Committee on Residues and Related Topics

STEPHEN G. CAPAR, CHAIR
U.S. Food and Drug Administration, Center for Food Safety and Applied Nutrition, 5100 Paint Branch Pkwy, College Park, MD 20740-3835

JOANNE M. COOK, SECRETARY
Florida Department of Agriculture, Chemical Residue Laboratory, 3125 Conner Blvd, Tallahassee, FL 32399-1650

JAMES BELL
2314 36th Ave, San Francisco, CA 94116

HEIDI HICKES
Montana Department of Agriculture, McCall Hall, Montana State University, Bozeman, MT 59717-0362

ALEXANDER J. KRYNITSKY
U.S. Food and Drug Administration, Center for Food Safety and Applied Nutrition, 5100 Paint Branch Pkwy, HFS-336, College Park, MD 20740-3835

STEVEN J. LEHOTAY
U.S. Department of Agriculture, ARS ERRC, 600 E. Mermaid Ln, Wyndmoor, PA 19038

CHRISTOPHER L. RITLAND
Nevada Division of Agriculture, 350 Capitol Hill Ave, Reno, NV 89502

FRANK J. SCHENCK
U.S. Food and Drug Administration, Southeast Regional Laboratory, 60 Eighth St NE, Atlanta, GA 30309

CHARLES WEISBURG, SAFETY ADVISOR
U.S. Environmental Protection Agency, Environmental Science Center, 701 Mapes Rd, Fort Meade, MD 20755-5350

RICHARD NEWELL, STATISTICAL ADVISOR
U.S. Food and Drug Administration, Center for Food Safety and Applied Nutrition, HFS-705, 5100 Paint Branch Pkwy, College Park, MD 20740-3835

Committee Actions

In 2004, a collaborative study with international participation was conducted to evaluate the method *Pesticides in Foods Using Acetonitrile Extraction and Partitioning with Magnesium Sulfate* (QuEChERS method) for pesticide residues in fruits and vegetables. In all, 13 laboratories in 7 countries provided results in the collaborative study, which entailed 20 fortified pesticides at 3 levels between 10–1000 ng/g, plus 7 incurred residues, in 3 matrixes (grape, lettuce, and orange). Preliminary statistical review of the results was conducted by the Study Director according to AOAC criteria and the results indicate that the method is acceptable for nearly all pesticides tested in each commodity. The Study Director is preparing the final report and is seeking Official AOAC status for the method with full support of the Committee.

A poster was presented at this meeting reporting results from an interlaboratory study of *Gamma Spectroscopic Determinations of Cesium-137 in Milk and Other Liquids Using Solid State Intrinsic Germanium Detector.* Milk and juice samples have been evaluated by 10 and 19 laboratories, respectively, with favorable results. The Committee will investigate the need for additional validation of this method. Stakeholders include members of federal agencies, state agencies, and the Food Emergency Response Network.

A new topic*, Ultra-Trace Method for Pesticides in Bottled Soft Drinks,* has been introduced. Study Director Paul Milne has been working with David Soderberg to plan a single-laboratory validation on 2 methods, one for GC/MS and a second for LC/MS detection of trace level pesticides. Methods are being developed. The Committee established a subcommittee of pesticide experts to review the proposed analytical approaches as well as other available methods and to propose the best analytical method for the needed pesticides and matrixes. The proposal will include a protocol for single-laboratory validation.

This past year, the OMB changed the applicability statements of Method **999.10** *Lead, Cadmium, Zinc, Copper, and Iron in Foods, Atomic Absorption Spectrophotometry after Microwave* and **999.11** *Determination of Lead, Cadmium, Copper, Iron, and Zinc in Foods, Atomic Absorption Spectrophotometry after Dry Ashing*. For Method **999.10**, considering the reported RSD_R values, the Pb applicability level was changed from 0.1 to 0.4 mg/kg and the Cu applicability level was changed from 0.2 to 3 mg/kg. For Method **999.11**, the applicability statement was expanded to the following:

[Applicable to determination of Zn, Cu, and Fe in a variety of foods by dry ashing and flame atomic absorption spectrometry (FAAS), and Cd and Pb by dry ashing and graphite furnace atomic absorption spectrometry (GFAAS). Method is capable of determining these elements at concentrations above approximately Pb (0.3), Cd (0.1), Zn (1), Cu (5), and Fe (7) mg/kg.]

