

# Report of the Instrumental Techniques Committee,

## AOCS 1970-1971

The Instrumental Techniques Committee was established by action of the Governing Board at its 53rd Annual Meeting on May 9, 1962. This action followed acceptance of a report by R. W. Bates of a committee which had been appointed to study the organization of the Society's various committees. The Instrumental Techniques Committee was created by combining into a single committee the Color, Spectroscopy and Gas Chromatography Committees and a Task Group of the Spectroscopy Committee to Prepare Methyl Esters From Free Acids and Triglycerides. Each of the earlier groups became a Subcommittee of the Instrumental Techniques Committee. The Instrumental Techniques Committee is thus a relatively new committee, just eight years old. But, through its Subcommittees, it has its roots deep into the history of the Society.

The objectives of the Instrumental Techniques Committee can be adequately described by quoting its scope as given in the "Manual of the Committee Scopes," a publication which resulted from the excellent work of our Secretary, Lois S. Crauer. It was accepted by the Governing Board at the AOCS Fall meeting in October 1967 as follows.

Scope, Instrumental Techniques Committee: "To promote the development and standardization of testing methods for fats and oils and their derivatives by means of applications of instrumental procedures such as: absorption spectroscopy, gas liquid chromatography, emission spectroscopy, mass spectroscopy, NMR spectroscopy, etc."

The Color Subcommittee of the Instrumental Techniques Committee predates its parent Committee by over 40 years. It was organized as a formal committee of the AOCS in 1918 under the title "Color of Cottonseed Oils and Meals" and is thus over 50 years old. The long list of Chairmen of the committee, which, following one reorganization or another, became known as either the Color Committee or the Oil Color Committee, reads something like a "Who's Who in AOCS." The first chairman was Frank Smalley, followed, in 1920, by David Wesson and, among several others, H. P. Trevithick in 1927 (again in 1941), W. D. Hutchins in 1928, G. W. Agee in 1943, and Ron Stillman in 1949. All of these became Presidents of AOCS. Its long history was one of some frustration between the desire for a truly objective instrumental evaluation of color and a wedlock to subjective measurements with visual glasses, particularly the Lovibond system. This conflict was never really resolved. In 1955 the Oil Color Committee, as it was then called, underwent the last of its many reorganizations before it became part of the Instrumental Techniques Committee. It was reorganized in 1955, with a name change back to Color Committee, with two subcommittees "Oil Color," the transmission measurement of transparent samples, and the "Color of Solids," the reflectance measurement of opaque samples. Its attempts to compromise between an objective and a subjective method resulted in development of the Society's Official Method Ce 13c-50 (Corrected 1963), "Color-Photometric Method," an attempt to tie a truly objective method of color measurement with the subjective Lovibond system. It was inactivated as a Subcommittee of the Instrumental Techniques Committee in April 1965 due to "lack of problems upon which it could work profitably."

The Spectroscopy Subcommittee is another group which predates the parent Instrumental Techniques Committee by several years. It was organized by the Governing Board at its Fall Meeting in Chicago in 1945, through the efforts of, among others, K. S. Markley, Reid Milner, and V. C. Melhenbacker. Its first Chairman was Ron Stillman, who served from 1946 to 1953. R. T. O'Connor was appointed Chairman in 1953 and served until this Committee met the fate of all those described in this brief history, becoming a part of the Instrumental Techniques Committee in 1962. The first achievement of the Committee was the establishment of method Cd 7-58 (Revised 1959) "Polyunsaturated Acids—Ultraviolet Spectrophotometric Method." About the

time it was incorporated into the Instrumental Techniques Committee, it had developed a method involving IR spectrophotometry for the determination of "Isolated Trans Isomers" Official Method Cd 14-61.

