

value, the two values rising together and confirming, at least for lard, the suggestion of Korpaczny that a Stamm value of 5 defines the boundary between rancid and non-rancid products.

Speaking of oils and fats as a whole, the Kreis test shows better correlation with peroxide value and rancid condition than the Stamm reaction. While this may not be the case with hog fat, it is quite clear in the case of all the vegetable, seed

and marine oils examined. An oxidized or rancid sample gave an intense Kreis reaction and a high peroxide value, but not necessarily a high Stamm value.

SUMMARY:

The Korpaczny modification of the Stamm reaction for detecting rancidity in fats has been applied to a variety of fats and oils, including hog and beet fat, cottonseed oil, a number of other vegetable and seed oils, and a group of marine oils.

This reaction has been shown to be generally inapplicable to vegetable, seed and marine oils but applicable to lard and beef fat.

REFERENCES:

1. J. Stamm—Bull. Soc. of Pharm. (Esthonia) 5, 181 (1925).
2. J. Stamm—Analyst 51, 416-7 (1926).
3. J. Stamm—Pharmacia (1926).
4. J. Stamm—Zeit. Unters. Lebensm. 62, 413 (1931).
5. I. Korpaczny — Kiserletugyi Kozle-menyek 36, 211-15 (1933).
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7. Wheeler—OIL & SOAP 9, 89-97 (1932).

REPORT OF THE SOAP ANALYSIS COMMITTEE 1936

M. L. SHEELY, Chairman

AT THE Fall Meeting of the Society held in Cincinnati October, 1935, the Soap Committee met and made the following recommendations:

1. That the standard methods for soap analysis adopted October, 1933, be made official.
2. That a new method for Volatile Hydrocarbons be adopted as a tentative method.
3. That a method for screen analysis be adopted including both a hand screening and a Ro-Tap procedure as an alternative, both methods to be tentative.

The above recommendations were approved by the Uniform Methods Committee and were officially adopted by the Society at the same meeting. The changes and new procedures are now being printed and will shortly be sent out for incorporation in the Lefax Method book.

During the past year the Committee has not conducted any further cooperative test work. Several discussions have been carried out by letter involving the following tests.

1. Water insoluble in built soaps.
2. Free alkali determination.
3. Moisture in paste soaps containing glycerine.

The discussions on these tests, while not accompanied by collaborative studies, developed some important facts and are consequently recorded in this report as a matter of general interest. In connection with Points No. 2 and No. 3 above, namely, free alkali and moisture determination in paste soaps containing glycerine, the Committee has voted to make minor changes in the present official methods as will be noted later in this report.

Water Insoluble in Built Soaps

One of the Committee members called attention to the fact that the amount of water insoluble in soaps containing high percentages of silicate of soda as determined by the present official method gives higher results than when determined by dissolving the soap directly in hot water. This difference was still greater if the alcohol insoluble, after filtration, is dried at 105° C. and then water insoluble determined thereon. As a confirmation of the above facts one member reported the following data:

pointed out by one of our Committee, namely, that the concentration of soap in the soap solution being filtered influenced results, higher results being obtained with increasing concentrations of soap used. The following data was submitted to substantiate this

TABLE II

Per cent Soap Concentration	Water Insoluble (Soap dissolved direct in the hot water)
1%	2.67%
2½%	4.71%
5%	5.12%

TABLE I—PER CENT OF WATER INSOLUBLE

Sample	Soap Dissolved in 95% Alcohol (Standard Method)	Soap Dissolved Directly in Hot Water
1. Built Flakes (10-30 days old).....	0.52%	0.14%
2. Built Flakes (10-30 days old).....	0.48%	0.22%
3. Built Flakes (10-30 days old).....	0.76%	0.30%
4. Built Flakes (10-30 days old).....	0.18%	0.02%
5. Built Flakes (Very old sample).....	15.40%	12.20%
6. Built Flakes (One day old).....	0.18%	0.00%
7. Built Flakes (One day old).....	0.00%	0.00%
8. Built Flakes (One day old).....	0.00%	0.00%
9. Built Flakes (One day old).....	0.00%	0.00%
10. Built Flakes (One day old).....	0.00%	0.00%
	Standard Method Not drying Alc. Insoluble	Standard Method Drying Alc. Ins. 16 hrs.—105° C.
11. Built Flakes (One day old).....	0.00%	3.00%
12. Built Flakes (Similar to No. 11).....	0.00%	1.34%

The above data indicate definitely that with the exception of very fresh soaps, the standard procedure gives higher results than the direct water method. Suggestions were made to revise the method to use a separate sample of soap and the direct hot water method, but our studies to date indicate that the method is impractical, since the rate of filtration is exceedingly slow. A fritted gooch filter was also suggested to replace the paper filter, but this does not apparently overcome the difficulty.

Another interesting fact was

The sample was an old silicated flake soap showing 5.68 per cent by the Standard Method.

