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The Raw Linseed Oil Acetone Foots Test. I. A Centrifuge Tube and Method for Rapid Determination[1]

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CRUDE or raw vegetable oils are generally graded by tests which reveal qualities of direct concern in the principal application of the oil. Practically all crude cottonseed, peanut, and nearly all crude coconut and soybean oils are first alkali refined, and hence the refining loss test is the principal grading factor. Although a considerable quantity of linseed oil used is also alkali refined for the production of high-grade varnish oils, or acid refined to make grinding oils, an even larger quantity, roughly one-half of the total amount produced, is sold by the processor as raw, probably in most cases after being given a very simple clarifying treatment. A very large part of this raw oil is used as such.

It is natural therefore that raw linseed oil should be evaluated by tests which show its quality as raw oil, rather than with particular reference to its refining loss. It is sold thus under standard specifications, generally those of the American Society for Testing Materials (hereinafter, A.S.T.M.), although the Federal Specifications (TT-O-369) are practically the same. The A.S.T.M. specifications follow:

Specific Gravity—15.5/15.5	0.931-0.936
Acid Number, maximum	4.0
Saponification Number	189.0-195.0
Unsaponifiable, maximum %	1.50
Iodine Value (Wijs), minimum	177.0
Loss on Heating at 105 to 110°C., max. %	0.2
Color—not darker than a freshly prepared solution of 1.0 g. of $K_2Cr_2O_7$ in 100 ml. of pure H_2SO_4 (sp. gr. 1.84)	
Foots, maximum, %—Heated oil	1.0
Chilled oil	4.0

In isolated cases buyers may have some modifications of these specifications.

It may be noted that several of the items in the above are inherent in the flaxseed and thus not subject to control by the processor except by blending with oil from other lots of flaxseed, an action which may not always be possible. These are: iodine value, specific gravity, saponification value, unsaponifiable content, and in a certain sense acid number. (The last is subject to control by neutralizing part of the oil though that part then ceases to be raw.) The other items are largely subject to control, but the chilled foots test only so by the application of treatments not ordinarily regarded as being expected or necessary for conditioning *raw* oil for shipment. (Such a treatment would be refrigerating the oil prior to filtering or centrifuging.)

Therefore the main properties determining raw linseed oil quality, not inherent in the flaxseed, are both controllable in the processing. These are the heated foots test and the "loss on heating at 105 to 110°C.," and since the latter, or the M. & V., must be held considerably below the A.S.T.M. maximum to assure reasonable clarity, it is generally not a factor. We are thus left with the heated foots test as the main criterion of quality in this oil, i.e., among those subject to convenient control in processing. The significance of the indications of this test and of the chilled foots test, as well as the nature of the test itself and the limitations to reproducibility of results, merit close consideration inasmuch as it was these things which led to the development of the centrifuge modification.

The official test procedure is as follows (1):

> *Foots*
>
> 14. a) With all materials at a temperature between 20 and 27°C., mix by shaking for exactly 1 min. in a graduated tube 25 ml. of the well-shaken sample of oil, 25 ml. of acetone, and 10 ml. of the acid calcium chloride solution. Clamp the tube in a vertical position where settling may take place for 24 hrs. The temperature during this period should be between 20 and 27°C.
>
> b) At the end of the 24 hrs. determine the volume of the stratum lying between the clear calcium chloride solution and the clear acetone and oil mixture to the nearest 0.1 ml. or fraction thereof. This volume multiplied by four expresses the amount of foots present as a percentage by volume.
>
> *Heated Oil Test*
>
> 15. Heat a portion of the oil to 65°C.; hold it within 2°C. of that temperature for 10 min.; then cool it to room temperature (20 to 27°C.). Subject the sample promptly to the foots test as described in Section 14.
>
> *Chilled Oil Test*
>
> 16. Heat a portion of the oil to 65°C.; hold it within 2°C. of that temperature for 10 min.; then place in a dry, clean bottle, stopper tightly, and place in a cracked ice and water mixture (0°C.) for exactly 2 hrs. At the end of this time place the bottle for exactly 30 min. in a water bath at 25°C.; then subject promptly to the foots test as described in Section 14.

