Jaocs News Feature

Instrumental Techniques Committee of the American Oil Chemists' Society[1]

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HISTORY AND OBJECTIVES

The Instrumental Techniques Committee was established by action of the Governing Board at its 53rd Annual Meeting in May 1962. This action followed a report by the late R.W. Bates, a former president of the Society, of a committee appointed to study the organization and structure of the Society's committees. The Instrumental Techniques Committee combined the Color, Spectrosocpy, and Gas Chromatography Committees which became subcommittees.

The objective of the committee was to replace classical methods of wet chemical analysis with methods based upon the use of modern instrumentation where they could be advantageous in terms of simplicity, accuracy, time, and, therefore, cost of analyses.

The Committee consists of four active and two inactive subcommittees. The active subcommittees are: Atomic Absorption Spectroscopy, Gas Liquid Chromatography, NMR Spectroscopy, and Spectroscopy. There are two inactive subcommittees: Color and X-ray Spectroscopy.

In March 1971 achievements of the Committee were reviewed in the *Journal*.

ACHIEVEMENTS

The Spectroscopy Committee was created by action of the Governing Board during the Fall Meeting in Chicago in 1945. The first achievement of the Spectroscopy Committee was the establishment of offical Method Cd 7-58 (Revised 1959), "Polyunsaturated Acids–Ultraviolet Spectrophotometric Method." As a subcommittee of the Instrumental Techniques Committee, it completed the development of an IR absorption method, official Method Cd 14-61, "Isolated *Trans*-Isomers."

The Gas Liquid Chromatography Committee became a subcommittee shortly after it had been organized as a joint committee with the Association of Official Analytical Chemists. The joint committee collaborated with American Society for Testing and Materials (ASTM) Committees D-1, "Paint, Varnish, Lacquer, and Related Products," and with E-19, "Chromatography." During its short existence as an independent committee, it developed a procedure for the determination of fatty acid composition by gas chromatography, which became an official method of the Society as AOCS official Method Ce 1-62, "Fatty Acid Composition of Gas Chromatography," after organization of the Instrumental Techniques Committee. This method has been revised and updated by the subcommittee on Gas Chromatography. The latest revision of the method was accepted by the Uniform Method Committee in 1968.

The Gas Chromatography Subcommittee completed, at the request of 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, specification for identification of specific vegetable oils or animal fats, based upon their fatty acid constituents, as determined by gas liquid chromatography.

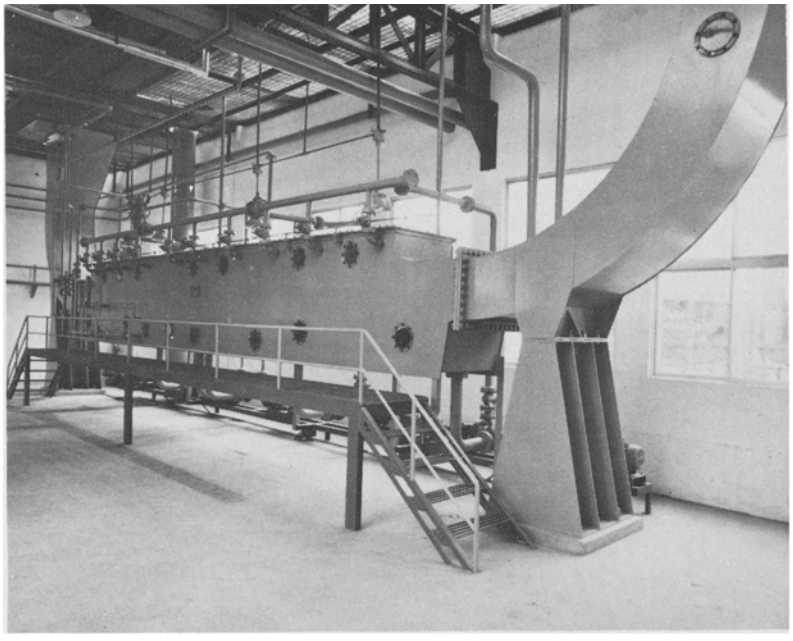
Just before incorporation into the Instrumental Techniques Committee, the Spectroscopy Committee had established a task group to investigate methods for the preparation of methyl esters from free fatty acids or from their triglycerides for the purpose of subsequent determination of *trans*-isomers. The committee had determined that, in specific cases, this determination could be made with greater accuracy by IR absorption measurement after conversion of the fatty acids or their triglycerides to the methyl esters. However, a procedure which would convert either the free acid or the triglyceride to the methyl esters without causing any *cis*- to *trans*-isomerization was essential. When this group was made part of the Instrumental Techniques Committee, it was apparent that the need for such a method was of equal, or greater, interest to the Gas Chromatography Subcommittee, because, in certain instances fatty acid composition could be obtained more satisfactorily by gas liquid chromatography if the sample were converted to the methyl ester. Thus, very early we had evidence of the wisdom of combining these committees concerned with instrumental methods of analysis.

