

## Report of the Soybean Analysis Committee

THE Soybean Analysis Committee has conducted no collaborative work during the year, and its report is based on work done at the U. S. Regional Soybean Industrial Products Laboratory.<sup>1</sup>

**Moisture.** It is recognized that the determination of the moisture content of a biological material, such as soybeans and soybean meals, by oven loss methods is a purely empirical procedure, governed by three variables, temperature, pressure, and time. Hence, in determining moisture by oven loss in weight methods it is necessary to learn what combination of these three variables may be used to give the most valid results without the results being influenced by oxidation or decomposition or both. In studying moisture methods for soybeans and soybean meals and flakes, an apparatus has been used which permits the continual observation of changes in weight at various oven temperatures, at atmospheric pressure, and under vacuum (less than 1 mm.), using one sample for a complete time run without disturbing the established temperature and pressure condition (Ind. Eng. Chem. Anal. Ed., 12, 45-47, 1940). The changes in weight were read to an accuracy of 0.07 percent on a 3-gram sample.

The samples were ground in a Wiley mill using a 1-mm. sieve and brought to the equilibrium moisture content corresponding to 50 percent relative humidity and 70° F. The moisture contents determined were calculated on the oil-free basis, in order to average and compare samples without influence of differences in oil contents.

The average results for five samples of soybeans, each a different variety, are given in Table I for the specified temperature, pressure, and time factors. The rate of loss per hour, observed by inspection of the data, indicates that the results obtained at 80° C. in vacuum for 24 hours, at 105° C. in vacuum for 6 hours, and at 130° C. in air for 2 hours are comparable and approximate the real moisture content. It is quite apparent that at 105° C. in air the moisture cannot be removed completely, even with 24 hours of heating.

TABLE I.—PERCENT OF MOISTURE IN SOYBEANS ON OIL-FREE BASIS.

Hours	Oven temperature			
	105° C.	80° C.	105° C.	130° C.
	Air	Vacuum	Vacuum	Air
2	9.15	9.23	9.69	9.70
4	9.24	9.52	9.81	9.80
6	9.24	9.60	9.85	9.87
24	9.38	9.78	9.96	10.97

The results obtained on one sample each of expeller meal, toasted solvent-extracted flakes, hydraulic meal, and white solvent-extracted flakes at specified temperatures, pressures, and atmospheres are given in Table II. The rate of loss per hour, observed by inspection of the data, indicates that the results obtained at 105° C. in vacuum for 6 hours, and at 130° C. in air for 2 hours, for expeller meal, toasted solvent-extracted flakes, and hydraulic meal are comparable and approximate the

real moisture contents of the samples. For the toasted flakes, it took 48 hours at 80° C. in vacuum, and 24 hours at 90° C. and 100° C. in vacuum to approximate the results by the conditions just mentioned. Six hours at 105° C. in vacuum appeared to give the best moisture values for the white flakes.

TABLE II.—PERCENT OF MOISTURE IN SOYBEAN MEALS AND FLAKES ON OIL-FREE BASIS.

Hours	Oven temperatures						
	80° C. Vacuum percent	90° C. Vacuum percent	100° C. Vacuum percent	105° C. Vacuum percent	130° C. Vacuum percent	130° C. Nitrogen percent	130° C. Air percent
<b>Expeller meal</b>							
1	.....	.....	.....	8.33	8.64	8.47	8.54
2	.....	.....	.....	8.47	8.85	8.57	8.60
3	.....	.....	.....	8.52	8.94	8.64	8.64
4	.....	.....	.....	8.52	9.00	8.68	8.68
5	.....	.....	.....	8.53	9.05	8.71	8.70
6	.....	.....	.....	8.54	9.12	8.73	8.70
7	.....	.....	.....	8.54	9.15	8.83	8.76
24	.....	.....	.....	8.82	9.58	9.35	9.13
<b>Toasted solvent-extracted flakes</b>							
1	6.98	7.33	7.62	8.04	8.60	8.48	8.41
2	7.56	7.78	8.15	8.35	8.75	8.64	8.64
3	7.95	8.08	8.27	8.42	8.87	8.70	8.68
4	8.05	8.17	8.34	8.47	8.95	8.77	8.71
5	8.14	8.28	8.45	8.53	9.00	8.84	8.78
6	8.20	8.33	8.48	8.58	9.03	8.85	8.85
7	8.22	8.34	8.49	8.67	9.05	8.92	8.92
24	8.55	8.70	8.73	8.86	9.57	9.50	9.56
48	8.62	8.70	8.80	9.05	.....	.....	.....
<b>Hydraulic meal</b>							
1	.....	.....	.....	8.85	9.10	8.88	8.98
2	.....	.....	.....	9.00	9.28	9.05	9.09
3	.....	.....	.....	9.06	9.35	9.10	9.16
4	.....	.....	.....	9.10	9.42	9.15	9.20
5	.....	.....	.....	9.14	9.48	9.22	9.26
6	.....	.....	.....	9.15	9.56	9.30	9.29
7	.....	.....	.....	9.15	9.61	9.37	9.29
24	.....	.....	.....	9.28	10.21	9.95	10.18
<b>White solvent-extracted flakes</b>							
1	.....	.....	.....	9.03	9.15	8.98	8.73
2	.....	.....	.....	9.10	9.27	9.15	8.86
3	.....	.....	.....	9.10	9.32	9.28	8.86
4	.....	.....	.....	9.11	9.38	9.38	9.01
5	.....	.....	.....	9.14	9.41	9.46	9.05
6	.....	.....	.....	9.15	9.44	9.52	9.09
7	.....	.....	.....	9.15	9.53	9.56	9.16
24	.....	.....	.....	9.28	10.02	10.10	9.78

The combination of 130° C. and vacuum gave higher results than those of 105° C. and vacuum. It is assumed that the elevated temperature caused either the volatilization of some constituents in the sample or some decomposition. The values at 130° C. in nitrogen are appreciably lower than those for 130° C. in vacuum for the expeller and hydraulic meals and toasted flakes but approximate them for the white flakes.

