Acta Cryst. (1988). C**44**, 978–979

Structure of (Methoxymethylethynyl)[tris(*p*-tolyl)phosphine]gold(I)

BY G. A. CARRIEDO AND V. RIERA

Departamento de Química Organometálica, Facultad de Química, 33071 *Oviedo, Spain*

AND X. SOLANS AND J. SOLANS

Departamento de Cristalografia y Mineralogia, Universidad de Barcelona, Gran Via 585, 08007 *Barcelona, Spain*

(*Received* 13 *October* 1987; *accepted* 4 *February* 1988)

Abstract. $[Au(C_4H_5O)\{(C_7H_7)_3P\}]$, $M_r = 570\cdot42$, triclinic, $P\bar{1}$, $a = 12\cdot012\ (1)$, $b = 10\cdot818\ (1)$, $c = 10\cdot263\ (1)$ Å, $\alpha = 108\cdot87\ (1)$, $\beta = 114\cdot90\ (1)$, $\gamma = 79\cdot93\ (1)°$, $V = 1143\cdot5\ (3)$ Å^3, $Z = 2$, $D_x = 1\cdot656$ g cm^{-3}, $F(000) = 556$, $\lambda(\text{Mo } K\alpha) = 0\cdot71069$ Å, $\mu(\text{Mo } K\alpha) = 73\cdot24$ cm^{-1}, $T = 288$ K, $R = 0\cdot015$ ($wR = 0\cdot015$) for 3376 observed reflections. The structure consists of discrete molecules with an almost linear ethynyl–gold group.

Introduction. Of the two gold(I) σ-acetylide derivatives that have been characterized by X-ray diffraction, one has a gold-atom chain structure $[Au(C_2Ph)(H_2NiPr)]$ (Corfield & Shearer, 1967) and the other consists of discrete molecules $[Au(C_2C_6F_5)(PPh_3)]$ (Bruce, Horn, Matison & Snow, 1984). We report here the structure determination of the analogous compound $\{Au(C_2CH_2OCH_3)[P(C_6H_4Me\text{-}4)_3]\}$, prepared by Carriedo & Riera (1988) following a method similar to that published recently for other ethynyl–gold complexes (Cross & Davidson, 1986).

Experimental. Colourless crystals were obtained by diffusion of hexane into a toluene solution of the complex at room temperature. A prismatic crystal ($0\cdot07 \times 0\cdot07 \times 0\cdot1$ mm) was selected and mounted on a Philips PW 1100 four-circle diffractometer. Unit-cell parameters were determined from 25 reflections ($4 \leq \theta \leq 12°$) and refined by least squares. Intensities were collected with graphite-monochromatized Mo $K\alpha_1$ radiation using the ω-scan technique, scan width 0·8°, scan speed 0·03° s^{-1}. 3422 reflections were measured in the range $2 \leq \theta \leq 25°$; *hkl* range: −12 to 12; 0 to 12; 0 to 12. Max. $(\sin\theta)/\lambda = 0\cdot595$ Å^{-1}; 3376 reflections were taken as observed applying the condition $I \geq 2\cdot5\sigma(I)$. Three reflections were measured every 2 h as orientation and intensity controls, significant intensity decay was not observed. Lorentz–polarization and absorption (*SHELX76*; Sheldrick, 1976) corrections were made (max. and min. transmission factors 0·86 and 0·74 respectively).

The Au atom was located from a Patterson synthesis and the remaining atoms from a weighted Fourier synthesis. The structure was refined by a least-squares method using the *SHELX76* computer program with all atoms anisotropic (Sheldrick, 1976). The function minimized was $\sum w||F_o| - |F_c||^2$, where $w = \sigma^{-2}(F_o)$. The final R factor was 0·015 ($wR = 0\cdot015$) for all observed reflections. H atoms were not included, f, f' and f'' were taken from *International Tables for X-ray Crystallography* (1974). Max. shift/e.s.d. = −0·4 in U_{13} of C(31), max. and min. peak in final difference synthesis 0·3 and −0·3 e Å^{-3}; number of parameters refined was 253. IBM-4033 computer. Final atomic coordinates are given in Table 1, selected bond distances and angles in Table 2.*

