Symposium

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The AOCS Smalley Program

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ABSTRACT

This program is named for Frank N. Smalley, a charter member of AOCS. In 1909, he was chief chemist for the Southern Cotton Oil Company, the same year in which AOCS was formed. Smalley checked the proficiency of his firm's laboratories by distributing weekly meal samples that had been prepared in his Savannah laboratory. Other analytical chemists saw the value of such a program and asked to participate. In 1915, the Uniform Methods Committee, under Dr. Smalley, adopted this program. In 1955, the name was formally shortened to the Smalley Committee. It continues to function largely with the aid of volunteer help. AOCS is greatly indebted to the people and industries who give of their time and talents. In 1981, an ad-hoc committee reviewed the Smalley Committee and each of the series involved and declared the program was functioning well and carrying out its intended purpose.

Origin

The Smalley Committee began in Memphis, Tennessee, in May 1909. A small group of nine chemists attending the thirteenth annual convention of the Interstate Cottonseed Crushers' Association met and adopted tentative plans for the organization, designed for the development and advancement of analytical methods for cottonseed products. Dr. Felix Paquin was the first president of the organization; Edward R. Barrow and G. Worthen Agee were appointed as a committee to draft a constitution and by-laws for presentation to the group the following year when the Interstate Cottonseed Crushers' Association met in Little Rock, Arkansas. During the year, the original group of chemists increased to twenty. These men became charter members of the organization which was known as the Society of Cotton Products Analysts and was a part of the Interstate Association. Within a few years, the society and its members had gained sufficient prestige to enable them to sever some connections with the Interstate Association and to hold meetings independently. The group set up three standing committees—the Governing Committee, the Membership Committee, and the Committee on Uniform Methods and Cooperative Work. This last committee was actually the forerunner of the present Smalley Committee, although it was several years before the name was established permanently.

Dr. Frank Smalley, chief chemist of the Southern Cotton Oil Company and a charter member of the society, used check samples for determining the proficiency of his company's district laboratories for a number of years. Check samples were prepared weekly at Savannah, Georgia, and were sent to the district laboratories. The analysts reported their findings on the determinations of oil and ammonia. This work was started by Dr. Smalley about 1905 and resulted in obtaining close agreement between his laboratories. Between 1912 and 1915, other commercial chemists began asking if they could participate in Dr. Smalley's program to ensure that their laboratories were doing efficient analytical work. The society's Committee on Uniform Methods and Cooperative Work, of which Dr. Smalley was chairman, started a general cooperative check sample program in 1915. The program not only included meal samples, but also crude cottonseed oil for free fatty acid and refining tests and some types of fertilizers. Dr. Smalley served as program chairman from 1915 until 1919, at which time the chairmanship was assumed by Thomas Law of Atlanta, Georgia. Initially, approximately 35 chemists participated in this check sample program; the number of participating chemists has now passed 300. Dr. Smalley, even after retiring as chairman, continued to exert efforts to develop the accuracy and dependability of the chemical analysis on meal, oil and other cottonseed products until his death in 1921.

During its initial decade, the Society of Cotton Products Analysts had shown constant growth in membership, and widening interests. In 1920, the name was formally changed to the American Oil Chemists' Society. In 1921, the American Oil Chemists' Society was incorporated under the laws of the state of Louisiana, severing all formal ties with the Interstate Cottonseed Crushers' Association. In 1921, the membership voted to divide the Committee on Uniform Methods and Cooperative Work to be known as the Smalley Foundation Committee in recognition of Dr. Smalley's contributions.

Growth

At AOCS' 65th spring meeting in 1974 at Mexico City, Bill Coleman, when reviewing Smalley functions, reported that there were fifteen series of samples of various commodities from which analysts could select.

In the relatively short span of time since then, seven new programs have been added, making a total of twenty-two programs now available to the analyst.

We also note that the Smalley programs are being utilized over larger geographical areas. Participants are widespread over the USA and are also to be found in Canada and Mexico and in about fifteen other countries.

Over the same seven-year span, since 1974, the total of series participants has grown steadily as indicated by these yearly numbers: 602, 634, 690, 818, and 908, with 999 in 1981. Of the 999, there were 155 from countries other than the USA.

Organization

The Smalley Committee is comprised of a chairman, first vice-chairman, second vice-chairman, liaison officer (second past president of the society), and chairmen and members of the following subcommittees: Oilseed and Oilseed Meals, Vegetable Oils, Drying Oils, Tallow and Grease, Edible Fats, Cellulose Yield, Gas Chromatography, N.I.O.P. Fats and Oils, Aflatoxins and Marine Products.

