

kept at this temperature, and cooled back to room temperature, there was no evidence of fat bloom.

Summary

Technical considerations indicate that cocoa butter-like mixtures can be prepared readily by the esterification of mixtures of oleic, palmitic, and stearic acids, or the interesterification of their glycerides, followed by the fractional crystallization of the reaction products.

Using the indicated procedures, three cocoa butter-like fractions were prepared. One consisted essentially of oleopalmitostearins, another consisted essentially of oleodistearins, while the third consisted mostly of oleodipalmitins.

On the basis of softening point curves, the oleopalmitostearin product was most compatible with cocoa butter, the oleodistearin product was the next most compatible, while the oleodipalmitin product was least compatible. When mixed with cocoa butter, all three of the products produced consistency *vs.* temperature curves whose shapes closely resembled that of cocoa butter. All of the mixtures softened

over a short temperature interval though the actual temperature at which softening occurred varied. The several products are believed to be satisfactory cocoa butter replacements.

Another cocoa butter-like fat was prepared by the interesterification of 70 parts of completely hydrogenated cottonseed oil and 30 parts of olive oil and the subsequent fractionation of the reaction product.

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Solution Hold-Up as a Factor in Oilseed Extractor Design¹

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FOR THE USUAL percolation extraction scheme, employing fixed or moving beds, the number of equilibrium contacts (or stages) is primarily a function of the hold-up of solution throughout the system. An equilibrium contact is defined as the contact after which the composition of the solution retained by the solids is the same as the wash solution. In addition, solvent ratio, concentration of final miscella, and carry-over of solution between stages and in the spent solids are all dependent upon hold-up. Obviously hold-up of solution by the solids undergoing extraction is one of the fundamental properties of the solvent-oil-solids system, and sufficient data to define this property quantitatively must be known before any rational approach to design can be made.

Although several methods for determining the number of stages required for a given system under both equilibrium and nonequilibrium conditions may be found in the literature (1, 2, 3, 6, 7), the number of references containing factual information on hold-up is not great. Graphs of hold-up during draining have been reported by Smith (8) and Karnovsky (4) for beds of soybean and cottonseed flakes. Recently Kocatas and Cornell (5) published the results of their work for the soybean flakes-hexane system under conditions very similar to an actual commercial unit. Even so, designers have had to obtain the necessary data themselves or to base their designs on what might be termed, at best, an "educated guess."

The purpose of this paper is to present the data and correlations on hold-up which were developed from a series of pilot-plant runs (8) carried out at

Armour Research Foundation, Chicago, Ill. In the interest of simplicity the corresponding extraction rates are not included, except for the illustration in Figure 4. A sample problem is worked through in order to point out the application of hold-up data to the design.

Equipment and Materials

As illustrated in Figure 1, the apparatus used in

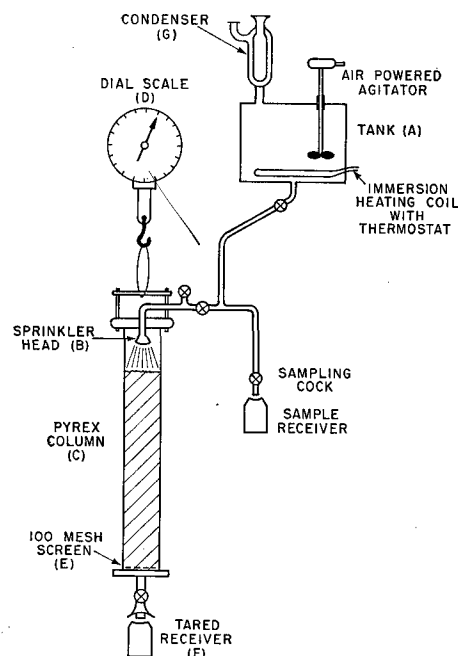


FIG. 1. Diagram of experimental equipment.

