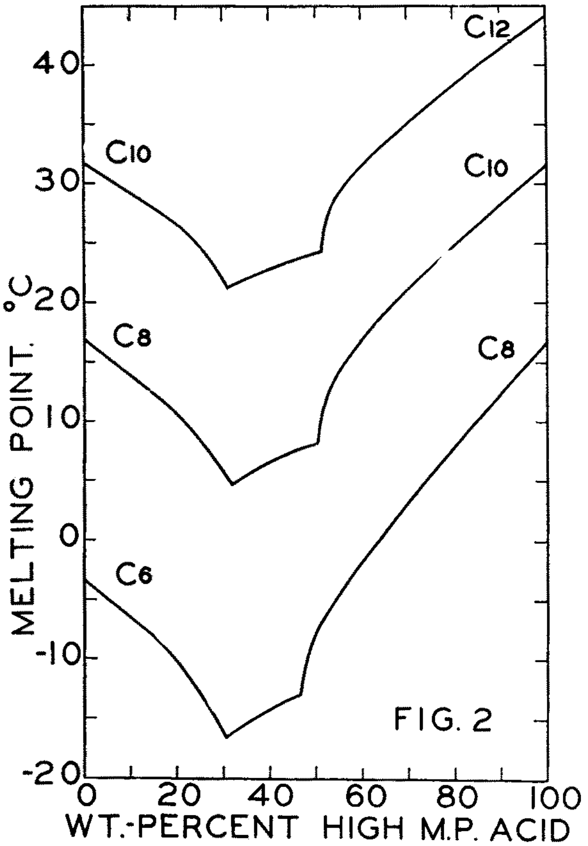

FIG. 2

of a mixture. Mixtures of pure acids were made in the proportions indicated. Their melting points were observed and molecular weight determined by titration.

In the calculations, mol per cent solute acids is obtained by dividing M.P. depression in °C. by Km, and mol per cent solvent acid by subtracting solute acids from 100. Weight per cent solvent acid is calculated by multiplying mol per cent solvent acid by the theoretical molecular weight of that acid and dividing by the molecular weight of the mixture.

TABLE III

Assay of Purity in a Mixture

Sample	1	2	3
Composition of Mixture			
Weight % C_6	0	2.95	5.78
Weight % C_8	4.01	94.01	87.71
Weight % C_{10}	91.94	3.04	6.51
Weight % C_{12}	4.05	0	0
Mol % Solvent Acid	91.74	93.81	87.42
Observed Data			
Melting Point °C	29.3	14.4	12.1
Molecular Weight	172.0	143.8	143.6
Calculated Data			
M.P. Depression °C	2.4	2.3	4.6
Km	0.285	0.36	0.36
Mol % Solute Acids	8.4	6.4	12.8
Mol % Solvent Acids	91.6	93.6	87.2
Weight % Solvent Acid	91.7	93.9	87.6
Deviation of Calculated from True Weight % Solvent Acid	−0.24	−0.11	−0.11

By the use of this method, the concentration of solvent acid may be determined with an accuracy of better than one-quarter of one per cent when present in more than 80%. For less precise work, the weight per cent solvent acid may be estimated from melting point only, using an empirical weight melting point depression constant.

Summary

The melting points of binary mixtures of adjacent even numbered carbon acids C_6 to C_{12} have been presented in table form and graphically. It has been demonstrated that by the use of the molar melting point depression constants, the purity of these acids may be readily determined to an accuracy of one-fourth of one per cent when present in a concentration of 80% or more.

The authors wish to thank Lever Brothers Company for permission to undertake and publish this work.

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Report of the Soybean Analysis Committee

(American Oil Chemists' Society)

During the past year, for the first time, soybeans have been sold on the basis of oil content as determined by the official A.O.C.S. method. A series of ten collaborative samples of soybeans was analyzed for oil by 79 laboratories. The results of these analyses permit an estimate of the reproducibility of these methods. Results on the first three samples may be neglected, since many laboratories had no previous experience in soybeans and the errors on these first three samples were correspondingly higher. On the last seven samples, ranging in oil content from 16.5 to 18.2 percent (14 percent moisture basis), the standard deviation in oil content ranged from 0.24 to 0.39 percent with an average of 0.34 percent. Elimination of the results of from one to six laboratories markedly improved these deviations. It was shown that the effect of the original grinding was quite important because much better agreement than the average was obtained in the case of one sample of ground soybeans submitted to the collaborators. These results compare favorably with those obtained on the 1943-1944 cottonseed check series where the average standard deviation on the first six samples is 0.31 percent.

An amplification of the present very brief official A.O.C.S. method is suggested for the benefit of laboratories using these methods for the first time. Such an amplification, based on the collaborative series, is given below. No changes are made, but the present method is given in greater detail. In view of the present successful use of these methods, any changes should be made with caution. Other types of extraction equipment and other extraction procedures may give the same results, but these should be carefully checked against the present official A.O.C.S. method before being used.

