The Centrifugal Acetone Foots Test Applied to Crude Soybean Oil. II. Rapid Method Compared With Other Measures of Phosphatides Content and Refining Loss

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MUCH work has been done in this country in the last 15 years on methods for the quality evaluation of crude soybean oil. Most of this work has been devoted to studying various methods for the laboratory refining loss determination since the practical yield of neutral oil is of primary interest to the main buyers of crude soybean oil. This effort led to the adoption of refining loss methods which are now embodied in a trading rule currently in its fourth season of application; and the consensus of opinion is that the methods have proved adequate for trading purposes. However neither the Refining Committee nor oil chemists interested in methods for refining control are satisfied that the present official methods represent the ultimate. The Refining Committee's preoccupation for several years with centrifugal refining tests suggests that one reason for this interest is the development of new commercial oil refining processes and improvement of older ones, advances which result in ever lower refining losses.

Now while no one expects the laboratory refining loss results to agree exactly with plant refining results, even as compared against a single plant process, it is very desirable that they should correlate with plant results reasonably well. Yet as the number of different kinds of commercial refining processes increases, and especially as the spread between the level of the laboratory official loss results and the plant results becomes wider, we can expect and actually find that the correlation becomes poorer. The worse it becomes, then the less reliable become the trading rules embodying such refining methods in fulfilling their intended function; i.e., to afford equitable price adjustment proportional to values obtainable from the oil. We are thus beginning to hear more discussion of other bases for crude oil evaluation; for example, neutral oil value, either as determined by the Wesson analysis or by separate determination of the three main loss components. Linteris and Handschumaker (8) recently developed these ideas and described a chromatographic method for the determination of loss components, using a much quicker and less involved technique than the Wesson loss determination. Their data compare values obtained by these three different measures of total loss components, and they discuss some sources of error in the acetone-insoluble and Wesson loss determinations.

The senior author of the present paper has been interested for about 12 years in the prediction of refining loss from the analysis of crude soybean oil and has used HCl break as the measure of gums or acetone-insoluble content (1). This was based on numerous comparative determinations in our own laboratory on crude oils and concentrated phosphatides and also on published results (7, 9). The main justification for this procedure has been in the way that "expected losses" calculated from an empirical equation embodying free fatty acids, break, and m&v have correlated with actual refining results. We sometimes call this the calculated loss and have found little difference in the way it predicts plant refining loss as compared with prediction from the laboratory test loss.

Centrifugal Foots as a Measure of Two Loss Factors

In referring to "loss components" we generally think of three, as mentioned above, and also by Linteris and Handschumaker (8): free fatty acid content, acetone-insoluble content, and moisture and volatile. Now these three are sufficient to cover the loss components when thinking in terms of the Wesson loss, or the theoretical minimum refining loss which it is possible to realize on a given oil based on its chemical composition. The main concern however is usually with refining losses which exceed the Wesson loss by appreciable amounts as do most commercial and laboratory refining losses, and hence we cannot ignore a fourth important "component" as a loss factor. This fourth factor may be called the emulsifying potential of the acetone insoluble material, and it is a fourth dimension which makes the refining loss turn out to be what it is on an oil with a given analysis when refined by a given process. Now the emulsifying capacity appears to be at least partially evaluated by the centrifugal foots method. This is deduced from circumstances inherent in the test; but, more convincing, we find some evidence for this idea in our data.

The empirical equation for predicting refining loss (introduced below) contains a factor which represents the average emulsifying potential of the gums in the oils covered by our experience. Thus the actual refining loss for an individual oil may deviate slightly above or below the loss predicted by the equation according to whether the emulsifying capacity of the contained gums is more or less than the average. In the centrifugal foots method we do not achieve complete separation of oil from the acetone-insoluble fraction, just as in degumming crude soybean oil the commercial lecithin produced is associated or emulsified with about one pound of oil for every two pounds of acetone-insoluble material. So oils containing relatively strongly emulsifying gums show a relatively high centrifugal foots value for a given acetone insoluble content, and vice versa. In short, we can conclude that the centrifugal foots value is a measure of a combination of two factors: a) the amount of acetone-insoluble material present and b) the relative emulsifying capacity of that material. Then, combined in a suitable manner with the other loss components, ffa and moisture and volatile, we obtain a quantity that covers all the factors which determine refining loss that are inherent in the oil itself. This is the basis of expecting a good correlation of refining loss calculated from the analysis embodying centrifugal foots value, with actual refining loss. Results to follow will show how nearly this is borne out by laboratory experience.

