DAMAGED SEED AND SEED ANALYSIS COMMITTEE REPORT 1921-22

By D. C. Picard, Birmingham, Ala.

The work of this Committee for the season 1920-21 was largely in the nature of a preliminary investigation of proposed new methods of analyzing whole seed with a view of supplanting the unwieldly and unhandy method of analysis by cutting seed, which had been the common practice for the last two decades. The methods investigated were (A) that devised by the late Dr. Smalley and Mr. Copes, in which 5 grams of seed are dried in canvas bags thoroughly pounded with a hammer, and extracted directly, with ammonia determinations on a separate undried portion; (B) that proposed by Mr. Malowan which consists, briefly, of treating a comparatively large sample of seed with hydrochloric acid for a minute or so and, after drying, grinding the whole sample in an ordinary coffee mill, making the analysis on the ground sample and figuring results back to the original moisture of the seed; (C) that proposed by Mr. Lehman Johnson in which, after being dried, the seed are ground up in a mortar, wrapped in filter paper and extracted, and the residue from the extract used for ammonia determination.

Of these three methods, the Smalley Copes and Malowan methods were deemed practicable for future study by the present Seed Committee because while the Johnson method gave as good results as any other, it was considered too time consuming and the personal equation was probably its largest factor of accuracy. What the Seed Committee desired to develop was a method that would give dependable results under the average working conditions of cotton seed products laboratories during the height of the season. The general opinion was that the Malowan method was the most accurate of any proposed, but on account of the large amount of seed used and the necessary drying equipment to handle a great number of samples per day, the method was regarded as better adapted to check and referee work than to daily routine testing. The Committee, therefore, gave most of its attention to the study of the Smalley-Copes method.

The greatest trouble that last year's Committee had with this method was to obtain a clear extraction. It seemed almost impossible to prevent meal from percolating into the extraction flasks. The method of preparing for extraction at that time was to tamp the meats down into the bags, place a small wad of cotton in the top to prevent washing, and wrap the bags in one filter paper, leaving the tops open so that the petroleum ether could drop directly on the cotton. From results obtained, it was evident that this manner of preparation was largely responsible for the variation in results, due to fine meal sifting through the cloth and filter paper.

During the early part of the summer, the chairman of the Committee and his personal assistant made an extended series of experiments to see if it were not possible to do away with the entrainment of the fine meal particles with the oil. It was found that by rolling the bags into tight cylinders and wrapping them in two thicknesses of filter paper just as meal samples are wrapped, a clear extraction and far more concordant results could be obtained. This gave the idea of taking an ordinary square of canvas about 5-inches to the side and folding it over to make a cylinder. The seeds were spread in a thin layer in the fold, rolled up and wrapped in filter paper in the same manner as the bags. Results were very good and there was the advantage of easy cleaning after being used.

On account of the difficulty in obtaining the right grade of canvas for Copes bags, samples of filter cloth were secured from one of the large filter cloth manufacturers and made into bags and squares for comparison with the canvas used in Copes bags and regular No. 12 duck used for the squares. However, about fifty tests showed that filter cloth was too loose in texture and could not stand pounding so was not as well adapted to this work as canvas.

The propositions to be studied for this year were (1) moisture, (2) oil, (3) ammonia and (4) free fatty acid in connection with off oils. For moisture, it was to be determined whether cutting seed in half preliminary to drying, was preferable to drying them whole; for oil, a study of the best way to get concordant results on not more than 5 grams of seed; and for ammonia, the size of sample best adapted to obtain good checks.

Seed sample No. 1 was sent out early in the fall and tested for moisture by cutting seed in half before drying; for oil, by using the Smalley-Copes bags and Picard cylinders on both 5 and 10 gram samples, and the Malowan method; for ammonia, by using the regular and the double factor weights. The moisture results were very good, being much more uniform than those obtained on whole seed. Ammonia results showed that there were mechanical difficulties almost insurmountable in handling the double factor weight on seed with practically identical results with those obtained on the single factor weight. Oil results, using the bags and squares, gave no particular difference in the amount of oil obtained, but the general objection was made that the squares took up too much space in the extraction tubes. The chairman of the Committee then hit upon the obvious idea of cutting down the size of the squares and sewing up the sides, leaving both ends open for ease of cleaning, making a sort of cylinder. The length of these cylinders was about 5 inches and the width about 2½ inches. The results on this first sample were used as a basis for work on all subsequent samples sent out.

