Chapter 1

The Commercial Significance of Oil Content Analysis: The Position of Official Methods

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Introduction

There are many anecdotal claims that “the error in the measurement of proximate X is costing/losing the industry millions.” Such a charge has been heard in the methods and commodity committees of many national and international organizations. On the other hand, contractual specifications between suppliers and consumers of raw materials are being written much more tightly than ever before. The result is that the precision and accuracy of the methods of analysis used to support these contracts are routinely being questioned. Such circumstances have led the Federation of Oils, Seeds and Fats Associations Ltd. (FOSFA International) to study the contractual method for sunflower seed oil content and modify it to include the determination of moisture, both before and after grinding before the oil extraction step. The original FOSFA Contractual Method was previously adopted by ISO/TC 34/SC 2 (Oleaginous Seeds and Fruits and Oilseed Meals) and developed as ISO 659; it is also reproduced as AOCS Am 2-93.

Other standards development organizations (SDO) such as AOCS, AOAC International, CEN, ISO, and Codex Alimentarius are faced with similar problems as the globalization of world standards follows the need to open up world trade. The existence of many versions of the same analytical method in the standards arena is complicated by the routine practice of translating these standards into company standard operating procedures (SOP) and the existence of more variant methodologies. Differences in regional customs, training, and language also contribute to the diversity of analytical methods.

All of these considerations have a large effect on both the trade of oilseeds and the introduction of new or modified, value-added crops into the specialty and niche markets and the acceptance of improvements to existing commodity oilseeds.

Oil Markets

Trade in oils and oilseeds depends on the purchaser being able to determine the yield and subsequently the price of value-added products. The oilseed crushing industry depends on the sale of crude oil and meal for its income stream. In commercial trade,
commodity prices are set by the major grain exchanges (e.g., Chicago Board of Trade) and by contractual agreement. Discounts and premiums may be paid on contract specifications depending on whether the purchased lot fails to meet, meets, or exceeds these specifications. The acceptability of the lot depends on the analytical results of samples collected according to specific sampling protocols, the price paid, and the anticipated yield of products.

Soybeans have a nominal composition of 38% protein, 15% soluble carbohydrate, 18% oil, 14% moisture and ash, and 15% insoluble carbohydrates (dietary fiber). Soybean crushers (processors) buy beans on the expectation of making a profit from the production and sale of soybean meal and oil.

The soy crush margin can be calculated as follows: Margin = [(price of soybean meal × pounds of meal per bushel) + (price of soybean oil × pounds of oil per bushel)] – price of soybeans per bushel (1). According to USDA statistics, the margin has rarely been above $1/bushel and has generally been ~$0.75 over the last 15 y (2). Crush margins assume that 1 bushel of soybean (60 lb) yields 48 lb of meal with 44% protein and 11 lb of oil. Because soybean quality varies among varieties and from region to region, the actual yield of oil and meal differs from the assumption above.

A calculator to determine the Expected Processing Value (EPV) may be found at www.stratsoy.uiuc.edu. This tool, based on a publication by Brumm and Hurburgh (3), determines the value of soybeans on the bases of their protein and oil content. It uses these values together with commodity prices quoted on the Chicago Board of Trade for soybean meal and oil to determine the processed value. Although there are some assumptions made in the calculation, valuable potential revenue information can be determined (Table 1.1). Profitability is further related to operational efficiency, transportation logistics, and capacity utilization. All of these factors play an important role in the final margin realization.

Any number of potential scenarios can be run using EPV to determine the magnitude of the effect of protein and oil content on profitability. At present, the value of the soybean is limited by the values of protein and oil content. Limits are set by the available commodity seed stocks. However, because greater profitability exists in sourcing higher-quality raw materials, soybean buyers are aware of this opportunity and search out these stocks.

