in accuracy but the procedure is admirably suited for either small laboratories that are not equipped with carbon dioxide trains or for the purpose of making quick checks.

The results tabulated in Table II show that a high order of precision is obtainable by an analyst skilled in the technic of the method. Table I shows what can be expected when the method is given to analysts previously unacquainted with the procedure. An inspection of the results given shows that satisfactory checks between the train and alkalimeter method were obtained in all cases.

Acknowledgments

The authors are indebted to Messrs. Boggie, Peluso, and Moore for their assistance in obtaining the analytical results incorporated in Table I.

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(1) Hitchcock and Divine, Oil and Soap, *15*, 8 (1938).

(2) Hitchcock and Divine, Oil and Soap, *18*, 80 (1941).

(3) Scott, "Standard Methods of Chemical Analysis," 4th edition, revised, Volume I, p. 124, D. Van Nostrand Company, Inc., New York (1925).

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Report of the Soybean Analysis Committee 1941-42

In the 1941 report of the Soybean Analysis Committee (1), it was pointed out that the determination of moisture in biological materials is a purely empirical procedure, governed by three variables—temperature, pressure, and time—and that in determining moisture by oven loss-in-weight methods it is necessary to learn what combination of these three may be used to give the most valid results without the results being influenced by oxidation or decomposition or both. It has been experimentally demonstrated that a reliable referee procedure for the determination of moisture in ground soybeans (1,2) and soybean meals (1,3) is to dry the samples for 6 hours at 105°C. in a vacuum oven maintained at less than 25 mm. Hg pressure. It has also been shown that results agreeing with this vacuum oven procedure are obtained by heating for 2 hours at 130°C. in an air oven and by heating for 1 hour at 130°C. in a vacuum oven at less than 25 mm. Hg pressure. The time factor must be strictly observed in the latter procedure.

The collaborative work conducted by the committee was for the purpose of comparing these and some other oven loss-in-weight methods for the determination of moisture in soybean meals.

One sample of each of the four types of soybean meals was ground to pass a 1 mm. sieve in a Wiley mill and was sent to each of ten collaborators with the request that the moisture be determined by six temperature, time, and pressure combinations after the sample had been exposed to assume the equilibrium moisture content of the laboratory. The procedure specified was as follows:

Weigh a 2-gram sample into a tared aluminum moisture dish (A.O.C.S.), 2 inches in diameter and ¾ inches high, fitted with a close-fitting cover. Remove cover, place in oven, and dry under specified oven conditions. The temperature of the oven is to be taken at the level of the samples. Remove pan from oven, cover, cool in desiccator for 15 or 20 minutes, and weigh. Calculate loss in weight as moisture. The specified conditions of drying were:

Method

No. 1. Regular laboratory procedure of collaborator
No. 2. 105°C. for 3 hours in air oven
No. 3. 130°C. for 1 hour in air oven
No. 4. 130°C. for 2 hours in air oven
No. 5. 105°C. for 6 hours in vacuum oven, less than 25 mm. Hg
No. 6. 130°C. for 1 hour in vacuum oven, less than 25 mm. Hg

The regular procedures used by the various collaborators were found to be quite different. Their reports indicated the following temperature, time, and pressure combinations:

Collaborators

No. 1. 130°C., 40 minutes in air oven
No. 2. 110°C., 18 hours in air oven
No. 3. 100°C., 2½ hours in vacuum oven at less than 50 mm. Hg
No. 4. 100°C., 5 hours in vacuum oven at less than 25 mm. Hg
No. 5. 108°C., 2 hours in air oven
No. 6. 135°C., 2 hours in air oven
No. 7.
No. 8. 100°C., 5 hours in vacuum oven at less than 25 mm. Hg
No. 9. 130°C., 1 hour in air oven
No. 10. 130°C., 3 hours in air oven

Collaborators 1 and 6 used forced draft air ovens, while all others used air ovens of the conventional type. The vacuum ovens used were of the externally-heated vacuum chamber type with the exception of the one used by collaborator 9 who used an oven having an internal hot plate. The low pressure desired in the vacuum ovens was not maintained in all cases with the equipment on hand.

The purpose of the study was to compare the temperature, time, and pressure combinations in determining the moisture in the meal samples and not to check the technique of the collaborators. There was considerable variation in the relative values obtained by the collaborators with the several methods. These variations may be due in part to ovens and thermometers. Collaborator 9 used a vacuum oven with an internal hot plate and obtained low results. Some collaborators did not have equipment that permitted the low vacuum pressure desired.

It was hoped that with uniform technique, including time, temperature, and pressure control, the average values obtained by each collaborator for each

sample by methods 4, 5, and 6 would agree within close limits. The averages of the results obtained by collaborators 1 to 7, inclusive, for all 4 samples by methods 4, 5, and 6 were approximately the same. However, in a majority of the 28 individual instances the results obtained by the 130°C. for 2 hours in an air oven (method 4) and by 130°C. for 1 hour in a vacuum oven (method 6) were higher than those obtained by the 105°C. for 6 hours in a vacuum oven (method 5). The exact time of heating is not critical for methods 4 and 5, but is critical for method 6.