Discussion. The structure consists of discrete molecules (Fig. 1) linked by van der Waals forces. The shortest intermolecular separation is C(5)—Au$^{\text{i}}$ [3·635 (3) Å; symmetry code: (i) $-x$, $1-y$, $1-z$]. The Au atom displays a linear coordination with a P—Au—C(1) angle of 178·3 (1)°. The distances P—Au, C(1)—Au and C(1)—C(2) (Table 2) are practically identical to those observed in the related complex $[Au(C_2C_6F_5)(PPh_3)]$ [2·274 (3), 1·993 (14) and 1·197 (16) Å respectively] (Bruce, Horn, Matison & Snow, 1984). While the angle C(1)—C(2)—C(3) is 178·2 (3)°, the angle Au—C(1)—C(2) [170·3 (2)°] is significantly different from linearity. This was also noted in $[Au(C_2C_6F_5)(PPh_3)]$ and attributed to packing forces. The angles C(2)—C(3)—O(4) and C(3)—O(4)—C(5) (Table 2) are very close to the expected values for the tetrahedral coordination of the C(3) and O(4) atoms (sp^3), and the dihedral angle C(2)—C(3)—O(4)—C(5) is −67·6°.

---

* Lists of structure factors, anisotropic thermal parameters and a complete list of bond lengths and angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44752 (18 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

0108-2701/88/060978-02$03.00

Table 1. *Final atomic coordinates* (× 10^4, Au × 10^5) *and equivalent isotropic thermal parameters, with e.s.d.'s in parentheses*

$$B_{eq} = \tfrac{8}{3}\pi^2 \textstyle\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}(Å^2)
Au	23649 (3)	23085 (3)	52522 (4)	3·88 (2)
P	3600 (3)	1585 (3)	7274 (3)	3·84 (12)
C(1)	1222 (10)	2946 (10)	3464 (10)	3·69 (41)
C(2)	678 (14)	3456 (11)	2514 (14)	5·40 (59)
C(3)	27 (19)	4099 (16)	1311 (16)	7·61 (84)
O(4)	212 (12)	5445 (11)	1788 (14)	8·16 (72)
C(5)	−328 (20)	6172 (16)	2812 (24)	8·63 (113)
C(11)	3146 (8)	−42 (9)	6995 (10)	3·44 (37)
C(12)	4039 (10)	−1074 (10)	7225 (12)	4·24 (47)
C(13)	3708 (12)	−2296 (11)	7070 (14)	4·89 (52)
C(14)	2500 (13)	2514 (11)	6746 (14)	4·89 (56)
C(15)	1585 (11)	−1475 (10)	6491 (14)	4·63 (50)
C(16)	1980 (11)	−257 (10)	6682 (14)	4·69 (49)
C(17)	2071 (16)	−3831 (12)	6615 (21)	7·21 (81)
C(21)	5257 (10)	1460 (10)	7668 (11)	3·66 (41)
C(22)	5590 (13)	1407 (11)	6495 (14)	5·06 (57)
C(23)	6872 (13)	1257 (12)	6760 (15)	5·06 (57)
C(24)	7775 (12)	1142 (11)	8123 (14)	4·84 (53)
C(25)	7388 (13)	1183 (13)	9268 (15)	5·55 (63)
C(26)	6139 (12)	1375 (12)	9039 (14)	4·91 (55)
C(27)	9105 (14)	897 (16)	8319 (21)	7·09 (82)
C(31)	3507 (10)	2595 (9)	9012 (13)	4·00 (45)
C(32)	3155 (9)	2130 (10)	9906 (12)	3·94 (43)
C(33)	3105 (11)	2951 (11)	11230 (14)	4·82 (53)
C(34)	3362 (12)	4283 (11)	11701 (16)	5·27 (58)
C(35)	3674 (12)	4763 (11)	10773 (14)	5·06 (55)
C(36)	3741 (12)	3928 (10)	9451 (14)	4·95 (54)
C(37)	3386 (18)	5191 (14)	13209 (17)	7·04 (81)

