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Synthesis and reactivity of neutral complexes of the types $[AuX_3(ylide)]$ and *trans*-$[Au(C_6F_5)X_2(ylide)]$ (X = halide or pseudohalide). X-ray structure of $[Au(SCN)_3(CH_2PPh_3)]$

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Abstract

Halogens (Cl_2 or Br_2) add oxidatively to ylide-gold(I) complexes $[AuX(CHRPR'_3)]$ or $[Au(C_6F_5)(CH_2PR_3)]$ to give stable gold(III) derivatives of the types $[AuX_3(CHRPR'_3)]$ (X = Cl, Br) or *trans*-$[Au(C_6F_5)X_2(CH_2PR_3)]$, which can be converted into other halo or pseudohalo complexes: $[AuI_3(CH_2PPh_3)]$, $[Au(SCN)_3(CH_2PR_3)]$ or *trans*-$[Au(C_6F_5)(SCN)_2(CH_2PPh_2Me)]$. The structures have been assigned on the basis of spectral (IR and NMR) and X-ray data.

Introduction

Many neutral or cationic ylide-gold(III) complexes are known [1], although some types are unrepresented or poorly represented. For instance, no neutral derivatives of the type $[AuRX_2(ylide)]$ (R = organic ligand, X = halide) have been reported, and complexes of the types $[AuX_2X'(ylide)]$ or $[AuX_3(ylide)]$ have been described only recently (viz. $[AuX_2(CN)(CH_2PPh_3)]$ (X = Cl, Br, I) [2] and $[AuCl_3\{CH(CO_2R)PPh_3\}]$ (R = Me, Et) [3]).

Here, we describe the preparation of $[AuX_3(ylide)]$ or *trans*-$[AuX_2(C_6F_5)(ylide)]$ by the oxidative addition of halogens to previously known ylide-gold(I) complexes $[AuX(ylide)]$ or $[Au(C_6F_5)(ylide)]$. They are stable enough to undergo substitution reactions by other halide or pseudohalide ions. No reduction to gold(I) has been observed.

Results and discussion

Stoichiometric amounts of Cl_2 or Br_2 added to dichloromethane solutions of [AuX(ylide)] [5] cause oxidative addition to give the corresponding trihalo(ylide) gold(III) complexes, according to eq. 1. An excess of halogen is not detrimental.

$$[\mathrm{AuX(CHRPR'_3)}] + \mathrm{X_2} \rightarrow \underset{\mathbf{(1\text{–}10)}}{[\mathrm{AuX_3(CHRPR'_3)}]} \qquad (1)$$

$X = Cl$, $R = H$, $PR'_3 = PPh_3$ (**1**); $X = Br$ (**6**)
$X = Cl$, $R = H$, $PR'_3 = PPh_2Me$ (**2**); $X = Br$ (**7**)
$X = Cl$, $R = H$, $PR'_3 = PPh_2Me_2$ (**3**); $X = Br$ (**8**)
$X = Cl$, $R = Me$, $PR'_3 = PPh_3$ (**4**); $X = Br$ (**9**)
$X = Cl$, $R = Ph$, $PR'_3 = PPh_3$ (**5**); $X = Br$ (**10**)

At room temperature, complexes **1–10** are air-, light- and moisture-stable solids. The chlorides are pale yellow, the bromides orange-red. They all are monomeric (in chloroform solution) and non-conducting (in acetone solution). The IR spectra of the chloro-derivatives **1–5** show two strong absorptions at 355 and 295 cm^{-1} (see Table 2), assignable to ν_{asymm}(ClAuCl) and ν(Au–Cl) *trans* to ylide, respectively. A weak band at 340 cm^{-1} can tentatively be assigned to ν_{symm}(ClAuCl) [3,6,7]. The bromo derivatives **6–10** show only one band at 250 (m,w) cm^{-1} assignable to ν_{asymm}(Br–Au–Br). The two additional expected bands may either be too weak or lie below 200 cm^{-1} (the lower limit of our spectrometer). In the ^1H NMR spectra of complexes **1–10** the resonances from CH_2 or CH are shifted towards lower fields (1.5–2.2 ppm for the chloro, 1.65–2.5 ppm for the bromo derivatives) with respect to the starting gold(I) complexes. A similar downfield shift, albeit less marked (0.3 ppm) is observed for the resonances of the P–CH_3 groups (complexes **2**, **3**, **7** and **8**). The ^1H NMR spectra of complex **9** shows another signal at 5.30 ppm (s, 1H) arising from dichloromethane of crystallization.

