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Preparation of Ethylaluminum Sesqui-iodide

A lecture demonstration

A great many organometallic compounds are markedly sensitive to air and moisture, and require delicate procedures in their preparation. Some also ignite spontaneously in air, and some react violently with liquid water. These are some of the reasons for the lack of lecture demonstrations in organometallic chemistry, no doubt, and yet the behavior toward air and water are important characteristics which should be emphasized in any classroom presentation of organometallic chemistry.

An opportunity to do a public demonstration of the direct synthesis of some organometallic compound[1] led us to search for a preparation which could be done in 30 minutes without fussy precautions, and which would be dramatic enough to warrant its inclusion in a crowded lecture. It was our belief that no matter how violent the reaction of choice might be, if the quantities were severely limited and if some simple safety rules were observed, it should be possible to demonstrate the principal points. The present demonstration has shown that this belief is justified, and we have used it repeatedly in classrooms.

We adopted the formation of ethylaluminum sesquiiodide, $(C_2H_5)_2AlI + C_2H_5AlI_2$, from aluminum and ethyl iodide, as the best method of demonstrating a direct synthesis.[2,3] Keeping in mind that the demonstration should take only a limited amount of time and should be understood at a glance, we tried to avoid complicated experimental setups. Fortunately, we found that the experiment could be carried out in air instead of using an inert atmosphere, because the vapor of ethyl iodide blankets it. The reaction proceeds according to the equation:

$$2Al + 3C_2H_5I = (C_2H_5)_2AlI + C_2H_5AlI_2$$

A 1-liter Erlenmeyer flask with standard taper joint, containing a 3-in. Teflon-coated magnetic stirring bar is used for the reaction. Two grams (0.074 mol) of household grade aluminum foil previously cut into pieces of about one-fourth inch square, 11 ml (20 g, 0.128 mol) of ethyl iodide, and one or two iodine crystals to start the reaction are placed in the flask. A reflux condenser of the cold-finger type, the outlet of which bears a small Drierite-filled drying tube, is connected to the Erlenmeyer flask and is kept filled with crushed ice throughout the experiment. The mixture is then stirred and heated to vigorous reflux by means of a magnetic stirrer–hot plate combination. When the reaction starts, the Erlenmeyer flask soon fills up with white fumes due to oxygen consumption by the aluminum–alkyl compounds being formed.

After 20 minutes most of the aluminum foil has disappeared. The operator may then put on asbestos gloves and pour the hot mixture into a porcelain evaporating dish. Instantly thick white fumes develop, occasionally turning to the violet color of iodine vapor when the oxygen supply is high enough to allow ignition.

The explosive reaction of aluminum alkyls with water can then be shown by adding 5 ml of water to the evaporating dish from a metal cup affixed to a handle or rod one meter long. This should be done between a pair of Plexiglas safety screens, or, if the screens are not available, by grasping the evaporating dish with crucible tongs and lowering it into a heavy corrugated-board box and then adding the water from a distance. A loud explosion occurs, and some water, aluminum oxide, and iodine are spattered about.

After the demonstration the Erlenmeyer is best cleaned by adding several successive small portions of acetone, then washing the flask with alcohol, and finally with water.

EDITOR'S NOTE: The cover photograph shows Professor Rochow performing this demonstration at his Harvard University lecture table.

WARNING

It is imperative that all safety precautions be followed during this demonstration. Always wear safety goggles, and use asbestos gloves while handling ethylaluminum iodide. ***Never exceed the quantities stated.*** It is best to use two safety screens during the demonstration.

Never add water to the flask after the reaction, or leave the flask where students may be tempted to do so. Stopper the flask with a glass stopper after pouring out the hot contents, and place it out of harm's way before proceeding further. **Be very cautious about cleaning the flask,** preferably carrying out the acetone addition behind a screen. The experiment can be conducted safely according to the directions, but the product is worthy of the greatest respect.

[1] At the Kipping Award lecture, 149th Meeting of the ACS, Detroit, Michigan, April 5, 1965.

[2] Hallwachs, W., and Schafarik, A., *Ann.*, **109**, 207 (1859).

[3] Grignard, V., and Jenkins, R. T., *Compt. Rend.*, **179**, 89 (1924).