Status of Meal and Cake Grading

A. S. RICHARDSON, Chairman of the Meal Grading Committee writes as follows:

"Although we are making progress in the study of meal grading, a large proportion if not all of the new season's meal will be graded before any official action can be taken by all the committees concerned with the development and adoption of the method.

"A search through the various journals suggests that just enough has been published on this subject to be confusing and this idea is further confirmed by several letters which I have received. Therefore we believe that a statement should be published in OIL & FAT INDUSTRIES, explaining the present status of meal and cake grading.

"I have taken the matter up with the other members of the committee on method of color comparison and we have prepared a statement which we enclose herewith in duplicate. Although it is merely a statement of the present situation and not a new committee report, we suggest that you pass it along for publication as a news item."

Interstate Rule 102 provides that prime cottonseed cake and meal of 41 per cent or higher protein content shall not be darker in color than Munsell Color Standard "2 yellow 6/5" and that 36 per cent protein cake and meal shall not be darker than Munsell Color Standard "10 yellow 5/5."

The Interstate rules do not yet provide a specific procedure for making the comparison between color standard and sample of meal or cake. In November, 1926, a special A.O.C. S. committee proposed two such procedures in a report which was subsequently published in the May 1927 issue of OIL & FAT INDUSTRIES (pages 187-8). Neither of these methods was definitely adopted, although it was the consensus of opinion of the Chemists' Committee of the Interstate that the simpler method (reproduced below) was preferable as a tentative method for the remainder of the 1926-7 season.

The special committee for study of methods of color comparison has been continued for another year and the adoption of an official procedure is awaiting the further study of that committee. In the absence of an official method, the most satisfactory procedure for general use will be method II of the report in the May issue of OIL & FAT INDUSTRIES. Method I of the same report, based upon the use of a rotating cup, has been adopted by the Texas Cotton Seed Crusher's Association in Rule 271, Supplement No. 1, 1927-8. The rotating cup method is believed to give more satisfactory results in doubtful cases but is less simple of manipulation. Method II, which was tentatively favored by the Chemists' Committee of the Interstate last season, is as follows:

"a. Meal. The meal to be graded should be placed in the center of a gray sheet or board at least eight inches square; it should be flattened out to make a level circle about three or four inches across, and a clean, one-inch square of the color standard laid on the center of

the meal. The meal and standard, lying in a horizontal plane, should then be observed, in good daylight, from a position directly above them and at least 36 inches distant. For making close decisions, it is best to lay the board on the floor and observe it from a standing position directly above. To be graded "prime," the meal must be as light or a lighter shade than the standard. If darker, it must be graded "off" in color.

"b. Cake. A representative portion of the cake to be graded should be ground so that 85 per cent will pass a 20-mesh screen.* Portions of sample used for screen test should not be used for color comparison. The ground sample should be graded as for meal.

"Note: Any samples of meal containing coarse particles should be ground to the standard for cake and this fact should be stated in the report."

* The committee report as published adds the words: "and 75% through 30 mesh." The committee has since concluded that the double mesh specification is objectionable.

Grading Standard Glasses

To All Members:

The Governing Board has employed Miss Geraldine K. Walker, formerly of the Munsell Color Co., as "Research Associate" at the Bureau of Standards to conduct "An Investigation into the Uniformity of the Lovibond Glasses at present in use in the Vegetable Oil Industry."

As a part of that work it will be necessary to grade all glasses submitted, and in due time Mr. A. W. Putland, who has consented to handle the matter will invite the members to send him any glass which they wish graded at $1.50 a glass. He will issue A. O. C. S. certificates of the value of each glass submitted.

We feel that this is a step in the right direction and that the results of this work will be of great benefit to the industry as a whole.

H. P. TREVITHICK,
President.

F. F. A. Co-operative Tests

This co-operative work has been conducted during the past season by the Refining Test Committee. The main object was to stimulate interest in getting correct F.F.A. tests because of their importance in refining work. The F.F.A. test is the first step in refining a crude oil, and if incorrectly made it will result in selecting an improper lye for refining, and this in turn will lead to improper refining results. Ten samples were sent out. The first went to nineteen laboratories, number two to twenty-nine, and the balance to thirty-two laboratories. Interest, however, fell off to such an extent that while all nineteen laboratories reported on the first sample, not more than twenty out of thirty-two reported on any of the last three samples.

