Determination of Neutral Fat in Soaps

A New Rapid Method Suitable for Control Laboratories.

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UNSAPONIFIED - saponifiable matter in soap includes free fatty acid if present and any neutral fat. In the case of a thoroughly uniform soap which contains free alkali, the unsaponified saponifiable matter would consist wholly of neutral fat, often referred to as "free fat."

The determination of neutral fat in soap by such standard methods as those of the Bureau of Standards (Circular 62, 3rd ed. of 1923, page 23, section (e), or of the American Chemical Society (J. Ind. Eng. Chem. Vol. 14, 1159 (1922)) involves a wet extraction process which is very tedious and requires a large amount of personal attention by a skilled analyst.

Both of the above standard methods provide for the correction of any free fatty acid contained in the unsaponified-saponifiable matter by titration and deduction of the amount found.

(Note: An extraction method is faulty which makes no provision for the correction of free fatty acid that is invariably contained, in the author's experience, in the unsaponified-unsaponifiable residue obtained by the wet extraction of soap solutions by ethyl ether. Analyses of soaps containing no free fatty acid whatever, made in this laboratory by wet extraction methods, have repeatedly yielded ether extracts in which the free fatty acid (calculated as oleic acid) amounted to 0.4 to 0.6 per cent, and even higher, of the weight of sample taken.)

It should be pointed out, however, that this correction cannot be made with strict and certain accuracy since the molecular weight of the free fatty acids involved is not known. Free fatty acids are ordinarily computed as oleic acid, but for example, in the extraction of a soap high in cocoanut oil the mixed free fatty acids obtained along with the neutral fat and unsaponifiable might have a mean molecular weight quite different from that of oleic acid. Furthermore, the residue containing an unknown amount of true unsaponifiable matter must be saponified and re-extracted to obtain the amount of unsaponifiable before the per cent of neutral fat can be computed.

Rapid Method of Determining Natural Fat in Soap

The following method for the determination of neutral fat in soap involves no new principles and is merely an adaptation of the method for determining saponification number.

Directions:—Weigh a sample of soap of from 10 to 15 grams (depending on moisture content) to 0.1 gram. Dissolve in 150-200 cc of hot neutral alcohol (94% or higher). If the alcoholic solution is not entirely clear and free from alcohol-insoluble matter, filter with suction through a Gooch crucible, protecting the solution from carbon dioxide or other acid fumes and wash the crucible with hot neutral alcohol until free from soap. Unless the resulting alcoholic soap

solution is neutral to phenolphthalein indicator, add just enough aqueous alkali or acid as required, to neutralize any free acidity or free alkali in the soap. To the neutral alcoholic soap solution, add 10 cc alcoholic KOH (40 grams per liter in aldehyde-free 94% alcohol) accurately measured with a pipette or burette. Attach to a reflux condenser and boil for 30 minutes. Run a blank in the same manner with 150 cc neutral alcohol and 10 cc alcoholic KOH accurately measured, boiling under reflux as with the sample. While still hot titrate both blank and sample with N/2 aqueous acid. From the number of cc of alcoholic potash absorbed calculate* the per cent of neutral fat in the sample.

$$\frac{\text{cc N/2 Alkali Consumed} \times 28.05}{\text{Saponification Number (mgs KOH)}} \times \frac{100}{\text{Wt. Sample (in grams)}} = \% \text{ Neutral Fat}$$

This method is particularly valuable for the rapid routine control of cold made soaps such as shaving soap, vegetable oil soaps, soap base for liquid soaps and the like, but can be applied equally well to boiled soaps.

It can also be applied with a fair degree of accuracy to soaps of unknown fat composition after obtaining the proper saponification number by either (1) or (2) as follows:

(1) Separate the fatty acids from a portion of the sample and determine their neutralization value, expressed as milligrams KOH per gram. Neutralization Value of Fatty Acids x 0.97 = Saponification Number (approx.) of neutral fat from which they were derived.

(2) Unless the sample contains a considerable proportion of such oils as coconut oil (avg. Sapon. No. 257) or of castor oil (avg. Sap. No. 181) its fat stock will probably have a mean saponification number of 195 to 200 and assumption of a value of 200 will usually yield results accurate within the limit of experimental error.

* This method was worked out for control purposes on soaps whose fat stock and whose saponification number were known.

Comparison of Methods for Determination of Neutral Fat in Soap

Standard A.C.S. Method	*Rapid Control Method*
Weigh sample, 5 grams	Weigh Sample 10 grams.
Dissolve in 100 cc hot 50% alcohol	Dissolve in 150-200 cc hot neutral 94% alcohol.
	Filter with suction, if not free from carbonates and other fillers.
If acid, neutralize with aqueous alkali.	If acid, or alkaline, titrate until neutral.
Evaporate bulk of alcohol.	
Redissolve in 200 cc hot water; transfer to separatory funnel, cool and carry out a complicated extraction process with ethyl ether, using 3 separatory funnels and repeated washings of ether extracts.	(no extraction, no evaporation)
Filter ether extract, evaporate and weigh (1).	

Dissolve residue in neutral alcohol and neutralize with standard alkali (3).	
Deduct free fatty acid found by titration (3). Residue is neutral fat plus unsaponifiable (3).	
Thoroughly saponify residue with alcoholic potash.	Thoroughly saponify with known excess of alcoholic potash.
Repeat the foregoing procedure.	Titrate excess of alkali with standard acid and from amount of alkali absorbed by sample calculate the % neutral fat in sample.
Weigh the residue of unsaponifiable matter (4).	
Neutral Fat in sample = (4)—(3).	
Summary:—	
5 Weighings.	1 Weighing.
1 Titration (2 if originally acid).	2 Titrations (or 1 if soap is neutral).
Complicated extraction procedure.	1 Titration of blank.
Considerable skilled attention required.	Minimum amount of attention.
Total elapsed time for analysis 3 hours or more.	Total elapsed time for analysis 1 hour or less.

By the rapid control method above described neutral fat in soap may be determined in an hour or less with an accuracy of about 0.05%, using a 10 gram sample and making titrations with N/2 acid.

Codliver Oil Equals Sun's Rays Effects

Experiments on albino rats by a group of Massachusetts chemists showed that codliver oil is more generally applicable in the treatment of rickets, which afflict children, than ultra violet light, which is a substitute for the sun, according to a report to the Division of Medicinal Products of the American Chemical society.

One group of rats was treated for fifteen minutes daily with ultra-violet light of known intensity, said the report presented by Dr. Arthur D. Holmes, director of the Patch Reserve Laboratory Boston, and his assistant, Miss Madaleine G. Pigott; and Dr. Edwin T. Wyman, associate professor of pediatrics, and Dr. Lawrence W. Smith, instructor in pathology, Harvard Medical School. Similar groups of rats during the same period were fed codliver oil in amounts varying from one-fifth of a drop to one drop daily.

The investigators determined the effect of the ultra-violet light treatment and the codliver oil feeding by their effect on the animal's body weight, the calcium and phosphorous content of their blood, X-ray pictures of the bones, by a pathological examination of the bones, and by their mineral content.

Judged by these five diagnostic features, it appeared that one-fifth to one-half of a drop daily of the oil was as effective in protecting the animals against rickets as ultra-violet treatment for fifteen minutes daily at a distance of three feet.

—*Drug Markets.*