Determination of Detergency of Soap Products

A Progress Report of the Work to Date by the Subcommittee of the American Oil Chemists Society

THE formation of a subcommittee of the A. O. C. S. on Determination of Detergency was one of the results of the annual meeting of the society in May, 1925. The urgent need of standard methods to evaluate detergents was admitted by all of the members of the original Detergents Committee and was stressed by the Chairman, Mr. Campbell, in his 1925 report.

A meeting of the subcommittee was held at the Bureau of Standards, Washington, D. C., on September 28, 1925, at which ten members were present. The membership was subsequently increased and the personnel of this subcommittee is now shown on the page following.

At the Washington meeting it was the consensus of opinion that the detergent value of soaps and soap products was of so complex a nature and involved so many factors and variables that it could not be properly judged by criteria such as lathering power, surface tension or other physical measurements.

An actual washing test of some sort was considered to be the ultimate test of detergency, although it was admitted by all that the difficulties in developing a satisfactory washing test would be enormous.

After discussion of various washing test procedures the committee decided that the laboratory washing test and machine described by A. K. Church of Lever Bros. Co., offered the best possibilities from the standpoint of simplicity, cost and small size of samples involved.

From the beginning, the need of standard methods to evaluate detergency has been before the American Oil Chemists Society. This paper constitutes, in a sense, a history of the detergency movement. And although the results to date have been somewhat contradictory and inconclusive, the society may justifiably take pride in the work of its subcommittee. Mr. Hoyt's report will be supplemented by comments of collaborators in Oils and Fats for February.

The problem of preparing secondary standards for quantitatively recording color values of the soiled and washed clothes was undertaken by Dr. P. H. Walker of the Bureau of Standards. Dr. Walker in collaboration with his associate, Mr. Bruce, worked out a series of gray pastes, of uniform gradations of color sensation, made with mixtures of zinc oxide, carbon black, and white mineral oil. The care, time, and labor involved in preparing such secondary standards is very great, and for the use of the large personnel of the committee arrangements were made with the Munsell Color Co., of Baltimore, to furnish at small cost sets of standard gray papers approximating closely to the gradations of gray pastes that

had been prepared by Dr. Walker.

In addition to the pastes, Dr. Walker and Mr. Bruce subsequently worked out a graded series of twelve cloths soiled with definite increments of India ink, as a possible secondary standard. Sets of 4 of these cloths of the shades most likely to be needed were prepared by the chairman according to directions furnished by Dr. Walker, and sent to each member of the committee. (In practice it is somewhat difficult to compare color of a woven fabric with the matte surface of a gray paper or paste and standard gray cloths are theoretically desirable. The chroma of the "India ink" cloths was so different from that of the bluish-gray soiled and washed cloths that these particular "India ink" cloths were generally not satisfactory.

Members of the Committee

Edward Bauer, Kirk & Co., Chicago, Ill.

H. C. Bennett, Los Angeles Soap Co., Los Angeles, Calif.

J. S. Boulden, Wm. Waltke & Co., St. Louis, Mo.

Dr. J. C. Brier, Prof. Chem. Eng., University of Mich., Ann Arbor, Mich.

R. K. Brodie, Chem. Supt., Procter & Gamble Co., Ivorydale, O.

V. K. Cassady, Chief Chemist, Palmolive Co., Milwaukee, Wis.

A. K. Church, Chief Chemist, Lever Bros., Cambridge, Mass.

R. E. Divine, 284 Beach St., Hackensack, N. J.

Dr. J. S. Goldbaum, Chief Chemist, Fels & Co., Phila., Pa.

F. H. Guernsey, Chief Chemist, Cowles Detergent Co., Lockport, N. Y.

Dr. M. H. Ittner, Chief Chemist, Colgate & Co., New York, N. Y.

G. H. Johnson, Director of Research, Laundryowners National Assoc., Mellon Institute, Pittsburgh, Pa.

Frederick Kenney, City of New York Board of Purchase, Central Testing Laboratory, New York, N. Y.

C. P. Long, Globe Soap Co., St. Bernard, Ohio.

E. T. Marceau, Chem. Director, Gold Dust Corp., New York, N. Y.

Prof. E. B. Millard, Mass. Institute of Technology, Cambridge, Mass.

H. S. Mitchell, Swift & Co., Chicago, Ill.

J. R. Powell, Chief Chemist, Armour Soap Works, 1355 West 31st St., Chicago, Ill.

W. S. Rapelje, Kirkman's & Son, Brooklyn, N. Y.

W. T. Reese, Chief Chemist, Peet Bros., Kansas City, Kansas.

J. G. Vail, Chemical Director, Philadlephia Quartz Co., Phila., Pa.

F. W. Smither, Bureau of Standards, Washington, D. C.

Dr. P. H. Walker, Bureau of Standards, Washington, D. C.

