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Acta Cryst. (1986). C**42**, 1128–1131

Structure of the First Example of an Organometallic Dinuclear Gold(II) Complex Possessing Bonds to Oxygen

BY LEIGH CHRISTOPHER PORTER AND JOHN P. FACKLER JR*

Laboratory for Molecular Structure and Bonding, Texas A&M University, College Station, TX 77843, *USA*

(*Received* 29 *October* 1985; *accepted* 17 *March* 1986)

Abstract. Bis(benzoato-*O*)bis-μ-(dimethylenediphenylphosphoranyl-*C*,*C'*)-digold(II)(*Au–Au*)-tetrahydrofuran (1/1), $[Au_2(C_7H_5O_2)_2\{P(CH_2)_2(C_6H_5)_2\}_2].C_4H_8O$, $M_r = 1134{\cdot}8$, triclinic, $P\bar{1}$, $a = 14{\cdot}464$ (7), $b = 15{\cdot}927$ (8), $c = 10{\cdot}853$ (4) Å, $\alpha = 99{\cdot}88$ (3), $\beta = 110{\cdot}95$ (3), $\gamma = 63{\cdot}60$ (3)°, $V = 2081$ (1) Å^3, $Z = 2$, $D_x = 1{\cdot}81$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 7{\cdot}146$ mm^{-1}, $F(000) = 1100$, $T = 298$ K, $R = 0{\cdot}0495$ and $wR = 0{\cdot}0472$ for 2986 reflections with $F_o^2 > 3\sigma(F_o^2)$. The structure consists of discrete dinuclear gold(II) ylide dimers containing unidentate benzoate ligands. The gold atoms have square-planar coordination geometries and are symmetrically bridged by ylide anion ligands. The asymmetric unit contains two crystallographically independent half-dimers and a molecule of solvent. In each dimer a metal–metal bond is present.

Introduction. The emergence of phosphorus ylides as ligands capable of forming strong metal–carbon bonds has led to the recent preparation of a large number of novel ylide coordination compounds. Examples of complexes containing ylide ligands can currently be found for most of the transition-metal series of elements, including several main-group and lanthanide elements (Schmidbaur, 1975, 1983; Kaska, 1983). Because of the importance of organometallic complexes in homogeneous catalysis, both the structures and the chemical reactivities of ylide complexes are of considerable interest.

The dimeric gold(I) ylide $[Au(CH_2)_2P(C_6H_5)_2]_2$ has been shown to have an extensive reaction chemistry. The chemistry, in general, is characterized by oxidative–addition reactions of the type frequently observed in binuclear organometallic complexes containing bridging bidentate phosphine ligands. Examples include two-center two-electron oxidative–addition reactions leading to dinuclear gold(II) products with discrete metal–metal bonds (Fackler & Basil, 1984) as well as the formation of molecular gold(III) *A*-frame species containing bridging methylene groups (Murray, Mazany & Fackler, 1985). Less frequently observed reactions include isomerizations (Dudis & Fackler, 1985) and cleavage reactions leading to mononuclear products (Porter, Knachel & Fackler, 1986).

As part of an on-going project aimed at exploring the reaction chemistry of dinuclear gold ylides, we recently examined the reaction of the dinuclear gold(I) ylide with benzoyl peroxide. In this paper we report the crystal structure of a dinuclear gold(II) ylide complex containing oxygen-bound carboxylate ligands and an exceptionally short gold–gold bond.

Experimental. The Au[I] ylide dimer, $[Au(CH_2)_2P\text{-}C_6H_5)_2]_2$, was prepared by a modification of the literature procedure (Schmidbaur & Franke, 1975). The benzoate adduct was obtained by adding an approximately equimolar amount of benzoyl peroxide (MCB Manufacturing Chemicals, Inc.) to a solution of the gold(I) dimer in benzene. Crystals suitable for X-ray analysis were obtained by crystallization from a tetrahydrofuran/diethyl ether solution. Single multifaceted red crystal of approximate dimensions 0·3 × 0·3 × 0·25 mm. Triclinic symmetry suggested on basis of interaxial angles and confirmed by Delaunay reduction. Axial lengths checked by comparison with interlayer spacings observed in axial photographs. Refined cell parameters from setting angles of 20 reflections with

* To whom correspondence should be addressed.