As a test of the above, we may consider that if acetone insoluble content measures only the quantity of gums and centrifugal foots measures both their quantity and their emulsifying capacity, then in a group of oils, as the ratio of acetone insoluble content to centrifugal foots decreases with an increase of the latter due to increased emulsifying capacity, the differences between the refining losses predicted by the centrifugal foots and actual refining losses should increase, i.e., trend toward the positive direction. In other words, the pattern of the plotted points, instead of being a random scatter covering a roughly circular or square form, should be flattened and show a slope. Figure 1 is the graph of this, and while a rather definite trend supporting the idea under test is evident in the shape of the pattern made by the points, it is apparent that the weight to be given this indication must be limited by the relatively small amount of data involved. It is at least highly suggestive that the centrifugal foots test when combined with ffa does measure the same factors as do laboratory refining loss determinations. The data plotted in Figure 1 were determined on the same samples covered in Table III plus four other crude soybean oil samples not shown.

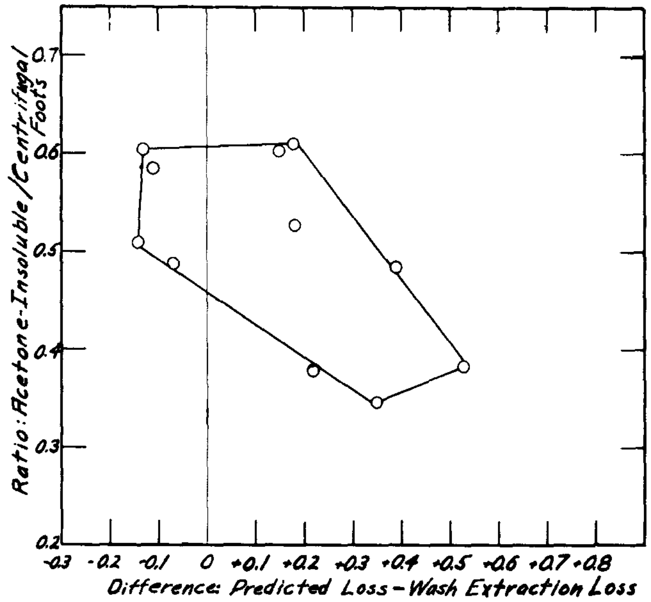

FIG. 1. Graphical test of the idea that the centrifugal foots value reflects the relative emulsifying capacity of the gums in crude soybean oil as well as the amount present.

This is given support by the asymmetrical pattern of the area covered by the plotted points, trending down at the right.

We may note that it is the assumed evaluation of emulsifying capacity of contained gums by laboratory refining loss tests which gives such tests better general acceptance for grading oils for plant refining than, say, indications based on analysis. Experience shows however that owing to differences in the conditions prevailing between laboratory and plant refining processes, the laboratory refining tests do not always properly measure the emulsifying potential with respect to some plant processes and thus may fail to predict the refining losses which are obtained by these plant processes. It remains to be determined whether, when correctly weighted in the combining equation, the refining loss scales derived from the centrifugal foots value, acetone insoluble contents, etc., better represent average commercial refining loss than does a gravity-separation cup refining loss method.

