PROCEEDINGS OF THE SIXTEENTH ANNUAL CONVENTION

SMALLEY FOUNDATION

Check Meal Samples for 1924-1925

Ammonia Committee Report

By H. C. Moore

In the following tables will be found a summary of the coöperative analytical work for 1924–25. These tables tell practically the whole story with reference to the results reported on the check meal samples, and very few additional comments are necessary.

During the past year seventy-five collaborators have participated, as compared with seventy-eight in each of the two previous years.

Table No. 1 shows the standing of the 29 collaborators who have determined oil in all of the samples. Last year 36 collaborators made the same determinations, as compared with 38 the previous year.

Table 1

Standing for Oil Results (30 Samples)

Place no.	Analyst no.	Points off	Av. error per sample	Efficiency per cent
1	26	28	0.0093	99.880
2	74	31	.0103	99.848
	80	31	.0103	99.848
4	6	39	.0130	99.808
5	37	43	.0143	99.789
6	20	45	.0150	99.778
7	21	47	.0157	99.768
8	19	54	.0180	99.734
9	5	73	.0243	99.640
10	23	83	.0277	99.590
11	22	90	.0300	99.556
12	42	93	.0310	99.542
13	4	103	.0343	99.493
	39	103	.0343	99.493
15	73	109	.0363	99.463
16	62	114	.0380	99.438
17	8	118	.0393	99.419
	49	118	.0393	99.419
19	79	146	.0487	99.280
20	25	152	.0507	99.250
21	65	157	.0523	99.227
22	3	200	.0667	99.013
23	35	206	.0687	98.885
24	40	222	.0740	98.905
25	67	274	.0913	98.648
26	2	317	.1057	98.447
27	70	398	.1327	98.036
28	61	469	.1563	97.688
29	46	521	.1737	97.431

Table No. 2 shows the corresponding standing of the 42 collaborators who reported ammonia determinations on all the samples, as compared to 50 for last year and 56 for the previous year.

TABLE 2

STANDING FOR AMMONIA RESULTS (30 SAMPLES)

Place no.	Analyst no.	Points off	Av. error per sample	Efficiency per cent
1	63	10	0.0033	99.956
2	80	13	.0043	99.942
3	74	15	.0050	99.933
4	12	17	.0057	99.924
5	31	21	.0070	99.906
6	32	23	.0077	99.897
7	20	25	.0083	99.889
8	6	27	.0090	99.880
9	4	33	.0110	99.853
10	37	35	.0117	99.843
11	11	36	.0120	99.839
12	23	37	.0123	99.835
13	49	38	.0127	99.830
	66	38	.0127	99.830
15	39	39	.0130	99.826
16	16	41	.0137	99.817
17	19	43	.0143	99.809
18	35	44	.0147	99 803
19	8	47	.0157	99.790
	62	47	.0157	99.790
21	21	50	.0167	99.776
	42	50	.0167	99.776
23	2	51	.0170	99.772
24	14	55	.0183	99.755
	65	55	.0183	99.755
26	26	56	.0187	99.749
	79	56	.0187	99.749
28	3	58	.0193	99.741
29	22	59	.0197	99.736
	40	59	.Q197	99.736
31	73	62	.0207	99.723
32	38	70	.0233	99.688
	44	70	.0233	99.688
34	67	72	.0240	99.679
35	27	74	.0247	99.669
36	5	83	.0277	99.629
	25	83	.0277	99.629
38	47	92	.0307	99.588
39	46	109	.0367	99.514
40	29	117	.0390	99.478
41	61	130	.0433	99.420
42	30	211	.0703	99.058

Table No. 3 shows the laboratory standing for both oil and ammonia results for the 28 collaborators who completed all determinations, as compared with 36 last year and 38 the year before.

TABLE 3

LABORATORY STANDING FOR BOTH OIL AND AMMONIA RESULTS

Place No.	Analyst No.	Efficiency per cent	Place No.	Analyst No.	Efficiency per cent
1	80	99.895	15	49	99.625
2	74	99.892	16	62	99.614
3	6	99.844	17	8	99.605
4	20	99.834	18	65	99.593
5	37	99.816	19	73	99.515
6	26	99.815	20	79	99.491
7	19	99.772	21	25	99.440
8	21	99.772	22	35	99.344
9	23	99.713	23	3	99.377
10	4	99.673	24	40	99.321
11	39	99.660	25	67	99.164
12	42	99.659	26	2	99.110
13	22	99.646	27	61	98.554
14	5	99.635	28	46	98.478

Table No. 4 summarizes the results of the other collaborators who failed to report on all the samples but whose results deserve recognition. It will be noted from Table 4 that several collaborators reported on all but one or two samples. In several cases this was due to failure to receive the samples, or the reports went astray in the mail. The Chairman has been as liberal as he felt possible in accepting late reports; in fact, all those received up to the time the report went to the printers on Tuesday morning have been accepted.

