Those members, not on the Bleaching Methods Committee who collaborated in this work, were the following:

R. R. King, Interstate Cotton Oil Refining Co., Sherman, Texas
P. A. Williams, South Texas Cotton Oil Co., Houston, Texas
J. J. Ganucheau, Southern Cotton Oil Co., Gretna, La.
W. T. Maxwell, Southern Cotton Oil Co., Savannah, Ga.
J. H. Sanders, The Procter and Gamble Company, Ivorydale, Ohio

There was no dissenting vote out of the eleven who voted to recommend that the bleach test procedure for cottonseed oil be unified with that now current for soybean oil. If adopted, this change would of course be tentative for at least a year.

Evaluating Bleaching Clay Activity by an Absolute Method. It was proposed that the committee study the possibility of developing some method of determining bleaching activity of fuller's earth independent of (a) a comparison of the activity with that of some other earth and (b) the necessity to use vegetable oils, which are notoriously unstable with respect to their bleaching response. There was some discussion of various approaches to this problem in correspondence but the consensus of opinion in the committee was that it represented a type of investigation which was beyond the scope of committee work, that it constituted really a research problem that might well be the subject of a fellowship investigation. However, two members objected that any method of evaluation which depended upon the bleaching power manifest in use on some "synthetic" oil which could be reproducibly constituted, would not be valid in evaluating bleaching earths as to their activity on natural vegetable and animal oils, which among themselves offer considerable diversity in their response to various bleaching earths.

Recommendations.

1. That in Cc 8a-28 (A-6), where now occurs "E. & D. 192," there shall be substituted the following: "E. & D. 192, R-A 871, Whatman, No. 12, S. & S. 596."
2. That the Society endorse the stock of activated clay which has been standardized and packed in sealed cans especially for use in grading the bleachability of soybean oil; and it is further recommended that this lot be designated Official A.O.C.S. Activated Clay, and a label to that effect be applied to the containers, assuming that suitable arrangement to permit this can be made with the present sponsors of that stock of clay.
3. Combine Methods Cc 8a-28 and Cc 8b-44, particularly with respect to (a) under Procedure in the latter, which will constitute the wording of the unified method. The statement of scope will therefore be broadened to read, "Applicable to refined cottonseed and soybean oils." The line at B. (b), Dc 8b-44, *For Green Oils—High Chlorophyll Content,* will have added to it, "Intended only to apply to soybean oil." This recommendation proposes that in the bleach test procedure for cottonseed oil the 6% official earth be added to the cold oil before heating to 120° C. with agitation in not over 5 minutes.[1]

The committee believes that it has completed the tasks which were assigned to it or which it had assumed, insofar as they can be considered to come within the scope of committee work, and therefore asks that it be discharged.

G. WORTHEN AGEE	A. D. RICH
N. F. KRUSE	HENRY ODEEN
R. A. MARMOR	EGBERT FREYER, chairman
R. J. HOULE	

[1] The recommendations embodied in this paragraph were subsequently approved, but on incorporating the indicated changes into the methods, (Second Edition), editorial requirements made it necessary to depart from the wording suggested here.

The Solvent Extraction of Oil From Acorns*

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Pratt Institute, Brooklyn, N. Y.

THE possibility of using acorns for food has often been discussed in the literature (18,21,27), but except in times of scarcity of other foods, acorns have been utilized in any quantity only by wild animals or semi-domestic pigs (22). There are some references (14,18) to the possible uses of acorn oil, but there is no record of any large scale work with this material. This investigation was undertaken to handle a quantity of acorns, 847 pounds, (a) to disclose what particular problems there are in processing this material for acorn oil, (b) to demonstrate a practical method of obtaining acorn oil, and (c) to relate solvent- extraction theory to the experimental extraction of this oil.

There are three general methods for obtaining oil from oleaginous seeds, expression, expulsion, and solvent-extraction. Solvent-extraction was selected as the most promising method for obtaining oil from acorns since it can be applied to any oil-bearing material and is particularly suited to the removal of oil from materials of low oil content. The oil content of acorns is reported to vary from 2.5% (25) to 16.0% (23).