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TABLE I
 Characteristics of Flaked Seed Materials

Flaked seed material	Flake thickness		Analysis ^b		Analysis on dry screens, U. S. standard sieve no.								
	Nominal	Average ^a	Oil	Water	3	4	6	8	12	30	100	Through 100	Total
	<i>mils.</i>	<i>mils.</i>	%	%	<i>weight %</i>								
Soybeans	8	9.3	21.6	8.2	1.5	0	21.0	20.0	22.0	24.5	9.5	1.5	100.0
	15	17.4	20.6	9.1	2.5	0	42.5	22.5	16.0	12.5	3.0	1.0	100.0
Cottonseeds	8	8.4	30.4	4.5	0	1.5	1.0	6.0	6.0	40.5	41.5	3.5	100.0
	10	10.6	31.2	5.2	0	1.5	2.5	7.0	11.0	53.0	20.0	5.0	100.0
	15	14.0	32.2	6.4	0	0	5.0	9.0	14.0	48.5	23.0	0.5	100.0
	20	21.8	31.3	6.4	0	0	2.0	8.0	15.0	60.5	13.0	1.5	100.0

^a Average of 50 flakes selected at random in each lot.

^b Wet basis.

this work consisted of a thermostatically controlled, agitated, overhead scale tank, A, arranged to feed through a sprinkler head, B, into a 4-in. in diameter Pyrex column, C. This column was fitted at the bottom with a wire screen, E, of approximately 100 mesh and was supported from a dial scale, D, so that the column and contents could be weighed at any time. Miscella was permitted to flow by gravity percolation through the bed and was collected in a tared container, F. To minimize solvent loss by evaporation the feed tank was surmounted by a dry ice condenser, G, and the column was gasket-sealed at top and bottom. The size of the outlet (1.5-in. in diameter) was made large enough to avoid resistance to flow at this point.

The oilseed materials included soybean and cottonseed flakes and the presscakes from flaxseeds, peanuts, and sesame seeds. Properties of these materials as prepared for extraction are listed in Tables I and II. Bed depths of 3, 6, and 12 in. were studied, with an additional 36-in. bed for all except sesame presscake. The soybean flakes were furnished by the A. E. Staley Company, Decatur, Ill., and the cottonseed flakes by the Texas Engineering Experiment Station, College Station, Tex. The raw presscakes were prepared by arrangement with the V. D. Anderson Company, Cleveland, O., and were later ground to size with a small Quaker City attrition mill at Armour Research Foundation, Chicago, Ill.

In all instances the solvent was a commercial n-hexane fraction, Skellysolve B, with a density of 0.6716 g./ml. at 25°C. (77°F.). Where necessary, miscellas were synthesized from solvent and solvent-extracted crude oils. The extraction temperature was 60°C. (140°F.) except with cottonseed flakes, where a temperature of 70°F. (21°C.) was used for reasons concerning the color of the extracted crude oil (9).

Procedure

A sample of oilseed material equivalent to the desired bed depth was weighed and poured carefully into the column. At the same time the concentration of miscella in the feed tank was adjusted to corre-

spond to the beginning of a countercurrent extraction system (most concentrated). Solids and liquids then were heated to extraction temperature, sampled, and weighed. Following these adjustments, the miscella was sprinkled rapidly over the solids but at a velocity low enough to avoid "hollowing out" the surface by impact. When the first liquid appeared at the base of the column, the flow rate was regulated to hold the meniscus at the original level of the solids.⁴ All of the effluent was collected at 1-min. intervals in tared sample bottles. These were weighed, and the contents were analyzed for oil.

After 5 to 8 min. of percolation the column discharge valve was closed, sufficient miscella was added to flood the bed, and the mixture was allowed to soak for another 30 min. At the conclusion of soaking the solution was allowed to drain by gravity, and the rate of drainage was measured by weighing miscella that had drained after zero, 1, 2, 4, 8, 16, and 32 min. of draining time. Total draining time was 32 min. for the 36-in. beds only. "Zero" time was the instant that solid and liquid levels coincided. Hold-up after 16 or 32 min. was determined by subtracting the tare weight (column weight plus solids, plus moisture, plus oil retained on the walls of the apparatus) from the weight of the column plus the drained solids. Hold-up at earlier periods was obtained by adding to this figure the weight of miscella that had drained in the interval.

