Whole seeds containing 7% and 12% moisture were stored for 4 weeks and seeds containing 17% moisture were stored for 2 weeks, during which periods the oils developed free fatty acids equivalent to acid values of 2.0 or less. Under none of the conditions used did the acid values of the oils exceed 8.0 after storage for 13 weeks.

Whole kernels developed even less free fatty acids than seeds stored under similar conditions. Kernels containing 4% and 6% moisture were stored for 12 weeks during which period the acid value of the oil never exceeded 1.5. Even in kernels containing 12% moisture the acid value of the oil did not exceed 6.0 at the end of 12 weeks.

Chopped kernels with moisture contents of 5% and 7% could be stored for 12 days without developing an acid value in the oil of more than 8.0. However chopped kernels with a moisture content of 12% developed an acid value in the oil in excess of 8.0 in less than a week.

Whole seeds with as much as 15% moisture could probably be stored for several weeks without developing an objectionable amount of free fatty acids. Since commercial hulled "nuts" practically always contain some broken kernels, to avoid development of free fatty acids in storage they should be dried to 10% or less moisture before storage.

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Pilot-Plant Application of Filtration-Extraction to Rice Bran[1]

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APPLICATION to rice bran of a simplified solvent process, filtration-extraction, which was developed originally for cottonseed (1), tends to overcome several of the technological difficulties that have hampered the solvent extraction of rice bran. Many of these difficulties have been investigated at this laboratory and the results reported, including work on the storage of rough rice bran (6) and on the composition and processing of rice bran (5, 8) and rice bran oil (2, 4, 7, 9, 11, 13).

The previous work indicated that most of the functional processing difficulties arise from the large amount of fine material in the bran. For example, in the percolation type of extractors, fines result in channeling; a high concentration of fines in the miscella presents clarification problems; and fines in the marc (solvent-damp extracted bran) tend to clog the vapor system during desolventization.

The general process as applied to rice bran consists of cooking the bran, cooling, slurrying with miscella (oil-hexane mixture), filtering, counter-currently washing the cake three times on a rotary, horizontal vacuum filter (1), and conventional recovery of oil and meal products. Cooking is responsible for practically eliminating the fines problem by agglomeration to form larger particles and for altering the physical characteristics of the particles so that the resistance to compression is appreciably increased, which, in turn, increases the filtration rate and minimizes the possibility of channeling. Cooking also inhibits any further rise of free fatty acids of the oil in the bran by substantially inactivating the lipolytic enzyme action (6).

The purpose of this paper is to report data obtained from pilot-plant-scale filtration-extraction runs, conducted following tests using small-scale batch equipment (12) which had indicated process advantages for rice bran similar to those found for cottonseed; such as rapid filtration rates, low fines content in the miscella, good oil extractability, high capacity, low solvent-to-meal ratio, low solvent content in the marc, and a good quality of oil and meal products.

Process and Equipment

Figure 1 is a flow diagram of the filtration-extraction process. Figures 2, 3, and 4 show the equipment —cooker and filter with auxiliary equipment—used in the two major operations of the process.

The cooker is a standard 5-high unit, such as is used in processing cottonseed. It consists of five steam-jacketed kettles, placed one above the other. The four lower kettles are joined by means of valved connections to a common vent stack, which is equipped with an exhaust blower. Moisture and temperature of the bran in the top kettle are adjusted and controlled by the addition of steam and water, and by steam jacket pressure. As the bran drops from the top to the four lower kettles, moisture removal, or drying, is controlled by the vent system and jacket temperatures.

The bran discharged from the cooker is shaken through a ¼-inch mesh screen to break the large lumps before spreading on trays to cool.

The cooked, cooled bran is charged continuously to one end of a paddle-type of mixing conveyor, 1 foot in diameter by 12 feet long. To the bran is added the second of the four filtrates (see flow diagram) to form a slurry which has a retention time of 15 to 20 minutes in the mixing conveyor. During this time the cooked bran and miscella are gently but thoroughly mixed and conveyed to the discharge end of the conveyor.

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A 3-in. solid-flight screw conveyor deposits the slurry onto the pan of the filter (1), which is a pilot-plant model of a continuous, horizontal, rotary, vacuum type commercially manufactured. Total filtering area of the pan is about 3.5 sq. ft. although the effective filtering area is 3.0 sq. ft. The filter medium is a 24 x 110 Dutch-weave screen.

Bulk of the liquid portion of the slurry is removed as the first filtrate—the product miscella—containing 12 to 17% oil. The first filtrate is pumped through a small pressure filter where the small amount of fines is removed. The filtered miscella is concentrated and stripped in conventional pilot-plant equipment. The filter cake, still containing some of the original miscella, received three successive countercurrent washes while the filter continues to rotate in its horizontal plane. The three washes are the third filtrate (2-4% oil), the fourth filtrate (0.5-1.5% oil), and oil-free commercial hexane. Drainage by vacuum filtration after each wash is so efficient that the cake contains only 30 to 40% solvent.

After the final washing and draining the cake is removed from the filter pan by means of a triple-flight scroll, 6 in. in diameter, rotating at 60 r.p.m. The scroll discharges the solvent-damp, extracted bran to a conventional-type, 4-stage, steam-jacketed dryer (4) for desolventization.

The filter is designed to permit continuous blowback at a point immediately underneath the filter section onto which the slurry is deposited. The blowback helps to keep the filter medium clean by remixing, with the oncoming slurry, the layer of meal, 1/16 to 1/8 in. thick, which passes underneath the discharge scroll. Nitrogen was used as the blowback gas in the pilot plant runs. In a commercial installation it would be more practical to use hexane saturated air from the filter hood. However gas from a noncombustible gas generator, superheated hexane vapor, or possibly liquid miscellas might be used.

A vacuum pump using hexane as its liquid seal is used to maintain vacuum at the filter through the four filtrate headers.

Operating Conditions and Results

Four pilot-plant scale runs were made: three with rice bran from standard milled rice, and one with bran from "Converted" rice (6).[3]

Cooking. In all four runs 230 lb. of bran per hour was fed continuously to the top kettle of the cooker. The total cooking time was 1 hour, or 12 minutes in each of the 5 kettles. Because of continuous feeding the temperature of the incoming bran was raised to about 203° F. and the moisture content to 25.9% almost instantaneously. Moisture content and temperature of the bran in each kettle for a typical run were as follows:

Kettle No.	Temperature	Moisture content
	°F.	%
1	203	25.9
2	212	24.5
3	212	21.7
4	210	20.5
5	219	16.0

The bran, which was discharged from the cooker at 219° F., cooled rapidly on the trays, after screening, to about 130° F., the highest temperaure at which the bran should be fed to the slurrying conveyor.

[3] The mention of trade products does not imply their endorsement by the Department of Agriculture over similar products not mentioned.

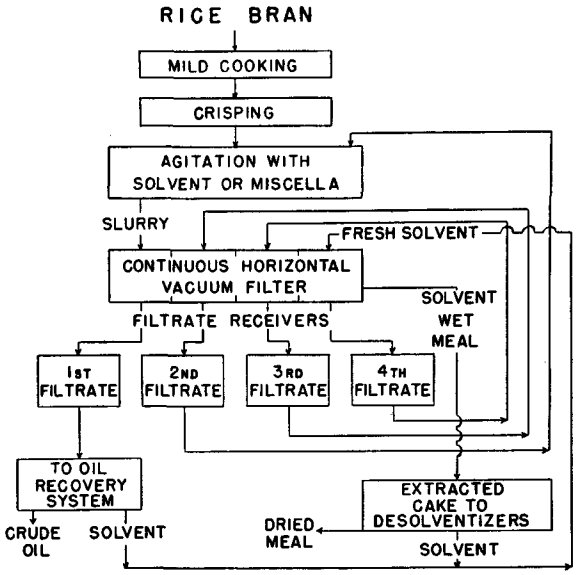

FIG. 1. Simplified flow diagram of the filtration-extraction process for rice bran.

Bulk densities of the bran, in lb. per cu. ft. were:

	Bran from standard milled rice		Bran from "Converted" rice	
	Uncooked	Cooked	Uncooked	Cooked
Unpacked	23.5	28.5	21.5	30.0
Packed	30.5	35.5	28.5	38.0

"Packed bran is bran which has been shaken down within a cubic-foot container. These figures are useful in determining the size of some of the processing equipment required for any given size plant.

Wet-screen analyses were made of uncooked and cooked bran from standard milled rice and from "Converted" rice. Results in Table I show seven

TABLE I

Wet Screen Analysis[a] of Rice Bran Before and After Cooking

Screen size, mesh	Screen openings, inches	Weight % of material retained on screen			
		Bran from standard milled rice		Bran from "Converted" rice	
		Uncooked bran	Cooked bran	Uncooked bran	Cooked bran
5	0.1570	0.0	0.8	0.0	0.5
8	0.0937	0.1	0.8	0.0	1.2
14	0.0555	0.2	1.4	0.0	1.2
20	0.0331	10.7	15.2	2.0	9.9
40	0.0165	25.5	39.3	24.5	38.2
60	0.0098	27.5	28.9	20.3	29.7
80	0.0070	10.9	6.7	10.0	7.3
120	0.0049	4.3	3.2	6.1	4.1
170	0.0035	3.8	1.1	3.9	2.0
200	0.0029	1.1	0.3	1.0	0.8
300	0.0018	3.6	0.8	3.8	1.3
Total		87.7	98.5	71.6	96.2
Through 300-mesh		12.3	1.5	28.4	3.8
Total		100.0	100.0	100.0	100.0

[a] Wet screening method developed at SRRL (3).

to eight times more through-300-mesh fines in uncooked bran from both standard and "Converted" rice than in the corresponding cooked bran; and a general particle size increase for the cooked as compared to the uncooked bran.

The cooking operation, besides reducing the amount of through-300-mesh fines and increasing the particle size, aids the release of oil from the bran and imparts a "crisping" or "hardening" effect to the particles.

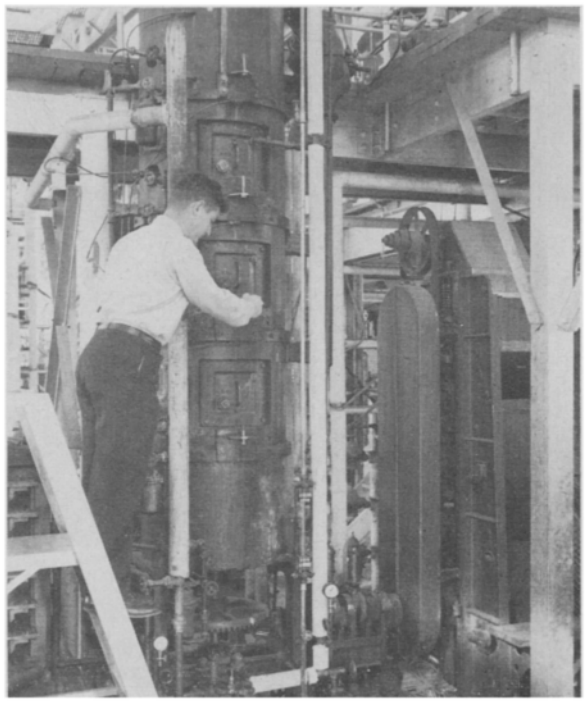

FIG. 2. Pilot-plant 5-high kettle-type of cooker.

These results are primarily responsible for shorter filtration cycle time, better extractability, and reduction of the fines problem. For example, in prepilot-plant-scale tests, the uncooked bran required filtration cycle times of more than 30 minutes compared to 30 to 60 seconds for the cooked material. Also the residual lipides in the extracted uncooked bran were about twice as high as that in the extracted cooked bran.

Extraction. Separation of the oil from the bran takes place during the slurrying and filtration steps. The greater part of the oil goes into solution as concentrated miscella during the 15 to 20 minutes of retention time in the mixing conveyor. The bulk of this miscella is separated and removed from the system during the initial slurry filtration on the vacuum filter. The remainder of the miscella and some additional oil are extracted by countercurrent washings during the less than two minutes of residence time on the filter.

