

NOTES AND CORRESPONDENCE

The Preparation of "Pure" Silver Iodide for Nucleation Studies

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1. Introduction

Silver iodide for use as a nucleant in the formation of ice from supercooled water vapor is normally prepared by precipitation from aqueous solution. It has been shown (Corrin and Storm, 1963) that such preparations contain small amounts of foreign ions present during the precipitation. The presence of these ions markedly affect the nature of the water adsorption isotherms (Corrin *et al.* 1964). These authors have also briefly described the preparation of "pure" silver iodide free of hygroscopic contaminants. It has been shown by Steele and Krebs (1965) that the "pure" material is a far less efficient nucleant for ice formation than the standard precipitated silver iodide. For example, with silver iodide smokes obtained with a liquid ammonia technique, the mass nucleation efficiency of the "pure" silver iodide at -10°C was about one-hundredth that of conventional silver iodide.

Since considerable interest has been shown in the

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"pure" preparation, and since samples of this material have been supplied to several investigators, it is of interest to describe the preparation in sufficient detail so that it can be employed by other workers. Use of this "pure" material is essential in the investigation of the nucleation mechanism of silver iodide. The pure material is also the starting point in "doping" experiments in which known amounts of known contaminants are added and the effect on nucleation efficiency determined.

2. Experimental procedure

Silver iodide is prepared by the direct reaction between pure silver powder and iodine in high vacuum. Excess silver is removed by treatment with liquid ammonia. It is necessary to avoid the use of grease during the preparation and to rigidly exclude water vapor. Extensive use is made of glass break seals and collapse seals. It is convenient to discuss the preparation in three phases.

1. The reaction phase. The reaction vessel is shown in Fig. 1. The empty vessel is connected to a vacuum sys-

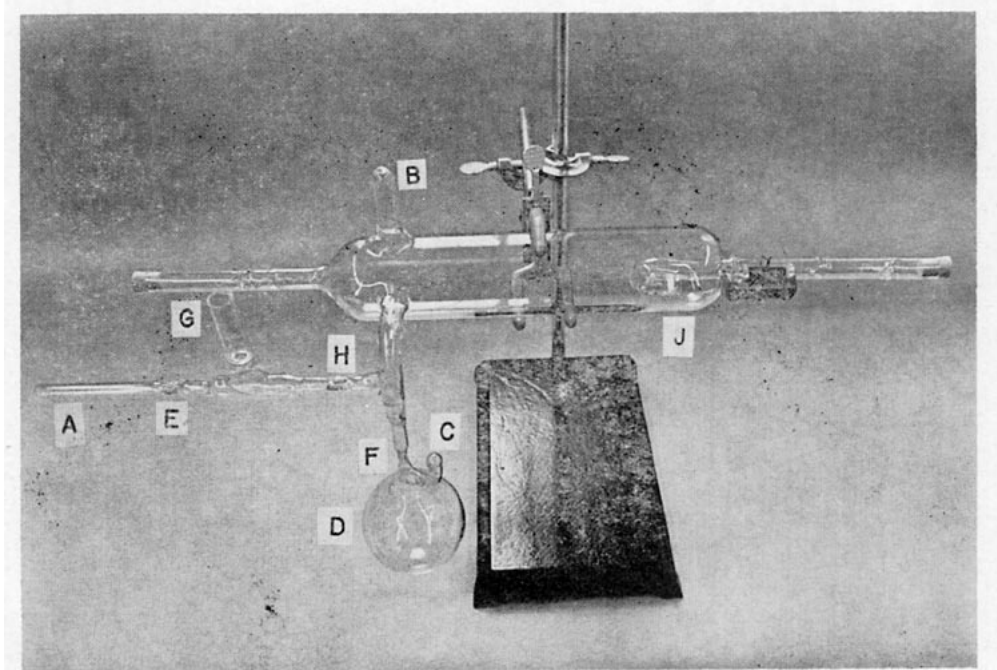


FIG. 1. Reaction vessel for the direct synthesis of silver iodide.

tem (mercury diffusion pump and liquid nitrogen trap) at A and pumped down to 10^{-6} torr as measured on an ionization gage; 24 hr of pumping serves for clean-up. The vessel is removed from the vacuum system at A, the filling tube at B blown open and 121 gm of silver powder (5% excess) introduced; the filling tube is then sealed off. Through the filling tube C, 135 gm of iodine are added to flask D and the tube sealed. The vessel is connected to the vacuum system at A and pumped down for 5 min. The iodine is cooled with acetone-dry ice and the system degassed for at least 48 hr (the pressure drops into the 10^{-7} torr range). The vessel is sealed off at E and placed in a 135C oven for 48 hr. At the end of this time the reaction is complete. The iodine flask is removed at F and the reaction vessel connected to the vacuum system at G. Unreacted iodine is removed by a 48-hr pump-off. It is essential that all excess iodine be removed to prevent the formation of nitrogen triiodide in the liquid ammonia treatment. The vessel is sealed off at H.

