

# Report of the Moisture Committee

BY W. H. IRWIN, CHAIRMAN

**A**T the 1927 May meeting, the Moisture Committee was asked to continue the study made the previous year of the oven method, using a jacketed oven with a glycerin solution in the jacket and electrically heated, and also the Kingman Distillation Method after modifying, as suggested in the previous report of the Committee.

It was decided to send out six samples to the members of the Committee for the moisture determination using the Kingman Distillation Method as modified. The results obtained on the first two samples, after the first modification of the method, were not satisfactory, and the method, as finally modified, is as follows and was applied to the last four samples sent out:

## Kingman Distillation Method (Modified) for Cotton Seed Meal, Cake, etc. (For Moisture Only)

### *Apparatus:*

Kingman Distillation Apparatus as shown in sketch. The apparatus should be thoroughly cleaned with cleaning solution before using. Follow by washing in alcohol and drying in an oven.

### *Reagents:*

Acetylene tetrachloride. Benzol.

### *Determination:*

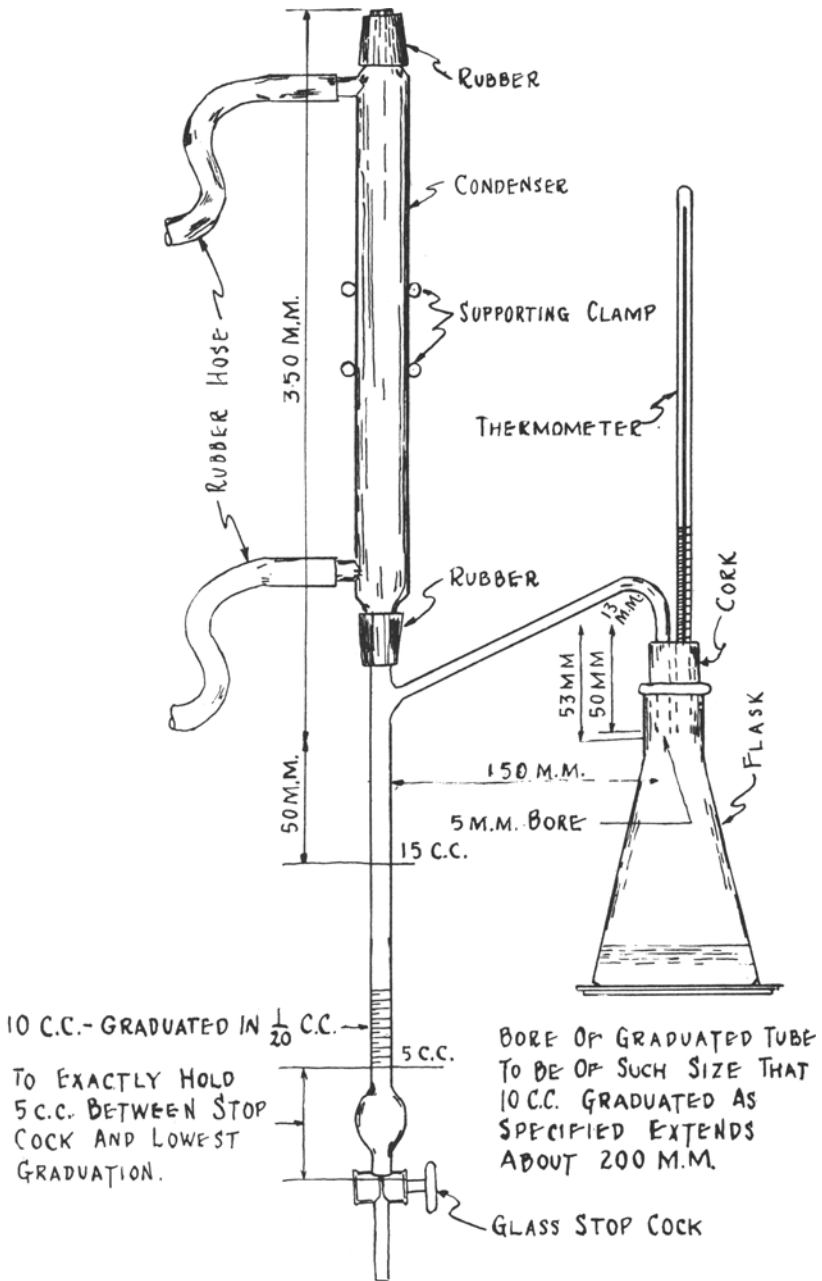
Weigh 40 grams of the sample into a 500 cc. Erlenmeyer flask. Add 125 cc. of acetylene tetrachloride to the sample in the distillation flask, connect with Kingman Distillation tube and condenser and distil as follows:

Heat with a Bunsen flame about 1 inch to 1¼ inch high placed so that the flame strikes the gauze protecting the flask. A thermometer calibrated at each degree should pass through the stopper of the Erlenmeyer flask with its bulb above the liquid. When the temperature rises to about 135°C. take temperature readings at one minute intervals using a watch for accurate timing. Draw off the solvent from time to time when the Kingman tube gets too full. When three successive temperature readings at one minute intervals show the same temperature, shut off the flame and allow the apparatus to cool. The constant boiling temperature is about 142°C. and should be reached in 30 minutes after heating is begun. A little practice and observation will enable the operator to judge the proper amount of heat necessary to finish the distillation in that time.

If the distillation is carried on properly, the water in the collecting tube will remain cool enough so that there is no danger of its redistilling.

After the apparatus has cooled, wash down the condenser and connecting tube into the graduated tube of the apparatus with a little benzol, pushing down any drops of water adhering to the upper part of the condenser tube with a copper wire with a loop on the end. When the water has settled completely, read the column of water, making due allowance for meniscus, and calculate the percentage.

The compilation of results given



Kingman's Moisture Apparatus

below shows the results obtained by the several members of the Committee:

2. Official aluminum moisture dish with cover, dimensions 2 inches outside diameter— $\frac{3}{4}$  inch

*A. O. C. S. CO-OPERATIVE CHECK MEAL RESULTS, 1927-28*

	1	Kingman Distillation Method				6
		2	3	4	5	
C. H. Cox, Barrow-Agee Lab's.....	9.37	11.62	9.35	9.12	9.62	9.75
N. C. Hammer, Southwestern Lab's.	9.75	11.00	9.13	8.89	9.75	10.25
W. D. Hutchins, Southern Cotton Oil Co. ....	10.52	13.12	9.00	9.06	9.29	10.12
C. P. Long, Globe Soap Co.....	9.31	11.37	9.52	9.48	10.14	10.75
M. L. Sheely, Armour Soap Works..	9.62	12.16	9.87	8.68	9.31	10.56
E. H. Tenent, International Sugar Feed Co. ....	8.47	10.50	9.25	9.08	9.38	9.96
J. J. Vollertsen, Armour & Co.....	9.38	11.48	9.50	9.19	9.42	9.47
W. H. Irwin, Swift & Company.....	9.32	11.75	9.38	9.19	9.75	10.50
Maximum .....	10.52	13.12	9.87	9.48	10.14	10.75
Minimum .....	8.47	10.50	9.00	8.68	9.29	9.47
Average .....	9.49	11.62	9.38	9.09	9.58	10.17

A review of these results indicates that the modification brought the results in better agreement than the results reported the previous year and also better than the results obtained on samples No. 1 and No. 2 of 1927-28 co-operative work. It should be noted that the Kingman moisture results are somewhat higher than the oven method figures, which is probably caused by a slight destructive distillation at the temperature of the solvent, 142°C. The Committee does not feel that it would be practical to use a distillation method except in special cases.

*Jacketed Oven:*

The same six samples were sent to sixteen Swift & Company laboratories for the moisture determination using the Standard Jacketed Oven (see sketch) and the following method:

**Moisture and Volatile for Cotton Seed Cake, Meal, Meats, etc.**

**Using the Standard Jacketed Oven**

*Apparatus:*

1. Balance accurate to one milligram.

high when completely covered.