Acorns. One of the problems of this investigation was to determine what type of material could be expected on offering a price for acorns. Potential suppliers in Arkansas, Virginia, New York State, Florida, and South Carolina expressed a willingness to collect acorns for three cents a pound. There were 361 pounds of acorns received from Gainesville, Florida, and 486 pounds from Anderson, South Carolina. It is our opinion, based on our personal observation and our attempts to get acorns, that they are seldom as abundant as popularly supposed and that harvesting for commercial utilization would be difficult.

* This work was done in the Department of Chemical Engineering, Virginia Polytechnic Institute, Blacksburg, Va.

The nuts which were supplied were of poor quality. The best lot obtained, the scrub oak acorns from Florida, *Quercus catesbaei,* consisted of 75% sound fruit, the rest was either wormy or sprouted. The South Carolina acorns were only 41% sound nuts. The quantity of oil which may be produced from sprouted or wormy nuts depends on the extent of growth or of worm damage. Most of the wormy nuts which were examined contained no meat at all. Fully sprouted nuts contain less than 1% of oil (16).

Before an acorn oil mill could be established on a commercial basis an extensive education campaign would have to be conducted in order to acquaint prospective acorn harvesters with the type of material for oil extraction. It is necessary that the nuts be gathered immediately after falling and stored in dry cribs.

Preparation of Acorns for Extraction. Most of the acorns were prepared for extraction by removing the shells in a winnower and fanner ordinarily used to remove the shells from cocoa beans. This machine combines a roll for cracking and crushing the nuts with a revolving screen for separation of different sized particles and an air blast for blowing the light hull fragments away from the heavier nut meats. The acorns were shelled at a rate of about 200 pounds per hour. Smaller quantities of acorns were shelled by crushing in a Sturtevant No. TR-32 two-roller mill, followed by separation of shells and meats in a Raymond No. 41344 laboratory mechanical separator with all the whizzers removed. The operations carried out in preparing the acorns for extraction are given in Fig. 1.

There are a number of methods of preparing seeds for oil extraction. The most satisfactory of these, according to the experience of soybean oil producers is to prepare a thin flake, the thinner the better. These flakes are readily penetrated by solvent but hold their shape after removal of the oil so that drainage of miscella from extracted solids is rapid. Others (3,4) have had good success in extracting crushed expeller cake, sized to be retained by an eighth-inch mesh screen and passed by a half-inch mesh screen. Expelling is a hot-pressing operation which produces a porous cake, probably due to the formation of steam within the material passing through the expeller. Solvent penetrates the porous expeller cake fragments easily and these fragments, like the soybean flakes, do not break up on removal of the oil. Grinding produces a large proportion of fine flour, evident after oil removal, which cannot be handled by any of the commercial extractors described in the literature as being in current use (4,8,10,11,12).

Acorn expeller cake was prepared by using an Anderson Duo Expeller on the acorn meats from the winnower and farmer. The expeller cake was crushed and the material retained by an eighth-inch screen and passed by a quarter-inch screen was extracted. Since at least 300 pounds of acorn meat were required for one expeller run most of the acorns were used unsorted for this experiment. Carefully selected acorns contain up to 40.0% of *n*-butanol extractable material whereas the unsorted material fed to the expeller contained only 13.3% of such material. The results of the expeller run show that acorn meats can be handled by an Anderson Duo Expeller. A good many runs would be required to establish optimum expelling conditions. During the first part of the 65-minute run the acorn meat was fed as it was. The first cake was crumbly and not firm. Water was added to the meat during the second part of the run and a much stronger and harder expeller cake was formed.

Methods of Extraction and Choice of Solvent. Two extractors were available for processing acorns on a pilot plant scale. The first of these consisted of a mixing tank (one-gallon porcelain ball mill jar), for solvent and meal, followed by a Tolhurst No. T-7893 centrifuge to separate extracted solids and miscella. Extraction followed Elgin's (5) pseudo-countercurrent extraction scheme. This scheme is presented in Fig. 2. Hunter (13) shows that such a scheme gives a deviation of 3 to 10%, depending on the stage, from a truly countercurrent extraction when five cycles are used with four stages. He demonstrates that this deviation becomes less than 2% for all stages with ten cycles. Three and four cycles were employed with two and three stages in these centrifuge extractions. A considerable deviation from a truly countercurrent process is therefore to be expected.

The second extractor was of the continuous chain type. It consisted of a 14-foot length of two-inch standard galvanized steel pipe bent so that a short section was vertical with the horizontal plane and a long inclined section beyond that bend made an angle of about 25° with this plane. A 28¾-foot continuous chain made up of No. 45 malleable detachable links with No. 45 C-1 flights every fourth link ran in at the top of the vertical section. up the inclined section and out at the top of this latter section. The extraction section, that part of the incline from the bottom of the bend to the solvent inlet, was 6½ feet long. The solid material being extracted was carried up the incline by the chain against a counterflow of solvent. There was a 3½-foot drainage section above the solvent inlet. The chain carried the solids through this drainage section before running out of the pipe and dropping the drained solids onto a discharge apron. The miscella flowed out of the vertical section at the solvent inlet level.

TABLE I.

Experimental Conditions for Centrifuge Extractions.

	Experiment		
	1	2	3
No. of stages	3	2	2
No. of cycles	3	3	3
Temp., °C.	25	25	25
Extraction time, hrs.	1	1	0.5
Feed			
Weight, lb.	2	2	4
Acorn oil, %	40.0	36.0	38.6
Solvent, lb.	4	4	4
Average overflow, lb.	3.81	3.28	4.07

Neither of these extractors was solvent-tight so that no volatile nor toxic solvent could be used with them. The usual commercial oil solvents are low-boiling hydrocarbons (15,19,20) and their chlorinated derivatives (4,7,26). These solvents are both volatile and dangerous, the hydrocarbons because of their flammability, and the chlorinated hydrocarbons because of their toxicity. *n*-Butanol was selected as solvent because it is not very volatile, it is not dangerous to use since its flash point is 35° C., and preliminary experiments indicated that it was a satisfactory solvent for acorn oil.

Centrifuge Extractions. The feed in the centrifuge extractions consisted of acorn meal of which 83.2% passed through a standard 35-mesh screen and 2%

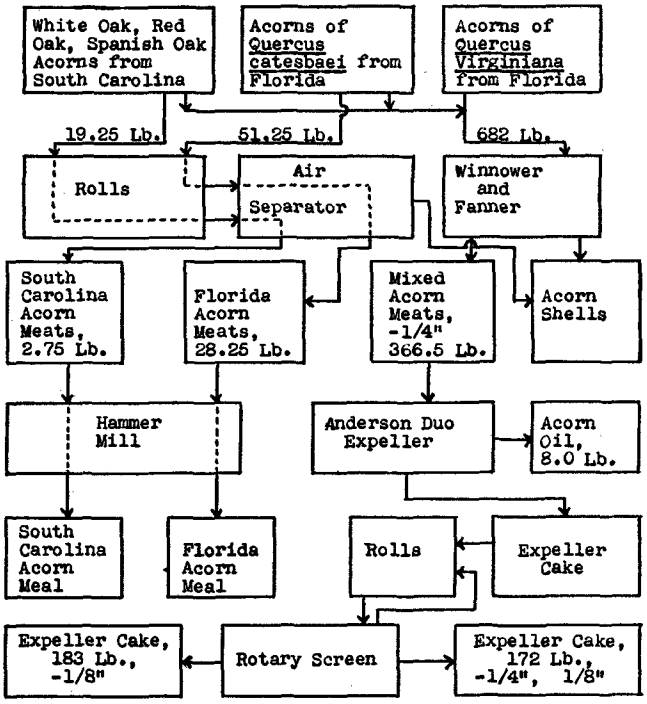


FIG. 1. Preparation of acorns for extraction.

was retained by an 8-mesh screen. The extractions were carried out under the conditions given in Table I to give the results of Table II.

Continuous Extractions. The continuous extractor was operated according to the conditions given in Table III to give the results of Table IV. It was difficult to obtain consistent results with this extractor. This was due to the tendency for solid material to hold up in the pipe and to progress irregularly through the system. This difficulty was particularly noticeable with acorn meal, which packed against the sides of the pipe so that the chain moved through a square duct composed of solvent-wet meal. This was an unstable condition. It was found possible to reach a rather unsteady equilibrium by cleaning the system well before a run, filling it with solvent and establishing the solvent feed rate, and then feeding in the solids at the selected rate for the run for a period of about an hour before taking samples and making readings. Under these conditions the fairly consistent results of Table IV were obtained.

Solvent and Oil Recovery. The miscella was distilled under a vacuum of 25 inches of mercury in a Stokes No. 84260 vacuum pan. The results of solvent and oil recovery are given in Fig. 4. The still was equipped so that steam could be blown through the charge and traces of solvent were removed from the acorn oil by steaming out under vacuum. After this operation a two-inch gate valve at the bottom of the still was opened and the residue was discharged. This residue divided into two layers, an upper acorn oil layer, and a lower water layer. The acorn oil was freed of impurities such as iron tannate and suspended solids by washing with water until the wash-water was uncolored.

TABLE II.

Results of Centrifuge Extractions.

	Experiment		
	1	2	3
Miscella, lb	3.44	3.5	3.94
Oil in miscella, %	18.63	18.4	28.6
Oil in miscella, lb	0.64	0.65	1.13
Oil in feed, lb	0.80	0.72	1.54
Oil experimentally extracted, %	80.0	89.5	73.0
Oil theoretically extracted, %	95.0	93.7	92.2

TABLE IV.

Results of the Continuous Extractions.

Experiment	Oil in feed, lb.	Oil extracted, experimental		Oil extracted, theoretical, %	Ratio of solvent to feed
		lb.	%		
1	0.46	0.19	41.3	52.0	2.0
2	0.46	0.21	45.6	74.5	2.0
3	0.23	0.09	39.2	57.8	2.0
4	0.77	0.13	16.9	31.5	2.0
5	2.70	1.13	41.8	44.5	2.3
6	1.54	0.55	35.8	52.2	2.6
7	1.54	0.32	20.8	32.5	2.2
8	1.54	0.53	22.8	45.4	1.3
9	0.16	0.10	62.3	70.3	2.7
10	0.82	0.55	67.0	72.3	1.5
11	0.71	0.51	71.8	87.0	1.0

Approximately three gallons of crude acorn oil were produced by these pilot-plant operations, about half of this oil resulted from the extractions reported in this paper and the rest came from acorn meal and expeller cake processed in preliminary experiments.

A sample of crude acorn oil from *Quercus catesbaei* had the following characteristics: d at 25° C., 0.907; n at 25° C., 1.4677; acid number 5.6; saponification number 190; and iodine number (Wijs) 106. It was refined to give an oil of d at 25° C., 0.903; n at 25° C., 1.4684; acid number 2.6; saponification number 188; and iodine number (Wijs) 107.

In addition to the oil prepared by the pilot-plant extractions there were the eight pounds of oil collected from the expelling operation with the Anderson Duo Expeller. This expelled oil was filtered to free it from a small amount of solid material to give an oil of d at 25° C. of 0.907, n at 25° C. of 1.4669, acid number of 29.5, saponification number 192, and iodine number (Wijs) 105.

TABLE III.

Experimental Conditions for the Continuous Extractions.

Experiment	Series	Feed	Length of run, min.	Solvent fed, lb.	Solid fed, lb.	Raffinate removed, lb.	Miscella out, lb.
1	I	Crushed cocoa expeller cake, 11.5% cocoa butter	120	8.0	4.0	8.4	4.4
2			120	8.0	4.0	6.3	6.3
3			30	4.0	2.0	2.7	2.4
4	II	Acorn meal, 38.5% acorn oil	60	4.0	2.0	3.6	1.5
5			210	16.2	7.0	14.7	8.4
6			120	10.5	4.0	6.7	6.3
7			120	8.6	4.0	7.6	3.3
8			120	5.3	4.0	7.7	3.1
9	III	Crushed acorn expeller cake, 17.8% acorn oil	20	2.4	0.9	1.3	1.8
10			120	7.2	4.6	6.4	5.8
11			90	4.0	4.0	3.4	4.1

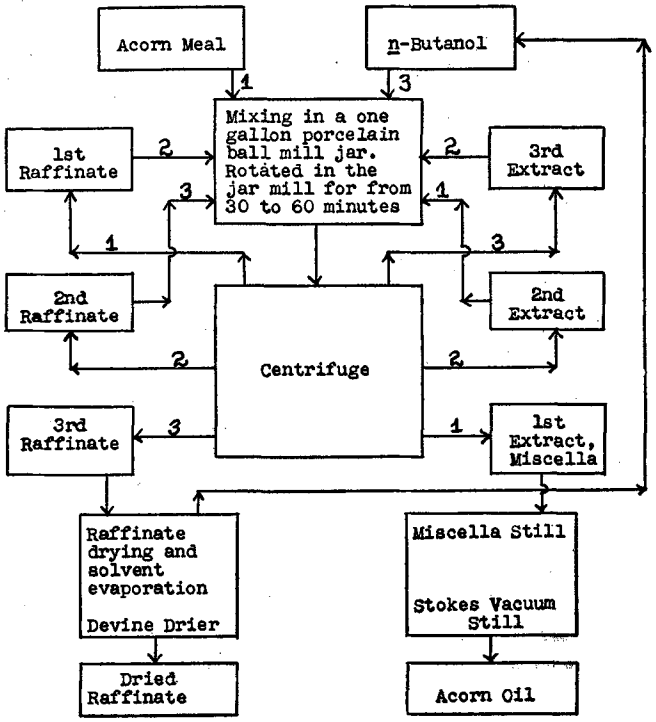


FIG. 2. Flow diagram of a three-stage centrifuge extraction.

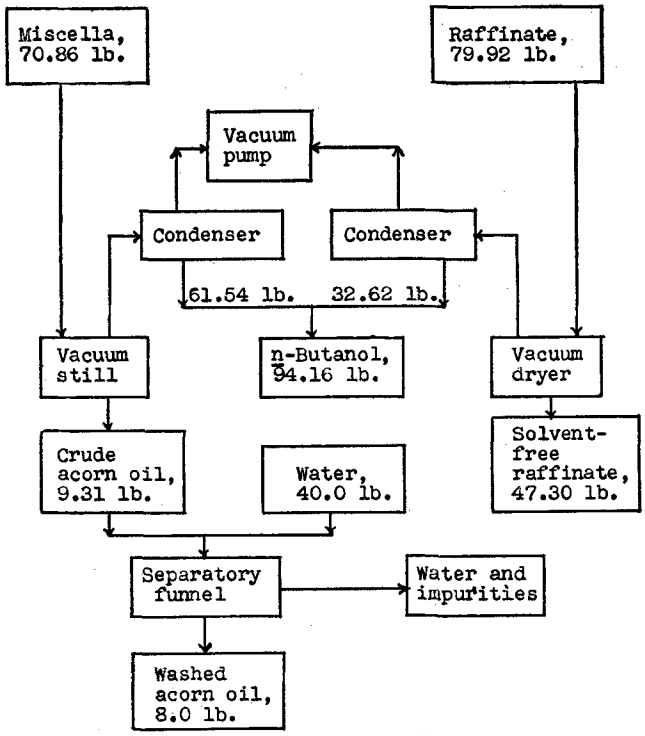


FIG. 4. Solvent and oil recovery.

Relation of Solvent Extraction Theory to the Experimental Extraction of Acorn Oil. Solvent extraction theory has been developed and discussed by Evans (7), Hawley (9), Baker (1,2), Ravenscroft (1,24), Elgin (6), and Kinney (17). Theoretical calculations, following this theory, were made for the pilot-plant extractions of acorn oil and cocoa butter with the results given in Tables II and IV. The calculations for the centrifuge extractions were for extractions of the experimental number of stages carried out under the experimental conditions. The number of stages in the continuous extractor was unknown until some of the experiments had been completed. It was found that there was slightly less than one theoretical stage in this extractor. The calculations for the continuous extractor are based on its containing one theoretical stage.

Increasing the solvent-solid ratio in the continuous extractions increased the extraction in the case of acorn meal—as would be expected. Contrary to expectation increasing this ratio led to decreased extraction in the case of the acorn expeller cake extractions. The effect of increasing solvent-solid ratios is plotted in Fig. 5. Solvent-solid ratio is not a sufficient cri-

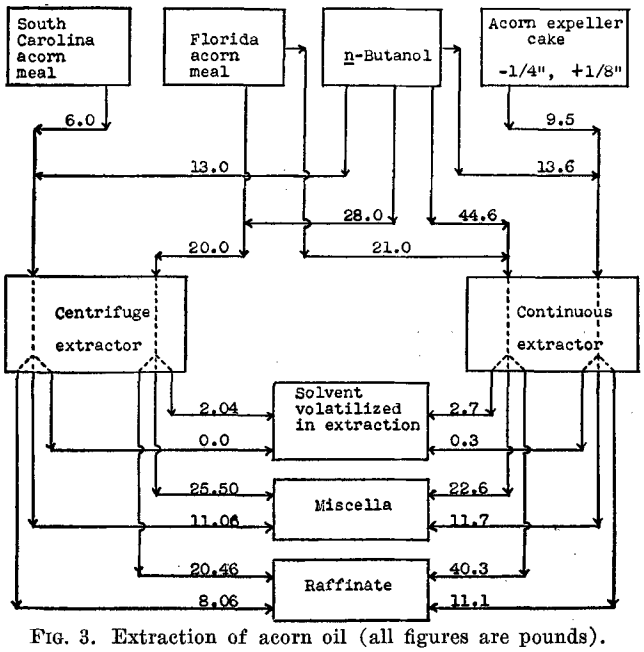
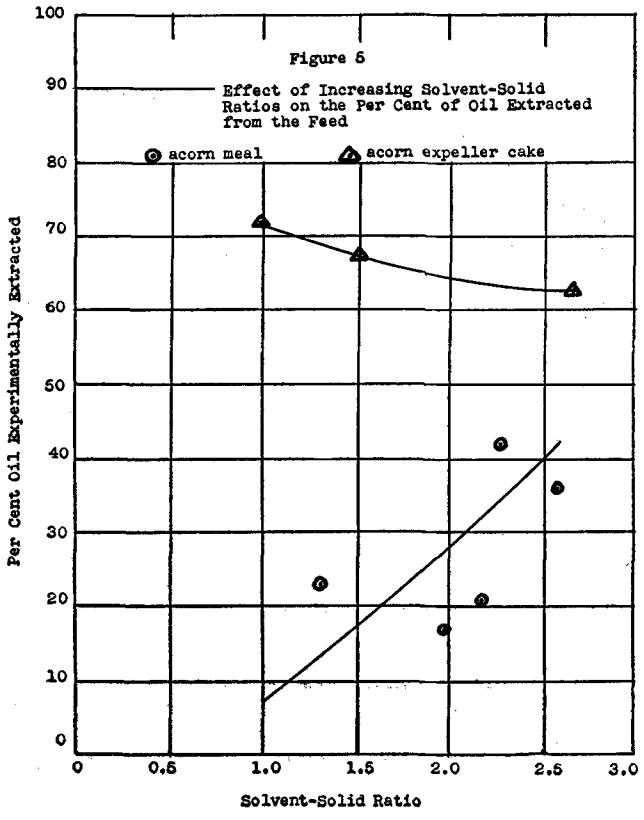
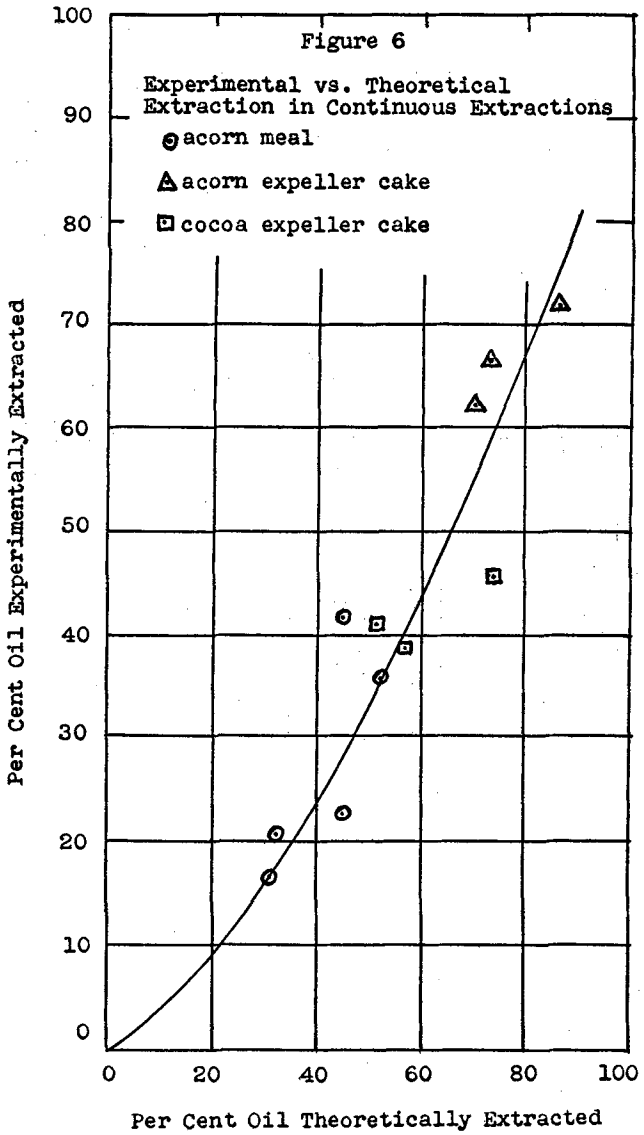