2. *Filtration phase.* Treatment of the silver iodide prepared in phase I serves two purposes: 1) removal of the excess silver and 2) recovery of the silver iodide in sufficiently finely divided form to permit gas adsorption measurements. The sintered disk is shown at J in Fig. 1.

A diagram of the filtration system is shown in Fig. 2. The sintered disk sealed into the reaction vessel is of medium porosity. Connections between the various vessels are made with polyethylene tubing; the control valves are of glass-Teflon construction (Fisher and Porter No. 795-500). Safety mercury blow-offs are employed and the system protected from mercury vapor through gold-leaf traps. The system is assembled and leak tested; about 8 gm of thinly sliced sodium is placed in the condensing vessel A and the system pumped down. An acetone-dry ice Dewar is placed around A and approximately 500 ml of liquid ammonia distilled from the tank into A. The sodium serves to dry the ammonia. The level of the mercury in the blow-offs may be used to estimate pressures in the system. With

proper valve manipulation the ammonia is distilled from the condensing vessel into the reaction vessel. A large acetone-dry ice Dewar is employed; it is necessary to open the break-seal at B. When the distillation is complete, the Dewar is removed briefly from the reaction vessel and the contents mixed with the aid of a jet of acetone directed at the bottom of the reaction vessel. The Dewar is replaced and the excess silver allowed to settle. This and the following operations require care and judgement in the adjustment of the Dewar height. When the solution in the reaction vessel is clear, the Dewar is lowered. Filtration occurs under the pressure head caused by vaporization of the ammonia in the reaction vessel. Excessive pressure is avoided by manipulation of the Dewar height. After filtration is completed the Dewar is lowered to the collapse seal point C and the system opened to the pumps through valves 2 and 4. The trap D is cooled in liquid nitrogen.

3. *The pump-off and storage phase.* The receiving vessel is sealed off at E and F after about 48 hr pump-off. The silver iodide is transferred from the receiving vessel into storage bulbs of suitable size in a dry box. The bulbs are connected to a high vacuum system and pumped down in the 10^{-7} torr range for at least 48 hr to remove the last trace of ammonia. The bulbs are then sealed off in vacuo.

3. Discussion

The silver iodide prepared by the above technique possesses specific surface areas on the order of $0.5\text{--}1.1\text{ m}^2\text{ gm}^{-1}$ as determined by krypton adsorption. Its behavior toward water adsorption differs considerably from that of the precipitated material. It is a relatively poor ice nucleant. It is stable toward light and does not darken appreciably after prolonged exposure. This photostability is lacking if the material is exposed to water vapor during the preparation. The sintering properties of the "pure" material differ markedly from those of the precipitated material. It is possible to "dope" the pure material through the addition of various doping substances in liquid ammonia solution.

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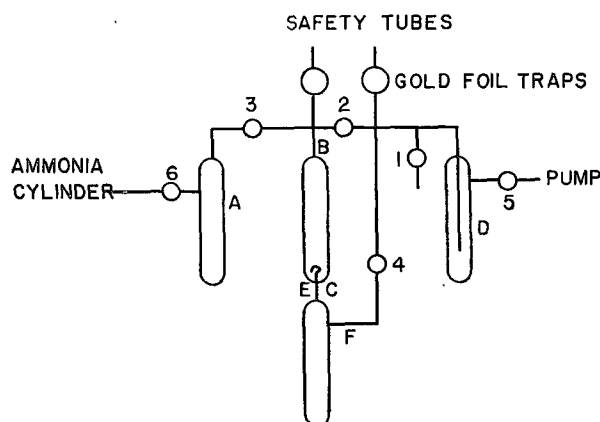


FIG. 2. Filtration system for liquid ammonia treatment of silver iodide.