Our efforts to compare the correlations of several different methods of predicting refining loss, including laboratory loss determination, with actual plant refining loss results have been inconclusive because none of them correlated particularly well. Since the plant results available to us represent routine day by day operations, we have concluded that this poor correlation must result from one or more of the following possibilities, probably a combination of all of them: a) the fact that three different commercial refining processes are involved; b) a possibility that the samples routinely drawn to represent the oil refined in the plant may not be uniformly accurate; c) the possibility that the weights reported for the crude oil started and the dry refined oil produced may not be uniformly accurate; d) the occurrence of ordinary laboratory testing errors; e) normal variability in the refining performance itself. We have been actively concerned with this last in developing the material covered in this paper. Admittedly most of these uncertainties could be eliminated from plant refining results by obtaining them under the direct close supervision of the laboratory, especially the operation of drawing the sample and checking the weights and accounting. There should also be a check for the occurrence of any abnormalities in a given run. The authors have not however been able to provide such close supervision to obtain unimpeachable plant refining loss data on which we have an array of analytical and other laboratory results, especially covering a wide range of quality of crudes. We have thus found it necessary in this study to eschew actual plant refining loss results and have chosen instead a particular laboratory test refining method as a primary criterion against which to compare and correlate various other methods of predicting refining loss.

The laboratory refining method chosen is one investigated some years ago by a subcommittee of the A.O.C.S. Refining Committee (2, 3). It was called the glass kettle method; and, while not developed beyond a preliminary stage then, it was modified in our laboratory and has since found useful application both in research and control. It represents a method of commercial refining widely used in Europe and to some extent in this country (6, 10). It is known on plant scale as the wash or water extraction method of caustic soda oil refining. Our experience with this laboratory test method convinces us that on the average it evaluates crude soybean as well as any other method; and because it gives results closer to the more efficient plant processes, we believe it to be a better indicator of probable plant loss than the official cup method. Accordingly, since the loss values of no single laboratory method will correlate with the different levels of losses which characterize the five or six distinct commercial processes in use, the choice of one found to be fairly reliable on the basis of our experience seemed justified. The reason the one chosen is not the official cup refining loss method used currently for grading crude soybean oil in trading is that most of our data already available are in terms of the glass kettle wash method, also because we want to consider the official method as one of those several

TABLE I

100—Neutral Oil Content, by Various Measures
(Values in parentheses are rank order numbers)
% Neutral Oil = 100 — (ffa% + b x Measure + m&v)

Oil No.	Loss Measure	Acetone Insoluble	Phosphorus	Centrifugal Foots	HCl Break	Ash	Wesson Loss	Chromatographic Loss
	Constant, b	1	26	0.55 extracted 0.38 expeller	4.37	6.76	......	......
1	Degummed Extracted	0.23(1)	0.40(1)	......(1)	0.36(1)	0.39(1)	0.32(1)	0.34(1)
2	Extracted	2.02(2)	2.23(3)	1.89(2)	2.06(2)	1.94(2)	1.65(2)	2.49(2)
3	Low Temperature Expeller	2.33(3)	2.10(2)	2.52(5)	2.38(3)	2.29(3)	1.74(3)	2.59(3)
4	Extracted	2.42(5)(4)	......(4)	2.31(4)	2.57(6)	2.66(5)	2.08(5)	2.87(6)
5	Extracted	2.42(5)(4)	2.61(6)	2.27(3)	2.55(5)	2.49(4)	2.39(6)	2.69(4)
6	Extracted	2.43(6)	2.39(5)	2.58(6)	2.50(4)	2.87(7)	2.04(4)	2.74(5)
7	Expeller	3.02(7)	2.77(7)	2.99(7)	2.75(7)	2.71(6)	2.43(7)	3.23(7)
8	Expeller	4.49(8)	3.84(8)	4.50(8)	4.15(8)	4.07(8)	3.42(8)	4.44(8)

to be compared against a single base, and finally because the losses indicated by it are generally somewhat higher than modern plant losses.

Experimental

Thirty-six crude soybean oils from a limited variety of sources but representing production covering a period of at least six months (1950 and 1951 crop) were analyzed for centrifugal foots content, HCl heat break, ffa, moisture and volatile (hereafter m&v), and laboratory wash extraction refining loss.