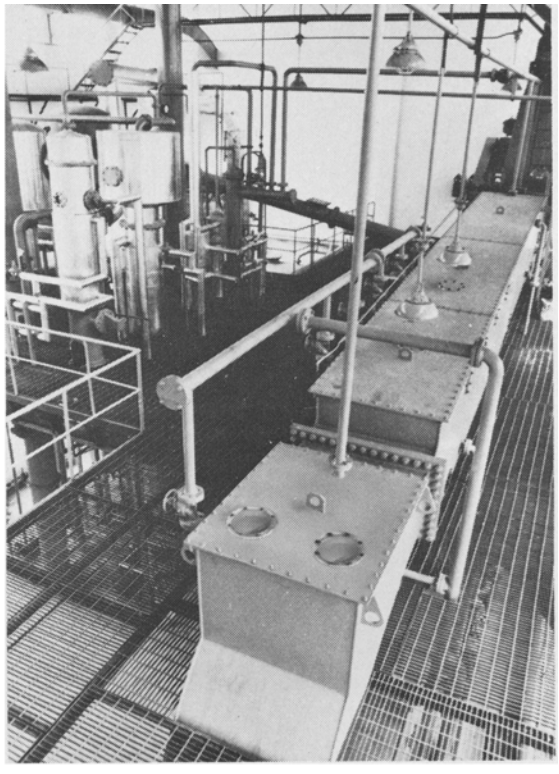
An Ad Hoc Committee was established to develop a procedure, whereby the methyl esters could be prepared either from the free fatty acids or from the triglycerides without any *cis*- to *trans*-isomerization. This committee recommended a technique involving the use of methanol and sulfuric acid, which became the Society's Method Ce 2-66. This method had hardly been adopted when simpler, faster methods were reported. The Ad Hoc Committee was reactiviated, and Method Ce 2-66 was replaced by a method employing BF_3 and methanol, Method Ce 2-66 (Revised 1969), "Preparation of Methyl Esters of Long Chain Fatty Acids." This procedure was adopted by the Association of Official Analytical Chemists (AOAC) and designated 28.052. This is one of several examples where an official method of AOCS, originating from its Instrumental Techniques Committee, has been adopted by other Societies, notably AOAC and ASTM.

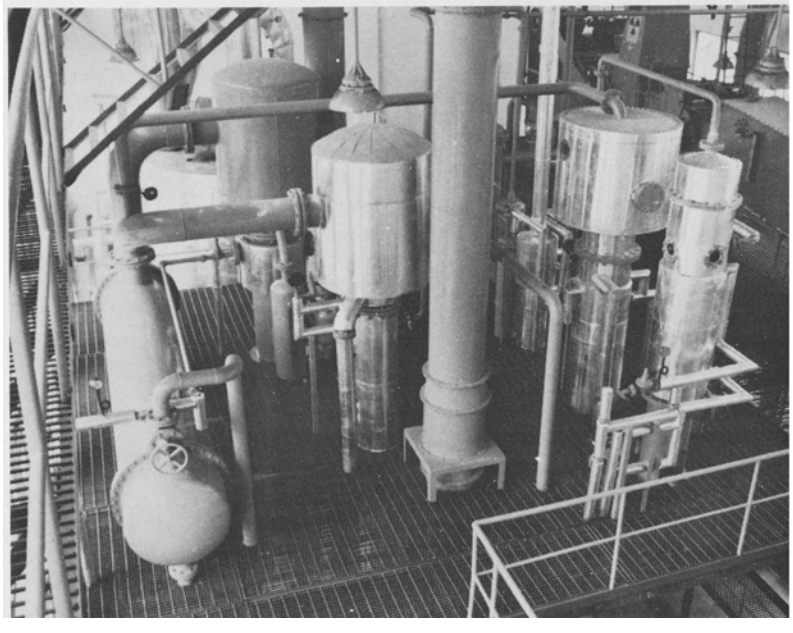
The objective of the X-Ray Diffraction Subcommittee, created in April 1965, was to develop standardization in analytical spectroscopy, particularly regarding terminology, symbols, etc., and to resolve apparent contradictions in the literature. A manuscript, entitled "The Role of X-Ray Diffraction

