Report of the Instrumental Techniques Committee, AOCS, 1967-1968[1]

Introduction

The Instrumental Techniques Committee met twice during the past year with all of its Subcommittees represented at each meeting. The first meeting was held October 16, 1967, in the Music Room of the Pick-Congress Hotel in Chicago, Illinois, during the 41st Fall Convention of the Society, with 13 members attending. The second meeting was held during the 58th Annual Convention, the Joint Meeting with the American Association of Cereal Chemists, in the Farragut Room of the Washington-Hilton Hotel, Washington, D.C., April 1, 1968, with 14 in attendance.

The past year has seen the creation of a new Subcommittee within the Instrumental Techniques Committee. In compliance with requests and agitation for collaborative testing within the Society of methods for the detection and quantitative estimation of trace elements in fats and oil by means of atomic absorption spectroscopy [see preceding reports of this committee (1) (2)] a new Atomic Absorption Spectroscopy Subcommittee has been formed. K. M. Brobst, A. E. Staley Manufacturing Company in Decatur, Illinois, has been appointed Subcommittee Chairman, and has established a ten-member Subcommittee.

The membership rolls of the Committee have been rather exhaustively checked during the year both to ascertain present activities of Committee Members and to verify all addresses. The complete up-to-date membership roster will be found in the 1968–1969 Directory of Membership. The newly revised roster shows that the Instrumental Techniques Committee consists of 58 members. The Gas Chromatography Subcommittee is the largest, with 26 members; the Spectroscopy Subcommittee and the Subcommittee for the Preparation of Methyl Esters each have 12 members; the X-Ray Subcommittee, 13; and the newly formed Atomic Absorption Subcommittee, 10. (Failure of the totals of each Subcommittee to agree with the total membership of the Committee indicates that several Committee Members are members of more than one Subcommittee.)

Subcommittee for the Preparation of Methyl Esters

This Subcommittee, by collaborative effort, has been studying methods for the preparation of methyl esters of long chain fatty acids, applicable to common fats, oils and fatty acids, which appear to be both simpler and much shorter than the present AOCS method Ce 2–66. Subsequent investigations have shown that one of the methods could not be scaled up to yield quantities of material sufficient for routine analysis by infrared absorption spectroscopic techniques. The second method investigated, involving the use of boron trifluoride in methanol for the esterification to replace the methanol-sulfuric acid reagent in the present AOCS method Ce 2–66, was found satisfactory. The Subcommittee's conclusions and recommendations, together with a complete description of the analytical procedure using BF_3-methanol has been given in the previous report of this Committee (1).

Subsequent to approval of the Subcommittee of the BF_3-methanol procedure, the Nederlands Normalisatie-Instituut reported a method they had been studying and were proposing as a standard procedure, which was very similar to that proposed by the Subcommittee for the Preparation of Methyl Esters. The Nederlands organization suggested that to promote international standardization, and to assist in international commerce in fats and oils, it would be an obvious advantage if the two methods were identical.

However, investigations of the two procedures by both groups led to some complications. The Nederlands Normalisatie-Instituut reported that the method proposed by the AOCS would not yield correct values unless the methyl ester solution was dried over anhydrous sodium sulfate before use. As this drying was not part of the procedure and had not been included in the collaborative work which resulted in satisfactory analyses, this point was checked by the Chairman of the Subcommittee by analyses of several samples of methyl esters with and without drying over sodium sulfate, and with water added after drying with sodium sulfate. The same values were obtained with all three procedures. There was general agreement among members of the Subcommittee with these results. However, further correspondence revealed that the Dutch Committee insisted that:

1) Analyses performed last fall strictly according to the AOCS method repeatedly showed that use of the wet esters gave broader peaks which resulted in poor separation of saturated and monounsaturated components of the same chain length and gave erroneous results for stearate and oleate.

2) More recently they were unable to repeat these observations with a different chromatographic column.

3) Compositions were calculated from values supplied by an electronic integrator.

