which no specifications or standards exist that have been developed by a public or unbiased body. This does not mean that governmental specifications are necessarily the ones which should be used to protect the consumers' interest for in many cases the government specifications are as unsuitable as those developed by industry or a trade association in which industrial groups exercise control.

It is to be hoped that manufacturers may shortly come to conclusions about nomenclature and labelling of detergent products so that consumers can tell whether the particular material is a soap or soap substitute, whether it is designed to be used on dishes, floors, and clothes, or for all household purposes. If, for example, a detergent is safe for use on painted walls, its label should so indicate in unequivocal terms, and the essential caution or special directions should be included. If it is not safe for fine fabrics or for silk or wool, the label should supply that information too and state clearly to what types of washing it may be applied safely and economically.

The label should also describe in reasonable detail the chemical nature of the product so that if the housewife finds a particular kind unsatisfactory for her purpose—harsh, say, on her hands or nails—she will not have to go through the process of discovering by trial and error that another product with a different name has the same undesirable property. Medical men in particular complain about the difficulty of getting information on the composition of various household materials for they often have the problem of dealing quickly with ill health or a skin lesion caused by a substance, the composition of which may be wholly unknown to them.

THROUGH careful study of the data available from scores of chemical laboratories we have succeeded in arranging connections with perhaps seven or eight analytical laboratories whose work is careful and responsible. Some of these laboratories specialize in detergents and cosmetics; others in solvents or petroleum products; still others in determination of contaminating substances in foods in micro amounts. One laboratory chiefly active in food and drug analysis and assays was recently engaged for a series of food analyses and for a literature search in a highly specialized field connected with food chemistry.

One of the most important aspects of the work of Consumers' Research is its broad use in educational fields, such as high schools and colleges. Its bulletins and other published material are used for various courses, such as economics of consumption, general economics, consumer education, marketing, and the like. There are now in existence at least a hundred theses, books, and articles written on problems of testing and rating of products from the consumers' standpoint. Consumers' Research has prepared a special bulletin of 38 pages entitled "Consumers' Test Manual," which includes a wide range of chemical tests that can be carried out by teachers and students to detect contaminating and preservative substances in foods, and metals and other contaminants in drinking water. There are also a few simple chemical test methods applying to soap and certain other cleaning agents.

The effect of Consumers' Research upon the goods available in the market is significant. Most of the distinctly fraudulent products—and there are a good many of these—have a much shorter run than in previous years because there are now many persons who are aware of the fields in which falsification or misleading use of scientific tests as a sales technique will commonly occur. A surprising number of manufacturers have written to Consumers' Research and conceded that the defects which we had reported in their products were actually present and that the findings had been valuable to them in pointing out deficiencies of which they had not been conscious.

There are few fields of manufacture which fully protect the consumers, but the need for modification of practices is much less in industries where there has been an extensive development of technical and scientific studies and of standard tests and specifications. In a very real way the engineer, chemist, and scientist are the defenders of the public interest, even at times when they are not aware of the relationship of what they are doing to the protection of consumers, for the effects of scientific and technical activity spread wide like the ripples cast in the water of a pond by a stone.

Soap by Saponification

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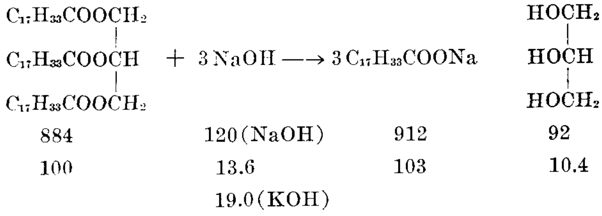
SOAP raw materials and their processing have already been treated. It remains to show how these materials are converted into finished soaps in various forms and, in particular, how fats and oils are converted into soaps. In brief, we will deal first with some fundamentals of the reaction; secondly with a typical kettle saponification, and thirdly with other methods of saponification.

Fundamentals

Natural oils and fats are triglycerides which react with an alkali to yield glycerine and salts of the fatty acids which may contain similar or different long chain alkyl groups.

Considering the case where the three alkyl groups are similar and equal to $C_{17}H_{33}$, the fat molecule becomes triolein and three molecules of sodium oleate soap are formed.



$$\begin{array}{cccc} C_{17}H_{33}COOCH_2 & & & HOCH_2 \\ | & & & | \\ C_{17}H_{33}COOCH & + \; 3\,NaOH \longrightarrow & 3\,C_{17}H_{33}COONa & HOCH \\ | & & & | \\ C_{17}H_{33}COOCH_2 & & & HOCH_2 \\ 884 & 120(NaOH) & 912 & 92 \\ 100 & 13.6 & 103 & 10.4 \\ & 19.0(KOH) & & \end{array}$$

Mathematically 884 pounds of such triolein will react with 120 pounds of anhydrous caustic soda to yield 912 pounds of anhydrous soap and 92 pounds of glycerine. For each 100 pounds of fat there is used 13.6 pounds of alkali to produce 103 pounds of soap and

10.4 pounds of glycerine. Such a fat would be said to contain 10.4% glycerine, and it would be easy to work out other relationships. For example, the total fatty acids in triolein would be 95.7%, and its saponification value would be 190. The fatty acids in tallow may roughly be taken as a mixture of palmitic, oleic, stearic, and a few other acids. A typical analysis is given in Table I.