Specific directions are given for preparing the saturated calcium chloride solution in hydrochloric acid. The acetone must conform to A.S.T.M. specifications for that solvent, and these permit up to 2% of moisture. All the work in our laboratory shows that variations of the moisture content in this range can significantly affect the foots test results, but a survey of the acetone stocks in several of our laboratories indicated that it appears ordinarily to be substantially dry. So this variable probably does not contribute to the poor reproducibility of foots test results experienced in checking between different laboratories. It may be noted here that before July

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[1] A modification of this test has been developed specifically for use on crude soybean oil and a paper describing it is in preparation.

1949 the directions for preparing the calcium chloride was such that wide variations in concentration could be expected, and indeed were experienced, as surveys of this factor by one of the authors revealed. This followed from the ease with which calcium-chloride forms a super-saturated solution.

Nature of the Foots Stratum. It is apparent that the foots test result is a highly empirical quantity, being percentage by volume of an emulsion consisting of a large part of the acetone-insoluble component of the raw oil, emulsified with the acetone-oil solution and a small amount of the calcium-chloride solution. The same is true of the chilled foots test result, except in this case we have the complicating effect of such wax and stearine as separate with the foots. Some of the results being reported here will show this to be a very serious complication indeed since the lack of reproducibility of chilled foots results is far worse than in the case of the heated foots test. (See the check sample results and discussion of these accompanying.) The lack of concordance and variability of results on a given sample therefore is clearly due to the variable amount of the oil-acetone and of the aqueous solution components in the emulsion stratum which is read and reported as *foots.* Since an emulsion composition formed under conditions such as prevail in this test can be expected to vary with the actual conditions applying in making a given test, it is not at all surprising to anyone familiar with the nature of emulsions that the results are of rather poor reproducibility. The statement of the conditions in the official method is such that significant variations may occur without violating the letter of the method. The effect of some of these variations will be illustrated in results given here, but at this point it will suffice merely to enumerate them with a few pertinent comments.

Temperature. A relatively wide temperature range is permitted, 20 to 27°C.(68 to 80.6°F.). Results presented below show that test indications may be significantly affected by variations within this range. It is to be noted that the top temperature named is one that cannot be maintained in most laboratories during the summer without employing a cooling bath, or air conditioning.

Agitation. The intensity is limited by specifying that the oil and reagents be mixed in the foots tube itself. Shaking these tubes is a somewhat awkward operation, and the senior author found in the only two laboratories using this test which he has visited outside of his employer's laboratories that agitation was performed by alternately inverting the foots tube. After each up-ending the bubble or air space passes from the lower to the upper position, thus giving a far less intense agitation than would be obtained by shaking. Although in many comparisons the degree of agitation seems to have little effect it has been found that it just as often does affect the results. It may be noted that the practice of mixing by simply inverting the tubes repeatedly does not conform literally to the condition in the method which directs, "mix by shaking." (See Table II).

Concentration of the Calcium-Chloride Solution. Experience has shown this to affect the results at times. This concentration is conveniently indicated by the specific gravity of the solution. While it would seem that this concentration must be constant in accordance with the precise and limiting directions for making the solution, such unfortunately is not the case. The solution may become supersaturated by cooling, say overnight. Then the excess salt may crystallize and concentrate in the bottom of the reagent bottle. As it is then warmed to 20°C. or higher, the crystals are slow in redissolving, in fact, have a tendency not to dissolve at all unless agitated for a considerable time. Thus an unwary chemist who uses the supernatant solution is obviously using one which is considerably depleted in salt content.

Worse, it was recently found that two different brands of calcium-chloride dihydrate gave solutions with significantly different specific gravities when made into the test reagent by the official directions. One of these was correct for the composition expected, the proper one, whereas the other was decidedly higher, indicating that the solution was highly supersaturated. This simply means that the salt used did not have the composition indicated on the reagent jar label as the dihydrate but contained an appreciable quantity of monohydrate.