Similarly, oxidative addition of chlorine to [Au(C_6F_5)(ylide)] [4,5] leads to the corresponding *trans*-dichloro(ylide)gold(III) complexes (eq. 2)

$$[\mathrm{Au(C_6F_5)(CH_2PR_3)}] + \mathrm{Cl_2} \rightarrow \underset{\mathbf{(11\text{–}13)}}{trans\text{-}[\mathrm{Au(C_6F_5)Cl_2(CH_2PR_3)}]} \qquad (2)$$

$PR_3 = PPh_3$ (**11**), $PR_3 = PPh_2Me$ (**12**), $PR_3 = PPhMe_2$ (**13**)

At room temperature complexes **11–13** are air-, light- and moisture-stable white solids. They are practically non-conducting (in acetone) and monomeric (in chloroform). The IR spectra show only one absorption in the 360 cm^{-1} region, as expected for *trans*-isomers [3,6]. A downfield shift of the ^1H NMR resonances from the groups CH_2 (0.8 ppm) or P–CH_3 (0.25 ppm) is observed (see above).

Complexes **1–13** undergo exchange reactions with the potassium salts of other halides or pseudohalides (eq. 3 and 4)

$$[\mathrm{AuCl_3(CH_2PR_3)}] + 3\mathrm{KX} \rightarrow \underset{\mathbf{(6,14\text{–}16)}}{[\mathrm{AuX_3(CH_2PPh_3)}]} + 3\ \mathrm{KCl} \qquad (3)$$

$PR_3 = PPh_3$, $X = Br$ (**6**),
$PR_3 = PPhMe_2$, $X = SCN$ (**16**)
$PR_3 = PPh_3$, $X = I$ (**14**)
$PR_3 = PPh_3$, $X = SCN$ (**15**)

$$[Au(C_6F_5)Cl_2(CH_2PPh_2Me)] + KSCN \rightarrow [Au(C_6F_5)(SCN)_2(CH_2PPh_2Me)] + 2\,KCl \quad (4)$$

(17)

Complex **14** is an air- and moisture-stable red-brown solid. It is monomeric (in $CHCl_3$) and moderately conducting (in acetone), although the measured Λ_M values are lower than expected for 1/1 electrolytes [8]. In the ^1H NMR spectrum of **14** the CH_2 resonance is shifted downfield, more so than in the trichloro or tribromo derivatives. In the IR spectrum the ν(Au–I) vibration was not observed because it fell outside the lower limit of our IR spectrophotometer (200 cm^{-1}).

Complexes **15** and **16** are stable yellow solids, and their acetone solutions are non-conducting. Complex **15** is monomeric in chloroform solution; the limited solubility of **16** prevents measurement. The IR spectra of both complexes show one strong absorption at 2106 and 2100 cm^{-1}, respectively, assignable to ν(CN) of the S-bonded SCN groups [9]. In their ^1H NMR spectra the resonances from the methylene and methyl groups are shifted to higher field with respect to the starting chloro complexes.

Finally, complex **17** is a stable pale yellow solid. It is monomeric and non-conducting. The IR spectrum shows one strong band at 2110 cm^{-1}, assignable to ν(CN) of the S-bonded SCN groups.

Crystals of complex **15** suitable for X-ray crystallography were obtained by slow diffusion of n-hexane into a layer of its dichloromethane solution.

