

$b$ ,  $\epsilon_1$ ,  $v_1$  and  $P_2$  obtained for each compound are shown in Table I along with the observed molecular refractions and the electric moments. The significance of the symbols and the method of calculation are the same as used previously.<sup>3</sup> The probable error in the dipole moments is about  $\pm 0.10 D$ .

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### Preparation of Pure, Dry Iodine

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It was suspected that some of the anomalous results observed in the kinetics of the exchange of iodine atoms between methyl iodide and free iodine are due to contamination by water, with the result that ionic exchange is made possible. To check the presence of water, iodine was recovered from an iodide solution to which radioactive water ( $T_2O$ ) had been added. The iodine was then dried in a conventional manner by sublimation *in vacuo* over  $P_2O_5$  and condensation in a trap cooled with Dry Ice-acetone or liquid air. The radioactivity of samples of iodine recovered in this manner corre-

sponded to a water content of about 0.1%. A modification of the palladous chloride method<sup>1</sup> which is sometimes used for the estimation of iodine was devised, which leads to recovery of pure, dry iodine.

The procedure recommended is as follows: sulfite ion is removed from the required amount of 0.5 *M* NaI solution (containing radioactive iodine as required) by addition of  $BaCl_2$  and removal of  $BaSO_3$  by centrifuging. About 10% excess  $PdCl_2 \cdot 2H_2O$  solution (10 g./liter *N* HCl) is added to a centrifuge tube containing the iodide solution, the tube is heated in a water-bath for 15 minutes, allowed to stand one hour, centrifuged, precipitate is washed free of  $Cl^-$  with water, washed with 95% EtOH and then with dry  $Et_2O$ . The centrifuge tube with the precipitate is then dried for one hour at 110° and sealed into a vacuum system free from mercury and grease.

The  $PdI_2$  is apparently moisture-free at this stage, but can be heated to 250° in a vacuum of  $10^{-6}$  mm. without loss of iodine. The  $PdI_2$  is then heated to 350° (mercury boiling at 1 atm.) and the iodine is recovered in a tube cooled with liquid air. Using this procedure the discrepancy in the recovery of 10-mg. samples of iodine was  $\pm 1\%$ . The iodine can also be recovered somewhat more quickly by heating the  $PdI_2$  to 350° in a stream of hydrogen. Samples of iodine recovered from NaI dissolved in water with a tritium activity of  $8.2 \times 10^8$  disintegrations/minute/gram had residual activities of 800 to 1800 d./min., indicating that the amount of water present had been reduced to the order of  $10^{-6}$  g.

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(1) N. H. Furman, Editor, "Scott's Standard Methods of Chemical Analysis," D. Van Nostrand Co., Inc., New York, N. Y., 1939.