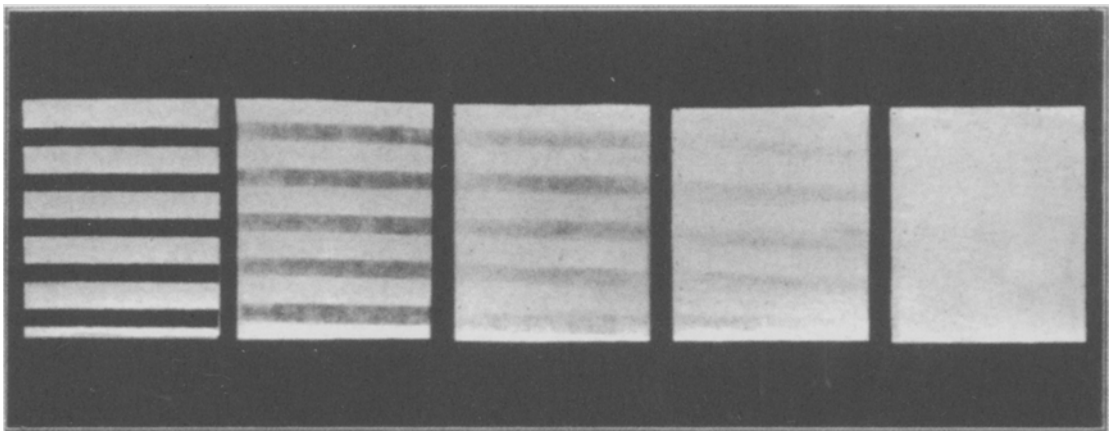
Determination of Detergency of Soap Products

A Progress Report by the Detergents Sub-Committee of the American Oil Chemists Society

By L. T. HOWELLS, *Chairman.*



Photographic record of results of progressive washings in Standard Detergency Test

THE report on the work accomplished by the Detergents Committee in 1926 (L. F. Hoyt, Journ. Oil & Fat Ind. Jan. & Feb. 1927) clearly indicated that the washing device and methods used in soiling and evaluating results were unsatisfactory. However, the work was essential because the difficulties encountered in desizing, soiling, washing and comparing results could not otherwise be realized in view of the many complexes involved. It was evident from the results that a uniform standard soil applied at one source would eliminate many of the difficulties and a change in the principle of the laboratory machine and method of evaluating results was necessary in order to justify further work in solving the problem before the committee.

In the preliminary report of the work accomplished by the committee in 1927 (Guernsey & Howells, Journ. Oil & Fat Ind. Nov. 1927) the idea of using a standard soiled cloth which would take care of both soiling and evaluating problems was advanced. A quantity of cloth was uniformly soiled by printing a design upon the fabric, using a printing paste composed of gum tragacanth, starch, mineral oil, tallow and lamp black. The number of washes necessary to remove all traces of the soil (contrast with the original unsoiled areas) was proposed as an index of detergency. Practical washing tests in standard laundry machines proved satisfactory and it became evident that a laboratory machine approaching the efficiency of a standard laundry machine was essential if tests were to yield useful results.

The work in 1927 (F. H. Guernsey, *Chairman*) was brought to a standstill because a suitable laboratory machine was not available and the members of the committee, who received samples of the cloth, were asked to submit suggestions for a more efficient machine.

Detergents Committee—1928

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

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

J. G. Vail, Philadelphia Quartz Co., Philadelphia, Penna.

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

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

W. D. Appel, Bureau of Standards, Washington, D. C.

W. C. Preston, Procter & Gamble Co., Ivorydale, Ohio.

W. H. Burkhardt, Gold Dust Corp., Baltimore, Md.

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

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

M. L. Sheely, Armour Soap Works, Chicago, Ill.

F. H. Rhodes, Cornell University, Ithaca, N. Y.

C. J. Post, National Oil Products Co., Harrison, N. J.

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

Foster D. Snell, Pratt Institute, Brooklyn, N. Y.

L. T. Howells, *Chairman,* Cowles Detergent Co., Cleveland, Ohio.

In the early part of 1928, two machines were proposed and a meeting was called at the Bureau of Standards, April 14th, for the purpose of discussing the proposed cloth, the most suitable machine and tests to be conducted.