The structure determination confirms the expected nature of compound **15** (Fig. 1); the coordination at gold is square planar (but with some small deviations of ca. 5° from ideal geometry) and the thiocyanate ligands are coordinated through sulphur. Structure determinations have been reported for only a few gold thiocyanate

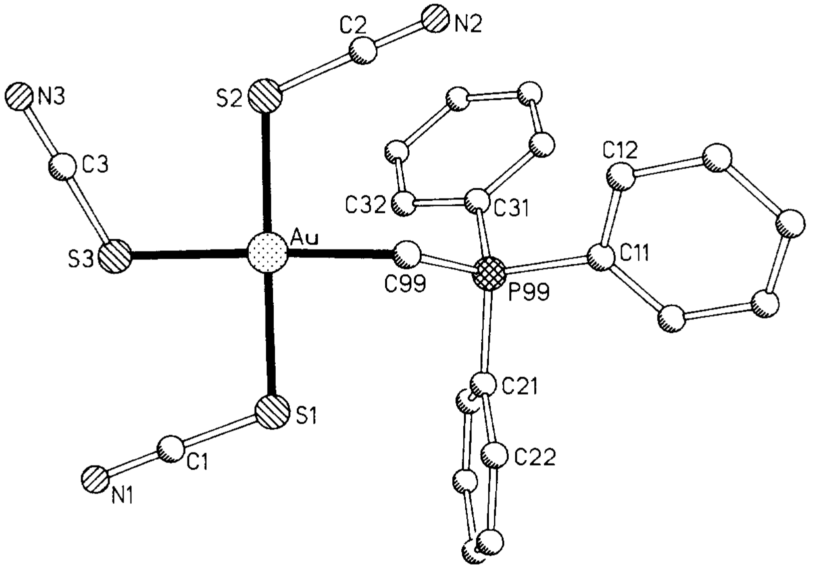

Fig. 1. The molecule of **15** in the crystal, showing the atom numbering scheme. Radii arbitrary, H atoms omitted.

complexes, and all of these were gold(I) complexes [11] with long Au–S bonds. The Au–S bond lengths of **15** are 2.398 (*trans* to C), 2.332 and 2.333 Å, indicating the greater *trans* influence of the ylide ligand. The thiocyanate ligands show some deviation from linearity (S–C–N angles 175, 176, 165°), but the standard deviations are high (2–3°) and the thermal motion of the N atoms is also high, so these deviations may not be very significant. The Au–C(ylide) bond length of 2.111 Å is similar to the values of 2.087(12) and 2.115(15) observed in a gold(II) methylenethiophosphinate complex [12]; there are few reports of Au–CH_2(ylide) bond lengths in the literature.

Experimental

The instrumentation employed and general techniques were as described earlier [4,5]. The yields, melting points, C, H and Au analyses, conductivities and molecular weights of the novel complexes are listed in Table 1. Table 2 gives the spectroscopic (^1H NMR and IR) data. All reactions were carried out at room temperature.

The starting compounds: [AuX(ylide)] (X = Cl, Br; ylide = CH_2PPh_3, CH_2PPh_2Me, CH_2PPhMe_2, $CH(Me)PPh_3$ and $CH(Ph)PPh_3$ and [Au(C_6F_5)(ylide)] (ylide = CH_2PPh_3, CH_2PPh_2Me and CH_2PPhMe_2) were prepared by the published methods [4,5].