TABLE I

Typical Analysis of a Tallow and a Coconut Oil

Mol. Wt.	Fatty Acid	Tallow[a]	Coconut Oil[b]
116	Caproic	....	0.8%
144	Caprylic	....	5.4%
172	Capric	....	8.4%
200	Lauric	....	45.5%
228	Myristic	6.3%	18.0%
256	Palmitic	27.4%	10.5%
284	Stearic	14.1%	2.3%
282	Oleic	49.6%	7.5%
280	Linoleic	2.5%	Trace
	Other	....	1.6%
	Unsaponifiable	0.1%	0.2%[c]
Average mol. wt. of F.A.		271.3	213.8
Average mol. wt. of glyceride		852	679
Saponification value		197	248
Alkali to saponify (NaOH)		14.1%	17.7%
Total fatty acids of glyceride		96.0%	94.5%
Glycerine		10.7%	13.5%

[a] Biochem. J. *25*, 1168-82(1931).
[b] J. Biol. Chem. *130*, 167(1939).
[c] Nominal value added.

The weighted average molecular weight of the fatty acids of the tallow is 271.3, which can be calculated to give an average glyceride wt. of 852 for a saponification value of 197. The saponification value of a neutral fat or oil then can be used to compute the average chain length of the constituent fatty acids, the average molecular weight of the glycerides, the percentage of total fatty acids, percentage of alkali required for saponification, and the percentage of glycerine present. In the above case the caustic to saponify is 14.1%, the total fatty acids 96.0%, and the glycerine 10.7%. The presence of unsaponifiables affects the calculation for the percentage of glycerine and must be taken into account when it is high or when very accurate results are necessary. Coconut oil, as a typical analysis shows, is largely lauric acid. A neutral glycerine of this fatty acid composition would have a saponification value of 248, an average glyceride molecular weight of 679, a caustic requirement for saponification of 17.7%, a glycerine content of 13.5%, and a total fatty acid content of 94.5%.

Fats and oils usually contain free fatty acids. For general purposes such acids may be considered as having the same average molecular weight as those present in the oil. When a fat is partially hydrolyzed to fatty acids, then the caustic to saponify still remains the same as that calculated from the saponification value. This value increases with the free acid and finally becomes 207 for a tallow of the composition given. The total fatty acids would finally become 100% less the percentage of unsaponifiable. The percentage of glycerine would be zero. For a 50% free fatty acid tallow of the above composition the saponification value would rise to 202, the total fatty acids to 98%, and the glycerine content would fall to 5.35%. This last value is only approximate since a fat does not hydrolyze with such uniformity and the only way to obtain the true glycerine content is by an analysis.

In normal soap boiling the alkali in the form of an aqueous solution is brought into contact with the fat at elevated temperatures. The first reaction takes place at the oil water interface to form some soap. If free fatty acids are absent as in a refined fat, this initial reaction can take considerable time. If, as is normally the case, free acids are present, soap is immedately formed. As soon as some soap is formed, it acts as an emulsifying and solubilizing agent and the reaction then proceeds smoothly as a homogeneous one. The work of McBain and others indicates that there is no relation between the molecular weight of the glyceride and the rate of saponification, the latter depending more on the ease with which the oil is emulsified after some soap is formed. Such factors are somewhat, although not wholly, dependent on the solubility of the soap, which in turn increases greatly as the molecular weight decreases. Sodium stearate, for example, is more insoluble in water than sodium laurate. The addition of salt or the reduction of water in the lye, which is another way of increasing the electrolyte content, can decrease this solubility. This decrease in water solubility of the soap is important and is under the control of the operator by the addition of brine along with the lye. Soap thus partially grained out is improved as an oil emulsifying agent, and for this reason the addition of salt is extremely important in kettle house boiling operations.

