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Preparation and Structure of $Au_2(\mu\text{-ylide})_2[S_2C_6H_4]_2$: A Dinuclear Gold(III) Ylide Complex with Cis,cis Geometry

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The preparation and crystal structure analysis of the cis,cis geometric isomer of the 1,2-benzenedithiolate Au^{III} complex $Au_2(\mu\text{-ylide})_2(S_2C_6H_4)_2$ (**1**), where μ-ylide = $(CH_2)_2P(C_6H_5)_2$, have been performed. Complex **1** crystallizes in the monoclinic space group $P2_1/c$ with $a = 7.635$ (1) Å, $b = 19.037$ (3) Å, $c = 13.210$ (2) Å, $\beta = 96.84$ (1), and $Z = 2$.[2] Refinement using 1794 reflections with $F_o^2 > 3\sigma(F_o^2)$ yielded conventional R values of $R = 0.0185$ and $R_w = 0.0182$. The Au^{III} metal centers have the expected square-planar coordination and are related to each other by a crystallographic inversion center. The two phosphine ylide ligands act as bidentate bridging groups, with methylene carbon atoms occupying cis positions on the metal centers. The chelating 1,2-benzenedithiolate ligands complete the coordination sphere of the metal atoms. The cis arrangement of methylene atoms at the metal centers and the chair configuration of the eight-membered $Au_2C_4P_2$ ring combine to separate the two metal atoms by a distance of 4.40 Å.

Introduction

Dinuclear Au^I phosphorus ylide complexes and their oxidative addition reactions with halogens and alkyl halides have been well studied. Stepwise reaction of a halogen with the dinuclear Au^I dimer $Au_2(\mu\text{-ylide})_2$ gives, first, the Au^{II} complex $Au_2(\mu\text{-ylide})_2[X]_2$ and, finally, a Au^{III} complex $Au_2(\mu\text{-ylide})_2[X]_4$ (X = Cl, Br).[1] In addition, Au^{III} "A-frame" compounds can be made by the reaction of dihalomethanes with the Au^I starting material $Au_2(\mu\text{-ylide})_2$.[2] A Au^{II} alkyl halide intermediate precedes the formation of the A-frame Au^{III} product.[3] While the chemistry of the Au^{II} metal–metal-bonded compounds has been explored extensively, their further reaction to form Au^{III} compounds has been only recently addressed.[4-6] One important aspect of this work has been the structural characterization of geometrical isomers associated with different ligand arrangements at the metal center in alkyl trihalide and tetrahalide complexes of the Au^{III} ylide dimers.[7] Figure 1 illustrates the three geometrical isomers that are expected for $Au_2(\mu\text{-ylide})_2X_4$ complexes. To date, only the trans,trans and cis,trans isomers have been described. This paper reports the preparation and structural characterization of $Au_2(\mu\text{-ylide})_2(S_2C_6H_4)_2$ (**1**), the first cis,cis Au^{III} μ-ylide complex, where the nonbridging ligands are 1,2-benzenedithiolate.

Experimental Section

Synthesis. $Au_2(\mu\text{-ylide})_2[O_6CC_6H_5]_2$ (**2**) was prepared by published procedures.[8] 1,2-Benzenedithiol was purchased from Aldrich and used as received. A 0.073-g sample (6.9×10^{-5} mol) of **2** was dissolved in 12 mL of tetrahydrofuran. To this light yellow solution was added 9 μL (7.6×10^{-5} mol) of 1,2-benzenedithiol. The color of the solution immediately became blood red. After 15 min of stirring, the THF solution was removed under vacuum. The product was taken into $CHCl_3$ and crystallized in low yield by layering with heptane. A plausible reaction leading to the observed products follows:

$$\underset{\mathbf{2}}{2[Au_2(\mu\text{-ylide})_2(O_2CC_6H_5)_2]} + 2[1,2\text{-}(HS)_2C_6H_4] \rightarrow \underset{\mathbf{1}}{Au_2(\mu\text{-ylide})_2(S_2C_6H_4)_2} + Au_2(\mu\text{-ylide})_2 + 2[HO_2C_6H_5]$$

