Report of the Instrumental Techniques Committee, AOCS 1968-1969[1]

Introduction

The Instrumental Techniques Committee met twice during the past year. On October 21, 1968, the Committee met in the Hudson Room of the Statler Hilton Hotel, in New York. Nine members attended the meeting, and four of the five Subcommittees were represented. The second meeting was held in the Shasta Room of the San Francisco Hilton Hotel in San Francisco, on April 21, 1969. Fourteen members attended this meeting and three of the Subcommittees were represented.

Subcommittee for the Preparation of Methyl Esters

The Subcommittee for the Preparation of Methyl Esters has completed its task of investigating, by collaborative effort, methods for the preparation of methyl esters of long chain fatty acids, applicable to common fats, oils and fatty acids, which appear to be simpler and much shorter than the present AOCS Method Ce 2-66, (See previous reports of the Committee (1,2).) As a result of these investigations, the Subcommittee recommended replacement of the present AOCS method with a more rapid and simplier method using BF_3-methanol. The recommendation had been approved by the Committee, and announcement was made during the Spring Meeting in San Francisco that the new method had been accepted by the Uniform Methods Committee of the Society and would become the official method of the Society replacing Method Ce 2-66.

During the Fall Meeting in New York, the Subcommittee Chairman recommended that the subcommittee be inactivated, since it had completed the task for which it was created. The Committee Chairman ruled that this action should not precede acceptance of the newly recommended method by the Uniform Methods Committee. The Subcommittee was not represented at the San Francisco Meeting, but subsequent to the announcement that the revised method had been accepted as the official method of the Society by the Uniform Methods Committee, the Committee Chairman announced that the Subcommittee would become inactive. It might be noted that the General Referee on Oils, Fats and Waxes of the Association of the Official Analytical Chemists has made a similar recommendation that the boron trifluoride method, as approved after collaborative investigation within the American Oil Chemists Society, should be adopted as the official method of the AOAC to replace the less convenient, more time-consuming methanolsulfuric acid reagent, AOAC 26.052 (3).

Spectroscopy Subcommittee

The Spectroscopy Subcommittee was represented at both of the Committee meetings during the year. The major activity of this Subcommittee has been completion of collaborative study of a method developed in the laboratory of the Subcommittee Chairman. The new method, if found satisfactory, would afford a much more rapid analysis for isolated *trans* isomers in long chain fatty acids, esters, or their triglycerides, and would have the advantage of eliminating the need for external standards, a requirement of the present AOCS Method Cd 14-61.

The newly proposed method has been submitted to the Journal for publication (4) therefore, will not be described in detail here. Collaborative investigations appear to suggest that, while permitting a more rapid analysis, it may not be as precise as AOCS Method Cd 14-61, principally because different instruments, even of the same make and model, usually give different absorptivities for the same sample. Furthermore, it may be considerably limited in scope. It would not appear to be applicable to samples of methyl esters or triglycerides which contain small amounts of free fatty acids, or to samples of free fatty acids which contain small amounts of methyl esters or triglycerides. It would not be applicable strictly to samples containing hydrocarbons or alcohols, which would be completely undetected and unaccounted for as part of the total sample.

A further objection to the proposed method at present is that it has not been sufficiently tested except for simple mixtures of oleic and elaidic derivatives. In addition, it is felt that mixtures containing stearates, linoleates, etc., as well as oleates and elaidates should be tested.

The new procedure does offer a much more rapid analysis which can be completed without the requirement for external standards. At present, its more limited scope and its lower precision appear to be factors against its adoption as a replacement for AOCS Method Cd 14-61. However, a rapid procedure to be used as an alternate to the procedure of AOCS Method 14-61, where highest precision is not required and where the limitations on scope are not particularly applicable, might enhance the official method and offer to many laboratories the advantages of a rapid and simple method. It was decided, within the committee meetings, that further consideration of the method should await its publication and then further action, either recommendation or additional collaborative study, should be reconsidered. The Subcommittee Chairman announced that a poll had failed to show sufficient interest to justify collaborative study of a method for the determination of conjugated *cis,trans-* or *trans-trans* acids, esters or glycerides, or to even permit any collaborative testing to be undertaken. It was agreed that any consideration of such a method should await evidence of increased need for and interest in it.

