REPORT OF SEED COMMITTEE FOR SEASON OF 1925-26

For the past decade, it has been the hope of everyone interested in the cottonseed products industry that an accurate and dependable method for seed analysis would ultimately be devised. It seemed hopeless, for years, that this would ever be accomplished, but the present Seed Committee believes that a method—easy of manipulation—which can be depended upon to give results comparable with mill yields has finally been worked out. Step by step, at the cost of much hard work by successive seed committees as well as by individual effort, this method has been evolved.

The chief trouble with seed analyses had always been the lint, as this prevented the grinding of a sample to sufficient fineness for analytical procedure. In addition to this, excessive moisture during the early part of the season was another problem, as wet seed simply mash and do not grind and therefore a representative sample for analysis could not be obtained on such seed. For many years the old method of cutting seed and analyzing the meats and hulls separately was the accepted procedure. The objection to this was the necessarily small sample—10 grams—which was of course not representative, as well as the time consumed in cutting. Also, oil was always lost in grinding the meats for analysis.

Various methods of analyzing whole seed have at times been proposed, to be tried out and discarded. First, there was the method of delinting seed with sulphuric acid and, after drying, grinding and analyzing the delinted seed. Unfortunately, the acid attacked the hulls as well as the lint so this method was quickly discarded as being impractical for handling seed in bulk. Later, placing 5 grams of seed in small canvas bags and extracting, after mashing the seed inside the bags with a hammer, was tried. Ammonia was determined on whole seed by this method. Concordant results were impossible, but this method was used for quite a while on the basis of making several extractions on each sample and averaging results. Numerous other methods have also been tried.

Of all the methods proposed during the past ten years, only one, the basic principle proposed by John Malowan, of hydrolyzing the lint of seed with hydrochloric acid, thus rendering it brittle and grindable and permitting large samples to be used, has survived the test of years of co-operative work. This method has been evolved from its crude beginnings to its present state of accuracy and ease of manipulation only as the result of patience and persistence in the face of discouragement and disappointment time after time. It is impossible for one who has not worked on seed analytical research for a number of years to fully understand the hope of achievement and subsequent disillusionment which followed in

rapid succession as the work was carried on. Each method, sooner or later, developed some weakness which necessitated discarding it.

From its beginning, the Malowan method gave closer checks and more concordance than any before proposed, but it was soon apparent that though ammonia results were accurate, oil results, for some reason not then understood, were usually too high. However, during the season of 1924-25 Messrs. Malowan and Picard, working together (there was no official Seed Committee that season), discovered that the use of liquid hydrochloric acid was the cause of the high oil results. The charring of the lint and hulls in part from the direct action of the acid while the seed were being dried, produced changes sufficient to raise the oil results. The remedy—to use gaseous acid—was obvious, though not quite so simple in its application as it might seem. The use of a false bottom to a beaker to elevate the seed above a small quantity of acid to be vaporized in the oven with simultaneous drying of the seed was not thoroughly satisfactory, as condensation of the acid in the lower part of the seed produced charring. However, when the discolored seed were discarded the results on the residual seed were excellent.

At this period of the committee work, the oil mill season of 1924-5 ended and Mr. Malowan accepted a position in a line of business not connected with cottonseed products, thus being compelled to leave unfinished on his part research on which he had spent much time and thought for a decade. For the season of 1925-26 the chairman was asked to choose his committee for seed work and selected the following men: C. H. Cox, of the Barrow-Agee Laboratories, Memphis, Tenn; G. R. Dunning, of the Fort Worth Laboratories, Fort Worth, Texas; W. D. Hutchins, of the Southern Cotton Oil Co., Savannah, Ga.; and C. M. Putland, of the Portsmouth Cotton Oil Refining Corp., Portsmouth, Va. This formed a well balanced committee composed of men from entirely different sections of the country and in addition, having two men from commercial laboratories and two from corporations, thus getting the benefit of the different view-point of each side.

