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Structure of a Dinuclear Gold(II) Ylide Complex Containing Unidentate Acetate Ligands

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Abstract. Bis(acetato-*O*)bis-μ-(dimethylenediphenylphosphoranyl-*C,C'*)digold(II)(*Au*–Au), $[Au_2(C_2H_3O_2)_2\{P(CH_2)_2(C_6H_5)_2\}_2]$, $M_r = 937{\cdot}9$, triclinic, $P\bar{1}$, $a = 10{\cdot}466\,(5)$, $b = 11{\cdot}581\,(5)$, $c = 14{\cdot}206\,(6)$ Å, $\alpha = 99{\cdot}72\,(3)$, $\beta = 98{\cdot}90\,(3)$, $\gamma = 107{\cdot}90\,(3)°$, $V = 1575\,(1)$ Å^3, $Z = 2$, $D_x = 1{\cdot}97$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 9{\cdot}403$ mm^{-1}, $F(000) = 892$, $T = 298$ K, $R = 0{\cdot}0338$ and $wR = 0{\cdot}0344$ for 361 variable parameters and 2070 reflections with $F_o^2 > 3\sigma(F_o^2)$. The structure of a dinuclear gold(II) ylide complex containing unidentate acetate ligands is reported. The asymmetric unit consists of two crystallographically independent half-dimers. The Au atoms have square-planar coordination geometries and are symmetrically bridged by ylide anion ligands. In both structures a metal–metal bond is formed.

Experimental. Green regularly shaped plate of approximate dimensions 0·01 × 0·05 × 0·2 mm. Triclinic symmetry suggested on the basis of interaxial angles and confirmed by a Delaunay reduction. Refined cell parameters obtained from setting angles of 20 reflections with $20 < 2\theta < 25°$. Data collection carried out at room temperature using ω-scanning technique in bisecting geometry. (Nicolet *R3m/E* diffractometer, graphite-monochromated Mo $K\alpha$ radiation.) Backgrounds estimated from 96-step profile. Scan rate variable, 2–30° min^{-1}, scan range −1·0° in ω from $K\alpha_1$ to +1·0° from $K\alpha_2$. 2902 reflections ($\pm h, \pm k, +l$; $h_{max} = 10$, $k_{max} = 11$, $l_{max} = 13$) measured with $0 < 2\theta < 40°$. Three standards (010, $\bar{1}00$, 112) measured every 100 data. Data corrected for standard decay (<6%), absorption, Lorentz and polarization effects. Absorption corrections applied empirically on the basis of azimuthal scans of 10 low-angle reflections (transmission range 0·195–0·261). Structure solution and refinement carried out using *SHELXTL* collection of crystallographic software (Sheldrick, 1978). Au-atom positions from sharpened Patterson map, remaining atoms from difference Fourier maps. Scattering factors, including terms for anomalous dispersion, from *International Tables for X-ray Crystallography* (1974). All atoms refined anisotropically, H atoms not included. Refinement based on F with $w^{-1} = [\sigma^2(F)$

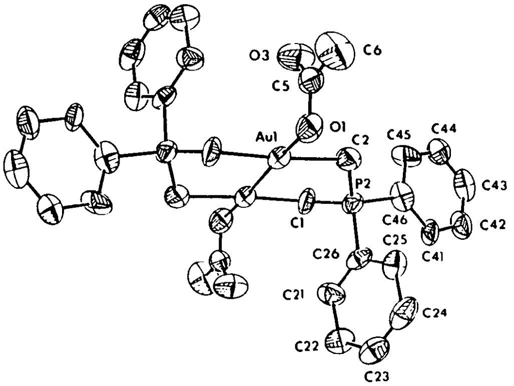

Fig. 1. A perspective view of one dimer illustrating the atomic-numbering scheme. Thermal ellipsoids are drawn at the 50% probability level.

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Table 1. *Atomic coordinates* ($\times 10^4$) *and isotropic thermal parameters* ($Å^2 \times 10^3$) *for* $[Au(CH_2)_2P(C_6H_5)_2]_2(CH_3CO_2)_2$