A second series of eight samples representing a variety of types and sources of oil was submitted to the following determinations: ffa, m&v, centrifugal foots, HCl heat break, acetone-insoluble, phosphorus content, and ash; losses were determined by the A.O.C.S. cup refining method, the wash extraction (glass kettle) method, the Wesson method, and by the chromatographic method. An additional four samples were analyzed for ffa, m&v, centrifugal foots, HCl heat break, and acetone-insoluble content, and the wash extraction loss was obtained on these samples.

Free fatty acids were determined by the A.S.T.M. method. M&v, HCl heat break, A.O.C.S. cup losses and ash were determined by A.O.C.S. procedures. Acetone-insoluble was determined by a modified lecithin analysis procedure commonly used in the industry, which is quite similar to the A.O.C.S. method; centrifugal foots, by the procedure previously described, (4); Wesson loss, according to the method described by Jamieson (5), with slight modification; chromatographic loss, by the procedure described by Linteris and Handschumaker (8); and wash extraction loss, by a modification of the procedure used by the Refining Committee in 1945 (2, 3). Phosphorus content was determined by a colorimetric procedure currently being studied by a committee of the A.O.C.S.

Derivation of Equations

The experience mentioned above in using HCl heat break as a measure of phosphatides and gums has shown that an equation of the form: expected loss = a x ffa + bP + m&v, where P is a measure of phosphatidic and gum constituents and *a* and *b* are constants, results in an acceptable approximation of refining loss. Occasionally other equation forms were investigated, but generally poorer correlation was obtained by their use. Accordingly in the work reported here this equation has been used to calculate loss, using the analytical factors shown in Table III as measures of the phosphatides and gums. Long use of HCl heat break in this equation fixed the constant *a* at a value of 3.0. In fitting the data reported here for acetone-insoluble into the equation with $a = 3$, it was necessary to use a value of $b = 0.77$. If the constant *b* was assigned the value of 1.0 when using acetone-insoluble content, then the value of *a* became 2.2, best to predict wash extraction loss. Accordingly the constant *a* was assigned the two values 2.2 and 3.0, and the corresponding value of *b* was calculated for these conditions in relating centrifugal foots, acetone insoluble, phosphorus content, and ash to wash extraction loss. Also the sum of the ffa, acetone-insoluble, and m&v contents was used to represent non-triglyceride constituents, and by using suitable factors relating centrifugal foots, phosphorus, HCl heat break, and ash contents to acetone insoluble content these latter four measures of phosphatides and gums were then used to calculate non-triglyceride constituents.

Discussion of Results

In Table I, showing this last mentioned data, it is found that on several oils there is excellent agreement between chromatographic loss and the minimum loss based on acetone-insoluble content. The chromatographic loss is higher when the agreement is not close, as on the first four oils. The Wesson losses are low except on the degummed oil and on the sixth oil.

TABLE II

Correlation of Predicted Loss With Wash Extraction Loss on Thirty-Six Samples. Predicted Loss Calculated From Two Measures of Phosphatides Content (P) Combined With Other Loss Components

Refining Loss = a ffa + bP + m&v

Constant, a.	3.0		2.2	
	HCl Heat Break	Centrifugal Foots	HCl Heat Break	Centrifugal Foots
Constant, b.	3.1	0.40	4.0	0.52
Deviation from Wash Extraction Ref'g Loss:				
Mean	±0.20	±0.17	±0.19	±0.16
Standard	±0.214	±0.181	±0.243	±0.205
Maximum	−0.42	+0.42	−0.65	+0.50

Table II is a summary of data obtained in routine application of the methods, comparing the relative accuracy with which HCl break and centrifugal foots, when combined with ffa and m&v, will predict the wash extraction loss. The summary includes two forms of the equation for predicting wash extraction loss. The constants used are those which, for the particular data shown, gave a direct as well as the best correlation with wash extraction loss. This data shows the somewhat better performance of centrifugal foots over HCl break in predicting refining loss.

