

## Trimethylsilylmethyl gold(I) complexes. X-ray structure of [Au(CH<sub>2</sub>SiMe<sub>3</sub>)PPh<sub>2</sub>CH<sub>2</sub>PPh<sub>2</sub>Me]ClO<sub>4</sub> · 0.25CH<sub>2</sub>Cl<sub>2</sub>

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Received 23 September 1996

### Abstract

The reaction of [AuCl(L)] (L = AsPh<sub>3</sub> or PPh<sub>3</sub>) with Mg(CH<sub>2</sub>SiMe<sub>3</sub>)Cl gives the neutral complexes [Au(CH<sub>2</sub>SiMe<sub>3</sub>)L] [L = AsPh<sub>3</sub> (1) or PPh<sub>3</sub> (2)]. Complex 1 undergoes substitution reactions with monodentate ligands to afford mononuclear complexes [Au(CH<sub>2</sub>SiMe<sub>3</sub>)PPh<sub>3</sub>] or [Au(CH<sub>2</sub>SiMe<sub>3</sub>)L']ClO<sub>4</sub> [L' = PPh<sub>2</sub>CH<sub>2</sub>PPh<sub>2</sub>Me (3) or PPh<sub>2</sub>CH<sub>2</sub>PPh<sub>2</sub>CH<sub>2</sub>C<sub>6</sub>F<sub>5</sub> (4)]. If a potentially bidentate ligand is used, the latter reaction gives mononuclear or dinuclear complexes, [Au(CH<sub>2</sub>SiMe<sub>3</sub>)dppe] (dppe = PPh<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>PPh<sub>2</sub>) (5) or [(Me<sub>3</sub>SiCH<sub>2</sub>)Au(L-L)Au(CH<sub>2</sub>SiMe<sub>3</sub>)] [L-L = dpmm (6) or dppe (7)] (dpmm = PPh<sub>2</sub>CH<sub>2</sub>PPh<sub>2</sub>), depending on the molar ratio. The structure of [Au(CH<sub>2</sub>SiMe<sub>3</sub>)PPh<sub>2</sub>CH<sub>2</sub>PPh<sub>2</sub>Me]ClO<sub>4</sub> has been established by an X-ray diffraction study. © 1997 Elsevier Science S.A.

### 1. Introduction

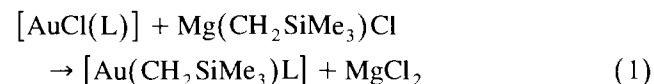
Gold(I) compounds containing the trimethylsilylmethyl group as a simple (terminal) ligand have been obtained by the reaction of corresponding alkylolithium or Grignard reagent with a complex gold(I) chloride [1–4], or by displacement of a phosphine ligand from [Au(CH<sub>2</sub>SiMe<sub>3</sub>)L] (L = PPh<sub>3</sub>, PMe<sub>3</sub>) by other more strongly coordinating donors such as ylide ligands [3]. It has been reported that the complex [Au(CH<sub>2</sub>SiMe<sub>3</sub>)(AsPh<sub>3</sub>)] cannot be obtained from the chlorogold(I) precursor and Li(CH<sub>2</sub>SiMe<sub>3</sub>), but the synthesis via the Grignard route has not been tested.

In this paper we describe the synthesis of this latter compound, [Au(CH<sub>2</sub>SiMe<sub>3</sub>)(AsPh<sub>3</sub>)], which is a better intermediate than the analogous phosphine derivatives since AsPh<sub>3</sub> is only weakly coordinating. Accordingly, the arsine can be easily displaced by monodentate or bidentate phosphine ligands to afford the new complexes [Au(CH<sub>2</sub>SiMe<sub>3</sub>)PPh<sub>2</sub>CH<sub>2</sub>PPh<sub>2</sub>R]ClO<sub>4</sub> [R = Me (3), CH<sub>2</sub>C<sub>6</sub>F<sub>5</sub> (4)], [Au(CH<sub>2</sub>SiMe<sub>3</sub>)dppe] (5) or

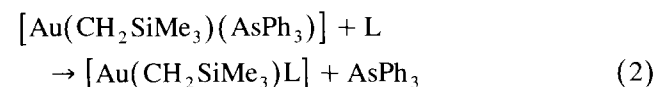
[(Me<sub>3</sub>SiCH<sub>2</sub>)Au(L-L)Au(CH<sub>2</sub>SiMe<sub>3</sub>)] [L-L = dpmm (6) or dppe (7)].