**TABLE 1.1**
Example of Output from an Expected Processing Value (EPV) Calculator

<table>
<thead>
<tr>
<th>Calculation inputs</th>
<th>$/bu</th>
</tr>
</thead>
<tbody>
<tr>
<td>Soybeans</td>
<td>4.40</td>
</tr>
<tr>
<td>Calculation results</td>
<td>$/bu</td>
</tr>
<tr>
<td>Meal</td>
<td>3.74</td>
</tr>
<tr>
<td>Oil</td>
<td>1.44</td>
</tr>
<tr>
<td>Hulls</td>
<td>0.14</td>
</tr>
<tr>
<td>Subtotal</td>
<td>5.32</td>
</tr>
<tr>
<td>Theoretical margin</td>
<td>0.92</td>
</tr>
</tbody>
</table>
Quality parameters also affect the international trade in oilseeds. The recent acknowledgment of genetically modified soybeans in Brazil removes some of the competitive advantage of soybeans from that area. However, increased yields and increased production in South America are driving U.S. producers to look at the quality of U.S. soybeans to determine a competitive advantage. Production and consumption of oilseeds generally go hand in hand. Reviews of the production of oilseed and oils and fats are regularly produced by world experts Frank Gunstone and Thomas Mielke and may be found in the AOCS membership publication inform (4–6).

**Development of a Quality Assurance Program**

Evidence is available from the USDA, the United Soybean Board (USB), and other sources detailing the effect of location within the United States on soybean quality. Each industry would, within the limitations of economic feasibility, source oilseeds with the highest quality. In a commodity-based industry in which the farmer is paid on yield, and profits are determined after the fact, sourcing suitable oilseeds for crushing is more of an art than a science. However, the oilseed trade could be revolutionized if oilseeds with higher levels of protein and oil or other desirable constituents were available on the U.S. market on a regular universal basis. This latter goal is the driving force for the USB Better Bean Initiative. For further details of this program visit www.unitedsoy.org. The program promotes the development of soybeans with enhanced quality traits such as increased oil and protein content, enhanced amino and fatty acid composition, and low phytate content while maintaining yield. Because differences in the precision and accuracy of methods of analysis will hinder the introduction and identification of new varieties of commodity oilseeds, this program has also recognized the need for analytical performance. In 2002, USB sponsored a 4-y program with AOCS to introduce the Soybean Quality Traits Analytical Standards Program (SQT) (Table 1.2).

With the initiation of a proficiency testing program to support SQT, AOCS is currently (end of 2003) proceeding with phases 3 and 4. In parallel, the development of a program to support the use of near infrared (NIR) spectroscopy for the analysis of soybean quality traits is underway. In an ambitious and highly cooperative program, the seed breeders, NIR equipment manufacturers, analytical laboratories, consultants, and academics have come together to foster the development of a library of diverse soybean samples to support NIR calibrations for protein, oil, and fatty acid composition. In future developments, it is anticipated that phytate, amino acid composition, and other analytes will be included.

**Factors Determining the Use of Globally Recognized Standards**

When considering how complex the soybean industry appears, how integrated the activities and the wealth of expertise available, it is easy to question the need for an analytical system to ensure confidence in the identification of crops with enhanced
traits. In fact it is the complexity of the current model that drives the need for standardization. Both internal and international trade in oilseeds require the assurance of analytical values.

When contracts specify the expected level of an analyte, they may also specify the method of analysis. This recognizes the variability among different methods of analysis and their performance characteristics, and many disputes are averted because of this understanding. However, these differences in the methods of analysis are inherent from their empirical nature. AOCS has methods for the determination of oilseed fat content dating from the 1930s. At the time of their introduction, they were considered state of the art and an industry standard. Still in use today, they remain the methods of choice in arbitration, and closely related versions of the same methods can be found in the collections of many SDOs.

