



Fig. 5. Effect of Renex on the detergency of sodium laurate in 300 p.p.m. hard water.

a superior detergent for general purpose use, particularly in hard water areas.

Summary

A non-ionic detergent, Renex, has been shown to improve markedly the detergency of alkylaryl sul-

TABLE III
Effect of Renex on the Detergency of Built Sodium Laurate in Washing Synthetically Soiled Cotton Fabric in Hard Water

Composition	Concentration (per cent)	Relative Detergency Hard Water (300 p.p.m.)
20% Sodium laurate 1% CMC (low visc.) 9% Water 55% Sodium carbonate 15% Tetrasodium pyrophosphate	0.25	100
10% Sodium laurate 10% Renex 1% CMC (low visc.) 9% Water 55% Sodium carbonate 15% Tetrasodium pyrophosphate	0.25	119

fonates in the washing of synthetically soiled cotton. This improvement was found to exist also when the alkylaryl sulfonates were built with CMC, salts, and alkalis.

A combination of tallow soap with the non-ionic detergent, Renex, has been shown to have greater effectiveness than tallow soap alone in hard water. Sodium laurate-Renex combinations show less improvement over straight sodium laurate. When Renex replaces half the tallow soap in a formula containing CMC, sodium carbonate, and tetrasodium pyrophosphate as builders, it improves the detergency in hard water. When Renex replaces half the sodium laurate in a formula containing CMC, sodium carbonate, and tetrasodium pyrophosphate as builders, it improves the detergency.

REFERENCES

1. Louis McDonald, Soap and Sanitary Chemicals, XXI, No. 12, 41 (1945).
2. G. E. Barker, Proceedings of the Scientific Section of the Toilet Goods Association, Number 9, May 1948.
3. Vaughn, Thomas H., and Smith, Clifton E., Journal of the Oil Chemists' Society, 25, 44 (1948).

Report of the Uniform Methods Committee 1948-49

THIS report briefly summarizes the activities of the Uniform Methods Committee and the 12 technical committees, including their subcommittees, for the past year. Its main purpose is to record action of the Uniform Methods Committee and the Society on any committee recommendations. Wherever the connotation "adopted" or "approved" is used it means that it carried the unanimous approval of the committee recommending, the unanimous approval of the Uniform Methods Committee, and the unanimous approval of the Society unless otherwise noted. Dates in parenthesis denote times of action by the Society in business session.

Official Natural Bleaching Earth. A 4,000-pound lot of this material that was placed in stock in November, 1947, was anticipated to be a five-year supply. However, this stock was exhausted by November, 1948. This was caused by the great increase in use due to the bleach color basis of trading on crude soybean oil for the first time. Active consideration is being given to start similarly bleach color basis of trading on crude cottonseed oil. This would further increase the demand.

A new lot of 24,000 pounds was prepared through the courtesy of L. A. Salomon and Bro., New York City, sole distributor of XL000 English earth in this country, the material used. It was placed in stock at the Central Scientific Company, Chicago, in April, 1949.

This new material was adopted as official (May, 1949) for use in the official bleaching methods Ce-8a-47 and Ce-8b-47 using 5% (15 grams) instead of the previously prescribed 6% (18 grams). Also approved (May, 1949) was a change in these two bleaching methods to delete mention of earth quantity and insert that the amount used shall be that stated on the label of the can of official earth. Labels will be so printed, carrying the statement of approval of the N.C.P.A. Chemists Committee and the N.S.P.A. Technical Committee when and if this is reported. Also approved (May, 1949) was that the above earth and procedures be recommended to the N.C.P.A. and N.S.P.A. for their approval and use in trading rules.

These changes had to be made because it was found impossible to secure a 24,000-pound quantity of XL000 English earth that was the exact equiva-

lent of the previous standard at a 6% level. The 5% level recommended was based on a large amount of comparative data in many laboratories. This lesser dosage was selected to maintain the same level of results, compensating for its greater bleaching activity.

It is not mandatory to use English XI.000 earth for this standard, but in fact it is the only material that has ever been used. There are reasons why it should not be continued in the future:

1. It rarely is used any more in present-day industry on cottonseed or soybean oil.
2. Quantities now required are so great that selection of identical lots are difficult. Its limited commercial use makes this more difficult.
3. It seems more logical to adopt a domestic earth having a bleaching activity more in line with what is now used industrially. This would be a much more active earth than English earth.
4. A more active standard would require less earth (on the order of 3% instead of 6%). Quantities required have already passed the realm of laboratory preparation into carload proportions.
5. By adopting a fairly active domestic earth, the need of our other sponsored official *activated* bleaching earth may become unnecessary. One standard earth might serve all purposes by adjusting the prescribed quantity in the test to maintain the desired bleaching level and repress the effect of green color on soybean oil.