X-ray Crystallography. An X-ray crystal structure analysis of compound **1** was performed by using a Nicolet R3m/E diffractometer with graphite-monochromated Mo Kα (λ = 0.71073 Å) radiation. The SHELXTL (version 5.1) collection of crystallographic software was used throughout. All calculations were performed on a Data General Eclipse S140 minicomputer. Table I contains a list of all pertinent data collection and structure solution parameters. A crystal of **1** was mounted on a glass fiber with epoxy. An initial unit cell was determined from 15 machine-centered reflections taken from a rotation photograph. Fifteen high-angle reflections were used to determine the final cell parameters for data collection. Systematic absences suggested the monoclinic space group $P2_1/c$, which was used in structure solution. Corrections for Lorentz and polarization effects were applied, as was an empirical absorption correction based on azimuthal (ψ) scans of 10 reflections (10.7° < θ < 32.8°) with Eulerian angle χ near 90 or 270°. A correction for an observed decay in check-reflection intensities (~6%) was also applied. The heavy metal atom position was found from a sharpened Patterson map. All other non-hydrogen atom positions were found by using difference maps generated after successive cycles of least-squares refinements. Hydrogen atoms were placed at calculated positions with C–H distances of 0.96 Å and thermal parameters set to 1.2 times that of the carbon atom to which they were attached. Refinement was based on F and used a weighting scheme to minimize $\sum w(|F_o| - |F_c|)^2$ with weights taking the form $w^{-1} = (\sigma^2|F_o| + |g|F_o^2)$, where g had the value 0.0001 determined by fitting $(F_o - F_c)^2$ to $(\sigma^2(F) + g(F)^2)/k$ (k = scale factor). Neutral-atom scattering factors, including terms for anomalous dispersion, were taken from ref 9. The refinement of the structure was unexceptional.

Table I. Crystallographic Data for $Au_2(\mu\text{-ylide})_2(S_2C_6H_4)_2$

$C_{40}H_{44}Au_2S_2P_4$	$P2_1/c$
fw 1108.91	$T = 25$ °C
$a = 7.635$ (1) Å	$\lambda = 0.71073$ Å
$b = 19.037$ (3) Å	$d_{calc} = 1.91$ g cm^{-3}
$c = 13.210$ (2) Å	$\mu = 80.06$ cm^{-1}
$\beta = 96.48$ (1)°	transm coeff = 0.720–0.549
$V = 1906.4$ (5) Å^3	$R(F_o) = 0.0185$
$Z = 2$	$R_w(F_o) = 0.0182$

Results and Discussion

Compound **1** was prepared in low yield from the reaction of 1,2-benzenedithiol with the Au^{II} dimer **2**, which contains terminal benzoate ligands. Although the formation of the Au^{III} product is not well understood, the initial product is probably a Au^{II} species, which disproportionates to the observed product and the well-characterized Au^I dimer.[1a] The reaction of **2** with monodentate thiols to give coordinated thiolates and free benzoic acid by ligand exchange has been observed for a variety of thiols.[10] None of these exchange reactions previously have given a Au^{III} product.

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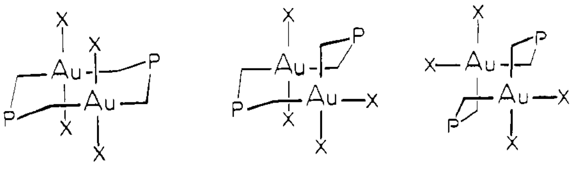

Figure 1. Geometrical isomers of symmetric Au^{III} ylide dimers: trans,trans; cis,trans; cis,cis.

