LXXI.—*Potassium Permanganate as a Reagent for the Detection of Lower Oxides in Phosphoric Acid.*

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DURING the course of other investigations, it became necessary to test samples of phosphoric oxide for traces of lower oxides. The usual tests recommended for this purpose are (*a*) boiling with a solution of mercuric chloride or (*b*) heating a neutralised solution of the phosphoric acid with silver nitrate, the production respectively of mercurous chloride or of a brown colour in the precipitate indicating the presence of lower oxides. Of these, the former seems to be the more sensitive, but is incapable of detecting less than about 0·05 mg. of phosphorous acid in 10 c.c.

The author has found the reduction of very dilute potassium permanganate solution to be a satisfactory test; this, however, is by no means an instantaneous reaction at the ordinary temperature, but occurs quite rapidly on gentle warming. In view of the extreme dilution of the potassium permanganate solution employed, it is essential to carry out an exactly parallel blank experiment and to compare the colour intensities of the two solutions. Where large quantities of phosphite are suspected, the test is much hastened by gentle heating, but for very small quantities it is preferable to allow the reduction to occur slowly at room temperature; in the latter case both the solution to be tested and the comparison solution should be made up with water from which all oxidisable material has been removed by distillation from potassium permanganate. Quite satisfactory results can be obtained, however, by using good ordinary distilled water and adding the potassium permanganate solution in slight excess.

The procedure adopted is as follows. About 1 g. of the phosphoric oxide is dissolved in 10 c.c. of water in a glass-stoppered test tube, and the solution is allowed to cool. An equal volume of a solution of phosphoric acid of about equal concentration is introduced into another similar tube. One c.c. of potassium permanganate solution of known concentration ($N/5000$–$N/1000$) is added to each. The solutions are mixed and kept for 2 hours. If there is any detectable colour difference between the two specimens, lower oxides are present. Certain specimens of distilled water cause a decrease in colour, but this is the same in each tube, so the test is not vitiated. In this manner it has been found possible to detect quite definitely 0·002 mg. of phosphorous oxide in 10 c.c. of solution.

For quantities of the order of 0·01 mg. per c.c. and upwards, for which 1 c.c. of $N/1000$-potassium permanganate solution can be used, a quicker method is to immerse the two tubes in a water-bath at about 60° for 2 minutes, remove them, and let them stand for 10 minutes; if there is no perceptible colour difference, the phosphite content is below about 0·005 mg. per 10 c.c. The quantity of phosphite can be estimated approximately by adding measured quantities of the standard *N*/1000-permanganate solution from a 1 c.c. pipette so as to restore the colour to the intensity of the " blank " sample. In this quantitative measurement, however, heating has to be continued for about 15 minutes, the colour intensities being matched, say, every 3 minutes. This gives a very good measure of the order of magnitude of the phosphite concentration when it is far too small to be estimated by the ordinary methods.

This method is obviously inapplicable in cases where other reducing agents are present, but has proved invaluable for confirming the absence of phosphorous oxide in resublimed phosphoric oxide.

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