TABLE II

Operating Conditions and Results of Four Pilot-Plant Filtration-Extraction Runs

Run No.	1[a]	2[a]	3[a]	4[b]
Conditions				
Wt. of cooked bran processed, lb.	525.0	370.0	412.0	400.0
Oil in cooked bran, %	15.7	14.3	13.8	16.5
Moisture in cooked bran, %	14.0	14.5	18.9	16.7
Feed rate of cooked bran, lb./min.	5.0	5.0	11.0	5.0
Solvent to bran ratio, lb./lb.	1.1:1	1.1:1	0.8:1	1.1:1
Solids in slurry, %	28.0	36.0	37.0	32.0
Results				
Vacuum required, inches mercury	0.5-1.0	0.5-1.0	0.5	2-4
Nitrogen blowback, cu. ft. per hr.	40	40	80	130
Cake thickness on filter, inches	1.5	1.5	1.5-2.0	1.5
Oil in filtrates, %				
1st	16.0	12.5	17.0	15.0
2nd	8.3	5.5	7.0	5.5
3rd	3.5	1.8	1.7	1.7
4th	1.3	0.4	0.2	0.5
Fines in first filtrate, wt., %	0.5	0.7	0.8	2.1
Solvent in extracted bran, %	29.0	41.0	36.0	42.0
Residual lipides in extracted cake, %	1.1	1.2	1.5	1.5

[a] Runs using bran from standard milled rice.
[b] Run using bran from "Converted" rice.

The filtration operating conditions for the four pilot-plant runs and the results obtained from these conditions are given in Table II. In the first three runs bran from standard milled rice was used whereas in the fourth run the bran used was bran from "Converted" rice which had been subjected to a severe "conversion" treatment and, in addition, contained a large proportion of rice "polish." Therefore it should contain as many fines as could be expected in any rice bran. In the third run the feed rate was increased from 300 to 660 lb. per hour and the solvent to bran ratio reduced from the initial 1.1 to 1 down to 0.8 to 1, to determine the effect of feed rate and of decreased solvent ratio.

Vacuum and blowback requirements in all cases are considered to be within practical commercial applicability. The requirements of a higher vacuum and an increased amount of blowback for the "Converted" rice bran run can be attributed to the higher percentage of finer particles present. However the 2 to 4 inches of mercury required for the "Converted" bran is well within practical operating range.

The solvent to bran ratio of 1.1 to 1 by weight is appreciably less than the approximate 2 to 1 used in many plants employing direct solvent extraction of rice bran. The low solvent ratio and the short slurry hold-up time should be conducive to reduction in solvent losses and to the enhancement of safety features of the process. Fresh solvent enters the system only as the final wash on the filter.

The concentration of oil in the first filtrate of each run is high for a low oil-bearing material, such as rice bran, and is due to the low solvent-to-bran ratio used. The amount of solvent to be evaporated is thereby decreased. Concentrations of oil decrease appreciably in the succeeding filtrates, which are subsequently used in the countercurrent extraction and washing procedure.

The fines content in the first filtrate is low compared to the 5 to 8% reported to be obtained in commercial practice; and the fines are readily filterable in the pressure-type of polishing filters. As noted in Table II, fines in the first filtrate for the "Converted" rice bran run are somewhat higher than for the other runs. It has been suggested that the fines in the mis-

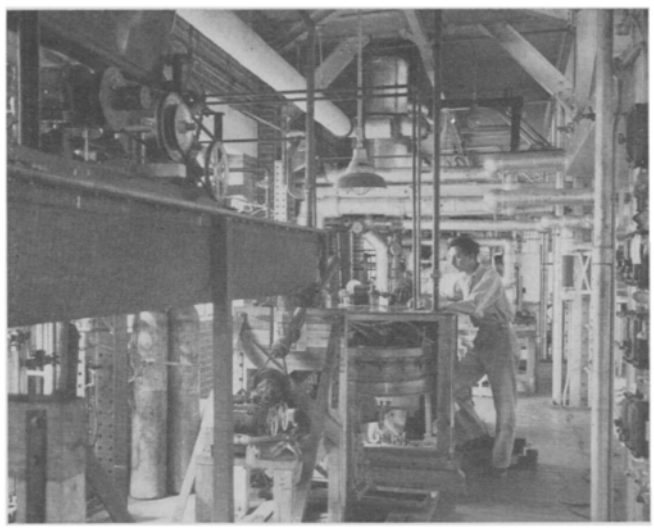

FIG. 3. Slurrying trough and pilot-plant, continuous, horizontal, rotary vacuum filter.