0108-2701/86/091128-04$01.50

$20 < 2\theta < 35°$. Data collection at room temperature using ω-scanning technique in bisecting geometry (Nicolet *R3m/E* diffractometer, graphite-monochromated Mo $K\alpha$ radiation). Intensities measured for 5437 unique reflections ($+h$, $\pm k$, $\pm l$; $h_{max} = 14$, $k_{max} = 17$, $l_{max} = 11$) with $0 < 2\theta < 45°$. Scan rate variable, 2–30° min^{-1}; scan range $-1{\cdot}0°$ in ω from $K\alpha_1$ to $+1{\cdot}0°$ from $K\alpha_2$. Backgrounds estimated from a 96-step peak profile. Three low-angle standards ($\bar{1}\bar{1}\bar{1}$, $22\bar{1}$, $1\bar{1}\bar{1}$) measured every 100 data. Data corrected for standard decay (<6%), absorption, Lorentz and polarization effects. No reflections had intensities exceeding range for valid coincidence correction. Corrections for absorption applied empirically on basis of azimuthal scans of ten low-angle reflections (transmission factors 0·154 to 0·297). Crystal solution and refinement using *SHELXTL* collection of crystallographic software (Sheldrick, 1978). Au-atom positions determined from sharpened Patterson map; all remaining non-H atoms located on difference Fourier maps. Phenyl rings refined as idealized polygons (C—C = 1·395 Å, C—C—C = 120°) with H atoms in calculated positions with fixed thermal parameters [U(H) = 0·08 Å^2]. Methylene H atoms not refined. All non-C heavy atoms refined anisotropically. Refinement of solvent-molecule occupancy factors did not lead to values significantly different from unity; atomic positional and thermal parameters refined at full occupancy. Refinement based on F with $w^{-1} = [\sigma(F) + 0{\cdot}0028(F^2)]$. Scattering factors, including terms for anomalous dispersion, from *International Tables for X-ray Crystallography* (1974). Convergence to conventional R values of $R = 0{\cdot}039$ and $wR = 0{\cdot}041$ using 189 variable parameters and 2986 reflections with $F_o^2 > 3\sigma(F_o^2)$. For final cycle, max. shift/σ = 0·008. Residual electron density on final difference Fourier map +1·20 and −0·91 e Å^{-3}.

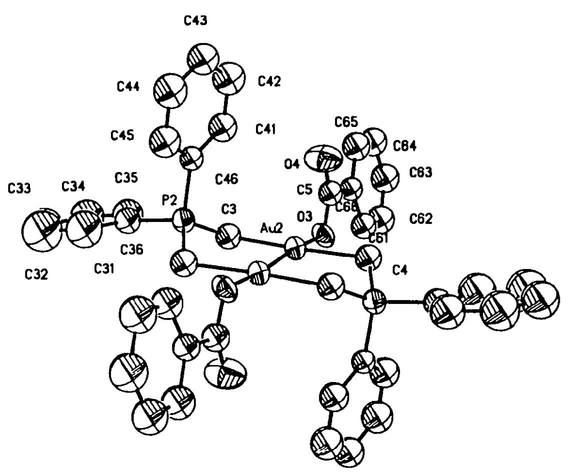

Fig. 1. A perspective view of the $[Au(CH_2)_2P(C_6H_5)_2(C_6H_5CO_2)]_2$ structure. Thermal ellipsoids are drawn at the 50% probability level.

Discussion. The product obtained from the reaction of benzoyl peroxide with the gold(I) ylide dimer consists of discrete dinuclear gold(II) molecules. The contents of the asymmetric unit include two crystallographically independent half-dimers and a single molecule of tetrahydrofuran. Both dimers possess a crystallographically imposed inversion center. The structures of the two independent molecules are essentially the same, therefore a perspective drawing of only one of them is shown here (Fig. 1). The tetrahydrofuran molecule has not been included. Atomic positional and equivalent isotropic thermal parameters for all three molecules are presented in Table 1.* Bond distances and angles are summarized in Table 2.

