

METHODS COMMITTEE REPORTS

Committee on Residues and Related Topics

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Committee Actions

Over the past year the committee recommended for adoption as First Action the following method: Determination of Pesticide Residues in Nonfatty Foods by SFE and GC/MS. The recommendation was approved during a committee conference call on May 9, 2002. During the Annual Meeting, the Committee proposed that 2 refereeships be combined. The new general refereeship will replace Multiresidue Methods and Single Class Multiresidue Methods with the single general refereeship: Pesticides and Other Chemical Contaminants. This title will parallel the other titles of general refereeships since it describes the analyte for which residue methods are applicable. All topics currently under "Multiresidue Methods" and "Single Class Multiresidue Methods" will be combined under the new refereeship. David Soderberg will be the General Referee. Charles Parfitt, GR for Multiresidue Methods, has resigned. A new topic has been proposed under the new general refereeship. Title of the new topic is "Rapid, Multiclass Methods for Pesticide Residues in Foods." Steven Lehotay will be the new Topic Advisor. Several other topic names were combined or changed and are listed in the discussion under each general refereeship.

Pesticides and Other Chemical Contaminants (formerly Multiresidue Methods), Charles Parfitt

(1) **998.01 Synthetic Pyrethroids:** Study Director Guo-Fang Pang, Qinhuangdao Entry-Exit and Quarantine Bureau, No. 39 Haibin Rd, P.C. 066002, Qinhuangdao, Peoples Republic of China, Tel-fax: 86-335-341-7119, E-mail: panggfciq@pang.com.cn. Method is recommended for Final Action. Continue study. Monitor performance of method.

(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 E. Mermaid

Ln, Wyndmoor, PA 19038, Tel: +1-215-233-6433, Fax: +1-215-233-6642, E-mail: slehotay@arserrc.gov. Method was adopted as First Action. Continue study. Monitor performance of method.

(3) *Miniaturized Methods*: Topic Advisor Frank Schenck, U.S. Food and Drug Administration, Southeastern Regional Laboratory, 60 Eighth St, NE, Atlanta, GA 30309, Tel: +1-404-253-1200, Fax: +1-404-253-1208, E-mail: fschenck@ora.fda.gov. Encourage development of a collaborative study protocol for SPE method. Continue study.

(4) *Comprehensive Multiresidue Methodology*: Topic Advisor S. Mark Lee, California Department of Food and Agriculture, Division of Inspection Services, 3292 Meadowview Rd, Sacramento, CA 95832, Tel: +1-916-262-1434, Fax: +1-916-262-1572, E-mail: mlee@cdfa.ca.gov. Since there has been no activity in this topic for the past 2 years, discontinue study.

Formerly Single Class Multiresidue Methods

General Referee David Soderberg, EPA-OPP, 403 W. Side Dr #102, Gaithersburg, MD 20828, Tel: +1-703-308-4137, Fax: +1-703-305-5147, E-mail: soderberg.david@epamail.epa.gov

(1) *Chlorinated Dioxins*: Topic Advisor Douglas Hayward, U.S. Food and Drug Administration, HFS-336, 5100 Paint Branch Pkwy, College Park, MD 20740-3835, Tel: +1-301-436-1654, Fax: +1-301-436-2632, E-mail: douglas.hayward@cfsan.fda.gov. Two FDA regional laboratories are now successfully analyzing food for dioxins using ion trap GC/MS. This success sets the stage for a possible AOAC interlaboratory study of the method. Continue topic.

(2) **2000.05** *Determination of Glyphosate and (Aminomethyl) Phosphonic Acid in Crops by Capillary Gas Chromatography with Selective Detection*: Study Director Phil Alferness, Biomarin Pharmaceuticals, Inc., Ste 210, Analytical Biochemistry Department, 371 Bel Marin Keys Blvd, Novato, CA 94949, Tel: +1-415-506-6121, Fax: +1-415-382-0113, E-mail: palferness@biomarinpharm.com. This method was adopted as First Action in January 2000. The collaborative study report was published in the May/June (2001) issue of *J. AOAC Int.* The Study Director has received no adverse comments about the method in the interim and recommends that this method be adopted Final Action. The General Referee and Committee concurs that method should be adopted Final Action. Continue topic and monitor method performance.

