

Method for Wool Oil Unsaponifiables

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WOOL greases and wool grease soaps are occasionally used as emulsifiers in emulsions of hydrocarbon oils sold for insecticidal purposes. Wool greases sometimes are adulterated with lubricating oil. Wool fat is not completely saponified by alcoholic potash solution, hence the usual method of determining mineral oils in the presence of fatty oils leads to high results, due to the inclusion of higher alcohols (cholesterol, ceryl alcohol, etc.) which are normal constituents of wool fat, in the unsaponified residue. Saponification in a pressure flask by a solution of metallic sodium in absolute alcohol, which has been recommended, is too tedious for a routine method of analysis.

The method described in this paper directs a preliminary treatment with alcoholic potash solution, if the sample is not already saponified, to separate the bulk of the fatty oils from the mineral oils. The residue from this treatment is purified by the use of ethyl-amyl alcohol mixture or by acetylation with acetic anhydride.

If the sample is a soap emulsion, weigh a portion sufficient to give 10 cc. of unsaponified oils, extract twice with 50 cc. and 30 cc. of ethyl ether, adding 1 to 2 cc. of 50 per cent sodium hydroxide solution to break the emulsion. As the mixture emulsifies readily, avoid too vigorous agitation. The ether extracts hydrocarbon oils, ceryl alcohol, etc., and small quantities of soap. Wash the extract with 10 per cent alcohol and 2 per cent sodium hydroxide solution until free of soaps. At this stage washing with pure water will form an emulsion which is difficult to break. Wash the combined alkaline and alcoholic layers successively with 2 portions of ether of 50 cc. each. Combine the ether extracts, evaporate the ether from the solution placed in a tared beaker and weigh the residue. Determine its specific gravity.

If the product under examination is a wool grease, weigh a portion sufficient to give 10 cc. of unsaponified oil, saponify in the usual way with alcoholic potash, evaporate off most of the alcohol on the steam bath, add 100 cc. water and evaporate to 50 cc. Cool the solution and transfer to a separatory funnel. Rinse out the saponification flask with 25 cc. ether and add to the separatory funnel. From this point, proceed as given in the directions for soap emulsions. This preliminary saponification separates most of

the fatty material from the hydrocarbons.

A close approximation of the quantity of hydrocarbon oils may be made by treating 10 cc. of the extracted oils with a mixture of 1 part amyl alcohol and 2 parts ethyl alcohol, using 40 cc. (adding from a buret). Place the mixture in a graduated cylinder, reading to 0.1 cc. Shake the mixture. The alcohols, etc., dissolve, and the hydrocarbons separate. Note the quantity of oils and determine the gravity and initial boiling point. Calculate percentage by weight, based on original sample.

A check on this determination may be made on the unsaponified material present in the sample or obtained after saponification with alcoholic potash or with metallic sodium and absolute alcohol. Determine the specific gravity of the unsaponifiable material, and pipet 10 cc. into a 50 or 100 cc. Florence or round-bottomed flask. Add twice its weight of acetic anhydride, connect with a reflux condenser, and boil for half an hour. Pour the acetylated mixture while hot, into a warmed small separatory funnel, rinsing the flask with the smallest possible quantity of acetic anhydride. If two layers separate from the hot liquid, hydrocarbon oils are present. If the solution is homogeneous, but crystals separate on cooling, cholesterol and higher alcohols, and no hydrocarbon oils, are present. As cholesterol is a normal constituent of wool fat, the solution must be kept warm during separation of the two layers, to avoid crystallization. Run off the lower layer into a small beaker, and save for tests for cholesterol, if necessary. Wash the portion left in the funnel once with a little acetic anhydride. If too much be used, notable quantities of hydrocarbon oils are dissolved. Wash the separated oils with hot water, and transfer them to a Babcock bottle. Wash the funnel with cold water, adding the washings to the Babcock bottle. Fill up to the graduations with cold water, allow to stand until oils separate in a clear layer, or centrifuge a few minutes, and note the number of cubic centimeters of the separated oils. Determine their specific gravity and calculate percentage by weight, based on the original sample.

As hydrocarbon oils are normally found in a distilled wool grease, caution should be exercised in reporting the oils thus found as mineral oils, added to the sample.