

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.

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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 hand-picking. 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 rewinding 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.

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