[$AuX_3(CHRPR'_3)$] (X = Cl, $CHRPR'_3$ = CH_2PPh_3 (**1**), CH_2PPh_2Me (**2**), CH_2PPhMe_2 (**3**), $CH(Me)PPh_3$ (**4**), $CH(Ph)PPh_3$ (**5**); X = Br, $CHRPR'_3$ = CH_2PPh_3 (**6**), CH_2PPh_2Me (**7**), CH_2PPhMe_2 (**8**), $CH(Me)PPh_3$ (**9**), $CH(Ph)PPh_3$ (**10**))

To a dichloromethane solution (30 ml) containing 0.3 mmol of each of the following precursors [AuCl(CH_2PPh_3)] (0.153 g), [AuCl(CH_2PPh_2Me)] (0.134 g), [AuCl(CH_2PPhMe_2)] (0.115 g), [AuCl{$CH(Me)PPh_3$}] (0.157 g), [AuCl{$CH(Ph)PPh_3$}] (0.175 g), [AuBr(CH_2PPh_3)] (0.166 g), [AuBr(CH_2PPh_2Me)] (0.147 g), [AuBr(CH_2PPhMe_2)] (0.129 g), [AuBr{$CH(Me)PPh_3$}] (0.170 g), or [AuBr{$CH(Ph)PPh_3$}] (0.189 g) were added 0.3 mmol of Cl_2 or Br_2 (0.84 ml of a CCl_4 solution 0.36 *M* Cl_2 or 0.67 ml of a CCl_4 solution 0.45 *M* Br_2). The colour of the solution changed immediately to pale yellow (chloride derivatives) or to orange-red (bromide derivatives). The mixture was stirred for 20 min and then vacuum concentrated to 5 ml. Addition of diethyl ether (20 ml) precipitated the complexes **1**–**10**.

[$AuBr_3(CH_2PPh_3)$] (**6**) was also prepared by reaction of an acetone solution (30 ml) of [$AuCl_3(CH_2PPh_3)$] (0.116 g, 0.2 mmol) with KBr (0.095 g, 0.8 mmol). The solution was stirred for 1 h and then evaporated to dryness, and the resulting solid extracted with 2 × 10 ml of dichloromethane. Evaporation of the solution to 5 ml and addition of diethyl ether (20 ml) gave **6** as a yellow solid (yield 72%).

[$Au(C_6F_5)Cl_2(CH_2PR_3)$] (PR_3 = PPh_3 (**11**), PPh_2Me (**12**), $PPhMe_2$ (**13**))

The white complexes **11**–**13** were obtained as described for complexes **1**–**5**, but starting from 0.3 mmol of [$Au(C_6F_5)(CH_2PPh_3)$] (0.192 g), [$Au(C_6F_5)(CH_2PPh_2Me)$] (0.155 g) or [$Au(C_6F_5)(CH_2PPhMe_2)$] (0.173 g) and 0.3 mmol of Cl_2 (0.84 ml of a CCl_4 solution 0.36 *M* Cl_2).

[$AuI_3(CH_2PPh_3)$] (**14**)

To an acetone solution (30 ml) of [$AuCl_3(CH_2PPh_3)$] (**1**) (0.116 g, 0.2 mmol) was added KI (0.133 g, 0.8 mmol). The mixture was stirred for 1 h and then evaporated

