH$_2$SiMe$_3$)$_2$](CF$_3$SO$_3$) (**3**): To a dichloromethane solution (20 ml) of $[Au(tht)_2](CF_3SO_3)$ (0.15 mmol, prepared "in situ") was added $PPh_2CH_2SiMe_3$ (82 mg, 0.3 mmol). After stirring for 2 h, the solution was concentrated to ca. 2 ml and diethyl ether (15 ml) was added to obtain **3** as a white solid. **3** was washed with diethyl ether. Yield: 70%, m.p. 136°C. − ^1H NMR: δ = 7.7−7.5 (m, 10 H, Ph), 2.08 (t, 2 H, $^2J_{HP}$ = 5.9 Hz, CH_2-P), −0.02 (s, 9 H, Me). − ^1H NMR (−60°C): δ = 7.7−7.5 (m, 10 H, Ph), 2.01 (t, 2 H, $^2J_{HP}$ = 5.7 Hz, CH_2-P), −0.01 (s, 9 H, Me). − $^{31}P\{^1H\}$ NMR (−60°C): δ = 35.8 (s). − $^1H\{^{31}P\}$ NMR (−60°C): δ = 7.8−7.5 (m, 10 H, Ph), 2.01 (s, 2 H, CH_2-P), −0.02 (s, 9 H, Me).− $C_{33}H_{42}AuF_3O_3P_2SSi_2$ (890.84) : calcd. C 44.5, H 4.75, S 3.6; found C 44.4, H 4.65, S 3.8.

[Au(PPh$_2$CH$_2$SiMe$_3$)(PPh$_3$)](CF$_3$SO$_3$) (**4**): This complex, mixed with the corresponding symmetric $[Au(PPh_2R)_2](CF_3SO_3)$ (R = Ph, CH_2SiMe_3), was prepared by two different ways. a) To a dichloromethane solution (20 ml) of $[Au(tht)(PPh_3)](CF_3SO_3)$ (0.15 mmol, prepared "in situ") was added $PPh_2CH_2SiMe_3$ (41 mg, 0.15 mmol). After stirring for 2 h, the solution was concentrated to ca. 2 ml and diethyl ether (15 ml) was added. The white solid was washed with diethyl ether. b) As for a) but using $[Au(thf)(PPh_3)](CF_3SO_3)$, prepared in situ in thf.

[Au$_2${μ-(CH$_2$)$_2$PPh$_2$}$_2$(PPh$_2$CH$_2$SiMe$_3$)$_2$](ClO$_4$)$_2$ (**5**): To a dichloromethane solution (10 ml) of $[Au_2\{\mu\text{-}(CH_2)_2PPh_2\}_2(tht)_2](ClO_4)_2$ (60 mg, 0.05 mmol) was added $PPh_2CH_2SiMe_3$ (27 mg, 0.1 mmol). After stirring for 2 h, the solution was concentrated to ca. 1 ml and diethyl ether (15 ml) was added to obtain **5** as a green solid. **5** was washed with diethyl ether. Yield: 85%, m.p. 120°C (decomp.). − ^1H NMR: δ = 7.5−7.0 (m, 40 H, Ph), 1.98 (t, 4 H, $^2J_{HP}$ = 5.4 Hz, $P-CH_2-Si$), 1.61 (d, 8 H, $^2J_{HP}$ = 11.5 Hz, $Au-CH_2-P$), −0.20 (s, 18 H, Me). − $^1H\{^{31}P\}$ NMR: δ = 7.5−7.0 (m, 40 H, Ph), 1.95 (s, 4 H, $P-CH_2-Si$), 1.60 (s, 8 H, $Au-CH_2-P$), −0.20 (s, 18 H, Me). − $C_{60}H_{70}Au_2Cl_2O_8P_4Si_2$ (1564.11) : calcd. C 46.05, H 4.5; found C 45.75, H 4.15.

Crystal Structure Determinations: Crystal data and refinement details are given in Table 1. Crystals of **1** were obtained by slow evaporation of a dichloromethane/ethanol slution; crystals of **3** were obtained by slow diffusion at −18°C of diethyl ether into a dichloromethane solution. − *Data collection:* Crystals were mounted in inert oil and transferred to the cold gas stream of a Siemens P4 diffractometer. Intensity measurements were performed using Mo-K_α radiation. Absorption corrections were based on ψ scans. − *Structure solution and refinement:* Solution by the heavy-atom method, anisotropic refinement on F^2 (program SHELXL-93, G.M. Sheldrick, University of Göttingen). H atoms with riding model or as rigid methyl groups. − *Special features*: For **1**, the second independent molecule is disordered, all atoms except Au and Cl occupying two alternative orientations. Nevertheless, the use of appropriate similarity restraints enabled a satisfactorily stable refinement to be performed. Disordered C and Si atoms were refined isotropically. − Full details of the crystal structures (except structure factors) have been deposited at the Cambridge Crystallo-

Table 1. Details of data collection and structure refinement for complexes **1** and **3**

Compound	**1**	**3**
Chemical Formula	$C_{16}H_{21}AuClPSi$	$C_{33}H_{42}AuF_3O_3P_2SSi_2$
Crystal habit	colourless prism	colourless tablet
Crystal size [mm]	0.55×0.20×0.10	0.60×0.30×0.15
Crystal system	triclinic	monoclinic
Space group	$P(-1)$	$P2_1/c$
a [Å]	9.9920(12)	11.378(2)
b [Å]	10.746(2)	18.880(2)
c [Å]	17.707(3)	17.993(2)
α [°]	94.99(2)	90
β [°]	104.97(2)	99.43(1)
γ [°]	94.34(2)	90
V [Å^3]	1820.4(5)	3813.2(9)
Z[a]	4	4
D_c [Mg m^{-3}]	1.842	1.552
M	504.80	890.81
F(000)	968	1776
T [°C]	−100	−100
$2\theta_{max}$ [°]	50	50
μ(Mo-K_α) [mm^{-1}]	8.37	4.11
Transmissions	0.49−1.00	0.63−0.96
No. of reflections measured	8494	7220
No. of unique reflections	6389	6714
R_{int}	0.020	0.022
R[a] [F, $F > 4\sigma(F)$]	0.029	0.026
wR[b] (F^2, all refl.)	0.058	0.047
No. of parameters	276	412
No. of restraints	244	280
S[c]	0.90	0.84
Max. Δρ [eÅ^{-3}]	0.6	0.63