It is evident from the above summarized discussion that various factors must be considered and studied further before any changes can be recommended in the present procedure. Consequently, no action has been taken by the Committee on this test.

Free Alkali Determination

In the cooperative work on this test last year it was found and reported that the present official

method is still the most satisfactory. However, a canvas of the Committee members during the present year developed the fact that practically all laboratories were using a slight modification, namely, bringing the alcoholic filtrate to incipient boiling before titration. A vote was taken and as a consequence, the Committee recommends modifying the method to include this procedure. As a matter of record the method on page 4 will then read as follows:

"Free Alkali or Free Acid. Heat the filtrate from the above nearly to boiling, add 0.5 c.c. of a 1% alcohol solution of phenolphthalein, and titrate with standard acid or alkali solution, and calculate the alkalinity to sodium hydroxide (or potassium hydroxide) or acidity to oleic acid."

Moisture in Paste Soaps Containing Glycerine

Several members of the Committee have pointed out that the oven method on these types of soaps is not satisfactory if accurate results are desired, since more or less glycerine is distilled off, depending upon the length of time the sample is left in the oven. Cooperative work on a sample of this type of soap was carried out several years ago when the distillation method for moisture was shown to be satisfactory. Consequently, the Committee recommends including a note to

this effect in the present method.

For record purposes the preliminary comments to this method on page 3 will be changed to read:

"Moisture. The oven method given below is generally applicable to all soaps. Experience has shown, however, that certain exceptions to this method must be made if accurate results are desired. These exceptions include:

- (a) For soaps containing appreciable amounts of sodium silicate the distillation method is preferred.
- (b) Soaps of linseed and other oxidizing oils absorb oxygen and if the oven method is used may gain in weight near the end of the test. Therefore, either an inert atmosphere or vacuum oven should be used. The distillation method is also applicable to these types of soaps.
- (c) Soaps containing appreciable amounts of glycerine, such as cold made and semi-boiled, (including paste soaps) usually give high results by the oven method. The distillation method is preferred for most accurate results on these types of soaps."

Minor Change in the Method for Rosin

An omission has occurred in the first printing of the Modified Wolff

Method for rosin. On page 6-a, under "Second Esterification" which proceeds as per "First Esterification," the final ether extract is obtained, and ether evaporated on the steam bath. At this point the method should have stated that alcohol should be added and the solution titrated as described. In order to clarify the procedure, the paragraph should read as follows:

"Second Esterification: Cool and dissolve the residue in 20 c.c. of absolute ethyl alcohol and then proceed as above under 'First Esterification.' Add 30 c.c. neutral alcohol (94% or higher) and titrate rosin and rosin soap as desired, using phenolphthalein as indicator."

The remainder of the paragraph remains unchanged.

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ABSTRACTS

Oils and Fats

Edited by

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The spoilage of fats and structural material by heat and light in regard to economy and life. I. H. Schmalfluss, H. Werner and A. Gehrke. *Fette u. Seife*, 43, 211-4 (1936).—A review, the major portion of which deals with authors' research on ketone rancidity.

Does babassu oil menace butter? Anon. *Food Indus.* 8, 617; 653 (1936).—Nowhere near the amt. of babassu oil that would be necessary to put it on a par with coconut oil in volume of consumption is available.

Unified work of D. G. F. I. communication: The determination of unsaponifiable. H. P. Kaufmann. *Fette u. Seifen*, 43, 218-22 (1936).—The petroleum ether, centralburo and English methods for detn. of unsapon. were compared in several laboratories. Conclusion: The petrol. ether method is quick and it yields good results for fats with low unsapon. percentage, however, it is not generally applicable to fats of high unsapon. percentage. Using ether as a solvent it becomes generally applicable, but requires more time and is tedious. It is recommended that the petrol. ether

method be retained for fats with low unsapon. percentage. It was suggested that in further work on the English method that 5 g. samples instead of the usual 2 g. be used. Further work will be on the comparison of the methods on difficulty saponifiable fats contg. small amts. of unsapon.

Hydroxylated acids of fats: An improved method of determination. P. G. Hafner, R. H. Swinney and K. S. West. *J. Biol. Chem.* 116, 691-697 (1936).—The authors present a modified West-Hoagland-Curtis method for detn. of Ac. no., which is claimed to be more convenient and accurate. It has been shown, apparently for the first time, that a number of the common animal and vegetable fats contain small but easily detectable, amts. of hydroxylated acids. Some Ac. values detd. are: Butter 2.7, castor oil 125.6, coconut 1.3, corn 3.6, cottonseed 4.4, lard 1.5, linseed raw 5.3, linseed boiled 7.9, neat's-foot 9.0, olive 3.1, peanut 2.6, and salmon 3.7.

Testing edible oils. H. Jesser and E. Thomas. *Angew. Chem.* 49, 846-7 (1936).—Color reactions of 23 oils were obtained by the 3 following procedures: