# An Improved Laboratory Method for Residual Linters on Cottonseed

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The A.O.C.S. Official Method Aa 7-44 is defined as a method to determine the residual lint (cellulose fiber) on the seed and is applicable to cottonseed. It is an adaptation or modification of A.O.C.S. Official Methods Aa 4-38 and Aa 5-38, which were originally developed in the Barrow-Agee Laboratories by C. H. Cox, then chief chemist. The method for lint determination, during a number of years of use, has proved to be reasonably satisfactory and reproducible but does not lend itself well to the making of a large number of lint determinations in a commercial or plant laboratory within a short period of time.

When it seemed probable that the percentage of residual lint on cottonseed after completion of the ginning process might be made a factor in the grading of cottonseed under the supervision of the Agricultural Marketing Service, U. S. Department of Agriculture, there was begun in the Barrow-Agee Laboratories a study of the official method for the purpose of improving or modifying this method, or developing a new one which would be more suitable for analyzing a large volume of samples.

The basic principles of the official method, that is, hydrolysis by means of heat and moist hydrochloric gas followed by mechanical brushing, were regarded as sound, and efforts were directed to improving the application of these principles.

At the beginning of the study attention was directed toward the application of infrared heat, because of its instantaneous radiation, as a means of replacing the slower convection type heat. Upon evidence of the feasibility of using infrared heat, a program was initiated to determine the variables and the limitations within which those variables could be expected to give accurate and reproducible results.

Consideration was given to the time, oven air temperature, and heat intensity, which combines lamp wattage and proximity to the sample. Many variations and combinations of these factors were attempted before establishing the optimum conditions suitable for the procedure. This phase of the study resulted in the selection of 125-watt lamps placed in opposing directions, each 6 in. from, and focussed to bear directly upon the sample. Oven air temperature was maintained at 118° ± 3° C. by a thermoregulator.

Because of the high transmission factor of glass toward infrared rays, heat-resistant glass dishes, 5 in. inside diameter, were selected as the container best suited for drying the sample. This particular diameter is relatively equivalent to the diameter of the lamp beam and also permits distribution of the sample to a thin depth for direct contact with the infrared rays for uniform drying.

It was noted, earlier in the study, that the lint is more readily hydrolyzed when the moisture in the seed is between 3%–9% at the beginning of the fuming phase. This fact tends to eliminate the necessity for drying low moisture seed. However the impracticability of segregating seed samples of low moisture from those of high moisture while handling large numbers is obvious. Recognizing the desirability of a procedure which would be applicable to extreme moisture percentages, a condition was sought whereby seed of low moisture content could be dried along with seed of high moisture content.

For the purpose of this test a 200-lb. sample, containing 5.8% moisture, was obtained. A portion of the sample was dampened and conditioned for several days to bring the moisture up to 30%. A series of tests were made in which half the samples, in the oven at any given time, were of the low moisture (5.8%) and half were of the high moisture (30%). It became apparent that, in order to dry the higher moisture samples sufficiently, the lower moisture samples would then become too dry to be hydrolyzed in the fuming operation. This effort was rewarding however when it was disclosed, through moisture graphs, that, in intermittent infrared drying by maintaining an air temperature of 118° ± 3°C., moisture reduction is approximately 50% at the end of 15 min. regardless of the original moisture percentage.

Since the general average moisture in seed is approximately 12%, a reduction of drying of 50% or near the 6% level, places the average seed moisture midway of the optimum range for hydrolyzing. It is obvious therefore that seed with moisture content from 6% to 18% may be expected to hydrolyze properly if 50% moisture reduction occurs during drying.

But for occasional exceptions the moisture percentages of the samples, received in a laboratory from a given area during a given period, remain well within an 8% range. A reduction of 50% in the moisture of each of the several samples would therefore reduce the range by 50%. Seed with moisture higher than 18% will necessarily have to be dried for a longer period. An additional 15-min. drying period has been found to be sufficient even for extremely high moisture (30%) and is not detrimental to seed with moisture above 15%. Upon this basis it was noted that the drying time could be adjusted in proportion to the moisture range of the seed being analyzed. However since the large majority of samples average well below 18% moisture, the time of 15 min., being suitable for such moisture, was selected as the most desirable.

The earlier study of the fuming phase, in which the lint becomes hydrolyzed by the hydrochloric acid gas, revealed that the official pots which had been previously used, did not lend themselves well toward a uniform distribution of heat, primarily because of their cylindrical shape. The supplier was then submitted a modified design which took into account the normal width of the infrared lamp beam. The new pot measured approximately 5½ in. in diameter and 1¼ in. in depth and was equipped with a flat lid, similar to the regular lid, but of the same diameter as the new pot.