Handling the Tubes Before and on Reading the Results. While such handling should perhaps be considered a violation of the spirit of the official method, which directs that the tubes shall be clamped in a vertical position during 24-hour settling period, it seems likely that chemists using the test may (and probably most of them do) carry them to a window or a good light to read the volume of the foots strata. To make this reading accurately may be a problem since both surfaces of the foots stratum may be irregular. Thus a reading of these surfaces to .025 ml. is a difficult matter at best, and the right kind of light is a necessity. Careful handling at the time of reading will not change the results, but vibration or jarring may do so, especially when visible pockets of bulk acetone-oil solution are trapped in the foots layer; for such oil pockets can be released, by a disturbance, into the phase where they rightfully belong. Indeed, at times the emulsion structure is what might be called *massive,* rather than colloidal or microscopic, and pockets of the oil phase may be held trapped in the foots layer by filmy interfaces having dome-shaped elevations irregularly spaced about the upper surface of the stratum. When this condition occurs and a reading is taken on the foots test (obviously a rough estimate under these conditions), this reading must be considered official as the official method is stated. However if the tube is rotated rapidly just once with the thumb and forefinger, disturbing and breaking down the films which hold the pockets of oil phase in the foots stratum, the dome structures will collapse with the release of the oil. Then, if the test is allowed to settle a few minutes longer and another reading taken, it will be decidedly lower than the first reading and of course more nearly representative of the quantity which it is the intent of the method to measure. Still, the higher, less representative and less accurate value must be reported under the present wording of the method.

Compacting the Foots by Centrifuging

The use of a centrifuge occurs naturally to anyone using this test, and 11 years ago the senior author developed a method based on centrifugal separation of raw linseed oil acetone foots by actually isolating the foots in a tared vessel, drying free of volatiles, and weighing. The method was not put into regular use however because of the time consumed and the amount of manipulation involved. This method was of particular interest to us in showing just how much real footy matter does occur in raw linseed oil on a weight basis and to show the relationship which this true foots content bears with the occasional fantastic indications of the official acetone foots test. On using it again about a year ago, the authors went further; they then treated the centrifuged and vacuum-dried "basic foots" with chilled acetone, following the procedure generally used in the analysis of lecithin. This gave the acetone-insoluble content of the original oil (to the extent that the acetone-insoluble matter was separated in the foots layer).

The values gravimetrically determined as "basic foots" averaged about one-fourth of the foots content, as percentage by volume, indicated by the official gravity-settled result. This shows that what is measured in the latter test is largely oil and acetone and calcium chloride emulsified with the true foots. The "basic foots" separated has an acetone-insoluble content roughly of 50%; or the foots in the gravity test is composed of only about one-eighth acetone-insoluble matter; i.e., phosphatides and other components generally referred to as "gums" or mucilage, material which is not triglyceride oil (and which for the most part does not belong in the unsaponifiable fraction). Space limitations do not permit publication of this method, but it is available to anyone interested upon request to the authors.

In 1948 there was access to some correspondence describing another simple method for separating the foots by centrifuging (3). The procedure involved centrifuging first in a separatory funnel, drawing off the aqueous phase, then transferring the oil and foots phases to a centrifuge tube with graduated tip. This was centrifuged again to re-compact the foots and the foots volume then read. The authors next recalled the idea which one of them conceived (on first being introduced to the foots test 13 years ago) of designing a centrifuge tube in which the oil and reagents could be mixed, then centrifuged, and the volume read accurately without any transfers. Recently the design for a suitable tube was made and several ordered for trial purposes. The results, given below, have seemed so promising that it appeared desirable to publish a description of this tube and its method of use and to discuss the significance of the results obtained, especially in relation to the results obtained by the present official method, which is called the gravity method hereafter.

In addition to precision of results, which was of paramount importance, the following three points were considered essential in developing the test:

a) Retain the general outline of the A.S.T.M. gravity test.
b) Make the manipulation required as simple as possible.
c) Reduce to a minimum the time required to obtain results (limited by size and speed of centrifuge).

