# Report of the AOCS Instrumental Techniques Committee, 1972-73

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*The Committee met twice during the year at the 46th Fall Meeting of the Society in Ottawa, Canada, and at the 64th Spring Meeting in New Orleans, La. The Spectroscopy Subcommittee had planned to study, in collaborative investigation, 2 published methods, 1 designed to provide a rapid determination of isolated* trans *isomers without recourse to external standards and the other to increase the sensitivity of these determinations. However, these investigations have been delayed because of lack of interest and inability to establish task groups within various laboratories. The Atomic Absorption Subcommittee completed collaborative testing of a method originating at the Southern Regional Research Center for the direct determination of trace metals in fats and oils by atomic absorption spectroscopy. The method will be submitted to the Uniform Methods Committee with recommendation for inclusion in the* AOCS Book of Official and Tentative Methods. *This Subcommittee has found that methods to provide increased sensitivity in determining small traces of metals by preconcentration by means of extraction procedures are not entirely satisfactory. Apparently this is because of inability to quantify the extraction preconcentration procedure. It will attempt to develop a more sensitive method, required for the analysis of refined oils, by the use of graphite furnace and carbon rods or carbon tube atomizers. Resignations of the chairman and 2 task group chairmen of the Gas Chromatography Subcommittee necessitated a reorganization of this Subcommittee, delaying plans to establish collaborative evaluation of methods for the determination of: (a) pesticides; (b) sterols; (c) resin acids in fats, oils, and other lipids; (d) the composition of turpentine by means of gas liquid chromatography; and (e) the composition of fatty acids by means of programed gas liquid chromatography. The NMR Spectroscopy Subcommittee conducted several collaborative tests to show that the instrumental method has advantages over the present dilatometric procedure in precision, scope, and time of analysis. The NMR procedure was found satisfactory with either wideline or pulsed-source instrumentation but was dependent upon the method of tempering of the sample prior to analysis. A method specifying a precise tempering procedure in detail will be studied in additional collaborative tests. Satisfactory results will permit development of a specific procedure to be recommended as an offical method of the Society.*

## INTRODUCTION

The Instrumental Techniques Committee met during both of the national meetings of the Society. The first meeting was held during the 46th Annual Fall Meeting, with 21 Committee members meeting in the McDonald Room of the Chateau Laurier Hotel in Ottawa, Canada, on September 25, 1972. The second meeting was held during the 64th Annual Spring Meeting in meeting room 2 of the Jung Hotel, New Orleans, La. on April 30, 1973, with 16 attending. Each of the subcommittee chairmen presented reports of progress and plans for the future.

## SPECTROSCOPY SUBCOMMITTEE

The Spectroscopy Subcommittee had been planning collaborative study of 2 proposals to modify AOCS Official Method Cd 14-61 (1). The first proposal was designed to permit rapid determination of *trans* isomers in fatty acids, esters, and triglycerides without recourse to external standards. The proposal would involve study of the published method entitled "A Rapid Method for the Determination of *trans* Unsaturation in Fats and Derivatives" (2). The objective of the second proposal was the inclusion of a technique to permit more accurate measurements of low concentrations of isolated *trans* content than are possible under the official method. Investigation of this proposal also would be based on testing a published method, "Determination of Low Level Isolated *trans* Isomers in Vegetable Oils and Derived Methyl Esters by Differential Infrared Spectrophotometry" (3).

The Subcommittee reported that it had been unable to generate significant interest in either of these proposals and that it had been unable to establish a task group of members in various laboratories to conduct the necessary collaborative studies. It recommended discontinuing consideration of both proposals, at least until such collaborative groups could be assembled. The Subcommittee did not present plans for future activities.

## ATOMIC ABSORPTION SUBCOMMITTEE

Collaborative investigations of a published method (4) have enabled the Atomic Absorption Subcommittee to evaluate and recommend a direct method for determining trace metals in vegetable oils and animal fats by atomic absorption spectrophotometry. A draft of the proposed method has been published (5) and will be submitted to the Atomic Absorption Subcommittee and the entire Instrumental Techniques Committee. Letter ballot, proposing that it be submitted to the Uniform Methods Committee with recommendation for its inclusion in *Official and Tentative Methods of AOCS*, will accompany the draft.

The Atomic Absorption Subcommittee has been considering the extension of its recommended method by including a procedure permitting quantitative determination of small traces of metals commonly found in refined vegetable oils. Collaborative studies and investigations in numerous laboratories have been made of proposed techniques for preconcentration or sample enrichment by such techniques as extracting the metals or ashing the oil sample. Preliminary collaborative results led to the selection of a specific enrichment procedure involving extraction with (ethylene dinitro) tetraaceticacid (EDTA)-hydrochloric acid mixtures at reflux temperatures. However, collaborative results have revealed that neither precision nor recov-

TABLE I

Proposed GLC Methods and Their Present Status

| Method | Status |
|---|---|
| (1) Determination of pesticides in fats, oils, and other lipids. | An AOAC method being evaluated for adoption as an AOCS method |
| (2) Determination of fatty acid composition by programed gas liquid chromatography | Collaborative study |
| (3) Determination of free sterols | Collaborative study |
| (4) Determination of resin acids in rosin | Method recently adopted by ASTM being evaluated for adoption as AOCS method |
| (5) Determination of turpentine | Ibid. |

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ery was satisfactory, evidently because of difficulties in quantification of the extraction procedure.

