Contributed and Selected

DETERMINATION OF ALCOHOL IN TINCTURE OF IODINE.

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As heretofore published[1] methods proposed for determining alcohol in tincture of iodine are very unsatisfactory, mainly owing to the time necessary for inversion of iodine with mercury as suggested by Alcock[2] or with iron filings or zinc as proposed by Roscoe & Schorlemmer[3].

The writers propose to fix the free iodine with sodium thiosulphate and then add sodium hydroxide to neutralize the sulphurous acid that may be formed when distilling. This procedure has the advantage of being rapid and giving practically accurate results.

The details are as follows: Decolorize 50 cubic centimeters of the tincture with a saturated solution of sodium thiosulphate, add 5 cubic centimeters saturated solution of sodium hydroxide and a few pieces of pumice to prevent bumping. Distill until all the alcohol comes over and make up the distillate with water to 100 cubic centimeters. Determine the alcohol in the usual manner with a pycnometer. Multiply the percentage of alcohol obtained by two and the result will be practically the percentage of absolute alcohol in the tincture.

Tincture of iodine prepared with alcohol of official strength should contain close to 92 per cent. of absolute alcohol by volume, as the iodine and iodide of potassium replaces from 3 to 4 cubic centimeters of alcohol in each 100 cubic centimeters of the finished product. There is, also, a difference in the temperature at which the tincture is prepared and that at which the alcohol is generally determined, therefore we think a tincture containing 90 to 91 per cent. of absolute alcohol by volume, should not be considered adulterated.

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A RAPID ACCURATE METHOD FOR THE QUANTITATIVE ESTIMATION OF CHLOROFORM IN CHLOROFORM LINIMENT.*

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A member of the Revision Committee of the Pharmacopœia, recently called my attention to the advantage of making official a method for the quantitative determination of chloroform in chloroform liniment, and the lack of a published process for the same.

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[1] Jr. Ind. and Eng. Chem., Vol. 1, 789. Merck's Report, Vol. 19, 35.

[2] Proc. A. Ph. A., 1904, 583.

[3] Treatise of Chemistry, Vol. 1, 157.

*Read before the New York State Pharmaceutical Association, June 25, 1912.

The subject being an important one, I began experimenting with the object in view of evolving a method whereby the pharmacist could easily and accurately make the estimation.

A method, which at first gave promise of yielding satisfactory results, was to precipitate the chloroform out of the liniment by means of 10 per cent. ammonium hydroxide, and while the results obtained were satisfactory when the sample was of U. S. P. strength, when the quantity of chloroform contained in the liniment was less than 25 per cent. or more than 30 per cent., the results were too far from the truth to be of value. The method was therefore abandoned. An effort to throw out the chloroform by means of centrifugal force did not yield concordant results.

It soon became apparent that the soap in the liniment was the disturbing factor, and that to obtain satisfactory results it was necessary to distill the chloroform. Remembering this fact, the following method was devised:

Into a test-tube having a capacity of about 85 cc. and about 24 mm. in diameter, place 10 cc. of distilled water and 10 cc. of liniment to be analyzed, accurately measured with a pipette; to prevent bumping, a small piece of pumice stone which has previously been heated to white heat and thrown into water, is added. The test-tube is connected with a Liebig condenser by means of corks and bent tube. For a receiver use an accurate 25 cc. cylinder graduated in tenths or fifths of a cc., containing 5 cc. distilled water. It is not necessary to have the condenser tube come in contact with the water. All that is required is to have it project into the cylinder. It is easy to know when the chloroform is all distilled by watching the receiving cylinder. As the chloroform distills it sinks to the bottom and then comes a lighter distillate, which remains on top and is perfectly clear, and then a distillate which forms a milky layer occupying about 1 cc.; after this turbid zone has appeared, remove the cylinder; stopper it with a sound cork and mix by shaking thoroughly; then remove the cork and add diluted sulphuric acid (10 per cent.) to the 25 cc.; mark and shake thoroughly. In a few moments the chloroform will have settled to the bottom in a clear layer, and all that remains is to multiply the cc. of chloroform by 10 to obtain the percentage of chloroform in the sample. The entire operation does not require over fifteen minutes.

The results obtained on a large number of samples of known but varying strengths, proved the method to yield such very accurate results that should the Revision Committee decide to make official a method for the quantitative estimation of chloroform in chloroform liniment, it is suggested that they adopt this one.

Of course, a description of the method for use in the Pharmacopoeia could be very much shortened, as I have purposely gone into detail in describing it.

In view of the accuracy of results, ease of application, and simplicity of apparatus, the method has everything to commend it.

I would take this opportunity of acknowledging my indebtedness to my assistant, Mr. I. Schwartz, for valuable aid rendered in connection with the work.