

We introduce the lightest liquid first and the heavier reagents on being added start to sink through it, but they mix on their way down and the shaking is unnecessary. It is very difficult to shake any nitrometer when making an assay and I have stopped doing it.

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### THE ANALYSIS OF EMULSIONS.\*

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The analysis of emulsions by the drug analyst is a branch of work not generally welcomed, because of the lack of specific information on the subject, in books devoted to the analysis of pharmaceutical preparations. Even such recent books as "The Analysis of Drugs and Medicines" by Nelson, and "The Qualitative Analysis of Medicinal Preparations" by Fuller, contain little or no specific information on the subject.

The necessity for doing some practical work along this line, recently led us to experiment with the method known as the Gottlieb Roese Method, which has been used successfully for some time in the estimation of fat in dairy products, and the results were so surprisingly successful, when working upon known mixtures, that the method is suggested for routine work in this connection.

As officially described in the Proceedings of the A. O. A. C. for 1909, Bulletin 132 of the Bureau of Chemistry, U. S. Dept. of Agriculture, an extraction apparatus known as a Rohrig tube, which is now a standard piece of apparatus, is directed to be used. We have found, however, that excellent results can be obtained by the use of a glass graduated cylinder, and the evaporation of an aliquot part of the extraction liquid, although the method as described in full may be used if desired.

The modified method used by us is as follows:

Prepare a mixture of the emulsion in distilled water, so that each 100 cc. of the liquid contains 40 gm. of the emulsion. Take two 100 cc. graduated cylinders and in one, place 10 cc. of the diluted emulsion and in the other, place 5 cc. of the diluted emulsion and 5 cc. of water. To each cylinder then add the following reagents in the order named, agitating thoroughly after each addition:

- 1 cc. stronger ammonia water.
- 10 cc. alcohol U. S. P.
- 25 cc. ether U. S. P.
- 25 cc. petroleum benzin U. S. P.

After the addition of the petroleum benzin, the agitation should be continuous for 10 minutes, after which the cylinders should be allowed to stand until the liquids have separated into two layers with a sharp dividing line, (this requires from 15 minutes to 1 hour). Then having observed the exact volume of the upper layer, draw off exactly one-half and transfer to a flat-bottomed glass capsule and evaporate quickly on a water bath to constant weight. In one of the duplicates the resulting fat will be from 2 gm. of the emulsion, in the other from 1 gm., which gives a satisfactory check upon the thoroughness of the extraction.

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\* Read before Scientific Section at Detroit Meeting.

Working with emulsions prepared from various oils, the following results were obtained by this process:—

KIND OF OIL	PERCENT OF OIL IN EMULSION	PERCENT OF OIL OBTAINED
Neatsfoot .....	40	39.87
Corn .....	35	34.00
Almond .....	25	24.60
Olive .....	31.50	31.48
Cod Liver .....	50	49.72

The emulsifying agent in some cases was tragacanth, and in others was acacia. So much for the quantitative determination of the oil.

Now, in order to ascertain whether the separated oil varied in the more important physical and chemical constants, separate extractions were made, using larger amounts of material and paying no attention to the quantitative feature, the effort being simply to obtain about 5 or 10 grammes of the separated oil. Upon each of these separated portions of oil, the following factors were determined: saponification value, iodine value and refractive index. These values on the samples obtained by extraction, were compared with the same values as obtained from the original oils from which the emulsions had been prepared. No greater deviation in results was noticed than is commonly observed in making duplicate determinations on the same oil.

The results warrant the conclusion that the modified Gottlieb Roese method may be successfully applied in the analysis of emulsions, both for the quantitative determination of the fat and for the separation of a sufficient amount of the fat for the determination of such constants as will lead to its identification.



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