On completing the first extraction and drainage run, the solids were contacted immediately with miscella of lower oil content, and the procedure was repeated. In all, three or four runs were made with each material at each bed depth. The compositions of the successive wash solutions were chosen to simulate those at the beginning, middle, and end of a countercurrent system so that the final wash in each series contained somewhat less than 0.7% oil.

When the oil content of the solids was reduced below 3%, this procedure was not found to be the most appropriate one for determining rates of extraction. This was particularly true of the more difficult-to-extract seeds. A small sample of the solids from the last run was therefore weighed into a Soxhlet thimble and extracted with Skellysolve B. At intervals of a few minutes the extract was removed, and its oil content was obtained by evaporation. After four hours of extraction with Skellysolve B, the sample was removed, ground, and extracted with petroleum ether. This latter portion of oil removed by petroleum ether was considered "una-

 TABLE II
 Characteristics of Ground Presscake Material

Ground presscake material	Analysis ^a		Analysis on Ro-tap dry screens						
	Oil	Water	6	8	12	50	100	Through 100	Total
	%	%	<i>weight %</i>						
Flaxseed									
Lot No. 2-3	11.7	8.3	0	0.6	11.7	78.2	9.4	0.1	100.0
Peanut									
Lot No. 4	17.0	7.2	0	0.9	10.3	78.7	10.6	0.1	100.0
Sesame seed									
Lot No. 15	18.4	5.4							

^a Wet basis.

⁴ To avoid channeling it was necessary to carry out these experiments near the flooding point of the bed. About 10% shrinkage occurred when the bed was first wet with solvent. The original solids level was merely a convenient point for flow regulation without introducing an excessive hydraulic head over the solids.

available oil," *i.e.*, oil which could not be extracted with Skellysolve B.

Correlation of Results

When hold-up, *W*, was plotted *versus* time, curves of the type shown in Figure 2 were obtained. These provided a convenient means for estimating the practical duration of interstage and final drainage periods. For this example, the rapid drainage which occurred at first suggested that a relatively short period, say, 2 min., would separate the stages reasonably well. Again, final drainage of more than about 12 min. would be unnecessary since very little solution would drain thereafter. It was evident also that concentration had only a slight effect on drainage rate since the curves tended to be displaced upward with increasing concentration of the wash solution. Such plots were obtained for all materials at all bed depths studied. All of the curves exhibited the same characteristic form, with rapid drainage occurring within the first 2 or 3 min. and decreasing at a much slower rate to some asymptotic value.⁵

For convenience, all of the drainage graphs were converted to the form shown in Figure 3. Here *W* was plotted against concentration of the solution, *X*, with the drainage times as parameters. These plots were much more adaptable to design calculations since the material balances required greater accuracy in reading *W* in terms of *X* than was possible from the rate curves. Also, nearly all parameter lines were virtually straight on linear coordinate paper, which permitted extrapolation in either direction.

Because of the almost linear relationship between *W* and *X* it was therefore possible to express all of the hold-up data simply by defining the slopes and intercepts characteristic of each bed depth and drain-

⁵ The asymptote is of significance only insofar as it represents the so-called static hold-up, or quantity of solution required to saturate the bed before drainage can occur. For a more complete discussion, see Karnovsky (4) and Smith (8).