Soap exists in a variety of forms and the application of phase diagrams helps us considerably in understanding just what happens, not only in the kettle but also in subsequent finishing operations such as plodding. The soap boiler recognizes such aqueous systems of soap. For example, there are neat soap, which is the finished kettle soap and is an anisotropic liquid; nigre soap, which is an isotropic solution; soap curd, which is dried soap consisting of fibrous crystals enmeshing a liquid phase; and finally middle soap, which is a viscous dilute crystalline phase. To these may be added such further phases as superneat soap and kettle wax. The following example (Figure 1) from the work of J. W. McBain and W. W. Lee, Oil & Soap *20*, 24 (1943), shows the system sodium oleate and water at varying concentrations and temperatures. The shaded areas represent mixtures of soap phases, determined from the boundary phases connected by each isothermal tie line. The unshaded portion represents single phases. At a temperature of 100° the various phases encountered with change in concentration are as follows:

0- 31%	isotropic solution
31- 34%	isotropic solution and middle soap
34- 42%	middle soap
42- 43%	middle soap and isotropic solution
43- 44%	isotropic solution
44- 69%	isotropic solution and neat soap
69- 86%	neat soap
86- 94%	neat soap and waxy soap
94- 96%	waxy soap
96- 98%	waxy and subwaxy soap
98-100%	subwaxy soap

Similarly, at any one concentration of soap, the various phases that may exist at varying temperatures may be found. At a 65% concentration, for example, soap boilers neat soap exists as a single phase at a temperature of 36 to 54°C. and again at 130 to 175°C. Other investigations including the work of McBain, Vold, Ferguson, and others have shown that anhydrous soaps exist in an even greater variety of phases and polymorphic forms, beyond the scope of this discussion.

Of more present help perhaps is the phase study of the system soap-water-salt at 100°C. An example cov-

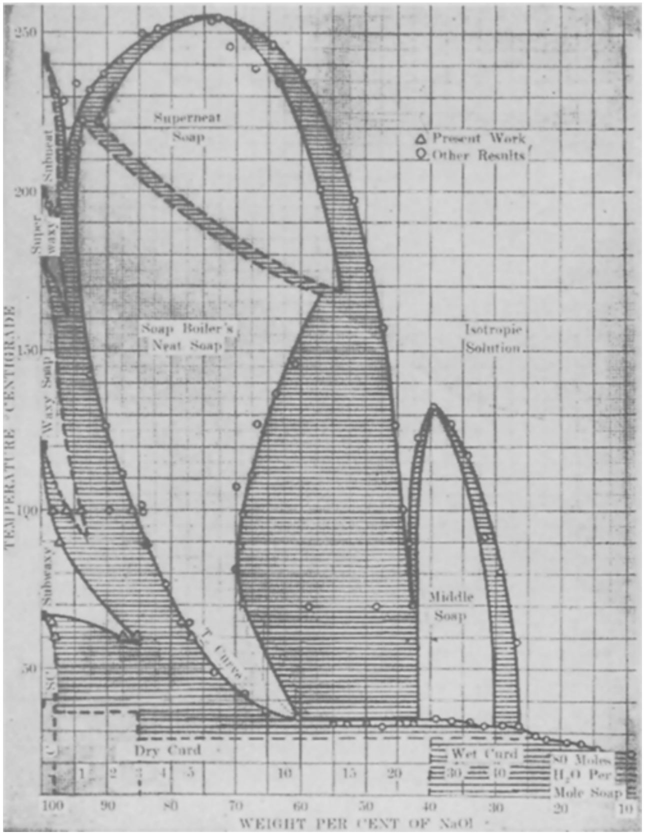


FIG. 1. Phase diagram for sodium oleate showing homogeneous fields of each of the different phases and the isothermal tie lines connecting the phases in heterogeneous equilibria.

ering tallow is shown in Figure 2 from the work of R. H. Ferguson and A. S. Richardson, Ind. & Eng. Chem., Vol. 24, page 1330 (1932).

Considering the diagram, Figure 2, it is seen that when zero salt is present, we pass through the following phases with increase in soap concentration as follows:

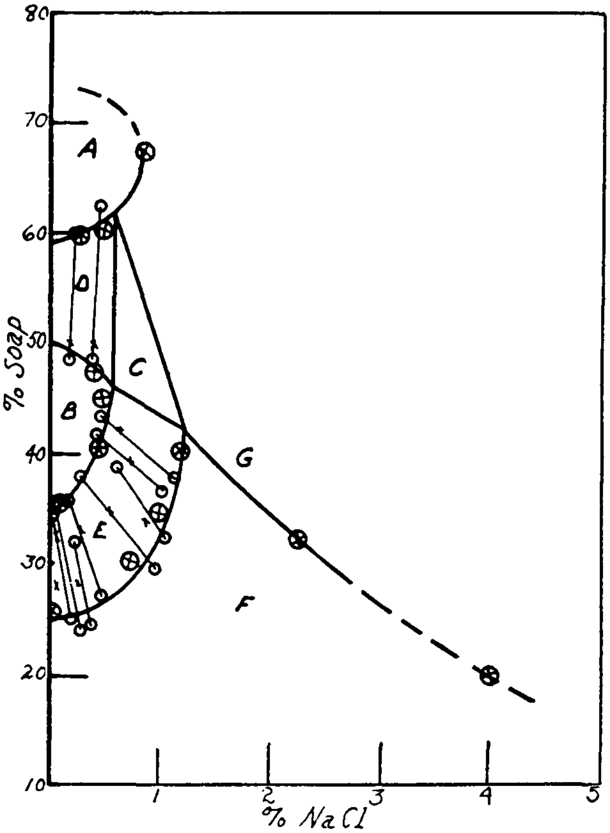
Area F	Niger	25%	(approx.) conc. of soap
Area E	Middle-Nigre	25-35%	(approx.) conc. of soap
Area B	Middle	35-50%	(approx.) conc. of soap
Area D	Neat and Middle	50-58%	(approx.) conc. of soap
Area A	Neat	58%+	