Upon putting these into use, it was soon discovered that the white surfaces, being highly reflective to the infrared rays, would have to be colored in order to increase the heat absorption sufficient to vaporize the acid absorbed therein. Many variations of colors were used, including black. The dark colors possessed such a high absorption rate that the lint on the seed nearest the surfaces appeared scorched. In order to have a color of the proper absorption quality, suitable for continual handling without severe color change, tan or light brown was selected for the outer surfaces of the pot and lid.

Many determinations were made with the use of the modified pots, and results were highly satisfactory. Although the time involved in making lint determinations had been substantially reduced by this improved procedure, it was felt that improvement in technique was desirable. This thought resulted in elimination of the earthenware pot altogether by allowing the seed to remain in the glass dishes used for drying. It was discovered that to absorb the acid into the inner surface of the lid when placed over the dish, the lint would become hydrolyzed with equal ease, provided no direct heat was applied from below.

The intermittent heat application from above is adequate to vaporize quickly the acid absorbed within the lid. The oven temperature, being controlled by a thermoregulator, is sufficient to prevent condensation within the dish. Upon completion of the required time, removal of the lids also removes the source of the acid, an action which prevents unnecessary contact of the seed with any remaining acid during cooling. Moreover this modification eliminates the necessity of transferring the seed from one type of container to another, reducing time, labor, and the possibility of spillage.

In view of the abnormal quantity of hydrochloric acid fumes dispelled from the oven, it was thought that a reduction of acid might be possible. There was then begun a progressive reduction of the quantity of acid used for fuming. Mechanically delinted seed and seed of low lint percentages responded satisfactorily to 1 ml. of acid. Some difficulty was encountered with seed of more than 11% lint. After several series of such tests it was decided to use a dilute hydrochloric acid. This resulted in trial of 1:10, 1:5, 1:2, and 1:1 solutions. In most cases the weaker solutions gave acceptable brushing. The 1:1 solution, made by adding one volume of concentrated hydrochloric acid to an equal volume of distilled water, gave best all-around results, without detrimental effest to the hull of the seed, if 2 ml. were used. The amount was reduced to 1 ml. for mechanically delinted seed.

THE percentage of moisture in the lint after removal from the seed is relatively constant regardless of the percentage moisture in the original seed and will be found to be approximately 2%. A variation of 1% moisture in the lint affects the lint percentage by about .1%.

The method, resulting from this study, is as follows:

A. APPARATUS

1. The infrared oven is of double-wall construction, insulated with 1-in. rock wool or fiber glass between the inner and outer wall. The inner wall should be of a highly reflective metal, such as aluminum or preferably stainless steel. The same material may also be used for the outer casing. The width and depth of oven are contingent upon the number of samples to be treated at one time.

Upper and lower heating banks, composed of 125-watt, 110–115-volt infrared lamps spaced 6 in–7 in. on centers, are opposite in direction and measure 13 in. between the convex surfaces of the opposing lamps.

An expanded metal tray, located 6 in. from the convex surface of the lower lamps, 7 in. from the upper lamps, is supported by channel brackets on both sides. The tray should be marked for the placement of dishes directly between the opposing lamps.

Oven temperature is maintained at 118° ± 3°C., by a thermoregulator located just below the tray and outside the direct lamp beam.

An exhaust opening of 1½-in. tubing is located in the top center of the oven and is discharged into an exhaust system for removal of fumes. Six ⅜-in. holes should be in the bottom of oven to permit passage of air.

The door, on the front of the oven, may be hinged at the top and retained in either open or closed position by tension springs or any other suitable arrangement. Two switches should be installed, the first of which to control both banks of lamps and the second to control only the lower bank of lamps. An interval timing-device may be wired into the circuit or attached to a signal system for convenience.

2. Heat-resistant glass dishes, 1-in. depth, 6 in-diameter (Pyrex No. 206 or equivalent).

3. Dish covers, unglazed porous earthenware, tan or light brown color on top side (Niloak Pottery, Little Rock, Ark.).

4. Aluminum moisture dishes, 30-gauge, 2 x ¾ in. (*ca.* 50 x 19 mm.) with tight-fitting slip-over covers.

5. Mechanical brushing machine equipped with Tyler 35-mesh (U. S. No. 40) wire screen.

B. REAGENTS

1. A diluted hydrochloric acid solution made by adding 1 vol. of concentrated reagent hydrochloric acid (37.0%–38% HCl) to an equal volume of distilled water.

C. PROCEDURE

1. Use a well mixed sample free of foreign matter.

2. Weigh 50 g. (to nearest ± .1 g.) of sample into glass dish, distribute evenly, and dry, uncovered, for 15 min. at 118° ± 3°C., in the oven, previously preheated to temperature. Dishes should be placed so that heat rays bear directly on samples. Samples containing more than 15% moisture shall be dried an additional 15 min.

