A MODIFIED PROCEDURE FOR
DETERMINING AMOUNT OF LINT
ON COTTONSEED*

The Development of More Rapid and More Accurate Technique

By EGBERT FREYER
South Texas Cotton Oil Company, Houston, Tex.

RECENT increase in the value of cottonseed linters has made it desirable to know more about the quantity of this material which the oil mill operator receives with his seed and the quantity that goes out with his hulls. In the absence of any analysis, the only figures available for linters are the actual amounts cut off from day to day. By weighing we determine what we get, but only by analysis plus weighing can we determine what we lose. Thus, chemical control affords a knowledge of what is available, the percentage of the available quantity recovered in yield, and the percentage lost.

Someone has said that we advance scientific knowledge in proportion to the extent that we learn to measure things. Chemistry was but a hit-or-miss, trial and error, test tube science before the development of the analytical balance by Lavoisier in the last years of the eighteenth century; while during the half century that followed it grew into something not far removed in the grandeur of its laws from the science we know today, and call *modern* chemistry. Much of this development was due to the balance. Testing methods and the instruments involved in their use make the difference between *quantitative* knowledge and *qualitative* knowledge; and where industrial processes depend upon such knowledge, this difference may often represent the difference between profit and loss.

Most industrial testing methods represent a compromise between speed, simplicity of manipulation, and accuracy. It is only in rare cases that extreme precision is required; so that in general, once the maximum limits of error are established, effort is concentrated on shortening the tests and making them easier. For in this way money can be saved.

When it was decided to study lint cutting in the writer's laboratory, a rapid method of estimation was required owing to the volume of work we found it necessary to handle. Our official method for lint on hulls was tried and found to require too much time, and not to afford the accuracy desired. In this method, triplicate or more tests are made on 5 gm. portions of predried hulls, removal of lint accomplished by treatment with concentrated sulphuric acid, which is washed out; and the delinted hulls are again dried. The average of the two determinations agreeing within 1 per cent is reported. This method was found to be much more easily applied to cottonseed, principally because the hull particles, especially fragments, are far more difficult to handle and transfer due to surface tension. After making a careful study of this method with especial reference to the conditions concerned in the possible errors, and making every effort to shorten it, a modification was adopted which the writer considers to be more accurate than the prototype. Certainly it requires much less time.

FACTORS AFFECTING THE RESULTS

In investigating the method the following variables had to be considered as being potential sources of error:

(1) Size of sample.
(2) The strength of the sulphuric acid.
(3) The temperature of the acid.
(4) The time of treatment with acid.
(5) The moisture content of the seed.
  (a) As affecting the strength of the acid.
  (b) As affecting the temperature of the acid.
(6) The condition of the drying.

Obviously a large sample is likely to be more representative than a small one; so 15 grams is the weight used in this modification. Weighings need only be made to the nearest 0.01 gram, which circumstance makes for speed; furthermore, the error resulting from accidental loss of small particles is reduced.

The errors associated with (2), (3), (4) and (5) will be due to two effects:

(1) Leaving more or less lint on the hulls.

(2) Dissolving or tearing off of more or less hull bran material.

These two errors tend to balance each other, and by checking the method against a mill's production, the conditions may be found under which this mutual compensation is complete.

Finally, the time consumed by predrying the seed may be saved by making a moisture determination along with the lint test, and treating the original seed with acid. To convert the percentage of dry lint thus determined to the percentage of lint containing the original moisture it is necessary to add 10 per cent of its value; no serious error accrues if the moisture content differs from 10 per cent by no more than 2 or 3 per cent. The large sample and the simplicity of the moisture test justifies in this work, at least for ordinary purposes, making the moisture determination in singles.

