Detergents Committee Report to Oil Chemists' Society

Standardization of Procedure in Washing Tests Will Promote Obtaining Comparable Results in Different Laboratories

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FURTHER work directed toward the development of a test of detergency leads the committee to believe that the task committed to its care has been advanced. It has also necessitated reconsideration of the objectives and the limitations which necessarily surround the problem. It is believed that no single test can be devised to measure detergent action under all the manifold conditions of practical cleansing. It is in the nature of the case impossible. If, however, a tool can be provided which will enable different laboratories to obtain concordant results, a new means of advancing the knowledge of detergents will be available. This appears worth doing and preliminary results indicate that it is possible. The present report is presented in the spirit of offering a proposal useful for study, rather than a standardized test from which exact quantitative checks are for the present to be expected.

In considering any test of detergency it is important that the scope of its usefulness should be understood. Washing is a complicated process dealing with many surfaces to be cleaned and many kinds of dirt to be removed. Both fabric and dirt differ chemically and physically and these differences affect profoundly the amount and kind of washing necessary to achieve the desired degree of cleanliness. Changes of temperature, concentration and composition of detergent solutions, the kind and degree of mechanical action, are all dictated by peculiarities of materials to be cleansed and will affect the choice of detergents. No one detergent is best for all kinds of work. Any test which indicates otherwise is false and misleading. Comparisons may nevertheless be made under conditions appropriate to the work in hand and a technique common to many workers may enable them to compare and coordinate their experiences.

In the attempt to reduce variables to a minimum, one type of pigment, one soiling method, and one type of fabric have been selected. The mechanical conditions, which are known to be important, have been arbitrarily standardized and cleanliness has been judged by the measured reflection of light from the fabric. If these conditions have yielded a procedure which will enable different workers to obtain the same results, a tool not hitherto available has been provided. It does not follow that all the important characteristics of detergents may thus be appraised. We would emphasize that the method must be used with discrimination.

It seems likely that a dark colored pigment will yield results more directly comparable to the miscellaneous dirt encountered in practice than any other simple soil. Carbon black is more difficult to remove from cotton fabric than most other types of dirt. Utica sheeting has been chosen as a readily available and well standardized cotton fabric and an extracted carbon black from one manufacturer has been used. Apparently different lots of this grade yield the same results and it may be that other grades of extracted lamp black can also be employed, but it should not be assumed without proof that carbon blacks generally are suited for the test.

WE MAY tentatively accept as a working hypothesis that such soil constituents as fats and oils, starches and albuminous materials which have not been coagulated will be removed before the fabric is free of carbon black. Carbon black offers the advantage of convenience in soiling fabric by working to what appears to be a definite point of saturation in a water suspension. (Numerous other pigments

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can be satisfactorily applied in this way.) The fabric thus soiled does not appreciably change if stored for as much as six months, but it is known that fabrics so soiled will yield values which are different from those obtained by soiling with the same carbon black in an oily vehicle. The selection of a soil harder to remove than ordinary laundry soil is open to criticism. Unless the soil is difficult to remove, differences in detergents cannot be determined accurately. Soiling to saturation reduces the danger of error. It has been necessary to abandon the idea of washing to complete cleanliness as errors in determination of end point are often greater than the differences between detergents.

The choice of a soiling material free from oily constituents and thus different from most dirt which laundries are called upon to remove is recognized as a radical departure which, however, will be justified if future work confirms our present belief that in ordinary cleansing processes the oily constituents are substantially, quantitatively removed before enough of the colored pigment has been removed to make what may be called commercially clean fabric, or that the cleaning of the test pieces evaluates detergents in the same order as does commercial practice. It is not contemplated that the test with carbon black will yield valuable comparisons of detergents with respect to their ability to remove stains or that detergents containing bleaching materials can be thus evaluated.

The question whether quantitative comparisons can be arrived at by the proposed method can only be answered when many more data are at hand. The results now available indicate that within a restricted set of conditions believed to be comparable with laundry practice, it will be possible to rate individual detergents and mixtures in terms of relative cleaning value. It may eventually be possible to refer these to a definite standard such as sodium stearate, but it must not be assumed that comparisons made at one temperature and one concentration will yield results which can be applied to other temperatures or other concentrations. In the interest of advancing the knowledge of detergency it is to be hoped that a wide variety of detergents and cleaning conditions will be studied.

The proposed method appears to have the advantage of yielding comparative results of the same order with considerable variation of mechanical conditions, in which respect it appears analogous to actual practice. In offering a progress report at this time it is with the hope that numerous workers will critically examine the suggestion and report their findings in detail to the committee to the end that in the not distant future a definite recommendation of procedure can be made.

TO BRING the work to the present state of development has involved extended studies in several laboratories in which different members of the committee work. The laborious trial of methods which have eventually been set aside as unsuitable has constituted no inconsiderable part of the work and contributed to the present state of knowledge. The most useful procedure so far found is herewith offered for purposes of study:

Suggested Procedure for the Study of Detergents

THIS test depends upon the soiling of cloth with a water suspension of carbon black to the point of saturation, removal of part of the pigment and comparison of the test pieces on the basis of color.

CLOTH: The cloth used is bleached cotton sheeting weighing about 1.5 grams per square decimeter and having about 27 threads per centimeter in the warp and 25 threads per centimeter in the filling. ("Utica" brand sheeting from the Utica Steam & Mohawk Valley Cotton Mills, Utica, N. Y., was used in the work of the committee.) The sheeting should be substantially free from size before soiling.

