Organometallics 1987, *6*, 1778–1780

Bimetallic Phosphorus Ylide Gold–Silver Complexes

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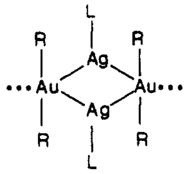
Received February 5, 1987

Cationic complexes $[Au(CH_2PR_3)_2]ClO_4$ ($PR_3 = PPh_3$, PPh_2Me, $PPhMe_2$), which can be prepared from $[Au(SC_4H_8)_2]ClO_4$ and CH_2PR_3, react with $AgClO_4$ or $(O_3ClO)AgPPh_3$ to give neutral $[(R_3PCH_2)_2Au\{\mu\text{-}Ag(OClO_3)_2\}_2Au(CH_2PR_3)_2]$ or cationic $[(Ph_3PCH_2)_2Au(\mu\text{-}AgPPh_3)_2Au(CH_2PPh_3)_2](ClO_4)_4$ complexes containing Au_2Ag_2 rings where the Au–Ag bonds are unsupported by any covalent bridge. The structure of one neutral complex ($PR_3 = PPh_3$) has been solved by X-ray crystallography. It crystallizes in space group $P2_1/n$ with $a = 13.282\ (3)$ Å, $b = 13.458\ (4)$ Å, $c = 22.016\ (5)$ Å, $\beta = 93.58\ (3)°$, and $Z = 2$.

Introduction

Recently we have reported the synthesis of a family of unprecedented clusters containing the unit



which forms polymeric neutral complexes through short Au⋯Au contacts. Some 35 complexes of this type (R = C_6F_5, $C_6F_3H_2$, C_6Cl_5: L = group VB (15) or VIB (16)[8] donors, olefins, acethylenes, arenes) have been prepared, and the structures of two examples (R = C_6F_5, L = SC_4H_8, C_6H_6) have been solved.[1,2] To what degree the polyhalophenyl groups R, the short Au⋯Au contacts, or both are responsible for the stability of this novel type of cluster is a problem that can be addressed by attempting the synthesis of other complexes lacking one or both characteristics but containing Au_2Ag_2 rings with direct Au–Ag bonds unsupported by any bridging ligand.

Here we report the synthesis of a new type of neutral nonpolymeric $[(\text{ylide})_2Au\{\mu\text{-}Ag(OClO_3)_2\}_2Au(\text{ylide})_2]$ complexes, containing the Au_2Ag_2 moiety and lacking any Au⋯Au interaction.

Results and Discussion

Addition of $[Au(SC_4H_8)_2]ClO_4$ to tetrahydrofuran solutions of CH_2PR_3 causes the precipitation of cationic bis(ylide)gold complexes (eq 1)

$$[Au(SC_4H_8)_2]ClO_4 + 2CH_2PR_3 \rightarrow [Au(CH_2PR_3)_2]ClO_4 + 2SC_4H_8 \quad (1)$$

$$PR_3 = PPh_3\ (I),\ PPh_2Me\ (II),\ PPhMe_2\ (III)$$

Complexes I–III are air- and moisture-stable white solids and 1:1 electrolytes in acetone solution (see Table I). Their IR spectra (Nujol mulls) show two bands at 1100 (s, br) and 625 (m) cm^{-1} due to the ClO_4^- anion,[3] a band due to ν(Au–C) appears[4] at 562–539 (m) cm^{-1}. These complexes (I–III) react (1:1) with $AgClO_4$ in CH_2Cl_2/OEt_2 solution to precipitate white solids (eq 2).

$$2[Au(CH_2PR_3)_2]ClO_4 + 2AgClO_4 \rightarrow [(R_3PCH_2)_2Au\{\mu\text{-}Ag(OClO_3)_2\}_2Au(CH_2PR_3)_2] \quad (2)$$

$$PR_3 = PPh_3\ (IV),\ PPh_2Me\ (V),\ PPhMe_2\ (VI)$$

Complexes IV–VI are air- and moisture-stable white solids, but their dichloromethane or acetone solutions decompose to re-form the starting products. Despite the difficulties arising from this lack of stability, single crystals of complex IV could be obtained by slow diffusion at −20 °C of an $AgClO_4$ solution (0.26 mmol in 12 mL of diethyl ether) into a dichloromethane solution of complex I (0.18 mmol in 6 mL of dichloromethane). Its structure has been established by X-ray crystallography (Figure 1).