An investigation was made of the variables which could affect the centrifugal test and a standard method of running the test was chosen. The variables tested were time of gravity pre-settling, speed of centrifuging, time of centrifuging, and effect of intensity of agitation.

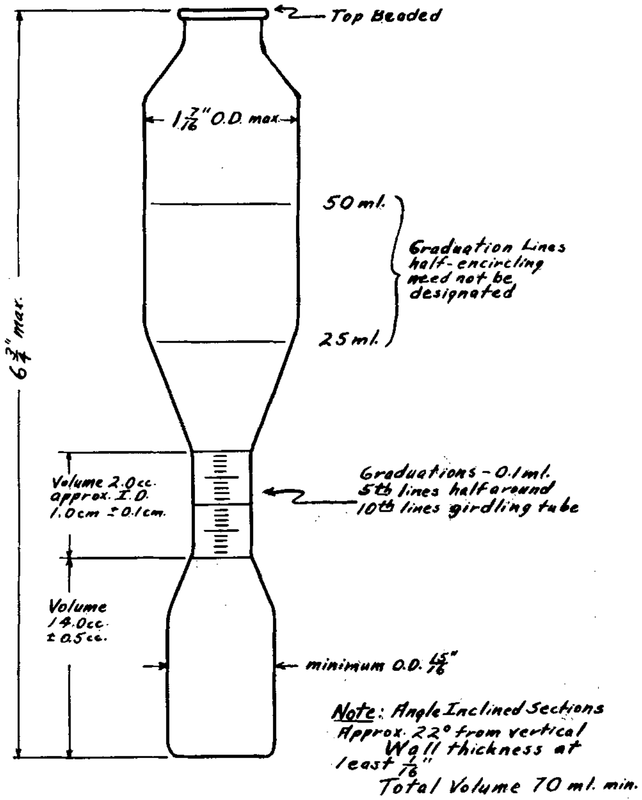

FIG. 1. Working drawing for the centrifugal foots test tube.

The Centrifugal Foots Method

Apparatus:

1. A graduated tube as shown in Figure 1. (Made by Ace Glass Corporation, Vineland, N. J.)
2. Bottom cushions, 1⅜" diameter by ¼" thick sponge rubber.
3. A centrifuge capable of swinging the tube in a 1½" diameter brass cup or tube holder. (A size 1, type C, International centrifuge equipped with a number 242 head and number 364 trunnions is satisfactory.)

Procedure:

Prepare the reagents and condition the oil sample for the heated or chilled test as specified in the A.S.T.M. official method (given above). Measure 25 ml. of the well mixed sample into the graduated tube, add 25 ml. of acetone to the upper mark, and add 10 ± 0.2 ml. of acid calcium chloride solution. Stopper the tube and mix the contents thoroughly for one minute. Place the tube in the brass cup and allow to stand for 5-15 minutes during which time the tubes and cups may be balanced for centrifuging. Centrifuge for 15 minutes at a speed determined by the distance (radius) of the bottom of the cups from the center of rotation of the centrifuge as follows:

For radius (R) of:	Use the following speed (N):
20 cm.	1850 RPM
23 cm.	1730 RPM
25 cm.	1660 RPM
28 cm.	1570 RPM
30.5 cm.	1500 RPM

Or for a centrifuge of a different dimension use the formula, $N = 8273\sqrt{1/R}$, where N = speed of operation, RPM, and R = distance from bottom of cup to center of centrifuge.

Remove the tubes from the supporting cups, read the volume of the foots layer, estimating both interfaces to the nearest 0.01 ml. and multiply by 4 to obtain per cent of centrifugal foots by volume.

