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Crystal Structure of Copper(I) Acetate

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Copper(I) acetate, $Cu(O_2CCH_3)$, forms monoclinic crystals in the space group $P2_1/m$ with $a = 5.221$ (2) Å, $b = 6.259$ (4) Å, $c = 9.928$ (4) Å, and $\beta = 93.63$ (3)° at 22°. For 4 formula units per cell, $\rho_{\text{calcd}} = 2.52$ g cm^{-3} and $1.56 < \rho_{\text{obsd}} < 2.97$ g cm^{-3}. Intensity data were collected by counter methods with Mo Kα radiation. The structure was solved by the usual Patterson and Fourier techniques and refined by full-matrix least-squares methods using 554 independent reflections with $F_o^2 > 3\sigma(F_o^2)$ and anisotropic temperature factors for all atoms except the carbon atoms of the methyl groups to an unweighted R factor of 0.046. The structure consists of infinite planar chains of binuclear dimeric units, $Cu_2(O_2CCH_3)_2$, which are bridged through copper and oxygen atoms to form a polymer. Each copper atom is bonded to three oxygen atoms and another copper atom in a distorted square-planar configuration. The Cu–Cu distance of 2.556 (2) Å is significantly shorter than the Cu–Cu distances found in any of the binuclear units of copper(II) carboxylates.

Introduction

A considerable amount of experimental data has been accumulated for the purpose of understanding the type of bonding that is present in copper(II) acetate.[1] Most of the evidence supports the idea that the quenching of the spin moment of copper(II) occurs *via* a direct interaction between the two copper atoms which are 2.64 Å apart in the dimer. An alternative explanation is that the interaction involves a superexchange mechanism through the bridging carboxylate groups. Additional information about the nature of the bonding in copper(II) acetate may be obtained from a study of copper complexes in which the number as well as the nature of the bridging carboxylate groups have been changed. The availability of one such compound was suggested in a report which described the identification of volatile binuclear copper(I) carboxylates from among the decomposition products of the corresponding copper(II) carboxylates.[2] It was expected, therefore, that the structures of these binuclear copper(I) carboxylates should bear some resemblance to the structures of the parent copper(II) carboxylates. On this basis we decided to carry out an X-ray structural investigation of copper(I) acetate which was one of the simplest copper(I) carboxylates in a series of saturated and unsaturated copper(I) carboxylates that was synthesized in our laboratory. A preliminary account of this structural investigation was reported in an earlier communication,[3] and a more detailed account of this work is presented below.

Experimental Section

Synthesis of Copper(I) Acetate. Copper(I) acetate is sensitive to air and moisture and, therefore, all preparative work was carried out in a glass vacuum system in which the pressure was reduced to less than 10^{-2} Torr. Anhydrous copper(II) acetate was reduced with copper metal in pyridine or dry acetonitrile solution until the original blue color of the solution had changed to a pale yellow. Slow evaporation of the solvent from the solution of copper(I) acetate usually resulted in a small amount of disproportionation into copper(II) acetate and copper metal. Crystals of copper(I) acetate obtained by this method were contaminated with trace amounts of impurities and were unsuitable for a structural study. Single crystals of copper(I) acetate that could be used for an X-ray structure determination were obtained as follows. A solution of copper(I) acetate in dry acetonitrile was prepared as described above, filtered through a sintered-glass frit in the vacuum line, and allowed to stand in an evacuated bulb at room temperature. An empty bulb in the same vacuum line was maintained at about 3° below room temperature for approximately 30 hr. During this time the solvent evaporated slowly from the solution of copper(I) acetate and condensed in the empty bulb, and platelike crystals of copper(I) acetate were formed. The crystals were separated by filtration and any excess solvent was removed under vacuum. The crystals were stored under dry nitrogen. Several small crystals were coated with a thin polyacrylate film which made the crystal impervious to air and moisture. No detectable color change occurred when these crystals were exposed to air for a period of several weeks.

Crystal Data for Copper(I) Acetate, $Cu(O_2CCH_3)$. One of the crystals was used for the determination of preliminary cell constants and the space group with Weissenberg and oscillation photographs using Cu Kα radiation. On the basis of the systematic absences ($0k0$, $k = 2n + 1$), it was deduced that copper(I) acetate, $Cu(O_2CCH_3)$, had crystallized in one of the monoclinic space groups, $P2_1/m$ or $P2_1$. The calculated density, 2.52 g cm^{-3}, for 4 formula units per cell agreed with the approximate density that was determined for the compound. The accuracy of the density determination was limited by the reactivity of the compound with many liquids. The upper and lower limits of the density that were determined in 1,2-dibromoethane and 1,1,2,2-tetrabromoethane were $1.56 < \rho_{\text{obsd}} < 2.97$ g cm^{-3}.