The other two subcommittees which are charter members of the Instrumental Techniques Committee are relatively new. The Gas Chromatography Subcommittee was organized as a Joint Committee with the Association of Official Analytical Chemists in 1964. This Joint Committee cooperated with the American Society for Testing and Materials through ASTM Committees D-1, Paint, Varnish, Lacquer and Related Products, and E-19, Chromatography. During the short period of its existence as an independent committee, it developed AOCS Method Ce 1-62 (Corrected 1964, Revised 1968) "Fatty Acid Composition by Gas Chromatography." This method has been revised and updated by the Subcommittee on Gas Chromatography, the most recent revision, to keep up with developments in gas chromatography particularly in the variation of detectors, has just been accepted by the Uniform Method Committee to replace the earlier modification of Ce 1-62.

The Gas Chromatography Subcommittee of the Instrumental Techniques Committee 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, specifications for the identification of specific vegetable oils or animal fats, based on their fatty acid constituents, as determined by GLC. These 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 of the Instrumental Techniques Committee, the Chairman of the American delegation expressed his thanks and those of the U.S. delegates to members of the Committee and through them to the Society for the "excellent cooperation furnished by you and your committee of the AOCS," and then he requested specifications for additional oils. Interest in these specifications among members of AOCS has prompted their publication in JAOCS 47:186A, 195A, 197A (1970). The Gas Chromatography subcommittee is also establishing plans for collaborative testing of methods of analysis, by means of GLC, of sterols and of pesticides in vegetable oils and animal fats.

In 1961, just prior to incorporation into the Instrumental Techniques Committee, the Spectroscopy Committee established a Task Group for the Preparation of Methyl Esters From Free Fatty Acids or From Triglycerides for the purpose of subsequent determination of *trans* isomers by means of the newly developed IR absorption method. This action appeared necessary when it was apparent that certain techniques for the preparation of methyl esters might create *trans* isomers which the IR method was attempting to measure. When this group was made a part of the Instrumental Techniques Committee, it was immediately apparent that the need for a method of preparing methyl esters which would not create isomeric artifacts was of equal or greater interest to the Gas Chromatography Subcommittee. Thus, very early we had evidence of the wisdom in combining these committees concerned with instrumental types of analyses. The subcommittee for the preparation of methyl esters, now working to develop a method for both the spectroscopy subcommittee and the gas chromatography subcommittee, 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 literature reports gave evidence of methods which would be considerably simpler and much faster. The original method Ce 2-66 with the methanol sulfuric acid techniques was entirely satisfactory and had been proven to be a technique which would not result in any isomeriza-

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tion. However, the demand for more rapid methods led the subcommittee to reconsider more recent literature reports and subsequently collaborative effort led to the recommendation of the present AOCS method employing BF<sub>3</sub> and methanol, method Ce 2-66 (Revised 1969) "Preparation of Methyl Esters of Long Chain Fatty Acids." This method was very promptly adopted by the AOCS under their designation 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.

Two additional Subcommittees have been created within the Instrumental Techniques Committee during its eight years of existence. The X-Ray Diffraction Subcommittee was created in April 1965 very shortly after the Instrumental Techniques Committee was organized. Its primary objective was to attempt to develop some standardization in this area of analytical spectroscopy, particularly as regards terminology, symbols, etc., and to attempt to solve some apparently contradictory results and values which have appeared in the literature. Work of the Subcommittee was delayed when its Chairman, Chuck Hoerr, was on a temporary leave of absence to attend to duties connected with his election as Vice President and President of the Society. Chuck is now back with the Subcommittee which has been studying the literature of x-ray diffraction in fats, oils and lipids. A manuscript by the Subcommittee Chairman, 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, entitled "The Role of X-Ray Diffraction in Studies of the Crystallography of Monoacid Saturated Triglycerides" is being used by the Subcommittee to establish greater uniformity in nomenclature, symbols and procedural techniques, much needed in this area, to afford maximum value of available data to the analytical spectroscopist.

A second Subcommittee has been established with the approval of the Governing Board to study methods of atomic absorption in fats, oils, lipids, etc. This subcommittee held its inaugural meeting in April 1968 and hence is only two years old. It is now engaged in its second collaborative effort preliminary to a plan to recommend a method based on atomic absorption for the analysis of the chemical elements in fatty acids and derivatives, in vegetable oils and animal fats, and in lipid materials.