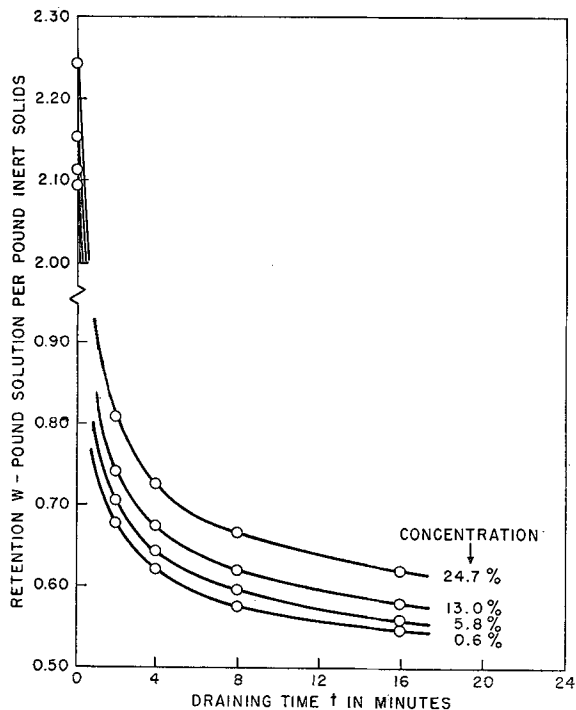


FIG. 2. Solution retention during draining at 60°C. (140°F.) (8-mil. soybean flakes, 12-in. bed depth).

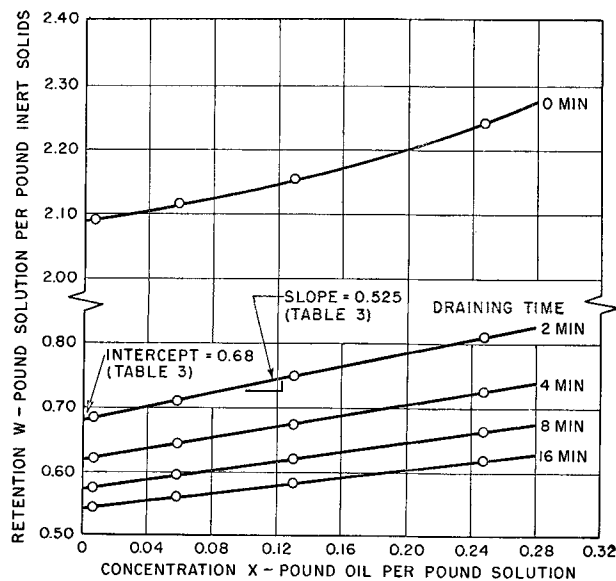


FIG. 3. Solution retention during draining at 60°C. (140°F.) (8-mil. soybean flakes, 12-in. bed depth).

ing time. The solution hold-up data have therefore been presented in this way in Tables III and IV. It is evident that a straight line can readily be constructed from each of these slope-intercept pairs relating *W* and *X* in the same way as illustrated in Figure 3.

Several observations are apparent from these tables. Hold-up at zero drainage time is, of course, a function of the porosity of a fully saturated bed and not

TABLE III
Cottonseed Flakes. Effect of Draining Time and Bed Depth upon Solution Hold-up Curves (*X versus W*)^a

Cottonseed flakes thickness	Bed depth	Draining time						
		0	2	4	8	16	32	
8 <i>mils</i>	3 <i>inches</i>	1.86 ^b	1.25	1.22	1.22	1.22		
		0.54	0.75	0.58	0.53	0.53		
	6	1.84	1.16	1.03	0.98	0.96		
		0.49	1.14	0.67	0.42	0.27		
	12	1.76	1.19	1.02	0.90	0.87		
		0.57	0.75	1.75	1.73	0.51		
	36	1.74	1.51	1.36	1.14	0.94	0.83	
		0.53	0.92	0.92	0.98	0.71	0.63	
	10	3	1.84	1.03	1.00	0.99	0.99	
			0.57	0.60	0.47	0.40	0.35	
		6	1.82	0.96	0.90	0.86	0.84	
			0.52	1.00	0.71	0.63	0.55	
12		1.74	1.00	0.93	0.87	0.83		
		0.56	1.01	0.63	0.52	0.44		
15	3	1.75	0.97	0.94	0.93	0.93		
		0.54	0.44	0.34	0.33	0.24		
	6	1.73	0.81	0.76	0.72	0.69		
		0.48	0.58	0.44	0.33	0.33		
	12	1.65	1.01	0.91	0.85	0.81		
		0.45	0.95	0.68	0.58	0.49		
20	3	1.61	0.84	0.81	0.80	0.79		
		0.47	0.62	0.48	0.37	0.36		
	6	1.58	0.77	0.73	0.71	0.69		
		0.44	0.38	0.34	0.27	0.25		
	12	1.51	0.78	0.71	0.66	0.64		
		0.41	0.54	0.47	0.44	0.31		