The overall geometries of the two independent adducts in this structure have configurations similar to those typically observed in other dinuclear gold(II) ylide complexes. These similarities include the chair configuration of the eight-membered ring and the linear arrangement of the *L*–Au–Au–*L* atoms. The Au atoms have square-planar coordination geometries and each forms bonds to a second Au center, two methylene C atoms, and one O atom of a benzoate ligand. The metal–metal bond lengths in the two independent dimers are not identical, differing by 0·026 (2) Å [Au(1)—Au(1′) = 2·587 (1); Au(2)—Au(2′) = 2·560 (2) Å]. Bonds from Au to the methylene C atoms of the bridging ylide ligands for both structures range from 2·077 (21) to 2·107 (22) Å, with bonds to O(1) and O(3) of the carboxylate ligands of 2·139 (14) and 2·11 (15) Å, respectively. The unidentate coordination geometry of the benzoate ligands makes the oxygen atoms in each carboxylate function inequivalent. As a result, C—O bonds to O atoms coordinated to the metal are longer by an average of 0·056 (35) Å compared with those to free carbonyl O atoms. For both independent molecules the O—Au—Au′ atoms are linear to within 5°. The phenyl rings of the benzoate groups are essentially coplanar with the O—C—O atoms of their respective carboxylate groups, with torsion angles ranging from 177 to 179°.

The P atoms in both molecules have tetrahedral geometries and form bonds to both methylene and phenyl C atoms. For both P centers the deviation from ideal geometry is minimal; the average C—P—C angle is 109·7 (10)° with a maximum variation of less than 4°. Bonds to the methylene C atoms in both adducts are not significantly shorter than those to phenyl C atoms, although in other ylides, both free and stabilized, some shortening of the methylene C—P bond has been observed (Bart, 1968, 1969).

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42921 (35 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

The structure of the complex described here constitutes a rare example of a gold complex containing carboxylate ligands and is the first example of a gold(II) complex containing bonds to oxygen. Other examples of gold–carboxylate complexes include two recently reported (triphenylphosphine)gold(I) derivatives containing acetate and benzoate ligands (Jones, 1984, 1985). The length of the metal–metal bond in these and other dinuclear transition-metal complexes containing metal–metal bonds is highly dependent on the nature of the ancillary ligands present and may be rationalized in terms of a structural *trans* effect (Basil, Murray, Fackler, Tocher, Mazany, Trzcinska-Bancroft, Knachel, Dudis, Delord & Marler, 1985). For both the dinuclear gold(II) ylides and a series of isoelectronic platinum(II) complexes the length of the metal–metal bond correlates with a ligand series ordered in terms of their *trans*-directing ability (Alexander, Bryan, Fronczek, Fulty, Rheingold, Roundhill, Stein & Watkins, 1985). The *trans*-directing ability of carboxylate ligands is weak compared with halides, chalcogens and alkyl groups. As a result, the Au—Au bonds in the two independent molecules described here are short and are, in fact, the shortest observed to date in any dinuclear gold(II) ylide complex.

Table 1. *Atomic coordinates* ($\times 10^4$) *and isotropic thermal parameters* ($Å^2 \times 10^3$) *for* $[Au(CH_2)_2P(C_6H_5)_2]_2(C_6H_5CO_2)_2$

