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The Measurement of Lather Quickness

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In the formulation of bar products (soap, combination, and detergent bars) a major property of interest is that of "lather quickness," *i.e.,* the ease with which the user can obtain an ample and voluminous lather. This can, of course, be measured subjectively, and major differences can easily be recognized, but it is often necessary to distinguish more subtle differences as a guide to formulation. Tests designed to measure the amount and stability of foam produced by the formulation, for example, the Ross-Miles pour-foam test (1), and the more recent Barnett-Powers "Latherometer" (2), while useful in many applications, do not give satisfactory correlation with performance of bar products.

Masters (3) has made a significant contribution by pointing out that ease of abrasion of the bar itself is important. This is quite clearly the case since a satisfactory lather cannot be obtained without abrading soap onto the hands. Masters describes a device in which bars can be abraded in a reasonably reproducible manner, and a wear number is defined from the relation

$$k = -(1/S) \ln (t_f/t_o)$$

where S = number of abrasions of the bar made by a sponge in a uniform manner, t_o = the original thickness of the bar, measured at the center, and t_f = the final thickness. The wear number W is defined as $1000k$ and is independent of S.

Unfortunately this technique has no provision for measuring the effect of water hardness or of the effect of changes in foam stability as a function of additives. It is the purpose of this paper to describe a test method and apparatus, developed in these laboratories over the years, which measures lather quickness in a way which combines the determination of rate of abrasion with that of foam stability. To be sure, as will be made clear in the following, it (like Masters' test) does not actually make measurements on lather; however the results of extensive testing have convinced us that a very exact correlation does exist.

Experimental

Description of Apparatus. The lather quickness tester consists of a device in which a bar of soap is uniformly abraded by a cellulose sponge, which is constantly wetted and rinsed in water of a definite hardness and temperature. The end-point is defined as the number of strokes of the sponge required to form a uniform layer of foam on the surface of the water in the test vessel into which the sponge dips.

Essentially the tester is a unit consisting of a reciprocating vertical shaft, which is activated by a Scotch yoke driven by a 1/20 h.p. motor through a chain drive (See Figure 1). A sponge-holder, consisting of a flat metal plate to which is cemented a cellulose sponge ($3\frac{1}{2} \times 3\frac{1}{4} \times 1$ in.), is fastened to the bottom of the shaft. Opposed to the sponge travel is a soap-holder designed to hold the bar under test. The downward stroke of the shaft carries the sponge past the soap-holder and down into a cylindrical stainless steel vessel holding one gallon of water at the prescribed hardness and temperature. The upward stroke carries the sponge up past the soap-holder and automatically records the stroke on a strategically placed Veeder Root counter. The device is designed for an operating speed of 22 strokes per minute.

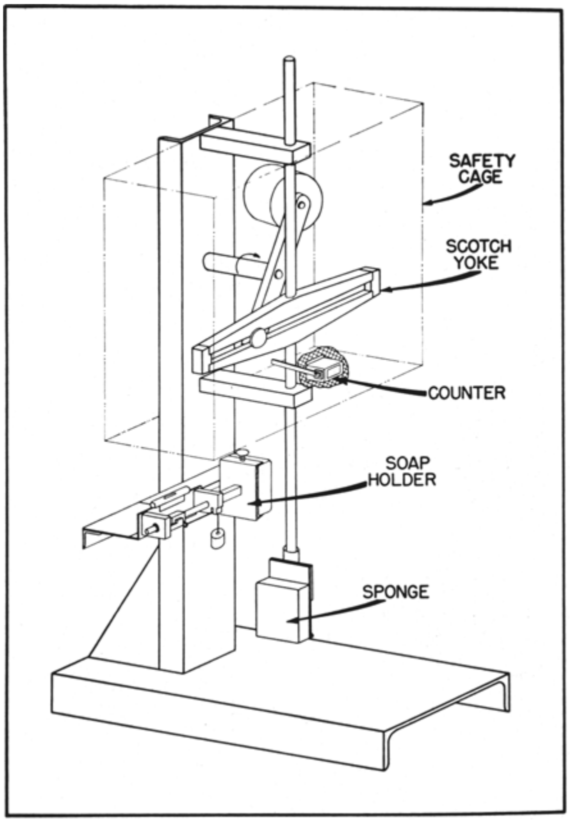

Fig. 1. Isometric drawing of test apparatus. The chain-drive and motor are concealed behind the vertical support.

