

ment to removing all the lint by rubbing or shaking, we would have to weigh the delinted seed to figure results back to the original seed and you have no idea how easy it is to lose a few seed in manipulation. In the Malowan method twenty to fifty seed can be handled a day if there is sufficient oven capacity and with the mill properly regulated, only about five minutes will be needed to prepare each sample for analysis after the seed have cooled. There is no weighing of seed before or after treatment and all calculations are figured on the moisture basis, therefore, after the seed are treated and prepared, the remainder of the work is routine.

L. F. Hoyt, Chairman of the Sub-Committee of the A.C.S. on the Determination of Detergency of Soap Products, reports progress on a standard method which is now being studied by the members of his committee. Special apparatus has been designed and the method has been standardized. It is believed we will find that with progress already made the final report will show that a thorough practical method has been worked out.

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### REPORT OF THE SEED COMMITTEE

The Seed Committee continued the work of its predecessors and worked on improvements of the Malowan method besides sending out seed samples to several chemists to obtain a better try-out of the method.

The following laboratories co-operated with your committee: Barrow-Agee Laboratories in Memphis, Tenn., Shreveport, La., Greenville, Miss.; Houston Laboratories, Houston, Tex.; Houston Cotton Oil Mill, Houston, Tex.; Law & Co., Atlanta, Ga.; Picard Laboratories, Birmingham, Ala., and the Portsmouth Cotton Oil Refining Corporation, Portsmouth, Va.

Seed Sample No. 1 was analyzed by the following two methods: (Method A was proposed by the Seed Committee of last year and method B is an improvement worked out since.)

**Method A:** Take approximately 50 grams of the sample and moisten for 2-3 minutes with HCl (2 parts acid to 3 parts water). Drain off the liquid and dry the sample at about 130 C. for two hours, or long enough to reduce the moisture to a maximum of 3 per cent. Allow the sample to cool and then grind in a mill or mortar to a degree of fineness that allows of thorough mixing.

**Method B:** Place into a 150 or 250 cc beaker 3 to 5 cc. conc HCL and insert a crucible cover to keep the seed of the acid and add about 50 grams of seed without packing. Cover with a watch glass and heat in an air bath, oven, or hot plate for about ten minutes, then remove watch glass and continue heating for about 30 minutes till seed are dry. Empty the beaker and permit to cool, then grind the seed and mix.

**Oil:** Take four or five grams of the ground seed and extract for two hours, then re-grind sample in mortar and extract two hours longer.  
**Ammonia:** Take the ground seed and use the same method as for meal.  
**Moisture:** Use the official method for moisture in the original seed and make a moisture determination in the ground seed.

Take the difference of moisture between the original seed and the ground seed into consideration to figure out the oil and ammonia content of the original seed.

The results obtained on Check Seed No. 1 were as follows:

Moisture in								
Original Seed	9.00	8.03	7.59	8.90	7.92	7.93	7.75	(e)
Oil in Original Seed.	{ a19.09	a18.38	17.98	....	19.45	....	19.14	....
by method A.....	{ b18.26	b18.25	....	....	....	....	....	....
NH3 in Original....	{ a4.39	a4.26	4.25	....	4.45	....	4.38	....
Seed by method A..	{ b4.20	b4.23	....	....	....	....	....	....
Moisture in Ground								
Seed by Method A....	2.51	4.81	1.50	....	1.55	....	5.40	....
Oil in Original								
Seed by Method B....	18.74	18.51	18.77	18.90	....	18.69	21.57	18.70
NH3 in Original								
Seed by Method B....	4.27	4.27	4.42	4.31	....	4.37	4.19	4.21
Moisture in Ground								
Seed by Method B....	7.00	6.77	9.07	2.68	....	7.48	6.86	(e)
Results by other....	{ c18.26	18.33	....	18.70	....	18.72	....	....
Methods	{ d18.59	....	....	....	....	....	....	....
	4.45	4.33	....	4.06	....	4.19	....	....
	C	C	....	D	....	D	....	....

Did you use the Malowan Method before? ..... yes    yes    yes    no    no    yes    yes    yes  
 What Method do you use? ..... A    A&C    A    D    E    D    D    ....

- Notes: A.—Malowan Method A, described above.  
 C.—Grinding 10 grams untreated seed in mortar, extracting twice.  
 D.—Smalley-Copes Method.  
 E.—Official Method of Georgia C. S. Crushers' Association.  
 a. Results figured according to methods A & B.  
 b. Results figured from actual loss in weight of seed during treating and drying. Seed were weighed before treatment and before grinding.  
 c. Method C, extracting twice with one re-grinding.  
 d. Method C2, extracting three times with two regrindings.  
 e. No moisture determinations were made; but as oil and ammonia results agree with average, it can be assumed that the moisture in the treated seed did not vary much from moisture in the original seed.