Data Collection and Structure Analysis. The oscillation and Weissenberg photographs indicated that the crystals tended to have a high degree of mosaicity. The mosaicity of the best crystal was checked with a Picker FACS-1 automated diffractometer equipped with a graphite monochromator, set for Mo Kα_1 radiation (λ 0.70926 Å), by recording the diffraction intensity while scanning through a dozen reflections in the ω mode. Although some splitting was observed in the selected crystal (0.16 × 0.18 × 0.56 mm), the peak width at half-height was less than 0.2°. Nine strong reflections were centered manually and used to calculate cell constants with two cycles of least-squares refinement. These refined cell constants were used to determine the crystal orientation matrix which was not altered throughout the data collection. Intensity data were obtained by integration of the scintillation count while scanning through each reflection in the 2θ mode at 2°/ min through a 2° range. Backgrounds were counted at each end of the scan for 10 sec, with the crystal and counter stationary, and no serious asymmetry was observed. In the range $4.2 < 2\theta < 52°$, intensity data from 782 reflections were collected over a period of 30 hr. Two reflections were monitored every 20 reflections for a total of 58 measurements. The intensities of these monitored reflections varied within a range of about 10% of the nominal intensity during the course of the data collection, but no trends in these variations were found and no attempt was made to correct the data for this variation. After the data collection, nine high-angle reflections were used to obtain new cell constants by least-squares refinement, for use in the solution of the structure. At 22°, $a = 5.221$ (2) Å, $b = 6.259$ (4) Å, $c = 9.928$ (4) Å, and $\beta = 93.63$ (3)°.

The systematic absences were removed from the data set and background correction was applied by use of the equation $F_o^2 = \text{CT} - 3(B_1 + B_2)$ and $\sigma(F_o^2) = [\text{CT} + 9(B_1 + B_2) + (0.04F_o^2)^2]^{1/2}$ where CT represents the total integrated count, B_1 and B_2 are the background counts and $\sigma(F_o^2)$ is the standard deviation of the observed intensity. Lorentz and polarization corrections were applied to all the data. Any data less than $3\sigma(F_o^2)$ were considered to be unobserved and were omitted from all further calculations. The 554 reflections greater than $3\sigma(F_o^2)$ were used for a three-

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(1) W. E. Hatfield and R. Whyman, *Transition Metal Chem.*, 5, 47 (1969).

(2) C. Reichert, D. K. C. Fung, D. C. K. Sin, and J. B. Westmore, *Chem. Commun.*, 1095 (1968).

(3) T. Ogura, R. D. Mounts, and Q. Fernando, *J. Amer. Chem. Soc.*, 95, 949 (1973).

dimensional Patterson synthesis which indicated that all atoms were located on planes perpendicular to the *b* axis and that the interplanar spacing was half the length of the *b* axis. Because the planes of atoms in the crystal were 3.13 Å apart, it is reasonable to assume that there is no bonding between atoms in adjacent planes. Hence, the centrosymmetric space group $P2_1/m$ was selected and all atoms were restricted to the mirror planes. The positions of the two copper atoms were determined from the Patterson map. A fourier synthesis based on F_o with the phases determined by the two copper atoms showed the positions of the eight additional atoms in the asymmetric unit. The atomic scattering factors were taken from ref 4. Two cycles of full-matrix least-squares refinement of two positional parameters and one isotropic temperature factor per atom, and the scale factor, gave a conventional *R* value of 0.153.

Anomalous dispersion corrections for the copper atoms were taken from ref 4. The analytical method of Tompa[5] was used to apply absorption corrections to all the data. The linear absorption coefficient for Mo Kα radiation was 72.2 cm^{-1}. The transmission factors varied from a maximum of 0.15 to a minimum of 0.12. No extinction corrections were made. Additional full-matrix least-squares refinement of a scale factor, two positional parameters per atom, four anisotropic temperature factors for the copper and oxygen atoms, and one isotropic temperature factor for the carbon atoms was continued until convergence occurred after four cycles. All shifts in the parameters were zero after the last refinement cycle and the conventional *R* value was 0.046.

