TABLE 7

Per Cent Linoleic in Edible Fats

	Lard	Butter	Margarine	Hydrogenated (100%)	Hydrogenated (<100%)	Mixed Animal and Vegetable
No. of samples..	27	41	57	60	31	11
High	13.7	4.8	23.4	22.4	38.2	26.2
Low	6.5	1.4	1.3	2.9	4.7	10.4
Median	11.8	3.3	9.9	12.8	21.5	23.2
Average	11.7	3.3	10.9	12.6	22.0	20.6

ings as a class than in butter, margarine, or lard, and higher still in shortenings of the groups which are made by blending hydrogenated fat or animal fat with unhydrogenated oils. The results are better summarized in Table 7 than in the concluding statement below.

Summary

Analysis of 227 samples of edible fats collected from widely scattered cities of the United States shows that the average percentage of glyceride derived from linoleic acid increases in the order: butter fat, margarine, lard, hydrogenated shortening, and blended shortening containing some hydrogenated fat or animal fat as stiffening agent.

BIBLIOGRAPHY

1. A. J. Carlson, Am. J. Public Health, *31*, 1181-90 (1941).
2. State of Kansas vs. Sage Stores and Carolene Products Co., Transcript of Testimony for Feb. 25, 1942, pp. 17-18, 24.
3. Official and Tentative Methods of the American Oil Chemists' Society, pp. 31-32 (1941).
4. *Ibid.*, pp. 44b-44d (1941).
5. Utilization of Fats, H. K. Dean, pp. 61-62 (1938).
6. Oil and Soap, *17*, 203-06 (October, 1941).

Some Observations On the Effect of Moisture On the Quantitative Extraction of Lipids From Soybeans

W. C. BULL

U. S. Regional Soybean Industrial Products Laboratory[1]

Urbana, Illinois

The fact that the amount of water present at the time of extraction has a definite effect on the amount and composition of the extract obtained from oil-seed materials by solvent extraction is well known. Milner,[2] writing as Chairman of the Soybean Analysis Committee of the American Oil Chemists Society, has given data showing that a variation of as little as one percent of moisture in certain ranges produces a difference as large as one-half of one percent in the total lipids or crude fat extracted when calculated to a moisture-free basis. Also, he indicated that the phosphorus in the higher yield of lipids may be ten times that in the lower yield. There can be no doubt that the materials removed by solvent extraction at various moisture levels are not all of the same composition. The total lipids in soybeans include, besides triglycerides of fatty acids, phosphatides, sterols, waxes, and less common substances. Soybeans and soybean oil are sources of commercial phosphatides. The variations in the phosphorus content of the lipid extract would indicate that more of the phosphatides are extracted at a high-moisture content than at a low-moisture content in the analytical sample. However, there is still no complete explanation of the variations obtained in quantitative extraction results when the moisture content of the sample has been varied over fairly wide limits. The present report does not attempt to explain these phenomena, but rather presents some further observations on the variations in quantity of lipids extracted due to moisture in samples that have had special treatment. The data obtained are presented in order that they may be of use to others doing work on methods of determining lipids in soybeans and other oil seeds and their meal products.

The soybeans used in all of the following series of samples were of the Illini variety and were from the 1938 crop. The original lot of beans was subsampled with a Boerner sampler to obtain uniform samples for the different phases of the work. In all cases the extraction solvent was a petroleum ether (Skellysolve F) and extractions were run for four hours using Butt extraction tubes. The standard quantitative oil-determination method used in this laboratory is a four-hour extraction of a 2-gram sample in a Butt extractor, the sample being removed after two hours and reground in a mortar before being replaced for the last two hours of extraction. The results are discussed on the basis of the percent of lipids in the moisture-free sample.

The samples were conditioned to various moisture contents either by being replaced for a suitable period in an atmosphere of 100 percent relative humidity at room temperature, or by drying over phosphorus pentoxide in vacuum at room temperature. The samples were ground fine enough in a Wiley mill to pass through a sieve having circular holes one millimeter in diameter before conditioning to shorten the time required.