Middle soap is a viscous mass which is very difficult to handle and which the soap boiler avoids at all costs. From the diagram it is seen that he can get into trouble if his salt content falls below 1¼%.

Kettle Soap

The main operations in kettle soap boiling may be classed in three parts: a) the saponification or killing change, followed by one or two washes; b) the strong change; and c) finishing operations. A typical kettle, shown in Figure 3, is an insulated cylindrical steel tank with a diameter about half its height, a cone bottom, open and closed steam coils controlled from the top, a bottom outlet and circulating pump, and a skimmer pipe for removal of the finished soap. The one diagrammed also has a cover and stack for vapor exit, a side entrance for salt, and a caution and warning light if the soap level exceeds a fixed height. The oil charge is roughly not more than 25% of the total water weight capacity of the kettle to allow plenty of boiling room.

As the oil is being pumped into the kettle, a very weak solution of about 1% caustic alkali is added. The rate of lye addition must be very carefully regulated with the flow of oil. Usually the high fatty acid oils are added to the kettle first. This is to enable the saponification to start quickly. The acids unite with the lye quite readily, forming soap, which emulsifies the fresh oil and enables them to come into intimate contact, thus aiding further saponification. If the lye is added too rapidly, the emulsion will be broken and the saponification retarded. This is brought about by the graining-out action that the lye has on the soap because of the common ion effect. (Salt has the same action as will be discussed later on.) If the lye solution is too dilute, saponification will also be retarded. After saponification has once begun or, in terms of the soap-boiler, a catch has been made, the heat of the action speeds it up. This heat is considerable and amounts to about 65 to 75 Cal. per kg. of fat saponified. The lye that is added is called killing-seat lye, and after a sufficient amount has been added to saponify 90% or more of the oil, enough salt is added to grain the soap. The amount of salt required is very small since the alkali added is generally strong in salt to keep the soap phase in a neat condition and promote saponification. If fresh caustic of 15° Bé or so is used, brine of 20° Bé or so is also added along with it. Where possible, the soap boiler saponifies his fresh charge on top of the nigre or bottom of a previous kettle. The presence of the soap increases the emulsification of the oil and lye and promotes a smooth and



SYSTEM TALLOW SOAP–SODIUM CHLORIDE–WATER

A.	Neat soap	*F.*	Nigres
B.	Middle soap	*G.*	Neat-nigre
C.	Neat-middle-nigre	*O-O.*	Two-phase systems
D.	Neat-middle	⊗.	Concentration at which second phase appears or disappears
E.	Middle-nigre		