Work at the laboratories of two of the Committee members has definitely shown that the methods used for moisture affect the oil content determined. Apparently, if the beans are heated (predried) a higher oil content is obtained. This effect, as well as that caused by relative humidity during extraction, as reported by Krober and Collins (Oil and Soap *21*, 1, 1944), emphasizes that the present methods for moisture and oil analysis are empirical and affected by factors which must be carefully studied. Further work on "true" moisture content and further study of factors influencing moisture and oil determinations are being carried out.

Summary

Collaborative analyses on seven samples by 79 laboratories have shown that the average standard deviation in the oil determination is about 0.34 percent, which compares with a standard deviation of 0.31 percent for six cottonseed samples.

The oil determined is dependent on many factors which must be studied further.

E. C. Ainslie
F. I. Collins
C. H. Cox
Egbert Freyer
Harold Ory
S. O. Sorensen
R. T. Milner, *Chairman.*

The Analysis of Soybeans for Oil Content

(An Amplification of the Present Official A. O. C. S. Method)

Preparation of Sample: The sample which has been cleaned of dockage is put through a divider or riffle until from one-half pound to one-fourth pound is obtained. This is weighed, spread on a large pan or tray, and all foreign matter removed by handpicking. The foreign matter is then weighed and its percentage of the original sample reported if requested.

Predrying: About 60 grams is then cut out by a divider or by quartering, and placed in a shallow container with a diameter of at least 3½ inches for drying. When possible and in every case if the beans contain over 14 percent moisture, they should be dried in a warm place overnight before oven-drying. The beans are dried for two hours at 130° C. ±3° C. The temperature drop in the oven is noted, and timing is started when the temperature returns to 130° C. The temperature should be measured by a calibrated thermometer, the bulb of which is nearly at shelf level, and, if possible, the uniformity of temperature within the oven should be measured. A mechanical convection oven is advantageous in returning to temperature more quickly and in obtaining more uniform temperatures. The dried beans are kept in a tight container until ground.

Grinding: In grinding, the dried beans are reduced to as fine a powder as possible. Preferably, all of the sample should pass through a screen with 40 meshes to the inch. Soybeans are too oily to screen very successfully, but to obtain a standard some ground soybeans may be put through a 40-mesh screen by shaking and brushing. The fineness of grinding of samples may be tested then by feeling, using a sample of the screened material as standard. The mill used for grinding must be carefully tested and adjusted, and the fineness of grinding tested daily (by feel). The dried beans are exposed to the air for as little time as possible while grinding, and the ground sample is kept in a tight container and must be at room temperature before weighing for analysis. The ground sample (60 grams) is thoroughly mixed by shaking in a bottle, by spatula, or by rolling, quartering, etc. Separate samples are taken for second moisture and oil determinations, preferably at the same time.

Second Moisture: Duplicate 5-gram samples for the second moisture are weighed in aluminum dishes, approximately 2 inches in diameter and ¾-inch high, with tight covers (official A.O.C.S. or A.A.C.C. dishes). These samples are heated in an oven at 130°C. ± 3°C. for 1 hour,* with the same precautions as in the first drying. The dishes are covered before removing from the oven and cooled in a desiccator. All weighings are made with an accuracy of at least 1 mg.

Oil: Duplicate 2-gram samples for oil determination are weighed accurately from the ground sample, and each portion spread in a thin layer on 12.5- or 15-cm. filter paper of medium, unwashed grade (S+S 597, Whatman 2, Reeve-Angel 211, or equivalent). The paper is folded about one-quarter of the distance from each of two opposite sides to the center over the sample, and then wrapped into a cylinder by coiling from one of the unfolded sides. This cylinder is then rewrapped in a second paper in such a manner as to prevent escape of the meal, leaving the top of the second paper open like a thimble. A piece of cotton is placed in the top of the thimble to distribute the solvent. The Butt-type extraction apparatus with tight, well-fitted, cork connections is used. Either 50- or 100-ml. extraction flasks with 25 to 50 cc. of solvent are used. The solvent used is petroleum ether, which should be tested for residue and must conform to the A.O.C.S. specifications. The refluxing solvent should drop in the center of the thimble at the rate of at least 150 drops per minute. After two hours' extraction, the sample is removed and reground with a porcelain mortar and pestle for at least 1 minute, or 100 vigorous strokes. No abrasive is used. The sample should not be dried in an oven before regrinding, and the mortar should be at least 4 inches inside diameter at the top, with a pestle large enough to permit a firm hand-grip and vigorous regrinding. After regrinding the sample and rewrapping in the same papers, extraction is continued as before for 3 hours additional. The solvent is evaporated until no trace remains. The procedure used for this purpose should be checked initially and rechecked at intervals by repeating the heating until constant weight is obtained. Traces of petroleum ether cannot always be detected by odor. Aeration of the extraction flask while heating is frequently desirable.

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* The official method calls for a drying time of 2 hours for the second moisture.