Gas Chromatography Subcommittee

Upon completion of their task to revise AOCS Method Ce 1-62 for the determination of fatty acids, by gas chromatography, the Gas Chromatography Subcommittee considered and proposed an inactive status. [See previous report (2)]. The Society President, however, requested further consideration of the consequences of this action, and in light of the activities of this Subcommittee since a year ago, the wisdom of this decision appears to have been confirmed.

Last year it was pointed out that the Society had no GLC method for fatty acids which permitted the use of flame detectors. The Gas Chromatography Subcommittee had no plans for any further work in this direction, but had considerable data from collaborative testing to compare flame and thermal conductivity detectors. It was agreed that these data would be studied in further detail and that a recommendation be made to determine whether AOCS Method Ce 1-62 should be further modified to permit the use of flame detectors, or whether this action should await additional collaborative testing. The Subcommittee was not represented by its Subcommittee Chairman at the Spring Meeting in San Francisco, but it was announced from a written report that the method was being rewritten to provide for the use of flame detectors. According to plans the revision of Ce 1-62 will be completed in time for Subcommittee action prior to the next committee meeting in Minneapolis, during the 43rd Fall Convention, in October 1969.

During the past year, the Society received a request from the Codex Committee on Fats and Oils of the Joint Food and Agricultural Organization of the United Nations and the World Health Organization, Codex Alimentarius Commission, for specifications to define a specific vegetable oil or animal fat, based on its constituents, as determined by GLC. Despite obvious limitations in this type of identification, arising from the composition differences

[1] Report of collaborative work from Government, Industrial, and Academic Laboratories by members of the ARS, USDA, Southern and Eastern Utilization Research and Development Divisions; Anderson, Clayton and Company, Food Division; The Hormel Institute, University of Minnesota; Durkee Fine Foods; and A. E. Staley Mfg. Co.

with species, variety, etc., and particularly from the modification of certain oils and fats by agronomists and geneticists to create oils or fats designed with a composition to fit specific end-uses, the Chromatography Subcommittee Chairman was able to furnish a set of specifications for the American and Canadian delegations on the Codex Committee on Fats and Oils for eight oils and two fats. These specifications were reviewed, at the request of the Committee Chairman, by selected members of the Society prior to submitting them to the Codex Committee. Specifications were furnished for the following oils and fats: arachis oil, cottonseed oil, maize oil, rapeseed oil, safflower oil, sesame seed oil, soya bean oil, sunflower seed oil, lard (rendered pork fat) and premier jus (an edible tallow).

The specifications were well received when presented by the American and Canadian delegations at the fifth session of the Codex Committee on Fats and Oils, sponsored by the Joint FAO/WHO Codex Alimentarius Commission held in London, England, September 16–20, 1968. In a letter to the Chairman, the Chairman of the American delegation expressed his thanks and those of the U.S. delegates to the members of the Committee, and through them to the Society, for the "excellent cooperation furnished by you and your committee of AOCS—". The U.S. delegation requested additional specifications for mustardseed oil and these were furnished to the new U.S. Delegate Chairman, sometime after the London meeting.

The specifications to identify a specific fat or oil, essentially gas chromatographic composition patterns of the constituents of the specific fat or oil, have received attention from a number of members of the Society. As a result of requests for these data, plans are being made to make them available to all members of the Society by publication in the Journal.

The Instrumental Techniques Committee has been receiving several requests and comments regarding the need for a GLC method for the determination of sterols in fats and oils, and for the determination of pesticides in fats and oils. These suggestions and requests were discussed at the Fall Meeting in New York and the Gas Chromatography Subcommittee Chairman was assigned the task of further determining these needs and interests by submitting questionnaires throughout the industry.