The soap-holder consists of a two-sided metal box, the top of which can be clamped down on the bar. This is supported at right angles by a bearing-mounted shaft which holds the bar (when at rest) at a definite distance from the sponge center and maintains the sponge-bar contact at a definite pressure by the use of a counter-balancing weight. The holder is located approximately six inches above the test vessel.

The sponge employed is a DuPont 6A cellulose sponge, cut to the desired size, and cemented to the plate with a water-proof asphalt-base cement.[1] There is some variability in the sponges, and they have to be standardized against a regular production soap of known properties. About 10% of the sponges are rejected as unsatisfactory upon standardization. The average life of the sponge is also variable, depending on the number and types of tests run; a hundred tests would be considered normal.

The assembled unit is shown in operation in Figure 2.

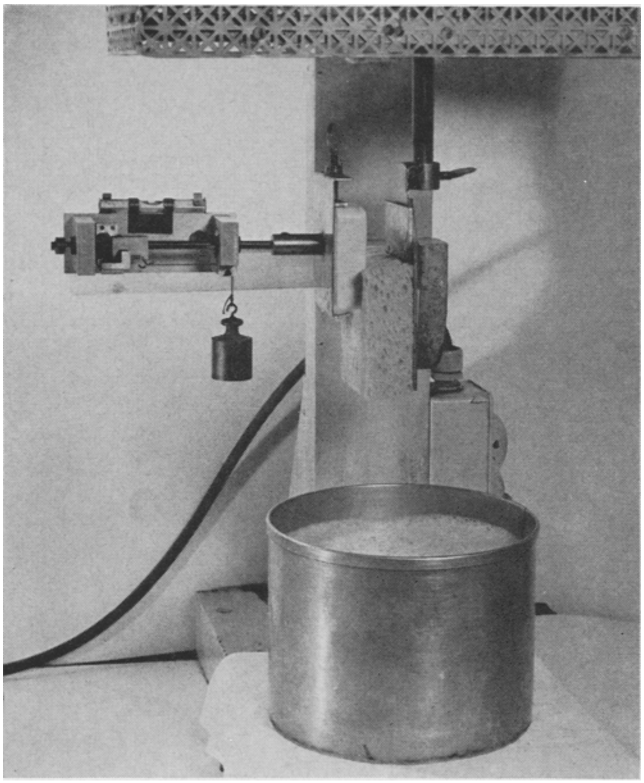

FIG. 2. The apparatus in use. The sponge has just begun to abrade the soap bar on the up-stroke.

Test Procedures. The actual testing is carried out in the following manner, with such variations as may be required for special cases:

1. The soap sample is prepared by cutting the bar to the dimensions: 3 x 2 x 1-in., with the side of the 3 x 2-in. surface which faces the sponge shaved plane. (Normally test bars in this laboratory are pressed in a 3 x 2-in. oblong shape; the flat surface and one-inch thickness are obtained by shaving the bar by moving it across the cutting edge of a 12-in. carpenter's plane, held in an inverted position.) If bars of odd shape are to be tested, the results are not strictly comparable unless a similarly shaped control is also run.[2]
2. The sponge is rinsed and squeezed out, and the sponge-holder is attached to the vertical shaft (stopped in "up" position).
3. One gallon of deionized or distilled water is measured out into the test vessel, and the requisite amount of stock hard water of 25,000 p.p.m. as $CaCO_3$ is added. For 125 p.p.m. (at which the majority of tests are run) 19.0 ml. are required. The stock hardness consists of a solution of calcium and magnesium chlorides with the Ca:Mg ratio 3:1; the technique of making up the solution has been previously described (4).

