Preparation of High Purity Copper Fluoride by Fluorinating Copper Hydroxyfluoride

The problem:
To prepare copper fluoride containing no more than 50 ppm of any contaminating element. Such high purity copper fluoride is needed as a cathode material for high energy density batteries. Copper fluoride which was obtained commercially did not meet these rigid specifications. In general, the concentrations of oxygen and iron were too high.

The solution:
Copper fluoride with the desired purity was prepared by the fluorination of copper hydroxyfluoride. The impurity content of copper fluoride prepared in this way was obtained by spark source mass spectrometry.

How it’s done:
Fluorine gas is passed over the copper hydroxyfluoride while raising the temperature to 525° and then holding the temperature at 525°C for 30 minutes. The copper hydroxyfluoride was contained in a copper boat to minimize contamination. The reaction vessel was fabricated from monel and monel tubing was used to carry the fluorine gas.

There are many advantages to using copper hydroxyfluoride as a starting material. One advantage is that copper hydroxyfluoride can be purchased commercially containing less than 10 ppm of any metallic impurity. Another advantage is that this compound is not appreciably passivated (as in the case of fluorinating copper metal) and the reaction goes to completion. The reason for this can be shown by reactions 1 and 2.

1. \( \text{CuOH}_2 \text{F} \rightarrow \text{CuO} + \text{HF} \)
2. \( \text{CuO} + \text{F}_2 \rightarrow \text{CuF}_2 + \frac{1}{2} \text{O}_2 \)

In reaction 1, finely divided and active copper oxide is formed which reacts with the fluorine as it is formed, thus minimizing the formation of clinkers (as occurs in the case of fluorinating copper sulfide). Furthermore, the liberation of hydrogen fluoride tends to break up clinkers. The decomposition products from reactions 1 and 2 are gaseous and easily removed; consequently, a pure copper fluoride product is left.

Notes:
1. The success with this method suggests that other high purity metallic fluorides could be prepared in a similar manner.
2. Two other ways of preparing copper fluoride were investigated in an attempt to obtain the desired purity. These two techniques were fluorinating copper sulfide and decomposing the double salt, copper ammonium fluoride, in the presence of fluorine. The desired purity was not obtained with either of these techniques.
3. No further documentation is available.
4. Technical questions may be directed to:
   Technology Utilization Officer
   Lewis Research Center
   21000 Brookpark Road
   Cleveland, Ohio 44135
   Reference: B69-10136

Patent status:
Inquiries about obtaining rights for the commercial use of this invention may be made to NASA, Code GP, Washington, D.C. 20546

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