The molecule is centrosymmetric; the Au–Ag distances (2.783, 2.760 (2) Å) in the central Au_2Ag_2 ring are within the previously observed range (2.70–2.79 Å).[1] The silver atoms are bonded to two gold atoms and two oxygen atoms (of two perchlorato ligands) in a distorted tetrahedral environment. The C(1)–Au–C(2) angle (173.9 (5)°) is almost linear. Au⋯Au contacts are not observed probably because of the barrier formed by the six phenyl groups of the two $CH_2P(C_6H_5)_3$ ligands linked to each gold center.

The ir spectra of complexes IV–VI show bands at 1130 (s), 1110 (s), 920 (m), and 620 (m, br) cm^{-1} due to the coordinated $-OClO_3$ groups.[3] The ν(Au–C) band appears at altmost the same position (564–544 cm^{-1}) as in the starting bis(ylide) complexes.

If $(O_3ClO)AgPPh_3$ is substituted for $AgClO_4$ in eq 2, the cationic $[(Ph_3PCH_2)_2Au(\mu\text{-}AgPPh_3)_2Au(CH_2PPh_3)_2](ClO_4)_4$ (VII) is obtained. Conductivity measurements (in acetone or nitromethane) cannot be carried out due to decomposition (see above), but the IR spectrum clearly shows the presence of the ClO_4^- anion [(one band at 1080 (s, br) cm^{-1} and another band at 620 (m) cm^{-1}].

Experimental Section

Instrumentation and general experimental techniques were as described earlier.[1] The yields, melting points, C, H, and Au (or Au + Ag) analyses, conductivities, and ν(Au–C) of the novel complexes are listed in Table I.

Preparation of the Complexes. $[Au(CH_2PR_3)_2]ClO_4$ [$PR_3 = PPh_3$ (I), PPh_2Me (II), or $PPhMe_2$ (III)]. To a tetrahydrofuran solution (40 mL) of CH_2PR_3 (1 mmol) (prepared by reacting equimolecular amounts of $[CH_3PR_3]ClO_4$ and BuLi) was added $[Au(SC_4H_8)_2]ClO_4$[5] (0.189 g, 0.4 mmol), and the mixture was stirred at room temperature for 1 h. Complexes I–III pre-

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Table I. Analytical Data for Complexes

complex	yield, %	anal. found (calcd) C	H	Au (or Au + Ag)	Λ_M[a]	mp, °C	ν(Au–C), cm^{-1}
$[Au(CH_2PPh_3)_2]ClO_4$ (I)	78	53.8 (53.8)	4.0 (4.05)	23.35 (23.2)	130	208 dec	562
$[Au(CH_2PPh_2Me)_2]ClO_4$ (II)	60	46.6 (46.4)	4.1 (4.2)	27.9 (27.2)	122	100	550
$[Au(CH_2PPhMe_2)_2]ClO_4$ (III)	80	35.9 (36.0)	2.5 (2.2)	32.4 (32.8)	155	104	539
$[Au(CH_2PPh_3)_2Ag(OClO_3)_2]_2$ (IV)	88	43.3 (43.2)	3.1 (3.2)	28.6 (28.9)		195 dec	564
$[Au(CH_2PPh_2Me)_2Ag(OClO_3)_2]_2$ (V)	87	36.0 (36.1)	3.1 (3.2)	32.5 (32.7)		186 dec	562
$[Au(CH_2PPhMe_2)_2Ag(OClO_3)_2]_2$ (VI)	77	26.6 (26.75)	3.2 (3.2)	38.0 (37.7)		159 dec	544
$[Au(CH_2PPh_3)_2Ag(PPh_3)]_2(ClO_4)_4$ (VII)	92	50.9 (51.0)	3.9 (3.75)	24.0 (23.1)		104	558

[a] In acetone, Ω^{-1} cm^2 mol^{-1}.