(3) *Triazine Residues in Raw Agricultural Products*. Change name to: *Determination of Residues of Triazines and Their Chloro-Metabolites in Raw Agricultural Commodities*: Study Director Vacant. All residues containing the chloro-triazine center have been expected to contribute to the same toxic endpoint for these compounds in food, and residues of the metabolites, especially diaminochlorotriazine, may predominate over residues of the parent triazines. Current multiresidue methods published by FDA in Volume 1 of the *Pesticide Analytical Manual* are capable of determining the parent triazines, but not of simultaneously also measuring all chloro-metabolites of these triazines. A method published by

J.R. Pardue, *J. AOAC Int.* **78**, 856–862 (1995), and used in a brief survey of triazine residues detects the mono-desalkylmetabolites of simazine and atrazine, but required a second method to determine diaminochlorotriazine. A collaborated method capable of measuring all of these residues together seems highly desirable and the topic should be continued with this clarification. Appoint Topic Advisor and continue topic with changed name.

(4) *Dioxins by GC/MS*: Vacant. AOAC INTERNATIONAL has advertised for a Study Director for this topic and at least one tentative response has been received. The possibility of a collaborative study is currently being discussed. Pending further resolution this topic should be continued. Continue topic.

(5) *Rapid Multiclass Methods for Pesticide Residues in Foods*: Topic Advisor Steve Lehotay, U.S. Department of Agriculture, Agricultural Research Service, Regional Research Center, 600 E. Mermaid Ln, Wyndmoor, PA 19038, Tel: +1-215-233-6433, Fax: +1-215-233-6642, E-mail: slehotay@arserrc.gov. New topic. A collaborative study is under discussion.

Radioactivity, Edmond J. Baratta

(1) *Iodine-131 and Cesium 137 in Milk and Foods*: Search for and appoint a new Study Director and Topic Advisor. Draw up a protocol for a collaborative study using the newer type Germanium detectors with computer assisted software. Continue topic.

(2) *Strontium-90*: Study Director Marina Silverstone, Washington Dept. of Health, Div. of Labs Radiation, 1610 NE 150th St, Seattle, Washington 98155-9701, Tel: +1-206-361-2894, Fax: +1-206-361-2899, E-mail: marina.silverstone@goh.wa.gov. "Rapid Method for Analyzing Strontium-90 in Water" is being developed. 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*: Study Director Milan Ihnat, Pacific Agri-Food Research Centre-Summerland, Agriculture and Agri-Food Canada, Summerland, British Columbia V0H 1Z0, Canada, Tel: +1-250-494-6411, Fax: +1-250-494-0755, E-mail: ihnatm@agr.gc.ca. Submit to *J. AOAC Int.* a report "Flame Atomic Absorption Spectrometric Methodologies for Food Analysis—A Review," comprehensively summarizing the status of current flame atomic absorption spectrometric methods used by AOAC INTERNATIONAL and other agencies for major, minor, and trace elemental levels in foods and agricultural products. Complete development of a unified, comprehensive, multielement flame atomic absorption scheme of analysis of foods for a range of major, minor, and trace elements and submit for collaborative study approval and publication in *J. AOAC Int.* Complete a report for submission to *J. AOAC Int.* on the development and application of a highly reliable flame AAS method for

multielement determinations in biological materials. Continue study.

(2) *Elements in Foods, Feeds, and Biological Materials by Inductively Coupled Plasma Atomic Emission Spectrometry*: Adopt new topic and appoint Study Directors.

(3) *Elements in Foods, Feeds, and Biological Materials by Inductively Coupled Plasma Mass Spectrometry*: Adopt new topic and appoint Study Directors.

(4) *LGraphite Furnace Atomic Absorption Spectrometry*: Topic Advisor Robert W. Dabeka, Health Canada, Food Research Division 2203D, Health Protection Branch, Ottawa, Ontario K1A 0L2, Canada, Tel: +1-613-957-0951, Fax: +1-613-941-4775, E-mail: Bob_Dabeka@hc-sc.gc.ca. Bring under this heading and this Advisor the topic Graphite Furnace Atomic Absorption Spectrometry and the topic Lead in Foods. Continue with research/development into methods for lead in foods utilizing determinative techniques of inductively coupled plasma mass spectrometry and graphite furnace atomic absorption spectrometric techniques and development of preconcentration techniques as may be required. Discontinue topic under this name. Begin new topic (Total Mercury in Foods by Cold Vapor AAS) with Robert Dabeka as Topic Advisor.

