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Preparation, Identification, and Reactivity of New Organogold(I) Halide Anion Complexes

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A previous work showed that copper(I) and silver(I) arylacetylides are fruitful sources of interesting organocopper and silver complexes [1]. In contrast to copper(I) and silver(I), gold(I) complexes have a great tendency to exhibit linear geometry [2]. In gold(I) phenylacetylide $[AuC_2Ph]_\eta$, linearity could be achieved by polymerisation through π-bonding between alkyne groups and gold atoms [3]. Treatment of this complex with tertiary phosphines and other neutral ligands gives monomeric acetylides such as Et_3PAuC_2Ph [3].

We report in this letter the reaction of halide ions, Cl^-, Br^-, or I^- with gold phenylacetylide. The normally insoluble gold complex dissolved within five minutes when added (1:1) to a solution of halide ions in acetone or dichloromethane. White crystals of phenylethynyl gold(I) halide anion complexes $[NR_4][XAuC_2Ph]$ (X = Cl or Br, R = Et; X = I, R = Bu^n) were obtained in ca. 90% yield on addition of hexane to the above solutions. Elemental analyses and ^1H n.m.r. spectroscopy indicate a 1:1 adduct. Molecular weight measurements, obtained by using a vaporimetric method, for the chloride and iodide complexes gave values characteristic of 1:1 electrolyte (Table I). Therefore, these complexes are monomeric with linear geometry.

TABLE I. Molecular Weight Data.

Complex	Measured *M* (acetone)	Calculated
$[NEt_4][ClAuC_2Ph]$	290	463.5
$[NBu^n_4][IAuC_2Ph]$	380	667

The i.r. spectra of these complexes contained a medium to a strong sharp absorption band at *ca.* 2120 cm^{-1} assigned for acetylide group. The $\nu(C\equiv C)$ band obtained does not change by altering the type of the halide ion in the complex, and is very close to $\nu(C\equiv C)$ absorption bands in the neutral complexes $LAuC_2Ph$ (L = tertiary phosphines, phosphites, arsines, *etc.*) [3]. This finding, with anionic complexes, confirms the availability of poor back-bonding from gold to π^* orbitals of the alkyne group described previously [3].

These complexes are stable and can be handled in the air in the solid state. After several weeks a slight change in colour of the salt is observed, probably due to slight sensitivity to light; this change, however, does not affect the other physical properties. Addition of water to acetone solution of the organogold(I) chloride or bromide regenerates the yellow gold phenylacetylide. Recently organogold(I) complexes $[PPh_4][RC_2AuC_2R]$ (R = H, Me, Ph) were reported [4] to be stable in contrast to their analogous potassium salts (R = H, Ph) [5]. The organogold(I) complexes $[NR_4][XAuC_6F_5]$ (X = Cl, R = Et; X = Br, R = Bu^n) were prepared by the reaction of $[NR_4][X]$ with $[thtAuC_6F_5]$ (tht = tetrahydrothiophen) in the presence of the corresponding concentrated HX [6, 7]. Nevertheless, the same method [7] failed to give the analogous organogold(I) iodide anion complex $[IAuC_6F_5]^-$. Lately, the complex anion $[IAuC_6F_5]^-$ was prepared by the reaction of QI{(Q = NBu^n_4,$(PPh_3)_2N$,Ph_3BzP} and $thtAuC_6F_5$ in ethanol or dichloromethane [8, 9].

When organogold(I) chloride and bromide complexes were treated with $[NEt_4][I]$, the iodide ions displaced both chloride and bromide ions. The major product was the corresponding linear organogold(I) iodide, as indicated by analytical data and i.r. spectroscopy. A minor product of an organogold(I) iodide complex with different stoichiometry was also obtained. The latter is currently being studied to determine its structure. The reaction of the complex $[ClAuC_2Ph]^-$ with PPh_3 gave PPh_3AuC_2Ph in very high yield. On the other hand, the reaction of the complex $[IAuC_2Ph]^-$ with PPh_3 gave different results. The first crop was PPh_3AuI (34% yield) and the second product was identified as $[(PPh_3)_2N][PhC_2AuC_2Ph]$ (43% yield) [4]. The latter reaction represents a novel method for the preparation of the organogold complex $[PhC_2AuC_2Ph]^-$.

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