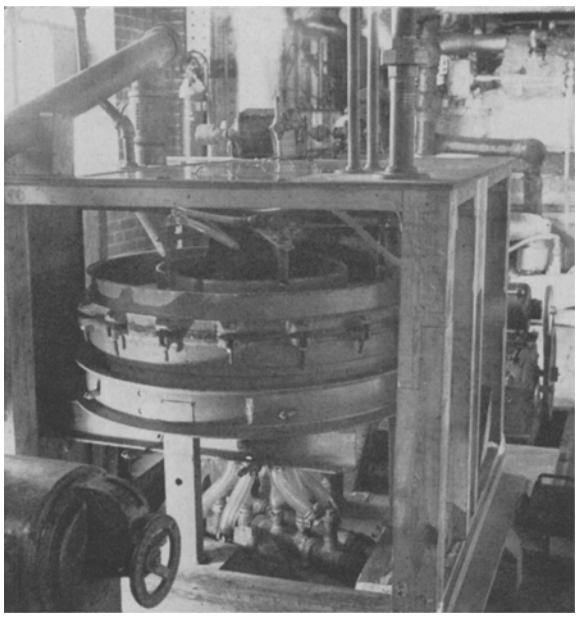

FIG. 4. Close-up view of filter.

cella before "polishing" could be further reduced by recirculating the first filtrate miscella onto the cake on the filter at a point between the oncoming slurry and first wash. The cake would act as a filter medium for the fines. The rapid filtration rate of rice bran makes this possible.

Solvent content of the extracted bran to the dryers ranged from 29 to 42% compared to the 50 to 60% obtained in commercial practice. Again this means less solvent to be evaporated, a saving in steam consumption and an increase in dryer capacity.

Residual lipides ranged from 1.1 to 1.5%. An increase in feed rate and a decrease in solvent-to-bran ratio, as shown in run No. 3, increased the residual lipides by only 0.4%. To determine residual lipides the official A.O.C.S. method, Ba 3-38, was modified in that the extraction time was increased from three to four hours.

Maximum capacity of the filter was not determined. However the 660 lb. per hour feed rate used in run No. 3 is equivalent to 7.9 tons of bran per day, or approximately 2.6 tons per day per square foot of filtering area. A 6-ft. diameter filter with a filtering area of 25 square feet would have a capacity of 65 tons per day; and one with a 10-ft. diameter and a filtering area of 65 sq. ft. would have a capacity of 169 tons per day.

Oil Evaluation

Oils used for refining and bleaching tests were obtained from a portion of the first miscella filtrate from each run. Before desolventization of the filtrate from run No. 1 it was observed that a rather heavy, firm layer of sediment had settled out. The clear miscella above the sediment was used. No sediment was removed from any of the other filtrates.

The filtrates were concentrated to about 90% oil under vacuum in glass laboratory apparatus and were vacuum-dried to remove the remainder of the solvent. The crude oils so obtained were refined and bleached by a modified A.O.C.S. method, as described by Pominski *et al.* (10). Other than removal of sediment from run No. 1 filtrate, the oils were not degummed or dewaxed before refining.

TABLE III

Refining and Bleaching Data and Results

Oil	F.F.A.	Refining data[a]			Photometric color[b]	
		Lye	Lye excess	Ref. loss	Refined oil	Bleached oil[c]
	%	°Bé	%	%	Red	Red
Run 1	2.8	12	0.5	18.22	3.7	4.1[d]
		14	0.5	18.64	4.7	3.4
		16	0.5	19.35	4.3	3.2
Runs 2 and 3	3.0	12	0.5	19.93	2.0	3.4[d]
		14	0.5	21.73	5.7	3.2
		16	0.5	22.24	6.8	3.6
Run 4	3.8	12	0.5	21.01	6.5	3.6
		14	0.5	25.20	8.5	4.2
		16	0.5	21.08	9.2	4.8

[a] Refined by official A.O.C.S. method Ca 9a-41 for slow break cottonseed oil using 0.5% excess lye.
[b] Determined by official A.O.C.S. method Cc 13c-50.
[c] Bleached by official A.O.C.S. method Cc 8a-49 for cottonseed.
[d] Increase in red color by bleaching is attributed to removal of green color by the bleaching earth.

Pertinent refining and bleaching data and results are given in Table III. It is seen that the filtration-extracted oils refined and bleached to acceptable colors, as judged in comparison with commercially produced rice bran oils. The refining losses were considerably higher than would be obtained with cottonseed oils of comparable free fatty acid content. Possibly the presence of the gums and waxes known to be present in crude rice bran oil may have contributed to the high refining losses.

Summary and Conclusion

The pilot-plant application to rice bran of a recently developed method of solvent extraction, called filtration-extraction, has been described.