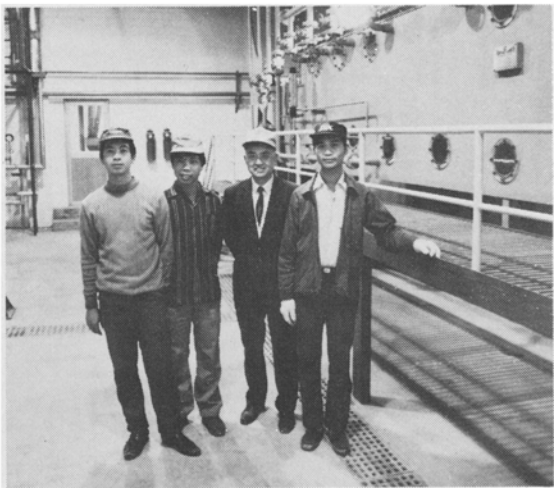
[1]Presented by T. Jacks at the AOCS Spring Meeting, Mexico City, April 1974.

[2]ARS, USDA.









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in Studies of the Crystallography of Monoacid Saturated Triglycerides," by the subcommittee chairman was published as part of a symposium on "Spectroscopy and X-Ray Diffraction," during the 59th AOCS Joint Meeting with the American Association of Cereal Chemists (AACC) in Washington, D.C., April 1968.

This manuscript was intended to be used as a guide to the subcommittee to standardize nomenclature, symbols, and procedural techniques. However, collaborative teams to pursue these plans were never established. Finally, after a survey of membership interest, the subcommittee chairman inactivated this Subcommittee in 1969. The recommendation was accepted by the entire committee and by the Uniform Methods Committee.

PRESENT ACTIVITIES AND PLANS

Two additional subcommittees have been created. A subcommittee to investigate atomic absorption methods for the identification and quantitative analysis of the chemical elements, both mineral and nonmineral, was added to the Instrumental Techniques in April 1968. This subcommittee has completed a detail investigation of a direct method for determination of chemical elements in fats and oils. The method has been approved by the entire Instrumental Techniques Committee and submitted to the Uniform Methods Committee with the recommendation that it be added to the Society's book of "Offical and Tentative Methods." The procedure has satisfactory precision, as determined by collaborative effort in various laboratories in this country and is rapid and conventient. However, it lacks sufficient sensitivity for analysis of refined and bleached oils. The subcommittee now is investigating methods to increase sensitivity. Techniques involving preconcentration by means of ashing or by solvent extraction procedures have not been entirely satisfactory, apparently because of inability to quantitize the ashing or preextraction procedures. The subcommittee in collaborative study, is testing the graphite furnace and carbon rods or carbon tube atomizers to develop a more sensitive method.

Between 1965 and ca. 1970, several groups urged the Instrumental Techniques Committee to form a Nuclear Magnetic Resonance Subcommittee and in 1970 the Nuclear Magnetic Resonance Subcommittee was formed. A chairman was appointed and plans were made to study, in collaborative investigations, methods for the determination of solid/fat index and for the determination of total oil content of vegetable oils by wideline NMR spectroscopy.

Unfortunately, the newly selected chairman was able to serve only a short term, and difficulty was encountered in developing collaborative groups. Finally, however, collaborative testing of methods for solid/fat index got underway with a task group leader from Unilever Research, Vlaardingen, Netherlands, and with three laboratories in the U.S., three in England, two in The Netherlands, and one each in Sweden and Canada. In early collaborative testing of wideline NMR techniques for the percentages of solids in such samples as liquid oil, hardened fish oil, margarine fat, etc., lack of precision among the various collaborating laboratories was a problem. Further tests showed that this lack of precision arose from sampling or presampling treatment. When collaborators were furnished specific samples or samples prepared from specific instructions, agreement among laboratories was good, with standard deviations among all laboratories of 1-2%.