The results agree quite well, although several chemists had no previous experience with the methods. Considering that method B is much easier to manipulate, besides giving better results, it was decided to discontinue experiments with method A. Mr. Picard modified the method B by using portions of exactly 10 grams instead of using about 50 grams.

Check Seed Sample No. 2 was run according to method B with the remark that neither the watch glass nor the crucible cover is required, but that too high a temperature in drying the seed should not be used and that the seed, which stuck to the container or have become dark brown should be discarded and not be ground. Method C is the Picard method

of treating and drying 10 grams, grinding in a mortar, extracting for three hours, re-grinding and re-extracting for two additional hours. For ammonia the double factor weight of the original seed is taken.

Results of Check Seed No. 2

Moisture in Original Seed .....	7.88	8.70	8.65	6.40	8.96
Oil in Original Seed, Method B.....	19.60	19.18	19.27	19.11	19.26
Ammonia in Original Seed, Method B.....	4.29	4.03	4.26	4.24	4.21
Oil, Method C.....	18.87	18.68	18.89	.....	18.84
Ammonia, Method C.....	4.06	4.04	4.10	.....	4.24
Copes Bag Method, Oil.....	19.06	.....	.....	.....	.....
Copes Bag Method, Ammonia.....	4.24	.....	.....	.....	.....

The results on this sample agree again quite well by either method, but there is a difference of 0.38 per cent between the results by the two methods.

A great disadvantage of the Malowan method is that a part of the seed become partly charred on the outside and it is necessary to separate the dark colored seed before analysis.

Some tests were made to determine the difference between the light colored seed and the dark colored seed. Twelve seed samples were treated and dried at 140 to 150 C. for one half an hour and the four most discolored samples taken for the test. Each sample was separated into a dark colored and a light colored portion and each of those portions weighed, ground and analyzed with the following results:

Weight of portion		Oil found in		Average oil figured from oil and weight
light	dark	light	dark	
30.3	13.6	19.86	21.01	20.21
33.6	19.6	19.61	20.60	19.97
28.2	14.9	17.97	17.94	17.96
26.3	15.7	19.18	19.86	19.43

If we consider that the oil found in the light colored portion represents the true oil content of the seed, then the maximum error would be about 0.35 per cent of oil. In the actual determination the possible error would be less because according to the method the dark colored seed ought to be discarded.

Several experiments were made to find out the causes of charring of the seed and ways to eliminate same. It was found that it is not the temperature by itself, nor the action of the hydrochloric acid; but it is the combined action of the aqueous HCl and high temperature which causes charring. This suggested three means to prevent charring, first: to lower the temperature; secondly: to reduce the amount of acid; and thirdly: to prevent the contact of seed with moist acid. The temperature cannot be reduced below 120 C. without prolonging the time of drying excessively. The acid can be reduced to 1 to 2 cc. and the contact of moist HCl with the seed can be prevented by using gaseous HCl or by preventing the condensation of the acid on either the walls of the vessel or on the seed.

Two methods have been worked out based on the above principles. Both methods use 1 to 2 cc. of acid; but in the one method the seed are dried in a beaker at 120 C., while in the other method the seed are treated in a funnel with the stem cut off short and dried at 130 to 140 C. In the latter case the funnel and the seed become hot before the acid begins to boil and therefore no condensation takes place.

Several tests were made to determine whether there is any loss in weight during treatment besides the one caused by the loss of moisture. Twenty gram portions of seed were treated and dried under various conditions and then placed into the regular moisture oven with other portions of untreated seed and weighed after five hours drying. If the latest two methods were used, the final weights of the treated and untreated seed were practically alike, showing that these methods give correct factors for the calculation of the oil and ammonia content in the original seed.

Untreated Seed 5 hours in Moisture Oven		Treated Seed After treatment five hours in Moisture Oven		Method of Treatment
% Dry Seed	% Dry Seed			
91.71	89.14	¾cc. HCl.	45 min. at 120C.	beaker without filter plate
91.71	89.24	¾cc. HCl.	45 min. at 130-140C.	beaker without filter plate
92.32	91.54	¾cc. HCl.	45 min. at 110-115C.	beaker without filter plate
91.10	90.80	½cc. HCl.	30 min. at 120-145C.	beaker with filter plate
91.10	90.85	½cc. HCl.	45 min. at 120-140C.	beaker with filter plate
91.40	91.40	½cc. HCl.	30 min. at 120C.	beaker with filter plate
91.07	90.70	½cc. HCl.	30 min. at 130-145C.	beaker with filter plate
91.07	91.13	½cc. HCl.	30 min. at 130-145C.	beaker with filter plate
91.07	90.85	½cc. HCl.	30 min. at 120C.	beaker with filter plate
90.90	90.35	½cc. HCl.	30 min. at 120C.	beaker with filter plate
90.90	90.88	½cc. HCl.	30 min. at 140C.	Funnel
90.90	90.45	½cc. HCl.	45 min. at 140C.	Funnel
90.90	91.23	½cc. HCl.	30 min. at 120C.	Funnel

To determine whether substances are formed during treatment which may be weighed as oil samples of hulls were treated like seed and no increase in the oil content could be noticed.