**Fats, Oils, and Lipids Methods Standardization**

There is a long list of developers of national and international standards. Although national standards bodies generally adopt standards methods that have been developed through international cooperation, they may also be developed internally in response to the needs of the trade or other organizations. These standard methods often form the basis of future international methods. In the United States, national standards are published by ANSI, although the developmental work may be carried out by any of a large number of professional organizations. However, in this arena,

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**TABLE 1.2**

<table>
<thead>
<tr>
<th>Phase</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>Phase 1</td>
<td>Identify analytical methods for Protein content, Oil content, Fatty acid composition analysis</td>
</tr>
<tr>
<td>Phase 2</td>
<td>Develop and validate methods of analysis including the evaluation of secondary methods</td>
</tr>
<tr>
<td>Phase 3</td>
<td>Identify users and their requirements Seed companies, Referee and private laboratories, End-user laboratories, Elevator and crop handling facilities, Establish a core group of expert laboratories</td>
</tr>
<tr>
<td>Phase 4</td>
<td>Develop Soybean Quality Traits Laboratory Program, including use of proficiency testing and standards</td>
</tr>
<tr>
<td>Phase 5</td>
<td>Implement laboratory quality assurance Standard methods, Certification, Proficiency participation, Results monitoring</td>
</tr>
<tr>
<td>Phase 6</td>
<td>Encourage incorporation of SQT methods of analysis into ISO 17025 certification and quality audits</td>
</tr>
</tbody>
</table>
AOCS is recognized as an international nongovernmental organization (NGO) with wide global representation.

Most standards organizations publish their own standards and rely on volunteer committees to carry out the developmental and technical work. This generally requires the provision of precision data that are based on collaborative studies as defined in either ISO 5725:1994 (7) or the IUPAC/AOAC Harmonized Protocol (8). Both standards allow the user to calculate the number of laboratories and samples required to provide a good estimate of precision. In most cases, results from 8–15 laboratories for 3–5 samples covering the level of expected values are required for each analyte and matrix covered by the method. The IUPAC/AOAC protocol requires the participation of 5 international laboratories, whereas ISO stipulates that 5 countries agree to participate in the development of the method. There are also minor differences in the statistical treatment of the data to determine outliers, although this does not generally affect the outcome of the analysis of the collaborative study.

ISO (International Organization for Standardization; www.iso.org) standards for the fats and oils community are developed by Subcommittees 11: Animal and Vegetable Fats and Oils (TC 34/SC 11) and 2: Oleaginous Seeds and Fruits and Oilseed Meals (TC 34/SC 2) of ISO Technical Committee 34 Food Products (ISO/TC 34). ISO uses a 6-step consultative process to ensure consensus and participation of interested parties. One of the characteristics of ISO standards is that the standard itself may contain only references to other appropriate ISO standards. In recent years, however, ISO has recognized that the source scientific literature is relevant to ISO standards and may be included in Appendices to methods.

CEN (European Committee for Standardization; www.cen.be) publishes standards that meet the specific needs of European countries in response to the needs of European industry and the regulations of the European Commission. Fats and oils are handled by TC 307 “Oilseeds, vegetable and animal fats and oils and their by-products—Methods of sampling and analysis.” Under the Vienna Agreement, ISO and CEN agree to cooperate in the development of standards without duplication. Work started by CEN may be transferred to ISO, and CEN adopts ISO standards wherever possible. Methods are developed by a process similar to the ISO process.

The International Union for Pure and Applied Chemistry, IUPAC (www.iupac.org) has had a long history of developing methods of analysis for fats and oils. Many of these have formed the basis for harmonization efforts in the fats and oils arena and have been adopted and refined by sister organizations. Although the members were a very active group of dedicated scientists, the Fats and Oils Commission was incorporated first into the Food Chemistry Division, and the latter group was absorbed into the Division for Chemistry and the Environment as IUPAC moves onto a grant-based/project-based program.

AOCS (www.aocs.org) supports and maintains an active standards development program and publishes methods for the fats and oils industry in the *Official Methods and Recommended Practices of the AOCS*. This compendium contains >400 methods of analysis for oilseeds, oils, fats, and their derivatives. *Additions and Revisions*
are published annually and the whole volume reviewed on a 5-y cycle. In line with the expectations of ISO 5725 and the IUPAC/AOAC harmonized protocol, new methods are studied collaboratively before publication as Official Methods. Recommended Practices are those methods either with a limited scope, incomplete validation, or urgently needed by the fats and oils industry. The methods program is supported by an Editor-in-Chief, currently David Firestone, the Uniform Methods Committee (UMC), and a number of technical subcommittees of experts in particular methods of analysis or matrices.