TABLE 4

RESULTS OF OTHER COLLABORATORS WHO FAILED TO REPORT ON ALL SAMPLES BUT WHOSE RESULTS DESERVE RECOGNITION

Analyst No.	No. of samples reported	Total points off	
		Oil	Ammonia
9	24	..	105
10	28	..	73
13	29	..	76
15	29	..	82
17	28	..	91
24	21	53	15
28	23	..	81
33	26	61	19
34	28	..	145
41	28	..	63
43	29	131	32
64	23	..	79
70	28	398 (30 samples)	144 (28 samples)

71	25	108	45
72	27	121	23
75	25	..	52
77	29	..	73
78	20	41	21
81	25	..	71
82	29	..	88

The prize awards for the best work done on these samples were outlined in the pamphlet sent out at the beginning of the year and originally printed in the *Cotton Oil Press* in 1923.

The winners of these prize awards are as follows:

The laboratory cup for the highest average for both oil and ammonia, E. H. Tenent, International Sugar Feed No. Two Co., Memphis, Tenn., Analyst No. 80, whose per cent efficiency is 99.895. The certificate for second place, L. B. Forbes, Dixie Cotton Oil Co., Memphis, Tenn., Analyst No. 74, whose per cent efficiency is 99.892. The certificates for the highest averages for the ammonia results: (1) Gascoyne & Co., Baltimore, Md., Analyst No. 63, whose per cent efficiency is 99.956; (2) E. H. Tenent, Memphis, Tenn., Analyst No. 80, whose per cent efficiency is 99.942. The certificates for the highest average for the oil results: (1) Southwestern Laboratories, Dallas, Tex., Analyst No. 26, whose per cent efficiency is 99.880; (2) L. B. Forbes, Analyst No. 74, and E. H. Tenent, Analyst No. 80, both of Memphis, Tenn. (tied for second place), whose per cent efficiency is 99.848.

The Society has decided that with the exception of those who have won the grand prize or certificates, the identity of the collaborators shall not be disclosed; that each shall be assigned a number and no key to these numbers shall be given out.

The method for determining the standing of the various collaborators and their per cent efficiency is the same as was used the past two years, and given in detail in the Jan., 1923, *Cotton Oil Press*, **6**, No. 9, p. 33.

It will be observed from Tables Nos. 1 to 3 that the highest averages this year are slightly lower than those last year. One reason for this difference is that the average of the accepted averages this year is slightly lower than for last year, which by the method of calculation makes the same number of "points" this year show a slightly lower percentage efficiency.

Judging from the comments of the collaborators during the past year there have been but few complaints on the uniformity of the samples. Several have felt that one or two of the samples were not uniform, but on the whole complaints have been very few. A few reports have come to the chairman that collaborators have failed to receive some of the samples, and for this reason have been unable to make their reports. Had they

notified Mr. R. F. Monsalvatge to this effect in proper time, as per notation on the report for sample No. 17, he could have sent them duplicate samples.

The Ammonia Committee acting on the suggestion of Mr. C. A. Butt decided to send to all collaborators a questionnaire inquiring into the details of the method used in the determination of ammonia. Mr. Butt's summary of the replies received follows in full:

"During the year a series of questions, relating to the various details in methods for the determination of ammonia in use at the laboratories participating in the meal series known as the SMALLEY FOUNDATION, were prepared and in the form of a questionnaire were mailed to each of the collaborators.

"The purpose of this work being mainly to show the trend in the methods and if possible to bring out new suggestions in connection with this determination.