FIG. 2

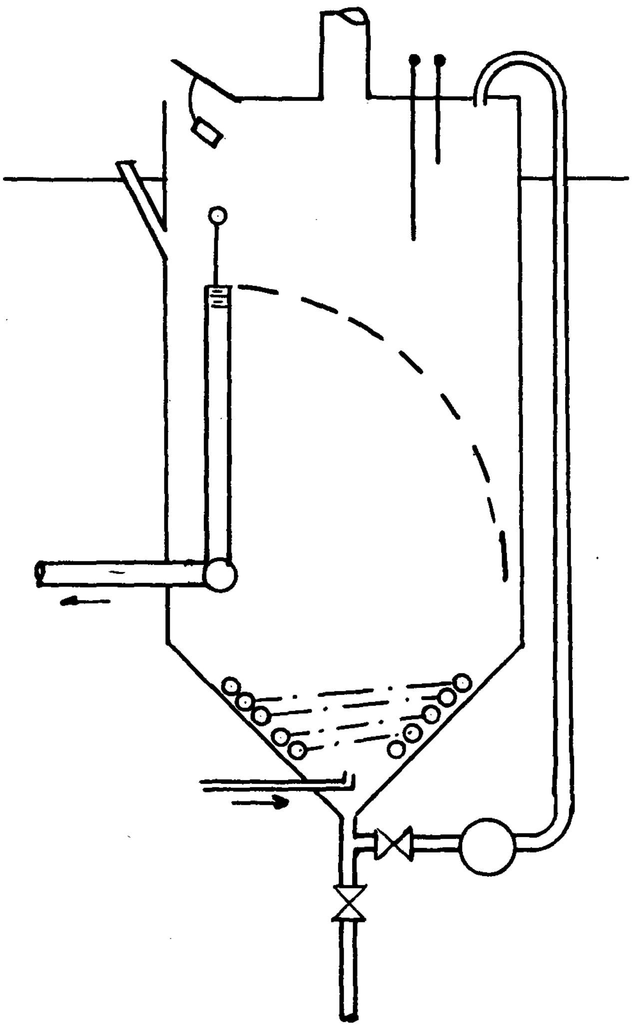


FIG. 3

rapid saponification. With coconut oil great care must be taken not to get too much stock into the kettle before saponification starts as the rapidity of saponification may release sufficient additional heat to cause the kettle to boil over. The curd soap after saponification is dry and hard when pressed between the fingers and slightly alkaline to the taste. After standing three or four hours the killing-seat lye is drawn off the bottom and transferred to storage for subsequent glycerine recovery. This lye will be less than ½% free alkali, will run about 20 to 25° Bé, and will contain anywhere from 10 to 15% glycerine, depending on the system of washing and reuse of the lyes. It will also contain dirt, coloring matter, organic soluble residues, etc.

The soap may or may not be washed several times, depending on the quality of the product desired. This is done by boiling up the soap with the addition of water until the soap is in a closed condition and will fall in a smooth film from the trowel. Weak alkali is then added and the soap circulated for some time, after which it is grained with salt and settled.

The strong change is now given the soap charge in a manner identical to that for the wash, except that alkali anywhere from 10 to 14% Na_2O may be added. This insures the complete saponification of all fats. The kettle is now grained as before and let stand. The lye drawn off to storage.

The finishing of the soap consists of one or more brine washes known as "pickling" to remove glycerine and free alkali, followed by a final "pitch," which consists of boiling up on water alone and allowing to settle several days. At this point the salt and alkali content must be such that the soap boiler arrives at point H of Figure 4 from Phase Phenomenon in "Commercial Soap Systems," R. H. Ferguson, Oil & Soap, January, 1932. This diagram is the same as Figure 2 except that it shows the area G under conditions of higher salt concentration, where lye exists as a separate phase. That is, he wishes to have a large neat soap plus a small nigre plus lye, and he must adjust his water and salt content so as to procure these conditions. It will be noted from the digram that finished kettle soap will run consistently from 65 to 68% anhydrous soap. The nigres are 35-40% soap solution and contain the dirt and considerable coloring matter from the oil. They are usually reworked in lower grade soaps. The nigres of such low grade soaps are then accumulated and reworked to yield a nigre of a nigre, which last is discarded.

Although the phase diagram points up a rigid scientific basis for boiling operations, the individual modifications of the process are quite extensive. For example, if a 98% or higher saponification is obtained on the killing operation, a comparatively weak alkali wash will suffice for the strong change. Sometimes a nigre is removed after the strong change and a second one on the finish.

Some soapers use only salt for graining, some use salt and brine and some only brine. The British soap-