Results and Discussion

A study made of the effect of gravity pre-settling on the indicated value of the centrifugal foots shows that the time of gravity pre-settling has no effect on the centrifugal foots test provided at least five minutes is allowed before centrifuging, which is about the required time for the two solution layers to separate. When centrifuging was started immediately after mixing the contents of the tube, there appeared to be a slight and unpredictable effect on the value of the foots test. In Figure 2 are plotted values of the indicated centrifugal foots against the time of centrifuging at speed for a single oil at various speeds of the centrifuge. Examination of the curves shows that at any given applied force there is an ultimate indicated foots content which is not changed by subjecting the foots to the force for longer periods of time. This is true even of the gravity test though a time of 24 hours does not seem long enough to obtain the ultimate value. It was from the data of Figure 2 that the condition, 15 minutes at 1850 RPM, was chosen as standard for the series of tests reported here. In Figure 3 are plotted additional data (chilled tests), showing that the conditions chosen for the centrifugal test do yield ultimate values of the foots content. It is particularly interesting to note that for the chilled gravity test even after 275 hours of settling it was not certain that the tests had reached an ultimate value.

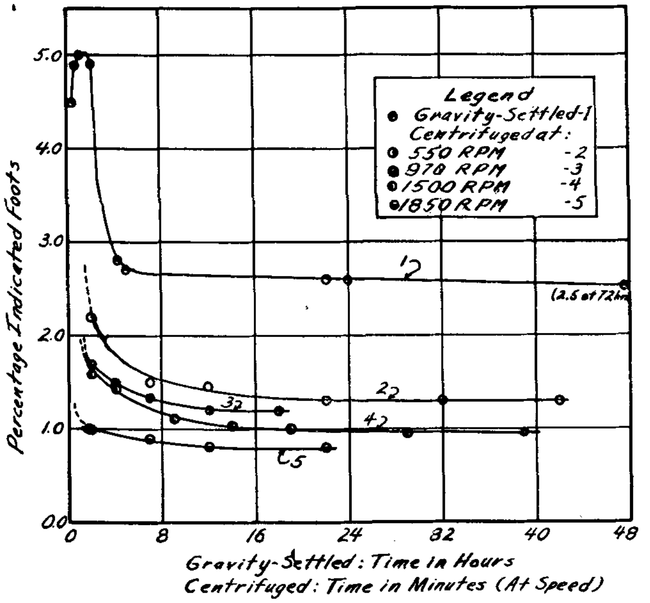

FIG. 2. Indicated percentage heated foots test versus time of gravity settling and time of centrifuging at various speeds. Scalped Linseed Sample 1.

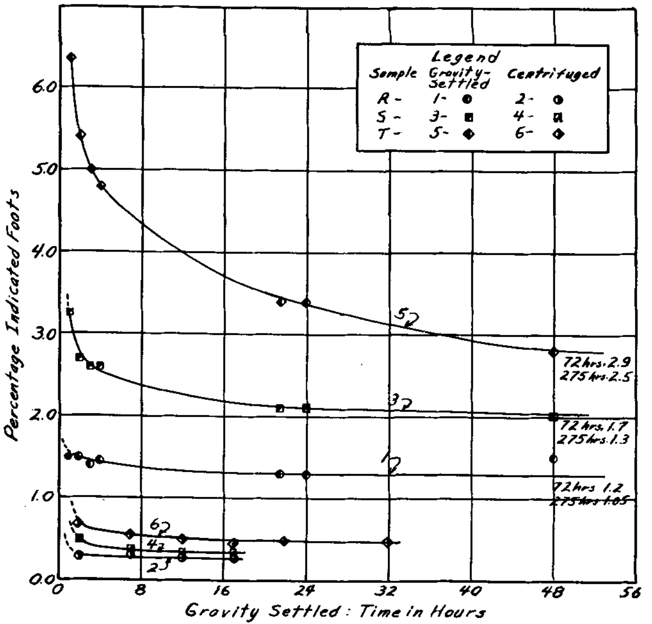

FIG. 3. Indicated percentage chilled foots test versus time of gravity settling and time of centrifuging at 1850 RPM. Refiltered Linseed Samples R, S, and T.

