09.4-12 THE CRYSTAL STRUCTURE OF TETRA-N-BUTYLAMMONIUM TETRABROMOAURATE III. Jeffrey P. Johnson*, Physics Dept., Northern Ill. U., DeKalb, IL, 60115, USA, H. Brigitte Krause, Northern Ill. U., DeKalb, IL, 60115, USA, and Elisabeth G. Sherry, Chemistry Division, Argonne Nat. Lab., Argonne, IL, 60439, USA.

Tetra-n-butylammonium tetrabromoaurate III, $C_{16}H_{36}AuBr_4N$ or $[Bu_4N]^+[AuBr_4]^-$ crystallizes tetragonally with a=12.135(5)Å and c=8.508(4)Å, $\frac{c}{a}$=0.7011. Two formula units are contained in a unit cell of volume U=1255Å^3. The probable space group is $P\bar{4}$ with atom parameters almost satisfying the higher symmetry of P4/n. The structure was refined in space group P4/n. It consists of discrete planar $[AuBr_4]^-$ ions which lie on fourfold axes and $[Bu_4N]^+$ ions which have $\bar{4}$ symmetry. The $[Bu_4N]^+$ ions are disordered with two distinct orientations for the carbon chains which are approximately mirror images of one another. The Au-Br bond distance is 2.404(1)Å, the N-C distance is 1.56(2)Å, and the C-C distances vary between 1.42Å and 1.56Å. The final R-value for 1450 reflections was 0.092.

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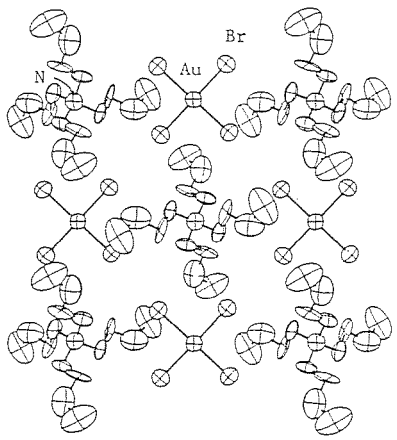

[001] projection of $[Bu_4N]^+[AuBr_4]^-$

09.4-13 THE STRUCTURE OF A FOUR COORDINATE GOLD(I) COMPLEX: $[Au(PPh_3)_3SCN]$. By J. A. Muir, M. M. Muir and S. Arias, Departments of Physics and Chemistry, University of Puerto Rico, Rio Piedras, PR 00931 USA, and C. F. Campana and S. K. Dwight, Nicolet XRD Corp., 10061 Bubb Road, Cupertino, CA 95014 USA.

Crystals of several colorless gold complexes have been obtained by reacting $Au(PPh_3)SCN$ with an excess of (PPh_3) in benzene solution. $[Au(PPh_3)_3SCN]$ is the first of these complexes whose crystal structure has been determined. It crystallizes in the space group $P2_1/n$, with a = 13.806(6), b = 22.110(9), c = 15.774(4) Å, β = 94.68(4)°, Z = 4, D_c = 1.44 and D_m = 1.46(1) g cm^{-3}. The structure was solved using heavy atom methods, and refined with SHELXTL. For 4599 observed structure amplitudes, using data up to 2θ = 45°, the final residuals were R = 0.066 and wR = 0.062. (MoK_α)

The coordination geometry about the gold is four-fold. The gold-phosphine geometry is nearly trigonal planar, with the gold atom only 0.38 Å out of the least-squares plane through the P atoms. Unlike the somewhat similar $[Au(PPh_3)_3]^+[B_9H_{12}S]^-$, however, the SCN^- ligand is coordinated to the gold, so the resulting structure is intermediate between trigonal pyramidal and tetrahedral. The Au-P and Au-S distances are exceptionally long: The Au-P range from 2.384(4) to 2.411(4) Å, while the Au-S distance is 2.787(4) Å. In $[Au(PPh_3)_3]^+[B_9H_{12}S]^-$, by comparison, the Au-P distances are less, ranging from 2.373(14) to 2.389(6), while the Au atom is 0.20 Å out of the least-squares plane through the P atoms.

09.4-14 THE CRYSTAL STRUCTURES OF TETRAPYRIDINE COPPER(I) PERCHLORATE AND TETRAPYRIDINE SILVER(I) PERCHLORATE AT 250 K.

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This study has been carried out as part of a research programme on structural and thermodynamic features of complexes formed in pyridine.

In a preliminary communication Lewin, Michl, Ganis, Lepore and Avitabile (Chem. Comm., 1971, 1400) reported the crystal structure of $Cu(C_5H_5N)_4ClO_4$. We have redetermined that structure and extended the measurements to the silver solvate. The compounds are isostructural, space group $I\bar{4}$ with Z=2 and a=12.471(3), c=6.894(2) and a=12.874(1), c=6.748(4) Å for the Cu and Ag compound, respectively.

Intensity data were collected at 250 K on a CAD-4 diffractometer using Mo-radiation. The refinements converged to R=0.042 (Cu) and R=0.026 (Ag).

The structure is composed of discrete $M(C_5H_5N)_4^+$ and ClO_4^- ions. The coordination geometry is tetrahedral with metal-nitrogen distances 2.047(4) (Cu) and 2.322(3) Å (Ag).

The bond distances in these and other copper(I) and silver(I) compounds will be compared. For a given coordination figure, the difference between the distances Ag-X and Cu-X decreases markedly as X becomes softer.