

few exceptions *failed to retain* the properties of tastelessness and freedom from odor. There is also a tendency to become yellowish or yellowish-brown in time. It would seem from some experiments which have been made that exposure to direct sunlight causes discoloration. This is remarkable because exposure to sunlight has been regarded for centuries as inducing a bleaching or decolorizing effect on most substances. At present it would seem that manufacturers of American oil have not entirely mastered the problems of purification.

At present buyers of American Liquid Petrolatum should be cautious about laying in a stock of the Oil in large lots, for they are likely to find that the last portions have acquired properties which will render it unsalable, since the slightest coal oil taste makes it unfit for internal administration.

The writer has faith in the American manufacturers' ability to overcome these defects and the greatest encouragement should be given them to produce an American Oil suitable for all purposes.

The new Pharmacopoeia will cover these points by appropriate tests. The sulphuric acid test to determine the presence of carbonizable impurities; the lead oxide test for sulphur compounds; a test for absence of acidity or alkalinity, and the physical tests to determine the tastelessness and freedom from odor and fluorescence will be given. A light Liquid Petrolatum will also be official for use in atomizers and nebulizers.

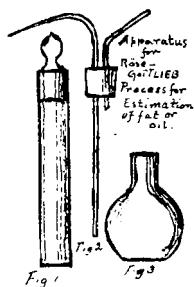
The definition will be changed simply to read, "A mixture of liquid hydrocarbons" as it is unnecessary to specify the source from which it is derived, either the methane or naphthene series.

FATS AND OILS.*

The Adaptability of the Röse-Gottlieb Method for the Estimation of Fat and Oil in Certain Pharmaceutical Preparations.

CHARLES A. HACKMAN, F. I. C.

Judging from statements recorded in past numbers of this journal (Chemist and Druggist), it does not appear to be generally known that the Röse-Gottlieb method affords the most rapid and accurate means for the determination of oil in many pharmaceutical materials and preparations. For example, the estimation of oil in cod-liver oil and malt presents many difficulties when the usual method of extraction with immiscible solvents is attempted; but with the Röse-Gottlieb method, suitably adapted, these difficulties are readily overcome, and the present note has been written with a view to drawing attention to the excellence and wide adaptability of the process. Originally designed by Gottlieb for the estimation of fat in milk (Analyst, 1898, 259), and subsequently modified by Röse, the method was prominently brought before analysts by H. Droop Richmond in "The Analyst" for October, 1908, p. 389. In that communication its suitability for the estimation of fat in dried milks was



* Chemist and Druggist, July 3, 1915.

especially pointed out, and in concluding his paper the author made the following remarks:

"The method is so convenient, accurate, and rapid, that I now use it in preference to all others for the analysis of milk (of which 5 cc. is taken and no water added) and all milk-products; it is also applicable to the analysis of eggs, egg-yolk, and dried egg-preparations, provided the quantities taken are not too large." Continued and extended use of the process for many years enables the writer of this note to confirm the above remarks in every particular, and for the benefit of those who have, perhaps, hitherto regarded the method as applicable only to milk, a detailed description of its working is here appended. The apparatus required is of the simplest description, and consists of Fig. 1, a narrow, accurately stoppered tube, approximately 7 in. high and 1 in. wide; Fig. 2, a cork to fit this tube carrying thin wash-bottle tubes; and Fig. 3, a light glass flask, and (4) a condenser. The reagents used are (1) solution of ammonia (0.880 ammonia diluted with an equal volume of water), (2) absolute alcohol, (3) ether (sp. gr.=0.720), and (4) petroleum ether (boiling point below 60° C.).