L. F. Hoyt, Larkin Co., Inc., Buffalo, N. Y., Chairman.

The manufacture of the minature washing machine, of slightly modified design, was undertaken by R. K. Brodie, Chemical Supt. of the Proctor and Gamble Co. Thru the efforts of Mr. Brodie a very satisfactory machine was made available to members of the committee at the low cost of $15.00.

The program agreed upon at the Washington meeting was restricted

primarily to a study of the tentative washing test procedure itself to determine its reliability in the hands of a number of investigators, and called for comparative washing tests at 100°, 122°, and 160°F in distilled water of two soaps of widely different composition.

There was considerable and unavoidable delay in getting started on this year's program, the first of its kind. Late in February members of the committee received from the Chairman the collection of materials for this year's program, including the Munsell and "India ink" cloth color scales. The Detergency Set included a 60-yd. roll of 4-inch cotton sheeting; lampblack, edible tallow and lubricating oil for the soiling mixtures; and samples of a granular tallow soap and of powdered olive castile soap. Ten members of the committee received sets of standard gray pastes from Dr. Walker for trial. Washing machines were supplied to members by Mr. Brodie, on order.

Directions for soiling and washing, as supplied to members of the committee were as follows:—

A. Procedure for preparing soiled cloths.

The cotton sheeting contains considerable starch sizing, which must be removed before uniform soiling can be attained. This can be accomplished as follows: cut the sheeting into convenient lengths and boil for about 5 minutes in dilute HCl, (10 cc. 1.20 HCl per liter), rinse, partially dry and iron out flat.

Experiments have indicated that it is not feasible to rigidly fix the amount of lampblack to be used in the soiling mixture since variation in technique of soiling will give different results with the same solution.

Soiling Mixture:—
5 grams Lubricating Oil
3 grams Edible Tallow
x grams Lampblack (x = 2 to 3)
2000 cc. Carbon Tetrachloride

Dissolve the oil, tallow, and lampblack in 1 liter of CCl_4 in a 2½ liter glass stoppered bottle (or a 2 quart jar provided with rubber ring and cover, if more convenient), shaking thoroly for 5 minutes. To the mixture add 1 liter of CCl_4 and filter rapidly thru several thicknesses of washed sheeting. Preserve the soiling solution in a closed container.

Pass a continuous strip of sheeting, of any convenient length, from which sizing has been removed as above described, through the soiling solution in a tray or dish and then thru a wringer. Dry the cloth rapidly with the aid of an electric fan or suspend for a few minutes in a drying chamber, and when dry press out flat with a warm iron.

This process should result in securing an even soil on the cloth and sufficient lampblack should be used (the amount can be determined only by trial) to produce a shade which will match the S48 of the Munsell gray scale.

Caution: Even with the standard soiling mixture containing no readily oxidizable constituents there is evidence that soiled cloths several days old do not wash out as white as freshly soiled cloths.

Therefore in this year's program, to avoid possible differences which might be caused by using soiled cloths of indefinite age, it has been considered advisable to specify that all washing tests should be run on soiled cloths not over one day old.

B. Procedure for washing test.

Connect up the washing machine to any convenient power source so that the wash wheel will have a speed of 250 r.p.m. with load. Provide a gas burner to maintain the desired degree of heat in the washing machine during the test.

Dissolve 5.00 grams of a standard soap sample, as received, in 500 cc. of distilled water, using heat, if necessary. Dilute to 1 liter with distilled water and adjust to the desired temperature. Fasten a strip of soiled cloth, shade S48, on the wash wheel, pour the soap solution rapidly into the machine, run for 5 minutes at 250 r.p.m. maintaining the temperature of the solution at one of the definite prescribed temperatures. Stop the machine and drain off the soap solution. Add 1 liter of rinse water (distilled water), run the wash wheel for 1 minute and drain. Repeat the rinsing twice more. Remove cloth and when nearly dry press with a warm iron, fold to four thicknesses and record its color value on the Munsell gray paper scale. Repeat the entire washing and rinsing process as above *on the same cloth* and record the color value after the third and fifth washing.

Note:—It should be distinctly understood that these washing test directions are to be considered *not* as final or standard, but rather as tentative directions subject to modification and improvement as the result of collaborative work.

Analyses of the Soaps used in this year's program as made by Messrs. Smither and Bower of the Bureau of Standards showed the composition given below.

Color	Powdered castile soap White	Granular tallow soap Light green
Volatile at 105° C.	2.5%	2.3%
Water Insoluble	0.1%	1.6%
Alcohol insoluable	1.2%	7.3% (5.8% Na_2CO_3, balance silicate of soda)
Sodium chloride	2.4%	0.8%
Anhydrous* soap, by difference	93.8%	88.0%
Free alkali	0.0%	0.0%
Free fatty acid	0.0%	0.0%
Titer of fatty acids	19.8° C.	40.2° C.

Reports of results were received from the committee and associates:

Mr. Boulden, Wm. Waltke & Co.