Attempts to locate the hydrogen atoms by means of difference Fourier maps were unsuccessful, but the low background electron density indicated that the nonhydrogen atoms were correctly located. Further refinement was carried out with the assignment of anisotropic temperature factors to all the atoms, which involved an increase in the number of variables from 49 to 61. No significant change in the model was produced, although the *R* factor was lowered slightly to 0.044. It was decided, therefore, that the introduction of 12 additional variables did not yield an increase in useful information.

The *R* factor was defined as $\Sigma||F_o|-|F_c||/\Sigma|F_o|$, and the quantity minimized was $\Sigma w(|F_o|-|F_c|)^2$ where $w = F_o^2/\sigma^2(F_o^2)$. The program FORDAP was used for the Fourier calculations and the program NUCLS was used for the structure factor and least-squares calculations.[5]

Space Group Assignment. The solution of the structure of this compound involved the choice between two monoclinic space groups, both of which have a twofold screw axis along *b*. The centrosymmetric space group, $P2_1/m$, has a mirror plane perpendicular to the screw axis, whereas the noncentrosymmetric space group $P2_1$ has only the twofold screw axis. The choice between these two space groups must of necessity be based on the best fit of a chemically reasonable model to the experimental data, provided that there is no other evidence that conflicts with the choice of such a model. The distinguishing feature of the centrosymmetric model is that all the atoms in the bonded unit are required to have their centers on the mirror plane. The acentric model is expected to resemble the centric model very closely, with the exception that all the bonded atoms are not restricted to lie on the mirror plane. The atomic positions, however, would be expected to deviate only slightly from the mirror plane; any large deviations would result in unreasonable bond distances and would not be in agreement with the three-dimensional Patterson synthesis.

The choice between the two space groups was governed by the following considerations: (a) indications of the presence of planar groups in the Patterson synthesis; (b) Wilson statistical tests of the distribution of intensities in the data set as compared with theoretical values for centric and acentric models; (c) a comparison of each of the observed and calculated structure factors; (d) temperature factors that result in ellipsoidal displacements perpendicular to the molecular plane; (e) reasonable values for bond lengths; and (f) refinement of the models in both space groups and a comparison of the respective *R* values.

The Wilson statistical tests confirmed that the distribution of the *E* values conformed most nearly to the centric model. Refinement of the centric model with 49 variables converged rapidly and there were no discrepancies in the list of structure factors. There were no unusually large displacements of the vibrational ellipsoids in a direction perpendicular to the mirror plane, and all the calculated bond distances were normal.

Table I. Atomic Fractional Coordinates, Thermal Parameters, and Their Standard Deviations

Atom	10^4x	10^4y	10^4z	B, Å^2
Cu(1)	−404 (2)	2500	−556 (1)	*a*
Cu(2)	4016 (2)	2500	709 (1)	*a*
O(1)	−1607 (12)	2500	1246 (6)	*a*
O(2)	2251 (12)	2500	2312 (6)	*a*
O(3)	1425 (12)	2500	−2170 (6)	*a*
O(4)	5229 (13)	2500	−1077 (6)	*a*
C(1)	−157 (18)	2500	2300 (8)	2.16 (0.16)
C(2)	−1356 (20)	2500	3656 (9)	2.85 (0.19)
C(3)	3800 (19)	2500	−2144 (9)	2.61 (0.18)
C(4)	−4836 (20)	2500	−3474 (9)	3.07 (0.19)

Atom	$10^4\beta_{11}$ [b]	$10^4\beta_{22}$	$10^4\beta_{33}$	$10^4\cdot\beta_{12}$	$10^4\beta_{13}$	$10^4\cdot\beta_{23}$
Cu(1)	167 (5)	349 (6)	63 (1)	0	19 (2)	0
Cu(2)	166 (5)	409 (6)	67 (2)	0	21 (2)	0
O(1)	152 (24)	298 (25)	53 (6)	0	18 (10)	0
O(2)	157 (26)	325 (26)	88 (7)	0	18 (12)	0
O(3)	130 (24)	362 (27)	75 (7)	0	7 (11)	0
O(4)	145 (26)	475 (34)	67 (7)	0	3 (11)	0

[a] Atom refined anisotropically. [b] Anisotropic thermal parameters are in the form $\exp[-(h^2\beta_{11} + k^2\beta_{22} + l^2\beta_{33} + 2hk\beta_{12} + 2hl\beta_{13} + 2kl\beta_{23})]$.

