

REFERENCES

1. Association of Official Agricultural Chemists. Official Methods of Analysis, 7th Ed., Method 13.19, p. 196-197, Washington, 1950.
2. Arthur, J. C. Jr., Crovetto, A. J., Molaison, L. J., Guilbeau, W. F., and Altschul, A. M., J. Am. Oil Chem. Soc., 25, 398-400 (1948).
3. Burnett, R. S., Chem. Eng. News, 24, 478-480 (1946).
4. Burnett, R. S., U. S. Patent 2,463,740 (March 8, 1949).
5. Burnett, R. S., and Fontaine, T. D., Ind. Eng. Chem., 36, 284-288 (1944).
6. Calculations based on private communications.
7. Irving, G. W. Jr., Merrifield, A. L., Burnett, R. S., and Parker, E. D., U. S. Patent 2,405,830 (Aug. 13, 1946).
8. Pominski, J., Molaison, L. J., Crovetto, A. J., Westbrook, R. D., D'Aquin, E. L., and Guilbeau, W. F., Oil Mill Gaz., 51 (12), 33-39 (1947).
9. U. S. Food Distribution Administration, "U. S. Standards for Shelled White Spanish Peanuts (effective Sept. 1, 1939)." 3 pp., processed (Aug. 15, 1939, reissued Oct. 11, 1943).
10. Gastrock, E. A., and D'Aquin, E. L., Oil Mill Gaz., 53 (4), 13-21 (1948).

[Received June 13, 1951]

Determination of Moisture and Oil in Sesame Seed

S. M. STARK JR. and CARROLL L. HOFFPAUIR, Southern Regional Research Laboratory,¹ New Orleans, Louisiana

THOUGH sesame is one of the oldest cultivated oilseed crops, no systematic investigation of analytical methods for the determination of moisture and volatile matter and of the oil content of the seed has been reported. Recent interest in the domestic production of sesame has made it desirable that such methods be developed for use in agronomic investigations and to provide a basis for adoption of methods for future needs in marketing and processing.

Sesame seed are small and have tough seed coats. The high oil content makes it impossible to prepare samples of them for analysis by grinding in most mills. However, the Henry nut slicer² was found satisfactory for the purpose, provided the blade is changed as soon as it becomes dulled.

Samples and Their Preparation

The variety and origin of five lots of sesame seed used are given in Table I. The last four are those used as sources of sesame oil in previously reported investigations (2, 5).

TABLE I
Description of Sesame Seed

No.	Variety	Crop year	Where grown	Weight per 1,000 seeds
				grams
1	Unknown	1946	Kansas	2.645
2	SO-4	Nicaragua	3.065
3	Nebraska 1025-3	1948	Nebraska	2.880
4	Clemson 4520	1948	South Carolina	2.765
5	Clemson 4522	1948	South Carolina	3.160

The seed were allowed to come to moisture equilibrium at a constant relative humidity of 65% and a temperature of 70°F. A portion of each lot was prepared for analysis with the Henry nut slicer under the same atmospheric conditions and allowed to equilibrate further for several days.

Determination of Moisture and Volatile Matter

Curves for the loss on drying vs. time were prepared from data obtained on these samples by heating 5-gram samples contained in the official A.O.C.S. moisture dishes at 101°C. and 130°C. in a forced-draft oven. The oven was equipped with a torsion balance sensitive to 5 mg. and a mechanism by which

the samples could be weighed at selected time intervals without removing them from the oven. Handling of the samples and adjustment of the oven were exactly as described previously for investigations on the determination of moisture in peanuts (3) and cottonseed (4).

