

## METHODS COMMITTEE REPORTS

## Committee on Commodity Foods and Commodity Products

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## Committee Actions

The method committee membership has been very steady this year. The terms of all existing members have been renewed and no new members have been added. The Committee Chair position will be taken over by Joanna Lynch. Darryl Sullivan will be the incoming chair of the Official Methods Board and will remain on the Method Committee as a member.

The committee did not approve any methods for First Action during the past year. The committee currently has 2 study protocols in review, one collaborative study in progress, and

3 collaborative study manuscripts in review. Several new study protocols are expected to be received in the near future.

The committee currently has 2 vacant General Referee positions that need to be filled: Meat and Meat Products, and Fruit and Fruit Products. The topics Egg and Egg Products, and Nuts and Nuts Products were discontinued.

### *Cereals and Cereal Products, Stephen R. Delwiche*

(1) *Total Folate in Cereal Products, Microbiological Assay Using Trienzyme Extraction*: Study Directors Jonathan DeVries, Medallion Laboratories, General Mills, Minneapolis, MN 55427, Tel: +1-763-764-2774, Fax: +1-763-764-7487, E-mail: jon.devries@genmills.com; Jeanne I. Rader, U.S. Food and Drug Administration (FDA), Center for Food Safety and Applied Nutrition, Washington, DC 20204, Tel: +1-202-205-5375, Fax: +1-202-205-4594, E-mail: jir@cfsan.fda.gov; Carol Hudson, U.S. Department of Agriculture (USDA), WRRRC, 800 Buchanan St, Berkeley, CA 94710, Tel: +1-510-559-5660, Fax: 510-559-3777, E-mail: cahudson@pw.usda.gov; Pamela Keagy, USDA-WRRRC 800 Buchanan St, Berkeley, CA 94710, Tel: +1-510-559-5664, Fax: +1-510-559-5626, E-mail: pkeagy@pw.usda.gov. Collaborative study report was submitted in Fall 2000 and reviewed by General Referee and Statistician in Spring 2001. Minor revisions needed, particularly clarification of outlier removal procedure. Continue study.

(2) *Measurement of Alpha-Amylase in Cereal Products and Fungal and Bacterial Enzyme Preparations*: Study Director Barry McCleary, Megazyme International Ireland, Ltd., Bray Business Park, Southern Cross Rd, Bray, County Wicklow, Ireland, Tel: +353-1-286-1220, Fax: +353-1-286-1264, E-mail: info@megazyme.com. The collaborative study, involving 15 international laboratories, was performed, and the statistical analyses have been completed. The Study Director planned to submit the collaborative study report in Summer 2001. Continue study.

(3) **991.19** *MOD-Gliadin as a Measure of Gluten in Foods, Color, Monoclonal Antibody Enzyme Test*: Study Director formerly Thomas Grace (resigned), presently none. The General Referee contacted former Study Director in June 2001 and learned that he is no longer affiliated with the company that was active in this method. Work on this study has ceased. Adopted Final Action in 2000. Discontinue study.

(4) *Assessment of Starch Pasting Quality by Rapid Viscosity Analysis*: Study Director Mark Bason, Newport Scientific, 1/2 Apollo St, Warriewood, NSW 2102, Australia, Tel: +61-2-9979 6992, Fax: +61-2-9979 6993, E-mail mark@newport.com.au. Method is designed to characterize starch pastes cooked under defined shear and temperature regimens. The protocol was originally assessed and revised in May and June 2000. It was resubmitted to AOAC INTERNATIONAL, which replied in November, requesting further substantial revisions. Further revision was delayed by staff changes and overseas postings at Newport but is now underway. Pending approval of the protocol, samples and instructions will be distributed to 16 participating laboratories in late 2001. Continue study.

(5) *Phytic Acid, LC Method*: Study Director Barbara F. Harland, Howard University, College of Pharmacy, Nursing and Allied Health Sciences, Department of Nutritional Sciences, Annex I, Rm 337, 6th and Bryant Sts NW, Washington, DC 20059, Tel: +1-202-806-5656, Fax: +1-202-806-9233, E-mail: bharland@fac.howard.edu. The Study Director is interested in continuing this study despite equipment malfunctions in her laboratory. Continue study.