A similar refinement of the acentric model was carried out with 68 variables, one scale factor, two positional parameters for one copper atom to fix the origin, three positional parameters for the remaining nine atoms, four anisotropic temperature factors for the first copper atom, six anisotropic temperature factors for the second copper atom and the four oxygen atoms, and one isotropic temperature factor for the four carbon atoms. The *y*-positional parameters as well as some of the temperature factors oscillated and did not converge when this model was refined, although the *R* factor was reduced to 0.043.

This data set, therefore, does not support a model in which any of the atoms is significantly displaced from the nominal plane of the molecule, and the structure consists of planar molecular units in the centrosymmetric space group $P2_1/m$. It must be emphasized, however, that the selection of this space group is not a certainty; it merely reflects the best fit of a model to a set of experimental data with a finite reliability.

Description of the Structure

The positional and thermal parameters together with their estimated standard deviations for the atoms in an asymmetric unit are listed in Table I. The bond distances and bond angles and their estimated standard deviations were calculated by the program ORFFE,[5] and the results are given in Table II. Copper(I) acetate has a planar polymeric structure which consists of infinite chains of eight-membered rings. Two of these eight-membered rings, linked together as shown in Figure 1, form a subunit of the polymeric chain. The polymeric chain must lie on mirror planes that are parallel to each other in the *b* direction at intervals of *b*/2. The coordination of the copper atom is, therefore, strictly planar. Copper(I) in this structure is four-coordinate since none of the atoms in adjacent mirror planes is sufficiently close to increase the coordination number of copper(I).

Each copper atom is coordinated to three oxygen atoms and a copper atom. Two of these oxygen atoms are in the eight-membered ring system and the average Cu–O distance is 1.91 Å. The third oxygen atom is in an adjacent eight-membered ring and the Cu–O distance is 2.31 Å. As a consequence of this interaction between a copper atom in one eight-membered ring and an oxygen atom in an adjacent eight-membered ring, the bond angles around the copper atom deviate considerably from 90°. The Cu–Cu distance,

(4) "International Tables for X-Ray Crystallography," Vol. III, Kynoch Press, Birmingham, England, 1962.

(5) Absorption corrections were made by using the general program AGNOST: P. Coppens, L. Lieserowitz, and D. Rabinovich, *Acta Crystallogr.*, 18, 1035 (1965). Other programs used in this work included UAFACS, for data reduction, NUCLS, for least-squares refinement, Zalkin's FORDAP Fourier summation program, Busing and Levy's ORFFE bond distance and bond angle program, and Johnson's ORTEP plotting program.

Table II. Bond Lengths (Å) and Angles (deg) with Estimated Standard Deviations in Parentheses

Atoms	Dist, Å	Dist,[a] Å	Atoms	Dist, Å	Dist,[a] Å
Cu(1)-Cu(2)	2.556 (2)	2.554 (4)	O(1)-C(1)	1.25 (1)	1.27 (3)
Cu(1)-O(1)	1.933 (6)	1.89 (2)	O(4)-C(3)	1.26 (1)	
Cu(1)-O(3)	1.917 (6)		O(3)-C(3)	1.24 (1)	1.26 (3)
Cu(1)-O(4)	2.306 (7)	2.31 (2)	O(2)-C(1)	1.26 (1)	
Cu(2)-O(1)	2.313 (6)		C(1)-C(2)	1.52 (1)	1.47 (3)
Cu(2)-O(2)	1.888 (7)	1.90 (2)	C(3)-C(4)	1.54 (1)	
Cu(2)-O(4)	1.920 (7)				

