XX.—*Note on the Estimation of Nitric Acid in Potable Waters.*

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A METHOD for the estimation of nitrogen existing as nitrates and nitrites in potable waters, should be at once delicate and simple. I have been using for some time past a method which, in common with Fr. Schulze's method, depends on the reduction of the nitric acid to ammonia by means of aluminium in alkaline solution. Instead, however, of using a quantity of aluminium known to liberate a known volume of hydrogen, when treated with an alkali, and observing the diminution caused by the nitric acid, I measure the ammonia formed, either by the Nessler-test or by titration.

The first thing to be done is to obtain some idea of the quantity of nitrate contained in the water. For this purpose boil a little of it in a flask with caustic soda, free from nitrate, until a sample of it does not colour the Nessler-test, cool, and then introduce a strip of aluminium foil. As soon as this is dissolved, decant the liquid into a test-tube, add Nessler-test,

and note the colour produced; should it be very dark, or should a precipitate be formed, either very little of the water (10 c.c. —25 c.c.) must be employed, or a tolerably large quantity, and the ammonia produced determined by titration. If, on the other hand, the colour is very pale, 200 c.c. will be a convenient quantity to employ; for common waters, such as those supplied to London, 100 c.c. is ample. For waters of the Loch Katrine class, 300 c.c. would be a convenient quantity.

Having learned what sort of water is in hand, measure off the volume indicated by the preliminary experiments—introduce it into a non-tubulated retort, and add 50 to 70 c.c. of a solution of caustic soda containing 100 grms. of soda in the litre. This soda must be free from nitrates. If very little water has been taken, add some distilled water. The contents of the retort are now to be distilled until they do not much exceed 100 c.c., and until no more ammonia can be detected in the distillate by the Nessler-test. The retort is now cooled and a piece of aluminium introduced into it (foil will answer very well with dilute solutions, but I much prefer thin sheet aluminium in all cases). The neck of the retort is now inclined a little upwards, and its mouth closed by a cork, through which passes the narrow end of a small tube filled with broken-up tobacco pipe, wet either with water, or better with very dilute hydrochloric acid free from ammonia. This tube need not be more than an inch and a half long, nor larger than a goose-quill. It is connected with a second tube containing pumice-stone moistened with strong sulphuric acid. This last tube serves to prevent any ammonia from the air entering the apparatus, which is allowed to stand in this way for a few hours or over night. The contents of the pipe-clay tube are now washed into the retort with a little distilled water, and the retort as adapted to a condenser, the other end of which dips beneath the surface of a little distilled water free from ammonia (about 70 to 80 c.c.).* The contents of the retort are now distilled to about half their original volume; the distillate is made up to 150 c.c.; 50 c.c. of this are taken out, and the Nessler-test added to them. If the colour so produced is not too strong the

* Condensers are very apt to contain a trace of ammonia if they have been standing all night, and must, therefore, be washed out with the utmost care. I prefer to distil a little water through them until I can no longer detect ammonia in the distillate.

estimation may be made at once; if it is, the remainder of the distillate must be diluted with the requisite quantity of water.

Should it be desired to determine the ammonia by titration, a much larger quantity of the water must be employed. Half a litre or a litre must be evaporated down to a small bulk, and this small bulk treated in exactly the same manner, except that the distillate is received in dilute standard acid instead of water.

With regard to the precision attainable by this method, all that I can say is, it is limited by the precision of the determination of ammonia by Hadow's quantitative method founded on the Nessler-test. If, therefore, the quantity of nitric acid be above $\cdot 0005$ grm. we may be sure of our result to 5 per cent., if the quantity be under this, the estimation will be somewhat less sharp. Even $\cdot 00004$ grm. nitric acid may be detected and an estimation made by this method.

I need hardly remark that the purity of the reagents employed must be ascertained by making blank experiments. I have found common caustic soda, sold in lump, to be free from nitrates. Should the caustic soda contain nitrate, it may be purified by dissolving a small quantity of aluminium in its cold aqueous solution, and then boiling to expel ammonia.