

## METHODS COMMITTEE REPORTS

## Committee on Residues and Related Topics

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### Committee Actions

Last year, the topic *Pesticides in Foods Using Acetonitrile Extraction and Partitioning with Magnesium Sulfate* was added to the Pesticides and Other Chemical Contaminants General Referee. Steve Lehotay, Study Director, prepared the study protocol and submitted it to the General Referee. The study did not officially proceed through the AOAC process since funding was not available. Therefore, committee

members did not review the protocol to assess conformance to AOAC requirements. The study has now proceeded through the collaborative study phase. The Study Director is awaiting results from 2 laboratories and will be preparing a report on the study. Further details are included in the General Referee report.

One additional topic has been proposed for inclusion in the Committee report this year: *Multiresidue Methods for Pesticides in Foods by GC/MS and LC/MS/MS* under the General Referee, Pesticides and Other Chemical Contaminants. Two Topic Advisors have been added for current topics: Neutron Activation Analysis and Determination of Residues of Triazines and Their Chloro-Metabolites in Raw Agricultural Commodities.

Three First Action methods have been recommended for approval as Final Action: **999.10**, *Zinc, Copper, and Iron in Foods, AA Spectrophotometric Method (NMKL)*; **999.11**, *Metals in Foods, AAS Method*; **2002.03**, *Pesticides in Nonfatty Foods Using SFE and GC/MS*.

### *Pesticides and Other Chemical Contaminants, David Soderberg*

(1) **998.01**, *Synthetic Pyrethroids*: Method Advisor Guo-Fang Pang, Qinhuangdao Entry-Exit and Quarantine Bureau, No. 39 Haibin Rd, P.C. 066002, Qinhuandao, People's Republic of China, Tel/Fax: 86-335-341-7119, E-mail: panggfciq@pang.com.cn. Adopted as Final Action with Comments May 2003. Continue monitoring any reports from users of Official Method **998.01** until publication of new edition of *Official Methods of Analysis*.

(2) **2002.03**, *Pesticides in Nonfatty Foods Using SFE and GC/MS*: Study Director Steven J. Lehotay, U.S. Department of Agriculture, Agricultural Research Service, Regional Research Center, Food Safety Research Unit, 600 East Mermaid Ln, Wyndmoor, PA 19038, Tel: 215-233-6433, Fax: 215-233-6642, E-mail: slehotay@errc.ars.usda.gov. Adopted as First Action, May 2002. Since no adverse reports have been received from users, recommend adoption as Final Action.

(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 control of matrix effects. Continue topic.

(4) *Chlorinated Dioxins*: Topic Advisor Douglas Hayward, U.S. Food and Drug Administration, 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. The Topic Advisor is revitalizing this topic. Continue topic.

(6) *Pesticides in Foods Using Acetonitrile Extraction and Partitioning with Magnesium Sulfate*: Topic Advisor Steven J. Lehotay. This very active topic has produced a study that is currently looking for support for a collaborative study. Continue topic.

(7) *Multiresidue Methods for Pesticides in Foods by GC/MS and LC/MS/MS*: Topic Advisor Guo-Fang Pang. This topic is very active and is expected to soon lead to a proposed collaborative study. Initiate topic.

#### *Radioactivity, Edmond J. Baratta*

(1) *Iodine-131 and Cesium-137 in Milk and Foods*: Prepare protocol for collaborative study for *Cesium-137 in Milk and Foods* using the newer type Germanium detectors with computer assisted software. The method has been tested using 10 laboratories and the results have been very favorable. Further action is contemplated. Search for and appoint a new Study Director and Topic Advisor. Continue topic.

(2) *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.

(3) *Plutonium-239*: Recommend a Study Director for the new method using ICP-MS. Continue topic.

