NOTE

A New Apparatus for Preparing Aluminum Chloride.—For the preparation of a very reactive aluminum chloride, much superior to the ordinary commercial article for the Friedel and Crafts reaction, the apparatus shown and described below has proved entirely satisfactory in this Laboratory. The product, obtained without difficulty as a uniform, fine powder, can be kept with good mechanical stirring in perfect suspension in the reaction mixture and so gives the best possible results.

The apparatus of Pyrex glass[1] may be made to any scale, but the specifications as given will meet all, ordinary laboratory requirements. The tube in which aluminum is heated has a wall thickness of an eighth of an inch, an internal diameter of 1 inch and a length of 22 inches. At one end it is bent at a right angle and expands into a funnel that has a depth of

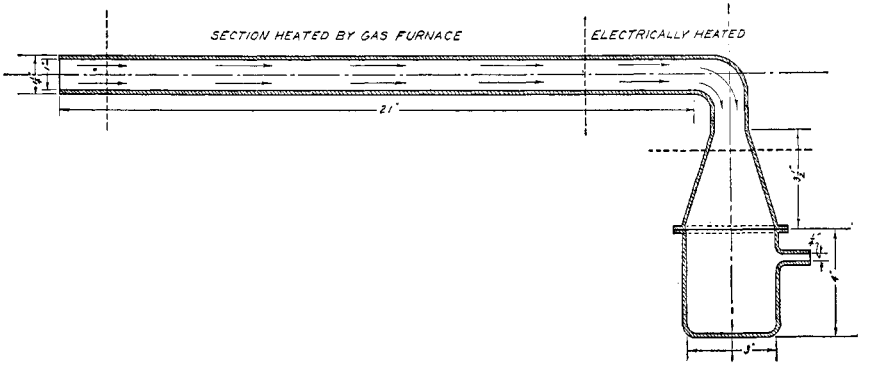

Fig. 1.—An apparatus for making aluminum chloride.

3.5 inches, a diameter of 3 inches at the bottom and is provided with a quarter-inch ground flange. The latter fits a similar flange on the jar where the product is collected. These surfaces, when thinly coated with vacuum grease, make a tight joint from which vapors do not escape while the reaction is going on. The jar has a diameter of 3 inches, a depth of 4 inches and is equipped with a quarter-inch side-outlet 1 inch below the flange. Such an apparatus is capable of turning out 120 g. of aluminum chloride at a single run. It is not expensive and with reasonable care should last for a long time. The original apparatus has been used in this Laboratory for a great many runs and apparently is as good now as in the beginning.

The tube containing the aluminum is heated in a small combustion furnace and the space between the furnace and the top of the funnel is covered with asbestos paper and then wound with 14 feet of 16 gage (B. and S.) nichrome resistance wire. This heating coil is connected with

[1] Supplied by the Corning Glass Company, Corning, New York.

a 110-volt line and the current regulated by a 110 ohm. 7 ampere cage rheostat to produce a temperature of 250° within the tube. The proper setting on the rheostat is determined by a blank trial with a thermometer inside the tube. An asbestos insulation around the electrically heated area is advisable. Since the receiving jar becomes quite hot during the experiment, it is well to set it in a pneumatic trough filled with water to within a half inch of the outlet-tube. To prevent staining of the heating area, aluminum is introduced into the tube in alundum boats of suitable size.

As soon as air is driven from the system by a rapid stream of dry hydrogen chloride, the flow of gas is checked somewhat and heating is begun at the funnel end by turning on the electric current. The resistance of the rheostat is gradually reduced until at the setting established in the blank trial. The inside of the tube reaches the desired temperature in about half an hour. The furnace burners are then lighted and the stream of hydrogen chloride is increased to its former velocity. At the temperature required for the reaction, white fumes appear in the funnel and settle as a powder on the bottom of the jar. A little of the sublimate collecting on the walls obscures the interior and some escapes through the outlet-tube with the hydrogen. The quantity, however, is negligible. It forms a loose deposit in the outlet-tube and can be easily removed by a stiff wire.

The reaction runs best in a rapid stream of hydrogen chloride at a dull red heat just below the fusion point of aluminum. With these conditions once established, the operation requires little attention. Aluminum chloride is obtained in nearly theoretical yield as an easily removable, white powder. It is at once transferred to the reaction flask, or kept in a desiccator in a tightly closed bottle until needed.

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