	x	*y*	*z*	*U*
Au(1)	540 (1)	335 (1)	4284 (1)	43 (1)*
Au(2)	−1058 (1)	347 (1)	30 (1)	46 (1)*
P(1)	−1171 (4)	−2017 (4)	930 (3)	49 (2)*
P(2)	−1738 (4)	1534 (4)	4746 (3)	45 (2)*
O(1)	1304 (13)	765 (11)	3039 (7)	63 (6)*
O(2)	−2874 (10)	821 (10)	−6 (8)	66 (5)*
O(3)	3173 (12)	2034 (12)	4124 (10)	84 (7)*
O(4)	8234 (12)	2354 (12)	1298 (10)	100 (7)*
C(1)	−1442 (15)	1057 (14)	5861 (11)	56 (8)*
C(2)	−242 (16)	1809 (15)	4241 (13)	65 (8)*
C(3)	−1727 (15)	−700 (15)	1032 (11)	54 (8)*
C(4)	−624 (15)	1471 (14)	−952 (11)	55 (7)*
C(5)	2509 (20)	1554 (16)	3264 (14)	55 (9)*
C(6)	3138 (21)	1927 (19)	2411 (14)	91 (11)*
C(7)	−2784 (17)	1767 (16)	606 (12)	75 (9)*
C(8)	−4076 (18)	2138 (18)	573 (15)	91 (11)*
C(11)	−1415 (17)	−4067 (16)	1741 (12)	55 (8)*
C(12)	−1803 (20)	−4747 (18)	2427 (16)	81 (11)*
C(13)	−2387 (19)	−4311 (22)	3199 (15)	88 (11)*
C(14)	−2587 (16)	−3164 (22)	3274 (12)	72 (10)*
C(15)	−2199 (15)	−2456 (16)	2625 (13)	59 (8)*
C(16)	−1631 (15)	−2933 (15)	1845 (11)	52 (8)*
C(21)	−4022 (16)	−627 (14)	4115 (12)	59 (8)*
C(22)	−5221 (17)	−1466 (15)	3374 (12)	58 (8)*
C(23)	−5502 (17)	−1278 (18)	2459 (13)	61 (9)*
C(24)	−4658 (20)	−235 (18)	2223 (12)	72 (10)*
C(25)	−3430 (17)	602 (15)	2907 (11)	53 (8)*
C(26)	−3185 (15)	388 (15)	3832 (11)	48 (8)*
C(31)	−3493 (15)	−3671 (15)	−355 (11)	58 (8)*
C(32)	−4234 (20)	−4632 (16)	−1227 (15)	81 (10)*
C(33)	−3535 (26)	−5101 (18)	−1843 (13)	86 (12)*
C(34)	−2059 (24)	−4610 (22)	−1677 (16)	106 (13)*
C(35)	−1353 (17)	−3588 (17)	−833 (13)	72 (9)*
C(36)	−2112 (15)	−3185 (14)	−199 (11)	53 (7)*
C(41)	−3591 (16)	2892 (15)	4720 (12)	56 (8)*
C(42)	−3894 (19)	3984 (17)	5071 (14)	69 (9)*
C(43)	−2846 (23)	5040 (17)	5648 (13)	72 (11)*
C(44)	−1502 (19)	5060 (15)	5892 (13)	66 (9)*
C(45)	−1175 (16)	4002 (15)	5612 (12)	61 (8)*
C(46)	−2209 (18)	2935 (15)	5008 (11)	58 (9)*

* Equivalent isotropic *U* defined as one third of the trace of the orthogonalized U_{ij} tensor.

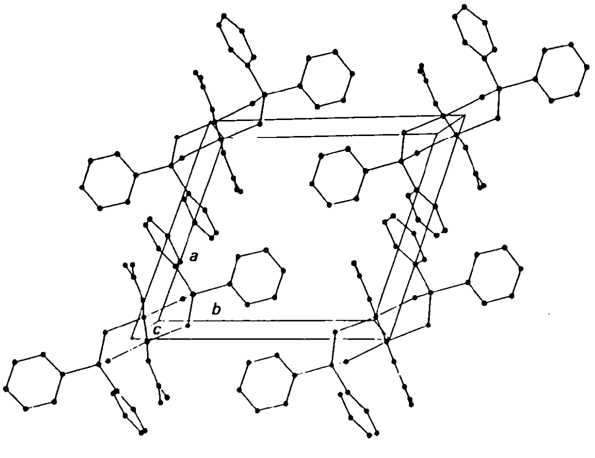

Fig. 2. A view of the cell contents projected down the *c* axis.

Table 2. *Bond lengths* (Å) *and angles* (°) *for* $[Au(CH_2)_2P(C_6H_5)_2]_2(CH_3CO_2)_2$