The following is a report on the meeting:

Soiled Cloth: It was agreed that the last traces of soil are hardest to remove and that the number of washes required to completely remove the soil is an index of detergency worthy of trial. The following points were cited in favour of the proposed cloth:

1. Mechanically soiled. (Preferably at one source) Personal equation is largely eliminated.
2. Can be applied on any kind of material.
3. Affords a simple means of measuring detergency. Delicate instruments not required.
4. Soil can be any specific dirt or mixture.
5. The cloth has practical possibilities.

To what extent aging will affect the soil is one of the problems to be studied after the method has been tested. Samples of washed cloth were exhibited. Mr. Appel furnished samples washed in the proposed machine and the chairman showed samples washed in a commercial laundry.

It was decided that in testing the method the experiments should be confined to one soil, such as lamp black, instead of complicating matters by adding kaolin, umber, rouge or other proposed soils. The only change to be made at present in the soiling mixture is the substitution of colourless Nujol for the A.O.C.S. mineral oil. The lamp black is to be No. 14, manufactured by Seaver & Company of Boston, a supply of which will be purchased by the chairman.

The composition of soil after drying will be:

Wheat Starch	56.82
Gum Tragacanth	3.41
Tallow (A.O.C.S.)	11.36
Nujol Oil	11.36
Lamp black	17.04
	99.99

For testing the method, the chairman will furnish two yards of the soiled cloth for each member.

Machine: It was agreed that last year's machine was unsuitable for this work. A small machine resembling a commercial wheel was exhibited by the chairman.

The merits of Mr. Appel's machine were very readily noted. It consists essentially of a battery of 20 pint jars revolving at a definite speed in a water bath which is kept at constant temperature. In each jar 15 rubber balls (d-3.2) are placed with a piece of standard soiled cloth three inches square in 100 cc. of solution. The speed of the jars should be in the neighborhood of 52 R.P.M. to insure greatest action.

Mr. Appel gave a talk on what he had accomplished in washing tests and gave the members present a photograph and blue print of the machine and a photograph showing the results of washing the cloth with cubes and balls. The tests proved that the balls were more effective.

The Research Committee of the American Association Textile Chemists and Colorists are planning to have several of these machines made. If the Detergents Committee have machines made up jointly with them the price will undoubtedly be much lower. The machines are not to be equipped with a driving mechanism or motor by the manufacturer. It will be up to the individuals to adjust the machines to the specified R.P.M. All members at the meeting signified that they would purchase one of these machines, providing the price was not unreasonable. The rubber balls will be furnished by Dr. Walker.

Tests: The following represents the approximate conditions under which the method is to be tested:

1. The soap will be furnished by the Chairman. It will have a titre of about 38 degrees. The percentage is not to be calculated on a bone-dry basis. The strength of the solution is to be .2% soap (soap as received taken as 100%).
2. The cloth samples are to be three inches square and cut in the same manner as a sample which will accompany the cloth.
3. A twenty-minute suds followed by two five-minute rinses (all at 160° F.) will constitute one wash. Distilled water is to be used throughout and all solutions are to be heated to temperature before use. The volume of solution in all cases will be 100 cc.
4. In each jar 15 balls are to be placed together with the cloth and 100 cc. of solution.
5. In conducting the test a piece of the cloth is placed in each of 10 jars. At the end of each wash one of the pieces is removed. After drying and ironing the samples are to be mounted on black cardboard. The number of washings corresponding to the piece which no

longer shows any visible trace of the soil is to be recorded as the index of detergency.

6. A duplicate test will be independently run within three days. To check up on aging, it is very important that the date of both runs be recorded.

The above procedure under uniform conditions will undoubtedly test this proposed method and if found favourable a more elaborate and practical set of experiments will follow.

Following this meeting W. D. Appel provided the members with a blue-print of the proposed machine and we are quoting below his specifications:

"The standard washing machine shall consist of a shaft which can be rotated at a constant speed of 42-3 R.P.M. Firmly attached to the shaft and rotating with it are one-pint glass topped Atlas E-Z Seal preserve jars in which the sample and wash solution are placed. The jars are 6 inches in height, 3¼ inches in diameter, and have a capacity of 1.2 pints. They are arranged radially about the shaft, the base of the jar being 2 inches from the center of the shaft. Provision shall be made for maintaining the temperature of the wash solution to within 2° F. of that specified for a given test."

The work in testing the method was held up because the members, at the meeting, decided to consider a machine being manufactured by the Atlas Co., for the American Textile Chemists. The machine known as the Launder-Ometer met Mr. Appel's specifications and sufficient members obtained machines to test the method. The necessary supplies (soiled cloth and soap) were put in the hands of the members having machines by the chairman and the following test was conducted.