Pesticides and Other Chemical Contaminants, David Soderberg

David Soderberg, United States Environmental Protection Agency, OPP, HED, RRB3, Room 821D, Crystal Mall II, 7509C, Ariel Rios Bldg, 1200 Pennsylvania Ave, Washington, DC 20460

(1) ***998.01*** *Synthetic Pyrethroids:* Method Advisor Guo-Fang Pang, Qinhuangdao Entry-Exit and Quarantine Bureau, No. 39 Haibin Rd, P.C. 066002, Qinhuandao, Peoples Republic of China, Tel/Fax: 86-335-341-7119, E-mail: panggfciq@pang.com.cn. Adopted as Final Action. Method is being published in the new edition of the *Official Methods of Analysis*. Recommend to continue topic.

(2) ***2002.03*** *Pesticides in Nonfatty Foods Using SFE and GC/MS:* Method Advisor Steven J. Lehotay, U.S. Department of Agriculture, Agricultural Research Service, Regional Research Center, Food Safety Research Unit, 600 E. Mermaid Ln, Wyndmoor, PA 19038, Tel: 215-233-6433, Fax: 215-233-6642, E-mail: slehotay@errc.ars.usda.gov. This method has been approved for Final Action and is being published in the new edition of the *Official Methods of Analysis*. Recommend continue topic until the method has been available through *Official Methods of Analysis* at least 1 year.

(3) *Miniaturized Methods*: Topic Advisor Frank Schenck, U.S. Food and Drug Administration, Southeast Regional Laboratory, 60 Eighth St NE, Atlanta, GA 30309, Tel: 404-253-1200, Fax: 404-253-1208, E-mail: fschenck@ora.fda.gov. This topic area is very active with current investigations into SPE cleanups for LC-postcolumn derivatization and other matrix interferences for LC and GC detectors. Continue topic.

(4) *Chlorinated Dioxins*: Topic Advisor Douglas Hayward, U.S. Food and Drug Administration, Center for Food Safety and Applied Nutrition, HFS-336, 5100 Paint Branch Pkwy, College Park, MD 20740-3835, Tel: 301-436-1654, Fax: 301-436-2632, E-mail: douglas.hayward@cfsan.fda.gov. This topic has been very active this year. Continue topic.

(5) *Determination of Residues of Triazines and Their Chloro-Metabolites in Raw Agricultural Commodities*: Topic Advisor Robert Yokley, Syngenta Crop Protection, Inc., PO Box 18300, Greensboro, NC 27409, Tel: 336-632-2142, Fax: 336-632-7645, E-mail: Robert.yokley@syngenta.com. Nine new methods were reported including multiresidue and immunoassay procedures. Continue topic.

(6) *Pesticides in Foods Using Acetonitrile Extraction and Partitioning with Magnesium Sulfate (QuEChERS Method):* Study Director Steven J. Lehotay. A collaborative study involving 13 laboratories has been completed. This very active topic continues to be highly productive and the method is coming into use in several laboratories. Recommend the collaborative study results be evaluated for acceptance by AOAC INTERNATIONAL.

(7) *Multiresidue Methods for Pesticides in Foods by GC/MS and LC/MS/MS:* Topic Advisor Guo-Fang Pang. Four methods have been developed. The Study Director will propose one method for collaborative study. Continue topic.

(8) *Ultra-Trace Method for Pesticides in Bottled Soft Drinks*: Study Director Paul Milne, Pepsi Cola Co., 100 Stevens Ave, Valhala, NY 10595, Tel: 914-742-4743, Fax: 914-0749-3323, E-mail: pmilne@pepsi.com. Analytical approaches have been proposed and are being evaluated by a subcommittee. Initiate topic.

