

NOTES ON THE PREPARATION OF ALKYL IODIDES.—By HAROLD S. KING, PH.D., Department of Chemistry, Dalhousie University, Halifax, N. S.

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ABSTRACT.

In the preparation of alkyl iodides by a modification of Adams' method the use of anhydrous alcohol is not necessary. The addition of 10 per cent. by weight of water to methyl alcohol did not reduce the yield of methyl iodide. The start of the reaction is smoother if part of the alcohol is put in the iodine container and allowed to run in on the phosphorus and the rest of the alcohol heated to 70-75°C.

Since we reported some years ago on "A Modification of the Adams' Method of Preparing Alkyl Iodides"¹, certain changes have been adopted. The notes here given are merely addenda to the above article, to which the reader is referred for details of the procedure and apparatus employed.

It was at first thought necessary to use absolute alcohol in these preparations. It has now been found that the presence of water is not deleterious. In the preparation of methyl iodide, the methyl alcohol was diluted with 10 per cent. of its weight of water. The yield was identical with that of a check experiment in which absolute methyl alcohol was used. The presence of water does not cause an evolution of hydrogen iodide. A tube from the top of the condenser dipped under water. After the experiment this water was found to be neutral to litmus. The methyl iodide, distilled from the reaction flask, was collected under a slush of ice and water. This also was neutral.

In the original procedure the alcohol was put in the reaction flask together with red and yellow phosphorus and the mixture heated to 100°C so that the liquid in refluxing dissolved the iodine on the way back to the reaction flask. At the beginning of the reaction methyl alcohol is the refluxing liquid; after some time methyl iodide takes its place. The temperature then must be lowered, partly because methyl iodide is

1. King, *These Transactions*, 16, Pt. 2, 87-91 (1924).

more volatile and partly because iodine is more soluble in it than in the alcohol. It would be an advantage if the refluxing began with methyl iodide so that no change in the temperature would be necessary. To accomplish this the iodine crystals are covered with part of the methyl alcohol and the alcoholic solution of iodine is run slowly into the reaction flask, which holds the phosphorus and the rest of the alcohol, and heated to between 70° and 75°C. By the time that all the solution has reacted, sufficient methyl iodide has been formed to start the refluxing. This modification is of even greater value in making the higher alkyl iodides, expediting their preparation to a considerable extent.