The accompanying table shows the total "points off" from the accepted average for each laboratory. A tolerance of two hundredths per cent from the accepted average is allowed in all cases, the same as in the Smalley Foundation work. This means that any test within .02% of the accepted average is considered perfect.

The names of the five laboratories making the best showing together with the average "points off" are also shown in a separate table. Laboratory No. 10 at the head of the list reported on eight

samples, and on three of these obtained perfect results by both brine and alcohol, and was also perfect on brine in a fourth sample, and perfect on alcohol on a fifth sample, thus scoring eight perfect results out of a possible sixteen. The other four laboratories on this list each scored from four to nine perfect results. All five of these laboratories averaged less than .05% off from the accepted average.

Our conclusions from this season's work are as follows:

(1) There is altogether too much variation among the different laboratories for such a simple test. This indicates a lack of care. There is no reason why all laboratories cannot average less than ten points off by using proper care.

(2) Alcohol gives slightly higher results on every sample in hands of every operator. The average of all ten samples shows .12% higher.

(3) Alcohol shows more concordant results among the various laboratories than brine on every sample without exception.

C. B. CLUFF.

Total "Points Off" from "Accepted Average"

A "tolerance" of ± .02% from "accepted average" allowed in all cases

	Brine		Alcohol		Combined		
Lab.	Samples Reported	Points Off	Samples Reported	Points Off	Sample Reported	Points Off	Av. Pts. Off
1	4	20	8	61	12	81	6.8
2	9	173	9	161	18	334	18.5
3	9	228	9	230	18	458	26.1
4	5	114	4	55	9	169	18.8
5	9	50	9	22	18	72	4.0
6	7	68	7	57	14	125	8.9
7	10	94	10	69	20	163	8.2
8	3	33	3	33	6	66	11.0
9	9	28	9	39	18	67	3.7
10	8	15	8	38	16	53	3.3
11	10	122	10	61	20	183	9.2
12	10	112	10	155	20	267	13.4
13	8	194	8	69	16	263	16.4
14	9	96	9	69	18	165	9.2
15	7	112	8	26	15	138	9.2
16	4	80	4	43	8	123	15.4
17	9	67	9	63	18	130	7.2
18	9	67	9	45	18	112	6.2
19	7	160	7	52	14	212	15.1
20	9	192	9	100	18	292	16.2
21	2	23	2	4	4	27	6.8
22	3	24	3	9	6	33	5.5
23	3	52	3	48	6	100	16.7
24	5	122	3	87	8	209	26.1
25	9	517	9	526	18	1,043	57.9
26	8	90	8	80	16	170	10.6
27	1	24	1	9	2	33	16.5
28	7	159	7	89	14	248	17.7
29	8	44	9	34	17	78	4.6
30	1	32	1	25	2	57	28.5
31	7	21	7	26	14	47	3.4
32	7	201	7	159	14	360	25.7

Best Laboratory Work

Consideration given only to laboratories reporting on at least 70% of the samples sent out

Order	Lab. No.	Name	Samples	Av. Pts. Off
1	10	P. & G. Co., Staten Island, N. Y.....	16	3.3
2	31	P. W. Tompkins, S. F., Col.....	14	3.4
3	9	P. & G. Co., Ivorydale, Ohio.....	18	3.7
4	5	H. P. Trevithick, New York.....	18	4.0
5	29	Lever Bros. Co., Cambridge, Mass....	17	4.6

The Editor,
OIL & FAT INDUSTRIES.
Sir:

Mr. Chapman's invitation for correspondence on his article in the May issue of OIL & FAT INDUSTRIES brings this from me, since accuracy of results cannot be overestimated.

In Mr. Chapman's directions under the heading of "Determination" the addition of the small piece of paraffine before digestion, that he calls for, is entirely useless, if not detrimental. Its effect is to retard the oxidation of the sample and to deposit carbon in the neck of the digestion flask.