At this point, objections were raised, because the proposed method was limited to wideline NMR and excluded pulsed source instruments. Pulsed source instruments were tested on a limited scale and preliminary results indicated that they might be superior to the wideline instruments. The subcommittee decided, however, that the method should not be limited

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to either wideline or pulsed source procedures for two reasons: (A) procedures based upon wideline NMR have been evaluated and gave satisfactory precision in collaborative tests, while pulsed source has been more recently introduced; and (B) even though pulsed source techniques eventually might be found superior to wideline techniques, they would probably be available to only a few laboratories.

The subcommittee decided to compare the results from the two techniques in another collaborative test. Although only a few collaborators had pulsed source instruments, comparison indicated that identical results could be obtained with either technique if sample preparation was uniform. The subcommittee is now engaged in the sixth test with collaborators in five countries and plans to recommend two procedures, one for wideline the other for pulsed source instruments.

The Nuclear Magnetic Resonance Subcommittee has not, as yet, established collaborative teams to test published procedures for measurement of total oil content of oilseeds by wideline NMR. Without the cooperation of collaborative groups we cannot proceed further.

Only two of the original, or charter, Subcommittees of the Instrumental Techniques Committee are still active. The last contribution from the Spectroscopy Subcommittee was the establishment of an official method for determining isolated *trans*-content of fats and oils, AOCS Method Cd 14-61, "Isolated *Trans*-Isomers." Collaborative tests showed that this method is accurate and, when instruments in various laboratories were calibrated with external standards, provided for use with the official method, precision and agreement among different laboratories was satisfactory. However, the procedure is not as rapid as more recently published methods and the use of external standards creates problems in routine determinations. The subcommittee, therefore, proposed to study collaboratively an alternate procedure which might be made part of the official method in cases where highest precision was not required. Because of insufficient interest, however, the subcommittee chairman recommended that the Spectroscopy Committee be inactivated. Action on this suggestion has been postponed, because there are methods that might be investigated by this subcommittee. For example, there is no official method for the determination of *trans*-content in conjugated samples. Attempts will be made to set up collaborative testing by this subcommittee. If sufficient interest cannot be generated, a proposal for inactivation will be forwarded to the Uniform Methods Committee.

The chairman of the Gas Chromatographic Subcommittee resigned because he has left the area of fats and oil research. A new chairman has been named, but activities of the subcommittee have been delayed while new task groups are being established and the subcommittee is being reorganized. Several interesting proposals for collaborative work to establish official methods within the area of GLC techniques have been proposed. These include (A) determination of pesticides in fats, oils, and other lipids; (B) determination of fatty acid composition by means of programed GLC; (C) quantitative determinations of free sterols by GLC; (D) determination of resin acids in rosin by means of GLC; and (E) composition of turpentine by GLC. The first of these proposals has been urged by, among other groups, the Fat and Oil Section of the International Union of Pure and Applied Chemistry, (IUPAC). This group has offered to assist in the development of an official method, probably furnishing suitable samples and entering into cooperative collaborative testing to lead, eventually, not only to an AOCS official method but an international method through IUPAC. The second proposal has been urged by several gas chromatographists. The AOCS offical