With sample No. 2 these new cylinders were substituted for squares and used throughout the remainder of the season. Results on this sample with bags and cylinders were about the same. Both the single and double factor weights for ammonia were again used but as the results were practically identical, it was decided to discontinue the use of the double factor weight. Mr. Malowan did some special work on moisture determinations from which it appears that drying cut seed for 5 hours gives some advantage over drying them whole for a longer period. In expelling the moisture before extracting, seed had been dried in the bags but Mr. Roettger made the suggestion that they be dried in the regulation tared moisture pans which not only permitted of more thorough drying, but also prolonged the life of the bags as in this way, they were not subjected to intense heat. This suggestion was adopted and used on all subsequent work. On seed sample No. 3, work was continued in comparing results between the bags and cylinders with results as before—practically the same. As Mr. Forbes raised the point that a third extraction produced more oil, three extractions were made on this sample instead of the usual two, with both bags and cylinders and an average of about 0.18% extractive matter was recovered in the third extraction. Study by Mr. Malowan, however, indicates that this last extraction is not oil, but a gummy substance not yet identified. Experiments by Messrs. Malowan and Picard showed that a preliminary treatment of 5 grams of seed with hydrochloric acid and drying would give excellent results, this treatment changing the lint into hydrocellulose and making it very brittle so that it would pound into a fine powder instead of lumping into balls like the untreated seed. On sample

No. 4, in addition to the regular methods, it was decided to have an extraction of 5 hours straight on seed so treated. The results were so excellent that with sample No. 5 the same treatment was again tried with two extractions instead of one and results were even better than with the previous sample and far better than any extractions on untreated seed.

During the progress of the work, Mr. Forbes suggested the use of 3 gram samples instead of 5 grams but on account of the fact that the Uniform Methods Committee has gone on record against using very small samples for oil, the Committee did not work on 3 gram samples. This is, nevertheless, well worth study because ammonia results have been perfectly concordant on the regular factor weight and it is very probable that better checks could be obtained on smaller samples on oil. The Southern Cotton Oil Company's head laboratory in Savannah has developed a home-made extraction thimble of which the directions for making were given by Mr. Roettger and are appended to this report with the methods of analysis. It is probable that the use of the thimble would give just as clear extractions as can be obtained by wrapping the bags in filter paper.

Conclusions:—Moisture is best determined by cutting seed in half and drying for 5 hours at 105°C. The single factor weight gives as accurate ammonia results as the double weight, with less trouble in manipulation. There is practically no difference in oil results where Copes bags and Picard cylinders are used but the latter permit of a more thorough cleaning. The average difference of the five chemists on five samples, leaving out the obviously high or low results, is 0.64% or 1.5 gallons of oil with Copes bags and 0.48% or 1.3 gallons with cylinders. The chemists checked themselves within 0.41% or 1.0 gallon with Copes bags and 0.37% or 1.0 gallon with cylinders. It is the opinion of the Committee that the cylinders are of somewhat easier manipulation than the bags but the use of either bags or cylinders is a matter of individual preference. The acid treatment of 5 gram samples before extraction gives closer checks than untreated seed. The chemists checked themselves on this method within 0.18% or 0.5 gallon and each other within 0.33% or 0.9 gallon. As only two samples were run by this method, nothing conclusive can be said to have been obtained. Nevertheless, this method should be carefully investigated.

The standardization of methods used for tests caused a great improvement in the checking of results between the chemists. Last season, the average difference between chemists on the Smalley-Copes method was 1.48% against this sason's average of 0.64% on the same method. The average difference then between checks by the same chemist was 0.50% and this year's, 0.48%. This evidently means that extractions on whole seed, as we are now doing, have a limitation of from 0.45% to 0.60% when handled by the same operator in precisely the same manner. It is also evident that if a strict procedure is outlined and followed faithfully, different operators can be expected to check each other within about the same limitations. It is quite possible that future study and further refinement will make even closer checks possible.