The curves for the loss during oven-drying for the Nicaraguan seed (No. 2) for temperatures of 101°C. and 130°C. are shown graphically in Figure 1. The

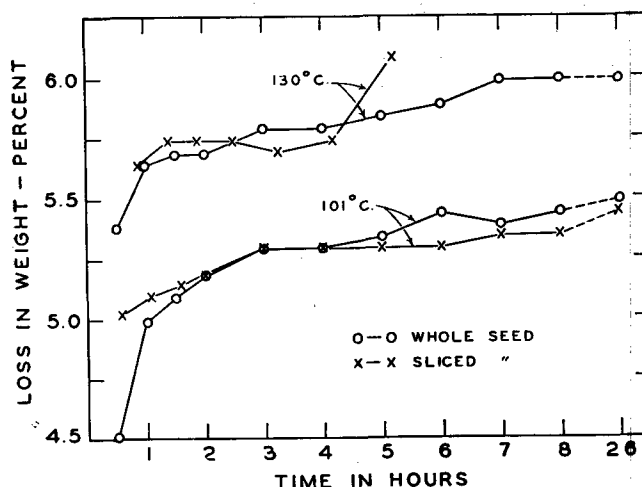


FIG. 1. Curves showing the loss in weight on oven-drying whole and sliced sesame seed.

individual points shown are averages of duplicate analyses generally agreeing within 0.1%. The curves for the other lots of the sesame seed were so similar to the ones shown that those in Figure 1 may be considered typical for all of the samples investigated.

In order to evaluate the degree of dehydration obtained under the specified drying conditions for different periods of heating, the residual moisture in the oven-dried samples was determined by the Karl Fischer volumetric method as described previously (4). The values obtained, together with those obtained by the oven-loss-in-weight methods, are shown in Table II.

Values obtained for residual moisture in samples dried at 101°C. for 4, 5, and 6 hours are essentially constant and show that complete removal of moisture was not accomplished under these conditions of heating a 5-gm. sample. These values are lower than those obtained by heating the samples at 130°C. for shorter periods. The sum of the values for residual moisture and for oven loss in weight for heating at

¹One of the laboratories of the Bureau of Agricultural and Industrial Chemistry, Agricultural Research Administration, U. S. Department of Agriculture.

²Manufactured by Davidson-Kennedy Company, Atlanta, Ga. The mention of firm names or trade products does not imply that they are endorsed or recommended by the Department of Agriculture over other firms or similar products not mentioned.

TABLE II

Moisture and Volatile Matter in Sesame Seed by Oven-Loss-in-Weight Method and Residual Moisture in Dried Sample by Karl Fischer Method

Sesame seed	Forced draft oven drying			Residual moisture in dried sample by K. F. reagent ¹
	Temperature	Time	Loss ¹	
No. 1 Whole	°C.	hours	%	%
	101	4	5.69	0.28
	101	5	5.69	0.27
	101	6	5.79	0.29
	130	1.5	6.13	0.08
	130	2	6.18	0.06
No. 2 Whole	130	3	6.18	0.02
	101	4	5.29	0.30
	101	5	5.34	0.28
	101	6	5.44	0.29
	130	1.5	5.68	0.07
	130	2	5.68	0.05
No. 1 Sliced	130	3	5.79	0.06
	101	4	5.64	0.32
	101	5	5.64	0.28
	101	6	5.74	0.31
	130	1	5.98	0.09
	130	1.5	6.03	0.14
No. 2 Sliced	130	2	6.08	0.15
	101	4	5.29	0.28
	101	5	5.29	0.26
	101	6	5.29	0.29
	130	1	5.64	0.11
	130	1.5	5.74	0.15
	130	2	5.74	0.14

¹All values are averages of closely agreeing duplicate determinations.

101°C. for 4 to 6 hours approaches those obtained for heating the samples at 130°C. for 1.5 and 2 hours.

The data indicate that the most satisfactory value for the determination of moisture and volatile matter in whole and ground or sliced sesame seed is obtained by heating 5-gm. samples for 2 hours at 130°C. in a forced-draft oven.

Determination of Oil

Preliminary experiments on the extraction of 2-gm. samples of sliced sesame seed indicated the necessity of regrinding the sample after the initial extraction in order to obtain complete removal of the oil by subsequent extraction. It was found necessary to use 0.25 gm. of 60- to 80-mesh sand to obtain adequate regrinding with a mortar and pestle. If sand were lacking or when coarser sand was used, the sample was not sufficiently reduced in particle size to allow complete extraction of the oil in a reasonable time.