Mr. Brodie and Dr. Preston, Proctor and Gamble Co.

Mr. Cassady, Palmolive Co.

Mr. Church and Mr. Boucher, Lever Bros.

Mr. Guernsey, Cowles Detergent Co.

Dr. Millard and Dr. Ashdown, Mass., Institute of Technology.

Mr. Mitchell, Swift & Co.

Mr. Powell, Armour & Co.

Mr. Reese, Peet Bros.

Mr. Smither and Mr. Bower, Bureau of Standards.

Mr. Vail and Mr. Carter, Philadelphia Quartz Co.

Mr. Hoyt, Larkin Co., Inc.

The time available for carrying

* Note: This value would include the small amount of glycerine present, for which no separate analysis was made.

out the program was limited and probably prevented more complete returns.

Comments and results of collaborators are as shown in the tables below and on the following pages.

A. O. C. S. Detergency Test Results 1926

Tallow Soap

(Munsell Paper Scale Readings)

Conditions of Test	1	2	3	4	5	6	7	8	9	10	11	12
Temp. 100° F.												
After 1st Wash	64	60	55	60	64	60	64	52	64	55	64	64
" 3rd "	67	64	55	64	71	67	67	67	67	60 —	67	71
" 5th "	71	64	55 +	64	76	71	71	71	67	60 +	73	76
Temp. 122° F.												
After 1st Wash	67	64	55	60	64	60	64	60	64	60 —	64	64
" 3rd "	71	67	55	64	71	71	67	71	67	60	71	71
" 5th "	71	67	55 +	64	76	76	71	76	67	64 —	76	76
Temp. 160° F.												
After 1st Wash	64	64	55 +	60	64	64	64	55	64	60 —	64	64
" 3rd "	71	67	60	64	67	71	71	64	67	60 +	71	67–71
" 5th "	71	67	60 +	67	71	80	76	71	67	64	76	76–80

A. O. C. S. Detergency Test Results 1926

Olive Castile Soap

(Munsell Paper Scale Readings)

Conditions of Test	1	2	3	4	5	6	7	8	9	10	11	12
Temp. 100° F.												
After 1st Wash	64	60	52	67	64	60	60	60	60	55 +	60	64
" 3rd "	71	64	52	67	67	67	64	67	64	60 —	67	67–71
" 5th "	71	67	55	71	71	67	67	76	67	60	71	76
Temp. 122° F.												
After 1st Wash	64	64	48 +	60	64	60	60	60	64	60 —	64	64
" 3rd "	71	64	52 —	64	71	64	64	64	67	60	71	67
" 5th "	71	67	52	67	76	67	67	67	67	60	76	71
Temp. 160 ° F.												
After 1st Wash	67	64	55	60	64	64	64	60	64	60 —	64	64
" 3rd "	71	67	55 +	64	71	67	67	67	67	60	71	67–71
" 5th "	71	67	55	67	76	71	67	71	67	60	76	76

A. O. C. S. Detergency Test Results 1926

Tallow Soap

Gray Pastes Readings

Conditions of Test	1	2	3	4	5
Note: Color of cloth at start of all tests reported as matching					
Paste No.	8	9	10	10	10
Temp. 100° F.					
After 1st Wash	6	5	5–6	5	5
" 3rd "	5 —	3	4	4	4
" 5th "	5	3	3	4	3
Temp. 122° F.					
After 1st Wash	5 —	5	5	5	5
" 3rd "	5	3	4	4	4
" 5th "	5	3	3	4	3
Temp. 160° F.					
After 1st Wash	5	4	5	5	5
" 3rd "	4	3	4	4	4–3
" 5th "	4	3	3	4	3–2

A. O. C. S. Detergency Test Results 1926

Olive Castile Soap

Gray Pastes Readings

Conditions of Test	1	2	3	4	5
Note: Color of cloth at start of all tests reported as matching					
Paste No.	8	9	10	10	10
Temp. 100° F.					
After 1st Wash	7	5	5–6	5	5
" 3rd "	6	3	5	5	4
" 5th "	6	3	5	4	3
Temp. 122° F.					
After 1st Wash	7	5	5–6	5	5–4
" 3rd "	7	3	5	4	4
" 5th "	6	3	5	4	3
Temp. 760° F.					
After 1st Wash	6	4	5–6	5	5
" 3rd "	5	3	4	4	4
" 5th "	6	3	4	4	3

This paper will be concluded in the February number of OILS AND FATS.

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a large bulk sample, and if the facilities are not available the figures submitted, whether from the pressed or extracted oil will have very little value as a reliable or just basis of consideration. It must be perfectly obvious, that to correctly represent a thousand or so tons of any material like copra in a 10 gram sample, it must be remarkably uniform, and to obtain this, must be prepared with all the precautions and care set forth, or else the results will be misleading.

David Wesson left for New Orleans, January 8. He will be gone a month, lecturing at several Agricultural Colleges in the South on Cottonseed and its Products.