Table III is a summary tabulation of the data collected on the series of eight oils arranged according

TABLE III

Analysis and Refining Losses on a Diverse Selection of Crude Soybean Oils

Oil No.	Type	% FFA	% M&V	HCl Break %	Cent. Foots %	Ash %	% Phosphorus × 26	Acet. Insol. %	Wesson Loss %	Chromatographic Loss %	A.O.C.S. Cup Loss %	Caustic Water Wash Extraction Loss %
1	Extracted Degummed	0.18	......	0.04	tr.	0.031	0.22	0.05	0.32	0.34	1.6	0.80
2	Extracted	0.47	0.10	0.34	2.4	0.202	1.66	1.45	1.65	2.49	3.2	2.60
3	Low Temp. Expeller	0.29	0.03	0.47	5.8	0.291	1.78	2.01	1.74	2.59	3.8	2.85
4	Extracted	0.50	0.10	0.45	3.1	0.305	......	1.82	2.08	2.87	4.0	2.95
5	Extracted	0.43	0.06	0.46	3.8	0.351	1.90	1.94	2.04	2.74	3.5	3.00
6	Extracted	0.75	0.14	0.38	2.5	0.237	1.72	1.53	2.39	2.69	4.3	3.20
7	Expeller	0.50	0.05	0.50	6.4	0.317	2.21	2.46	2.43	3.23	5.8	3.60
8	Expeller	0.66	0.08	0.78	9.9	0.492	3.10	3.75	3.42	4.44	7.3	5.78

to increasing wash extraction loss. While included mainly to show the basic data obtained by the several analytical determinations, it also shows how the different refining loss methods vary among themselves in estimating the toll taken in refining. This estimate becomes progressively greater as we go from Wesson loss to chromatographic loss to extraction loss, and finally to the A.O.C.S. cup loss. We are unable to explain the unusual relationship between the Wesson loss values and acetone-insoluble content, but, on first noting this, the Wesson loss determinations were carefully repeated in a different laboratory with similar results.

In Table IV all of the loss measures are compared to the wash extraction loss as a base. The average ratio of the calculated refining losses or the directly determined losses to wash extraction loss was calculated for each loss measure. (Since the refining losses for the degummed oil determined by all measures departed widely from the wash extraction loss, it has been omitted from this table.) This average ratio was then used to adjust each of the refining losses to the same approximate numerical level as that obtained in the wash extraction loss, and the deviations of these calculated losses from the wash extraction loss for each oil sample was then determined. The various loss measures are arranged in order according to the standard deviations from wash extraction loss. Changes in the constants *a* and *b* in the equation can cause marked differences in the precision with which each loss measure predicts wash extraction loss, so only the constants which give the best correlation are shown in Table IV. It is observed here that whereas the refining loss based on acetone-insoluble content correlates best with the actual test loss on the seven crudes, the centrifugal foots measure outranks acetone-insoluble when including the 12 oils on which acetone-insoluble contents were available. This good correlation of the refining loss as predicted from the analysis with centrifugal foots as the measure of gums content may be considered evidence that the centrifugal foots value measures the qualitative characteristics of the gums which contribute to establishing the value of the refining loss, for a given amount present.

Table V shows, for the eight oils on which complete data is available, the relative ranks of the loss measures according to their standard deviations from the wash extraction loss (Column A), and to their ability to rank the oils in the same order as wash extraction loss (Column B), and the same as A.O.C.S. cup loss (Column C). The total number of places that the oils were displaced from correct rank order by each loss measure was used to obtain the rankings shown in Columns B and C. In Column D the cumulative rankings of the loss measures according to the values of the standard deviation and to displacement from wash extraction loss are shown.

In Figure II the displacement from correct rank order is illustrated by the occurrence of a negative slope in the line connecting the points. Examination of Figure II shows that all of the disagreement in relative ranks were on oil 3, 4, 5, and 6. This is not unexpected since all of these particular oils had very nearly the same refining loss by all tests, thus slight differences became magnified. Even so, if we except the one sample of low temperature expeller oil, losses calculated from centrifugal foots would agree perfectly with wash extraction loss in terms of grade rank order. Note that the A.O.C.S. cup loss upgrades Sample No. 5 appreciably in comparison with the way all of the other determinations rank this oil and also downgrades the expeller oil No. 7 significantly in comparison with all other determinations. It is pertinent