#### *Metals and Other Elements, Milan Ihnat*

(1) *Atomic Absorption Spectrometry*: Topic Advisor Milan Ihnat, Pacific Agri-Food Research Centre-Summerland, Agriculture and Agri-Food Canada, Summerland, British Columbia V0H 1Z0, Canada, Tel: 250-494-6411, Fax: 250-494-0755, E-mail: ihnatm@agr.gc.ca. Submit to *J. AOAC Int.* the manuscript "High Reliability Analysis of Biological Materials by Acid Decomposition Flame Atomic Absorption Spectrometry"; complete and submit to *J. AOAC Int.* a report with the working title "Determination of Aluminium, Calcium, Copper, Iron, Potassium, Magnesium, Manganese, Sodium, Strontium, and Zinc in Agricultural/Food Reference Materials by Acid Decomposition-Flame Atomic Absorption Spectrometry"; complete and submit to *J. AOAC Int.* a report "Flame Atomic Absorption Spectrometric Methodologies for Food Analysis-A Review". Complete development of a unified, flame atomic absorption scheme of analysis of foods for a range of elements and submit for collaborative study approval and publication in *J. AOAC Int.* Coordinate these developments on AAS with ICP-AES, ICP-MS, and INAA/RNAA methods reported below. Continue study.

(2) *Elements in Foods, Feeds, and Biological Materials by Inductively Coupled Plasma-Atomic Emission Spectrometry*: Topic Advisors Milan Ihnat and Victor J. Boyko 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 planning of study including selection of elements, materials, decomposition, and measurement procedures to be investigated and complete initial method evaluation and experimental phases. Integrate with studies on atomic absorption spectrometry and inductively coupled plasma-mass spectrometry. Continue study.

(3) *Elements in Foods, Feeds, and Biological Materials by Inductively Coupled Plasma-Mass Spectrometry*: Topic Advisors Milan Ihnat, Lu Yang, 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 planning of study including selection of elements, materials, decomposition, and measurement procedures to be investigated and complete initial method evaluation and experimental phases. Integrate with studies on atomic absorption spectrometry and inductively coupled plasma-atomic emission spectrometry. Continue study.

(4) *Graphite Furnace Atomic Absorption Spectrometric Determination of Chromium in Foods*: Study Director Nancy J. Miller-Ihli, Nutrient Composition Laboratory, Beltsville Human Nutrition Research Center, U.S. Department of Agriculture, Building 161, BARC-East, Beltsville, MD 20705, Tel: 301-504-8252, Fax: 301-504-8314, E-mail: miller-ihli@bhnrc.usda.gov. 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.* **75**, 354-359). Continue study.

(5) **997.15**, *Lead in Sugars and Syrups, Graphite Furnace Atomic Absorption Method*: Study Director Nancy J. Miller-Ihli. Adopted as Final Action in January 2004, with changes. Applicability statement changed to specify high fructose corn syrup and to include levels of lead which can be determined. Discontinue topic.

(6) **999.17**, *Lead and Cadmium Extracted from Ceramic Foodware, Graphite Furnace (GFAAS) Method*: Study Director Susan C. Hight, Elemental Research Branch, Center for Food Safety and Applied Nutrition, U.S. Food and Drug Administration, HFS-338, 5100 Paint Branch Pkwy, College Park, MD 20740-3835, Tel: 301-436-1652, Fax: 301-436-2632, E-mail: susan.hight@cfsan.fda.gov. Adopted as Final Action in January 2004. Discontinue topic.

(7) *Lead in Calcium Supplements*: Study Director vacant. This topic is adequately covered in existing topics and is unlikely to be collaboratively studied as a separate method. Discontinue topic.

(8) *Lead in Wines*: Study Director Alan L. Reisig, BATF Laboratory, 1401 Research Blvd, Rockville, MD 20850, Tel: 301-413-5227, Fax: 301-413-4419, E-mail: Alreisig@atfhq.atf.treas.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 and resubmit modified method; settle collaborative study funding issues. Continue study.

(9) *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. Investigate the applicability of the 2 major subdivisions of neutron activation analysis, namely, instrumental neutron activation analysis (INAA) and radiochemical separation neutron activation analysis (RNAA) to the determination of important elements in foods and related biological materials. Complete initial study planning stages dealing with selection of elements, materials, and measurement procedures to be investigated. Integrate

activities with this study with studies on atomic absorption spectrometry and inductively coupled plasma-atomic emission and -mass spectrometries. Continue topic.

(10) **999.10**, *Zinc, Copper, and Iron in Foods, AA Spectrophotometric Method (NMKL)*: 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. Since no adverse reports have been received from method users, recommend adoption as Final Action.

(11) **999.11**, *Metals in Foods, AAS Method*: Method Advisor Lars Jorhem. Since no adverse reports have been received from method users, recommend adoption as Final Action.

(12) *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 into method for total mercury in foods utilizing determinative techniques of cold vapor atomic absorption spectrometry and pretreatment and digestion methods as may be required. Continue topic.