As a standard, a series of samples having various moisture contents was prepared in which the oil was determined by the regular analytical procedure, in-

[1] The Chemical and Engineering Sections of the U. S. Regional Soybean Industrial Products Laboratory, Urbana, Illinois, were merged with the Northern Regional Research Laboratory, Peoria, Illinois, July 1, 1942. The Soybean Industrial Products Laboratory was a cooperative organization participated in by the Bureaus of Agricultural Chemistry and Engineering and Plant Industry of the Agricultural Research Administration, U. S. Department of Agriculture, and the Agricultural Experiment Stations of Illinois, Indiana, Iowa, Kansas, Michigan, Minnesota, Missouri, Nebraska, North Dakota, Ohio, South Dakota and Wisconsin.

[2] Milner, R. T., *et al.* Oil and Soap, *16*, pp. 129-131 (1939).

volving regrinding of the samples after the first two hours of extraction. The values obtained are shown in Table I.

TABLE I

Influence of Moisture on the Quantitative Determination of Lipids in Soybeans. (Samples *Reground* During Extraction.)

Moisture Contents of Samples	Lipids Extracted (Original Moisture Basis)	Lipids Extracted (Moisture-Free Basis)
Percent	*Percent*	*Percent*
5.0	19.14	20.14
8.2	18.86	20.54
8.9	18.74	20.58
9.8	18.53	20.55
10.6	18.40	20.57
11.4	18.22	20.57
13.8	17.81	20.68
16.8	17.35	20.85
23.4	15.90	20.75

Between 8.2 and 11.4 percent of moisture, the percent of lipids on moisture-free basis is practically constant. Lower moisture content gives lower percent of lipids and higher moisture content gives higher percent of lipids. These results are quite similar to those obtained by Milner (Loc. cit.).

To show the effect of regrinding, a comparison series was prepared in which the samples were extracted for four hours without regrinding. As shown in Table II, the effect of the moisture content of the sample on the percent of lipids, on the moisture-free basis extracted without regrinding, is much greater than when the samples are reground. The results are erratic and are lower than those obtained on the samples reground, indicating that all of the lipoid materials were not extracted by the procedure used.

One series of samples was pretreated before extraction by flooding the 2-gram samples with varying amounts of a 60-40 percent mixture of benzene and methyl alcohol in a tinfoil dish and heating on a hot-plate at 150° C. for 5 minutes to remove the solvent. The samples were conditioned to the desired moisture contents before the pretreatment indicated. In this case the variations due to the moisture content are complicated by the variation due to the amount of solvent added in the pretreatment and to the heating for the removal of the solvent mixture. The results of these determinations are shown in Table III. The pretreatment with no solvent mixture, i.e., heating sample at 150° C. on hot-plate without solvent for 5 minutes, gave higher results than were obtained without the pretreatment (Table II) for corresponding moisture contents of the samples, but lower results than were obtained by the procedure involving regrinding (Table I). For corresponding moisture contents the pretreatments involving the larger amounts of the solvent mixture gave results approximating those obtained by the regrinding method (Table I), except in the case of the two highest moisture levels.

A fourth series of samples was chosen from a large number which had been prepared to determine the effect of heat, time of heating, and moisture on the denaturation of the protein in ground whole soybeans. The relative humidity of the atmosphere was maintained at 100 percent for the temperatures employed.

TABLE II

Influence of Moisture on the Quantitative Determination of Lipids in Soybeans. (Samples *Not* Reground During Extraction.)