It has been noticed that sometimes no additional oil is extracted after re-grinding which would show that the dried seed can be ground in a suitable mill fine enough that it is possible to dispense with the re-grinding.

The last improvements were developed so late that it was impossible to have some seed samples ran by the collaborators by those methods; but same were thoroughly tested by the seed committee and it was found that the results by either method agree very well with those obtained by any other method.

A description of the two methods was sent out with a questionnaire to those chemists who did the co-operative seed work and answers were received. The questionnaire was as follows:

Are you in favor of having the method using about 50 gram of seed adopted?

Are you in favor of having the method of using 10 gram of seed adopted?

Are you in favor of having both methods adopted, leaving it to the chemist to choose either method?

Are you not in favor of having either method adopted?

Are you in favor of any other method?

The answers received were as follows:

C. W. Putland: I prefer the method using 50 grams to the one using 10 grams. I am not in favor of having both methods adopted. I have not done sufficient samples to judge accuracy of the method. Believe it should be tentatively adopted for one year and given a thorough trial.

B. L. Caldwell: I am in favor of the 50 gram method and opposed to the 10 gram method; but, I favor permitting a chemist to use both methods if he is satisfied with a 10 gram basis. My only objection to the second method is the smallness of the sample. I think the 50 gram method much better than the present Copes bag method.

J. R. Mays: We do not feel that we have had sufficient experience with either of the two methods yet. However, we believe that they are worthy of further study. We are not yet qualified by experience with the two methods to state whether they should be adopted or not.

P. S. Tilson: I am in favor of having both methods adopted, permitting the chemists to use either method.

G. Worthen Agee: We are in favor using the 50 gram method and not in favor of the 10 gram method for the reason that too small a weight of seed is used. We think that not less than 50 grams of seed should be treated. We see no advantage whatever in the method if small samples are used. We might add that in our experience, if the seed are reasonably dry when ground and are ground in a suitable mill, re-grinding is not necessary.

Considering the results obtained and the answers received the Seed Committee believes it can propose the following method for cotton seed analysis for adoption.

### **Proposed Method for the Analysis of Cotton Seed**

*Preparation of Sample and Determination of Impurities:* Pass the whole sample through a one-half inch sieve to separate the seeds from possible clusters of a similar kind. Then shake on a six mesh sieve to remove sand and dirt. For the determination of impurities, which can

be made at this point, shake a weighed portion on a sieve to remove sand and pick out the bolls and trash, weighing all the impurities together.

*Moisture:* (same as last year) Mash (do not crush or grind, simply flatten) a little more than 5 grams of seed in an iron mortar. Weigh out exactly 5 grams of the mashed seed into an official moisture dish and dry at 102 degrees C. for 5 hours. With official or referee samples make duplicate determinations and report as moisture content the average of at least two fairly concordant results.

*Oil and Ammonia* (Malowan-Picard Method):

**Method A:** Take a funnel of about  $3\frac{1}{2}$  inches diameter and cut off the stem to a length of  $1\frac{1}{2}$  inches. Close the stem by fusing together or by any other means. The stem shall hold one to two cc. of acid and the funnel 50 grams seed.

For the preparation of the seed, fill the stem with 1 to 2 cc. of concentrated HCl. Put into the funnel about 50 grams of seed and place the funnel into an asbestos oven at 130 to 140 C. for about half an hour, depending on the moisture content of the seed. It is advantageous to stir the seed after 10 minutes, so that the seed on top receive a better acid treatment. When the seed are dry remove and empty the funnel. Spread out the seed to permit the seed to cool and the last traces of acid to escape. Discard all seed which have become dark during treatment, usually only the two or three seed in the neck of the funnel, and grind in a mortar or mill to a degree of fineness which permits thorough mixing. The fineness of the ground seed will depend largely on the amount of moisture remaining in the seed. It is impossible to pass the ground seed through the mill a second time without gumming up the plates. After grinding the mill has to be cleaned and any larger pieces of seed remaining in the mill are ground in a mortar and added to the other portion.

