

A METHOD FOR THE ANALYSIS OF COTTON SEED

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The perfection of a method for the analysis of cotton seed that will accurately represent the value of a given lot of seed in terms of pounds of cotton seed oil and cake that can be produced, has been the subject of a large amount of work by many analysts.

Several methods have been, and still are in use with varying degrees of success.

The method of weighing a portion of seed, cutting by hand, then picking out the meats for analysis and reweighing the picked hulls, to determine the percentage of meats and hulls, has probably been in use the longest and by the greatest number of operators. This method, however, is subject to numerous errors, probably the greatest being in getting the correct percentage of meats and hulls. Careless picking and a change in the moisture content of the hulls while being worked, are two factors very difficult to control. It is also obviously impossible to work on a sample of sufficient size to insure accuracy.

The various methods of extracting a weighed sample of whole seed after grinding or crushing are subject to the same error—the small size of the sample—a few more or less than the average number of immature seed giving erroneous results.

The solution of the problem seemed to be in some method by which a sample of seed, large enough to be representative, could be ground to a homogeneous mixture. The analysis of such a mixture would be comparatively simple.

Every analyst who has worked with cotton seed is familiar with the fact that the lint adhering to seed makes it impossible to grind them to a homogeneous mixture, for the lint, the hull and the kernel separate and no amount of turning, shaking or rolling will remix them well enough to allow of representative samples being weighed from the mass.

Several years ago Malowan, of Houston, Texas, proposed to treat seed with hydrochloric acid, dry and then grind for analysis; the acid attacking the lint and so changing its character that after partial drying the seed can easily be ground and mixed to a product entirely suitable for analysis.

The idea was entirely practical, but the difficulty in this procedure lay only with the method of applying the acid. Too much acid or too close contact invariably charred a portion of the sample.

The problem of finding a method of bringing the gaseous acid in contact with the lint on the seed was attacked from a number of angles. Seed were placed in several different types of containers over an aqueous

solution of hydrochloric acid, and the acid volatilized by heating. In every instance it was found impossible to treat the seed evenly throughout, some getting too much acid and some too little, and where the acid in too concentrated form came in contact with the seed charring always took place. Passing the gas from an outside generator into a vessel containing the seed was tried, but is subject to the objection that it is hardly adaptable to a large number of seed samples, and further, it is wasteful, in that it is difficult to regulate the amount of acid to varying numbers of samples; it being necessary to obtain a sufficient concentration of gas in the vessel containing the samples regardless of the number of samples contained.

After some work we decided that if the acid were absorbed into the walls of a porous vessel filled with seed and then this vessel were heated slowly, the acid would be liberated in such a manner that all of the seed would be acted upon in about the same degree.

The absorbent vessel finally selected is an ordinary two and a half inch flower pot—the kind used by florists for small plants. They readily absorb the acid, hold fifty to sixty grams of cotton seed, and a large number can be packed at one time into a suitable oven. They are easily obtainable and inexpensive, so that when they cease to absorb the acid through glazing of the surface, they can be replaced.

From 1.0 to 1.5 cc of concentrated Hydrochloric acid are allowed to run down the sides of the pot from a pipette or burette and the pot turned about until it has all been absorbed. It is then loosely filled with fifty to sixty grams of seed and placed in a ventilated air oven and heated to 115 deg. to 125 deg. C from one to one and a half hours, depending on the moisture content of the seed. This treatment leaves the seed with a moisture content of from 3 per cent to 8 per cent and in such condition that they can be readily ground and mixed without danger of subsequent separation of the hulls and lint from the meat. The usual quantities are then weighed out for moisture, oil and ammonia and the moisture and ammonia are determined exactly as in a cotton seed meal. The oil determination requires a longer extraction and unless the grinding is very well done—so that everything will pass a 30 mesh screen—the partially extracted material must be taken down at the end of two hours, ground in a porcelain mortar and re-extracted for two hours more. If the seed are well dried and ground, however, a rapid percolating extraction will get practically all the oil in four hours.

A moisture determination is made on the original seed by drying at least eight hours at 100 deg. to 102 deg. C and the determinations made on the partially dried seed are reduced to the original moisture basis as follows:

M = Moisture in original seed

P = Moisture in partially dried seed

F = Factor to use to reduce to original basis

$$(100 - M \div (100 - P)) = F.$$

These calculations are easily made on a twenty-inch slide rule with sufficient accuracy. In reporting the figures to the oil mill or seed shipper, we report available ammonia, this figure being obtained by subtracting 0.21 per cent from the total ammonia.

To obtain the available oil in the seed we must first find how much oil will be left in the cake produced. As cotton seed contain anywhere from three to four and one-half per cent ammonia and produce from 750 to 1050 pounds of 8.37 per cent ammonia cake in calculating the oil remaining in the cake we must always take into account the indicated yield as shown by the ammonia determination. Then if we assume a constant relation between the oil and ammonia content of the cake we can easily find the oil left in the cake by multiplying the pounds of cake produced by the percentage of oil remaining in the cake. This oil and ammonia relation is based on a standard figure of 80 to bring the oil yields of all seed to the same basis regardless of the grade of cake being produced. That is if 7 per cent ammonia cake is being made the oil content is assumed to be 5.60 per cent; if 8 per cent ammonia cake, the oil is 6.40 per cent.

The resultant figure obtained by multiplying the percentage of oil in the cake by the indicated yield of cake is subtracted from the total oil and this figure reported as available oil.

This method has been in use in our laboratories during the season just closing and has stood the test in every way. It readily adapts itself to volume. It is very easy to keep account of samples by numbering the pots. Duplicate determinations give close checks and abnormally high or low results are almost unknown.

On several test runs, where we have analyzed every car of seed worked, we have calculated within a very close margin, the yields of cake and oil obtained by the mills.

This method is now in the hands of the Seed Committee of the American Oil Chemists' Society and is being tried out by them.

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