Chromatograms furnished by the Dutch Committee clearly showed broader peaks obtained with the undried esters. Retention times were unaltered, and, although the amounts of stearate and oleate in the sample were identical, the stearate peak was broader than the oleate, but it was also lower. This suggested that the relative areas of the two peaks may not have been changed greatly and prompted a calculation of these areas by triangulation. This procedure gave correct values for both the wet and dry esters, leading to the conclusions that the errors reported by the Dutch Committee arise from a combination of two factors: the presence of moisture, which in their particular column increased the breadth of the peaks, and an integrator incapable of evaluating accurately the areas of these abnormal peaks.

These factors were considered by the Subcommittee and members of the entire Committee at the above mentioned meetings. It was concluded that while sodium sulfate drying was not necessary to obtain accurate results, inclusion of this step might prevent similar problems in other laboratories and therefore might keep results obtained in a number of laboratories in better agreement (that is, enhance the precision of the method). Furthermore, addition of the sodium sulfate drying step was a simple procedure

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[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 Co., Food Division; The Hormel Institute, University of Minnesota; Durkee Fine Foods; and A. E. Staley Mfg. Co.

which would not detract from the attractive feature of the BF_3-methanol method of permitting a rapid preparation. It was, therefore, decided that this drying step would be inserted into the proposed procedure.

The proposed AOCS method is not identical to the Dutch method because its scope is considerably broader and it gives directions for preparing methyl esters for purposes other than GLC. Furthermore, the Dutch method makes no distinction between fatty acids and glycerides, applying the saponification step to both. This is a step, in the preparation of methyl esters from their fatty acids, which is considered not only unnecessary but undesirable.

The Subcommittee for the Preparation of Methyl Esters voted to propose to the Instrumental Techniques Committee that the BF_3-methanol method be substituted in the AOCS Book of Official Methods for the present methanol-sulfuric acid method, Ce 2–66. The proposed method is essentially identical to the procedure published with the last report of this Committee (1). The recommendation of the Subcommittee has been adopted by the 54-member (at the time of the letter ballot): Approve, 34; Approve with reservation, 0; Disapprove, 0; Abstain, 1; Not voting, 19. The new procedure will be recommended to the Uniform Methods Committee for adoption as an official method of the Society to replace the Ce 2–66 Method.

Spectroscopy Subcommittee

The Spectroscopy Subcommittee has been collaboratively studying a method, developed in the laboratory of the Subcommittee Chairman, with the possible objective of recommending its adoption as an official method to replace AOCS method Cd 14–61. The advantage of the newly proposed procedure is that it would eliminate the need for external standards, a requirement of the present AOCS method.

The Subcommittee has been investigating the precision with which the constants required in the new procedure can be determined within different laboratories using different infrared spectrophotometers. These data will be evaluated and, probably, analyses will be made to compare results with the present AOCS method before any Subcommittee recommendations can be made. Details of the proposed method will be published in a future issue of JAOCS (3).

Gas Chromatography Subcommittee

Following adoption of a revised edition of AOCS Tentative Method Ce 1–62, completed by this Subcommittee last year, (1), the Gas Chromatography Subcommittee had no definite plans for further collaborative investigations and a proposal that it be inactivated was suggested (1). However, the Society President did not agree with this proposal, at least without further detailed consideration of its consequences. In the meantime, it has been found that presently there is no work being done on a GLC method for fatty acids. The AOCS has no GLC method for fatty acids that permits the use of the flame detector and no work in this area is now contemplated, as far as can be ascertained. The Gas Chromatography Subcommittee has considerable data from collaborative testing to compare flame and thermal conductivity detectors. Decisions will be made as to whether these data, with or without additional collaborative testing, will be adequate for recommendation of further modification of AOCS method Ce 1–62 to permit the use of flame detectors.