### 2. Results and discussion

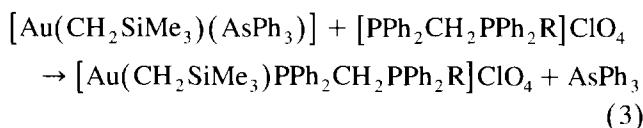
The reaction in diethyl ether of [AuCl(L)] (L = AsPh<sub>3</sub> or PPh<sub>3</sub>) with Mg(CH<sub>2</sub>SiMe<sub>3</sub>)Cl in molar ratio 1:1.5 at 0 °C leads to the formation of [Au(CH<sub>2</sub>SiMe<sub>3</sub>)L] [L = AsPh<sub>3</sub> (1) or PPh<sub>3</sub> (2)] [Eq. (1)]:



In the chemistry of gold(I), the ligand AsPh<sub>3</sub> is weakly coordinating and can therefore be readily displaced by most other ligands. The complex [Au(CH<sub>2</sub>SiMe<sub>3</sub>)(AsPh<sub>3</sub>)] (1) behaves similarly and reacts with PPh<sub>3</sub>, dppe or [PPh<sub>2</sub>CH<sub>2</sub>PPh<sub>2</sub>R]ClO<sub>4</sub> [R = Me or CH<sub>2</sub>C<sub>6</sub>F<sub>5</sub>], in 1:1 ratio, to give mononuclear complexes [Au(CH<sub>2</sub>SiMe<sub>3</sub>)(PPh<sub>3</sub>)] (2), [Au(CH<sub>2</sub>SiMe<sub>3</sub>)dppe] (5) or [Au(CH<sub>2</sub>SiMe<sub>3</sub>)PPh<sub>2</sub>CH<sub>2</sub>PPh<sub>2</sub>R]ClO<sub>4</sub> [R = Me (3), CH<sub>2</sub>C<sub>6</sub>F<sub>5</sub> (4)] [Eqs. (2) and (3)]:

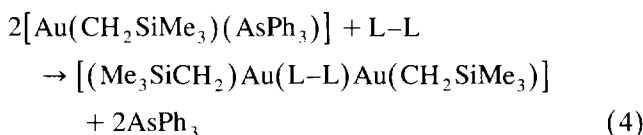


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Complex **2** has previously been prepared by metathesis between the chlorogold(I) precursor and  $\text{Li}(\text{CH}_2\text{SiMe}_3)$  [1,2] or by reaction of  $\text{H}[\text{AuCl}_4]$  with  $\text{PPh}_3$  and  $\text{Mg}(\text{CH}_2\text{SiMe}_3)\text{Cl}$  [2].

The treatment of  $[\text{Au}(\text{CH}_2\text{SiMe}_3)(\text{AsPh}_3)]$  with potentially bidentate ligands such as *dppm* or *dppe*, in molar ratio 2:1, in dichloromethane leads to the formation of the dinuclear complexes  $[(\text{Me}_3\text{SiCH}_2)\text{Au}(\text{L}-\text{L})\text{Au}(\text{CH}_2\text{SiMe}_3)]$  [ $\text{L}-\text{L} = \text{dppm}$  (**6**) or *dppe* (**7**)] [Eq. (4)]:



All complexes are unstable in the solid state or in solution at room temperature, but they can be stored for several weeks at  $-20^\circ\text{C}$ . Acetone solutions of **1**, **2**, **5–7** are non-conducting and those of **4** and **5** display conductivities typical of 1:1 electrolytes [5] (Table 1). The IR spectra show a weak band at  $\sim 540\text{cm}^{-1}$  assignable to  $\nu(\text{Au}-\text{C})$  [2],  $\nu(\text{Si}-\text{CH}_3)$  absorptions from trimethylsilylmethyl ligand at 825(s), 845(m), 860(s) (**1**), 826(s), 844(m), 859(s) (**2**), 828(s), 855(w) (**3**), 824(s), 857(s) (**4**), 826(s), 859(s) (**5**), 823(s), 842(m), 856(m) (**6**) or at 828(s), 855(s)  $\text{cm}^{-1}$  (**7**), and a medium band at  $\sim 1240\text{cm}^{-1}$  assignable to  $\delta(\text{CH}_3)$ . Furthermore, **3** and **4** show two bands due to the  $\text{ClO}_4$  anion at

1072(s,br), 623(m) (**3**) or at 1096(s,br), 623(m)  $\text{cm}^{-1}$  (**4**) [6].