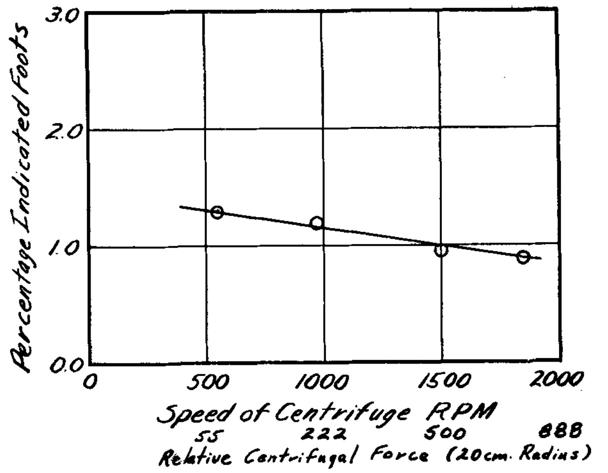

FIG. 4. Indicated heated foots test at ultimate compaction versus speed of centrifuging and relative centrifugal force for Scalped Linseed Sample 1.

In Figure 4 data derived from Figure 2 is plotted as ultimate percentage centrifugal foots against speed of centrifuging. For convenience the relative centrifugal force is shown on the same axis.

TABLE I

Effect of Intensity of Agitation on Percentage of Foots by the Chilled Gravity Method

Sample	Specified Agitation (Shaking in Tube)	Alternately Inverting Tube	Difference % Foots
1	3.6%	3.2	0.4
Recheck 1	4.4	3.2	1.2
H	4.0	3.4	0.6
I	3.4	3.1	0.3
J	3.7	3.2	0.5
K	4.8	3.6	1.2
U	4.0	2.8	1.2
V	4.0	3.6	0.4

Studies of the effect of intensity of agitation show that it has little if any effect on the results of the centrifugal test. Table I shows that intensity of agitation quite markedly and uniformly affects the results of the chilled gravity test. The more violent the agitation, the higher the result, an effect undoubtedly caused by occluded air bubbles, which may even be seen in the foots layer. Studies were made on the effect of intensity of agitation on the heated gravity test. Results were quite variable and indicated that the relative effect may depend upon the value of the result. This effect on both the heated and chilled gravity test has been studied by the A.S.T.M. Subcommittee II (D-1) on Drying Oils (2).

Table II shows the degree of correlation of the heated centrifugal test with the heated gravity test. The ratio of the gravity test to the centrifugal test (hereafter, Heated Ratio G/C) has been calculated for each oil and its average value shown together with the high and low results. This is done principally to enable the centrifugal test to be evaluated in terms of the gravity test; however its value falling in the neighborhood of 3 is of particular and significant practical interest. Actual processing losses associated with the removal of a given percentage of acetone foots lie in the same range (i.e., official gravity-settled acetone foots reduction divided by processing shrink = 3.5 as a rough average but may vary from 2.5 to 4.5). The latter ratio is of the same order of magnitude regardless of whether the acetone foots reduction is accomplished by complete or partial degumming (refining) or by filtering. The percentage of *centrifugal foots* is therefore directly a very useful indication of probable or expected processing loss incident to gum removal alone.

TABLE II

Correlation of Percentage of Gravity-Settled Foots With Percentage of Centrifugal Foots

	Ratio of Gravity-Settled to Centrifugal Foots	
	Heated Test	Chilled Test
	(Heated Ratio G/C)	*(Chilled Ratio G/C)*
Average Ratio	2.82	6.78
Low Value Ratio	2.25	3.28
High Value Ratio	3.67	9.6
Average Deviation Ratio	±0.26	±1.29
Number of Comparisons	21	16
Number Oils Compared	13	14

Table II also shows the degree of correlation of the chilled gravity test with the chilled centrifugal test. The ratio chilled gravity-settled foots to chilled centrifugal foots (Chilled Ratio G/C) is quite close to 7 as an average and varies through a wide range. The obvious explanation is that the foots layer read in the chilled gravity test is an indefinite and voluminous emulsion of acetone-oil and acid-calcium chloride solution with the actual footy and waxy material. This undoubtedly accounts for the lack of precision in the gravity test, as strikingly illustrated by the poor concordance of results on check samples given in Table IV.

