

TABLE I

The Effect of the Route Taken to Reach Equilibrium (Partial Acidification) on the Resultant Fractionation

Starting material	Neutralization history	IV of liquid acids
Tallow fatty acids IV 63	(a) 1.0 eq. NaOH, then 0.5 eq. HCl	91.4
	(b) 1.0 eq. NaOH, then CO ₂ to pH 7.5	98.8
Fish fatty acids IV 196	(a) 1.0 eq. NaOH, then 0.5 eq. HCl	233
	(b) 0.5 eq. NaOH	236
Mixed vegetable Dist. fatty acids IV 83	(c) 1.0 eq. NaOH, then CO ₂ to pH 7.5	238
	(a) 1.0 eq. NaOH, then 0.5 eq. HCl	119
Soybean fatty acids IV 122	(b) 0.5 eq. NaOH	117
	(a) 1.0 eq. NaOH, then 0.5 eq. HCl	146
	(b) 0.5 eq. NaOH	143

Obviously the required equilibrium for the formation of acid soaps can be approached by several means without greatly affecting the resultant fractionation, and that the cheaper inorganics can be used instead of CO₂. It was surprising that one equivalent of fatty acids would dissolve in only 1/2 an equivalent of aqueous alkali.

The Physical Separation of Acid Soaps on a Larger Scale. While with flotation and centrifugation some separation of the solid acid soaps was effected, it was felt that a more positive separation method was required. Consequently, filtration was again studied.

As reported in Part I, filtration without a filter aid was impossible. Even with a filter aid in considerable quantity, the filtration rate was slow and produced unacceptable solids. However the work on settling gave the know-how necessary to grow a superior type of crystal, and it was expected that some degree of success would be attained with filtration. Since washing was essential to produce low IV solids, a continuous precoat vacuum filter was selected for the test work. This was an Oliver 12 x 2 inch Laboratory Precoat Filter. Table II gives the filtration rates obtained.

TABLE II

Pilot Plant Filtrations of Acid Soaps on an Oliver Continuous Precoat Filter (Drum Speed—15 rph)

Fatty acid raw material	Soap soln. %	Degree of shave in./rev.	Duration of test hr.	Filtration rate gal/ft ² /hr	IV of Solid acids	IV of liquid acids
Fish oil IV 196	6.4	0.010	1 1/2	6.5	88	238
	4.0	0.010	1 3/4	9.4	72	236
	5.3	0.005	1 1/2	5.9	41	225
Cottonseed Fatty acids IV 96	4.7	0.010	3/4	13.5	24	122
	4.7	0.005	2	6.0	25	122
Soybean fatty acids IV 122	5.6	0.010	2 5/8	9.8	70	136

While the filtration rates were only fair, it was found that the use of a precoat continuous filter for the solid acid soaps was practical.

The Application of the Acid Soap Separation to Stocks Other than Tallow. Since this process works for separating the fatty acids present in tallow, it would be expected to perform similarly on cottonseed, fish, and soybean fatty acids. A number of examples of the fractionations of other fatty acids have pre-

viously been cited. Table III shows further results on the various raw materials tested.

TABLE III

The Separation of Distilled Fatty Acid Mixtures

Fatty acid raw material		Separation temp. °C.	Solid acids IV	Liquid acids		
Type	IV			IV	% Yield	
Cottonseed	96.0	6	24.2	121	74	
	96.0	10	24.4	122	73	
	98.8	5	41.7	135	61	
	98.8	5	25.9 ^a	135	67	
	98.8	15	35.8	129	68	
	98.8	15	20.6 ^a	128	73	
	98.8	25	32.1	119	77	
	98.8	25	16.0 ^a	118	81	
	Fish	196	5	88.4	238	72
	Mixed vegetable	82.5	9	13.2	122	64
Soybean	120	10	57.0	140	76	
Tallow	43.4	12	17.8	85.2	38	
	56.9	5	20.0	101	46	
	56.9	15	17.5	96.4	50	
	56.9	25	19.4	86.2	56	
	56.9	25	14.9 ^a	88.0	57	
	63.0	11	30.4	98.8	48	
	63.0	11	25.7 ^a	98.8	51	

^a Indicates that the solid acids were washed to free them of mother liquor.

Since this process entails the formation of a soap solution, it might naturally be expected that it would find its most economic application in the fractionation of the foots produced on refining vegetable and fish oils. Here the alkali necessary for the operation of the process is already present, and acid has to be added in any case. Consequently, it was attempted to fractionate raw soapstocks that had been further treated with alkali to saponify the neutral oil present. See Table IV.

TABLE IV

The Separation of Raw Soapstock

Soapstock raw material		Separation temp. °C.	Solid acids IV	Liquid acids	
Type	IV ^a			IV	% Yield
Fish	172	9	33.5	227	72
Cottonseed	103	5	41.6	132	67
	103	15	72.1	127	54
	103	15	42.8 ^b	129	69
	103	25	45.9	114	84
	104	8	47.3	136	64
Peanut	90.5	15	16.9	103	86

^a IV of the total fatty matter present.

^b Indicates solids acids were washed to free them of mother liquor.

It is evident that this separation procedure can be used to advantage on foots as well as distilled and undistilled fatty acids (2).

In handling soap stocks, the electrolyte content of the completely saponified solution prior to fractionation must be watched. Whereas the fractionation will work in the presence of 5 g per l of sodium chloride or sulphate, twice this amount will prevent the process from operating at all.

REFERENCES

- Munns, W. O., S. Kairys, Dilys A. Manion, and E. M. Meade, *JAOCS*, **39**, (1962).
- U. S. Patent No. 2,895,976—S. Kairys, E. M. Meade, W. O. Munns, and Dilys A. Walder.

[Received November 27, 1961]

Erratum. Vol. 39, page 70, January, 1962, LYON ET AL.: SOLVENT-BLOWN, RIGID URETHANE FOAMS. The passage beginning with the last 1 lines, first column, on page 70 should read:

The average equivalent weight of the polyol mixtures was varied from 70 to 120 and castor oil content from 11 to 58%.

The compressive strengths of foams prepared from

these polyol mixtures are shown in Fig. 1. Here the compressive strength of foams calculated to 2 lb./ft.³ density is plotted *versus* average polyol equivalent weight. The castor oil content of the polyols used is indicated below the curve. The prepolymer used was the same as that described previously (6) and contained 10.3% pentaerythritol monoricinoleate, 10.3% trimethylolpropane and 79.4% toluenediisocyanate.