TABLE IV

Deviations of Predicted Refining Losses Determined by Various Measures from the Laboratory Extraction Refining Loss

Loss Measure	Loss Formula	Multiplier[a]	Deviations from Extraction Refining Loss			
			Standard[c]	Average	Maximum	Range
Group A: For Seven Crude Soybean Oils						
Acetone Insoluble Content	ffa + AI + m&v	1.25	±0.132	±0.11	+0.18	0.35
Centrifugal Foots	3 ffa + 0.40 CF + m&v	0.974	0.251	0.21	+0.42	0.64
HCl Break	ffa + 4.37 Br + m&v	1.25	0.281	0.19	—0.59	0.85
Wesson Loss	none	1.52	0.328	0.25	—0.59	1.02
Chromatographic Loss	none	1.13	0.354	0.23	—0.78	1.06
Phosphorus Content[b]	ffa + 26 P + m&v	1.30	0.388	0.26	—0.77	1.08
A.O.C.S. Cup Loss	none	0.760	0.389	0.25	+0.84	1.18
Ash Content	ffa + 6.76 ash + m&v	1.25	0.418	0.31	—0.69	1.28
Group B: For Twelve Oils, Including the Above Seven						
Centrifugal Foots	3 ffa + 0.40 CF + m&v	0.965	±0.211	±0.16	+0.39	0.63
Acetone Insoluble Content	ffa + AI + m&v	1.24	0.274	0.20	—0.57	1.10
HCl Break	ffa + 4.37 Br + m&v	1.28	0.293	0.23	—0.60	0.93

[a] Multiplier = the average ratio of extraction refining loss to loss by loss measure.

[b] Data based on six of the samples.

[c] The calculation of standard deviation is based on the premise that each of the loss measures, when adjusted by the multiplier, constitutes in effect another method for determining the refining loss.

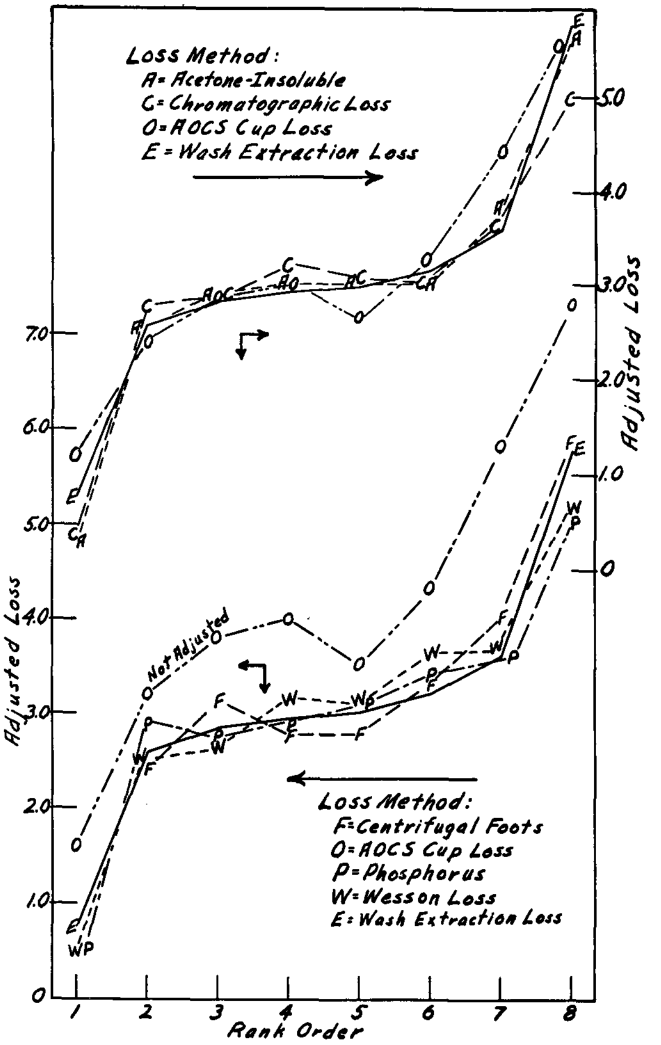

FIG. 2. Correlation with wash extraction refining losses of refining losses predicted by various measures. (Data are plotted as two groups to avoid overcrowding.)