EXPERIMENTS MADE TO STUDY THE VARIABLES OF THE TEST

The following experiments were made to study the influences discussed above and to ascertain just how rigidly the conditions and procedure finally adopted would have to be followed to secure accurate results, and to learn what errors would result from deviations from them:

DRYING OVEN CONDITIONS

Though undoubtedly more ac-

---

*Paper presented as a part of the Report of the Crude Mill Operations Committee, Memphis, Tenn., May 23-24, 1935.

curate results would obtain by the use of an oven temperature of 101-105° C., the time saving effected by going to a higher temperature, i.e., 130° C., especially in a forced draft oven, caused the writer to adopt the latter condition. He felt justified in doing so by the following considerations:

(1) The final results depend upon the *difference* between two weight losses rather than upon a single loss. That is (moisture + lint) — (moisture) = dry lint. Therefore, so long as both portions are thoroughly freed of moisture, any additional slight loss of weight by decomposition ought to be the same in both cases, and consequently not appear in the difference. Figure I shows the indicated % H_2O, % H_2O + % lint, and % lint as a function of time in the oven. The moisture curves show a tendency to flatten as the last traces of water leave, followed by a continued rise, which indicates the beginning of thermal decomposition. Three hours was chosen as the time for the test. Below are given the indicated percentages of lint for three different periods in the oven.

Time in Oven	% Lint
2.0 hrs.	4.35
2.5	4.40
3.0	4.45

(2) The second reason why the advantages of using high oven temperature are justifiable is that the error accruing, as indicated in the above figures, is small compared with the other chief source of error in the test; that is, variations of actual lint content in different portions of one sample.

EFFECT OF THE CONCENTRATION OF THE SULPHURIC ACID

Effect of Predrying the Seed

To determine if the naturally occurring moisture of the seed has any effect on the results four samples were tested in two ways: (1) Predrying, delinting, etc., and (2) Acid-treating the original seed and making separate moisture determination. The results follow:

% INDICATED LINT
(Dry Lint)

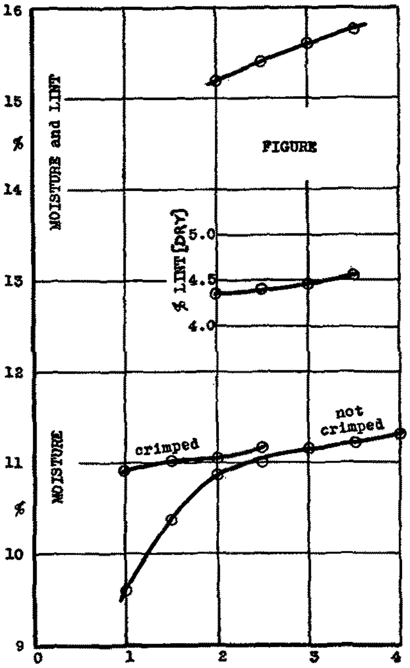
Predried 3 hrs. @ 130° C. in Forced Draft Oven	Seed Treated as Received	Difference
4.5%	4.2%	0.3
6.3	5.9	0.4
7.4	6.6	0.8
7.7	7.6	0.1

(Basis Original Moisture Content)

The differences are relatively large and illustrate the necessity of standardizing on conditions. Which method gives correct results can be determined with a check by a combination of analysis and lint production figures of some oil mill.

EFFECT OF TIME OF ACID TREATMENT

This is indicated in the following experiment in which, after delinting a portion of seed according to the method adopted as standard, the dried denuded seed were subjected to subsequent acid treatments, followed by washing and drying. In each case appreciable additional loss of weight occurred corresponding to the following apparent "lint" contents of the completely denuded seed.



Hours in Forced Draft Oven at 130° C.

Treatment	Second·	Second	Third
Time of	30 sec.	15	15
% weight loss ..	0.85%	0.6%	1.0

On the average, therefore, for every five seconds' error in the timing of the acid contacting period, an error of 0.2 per cent lint content will appear in the result. Since this figure will be somewhat smaller in the case of seed containing some moisture, in accordance with the preceding experiment, this source of error becomes negligible providing the acid contracting period be timed to within two or three seconds, which is not difficult to do.