The process consists simply of mildly cooking the rice bran, cooling to about 130° F., slurrying the cooked bran with a miscella filtrate, filtering the slurry and countercurrently washing the cake three times on a continuous, rotary, vacuum filter, followed by conventional recovery of oil and meal products. The advantageous effects of cooking under the conditions described in the paper are reflected in the shorter filtration cycle time, better extractability, and virtual elimination of the fines problem. For example, tests showed that uncooked bran required filtration cycle times of more than 30 minutes compared to 30 to 60 seconds for cooked bran.

This development makes available to the industry a feasible continuous solvent-extraction process for rice bran. Feasibility of the process is due primarily to the practical elimination of the inherently serious fines problem and to other advantages of the process, such as the possibility of attaining high capacities for small units; lower solvent content of extracted cake and of final miscella, which should decrease recovery costs; lower solvent requirements; and extraction to a residual lipides of 1 to 1.5%. Because of the reduced equipment size and simplicity of the process, it should prove economically feasible for the small and medium-sized plant, where heretofore a solvent-extraction plant required the processing of relatively large quantities of material daily.

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The Flavor Problem of Soybean Oil. XI. Phytic Acid as an Inactivating Agent for Trace Metals[1]

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WITHIN the past few years the addition of inactivating agents to inhibit the possible chemical effect of trace metals in soybean oil has become an accepted processing technique. Soybean oil has a high tocopherol content and is very sensitive to metallic contamination, thus making it an excellent oil in which to study trace-metal inactivation. The removal of pro-oxidant metal traces reduces the rate of oxidation and allows for the extended functioning of the antioxidant. This paper reports on our studies with phytic acid as a metal-inactivator for soybean and other edible oils.

Phytic Acid

Interest in phytic acid previously has centered largely in nutritional and biochemical investigations, and it was in such studies that the metal-binding capacity of phytic acid was first observed. The ability of phytic acid to remove catalytic amounts of iron and copper from gelatin has been patented by Grettie (9), also by Pedersen (18) for the stabilization of mercaptans. Both rice bran (13) and oat "flour" (16), which are the finely ground bran coat, have received some attention as antioxidants for fats and foods. It is likely that phytic acid was an important constituent of these preparations. Other examples of phosphorus containing products which were active as antioxidants were reported by Hilditch, *et al.* (8, 12).

Phytic acid, the hexaphosphoric acid ester of inositol, occurs as the mixed calcium, magnesium, or potassium salts in all seeds, and these salts are collectively referred to as phytin. In soybeans, phytin is reported (6) to make up about 75% of the phosphorus content of the seed. An excellent literature review on phytin and phytic acid has been prepared (20) covering references up to 1950.

The present commercial source of phytin is corn steep liquor, from which phytic acid is precipitated by the addition of lime. Crude phytic acid can be prepared easily in the laboratory by dissolving the commercial phytin in a minimum amount of 6% hydrochloric acid. The solution is then diluted to reduce the concentration of hydrochloric acid to less than 1%, filtered, and demineralized by use of a high capacity ion exchange resin. Later the hydrochloric acid is removed by one or two vacuum evaporations. The darkening which occurs during stripping of the hydrochloric acid is removed by slurrying with carbon and filtering. In this simplified procedure, nonvolatile acids present in the original phytin will also tend to concentrate with the phytic acid. To obtain pure phytic acid repeated precipitations with barium or iron must subsequently be carried out.

Methods for the determination of phytic acid leave much to be desired. All methods depend upon an iron titration or some modification of the original method of Heubner and Stadler (11). In addition to the iron titration, the determination of total phosphorus, free orthophosphate, and an acidity titration to the methyl orange end-point are helpful in characterizing phytic acid solutions. The first replaceable hydrogen shows a distinct end-point, and apparently quantitative results are obtainable. The titration of the second replaceable hydrogen (phenolphthalein end-point) is not equivalent to the first, and the titration end-point is very poor. Electrometric titration curves show only a small dip at this point. It has been found that the phosphoric acid content of various samples of phytic acid have varied from 2.0 to 17.0%. A further complication in the scheme of analysis arises because the phosphoric acid content of phytic acid solutions increases as the sample ages and darkens. Apparently the phytic acid molecule retrogrades, splitting off phosphoric acid. In a phytic acid sample stripped of hydrochloric acid, the phosphoric acid increased from 5.88 to 6.80% within 4 months. Phytic acid does not give the molybdenum blue test,

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