Laboratory Scale Direct Synthesis of Pure AuF_3☆

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Pure AuF_3 has been prepared in high yield from the elements in a convenient and safe one-pot laboratory scale reaction. The identity and purity was clearly established by vibrational spectroscopy, mass spectrometry and microanalytical data.

The activation of the strong carbon-fluorine bond by transition metal complexes is a formidable task in organometallic chemistry[1]. In this context, gas phase studies on the intrinsic reactivities of "bare" and ligated transition metal cations[2] are expected to help in the understanding of molecular mechanisms in the transition metal-assisted cleavage of a C−F bond. Quite recently, Schwarz et al.[3,4] reported on the activation of various aliphatic and aromatic C−F bonds by M^+ ions and small MF^+ and MF_2^+ complexes (M = Au, Pr). In these experiments, the ions Au^+, AuF^+ and AuF_2^+ were produced by fast atom bombardment, electron impact ionization or chemical ionization of AuF_3.

Since AuF_3 is commercially not readily available, and this compound may very well have a wider synthetic and theoretical impact, we designed a convenient high-yield laboratory scale synthesis for AuF_3 (using commercially readily-available equipment). Most of the earlier laboratory scale syntheses of AuF_3 refer either to the thermal decomposition of $AuBrF_6$ to give AuF_3 and BrF_3[5,6] (N. B. $AuBrF_6$ is also not commercially available) or to the preparation of AuF_3 from Au powder und F_2 or ClF_3[7,8]. Especially the product obtained from an oxidation with ClF_3 is usually of lower purity[7] and the reaction of gold powder can be violent and may proceed uncontrollably. High-yield preparations for AuF_3 have also been reported in the literature; however, these methods are not one-pot syntheses (usually three steps) and require reaction times of several days[9,10].

In this paper we report on a convenient one-pot laboratory-scale preparation yielding AuF_3 as an analytically pure material in high yield.

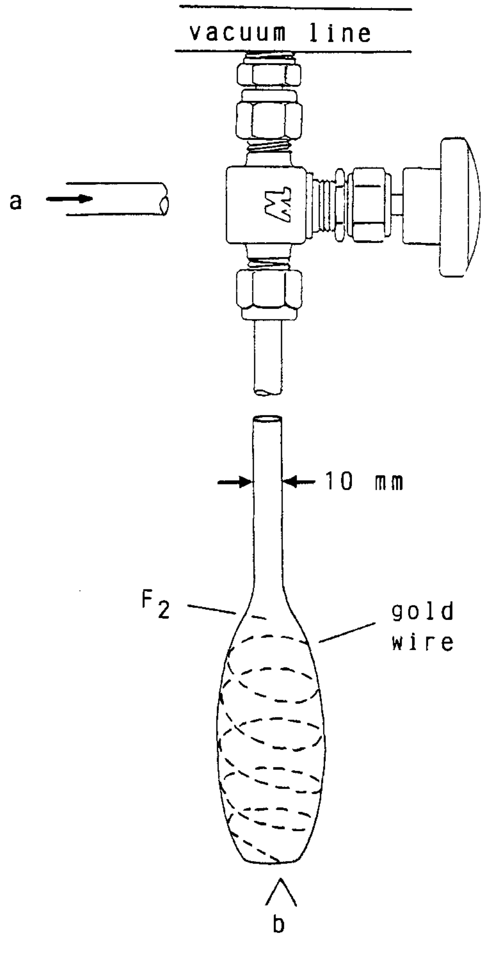
Results

Gold(III) fluoride was prepared by fluorinating elemental gold wire with an excess of elemental fluorine in a thick-walled quartz reactor in a low pressure reaction [eq. (a)] (Figure 1).

$$Au + 3/2\,F_2 \xrightarrow[p_{max} = 15\ \text{bar}]{T_{max} = 1000\,°C,} AuF_3 \qquad \text{(a)}$$

AuF_3 was obtained as an analytically pure compound in high yield in a convenient one-pot reaction. Therefore, in our opinion, the synthesis described above is suitable for the laboratory-scale preparation of this fluoride and represents a safe method for the synthesis of pure gold(III) fluoride. We have repeated the preparation several times with equally good results.

Figure 1. 100-ml thick-walled quartz reaction vessel for the preparation of AuF_3.
a: One of the three nozzles cooling the valve with compressed air.
b: To initiate the reaction the bottom of the quartz vessel is rapidly heated for 10 s to 800 °C.



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Experimental

Caution: Fluorine and chlorine trifluoride are powerful oxidizers; all these materials (including AuF_3) are toxic. Suitable shielding is required, and protective clothing and face masks should be worn all times. Extensive care must be taken to avoid contact between fluorides and oxidizable materials.

All aparatus (including the quartz reaction vessel) were rinsed several times with $CFCl_3$ (Freon-11, Merck), dried and deactivated with ClF_3 (Air Products) at 100°C and 2 bar for 8 h.

Gold(III) Fluoride: In a typical experiment, gold wire (Aldrich, >99.9%, ∅ = 0.25 mm; 0.50 g, 2.54 mmol) was transferred into the prepassivated reactor (100-ml thick-walled quartz reaction vessel closed by a monel needle valve (Whitey), see Fig. 1). Fluorine (Solvay, 0.42 g, 11.0 mmol) was condensed at −196°C on the nickel vacuum line, and the mixture was warmed to room temperature. The bottom of the quartz reactor (Figure 1, b) was rapidly heated for 10 s to 800°C to ignite the gold wire in the F_2 atmosphere. After the reaction was completed, the vessel was recooled to room temperature. During the heating and the exothermic reaction, the needle valve was cooled with compressed air by a system of three nozzles (Fig. 1, a). All material volatile at 20°C was pumped off. The quartz vessel was taken into the dry box (N_2, 99.999%) opened and the light orange-yellow AuF_3 was immediately transferred into flamed-out ampoules of Pyrex which were flame-sealed. Since AuF_3 is extremely sensitive to atmospheric moisture and water, all handling and storage must be done under the utmost anhydrous conditions. – Yield (isolated): 0.42 g (65%). – AuF_3 [253.96] F 22.8 (calcd 22.4%). – MS (EI, 70 eV, 150°C) *m/e*: 508 (Au_2F_6), 254 (AuF_3), 235 (AuF_2), 216 (AuF), 197 (Au). – T_{subl} = 300°C; IR data in agreement with reference 7.

☆ Dedicated to Professor *Robert Schöllhorn* on the occasion of his 60th birthday.

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