An Evaluation of the Soxtec System for Oil Determination in Soybeans

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The Soxtec System and the AOCS method were compared for oil determination in soybeans, and no significant difference was found between the two methods. The Root Mean Square Error (RMSE) and standard deviation for individual samples were higher for the Soxtec, but this was attributable to the regrinding step used. The study suggests that the Soxtec System is faster and just as accurate as the AOCS method.

The standard AOCS method for the determination of oil content in oil seeds by extraction can be extremely time-consuming, requiring 5 hr of extraction time. A more rapid extraction method in which solvent is equilibrated with dissolved oil inside and outside the soybean particles rather than an exhaustive extraction to remove oil has been reported (1). Nuclear magnetic resonance (NMR) and near infrared reflectance (NIR) have been used to reduce the time needed for this determination requiring only seconds compared to hours for a determination (2). However, both instrumental methods depend on an extraction method to provide a primary standard or reference sample.

It became necessary to determine the oil content of a large number of soybean samples in a short period of time. It was necessary to use an extraction procedure which was as accurate as the standard AOCS method but required less analysis time to serve as a primary standard for two instrumental methods. The method we developed involves an extraction, regrinding, and another extraction using the Soxtec System (Soxtec) produced by Tecator. The Soxtec is an extraction system in which a thimble containing the oil-bearing matrix is immersed in a boiling solvent. The thimble can be raised so that the refluxing solvent can wash or rinse the last traces of oil from the thimble. The Soxtec has been compared with the Goldfisch apparatus for oil determination in a number of oil-containing materials (3). It was found that immersion time, rinse time and the temperature of the circulating water for the heating plates were critical factors in oil determinations. The methods were considered equivalent; however, the Soxtec gives slightly lower values. When particle size was reduced and optimum conditions for each individual sample developed, this difference could be reduced. This report will cover the method developed using the Soxtec, and its comparison with the standard AOCS method for oil determination for soybeans.

MATERIALS AND METHODS

Soybeans were stored in sealed plastic bags until grinding. Samples were ground in a Cyclotec 1093 Sample Mill to pass a 2mm screen (74 holes/sq in) and stored in sealed jars. Samples (2 g) were taken from the jar alternating between weighing for the AOCS and the Soxtec methods in order to offset any change in moisture due to exposure to the atmosphere. Oil determinations were run in triplicate for each method. Only one set of two samples run in triplicate were run by each method each day. Moisture samples (5 g) were run in duplicate in a forced draft oven at 130°C for 2 hr and were taken as the first and last samples weighed from each bottle. Differences between methods of oil determination were analyzed by one-way analysis of variance using SAS (4).

Extraction. The Official AOCS Method Ac 3-44 (5) using the Butt type extractor was followed except for the grinder used and the samples not being predried.

Soxtec method. The ground samples were added to tared thimbles (Whatman cellulose thimbles, 26mm × 60mm, single thickness), cotton was placed in the top of each thimble, and the thimbles were placed in the apparatus. A tared cup containing a boiling stone and 45 mL 30–60°C petroleum ether was then placed in the apparatus. The thimbles were lowered to the "boiling" position, and heat was applied to the plates. The temperature of the circulating heating fluid was 68°C. The condensers were connected to a recirculating cold bath maintained at 10°C. After 1 hr the thimbles were raised to the "rinsing" position for 5 min and the heat reduced. The thimbles were removed and placed in the hood, and each sample was reground in a mortar and pestle for 30 sec. The sample was returned to the thimble, and fresh cotton was placed in the top of each thimble. Five to 15 mL of solvent were added to make up for that lost during the first boiling and evaporation before regrinding. Samples were boiled for an additional hr, and raised to the "rinsing" position for 20 min. The solvent collection knob was closed and when no additional solvent could be seen collecting in the condensers, the cups were removed and placed in the hood to remove last traces of solvent before final drying for 30 min in a forced draft oven at 130°C. After cooling in a desiccator, the samples were weighed and dried a second time for 15 min or until a constant weight was reached.

