Determination of Moisture in Cottonseed and Cottonseed Products

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IN determining oil and nitrogen in cottonseed (1) the sample is fumed and ground to give a finely divided material suitable for analysis. This pretreatment makes necessary the determination of original moisture on the seed and second moisture on the prepared sample in order to calculate the analysis to the basis of the original cottonseed. Jamieson and McKinney (2) outlined methods of analysis for cottonseed which specified 5 hours' drying at 101° C. of crimped cottonseed for original moisture and 3 hours' drying at 101° C. of fumed and ground cottonseed for second moisture. They do not give, however, the experimental data to support these drying times. In addition the moisture committee of the American Oil Chemists' Society (3) carried on a program of collaborative investigation of moisture in crimped cottonseed and in cottonseed meal.

As a result of these findings the present official method (1) specifies for original moisture the loss in weight on drying 5-gram samples of crimped cottonseed for 5 hours or 50- to 60-gram samples of whole cottonseed for 12 to 16 hours at 101° C. For second moisture the loss in weight determined on 5-gram samples of fumed and ground cottonseed dried for 2 hours at 101° C. is specified. The moisture content of cottonseed meal is obtained by drying 5-gram samples at 101° C. for 3 hours. The published reports of the moisture committee (3) do not record investigation of time, temperature, and moisture relations on fumed and ground cottonseed, whole or ground cottonseed meats. The only oven conditions reported in their investigations on whole and crimped cottonseed and on cottonseed meal are at 101° C. in either forced draft ovens or in convection ovens. Some of their reports suggested the possibility of shortening the drying time for crimped cottonseed and cottonseed meal. It therefore seemed advisable to re-examine oven methods for determining moisture under different conditions of time, temperature and pressure on whole, crimped and fumed and ground cottonseed; whole and ground cottonseed meats; and cottonseed meal. Since the determination of moisture in biological materials by oven-loss-in-weight methods is an empirical procedure, it is essential that suitable oven conditions be determined for each commodity.

Experimental and Discussion

Fresh cottonseed (1035 seeds per 100 grams), commercially hulled cottonseed meats, both from the 1945 crop, and commercial cottonseed meal were used as sample materials. Several kilograms of the seeds were crimped; another portion was fumed and ground as directed in the Official Methods of the American Oil Chemists' Society (1); and a third portion was retained whole. Part of the cottonseed meats was ground through a Wiley mill using the 2-mm. screen and the remainder was used whole. The meal was used without further grinding.

Each of the sample materials was placed in a cheesecloth-lined wire tray, allowed to come to moisture equilibrium with the air of the laboratory, and then stored in a sealed container until analytical samples were weighed. While the whole and the crimped cottonseed samples were expected to have the same moisture content, this moisture content was not expected to be necessarily that of the fumed and ground seed sample. Both the whole and the ground cottonseed meats were expected to have the same moisture content but not necessarily identical with that of the seed or meal samples. Sieve analyses on the fumed and ground seed, the ground meats, and the meal are shown in Table I.

TABLE I.
Sieve Analyses of Cottonseed Products.

Material	Retained by			Not retained by 60 mesh
	20 mesh	40 mesh	60 mesh	
	%	%	%	%
Cottonseed meal	9.5	17.0	17.9	55.6
Cottonseed meats ground through Wiley mill, 2-mm. screen	7.8	53.0	18.1	21.1
Fumed and ground cottonseed	17.5	62.7	14.2	5.6

The time-moisture loss data were obtained on both 5- and 50-gram samples of the whole and the crimped cottonseed; and on 5-gram samples only of the other cottonseed, meal, and meat samples. Data were obtained for all the materials in forced draft ovens at 101°C. and also at 130°C., and in a vacuum oven held at 101°C. (Figs. 1 to 5, inclusive). The forced draft oven used was equipped with a torsion balance sensitive to 5 mg. and a mechanism by use of which the samples could be weighed at the specified time intervals without removing them from the oven. The vacuum oven was evacuated to a pressure of less than 5 mm. of mercury. For the 50-gram samples, metal dishes, 9 cm. in diameter and 3 cm. deep, having tight-fitting covers, were used as containers; for the 5-gram samples, the official A.O.C.S. moisture dishes were used.

The handling of the samples and the adjustment of the ovens were exactly as previously described for peanuts (4). In all cases when samples were removed from the oven they were allowed to cool for approximately ½ hour in desiccators over phosphorous pentoxide before weighing. The points shown in Figures 1 to 5 are averages of duplicate analyses which usually agreed within 0.04%.