Formation of a third new Subcommittee is being considered. A group of Society members have petitioned the formation of a group to investigate the use of wide-line nuclear magnetic spectroscopy as a technique to measure the oil content of oilseeds or to measure the solid fat index, with the aim of ultimately recommending an official method. If a sufficient number of members are interested and willing to participate in the collaborative investigation necessary to select the most appropriate method, a Nuclear Magnetic Resonance Subcommittee may be formed.

From time to time during the existence of the Instrumental Techniques Committee and of its Subcommittees, particularly the Spectroscopy and Gas Chromatography Subcommittees, the question of overlapping or duplication of scope of the Technical Committees of the Society has been raised. Comparison of the scope of the Instrumental Techniques Committee, as given above, with that of: (a) Biochemical Methods Committee—To survey, study, encourage development of and recommend preparative analytical and other procedures to use in (biochemical) research; (b) Commercial Fats and Oils Analysis Committee—To investigate methods concerned with the analysis of fats and oils sold on the commodity market; or (c) Fat and Oil By-Product Analysis Committee—To develop methods and sampling procedures for soapstocks, glycerine, lecithin and other fat and oil by-products, makes it quite obvious that any method involving instrumentation falls jointly within the scope of all of these committees. As a result, with greater use of instrumentation in analyses, the problem of conflict in scope and duplication of effort has been raised.

This problem has created the need for study by ad hoc committees during the term of office of several AOCS Presidents, the most recent being during the tenure of Ron Stillman and of Ray Reiser. The Instrumental Techniques Committee is a techniques committee, as note the titles of its subcommittees, while most other committees are oriented about specific commodities. Hence with instrumental techniques becoming more and more common, it is certainly possible, even probable, that the Instrumental Techniques Committee and one of the Commodity Committees could be investigating the same method by the same technique. For example, a method to promote the development and standardized test methods for lipids by means of gas chromatography is clearly within the wording of the scopes of the Biochemical Methods Committee and the Instrumental Techniques Committee. A collaborative endeavor to devise a method involving infrared absorption spectroscopy in the area of soaps and detergents clearly falls jointly within the scope of the Soaps and Detergents Analysis Committee and the Instrumental Techniques Committee.

In the final analysis, there appears to be the choice of accepting a potential duplication of effort or facing loss of opportunity to devise a much needed method. For example, the Instrumental Techniques Committee has recently activated a Subcommittee to investigate atomic absorption as a technique for the identification and quantitative measurement of the metal content of oil and lipid materials. The subcommittee has been created from among available specialists within the Society in the field of atomic absorption. This is, perhaps, the only manner in which a truly satisfactory method involving atomic absorption can be investigated. The Instrumental Techniques Committee will thus generate general techniques or methods applicable to most fatty acid, oil or lipid material or by-products, while the Commodity Committees will develop methods for their special products. We have never had an example of too many good methods for consideration within the Uniform Methods Committee—and until this becomes a more real problem, it is doubtful if any reorganization of the Technical Committees is advisable. We are attempting to establish cooperation among other Societies through the Inter-society Relations Committee with the main purpose of eliminating duplication of effort. It is unrealistic to believe that we can accomplish this in InterSociety Relations but not in IntraRelations within our own Society. When potential duplication of effort becomes a reality, it would appear reasonable to assume that with the cooperation between two committees, a pooling of effort will result, and will result in better methods for the Society.

### AOCS Official Methods Developed and Recommended by the Instrumental Techniques Committee

- Cc 13c-50 (Corrected 1963) "Color-Photometric Method."
- Cd 7-58 (Revised 1959) "Polyunsaturated Acids-Ultraviolet Spectrophotometric Method."
- Cd 14-61 "Isolated Trans Isomers."
- Ce 1-62 (Corrected 1964, Revised 1968) "Fatty Acid Composition by Gas Chromatography."
- Ce 2-66 (Revised 1969) "Preparation of Methyl Esters of Long Chain Fatty Acids."

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