Official Ammonium Sulfate. A new lot of this material was standardized and placed in stock in October, 1948, under the direction of the Seed and Meal Analysis Committee, being the first new standard lot in many years.

Standard Crude Glycerine. On recommendation of the Glycerine Analysis Committee a standard crude glycerine with a certificate of analysis was adopted (November, 1948) for sale by the Society. Samples and certified analyses were prepared by this committee and the material put in stock for sale January, 1949. This is the second lot of this type of material sponsored by the Society.

Gossypol Committee. As the result of several years of collaborative work on several different methods the P-Anisidine method for determination of free gossypol was adopted (May, 1949) as a tentative method. Collaborative work will be continued on slight modification of this method. This method is thoroughly described in a paper entitled "Determination of Free Gossypol in Cottonseed Materials" by Pons and Guthrie, presented at the spring meeting of the Society and soon to be published in the journal.

Fat and Oil Analysis Committee. The following methods were adopted (November, 1948):

1. Tentative methods for flash and fire point, saponification value, and viscosity, all applicable to drying oils.
2. A tentative method for acid value on lecithin.
3. A tentative method for closed cup flash point subject to approval of final, edited, detailed copy by the Uniform Methods Committee, which was later done.

Due to a minority dissenting opinion in the committee a matter of F.F.A. determination was referred to the Uniform Methods Committee, which unanimously approved changing the conflicting method of determining F.F.A. on crude oils in the refining loss tests to conform strictly to the F.F.A. method Ca-5a-40. This merely means that sample weights of 28.2 gms. are to be used on all oils having F.F.A. under 1.0% instead of 7.05 gms. now specified in the refining loss methods Ca-9a-41, Ca-9b-46, and Ca-9c-46. The change was approved (May, 1949).

Soapstock Analysis Committee. This committee was just formed less than six months ago for purposes of improving the significance of soapstock analyses; especially to recognize and develop methods for determining non-fatty material in soapstock. Some collaborative work has already been started.

Refining Committee. A change in method Ca-9c-46 for refining degummed expeller soybean oils was approved wherein the lye dosage of from .1% to .2% excess was changed to .15% and .20% excess, other types of degummed oil remaining unchanged. This means a new method for degummed expeller soybean oil and was so approved (May, 1949) as a tentative method. Several subcommittees are continuing work on various aspects of centrifugal refining methods.

Oil Color Committee. By a vote of 13 to 2 the committee recommended the tentative adoption of the spectrophotometric method for determining oil color. This method is detailed in their annual report based on the transmission of light at the single wave length of 525 millimicrons. The Uniform Methods Committee approved this by a vote of 5 to 2. The Society adopted it (May, 1949) unanimously. The details of this method are published elsewhere in this issue of the Journal.

Meal Analysis Committee. As the result of a subcommittee's work the method presented in their annual report for sampling soya flour was adopted (May, 1949) as tentative. The subcommittee on soya flour sieving method is continuing work for another year on the development of a uniform method.

A subcommittee has developed some information on water absorption of soya flour but no satisfactory method has been evolved; and since there is no apparent need for such a method, no further work is contemplated.

The subcommittee on Copra Meal is continuing its investigations on analytical methods for moisture, protein, fat, crude fibre, and ash, but so far no uniform method has been developed.

The subcommittee on Tung Fruit and Meal analysis has conducted collaborative tests on the tentative methods of analysis on tung meal adopted a year ago, but no complete report on this can be expected before next fall.

The following methods, having been tentative for two years and recently adopted as official by the A.O.A.C., were adopted (May, 1949) as official:

- Be-2-47—Moisture and Volatile Matter
- Be-3-47—Oil
- Be-4-47—Nitrogen and Protein
- Be-5-47—Ash
- Be-6-47—Crude Fibre
- All in soya flour.

Tentative method Ba-5-47 for ash and Ba-6-47 for crude fibre, both in oil seed meals, have been tentative for two years and agree with the corresponding A.O.A.C. methods and were adopted (May, 1949) as official.

Cellulose Yield Committee. This committee continues to sponsor collaborative sample testing on official method Bb-3-47 and recommends its continuation.