Their  $^1\text{H}$  NMR spectra are as expected (Table 1) showing two resonances for the trimethylsilylmethyl ligand. The  $^{31}\text{P}\{^1\text{H}\}$  NMR spectra show a singlet for the phosphine in **2**, **6** and **7** [ $\delta$  46.3 (**2**), 34.8 (**6**) and 43.9 (**7**)], and two signals for the diphosphine in complexes **3** and **4** at 30.0(br), 21.9(br) ppm (**3**) or at 28.5(d), 24.3(d) ppm [ $^2J_{\text{P}-\text{P}} = 21\text{ Hz}$ ] (**4**). Complex **5** shows a single signal at 21.5 ppm for the two phosphorus atoms even at  $-50^\circ\text{C}$  in deuteriochloroform, but this signal splits at  $-80^\circ\text{C}$  in deuterotoluene (42.2(d) and  $-15.3(\text{d})$  ppm,  $^2J_{\text{P}-\text{P}} = 42\text{ Hz}$ ). Therefore, tricoordination around the gold atom can be rejected.

The positive-ion fast atom bombardment mass spectra show the parent ion for complexes **3** [ $m/z = 683$  (70%)], **4** [ $m/z = 850$  (43%)], **5** [ $m/z = 683$  (15%)] and **7** [ $m/z = 966$  (20%)]. Other peaks appear at  $m/z = 576$  (62%,  $[\text{M}-\text{CH}_3]^+$ ) for **1**, 531 (26%,  $[\text{M}-\text{CH}_3]^+$ ) for **2**, 399 (100%,  $[\text{PPh}_2\text{CH}_2\text{PPh}_2\text{Me}]^+$ ) for **3**, 565 (100%,  $[\text{PPh}_2\text{CH}_2\text{PPh}_2\text{CH}_2\text{C}_6\text{F}_5]^+$ ) for **4**, 668 (95%,  $[\text{M}-\text{CH}_3]^+$ ) for **5**, 1537 (100%,  $[\text{M}+\text{Audppm}]^+$ ) and 867 (52%,  $[\text{M}-(\text{CH}_2\text{SiMe}_3)]^+$ ) for **6** and 879 (69%,  $[\text{M}-(\text{CH}_2\text{SiMe}_3)]^+$ ) for **7**.

The structure of complex **3** has been determined by single-crystal X-ray diffraction (Fig. 1). The unit cell contains 16 molecules of **3** (two independent) and four of dichloromethane (with crystallographic two-fold symmetry). The structure is however not very precise, because, despite low temperature measurements, the trimethylsilyl group of one molecule displays high displacement parameters and that of the other is disordered

