

METHODS COMMITTEE REPORTS

Committee on Residues and Related Topics

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Committee Actions

The Committee Chair and Secretary presented a proposal to the Official Methods Board (OMB) and the AOAC Board of Directors (September 2006) to revise the way the members

of several AOAC committees prioritize methods for analysis of chemical contaminants and residues in food. These changes, if implemented, will establish procedures for working with the community of stakeholders interested in trace level analysis of foods. They will include method needs statements and method priority setting by members of multiple committees. These procedures will include solicitation of comment by stakeholders and development of priority methods through the establishment of expert panels. The Committee Secretary assisted on preparation of an article for AOAC's *Inside Laboratory Management* on how the Committee may transform into a community (*Inside Laboratory Management*, July/August 2006, pp 10–11).

During the January 2006 meeting of the OMB, the Committee Chair recommended and the OMB approved the correction of an editorial error in the applicability statement of Method **999.11** *Determination of Lead, Cadmium, Copper, Iron, and Zinc in Foods, Atomic Absorption Spectrophotometry after Dry Ashing* to change the iron level from 7 to 4 mg/kg.

The Committee continued to encourage Study Director, Steven Lehotay, to submit the report on the 2004 collaborative study on the method *Determination of Pesticide Residues in Foods by Acetonitrile Extraction and Partitioning with Magnesium Sulfate* (nicknamed QuEChERS) for pesticide residues in fruits and vegetables. The Study Director has sent the final draft of the report to his collaborators for comments prior to submitting the report to AOAC.

During last year's Annual Meeting, as a result of a need presented to AOAC INTERNATIONAL, the Committee established a subcommittee of pesticide residue experts assigned to devise analytical protocols for analysis of bottled soft drinks for low levels of pesticide residues. The subcommittee composed 2 methods and wrote single-laboratory validation (SLV) protocols for each method: *Low-level Agricultural Residues in Nonalcoholic, Nondairy, Nonjuice Beverages by LC/MS/MS* and *Low-Level Agricultural Residues in Nonalcoholic, Nondairy, Nonjuice Beverages by Gas Chromatography with Mass-Selective Detection*. Study Director, Paul Milne, has completed the SLV on both methods. Reports will be submitted to the subcommittee for review. A collaborative study protocol is anticipated soon.

The General Referee (GR) for Radioactivity has retired but did not resign as the GR. There continues to be considerable interest in this field of study including a U.S. Food and Drug Administration announcement of cooperative agreements to expand these capabilities. Stakeholders include members of federal agencies, state agencies, and the Food Emergency Response Network. The Committee will seek to prioritize methods for study under this topic.

The GR for Metals and Other Elements suggested that the priority in this area is the integration of activities involving 5 analytical techniques of flame atomic absorption spectrometry (FAAS), inductively coupled plasma-atomic emission spectrometry (ICP-AES), inductively coupled plasma-mass spectrometry (ICP-MS), instrumental neutron activation analysis (INAA), and radiochemical separation neutron activation analysis (RNAA). The goal is to coordinate and integrate the 3 atomic spectrometric and 2 neutron activation topics to yield several integrated, comprehensive, multi-element analytical methods for *Elements in Foods, Feeds, Food Supplements, and Biological Materials Utilizing FAAS, ICP-AES, ICP-MS, INAA, and RNAA*. These 5 interrelated topics are believed to be of the greatest interest to the analytical/agricultural communities and are given the highest priority within this GR topic. The Committee will solicit comment from stakeholders in an effort to identify the best method of highest priority to scientists in this topic area.

David Soderberg's term as a GR has expired. He has been appointed a member of the Methods Committee on Residues and Related Topics. While we will miss his excellent GR summaries of the work in pesticide residue analysis, he will continue to contribute to the work of the Committee. In his place, Jon Wong of FDA, Center for Food Safety and Applied Nutrition (CFSAN), has been appointed as GR for the topic, "Pesticides and Other Chemical Contaminants." Wong brings extensive experience in pesticide analysis to the job.