   The temperature of the water is adjusted to 85°F. by placing the test vessel on the steam bath.
4. The test vessel is placed in position beneath the shaft and, keeping the soap-holder away from the sponge, the unit is started and sponge is allowed to pass in and out of the water three times to saturate it. The sponge is stopped above the water level, but below the soap-holder. The counter is then set to zero, and the test is started.
5. The test is continued until the surface of the water in the test vessel is covered with foam (except for an area near the center, where the foam is broken down by the drip from the sponge). A characteristic end-point is illustrated in Figure 3.
6. The number of strokes is recorded, and the appearance and qualitative stability of the foam are noted.

The test is normally run in duplicate, and a trained operator will obtain agreement to within ±1 stroke. Obviously the lower the number of strokes required, the more satisfactory is the product with respect to lather quickness.

Discussion

Experimental Results. The type of data which can be obtained with the use of the above-described apparatus is shown in Figures 4 to 6. Figure 4 illustrates the change in lather quickness as measured by the test as a function of soap-stock composition. The soaps tested consist of a series of coco-tallow soaps in the range of 0% to 50% coco. The remarkable aspect of these data resides in the minimum in the end-point values (maximum in lather quickness) at about 15% coco content. However the data indicate a tendency toward lower values at much higher coco contents. It also should be pointed out that, in evaluating compositions for optimum lathering, lather quickness is only one property to be considered. Such things as lather consistency and bubble size (which are not measured by this test) may be significant. This statement applies not only to the data

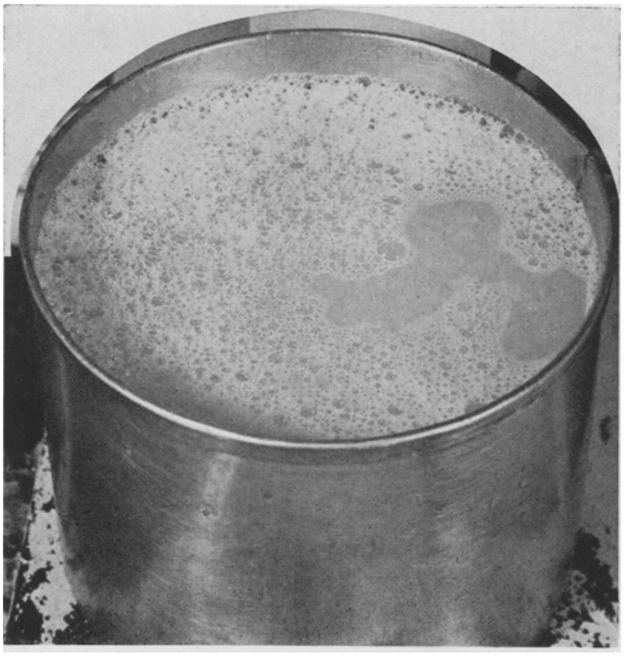

FIG. 3. A characteristic end-point. The area not covered by foam is caused by drip from the sponge.

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[1] A satisfactory cement is "Iron Bound Cement," manufactured by Slomons Laboratories Inc., Long Island City, N. Y.

[2] In the case of fresh bars, shaving the bar can have no effect on the determination. It might be supposed that a slight effect would be observable in the case of aged bars since the surface would be at a lower moisture than the body of the bar. However this drier layer is quite thin and, if not shaved off, would be removed in the first few strokes of the sponge; thus it could not materially affect the result of the test.

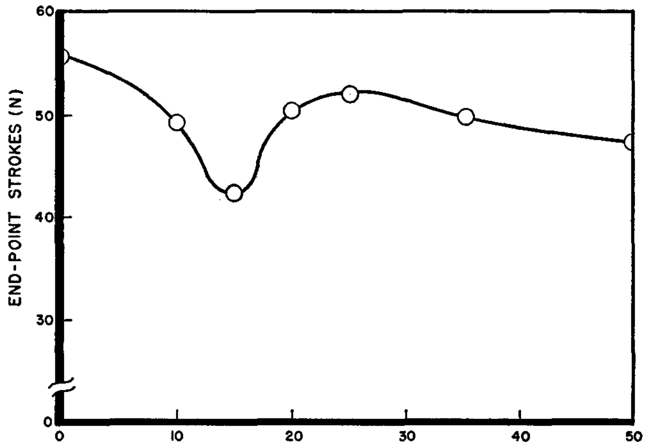

Fig. 4. The effect of coco-tallow ratio on lather quickness.