Results of this questionnaire were received from 31 laboratories and indicated a high degree of interest. In the absence of the Subcommittee Chairman, the results were announced by the Committee Chairman:

	Sterols			Pesticides		
	Yes	No	Don't know	Yes	No	Don't know
Do you have any interest in a GLC method for the determination of sterols and pesticides or both in fats and oils?	21	2	6	22	3	5
Are you willing to actively cooperate in a collaborative effort to establish an official AOCS method for sterols and pesticides or both in fats and oils	16			13		

If we assume that the poll of 31 laboratories is representative of the industry, it must be concluded that these problems are of interest to a substantial majority, about 70% indicating such an interest. More important, probably to the Instrumental Techniques Committee, is the fact that about 50% of those responding indicate a willingness to actively cooperate in collaborative efforts to establish an official method. Based on these results, the Committee voted unanimously that the Gas Chromatography Subcommittee should proceed to select methods for collaborative study with the objective of establishing official methods for the determination of both sterols and pesticides in fats and oils.

This decision of the Committee has raised questions for consideration by its Gas Chromatography Subcommittee. Analytical technology is available for both of these determinations, but considerable time, effort and study will have to be made to decide which will best fit the needs of the AOCS before any collaborative effort can be initiated. There is also the question of what is being done in other Societies, questions which can probably be settled through the Intersociety Relations Committee (AOCS, AOAC, ASTM and AACC). Another problem for consideration by the Subcommittee is the scope of any general method. Probably a method suitable for the determination of sterols and pesticides in fats and oils need not encompass all possible sterols or all possible pesticides but might preferably be restricted to analyses for the sterols or pesticides commonly associated with fats and oils. Specific suggestions of the Subcommittee will be discussed at the next meeting of the Committee.

X-Ray Diffraction Subcommittee

Subcommittee Chairman, C. W. Hoerr, distributed copies of a manuscript entitled, "The Role of X-Ray Diffraction in Studies of the Crystallography of Monoacid Saturated Triglycerides." This review has been published as part of the Symposium on Spectroscopy and X-Ray Diffraction, held during the AOCS-AACC Joint Meeting in Washington, D.C., March 31–April 4, 1968 (5). Reprints of the article have been distributed to all members of the X-Ray Diffraction Subcommittee for comment and suggestions as to what action the Subcommittee can take to establish greater uniformity in nomenclature, symbols and precedural techniques in the entire area of x-ray diffraction.

Atomic Absorption Subcommittee

This newest of the Subcommittees of the Instrumental Techniques Committee embarked on their first attempted collaborative efforts following discussions at the Fall Meeting. Very limited quantities of standards, consisting of metals dissolved in a metal-free molecularly distilled oil, by the use of oil-soluble compounds, were sent to each collaborator. These standards, at the 500 ppm level (for dilution to prepare working or calibration curves), proved to be unstable. The small quantities of samples available prevented any repetitive investigations by the method the Committee proposed to investigate, essentially that described by Piccolo and O'Connor at the Syposium on Spectroscopy and X-Ray Diffraction, held during the AOCS-AACC Joint Meeting in Washington, D.C., March 31–April 4, 1968 (6).

Results were submitted by only a few collaborators and further collaborative work is required before any recommendations regarding a suitable procedure can be made. The Subcommittee, however, has made some progress. A sufficient supply of molecularly distilled metal-free oil now appears to be available to permit preparation of adequate quantities of standard, and oil-soluble compounds of most of the metals of immediate interest are now available. The deterioration of standards containing high (500 ppm) quantities of these metals has been established, and methods to prepare suitable standards at considerably lower levels is being attempted. The Subcommittee has also encountered difficulties resulting from differences in viscosity between standard and samples. It appears that the viscosity of both standards and samples to be analyzed will have to be carefully controlled.

Work is being undertaken to prepare adequate standards and suitable samples in sufficient quantities for another attempt at collaborative testing.

R. T. O'Connor, Chairman
R. R. Allen, Subcommittee Chairman
K. M. Brobst, Subcommittee Chairman
J. R. Chipault, Subcommittee Chairman
S. F. Herb, Subcommittee Chairman
C. W. Hoerr, Subcommittee Chairman

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