On account of the fact that this was a prime seed season, nothing could be done on what is probably the most important work of the Committee, the investigation of the relationship between the free fatty acid in the seed and off oil produced in the mill. During an off season, the majority of the Seed Committee's time should be spent in investigating this most important matter.

The Seed Committee feels that enough work has been done to recommend as provisional, the following methods: for moisture, the drying of 5 grams of cut seed for 5 hours at 105°C; for oil, the extraction of 5 grams of whole seed for 5 hours (one extraction of 3 hours then respound and reextract for 2 hours) for routine testing, but duplicate and even triplicate determinations are necessary because of the fact that the present limitation of the method recommended is within 0.60% or 1.5 gallons. Even under the most careful manipulation the checks have been as far apart as 1.0% or 2.5 gallons by individual operators, and it is obvious that unless duplicate or triplicate determinations are made, there is no guarantee of absolute accuracy. For ammonia, the use of the single factor weight, 1.7034 is recommended. For all large sales samples the Malowan method is recommended.

Below are given the outlines of the methods of analysis recommended together with the tabulated results of the Seed Committee's work during the year:

Methods of Analysis

Method of Sampling

Pass the whole sample through a one-half inch sieve to separate the seed 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 the six mesh sieve to remove the sand and pick out the bolls and trash, weighing all the impurities together.

Moisture

Weigh exactly 5 grams of seed cut in half into an official tared moisture dish and dry in the oven for five hours at 100-105°C.

Oil—(*Smalley-Copes Method*)

Weigh exactly 5 grams of whole seed, cutting a seed lengthwise, if necessary, to get accurate weight, into an official tared moisture dish and dry in an oven at 130°C for one-half to one hour, depending on the moist condition of the seed. After this partial drying, which is designed to remove enough moisture to make the sample brittle enough to disintegrate readily, place the seed in either the Copes bags or the Picard cylinder modification and pound thoroughly with a hammer or mallet to reduce them to a fine powder. Spread the pounded mass evenly in the bags or cylinders and roll them up tightly, wrapping in two thicknesses of filter paper just as meal samples are wrapped, and extract for three hours. Remove the bag from extraction tube and paper, allow the gasoline on the bag to evaporate, repound and reextract as before for two hours. Evaporate gasoline off the extraction, place flask in moisture oven at 105°C for one-half hour, cool and weigh. An optional method of making extraction is to place the bag or cylinder in the home-made paper extraction thimbles developed by the head laboratory of the Southern Cotton Oil Company, directions for the making of which are appended. If the thimbles are used, the Copes bags are not rolled up, but the pounded mass is well tamped in the bag and the top of the bag turned over and tucked into the thimble. If the cylinders are used, they are rolled up and tied with a string and then placed in the thimbles. *All determinations must be made in duplicate or triplicate.*

The Copes bags are made by folding a piece of No. 10 canvas 4 x 3 inches, in the middle and sewing one end and one side together with a strong twine, leaving one end open. The Picard cylinders are made by folding a piece of No. 12 canvas 4½ x 4½ inches, and sewing it up lengthwise, leaving both ends open.

Ammonia

Make duplicate determinations on whole seed, using 1.7034 grams. There is considerable foaming in dis-

tilling, which can be controlled by placing a piece of paraffin the size of a pea in each flask just before attaching to the still.

Malowan Method

Weigh 25 grams of seed on a balance sensitive to 0.1 gram, not necessarily selecting whole seed, as free meats, broken seed or empty hulls do not interfere. Stir in a beaker with diluted hydrochloric acid (2 parts acid to 3 parts water) just sufficiently to dampen the lint, then remove the seed and permit them to drain. The whole procedure should not take five minutes. Dry the seed in an asbestos oven at about 130°C for one hour then remove and weigh on same balance to find loss on this preliminary drying. Drying the seed on a porous plate is preferable to drying on a watch glass because some of the lint will stick to the glass in drying. A porous plate 8 x 12 inches is sufficiently large for four to six samples. Seed do not need to be spread out but can be placed on the plate in a small pile. The lint has been changed to hydrocellulose and can be easily ground. Grind seed in a mill and mix well together.

