technology at the University of Guelph in Ontario in 1969 and his doctorate in nutrition-lipid biochemistry at McGill University in 1971.

Known as a defender of the natural qualities of milk and dairy foods, he once wrote in a scholarly article concerning a controversial food stabilizer: "There is no reason to use a non-nutritive stabilizer when a nutritional product of higher value can perform the same function or even better." He regularly attended the scientific and administrative meetings of the American Dairy Science Association, American Cultured Dairy Products Institute, Institute of Food Technologists and AOCS, presented papers at their meetings and published extensively in their journals.

Dr. Amer was first recognized for his work on yogurt, which showed that the natural beneficial bacteria used to make yogurt liberated higher levels of essential amino acids. He also was interested in the scientific and medical aspects of fats. In 1973, he and his colleagues produced a "new milk" with reduced levels of saturated fats. He also did significant work on new types of foods, such as spreads combining butter and vegetable oils. Most recently, he helped establish a program of medical and technological research for the Dairy Bureau of Canada.

Fellowship set up

The Anthony M. Schwartz Fellowship in Chemistry has been established at the American University in Washington, D.C., in memory of Dr. Schwartz, who died earlier this year. He had been a member of AOCS from 1950 until his death.

Anyone wishing to make contributions to this fellowship may contact Jane K. Schwartz, 2260 Glenmore Terrace, Rockville, MD 20850.

AOCS Deadlines

The following deadlines have been set for various AOCS activities. For more information, contact AOCS, PO Box 3489, Champaign, IL 61826-3489, USA, telephone 217-359-2344.

Sept. 1 — Early registration cut-off for the 1989 AOCS world conference on processing to be held in Maastricht, The Netherlands. After this date, prices increase. Also, submission of AOCS officer candidate nominations for the Governing Board for 1990-1991.

Sept. 29 — Early registration cut-off for the AOCS/CSMA Surfactants and Detergents Industry Conference to be held Oct. 29-Nov. 1, 1989, in Hershey, Pennsylvania.

Oct. 3 — Submission of nominations for the 1990 AOCS Honored Student Awards, the 1990 Potts Memorial Fellowship and the Northeast Section's 1990 Hans Kaunitz Award.

Oct. 15 — Submission of paper proposals for the 1990 AOCS annual meeting.

Oct. 26 — Early registration cut-off for the Short Courses on Analysis of Fats, Oils and Lipoproteins to be held Nov. 26-30, 1989, in Rosemont, Illinois.

Nov. 1 — Submission of nominations for the 1990 AOCS Award of Merit, the 1990 Supelco AOCS Research Award, the A. Richard Baldwin Distinguished Service Award, and the North Central Section's 1990 Alton E. Bailey Award.

METHODOLOGY

Total oil analysis of oilseeds

The following article is based on highlights of a report on the status of total oil analysis of oilseeds. The report was prepared for the AOCS Seed and Meal Analysis Committee for the May 1989 meeting in Cincinnati by committee chairman James Daun of the Canadian Grain Commission and Harry Snyder of the University of Arkansas.

A number of reasons that have developed over the past few years make it necessary to modify the American Oil Chemists' Society's methods for determining oil content in oilseeds. The first section of this report discusses these reasons. In the second section, some recommendations are offered for consideration.

There seems to be a lack of precision in the total oil analysis of soybeans and other oilseeds. Doughtie (1) in 1947 pointed out that a reproducibility of ±0.2% oil could be achieved for the analysis of soybeans by different laboratories. Collins (2), commenting in 1953 that the range in Smalley results was about 1%, attributed much of the variation to the method of preparing the sample rather than to the extraction technique. The few standard methods including error statements mostly show reproducibilities in the order of 1% to 2% (Table 1). Current Smalley data for oil content determination (Table 2) show reproducibility on the order of 1-3% for all series except cottonseed, where the reproducibility (0.4% to 0.8%) is close to the stated 0.73%, and sunflowerseed, where the reproducibility (2.2%) equals the stated 2.2%. Reproducibility for the latter seems larger than it could be.