Table 1

Analytical data for complexes **1–17**

Complex	Yield (%)	Anal. (Found (calcd.)(%))				Λ_M [a]	Molecular weight [b]	M.p. [d] (°C)
		C	H	N	Au			
$[AuCl_3(CH_2PPh_3)]$	88	39.7	2.95		33.5	2	584	208
1		(39.35)	(2.95)		(34.0)		(580)	
$[AuCl_3(CH_2PPh_2Me)]$	88	32.8	2.95		38.55	2	526	192
2		(32.5)	(2.85)		(38.05)		(518)	
$[AuCl_3(CH_2PPhMe_2)]$	85	23.85	2.85		43.35	3	[c]	156(d)
3		(23.75)	(2.85)		(43.25)			
$[AuCl_3(CH_2(Me)PPh_3\}]$	89	40.4	3.2		33.6	4	594	178(d)
4		(40.45)	(3.2)		(33.15)		(594)	
$[AuCl_3\{CH(Ph)PPh_3\}]$	84	46.25	3.5		29.7	16	650	170
5		(45.8)	(3.15)		(30.05)		(656)	
$[AuBr_3(CH_2PPh_3)]$	85	31.6	2.5		27.35	4	700	196
6		(32.0)	(2.4)		(27.6)		(713)	
$[AuBr_3(CH_2PPh_2Me)]$	89	26.1	2.3		29.9	8	661	152
7		(25.85)	(2.3)		(30.25)		(651)	
$[AuBr_3(CH_2PPhMe_2)]$	91	18.75	2.3		33.35	3	[c]	145
8		(18.35)	(2.2)		(33.45)			
$[AuBr_3\{CH(Me)PPh_3\}]$								
$\cdot\frac{1}{2}CH_2Cl_2$	92	32.05	2.6		26.05	11	729	130
9		(32.0)	(2.6)		(25.6)		(727)	
$[AuBr_3\{CH(Ph)PPh_3\}]$	90	37.95	2.6		24.75	7	751	173(d)
10		(37.6)	(2.65)		(24.95)		(789)	
$[AuRCl_2(CH_2PPh_3)]$ [e]	66	42.5	2.7		28.15	11	696	135(d)
11		(42.2)	(2.4)		(27.7)		(711)	
$[AuRCl_2(CH_2PPh_2Me)]$ [e]	75	36.95	2.35		30.75	3	676	182(d)
12		(37.0)	(2.35)		(30.35)		(649)	
$[AuRCl_2(CH_2PPhMe_2)]$ [e]	76	30.25	2.1		34.05	7	625	137(d)
13		(30.7)	(2.25)		(33.55)		(587)	
$[AuI_3(CH_2PPh_3)]$	63	26.8	2.2		22.65	50	915	133(d)
14		(26.7)	(2.05)		(23.05)		(854)	
$[Au(SCN)_3(CH_2PPh_3)]$	71	40.45	2.8	6.25	31.0	3	647	140(d)
15		(40.8)	(2.65)	(6.5)	(30.4)		(643)	
$[Au(SCN)_3(CH_2PPhMe_2)]$	75	28.0	2.45	7.5	38.15	9	[c]	133(d)
16		(27.55)	(2.5)	(8.0)	(37.65)			
$[AuR(SCN)_2(CH_2PPh_2Me)]$	61	38.1	2.0	4.4	28.9	5	719	160(d)
17		(38.05)	(2.2)	(4.05)	(28.35)		(694)	

[a] In acetone, ohm^{-1} cm^2 mol^{-1}. [b] In chloroform. [c] Not soluble enough. [d] Or decomposition. [e] R = C_6F_5.

to dryness. The resulting solid was extracted with dichloromethane (2 × 15 ml) and the extract evaporated to 5 ml. Addition of diethyl ether (15 ml) precipitated **14** as a brown solid.

$[Au(SCN)_3(CH_2PR_3)]$ ($PR_3 = PPh_3$ (15), $PPhMe_2$ (16))

The yellow complexes of **15** and **16** were obtained from $[AuCl_3(CH_2PPh_3)]$ (**1**) (0.116 g, 0.2 mmol), or $[AuCl_3(CH_2PPhMe_2)]$ (**3**) (0.091 g, 0.2 mmol) and 0.8 mmol of KSCN (0.078 g) by the procedure described for **14**.

The same procedure starting from $[AuBr_3(CH_2PPh_3)]$ (**6**) gave complex $[Au(SCN)_3(CH_2PPh_3)]$ (**15**) (64% yield).