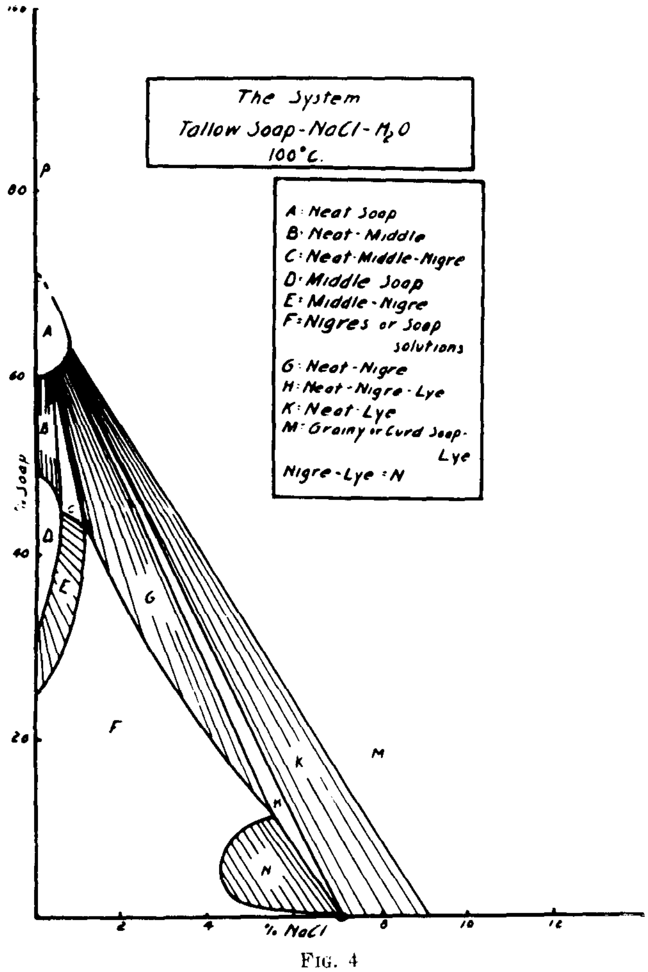


FIG. 4

makers use square shallow "pans" for soap boiling. For all these differences however each manufacturer follows a very definite procedure depending on the product to be made. His toilet soap base, for example, may get three weak washes before the strong change followed by a pickle. His laundry soap may get only one wash before the strong change and may be finished without a pickle. In each case however his operations will be controlled by the volume, baumé, and alkalinity of the lyes dropped.

Variations in the procedure are dictated by finished specifications, lye space, available glycerine recovery, and other factors. For example, the tabulation below shows typical kettle-house soap specifications on three classes of soap.

Typical Specifications for Kettle House Soaps

	Toilet Base	Laundry Bar Base	Industrial Flakes Base
Real Soap	67.0% min.	67.0% min.	65.5% min.
Free Na_2O	0.03-0.05%	0.3-0.5%	0.2-0.4%
Glycerine	0.3% max.	0.2% max.	0.7% max.
NaCl	0.40-0.50%	....	....

When rosin is used in the charge, it may be added over the top if small, such as 10% of the total charge. In this case it usually is added with the fats or directly after the saponification. When large amounts of rosin are used, they are generally saponified separately with less expensive soda ash, washed, and grained. The lyes are dropped to the sewer since they contain no glycerine. The soap is then half-grained and pumped on top of the saponified fat charge, where it is strong-changed and finished in the usual manner.

The amount of glycerine recovered is of very great importance, and as little as possible is left in the soap commensurate with economical recovery. For example, it was shown previously that an average neutral tallow contained about 10.7% glycerine. If such tallow is converted to a soap analyzing 0.5% glycerine and 67% anhydrous soap, the overall loss of glycerine left in the soap is 7% of the total. It is not uncommon for a well-run soap plant to lose as much as 10% glycerine by leaving it in their kettles. On the other hand, it is possible to pursue this recovery to a point where the increase in lye volume may overtax the equipment available, or cost of the additional evaporation may exceed the price of the crude obtained. For example, the steam consumption required for evaporation of a 5% glycerine lye is about three times as much as it is for a 12% glycerine lye. Cases have been known where soap washing operations were cut to avoid high costs of recovery. The total volume of lye evaporated bears a very simple relation to the fats saponified. For example, if the average fat charge contains 11% glycerine and the lye produced also contains 11% glycerine, then there is one pound of lye produced for each pound of fat saponified. If such conditions produce an average known loss of glycerine left in soap of say 7%, then greater washing of the soap will produce a smaller loss (say 5%), a lower glycerine content of the lyes, and a greater volume of lye, up to say 1.3 lbs. of lye per lb. of fat saponified if kettle-house conditions remain the same. The system of lye handling in the kettle house also determines largely the efficiency of recovery, and it is frequently the practice to use the lye from the strong change on the second wash of the next kettle and the first wash of a third, etc. This is