^a Each intercept-slope pair defines a linear plot of solution hold-up, *W* (pounds of solution/pound of solids), *versus* solution concentration, *X* (pounds of oil/pound of solution), for one oilseed, bed depth, and draining time. A typical *W versus X* plot is shown in Figure 3.

^b The number in italics (top line) is the intercept, the number in roman type (bottom line) is the slope.

TABLE IV
Effect of Draining Time and Bed Depth upon Solution
Hold-up Curves (*N* versus *W*)^a

Material	Bed depth	Draining time						
		0	2	4	8	16	32	
Ground flaxseed presscake	inches	<i>minutes</i>						
		3	1.23 ^b 0.38	0.48 0.53	0.46 0.38	0.46 0.38	0.46 0.38	
		6	1.11 0.35	0.42 0.78	0.39 0.55	0.38 0.38	0.37 0.30	
		12	1.01 0.33	0.44 0.60	0.40 0.50	0.37 0.38	0.34 0.28	
8-mil. soybean flakes	inches	3	2.14 0.57	0.82 0.50	0.79 0.44	0.77 0.38	0.75 0.31	
		6	2.12 0.58	0.70 0.47	0.66 0.42	0.63 0.35	0.61 0.30	
		12	2.09 0.57	0.68 0.53	0.62 0.43	0.57 0.37	0.54 0.31	
		36	1.90 0.54	0.73 0.79	0.63 0.62	0.56 0.51	0.51 0.41	0.48 0.35
Ground peanut presscake	inches	3	1.08 0.37	0.68 0.61	0.67 0.41	0.67 0.41	0.67 0.41	
		6	0.93 0.24	0.54 0.72	0.52 0.55	0.50 0.41	0.49 0.31	
		12	0.89 0.24	0.55 0.51	0.49 0.41	0.45 0.31	0.42 0.25	
		36	0.81 0.24	0.53 0.38	0.45 0.31	0.39 0.28	0.36 0.24	
Ground sesame presscake	inches	3	1.17 0.37	0.70 0.96	0.69 0.39	0.68 0.39	0.68 0.39	
		6	1.14 0.35	0.58 0.73	0.55 0.59	0.52 0.41	0.51 0.30	
		12	1.11 0.33	0.58 0.57	0.53 0.43	0.48 0.32	0.44 0.26	

^a Each intercept-slope pair defines a linear plot of solution hold-up, *W* (pounds of solution/pound of solids), versus solution concentration, *X* (pounds of oil/pound of solution), for one oilseed, bed depth, and draining time. A typical *W* versus *X* plot is shown in Figure 3.

^b The number in italics (top line) is the intercept, the number in roman type (bottom line) is the slope.

of flow. Thus intercepts and slopes decrease as the bed depth increases, at zero time, due to packing effects. Under draining conditions, slope decreases in regular fashion with length of draining period as might be expected. The effects of concentration are clearly shown and will depend upon density, viscosity, wetting properties, and surface tension of the miscella. Hold-up passes through a minimum at about 12-in. bed depth for any given draining time.

In the course of this work detailed extraction data were also obtained and correlated for each type of oilseed, as illustrated in Figure 4. These rates also profoundly affect extractor design, but the present discussion will be limited to the effect of solution retention or drainage. The term, *E*, is the ratio of extractable oil remaining in the material to that at zero time.

A nomographic method of correlating the various factors affecting hold-up is illustrated in Figure 5, which pertains to 15-mil. soybean flakes. This chart is accurate to within about 2% of the measured values.

Example Problem

The effect of solution retention upon extractor design is best illustrated by a sample calculation. In the following example it is assumed for simplicity of computation that sufficient soaking and extraction have been allowed so that the "free" solution is in equilibrium with the retained solution. The nomenclature is similar to that of Ruth (7).