	x	*y*	*z*	*U*
Au(1)	4899 (1)	5434 (1)	1103 (1)	43 (1)*
Au(2)	−385 (1)	771 (1)	−601 (1)	43 (1)*
P(1)	3103 (4)	6558 (4)	−1464 (5)	49 (3)*
P(2)	1416 (4)	624 (4)	2264 (5)	48 (3)*
O(1)	4695 (9)	6169 (9)	2910 (11)	50 (7)*
O(2)	5990 (11)	6563 (11)	2993 (15)	86 (10)*
O(3)	−1148 (9)	2071 (9)	−1574 (11)	50 (7)*
O(4)	369 (11)	1749 (11)	−1997 (15)	87 (9)*
C(1)	3805 (15)	6746 (14)	227 (19)	56 (6)
C(2)	5919 (15)	4213 (14)	2219 (19)	55 (6)
C(3)	250 (14)	1443 (14)	1128 (18)	53 (5)
C(4)	−1121 (14)	290 (14)	−2476 (18)	55 (5)
C(5)	−568 (16)	2243 (14)	−2047 (19)	51 (5)
C(6)	5291 (15)	6602 (14)	3382 (19)	54 (5)
C(11)	1700 (10)	7651 (8)	−3664 (12)	64 (6)
C(12)	1156	8482	−4376	76 (7)
C(13)	1196	9311	−3742	73 (7)
C(14)	1780	9309	−2396	57 (5)
C(15)	2324	8478	−1684	58 (6)
C(16)	2284	7649	−2318	48 (5)
C(21)	1645 (10)	6337 (8)	−592 (11)	53 (5)
C(22)	872	6008	−699	67 (6)
C(23)	662	5388	−1723	76 (7)
C(24)	1224	5097	−2641	99 (8)
C(25)	1997	5426	−2534	74 (7)
C(26)	2207	6046	−1510	52 (5)
C(31)	2321 (12)	731 (8)	4985 (17)	107 (9)
C(32)	2560	1203	6187	133 (11)
C(33)	2246	2169	6203	98 (8)
C(34)	1692	2663	5018	103 (9)
C(35)	1453	2192	3817	81 (7)
C(36)	1767	1226	3800	55 (5)
C(41)	2595 (9)	563 (8)	686 (12)	63 (6)
C(42)	3510	189	268	82 (7)
C(43)	4414	−611	870	72 (6)
C(44)	4403	−1037	1889	82 (7)
C(45)	3489	−663	2307	70 (6)
C(46)	2585	137	1705	43 (5)
C(51)	4295 (10)	7312 (9)	5069 (13)	61 (6)
C(52)	4071	7931	6112	88 (8)
C(53)	4634	8497	6648	85 (7)
C(54)	5420	8443	6141	97 (8)
C(55)	5644	7823	5099	86 (7)
C(56)	5082	7257	4563	58 (6)
C(61)	−2307 (9)	3697 (9)	−3003 (12)	61 (6)
C(62)	−2839	4544	−3675	78 (7)
C(63)	−2259	4857	−4130	81 (7)
C(64)	−1148	4324	−3913	74 (7)
C(65)	−616	3477	−3241	71 (6)
C(66)	−1196	3163	−2786	47 (5)
O(7)	3055 (22)	2563 (18)	9562 (25)	179 (10)
C(71)	3746 (26)	3034 (25)	10099 (33)	139 (12)
C(72)	3246 (23)	3743 (21)	11120 (29)	117 (10)
C(73)	2268 (26)	3554 (24)	11100 (33)	141 (12)
C(74)	2072 (31)	2945 (28)	9941 (39)	170 (15)

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

Table 2. *Bond lengths* (Å) *and angles* (°) *for* $[Au(CH_2)_2P(C_6H_5)_2]_2(C_6H_5CO_2)_2$