Soap Analysis Committee. Two new methods, one for per cent anhydrous synthetic detergent and the other for per cent neutral inorganic salts, were adopted as tentative (May, 1949). Both methods in-

volve calculations only, based on reference to results of existing official methods.

Bleaching Methods Committee. This committee continues to be rather inactive as there is no specific problem before them although one member of the committee is making a spectroscopic study of bleaching response of various oils. In view of the problems on standard bleaching earth presented in the first of this report, the committee may soon have a very active problem.

Vitamin Committee. This committee is rather inactive pending action that the U. S. Pharmacopoeia may take in their contemplated consideration of the physicochemical methods of Vitamin "A" assay that the committee has previously developed.

Glycerine Analysis Committee. Revision in tentative method Ea-6-46 for the periodic acid determination of glycerine was adopted (November, 1948).

Spectroscopy Committee. A spectrophotometric method of analysis for constituent fatty acids was adopted (November, 1948) as tentative.

General. The foregoing reviews of committee activities are in most cases as complete as information has been made available to the Uniform Methods Committee. It is entirely complete with respect to all official actions of the Society on analytical methods.

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The Determination of Potassium in Soap and Mixed Caustic Lye*

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A MODIFICATION of the method for the determination of potassium as the periodate, suggested by Willard and Boyle (34), is presented. The modification increases the accuracy and sensitivity of the original method and at the same time contributes to its usefulness as an industrial control procedure. A literature survey is presented as an aid for those who wish to evaluate the proposed method by comparison with other methods.

Review of Methods

Cobaltinitrite. The determination of potassium with sodium cobaltinitrite, as first suggested by de Koninck (11), has disadvantages that cannot be overlooked when extreme accuracy is desired. The precipitate of dipotassium sodium cobaltinitrite has been studied quite completely by Robinson and Hauschildt (22). Their investigations show the composition of the precipitate to vary, depending upon the conditions of the precipitation; the most important is the sodium and alcohol concentrations of the precipitating medium and the temperature at which the precipitation is made. Others (19, 25, 26, 30) have confirmed this work insofar as agreeing with the qualitative effect of these variables. There is wide discrepancy, however, in the literature, concerning the quantitative effects of Na:K ratio, temperature, and concentration.

Sodium cobaltinitrite reagent is generally prepared by mixing a solution of sodium nitrite with a solution of cobaltous acetate in acetic acid. Since precipitation by this reagent usually gives high results, it is felt that a reagent containing less extraneous materials would be desirable. This is accomplished by Wilcox (33), who employs an aqueous solution of trisodium cobaltinitrite as the precipitating reagent, the reaction being carried out in a nitric acid medium. The precipitate is crystalline and allegedly of constant composition regardless of the sodium concentration.

The precipitate of dipotassium sodium cobaltinitrite obtained through any scheme of precipitation may be finally determined either gravimetrically or volumetrically. One of the best known methods is that of Kramer and Tisdall (18), who decompose the precipitate with alkali, oxidize the liberated nitrite with potassium permanganate, and back titrate the excess with oxalate.

There are certain disadvantages in this method, and these are outlined by Brown, Robinson, and Browning (5), who have suggested an alternative procedure. They dissolve the precipitate in ceric sulfate and back titrate the excess with ferrous ammonium sulfate, using ortho-phenanthroline as the indicator. An average error of 2% is reported in determinations of potassium from 0.2 to 1.0 mg.

A variation of the cerium procedure is presented by Kaye (16). Potassium iodide is added to the excess ceric sulfate, and the liberated iodine is titrated to a starch endpoint using sodium thiosulfate. The obvious advantage of such procedure lies in the sharpness of the endpoint, even in very dilute solutions.

Further variations of the cobaltinitrite method include the use of silver cobaltinitrite reagent and the final determination made with ceric sulfate and ferroin (17). Determination of the precipitate formed from the same reagent is also made through the estimation of silver in the insoluble complex using the Volhard method (14). An alternative procedure is the determination by distillation and titration of ammonia (10). The formation of the silver complex is preferred by some because of its greater insolubility, more constant composition, and ease of manipulation.

Colorimetry. Colorimetric methods applicable to the determination of potassium are many and include further variations of the precipitation of the cobaltinitrite. One of these is suggested by Eden (12) and is a variation of the method of Jacobs and Hoffman (15), in which a brilliant green complex is formed with choline hydrochloride and potassium ferrocyanide. Wander (31) suggests the oxidation of dipotassium sodium cobaltinitrite with dichromate and

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