struck. Soap in this condition is termed "crystal" or "crystal base." Liquid potash soap made from coconut oil fatty acids and free from glycerol begins to gel at about 37.5% and is a firm crystal base at 40.5% concentration. Hence a glycerol-free crystal base has less total solids by oven dry test than one made from coconut oils, but the two are practically equal in the amount of combined fatty acids.

These gelled soaps of the crystal type have some peculiar properties. They are liquid when hot and solid when cold. They remain in the crystal state through the range of useful temperatures from about 5°C. to 80°C. When the crystal base type of soap is cooled to a low temperature, these soaps again liquefy and become quite thin. Liquid soaps that are so concentrated that they are passing from the liquid state to the crystal state first become thick and viscous but upon chilling return to a thinner liquid. For the present it is assumed that this condition explains the reversal in the viscosity of a liquid soap, curve 1, below 15°C. (Figure 4).

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Methanolysis of Triglycerides by an Anion Exchange Resin[1]

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IN a preparation of radioactive glycerol (1) Amberlite IRA-400-OH was used to liberate the carbon-labelled glycerol from dicaproin. The reaction was carried out in methanol, and the water-soaked resin provided more than the amount of water required for the hydrolysis. In addition to the free glycerol a large amount of methyl caproate was found, indicating that interesterification had occurred rather than the expected saponification.

Catalysis by cation exchange resins in esterification, interesterification, and saponification has drawn considerable attention recently (2, 3, 4). Interesterification by anion exchange resins, to our knowledge, has not been reported. In addition to any possible preparative value it seemed to be desirable to learn more about the effect of anion exchange resins upon fats for anion exchange resins have already proven to be of value in the analytical phase of metabolic studies (5).

Two detailed experiments were conducted on cottonseed oil, identical in all respects except that the same catalyst resin recovered from the first experiment was used in the second experiment rather than fresh resin. Cottonseed oil, 55 g., was dissolved in 250 ml. ether plus 250 ml. ethanol. When 20 g. of moist Amberlite IRA-400-OH-AG resin were added to the solution, two liquid layers formed. As the reaction proceeded, the liquid again became homogeneous. After three days' shaking the resin was filtered off and washed with the same solvent. When the sample was reduced to dryness under nitrogen, the oil contained some large drops of glycerol. Free glycerol and monoglyceride were determined according to the procedure of Pohle and Mehlenbacher (6) on the aqueous extract of this mixture and upon the residual oil. Total bound glycerol was determined by periodate titration after saponification of the oil and separation of the fatty acids from the glycerol (7). Blanks run with methyl palmitate gave negligible values. The recovery of oil was 52.9 and 53.9 g. in the two experiments. The analytical results are listed in Table I. Probably the interesterification proceeds

TABLE I

Methanolysis of Cottonseed Oil by Amberlite IRA-400-OH-AG

	Mg. Free Glycerol per g. Oil	Mg. Bound Glycerol per g. Oil	Minimum Interesterification
			%
Cottonseed Oil	.....	108.2, 104.5	....
Interesterified			
Mixture I	69.5	16.9, 17.3	84
Mixture II	75.0	30.2, 29.5	72

stepwise. The periodate values however were raised only slightly from 0.071 meq. per g. cottonseed oil to 0.098 and 0.090 meq. per g. interesterified oil, indicating that under the above conditions the larger portion of acids not converted to methyl esters remained as tri- and diglycerides. Since the method of determining the total bound glycerol does not give account of partial interesterification, the values calculated on that basis are minimum values. It seems justifiable to base this calculation on the residual bound glycerol rather than on the recovered free glycerol. The amount of glycerol held back by the resin has not been determined, and the increase of both free and bound glycerol in the second experiment would be explained by such effect.

Free fatty acids were found only in the resin. After the second catalysis the resin was shaken with aqueous hydrochloric acid and ether for several hours, and 0.89 g. of a semisolid oil was obtained, having an acid value of 166. The capacity of 20 g. of resin at saturation is about 6.9 g. stearic acid.