Several of the collaborating teams attempted preliminary tests involving an alternate approach to greatly increased sensitivities through the use of a graphite furnace with carbon rods or a carbon tube atomizer. Since these techniques do not involve extraction, they are more precise and rapid. If a sufficient number of collaborators possessing the required graphite furnace with carbon rods or a carbon tube atomizer can be found, a collaborative investigation of this technique will be conducted.

## GAS CHROMATOGRAPHY SUBCOMMITTEE

S.F. Herb, Subcommittee chairman, has resigned, since his new work assignment will not be concerned with fat, oil, or lipid research. Also resigning were J.L. Iverson, chairman of a task group to investigate a procedure involving programed temperature analysis of fatty acid composition designed to modify or augment AOCS Tentative Method Ce 1-62 Rev. 1970, and B.D. Thomas who had taken over activities to investigate and evaluate gas liquid chromatographic (GLC) methods for determining resin acid in rosin and of turpentine. These resignations will require an almost complete reorganization and restructuring of the Gas Chromatography Subcommittee if the objectives (as outlined in the previous report (5), all involving collaborative effort, are to be achieved. See Table I. Major activities within this Subcommittee during the coming year will probably be confined to this reorganization and will include the appointment of a new Subcommittee chairman and new collaborative investigation or task group leaders.

## NMR SPECTROSCOPY SUBCOMMITTEE

AOCS has not found a member willing to accept appointment as chairman of the NMR Spectroscopy Subcommittee. This vacancy has handicapped the progress of this group, particularly in establishing a collaborative task group to investigate published methods and select a preferred technique for the determination of total oil in oilseeds by wide-line NMR spectroscopy.

A.J. Haighton, Unilever Research, Vlaardingen, The Netherlands, has served as chairman of a task group established to study a method for the determination of solid/fat index by NMR. Despite the handicap of distance, he has made considerable progress with the task group. (See "Report of the Instrumental Techniques Committee 1971-72" for a summary of earlier collaborative tests [5]). This collaborative testing has been international with 2 collaborative teams from the USA, 3 from the UK, and 1 each from Sweden, Holland, Canada, and Switzerland.

In a report of latest collaborative investigation, Haighton stated that the instrumental precision was satisfactory and that the method could readily include procedures permitting the use of wide-line or pulsed-source instruments. A collaborator pointed out one "great advantage of the NMR instrument, namely that measurement can be made on texturized fat without altering the sample's crystalline state." These collaborative studies have demonstrated that the NMR procedures have advantages over the dilatometric techniques (AOCS tentative method Cd 10-57) in precision, scope, and time of analysis. However, the studies are revealing that precision is related to the method of tempering. Since tempering affects the solid/fat index, precision can be obtained among collaborators if, and only if, the method includes a precise, detailed description of a recommended method of tempering. Thus far there has been little, or no, agreement regarding the preferred method. An additional collaborative effort is being organized to test the method when a specific tempering procedure is followed. Collaborators will be asked to follow the method whether or not the tempering procedure agrees with that customarily used in their respective laboratories. Hopefully this method will reveal agreement among collaborators and can be recommended to the Uniform Methods Committee for inclusion as a standard or official method of the Society.

R.T. O'CONNOR, chairman
R.R. ALLEN, subcommittee chairman
K.M. BROBST, subcommittee chairman
S.F. HERB, subcommittee chairman

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lis, Minn.; P.L. Maiers, Doty-Wilhoit Labs, Inc., Minneapolis, Minn.; P.L. Maiers, Doty-Wilhoit Labs, Inc., Minneapolis, Minn.; D.C. Melear, Jr., Southwestern Labs, Fort Worth, Texas; J. Ridlehuber, Plains Cooperative Oil Mill, Lubbock, Texas; J.K. Thomas, USDA, ARS, Russell Research Center, Athens, Ga.; and B.D. Deacon, USDA, ARS, AMRI, Beltsville, Md.

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# Call for Papers

## AOCS 48TH ANNUAL FALL MEETING

**DEADLINE: MAY 3, 1974**

The Technical Program Committee has issued a call for papers to be presented at the AOCS Fall Meeting, September 29-October 2, 1974, in the Sheraton Hotel, Philadelphia, Pennsylvania. Papers on lipids, fats, and oils and all related areas are welcome. Submit three copies of a 100-300 word abstract with title, authors, and speaker to: Gerhard Maerker, Eastern Regional Research Center, U.S. Department of Agriculture, 600 East Mermaid Lane, Philadelphia, Pennsylvania 19118. ■