AOAC referee report on fats, oils

David Firestone, chairman of the AOCS Uniform Methods Committee, is the General Referee for Fats and Oils for the Association of Official Analytical Chemists (AOAC). The following is a summary of Firestone's General Referee report presented at the 1988 AOAC annual international meeting. It includes a summary of actions by the International Union for Pure and Applied Chemistry's (IUPAC) Commission on Fats and Oils.

Three procedures for extracting antioxidants from butter oil have been evaluated, using HPLC to determine extraction efficiency. The International Dairy Federation (IDF) is planning a collaborative study of the determination of BHA, BHT and TBHQ in butter oil by AOAC method 20.009-20.013. It is anticipated that butter oil will be included in the scope of the AOAC method. Studies on the HPLC separation and "tailing" phenomena of antioxidants are continuing. Caution is required regarding OG and Ionox-100 antioxidants, which coelute on most C-18 HPLC columns.

AOAC Associate Referee R.A. DePalma of Procter & Gamble has completed a collaborative study of a capillary column GC method for the determination of C-18 **trans** monoene and **cis-cis** methylene interrupted unsaturated diene and triene fatty acids, as well as general fatty acid composition of vegetable oils, shortenings and margarines. Collaborative results received from 14 laboratories indicate good repeatability and reproducibility. Recovery of analyte from the fortified samples was generally below 100%. A full report of the joint AOAC/AOCS study is in preparation.

The IUPAC Commission on Oils, Fats and Derivatives has drafted a capillary column GLC method for the determination of the fatty acid composition of edible oils and fats (IUPAC Working Report, June 24, 1988, pp. M9-M10).

The IUPAC Commission on Oils, Fats and Derivatives validated and adopted the Phillips and Sanders procedure for the determination of butyric acid in fats containing butterfat [W.D. Pockington and A. Hautfenne, *Pure Appl. Chem. 58:*1419 (1986)].

Robert Ackman and Jeanne Joseph have completed a collaborative study of a capillary column GLC method for the determination of the fatty acid composition of marine oils [R.G. Einig and R.G. Ackman, *JAOCS 64:*499 (1987)]. Theoretical correction factors relative to a C23:0 internal standard are used to calculate the levels of EPA and DHA in each sample. The collaborative study data are under review.

Regarding olive oil adulteration, E. Fedeli and colleagues have developed standard procedures for determining the sterol [NGD Method C 72-1987, *La Riv. delle Sost. Grasse 64:*553 (1987)] and the aliphatic alcohol [NGD Method C 75-1987, *La Riv. delle Sost. Grasse 64:*561 (1987)]. HPLC has been used to detect the presence of undeclared esterified olive oil in olive oil products [R. Flor, *JAOCS 65:*487 (1988), Abstract].

For oxidized fats, M.M. Blumenthal reported on a new colorimetric rapid test for monitoring the cumulative changes in frying oils [*JAOCS 65:*482 (1988), Abstract]. J.M. Snyder [*JAOCS 65:*511 (1988), Abstract] reported on a headspace extraction technique for the quantitative determination of volatiles produced from oxidized vegetable oils. Perkins and Pinter [*JAOCS 65:*783 (1988)] reported on the efficiency of five separation techniques—low-temperature crystallization, countercurrent distribution (aqueous ethanol/hexane and acetonitrile/hexane), adsorption chromatography and partition chromatography. A batch-type distribution method using acetonitrile/hexane was the most effective in concentrating polar products.

The IUPAC Commission on Oils, Fats and Derivatives carried out collaborative studies of a direct

injection method and a headspace method for GC determination of volatile hydrocarbon (hexane) residue in vegetable oils using either a packed or capillary column [A. Hautfenne, W. D. Pocklington and J. P. Wolfe, *Pure Appl. Chem. 59*:1561 (1977)]. The headspace method (IUPAC Method 2.607) was adopted by the commission.

Firestone recommended the following actions:

- Adopt as official first action IUPAC Method 2.310, Determination of Butyric Acid.
- Adopt as official first action IUPAC Method 2.607, Determination of Volatile Hydrocarbon Residues in Fats and Oils.
- Revise 28.B01-28.B05, Triglycerides in Fats and Oils—GC Method, to add the following at the end of section 28.B02(b): "(Equivalent results may be obtained with use of a short capillary column, i.e., 6 m or less.)"
- Adopt as official final action the following official first action methods: 28.104-28.109, β Sitosterol in Butter Oil; 28.120-28.123, Cyclopropene Fatty Acids in Oils; 28.130-28.131, Foreign Fats Containing Tristearin in Lard; and 28.139-28.141, Chick Edema Factor (Dioxins) in Oils and Fats.
- Continue study on all topics.

IUPAC Commission

The 41st meeting of the IUPAC Commission on Oils, Fats and Derivatives was held at the University of St. Andrews, St. Andrews, Scotland, August 16-18, 1988. The meeting was chaired by commission chairman Joyce Beare-Rogers of the Food Directorate, Department of National Health and Welfare, Ottawa, Canada.

The commission reviewed reports submitted by the coordinators of eight working groups covering 1987-88 studies of methods for determination of mineral oil residues, color of commercial lecithins, lead in oils and fats by AAS, triglycerides as ECN (equivalent carbon numbers) by RP-HPLC, polymerized triglycerides by gel permeation HPLC, phospholipids by HPLC, fatty acids (including n-3 and n-6 fatty acids in animal fats) by capillary GC, and benzo-a-pyrene (rapid method). Results of collaborative studies of methods for determination of triglycerides by RP-HPLC and lead in oils and fats by AAS were satisfactory, and these methods were adopted.

Twenty laboratories from 12 countries participated in a third study of the HPLC triglyceride method. Four samples (palm-sunflower oil blend used as a training sample, olive oil, rapeseed oil and palm oil) were sent to collaborators for analysis using a 5-μm C-18 column with refractive index detector [acetone-acetonitrile (50:50) elution solvent]. Results from 16 laboratories were subjected to statistical analysis. For ECN 40 to 48, reproducibility relative standard deviation (RSDR) varied with concentration as follows: 0.5%, RSDR=21; 3-5%, RSDR=8-16; 5-10%, RSDR=3-6; 10-30%, RSDR=2-4; 30-65%, RSDR=1.6-2.5. RSDR values for ECN 50 (concentration range, 5-10%) were 7-10. All but two participating laboratories identified peaks eluting before ECN 38 as mono- and diglycerides. Good results were also obtained with the rapid method for benzo-a-pyrene. However, little progress was made with the method for determination of mineral oil residues.

New topics to be considered by the commission include determination of contaminants in oils shipped in bulk, halogenated solvents in olive oils, precision data for the peroxide value and acidity value methods, fat and oil refining, and phosphorus in oils. The commission also agreed to support the ISO study of the proposed replacement of carbon tetrachloride with cyclohexane in the procedure for determination of iodine value, and to cooperate with the Commission on Food Chemistry in a project assessing lipid oxidation. The commission agreed to establish a joint committee with the Commission on Food Chemistry on the topic of lipid oxidation, and Prof. H. Wessels accepted the position of cochairman of this committee.