In a further study of this problem we passed the ether–diazomethane mixture through a furnace heated only to 500°, under which conditions we had previously proved that no methyl groups are produced—and tried the effect of the fragments on metallic mirrors. We found that some metals were attacked and others not. Antimony and tellurium are two typical members of the first group, while zinc, cadmium and lead belong to the second group.

It seems possible to identify the fragments by combining them with tellurium and isolating the compound formed, or by combining them with carbon monoxide, in which case ketene, $CH_2{=}CO$, should be formed if we are dealing with a methylene radical. These experiments are now in progress.

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AN APPARATUS FOR THE SEPARATION OF ISOHYDROGEN (DEUTERIUM) OXIDE BY ELECTROLYSIS

Sir:

The writers have not been able to find in the literature any description of an apparatus suited for the moderate scale production of heavy water (isohydrogen or deuterium oxide) by the method discovered by Washburn, that is, the electrolysis of water. An apparatus used in this Laboratory has proved so convenient for this purpose that some of its details may be of sufficient value to be presented.

Two concentric tubes of nickel, 2.5 and 10.6 cm. in diameter (Fig. 1) are used both as the electrodes and the vessel in which the aqueous solution is contained. The portion IJ, 100 cm. in height, holds 3.5 liters of the alkaline solution, supplied by the Burdette Oxygen Company. About 1 part in 2000 of the hydrogen present in this solution initially is deuterium.

The two tubes are insulated from each other by heavy rubber washers, H, Q and K. The washer K is cemented to the metal on which it rests. The screws which hold the apparatus together are insulated on one side by the Bakelite insulators D. The solution is put in or taken out at R, and a condenser and apparatus for the prevention of the escape of mist are attached at B. Electrical connections are made at A and E. The 2.5-cm. tube P is filled with water for cooling which is admitted at M. With 100 amperes operating current the water which escapes from N has a temperature only 0.3° above that which enters at M. The apparatus is made gas tight, except for the opening into the condenser, by the packing C.

The upper length of 10-cm. tubing, 15 cm. in length, is covered with rubber (F) from the inner tube of an automobile tire, and most of the

wall of the steel tube is cut away. The purpose of the rubber is to avoid danger in any accidental explosion of the gas. The lower tube IJ is supposed to be kept as full of solution as is possible under the conditions of operation. The joint at H is not essential, as the upper part of the outer tube IJ may be cut away instead. One apparatus was built in which the upper tube was made wholly of rubber, and the connection between each D and H was made by an insulating post.

The apparatus was designed to operate at 400 amperes, but this large a current has not been available.

To save expense the outer tube of our apparatus was made of steel, nickel plated on the inside, and this tube is therefore used as the electrode at which the hydrogen is liberated. The use of steel or iron for the inner tube is not recommended unless a very thick and dense nickel coating can be obtained.

Ten units such as that described are used for the preliminary separation. It is obvious that smaller units must be employed as the separation proceeds. The solution from one unit is distributed among the other nine as soon as space in them becomes available. It is planned to operate a second set of units of a different type, with acid solutions, and to use mutual neutralization as an aid to the separation of the water from the electrolyte.

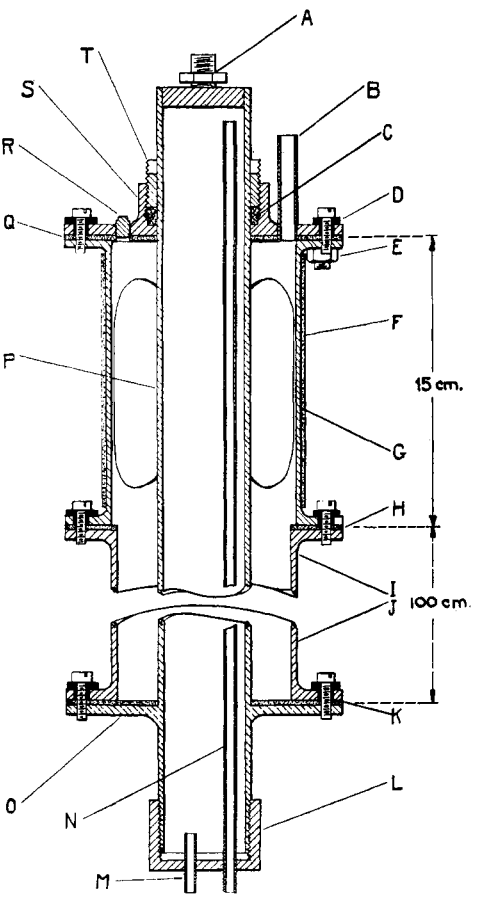

Fig. 1.—Apparatus for the separation of deuterium oxide.

In April, Dr. Washburn stated to one of us (Harkins) that he had tried the use of concentric tubes for the electrolysis, but we have no other knowledge of the apparatus employed by him.

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