Moisture Contents of Samples	Lipids Extracted (Original Moisture Basis)	Lipids Extracted (Moisture-Free Basis)
Percent	*Percent*	*Percent*
5.4	15.92	16.83
6.4	15.84	16.92
8.2	16.03	17.46
9.2	15.91	17.52
9.4	15.94	17.60
10.0	15.83	17.60
13.3	15.38	17.75
15.15	.....	18.59
16.8	13.58	16.31
23.4	11.49	14.99

TABLE III

Influence of Moisture on the Extraction of Lipids From Samples of Soybeans Pretreated With Varying Amounts of 60-40 Percent Mixture of Benzene and Methyl Alcohol and Subsequently Heated for 5 Minutes at 150° C. (Samples Not Reground During Extraction.)

Solvent in Pretreatment (In 2 gm. Sample)	Moisture Contents of Samples (Before Pretreatment)	Lipids Extracted (Original Moisture Basis)	Lipids Extracted (Moisture-Free Basis)
C.C.	*Percent*	*Percent*	*Percent*
0	1.5	18.30	18.57
2		19.42	19.71
2.5		19.59	19.88
3		19.80	20.10
2	2.5	19.12	19.62
2.5		19.37	19.88
3		19.51	20.02
2	4.6	19.40	20.33
2.5		19.63	20.57
3		19.76	20.71
0	6.35	17.10	18.26
2		18.78	20.06
2.5		19.22	20.53
3		19.38	20.70
0	8.25	16.61	18.09
2		18.65	20.31
2.5		19.01	20.70
3		18.92	20.60
0	9.15	16.34	17.99
2		18.36	20.21
2.5		.....	20.19
3		18.59	20.47
0	9.30	16.16	17.81
2		18.66	20.56
2.5		.....	20.52
3		18.78	20.70
0	10.05	16.20	18.01
2		18.35	20.40
2.5		18.44	20.50
3		18.36	20.42
0	13.35	15.59	17.99
2		17.40	20.08
2.5		.....	19.74
3		17.02	19.64
0	15.15	15.22	17.93
2		15.96	18.80
2.5		15.64	18.42
3		15.44	18.19

TABLE IV

Influence of Heat-Moisture Denaturation of Protein in the Soybean Sample on the Extraction of the Lipids (Samples Not Reground.)

Protein-Denaturation Condition			Moisture Contents of Samples	Lipids Extracted (Original Moisture Basis)	Lipids Extracted (Moisture-Free Basis)
Time	Temperature	Relative Humidity			
Hours	*°C.*	*Percent*	*Percent*	*Percent*	*Percent*
2.5	100	100	3.25	19.42	20.08
			5.60	18.90	20.02
			6.75	18.34	19.66
0.5	127	100	4.30	18.34	19.16
			5.25	18.21	19.21
			7.40	17.72	19.14
1.0	127	100	4.65	18.80	19.72
			6.57	17.52	18.74
			8.80	17.18	18.83
2.5	127	100	0 *	.....	19.98
			4.20	18.78	19.61
			5.70	18.16	19.25
			6.60	.....	18.66
			8.30	16.84	18.36
			17.80	15.39	18.71

* By drying at 130° for 40 minutes in oven.

After the denaturation treatment described in the first three columns of Table IV, the samples were adjusted to the moisture contents indicated in column 4, and then extracted as previously. The data obtained on these samples are given in Table IV. The values, calculated on a dry basis, are low as compared to those obtained by the method involving regrinding, and do not indicate any positive trends. It is remarkable that, in general, the lipids extracted decrease with increasing moisture content of the samples in which the protein was denatured.

The quantity of lipids obtained by solvent extraction appeared to have little relation to the treatments used and the moisture content of the samples studied. Other methods and conditions of pretreatment might be devised which would show some consistent effect. The data emphasize the fact that the determination of lipids in soybeans is empirical, with the methods employed, and does not necessarily represent the total amount of this group of constituents present. They also suggest that variations in the method of processing soybeans for oil and meal may influence the amount of crude lipids or oil extracted from soybean meal by petroleum ether (Skellysolve F).

Summary

Data are presented to show the effect of moisture on the quantitative determination of lipids in soybean samples which were pretreated by: (1) Conditioning to a range of moisture contents, (2) by heating in the presence of 60-40 percent mixture of benzene and methyl alcohol, and (3) by heat-denaturation of the protein in the sample in the presence of water vapor.