Table 1  
Analytical and spectroscopic data for complexes **1–7**

Complex	Yield (%)	Analysis (%) <sup>a</sup>		$A_{\text{M}}$ <sup>b</sup> ( $\text{ohm}^{-1}\text{cm}^2$ $\text{mol}^{-1}$ )	$^1\text{H}$ NMR <sup>c</sup> , $\delta(\text{CH}_2\text{SiMe}_3)$		$\nu(\text{Au}-\text{C})$ ( $\text{cm}^{-1}$ )
		C	H		$\delta(\text{CH}_2)$ <sup>d</sup>	$\delta(\text{CH}_3)$ <sup>e</sup>	
<b>1</b> $[\text{Au}(\text{CH}_2\text{SiMe}_3)(\text{AsPh}_3)]$	95	44.5 (44.75)	4.3 (4.45)	0.4	0.42(s)	0.05(s)	548
<b>2</b> $[\text{Au}(\text{CH}_2\text{SiMe}_3)(\text{PPh}_3)]$	86	48.8 (48.35)	5.0 (4.8)	0.3	0.32(d) [9.6]	0.05(s)	534
<b>3</b> $[\text{Au}(\text{CH}_2\text{SiMe}_3)\text{PPh}_2\text{CH}_2\text{PPh}_2\text{Me}]\text{ClO}_4$	91	45.75 (46.0)	4.5 (4.6)	108	0.04(d) [5.1]	$-0.03(\text{s})$ <sup>e</sup>	546
<b>4</b> $[\text{Au}(\text{CH}_2\text{SiMe}_3)\text{PPh}_2\text{CH}_2\text{PPh}_2\text{CH}_2\text{C}_6\text{F}_5]\text{ClO}_4$	83	45.2 (45.6)	3.8 (3.7)	112	$-0.16(\text{d})$ [10.3]	$-0.07(\text{s})$ <sup>f</sup>	546
<b>5</b> $[\text{Au}(\text{CH}_2\text{SiMe}_3)(\text{dppe})]$	70	51.9 (52.8)	5.25 (5.15)	4	0.28(m)	0.05(s) <sup>g</sup>	521
<b>6</b> $[(\text{Me}_3\text{SiCH}_2)\text{Au}(\text{dppm})\text{Au}(\text{CH}_2\text{SiMe}_3)]$	81	42.05 (41.6)	5.05 (4.65)	0.8	0.4('t') [9.9] <sup>h</sup>	0.02(s) <sup>i</sup>	523
<b>7</b> $[(\text{Me}_3\text{SiCH}_2)\text{Au}(\text{dppe})\text{Au}(\text{CH}_2\text{SiMe}_3)]$	50	42.75 (42.25)	5.0 (4.8)	0.9	0.28('t') [10] <sup>h</sup>	0.05(s) <sup>j</sup>	521

<sup>a</sup> Calculated values are given in parentheses.

<sup>b</sup> In acetone ( $5 \times 10^{-4}\text{ mol l}^{-1}$ ).

<sup>c</sup> In  $\text{CDCl}_3$ , values in ppm.

<sup>d</sup> Values of  $J$  and  $N$  in hertz.

<sup>e</sup>  $\delta(\text{CH}_3$  of  $\text{PPh}_2\text{CH}_2\text{PPh}_2\text{Me}^+) = 2.60(\text{d})$  [ $^2J_{\text{P}-\text{H}} = 13.3\text{ Hz}$ ],  $\delta(\text{CH}_2$  of  $\text{PPh}_2\text{CH}_2\text{PPh}_2\text{Me}^+) = 4.3(\text{dd})$  [ $^2J_{\text{P}-\text{H}} = 14.7\text{ Hz}$ ,  $^2J_{\text{P}-\text{H}} = 8.8\text{ Hz}$ ].

<sup>f</sup>  $\delta(\text{CH}_2$  of  $\text{PPh}_2\text{CH}_2\text{PPh}_2\text{CH}_2\text{C}_6\text{F}_5^+) = 4.53(\text{dd})$  [ $^2J_{\text{P}-\text{H}} = 13.9\text{ Hz}$ ,  $^2J_{\text{P}-\text{H}} = 8.0\text{ Hz}$ ],  $\delta(\text{CH}_2$  of  $\text{PPh}_2\text{CH}_2\text{PPh}_2\text{CH}_2\text{C}_6\text{F}_5^+) = 4.76(\text{d})$  [ $^2J_{\text{P}-\text{H}} = 13.9\text{ Hz}$ ].

<sup>g</sup>  $\delta(\text{CH}_2$  of *dppe*) = 2.39(m).

<sup>h</sup>  $N$  is the splitting between the external lines.

<sup>i</sup>  $\delta(\text{CH}_2$  of *dppm*) = 3.40(t) [ $^2J_{\text{P}-\text{H}} = 9.7\text{ Hz}$ ].

<sup>j</sup>  $\delta(\text{CH}_2$  of *dppe*) = 2.39(m).

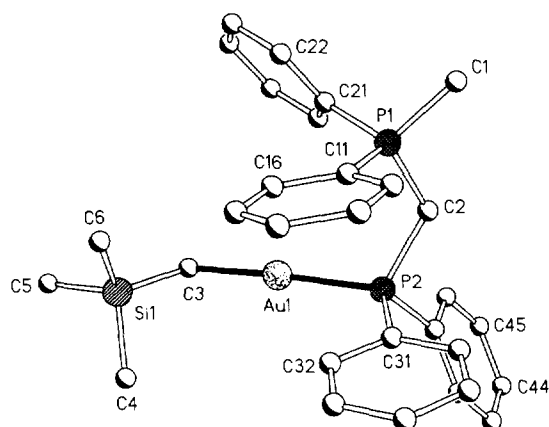


Fig. 1. Molecular structure of complex **3** showing the atom numbering scheme. Radii are arbitrary, hydrogen atoms are omitted for clarity.