(5) *Graphite Furnace Atomic Absorption Spectrometric Determination of Chromium in Foods*: Study Director Nancy J. Miller-Ihli, U.S. Department of Agriculture, Nutrient Composition Laboratory, Beltsville Human Nutrition Research Center, Bldg 161, BARC-East, Beltsville, MD 20705, Tel: +1-301-504-8252, Fax: +1-301-504-8314, 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 (*JAOAC* **75**, 354–359 [1992]). Continue study.

(6) **997.15** *Lead in Sugars and Syrups*: Study Director Nancy J. Miller-Ihli. Continue topic.

(7) **999.17** *Lead and Cadmium Extracted from Ceramic Foodware*: Study Director Susan C. Hight, U.S. Food and Drug Administration, Center for Food Safety and Applied Nutrition, Elemental Research Branch, HFS-338, 5100 Paint Branch Pkwy, College Park, MD 20740-3835, Tel: +1-301-436-1652, Fax: +1-301-436-2632, E-mail: susan.hight@cfsan.fda.gov. First Action, 1999. Continue monitoring any reports from users. Continue study.

(8) *Lead in Calcium Supplements*: Study Director Paul H. Siitonen, U.S. Food and Drug Administration, National Center for Toxicological Research, Division of Chemistry, 3900 NCTR Dr, HFT-230, Jefferson, AR 72079-9502, Tel: +1-870-543-7656, Fax: +1-870-543-7686, E-mail: psitonen@nctr.fda.gov. Complete evaluation of the precollaborative trial based on the proposed collaborative

study method, "Determination of Calcium by Inductively Coupled Plasma Atomic Emission Spectrometry and Lead by Graphite Furnace Atomic Absorption Spectrophotometry in Ca Supplements after Microwave Dissolution or Dry-Ash Digestion: Method Trial." Revise the collaborative study protocol to include the microwave dissolution procedure and prepare a protocol for collaborative study for approval by the General Referee, Committee Statistical Advisor, and Committee on Residues. Continue study.

(9) *Lead in Wines*: Study Director Alan L. Reising, BATF Laboratory, 1401 Research Blvd, Rockville, MD 20850, Tel: +1-301-762-9800, Fax: +1-301-413-9463, E-mail: Alreising@atfhq.atf.treas.gov. Complete revision of the collaborative study, "Lead in Beverage Alcohol, Graphite Furnace Atomic Absorption Spectrometric Method," following additional statistical analysis which were performed and resubmit modified method. Continue study.

(10) *Neutron Activation Analysis*: Study Director William C. Cunningham, U.S. Food and Drug Administration, Center for Food Safety and Applied Nutrition, Elemental Research Branch [mailing address: National Institute of Standards and Technology, Mailstop 8395, Gaithersburg, MD 20899-8395], Tel: +1-301-975-6271, Fax: +1-301-208-9297, E-mail: william.cunningham@nist.gov. Accept resignation of Study Director Cunningham with thanks for past activities on this topic. Methods based on neutron activation analysis may be looked upon as more specialized than most other methods due to the requirement, for typical applications, of a nuclear reactor as a source of neutrons. Instrumental neutron activation analysis (INAA) and neutron activation analysis with radiochemical separation (RNAA) are 2 common variants. Other subdivisions include thermal, epithermal, and prompt gamma versions of INAA, photon-induced activation analysis and proton activation analysis. In addition, nonreactor sources can be used to provide excitation. The excellent multielement performance of activation-based methods and moderately wide use makes it desirable to obtain at least generic versions of neutron activation methods for foods into official status. Continue topic; search for new Study Director.

(11) *Organometallics in Fish*: Continue Official First Action status of Mercury (Methyl) in Seafood, Liquid Chromatographic–Atomic Absorption Spectrophotometric Method, **990.04**. Discontinue topic.

(12) **999.10** and **999.11** *Metals in Foods by Atomic Absorption Spectrometry*: Method Advisor Lars Jorhem, National Food Administration, Box 622, S-751 26 Uppsala, Sweden, Tel: +011-46 18 17 55 00, Fax: +011-46 18 10 58 48, E-mail: lajo@slv.se. Continue monitoring any reports from users of the Official Methods.

(13) *Total Mercury in Foods by Cold Vapor Atomic Absorption Spectrometry*: Adopt new topic and invite Robert W. Dabeka to be Study Director.