**Or:** Instead of the funnel, take a 250 cc. beaker. Add 1 to  $1\frac{1}{2}$  cc. of concentrated HCl and place a perforated filterplate  $2\frac{1}{4}$  inches at the bottom, fill loosely with about 50 grams of seed and dry in an oven 120 C. for about half an hour. Empty, discard any dark colored seed and any seed sticking to the beaker and proceed as described above.

*Oil:* Take 5 grams of the ground seed and extract for two hours like c. s. meal, then re-grind the extract two additional hours.

*Ammonia:* Use the same method as for meal. (The addition of paraffin or amyl alcohol is not necessary, because there is no danger of excessive foaming).

Make a moisture determination of the ground seed like of meal to be used for figuring the results back to the original seed.

Calculate results by the formula:

$100 - (\% \text{ H}_2\text{O in original seed} - \% \text{ H}_2\text{O in ground seed}) \text{ equals } X.$

X times the oil and NH<sub>3</sub> values obtained gives the true values in the original seed.

A table has been figured out which will give the correction directly.

### Method B:

*Oil:* Weigh 10 grams of seed, discarding any palpably defective seed or empty hulls, but make no selection otherwise. Place not more than 7 drops of Conc. HCl at the bottom of a 100 cc. beaker, then add the seed and place into an oven or on a hot plate till the acid has evaporated and the seed are dry. Spread the seed on a piece of paper to cool. Then grind the seed in an iron mortar until they are approximately as fine as meal. Wrap the ground seed just like meal and extract for three hours, regrind and extract for two hours longer.

*Ammonia:* Use double factor weight. To prevent foaming during distillation use a piece of paraffin the size of a pea or a few drops of amyl alcohol.

*Committee:* JOHN MALOWAN, Chairman; D. C. PICARD.

TABLE OF CORRECTION FOR OIL AND AMMONIA. FOR ANALYSIS OF COTTON SEED.

Ammonia in Treated Seed, Per Cent	Difference		in		Moisture		Content		of		Original		and Treated		Seed		in Tens of One		Per Cent	
	00	02	04	06	08	10	12	14	16	18	20	22	24	26	28	30	32	34	36	38
	Corrections in hundred of one per cent																			
3.50	00	01	01	02	03	04	04	05	06	06	07	08	08	09	10	11	12	13	13	14
3.75	00	01	02	02	03	04	05	05	06	07	08	08	09	10	11	11	12	13	14	15
4.00	00	01	02	02	03	04	05	06	06	07	08	09	10	10	11	12	13	14	15	16
4.25	00	01	02	03	03	04	05	06	07	08	09	09	10	11	12	13	14	15	16	17
4.50	00	01	02	03	04	05	06	07	08	09	09	10	11	12	13	14	15	16	17	18
4.75	00	01	02	03	04	05	06	07	08	09	10	10	11	12	13	14	15	16	17	18
5.00	00	01	02	03	04	05	06	07	08	09	10	11	12	13	14	15	16	17	18	19

  

Oil in Treated Seed, Per Cent	Difference		in		Moisture		Content		of		Original		and Treated		Seed		in Tens of One		Per Cent		
	00	02	04	06	08	10	12	14	16	18	20	22	24	26	28	30	32	34	36	38	40
	Corrections in hundreds of one per cent																				
17.00	00	03	07	10	14	17	20	24	27	31	34	37	41	44	48	51	54	58	61	65	68
17.50	00	04	07	11	14	18	21	25	28	32	35	39	42	46	49	53	56	60	63	67	70
18.00	00	04	07	11	14	18	22	25	29	32	36	40	43	47	50	54	58	61	65	68	72
18.50	00	04	07	11	15	19	23	26	30	33	37	41	44	48	52	56	59	63	67	70	74
19.00	00	04	07	11	15	19	23	26	30	34	38	42	46	49	53	57	61	65	68	72	76
19.50	00	04	08	12	16	20	23	27	31	35	39	43	47	51	55	59	62	66	70	74	78
20.00	00	04	08	12	16	20	24	28	32	36	40	44	48	52	56	60	64	68	72	76	80
20.50	00	04	08	12	16	21	25	29	33	37	41	45	49	53	57	62	66	70	74	78	82
21.00	00	04	08	13	17	21	25	29	34	38	42	46	50	55	59	63	67	71	76	80	84
21.50	00	04	09	13	17	22	26	30	35	39	43	47	52	56	60	65	69	73	77	82	86

If treated seed are lower in moisture content than the original seed subtract correction, if the moisture content is higher add correction.

## ELECTED TO HONORARY MEMBERSHIP

The American Oil Chemists' Society, at its annual convention in New Orleans, unanimously elected to honorary membership Dr. C. A. Browne, Chief of the Bureau of Chemistry, U. S. Department of Agriculture.