Table 2

^{1}H NMR data [a] and some significant IR bands

Complex	CH_2 or CH (ppm)	^2J(P–H) (Hz)	P–*Me* (ppm)	^2J(P–H) (Hz)	CH–*Me* (ppm)	^3J(P–H) (Hz)	ν(Au–C)	ν(Au–X)		
$[AuCl_3(CH_2PPh_3)]$	3.65(d)	8.6	–	–	–	–	590	302	335	352
$[AuCl_3(CH_2PPh_2Me)]$	3.30(d)	9.4	2.62(d)	13.5	–	–	590	299	334	359
$[AuCl_3(CH_2PPhMe_2)]$	3.01(d)	10.1	2.34(d)	13.6	–	–	575	289	336	358
$[AuCl_3\{CH(Me)PPh_3\}]$	4.90("q")	7.5	–	–	1.72(dd)	19.9	568	295	337	355
$[AuCl_3\{CH(Ph)PPh_3\}]$	6.22(d)	9.1	–	–	–	–	591	291	339	356
$[AuBr_3(CH_2PPh_3)]$	3.94(d)	8.6	–	–	–	–	583			247
$[AuBr_3(CH_2PPh_2Me)]$	3.62(d)	9.8	2.65(d)	13.5	–	–	579			252
$[AuBr_3(CH_2PPhMe_2)]$	3.30(d)	10.2	2.36(d)	13.5	–	–	573			253
$[AuBr_3\{CH(Me)PPh_3\}]$	5.27("q")	7.4	–	–	1.82(dd)	19.8	555		244	
$[AuBr_3\{CH(Ph)PPh_3\}]$	6.67(d)	10.4	–	–	–	–	587		245	
$[Au(C_6F_5)Cl_2(CH_2PPh_3)]$	2.72(d)	11.8	–	–	–	–	575		361	
$[Au(C_6F_5)Cl_2(CH_2PPh_2Me)]$	2.35(d)	12.4	2.52(d)	13.4	–	–	575		355	
$[Au(C_6F_5)Cl_2(CH_2PPhMe_2)]$	2.10(d)	12.7	2.22(d)	13.4	–	–	560		360	
$[AuI_3(CH_2PPh_3)]$	4.42(d)	9.4	–	–	–	–	565			
$[Au(SCN)_3(CH_2PPh_3)]$	3.18(d)	10.3	–	–	–	–	570			
$[Au(SCN)_3(CH_2PPhMe_2)]$	2.50(d)	13.7	2.41(d)	13.2	–	–	557			
$[Au(C_6F_5)(SCN)_2(CH_2PPh_2Me)]$	2.97(d)	11.4	2.59(d)	13.7	–	–	565			

[a] In $CDCl_3$ using TMS as internal reference. d: doublet, dd: doublet of doublets; "q": apparent quintet.

$[Au(C_6F_5)(SCN)_2(CH_2PPh_2Me)]$ (17)

The procedure used for **14** and **15**, but starting from *trans*-$[Au(C_6F_5)Cl_2(CH_2PPh_2Me)]$ (**12**) (0.130 g, 0.2 mmol) and KSCN (0.6 mmol, 0.058 g) gave **17** as a yellow solid.

X-Ray structure determination of 15

Crystal data. $C_{22}H_{17}AuN_3PS_3$, $M = 647.5$, monoclinic, $P2_1/n$, a 8.197(3), b 17.680(6), c 16.235(5) Å, β 91.42(3)°, V 2352 Å^3, $Z = 4$, D_x 1.83 g cm^{-3}, μ(Mo-K_α) 6.6 mm^{-1}, F(000) 1248.

Data collection and processing. A prism 0.35 × 0.2 × 0.2 mm was used to record 6657 profile-fitted intensities [10] on a Stoe-Siemens four-circle diffractometer (Mo-K_α radiation, $2\theta_{max}$ 50°). Of 4128 unique reflections, 2042 with $F > 4\sigma(F)$ were used for all calculations (program system SHELXTL). Absorption corrections based on ψ-scans were applied (transmission factors 0.75–0.82). Cell constants were refined from 2θ values of 41 reflections in the range 20–23°.