Table II. Atomic Coordinates ($\times 10^4$) and Isotropic Thermal Parameters ($Å^2 \times 10^3$)

	x	*y*	*z*	*U*
Ag	6144 (1)	839 (1)	5088 (1)	63 (1)[a]
Au	4257 (1)	875 (1)	5572 (1)	44 (1)[a]
Cl(1)	6929 (4)	2706 (5)	4224 (3)	139 (3)[a]
O(11)	7228 (10)	1759 (8)	4409 (6)	130 (5)
O(12)	6876 (17)	3321 (13)	4724 (7)	411 (25)
O(13)	7637 (12)	3085 (15)	3846 (9)	295 (15)
O(14)	6004 (10)	2662 (13)	3900 (8).	224 (10)
Cl(2)	7922 (3)	281 (3)	6270 (2)	66 (2)[a]
O(21)	7552 (11)	560 (12)	5694 (5)	193 (8)
O(22)	7250 (8)	−370 (9)	6516 (5)	117 (5)
O(23)	8049 (15)	1114 (10)	6636 (8)	371 (20)
O(24)	8856 (8)	−171 (11)	6231 (7)	168 (7)
C(1)	3890 (10)	1698 (10)	4790 (6)	49 (4)
P(1)	3034 (3)	2698 (3)	4918 (2)	47 (1)[a]
C(2)	4737 (10)	−42 (11)	6305 (6)	51 (4)
P(2)	4405 (3)	366 (3)	7024 (2)	49 (1)[a]
C(12)	963 (7)	2727 (7)	4724 (4)	65 (4)
C(13)	−13	2370	4778	79 (5)
C(14)	−170	1522	5124	76 (5)
C(15)	649	1033	5417	88 (6)
C(16)	1625	1390	5363	70 (5)
C(11)	1782	2238	5016	48 (3)
C(22)	2632 (8)	4511 (8)	4378 (4)	72 (5)
C(23)	2439	5132	3878	93 (6)
C(24)	2555	4777	3291	83 (5)
C(25)	2865	3799	3205	88 (6)
C(26)	3059	3177	3705	75 (5)
C(21)	2942	3533	4292	58 (4)
C(32)	4356 (8)	3980 (8)	5493 (4)	76 (5)
C(33)	4724	4580	5973	99 (6)
C(34)	4227	4610	6513	103 (7)
C(35)	3362	4040	6572	110 (7)
C(36)	2994	3439	6091	79 (5)
C(31)	3491	3409	5552	48 (4)
C(42)	5469 (8)	−1243 (8)	7487 (4)	71 (5)
C(43)	5796	−1910	7942	79 (5)
C(44)	5388	−1873	8510	95 (6)
C(45)	4653	−1168	8623	91 (6)
C(46)	4325	−500	8168	75 (5)
C(41)	4734	−538	7600	60 (4)
C(52)	2455 (9)	−229 (7)	6822 (5)	79 (5)
C(53)	1410	−113	6808	99 (6)
C(54)	996	767	7015	85 (5)
C(55)	1626	1531	7237	100 (6)
C(56)	2671	1415	7251	83 (5)
C(51)	3085	535	7043	62 (4)
C(62)	5586 (9)	2020 (9)	6816 (4)	87 (6)
C(63)	6098	2891	6988	101 (6)
C(64)	6080	3238	7584	83 (5)
C(65)	5549	2716	8008	94 (6)
C(66)	5037	1845	7836	80 (5)
C(61)	5055	1497	7240	61 (4)

[a] Equivalent isotropic *U* defined as one-third of the trace of the orthogonalized $\mathbf{U}_{ij}$ tensor.

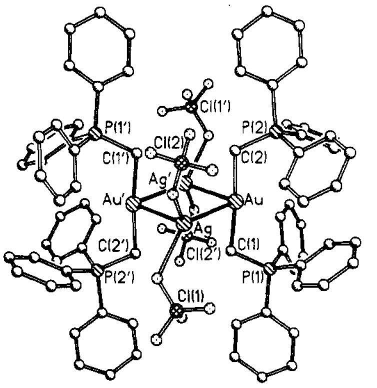

Figure 1. The molecule of IV in the crystal, showing the atom numbering scheme. Radii are arbitrary; H atoms are omitted. Primes indicate symmetry-equivalent atoms (symmetry operator $1 - x, -y, 1 - z$); the crystallographic center of symmetry imposes exact planarity on the Au_2Ag_2 ring.