The Conditions Governing Test

1. Distilled water shall be used thruout, and all solutions shall be heated to temperature before use. (150° F)

2. The strength of suds solutions shall be .2%. The percentage is not to be calculated on a bone-dry basis (soap as received taken as 100%).

3. The test pieces shall be 2½" x 4", and cut in the same manner as the sample received.

4. In each jar 10 rubber spheres, 1 cm. in diameter and weighing approximately 1.5 grms. shall be placed together with the cloth in suds solution.

5. A 20 minute suds, followed by two 5 minute rinses, (all kept constant at 150° F) shall constitute one wash. The volume of the suds bath shall be 100 cc. and the volume of the rinses 200 cc.

6. In conducting the test, a piece of test cloth shall be placed in each jar as specified above, and at the end of every wash, one of the pieces shall be removed. After drying and ironing, the test pieces shall be mounted on black cardboard in the order washed. The number of washings corresponding to the piece which no longer shows any visible trace of the soil shall be recorded as the index of detergency.

7. The test shall be run without interruption (finished the same day as started).

8. A duplicate test shall be independently run within three days. To check up on aging, it is very important that the date of both runs be recorded. Members of the Committee are requested to keep mounted samples for comparison at a meeting scheduled to follow tests.

Results of Tests:

IN determining the wash at which all traces of the stripe were removed the background and light reflection greatly influenced the readings. When mounted on black cardboard as instructed in the proposed method the results of the collaborators, who took part checked well. The majority of readings were between 4 and 5 washes and in most cases the workers checked themselves in both tests outlined. However, when the same cloth samples were placed on a white background and shaded from direct light outlines of the stripes could be seen up to 15-16 washes in cases where that many washes were made.

The following comments were taken from reports received by the chairman:

* * *

1. We have washed the soiled cloth in accordance with the instructions given in your letter of February 26.

2. Most of the soil is removed in the first wash. After four washes the soil may be said to be completely removed although under certain conditions of illumination streaks still can be made out. This is true however even after nine washes.

3. The streaks are easy to see when one knows where to expect them. An irregular pattern would be better than the present one.

4. These streaks appear to be attributable to a difference in the cloth where the soil was applied rather than to soil remaining in it.

* * *

When the samples were shuffled and viewed against the white background thru the hole in the black cardboard, shaded as you suggested, the marks could be identified on all the samples up to and including No. 15. No marks could be detected on No. 16 but a stripe was visible on No. 17.

This seems to indicate that a perfectly definite end point will be hard to arrive at, but

it may be that by increasing mechanical action, results more like those obtained by you in the commercial laundry can be secured and in this case the steps might be expected to be long enough so that the end point could be established with a satisfactory degree of concordance between different tests or different workers.

We shall await your further advices before doing additional work along this line, but suggest for your consideration the use of some sort of heavier ball and a greater number of them in each jar.

* * *

We encountered some difficulty in selecting the test piece which did not show any visible trace of the soil. We found it necessary to have a number of men make the selection, and took as a final result the conclusions reached by a majority. This conclusion was that the index of detergency in the work as conducted in our laboratory would be seven; that is, the seventh test piece was the first one which did not show any traces of the lines of soil.

* * *

TEST "A"—3/11/29: Looking at the darkest samples from Test "A," we find that after 3 washes a trace of the soil still remains but that practically nothing can be seen on the piece washed 4 times. In the case of the lighter samples from Test "A," nothing can be seen on the piece washed 3 times.

TEST "B"—3/14/29: Looking at the darkest samples we find a faint suggestion of soil remaining after 4 washes, while on comparing the lighter samples from this wash we find only a faint suggestion after 3 washes, and nothing after 4 washes.

TEST "C"—3/18/29: Looking at the darkest samples we find a trace still remains after 4 washes, and after 5 and 6 washes a suggestion of the line is still present if the cloth is examined extremely carefully. Looking at the lighter samples we find practically no soil left after 3 washes, and a mere suggestion after 4 washes, and nothing after 5 or 6 washes.