Atoms	Angle, deg	Angle,[a] deg	Atoms	Angle, deg	Angle,[a] deg
Cu(2)-Cu(1)-O(1)	83.2 (2)	83.0 (3)	Cu(2)-O(1)-C(1)	136.8 (6)	132.0 (6)
Cu(1)-Cu(2)-O(4)	83.5 (2)		Cu(1)-O(4)-C(3)	135.7 (6)	
Cu(2)-Cu(1)-O(3)	85.9 (2)	86.5 (3)	Cu(2)-O(2)-C(1)	122.2 (6)	123.0 (6)
Cu(1)-Cu(2)-O(2)	86.6 (2)		Cu(1)-O(3)-C(3)	122.2 (6)	
O(1)-Cu(1)-O(4)	80.4 (2)	79.8 (6)	O(1)-C(1)-O(2)	124.0 (9)	120 (2)
O(1)-Cu(2)-O(4)	80.4 (2)		O(3)-C(3)-O(4)	123.9 (9)	
O(3)-Cu(1)-O(4)	110.5 (3)	110.7 (6)	O(1)-C(1)-C(2)	118.6 (8)	121 (2)
O(1)-Cu(2)-O(2)	109.5 (2)		O(4)-C(3)-C(4)	116.2 (9)	
Cu(1)-O(1)-Cu(2)	99.3 (2)	100.0 (6)	O(2)-C(1)-C(2)	117.3 (8)	119 (2)
Cu(1)-O(4)-Cu(2)	99.9 (3)		O(3)-C(3)-C(4)	119.9 (8)	
Cu(1)-O(1)-C(1)	124.0 (6)	128.0 (6)			
Cu(2)-O(4)-C(3)	124.4 (7)				

[a] Mean values of the distances and angles determined independently by M. G. B. Drew, D. A. Edwards, and R. Richards, *J. Chem. Soc., Chem. Commun.*, 124 (1973).