Refining losses based on various measures of phosphatides were calculated as indicated in Table IV. Note in top group the excellent agreement of the A and the E graphs, also the rather wide departure of the official cup loss values on crudes 1, 5, and 7.

that the low temperature expeller oil is commercially rare, and moreover other data have shown that the centrifugal foots test gives abnormally high values on this type.

Conclusions

It is realized that the weight to be given the indications of some of these data is somewhat limited by the relatively small number of samples on which all of the determinations were made though a far larger number of crudes was covered by three of the loss measures, including centrifugal foots. While the rank order of the various refining loss measures might turn out to be somewhat different if determined on a different series of crudes, especially as those at the center of the scale in this series are very similar in quality, this work shows that the centrifugal foots method ranks as one of the best of the eight studied. Moreover, considering the amount of laboratory work required to perform any of these determinations other than centrifugal foots and the time lapse incident to refining loss determinations, this new quick method may be of significant interest to those concerned with grading crude soybean oil, even if only for refinery control purposes and on occasions when it is desired to grade crude soybean oil in the shortest possible time.

TABLE V

Rank Orders of Various Loss Measures

	Basis of Stand. Dev'n from Extr'n Ref'g Loss[a]	Basis total number of places oils were displaced from correct order		Composite Rank (*A&B*)
		Referred to Extr'n Ref'g Loss	Referred to A.O.C.S. Cup Loss	
	A	*B*	*C*	*D*
Method				
Extraction Refining Loss	....	....	3	0
Acetone Insoluble	1	1	4	1
Centrifugal Foots	2	2	3	2
HCl Break	3	2	2	3
Wesson Loss	4	1	1	3
Chromatographic Loss	5	3	5	5
Phosphorus	6	1	3	4
A.O.C.S. Cup Loss	7	2	....	6
Ash	8	4	6	7

[a] *Based on the seven crude samples.* See Table IV.

A limitation of the centrifugal foots method is that, in its present form, it gives only trace or zero results on most degummed oils. It is hoped that further work will develop a modification which will better apply to such oils. The method shares this limitation with the acetone-insoluble determination; for, while determinations of acetone-insoluble content are sometimes made on degummed oils, the results are unrealistically low due to partial, if slight, solubility of the phosphatides in acetone.

When color evaluation of the crude is a necessary adjunct to refining loss evaluation, all methods except those involving an actual refining of the oil are at a disadvantage. The centrifugal foots method is included in this category with seven out of the nine methods covered here, or all except the glass kettle and the A.O.C.S. cup refining methods. However it has been found feasible to develop a fairly rapid method for obtaining refined oil just for a small scale bleach test, for example, along the lines of those employing centrifugal separation of soap stock studied by the Refining Committee in recent years.

Summary

Comparisons are made between eight methods of grading crude soybean oil with respect to a laboratory refining loss determination which closely simulates commercial wash kettle practice. This list of methods includes the five analysis factors, acetone-insoluble, HCl heat break, centrifugal foots, phosphorus and ash contents, *each combined with* percentage of ffa and moisture and volatile. It includes the official A.O.C.S. cup loss, the Wesson loss, and the chromatographic absorption loss determinations. On 12 crudes on which laboratory wash extraction loss and losses determined from centrifugal foots values, from acetone-insoluble contents, and from HCl break values were available, the centrifugal foots values best predicted the refining loss. The standard deviations of each method from the wash extraction loss were, respectively, 0.21%, 0.27%, and 0.29%. On seven of these crudes on which all nine determinations were available, acetone insoluble content showed the best correlation, followed by centrifugal foots and HCl heat break. The centrifugal foots test generally gives zero or trace results on degummed oil. By use

of this method however the refining loss grade of crude soybean oils may be estimated in as little as one hour of elapsed time, and it requires only 5 to 10 minutes of the operator's time.