The chairman is appointed annually by the incoming president of the society, and his appointment must be approved by the governing board. The vice-chairman and chairman of subcommittees are appointed by the committee chairman. Each subcommittee chairman elects subcommittee members, subject to the approval of the chairman. The board usually acts on the approval of the committee members at the annual meeting. The term of office for all members begins immediately following their approval by the board and continues for one year.

The scope of the Smalley Committee is to conduct a cooperative analytical program, stimulating the upgrading of analytical work through public acknowledgement of individual excellence. There are approximately sixty volunteers who are directly involved with the administration of the Smalley Program, which is placed under administrative committees along with the Examination Board and the Uniform Methods Committee.

All of these groups serve at the discretion of the governing board with the Chairman of each group appointed by the Society President and approved by the governing board.

Recently an ad-hoc committee conducted an extensive review of the Smalley Program at the request of the Governing Board. The review was conducted in two steps, the final assessment being that the Smalley Program was serving its intended purpose and was being kept up to date by the chairman.

Problems

A serious problem with which the Smalley Series has to contend is poor mail service. This occurs on distribution of samples and also on mailing back of reports. This problem is intensified in foreign mail service and is considered a deterrent to greater participation outside of the USA. Discussions have focussed on this problem and setting up distribution centers in a few foreign countries may be a possible solution.

There are differing participant requirements even within a single series—for example, the meal series. Currently, ten samples are offered over several months, from July to February. Some want more, some less. The committee is considering the possibility of having two 10-sample series—one 10-sample series required for possible referee certification, and a second 10-sample series which would effect coverage over a longer time period. Random samples and/or blind samples have also been discussed in connection with the series.

Statistical Treatment

Currently, a nonuniform system is being used for evaluation of check sample data. This system evolved under the direction of many committee chairmen. This is not to say the system doesn't work—it does work very well. Most of the long-term Smalley participants are familiar with the statistical treatment of data for the particular series in which they participate. Newcomers may have difficulties with the various methods of evaluation.

Although a uniform system would be beneficial, the data from some series would require treatment in a different manner. Whether this direction will be taken has not yet been decided.

Data processing is currently being applied in the following locations—each following somewhat different practices: Business Solutions, Lubbock, TX; General Mills, Minneapolis, MN; Honeymead, Minneapolis, MN; and Buckeye Cellulose, Memphis, TN.

In the early years, several questions arose about large distortions due to skewed distributions contributing to validity of decisions as to outliers. The first accepted averages were probably means, which disregarded outliers. Problems also were encountered in the early stages regarding development of a method for grading relative proficiencies. A system evolved over the years which has changed with the availability of computers.

Although each computer program has some distinctive features, they are all relatively similar: tabulation of reported data, computation of mean, determination of standard deviation, deviation from mean and ratios of these deviations to the standard deviation calculated, outliers discarded and process repeats until no outliers are found.

There are several variations on criteria for outliers. Some programs incorporate the term "typical normalized deviation" (TND). Usually these programs rank TND by magnitude and assign an inverse percentile which are averaged and expressed as a percentage.

Other programs calculate grades by subtracting the sum of the deviations from 100.

Usefulness of Smalley

Check samples, sponsored by the committee, are used widely by industry and commercial analysts as an indicator of how their results on replicate samples compare with those of other analysts. It is vital to industry today to know what their plants are doing in quality control and whether their products meet required specifications. The Smalley samples provide a means of measuring an individual analyst's and a laboratory's analytical proficiency.

The examination board of the society certifies analysts annually in commercial laboratories as "referee chemists" for high proficiencies in the analysis of certain oilseeds and oilseed products. These certifications are based on the final Smalley check sample ratings of these individuals as determined by the Smalley Committee. A number of trade associations require their chemists to hold certification from the Examination Board in order to become listed as "official chemists" of their respective associations.

It is difficult to assess the full value of Smalley for use by the Examination Board, however, the program is regarded highly.

If there is a desire for other types of collaborative work on the highest level, people should contact the Smalley Committee and make their needs known. It is by this means the program is kept viable and useful.

The American Oil Chemists' Society's Official Methods, Smalley Check Sample, and Official Referee Chemists programs form a vital foundation for the nation's multibillion-dollar oilseed industry, but the programs may be little known or understood beyond the core of persons who participate in them. More than two-thirds of Smalley program participants are not independent chemists. Employees

of buyers and sellers of oilseed products enroll solely to maintain and to verify their own analytical proficiency. Independent chemists—those who do not work for buyers or sellers of oilseeds or oilseed products—who do well enough in the Smalley Program can be certified as AOCS official referee chemists. Applicants must be AOCS members whose labs have been inspected to ensure the labs are properly equipped to perform AOCS analytical methods.