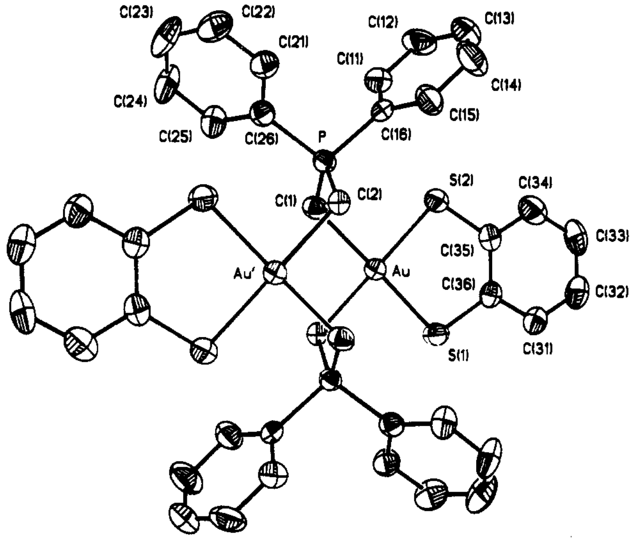

Figure 2. Molecular structure of and atomic labeling scheme for *cis*, *cis*-$Au_2(\mu\text{-ylide})_2[S_2C_6H_4]_2$ (**1**). Thermal ellipsoids are drawn at the 50% probability level.

It seems likely that initially a ligand-exchange reaction takes place in the reaction of **2** with 1,2-benzenedithiol to give a Au^{II} product, possibly a product related to $Au_2(\mu\text{-ylide})_2[S_4]_2$, which contains two dinuclear Au^{II} fragments linked by two four-atom polysulfide chains.[11] In the title compound, two Au^{II} fragments would be linked by two 1,2-dithiolate ligands. Compounds containing dithiolates and dithiolenes are well-known with dinuclear Au^{I} compounds, where these ligands act as the bridge between the two metal centers.[12,13] However, bridging dithiolate Au^{II} compounds disproportionate in solution to a mixture of mononuclear Au^{I} and Au^{III} compounds.[13,14] A similar process may occur here, whereby the initial Au^{II} product disproportionates to a mixture of the Au^{I} dimer and the observed Au^{III} dimer. Although the Au^{III} product is the only crystalline compound isolated upon crystallization, the Au^{I} dimer is observed by ^1H NMR spectroscopy in the bulk reaction mixture.[15] The initial geometry of the Au^{III} product is unclear. The possibility of rearrangement during crystallization cannot be addressed from available data.

Table II. Atomic Coordinates ($\times 10^4$) and Isotropic Thermal Parameters ($Å^2 \times 10^3$)[a] for $Au_2(\mu\text{-ylide})_2(S_2C_6H_4)_2$

atom	*x*	*y*	*z*	U_{iso}[b]
Au	2353 (1)	5475 (1)	9818 (1)	27 (1)
P	4513 (2)	4524 (1)	11694 (1)	29 (1)
S(1)	1476 (2)	6476 (1)	8898 (1)	42 (1)
S(2)	946 (2)	5899 (1)	11158 (1)	37 (1)
C(1)	2953 (7)	4520 (3)	10604 (4)	33 (2)
C(2)	6547 (6)	4924 (3)	11458 (4)	30 (2)
C(11)	1999 (8)	4728 (3)	12998 (4)	44 (2)
C(12)	1275 (8)	5041 (4)	13798 (5)	57 (3)
C(13)	2166 (10)	5573 (4)	14334 (5)	65 (3)
C(14)	3788 (10)	5787 (4)	14108 (5)	69 (3)
C(15)	4503 (8)	5482 (3)	13293 (4)	51 (2)
C(16)	3620 (6)	4957 (3)	12738 (4)	30 (2)
C(21)	5943 (7)	3553 (3)	13101 (4)	43 (2)
C(22)	6341 (8)	2892 (4)	13468 (5)	55 (3)
C(23)	5662 (9)	2315 (4)	12936 (6)	65 (3)
C(24)	4608 (9)	2396 (3)	12032 (5)	60 (3)
C(25)	4234 (7)	3057 (3)	11638 (5)	45 (2)
C(26)	4923 (6)	3645 (3)	12166 (4)	33 (2)
C(31)	−222 (7)	7642 (3)	9465 (4)	43 (2)
C(32)	−1123 (8)	8046 (3)	10095 (5)	52 (3)
C(33)	−1475 (8)	7783 (4)	11021 (5)	60 (3)
C(34)	−879 (8)	7116 (4)	11321 (5)	55 (3)
C(35)	89 (6)	6709 (3)	10706 (4)	34 (2)
C(36)	379 (6)	6971 (3)	9750 (4)	34 (2)

[a] Estimated standard deviations in the least significant digits are given in parentheses. [b] The equivalent isotropic *U* is defined as one-third of the trace of $\mathbf{U}_{ij}$ tensor.