over two positions. The gold atom is two-coordinate with P–Au–C angles of 175.4, 176.4(4)°, close to the linear stereochemistry preferred by Au<sup>I</sup>. The Au–P bond lengths of 2.273, 2.282(3) Å are similar to those observed in other P–Au–C systems such as [Au(C<sub>6</sub>F<sub>5</sub>)PPh<sub>2</sub>CHPPh<sub>2</sub>Me] (2.287(2) Å) [7], [Au(C<sub>6</sub>F<sub>5</sub>)PPh<sub>3</sub>] (2.27(1) Å) [8], [(Ph<sub>3</sub>P)Au(mes)Ag(tht)]<sub>2</sub>[SO<sub>3</sub>CF<sub>3</sub>]<sub>2</sub> (2.2886(9) Å) [9], or [Ag(μ-dppm)<sub>2</sub>{Au(mes)}<sub>2</sub>]ClO<sub>4</sub> (2.315(5) Å) [10]. The Au–C bond distances, 2.070(11) and 2.071(12) Å, are of the same order as those in [Au{2,6-(MeO)<sub>2</sub>C<sub>6</sub>H<sub>3</sub>}PPh<sub>3</sub>] (2.050(4) Å) [11], [Au(C<sub>6</sub>F<sub>5</sub>)<sub>2</sub>]<sup>−</sup> (2.062(8) and 2.041(9) Å) [12] or [(Ph<sub>3</sub>P)Au(mes)Ag(tht)]<sub>2</sub>[SO<sub>3</sub>CF<sub>3</sub>]<sub>2</sub> (2.086(3) Å) [9].

### 3. Experimental section

Instrumentation and general experimental techniques were as described earlier [10]. The yields, C and H analyses, proton NMR and conductivity data are listed in Table 1. All the reactions were performed at room temperature except that of [AuCl(L)] (L = AsPh<sub>3</sub> or PPh<sub>3</sub>) with Mg(CH<sub>2</sub>SiMe<sub>3</sub>)Cl.

#### 3.1. Syntheses

##### 3.1.1. [Au(CH<sub>2</sub>SiMe<sub>3</sub>)L] [L = AsPh<sub>3</sub> (1) or PPh<sub>3</sub> (2)]

To a solution of [AuCl(L)] [L = AsPh<sub>3</sub> [13] (0.538 g, 1 mmol) or PPh<sub>3</sub> [14] (0.495 g, 1 mmol)] in 30 cm<sup>3</sup> of diethyl ether was added a solution of Mg(CH<sub>2</sub>SiMe<sub>3</sub>)Cl (1.5 mmol) in tetrahydrofuran (1.5 cm<sup>3</sup>) at 0°C under nitrogen. The mixture was stirred for 2 h at this temperature and then a drop of water was added. The solution was evaporated to dryness and dichloromethane (20 cm<sup>3</sup>) was added. Filtration and subsequent evaporation to dryness led to complexes **1** or **2** as white solids.

##### 3.1.2. [Au(CH<sub>2</sub>SiMe<sub>3</sub>)L] [L = PPh<sub>3</sub> (2) or dppe (5)] or [Au(CH<sub>2</sub>SiMe<sub>3</sub>)L']ClO<sub>4</sub> [L' = PPh<sub>2</sub>CH<sub>2</sub>PPh<sub>2</sub>Me (3) or PPh<sub>2</sub>CH<sub>2</sub>PPh<sub>2</sub>CH<sub>2</sub>C<sub>6</sub>F<sub>5</sub> (4)]

To a dichloromethane solution (20 cm<sup>3</sup>) of complex **1** (0.059 g, 0.1 mmol) was added PPh<sub>3</sub> (0.026 g, 0.1 mmol), dppe (0.040 g, 0.1 mmol) or [PPh<sub>2</sub>CH<sub>2</sub>PPh<sub>2</sub>R]ClO<sub>4</sub> [R = Me (0.050 g, 0.1 mmol) or CH<sub>2</sub>C<sub>6</sub>F<sub>5</sub> (0.067 g, 0.1 mmol)]. After stirring for 20 min the solution was evaporated to dryness to obtain complex **2** or concentrated to ca. 5 cm<sup>3</sup> and n-hexane (20 cm<sup>3</sup>) added to precipitate complexes **3–5** as white solids.