AOAC International (www.aoac.org) has a long history of providing methods of analysis. Its method validation programs comprise the AOAC® Official MethodsSM Program®, a Peer-Verified MethodsSM Program®, and the AOAC® Performance Tested MethodsSM Program. In addition, AOAC International is in the process of developing an online methods resource (eCAM) that will be a compilation of analytical methods from many organizations. A method published under the AOAC® Official MethodsSM Program® requires collaborative study data from a minimum of 8 independent laboratories. Peer-Verified MethodsSM provide a rapid way for methods to be recognized by a standards writing body at an entry level of validation.

National Professional Associations

IUPAC, AOCS, and AOAC International are some of the international professional associations with standard methods development programs; however, there are also many national associations that publish standard methods either in the national language or because they meet specific regional needs.

Trade Associations

Industrial trade associations may require special methods to be used by their members to support the exchange of goods. Although many methods have been proposed for adoption as national or international standards, for others, there is an insufficient amount of precision data available or a more generic method is already available on a national or international basis. Some trade methods have been retained to ensure the continuity of trade because a newer international standard may give slightly different results.

In the area of fats and oils, several trade associations play a role in developing analytical methods for their members. FOSFA International (www.fosfa.org), a world leader in the area of fats, oils, and oilseeds, maintains a technical manual that lists methods that are to be used as part of trading contracts among its members. The manual lists methods developed by international groups such as ISO, IUPAC, and AOCS, and maintains FOSFA methods when no official methods are available.

Process for AOCS Approval of Official Methods

Methods submitted for inclusion are first screened by the Technical Department of AOCS and then evaluated by one of the subcommittees. The response of the sub-
committee is relayed to the author or proposer, and the revised proposal is considered by the subcommittee. If more validation data are required, the method may be considered as a Recommended Practice and forwarded to the UMC or a collaborative study will be proposed. The Technical Department is available to conduct the trial or analyze the data. Once the method is approved by the subcommittee, it is passed to the UMC for consideration and voting. With the use of electronic manuscript transmission, the whole process can be achieved within 6 mo to 1 y and a new method can be incorporated into the next set of annual *Additions and Revisions*. In 2001, in response to consumer requests and the trend toward the purchase of individual methods brought about by the requirements of ISO 17025, AOCS introduced *Methods Online*, which allows users to search the AOCS Methods of Analysis and select individual methods on the AOCS website and receive them in electronic format. AOCS harmonizes and maintains its methods through active participation in ISO, CEN, Codex Alimentarius, IUPAC, AOAC, AACC, and IOOC.

**Process for the Maintenance of Standard Methods**

Most standards writing organizations have instituted a process for review of methods with the aim of confirming, revising or deleting them. These reviews usually take place on a regular schedule of 3–5 y. All ISO standards must be reviewed once every 5 y. At this time, the voting committee members are asked to decide whether a standard should be confirmed, revised, or withdrawn on the bases of its relevance, usage, and technical merit.

**Interactions Between Standards Developing Organizations**

The last 15 y have seen a major increase in the harmonization of methodologies among SDOs. IUPAC, ISO, AOAC International, and AOCS have actively pursued a program of harmonization of methodologies. The provision of lists of methods of analysis in many Codex Alimentarius documents has done much to foster this process. Codex Alimentarius, a governmental organization set up under the auspices of FAO (Food and Agriculture Organization of the United Nations) and the WHO (World Health Organization) sets guidelines for food safety and trade in food. The activities of the Codex Committee on Fats and Oils (CCFQ) and the Codex Committee on Measurement, Analysis and Sampling (CCMAS) are of particular importance to the harmonization activities of AOCS and its partners. As a recognized international NGO, AOCS participates actively in these activities and provides comments in areas in which agenda items affect the interests of the AOCS membership. AOCS methods of analysis can be found listed in relevant Codex documents.

