Methodology

Methods development update

Alternate solvents
Past recommendations for alternate solvents in the iodine value method (AOCS Official Method Cd 1-25) have included trichloroethane (TCE), 1,1,2-trichloro-1,2,2-trifluoroethane (Freon 113), glacial acetic acid alone and a 1:4 mixture of acetic acid/cyclohexane. Based on comments expressed at AOCS technical committee meetings, the use of Freon 113 should not be considered due to environmental concerns.

Several laboratories have made a comparison between TCE and carbon tetrachloride in the iodine value method. The reports from the laboratories show that TCE is most likely not a suitable substitute in the method because TCE consistently gave values that were two iodine value units lower than when carbon tetrachloride was used. Therefore, at this time, comparison studies are concentrating on the use of cyclohexane as an alternate solvent in the iodine value method.

Participants in the Smalley Check Sample Program series on edible fats, NIOP fats & oils and fish oil have been asked, on a volunteer basis, to perform comparison studies between cyclohexane and carbon tetrachloride when analyzing the iodine value of the check samples. To date, favorable results have been reported except for fish oil (high iodine value) and emulsified shortenings. One laboratory is undertaking a study to determine the problem with emulsified shortenings.

From a safety and environmental point of view, the use of cyclohexane appears to be worth serious consideration. The *Chemical Regulation Reporter*, No. 30, Vol 11 (Oct. 23, 1987), notes that "cyclohexane (CAS No.110-82-7) has been produced and used in industry for 40 years with no identified health hazards. The industry group members also presented results of three surveys on occupational exposure to the chemical, and claimed there is no significant consumer exposure to it."

The use of cyclohexane as an alternate solvent to carbon tetrachloride in the iodine value method has been proposed as a Recommended Practice in the *1987 Additions and Revisions to Methods*. This method will appear as AOCS Recommended Practice Cd 1b-25.

Smalley Check Sample Program
At the AOCS Governing Board meeting in November 1987, the Technical Activities Coordinating Committee recommended that the scope of the Smalley Check Sample Program be expanded to allow collaborative studies for the validation of new methodology and continued validation of existing AOCS methods. This recommendation was approved by the Governing Board.

The first project to be undertaken concerns the validation of the copper sulfate catalyzed TKN method vs. the presently used mercuric oxide catalyzed method. While the copper sulfate method has been studied collaboratively among 15 laboratories (*JAOCS 64*: 511 [1987]), there is not sufficient data to permit a decision about which is *the* official method that should be used for referee analysis. At this time, the plan is to incorporate the collaborative study of the two TKN methods (copper sulfate vs. mercuric oxide) into the Smalley Check Sample Program for the 1988-1989 year. The copper sulfate method has been adopted as an official AOCS method and will appear in the *1987 Additions and Revisions* as Method Ba 4b-87.

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Publications

Book reviews

Chromatography of Lipids in Biomedical Research and Clinical Diagnosis (Journal of Chromatography Library, 37), edited by Arnis Kuksis (Elsevier Science Publishing Co. Inc., PO Box 1663, Grand Central Station, New York, NY 10163, 1987, 460 pp., $97.75).

This book is divided into 13 major chapters; each of these is further subdivided into sections discussing specific topics concerning lipid chromatography. Topics covered are general strategies for practical chromatographic analysis of lipids, polar capillary chromatography of intact natural diacyl and triacylglycerols, HPLC of arachidonic acid metabolites involved in inflammation, application of GC-MS techniques to the analysis of prostaglandins and related substances, GLC of plasma intact lipids in clinical research, HPLC of the arachidonyl molecular species of glycerophospholipids in alveolar macrophages and immune responses, HPLC of diacylglycerol and phospholipase C-sensitive glycerolipids in microsomes of normal tissues and dystrophic muscle, chromatographic analysis of phosphoinositides and their breakdown products in activated blood platelets/neutrophils, TLC and HPTLC of phospholipids and glycolipids in health and disease, HPLC of molecular species of glycero-containing phospholipids in studies of lipoproteins and lipid transport, HPLC of glycosphingolipids in brain disease, GC-MS of molecular species of glycerophospholipids and LC-MS of natural glycerolipids.

The above listing is but a fraction of the information presented in this book. Much more detailed information can be found in the many subsections. This book contains a wealth of information of