##### 3.1.3. [(Me<sub>3</sub>SiCH<sub>2</sub>)Au(L–L)Au(CH<sub>2</sub>SiMe<sub>3</sub>)] [L–L = dppm (6) or dppe (7)]

To a solution of complex **1** (0.118 g, 0.2 mmol) in dichloromethane (20 cm<sup>3</sup>) was added dppm (0.038 g, 0.1 mmol) or dppe (0.040 g, 0.1 mmol) and the mixture was stirred for 20 min. Partial concentration of the solution to ca. 5 cm<sup>3</sup> and addition of n-hexane (20 cm<sup>3</sup>) led to the precipitation of complexes **6** and **7** as white solids.

### 3.2. Crystal structure determination of compound **3**

#### 3.2.1. Crystal data

**3**. 0.25CH<sub>2</sub>Cl<sub>2</sub>: C<sub>30.25</sub>H<sub>36.5</sub>AuCl<sub>1.5</sub>O<sub>4</sub>P<sub>2</sub>Si, *M* = 804.27, monoclinic, space group *C2/c*, *a* = 38.892(5), *b* = 15.048(3), *c* = 24.655(3) Å, β = 103.120(10)°, *U* = 14053(4) Å<sup>3</sup>, *Z* = 16, *D*<sub>c</sub> = 1.521 Mg m<sup>−3</sup>, λ(Mo Kα) = 0.71073 Å, μ = 4.46 mm<sup>−1</sup>, *F*(000) = 6376, *T* = −100°C.

#### 3.2.2. Data collection and reduction

A colourless tablet ca. 0.70 × 0.35 × 0.15 mm<sup>3</sup> was mounted in inert oil on a glass fibre. A total of 12982 intensities were measured on a Siemens R3 diffractometer to 2θ<sub>max</sub> 50°. A 'Δ*F*'-type absorption correction was applied using the program SHELXA (G.M. Sheldrick, unpublished), with transmission factors 0.197–0.723. Merging equivalents gave 12168 unique reflections (*R*<sub>int</sub> 0.046), of which 12144 were used for all calculations.

#### 3.2.3. Structure solution and refinement

The structure was solved by the heavy-atom method and subjected to full-matrix least-squares refinement on *F*<sup>2</sup> (program SHELXL-93 [15]). The heaviest atoms Au, P, Si and Cl were refined anisotropically, others isotropically. Phenyl groups were refined with idealised geometry. Other hydrogen atoms were included using a riding model. The SiMe<sub>3</sub> group of one molecule was refined over two alternative disorder sites. The weighting scheme was *w*<sup>−1</sup> = σ<sup>2</sup>(*F*<sup>2</sup>) + (*aP*)<sup>2</sup>, where *P* = (*F*<sub>o</sub><sup>2</sup> + 2*F*<sub>c</sub><sup>2</sup>)/3 and *a* is a constant adjusted by the program. The final *wR*(*F*<sup>2</sup>) was 0.123 for 329 parameters and 113 restraints, conventional *R*(*F*) 0.057. *S* = 0.84, maximum Δρ 1.66 e Å<sup>−3</sup>. Final atomic coordi-

Table 2

Atomic coordinates [ $\times 10^4$ ] and equivalent isotropic displacement parameters [ $\text{\AA}^2 \times 10^3$ ]