The current edition of *Official Methods and Recommended Practices of the American Oil Chemists' Society* lists 10 analytical procedures for determining the oil content of specific oilseeds, with no method listed for rapeseed or canola. Some of these methods require heating of the samples, some require regrinding between extraction, and most vary in the method used to prepare the sample. Although each of these methods has been developed for a particular oilseed, many (such as the method for flaxseed or tung fruit) are not in common use. There is an opportunity to combine many of these methods and simplify the analysis for total oil in oilseeds.

There are four major international organizations which deal with methods for oilseed analysis. They are the Federation of Oils, Seeds and Fats Associations (FOSFA), the International Association of Seed Crushers (IASC), the International Standards Organization (ISO) and the International Union of Pure and Applied Chemistry (IUPAC). All have similar methods for determining total oil content, reflecting the recent trend towards normalization of international methods. A recent comparison of three of these methods (FOSFA, IASC and ISO) showed little difference in results obtained in a single laboratory (3).

All four methods recommended by the international agencies allow the use of a Butt-type extractor; only the FOSFA method does not allow the use of a Soxhlet extractor. They all require exhaustive extraction with an initial extraction period of four hours (two hours for IASC) followed by at least two further two-hour extractions, each of which is preceded by micro-grinding of the previously extracted material. The methods are designed for oilseeds in general; although the extraction procedure is the same for all oilseeds, different sample preparation procedures are set forth for different types of oilseed.

Repeatability of the methods from these international organizations is in the order of 0.4–0.6% (about the same as AOCS Method Aa 4-38 for cottonseed, which is similar in its sample treatment and extraction technique). Methods requiring re-extraction and regrinding are time-consuming and laborious, but have the advantage of uniformity and complete extraction.

TABLE 1

Some Current Methods for Determining Oil Content in Oilseeds[a]

Method	Grinders	Particle	Extractor	Solvent	Error
FOSFA	Mechanical mill Mechanical micro-grinder	<160 μM <400 μM (shell)	Butt No Soxhlet	Hexane or petroleum ether <70°C	0.6% repeat
IASC	Mortar, pestle, sand or mechanical mortar or micro-grinder		Butt or Soxhlet	Petroleum ether 40–60°C or hexane	0.4% repeat
ISO	Mechanical mill Micro-grinder or mortar & pestle	<160 μM <400 μM (shell)	Suitable	Hexane or petroleum ether <70°C	0.4% repeat
IUPAC	Mechanical crushing mill (large seeds) Micro-grinder		Suitable	Hexane or petroleum ether 50–70°C	–

[a]All methods specify 10 g sample.

TABLE 2

Variance and Reproducibility of Oil Content Determinations from Smalley Check Sample Program (1987)

Seed	S_R[a] Min.	S_R Max.	R[b] Min. (%)	R Max. (%)
Cottonseed	.152	.288	.43	.82
Soybeans	.241	.542	.68	1.53
Peanuts	.285	.567	.81	1.24
Sunflower,				
clean	.338	.803	.96	2.27
as is	.380	.810	1.08	2.29
NMR	.203	1.101	.57	2.83
Safflower/Rapeseed	.241	.817	.68	2.31

[a] S_R = Standard error between laboratories. [b] R = Reproducibility.

Source: Firestone, D., J. Ridlehuber and D. Berner, *J. Am. Oil Chem. Soc. 65*:1427 (1988).

Several recent developments should be considered when contemplating changes in methodology:

- The SoxTec Extractor allows the sample to be immersed in boiling solvent during the first part of the extraction. The sample may then be lifted above the solvent and held for washing free of oil by condensing solvent as in a conventional Butt tube extraction. The advantage is decreased extraction time (4).
- Snyder and colleagues (5) investigated particle size as a variable in oil extraction of soybeans and reported that particles of about 150 μM (passing through a 100-mesh screen) or less in diameter gave complete extraction in four hours. Between 2% and 3% less oil was extracted from particles obtained by grinding soybeans with a UDY cyclone mill with no screen than from particles that had passed through a 100-mesh screen.
- Results comparable to those from exhaustive extractions can be obtained from equilibrium procedures in much less time. In equilibrium procedures (5–8), solvent is equilibrated with oilseed tissue, and the tissue-free solution is analyzed for total oil content. Provided that the particle size is small, equilibrium can be achieved in a few minutes to about an hour. The grinding may be carried out in the presence of the solvent.