If, as supposed, the chilled test is a measure of the quantity of saturated triglycerides and waxy materials in the oil a consideration of the ratio for the single scalped raw sample on which a chilled test was obtained is of interest. The Chilled Ratio G/C for this oil averaged 3.64, far lower than the values obtained for refiltered raw oils. It is thought that the presence of the larger quantity of acetone-insoluble material in scalped oil either acts to compact the foots layer separated (say, as a binder) or inhibits crystallization of the saturated components, i.e., the chilled gravity test for this oil should be much higher, giving a more normal G/C ratio, if it is a true comparative measure of saturated fats and waxes. This effect is being studied further and will be reported separately.

TABLE III

Effect of Temperature Prevailing During Settling on the Chilled Gravity Foots Test

Sample	Settling Temperature in °C.		Difference 27°C. as Base
	20-23°	27°	
B[a]	4.3	4.1	+0.2
P[a]	2.9	2.2	+0.7
1	5.2	3.6	+1.6
2	4.0	3.8	+0.2
3	7.4	7.8	−0.4
4	4.8	4.4	+0.4
5[a]	0.3	0.4	−0.1
6[a]	2.7	3.3	−0.6
7[a]	4.8	3.9	+0.9
8[a]	2.4	2.1	+0.3

[a] Values shown are averages of two duplicates checking within 0.2%.

Table III presents data obtained in a study made of the effect of temperature prevailing during the settling period on the chilled gravity foots test. The effect of reagent temperature was also studied and seems to be small when it falls in the official range, but the effect of the temperature of settling is not negligible on some oils. Temperatures outside of the official range may conceivably occur when the tubes are placed next to a window or to heating equipment in winter. Though the effect of temperature has not been specifically studied in its relation to the heated gravity test, it is not thought to be a large factor in low foots oil but may conceivably affect the results when the foots run around 3 to 4%, well outside the commercially significant range. In any event the effect of temperature may be ruled out of the centrifugal test as the time required to make the test is so short that the temperature may be controlled even by crude methods.

The effect of handling the tubes on the results of the gravity test is of interest. In a test made of this effect two oils tested 2.3% and 1.2% foots before handling whereas, after twirling the tubes between thumb and forefinger and settling an additional 10 minutes, the same tests were read as 1.6% and 0.9%, respectively. No amount of rough handling can decrease the centrifugal test result as the forces which have acted on the foots layer are greater than any which could be applied by the strength of man.

Table IV presents results on a series of check samples sent out to six different laboratories. All laboratories were known to be using the official A.S.T.M. Method D555-41. Clearly, the results show lack of agreement, especially on the chill test, but presumably represent the normal variation to be expected

TABLE IV

Comparison of Chilled and Heated Gravity-Settled Foots Test Results as Determined in Different Laboratories Reported to be Using the Official Methods

Laboratory	Chilled Foots, %				Heated Gravity Foots, %					
	Sample No.				Sample No.					
	3	4	5	6	1	6[c]	2	3	4	5
1	2.8	4.0	1.7	4.2[b]	0.8	0.7	0.6	0.8	0.5	0.65
2	2.2	7.0[a]	....	2.2	0.6	0.75	0.6	0.9	0.8	....
3	2.3	3.5	3.2	3.7	0.75	0.5	0.75	0.5	0.5	1.0
4	3.2	2.0	0.7	3.2	0.8	0.6	0.6	0.4	0.4	0.6
5[d]	....	....	1.12	2.0	1.2	1.4	0.8	....	....	0.4
6	2.4	3.2	1.6	3.2	0.6	0.6	0.4	0.8	0.5	0.5

[a] This laboratory reported a breakdown of heating plant during cold weather when this test was made, so room temperature was probably well below the official minimum.

[b] Average of ten tests.

[c] Same as Sample No. 1 but sent three months later.

[d] This is the only laboratory in the group which does not run foots tests routinely.