RESULTS AND DISCUSSION

In preliminary studies, we found no statistical difference in oil content between samples with an "as is" moisture of 8.5 to 11% run on the Soxtec System and samples which had been predried and run by the AOCS method. In addition, no difference in oil content was found using the Soxtec varying the time from a 60 min "boiling," 20 min "rinsing" to a 2 hr "boiling," 20 min "rinsing" with no regrinding. However, with the addition of a regrinding step, the oil content increased (Table 1). Six samples were run in duplicate. One set was boiled for 2 hr followed by a 20 min rinse. The second set was boiled for 1 hr, reground, boiled an additional hr and rinsed for 20 min. The results show

TABLE 1

Effect of Regrinding on Soxtec Oil Determination[a]

Sample	No regrinding	Regrinding	Change in oil with regrinding
1	19.86 ± 0.08	20.50 ± 0.11	+0.64
2	19.13 ± 0.09	19.48 ± 0.18	+0.35
3	19.36 ± 0.01	19.38 ± 0.01	+0.02
4	18.65 ± 0.05	19.38 ± 0.19	−0.22
5	18.80 ± 0.01	19.04 ± 0.06	+0.24
6	19.85 ± 0.01	20.08 ± 0.00	+0.23
Average	19.26	19.47	+0.21

[a] Oil % reported on "as is" basis ± standard deviation of duplicate analysis.

TABLE 2

Comparison of Oil Determination by Soxtec System and AOCS Butt Tube Extraction[a]

Sample	Soxtec	Butt Tube	Moisture
1	18.35 ± 0.11	18.17 ± 0.04	9.27
2	20.24 ± 0.10	20.17 ± 0.03	8.59
3	18.96 ± 0.33	18.81 ± 0.03	9.25
4	19.89 ± 0.14	19.83 ± 0.12	10.21
5	20.58 ± 0.09	20.50 ± 0.06	9.81
6	18.33 ± 0.22	18.57 ± 0.05	10.37
7	18.29 ± 0.04	18.28 ± 0.02	11.12
8	19.51 ± 0.12	19.48 ± 0.03	9.38
9	20.05 ± 0.22	20.02 ± 0.05	10.31
10	18.65 ± 0.08	18.85 ± 0.04	9.73
Average	19.28 ± 0.85	19.27 ± 0.81	
RMSE	0.17	0.05	

[a] Oil % reported on "as is" basis ± the standard deviation of triplicate analysis.

the oil content of the reground samples averaging 0.21% higher. Although this difference was not statistically significant, a regrinding step was added. We also found that a heating temperature just high enough to produce a vapor ring in the lower third of the condenser was sufficient without causing foaming or bubbling above the thimbles. Excessive solvent was lost if the water temperature in the condensers was much above 20°C.

The results of the comparison of the AOCS and Soxtec methods for oil determination in soybeans are shown in Table 2. Statistical analysis shows no difference ($p > .05$) in the two methods for oil determination. The root mean square error (RMSE) for the Soxtec is 0.17 compared to 0.05 for the AOCS method. The average standard deviation for individual samples is also higher for the Soxtec. This could be attributable to sample manipulation involved in the regrinding step which is not needed for the butt tube extraction.

In an unpublished study 120 soybean samples ranging from 13 to 24.2% oil and 8.4 to 11.6% moisture were evaluated for oil content in the same ground sample—first by pulse NMR and then by the Soxtec system described. Since the NMR was calibrated using oil as the standard, this procedure measures only oil; the oil value is independent of an extraction value. There was no statistical difference ($p > .05$) between the two methods for oil determination. Details of the NMR method will be published in a separate study.

In this study only two samples run in triplicate were evaluated each day by each method in order that sample preparation be identical for each method. Using a Soxtec System with a capacity for six determinations, three sets of six determinations could be run per day as compared to only one set for a Butt tube apparatus with a six sample capacity; however, this required meticulous planning. Also some improvement in time utilization could be realized by undertaking extensive studies to find the best grinder and shortest exposure times to boiling that would give results equivalent to the AOCS values.

This study suggests that, under the conditions in this study, the Soxtec System provides oil determination results that are in good agreement with those obtained from the AOCS method. It must be pointed out that there were no detailed studies to optimize conditions in order to minimize time requirements or the degree to which the sample must be handled (regrinding). This study suggests that the Soxtec System could well be used as a primary standard for other methods.

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