The Interagency Meeting (IAM), a subcommittee of CCMAS, currently comprises more than 30 NGOs listed in the CCMAS Directory of Organizations, “known to be active in the field of methods of analysis and sampling” or that have had a method adopted by the Codex Commission. Harmonization is also accomplished by the inclusion of a significant number of representatives from many dif-
ferent organizations on several of the major committees. An impetus for harmonization is the desire to avoid holding more than one expensive collaborative trial for new methodologies (9).

AOCS continues to focus on the needs of the fats and oils community and is seen as an advocate for fats and oils methods. Although there is much to be gained from the harmonization of methods of analysis, there remains a regional preference for methods of analysis from different organizations. It is not clear whether this is based on language, style, the experience and training of laboratory staff and assessors, or habit. These questions are frequently addressed by those concerned with the market share of the methods of a particular organization.

**Issues Related to Differences in Analysis Procedures**

The list of reasons for the establishment of several different methods for the determination of the same analyte can be very long. Historically, different techniques grew up around a specific matrix (oilseed) and location. The development of general methods has been one of trial and error. Even among the AOCS methods, where many simple methods are repeated for single oilseeds, it has proved to be a difficult task to develop generic methods and then compile the special considerations for each application.

Sampling and laboratory sample preparation are central to the performance of any method and are important considerations when determining the precision of an analytical technique. In the development of oilseed extraction technology, the choice of solvents and the type of equipment play an important role. Many methods committees have devoted considerable time to the discussion of the philosophical question “what is oil?” Because the degree of lipid extraction is highly solvent dependent, answering this question is at the center of determining the equivalence of different methods whether they are standard methods or equipment-based or semiautomated systems.

Up to now, standardization procedures have been developed to consider only generic public-domain methods in a recipe-like prescriptive manner. Comparison with proprietary methods has not been performed on a large scale. This book describes one attempt to conduct such a study using the same samples in parallel (see Chapter 3 of this book, Accelerated Solvent Extraction). It takes considerable time, money and goodwill to carry out successful comparisons. Without the help of major corporations and equipment vendors, it is not possible to reach statistically sound conclusions. AOAC has addressed this need through its Research Institute and Peer-Verified Methods program, whereas AOCS has established AOCS Approved Procedures as a category of official methods.

**Need for Harmonization of Procedures**

In the face of such a large number of different methods and technologies to determine the oil content of oilseeds, why should there be methods harmonization? The answer is clearly financial. If a seed company develops an oilseed with enhanced quality traits, then to make a return on its investment, it must have market penetra-
tion and be able to assure the buyer that these traits exist and represent a benefit to the value chain. Without supporting analytical data, these claims cannot be substantiated. The use of different methodologies to determine the trait can easily lead to loss of confidence. Hence, there is a need for consistent methodology. In the case of a dispute between the buyer and seller, the dispute may be settled by arbitration; in that case, a referee laboratory will be selected to perform the analysis according to an agreed reference methodology.

In recent meetings, the Codex Committee on Measuring, Analysis and Sampling has proposed the use of performance-based criteria in the establishment and use of methods of analysis. Although this appears to be a challenge to the adoption and use of official methods and international standards, the list of required performance criteria is long and onerous. The degree of validation required may convince the laboratory to adopt and use official methods. The determination of performance criteria or the publication of precision data from a collaborative study in an official method allows the user to determine whether the method will fulfill his needs and whether it is fit-for-purpose. Indeed, the development and interpretation of performance criteria may be considered another way of looking at fitness-for-purpose.

**Challenges for Harmonization**

For an SDO and its volunteer members, there is no difficulty in deciding whether the standard developed by another organization is similar enough to its own to consider harmonizing with it. The challenge is to convince current method users that there is an improvement in performance if certain changes are made. When industries are consolidating and looking for ways to streamline and economize, they are less likely to accept the advice of standards organizations. They are more likely to implement vertical integration within the different business units, thereby making official methods secondary to in-house methodology.

This may be seen as a problem for the SDO in the short term, but the streamlining of company activities is also a fertile area for invention through miniaturization, high-throughput, real-time, and novel in-line technologies. In the future, these technologies will require recognition and validation through collaborative trial, a role that many SDOs have been performing for the last century.

**Acknowledgments**

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**References**