(6) *Crude Fat in Cereal Foods by Supercritical Fluid Extraction*: Study Director Cynthia Kradjel, Integrated Tech Solutions, 32 Halstead Ave, Port Chester, NY 10573-2706, Tel: +1-914-934-0696, Fax: +1-914-934-8843, E-mail: kradjel@branluebbe.com. Study Director indicates that planning for this study is stalled. Cooperation from other scientists interested in drafting the protocol is necessary but not forthcoming. Discontinue study.

(7) *Determination of Fat Content in Baked Cereal Products by Supercritical Fluid Extraction*: Study Director Les J.D. Meyer, Leco Corp., 3000 Lakeview Ave, St. Joseph, MI 49085, Tel: +1-616-983-5531, Fax: +1-616-982-0631, E-mail: les\_myer@leco.com. Study Director is no longer employed by Leco. Other scientists at the company do not wish to pursue this method at this time. Discontinue study.

(8) *Protein, NIR Method for Wheat Flour*: Topic discontinued per recommendations made in 2000.

(9) *Fat Acidity*: No Study Director for this method. Discontinue study.

#### **Dairy Chemistry, Robert L. Bradley, GR**

(1) *Trans-Retinyl Palmitate (Vitamin A) in Fluid Milks by LC*: Study Director Douglas Hite, Technical Services, Tennessee Department of Agriculture, PO Box 40627, Melrose Station, Nashville, TN 37220, Tel: +1-615-837-5317, Fax: +1-615-837-5516, E-mail: dhite@mail.state.tn.us, has finished preliminary trials and the collaborative study protocol has been approved. Continue study.

(2) **2000.18** *Fat in Raw and Processed Whole Milk by the Gerber Method*: Study Director Martin Mitchell, Certified Laboratories, Inc., 200 Express St, Plainview, NY 11803, Tel: +1-516-576-1400, Fax: +1-516-576-1410, E-mail: corp@800certlab.com. The method was approved First Action last year and published in the J. AOAC Int. Continue study.

(3) *Total Nitrogen (Protein) Content of Cheese*: Study Directors D.M. Barbano, Department of Food Science, Cornell University, 118 Stocking Hall, Ithaca, NY 14853, Tel: +1-607-255-5482, Fax: +1-607-254-4868, E-mail: dmb37@cornell.edu; and Joanna M. Lynch, Department of Food Science, Cornell University, 207 Stocking Hall, Ithaca, NY 14853, Tel: +1-607-255-7903, Fax: +1-607-254-4868, E-mail: JL72@cornell.edu, have complete the collaborative study. The finished report is in the approval process. A poster describing these results was scheduled for the 2001 Annual Meeting. Continue study.

(4) **972.16** *Infrared Spectroscopic Methods for the Determination of Fat, Lactose, Protein, and Solids in Milk*: Study Directors D.M. Barbano (*see 3*) and J.M. Lynch (*see 3*) report continued effort in this topic. Following completion of the de-

velopmental program, a collaborative study proposal will be drafted and submitted. Continue study.

(5) **991.24** *Alkaline Phosphatase Activity in Dairy Foods*: Study Director and Topic Advisor R.M. Rocco, Biometrics, Inc., PO Box 62379, Sunnyvale, CA 94088, Tel: +1-650-943-4515, is preparing an expansion of the method. This method was erroneously discontinued last year. Reactivate topic, alter heading to show that it is an IDF-ISO-AOAC Method, and continue study.

(6) **998.05** *Non-Casein Nitrogen in Whole Milk, Kjeldahl Method*: Move to Final Action status and approve Study Directors D. M. Barbano (*see 3*) and J.M. Lynch (*see 3*) as Method Advisors.

(7) **998.07** *Casein Nitrogen in Milk, Kjeldahl Method, Indirect Method*: Move to Final Action status and approve Study Directors D.M. Barbano (*see 3*) and J.M. Lynch (*see 3*) as Method Advisors.

(8) **998.06** *Casein Nitrogen in Milk, Kjeldahl Method, Direct Method*: Move to Final Action status and approve Study Directors D.M. Barbano (*see 3*) and J.M. Lynch (*see 3*) as Method Advisors.