The wide variation in results for lipid content indicates that the lipids removed cannot be considered as either triglycerides alone, or total lipids, but an empirical value given by a rigidly controlled procedure. It is apparent that methods are needed to determine the triglycerides and the total lipid content of soybeans and soybean meal.

Abstracts

Oils and Fats

Edited by
M. M. PISKUR and SARAH HICKS

VEGETABLE-OIL PROSPECTS IN FRENCH NORTH AND WEST AFRICA. W. N. Small. *Foreign Commerce Weekly 10*, No. 4, 8-9, 29 (1943). In return for olive oil from Algeria and French Morocco, the United Nations will supply other fats and oils.

QUANTITATIVE SPECTRAL ANALYSIS OF FATS. J. H. Mitchell, Jr., H. R. Kraybill and F. P. Zscheile. *Ind. Eng. Chem., Anal. Ed. 15*, 1-3 (1943). A spectroscopic method is described for direct detn. of the linoleic and linolenic acid content of a fat. These acids can be detd. very simply and as accurately as standard value for the pure acids can be obtained, when the fats do not contain other acids with 2 or more double bonds. Making use of the I no., the oleic acid content can be obtained; the satd. acids are then obtained by difference. Thus an analysis can be obtained on many fats (those contg. chiefly satd. acids, oleic, linoleic and linolenic acids) with as little as 0.2 g. of sample. The method is more rapid than the Kaufmann method and involves fewer detns.

COMPARATIVE SHORTENING VALUE OF SOME COMMERCIAL FATS. L. R. Hornstein, F. B. King and F. Benedict. *Food Research 8*, 1-12 (1943). Although prime steam-rendered lard ranked first in shortening value in the present expt., hydrogenated vegetable oil No. 3 was about as good, and all 3 of the hydrogenated vegetable oils were found to equal or to excel leaf lard in shortening power. Improvement in the quality of hardened fats may be responsible for this change. The butterfats were also found to be very good shortening agents in this study, particularly at the higher temps. Because of correction of the formula for the increased water and lower fat content of butter, the pastries made from butterfats in this expt. contd. a higher proportion of fat than those reported on by Lowe, so that results are not strictly comparable. Finally, there was no correlation between the breaking strengths of pastries and either the congealing pts. or the I nos. of the fats. Of the fundamental properties of the fats, only the consistency of the worked fats, probably a measure of what has been termed "plasticity," was found to correlate with shortening power. The behavior of hydrogenated vegetable oil No. 3 as a shortening agent was in accord with its known glyceride compn. This fat was an excellent shortening agent at all temps. and showed very little change in shortening power with changes in temp. Since it consisted of about 10% of fully hydrogenated cottonseed oil dispersed in the oil, the ratio of liquid to solid glycerides was high. Owing to the high m.p. of the solid material, this ratio was subject to very little change in the temp. range used in this expt. The greatly increased shortening power of the butterfats and particularly of butter oil with increase in temp. can only be explained by their phys. structure. The large quantity of low-melting compds. present in these fats probably causes the liquid glyceride content to increase more rapidly with rise in temp. than is true of most other fats and consequently causes a greater increase in the shortening value.

COMPARATIVE NUTRITIVE VALUE OF BUTTER FAT AND VEGETABLE OILS. E. B. Hart. *Am. J. Pub. Health 33*, 265-6 (1943). Expts. with young rats indicate the superiority for growth of butter fat as compared with certain vegetable oils. This superiority rests upon a constitutional difference and is not related to the differences in vitamin content. As the sole article of nutrition a "filled milk" in which a vegetable oil has been substituted for the butter fat, based on rat expts., could give inferior growth with the infant; but no such expts. have been carried out. Until they have and an equality is established, if it exists, the burden of proof of equal value must rest with the purveyor of filled milk, and the public should be protected from a substitute for natural milk.