The directions to digest only 45 minutes leads to low results as all the nitrogen of the protein has not by this time, been converted into ammonia. If the digestion is stopped at the end of 45 minutes, only 94% to 97% of the total protein nitrogen has been converted into ammonia, and the report of the protein content is correspondingly low. Where 50 to 80 samples were run per day, the time of digestion being 1 hour and 10 minutes, fully one-third of the samples failed to check within 0.3% protein.

It takes a digestion of an hour and a quarter under the best regulated conditions to effect the change of all the protein nitrogen into ammonia, and where some burners do not give as hot a flame as others in the bank, the digestion should be carried on for an hour and a half. After the boiling acid begins to clear, the heat should be increased until the condensing ring is about an inch up in the neck of the flask.

Guard against the use of too much potassium (or sodium) sulfate. Five grams of potassium sulfate will give the same results as ten grams, but if more than ten grams are used with twenty-five cc of sulfuric acid, a slightly lower percent of nitrogen will be found.

Another caution, do not use such accelerators such as permanganates or perchlorates, as is sometimes recommended. These will oxidize some of the ammonia and lead to low results. Very truly yours,

PAUL L. MENAUL.

The Editor,
OIL & FAT INDUSTRIES.
Sir:

I have received Mr. Menaul's letter regarding the publication of my method for protein in your May issue. I have checked this method many times but in order to give you some late comparative figures I had our Mr. John T. Scott run check meal sample No. 13 four different ways. Probably the sample had dried out slightly as the ammonia was slightly higher than accepted average for this sample.

In making the following determinations the weight taken was 2.5g. The weight of sodium sulphate used 7.0g. Other variables are shown in the table which follows:

Time of Dig.	Mercuric Oxide used grams	Titr. cc			Protein %	Ave. %	Ammonia %
30 Minutes	1.00	5.4	50 — 5.4	=	44.60		
30 Minutes	1.00	5.4	50 — 5.4	=	44.60	44.67	8.69
30 Minutes	1.00	5.2	50 — 5.2	=	44.80		
45 Minutes	.70	5.4	50 — 5.4	=	44.60		
45 Minutes	.70	5.4	50 — 5.4	=	44.60	44.67	8.69
45 Minutes	.70	5.2	50 — 5.2	=	44.80		
75 Minutes	.70	5.4	50 — 5.4	=	44.60		
75 Minutes	.70	5.1	50 — 5.1	=	44.90	44.73	8.71
75 Minutes	.70	5.3	50 — 5.3	=	44.70		
3 Hours	.70	5.2	50 — 5.2	=	44.80		
3 Hours	.70	5.2	50 — 5.2	=	44.80	44.80	8.72
3 Hours	.70	5.2	50 — 5.2	=	44.80		

From the results obtained it will be seen that the difference between 45 minutes digestion and one hour and fifteen minutes digestion is very slight. Using a larger amount of mercuric oxide also decreases the time needed for digestion without any resultant loss of accuracy. Three hours' digestion does however, seem to give better checks but for most purposes the shorter time should suffice. It was not the intention to intimate that all samples should be digested for 45 minutes only. As Mr. Menaul has indicated, flames and other conditions vary and the time of digestion should always be left to the judgment of the operator. We mentioned 45 minutes because we had found that this was about the average for our own work.

We have never found that the use of paraffine had anything to do with the time or completeness of oxidation. With certain classes of material it helps to prevent foaming in the digestion and it always prevents foaming in the distillation, especially if there has been added an excess of caustic soda solution. It is merely used as a precautionary measure and if proper quantities of reagents are used, the paraffine may be dispensed with.

Since the chief use of the sulphates of Sodium or Potassium is to raise the boiling point of the digestion there is no object in using an excess. In order to get the greatest efficiency the proper amount must be used.

I agree that the use of accelerators such as mentioned by Mr. Menaul is to be condemned.

I should like to hear from anyone who has suggestions or criticisms to make on this article or on the method.

Very truly yours,

R. M. CHAPMAN.

Referee Applicants

Applicants for Referee Chemist Certificates are Herman A. Nester, San Antonio, Texas, (second publication), and (first publication), A. W. Horrell, Jackson, Miss.