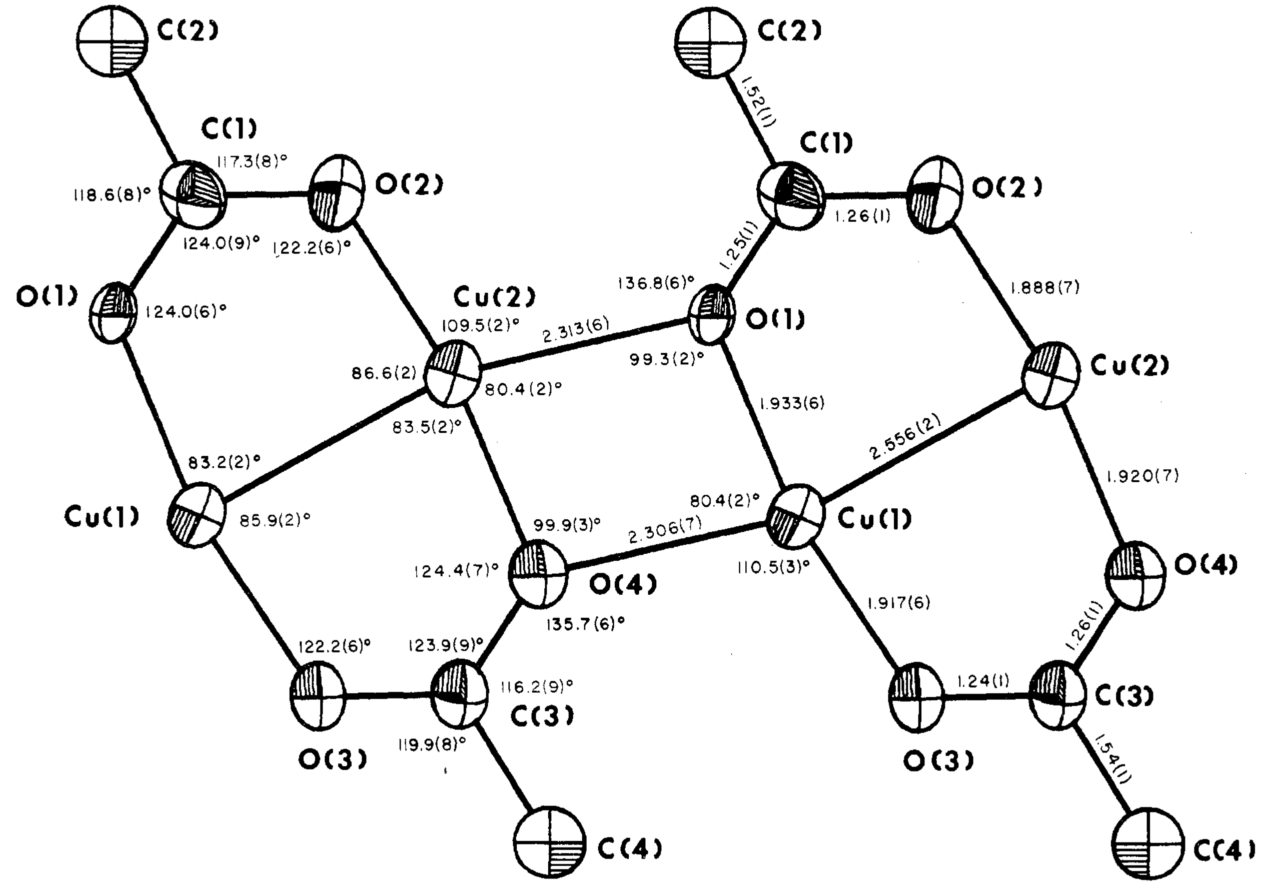

Figure 1. ORTEP plot of the structure of Cu(O_2CCH_3). The distances are in angstroms and the angles are in degrees with the estimated standard deviations in parentheses.

2.556 (2) Å, is significantly shorter than the Cu-Cu distances found in copper(II) carboxylic acid complexes that contain similar eight-membered ring systems: 2.64 Å in copper(II) acetate monohydrate,[6] 2.645 (3) Å in orthorhombic monopyridine copper(II) acetate,[7] 2.630 (3) Å in monoclinic monopyridine copper(II) acetate,[8] and 2.610 (1) Å in copper(II) succinate dihydrate.[9] It is evident, therefore, that the extent of the Cu-Cu interaction is greater in the complex with the d^{10}-electron configuration than in the complex with the d^9-electron configuration. The bond

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[6] J. N. van Niekerk and F. R. L. Schoening, *Acta Crystallogr.*, **6**, 227 (1953).

[7] F. Hanic, D. Stempelova, and K. Hanicova, *Acta Crystallogr.*, **17**, 633 (1964).

[8] G. A. Barclay and C. H. L. Kennard, *J. Chem. Soc.*, 5244 (1961).

[9] B. H. O'Connor and E. N. Maslen, *Acta Crystallogr.*, **20**, 824 (1966).

distances in the bridging acetate groups with binuclear units are essentially the same in the copper(I) and in the copper(II) acetate structures[6,7] although the Cu-O bonds in the copper(II) complexes (1.96-1.98 Å) are significantly longer than the corresponding average Cu-O bond distance (1.91 Å) in copper(I) acetate.

The results of an independent structure determination of copper(I) acetate by Drew and co-workers was published almost simultaneously with our communication.[3] The mean values of the bond distances and bond angles that were reported are shown in Table II and are in essential agreement with our values.

Acknowledgment. The authors are grateful to M. G. B. Drew, D. A. Edwards, and R. Richards for permission to quote results of their structure determination.[10]

Registry No. $Cu(O_2CCH_3)$, 49830-22-0.

Supplementary Material Available. A listing of structure factor amplitudes will appear following these pages in the microfilm edition of this volume of the journal. Photocopies of the supplementary material from this paper only or microfiche (105 × 148 mm, 24× reduction, negatives) containing all of the supplementary material for the papers in this issue may be obtained from the Journals Department, American Chemical Society, 1155 16th Street, N.W. Washington, D. C. 20036. Remit check or money order for $3.00 for photocopy or $2.00 for microfiche, referring to code number INORG-74-802.

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(10) M. G. B. Drew, D. A. Edwards, and R. Richards, *J. Chem. Soc., Chem. Commun.*, 124 (1973).

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Crystal and Molecular Structure of Chlorobis(triphenylphosphine)gold(I) Hemibenzenate

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The crystal and molecular structure of chlorobis(triphenylphosphine)gold(I) hemibenzenate, $[(C_6H_5)_3P]_2AuCl\cdot{}^1/_2C_6H_6$, has been solved using X-ray crystallographic data collected on a diffractometer. The colorless, bladed, triclinic crystals crystallize in the space group $P\bar{1}$ with $a = 10.187$ (1) A, $b = 12.997$ (1) A, $c = 16.596$ (1) A, $\alpha = 52.54$ (1)°, $\beta = 90.68$ (1)°, $\gamma = 75.00$ (1)°, and $Z = 2$. The structure was refined, using block diagonal least squares, to a final R factor of 0.064 on 3661 independent reflections. All of the hydrogen atoms were included in the structure determination but their parameters were not refined. The structure consists of discrete units of $[(C_6H_5)_3P]_2AuCl$. This is the first X-ray crystallographic study in which the coordination about the gold atom has been found to be essentially trigonal planar. The Au-Cl distance, 2.500 (4) A, and the average Au-P distance, 2.331 (4) A, are both longer than the corresponding distances reported for other gold(I) complexes. The P-Au-P angle, 132.1 (1)°, is larger than the P-Cu-P angle reported for the isostructural copper compound.