Acknowledgments

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Notes on the Centrifugal Foots Test Applied to Crude Cottonseed Oil

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AFTER modifying the centrifugal foots method originally developed for raw linseed oil (1) so that it gave satisfactory results on crude soybean oil (2) (and indeed proved to be in the second case a more useful test for control purposes than when applied to raw linseed oil), we considered that it might have some usefulness for the approximate evaluation of quality in crude cottonseed oil, if applicable.

To determine this, a group of crude samples of different origins and varying quality was requested of John J. Thoede of the South Texas Cotton Oil Company, who also supplied the f.f.a. and refining loss values on the oils. Centrifugal foots tests were run on a few of these under different conditions. The results which follow are decidedly preliminary and are presented merely as a point of departure for any other investigators who may be interested in working on the development of a quick easy test for estimating the phosphatides content of crude cottonseed oil since it is unlikely that we will do any further work on this particular application of the method.

In tests run to determine effect of oil to acetone ratio, it was found that the use of 10 ml. of oil to 40 ml. of acetone (ratio originally developed for soybean oil) resulted in a higher percentage of separated foots than obtained with any other ratio. In a test to determine the minimum time of centrifuging, four oils were run for 15 minutes and 30 minutes. Only very slightly lower results were obtained after 30 minutes. The reduction ranged 0.1 to 0.3% on values of 3.3 to 5.1% foots, due to the extra 15 minutes of centrifuging.

Briefly, the method discussed here involves the precipitation of the phosphatides, etc., in a 10-ml. portion of the oil with 40 ml. of C. P. acetone and in the presence of 10 ml. of a saturated and acidified solution of calcium chloride. (See A.S.T.M. Method D555-47.) The reagents and oil are mixed by violent shaking for one minute in a specially designed centrifuge tube and, after settling 5 minutes, are centrifuged for 15 minutes under a given force, determined by a specified r.p.m. for the radius used. Then the volume of the separated foots stratum is read to the nearest 0.01 ml. For a detailed statement of the method (bearing in mind the qualifications discussed here), reference is made to a previous paper (2) by the authors.

Results and Discussion

Referring to Table I, the heating cycle may not always be necessary, but as the amount of data here is quite limited, this point should be investigated further. On high foots oil it would be advisable to spin for a longer time as complete compaction was probably not attained in 15 minutes of centrifuging; or, alternately, the crude could be diluted with 100 to 300% of its volume of refined oil before subjecting the oil to the test, in which case appropriate factors would have to be used to convert the volume separated to percentage by volume of the original oil.

TABLE I

Relation of Centrifugal Foots Results to Refining Loss. Effect of Preheating and Cooling

Type	F.F.A.	Centrifugal Foots			Calculated Loss[b]	A.O.C.S. Loss
		Not Heated	Heated to 65°C. and Cooled			
		Average	Checks	Average		
	%	%	%	%	%	%
Hyd.	0.7	0.3	0.3, 0.3	0.3	2.3	2.6
Exp.	0.7	3.3	3.3, 3.2	3.3	4.1	4.8
Hyd.	0.8	4.5	4.2, 5.2, 5.1	4.8	5.3	4.9
Exp.	0.9	2.3	2.2, 2.2	2.2	4.0	5.1
Hyd.	1.4	2.9	3.2, 3.2	3.2	6.1	5.4
Exp.	1.2	3.6	3.7, 3.8, 3.9, 3.7	3.8	5.9	6.5
Hyd.	0.8	3.7	5.1,[a] 3.9, 3.2, 3.5	3.5	4.5	6.9
Hyd.	1.4	4.9	4.2, 4.4	4.3	6.8	7.7
Hyd.	1.0	17.0	17.5, 17.0	17.3	13.4	11.4
Hyd.	2.0	>20	17.8, 18.5	18.2	16.9	14.2

[a] Value not in average.
[b] = 3 × f.f.a. % + 0.6 × centrifugal foots % (heated).

We know that the principal reason for some lack of precision in these results was the presence of quantities of meal in some of the oils. Much, but not all, of this meal separated from the foots layer and settled to the bottom of the centrifuge tubes. When a large quantity of meal was present or when centrifuging