3. Toward the end of the drying period, absorb into the inner side of the clay cover, 2 ml. of the diluted solution of HCl (B 1), (1 ml. for mechanically delinted seed).

4. At the expiration of the drying period place the cover on the glass dish with the treated side toward the seed. Heat for 20 min. at 118° ± 3°C., using top lamps only. (The oven may be the same as the one used for drying,)

5. Remove and cool to room temperature without desiccation.

6. Weigh seed, recording weight as A.

7. Remove lint, by means of a mechanical brushing machine.

8. Weigh lint-free seed, recording weight as B.

9. Determine moisture in average lint removed at intervals, recording as C.

D. CALCULATION

The residual lint, that is, the lint remaining on the original sample of seed, is conventionally calculated to an 8% moisture basis.

Therefore when 50-g. sample is used

$$\text{Percentage lint} = 2(A - B) \times \frac{100 - C}{92}$$

## Summary

The several advantages of the infrared method over the official method Aa 7-44 are as follows:

1. In the infrared method an error in the original weight of the seed is reflected only as that percentage lint of the errata from the 50 g. In the present official method such an error is doubled in the final percentage.
2. An error in the original moisture determination is reflected as an equal error of lint percentage when the official method is used. In the infrared method the original moisture percentage does not enter into the calculation, and therefore such an error is not reflected.
3. A single oven for the infrared method, as compared to three required for the official method (one drying, one fuming, and one overnight drying).
4. Power consumption is substantially less as a result of the reduced total oven time of from 18 or 20 hrs. to 35 min. Actual cost varies with KWH rates. Duplicate determinations may be expected to use about .1 KWH.
5. Reduction of HCl by 50%, which also results in less breakage of hull during brushing.
6. Reduced cost of containers required for the determinations.

THE basic procedure was presented to the subcommittee on residual linters for study in the early months of 1954. During the months that followed many determinations were made through the collaborative effort of the subcommittee. In addition, some of the individual laboratories accumulated data comparing results of the infrared method to those of the official method. A typical portion of the data thus obtained was reported to the subcommittee for tabulation and evaluation.

The close agreement obtained between duplicate determinations of many thousands of samples and the reproducibility of the infrared method warrants adoption as an official method.

NOTE: Preliminary study leads to the belief that it is possible to substitute the infrared method of treatment reported here for the present pre-drying and lint hydrolysis procedure of the A.O.C.S. Official Methods (Aa 4-38 and Aa 5-38) for the analysis of cottonseed for oil and ammonia (N equivalent).

Further investigation of this possibility is being carried on in these laboratories. If successful, it will be the first important improvement and change in cottonseed analysis since the present official method was announced in 1928.

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# Effect of Heat on the Color of Trichloroethylene-Extracted Cottonseed Oil

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PREVIOUS WORK in this laboratory (5) has shown that the color of trichloroethylene-extracted cottonseed oil extracted and desolventized in the pilot plant equipment under conditions producing a very satisfactory soybean oil and refined by the ordinary refining method is too dark to be acceptable. Duncan (2) has reported that a prime oil can be produced by extraction with trichloroethylene but gave no operating conditions. Vix, Pollard, Spadaro, and Gastrock (6), after a systematic study of the effect of heat on hexane-cottonseed oil miscellas, reported that color fixation became objectionable between 150° and 180°F. and that beyond 180°F. it increased rapidly. The present studies were for the purpose of obtaining similar data for trichloroethylene-cottonseed oil miscellas to determine whether the effect of heat is similar with the two solvents, that is, whether it is primarily a heat effect.

## Effect of Desolventization Temperature on the Color of the Oil

*The Miscella.* Prime delinted cottonseed were dehulled, flaked, and extracted with trichloroethylene at room temperature in the countercurrent extractor described by Arnold and P'Pool (1) to produce the miscella.

*Desolventizing the Miscella.* The miscella was desolventized in the equipment, shown in the flow sheet in Figure 1, consisting essentially of a climbing film evaporator and a packed steam-stripping column, together with the necessary auxiliary equipment. The evaporator consisted of a 3/8-in. pipe, 6 ft. long, heated by an electric-resistance wire-winding controlled by a variable transformer. This evaporator discharged into a flash chamber, from which the hot concentrated miscella flowed by gravity through a pre-heater into the stripping column.

This stripping column was a 2-in. glass pipe packed with a 4-ft. bed of 1/2-in. Berl saddles and heated by electric-resistance wire. Stripping steam was generated from distilled water in a steam-jacketed pipe still and superheated in an electrically heated pipe. The equipment was operated under reduced pressure produced by a water jet ejector and controlled by air bleed-in valves.

*Results.* Batches of the miscella were desolventized at four temperature ranges: 150°–160°F.; 170°–180°