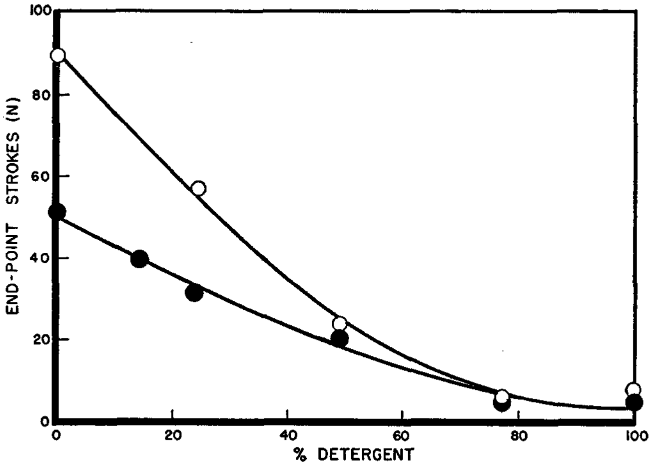

Fig. 5. The effect of added detergent (sodium H-coco monoglyceride sulfate) on lather quickness; ● — at 125 p.p.m.; ○ — at 250 p.p.m.

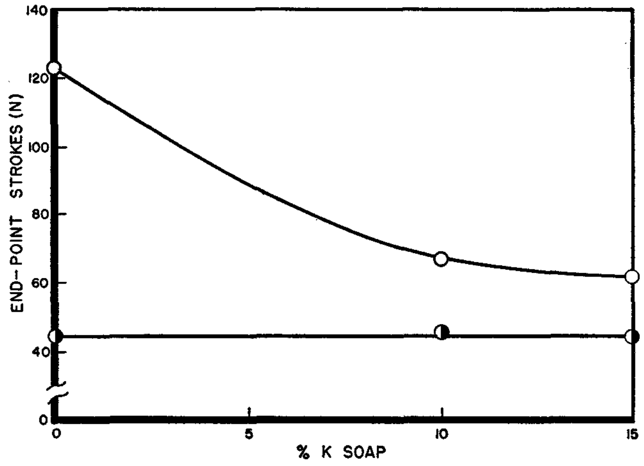

Fig. 6. Effect of potash soap on lather quickness; ◑ — at 85° F.; ○ — at 55°F.

of Figure 4 but to many of the compositions considered below.

Figure 5 illustrates two effects: the effect of the addition of an extremely soluble synthetic detergent (sodium H-coco monoglyceride sulfate) and the effect of different water hardnesses (125 p.p.m. and 250 p.p.m. as $CaCO_3$). This is especially instructive in the case of a soap-detergent combination of this type. It will be observed that, at about 50% detergent, the hardness of the water has scarcely any effect on lather quickness.

Since the detergent used in obtaining the data of Figure 5 is quite soluble, it might be expected that lather improvement would result on its incorporation. However it is a remarkable fact that the addition of relatively insoluble detergents also has a lather-boosting effect, at low concentrations at least. At high concentrations however the insolubility of this material has a significantly adverse effect on the lather quickness.

It has long been the feeling that the incorporation of potash soaps would lead to a gain in lather quickness. The data presented in Figure 6 shows the effect of varying percentages of potash soap on the lather of a coco-tallow soap containing 15% coco, both at the regular temperature of 85°F. and at the lower temperature of 55°F. As can be seen, the potash soap has no particular effect on lather quickness at normal use temperature but is of value in cooler water.