By happy chance, unknown to the chairman at the time of the selection of the committee, Messrs. Cox and Dunning had been doing independent work on seed analysis and much of the Seed Committee's time was spent in studying modifications of the Malowan method proposed by these two gentlemen. The chairman of the committee had devised a special glass tube for treating seed, which gave excellent results, though rather unhandy to manipulate. These tubes were 60 mm. x 60 mm., drawn to a funnel shape at the bottom and terminating with a small well to hold 3 cc. of hydrochloric acid. The seed were supported on a porcelain filter plate. That this treatment was good is shown by the results on seed samples 1 to 4 which were run, in part, in these tubes.

However, it is not enough that any method for seed analysis be merely accurate. It must also be practicable for handling a large number of samples at one time with a minimum of effort. Nothing is to be gained by advocating any method, no matter how accurate, if it is not adapted to the working conditions of the average cottonseed products laboratory during the rush of the season when minutes are precious. Therefore, when Mr. Cox brought to the attention of the chairman of the committee a most ingenious modification of the Malowan method devised by him, the chairman, after trying it out, saw at once that after many years, a practical and accurate method of treating seed had at last been devised, adaptable in every way to work on a large scale. This method of treatment is simply to pour a few cc. of hydrochloric acid around the sides and bottom of a 3-inch flower pot and after it has been absorbed into the pores place 50 grams (or more) of seed in the pot and heat at 130° C. for an hour until the acid has been expelled. This treatment *absolutely* does away with any charring of the lint but leaves the lint more brittle than any other method and the seed can be ground to a meal of uniform composition. After the proposal of this method, the glass tubes were withdrawn from experimental use.

At about the same time, Mr. Dunning proposed a trial on the method used at the Fort Worth Laboratories, which consists of placing exactly 50 grams of seed on watch glasses in a suitable glass container and passing gaseous hydrochloric acid through the samples. After treatment, the samples are placed in an oven at 110-130° C. for ten minutes and then allowed to cool. When cold, all lint is rubbed off the seed by hand and the delinted seed are carefully weighed and ground. Results are figured on the difference in weight of the raw and the delinted seed. This procedure was submitted to the committee for study and trial.

Now, in comparing the two methods studied, it must be remembered that cottonseed is very variable in moisture and at times may be so wet as to be ungrindable. A method of analysis acceptable to the Society must be suitable for handling all degrees of moisture *alike*—that is, after the seed are prepared for analysis the analytical procedure should be the same in all cases. In the Cox procedure the seed may remain in the oven until sufficiently dry (about 6 per cent) to grind to a uniform meal, taking but a single extraction. The basis of calculation of this method is in the moisture in the whole seed and the moisture in the treated seed. As these determinations are run side by side, any actual errors in moisture determinations are relative and are about the same on both the whole seed and the treated seed, therefore the moisture element as a source of error in calculation can be practically eliminated. The Cox modification, therefore, is adaptable to seed in any range of moisture.

The Dunning modification, on account of the very short time the seed remain in the oven, does not seem as widely applicable to varying moisture conditions as the Cox modification. Though the Fort Worth Laboratories obtain good results with it on high moisture seed, it is obvious that double extractions would have to be used on wet seed as the short heating would not dry the seed sufficiently to grind to a uniform meal. One point brought up by Mr. Hutchins is that there is a possible source of error due to the change in moisture conditions during grinding of the delinted seed which he thinks is likely to be larger than can exist in the standard Malowan method—unless the seed are allowed to reach a moisture equilibrium before grinding. This seems logical because the Malowan method, as orginally proposed, was figured on the difference in the weight of the raw and treated seed and results did not check well among different operators until this was discarded and the calculation based on difference in moisture.

The committee finds that the Cox modification offers much more ease of manipulation than the Dunning procedure. There is less of the personal element. This might be overcome in the Dunning method by using mechanical shaking instead of rubbing by hand to remove the lint from the seed, but the change of the sample from one container to another in the preliminary treatment is unnecessarily time consuming. The Cox modification uses one container only and the sample goes from the oven treatment directly to the mill.

It is not a question of accuracy alone, as pointed out above, for the Seed Committee agrees that the Dunning modification, when carried out properly, is fully as accurate as any method proposed, but adaptability and ease of manipulation under average laboratory conditions must be considered as well in advising the adoption of any method by the Society.