Au(1)—O(1)	2·144 (12)	Au(1)—C(1)	2·102 (18)
Au(1)—C(2)	2·094 (19)	Au(1)—Au(1′)	2·587 (1)
Au(2)—O(3)	2·117 (13)	Au(2)—C(3)	2·107 (19)
Au(2)—C(4)	2·115 (18)	Au(2)—Au(2′)	2·561 (2)
P(1)—C(1)	1·792 (18)	P(1)—C(16)	1·790 (12)
P(1)—C(26)	1·797 (18)	P(1)—C(2′)	1·765 (21)
P(2)—C(3)	1·777 (16)	P(2)—C(36)	1·794 (16)
P(2)—C(46)	1·798 (15)	P(2)—C(4′)	1·759 (27)
O(1)—C(6)	1·255 (29)	O(2)—C(6)	1·201 (32)
O(3)—C(5)	1·265 (33)	O(4)—C(5)	1·214 (24)
C(2)—P(1′)	1·765 (21)	C(4)—P(2′)	1·759 (27)
C(5)—C(66)	1·517 (24)	C(6)—C(56)	1·533 (24)
O(7)—C(71)	1·415 (55)	O(7)—C(74)	1·458 (56)
C(71)—C(72)	1·560 (46)	C(72)—C(73)	1·565 (60)
C(73)—C(74)	1·471 (52)		
O(1)—Au(1)—C(1)	84·1 (6)	O(1)—Au(1)—C(2)	87·9 (6)
C(1)—Au(1)—C(2)	171·7 (8)	O(1)—Au(1)—Au(1′)	178·8 (3)
C(1)—Au(1)—Au(1′)	95·1 (5)	C(2)—Au(1)—Au(1′)	93·0 (5)
O(3)—Au(2)—C(3)	87·7 (6)	O(3)—Au(2)—C(4)	83·7 (7)
C(3)—Au(2)—C(4)	171·4 (8)	O(3)—Au(2)—Au(2′)	175·2 (4)
C(3)—Au(2)—Au(2′)	93·0 (6)	C(4)—Au(2)—Au(2′)	95·6 (6)
C(1)—P(1)—C(16)	110·5 (8)	C(1)—P(1)—C(26)	109·4 (10)
C(1)—P(1)—C(2′)	108·3 (9)	C(3)—P(2)—C(36)	108·5 (8)
C(3)—P(2)—C(46)	112·3 (9)	C(3)—P(2)—C(4′)	109·0 (10)
Au(1)—O(1)—C(6)	112·4 (14)	Au(2)—O(3)—C(5)	115·6 (10)
Au(1)—C(1)—P(1)	108·5 (10)	Au(1)—C(2)—P(1′)	110·0 (12)
Au(2)—C(3)—P(2)	111·5 (10)	Au(2)—C(4)—P(2′)	109·6 (10)
O(3)—C(5)—O(4)	127·2 (21)	O(3)—C(5)—C(66)	112·0 (15)
O(4)—C(5)—C(66)	120·8 (24)	O(1)—C(6)—O(2)	126·7 (19)
O(1)—C(6)—C(56)	115·8 (20)	O(2)—C(6)—C(56)	117·5 (20)
P(1)—C(16)—C(11)	119·4 (4)	P(1)—C(16)—C(15)	120·2 (4)
P(1)—C(26)—C(21)	119·7 (4)	P(1)—C(26)—C(25)	120·0 (4)
P(2)—C(36)—C(31)	119·9 (5)	P(2)—C(36)—C(35)	120·1 (5)
P(2)—C(46)—C(41)	120·3 (4)	P(2)—C(46)—C(45)	119·7 (4)
C(6)—C(56)—C(51)	119·6 (12)	C(6)—C(56)—C(55)	120·3 (12)
C(5)—C(66)—C(61)	122·6 (12)	C(5)—C(66)—C(65)	117·4 (12)
C(71)—O(7)—C(74)	115·7 (28)	O(7)—C(71)—C(72)	103·8 (32)
C(71)—C(72)—C(73)	105·7 (28)	C(72)—C(73)—C(74)	106·7 (34)
O(7)—C(74)—C(73)	105·2 (35)		

These studies are supported by the National Science Foundation, grant CHE-8408414, the donors of the Petroleum Research Foundation as administered by the American Chemical Society and the Welch Foundation. Preparation of the starting material used in this work by H. H. Murray is gratefully acknowledged. LCP is supported by a Welch Fellowship.