Because of the slower rate of loss of the last portion of the moisture driven off it is difficult to establish rigidly from rate-of-loss time curves the minimum time required to dry a given material under a given oven condition. As an aid to interpreting the curves shown in the figures, therefore, moisture was determined in oven-dried samples by the Karl Fischer

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volumetric method. Duplicate 5-gram samples of the fumed and ground cottonseed, the ground cottonseed meats, and the cottonseed meal previously used were dried in official moisture dishes under each of the oven conditions investigated for two different time intervals. The drying curves indicate that the samples were not completely dried at the shorter but were probably dried at the longer time interval. Directly upon removal from the oven the samples were transferred to oven-dried glass-stoppered Erlenmeyer flasks, which were then immediately stoppered. With minimum exposure to the atmosphere, to each flask was added 25 ml. of absolute methanol, the flasks then being placed in desiccators over phosphorous pentoxide to avoid diffusion of atmospheric moisture around the stopper. After extraction for 3 hours with frequent shaking the samples were titrated with Karl Fischer reagent (5, 6) to the appearance of an iodine color. Blanks were run on the absolute methanol. The extraction procedure employed proved to yield maximum values for the sample materials used. For purposes of comparison 1-gram samples of the undried materials were also analyzed for moisture by the Karl Fischer method in the same way. The values obtained, together with the values obtained by oven-loss-in-weight methods, are shown in Table II.

TABLE II.

Comparison of Moisture by Selected Oven Methods With Moisture by Karl Fischer Method[1]

Material	Moisture in original material. Karl Fischer method	Oven condition	Loss in weight at oven condition	Moisture in dried material. Karl Fischer method
	%		%	%
Fumed and ground cottonseed	5.54	101° F.D.[2] 3 hrs.	5.81	0.45
		101° F.D. 5 hrs.	5.99	0.01
		101° Vac. 3 hrs.	5.60	0.29
		101° Vac. 5 hrs.	5.85	0.02
		130° F.D. 1.5 hrs.	6.26	0.19
		130° F.D. 3 hrs.	6.40	0.01
Ground cottonseed meats	6.11	101° F.D. 3 hrs.	5.99	0.33
		101° F.D. 5 hrs.	6.15	0.03
		101° Vac. 3 hrs.	5.77	0.13
		101° Vac. 5 hrs.	5.96	0.05
		130° F.D. 1.5 hrs.	6.35	0.12
		130° F.D. 3 hrs.	6.44	0.03
Cottonseed meal	7.27	101° F.D. 3 hrs.	7.40	0.41
		101° F.D. 5 hrs.	7.52	0.07
		101° Vac. 3 hrs.	7.11	0.12
		101° Vac. 5 hrs.	7.39	0.04
		130° F.D. 1.5 hrs.	8.00	0.13
		130° F.D. 3 hrs.	8.07	0.00

[1]All values are the average of closely agreeing duplicate determinations.
[2]Forced draft.

The values obtained by the Karl Fischer method on the partially dried and dried material are in agreement with the losses indicated by the curves for all three oven conditions. The higher values of oven loss in weight at 130° may be due to some decomposition at the higher temperature; and the lower values by the Karl Fischer method on fumed and ground cottonseed and on cottonseed meal at 101° may be due to incomplete extraction of water from these materials by methanol. The discrepancies serve to emphasize the empirical nature of the determination of moisture in biological materials such as cottonseed.

To further check the influence of the moisture content of cottonseed materials on the values found on analysis, moisture was determined by a number of selected oven procedures before and after known amounts of moisture were added to samples of the conditioned lots of cottonseed and cottonseed products previously used. In each case moisture was determined in duplicate on the conditioned samples (Table III) and two additional weighed samples were placed in desiccators over water for 48 hours at 25° C. to increase the moisture content by absorption. These samples were then reweighed and dried under the oven conditions indicated in Table III.

Attempts to raise moisture to higher levels than those shown in Table III, by longer exposure of the samples over water, resulted in mold growth and were therefore unsuccessful. The differences between moisture calculated and that found may be in part due to the activity of molds and enzymes during exposure.