Table III. Bond Lengths (Å) and Angles (deg)[a] for $Au_2(\mu\text{-ylide})_2(S_2C_6H_4)_2$

Au–S(1)	2.317 (2)	Au–S(2)	2.322 (1)
Au–C(1)	2.116 (5)	Au–C(2a)	2.111 (5)
P–C(1)	1.755 (5)	P–C(2)	1.787 (5)
P–C(16)	1.808 (5)	P–C(26)	1.802 (6)
S(1)–C(36)	1.755 (6)	S(2)–C(35)	1.752 (5)
C(2)–Au′	2.111 (5)	C(11)–C(12)	1.384 (9)
C(11)–C(16)	1.392 (8)	C(12)–C(13)	1.369 (10)
C(13)–C(14)	1.368 (11)	C(14)–C(15)	1.391 (9)
C(15)–C(16)	1.369 (8)	C(21)–C(22)	1.370 (9)
C(21)–C(26)	1.389 (7)	C(22)–C(23)	1.371 (10)
C(23)–C(24)	1.366 (10)	C(24)–C(25)	1.378 (9)
C(25)–C(26)	1.388 (8)	C(31)–C(32)	1.376 (9)
C(31)–C(36)	1.395 (8)	C(32)–C(33)	1.378 (10)
C(33)–C(34)	1.390 (10)	C(34)–C(35)	1.396 (9)
C(35)–C(36)	1.399 (8)		

S(1)–Au–S(2)	89.0 (1)	S(1)–Au–C(1)	175.2 (1)
S(2)–Au–C(1)	91.0 (2)	S(1)–Au–C(2a)	89.7 (1)
S(2)–Au–C(2a)	175.9 (1)	C(1)–Au–C(2a)	90.0 (2)
C(1)–P–C(2)	112.1 (2)	C(1)–P–C(16)	110.4 (2)
C(2)–P–C(16)	110.6 (2)	C(1)–P–C(26)	110.6 (2)
C(2)–P–C(26)	109.7 (2)	C(16)–P–C(26)	103.0 (2)
Au–S(1)–C(36)	103.6 (2)	Au–S(2)–C(35)	103.5 (2)
Au–C(1)–P	119.0 (3)	P–C(2)–Au′	114.9 (3)
C(12)–C(11)–C(16)	120.0 (5)	C(11)–C(12)–C(13)	119.6 (6)
C(12)–C(13)–C(14)	121.0 (7)	C(13)–C(14)–C(15)	119.5 (6)
C(14)–C(15)–C(16)	120.5 (6)	P–C(16)–C(11)	118.2 (4)
P–C(16)–C(15)	122.4 (4)	C(11)–C(16)–C(15)	119.4 (5)
C(22)–C(21)–C(26)	120.4 (5)	C(21)–C(22)–C(23)	120.0 (6)
C(22)–C(23)–C(24)	120.3 (6)	C(23)–C(24)–C(25)	120.4 (6)
C(24)–C(25)–C(26)	119.8 (5)	P–C(26)–C(21)	118.7 (4)
P–C(26)–C(25)	122.4 (4)	C(21)–C(26)–C(25)	119.0 (5)
C(32)–C(31)–C(36)	121.4 (5)	C(31)–C(32)–C(33)	119.9 (6)
C(32)–C(33)–C(34)	119.6 (6)	C(33)–C(34)–C(35)	121.3 (6)
S(2)–C(35)–C(34)	119.5 (4)	S(2)–C(35)–C(36)	121.9 (4)
C(34)–C(35)–C(36)	118.6 (5)	S(1)–C(36)–C(31)	119.0 (4)
S(1)–C(36)–C(35)	121.8 (4)	C(31)–C(36)–C(35)	119.2 (5)

[a] Estimated standard deviations in the least significant digits are given in parentheses.