3. Standard Oven (or equivalent). See sketch.

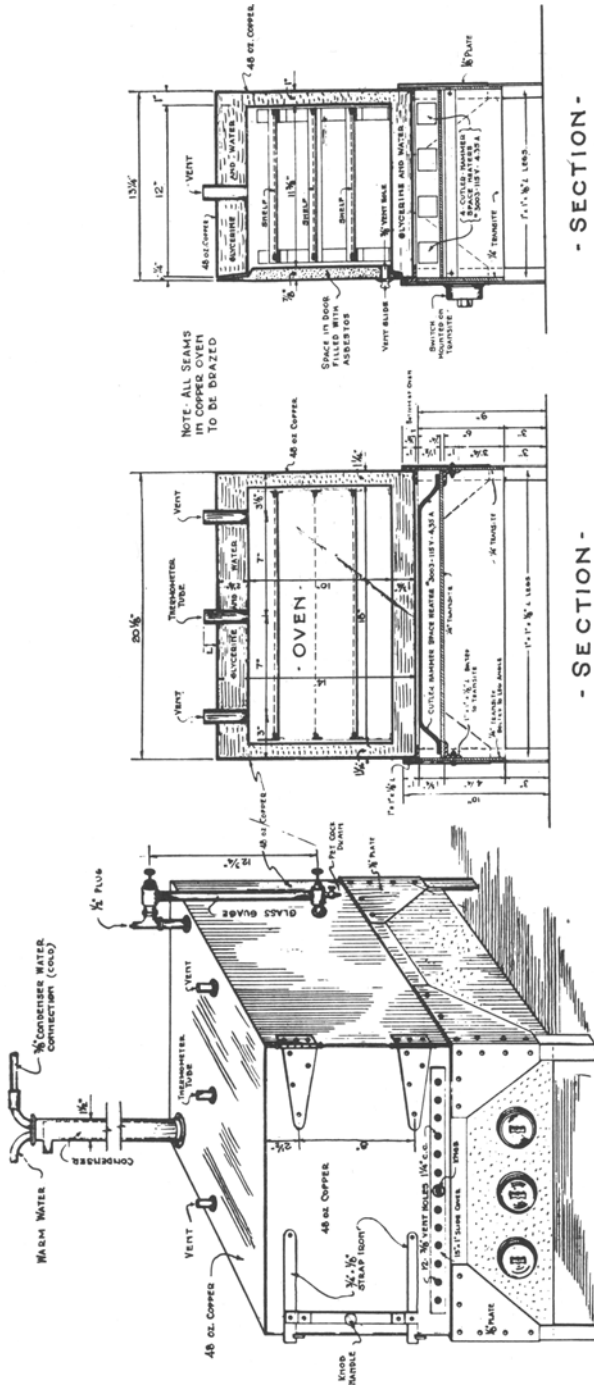
A double walled oven containing a glycerin solution in the jacket; this oven supported on an angle iron base equipped with electric space heating units for heating the glycerin solution.

The jacket of the oven is so constructed that the glycerin solution fills not only the side walls and bottom but also covers the top inner wall to a depth of about one inch. The door is well insulated by means of asbestos or other suitable insulating material and is equipped with a ventilating shutter. All the seams of the oven are brazed.

*Determination:*

Weigh five grams of the well mixed sample into a tarred aluminum moisture box. Place the sample in the standard oven and heat for five hours at a temperature of 101°C. At the expiration of five hours, remove the box from the oven, cover immediately, and cool in a desiccator. Weigh as soon as cool (within 30 minutes).

Loss in weight divided by 5, times 100 = percentage moisture.



NOTE - ALL SEAMS  
IN COPPER OVEN  
TO BE BRAZED

SPACE IN DOOR  
FILLED WITH  
ASBESTOS

- SECTION -

- SECTION -

- JACKETED OVEN -

*Note:*

The calcium chloride in the desiccators must be changed frequently in order to insure a dry atmosphere. The samples must be weighed back promptly or an increase in weight may occur in the desiccator.

The results given in the compilation below, in the opinion of the Committee, are remarkably good and are very much on the same order as were reported on last year's co-operative work:

*A. O. C. S. Co-Operative Samples of Cottonseed Meal, Numbers 1 to 6, for Moisture Determinations, Swift & Company Laboratories, Using Swift & Company Standard Jacketed Glycerin Oven, Temperature 101°C.*