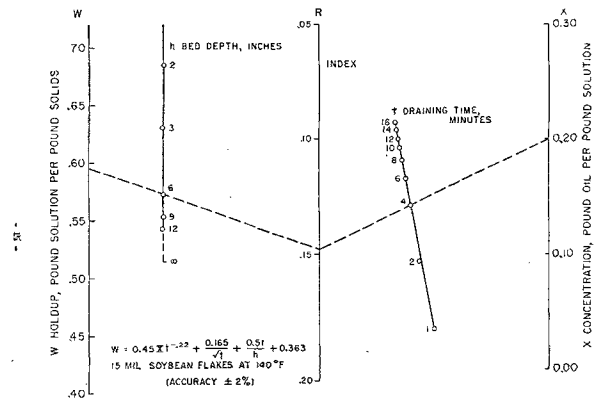


FIG. 4. Extraction rate of 8-mil. soybean flakes at 60°C. (140°F.) (8-mil. soybean flakes, 12-in. bed depth).

Soybean flakes having an average thickness of 8 mils., a moisture content of 9%, and an initial oil content of 20% are to be extracted with commercial hexane to a final oil content of 0.5%. A stagewise countercurrent system is to be used, employing alternate extraction of a bed 12 in. in depth followed by 4 min. of draining per stage. Sufficient soaking and extraction time are given so that equilibrium is reached at each stage. The amount of solvent and the number of stages required in order to produce a miscella containing 22.0% are to be determined. Also, the number of stages are to be calculated if 2, 1, or 0 min. of draining time are used in each stage instead of 4 min.

A material balance gives:

$$0.22 \left(F + \frac{0.20}{1.0 - 0.2 - 0.09} - W_F \right) = \frac{0.20}{0.71} - \frac{0.005 (0.80)}{0.995 (0.71)} \quad (1)$$

or

$$F - W_F = 0.976$$

where

F = pounds of solvent required per pound of inert solids

and

W_F = pounds of solution retained per pound of inert solids in final discharge flakes.

From Figure 3, $W_F = 0.62$ when draining time = 4 min. and when solution concentration $X_F = 0$. Then $F = 1.60$ pounds of solvent/pound of inert solids. For Stage 1, $W_1 = 0.72$ when $X_1 = 0.22$ (Figure 3). The concentration of S_1 in flakes = $0.22 (0.72) = 0.1584$ lb. of oil/pound of inert solids, and $X_2 = (0.1584 - 0.0057) / (1.60 - 0.62 + 0.72) = 0.090$ lb. of oil/pound of solution. By repeating the calculation for additional stages, the summary shown in Table V is obtained.

TABLE V
Summary of Example Calculation

Stage	Solution concentration, X_N	Oil retention, W_N	Unextracted oil, S_N
	lb. oil/ lb. soln.	lb. soln./ lb. solids	lb. oil/ lb. solids
Final.....	0.22	0.72	0.282
Discharge.....	0	0.62	0.0057
1.....	0.22	0.72	0.1584
2.....	0.090	0.66	0.0592
3.....	0.032	0.63	0.0201
4.....	0.0095	0.62	0.0056

