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Report of the Soap Analysis Committee

THE Soap Analysis Committee was organized as a separate committee of the Soap Section early in 1930. Until the present time the official soap analysis methods of the American Chemical Society have been in general use by all soap manufacturers and consumers since they were originally adopted in 1919 and revised in 1922. When the Soap Analysis Committee of this so-

ciety was organized, it was felt that several of these methods needed further study with a view to revising them to fit the requirements of the industry. To summarize briefly, their work has been confined to studies of several of these methods which have generally given trouble in the past. Among the methods studied by this committee two have been revised and recommended for tentative adoption by the society. The first of these was the method for unsaponified and unsaponifiable matter

*Presented at the meeting of the American Oil Chemists' Society Oct. 12, 1933.

in soap (SOAP, 7, 29 (1931)). This was recommended as a tentative method a year ago and is now ready for official adoption by the society replacing the original A. C. S. method. The second method studied was that covering moisture content in highly filled soaps and this was also recommended for tentative adoption a year ago and is also in order for adoption as an official method.

Until the present time no action has ever been taken by our society towards adopting a complete set of soap analysis methods which might be considered standard or official. A meeting of the Soap Analysis Committee was held on October 11th and the following plan of action was voted on and is now recommended for action by the society.

1. That we adopt the present official A. C. S. Soap Methods (Ind. Eng. Chem., 14, 1159, [1922]) as the tentative standard methods of this society with the exception of the two methods previously mentioned, namely the unsaponified and unsaponifiable matter and moisture in highly filled soaps. It is proposed that the A. C. S. methods with these exceptions be published as our standard methods but in each case where the individual method has not been studied by this committee to designate it as a tentative method only.

Due to general conditions in most laboratories, very little cooperative work has been possible during the past year. However, some work has been done by a sub-committee of the F. A. C. which happened to consist of three members of the soap committee which bears directly upon the program of the Soap Analysis Committee. Several years ago the F. A. C. Committee initiated a study of the various methods for the determination of rosin in fatty acid. This work has been continued this year by this sub-committee of the F. A. C. but Mr. Irwin, who is chairman of that committee, has recommended that the work be carried to a conclusion by the Soap Committee, inasmuch as this test is principally used by the soap industry. Accordingly samples containing various amounts of rosin were sent to the three designated laboratories for study. The methods tried included the German official, volumetric and gravimetric methods, the Twitchell method, and the Wolff method. The preliminary figures on this cooperative work are very encouraging. Incidentally, the German official methods follow very closely the Wolff method for rosin determination but employ methanol instead of ethanol as the esterifying medium. Also it includes a double esterification process in order to eliminate the last traces of unesterified fatty acid. In our study of this method it was found that this double esterification is very important and gives indication of being a solution to the rosin determination. With this as a background, it was felt that the Wolff method, which is a single esterification method, could be revised to give as accurate results as the German method. Consequently, this method was modified to include a double esterification procedure and the results of the three laboratories on the cooperative samples indicate fairly close agreement especially on the samples containing high rosin content. For example, on the sample containing 25 per cent added rosin there was found by the three collaborators 24.3, 24.9 and 25.0 per cent rosin or an average of 24.7 per cent. On the lower percentages of rosin two laboratories show close agreement but the third laboratory appears to be appreciably out of line, although it is considered that some fault in the technique or reagents is responsible for this discrepancy. Incidentally, all collaborators report a blank of approximately one per cent on the sample containing no rosin indicating the limitation of the method on low rosin contents. This was true with all four methods studied. Consequently, a note to this effect

ROSIN COOPERATIVE WORK

	WOLFF METHOD							
	0.0% Rosin		5% Rosin		10% Rosin		25% Rosin	
	1 Est.	2 Est.	1 Est.	2 Est.	1 Est.	2 Est.	1 Est.	2 Est.
Lab. 1	5.2	1.0	10.7	5.2	15.3	10.1	30.0	24.3
Lab. 2	3.3	1.1	9.0	5.7	12.6	10.3	29.1	24.9
Lab. 3	3.3	3.5	8.1	8.0	12.9	10.6	26.2	23.8

	GERMAN METHOD—VOLUMETRIC							
	0.9*		1.1		5.5*		9.3	
	1 Est.	2 Est.	1 Est.	2 Est.	1 Est.	2 Est.	1 Est.	2 Est.
Lab. 1	0.9*	1.1	5.5*	5.2	10.5*	9.3	24.9*	23.3
Lab. 2	2.2	1.0	5.7	5.3	12.5	9.9	25.1	23.7
Lab. 3

*Single esterification. Before calculating to rosin 1.5% was subtracted from the rosin acids. This was to compensate for unesterified fatty acids.

	GERMAN METHOD—GRAVIMETRIC			
	0.4	4.0	7.5	19.0
Lab. 1	0.4	4.0	7.5	19.0
Lab. 2	1.0	5.4	10.2	24.7
Lab. 3	0.8	4.3	8.6	22.7

	TWITCHELL METHOD—GRAVIMETRIC			
	2.3	6.8	11.7	26.1
Lab. 1	2.3	6.8	11.7	26.1
Lab. 2	1.0	5.7	10.1	24.9
Lab. 3	1.9	6.0	10.9	24.7

Sap. No. Rosin	177.18
Unsaponifiable	3.35
Mol. Wt. Rosin	316.63
Mol. Wt. Rosin Acids	327.6
Factor for N/2	1638
Factor Rosin Acid to Rosin	103.4

NOTE: Factor 1638 was used in Wolff and German and Volumetric Methods.

Factor 107. is commonly used for calculating rosin acids to rosin. However, all above figures are based on 103.4.

will necessarily have to be included with any method adopted as well as a warning to check any figure of this order by means of the qualitative test for rosin. The detailed results of the cooperative work on these methods are published as a part of this report.

Since the Wolff method as it now stands gives very erratic results, it is the recommendation of the committee that the method be adopted as a tentative method in modified form, namely, to include a double esterification procedure. Further study during the coming year should establish the accuracy of the proposed method before final adoption is considered.

Summarizing, we wish to recommend:

- (1) Official adoption of the present A. C. S. methods as tentative methods of the American Oil Chemists' Society with the following exceptions:
 - (a) Unsaponified and unsaponifiable matter as an official method.
 - (b) Distillation moisture in highly filled soaps, as an official method.
 - (c) Rosin determination as a tentative method by the double esterification procedure. (Wolff method, modified.)
 - (d) Several minor changes in the A. C. S. methods to conform to general practice. These to include such items as size of sample taken for analysis change in regular oven method for moisture determination and temperature of digestion of sample in glycerol determination.
- (2) That a further study be undertaken of the rosin determination to improve the accuracy of this test.
- (3) That further studies be undertaken during the coming year to include free alkali in soap since this is a very important test both in the manufacture and use of soap and soap products.

With the above program in effect, it is hoped that from time to time revision can be made of the various methods to keep them in line with good commercial practice.

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