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A Rapid Volumetric Test for Anhydrous Soap

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INTRODUCTION

This paper treats of a rapid, volumetric method for determining anhydrous soap. The procedure and equipment are adapted from the Babcock test for butterfat used in the dairy industry.

The test has been given a fair trial for a period of six months in the author's laboratory and has developed to the point where it largely supersedes the long, unwieldy A.O.C.S. ether-extraction method.

EQUIPMENT AND DETAILS OF PROCEDURE

The equipment consists of a hand driven Babcock milk-test centrifuge, a liter Berzelius pyrex beaker which is used as a boiling-water bath,* pyrex test bottles made up according to the specifications listed in the accompanying sketch. The 4 ml. graduated necks of the latter are calibrated at 100°C. to contain milliters at 4°C. Usually a correction factor has to be determined for each test bottle, which should not exceed 1±.004 for the 4 ml. graduation.

The routine of the test is as follows:

Weigh in the tared test bottle a 3.5 to 5 gram sample (depending on anhydrous soap content) to the nearest milligram. Add about 5 ml. (an excess) of 37% hydrochloric acid and place in water bath until fatty acids clarify. Twirling the bottle prevents carbonates from foaming over after addition of acid. Glycerin C.P. at a temperature of 110-115°C. is added from a wash bottle until the fatty acids are brought into the graduated portion of the neck. The test bottle is centrifuged for a few seconds and then placed in the boiling water bath the upper level of which should lie above the column of fatty acids. Equilibrium in temperature between the fatty acids and the boiling water should not take more than 10 or 15 minutes. At the end of this period, the reading is taken. First the test bottle is partially withdrawn to allow the reading of the bottom of the upper meniscus of the fatty acids, then the bottle is completely withdrawn to allow the reading of the bottom of the lower meniscus. The difference between the readings represents the observed volume in milliters, which multiplied by the correction factor of the test bottle gives the actual volume of fatty acids at 100°C./4°C. The actual volume multiplied by a predetermined factor gives the weight of anhydrous soap. The latter factor is derived from the specific gravity of the fatty acids at 100°C./4°C. and the acid value of the fatty acids.

Special technic is required in introducing some soap samples into the test bottle. Flake soap may be finely comminuted. Bar soap can be cut into small strips. Hot neat or kettle soap may be introduced into the tared and stoppered bottle by means of a preheated 10 ml. pipette, the tip of which has been cut off.

Often times the reading of the lower meniscus of the fatty acids is obscured by a layer of organic impurity. A few drops of hot glycerin added just before the reading will separate this layer from the fatty acids and allow a clear reading.

* Pellets of copper wire are useful to prevent "bumping" in the bath.

EXPERIMENTAL

The specific gravity determinations of fatty acids were made in a 10 ml. pyrex pycnometer calibrated at 100°/4°C. The fatty acids were prepared from boiled soap as in the official A.O.C.S. titre test. These were placed in the pycnometer which was immersed in a bath of boiling water until equilibrium was reached. Determinations were made on a number of samples such as tallow soaps of varying titres and tallow-coconut oil soaps having coconut oil up to 50%. The variation in specific gravity of all these fatty acids did not exceed .0010 from the mean value which was .8342 at 100°C./4°C. The maximum error from this source is not more than 1.3 parts per thousand.

Fatty acids were weighed into a test bottle. Sodium chloride, water, and hydrochloric acid were added in amounts to correspond with the conditions of an actual test. Glycerin was added and the test bottle immersed in the boiling bath until equilibrium was obtained. The actual volume of fatty acids was compared with the original weight of the fatty acids divided by their specific gravity at 100°C./4°C. A number of such tests on fatty acids of tallow and tallow-coconut oil soaps showed no discrepancy even in the 1/100 ml. place between the actual volume and the volume calculated from the weight of fatty acids and their specific gravity. Therefore, the solubility of the fatty acids in glycerin under the conditions of the test gives no readable error.

The determination of acid value of the fatty acids was done according to the A.O.C.S. method. When this value is used in converting weight of fatty acid to weight of soap according to the stoichiometric principle of the law of definite proportions, the error introduced is considerably less than one part per thousand. Even if the acid value is arbitrarily assumed from the kind of soap stock used, the error need not exceed one part per thousand.

The only other source of error is a personal one. As in all volumetric work, there is a reading error. However, with care, the reading error for this apparatus should not exceed 1/100 milliters. This is equivalent to a maximum error of three parts per thousand.

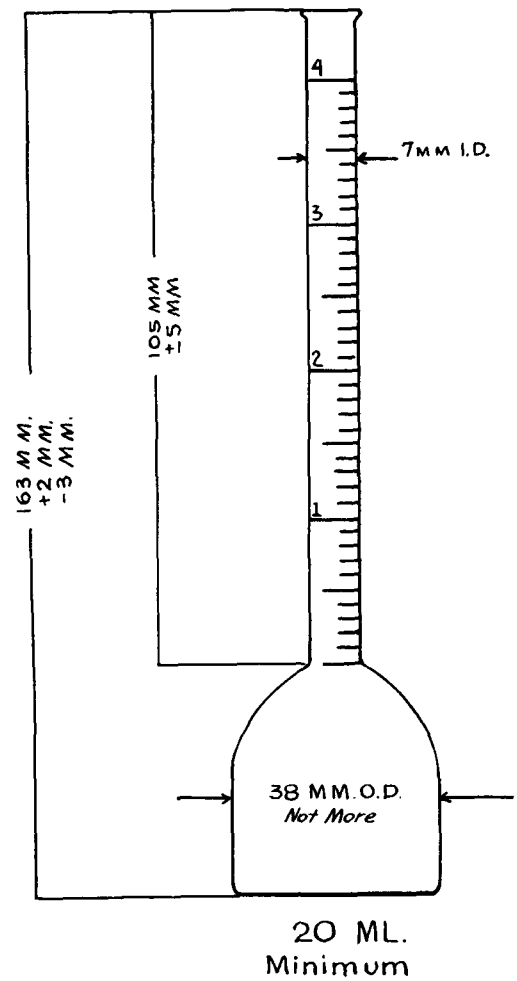
To sum it up, the volumetric method allows a total maximum error of five parts per thousand.

A factor for directly converting volume of fatty acids to weight of anhydrous soap may be obtained for each kind of soap base. First, the "soap-equivalent" factor of one gram of fatty acids must be calculated. For example, one gram of tallow fatty acids is equivalent to 1.082 grams of sodium soap of those fatty acids. It follows then that

$$A \times B = D$$
$$D \times K = C$$
$$\therefore A \times (BK) = C$$

where A = volume of fatty acids at 100°/4°C
B = specific gravity of fatty acids at 100°/4°C
D = weight of fatty acids
C = weight of anhydrous soap
K = "soap-equivalent" factor of fatty acids.

The value BK is the conversion factor and for straight tallow soaps is .902. For the kettle formula 65 parts of tallow to 35 parts coconut oil, the factor is .909. For equal parts of tallow and coconut oil, it is .912.



PYREX GLASS TEST BOTTLE

Furnished by the Scientific Glass Apparatus Co., Bloomfield, N. J., according to the following specifications:

Neck is graduated to 4 ml. in tenths.
Each ml. is a full circle.
Each ½ ml. is a ¾ circle.
Each 1/10 ml. is a ½ circle.
Each bottle is to have an identification number.
Tolerance of graduated portion not to exceed ± 0.03 *ml.*

SUMMARY

The preceding describes a method for obtaining anhydrous soap in an elapsed time of less than a half-hour with a maximum error of less than five parts per thousand. The method agrees well with simultaneous analyses made by the official ether extraction method and with total analyses. The equipment is inexpensive and the technic is simple. The volumetric method is particularly well-suited to process control. Though its use in tallow and coconut oil soaps only has been attempted, the author feels that its scope may be extended to other soap bases.