Table 3

Atomic coordinates (×10^4) and isotropic thermal parameters (Å^2 ×10^3) for compound **15**

	x	y	z	U
Au	3507(1)	4677(1)	6004(1)	51(1) [a]
P(99)	3726(7)	6058(3)	7398(4)	62(2) [a]
C(99)	4810(20)	5524(10)	6658(11)	45(6)
S(1)	2798(8)	5648(3)	5090(4)	72(3) [a]
S(2)	4195(8)	3717(3)	6934(4)	74(3) [a]
S(3)	2093(8)	3748(4)	5191(5)	93(3) [a]
C(12)	6670(19)	6295(7)	8207(9)	76(7)
C(13)	7826	6726	8643	93(8)
C(14)	7535	7490	8793	92(8)
C(15)	6087	7824	8507	111(10)
C(16)	4930	7394	8071	101(9)
C(11)	5222	6629	7921	67(7)
C(22)	2930(15)	7175(10)	6304(11)	113(10)
C(23)	1899	7675	5878	122(11)
C(24)	233	7683	6035	100(9)
C(25)	−401	7191	6617	109(10)
C(26)	631	6691	7044	103(9)
C(21)	2296	6683	6887	66(7)
C(32)	1510(18)	4998(9)	7874(7)	84(8)
C(33)	738	4541	8446	94(8)
C(34)	1206	4575	9277	105(9)
C(35)	2447	5067	9536	100(9)
C(36)	3219	5524	8964	91(8)
C(31)	2751	5490	8133	64(6)
C(1)	1735(25)	5268(14)	4333(14)	73(7)
N(1)	1037(24)	5003(11)	3729(14)	93(7)
C(2)	5522(28)	4075(14)	7618(15)	78(8)
N(2)	6363(23)	4331(11)	8127(13)	92(7)
C(3)	3208(33)	3006(18)	5230(18)	101(9)
N(3)	3701(32)	2387(17)	5310(18)	157(11)

[a] Equivalent isotropic U defined as one third of the trace of the orthogonalised U_{ij} tensor

Table 4

Bond lengths (Å) and angles (deg.) for compound **15**

Au–C(99)	2.111(17)	Au–S(1)	2.333(6)
Au–S(2)	2.332(6)	Au–S(3)	2.389(7)
P(99)–C(99)	1.782(18)	P(99)–C(11)	1.788(15)
P(99)–C(21)	1.798(17)	P(99)–C(31)	1.766(15)
S(1)–C(1)	1.634(23)	S(2)–C(2)	1.661(25)
S(3)–C(3)	1.599(31)	C(1)–N(1)	1.216(31)
C(2)–N(2)	1.155(31)	C(3)–N(3)	1.174(43)
C(99)–Au–S(2)	94.5(5)	S(1)–Au–S(2)	179.1(2)
C(99)–Au–S(3)	176.7(5)	S(1)–Au–S(3)	92.6(2)
S(2)–Au–S(3)	87.9(2)	Au–S(1)–C(1)	107.1(9)
Au–S(2)–C(2)	107.5(9)	Au–S(3)–C(3)	105.9(11)
S(1)–C(1)–N(1)	175.0(22)	S(2)–C(2)–N(2)	175.7(24)
S(3)–C(3)–N(3)	164.8(27)	C(99)–Au–S(1)	85.0(5)
Au–C(99)–P(99)	117.4(9)	C(99)–P(99)–C(11)	105.8(8)
C(99)–P(99)–C(21)	110.2(8)	C(99)–P(99)–C(31)	113.3(8)

Structure solution and refinement. The structure was solved by the heavy-atom method and refined on F to R 0.080, R_w 0.063. Au, P and S atoms were anisotropic; phenyl groups were refined with C–C 1.395, C–H 0.96 Å, all angles 120°; methylene H were refined using a riding model; the weighting scheme was $w^{-1} = \sigma^2(F) + 0.0003\ F^2$; 110 parameters.

Final atom coordinates are presented in Table 3, with derived bond lengths and angles in Table 4. Further details of the structure determination (structure factors, temperature factors, H atom coordinates) have been deposited at the Fachinformationszentrum Energie Physik Mathematik, 7514 Eggenstein-Leopoldshafen 2, W. Germany. Any request for this material should quote reference number CSD 52563 and the complete literature citation.

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