(9) *Post-Extraction GPC Cleanup for Pesticide Residues*: Topic Advisor Michael Halvorson, OI Analytical, 151 Graham Rd, College Station, TX 77842-9010, Tel: 800-653-1711, Fax: 979-690-0440, E-mail: mhalvorson@oico.com. This topic has been very active for several years. Recommend Halvorson as a new Topic Advisor and initiate topic.

Radioactivity, Edmond J. Baratta

Edmond J. Baratta, U.S. Food and Drug Administrtation, Winchester Engineering and Analytical Center, 109 Holton St, Winchester, MA 01890

(1) *Iodine-131 in Milk and Foods*: Work is needed in this area. Further action is contemplated. Search for a Topic Advisor continues. Continue topic.

(2) *Gamma Spectroscopic Determinations of Cesium-137 in Milk and Other Liquids Using Solid State Intrinsic Germanium Detector*: Milk and juice samples have been evaluated by 10 and 19 laboratories, respectively, with favorable results. A poster reporting the results of these studies was presented at the Annual Meeting by the General Referee. Search for a Topic Advisor continues. Recommend a protocol be prepared for a collaborative study.

(3) *Strontium-90*: Study Director Marina Silverstone, Washington Department of Health, Division of Laboratory Radiation, 1610 NE 150th St, Seattle, WA 98155-7224, Tel: 206-361-2894, Fax: 206-361-2899, E-mail: marina.silverstone@goh.wa.gov. A "Rapid Method for Analyzing Strontium-90 in Water" is being developed using Eichrom® cartridges. Continue topic.

(4) *Plutonium-239:* A promising method using ICP-MS should be studied further. Search for Topic Advisor continues. Continue topic.

Metals and Other Elements, Milan Ihnat

Milan Ihnat, Pacific Agri-Food Research Centre-Summerland, Agriculture and Agri-Food Canada, Summerland, British Columbia, V0H 1Z0, Canada

(1) ***999.10*** *Lead, Cadmium, Zinc, Copper, and Iron in Foods, Atomic Absorption Spectrophotometry after Microwave:* Method Advisor Lars Jorhem, National Food Administration, Box 622, S-751 26 Uppsala, Sweden, Tel: 46 18 17 55 00, Fax: 46 18 10 58 48, E-mail: lajo@slv.se. Approved for Final Action. Continue monitoring.

(2) ***999.11*** *Determination of Lead, Cadmium, Copper, Iron, and Zinc in Foods, Atomic Absorption Spectrophotometry after Dry Ashing:* Method Advisor Lars Jorhem. Approved for Final Action. Consider whether a modification is required to the applicability statement in light

of recent observations by the Method Advisor of the interference of tin on iron determination in fruit and vegetables preserved in tin cans. Continue monitoring any reports from users of the Official Method.

(3) *Atomic Absorption Spectrometry*: Topic Advisor Milan Ihnat. Submit to *J. AOAC Int.* the manuscripts "Elemental Calibration Solutions for Atomic Absorption Spectrometry" and "Development of a Reliable Acid Decomposition-Direct Aspiration Flame Atomic Absorption Spectrometric Method for the Analysis of Biological Materials"; complete preparation of reports "Application of Acid Decomposition-Direct Aspiration Flame Atomic Absorption Spectrometry to the Elemental Characterization of Agricultural/Food Reference Materials in a Certification Campaign" and "Flame Atomic Absorption Spectrometric Methodologies for Food Analysis-A Review". Complete development of a proposed unified, flame atomic absorption spectrometric (FAAS) scheme of analysis of foods, feeds, food supplements, and biological materials for a range of elements and submit for collaborative study approval and publication in *J. AOAC Int.* Coordinate these developments on FAAS with ICP-AES, ICP-MS, and INAA/RNAA methods reported on below. Continue study.