To establish the time required for complete extraction of the oil in sesame seed, 2-gm. samples of the sliced seed were extracted in a Butt extractor as specified in A.O.C.S. official method Aa 4-38 (1) for 2 hours. The partially extracted sample was removed from the extractor and transferred to a porcelain mortar having an internal diameter of 4 inches. After the addition of 0.25 gm. of 60- to 80-mesh sand the sample was reground with approximately 250 strokes of the pestle during a period of from 2 to 2.5 minutes. The reground material was quantitatively returned to the original filter paper, rewrapped, and replaced in the extractor. The extraction with the official A.O.C.S. solvent (1) was continued for selected time intervals of from 2 to 16 hours. The results obtained by regrinding the sample with and without sand are given in Table III. They indicate that for the determination of oil in sesame seed, a 2-gm. sample of the sliced seed should be extracted for 2 hours as specified in A.O.C.S. official method Aa 4-38, reground with 60- to 80-mesh sand as described above, and re-extracted for 3 hours.

TABLE III

Yield of Oil from Sesame Seed by Varying Extraction Time

Sesame seed	Extraction after regrinding	Yield of oil ¹	
		Reground with sand ²	Reground without sand ²
No. 1	hours	%	%
	2	52.38
	3	52.55
	6	52.38
No. 2	16	52.56	52.54
	2	53.36
	3	53.48
	6	53.38
No. 3	16	53.72	53.91
	2	50.56
	3	51.07
	6	50.73
No. 4	16	51.17	51.09
	2	55.32
	3	55.51
	6	55.23
No. 5	16	55.36	55.43
	2	55.06
	3	55.19
	6	54.99
	16	55.43	55.02

¹Averages of closely agreeing duplicate determinations.

²All samples were extracted 2 hours, reground and re-extracted for the time specified.

Application of the Methods

The methods for determining moisture and volatile matter and for oil have been used to analyze 95 samples of sesame seed including those of different varieties and origins. Nitrogen was also determined on 1-gm. samples by the Kjeldahl method, using mercury as a catalyst.

General conclusions with reference to the use of these methods for the analysis of these samples are as follows: The values for moisture ranged from 4.19% to 5.97%. The average variation between duplicate moisture values was 0.02%, and the maximum variation was 0.09%. Similarly the range in values of oil content was 42.62% to 60.73%, with the average variation being 0.14% and a maximum variation of 0.36% between duplicates. Values for nitrogen content ranged from 2.54% to 4.80% with an average variation of 0.02% and a maximum variation of 0.07% between duplicates.

Summary

Sesame seed may be satisfactorily prepared for analysis by use of the Henry nut slicer equipped with a sharp blade.

For the determination of combined moisture and volatile matter in both the whole and ground seed, a 5-gram sample should be dried for 2 hours at 130°C. in a forced-draft oven.

The A.O.C.S. official method Aa 4-38 for cottonseed is satisfactory for the determination of oil in sesame seed, provided a 2-gm. sample is reground with 0.25 gm. of 60- to 80-mesh sand after an initial 2-hour extraction and subsequently re-extracted for 3 hours.

REFERENCES

1. American Oil Chemists' Society, Official and Tentative Methods, 2nd ed., edited by V. C. Mehlenbacher, Chicago, 1946, rev. to 1950.
2. Andraos, V., Swift, C. E., and Dollear, F. G., J. Am. Oil Chem. Soc., 27, 31-34 (1950).
3. Hoffpauir, C. L., Oil and Soap, 22, 283-286 (1945).
4. Hoffpauir, C. L., and Petty, Dorothy H., Oil and Soap, 23, 285-288 (1946).
5. Menezes, F. G. T., Budowski, P., and Dollear, F. G., J. Am. Oil Chem. Soc., 27, 184-186 (1950).

[Received June 21, 1951]