Atom	x	y	z	$U_{\text{eq}}$
Au(1)	5940.4(1)	3830.4(3)	4208.6(2)	44.1(1)
P(1)	6558.7(7)	5908(2)	4093.0(10)	35.1(7)
P(2)	6463.4(7)	4223(2)	4779.4(11)	35.0(7)
C(1)	6924(3)	6579(7)	4006(4)	44(3)
C(2)	6751(2)	4926(6)	4462(4)	35(2)
C(3)	5475(3)	3369(9)	3704(5)	61(3)
Si(1)	5070.2(9)	3827(3)	3861(2)	69.9(11)
C(4)	5034(5)	3534(13)	4566(6)	133(7)
C(5)	4684(3)	3366(10)	3394(5)	88(5)
C(6)	5049(5)	5025(10)	3798(7)	128(7)
C(11)	6295(2)	6517(4)	4474(3)	35(2)
C(12)	6462.2(12)	7081(5)	4899(3)	45(3)
C(13)	6264(2)	7578(4)	5192(3)	56(3)
C(14)	5898(2)	7512(5)	5060(3)	54(3)
C(15)	5730.1(12)	6948(5)	4635(3)	54(3)
C(16)	5929(2)	6450(4)	4342(2)	49(3)
C(21)	6290(2)	5637(4)	3425(2)	39(3)
C(22)	6087(2)	6313(4)	3128(3)	53(3)
C(23)	5869(2)	6136(4)	2609(3)	62(3)
C(24)	5854(2)	5284(5)	2388(2)	71(4)
C(25)	6057(2)	4608(4)	2685(3)	56(3)
C(26)	6275(2)	4785(4)	3203(3)	40(3)
C(31)	6433(2)	4835(4)	5402(2)	40(3)
C(32)	6101.5(14)	5034(5)	5491(3)	55(3)
C(33)	6070.9(15)	5534(5)	5952(3)	63(3)
C(34)	6372(2)	5835(5)	6324(2)	65(4)
C(35)	6704(2)	5636(5)	6235(3)	52(3)
C(36)	6734.8(14)	5136(5)	5775(3)	43(3)
C(41)	6750(2)	3274(4)	5013(3)	40(3)
C(42)	6818(2)	2990(5)	5564(3)	54(3)
C(43)	7020(2)	2233(5)	5725(2)	69(4)
C(44)	7154(2)	1759(4)	5336(3)	68(4)
C(45)	7087(2)	2042(5)	4785(3)	66(4)
C(46)	6885(2)	2800(5)	4624(2)	51(3)
Au(1')	6200.2(1)	339.8(3)	6469.4(2)	63.3(2)
P(1')	7002.5(7)	-716(2)	7589.0(10)	30.3(6)
P(2')	6512.8(7)	914(2)	7290.9(11)	36.3(7)
C(1')	7388(2)	-1002(7)	8114(4)	35(2)
C(2')	6954(2)	452(6)	7606(4)	34(2)
C(3')	5886(4)	-76(10)	5718(5)	92(5)
Si(1')	5890(3)	693(7)	5163(4)	98(3)
C(4')	5523(8)	557(22)	4527(12)	112(12)
C(5')	6292(7)	385(20)	4973(12)	103(10)
C(6')	5918(9)	1940(20)	5365(15)	130(15)
C(11')	6628(2)	-1237(5)	7744(3)	38(2)
C(12')	6595(2)	-1261(5)	8293(2)	61(3)
C(13')	6298(2)	-1642(6)	8423(3)	89(5)
C(14')	6035(2)	-2000(6)	8003(4)	86(4)
C(15')	6068(2)	-1977(5)	7453(3)	73(4)
C(16')	6365(2)	-1596(5)	7324(2)	50(3)
C(21')	7054(2)	-1077(4)	6922(2)	35(2)
C(22')	7122(2)	-1971(4)	6850(2)	41(3)
C(23')	7180(2)	-2268(3)	6344(3)	51(3)
C(24')	7170(2)	-1671(5)	5911(2)	47(3)
C(25')	7103(2)	-777(4)	5983(2)	48(3)
C(26')	7045(2)	-480(3)	6488(3)	42(3)
C(31')	6276(2)	871(5)	7845(3)	44(3)
C(32')	6435(2)	1104(5)	8390(3)	53(3)
C(33')	6238(2)	1106(6)	8796(2)	77(4)
C(34')	5883(2)	875(7)	8657(4)	108(6)
C(35')	5724(2)	642(7)	8112(4)	122(6)

Table 2 (continued)