SHARPLES CENTRIFUGAL SOAP PROCESS

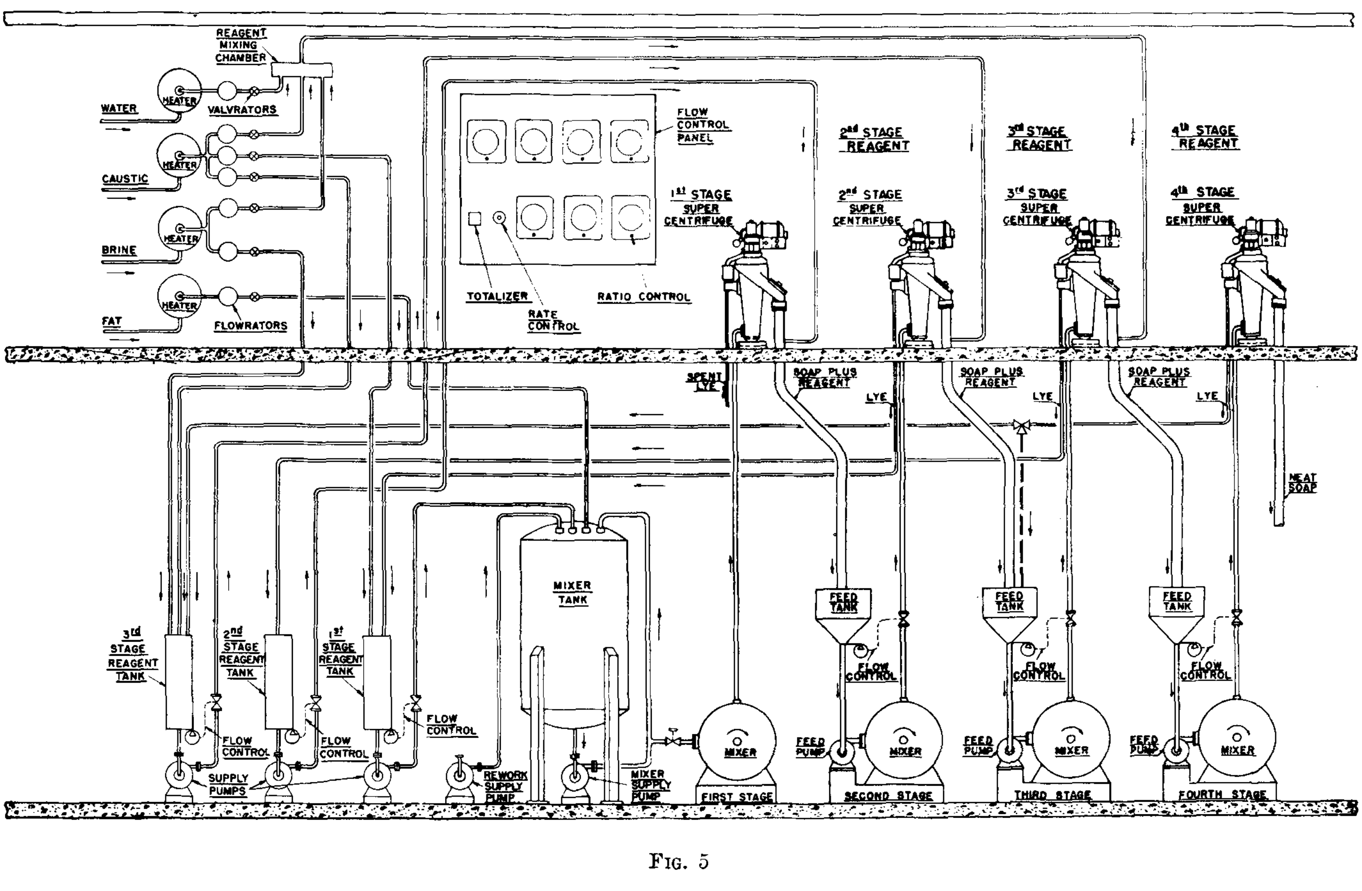


FIG. 5

known as the countercurrent system of lye handling. Cases have been known where such a system will reduce the lye to fat volume from a ratio of 1.0 to 0.7, yielding glycerine lyes as high as 15% while still keeping the known loss of glycerine below 7%. In certain types of operation the countercurrent system of lye transfer is not always feasible as, for example, when production is limited and contains various types of soaps.

Generally speaking, the yield of kettle soap will be between 98.5 and 99.5% of theory with primary fats and oils as raw materials. With large quantities of foots, yields of both glycerine and soap will be adversely affected. The quality of the finished soap is a joint responsibility of the boiling operations and raw material selection, and each must stand on its own feet.

Other methods of saponification exist which modify regular kettle house practice. In the semi-boiled process for example, the soap is saponified in a crutcher with the requisite amount of lye added. Builders are added and the whole mixed and dropped to frames. The entire glycerine goes with the soap. This type of operation is exceedingly rare.

Cold-made soap is processed on the same principle as semi-boiled soap. For example, coconut oil having a saponification value of 260 would require 18.5 pounds of NaOH for each 100 pounds of oil used. If a 60% superfatted soap product is desired, a little arithmetic shows that 56.5 pounds of water will be needed for the alkali. By holding out one pound of NaOH and replacing it with an equal weight of water, the desired product is obtained having a superfat of 5.3%. The fat is charged to the crutcher and the alkali solution added slowly. When saponification begins, it may be necessary to cool the crutcher with water due to the heat released through the saponification.

Continuous soap making deserves special attention. The Emery-Colgate and Procter and Gamble methods go through the hydrolysis to fatty acids and therefore do not form a part of this discussion. In the Sharples method the fat charge plus alkali and brine solutions of the proper strength flows continuously through a mixer. The resultant soap is then separated by centrifuges into neat soap and lye. The soap is then strong-changed and washed several times in succession by means of intermediate mixers and centrifuges and comes out a finished product, having the same specifications as a kettle soap.