FIG. 3. Extraction of acorn oil (all figures are pounds).



terion for the prediction of the results of an oil extraction. Solvent-extraction theory, however, can be used successfully to predict the results of such extractions. Experimental vs. theoretical extraction is plotted in Fig. 6 and this figure shows that this theory is capable of predicting the results of such extractions as those of acorn oil from acorn meal and acorn expeller cake as well as the extraction of cocoa butter from cocoa bean expeller cake. A comparison of the results for extraction of cocoa butter and acorn oil shows that, if the same extractor is operated under similar conditions on these materials, there are the same number of theoretical stages in that extractor whether the feed is acorn meal, acorn expeller cake, or cocoa bean expeller cake.



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Report of Seed and Meal Analysis Committee

Methods of Analysis of Soyflours

THE Seed and Meal Analysis Committee has been directed by the Governing Board of the Society to investigate and recommend methods for the determination of moisture, oil, nitrogen, ash, crude fiber, water absorption, phosphatides and particle size in soyflours. The initial step taken was to conduct a survey of experiences in the analysis of soyflours to direct the action of the Committee. The work, considerations, and recommendations regarding the first five of these methods are given in this report.

Moisture and Volatile Matter

It is recognized that oven moisture values do not actually represent moisture but a combination of moisture and volatile matter influenced by decomposition and oxidation that may occur under conditions of the test. For this reason the method is empirical and must be specific and followed rigidly. Majority of practices and experiences, revealed by the survey, indicated the satisfaction of the use of the forced draft oven (A.O.C.S. Specification H 1-39) and an oven temperature of 130° C. Two combinations of sample weight and time of heating have been used, namely, 2 grams for one hour and 5 grams for 2 hours. This point was studied collaboratively by four laboratories using samples of each of full fat, low fat, and de-fatted soyflours and heating at 130° C. in the forced draft oven. Observations made from the results reported are as follows:

1. For individual periods of heating, varying from 30 minutes to 2 hours, there was but little difference in the results obtained on 2- and on 5-gram samples.
2. A low rate of loss which can be tolerated in approaching a constant weight appears to occur at or soon after one hour of heating.