Table 2. *Selected bond distances* (Å) *and angles* (°)

P–Au	2·274 (1)	C(2)–C(3)	1·474 (4)
Au–C(1)	2·024 (2)	C(3)–O(4)	1·402 (4)
C(1)–C(2)	1·169 (3)	O(4)–C(5)	1·415 (4)
P–Au–C(1)	178·3 (1)	C(2)–C(3)–C(4)	113·1 (3)
Au–C(1)–C(2)	170·3 (2)	C(3)–O(4)–C(5)	113·7 (3)
C(1)–C(2)–C(3)	178·2 (3)		

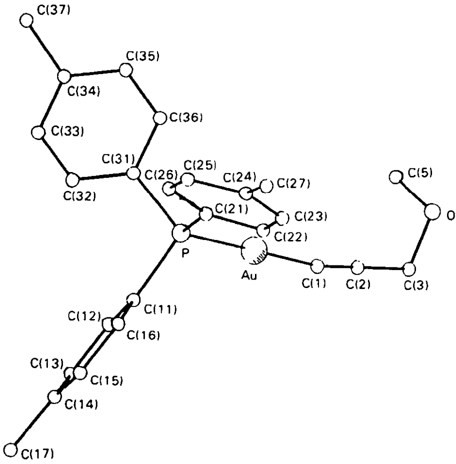

Fig. 1. One molecule of the title complex showing the atom numbering.

We thank the Spanish CAICYT for financial support and the University of Barcelona for a grant.

References

Bruce, M. I., Horn, E., Matison, J. G. & Snow, M. R. (1984). *Aust. J. Chem.* **37**, 1163–1170.

Carriedo, G. A. & Riera, V. (1988). In preparation.

Corfield, P. W. R. & Shearer, H. M. M. (1967). *Acta Cryst.* **23**, 156–162.

Cross, R. J. & Davidson, M. F. (1986). *J. Chem. Soc. Dalton Trans.* pp. 411–414.

International Tables for X-ray Crystallography (1974). Vol. IV, pp. 99–100, 149. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

Sheldrick, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.

Acta Cryst. (1988). C**44**, 979–981

Structure of a Dinuclear Osmium Complex Containing a Carboxamido and Three Bromine Ligands

By Kuang-Lieh Lu,* Ying-Chih Lin, Ming-Chu Cheng and Yu Wang

Department of Chemistry, National Taiwan University, Taipei, Taiwan

(*Received* 24 *November* 1987; *accepted* 3 *February* 1988)

Abstract. μ-Bromo-μ-(isopropylcarbamoyl-*O*,*C*)-bis(bromotricarbonylosmium), $(\mu\text{-Br})Os_2(CO)_6[\mu\text{-OCNHCH}(CH_3)_2]Br_2$, $M_r = 874{\cdot}1$, monoclinic, $P2_1/c$, $a = 14{\cdot}579\ (3)$, $b = 10{\cdot}783\ (1)$, $c = 11{\cdot}987\ (4)$ Å, $\beta = 92{\cdot}08\ (2)°$, $V = 1883\ (1)$ Å^3, $Z = 4$, $D_x = 3{\cdot}084$ Mg m^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}7107$ Å, $\mu = 19{\cdot}831$ mm^{-1}, $F(000) = 1552$, $T = 298$ K. $R = 0{\cdot}061$ for 2358 observed reflections. The structure consists of two Os metal centers, each with three terminal carbonyl ligands, bridged by both a Br atom and a μ-η^2 carboxamido ligand; two terminal Br atoms are *trans* to each other. The two Os atoms are separated by 3·956 (2) Å, indicating no metal–metal interaction.

---

* Permanent address: Institute of Chemistry, Academia Sinica, Nankang, Taipei, Taiwan.

0108-2701/88/060979-03$03.00