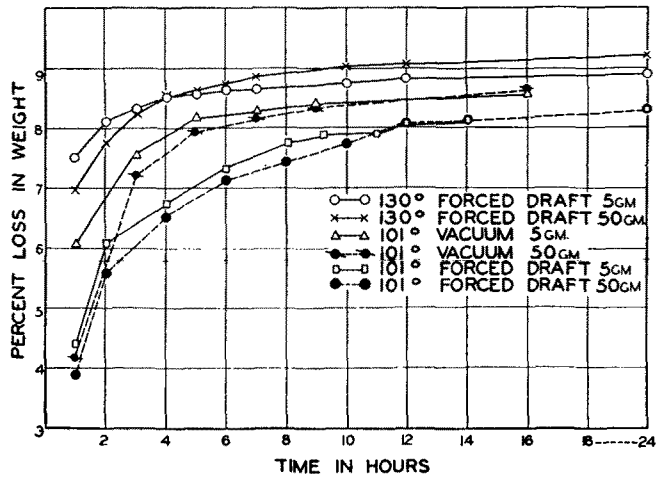

FIG. 1. Loss in weight on drying whole cottonseed under different oven conditions.

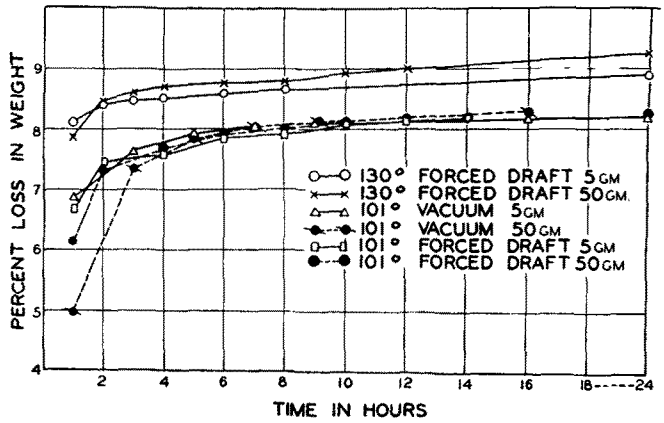

FIG. 2. Loss in weight on drying crimped cottonseed under different oven conditions.

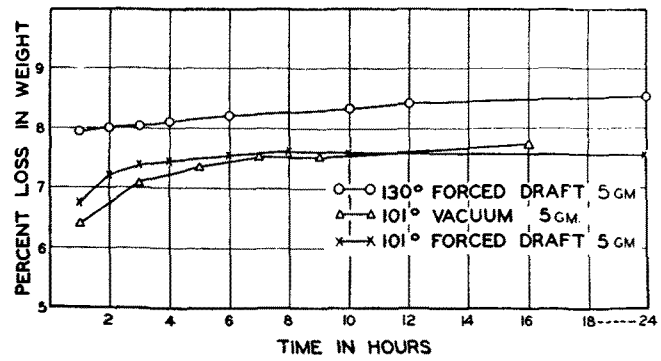

FIG. 3. Loss in weight on drying fumed and ground cottonseed under different oven conditions.

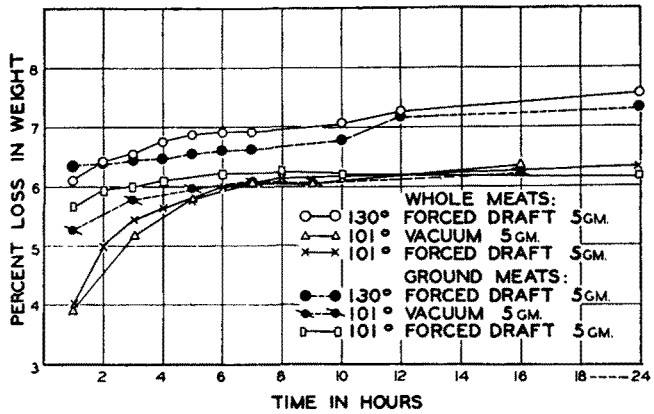

FIG. 4. Loss in weight on drying whole and ground cottonseed meats under different oven conditions.

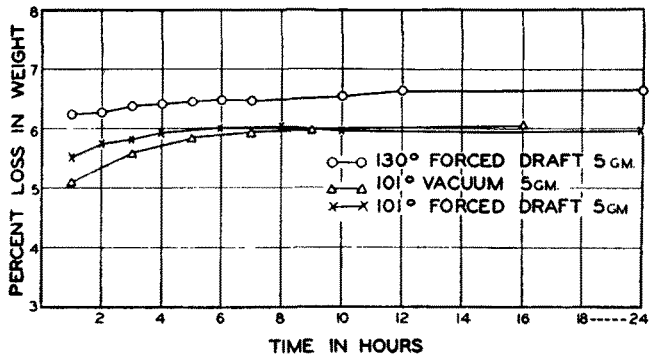

FIG. 5. Loss in weight on drying cottonseed meal under different oven conditions.