In other experiments corn oil, coconut oil, and cod liver oil were treated. The interesterified oils were analyzed for bound glycerol only, calculating therefrom the percentage of interesterification. The re-

TABLE II

Alcoholysis of Triglycerides

Oil	Solvent	Resin Wt.	Time	Interesterification
			hrs.	%
Cottonseed 50 g.	Methanol, Skelly B.	20 g.	48	73
Corn 45 g.	Ethanol, ether	20 g.	72	61
Cod liver 50 g.	Methanol, ether	20 g.	48	67
Coconut 50 g.	Methanol, Skelly B.	20 g.	48	74

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sults are shown in Table II. When only methanol was used as solvent during the interesterification, the results were inconsistent. Addition of another solvent to increase solubility of the fat improved the results. The reaction proceeded rather slowly with most of the fats. However when tricaproin was treated, the characteristic odor of methyl caproate was immediately apparent. The water content of the resin did not markedly affect the course of the reaction since catalyst which had been repeatedly soaked in methanol gave essentially the same results. The amount of monoglyceride found in these experiments was very low. Resin saturated with fatty acid has no catalytic activity. The resin does not catalyze the esterification of free fatty acids with methanol, and saponification of esters occurs to only a negligible extent.

From these observations it appears certain that the free fatty acids have no role in the course of the interesterification.

The interesterification by resin catalysis suggests a strong parallel to the interesterification catalyzed by alkali (8, 9). The heterogeneous system of solid caustic resin and the fat solution may account for the low reaction velocity. The amount of resin used, when expressed in equivalents of alkali, is about four times the amount recommended for interesterification by means of sodium ethylate or sodium hydroxide. A remarkable distinction between the resin catalysis and alkali catalysis is the indifference of the resin catalysis to amounts of water which would interfere with the latter type of reaction.

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A New Alkali Isomerization Procedure

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THE standard procedure described in the 1948 Report of the Spectroscopy Committee (2) has many generally recognized shortcomings. Improvements have been proposed. We are indebted to Brice *et al.* (1) for more adequate spectroscopic constants and for a pertinent suggestion as to the optimum time of reaction. The application of the procedure remained however what it was originally; a long, complicated, exacting series of manipulations requiring above-average skill and experience. Even under the best circumstances the accuracy is in no way remarkable. As a tool in the kinetic study of the alkali-isomerization reactions it is poor. A time lag results from the fact that the sample does not dissolve instantaneously on coming in contact with the hot reagent. This lag cannot be determined because of undefined variations, resulting from the manual handling of the reaction tubes at the time solution takes place. Clearly there was a need for:

a) An apparatus allowing simultaneous application of an identical treatment to samples and blanks
b) A procedure which would eliminate the time lag resulting from delayed solution
c) A procedure allowing the same treatment to be easily reproduced from run to run.

Moreover, to make it a more practical, reliable, and accurate method, steps should be eliminated entirely or extensively modified. Among the steps to be eliminated were:

a) The cumbersome and time-consuming transfer of the reaction mixture to the volumetric flasks
b) The use of specially purified absolute alcohol as solvent.

Among the features needing improvements were:

a) The nitrogen purifying train
b) The handling of the reagent.

The authors undertook the present work in order to find a solution to these problems. The present article describes the outcome as follows:

Under Apparatus:

a) A reagent handling unit allowing preparation, storage, and dispensing
b) A new nitrogen purifying train
c) A new alkali-isomerization apparatus.

Under Determination of Δ m:

A procedure for the determination of a correction, Δ m, bearing on the reaction time and allowing lag effects to be eliminated.

Under Suggestion for a New Procedure:

A simple protocol, allowing the faithful reproduction of conditions.

Apparatus

Figure 1 shows the relative position of each piece of apparatus on the laboratory table (top view). The

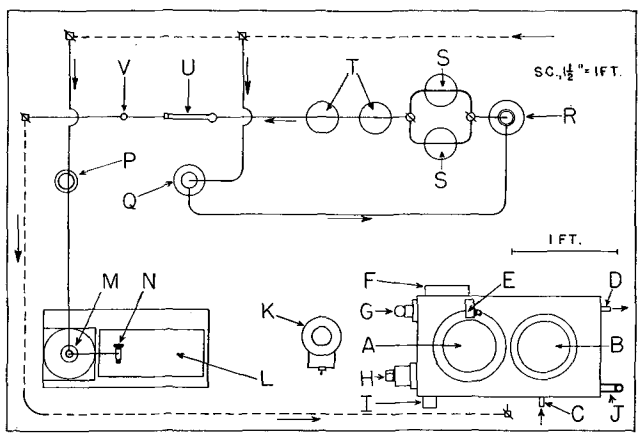

FIG. 1. General lay-out of isomerization equipment.