A thermal ellipsoid drawing of **1** is presented in Figure 2. The atomic coordinates and isotropic thermal parameters and the selected bond distances and angles are given in Tables II and III, respectively. In compound **1** the two metal centers of the dimer are related by an inversion center. The eight-membered ring consisting of two Au atoms, two P atoms, and four C atoms, which holds the metal centers together, crystallizes in a pseudochair configuration. The coordination geometry each Au atom is square planar. The four cis angles at Au (C(1)–Au–S(2), S(2)–Au–S(1), S(1)–Au–C(2a), C(1)–Au–C(2a)) are nearly ideal, ranging from 91.0 (2) to 89.0 (1)°. The trans angles (C(1)–Au–S(1) and S(2)–Au–C(2a)) average 175.6°. The Au–C distances of 2.116 (5) and 2.111 (5) Å are well within the range of distances found

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(15) The ^1H NMR spectrum of the bulk unrecrystallized product shows a doublet (1.35 ppm J_{P-H} = 12.1 Hz) corresponding to $Au_2(\mu\text{-ylide})_2$ and a multiplet (centered at 1.24 ppm, broad). In a similar reaction using 1,3-propanedithiol, the only crystalline product isolated was $Au_2(\mu\text{-ylide})_2$.

in other Au^{III} ylide complexes (2.04–2.16 Å).[7] The P–C(methylene) and P–C(phenyl) distances are also within expected ranges, with average lengths of 1.771 (5) and 1.805 (6) Å, respectively.

The geometry of **1** is composed of essentially planar units involving a 1,2-benzenedithiolate ligand, a gold atom, and two methylene carbon atoms. The ylide ligands and the planar $Au(S_2C_6H_4)$ units form a stair step molecule with a geometry that gives excellent crystal packing. The pseudochair conformation of the central eight-membered ring allows this stair step arrangement and as a result separates the two metal atoms to a distance of 4.40 Å. Conversely, a boat conformation around the eight-membered ring with the same cis,cis geometry would place the square-planar metal atoms on top of each other and give a metal–metal separation of ~3.1 Å, a distance commonly seen in trans,trans geometries of Au^{III} ylide dimers.

The 1,2-dithiolate ligand in complex **1** chelates the gold atoms. The preference for a chelating coordination by 1,2-dithiolate ligands is well-known, especially[16] for Au^{III}. The achievement of such a coordination arrangement probably contributes significantly to the formation of the observed cis,cis geometry. The Au–S bond distances of 2.317 (2) and 2.322 (1) Å are shorter than Au–S distances observed in a series of Au^{II} ylide dimers,[10] where the distances average ~2.40 Å. The observed distances are similar, however, to Au–S distances observed in monomeric Au^{III} compounds containing thiolates.[16]

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Supplementary Material Available: Listings of crystal data, thermal parameters, and hydrogen coordinates (4 pages); a table of calculated and observed structure factors (13 pages). Ordering information is given on any current masthead page.

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Contribution from the Department of Chemistry and Laboratory for Molecular Structure and Bonding, Texas A&M University, College Station, Texas 77843

Gold Thiolate Complexes with Short Intermolecular Au–Au Distances from Reactions of Organic Disulfides with Gold(I) Complexes. Syntheses and Crystal Structures of $[Au^{I}_2(PPh_3)_2(\mu\text{-}SCH_2Ph)](NO_3)$ and $Au^{III}_2Cl_4(\mu\text{-}SPh)_2$