Au(1)—O(1)	2·124 (12)	Au(1)—C(2)	2·114 (19)
Au(1)—Au(1′)	2·592 (1)	Au(1)—C(1′)	2·102 (19)
Au(2)—O(2)	2·130 (12)	Au(2)—C(3)	2·087 (17)
Au(2)—C(4)	2·072 (17)	Au(2)—Au(2′)	2·586 (1)
P(1)—C(3)	1·784 (19)	P(1)—C(16)	1·841 (18)
P(1)—C(36)	1·825 (13)	P(1)—C(4′)	1·781 (15)
P(2)—C(1)	1·778 (18)	P(2)—C(2)	1·784 (19)
P(2)—C(26)	1·821 (13)	P(2)—C(46)	1·833 (20)
O(1)—C(5)	1·264 (20)	O(2)—C(7)	1·247 (21)
O(3)—C(5)	1·247 (21)	O(4)—C(7′)	1·253 (18)
C(1)—Au(1′)	2·102 (19)	C(4)—P(1′)	1·781 (15)
C(5)—C(6)	1·530 (30)	C(7)—C(8)	1·537 (29)
C(7)—O(4′)	1·252 (18)	C(11)—C(12)	1·385 (29)
C(11)—C(16)	1·388 (27)	C(12)—C(13)	1·429 (33)
C(13)—C(14)	1·396 (38)	C(14)—C(15)	1·361 (30)
C(15)—C(16)	1·445 (25)	C(21)—C(22)	1·457 (19)
C(21)—C(26)	1·395 (23)	C(22)—C(23)	1·355 (26)
C(23)—C(24)	1·387 (27)	C(24)—C(25)	1·437 (20)
C(25)—C(26)	1·378 (24)	C(31)—C(32)	1·442 (22)
C(31)—C(36)	1·347 (20)	C(32)—C(33)	1·371 (34)
C(33)—C(34)	1·438 (35)	C(34)—C(35)	1·447 (25)
C(35)—C(36)	1·403 (26)	C(41)—C(42)	1·425 (28)
C(41)—C(46)	1·423 (26)	C(42)—C(43)	1·389 (22)
C(43)—C(44)	1·387 (31)	C(44)—C(45)	1·382 (27)
C(45)—C(46)	1·403 (19)		
O(1)—Au(1)—C(2)	86·1 (7)	O(1)—Au(1)—Au(1′)	175·6 (3)
C(2)—Au(1)—Au(1′)	95·0 (5)	O(1)—Au(1)—C(1′)	85·0 (6)
C(2)—Au(1)—C(1′)	171·0 (7)	O(2)—Au(2)—C(3)	86·5 (6)
O(2)—Au(2)—C(4)	85·6 (6)	C(3)—Au(2)—C(4)	172·0 (7)
O(2)—Au(2)—Au(2′)	176·1 (3)	C(3)—Au(2)—Au(2′)	93·9 (5)
C(4)—Au(2)—Au(2′)	94·0 (5)	C(3)—P(1)—C(16)	113·2 (8)
C(3)—P(1)—C(36)	111·2 (7)	C(16)—P(1)—C(36)	100·5 (7)
C(3)—P(1)—C(4′)	107·5 (7)	C(16)—P(1)—C(4′)	114·1 (8)
C(36)—P(1)—C(4′)	110·4 (7)	C(1)—P(2)—C(2)	110·2 (8)
C(1)—P(2)—C(26)	112·9 (7)	C(2)—P(2)—C(26)	108·3 (7)
C(1)—P(2)—C(46)	107·7 (8)	C(2)—P(2)—C(46)	111·9 (8)
C(26)—P(2)—C(46)	105·8 (8)	Au(1)—O(1)—C(5)	112·6 (11)
Au(2)—O(2)—C(7)	116·5 (10)	P(2)—C(1)—Au(1′)	111·3 (8)
Au(1)—C(2)—P(2)	110·1 (9)	Au(2)—C(3)—P(1)	110·7 (9)
Au(2)—C(4)—P(1′)	111·3 (8)	O(1)—C(5)—O(3)	123·7 (19)
O(1)—C(5)—C(6)	116·1 (15)	O(3)—C(5)—C(6)	120·2 (15)
O(2)—C(7)—C(8)	117·1 (13)	O(2)—C(7)—O(4′)	124·4 (18)
C(8)—C(7)—O(4′)	118·0 (16)	C(12)—C(11)—C(16)	116·0 (17)
C(11)—C(12)—C(13)	122·3 (21)	C(12)—C(13)—C(14)	119·6 (21)
C(13)—C(14)—C(15)	120·0 (19)	C(14)—C(15)—C(16)	118·9 (18)
P(1)—C(16)—C(11)	117·4 (13)	P(1)—C(16)—C(15)	119·5 (14)
C(11)—C(16)—C(15)	123·1 (16)	C(22)—C(21)—C(26)	116·1 (15)
C(21)—C(22)—C(23)	122·0 (15)	C(22)—C(23)—C(24)	119·6 (14)
C(23)—C(24)—C(25)	121·1 (16)	C(24)—C(25)—C(26)	117·4 (15)
P(2)—C(26)—C(21)	118·4 (12)	P(2)—C(26)—C(25)	118·0 (11)
C(21)—C(26)—C(25)	123·5 (12)	C(32)—C(31)—C(36)	118·6 (17)
C(31)—C(32)—C(33)	120·3 (17)	C(32)—C(33)—C(34)	121·6 (16)
C(33)—C(34)—C(35)	116·6 (21)	C(34)—C(35)—C(36)	119·5 (16)
P(1)—C(36)—C(31)	118·6 (13)	P(1)—C(36)—C(35)	118·2 (10)
C(31)—C(36)—C(35)	123·1 (13)	C(42)—C(41)—C(46)	117·4 (13)
C(41)—C(42)—C(43)	119·6 (19)	C(42)—C(43)—C(44)	121·5 (20)
C(43)—C(44)—C(45)	120·8 (14)	C(44)—C(45)—C(46)	118·5 (17)
P(2)—C(46)—C(41)	121·5 (11)	P(2)—C(46)—C(45)	116·0 (15)
C(41)—C(46)—C(45)	122·0 (17)		