Table III. Bond Lengths (Å)

Ag–Au	2.783 (1)	Ag–O(11)	2.473 (13)
Ag–O(21)	2.260 (13)	Ag–Au′	2.760 (1)
Au–C(1)	2.079 (13)	Au–C(2)	2.099 (13)
Cl(1)–O(12)	1.382 (18)	Cl(1)–O(11)	1.389 (13)
Cl(1)–O(14)	1.383 (15)	Cl(1)–O(13)	1.391 (20)
Cl(2)–O(22)	1.384 (12)	Cl(2)–O(21)	1.383 (12)
Cl(2)–O(24)	1.389 (12)	Cl(2)–O(23)	1.384 (16)
P(1)–C(11)	1.799 (11)	C(1)–P(1)	1.796 (14)
P(1)–C(31)	1.768 (11)	P(1)–C(21)	1.777 (10)
P(2)–C(41)	1.791 (10)	C(2)–P(2)	1.757 (13)
P(2)–C(61)	1.800 (12)	P(2)–C(51)	1.770 (12)

cipitated as white solids which were filtered off and recrystallized from acetone/diethyl ether.

$[(R_3PCH_2)_2Au\{\mu\text{-}Ag(OClO_3)_2\}_2Au(CH_2PR_3)_2]$ [$PR_3 = PPh_3$ (IV), PPh_2Me (V), or $PPhMe_2$ (VI)]. To a dichloromethane solution (25 mL) of I (0.255 g, 0.3 mmol), II (0.217 g, 0.3 mmol), or III (0.180 g, 0.3 mmol) was added a diethyl ether solution (15 mL) of $AgClO_4$ (0.062 g, 0.3 mmol). The mixture was stirred for 2 h at room temperature. Complexes IV–VI precipitated as white solids which were filtered off.

$[(Ph_3PCH_2)_2Au(\mu\text{-}AgPPh_3)_2Au(CH_2PPh_3)_2](ClO_4)_4$ (VII). To a dichloromethane solution (25 mL) of I (0.255 g, 0.3 mmol) was added $Ag(OClO_3)(PPh_3)$[6] (0.141 g, 0.3 mmol), and the mixture was stirred for 4 h at room temperature, when the solution was evaporated to ca. 4 mL and addition of diethyl ether (20 mL) led to precipitation of complex VII as a white solid.

Crystal Structure Determination of IV. Crystal data: $C_{76}H_{68}AgAuCl_4P_4O_{16}$; M_r 2112.7; monoclinic; space group $P2_1/n$; $a = 13.282$ (3) Å, $b = 13.458$ (4) Å, $c = 22.016$ (5) Å, $\beta = 93.58$ (3)°; $U = 3927$ $Å^3$; $Z = 2$, $D_{calcd} = 1.79$ g cm^{-3}; λ(Mo Kα) = 0.71069 Å; $\mu = 4.5$ mm^{-1}; $F(000) = 2064$.

Data were collected on a Stoe-Siemens four-circle diffractometer in profile-fitting mode.[7] $2\theta_{max} = 50°$, 8960 intensities, 6896 unique,

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[8] In this paper the periodic group notation in parentheses is in accord with recent actions by IUPAC and ACS nomenclature committees. A and B notation is eliminated because of wide confusion. Groups IA and IIA become groups 1 and 2. The d-transition elements comprise groups 3 through 12, and the p-block elements comprise groups 13–18. (Note that the former Roman number designation is preserved in the last digit of the new numbering: e.g., III ⇒ 3 and 13.)

Table IV. Bond Angles (deg)