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Acta Cryst. (1986). C**42**, 1131–1133

Structure of a Seven-Coordinated Complex of Nickel, Diaqua[2,6-diacetylpyridine bis(2-hydroxybenzoylhydrazone)]nickel(II) Nitrate Sesquihydrate*

BY CORRADO PELIZZI, GIANCARLO PELIZZI, SEBASTIANO PORRETTA AND FRANCESCA VITALI

Istituto di Chimica Generale, Università di Parma, Centro di Studio per la Strutturistica Diffrattometrica del CNR, Via M.D'Azeglio 85, 43100 *Parma, Italy*

(*Received* 8 *July* 1985; *accepted* 28 *April* 1986)

Abstract. $[Ni(H_2O)_2(C_{23}H_{21}N_5O_4)](NO_3)_2.1\cdot5H_2O$, $M_r = 677\cdot22$, orthorhombic, *Pbca*, $a = 17\cdot297$ (6), $b = 13\cdot614$ (5), $c = 24\cdot266$ (11) Å, $V = 5714$ (4) Å^3, $Z = 8$, $D_x = 1\cdot574$ g cm^{-3}, D_m not measured, λ(Mo $K\alpha$) = 0·71707 Å, $\mu = 7\cdot57$ cm^{-1}, $F(000) = 2808$, $T = 295$ K, final $R = 0\cdot0578$ for 2277 unique observed reflections. The structure consists of pentagonal-bipyramidal $[Ni(H_2daps)(OH_2)_2]^{2+}$ cations [H_2daps = 2,6-diacetylpyridine bis(2-hydroxybenzoylhydrazone)], NO_3^- anions and uncoordinated H_2O molecules held together by a three-dimensional network of hydrogen bonds. The H_2daps molecule functions as an N_3O_2 quinquedentate ligand defining the equatorial plane of the bipyramid, the apices of which are occupied by two H_2O molecules.

Introduction. This work is part of a programme of research into the coordinating properties of 2,6-diacetylpyridine bis(acylhydrazones). These molecules form an interesting class of compounds because of their ability to form stable metal complexes, their versatility as chelating agents, their flexibility in assuming different conformations, and their tendency to act as approximately planar quinquedentate ligands, so favouring seven-coordinate pentagonal-bipyramidal structures (Pelizzi, Pelizzi & Predieri, 1984). As an extension, we report here the crystal and molecular structure of the seven-coordinated $[Ni(H_2daps)(OH_2)_2](NO_3)_2.\frac{3}{2}H_2O$ complex, which was determined in order to provide structural data for comparison with previous X-ray work on the ligand behaviour of hydrazones towards different transition- or non-transition-metal ions. This study also provides the opportunity to supply some information about seven coordination for the Ni^{2+} ion.

Experimental. Brown-green crystals prepared by reacting equimolar amounts of 2,6-diacetylpyridine bis(2-hydroxybenzoylhydrazone) and $Ni(NO_3)_2.6H_2O$ in ethanol. Parallelepipedal crystal, dimensions 0·33 × 0·49 × 0·52 mm. Siemens AED three-circle diffractometer, General Automation Jumbo 220 computer, room temperature, Nb-filtered Mo $K\alpha$ radiation. Unit-cell dimensions determined from least-squares refinement of θ values of 26 centred high-angle reflections, θ range: 12·8–18·7°. Space group inferred from systematic extinctions ($hk0$ with $h = 2n + 1$, $h0l$ with $l = 2n + 1$, and $0kl$ with $k = 2n + 1$). Intensity data collected by θ–2θ scans to $\theta_{max} = 26°$; range of hkl: h 0 to 21, k 0 to 14, l 0 to 29. A standard reflection every 50 measurements: no intensity change. 5994 reflections collected, 5417 unique and allowed by symmetry, 2283 with $I > 2\sigma(I)$. Data corrected for Lorentz, polarization and background effects. No correction for absorption or extinction. Structure solved by *MULTAN*74

* IUPAC name: diaqua[2'-(1-{6-[1-(salicyloylhydrazono)ethyl]-pyrid-2-yl}ethylidene)salicylohydrazide]nickel(II) nitrate sesquihydrate.

0108-2701/86/091131-03$01.50