$+ 0{\cdot}0012(F^2)]$. Convergence to conventional *R* values of $R = 0{\cdot}0338$ and $wR = 0{\cdot}0344$ using 2070 unique reflections with $F_o^2 > 3\sigma(F_o^2)$. Max. shift/σ for final cycle $-0{\cdot}01$. Difference Fourier map calculated after last cycle of refinement featureless with min. and max. residual electron densities of $+0{\cdot}77$ and $-0{\cdot}74$ e $Å^{-3}$. A perspective view of one entire dimer is presented in Fig. 1. In Fig. 2 is shown a view of the unit-cell contents projected down the *c* axis. Atomic positional and equivalent isotropic thermal parameters for both molecules are presented in Table 1.* Intramolecular bond distances and angles are summarized in Table 2.

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 42923 (20 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Related literature. This complex is the second example of a gold(II)–carboxylate complex and is one in a series of dinuclear gold(II) ylide complexes containing pseudo-halogen ligands (Porter, Knachel & Fackler, 1986; Murray, Mazany & Fackler, 1985; Porter & Fackler, 1986).

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Structure of the Propellant Oxalylhydroxamic Acid

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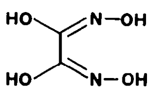
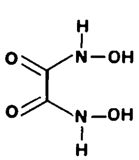
Abstract. $C_2H_4N_2O_4$, $M_r = 120{\cdot}06$, monoclinic, $P2_1/n$, $a = 5{\cdot}210\,(2)$, $b = 3{\cdot}871\,(1)$, $c = 10{\cdot}730\,(3)$ Å, $\beta = 95{\cdot}45\,(3)°$, $V = 215{\cdot}41\,(11)$ Å^3, $Z = 2$, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71069$ Å (graphite monochromator), $\mu = 1{\cdot}69$ cm^{-1}, $D_m = 1{\cdot}85$, $D_x = 1{\cdot}852$ g cm^{-3}, $F(000) = 124$, room temperature (291 K), least-squares refinement, final $R = 0{\cdot}037$ for 563 reflections. The molecule contains a crystallographic center of symmetry. The —C=O bond length of 1·232 (2) Å and the presence of an experimentally determined $\gt$N—H group indicate that the molecule exists as the keto tautomer rather than as the glyoxime form. There is intermolecular hydrogen bonding between the H of the hydroxy group and the O of the carbonyl group [=O···H—O, 1·725 (2) Å].

Experimental. Crystals of the title compound and the value for D_m were provided by Dr R. L. Willer of Morton Thiokol. Experimental conditions for data collection using a Nicolet $R3$ diffractometer are given in Table 1. Three check reflections (0,0,10, 020, $\bar{1}23$) were monitored every 45 reflections. No decay was observed. Absorption corrections were not applied. Lorentz and polarization corrections, including those for the graphite monochromator, were applied. Systematic absences: $h0l$, $h+l = 2n+1$; $0k0$, $k = 2n+1$. Solution with default parameters for the multi-solution program of *SHELXTL* (Sheldrick, 1983). The C, N, and O atoms were all refined anisotropically; H atoms observed on difference Fourier maps refined isotropically without constraints; minimized $[\sum w(|F_o| - k|F_c|)^2]$ by blocked-cascade least-squares algorithm of *SHELXTL*; $w = 1/[\sigma^2(F) + 0{\cdot}0001F^2]$. 45 parameters varied during the final cycle. Maximum shift to e.s.d. was 0·01 in the last cycle. The final difference Fourier map exhibited peaks and troughs ranging from +0·39 to −0·27 e Å^{-3}. Atomic scattering factors as supplied within *SHELXTL*.

Atomic coordinates and thermal parameters are given in Table 2.* A plot of the molecule with bond lengths and angles is shown in Fig. 1. The title compound is used extensively in gas generator propellant formulations. Because of its wide use, it was of interest to determine whether it exists in the solid state as the glyoxime (*a*) or the di-keto tautomer (*b*).



(*a*)



(*b*)

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43127 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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