Au–Ag–O(11)	142.6 (3)	Au–Ag–O(21)	120.6 (3)
O(11)–Ag–O(21)	87.1 (5)	Au–Ag–Au′	93.8 (1)
O(11)–Ag–Au′	101.6 (3)	O(21)–Ag–Au′	107.3 (4)
Ag–Au–C(1)	82.1 (4)	Ag–Au–C(2)	92.7 (4)
C(1)–Au–C(2)	173.9 (5)	Ag–Au–Ag′	86.2 (1)
C(1)–Au–Ag′	89.0 (4)	C(2)–Au–Ag′	87.3 (4)
O(11)–Cl(1)–O(12)	110.2 (10)	O(11)–Cl(1)–O(13)	108.6 (10)
O(12)–Cl(1)–O(13)	109.0 (12)	O(11)–Cl(1)–O(14)	109.9 (10)
O(12)–Cl(1)–O(14)	110.5 (12)	O(13)–Cl(1)–O(14)	108.6 (11)
Ag–O(11)–Cl(1)	118.0 (8)	O(21)–Cl(2)–O(22)	109.2 (8)
O(21)–Cl(2)–O(23)	109.7 (10)	O(22)–Cl(2)–O(23)	110.1 (9)
O(21)–Cl(2)–O(24)	109.2 (9)	O(22)–Cl(2)–O(24)	110.3 (8)
O(23)–Cl(2)–O(24)	108.3 (10)	Ag–O(21)–Cl(2)	145.1 (9)
Au–C(1)–P(1)	112.8 (6)	C(1)–P(1)–C(11)	111.1 (6)
C(1)–P(1)–C(21)	111.4 (6)	C(1)–P(1)–C(31)	109.9 (6)
Au–C(2)–P(2)	115.4 (7)	C(2)–P(2)–C(41)	111.2 (6)
C(2)–P(2)–C(51)	111.5 (6)	C(2)–P(2)–C(61)	111.0 (6)
P(1)–C(11)–C(12)	118.8 (3)	P(1)–C(11)–C(16)	121.2 (3)
P(1)–C(21)–C(22)	119.8 (3)	P(1)–C(21)–C(26)	119.7 (3)
P(1)–C(31)–C(32)	118.0 (4)	P(1)–C(31)–C(36)	122.0 (4)
P(2)–C(41)–C(42)	118.6 (3)	P(2)–C(41)–C(46)	121.3 (3)
P(2)–C(51)–C(52)	118.1 (3)	P(2)–C(51)–C(56)	121.9 (3)
P(2)–C(61)–C(62)	120.3 (3)	P(2)–C(61)–C(66)	119.7 (3)

4569 with $F > 4\sigma(F)$ were used for all calculations (program system SHELXTL). Cell constants were refined from 2θ values of 56 reflections in the range 20–23°. An absorption correction based on ψ-scans was applied (transmissions 0.37–0.66; crystal size 0.45 × 0.45 × 0.4 mm).

The structure was solved by the heavy-atom method and refined on F to $R = 0.075$ and $R_w = 0.071$ [Au, Ag, Cl, and P atoms anisotropic; phenyl rings as idealized rigid groups with C–C = 1.395 Å and C–H = 0.96 Å; methylene H included by using a riding model; weighting scheme $w^{-1} = \sigma^2(F) + 0.0005F^2$; 168 parameters]. Final atomic coordinates are given in table II (see also Figure 1).

The high-temperature factors of the O atoms and the presence of significant electron density associated with the perchlorate groups probably indicate disorder, but no sensible disorder model could be refined. The corresponding bond lengths and angles of the ClO_4^- groups should therefore be interpreted with caution. These problems are also reflected in the rather high R value.

Single crystals of V could be obtained but lost solvent too readily to permit X-ray analysis. The cell constants of V are a = 17.5 Å, b = 17.1 Å, c = 25.25 Å, and β = 102.5° (monoclinic P).

Acknowledgment. We thank the CAICYT (Spain) and the Fonds der Chemischen Industrie for financial support.

Supplementary Material Available: Tables of positional and thermal parameters of calculated hydrogen atoms and anisotropic thermal parameters for IV (2 pages); a listing of structure factor amplitudes for IV (41 pages). Ordering information is given on any current masthead page.

Mercury in Organic Chemistry. 34.[1] Synthesis of Vinylmercurials via Mercuration of Propargylic Amines

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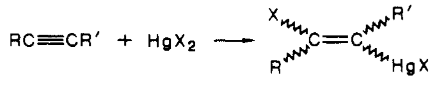
Propargylic amines react readily with 1–2 M $HgCl_2$ in 2–6 M HCl to precipitate the ammonium trichloromercurate, bis(ammonium) tetrachloromercurate, or bis(ammonium) hexachlorodimercurate salts **1–10** in which mercuric chloride has added in a trans manner across the original carbon–carbon triple bond placing the mercury nearer to nitrogen. X-ray crystallographic data on one of each type of these salts are provided.

Introduction

Vinylmercurials have proven to be versatile intermediates in organic synthesis.[2–4] While a number of methods presently exist for the preparation of vinylmercurials, one of the simplest and most important approaches involves the direct addition of mercury(II) salts to acetylenes (eq 1). For instance, mercuric acetate adds primarily trans



(1)

to internal dialkyl-[5–10] and alkylarylacetylenes[11,12] and cis to diphenylacetylene[5,7,13,14] to afford stable, isolable (β-acetoxyvinyl)mercurials. Mercuric chloride adds to internal acetylenic halides,[15,16] ethers,[17–19] alcohols,[15,20,21]

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