(Continued)

- Physical methods making use of nuclear magnetic resonance (NMR) and near-infrared reflectance (NIR) are rapid and nondestructive. NMR has been used by the U.S. Federal Grain Inspection Service in the routine determination of oil content in sunflowerseed for establishing a premium or discount. NIR will likely be the method for rapid analysis of soybeans for oil, protein and moisture in a proposed system to compensate for quality differences in buying and selling of soybeans (9). Reliable wet chemistry procedures are required for calibration of NIR equipment.

The "total oil" determined by any oil content method is a mixture of lipid material having different polarities and hence different solubilities in different solvents. Sample pretreatment, particle size and solvent characteristics all play a role in determining how much lipid is removed from the seed. "Oil content" is not easy to define in terms of any given lipid components. The "oil content" of any oilseed is thus a function of the method used in the determination, and an "accurate" measure of oil content should include the method used as part of the result. The most commonly used solvents in the determination of oil content are diethyl ether, hexane and petroleum ether.

Hexane or petroleum ether has been preferred over diethyl ether as a solvent for determining oil content mainly for safety reasons because diethyl ether tends to form dangerous peroxides and must be kept dry.

Studies are required to quantify any differences between the solvents listed in their efficiency for extracting lipid material from oilseeds. More polar lipid is extracted with the diethyl ether due to its polarity.

Clark and Snyder (10) showed the amount of phospholipid extracted during analysis is dependent on the temperature of pretreatment of soybeans and on the moisture content of the soybeans. The higher the temperature or moisture content, the more phospholipid is extracted.

A final factor to be considered is the relationship of the analysis of oil content to the purpose of the analysis. If the purpose is excellent accuracy and precision, a method using multiple extractions, such as the FOSFA method, would be the method of choice. If the purpose is to reproduce what is going to be extracted commercially, the seeds should be cooked and flaked before extraction. If the purpose is to extract all the lipid material, a polar solvent such as chloroform:methanol (2:1) would be appropriate.

Recommendations for action

AOCS methods Ai 3-75 (sunflower), Ac 3-44 (soybean), Af 3-54 (flaxseed), Ag 1-65 (safflowerseed), Aa 4-38 (cottonseed) and Ab 3-49 (peanut) should be replaced with a single method such as the FOSFA method. This move should be done in conjunction with the movement within Europe to produce normalized standards by 1992 and, if possible, the AOCS method should be the same as the method adopted by the EEC. Studies should be carried out to determine if the times for the extraction could be reduced with no loss of accuracy and precision. Since large particles extract more slowly than small particles, long extraction times should not be necessary if fine grinding is achieved. The use of Soxhlet-type analyzers could be included, provided sufficient evidence is given of their equivalency to Butt tubes.

The three AOCS methods for tung fruit and related products (Ad 3-52, Ad 5-52 and Ad 6-52) should be made into one method which includes the differences in handling.

Experimental investigations should be carried out to see if the AOCS method for determining oil content of castor beans (Ae 3-52) could be incorporated into the general method.

Further experimentation may allow the development of a rapid equilibrium method by which the oilseed tissue is broken up in a closed homogenizer system. This method could be adopted as a supplement to the general method once sufficient experimentation is carried out.

Further experiments need to be conducted to evaluate the effectiveness of different solvents (11). These experiments should be carried out on as many different oilseed types as possible, using very fine flours so that particle size would not be an uncontrolled variable.

Instrumental methods such as the ISO NMR procedure and the BSI NIR procedure should be adopted immediately as part of AOCS recommended practices. Work is required to modify the calibration procedure for the NMR method to allow the use of whole seed standards (as in the AOF method).

The overall goal should be to develop several good general methods for oil content determination, each of which has a particular usefulness that is spelled out in the scope of the method.

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Committee meetings

The following AOCS technical committees have elected to meet in Chicago this fall: Commercial Fats and Oils, Seed and Meal Analysis, NMR and Uniform Methods. The tentative meeting date will be Friday, Dec. 1, 1989, in the vicinity of Chicago O'Hare Airport. Further details will appear in this column as soon as they are finalized.

The AOCS technical committees are open committees. Anyone wishing to participate in committee projects and meetings may do so. Please contact AOCS Technical Director Dave Berner at AOCS headquarters, PO Box 3489, Champaign, IL 61826-3489, USA, telephone 217-359-2344, for further details.