Laboratory	No. 1	No. 2	No. 3	No. 4	No. 5	No. 6
1 .....	7.69	10.22	8.90	8.70	9.26	9.70
2 .....	7.70	10.22	9.00	8.72	9.05	9.52
3 .....	7.83	10.33	8.86	8.58	9.05	9.67
4 .....	7.83	10.30	8.98	8.70	9.20	9.60
5 .....	7.96	10.33	8.76	8.48	9.33	9.66
6 .....	7.83	10.35	8.90	8.64	9.18	9.45
7 .....	8.09	10.53	8.91	8.69	9.35	9.66
8 .....	7.73	10.29	8.82	8.55	9.24	9.40
9 .....	7.88	10.33	8.81	8.56	9.05	9.52
10 .....	7.93	10.22	*8.49	8.50	8.91	9.33
11 .....	7.94	10.11	8.74	8.48	9.16	9.59
12 .....	8.01	10.45	8.69	8.44	9.09	9.47
13 .....	8.05	10.43	8.90	8.63	9.24	9.54
14 .....	7.90	10.35	8.93	8.73	9.15	9.71
15 .....	8.07	10.53	8.92	8.69	9.42	9.77
16 .....	7.70	10.20	8.67	8.45	9.00	9.30
Maximum .....	8.09	10.53	9.00	8.73	9.42	9.77
Minimum .....	7.69	10.11	8.67	8.44	8.91	9.30
Average .....	7.88	10.32	8.83	8.58	9.15	9.57

\* Not included in maximum, minimum or average.

All of the above samples were dried for a period of five hours. Since there is some objection offered to the length of time required, the same six samples were dried for periods of two, three, four and five hours. The three hour drying time gave results which were very close to the five-hour period, the average difference being only .06%.

An examination of the check meal moisture results on the first twenty-four Smalley Foundation samples for the season 1927-28, in

which ovens of various types were used, shows a maximum range of 4.35%, a minimum of 1.48%, with an average range of 2.67%.

The maximum range of the co-operative work sent out by the Moisture Committee, in which the jacketed oven was used, was 0.51%, the minimum 0.29%, and the average 0.40%.

The Committee has been in touch with several chemical supply houses with a view of getting ovens, of the type used in the co-

operative work this year, built at a reasonable figure and has assurances that bids and possibly specimen ovens will be in the hands of the Committee prior to the May Meeting of the Society. It is hoped to have one or more ovens at the May Meeting for inspection.

The Committee feels that the co-operative work warrants the following recommendations:

1. That a jacketed oven of the type shown in the sketch be adopted as standard.

2. That the moisture method us-

ing the standard jacketed oven be adopted and the drying time be fixed at three hours.

3. That the Kingman Distillation Method be suggested for use in special cases where, for any reason, a distillation moisture method appears advisable.

Moisture Committee, A. O. C. S.:

W. H. Irwin, Chairman,

C. H. Cox,

N. C. Hamner,

W. D. Hutchins,

C. P. Long,

M. L. Sheely,

E. H. Tenent,

J. J. Vollertsen.

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## Report of Committee on Revision of Methods of Analysis

**T**HIS Committee was appointed to eliminate from our Methods all reference to Trading Rules, leaving merely a description of procedure, and to simplify the wording where possible. Its duties were merely editorial and no changes in procedure are made by this Committee. The object of this work was to put the Methods into such shape that they could be adopted and recommended by the American Chemical Society for universal use by all chemists concerned with analyses of any kind where these Methods will apply, even though such chemists are not concerned with cottonseed products or Interstate Rules.

Our present Methods for 1927-28 have been thus revised and sample copies printed. Suggestions for improvement will be welcomed. A few changes in procedure will undoubtedly be made at our 1928 meeting, after which it is proposed to have the complete, up-to-date Methods reprinted in pamphlet form, as the official Methods of this Society, omitting the Interstate Rule numbering. They will then be obtainable from our

Secretary at a very small charge.

While Rules 260, 261, 262, 263 and 264 in Chapter XII have been printed with the sample copies as a matter of convenience, it is not intended to include these in the final edition, as they relate only to settlements of trades, specifying which refining test to report, etc., and they can be found in the Interstate Rule Book by those who need them.

The Interstate will doubtless continue to include our Methods in its annual Rule Book for convenience.

It has been suggested that we should also have the "Standard Methods for the Sampling and Analysis of Commercial Fats and Oils" of the American Chemical Society, printed in the same pamphlet as our present rules, for these are also official Methods of this Society, as well as of the American Chemical Society. These will be included if the Society so directs.

E. R. BARROW

W. H. IRWIN

C. B. CLUFF, *Chairman.*

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