

The test is very simple and only about five minutes is required to make it. A further classification of those preparations within the strong group could be obtained by allowing, in all cases, the filter to drain completely, and then titrating a definite volume of the filtrate against a dilute standard solution of thio-cyanate. The turbidity noticed in the case of proganol as compared to the heavy precipitate obtained when protargol and protargentum are tested in this manner, shows a silver-ion concentration lower than the last two. This has been substantiated by electrometric determination made in this laboratory.

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THE DETERMINATION OF THE AMOUNT OF OIL IN SPIRIT OF PEPPERMINT.*

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Spirit, or Essence of Peppermint, when manufactured according to the Pharmacopœia, is an alcoholic preparation containing approximately 10 per cent of Oil of Peppermint, with chlorophyll, extracted from the herb, as color. The Pharmacopœia directs the maceration of the moist herb with the mixture of alcohol and oil, filtration of the mixture to remove the herb and, finally, the completion of the volume by passing sufficient alcohol through the filter. The completed preparation is leaf green in color, due to the alcohol-soluble chlorophyll.

The therapeutically active ingredient is the Oil of Peppermint, in which the important constituent is the complex alcohol, menthol, of which the U. S. P. requires not less than 50 per cent. With the present high price of peppermint oil, the manufacturer is tempted to reduce the amount in the spirit below the official requirement in order to increase his profit or to enable him to meet cut price competition. The ethical way for the retailer to insure his dispensing the U. S. P. Spirit of Peppermint is to manufacture it himself. But because of the present method of taxing alcohol, the manufacturer can make a U. S. P. preparation and sell it for considerably less than the cost of the ingredients to the retailer, so one can hardly blame the thrifty pharmacist for purchasing his requirement of Spirit of Peppermint, instead of manufacturing it.

The analysis of Spirit of Peppermint for the percentage of oil is a simple matter, requiring only one piece of apparatus, a Cassia flask, which is not ordinarily possessed by every pharmacist. This flask is conical in shape, with a long narrow neck, which is graduated from 0 to 10 cc., and holds about 125 cc. up to the 0 graduation on the neck, the exact capacity being immaterial.

The method as given by Charles H. LaWall and LeRoy Forman, in the JOURNAL OF THE AMERICAN PHARMACEUTICAL ASSOCIATION about ten years ago, is as follows:

Introduce 25 cc. of the Spirit, accurately measured, into a Cassia flask which holds approximately 125 cc. to the 0 graduation in the neck. If a graduate is used to measure the Spirit, drain it well and rinse with portions of warm water, adding rinsings to the contents of the flask. Add 5 cc. of diluted hydrochloric acid to the Spirit and rinsings in the flask, and then follow with sufficient water at 80° C. to bring the upper level of the liquid to about the 8 cc.

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graduation in the neck. The oil will immediately separate and rise to the top. Tap the flask gently upon the table top to loosen any globules of oil that may be adhering to the sides of the flask, or impart a rotary motion to the flask by rolling it between the palms of the hands. Immerse the flask in a dish of water at 80° C. for 15 minutes, and again tap the flask to loosen any more particles of oil. Allow to stand at room temperature for 12 hours or longer and take the reading of the volume of separated oil by subtracting the reading of the extreme lower limit of the meniscus at the lower level of the oil from the extreme upper limit of the meniscus at the top of the layer. Divide the volume of oil by 25 cc. and multiply by 100 to get the per cent of oil in the Spirit.

The method is simply the measurement of the volume of oil which separates from a measured volume of Spirit, upon largely diluting with water. It will give fairly accurate results if ordinary care is used in measuring out the Spirit and in reading the volume of separated oil. The largest source of error, aside from that from manipulation, is the oil that remains in solution in the diluted alcohol.

To determine whether or not this oil in solution was sufficient to produce appreciable error in the determination a ten per cent solution of U. S. P. Oil of Peppermint in 95 per cent alcohol, without the herb, was tested by the above method and results giving 10 per cent of oil were obtained, thus showing that the method was accurate within 0.1 per cent. Next a number of samples of Spirit of Peppermint were carefully made up according to the U. S. P. directions and using the same oil as in the above solution, with peppermint leaves for coloring. These samples gave only 9.8 per cent of oil upon analysis. More samples were made up in which the herb and filter paper were carefully rinsed with the available amount of alcohol to bring all the oil through into the preparation, but the results were still 9.8 per cent. The conclusion was that the herb and filter paper held some of the oil, and that the amount of alcohol allowable to complete the volume is insufficient to wash the entire amount of oil into the filtrate.

Recognition of this fact must be made when testing a sample of Spirit. A preparation yielding 9.8 per cent of oil may still be within the U. S. P. requirement, as it specifies that 10 per cent of oil must be used and it does not say that the Spirit must yield 10 per cent of oil. The above experiments showed that one may put 10 per cent of oil into a spirit and obtain only 9.8 per cent upon analysis.

The analysis of 33 samples of Spirit of Peppermint indicated that there are very few poor preparations on the market. Seventeen samples yielded between 9.8 per cent and 10.2 per cent of oil; three samples gave 11 per cent or over; thirteen samples were below 9.8 per cent in strength, only four or five, however, being below sufficiently to consider them of poor quality. The poorest sample gave 6.8 per cent of oil.

The universal charge for an ounce of this preparation was 40c in all except one instance—a cut-rate store—where 25c purchased a bottle labeled "1 fluid-ounce," which actually yielded only $\frac{5}{6}$ of an ounce, containing about 8.6 per cent of oil.

It might be interesting to note that about a year ago a retailer tested 2 gallons of Spirit of Peppermint by this method, which he had purchased from an eastern wholesaler, and found that it contained only 8 per cent of oil. The manufacturer, upon being informed of this, immediately sent 2 more gallons, and admitted that because of inefficiency in their manufacturing department they, unknowingly, had placed some sub-standard spirit on the market.