The agreement between the calculated and the determined moisture values for the 101°-forced draft and the 101°-vacuum ovens indicate that the drying times required under these oven conditions are independent of the amount of moisture present over fairly large variations of moisture. The values obtained in the 130°-forced draft for whole and crimped cottonseed are also in agreement with those calculated. The remainder of the samples, however, gave high values in the 130°-forced draft oven, probably another indication of decomposition at that temperature.

Thus, Figures 1 to 5 show that higher temperatures not only increase the initial rate of loss but also increase the total loss on heating. When reduced pressure is used the initial rate of loss is slightly lower than that at atmospheric pressure, undoubtedly because of the slower rate of heat transfer in the vacuum. The loss in weight in the vacuum oven soon exceeds that in the forced draft oven and continues at a higher value with prolonged heating. With samples of whole and of crimped seeds the rate of loss for 5- and 50-gram samples is very nearly the same. In the 130°-forced draft oven the loss of the 50-gram samples of both whole and crimped cottonseed after about 4 hours' heating exceeds slightly that of the corresponding 5-gram samples. Measurement of temperature in the sample during heating showed that during the first 6 hours the temperature of the 5-gram samples not only exceeded that of the 50-gram samples but even exceeded slightly the temperature of the shelf of the oven. Though this temperature increase is small (about 1° C.) it may induce an oxidative effect on the 5-gram sample.

TABLE III.

Recovery of Moisture Added to Specified Cottonseed Materials by Specified Procedures. Values Expressed on Basis of Conditioned Samples.

Cottonseed material	Moisture in conditioned sample[1]	Moisture added[2]	Total moisture calculated[3]	Total moisture found
	%	%	%	%
At 101° C. for 16 hours in forced draft oven:				
Whole cottonseed, 50-gram sample	8.07	3.77	11.84	12.09
		4.07	12.14	12.39
Whole cottonseed, 5-gram sample	8.11	4.97	13.08	13.33
		3.20	11.31	11.59
At 101° C. for 8 hours in forced draft oven:				
Crimped cottonseed, 50-gram sample	8.03	1.24	9.27	9.37
		2.24	10.27	10.35
Crimped cottonseed, 5-gram sample	7.92	4.38	12.30	12.40
		4.97	12.89	12.77
At 101° C. for 5 hours in forced draft oven:				
Fumed cottonseed, 5-gram sample	5.99	4.48	10.47	10.51
		4.35	10.34	10.36
Whole meats, 5-gram sample	5.74	3.13	8.87	9.11
		2.69	8.43	8.66
Ground meats, 5-gram sample	6.15	3.71	9.86	9.78
		5.12	11.27	11.22
Cottonseed meal, 5-gram sample	7.52	2.99	10.51	10.67
		3.02	10.54	10.69
At 101° C. for 7 hours in vacuum oven:				
Whole cottonseed, 50-gram sample	8.18	2.19	10.37	10.40
Crimped cottonseed, 50-gram sample	8.11	3.77	11.95	12.01
		1.24	9.35	9.30
		3.84	11.95	12.08
At 101° C. for 5 hours in vacuum oven:				
Whole cottonseed, 5-gram sample	8.18	4.18	12.36	12.20
Crimped cottonseed, 5-gram sample	7.95	1.74	9.92	9.97
		3.01	10.96	10.94
Fumed cottonseed, 5-gram sample	5.85	5.08	13.03	12.95
		5.01	10.86	10.86
Whole meats, 5-gram sample	5.76	4.64	10.49	10.51
		3.38	9.14	9.04
Ground meats, 5-gram sample	5.96	5.69	11.45	11.61
		5.67	11.63	11.67
Cottonseed meal, 5-gram sample	7.39	5.83	11.79	11.83
		7.02	14.41	14.75
		7.40	14.79	15.15
At 130° C. for 5 hours in forced draft oven:				
Whole cottonseed, 50-gram sample	8.66	2.32	10.98	11.03
Whole cottonseed, 5-gram sample	8.59	3.68	12.34	12.43
		2.36	10.95	11.26
		3.21	11.80	11.99
At 130° C. for 3 hours in forced draft oven:				
Crimped cottonseed, 50-gram sample	8.64	2.29	10.93	10.94
		3.31	11.95	11.89
Crimped cottonseed, 5-gram sample	8.46	1.50	9.96	11.98
		3.41	11.87	12.21
Fumed cottonseed, 5-gram sample	6.40	3.84	10.24	10.49
		3.54	9.94	10.24
Whole meats, 5-gram sample	6.54	1.93	8.47	9.04
		1.62	8.16	8.72
Ground meats, 5-gram sample	6.44	3.40	9.84	10.30
		4.36	10.80	11.28
Cottonseed meal, 5-gram sample	8.07	5.97	14.04	14.65
		6.37	14.44	15.03

[1]Obtained by procedure specified.
[2]Moisture added by exposure of weighed sample.
[3]Calculated by addition of values found in first two columns.