"Fifty-one replies were received and the data is summarized in the following:

1. *Name of Method*

Number using Kjeldahl-Gunning mercury method (H_2SO_4, sodium or pot. sulfate and mercury)	40
Number using Kjeldahl-Gunning copper method (H_2SO_4, sodium or pot. sulfate and copper)	10
Number using Kjeldahl method (H_2SO_4, mercury and $KMnO_4$ to complete oxidation)	1

2. *Weight of Sample Taken*

Number using less than 1 gram	4
Number using 1 to 2 grams	46
Number using 2.5 grams	1

3. *C. C. Sulfuric Acid Used in Digestion*

Number using 7 cc	1
Number using 15 cc	1
Number using 20 cc	3
Number using 25 cc	30
Number using 30 cc	13
Number using 35 cc	3

4. *Sodium or Potassium Sulfate*

Number using sodium sulfate	18
Number using potassium sulfate	31
Number using either	1

5. *Quantity of Sodium or Potassium Sulfate*

Number using 5 grams or less	2
Number using 6 to 8 grams	6
Number using 9 to 10 grams	35
Number using 11 to 12 grams	2
Number using 13 grams or more	5

6. Grams Mercury or Mercuric Oxide

Number using 0.5 to 0.6 gram mercury	20
Number using 0.7 to 1 gram mercury	11
Number using 0.25 gram mercuric oxide	1
Number using 0.5 to 0.6 gram mercuric oxide	2
Number using 0.7 to 1 gram mercuric oxide	7

7. Grams Copper or Copper Sulfate

Number using 0.1 to 0.2 gram copper	2
Number using 0.2 to 0.5 gram copper	4
Number using 0.5 to 0.7 gram copper	2
Number using 0.2 to 0.3 gram copper sulfate	1
Number using 0.5 to 0.7 gram copper sulfate	1

8. Time of Digestion—Total

Number digesting 3/4 hour or less	1
Number digesting from 3/4 to 1 hour	8
Number digesting from 1 to 1 1/2 hours	17
Number digesting from 1 1/2 to 2 hours	14
Number digesting from 2 to 3 hours	10
Number digesting 3 hours or more	1

9. Time of Digestion—After Clear

Number digesting 1/4 hour or less	5
Number digesting 1/4 to 1/2 hour	13
Number digesting 1/2 to 1 hour	13
Number digesting 1 to 2 hours	8
Number digesting 2 hours or more	2
Number not stating	10

10. Rate of Boiling during Digestion

Gently or slowly	7
Medium	9
Briskly	34
Various rates	1

11. Time of Distillation

Number distilling 1/2 hour or less	15
Number distilling 1/2 to 1 hour	31
Number distilling 1 hour or more	4
Number not stating	1

12. Using Gas or Electric Heat

Number using gas for both digestion and distillation	33
Number using electric for both digestion and distillation	16
Number using electric for digestion gas for distillation	2

13. Sodium or Potassium Sulfide

Number using sodium sulfide	19
Number using potassium sulfide	21
Number using neither (copper method contestants)	10
Number using neither (sodium thiosulfate used instead)	1

14. Quantity of Sodium or Potassium Sulfide

Number using solution containing 0.4 gram	1
Number using solution containing 0.5 to 0 7 gram	3
Number using solution containing 0.7 to 1 gram	26
Number using solution containing 1.0 to 1.5 grams	8
Number using solution containing 1.5 to 2.2 grams	2

15. To Prevent Bumping during Distillation

Number using zinc	49
Number using talcum	1
Number not using anything (copper method)	1

16. Connecting Bulbs

Number using Kjeldahl, Hopkins or modification	33
Number using bulbs of special design, including Barrow-Agee's, Clark's, Davisson Scrubber, Law & Co., and Magruder's	16
Number using none	1
Number not stating	1

17. Condenser Tube or Coil—Inside Diameter

Number using tube $^3/_{16}$ to $^1/_4$ inch diameter	20
Number using tube $^5/_{16}$ to $^3/_8$ inch diameter	23
Number using tube $1^1/_2$ inch diameter	1
Number using miscellaneous sizes	1
Number not stating	1
Number using $^7/_{16}$ to $^1/_2$ inch diameter	5

18. Condenser Tube or Coil—Length

Number using from 1 to 2 ft	13
Number using from 2 to 3 ft	20
Number using from 3 to 4 ft	9
Number using from 4 to 5 ft	1
Number using 5 ft. or more	7
Number not stating	1

19. Capacity of Kjeldahl Flask Used

Number using 500cc. flasks	15
Number using 650cc. flasks	8
Number using 700cc. flasks	1
Number using 800cc. flasks	27