SOILING MATERIAL: The soiling material is an extracted carbon black. (Grade "J", supplied by L. Martin Co. of Philadelphia, has been found satisfactory.) It is essential that the pigment shall disperse readily in water without the addition of a deflocculating agent.

WASHING APPARATUS: The washing machine consists of a horizontal shaft that can be rotated at a speed of 40 r.p.m. arranged to hold one-pint jars placed radially about the shaft with the base of the jars two inches from the center of the shaft. Provision is made for maintaining the temperature of the wash solution in the jars to within plus or minus 2° F., of that specified for a given test. One-pint glass-topped jars about 6 inches in height and about 3½ inches in diameter are used. (The Launderometer manufactured by the Atlas Electrical Devices Company, Chicago, Illinois and Atlas E-Z Seal jars were used by the committee.)

METHOD OF SOILING: The cloth is cut into strips, about 3½" wide and 13" long. Not less than 10 nor more than 12 grams of carbon black are suspended in 500 ml. of distilled

water in any suitable vessel. Each strip of cloth is thoroughly wet in distilled water, then stirred for a few moments in the suspension of pigment, and passed between the closely-set rolls of a clothes wringer or similar device. The treatment with pigment and the rolling are repeated until the color of the cloth ceases to become deeper. Five passes will usually be found sufficient. Without drying, each piece is shaken in a jar or wide-mouthed bottle, with successive portions of distilled water, until pigment no longer appears in the wash-water. The strips are then dried at room temperature. This method should give soiled specimens uniform and almost black in color. The brightness readings on soiled unwashed specimens should show very close to 8% whiteness as compared to magnesium carbonate with Hess Ives photometer. Samples of properly soiled specimens for comparison may be had on application to the *Chairman*. An alternative method of soiling suggested on the basis of experience in one laboratory is as follows:

Bags are made *before* the cloth is soiled rather than after. In each jar of the launderometer are placed 100 balls, a weighed amount of the carbon, 100 c.c. distilled water and two of the bags each containing 50 balls. The jars are then run in the machine at 60° C for one hour. Three 15-minute rinses with water are given. The bags are then ready for use without drying. Several bags may be dried if the brightness of the soiled material is to be measured. It may be found possible to reduce the time of the soiling and of the rinses. It is recommended that experiments with this method be made.

PREPARING THE SPECIMEN: The soiled cloth is cut into pieces of 3″ x 6″ size, folded crosswise and sewed along three sides to form a bag, 3″ x 3″ in dimension. Enclosed in each bag are 50 balls of Monel metal of ¼″ diameter, weighing approximately 60 grams, (Atlas Ball Co. of Glenwood Avenue and 4th Street, Philadelphia, Pa. are suppliers.)

PREPARING THE DETERGENT SOLUTION: The preliminary work on this test has been done with soap solutions made by dissolving 2 grams of the detergent in 100 ml. of boiling-hot distilled water, and diluting to 1,000 ml. The washing has been done at 60°C. (150°F.) Obviously, other concentrations, other temperatures, and other detergents can be studied by this method. The effect of hardness of water is also an important consideration which should be investigated.

THE WASHING TEST: (*In this test it is intended that a washing machine of the type described above shall be used. There is some evidence to show that satisfactory results may be obtained with other forms of mechanical washers.*) Heat the water in the Launderometer to 150°F. (60° C.) In each jar place 2 of the specimen bags. Heat the detergent solution to 150°F., and add 100 ml. to each jar. Close the jars and make them fast in the Launderometer, loading the shaft so as to secure approximate balance. (Tendency of the jars to leak may be checked by forcing down the wing-nuts.) Rotate by means of motor for 20 minutes at 40 r.p.m. Remove the jars, pour out the liquids, squeeze the bags by hand, recharge as before with detergent solution and repeat the process until 6 washes have been given. The bags should not be allowed to dry between treatments. After the sixth wash, each pair of bags is rinsed by placing for a few minutes in 3 successive 100 ml. portions of distilled water at 150°F. The bags are then cut open and spread on a flat surface of glass or enamel, to dry. Not less than 10 bags should be used with each detergent.

JUDGING THE RESULTS: When dry, the specimens may be compared by any method which is available to the operator. When this is done directly by the eye, care must be taken that the pieces receive exactly the same illumination. Mechanical devices are very helpful, especially in giving numerical values and in obtaining averages. The Ives tint photometer has been found satisfactory. (Palo-Meyers, Inc., of 81 Reade Street, New York City are suppliers of this device.) Plotting of results in terms of percentage of white has been found useful.

Alternatively, it has been suggested that the results be expressed as the efficiency E of a detergent under specified conditions of use by the following equation;

$$E = \frac{Bs - Bw}{Ss - Bo} \times 100,$$

in which Bs, Bw and Bo are respectively the negative logarithms of the ratios of the brightness of the soiled, the washed, and the original unsoiled cloth to that of a white standard.

The Detergents Committee now solicits the cooperation of a numerous group of volunteers each of whom is prepared to devote about two days to carrying out the proposed test in his laboratory. Identical materials and directions will be supplied with forms for reporting and the summarized results will be sent to each cooperator.