The majority of the committee, composed of Messrs. Hutchins, Putland and Picard (Messrs. Cox and Dunning, as the proposers of the two methods under consideration, not voting), unanimously feel that the modification of the Malowan method as devised by C. H. Cox, should be be adopted as official and recommended for use for the season 1926-27. This method eliminates the large source of error in the original Malowan method—the charring of the seed in treating—and has also been tested against actual mill yields with thoroughly satisfactory results.

It is also recommended that the Dunning modification be studied by next year's committee with especial reference to mechanical delinting of seed and a report made as to its adaptability as an alternate method for official use. It would be well, also, to test it against mill yields, for this is the final proof of the value of any method.

Below are given tabulated reports, with explanatory notes, covering the actual work done by this year's committee.

Samples 1 to 4, whole unground seed, were run partly by the special tube method and partly by the flower pot and Fort Worth methods. The method used is given. It will be noted that agreement is close throughout. The seed for these four samples were carefully selected for uniformity.

RESULTS ON SAMPLE NO. 1

Analyst	Cox	Dunning	Hutchins	Putland	Picard	Aver-
Method	Tube	Tube	Tube	Tube	Tube	age
Moisture	7.20	7.51	7.45	6.33	7.02	7.10
Oil	19.91	20.60	19.79	19.57	19.87	19.95
Ammonia	4.05	4.03	4.14	4.07	4.09	4.09

RESULTS ON SAMPLE NO. 2

Analyst	Cox	Dunning		Hutchins	Putland	Picard	Aver-
Method	Flower Pot	Tube	Delinted	Tube	Tube	Tube	age
Moisture	11.48	10.17	10.17	10.72	11.11	11.70	10.89
Oil	17.50	18.16	17.50	18.16	18.13	17.81	17.88
Ammonia	4.03	4.28	4.04	3.99	3.98	4.09	4.07

RESULTS ON SAMPLE NO. 3

Analyst	Cox	Dunning		Hutchins	Putland	Picard	Aver-
Method	Flower Pot	Tube	Delinted	Tube	Tube	Tube	age
Moisture	11.10	9.78	9.78	10.15	10.45	10.88	10.37
Oil	17.41	17.96	18.18	18.55	18.09	18.14	18.06
Ammonia	4.00	4.12	4.18	4.10	3.91	4.09	4.07

RESULTS ON SAMPLE NO. 4

Analyst	Cox	Dunning	Hutchins	Putland	Picard	Aver-
Method	Flower Pot	Tube	Tube	Tube	Tube	age
Moisture	12.05	12.49	10.70	11.95	12.08	11.86
Oil	17.45	17.80	18.20	18.39	18.08	17.98
Ammonia	4.10	4.15	4.05	3.99	4.08	4.07

Seed samples 5 to 9 were treated with hydrochloric acid by the flower pot method and ground so that all meats passed a 30 mesh sieve. This fineness of grinding of the meats granulates the hulls so that on remixing, a meal of uniform texture is produced. In none of the samples was there any charring of lint by the acid. The lint was completely attacked by the acid and was thoroughly disintegrated by the mill so that no traces of lint were visible in the ground material. The double extraction consisted of two extractions of 2 hours each with a regrinding between. The single extraction was of 5 hours duration. It will be noticed that with the proper preparation of the sample, single extractions check double extractions closely. It must be strongly stressed, however, that unless samples are *uniformly ground* to a certain degree of fineness, double extractions are necessary.

RESULTS ON SAMPLE NO. 5

Analyst	Cox	Dunning	Hutchins	Putland	Picard	Av.
Oil: Double Ext.	19.38	19.37	19.30	19.63	19.24	
	19.45	19.25	19.28	19.70	19.28	
	19.43	19.31	19.29	19.67	19.26	19.39
Single Ext.	19.33	19.30	19.12	19.33	1929	
	19.23	18.96	19.32	19.14	19.09	
	19.28	19.13	19.22	19.23	19.19	19.21

RESULTS ON SAMPLE NO. 6

Analyst	Cox	Dunning	Hutchins	Putland	Picard	Av.
Oil: Double Ext.	21.08	20.80	21.18	21.00	21.09	
	21.05	20.78	21.20	20.94	21.05	
	21.07	20.79	21.19	20.97	21.07	21.02
Single Ext.	21.15	20.55	21.17	21.10	20.89	
	21.03	20.79	21.24	21.17	20.97	
	21.09	20.68	21.18	21.13	20.83	20.98