EFFECT OF THE TEMPERATURE OF THE SULPHURIC ACID

Portions of a sample of seed were delinted with acid at different temperatures. The results follow,

Temperature	15° C.	30° C.
Indicated % lint	5.6%	6.0%

It should be mentioned that all of these experimental test data are the average of two or more determinations. No serious error will result from ordinary variations of room temperature. Taking 25° C. as standard, 0.2 per cent indicated lint content should be the limit of error from this cause.

The moisture content of the seed may be expected to have some influence on the temperature of treatment due to the strong evolution of heat which occurs when sulphuric acid dissolves water. However, since the moisture dilutes the acid with a tendency towards low results, whereas the heating tends to give high values, the two influences should balance one another, at least partially. A test was made to ascertain the effect of moisture content on the temperature rise. A sample was divided into four 15 gram portions, one of which was left unchanged; another was partially dried; a third, completely dried; to the fourth was added water in such amount as to add 8.5 per cent to the original moisture content. Each portion was treated with acid for 45 sec. and the temperature noted.

% moisture..	0	2.0	8.0	16.5%
Temp. rise ..	6.1	6.1	5.9°	13.8° C.

As the wetted portion was not allowed to stand until the added moisture had been absorbed into the seed and reached a state of equilibrium, the rise indicated represents an extreme; and in view of the absence of any effect of naturally occurring moisture on the temperature, we may dismiss this temperature effect from consideration insofar as it is due to the natural moisture of the seed. It is still not clear, however, to what extent varying moisture contents may affect the results by virtue of the diluting or other inhibiting effect.

TIME FOR WASHING THE DENUDED SEED FREE OF ACID

Using a water flow of 3 liters per minute and the washing apparatus described below, it was shown that 45 to 60 sec. is sufficient time to wash out all of the sulphuric acid, at least for the purpose of this test. Four portions of a sample were treated, then washed four different times, with the following results,

Liters
Rate of flow: 3 1/min.

Time of washing (min.)	0.5	1.0	2.0	3.0 min.
% indicated lint	8.2	7.6	7.7	7.6%

Failure to remove all the sulphuric acid causes this to be weighted, resulting in a high value.

PROCEDURE AND FORM OF APPARATUS ADOPTED

The Method: The net result of the foregoing experiments was the adoption of the conditions named in the following statement of the method: Weigh three 15 gram (± 0.01) portions of the sample and place two of them in 150 cc. beakers for duplicate delinting treatments. The third portion is placed in a moisture dish. Pour 40 cc. of concentrated sulphuric acid (98.5 per cent) onto the seed in the beaker and submerge immediately by stirring vigorously with a bent end glass rod, using an up and down as well as a rotating motion. At the end of 45 sec. pour the mixture into 500 cc. of water and pour this immediately into a perforated washing cup standing under the flowing tap. When free of acid, drain off the excess water, transfer to a moisture dish, and dry, with the portion of original seed, for three hours in a forced draft oven at 130° C., or in any other type of oven for a period sufficient to remove all the moisture. After cooling, weigh. The lint content is determined by the difference between the loss in weight of the acid treated sample and of the untreated sample.

Reproducibility of Results

Since different small portions of a sample, especially of mill delinted seed, may vary appreciably in lint content, bad checks are occasionally obtained, but about 90 per cent of the tests show agreement between duplicates within 0.3 per cent indicated lint. When duplicates do not agree to at least 0.5 per cent, the determination is repeated. A table is given showing the kind of agreement that might be expected. The figures were taken at random from the routine note book.

It might be remarked that the figures given for raw seed practically cover the entire range of lint contents determined for cottonseed collected from various sources in southeastern and southern Texas.