20. Standard Acid Solution—Kind

Number using standard solution of sulfuric acid	45
Number using standard solution of hydrochloric acid	6

21. Standard Acid Solution—Strength

Number using normal solution	3
Number using $^1/_2$ normal solution	34
Number using $^1/_4$ normal solution	2
Number using $^1/_5$ normal solution	4
Number using various other strengths	8

22. Standard Alkali Solution—Kind

Number using standard solution of sodium hydroxide	44
Number using standard solution of potassium hydroxide	3
Number using standard solution of ammonium hydroxide	3
Number using none (ammonia absorbed in nearly saturated solution of boric acid and titrated direct with standard acid using brom-phenol-blue indicator)	1

23. Standard Alkali Solution—Strength

Number using $^1/_2$ normal solution	5
Number using $^1/_4$ normal solution	23
Number using $^1/_6$ normal solution	5
Number using $^1/_8$ normal solution	1
Number using $^1/_{10}$ normal solution	6
Number using various other strengths	11

24. Kind of Indicator

Number using methyl red	20
Number using sodium alizarine sulfonate	15
Number using cochineal	13
Number using methyl orange	2
Number using brom-phenol-blue	1

25. Reagents Used in Standardizing Solutions

Number using sodium carbonate and ammonium sulfate	11
Number using sodium carbonate and ferrous ammonium sulfate	1
Number using sodium carbonate alone	3
Number using ammonium sulfate alone	2
Number using ammonium chloride alone	1
Number using benzoic acid alone	2
Number using acid potassium phthalate alone	1
Number using acid potassium phthalate and sodium carbonate	2
Number using potassium bitartrate and succinic acid	1
Number using barium chloride alone	14
Number using barium chloride and ammonium sulfate	1
Number using barium chloride and sodium carbonate	4
Number using silver nitrate alone	3
Number using silver nitrate and sodium carbonate	2
Number using silver nitrate, sodium carbonate and ammonium sulfate	1
Number using silver nitrate, calcium carbonate and ammonium chloride	1

From these data it will be noted that there are in use procedures for the ammonia determination with numerous variations as to detail.

It may be interesting to note that by taking the most popular manner in which each step in the determination is carried out and making a method in this way, the following would be the procedure thus constructed:

"Weigh 1 to 2 grams of sample into an 800 cc. Kjeldahl flask and add 25 cc. sulfuric acid (sp. gr. 1.84), 9 to 10 grams potassium sulfate and 0.5 to 0.6 gram mercury. Heat to boiling and continue digestion, boiling briskly, from 1 to $1^1/_2$ hours. Cool, dilute with water and precipitate

TABLE 5

SUMMARY OF REPLIES TO QUESTIONNAIRE FROM THE TEN HAVING THE HIGHEST AMMONIA STANDING

Details of analysis	Analyst's number									
	No. 63	No. 80	No. 74	No. 31	No. 6	No. 4	No. 11	No. 23	No. 49	No. 66
Wt. of samp. gr.	1.7036	1.401	1.7034	1.000	1.7034	1.7034	1.7034	1.401	1.7034	2.0000
Cc. H_2SO_4	30	25	25	20	25	25	30	25	30	30
Gr. K_2SO_4	10	..	8–10	..	10	10	8–10	18–20	9–10	..
Gr. Na_2SO_4	..	9	..	..	..	..	..	..	..	10
Gr. Cu added	..	..	..	0.7	..	..	..	..	..	..
Gr. $KMnO_4$ added	..	..	..	.25	..	..	..	..	..	..
Gr. Hg	..	0.5	0.5	..	0.5	0.5	..	0.6	0.5–0.6	0.7
Gr. HgO	1	..	..	..	..	..	.8	..	..	..
Total time dig. hrs.	2	1	1	3	1 1/2	1 1/4	2 1/2	1 1/2	1 1/2–2	1 3/4
Time after clear, hrs.	1 1/2	..	..	1 1/2	..	1/2	1 1/2	3/4	1–1 1/4	..
Rate of boiling	Brisk	Brisk	Brisk	Brisk	Brisk	Brisk	Slow	Med.	Med.	Brisk
Kind of heaters, digest.	Gas	Elec	Elec.	Gas	Elec.	Gas	Elec.	Elec.	Elec.	Gas
Kind of heaters, distil.	Gas	Elec.	Elec.	Gas	Elec.	Gas	Elec.	Gas	Elec.	Gas
Time for distn., hrs.	1	1/2	3/4–1	3/4	1 1/4–1 1/2	1/4	1 1/2	40 m.	30–40 m.	1
Gr. K_2S	1	0.9	1.5	(a)	1	1	..	..	..	..
Gr. Na_2S	..	..	..	..	..	..	1	1.25	1.2	1
To prevent bumping	Zinc	Zinc	Zinc	Zinc	Zinc	Zinc	Zinc	Zinc	Zinc	Zinc
Connecting bulbs	E&A1216	Hopk.	SCo	Hopk.	B&A	Hopk.	Kjel.	Kjel.	Hopk.	Double
Diam. condens. tube, in.	1/4	1/2	3/8	1/4	3/8	3/8	1/4	3/8	1/4	3/8
Length cond. tube, ft.	2	3 1/2	5	2 1/2	4	2 1/2	2	2 1/2	3	2

