

79.0° was obtained. (Swarts¹⁷ reports 74°.) Anhydrous ammonia was passed into the CCl₄ solution of the ester to give the amide CFHICONH₂ melting at 93.0° (Swarts reports 92.5°).

Infrared spectra were taken with a Perkin-Elmer Model 12 C spectrophotometer. A Beckman quartz spectro-

(17) F. Swarts, "Organic Fluorine Compounds: A Review," *Mem. Couronnes, Acad. roy. Belg.*, **61**, 94 (1901); *Chem. Zentr.*, **74**, 12 (1903).

tometer, Model D.U., was used for the ultraviolet spectrum.

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[CONTRIBUTION FROM LOS ALAMOS SCIENTIFIC LABORATORY OF THE UNIVERSITY OF CALIFORNIA]

Preparation and Some Properties of H₂Au(CN)₂¹

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The existence of H₂Au(CN)₂ has been suggested in the literature, for example, in the work of Bassett and Corbet² on KAu(CN)₂, but the compound was not isolated. The preparation of a dilute solution (10⁻³ M) of the analogous compound, H₂Cu(CN)₂, by the dissolution of CuCN in aqueous HCN has been reported.³ These workers found that H₂Cu(CN)₂ behaved like a strong acid, having the limiting equivalent conductance, Λ_∞ 389.

Our interest in H₂Au(CN)₂ arose from a discussion⁴ concerning work of L. H. Jones on the polarized infrared spectrum of KAu(CN)₂.⁵

Experimental

The preparation of H₂Au(CN)₂ is achieved readily by passage of a solution containing KAu(CN)₂ through a column containing H⁺-form Dowex-50 resin. In a typical experiment, 1 g. of KAu(CN)₂ was dissolved in 15 ml. of water and passed slowly through a resin column (1.5 cm. i.d. × 13 cm.), previously washed to pH 6 with water. When H₂Au(CN)₂ appeared in the eluate, the pH dropped abruptly.

A purer preparation (containing only negligible AuCN) was obtained when a saturated solution containing equivalent amounts of KAu(CN)₂ and KCN was passed through the resin so that both H₂Au(CN)₂ and HCN were produced. Under these conditions, less decomposition occurred when the solution was evaporated.

Colorless crystals of H₂Au(CN)₂ were deposited when a drop of solution was evaporated rapidly in a stream of dry air at room temperature. A microchemical test for potassium was negative. H₂Au(CN)₂ is readily soluble in water. If the solid is heated to 120° it alters rapidly, forming AuCN; decomposition is slower at 103°.

Preparations were examined with the polarizing microscope, with infrared spectroscopy and by X-ray.

The crystals of H₂Au(CN)₂ examined were small, had poorly developed faces, and gave irregular extinctions in crossed polarized light. Observed extinction angles and interference figures make it probable that H₂Au(CN)₂ crystallizes in the monoclinic system. The crystals are

optically biaxial positive with a large axial optic angle (estimated 2V_Z = 70°) and with very strong dispersion of optic axes $r > v$. The principal refractive indices are $n_x = 1.95$; $n_z = 1.96$; $n_y = 1.98$.

The infrared spectrum of an aqueous solution of H₂Au(CN)₂ has a strong absorption peak at 2147 cm.⁻¹, as does aqueous KAu(CN)₂. A sample of solid H₂Au(CN)₂, dried on polythene film, showed absorption bands at 2146 and 399 cm.⁻¹. Neither the bending frequencies nor a hydrogen stretching frequency for solid H₂Au(CN)₂ were observed at path lengths up to 0.5 mm. Thicker samples were too opaque to infrared radiation to obtain their spectra. Solid KAu(CN)₂ shows absorption peaks at 2141 and 427 cm.⁻¹ (see reference 5). These peaks correspond to the asymmetric C-N stretching and asymmetric C-Au-C stretching vibrations, respectively. This indicates that probably the Au-C force constant is lower in solid H₂Au(CN)₂ than in solid KAu(CN)₂. In solid KAu(CN)₂ the peak at 2141 cm.⁻¹ has a much higher extinction coefficient than the analogous peak of solid H₂Au(CN)₂ at 2146 cm.⁻¹. This indicates that although H₂Au(CN)₂ is highly ionized to H⁺ and Au(CN)₂⁻ in aqueous solution, in the solid it is probably essentially un-ionized, thus greatly decreasing the extinction coefficient.

A sample of H₂Au(CN)₂ gave a powder X-ray diffraction pattern in which no lines characteristic of KAu(CN)₂ or of AuCN were recognized (see Table I). After 20 minutes heating at 120° in the open capillary, the residue gave an X-ray diffraction pattern characteristic of AuCN and no other recognized lines.

TABLE I

PARTIAL POWDER X-RAY DIFFRACTION PATTERN OF H ₂ Au(CN) ₂			
<i>d</i> (Å) ^a	<i>I</i> / <i>I</i> ₁ ^b	<i>d</i> (Å) ^a	<i>I</i> / <i>I</i> ₁ ^b
8.67	100	2.286	5
4.76	<5	2.160	10
4.49	<5	1.921	<5
4.36	<5	1.745	5
4.30	30	1.729	5
3.35	<5	1.618	<5
3.02	45	1.510	5
2.94	<5	1.441	5
2.88	20	1.392	<5
2.70	5		

^a Philips 114.6 mm. diameter powder camera, Straumanis mounting (CuKα) = 1.5418 Å. ^b Relative peak intensities above background from densitometer measurements.

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(1) This work was sponsored by the United States Atomic Energy Commission.

(2) H. Bassett and A. S. Corbet, *J. Chem. Soc.*, **125**, 1660 (1924).

(3) M. G. Vladimirov and I. A. Kakovsky, *Zhurnal Prikladnoi Khimii (Journal of Applied Chemistry)*, **23**, (6) 580 (1950).

(4) Prof. John C. Bailar, Jr., University of Illinois suggested H₂Au(CN)₂ while consulting at LASL.

(5) L. H. Jones, *J. Chem. Phys.*, **22**, 1135 (1954); **21**, 1891 (1953).