Introduction

Very little is known concerning the stereochemistry of Au(I) complexes. Recently the X-ray crystal structures of cyano(triphenylphosphine)gold(I)[1] and chloro(triphenylphosphine)gold(I)[2] have been reported. Both of these two-coordinated gold compounds are essentially linear. The P-Au-CN angle and the P-Au-Cl angle are 169 (2) and 179.63 (8)°, respectively. With chelating ligands, Au(I) has been found to form four-coordinated complexes which are tetrahedral.[3]

Although X-ray structure studies have demonstrated the ability of Au(I) to show linear and tetrahedral coordination, none has—until this work—shown Au(I) with trigonal-planar coordination. Chlorobis(triphenylphosphine)gold(I) seemed to be a compound which might exhibit a three-coordinate Au(I) atom. To investigate this possibility, the X-ray crystal structure of this compound was done. While the study was in progress, the structure of bromobis(triphenylphosphine)copper(I) hemibenzenate was published.[4] A comparison of the structure of chlorobis(triphenylphosphine)gold(I) hemibenzenate with the structure of bromobis(triphenylphosphine)copper(I) hemibenzenate is of interest since in both compounds the central atom is a coinage metal which is coordinated with two triphenylphosphine ligands and a halogen.

Experimental Section

Chlorobis(triphenylphosphine)gold(I) Hemibenzenate. Attempts to synthesize single crystals of chlorobis(triphenylphosphine)gold(I), in solvents other than benzene, yielded only twinned crystals. It was decided, therefore, to attempt the synthesis of chlorotris(triphenylphosphine)gold(I) which should also demonstrate a possible coordination about the Au(I) atom. The procedure for the preparation of chlorotris(triphenylphosphine)copper(I)[5] was followed. The method yielded *not* the expected tris compound but rather single crystals which analyzed as chlorobis(triphenylphosphine)gold(I) with 0.5 mol of benzene of crystallization per mole of complex.

A suspension of $[(C_6H_5)_3P]AuCl$ (1.9140 g, 0.004 mol) was refluxed with excess triphenylphosphine (8.3002 g, 0.03 mol) in 100 ml of benzene until the solution became clear. The hot solution was filtered. After 3 hr the first set of crystals was filtered, dried, and submitted for elemental analysis. *Anal.* Calcd for $[(C_6H_5)_3P]_2AuCl\cdot{}^1/_2C_6H_6$: C, 58.65; H, 4.18. Found: C, 58.56; H, 4.00. The benzene of crystallization could be removed by heating overnight in an Abderhalden. *Anal.* Calcd for $[(C_6H_5)_3P]_2AuCl$: C, 57.21; H, 3.99. Found: C, 57.18; H, 3.93.

Crystal Data. For $[(C_6H_5)_3P]_2AuCl\cdot{}^1/_2C_6H_6$, mol wt 796.1, the crystals are triclinic with $a = 10.187$ (1) A, $b = 12.997$ (1) A, $c = 16.596$ (1) A, $\alpha = 52.54$ (1)°, $\beta = 90.68$ (1)°, $\gamma = 75.00$ (1)°, $V = 1643.2$ Å^3, ρ_{measd}(pycnometrically in Skellysolve at 32.6°) = 1.59 g/cm^3, $Z = 2$, $\rho_{calcd} = 1.61$ g/cm^3, $F(000) = 786$, and μ(Mo Kα) = 48.1 cm^{-1}. Cell dimensions chosen to conform with those chosen

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(1) P. L. Bellon, M. Manaserro, and M. Sonsoni, *Ric. Sci.*, **39**, 173 (1969).

(2) D. M. Soboroff, Ph.D. Thesis, The University of Iowa, 1973; W. E. Bennett, D. M. Soboroff, and N. C. Baenziger to be submitted for publication.

(3) W. Cochran, F. A. Hart, and F. G. Mann, *J. Chem. Soc.*, 2816 (1957).

(4) P. H. Davis, R. L. Belford, and I. C. Paul, *Inorg. Chem.*, **12**, 213 (1973).

(5) G. Costa, E. Reisenhofer, and L. Stefani, *J. Inorg. Nucl. Chem.*, **27**, 2581 (1965).