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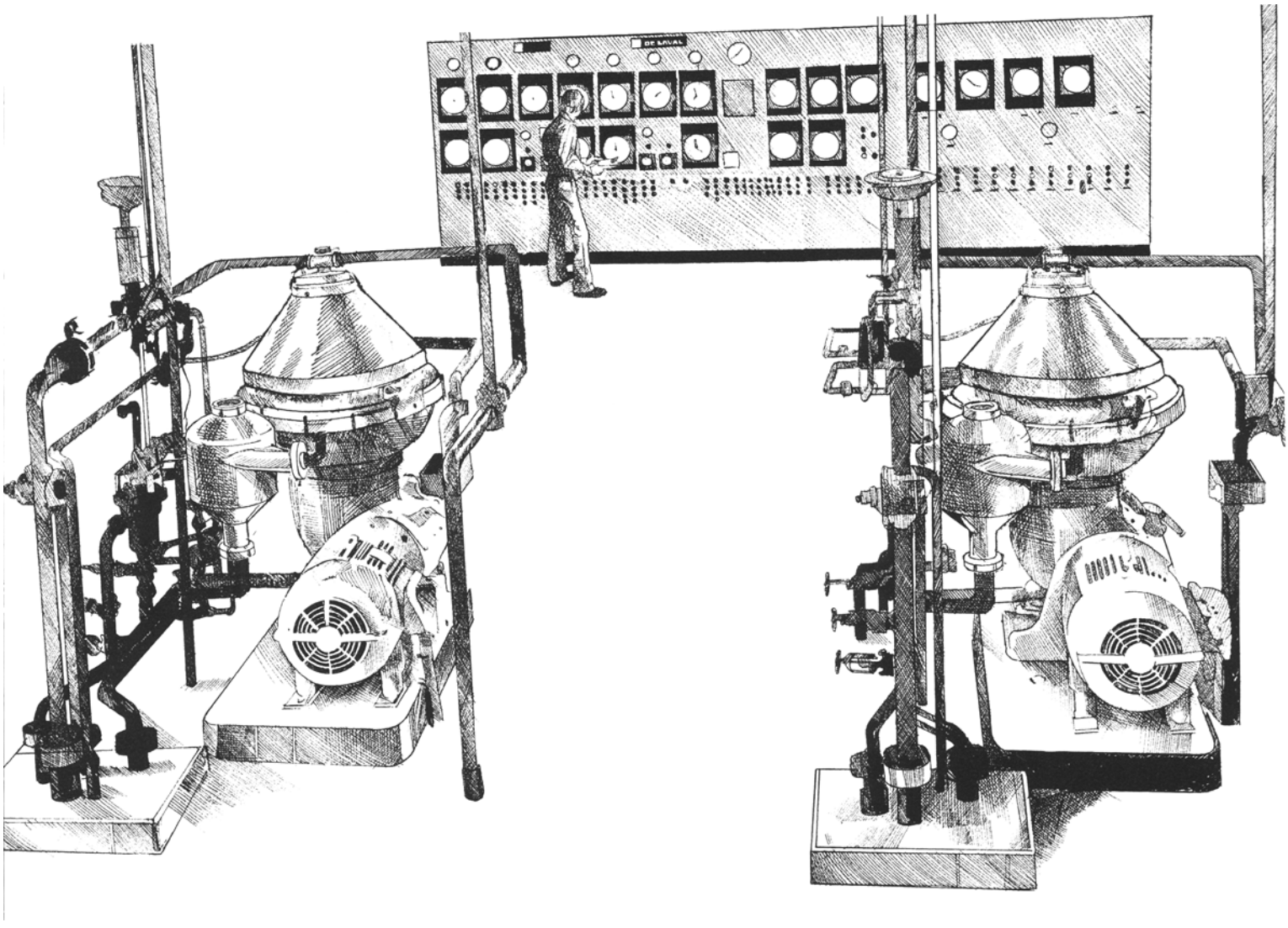

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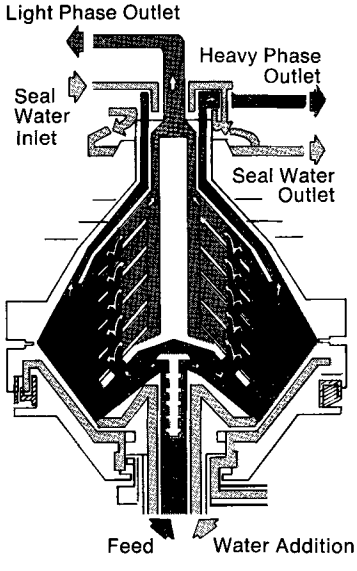

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Method Ce 1-62, "Fatty Acid Composition by Gas Chromatography," does not provide for temperature programing and should be brought up to date. The third proposal has strong backing by the Codex Committee on Fats and Oils of the Joint Food and Agricultural Organization of the United Nations and the World Health Organization Codex Alimentarius Commission. This Commission adopted the AOCS procedure for obtaining GLC patterns of the fatty acids in various fats and oils as a means to identify an unknown fat or oil. There are, however, instances where such patterns for specific fats or oils are somewhat ambiguous. Among several proposals to avoid this ambiguity, (including one to attempt to make all the data truly quantitative) was one to include the patterns for the free sterols in the specific fat or oil to be identified. The quantitative procedure would complicate the method designed for qualitative identification of a specific fat or oil. The sterol patterns will complement the free fatty acid patterns and provide a unique identification for any fat and oil. The Codex Commission feels that a standard or official method for determining such patterns, as is available for fatty acids in AOCS Method Ce 1-62, is required for obtaining the sterol patterns for the proposed qualitative identification of the fats and oils.