NOTE: On chilled foots Sample No. 3, three buyers' laboratories obtained values of 2.0, 2.0, and 2.4% and on Sample No. 4 a commercial laboratory obtained a value of 2.8%.

TABLE V

Comparison of Results of the Heated Centrifugal Test and the Heated Gravity Test on Two Samples of Raw Linseed oil, by Different Analysts

	% Foots by Centrifugal Test			% Foots by Gravity Test	
Analyst No.	1	2	3	1	4
Refiltered Q	0.4	0.4	0.4	1.0	0.8
	0.4	0.4	0.4	1.1	0.8
	0.4	0.4	0.45	1.2	1.2
	0.4	0.4	0.4	0.9	0.8
	0.28	0.4	0.4	1.2	0.8
Average	0.38	0.4	0.4	1.08	0.88
Scalped 8	0.8	0.92	0.88	2.4	1.8
	0.84	0.90	0.68	2.4	2.0
	0.80	0.90	0.84	2.0	2.0
	0.88	0.92	0.80	2.2	1.8
	0.88	1.1	0.80	2.3	1.8
Average	0.86	0.95	0.80	2.3	1.9

when different laboratories run the test, even with careful attention to detail.

Table V presents the results of the analysis by the centrifugal method and by the gravity method of a series of unknown samples by different analysts in our laboratory. Average differences between analysts using the centrifugal test method when multiplied by the Heated Ratio G/C are as good or better than the average differences between analysts using the gravity method.

While the reproducibility of results by the centrifugal method is somewhat disappointing when magnified several-fold through multiplying by the G/C ratio or when the precision is expressed as a percentage deviation from the test result, yet when the absolute magnitude of the deviations in terms of differences is the main consideration, the centrifugal method is obviously superior to the gravity separation foots method. A collaborative testing program involving several laboratories would be required to establish conclusively whether the centrifugal method really does represent a significant gain in reproducibility in practical commercial oil grading. Moreover it must not be overlooked that a new variable has been introduced, centrifugal force, which is not encountered in the gravity method.

In this connection a tachometer affording direct and easy reading of the speed was installed on our centrifuge only after completion of the experimental work. Subsequent checks of the speed made while routine tests were being run indicated that for a given rheostat setting the speed could vary from 1850 to 2100 RPM, probably as a result of line voltage variations. It is now believed that this uncontrolled speed variation may account for some (if not most) of the occasional puzzling discrepancies noted, and it seems reasonable to suppose that by controlling the RPM within ± 50 the concordance of results could be significantly improved over those reported here. This probably would require voltage control for certain motors.

Nevertheless as it stands, the centrifugal method does possess certain inherent advantages which would seem to justify its meriting serious consideration. These are:

a) The results are available in less than one hour whereas more than 24 hours must elapse before knowing the results of the gravity-settled test.
b) The magnitude of deviations or discrepancies, expressed as differences, is smaller.
c) The magnitude of the test results is such that it has real practical significance since it corresponds closely to the processing loss that would obtain in treating the oil to remove the corresponding quantity of gums, either by refining, washing, or filtering.
d) The test is independent of some of the variable conditions known to affect the results obtained by the gravity method.
e) The interfaces of the foots layer with the two liquid phases are sharp and parallel, affording accurate readings of the foots volume — a condition frequently not obtained in the gravity test.

Summary

A rapid method, requiring less than one hour to apply, has been developed for determining the acetone foots content of linseed oils using centrifugal force to compact the foots layer instead of the force of gravity. Application of the method requires the use of a centrifuge tube of novel design. Conditions known to affect the results obtained by the A.S.T.M. gravity test have been investigated as they apply to the centrifuge test and found to have little or no effect on the results of the latter test. Correlation of the percentage of centrifugal foots under stated conditions with percentage of foots by the gravity method has been determined for both the heated and chilled tests. Under the conditions chosen for the centrifugal test the magnitude of the heated test result is about the same as the processing shrink sustained when these foots are removed in practice.

The reproducibility of the method, based on this work, is equal to or better than the gravity test now in use for trading linseed oil.

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