Size flask	500	800	800	700	800	800	650	800	650	500
Standard H_2SO_4	..	N/2	N/2	..	N/2	N/2	N/2	N	N/2	N/1.7
Standard HCl	N	..	..	N/2(*b*)	..	..	..	..	..	..
Standard NH_4OH	..	..	..	N/5(*c*)	..	..	..	..	..	..
Standard NaOH	N/10	N/4	N/4	..	N/4	N/4	N/8	N/5	..	N/1.7
Indicator	SAS	MR	MR	MR	MR	MO	MR	MR	BPB	MR
Method standardizing acid	Ag	FAS	APT	Ag	SC	SA	SC	NC	SA	Ba

(*a*) Uses hypo.
(*b*) About N/2.
(*c*) About N/5.

SAS—Sodium alizarin sulfonate
MR—Methyl red
Ag—Silver nitrate
Ba—Barium sulfate
APT—Acid potassium phthalate
SA—Ammonium sulfate (ACCS standard)
BPB—Brom-phenol-blue
MO—Methyl orange
FAS—Ferrous ammonium sulfate
SC—Sodium carbonate.

mercury by adding solution containing 0.7 to 1 gram potassium sulfide. Add a few particles of granulated zinc, make alkaline and distill ammonia through 2 to 3 ft. of condenser tube of $^5/_{16}$ to $^3/_8$ in. inside diam. into a $^1/_2$ normal solution of sulfuric acid which has previously been standardized by precipitation with barium chloride. Use methyl red as indicator and titrate excess acid with $^1/_4$ normal sodium hydroxide."

In submitting the above summary Mr. Butt suggested that it might be of interest to note how closely the average or cross section method which he devised from the replies was followed by the leaders in the ammonia series. Not all of the leaders replied to the questionnaire, but Table No. 5 gives a summary of replies from the ten highest. It is interesting to note that the details used by these ten collaborators are in the main very close to the average method used by all, and it is believed that Table No. 5 will prove interesting in connection with Table No. 2 and the summary to the questionnaires.

Committee: H. C. MOORE (Armour Fert. Works, Chicago): C. A. BUTT; L. B. FORBES; JOHN MALOWAN; H. B. BATTLE.

DETERGENTS COMMITTEE REPORT FOR 1924-25

BY ARCHIBALD CAMPBELL

The Detergents Committee of the American Oil Chemists' Society includes in its membership the members of the Soap and Soap Products Committee of the American Chemical Society, representatives of most of the large soap manufacturers, also of the Bureau of Standards, as well as several large soap consumers of the country. It is thus an interlocking committee organized with a view of correlating the work done by the Soap and Soap Products Committee on Methods of Sampling and Analysis with the work done by the Soap Committee of the Soap Section of the American Specialty Manufacturers' Association working in conjunction with the Federal Specifications Board on Soap Specifications. The Soap and Soap Products Committee is likewise an interlocking committee with the Glycerine and F. A. C. Committees of the American Chemical Society. By this interlocking system of committees it is hoped to avoid duplication of efforts and effect correlation of results.

As it was late in the season before the Detergents Committee was organized it confined its efforts this year to the following work:

1. Organization.
2. Discussion and criticism of the Standard Methods for the Sampling and Analysis of Commercial Soaps and Soap Products as adopted by the American Chemical Society.
3. Presentation of these Standard Methods to the Uniform Method