In the meantime the Subcommittee has been asked to prepare specifications which will define a specific vegetable oil or animal fat based on its constituents as determined by gas chromatography. The Society has received a request for such specifications 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.

The proposal contemplates establishment of a fatty-acid composition pattern, as established by gas chromatographic analyses, for each specific vegetable oil and animal fat. From such a pattern or specification the individual fat or oil will be identified. As search of such compositions continues, it becomes more evident that work—particularly recent work by agronomists and geneticists—to create oils or fats designed with a composition to fit specific end-uses, for example, safflower oil with high oleic or high linoleic acid content, rapeseed oil with no erucic acid, and many others, will greatly complicate such a project. No reasonable set of specifications can be established with a range of fatty acid content within which every sample of the oil or fat will fit. However, a set of specifications for some dozen fats and oils has been compiled, which will be valid for most samples of these specific fats and oils. These specifications will be furnished to the American and Canadian delegations on the Codex Committee on Fats and Oils prior to the International Meeting in London in September, 1968.

X-Ray Subcommittee

This Subcommittee, was somewhat dormat while its Subcommittee Chairman was busily engaged with the operation of the Society as its Vice-President and President. Since the announcement of the reappointment of C. W. Hoerr as its Subcommittee Chairman it has been concerned with the problem as to how it can best function to achieve standard nomenclature, symbols, techniques etc., within the general area of x-ray diffraction.

As a result of a canvas of the members, a considerable number felt that no useful purpose would be accomplished by a collaborative measurement of a very pure, well-characterized triglyceride, highly purified tristearin. The Subcommittee Chairman, C. W. Hoerr, with another member from his laboratory, F. R. Paulicka, reviewed the "Role of X-Ray Diffraction in Studies of the Crystalography of Triglycerides" as one of the papers presented at the Symposium on "Spectroscopy and X-Ray Diffraction" held during the AOCS-AACC 1968 Joint Meeting, March 31–April 4, 1968, in Washington, D.C. The Subcommittee Chairman plans to distribute copies of this review to all Subcommittee members in an attempt to determine the extent to which members agree with the conclusions, and formulate a program of further study.

Atomic Absorption Subcommittee

Atomic Absorption has been the subject of considerable attention and interest at all recent meetings of the Instrumental Techniques Committee, either by individuals appearing informally, or by organized groups appearing by invitation following formal requests to be allowed to discuss the topic (1,2).

At the 58th Annual Meeting of the Society it was announced that a new Subcommittee of the Instrumental Techniques Committee had been formed and that K. M. Brobst, A. E. Staley Mfg. Co., accepted appointment as Chairman.

The new Subcommittee, composed of 10 members, is planning collaborative study of methods for the detection and quantitative measurement of various elements, metallic and nonmetallic, vegetable oils, animal fats and their constituents. A molecularly distilled, metal-free oil has been obtained and is being used to prepare standards by the addition of known quantities of metals in the form of oil-soluble salts. With the cooperation of several organizations, samples for analysis by collaborative effort in several laboratories have been collected. Collaborative work is planned, probably to investigate in each laboratory a single common method and the method used by the specific laboratory. This cooperative effort will constitute the first step toward establishment of a standard or official Society method.

The activities of technical committees to test the precision of specific analytical procedures in collaborative investigation and to ascertain agreement of results and satisfactory procedure by different individuals using different instruments in different laboratories, is one of the oldest and still one of the more important functions of the AOCS. The Instrumental Techniques Committee invites any member of the Society interested in and using instrumental method to join us in this important work. Application for membership on the Committee can be made by corresponding with the Chairman of the Committee or the Chairman of any of its Subcommittees.

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

REFERENCES

1. O'Connor, R. T., R. R. Allen, J. R. Chipault, S. F. Herb and C. W. Hoerr, JAOCS *45*, 103–106 (1968).
2. O'Connor, R. T., R. R. Allen, J. R. Chipault and S. F. Herb, JAOCS *44*, 215–217 (1967).
3. Allen, R. R., in press.