General Referee Reports

Pesticides and Other Chemical Contaminants

General Referee David Soderberg, U.S. Environmental Protection Agency, OPP, HED, RRB3, Rm 821D, 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, Qinhuangdao, People's Republic of China, Tel/Fax: 86-335-341-7119, E-mail: gfpang@heinfo.net. Adopted Final Action in 2003 and appears in the 18th Edition of *Official Methods of Analysis*. Discontinue 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, Eastern Regional Research Center, Food Safety Research Unit, 600 E. Mermaid Ln, Wyndmoor, PA 19038, Tel: 215-233-6433, Fax: 215-233-6642, E-mail: Steven.Lehotay@ars.usda.gov. Adopted Final Action in 2005 and appears in the 18th Edition of *Official Methods of Analysis*. The method is in regular use in Japan. Recommend continue topic until the method has been available through *Official Methods of Analysis* at least 1 more year.

(3) **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. A single automated approach for all food matrixes requiring PCDD/F, PCB, and

PBDE measurement is now being tested. The results were promising with corn oil, fish oil, butter, fish fillet, and carrots. Continue topic.

(4) **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 analysis of triazines in foods using GC or LC/MS techniques. Several immunoassay methods have also been developed. Continue topic.

(5) **Pesticides in Foods Using Acetonitrile Extraction and Partitioning with Magnesium Sulfate.**—Study Director Steven J. Lehotay. A collaborative study involving 13 laboratories has been completed. The collaborative study report will be evaluated by the Committee this year for acceptance by AOAC INTERNATIONAL.

(6) **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: frank.schenck@fda.hhs.gov. The Topic Advisor has studied the effect of the solvents used during SPE cleanup on the stability of base sensitive organochlorine pesticides. His work is improving the recoveries for these classes of compounds. Continue topic.

(7) **Multiresidue Methods for Pesticides in Foods by GC/MS and LC/MS/MS.**—Topic Advisor Guo-Fang Pang. This Topic Advisor has proposed a collaborative study. He expects publication of all 4 of his methods this year. He is temporarily distracted by other studies, but intends to return to doing a collaborative study when time is available and if the community supports that study. Continue topic.

(8) **Ultra-Trace Method for Pesticides in Bottled Soft Drinks.**—Study Director Paul Milne, Pepsi Cola Co., Valhalla, NY, 10595, Tel: 914-7424743, Fax: 914-749-3323, E-mail: pmilne@pepsi.com. An expert panel has worked with the Study Director to develop methods that best meet the needs of the analysis. Methods have been developed and SLVs have been completed. The Study Director, together with the Expert Review Panel and the General Referee, plan to submit a protocol for multilaboratory validation. Continue topic.

(9) **Post Extraction GPC Cleanup for Pesticide Residues.**—Topic Advisor Michael Halvorson, Gilson, Inc., 3000 Parmenter St, PO Box 620027, Middleton, WI 53562-0027, Tel: 608-828-3226, Fax: 608-831-4451, E-mail: mhalvorson@gilson.com. Extensive work on the GPC cleanup of fatty food matrixes is reported. Continue topic.

Radioactivity

General Referee Edmond J. Baratta (retired), formerly of U.S. Food and Drug Administration, 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 GR. 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

General Referee 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: lars.jorhem@slv.se. Adopted Final Action in 2005. Continue monitoring any reports from users of the Official Method.

(2) **999.11** *Determination of Lead, Cadmium, Copper, Iron, and Zinc in Foods, Atomic Absorption Spectrophotometry after Dry Ashing*.—Method Advisor Lars Jorhem. Adopted Final Action in 2005. 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 FAAS developments 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 spectrometry and inductively coupled plasma-mass spectrometry. 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 (DLs) and isobaric and polyatomic interference corrections required for potential analyte elements of interest. Integrate activities with concurrent studies on atomic absorption spectrometry and inductively coupled plasma-atomic emission spectrometry. Continue study.

(6) *Lead in Wines*.—Study Director Alan L. Reisig, Alcohol and Tobacco Laboratory, 6000 Ammendale Rd, Beltsville, 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. With assistance of the GR, make a last concerted effort to settle evaluation of the collaborative study, subject to funding issues, and bring this study to a conclusion. Continue study.

(7) *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 the techniques of INAA and RNAA. Continue with preparation of a manuscript reviewing neutron activation analytical methods, setting the stage for proposed INAA and RNAA methodologies. Integrate activities within this study with studies on atomic absorption spectrometry, ICP-AES, and ICP-MS. Continue study.

(8) *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.