(9) **998.04** *Neutral  $\beta$ -Galactosidase (Lactase) Activity in Industrial Enzyme Preparations*: Study Director A. J. Engelen, DMS Food Specialties, Dept. ISL PP 010-0585, PO Box 1, Delft, 2600-MA, The Netherlands, Tel: +31-15-2793317, Fax: +31-15-2792357, E-mail: jos.engelen@dsm-group.com, has retired and a new SD is being sought. Move to Final Action status.

(10) **989.04** *Fat in Raw Milk, Babcock Method*: Method was approved to Final Action in 2000 based on 1997 method revisions and topic was discontinued. Approve Study Directors D.M. Barbano (*see 3*) and J.M. Lynch (*see 3*) as Method Advisors.

(11) **995.18** *Fat in Cream, Babcock Method*: Method was approved to Final Action in 2000 based on 1997 method revisions and topic was discontinued. Approve Study Directors D. M. Barbano (*see 3*) and J.M. Lynch (*see 3*) as Method Advisors.

(12) **970.26** *Sampling of Milk from Bulk Tanks and Other Storage Equipment, Manual Method*: Discontinued per 2000 recommendations.

(13) **905.02** *Fat in Milk, Roese-Gottlieb Method*: Repealed per 2000 recommendations.

(14) **948.12** *Moisture in Cheese, Method II (Rapid Screening Method)*: Repeal.

(15) **972.17** *Phosphatase (Residual) in Milk*: Repeal.

(16) *Vitamins A and D in Milk Powders, Colorimetric and LC Methods*: Study Director Ellen Jan De Vries, Solvay Pharmaceuticals BV, Fideliolaan 41, Amstelveen, 1183-PH, The Netherlands, Tel: +31-20-6411211, Fax: +31-20-6411211, E-mail: ej.devries@wxs.nl. No report received. Discontinue topic.

(17) *Full Spectrum Infrared Analysis of Dairy Foods*: Ap- point David McKenna, Food Technology, 25710 Velan Dr, Valencia, CA 91355, Tel: +1-805-255-7454, Fax: +1-805-259-6258, E-mail: dmckenna@fosnorthamerica.com, as Topic Advisor.

#### **Processed Vegetable Products, Michael Jantschke**

**985.26** *Solids (Total) in Processed Tomato Product, - Microwave Oven Drying Method*: Study Director Henry Chin, National Food Processors Association, 6363 Clark Ave, Dublin CA 94568, Tel: +1-925-551-4234, Fax: +1-925-833-8795, E-mail: hchin@nfpa-food.org. Recommended adoption as Final

Action last year but this was not acted upon. Again, it is recommended for adoption as Final Action.

### Seafoods, John M. Tennyson

(1) **977.13 Histamine in Seafood, Fluorometric Method:** Study Director Patricia L. Rogers, U.S. Food and Drug Administration, HFS-426, 200 C St SW, Washington, DC 20204, Tel: +1-202-205-4385, Fax: +1-202-260-2511, E-mail: progers@cfsan.fda.gov, reports that the method was revised in March 1997. This method was modified Final Action in 1997. Discontinue study.

(2) **996.07 Putrescine in Canned Tuna and Cadaverine in Canned Tuna and Mahi-Mahi, Gas Chromatographic Method:** Study Director Patricia L. Rogers (*see* 1). A modification in column solvents to elute compounds was submitted last year and the modification is pending. This method is being applied to shrimp with a modification of the extraction procedure. A full collaborative study is expected during 2001–2002. Submit collaborative study protocol and continue study.

(3) **996.15 Fish Flesh Content (FFC) in Frozen Coated Fishery Products:** Study Director Jane E. Fox-Dobson, c/o Massachusetts Division of Marine Fisheries, Annisquam River Marine Fisheries Station, 30 Emerson Ave, Gloucester, MA 01930, Tel: +1-978-282-7463, Fax: +1-978-282-7463, E-mail: Jane.E.Fox-Dobson@noaa.gov. Method was approved First Action in 1996 and no comments were received. The Study Director does not recommend Final Action at this time. Continue study.

(4) **999.01 Volatile Bases in Fish, Ammonia Ion Selective Electrode Method:** Study Director P. Christopher Ellis, Rhode Island Department of Health Laboratories, 50 Orms St, Providence, RI 02904, Tel: +1-401-222-5589, Fax: +1-401-222-6985, E-mail: ChrisE@doh.state.ri.us, reports that the method was adopted First Action in 1999. Additional research is underway on the use of the electrode for simple and rapid determination of seafood quality. Continue study.