RESULTS ON SAMPLE NO. 7

Oil: Double Ext.	19.73	19.25	19.64	19.97	19.69	
	19.58	.....	19.70	19.90	19.67	
	19.65	19.25	19.67	19.94	19.68	19.64
Single Ext.	19.65	19.49	19.44	20.17	19.50	
	19.63	.....	19.40	19.83	19.38	
	19.64	19.49	19.42	20.00	19.44	19.59

RESULTS ON SAMPLE NO. 8

Moisture	5.12	5.06	5.08	5.16	5.19	5.12
Ammonia	4.30	4.34	4.24	4.20	4.29	4.27
Oil (Double Ext.)	19.54	20.22	19.97	.....	19.50	19.82
(Single Ext.)	19.64	19.80	19.74	19.66	19.50	19.67

RESULTS ON SAMPLES NO. 9

Moisture	5.50	.....	5.58	.....	5.66	5.58
Ammonia	.....	4.55	4.40	.....	4.50	4.48
Oil (Double Ext.)	18.07	17.88	18.17	.....	18.02	18.04
(Single Ext.)	18.05	17.73	18.20	.....	17.98	17.99

When these four samples were sent out the season was far advanced and practically no prime seed could be obtained. The samples were composed of off seed and in spite of all care in preparing, the resulting material was not uniform, yet results agreed fairly well—in fact, better than on prime seed by older methods.

RESULTS ON SAMPLE NO. 10

Analyst	Cox	Dunning	Hutchins	Putland	Picard	Av.
Flower Pot Method						
Moisture	7.10	.....	7.36	6.12	6.28	6.72
Ammonia	4.20	.....	4.12	4.25	4.16	4.18
Oil: (Double Ext.)	19.42	.....	18.96	19.46	19.44	19.32
(Single Ext.)	19.42	.....	18.77	19.33	19.44	19.24
Delinted Method						
Moisture	7.10	.....	7.36	.....	6.28	6.72
Ammonia	4.02	4.00	4.08	.....	4.04	4.04
Oil: (Double Ext.)	19.42	.....	18.53	.....	18.63	18.86
(Single Ext.)	19.35	17.17	18.58	.....	18.63	18.43

RESULTS ON SAMPLE NO. 11

Flower Pot Method						
Moisture	6.65	.....	6.04	7.04	6.99	6.68
Ammonia	4.41	.....	4.28	4.30	4.31	4.31
Oil: (Double Ext.)	20.26	.....	20.38	21.22	19.90	20.18
(Single Ext.)	20.25	.....	20.46	20.96	19.89	20.39
Delinted Method						
Moisture	6.65	.....	6.04	.....	6.99	6.68
Ammonia	4.12	4.18	3.92	.....	4.23	4.11
Oil: (Double Ext.)	.....	19.06	.....	.....	19.62	19.34
(Single Ext.)	19.72	.....	18.23	.....	.....	18.97

RESULTS ON SAMPLE NO. 12

Analyst	Cox	Dunning	Hutchins	Putland	Picard	Av.
Flower Pot Method						
Moisture	7.20	.....	6.29	.....	7.77	7.04
Ammonia	4.57	.....	4.32	.....	4.55	4.48
Oil	16.64	.....	18.06	17.76	16.80	17.32
Delinted Method						
Moisture	7.20	.....	6.29	.....	7.77	7.04
Ammonia	4.45	4.46	4.44	.....	4.58	4.48
Oil	18.02	17.44	17.88	.....	17.33	17.69

RESULTS ON SAMPLE NO. 13

Flower Pot Method						
Moisture	6.60	.....	6.04	.....	6.88	6.51
Ammonia	4.12	.....	4.28	.....	4.21	4.15
Oil	19.70	.....	20.17	19.92	19.80	19.87
Delinted Method						
Moisture	.....	.....	6.04	.....	6.88	6.46
Ammonia	.....	4.19	4.19	.....	4.21	4.20
Oil	.....	19.22	19.92	.....	19.17	19.45

Respectfully submitted,

Seed Committee, 1925-26.
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