Interpretation of Test. Since foam is used as the end-point indication for this test, no end-point can be observed (for an all-soap composition) until the hardness present is titrated by the soap. Following this, additional soap has to be abraded and air whipped into the solution by the action of the sponge to produce enough foam to constitute an end-point. Thus N, the number of strokes at the end-point, is given by

$$N = n' + n \qquad (1)$$

where n' is the number of strokes required to titrate the hardness, and n the additional number of strokes required to generate the foam end-point. If C_{eq} is the concentration of soap which is equivalent to (titrates) the hardness, and R is the rate of abrasion, expressed in units of g. per 100 ml./stroke, then Equation 1 becomes

$$N = C_{eq}/R + n \qquad (2)$$

Thus, if one plots the number of strokes required for the end-point against the reciprocal rate of abrasion for a number of soaps, a straight line should result. The rate of abrasion is calculated from the relation $R = C/N$, where C is the soap concentration in the test vessel at the end-point. This is based on

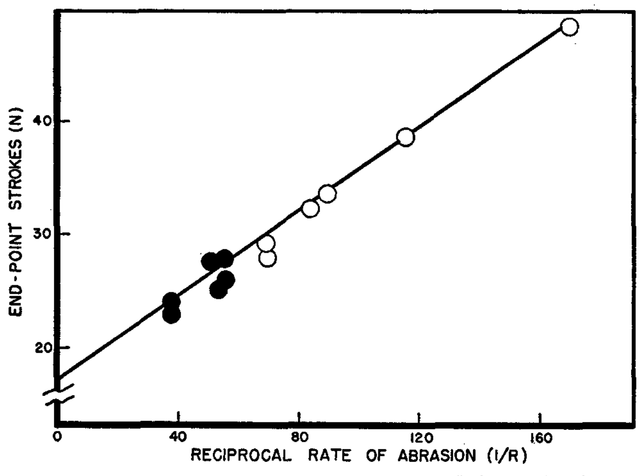

Fig. 7. Number of sponge strokes required for end-point *vs.* reciprocal rate of abrasion for a number of soaps; ○ — at 85°F.; ● — at 110°F.

the assumption that the sponge abrades the bar at a uniform rate, *i.e.*, that the amount of soap introduced into the test vessel per stroke does not vary with the number of strokes for a given composition.[3] Work done in these laboratories some years ago (5), in which end-point concentrations were determined, have been plotted according to Equation 2, with the results shown in Figure 7. As can be seen, there is good agreement with the theory, and the relation is apparently independent of temperature. Unfortunately these results were obtained in a somewhat different way from the standard method described above and thus cannot be used for the calculation of the constants of Equation 2. However the slope C_{eq} can be calculated from the known equivalence of the soap and calcium carbonate hardness while the intercept n should be equal to the number of strokes required for an end-point in soft water. The former is found to be about 0.069 g. per 100 ml. for soaps containing about 20% coconut oil; the latter is found experimentally to be 10–12 strokes for most soaps.

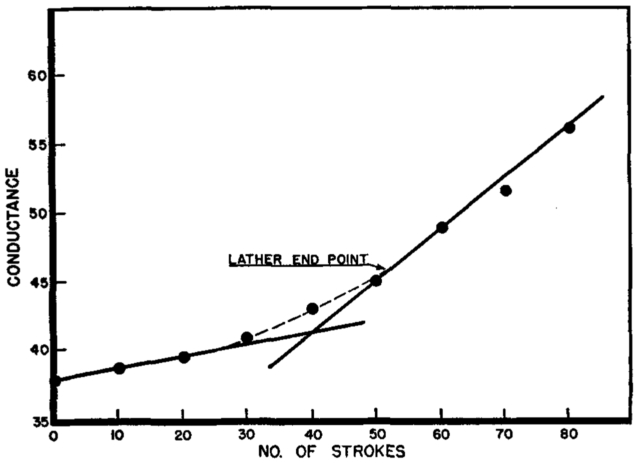

FIG. 8. Conductance of test solution as a function of number of sponge strokes at 125 p.p.m.

Another method of verifying Equation 2 is indicated by the data of Figure 8. In this experiment the lather test was run on an ordinary soap bar (20% coco–80% tallow) at a hardness of 125 p.p.m. The conductance of the solution was measured by means of a dipping electrode in the test vessel and a Model RC M15 Serfass Conductivity Bridge. Since the absolute values of the conductivities are not required, the ordinate is plotted in arbritary scale readings.

