Remarks.—Unpurified diphenyl from run 1 had a melting point of 69–69.5°. The temperature of 252° was the lowest at which observable decomposition took place. The lead deposit in run 1 was bright. All other runs exhibited more or less charring. In runs 2 and 6 the bulb was placed in the cold furnace and then rapidly heated. In the other runs the bulb was placed in a hot furnace. The benzene was determined by the difference in weight caused by passing a stream of air through the bulb. These values are probably high, due to the volatility of diphenyl. The undecomposed lead tetraphenyl was recovered by precipitation with acetone, and the *p*-diphenylbenzene and diphenyl separated by solution in ethyl alcohol.

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COMMUNICATIONS TO THE EDITOR

THE FREE METHYLENE RADICAL

Sir:

We have been decomposing diazomethane in a current of ether since some time, in attempts to isolate the methylene radical. Blank runs showed that ether alone, under the conditions of our experiments, does not decompose appreciably under 750°. Using the ether–diazomethane mixture and combining the fragments with metallic mercury [see Rice, Johnston and Evering, THIS JOURNAL, **54**, 3529 (1932)] we obtained no trace of any organic compound of mercury with the furnace below 650°, although a considerable portion of the diazomethane decomposed, and antimony mirrors could be readily removed at furnace temperatures as low as 450°. A run at 700° gave an appreciable yield of an organo-mercury compound which was identified as dimethylmercury by adding iodine to the contents of the liquid air trap after distilling off the undecomposed diazomethane; in this way we obtained pure methylmercuric iodide, CH_3HgI, identified by its mixed melting point.

It seems reasonable to infer from these experiments that, if the CH_2 group has been formed, it does not combine with mercury to form $Hg{=}CH_2$, since the addition of iodine to this compound should give CH_2IHgI. Furthermore, it seems very probable that at temperatures of 700° and higher, the methylene group picks off a hydrogen atom from one of the surrounding ether molecules, thus producing a free methyl group.

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