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The reaction of $PhCH_2SSCH_2Ph$ or $Na(SCH_2Ph)$ with $Au(PPh_3)NO_3$ in CH_2Cl_2 yields the dinuclear compound $[Au^{I}_2(PPh_3)_2(\mu\text{-}SCH_2Ph)](NO_3)$ (**1**). The reaction of PhSSPh with Au(CO)Cl in CH_2Cl_2 yields the dinuclear compound $Au^{III}_2Cl_4(\mu\text{-}SPh)_2$ (**2**). The crystal structures of **1** and **2** have been determined by single-crystal X-ray diffraction analysis. Compound **1** dimerizes in the solid to form a tetranuclear cluster with intermolecular Au–Au separations of 3.077 (2) and 3.194 (2) Å. Compound **2** also dimerizes through the eclipse of two square-planar Au^{III} units with an Au^{III}–Au^{III} separation of 3.416 (1) Å. **1** crystallizes in a monoclinic space group $P2_1/n$ with $a = 17.743$ (6) Å, $b = 27.03$ (1) Å, $c = 17.926$ (4) Å, $\beta = 95.29$ (2)°, $V = 8560$ (5) Å^3, and $z = 4$. Refinement using 4131 reflections with $F_o^2 \geq 3\sigma(F_o^2)$ gave $R = 0.078$ and $R_w = 0.0881$. **2** crystallizes in a triclinic space group $P1$ with $a = 7.435$ (2) Å, $b = 10.046$ (2) Å, $c = 12.611$ (3) Å, $\alpha = 85.02$ (2)°, $\beta = 88.81$ (2)°, $\gamma = 69.68$ (2)°, $V = 880.0$ (4) Å^3, and $Z = 2$. Refinement using 1670 reflections with $F_o^2 \geq 3\sigma(F_o^2)$ yielded $R = 0.0498$ and $R_w = 0.0658$.

Introduction

Chemists have been fascinated by the potential importance of gold(I) compounds as antiarthritis medicine. Gold(I) compounds with phosphine ligands or sulfur-containing ligands are most commonly used in the treatment of arthritis. However, very little is known about how gold compounds perform their functions in vivo, despite the fact that numerous studies have been carried out in this area.[1] Some studies suggest that the interactions of the Au^{I} center with thiol or disulfide linkages in biomolecules may play an important role. An important paper by Shaw et al.[1e] has appeared very recently. For these reasons we have been interested in the reactions of organic disulfides or polysulfides with gold(I) complexes, since such reactions may not only produce new gold thiolate compounds with the potential use in medicine but also provide information on the possible function of Au^{I} compounds in biological systems. Organic disulfides and polysulfides have been found to undergo oxidative addition reactions[2a,b,d] with ylide gold(I) dimers to form metal–metal-bonded gold(II) species.[2] Reactions of organic disulfides with monomeric gold(I) complexes have been described.[1e,2c] In this paper, we describe the syntheses and crystal structures of two new thiolate gold compounds, $[Au^{I}_2(PPh_3)_2(\mu\text{-}SCH_2Ph)](NO_3)$ (**1**) and $Au^{III}_2Cl_4(\mu\text{-}SPh)_2$ (**2**), obtained from the reactions of $PhCH_2SSCH_2Ph$ and PhSSPh with $Au(PPh_3)NO_3$ and Au(CO)Cl, respectively.

Experimental Section

All reactions were carried out under an N_2 atmosphere. All solvents were freshly dried before use. Au(CO)Cl and $Au(PPh_3)NO_3$ were prepared according to the literature.[3] ^1H NMR spectra were taken on a Varian XL-200 spectrometer at 200 MHz. Elemental analyses were done by Desert Analytics Co., Tucson, AZ.

Synthesis of $[Au_2(PPh_3)_2(\mu\text{-}SCH_2Ph)]NO_3$ (1). (a) A 10-mg sample of $Au(PPh_3)NO_3$ (0.019 mmol) was dissolved in 3 mL of CH_2Cl_2, and 5 mg of $PhCH_2SSCH_2Ph$ (0.020 mmol) was added to the solution. After the mixture was stirred for 1 h, the solution was concentrated to 1 mL in vacuo. A 1-mL aliquot of diethyl ether was added. After a few days of standing at 0 °C, colorless crystals of **1** precipitated from the solution.

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