Moisture—Take 4 grams of the ground seed and run as usual at 100-105°C.

Oil—Weigh 4 grams of ground seed into either Copes bags or Picard cylinders and proceed exactly as described for the Smalley-Copes method.

Ammonia—Weigh 1.7034 grams of ground seed and treat as usual.

Calculation—Figure all percentage results back to the original weight, taking loss on the preliminary drying into consideration. For instance, if 25 grams became after drying, 23.2 grams, the loss was 7.20%. If the analytical results on the ground sample were: moisture, 4.21%, oil 21.06% and ammonia 7.11%, calculate as follows: subtract 7.20% from 100% which gives the amount of material worked on in relation to the original weight, in this case, 92.80%; multiply the percentage results by this figure which will give directly the oil and ammonia on the original seed, but the moisture result must be added to the moisture obtained on the preliminary drying, the final results being as follows: moisture 11.11%, oil 19.54% and ammonia 6.60%.

Results on Seed Sample No. 1

	Malowan	Picard	Roettger	Strack
Moisture				
Regular Method	10.44	10.22	10.09	7.87*
Malowan Method	10.36	10.67	11.03	8.95
Oil				
Smalley-Copes				
Method, 5 grams	17.68	17.58	17.38	17.84
	17.90	17.42	17.60	17.67
		17.58	17.84	
			17.84	
Average	17.79	17.53	17.66	17.75
Method, 10 grams	16.56	17.56	17.50	17.23
		17.74	17.68	17.20
		17.82	17.90	
		17.70		
Average	16.56	17.71	17.69	17.22
Malowan Method	17.56	17.11	16.91	17.36
	17.48	17.03	17.08	18.20
		17.43		
		17.28		
Average	17.52	17.21	16.99	17.78
Special Method				
(Picard) 5 grams	17.58	17.84	17.54	18.62
	17.72	17.76	17.84	17.52
		17.86	17.84	
		17.46		
Average	17.65	17.73	17.77	18.07
Picard 10 grams	17.75	17.39	17.60	17.25
		17.48	17.87	17.75
Average	17.75	17.43	17.73	17.50
Ammonia				
1.7034 grams	4.40	4.25	4.07	4.27
	4.25	4.29	4.20	4.26
	4.36			
Average	4.34	4.27	4.14	4.27
3.4068 grams	4.39	4.21	4.13	4.22
	4.37	4.25	4.25	4.19
	4.45			
Average	4.40	4.23	4.19	4.21
Malowan Method	4.43	4.34	4.31	4.29
	4.40	4.26	4.34	
	4.44	4.28		
	4.29			
Average	4.42	4.29	4.33	4.29

* Note—Sample held some time before testing.

Results on Seed Sample No. 2

	Forbes	Malowan	Picard	Roettger	Strack
Moisture					
Regular Method	8.80	(Seebelow)	8.60	9.24	8.98
			8.84		8.86
					9.10
Average	8.80		8.72	9.24	8.98
Malowan Method			9.60		9.06
					9.12
Average			9.60		9.09
Oil					
Smalley-Copes	18.80	20.33	19.63	18.60	19.56
	19.10	20.70	20.10	18.80	19.64
	18.70	18.80	19.20	19.06	
		19.75			
		20.04			
Average	18.86	19.92	19.64	18.82	19.60
Picard Cylinders	18.40	19.68	19.43	18.72	19.67
	18.40	19.88	19.83	19.08	19.41
		19.39	19.74	19.12	
		20.01			
		19.72			
Average	18.40	19.74	19.67	18.97	19.54
Malowan Method			20.15		18.92
			20.06	Copes bags	18.08
			20.31		
					18.50
			20.17		
					19.12
				Picard	19.49
				Cylinders	19.30
				Average	18.90

AMMONIA					
1.7034 grams	4.23	4.00	4.05	3.98	4.09
	4.28	4.05	4.07	4.02	3.91
		4.29	4.04	3.98	
		4.36			
Average	4.25	4.17	4.05	3.99	4.00
3.4068 grams	4.29	3.96			3.98
		4.05			3.98
		4.15			
		4.35			
Average	4.29	4.13			3.98
Malowan Method			4.08		3.96
			4.15		3.93
			4.14		
Average			4.12		3.94

Special Work Done by Mr. Malowan on Oil and Moisture

MOISTURE—5 grams of seed, cut in half, were dried at 105°C with the following results: first five hours, 8.76; ten hours, 9.42; fifteen hours, 9.43; twenty hours, 9.20; average, 9.20. Results on seed dried whole at same temperature, as follows: first five hours, 8.27; ten hours, 8.98; fifteen hours, 9.16; twenty hours, 9.08; average, 8.87.