Trade associations' "official chemists" serve as objective arbitrators or mediators to help resolve disputes about product quality. If analysts for buyer and seller agree on quality, there is no problem. If they disagree, a mutually selected "official chemist" can provide an objective analysis. Although the official chemist's analysis may be binding, the disputants also may use it as the basis for further negotiating. Thousands of dollars in a single soybean oil sale may hinge on analytical discrepancies. Buyers and sellers both must have confidence in the method used to resolve disputes.

Let me again state that the Smalley Program is viable and urge your inspection of and participation in several series of samples.

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Quality Control in Processing Drying Oils

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ABSTRACT

A brief history of linseed oil, its production trends, uses and prospects for the future are discussed. Methods of processing, effects of process variables on oil quality, how quality is measured and how it is maintained are the main topics of this paper. Although linseed oil production declined steadily for many years after World War II, it has stabilized in recent years and shortages of petroleum products could cause a return to linseed oil in the form of water emulsion. Brief references to other drying oils will be included. Quality control begins with seed quality and includes storage, cleaning, processing and degumming. Refined oils described include alkali-refined, blown, heat-bodied and chemically modified oils. Present quality control methods are described. Problem methods such as the "foots" test for degummed oils are described. Recent developments used on occasion included liquid chromatography, thin layer chromatography (TLC), ultraviolet (UV) and infrared (IR) spectroscopy, but the most predominantly used technique has been gas chromatography (GC). Recent developments in capillary GC and fused silica columns have improved separations and reduced analysis times. Near infrared reflectance shows some promise for future quality control work.

INTRODUCTION

Many years have passed since the subject of drying oils (1), and many more years since quality control in drying oils, has been a topic at an AOCS annual meeting (2). One might well ask: in view of current production volumes, who cares?

There are some reasons for hope for the future of drying oils. Renewable resources can supply virtually all the raw materials needed for making coatings if and when petroleum is no longer available (3). The major source of traditional agricultural raw materials for coatings is the seed oils. Alkyd resins are the major market for the seed oils, with linseed, soybean and sunflower the principal oils used in alkyd resin technology. Future price increases or petroleum shortages may add impetus to the development of substitutes for petroleum-based monomers and polymers for coatings. In recent years, vegetable oils were replaced by the, then, less expensive petrochemicals, but now vegetable oils may need to be reconsidered. Linseed oil paints, in water emulsion form, with their well known performance advantages, are ready and waiting to be rediscovered. Several long oil emulsifiable linseed alkyds have been developed and paints using them have been formulated and evaluated. Test fence data looked promising. The well known advantages of linseed oil for exterior coatings are retained and the clean-up advantages of latex coatings are added. The coatings industry showed little interest when crude oil was $3.00 per barrel, but this could change in the future.

Drying oils include linseed oil, tung oil, castor oil and oiticica oil. Since the early 1970s, only linseed oil is shown in US production figures. Small quantities of the others are still imported for specialty uses. These include alkyds, urethanes and varnishes. Many of the control methods used for linseed oils apply to the other drying oils as well.

Foreign linseed oil production is down about 25% over the past 10 years (4,5), but showing signs of leveling out. Domestic production has declined about 75% over the same period, from 27% of total world production to about 6% of the total production. This, too, shows signs of stabilizing, thus providing a base for any future expansion of production.

The topic of this paper is quality control in drying oils, although a valid assumption is that US linseed oil plants and refineries are multi-product plants and probably have frequent product changes. Therefore, product separation and purity become all-important and the quality control function is a key one. Multi-product plants must be monitored throughout all parts of the process. Lines, storage, blending, loading and shipments must be checked for contamination.

By far the most important quality control tool is gas chromatography (GC); we could not operate a plant without it. Recent developments provide good separation with easy-to-use columns (fused silica Wall-Coated Open Tubular [WCOT]) and elution of the linolenic ester in less than 5 min. This means a sample can be analyzed in 30 min, including methyl ester prepartion, chromatographic separation and calculation of results with the improved electronic integrators available today.

The most frequently used control methods are those of AOCS, ASTM, AACC, AOAC and IUPAC. The first two are most frequently used. Table I lists the AOCS methods most frequently used in quality control of drying oils. These methods are constantly being evaluated by participants in the Smalley Check Series Program, a very valuable tool with which to evaluate methods and laboratories.

Raw linseed oil is usually sold on the basis of ASTM methods, Part 29, shown in Table II. These methods are