

XXXI.—*The Preparation of Iodic Acid.*

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In a note on the preparation of iodic acid appended to the first part of his "*Nouvelles Recherches*," Stas states that the yield of iodic acid obtainable by oxidising iodine by means of nitric acid has been much exaggerated, and that the quantity of pure iodic acid obtained does not represent a quarter of the iodine employed. If this statement is correct, the question arises, What becomes of the rest of the iodine? Experiments were therefore made to test its truth. Into a round bottomed flask with a very long neck, 100 grams of iodine were placed, and boiled gently with nitric acid (sp. gr. 1.5) repeatedly renewed, the more or less spent acid being removed each morning and replaced with strong acid. In order to oxidise all the iodine this had to be continued for about ten days of eight hours each. By careful regulation of the flame so as to minimise the loss of iodine due to its being carried off with the steam and oxides of nitrogen, 102 grams of iodic acid were obtained, that is, about 74 per cent. of the iodine employed had been obtained as iodic acid.

As this method is very tedious and, even as carried out above, wasteful both as regards iodine and nitric acid, other forms of apparatus were tried with varying degrees of success.

The first apparatus employed was a Soxhlet's fat extraction apparatus, all the joints being glass to glass, the iodine being treated as if it had to be extracted, and the fuming nitric acid placed in the flask. In the first experiment, 20 grams of iodine and 150 c.c. of fuming nitric acid were employed, and 26 grams of iodic acid were obtained (93 per cent.). Next 50 grams of iodine and 200 c.c. of nitric acid were employed and 60 grams of iodic acid obtained (86 per cent.). The action was rapid, the only, but very serious, objection to this form of apparatus being the liability of the narrow siphon tube to become choked by the deposition in it of crystals of iodic acid. When this occurs, the operation must of course be stopped.

By varying the diameter of the tubes, some improvements were effected, but, as much simpler and more efficient forms of apparatus have been devised, it is unnecessary to describe these intermediate forms.

The best form of apparatus is a flask like that recommended for the preparation of pure hydrobromic acid (Trans., 1900, 77, 649), but having the tube *C* sealed to a reflux condenser; through *B* a tube is fitted (by means of a small piece of rubber tubing) by which a current of oxygen may be passed through the boiling acid, the point

of the tube reaching almost to the bottom of the flask. With this apparatus, iodine *in fine powder* boiled with ten to twelve times its weight of nitric acid (sp. gr. 1.5) is completely converted into iodic acid in from twenty minutes to half an hour.

The other and more easily obtainable form of apparatus is a long necked flask such as was employed in the first experiment. In the neck of this flask is inserted a condenser of the well-known form made of quill tubing bent so as to form a long double U-tube and a brisk current of cold water is kept circulating through it. A current of oxygen should be passed through the acid during the oxidation, care being taken that the tube very nearly touches the bottom of the flask, otherwise very violent bumping takes place owing to the heavy deposit of iodic acid crystals. With this apparatus, to get the reaction to go completely and quickly, a greater proportion of nitric acid is required. Moreover, it does not work well with larger quantities than 20 grams of iodine at a time, as the following results show: 20 grams of iodine gave 27 grams of iodic acid in 25 minutes, but from 40 grams of iodine only 49 grams were obtained after oxidation for an hour. A current of air is also of assistance if oxygen is not available, but either a somewhat larger proportion of nitric acid or a longer time must be employed to complete the oxidation.

Experiments in which the iodine was volatilised in the current of oxygen and passed as vapour into the boiling nitric acid were tried, but without much success.

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