I am inclined to consider the results of this test unsatisfactory for the reason that the soil was too easily removed, most of it being removed in the first wash; and after the third wash only a trace remained. Furthermore, the soil is not evenly removed, that is, a piece washed in one jar may be a little lighter than a piece washed in another jar. This result may be due to unevenness in the printing of the soil on the cloth, or an unequal mechanical action in the jars. If all the soil washes out after three or four washes, it appears to us that the detergent efficiency of different soaps could not be graded closely enough to be of the practical value contemplated, especially if the same soap will, in one test, wash the fabric clean in four washes and in another test wash it clean in three washes. However, we understand that the main purpose of this series of tests is to determine whether the machine itself, that is the Launder-Ometer type, is a machine of satisfactory type to use in future work on detergency. We have used our Launder-Ometer considerably and while certain improvements are indicated, we feel that it is a satisfactory type of machine for future work on detergency —that is, until a better is devised. For example, the Launder-Ometer permits the running of 20 tests at once under identical conditions and this is a decided advantage.

The objections to the machine which we have noticed are as follows:

The sample piece shows a tendency to stick to the inside of the cover, allowing the balls to beat on it excessively, thus tending to produce uneven washing. This was especially noticeable when using soiled cotton sheeting and steel balls. The washed test pieces were so uneven that these particular tests had to be discarded. Rubber balls give a more even soil removal; also the thinner fabrics wash more evenly. When conducting tests at 150° F and over, it is difficult to keep the jars tight, that is, the solution tends to leak out of the jar. At 170°F, this leaking is very troublesome. At times we have found some jars entirely empty at the end of the wash, and other jars containing 300 or 400 cc of water starting with 100cc. This trouble occurs even when new rubber rings are used and the cover apparently clamped down tightly. The clips which hold the jars in place are easily bent out of shape and should be made stronger. The inside of the water bath of our Launder-Ometer is beginning to rust. We have little fault to find, on the whole, with the construction of the Launder-Ometer made by the Atlas Electric Devices Co. except as mentioned above.

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The number of washes necessary in each case to remove the soil was four when test pieces were mounted on black cardboard and five when observed over a white background. There was a lack of uniformity in results between the two tests, e. g. one wash in test number one did not remove as much soil as in test number two and the same effect was observed after two washes. (In test number two the soil was more completely removed on the test piece which was washed twice then on the one that had had three washes.)

We have run the tests as nearly as possible under the conditions specified. Our temperatures probably did not vary from 150° F. by more than 2 degrees, but the speed of rotation of our machine, which was not purchased from the Atlas Co., but was made in our own shops, proved to be 59 R. P. M. This is, of course, higher than it should be but could not be remedied. Comparison with the results of other members will show whether this had any effect.

Our two tests, run on March 6 and 8 respectively, check each other very well, but we cannot say that we are optimistic over the prospect of differentiating between soaps by this procedure. It is difficult to decide just when the black stripe has been completely washed out. We should consider nine washes to be needed for complete whiteness but we fear that the personal element is considerable in this judgment, and that it will be precarious to judge between soaps A and B when with soap A alone it is so hard to tell whether six, eight, or ten washes are required for complete whiteness. Unfortunately we have little constructive criticism, other than to repeat our opinion that the degree of whiteness resulting from one or two washes is a better criterion of detergent power than is an attempt to determine the number of washes required for complete whiteness. We believe that a visual comparison of cloths washed once with each of two soaps would show the difference, if any, between the soaps better than would a comparison after nine or ten washes. We report nine washes as being needed for complete whiteness, and we shall be interested to hear the findings of other members.

* * *

As instructed in the direction for testing the proposed method of detergency, we cannot see any trace of the design on the 5th wash when the pieces are mounted on black cardboard and viewed in direct light. Our tests were run March 19th and 21st and both tests check at the 5th wash. However, when the samples are viewed against a white background thru a hole in black carboard and shaded from direct light, the stripes can be identified up to and including the 13th but cannot be seen on the 14th to 20th. Ten individuals read the same test pieces and it was found that those trained in judging laundry quality could detect traces that were not caught by the untrained and altho the readings of individuals differed it was found in all cases each individual was able to check himself on shuffled samples.