As is evident, what is obtained is the well-known curve for a conductometric precipitation titration (6), with the break occurring at 40 strokes (the lather end-point for this soap sample occurs at 52 strokes). If a similar determination is carried out in soft water, a straight line should result. This is shown in Figure 9. In this experiment 0.25% ethanol was added as a foam depressant since it had been found that the high foaming experienced in the soft water gave spurious results. The slight deviation from the straight line at 50 strokes may be ascribed to the loss in effectiveness of the ethanol as the soap concentration increases.

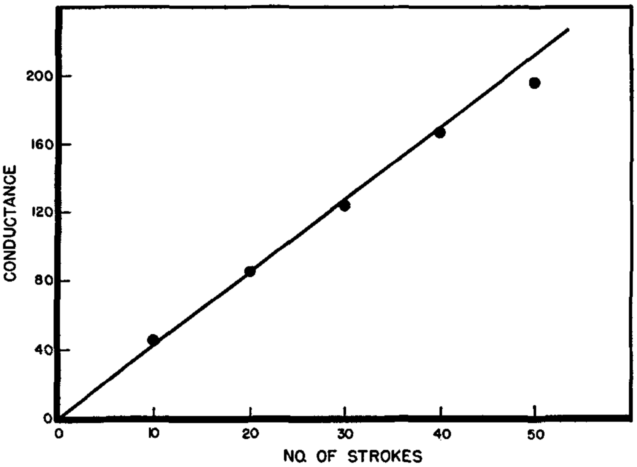

FIG. 9. Conductance of test solution as a function of number of sponge strokes at 0 p.p.m. (0.25% ethanol added as foam depressant).

From Figure 9 and a suitable calibration curve it is possible to determine the rate of abrasion and thus calculate the soap concentration at the break-point of Figure 8. If this is done, the concentration is found to be 0.064 g. per 100 ml., which is in good agreement with the calculated theoretical value for C_{eq}; subtraction of the number of strokes at the break-point from those for the lather end-point yields 12 strokes, which, again, is in agreement with the expected result.

From the above discussion it might appear that results equivalent to this test could be obtained by use of the abrasion technique of Masters, together with a lather-quickness test run in soft water. This however is only true if one is dealing with simple soap compositions; as soon as important amounts of additives are included, the simple interpretation which leads to Equation 2 no longer applies. That this is the case is seen from the data of Figure 5, where end-points are found lower than that required for C_{eq} to be reached. Obviously what is now being measured is a more complex process involving complexing of hardness, dispersion of lime soaps, and the foaming properties (if any) of the additives, all in the presence of soap and of hard water. Equation 1 still applies, but the definition of the quantity n' is no longer a simple matter and n will vary with the composition. However, while the theoretical interpretation is difficult under these conditions, the method is still felt to measure lather quickness. No simple abrasion measurement is capable of this flexibility.

Acknowledgments

It is a pleasure to acknowledge the cooperation of the mechanical Engineering Division of the Research and Development Department (and, in particular, that of the Division Head, E. J. Gibbons) in the design and construction of the test unit. Most of the experimental data reported herein were obtained by Mrs. Alice M. Helble.

Summary

A method and an apparatus are described for the measurement of lather quickness of bar soaps and combinations. This method is capable of distinguishing differences in lather quickness as a function of

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[3] This is demonstrably true under the conditions of test described herein; it will not be true for a large number of strokes (several hundred) since the thickness of the bar will then be changed materially and the pressure exerted by the sponge will change accordingly.

soap stock composition, presence of additives, hardness, and temperature. Representative data obtained by the method are presented, and a theoretical interpretation of the test is given.

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Epoxidation of Polyesters of Tetrahydrophthalic Acid and Unsaturated Alkyd Resins