Atom	x	y	z	$U_{\text{eq}}$
C(36')	5921(2)	640(6)	7706(3)	88(5)
C(41')	6615(2)	2090(3)	7234(3)	35(2)
C(42')	6386.0(15)	2574(4)	6825(2)	50(3)
C(43')	6426(2)	3489(4)	6792(2)	53(3)
C(44')	6694(2)	3920(3)	7169(3)	50(3)
C(45')	6923.1(15)	3436(4)	7578(2)	43(3)
C(46')	6883.2(15)	2521(4)	7611(2)	41(3)
Si(1'')	5668(3)	844(7)	5296(4)	94(3)
C(4'')	5960(8)	1679(23)	5091(15)	130(15)
C(5'')	5433(8)	1317(22)	5776(12)	135(14)
C(6'')	5317(9)	411(24)	4658(13)	140(16)
Cl(1)	7171.8(7)	6116(2)	8015.7(11)	43.0(6)
O(1)	7228(3)	5493(8)	7610(4)	115(4)
O(2)	7157(2)	5593(8)	8474(4)	100(4)
O(3)	7477(3)	6642(7)	8084(4)	98(3)
O(4)	6859(3)	6534(8)	7779(6)	142(5)
Cl(2)	7647.6(8)	4102(2)	569.5(10)	42.4(7)
O(5)	7957(2)	3755(7)	455(3)	81(3)
O(6)	7352(2)	3606(5)	284(3)	64(2)
O(7)	7667(2)	4054(6)	1156(3)	66(3)
O(8)	7597(3)	4986(6)	396(5)	103(4)
C(99)	5000	8461(24)	7500	165(13)
Cl(3)	5117(2)	9145(5)	6973(3)	211(4)

$U_{\text{eq}}$  is defined as one third of the trace of the orthogonalized  $U_{ij}$  tensor.

nates are given in Table 2, with derived bond lengths and angles in Table 3. See also Section 4.

#### 4. Supplementary material

Full details of the structure determination have been deposited at the Fachinformationszentrum Karlsruhe.

Table 3

Selected bond lengths [ $\text{\AA}$ ] and angles [ $^\circ$ ]

Au(1)–C(3)	2.070(11)	Au(1)–P(2)	2.273(3)
P(1)–C(21)	1.786(5)	P(1)–C(11)	1.793(6)
P(1)–C(1)	1.794(10)	P(1)–C(2)	1.805(9)
P(2)–C(31)	1.817(6)	P(2)–C(41)	1.823(6)
P(2)–C(2)	1.841(10)	Au(1')–C(3')	2.071(12)
Au(1')–P(2')	2.282(3)	P(1')–C(11')	1.769(6)
P(1')–C(2')	1.768(10)	P(1')–C(21')	1.787(5)
P(1')–C(1')	1.796(8)	P(2')–C(31')	1.814(7)
P(2')–C(41')	1.826(5)	P(2')–C(2')	1.850(9)
C(3)–Au(1)–P(2)	175.4(4)	C(21)–P(1)–C(11)	108.4(3)
C(21)–P(1)–C(1)	109.4(4)	C(11)–P(1)–C(2)	109.7(4)
C(1)–P(1)–C(2)	111.5(4)	C(11)–P(1)–C(2)	112.1(4)
C(1)–P(1)–C(2)	105.8(5)	C(31)–P(2)–C(41)	106.4(3)
C(31)–P(2)–C(2)	103.3(4)	C(41)–P(2)–C(2)	101.4(4)
C(31)–P(2)–Au(1)	115.8(2)	C(41)–P(2)–Au(1)	112.9(3)
C(2)–P(2)–Au(1)	115.6(3)	P(1)–C(2)–P(2)	117.5(5)
C(3')–Au(1')–P(2')	174.6(4)	C(11')–P(1')–C(2')	109.7(4)
C(11')–P(1')–C(21')	109.5(3)	C(2')–P(1')–C(21')	111.1(4)
C(11')–P(1')–C(1')	109.3(4)	C(2')–P(1')–C(1')	107.3(4)
C(21')–P(1')–C(1')	109.9(4)	C(31')–P(2')–C(41')	104.3(4)
C(31')–P(2')–C(2')	104.5(4)	C(41')–P(2')–C(2')	101.5(4)
C(31')–P(2')–Au(1')	113.4(3)	C(41')–P(2')–Au(1')	112.3(2)
C(2')–P(2')–Au(1')	119.1(3)	P(1')–C(2')–P(2')	117.2(5)

Gesellschaft für Wissenschaftlich-technische information mbH, 76344 Eggenstein-Leopoldshafen, Germany. Any request for this material should quote a full literature citation and the reference number CSD-405719.

### Acknowledgements

We thank the Fonds der Chemischen Industrie and the Dirección General de Investigación Científica y Técnica (No. PB95-0140-A) for financial support and the Universidad Pública de Navarra for a grant (to M.C.).

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