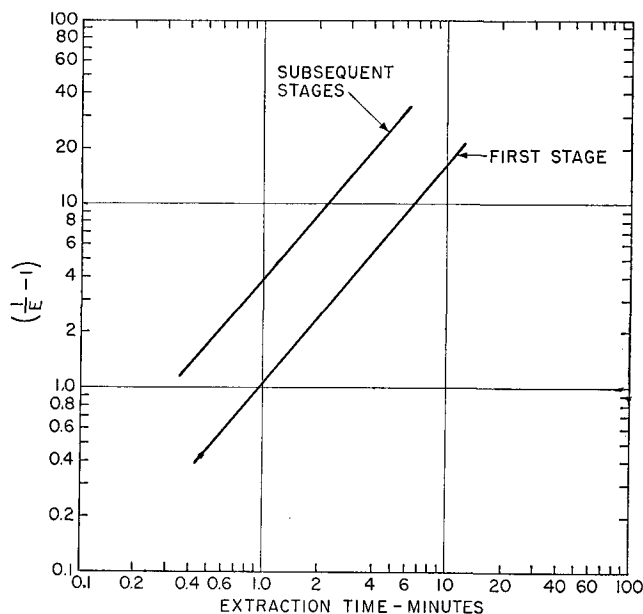


Fig. 5. Effect of design parameters upon solution hold-up of 15-mil. soybean flakes at 60°C. (140°F.).

Thus 4.0 equilibrium stages are required if a draining time of 4 min. is used between stages. For 2 min. of drainage approximately 4.5 stages would be re-

quired; for 1 min. of drainage approximately 5 stages; and for 0-min. drainage, approximately 10 stages.

Acknowledgment

This work was part of a project conducted by the Armour Research Foundation, Chicago, Ill., for Wurstter and Sanger Inc., Chicago, Ill. Those who participated in the experimental work, correlation of data, and analytical work included: L. H. D. Fraser, R. A. Peterson, Miss E. Grimm, S. Hu, R. Snow, and A. Weiner.

Summary

Hold-up of solution by the solids undergoing extraction has been shown to be an important factor in the design of percolation extraction systems. By means of a laboratory technique, hold-up was measured for a wide variety of prepared oilseeds. Empirical correlations of the data were obtained, and their significance was illustrated in a sample design calculation.

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The Metabolism of Triglycerides Containing *cis* and *trans* Octadecenoic Fatty Acids

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THE GEOMETRIC STRUCTURE of the unsaturated fatty acids in edible oils can be shifted from a *cis* to a *trans* configuration during hydrogenation (1). The amount of high-melting *trans* or "iso-oleic" acids formed is in direct proportion to the selectivity of the reaction, and, as recently pointed out (2), greater geometrical and positional isomerization than heretofore suspected occurs under conditions of high selectivity. It remains to be determined however whether the *trans* isomers are metabolized as efficiently as the *cis*. Kohl (3) reported in 1938 that it took over 30 days to clear the elaidic-acid reserves from the rat body after the acid had been fed for three days. Sinclair (4) made use of elaidic acid as a "label" to demonstrate the incorporation of fatty acids into the phospholipid fractions of the intestinal mucosa.

Both of these studies were made before the more accurate determination of *trans* acids by infrared analysis had been perfected. Phatak and Patwardhan (5) fed rats iso-oleic acids from hydrogenated peanut oil and reported that 94% was metabolized and 6% was found in carcass lipides. That elaidic acid is selectively deposited and that phospholipides contain less elaidic acid than neutral fat were reported by Collet and Favarger (6). Paul and McCay (7) have

shown that guinea pigs do not utilize elaidic acid as efficiently as rats. Furthermore Aaes-Jorgensen and Dam (8) found a decrease in the growth rate of rats fed hydrogenated peanut oil. More recently Melnick and Deuel (9) demonstrated that iso-oleic acids are not antimetabolites for oleic acid in microorganisms.

In all studies reported to date the *trans* acids were mixtures obtained from hydrogenated-oil sources or were pure elaidic acid. In the present study known amounts of synthetic triglycerides containing unsaturated fatty acids with the *trans* bond in the 9 or 8 position were fed to rats, and the results were compared with those from hydrogenated oil. The total amount of fat excreted was determined, and various tissues were analyzed for the total amount of *trans* acids deposited.

Experimental

Weanling female rats were fed a synthetic basal diet composed of 31% casein, 63.8% cerelese, 5% Wesson salts (10), and 0.4% of a water-soluble vitamin mix of the following composition: choline 93.5%, thiamine 1.24%, riboflavin 1.24%, pyridoxine 1.24%, Ca pantothenate 2.48%, and folic acid 0.3%. In addition, each animal received approximately 100 mg. per day of linoleic acid (as trilinolein) and approximately twice the minimum daily requirements

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