Delinted Seed % Indicated Lint (Dry)		Raw Seed % Indicated Lint (Dry)		
6.4%	6.7%	9.1%	9.4%	
6.0	7.4 n.g.	10.4	10.4	10.6
6.0	6.0	10.1	10.1	10.2
4.7	4.7	11.0	11.1	11.2
4.9	5.0	10.0	9.9	(10.6)
4.7	8.2 n.g.	10.4	10.4	10.5
4.9	5.0	11.3	11.0	10.9
4.8	4.9	11.1	11.1	11.0
4.8	4.9	9.7	9.4	9.3
5.2	5.3	8.8	8.8	8.6
6.4	6.6			

Note: Parenthesis indicates results not averaged. Brackets enclose results of two separate duplicate tests, one of which failed to check.

NOTES OF THE APPARATUS AND PROCEDURE

For handling a large volume of work, for example, shift samples from five or six oil mills, the following devices are used:

(1) A large, tared weighing scoop in which the denuded, dried seed are weighed, as well as, of course, the original 15 gram samples.

(2) Two washing cups; for, while one portion is being acid treated another can be washed. These cups should be about 3 in. in diameter and 3 in. in height, and perforated freely around the sides to within an inch of the bottom so that the copious flow of water will pass through. The moisture dishes* should be slightly larger in diameter than the washing cups so that the washed seed may be transferred quickly by inverting the former over the latter; then reversing and slamming down on the table. The water that will not drain off need not be removed with a cloth if a forced draft oven at 130° C. is used for drying.

The weighing is done all at one time; then all tests are acid treated. This requires a 150 cc. beaker for each test. An acid dispensing burette is a very convenient accessory. For starting the acid-contacting time sharply, the acid is measured into a 100 cc. beaker from which it is rather dumped than poured onto the seed. It is advisable to wear a rubber apron, although acid burns on the hands are rare. By overlapping the treating and washing times one man can treat tests on an average about a test every one and a half minutes, whereas with a helper to draw acid and make the transfers, one test a minute can be treated, washed, and made ready for the oven. This is the minimum safe washing time. A very simple timing device is made by blowing a hole in the bottom of a flask and adjusting its size so that the overflowing flask drains completely in 45 sec. The flask is clamped in such a position that it may be swung under a tap flowing at a rate somewhat greater than the rate of flow from the hole. It is normally kept under this stream, and therefore overflowing. At the start of a test, as the acid is poured onto the seed with one hand the flask is swung out from under the tap with the other, and the acid treatment is ended when the flask is empty.

SUMMARY

The American Oil Chemists Society's method for estimating lint on cottonseed hulls has been modified for application to cottonseed. The details of the method have been studied with especial reference to the effect of the several variables on the accuracy of the results, and of saving time in the procedure.

Apparatus is suggested for the handling of samples in quantity without undue consumption of time, and data are presented which show that the modification is about as accurate, in the case of the whole seed, as the seed moisture determination; whereas in the case of mill delinted seed, duplicate results should agree within 0.3 per cent lint, except occasionally wider deviations occur, perhaps as much as 1.0 per cent difference, which are presumably due to variations in the sample.

DISCUSSIONS

Mr. Cox: To clear up one point, did you say the difference between the loss of moisture and the loss of moisture and lint is lint?

Mr. Freyer: That is the way we figured.

Mr. Cox: How about the moisture that was in the lint originally, which you are now figuring as dry lint?

Mr. Freyer: That would cause a slight error.

Mr. Cox: About ten per cent?

Mr. Freyer: The accuracy of that method is not much better than ten per cent.

Mr. Malloy: We have become interested in this question of the lint content. Particularly you will find that we have greater differences in the residual lint content of the cottonseed in some sections of the

---

*The writer uses the friction covers from cans designed for soap paste.

country than in others. We have been able to get some very good checks by taking fifty-gram samples and first making a moisture determination, then fuming with hydrochloric acid at 130 for one hour, putting them in the ten-mesh sieve and rubbing them with a large rubber stopper. Then make a final polish by putting the seed on a piece of cloth. Then we have determined the moisture content of the lint that is taken off a number of samples and got an average. Using that average, and our original moisture, we have been able to calculate the percentage of lint. We have made some very good checks. We believe following so closely the method used in the analysis of seed will make a better series of determinations than using the acid, which has its dangers and is somewhat more expensive.