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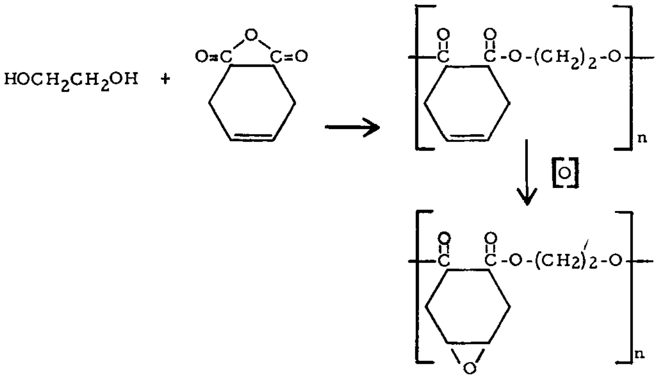
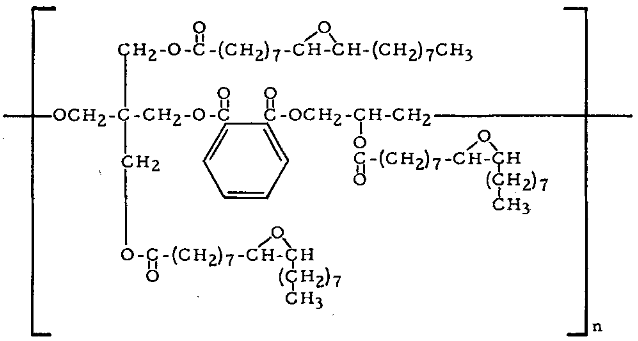
IN THE COURSE of an investigation carried out in our laboratory, directed toward synthesis of molecules containing a large number of epoxide groups, oxidation of unsaturated polyesters to the corresponding epoxide derivatives offered considerable interest. Particularly, epoxidized derivatives of tetrahydrophthalic acid polyesters and unsaturated alkyd resins could be important intermediate compositions from a practical standpoint. We wish to report not only the subject compositions but also processes suitable for their preparation and purification in high yield.

The efficient epoxidation of the olefin bonds of these polyesters offers the opportunity to synthesize molecules containing several epoxide groups. For example, polyesters of tetrahydrophthalic acid can be prepared containing at least 10 to 15 recurring units. Molecules containing this large number of recurring units would contain a significantly larger number of epoxide groups than herebefore commonly known, provided efficient esterification without appreciable loss in olefin and epoxidation was accomplished. Similarly, complex esters of a "medium" to "long oil" alkyd type of resin, likewise epoxidized, would give a structure of analogous character. The reaction of ethylene glycol and tetrahydrophthalic anhydride and oxidation of the resulting polyester to the corresponding polyepoxide illustrates the polyester type.



The epoxidation product of a complex ester made from pentaerythritol, phthalic anhydride, and glycerol trioleate demonstrates the oil-modified polyepoxide alkyd type.



In order to obtain polyepoxides, of the type illustrated, basic epoxidation processes already known were evaluated. However epoxidation of these polyester and alkyd type of resin compositions by a preformed peracetic acid process of Findley and Swern (1) or of Terry and Wheeler (2) could not be advantageously used because of the difficulty of isolation and purification of the epoxidized product in the first case and side reaction of the epoxide as well in the second case. A newer *in situ* process, using a cation exchange resin of the sulfonic acid type (3, 4), has also proved unsatisfactory in yielding desired epoxides. The reduced quantity of acetic acid required by this latter process is a desirable advantage if it could be made operable for the epoxidation of the subject compositions. Schmitz and Wallace have indicated its commercial feasibility (5). We have found that the use of dehydration in vacuum at 80 to 85°C. of a carefully washed acid form of a cation exchange resin of the styrene-divinyl benzene sulfonic acid type permits efficient *in situ* epoxidation of the subject unsaturated polyesters. The dehydrated property of the dry cation exchange resin apparently is essential to their successful epoxidation.

Table I compares the use of a wet and dry cation exchange resin of the sulfonic acid type (Dowex 50-X8, 50-100 mesh). In all cases the use of a dried cation exchange resin resulted in more efficient epoxidation, and clear transparent reaction products were isolated. In the case where the wet ion exchange resin was used, milky turbid reaction products were

TABLE I
A Comparison of the Use of Wet and Dry Cation Exchange Resin

Species epoxidized	Percentage conversion to epoxide	
	Wet ion exchange resin	Dry ion exchange resin
Tetrahydrophthalic-1,5-pentanediol polyester	4.3	81.4
Unsaturated alkyd resin	53.1	71.5
Butyl oleate	79.0	86.6