The details of procedure in the case of milk are as follows:

The tube, without its stopper, is first accurately weighed; 5 cc. of the sample is then pipetted in, and the whole weighed again, the difference giving the exact weight taken. Next 0.5 cc. of the ammonia solution is run into the tube, followed by 5.0 cc. of alcohol, and the whole thoroughly mixed. Then 12.5 cc. of ether (measured out in a small cylinder) is added in approximately 5 cc. quantities at a time, holding the tube in the right hand and imparting to it a rotary motion after each addition in order to secure thorough admixture. A similar quantity (12.5 cc) of petroleum ether, measured in the same way, is finally added and mixed. The tube, with the stopper loosely in position, is placed in a water-bath containing lukewarm water and gently rotated until the ether-vapour just commences to escape and condenses around the ground-in portion of the stopper. As soon as this happens the stopper is pushed home, and the tube and its contents are cooled under the tap. After gently inverting once or twice, the tube may either be set aside for the mixed layer to separate, or preferably "whirled" immediately in a centrifuge until this separation is complete.

The stopper is now cautiously withdrawn, and its underside rinsed off into the tube with a few cubic centimeters of mixed ethers contained in a small wash-bottle. The use of the warm-water bath employed above is apparent at this stage, since the slight vacuum produced prevents any of the contents of the tube being ejected—as would otherwise be the case—when the stopper is withdrawn. The cork carrying the wash-bottle tubes is now inserted in the place of the stopper, and the long tube adjusted until it is about one-fourth inch above the line of demarcation of the two liquids. On applying gentle pressure to the shorter tube, either by the mouth or by means of a small pressure-tank provided with a finely acting valve, the whole of the ethereal layer is blown off into the light glass flask. The cork is next carefully removed, the outside of the longer wash-bottle tube rinsed back into the Gottlieb tube, 25 cc. of mixed ethers added, the process described above repeated in exactly the same manner and the separated ethereal layer again blown off into the flask containing the first portion.

In the case of milk two "blow-offs" are sufficient, but in some substances richer in fat or oil three or even four are necessary.

The thin glass flask containing the united ethereal extracts is now attached to a condenser and the mixed ethers are distilled off. After detaching the flask the residual alcoholic and ether is blown off by means of a current of air, and the residue dried to constant weight in the water-oven. The fat is next dissolved in a little petrolatum ether and cautiously decanted from any insoluble residue. After several rinsings to remove the whole of the fat, the flask is again dried and weighed, the difference between the two weighings giving the amount of fat in the weight of sample taken.

In adapting this process to substances other than milk it is important to keep the relative proportions between the water and immiscible solvents constant, and to secure thorough admixture of each reagent. Thus in the case of extract of malt with cod-liver oil a weighed quantity approximating to 1 gram is taken and dissolved in sufficient warm water to occupy 5 cc. The remaining procedure is then as described above. For all emulsions the process will also be found eminently suitable. The estimation of the proportion of cacao-butter in cacao and chocolate is possibly of minor importance to pharmacists, but the Röse-Gottlieb process in the hands of the writer, employed with slight modification in the order of adding the reagents, has proved the quickest and most accurate process for this estimation that is known to him.

LIMITATIONS OF THE REACTION WITH NINHYDRIN.

Current medical literature bears witness to the interest which has attached quite recently to the triketohydrinden reaction, more popularly known by the name of ninhydrin reaction. Until very lately it has been regarded as a characteristic test for amino-acids or compounds of them, and consequently has found a wide application in some of the modern serum reactions, such as those introduced by Abderhalden in relation to pregnancy, cancer, etc. In view of the growing use of the reagent, it deserves to be pointed out that many substances which in no sense exhibit a combination of amino and carboxyl groups, as do the amino-acids and proteins, nevertheless give a characteristic reaction. Amines, amino-aldehyds, aminosulphonic acids, urea derivatives and certain organic acids may be mentioned in illustration of this statement. To these known exceptions Neuberg has added a new series of both organic and inorganic substances, some of which might lead to deception in respect to the reaction. It would be of little advantage to recite here this list of compounds, which the specialist in this field should learn at first hand. Perhaps it is worthy of mention, however, that a minimal putrefactive change in proteins is sufficient to provoke the appearance of substances which give a strongly positive test with the ninhydrin reagent. They are presumably putrefactive bases which are in part of a volatile nature. These facts point to the necessity of caution in the interpretation of findings based on the use of the ninhydrin test.—*Journal A. M. A.*