The tests have clearly shown that it is the very last traces of soil that are hardest to remove, and that the percentage of soil removed in the beginning has very little bearing on the final efficiency of a detergent and methods of laundering. So long as a trace of soil is discernible with the naked eye, the cloth cannot be considered clean. The degree of whiteness tells absolutely nothing about the effectiveness of the washing formula on the remaining soil, and since we are not interested in methods that almost clean soiled articles, we believe it is of very little value when used alone. We find a white background (such as a clean cuff) in shaded light shows up traces very well. We find one of the drawbacks of the machine is that it washes the soil from the surface but does not have sufficient mechanical action to be effective on soil between the fibres. In this respect, we find the laboratory machine less efficient than a laundry wheel, and suggest that steps be taken to overcome this difference. In direct light on a black background, the cloth may appear white, but still show traces when shaded on a white background. It is our opinion that the latter method is the better, and we should like the opinion of all the workers on this point. We have all learned a great deal about the Launder-Ometer, and perhaps an open discussion will furnish ideas by which it can be made more efficient. We believe that the abrasive load can be improved, because the smooth surfaced rubber spheres do not approach the abrasive rubbing action brought about by friction in laundering. Perhaps covering the individual spheres with cloth, using cloth covered Monel metal spheres, cubes, or increasing the number per jar will prove more satisfactory. The mechanical action in the tests is not uniform because the cloth floats in the solution. We found that this very undesirable feature can be eliminated by using the method of Rhodes & Brainard (Ind. & Eng. Chem. Vol. 1 Jan. 1929) in which the abrasive load is inserted in a small bag made of the standard soiled cloth. We find the double belt drive requires considerable regulating to maintain the specified R. P. M., and believe a chain or gear drive more serviceable. The speed in carrying out the tests could undoubtedly be improved if a hinged bar held each row of five jars in place with a single clamp or wing nut at one end. There are many things of this nature which we hope will be brought up in the proposed meeting after the tests, and it is hoped that the members will agree that the method can be made useful.

* * *

Discussion of Results

IT is evident from the results that the machine, as used, was found unsatisfactory

and the method of reading the index likewise because the end point was drawn out and the washing was not uniform.

Since practical laundry tests in standard washing machines give sharp end points and consistent results, the problem of producing a satisfactory laboratory machine must first be solved if progress is to be made on a laboratory scale. A satisfactory laboratory machine should be capable of removing all traces of soil in a reasonable length of time and its action should be such that it readily removes traces held between the fibres as well as impressions made in cloth at time of printing, if any. This year's work has clearly indicated that the very last traces of soil are hardest to remove and since the committee is working to establish a method for determining the detergency of soap products, it is interested in removing these last traces and not in a method that almost cleans soiled cloth. It has been suggested that mechanical action of the machine be increased by increasing the abrasive load (kind, size and number of balls) and also placing the balls in a bag made up of the standard cloth. It is evident that the method of reading the index can be made satisfactory on properly washed samples if observed under identical conditions. In this respect a white background in shaded light has proved the best so far suggested.

As a check up on the index, it has also been suggested that a substance be put in the soil composition that can easily be detected by chemical means directly on the cloth or in the ash of the cloth. Materials other than lampblack have also been proposed with the idea that a material which will wash out easier will make up for the deficiency of the machine. It has also been suggested that the degree of whiteness of the washed cloth be used along with the proposed index reading in order to account for any possible redeposition of soil. The chairman, as well as other members, believes that the proposed method of soiling and evaluating detergency has not yet been thoroughly tested and work along this line should be continued. The principle is sound and modifications in the composition of soil and method of reading should yield useful results in tests approximating commercial laundering. The machine used in this year's work makes simultaneously possible a series of tests under identical conditions and is undoubtedly the best so far presented. Mechanical improvements such as using metal shot abrasive in a bag made of the soil cloth and adopting square bodied corrugated jars are worthy of trial.

In view of past work and the complexes involved, it is evident that detergents should preferably be tested under conditions in which they are generally used. If laboratory equipment cannot be made a reduction to practice, surely, the standard washwheels of industry are accessible and offer an excellent field for this class of work. The chairman wishes to express his sincere appreciation to the members of the committee and their assistants for the large amount of time and work devoted to detergency problems in the past year.

L. T. HOWELLS, *Chairman,*
Detergents Sub-Committee
American Oil Chemists' Society.

Offer New Open-Head Drum

Republic Steel Package Co., Cleveland, is now offering a new type open or removable head light weight drum to producers of vegetable oils, fats, greases and allied products. The new drum, which is designed as a one time shipper, makes removal of fats, hardened oils, greases, etc., possible without the use of heat and, according to the makers, sells at a substantially lower price than heavier containers. It is available in 15, 30 and 55 gallon sizes. The company, of which F. E. Rennebaum is president, also makes standard I.C.C. removable head drums and steel pails in various sizes.

Brown-Edwards Co., well known New York brokers and commission merchants in all classes of oils and fats, have removed their offices from 2 Rector St. to 40 Rector St.

The offices of Frank Honicker, Executive Secretary of the Mayonnaise Products Manufacturers Association of America, Inc., are now located at 1500 Walnut Street, Philadelphia.