There appears to be little, if any, oxidation of the expeller and hydraulic meals and the toasted flakes at 130° C. in air as the results agree surprisingly well with those obtained at 130° C. in nitrogen, where the absence of oxygen should preclude oxidation. For these three samples the increase in loss in weight with prolonged heating is assumed to be due to decomposition.

The loss in weight for the white flakes at any given time of heating at 130° C. in air was not as high as that at 130° C. in nitrogen. It therefore appears that there must have been some oxidation of this sample and a gain in weight which was not offset by decomposition.

The data significantly indicate that a very satisfactory method for the determination of moisture in ground soybeans, expeller and hydraulic meals, and in toasted and white solvent-extracted soybean flakes is to heat

<sup>1</sup> A cooperative organization participated in by the Bureaus of Agricultural Chemistry and Engineering and Plant Industry of the U. S. Department of Agriculture, and the Agricultural Experiment Stations of the North Central States of Illinois, Indiana, Iowa, Kansas, Michigan, Minnesota, Missouri, Nebraska, North Dakota, Ohio, South Dakota, and Wisconsin.

<sup>2</sup> Presented before the American Oil Chemists' Society at New Orleans, Louisiana, May 15-16, 1941.

the sample for 6 hours at 105° C. in vacuum and to report the observed loss in weight as moisture.

The tentative procedure for determining moisture in soybeans by drying at 130° C. for 2 hours in an air oven at atmospheric pressure is confirmed for soybeans, expeller and hydraulic soybean meals, and toasted solvent-extracted soybean flakes. In following this procedure for these products the time period should be counted after the oven returns to 130° C.

The suggested referee method of heating for 6 hours at 105° C. in vacuum is the only procedure that appears satisfactory for the white solvent-extracted soybean flakes.

For a rapid method of determining moisture in these products, where highest reproducible precision is not

required, it is suggested that the samples be heated for 1 hour at 130° C. in vacuum.

Repeated tests have shown that results obtained at 130° C. in a convection air oven and in a horizontal forced draft oven are identical.

*Lipids.* A considerable amount of work has been done on the study of the improvement of the present methods of determining lipids in soybeans and soybean products. Results so far are not conclusive, and no new modifications have been suggested for collaborative study.

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## Rosin Cleanliness

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**M**OST producers are boastful when making "X" rosin, but are you proud of the cleanliness of your ordinary rosin?

Rosin containing a noticeable amount of trash, which heretofore has been known as "circled" rosin, is no longer acceptable on the market. This exclusion was brought about by "loan" stipulations and a desire to place a better product on the market. A second step toward better rosins has been taken by the Naval Stores Station which aims to produce a rosin that is free of the "haze" due to fine trash particles that are almost microscopic in size. This can be done by cleaning the gum by filtration and washing before distillation, and can be approached by careful straining of fire-still rosin, using a high grade and heavy cotton batting.



Fig. 1—This rosin was WG grade, having a cleanliness of about 57% and with some noticeably large trash particles of "pebbly" appearance. This rosin would not be accepted on the market

One of the finer qualities of good rosin, regardless of grade, is its "brightness" or cleanliness, which is in reality a measure of its freedom from very fine trash or extraneous matter. A rosin containing no extraneous material would be considered as having a brightness of 100%. A poorly strained fire-still rosin, having a hazy or slightly smoky cast, would have a brightness of 50 to 60%, based on the same arbitrary standard.

Following are some photographs showing poorly strained rosin from a fire still, a well strained rosin

from a fire still where a heavy batting was used, and a rosin made from cleaned gum.

These pieces of rosin, when photographed, were held in front of a sign so the lettering could be seen through the rosin in order to better illustrate its cleanliness.

The technical staff of the Naval Stores station will be glad to discuss rosin straining and gum cleaning problems with representatives of industrial concerns which are interested in the subject.

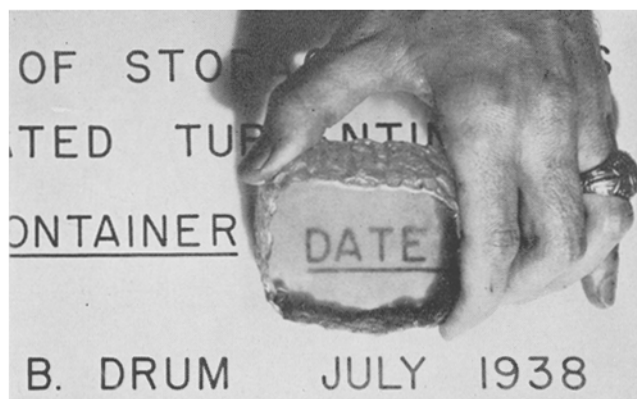


Fig. 2—This rosin was WG grade, having a cleanliness of about 83%. This compares with the best strained fire-still rosin when using a heavy batting



Fig. 3—This rosin was WG grade, having a cleanliness of about 96%. This represents the brightest rosin obtainable from cleaned gum