INTERNATIONAL ASPECTS OF ACTIVITIES OF THE AMERICAN OIL CHEMISTS' SOCIETY INSTRUMENTAL TECHNIQUES COMMITTEE

When describing various investigations of the subcommittees of the Instrumental Techniques Committee, several references have been made to the international aspects of these activities. A report, entitled "International Aspects of the Activities of the American Oil Chemists' Society's Instrumental Techniques Committee," was furnished by Norris Embree, chairman of the Fats and Oils Section, IUPAC, for presentation at the Oils and Fats Section of the 27th IUPAC Conference in Munich, Germany, August 1973.

As indicated earlier, the Gas Chromatographic Subcommittee has been asked by the Fat and Oil Section of the IUPAC, for details of their proposed investigations to develop a standard method for the determination of pesticides in fats and oils. This group has offered to work with the subcommittee in the development of a method which could be recommended as an official method of AOCS and for consideration as a standard method by IUPAC.

Earlier, this subcommittee had been asked for a specific standard method for the production of GLC patterns of the fatty acids in oils and fats to be used to identify specific vegetable oils or animal fats. These patterns were well received, when furnished to the Codex Commission with copies of offical Method Ce 1-62 (Revised 1970), as the standard procedure for producing them. The Codex Committee has requested an official method for the production of GLC patterns of free sterols in these fats and oils to complement the fatty acid patterns to afford an unambiguous procedure for the identification of a specific vegetable oil or animal fat.

The collaborative effort of the NMR task group to develop a standard method for the determination of solid/fat index by means of NMR has attracted international interest because its collaborative studies are being conducted in five countries. The Oil and Fats Section of IUPAC has requested copies of this method as soon as they become available. They are interested in the method, especially in pretreatment of samples, which they feel could be made a standard procedure for samples for melting point determination where a lack of precision appears to arise from the lack of uniformity in sample preparation.

The Ad Hoc Committee for the preparation of methyl esters from their free fatty acids or from their triglycerides cooperated with the Nederlands-Normalisatie-Institute. The Institute proposed a standard method and suggested that, to promote international standardization and to assist in international commerce in fats and oils, it would be an advantage if their proposed standard and the AOCS method could be made identical. However, the Nederlands-Normalisatie-Institute reported that the method by AOCS would not yield correct values unless the methyl ester solution was dried over anhydrous sodium sulfate. Repeated investigations by the Instrumental Techniques Committee's Ad Hoc Committee gave identical results whether sodium sulfate was used or omitted. Examination of the chromatograms furnished by the Dutch Committee showed that peaks were broader 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 had not changed and prompted a calculation, from the Dutch chromatograms, of these areas by triangulation. This procedure gave correct values for both the wet and dry esters, leading to the conclusion that the errors reported by the Nederlands-Normalisatie-Institute 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 Instrumental Techniques Committee which concluded that, while sodium sulfate drying was not necessary for accurate results, inclusion of this step might prevent similar problems in other laboratories, and, therefore, give better agreement among laboratories, i.e. enhance the precision of the method. Furthermore, inclusion of the sodium sulfate drying step would bring the AOCS method in closer agreement with the Dutch method and such a simple operation would not detract from the attractive features of the BF_3-methanol procedure of providing a rapid preparation of the methyl esters.

It appears that we may be on the verge of establishing international, as well as national, standard methods of analysis. Such world standards would be of obvious advantage in the international commerce in fats and oils. Apparently, international cooperation is available, if we in AOCS want to take advantage of it.

[Received January 31, 1974]

R.J. Limon named marketing manager

Robert J. Limon has been appointed marketing manager for the Chemical Specialties Division, Henkel, Inc.

In his new post, he will be responsible for current and future marketing of Henkel products for plastics and polymers, cosmetics and detergents, paints and pigments, and for metal treatment. He previously had been manager for future market development, following two years as product manager of Henkel's Plastics Department. ■

Jakob Jakobsen dies

Jakob Jakobsen, who first joined AOCS in 1943, died on July 9, 1974, in Denmark.

Mr. Jakobsen, who particiated in a variety of AOCS activities before his retirement, was formerly chief fat technologist for General Mills.

After leaving industry, he worked as a consultant for some time before retiring to Denmark. ■