A flow sheet of this very interesting process is shown in Figure 5. It is not difficult to trace the various steps back to fundamentals of kettle soap practice. Stage 1 for example is the saponification, stage 2 corresponds to the 1st, 2nd, and 3rd wash, stage 3 corresponds to the strong change, and stage 4 to the finish. The principal point of interest to note in this method is the lack of a nigre. This is not necessary as the dirt and coloring matter usually associated with nigres in kettle boiling are accumulated on the inside of the centrifugal bowls and are removed periodically. All equipment is integrated into a single complete unit by means of electric controls and proportioning pumps.

The Clayton Process, which has not been widely used, saponifies the fat and alkali in a mixer under high temperature and pressure. The product is then sprayed into a vacuum chamber where the water and glycerine are stripped off. The anhydrous soap is then hydrated to the proper degree and finished by plodding, etc.

A similar system is that described by Lorenz (U. S. Patents 2,084,446, 2,096,188, 2,232,544, and 2,262,950), and others (U. S. Patents 1,874,388 and 2,136,283), where the fat and alkali are brought together in the spraying tower itself.

The Monsavon system of soap making (U. S. Patents 2,485,204-5) utilizes a colloid mill to emulsify the fat and the aqueous alkali phase. These are discharged to a vertical reaction column. The saponified fat then passes into a washing column and hence to a settling tank for separation into neat soap and nigre.

Still another method reacts the alkali and fat in a nonaqueous medium, such as kerosene (U. S. Patents 1,753,659, 1,813,454, and 2,380,650). The kerosene solvent-fat-alkali mixture is sprayed into a vacuum chamber as in the case of the Clayton process, and the separation of solvent, water, and glycerol from anhydrous soap is made. Also J. K. Gunther in U. S. Patent 2,401,756 describes a similar type of process. Doubtless some of these methods may be used in the future when technical difficulties have been overcome.

Other methods of making hard soaps is by the saponification of esters formed in turn by alcoholysis of a fat (U. S. Patents 2,271,619, 2,360,844, 2,452, 724-5, and 2,383,632). This is not believed to be in commercial use at present.

The following U. S. Patents on continuous and semicontinuous methods of saponification may be of interest to those who would like to pursue this subject somewhat further.

A Few Select U. S. Patent References on Continuous and Semi-Continuous Methods for Soap Making.

1,753,659 (Kokatnur—kerosene)
1,813,454 (Kokatnur—kerosene)
1,874,388
1,918,603 (Colgate)
2,019,775-6 (Clayton)
2,037,004 (Clayton)
2,037,006 (Clayton)
2.084,446 (Lorenz)
2,096,188 (Lorenz)
2,123,647 (Clayton)

2,136,283
2,137,213 (Clayton)
2,139,589 (Colgate)
2,142,982 (Clayton)
2,146,661 (Clayton)
2,154,835 (P & G)
2,156,863 (P & G)
2,159,397 (P & G)
2,178,532 (Clayton)
2,178,987-8 (Clayton)

2,179,001 (Clayton)
2,185,653 (Clayton)
2.190,591-2 (Clayton)
2,190,615-6 (Clayton)
2,193,786 (Clayton)
2,195,377 (Clayton)
2,218,279 (Clayton)
2,221,799 (Colgate)
2,232,544 (Lorenz)
2,233,845 (P & G)

2,235,628 (Clayton)
2.239,131 (Clayton)
2,242,187 (Clayton)
2,245,536 (Clayton)
2,245,538 (Clayton)
2,249,675-6 (Clayton)
2,254,996 (Clayton)
2,262,950 (Lorenz)
2,267,750 (P & G)
2,271,406-7-8 (Clayton)

2,271,619 (DuPont-Bradshaw & Meuly)
2,281,534 (Colgate)
2,283,776 (Clayton)
2,287,884 (P & G)
2,299,603 (Clayton)
2,300,749-50-1 (Sharples)
2,302,382 (Sharples)
2,321,947 (Sharples)
2,335,457 (Sharples)
2,336,893 (Sharples)

2,343,829 (Clayton)
2,348,855 (Sharples)
2,360,844 (DuPont-Bradshaw & Meuly)
2,369,257 (Sharples)
2,369,372 (Sharples)
2,380,650 (Jacobs—kerosene Cont.)
2,383,632 (Colgate Alcoholysis)
2,397,161-2 (Sharples)
2,401,756 (Gunther)
2,411,468-9 (Sharples)

2,435,745 (Colgate)
2,452,724-5 (Bradshaw Alcoholysis)
2,458,170 (Colgate)
2,480,471 (Colgate)
2,485,204-5 (Monsavon)
2,486,630 (Emery)