(4) *Elements in Foods, Feeds, Food Supplements, and Biological Materials by Inductively Coupled Plasma-Atomic Emission Spectrometry*: Topic Advisors Milan Ihnat and Ralph E. Sturgeon, Chemical Metrology, Institute for National Measurement Standards, National Research Council of Canada, Ottawa, ON K1A 0R9, Canada, Tel: 613-993-6395, Fax: 613-993-2451, E-mail: Ralph.Sturgeon@nrc.ca. Complete the planning phase of this study with selection of elements, materials, decomposition, and measurement procedures to be investigated and delineation of methodological details. Integrate activities with concurrent studies on atomic absorption and inductively coupled plasma-mass spectrometries. Continue study.

(5) *Elements in Foods, Feeds, Food Supplements and Biological Materials by Inductively Coupled Plasma-Mass Spectrometry:* Topic Advisors Milan Ihnat, Lu Yang, and Ralph E. Sturgeon. Complete the planning phase of this study with selection of elements, materials, decomposition, and measurement procedures to be investigated and delineation of methodological and experimental details. Carry out preliminary determinations on a variety of digested matrixes, assessing practical detection limits and isobaric and polyatomic interference corrections required for potential analyte elements of interest using quadrupole and magnetic sector ICP mass spectrometers, different spectrometer resolutions, and different argon plasmas. Integrate activities with concurrent studies on atomic absorption and inductively coupled plasma-atomic emission spectrometries. Continue study.

(6) *Graphite Furnace Atomic Absorption Spectrometric Determination of Chromium in Foods:* Study Director Nancy J. Miller-Ihli, U.S. Department of Agriculture, Nutrient Composition Laboratory, Human Nutrition Research Center, Building 161, BARC-East, Beltsville, MD 20705, Tel: 301-504-8252, Fax: 301-504-8314, E-mail: miller-ihli@bhnrc.usda.gov. Due to the retirement of Miller-Ihli, it may not be feasible to complete the collaborative study or peer-validation on the graphite furnace atomic absorption method for the determination of chromium in foods and biological materials, based on the method published (*J. AOAC Int.* (1992) **75**, 354–359). Discontinue topic.

(7) *Lead in Wines:* Study Director Alan L. Reisig, Alcohol and Tobacco Laboratory, 6000 Ammendale Rd, Ammendale, MD 20705-1250, Tel: 240-264-1436, E-mail: Alan.Reisig@ttb.gov. Complete revision of the collaborative study, *Lead in Beverage Alcohol, Graphite Furnace Atomic Absorption Spectrometric Method (G-21)*, following additional statistical analysis carried out previously and resubmit modified method. Make a last concerted effort, with assistance of the General Referee, to settle the collaborative study evaluation funding issues and bring this study to a conclusion. Continue study.

(8) *Neutron Activation Analysis:* Topic Advisors Borut Smodis, Jozef Stefan Institute, Jamova 39, SI-1000 Ljubljana, Slovenia, E-mail: Borut.Smodis@IJS.SI and Jan Kucera, Nuclear Physics Institute, Academy of Sciences of the Czech Republic, CZ-250 68 Rez near Prague, Czech Republic, Tel: 42-2-66172268, Fax: 42-2-6857003, E-mail: Kucera@ujf.cas.cz. Continue with joint cooperative pursuit of method development involving one or both of the techniques of instrumental neutron activation analysis (INAA) and radiochemical separation neutron activation analysis (RNAA). Complete initial study planning stages dealing with selection of elements, materials, and experimental measurement procedures to be investigated. Integrate activities within this study with studies on atomic absorption spectrometry and inductively coupled atomic emission and plasma mass spectrometries to benefit from elemental, material, and methodological considerations formulated in these other studies. Continue study.

(9) *Total Mercury in Food by Cold Vapor Atomic Absorption Spectrometry*: Topic Advisor Robert W. Dabeka, Food Research Division 2203D, Health Protection Branch, Health Canada, Ottawa, Ontario K1A 0L2, Canada, Tel: 613-957-0951, Fax: 613-941-4775, E-mail: Bob_Dabeka@hc-sc.gc.ca. Continue with research/development of method for total mercury in foods utilizing determinative techniques of cold vapor atomic absorption spectrometry and pretreatment and digestion methods as may be required. Prepare a detailed version of the method for validation. Continue study.