In contrast to other oilseed (4, 7), in all the cottonseed materials, with the exception of whole cottonseed, the total losses in weight attained in both vacuum and atmospheric ovens at 101° are in good agreement. Since values for moisture by the Karl Fischer method are lower for fumed and ground cottonseed and for cottonseed meal, it is possible that the extraction of water from these materials with methanol is inadequate. It is equally conceivable, however, that some volatile constituent other than water is lost in the oven methods.

Summary and Conclusions

Losses in weight of whole, crimped, and fumed and ground cottonseed, whole and ground cottonseed

meats, and cottonseed meal have been studied in both vacuum and forced draft ovens at 101° C. and in forced draft ovens at 130° C. The values obtained have been compared with values for moisture obtained by the Karl Fischer method. The amount of moisture in the sample had very little or no influence on the length of time required to attain a constant dehydration level at the times and temperatures studied. The rate-of-loss curves indicated that it is not feasible to reduce the drying times required by the official methods for determination of moisture in cottonseed materials.

With whole cottonseed 12 to 16 hours' drying at 101° in the forced draft oven as required by the present official method was found satisfactory. With crimped seed, however, 8 hours' drying in the 101°-forced draft oven was required to attain an equivalent degree of dehydration. Values approximately 0.2% lower were obtained in the 5 hours specified in the official method. For fumed and ground cottonseed, whole or ground meats, and cottonseed meal, the oven loss-time curves indicated that 5 hours at 101° C. are necessary to reduce the rate of loss in weight to a minimum. This time interval, confirmed by the data obtained by the Karl Fischer method, is somewhat longer than the time specified in the official method, namely, 2 hours for fumed and ground cottonseed and 3 hours for the meal and meats.

On heating cottonseed materials in forced draft ovens, larger losses in weight were obtained at 130° than at 101° C.

The results obtained indicated that while at 101° C. both the vacuum and the forced draft ovens reduce cottonseed materials to equivalent degrees of dehydration, the times required are different.

Any changes which appear to be indicated in the official methods by the present work should be carefully studied and checked by collaborative investigations, taking into account the wide range of samples and the variations of individual workers in the application of recommended procedures.

Acknowledgment

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Report of the Soybean Analysis Committee 1945-46

DURING the past year no collaborative work on soybeans has been carried out by this committee; however R. T. Doughtie, Jr., agent of the Commodity Credit Corporation, has conducted an extensive test of the present official AOCS procedure for determining the oil content of soybeans. Results of this test, participated in by 38 of the leading soybean chemists of the country, have been made available through the courtesy of Mr. Doughtie. Two series of 20 samples were studied with results furnished by all 38 chemists on each of the 40 samples.

In the first series of 20 samples, standard deviation averaged 0.28, with the highest standard deviation 0.46. The sample giving this extremely high standard deviation contained an excessive amount of dockage (5.2%) as compared with all of the remaining samples. The difficulties found by the chemists on this sample were attributed to this cause, and the analysts were cautioned to obtain proper screens and follow the official procedure for dockage removal more closely.

On the second series of 20 samples the standard deviation averaged 0.22 (a distinct improvement) and the highest standard deviation was 0.29. This value of 0.22 may appear somewhat high but represents the agreement between results when a large group of chemists (some very experienced and some rather inexperienced) uses the present official AOCS method for this determination. It should be remembered that on any given sample two-thirds of the results of individual chemists will lie within plus or minus one standard deviation of the general average. In an effort to estimate the precision of the method when used by the most experienced workers, the average standard deviation was calculated for the second series of 20 samples using the 19 best chemists (half of total). This average was found to be only 0.14, and this improvement shows that the method of analysis is still responsive to expert treatment.

During the year, in addition to the above series of tests, Mr. Doughtie and his collaborators also studied the effect of regrinding and the increase of sample size from 2 to 5 grams. All analysts would like, if possible, to eliminate the regrinding procedure, and many workers feel that increased accuracy would result from the use of 5-gram samples. The results obtained in Mr. Doughtie's study were too inconclusive to suggest changes at this time.

It should also be noted that during the past year there has been more general satisfaction with this method of analysis, and more confidence in it, than in the past. This is reflected by fewer variations in the check analyses performed by CCC and by fewer appeal analyses requested both by the processors and CCC.

At the Northern Regional Research Laboratory studies on the grinding of soybeans with a hammer-type of mill have been carried out. This mill shows sufficient promise so that it will be submitted to a