[a] $R(F) = \Sigma \| F_o| - |F_c\| / \Sigma |F_o|$. − [b] $wR(F^2) = [\Sigma\{w(F_o^2 - F_c^2)^2\}/\Sigma\{w(F_o^2)^2\}]^{0.5}$; $w^{-1} = \sigma^2(F_o^2) + (aP)^2 + bP$, where $P = [F_o^2 + 2F_c^2]/3$ and a and b are constants adjusted by the program. − [c] $S = [\Sigma\{w(F_o^2 - F_c^2)^2\}/(n - p)]^{0.5}$, where n is the number of data and p the number of parameters.

graphic Data Centre, and can be obtained free of charge from CCDC, 12 Union Road, Cambridge CB2 1EZ, England [Fax: (internat.) + 44(0)1223/336033; E-mail: deposit@ccdc.cam.ac.uk] on quoting the reference number CCDC-101120.

[1] P. Pyykkö, N. Runeberg, F. Mendizabal, *Chem. Eur. J.* **1997**, *3*, 1451.

[2] W. Schneider, K. Angermaier, A. Sladek, H. Schmidbaur, *Z. Naturfosch* **1996**, *51b*, 790.

[3] A. Bauer, H. Schmidbaur, *J. Am. Chem. Soc.* **1996**, *118*, 5324.

[4] F.P. Gabbaı, A. Schier, J. Riede, H. Schmidbaur, *Chem. Ber.* **1997**, *130*, 111.

[5] H. Schmidbaur, F.P. Gabbaı, A. Schier, J. Riede, *Organometallics* **1995**, *14*, 4969.

[6] K. Angermaier, H. Schmidbaur, *Chem. Ber.* **1995**, *128*, 817.

[7] S. Bommers, H. Beruda, N. Dufour, M. Paul, A. Schier, H. Schmidbaur, *Chem. Ber.* **1995**, *128*, 137.

[8] A. Bauer, W. Schneider, K. Angermaier, A. Schier, H. Schmidbaur, *Inorg. Chim. Acta* **1996**, *251*, 249.

[9] W. Wojnowski, B. Becker, J. Sassmannhausen, E. M. Peters, K. Peters, H. G. von Schnering, *Z. Anorg. Allg. Chem.* **1994**, *620*, 1417.

[10] P. J. Bonasia, D. E. Gindelberger, J. Arnold, *Inorg. Chem.* **1993**, *32*, 5126.

[11] W. Eikens, P. G. Jones, C. Thone, *Z. Anorg. Allg. Chem.* **1997**, *623*, 735.

[12] D. Fenske, F. Simon, *Z. Anorg. Allg. Chem.* **1996**, *622*, 45.

[13] R. Uson, A. Laguna, M. Laguna, M. N. Fraile, P. G. Jones, G. M. Sheldrick, *J. Chem. Soc., Dalton Trans.* **1986**, 291.

[14] P. J. Stang, Y. Huang, A. M. Arif, *Organometallics* **1992**, *11*, 231.

[15] N.C. Baenziguer, W. E. Bennett, D. M. Soboroff, *Acta Crystallogr., Sect. B* **1976**, *32*, 962.

[16] D. Toronto, B. Weissbart, D. Tinti, A. Balch, *Inorg. Chem.* **1996**, *35*, 2484.

[17] E. R. T. Tiekink, *Acta Crystallogr.* **1989**, *C45*, 1233.

[18] P. Lange, A. Schier, H. Schmidbaur, *Inorg. Chim. Acta* **1995**, *235*, 263.

[19] B. Weissbart, L. J. Larson, M. M. Olmstead, C.P. Nash, D.S. Tinti, *Inorg. Chem.* **1995**, *34*, 393.

[20] M. C. Gimeno, P. G. Jones, A. Laguna, M. Laguna, R. Terroba, *Inorg. Chem.* **1994**, *33*, 3932.

[21] J. J. Guy, P. G. Jones, G. M. Sheldrick, *Acta Crystallogr., Sect. B* **1976**, *32*, 1937.

[22] S. M. Draper, C. E. Housecroft, J. E. Rees, M. S. Shongwe, B. S. Haggerty, A. L. Rheingold, *Organometallics* **1992**, *11*, 2356.

[23] A. Bayler, G. A. Bowmaker , H. Schmidbaur, *Inorg. Chem.* **1996**, *35*, 5959.

[24] R. Uson, A. Laguna, M. Laguna, J. Jimenez, P. G. Jones, *J. Chem. Soc., Dalton Trans.* **1991**, 1361.

[25] M. N. Gowda, S. B. Naikar, G. K. N. Reddy, *Adv. Inorg. Chem. Radiochem.* **1984**, *28*, 255.

[26] H. Schmidbaur, R. Franke, *Chem. Ber.* **1975**, *108*, 1321.

[27] R. Uson, A. Laguna, M. Laguna, *Inorg. Synth.* **1989**, *26*, 85.

[28] B. E. Cooper, W. J. Owen, *J. Organomet. Chem.* **1970**, *21*, 329.

[98033]