The results reported for method 3, heating at 130°C. for 1 hour in an air oven, are lower than those obtained by heating for 2 hours at the same temperature (method 4) and those obtained by heating at 105°C. for 6 hours in a vacuum oven. The time element is more important in the case of method 3 than in the case of methods 4 and 5 and with heating for the full hour period lower results are to be expected. As was anticipated, still lower values were obtained by heating at 105°C. for 3 hours in an air oven.

Collaborators 4 and 8 regularly determine moisture by heating the sample at 100°C. for 5 hours in a vacuum oven (method 1). Their results with this method were lower than the ones they obtained by heating at 130°C. for 2 hours in an air oven (method 4). The values obtained by collaborator 2 (method 1) by heating the samples at 110°C. for 18 hours in an air oven, agree remarkably well with the values he obtained by methods 4 and 5.

On the basis of the experimental work cited and the collaborative work done it appears that the most satisfactory procedures for determining moisture in soybean meals are heating at 105°C. for 6 hours in a vacuum oven and at 130°C. for 2 hours in an air oven. If the Uniform Methods and Planning Committee deems it advisable to recommend to the Society methods for the determination of moisture in soybean meals based on the present collaborative study, the following procedures are recommended for the consideration of the committee:

1. Referee Procedure: Heating a 2-gram sample in an A.O.C.S. moisture dish at 105°C. for 6 hours in a vacuum oven of the externally-heated chamber type in which pressure of less than 25 mm. Hg is maintained.
2. Routine Procedure: Heating a 2-gram sample in an A.O.A.C. moisture dish in an air oven at 130°C. for 2 hours.
3. Alternate Procedure: Using any temperature, time, and pressure combination that will give the same values as the referee procedure.

In the interest of uniformity it is further recommended that the Society collaborate in the adoption of moisture methods for soybean meals with the Association of Official Agricultural Chemists.

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TABLE I

Results of collaborative moisture determination in soybean meals (averages of duplicate determinations)

Method No.	Collaborator 1	2	3	4	5	6	7	8	9	10
Expeller meal	*per cent*	*per cent*	*per cent*	*per cent*	*per cent*	*per cent*	*per cent*	*per cent*	*per cent*	*per cent*
1	5.90	5.74	5.83	6.28	6.32	.....	.....	6.38	6.54	6.21
2	5.64	5.28	5.73	5.88	6.40	6.04	6.09	6.48	6.00	5.72
3	5.94	5.65	6.03	6.24	6.25	6.46	6.26	6.87	6.52	6.05
4	6.07	5.92	6.08	6.51	6.80	6.52	6.41	6.90	6.62	6.20
5	6.05	5.88	6.15	6.41	6.52	6.21	6.72	.....	6.35	.....
6	6.13	5.98	6.28	6.26	6.72	6.28	6.36	6.93	6.40	.....
Toasted solvent extracted flakes										
1	6.00	6.22	5.85	6.61	6.50	.....	.....	6.56	6.96	6.48
2	5.78	5.50	6.03	6.21	6.55	5.99	6.29	6.61	6.36	5.82
3	6.14	6.05	6.00	6.48	6.68	6.66	6.65	7.16	6.98	6.08
4	6.35	6.10	6.18	6.96	7.15	6.77	6.73	7.26	7.11	6.36
5	6.30	6.14	6.38	6.77	6.85	6.56	7.00	.....	6.24	.....
6	6.39	6.22	6.60	6.68	7.08	6.50	6.94	7.16	6.70	.....
Hydraulic meal										
1	5.94	6.01	5.83	6.48	6.58	.....	.....	6.50	6.98	6.40
2	5.69	5.39	5.90	5.96	6.65	6.17	6.09	6.54	6.04	5.70
3	6.05	5.74	6.25	6.46	6.62	6.63	6.99	7.09	6.98	6.08
4	6.22	6.12	6.33	6.88	7.18	6.74	6.45	7.14	6.95	6.22
5	6.20	6.01	6.33	6.60	6.80	6.26	6.91	.....	6.09	.....
6	6.34	6.20	6.35	6.52	6.98	6.55	6.46	7.22	6.46	.....
Solvent extracted flakes										
1	6.47	6.44	6.28	7.01	6.78	.....	.....	6.99	7.16	6.76
2	6.18	5.82	6.33	6.68	6.90	6.33	6.67	7.02	6.55	6.10
3	6.51	6.32	6.38	6.86	6.90	7.07	7.07	7.48	7.20	6.52
4	6.64	6.45	6.48	7.34	7.32	7.08	7.13	7.54	7.38	6.62
5	6.53	6.30	6.33	7.14	7.00	6.58	7.32	.....	6.99	.....
6	6.71	6.60	6.83	7.08	7.30	6.87	6.85	7.56	7.26	.....
Deviation between duplicates (all samples)										
Highest	.09	.20	.25	.18	.15	.09	.28	.04	.12	.10
Lowest	.00	.00	.00	.00	.00	.00	.02	.00	.00	.00
Average	.015	.065	.098	.068	.027	.032	.108	.018	.058	.023
Vacuum oven pressure Mm. Hg	<10	<150	<50	<30	<15	125	<12	<25	100	.....