ESTIMATION OF OIL OF PEPPERMINT IN SPIRIT OF PEPPERMINT.

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The estimation of oil of peppermint in Spirit of Peppermint has usually been carried out by means of the precipitation method as used for Spirit of Lemon, using a Babcock milk flask with a graduated neck. The varying results, however, which have been obtained have caused criticism of the method as inaccurate, and upon investigation of the subject we have found that the fault lies not with the principle of the method but with the manner in which it is carried out. The use of a Babcock milk flask, holding as it does only a little more than 25 cc. of liquid, does not permit of sufficient dilution with water in preparations containing a high amount of alcohol and a low amount of oil, for peppermint oil is distinctly more soluble in diluted alcohol menstrua than is lemon oil, to which the method is particularly applicable in its form adopted in the U. S. Dept. of Agriculture Bulletin No. 107.

Experiments have shown us that where the alcoholic strength is reduced below 25 percent, the amount of oil dissolved is negligible in amount, but the use of so small a proportion of the Spirit in a Babcock milk bottle makes the separated volume of oil so small in amount as to seriously interfere with the sensitiveness of the method to within one or two percent.

A larger form of flask was designed by us which gives very good results and which consists of a conical flask of 100 cc. capacity terminating in a long narrow tubular neck not over 12.5 mm. in diameter and graduated up to 10 cc. in one-tenths.

The introduction into such a flask of 25 cc. of Spirit of Peppermint, followed by the addition of 5 cc. of hydrochloric acid and sufficient warm water to fill the flask and bring the oil up into the neck, suffices for the determination within one-tenth of one percent upon all stregths from 10 percent down to 1 percent, all in strong alcohol. The addition of salt which was thought would be of value in hastening the separation of the oil is not permissible, for in the salting out by such a process some of the alcohol is separated with the oil and the results run high to the extent of several percent in the several experiments tried.

With such a flask, gravitation alone suffices to bring the oil up into the neck of the flask within several hours, occasionally rotating to lessen the tendency of the globules to adhere along the sides of the flask and neck. If the flask were constructed, as could easily be done, so as to permit of whirling in a centrifuge, the estimation could be made accurately and satisfactorily within a very few minutes.

Such flasks are in use by us for the determination of all of the spirits of oils lighter than water, excepting almond, and the additional advantage is gained that after the volume of the separated oil has been accurately observed and noted, the oil itself may be easily and completely removed by means of dry filter paper or blotting paper inserted in rolled strips, without the removal of any of the hydro-alcoholic liquid beneath. The contents of the flask after the

removal of the oil may be transferred to a distilling flask, the hydrochloric acid neutralized and the alcoholic distillate from 25 cc. of the original spirit obtained in better condition, as regards freedom from oil, than usually results by following the official method of diluting the original spirit and filtering through magnesium carbonate, which always occasions a slight loss by evaporation which cannot take place to the same extent under the procedure given above.

PHOSPHORIC ANHYDRIDE CONTENT OF SIMPLE AND COMPOUND SYRUP OF HYPOPHOSPHITES.

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The attention of this department was recently directed to the great difficulty of holding all the ingredients of Syrup of Hypophosphites, both simple and compound, in solution. The solution of the various salts in water is easily accomplished but the addition of the sugar precipitates a part and if the product is immediately strained, this precipitate is removed and the resulting solution is lower in strength than was originally intended. Upon inquiry, we found that several manufacturers had noted this fact and were somewhat in doubt as to the actual content of finished product, but had assumed the loss to be immaterial.

The first difficulty encountered in our investigation was the adoption or formation of a suitable method for the determination of the amount of phosphorus present. It was at once apparent that the great amount of organic matter must be destroyed by some means that would not interfere later in the phosphate precipitation and for this reason the simple process of ashing was not applicable because all hypophosphites decompose at low heat into pyrophosphates with the evolution of hydrogen phosphide. The reaction is quite variable and the remaining phosphorus cannot be used as an index of the amount originally present.

A search of the literature revealed the fact that while no method was recommended, the National Dispensatory states that the one of H. A. D. Jewett is "seemingly" accurate for the estimation of phosphorus in calcium hypophosphite. Briefly this method consists in the liberation and oxidation of the hypophosphorus acid to phosphoric acid by means of bromine and the determination of the phosphoric anhydride in this condition. Any phosphites that might be present as impurities are removed by precipitation with lead acetate. We experimented with this method as outlined and obtained very satisfactory results on calcium, sodium and potassium hypophosphites alone and in the presence of a large amount of sugar. In the course of our experiments we found that the lead acetate gave but a slight precipitate even in a concentrated solution of a hypophosphite and the percent of phosphites in the dilute syrup could be neglected. We also, for convenience, substituted concentrated nitric acid for the bromine and evolved the following method in detail:

Determine the specific gravity with a Westphal balance or pycnometer. Weigh accurately two grams of the sample into a one hundred cubic centimeter beaker, add ten cubic centimeters concentrated nitric acid and cautiously bring to boiling.