OIL—Acid treatment was used with 10 grams delinted seed, first extraction five hours, reextraction, three hours, with following results; on first extraction, 19.53; 19.37; 19.63; 19.42; average, 19.49. On reextraction, 0.09; 0.10; 0.02; 0.03; average, 0.06. Acid method used without delinting, first extraction, 19.41; 19.21; 19.28; average, 19.30. On reextraction, 0.02; 0.16; 0.16; average, 0.11.

Results on Seed Sample No. 3

	Forbes	Malowan	Picard	Roettger	Strack
MOISTURE					
Regular Method	7.90		7.82	7.80	8.32
			7.78	7.60	8.48
					8.30
Average	7.90		7.80	7.70	8.36
Malowan Method			9.38		9.41
OIL					
Smalley-Copes	20.13	20.16	10.86	19.00	19.53
	20.17	20.05	19.42	19.00	19.96
	20.14	19.92	19.12	18.72	
	19.94			19.26	
	19.56				
	19.93				
Average	19.98	20.04	10.27	19.00	19.74
Picard Cylinders	19.80	19.63	19.90	18.92	20.34
	19.90	19.85	19.50	18.84	20.06
		19.78	20.42	18.80	
Average	19.85	19.75	19.94	18.85	20.20
Malowan Method			19.22		19.06
			19.16		
Average			19.19		19.06
Acid Method					
(5 hrs. straight		19.55		18.81	
Undelinted)		19.78			
Average		19.66		18.81	
				18.64	
Copes bag in				19.26	
Thimble					19.58
					19.60
					19.27
AMMONIA					
1.7034 grams	3.95	3.75	3.91	4.02	4.00
	3.95	3.76	3.99	4.12	3.85
	4.00	4.00		4.20	3.70
	4.03				
	4.08				
	4.13				
Average	4.04	3.83	3.95	4.11	3.85
Malowan			3.99		
			3.98		4.11
					4.16
					4.11
Average			3.99		4.12

Work by Mr. Strack

Moisture was run on whole seed dried for five hours with the following results: 8.04; 7.90; 7.94; average, 7.96.

Additional Work on Extractions Sample No. 3

	Oil after two poundings	Oil on reextraction	Total oil
FORBES			
Smalley Copes	20.13	0.13	20.26
	20.17	0.10	20.27
	20.14	0.03	20.17
	19.94	0.20	20.14
	19.56	0.44	20.00
	19.93	0.17	20.10
Average	19.98	0.18	20.16
MALOWAN			
Smalley-Copes	20.16	0.22	20.38
	20.05	0.16	20.21
	19.92	0.23	20.15
Average	20.04	0.20	20.24
Picard Cylinders	19.63	0.20	19.83
	19.85	0.14	19.99
	19.78	0.16	19.94
Average	19.75	0.17	19.92
PICARD			
Smalley-Copes	19.86	0.12	19.98
	19.42	0.14	19.56
	19.12	0.16	19.28
Average	19.47	0.14	19.60
Picard Cylinders	19.90	0.18	20.08
	19.50	0.24	19.74
	20.42	0.18	20.60
Average	19.94	0.20	20.14
ROETTGER			
Smalley-Copes		0.26	
		0.32	
		0.08	
		0.10	
		0.19	
Picard Cylinders		**0.10**	
		0.10	
Average		0.10	