SOME NOTES ON THE THEORY AND PRACTICE OF BLEACHING FATTY OILS*

By HENRY ODEEN and H. D. SLOSSON
Filtrol Company of California

THE purification of oils by the removal of many impurities occurring as finely divided suspensions, colloidal bodies and soluble matter has brought into wide use such materials as carbon, clay and silicia in a variety of form and quality. The displacement of colloids and solutes from the oil to the surface of the solid adsorbent particle, to be held there tenaciously, is termed adsorption.

ADSORPTION THEORIES

The removal of such colloidal and soluble impurities from oil as, for example, mucilaginous and coloring matter, is, in the practice of the art, a relatively simple operation, but underlying this simple performance, science has found physico-chemical law and theory to explain the behavior and mathematically to measure the interplay of surface energy at work. One of our earliest references to the scientific study of adsorption is Scheele's research in 1777 on the adsorption of gas on charcoal. Willard Gibbs, eminent American scientist of his time, promulgated the adsorption theorem in 1876 and later H. Freundlich in 1909 published his work on colloidal chemistry giving mathematical expression for the measurement of the amount of adsorption at constant temperature.

For our purpose the theory may be stated that: solutes which lower the surface tension of the solvent will be concentrated at the liquid-solid interface. The theory may be demonstrated as follows:

(1) Assume a pure, colorless oil. Within the body of this oil the molecules have neighbors above, below and on all sides to attract each other. But the molecules in the top or surface layer cannot have this molecular pull upward; they do have an extra attraction downward into the mass of the oil molecules. This inward pull makes the surface act like a stretched membrane or elastic skin, contracting when it can. There is potential energy or ability to do work resting in this surface tension which is trying to expend itself.

(2) Assume the same oil with coloring matter present. The color bodies are larger in size than the oil molecules and while not as numerous, yet they do disturb the lines of attraction which previously held the oil molecules so closely together. If we measure the surface tension now we will find it less than it is in the case with pure oil as in (1) above. We have now come to the meaning of "solutes which decrease the surface tension" for the color bodies are the solutes (dissolved substances).

(3) Assume that adsorbent material has been added to oil containing coloring matter. For conveience let us take only a small portion of this oil wherein we have many oil molecules, a few color bodies and one adsorbent particle.

The main thing to note is that there is another kind of surface aside from the top surface of the oil. Whenever the oil is displaced to make room for the adsorbent particle, including any penetration into its porous structure, there we have other free surface which, in the aggregate, is very large. This surface is called "liquid-solid interface." Probably the first thing which happens on the addition of the adsorbent particle is a momentary change in the surface tension of the surrounding oil—there is a lowering at the particle surface, accompanied by an equivalent increase of intermolecular forces in the adjacent layers. Adjustment of these forces to equilibrium causes a counter-flow back to the adsorbent surface.

(4) Apply heat and agitation to the oil. The color bodies are now presumed to be concentrated at the liquid-solid interface. The agitation has brought the color bodies into intimate contact with the adsorbent particles permitting their concentration at this point.

These interfering color bodies have now been removed so that they can no longer break the intermolecular force of cohesion; accordingly the surface tension again increases. Again the oil molecules, relieved of this interference to some extent, exert their natural, mutual cohesion to each other.

Freundlich's absorption isotherm concerns changes which take place when the amount of adsorbent is progressively increased but the temperature is held constant. The essential formula is:

$$X/M = K\ C^n$$

Where X is amount of solute adsorbed
M is amount of adsorbent used
C is concentration of unadsorbed solute
K and n are constants

---

*Presented at the Twenty-sixth Annual Meeting of the American Oil Chemists' Society at Memphis, Tennessee, May 23-24, 1935.