Results on Seed Sample No. 4

	Forbes	Malowan	Picard	Roettger	Strack
MOISTURE					
Regular Method	8.20 Whole	8.25	8.20	8.60	9.42
	8.00 Cut	8.73	8.28		9.28
					9.46
Average	8.10	8.49	8.24	8.60	9.38
Malowan Method					10.51
					9.02
Whole seed dried					8.98
8 hrs. at 105°C					9.24
					9.08
20 hrs.					9.10
OIL					
Smalley-Copes		20.52	21.10	20.86	19.48
		20.20	20.08	21.34	
		20.39	19.90	21.48	
Average	20.30	20.37	20.36	21.23	19.48
Picard Cylinders		20.12	20.54	20.78	20.13
		20.23	20.72	20.90	
		20.48	20.58	21.42	
Average	20.20	20.28	20.61	21.03	20.13
Malowan Method					18.51
Acid Treatment					
5 hrs. straight		20.44	20.16	19.80	20.19
		20.30	20.18	20.40	19.78
				20.60	
Average	20.20	20.37	20.17	20.27	19.98
					19.27
S-C extraction					20.24
on seed used					20.58
for moisture					19.18
					19.82
Same, using					19.24
100 cc. flasks					20.35
					19.79
				20.86	
C bag & thimble				21.10	
for 5 hrs. with				21.32	
two poundings				21.09	
AMMONIA					
1.7034 grams		3.94	4.06	4.20	3.82
		3.95	4.12	4.25	3.90
		3.88			4.00
Average	3.88	3.92	4.09	4.23	3.91
Malowan Method					3.87
					3.92
					3.92
					3.90

Mr. Roettger ran a number of extractions on Sample No. 3 in Copes bags for 5 hours with no repounding, with the following results: 18.98; 18.66; 18.63; 19.10; 19.30; 18.44; average, 18.81.

Results on Seed Sample No. 5

	Forbes	Malowan	Picard	Roettger	Strack
MOISTURE					
Regular Method	9.00	9.20	8.98	8.90	8.88
	9.00		8.90	8.98	9.96
					8.60
					9.18
					8.96
Average	9.00	9.20	8.94	8.94	9.11
Malowan Method					9.48
Whole seed		8.72			
OIL					
Smalley-Copes	20.25	20.09	19.40	20.04	20.18
	20.30	19.79	20.22	20.20	19.82
	20.30	19.89	19.82	20.34	
	20.40				
	20.70				
	20.75				
Average	20.45	19.92	19.81	20.19	20.00
Picard Cylinders	20.30	20.07	19.98	20.54	19.70
		19.77	20.16	20.38	20.54
		19.79	19.76	19.86	20.56
Average	20.30	19.88	19.97	20.26	20.27
Malowan Method					19.51
					20.34
					20.25
					20.03
Acid treatment					
5 hrs. with two		20.06	19.98	20.04	20.04
poundings		19.98	20.02	20.10C. bag	20.24
Average	20.30	20.02	20.00	20.07	20.14
P. cylinder					21.08
5 hrs. with		19.84			
1 pounding		19.74			
Average		19.79			
On repounding		0.22			
& reextracting		0.24			
Average		0.23			
		20.06			
Total oil		19.98			
Average		20.02			
AMMONIA					
1.7034 grams	3.95	3.83	4.07	4.00	4.25
	3.98	4.09	4.13	4.07	4.14
	4.00	4.09			4.10
	4.08				4.20
	4.10				
Average	4.02	4.00	4.10	4.03	4.17
Malowan Method					4.16
					4.11
					4.14

SOAPSTOCK COMMITTEE REPORT 1921-1922

By W. J. Reese, Peet Brothers, Kansas City, Kans. Chairman

Your Soap-stock Committee has rather an unsatisfactory report to make this year.

The Committee of last year, under the chairmanship of Mr. C. P. Long, submitted recommendations for the radical revision of the present filtration method for the determination of Total Fatty Acids in Soap-stock by the substitution of an extraction method. (See Soap-stock Committee report, COTTON OIL PRESS, Vol. V. No. 1.) Because of this probable change of method, it was necessary to obtain a ruling on the proposed changes before it would be possible to proceed with the analytical work on a satisfactory basis.

On this account, the Committee work has centered upon urging the adoption of the proposed new method with the hope of obtaining a final ruling in sufficient time to use it as a basis for the work for the current year. The new method has been approved by the