(5) **Arsenic in Seafood:** Study Director Kaare Julshamn, Institute of Nutrition, Directorate of Fisheries, PO Box 185, Sentrum, N-5804 Bergen, Norway, Tel: +47-55-238290, Fax: +47-55-238095, E-mail: kare.julshamn@nutr.fiskeridir.no, reported that the analytical method entitled "Determination of Arsenic in Seafood by Electrothermal Atomic Absorption Spectrometry after Microwave Digestion" was accepted as an official Nordic Committee on Food Analysis (NMKL) method and published by NMKL in 2000. The AOAC General Referee and Statistical Advisor scientifically and statistically reviewed the NMKL collaborative study results in November 2000. Subsequently, the NMKL collaborative study article was published in *J. AOAC Int.* **83** (2000). Continue study.

(6) **Chemometric Methods:** Topic Advisors Bradley J. Tenge, FDA, Pacific Region, Regional Information Technology Staff, 22201 23rd Dr SE, Bothell, WA 98021, Tel: +1-425-402-3167 ext. 2167, Fax: +1-425-483-4996, E-mail: btenge@ora.fda.gov; and Frederick S. Fry, Jr, FDA, Center for Food Safety and Applied Nutrition, 200 C St SW, Washington, DC 20204, Tel: +1-202-205-4003, Fax: +1-202-205-4620, E-mail: FRF@cfsan.fda.gov, report that work on development of numerical method for the analy-

sis of seafood-related biochemical fingerprint data is static due to staff reassignments. However, chemometric methods are being applied to 3 areas: (1) seafood decomposition, (2) rapid identification of pathogenic bacteria at the species and strain levels using mass spectrometric techniques, and (3) a microbiological spectral library or chemotaxonomic database for bacterial strain identification. Continue study.

(7) **Histamine Determination by Flow Injection Analysis:** Study Director James M. Hungerford, FDA, Seafood Products Research Center, 22201 23rd Dr SE, Bothell, WA 98021, Tel: +1-425-483-4894, Fax: +1-425-483-4996, E-mail: jhungerf@ora.fda.gov, recommends a peer validation study of the flow injection method for histamine, and recruitment of laboratories for a full collaborative study. Continue study.

(8) **Histamine in Tuna, Capillary Electrophoresis:** Topic Advisor Barry Mopper, FDA, New York Regional Laboratory, HFR-NE, 158-15 Liberty Ave, Jamaica, NY 11433, Tel: +1-718-340-7040, Fax: +1-718-662-5439, E-mail: bmopper@ora.fda.gov, reports that no new work was performed with histamine by capillary electrophoresis. However, the Topic Advisor's laboratory has set up a flow injection analyzer for histamine samples. Continue study.

(9) **Moisture, Microwave Method:** Topic Advisor Robert A. Fisher, Virginia Institute of Marine Science, College of William and Mary, Rt. 1208 Greate Rd, Gloucester Point, VA 23062, Tel: +1-804-684-7168, Fax: +1-804-684-7161, E-mail: rfisher@vims.edu, indicates that he has not initiated the collaborative study process. Recommend continuation of the solicitation of new collaborators. Continue study.

(10) **Noninvasive Assessment of Lipid Oxidation in Fish by Near-Infrared Spectroscopy:** No Study Director for this topic. Discontinue study.

(11) **Oxytetracycline in Farm-Raised Fish:** Topic Advisor Elaine A. Bunch, FDA, SPRC, 22201 23rd Dr SE, Bothell, WA 98021, Tel: +1-425-483-4887, Fax: +1-425-483-4996, E-mail: ebunch@ora.fda.gov, states that FDA has issued a program to survey seafood for oxytetracycline residues in 2001. Continue study.

(12) **F-29-Veratox for Histamine:** Study Director Chuck B. Bird, Neogen Corp., 620 Leshner Pl, Lansing, MI 48912, Tel: +1-517-372-9200 ext. 240, Fax: +1-517-372-0108, E-mail: cbird@neogen.com. No comments received. On May 15, 2001, the Study Director submitted a collaborative study manuscript for Method F-29 entitled "Determination of Histamine in Fresh Tuna, Fresh Mahi, and Canned Tuna by a Competitive Direct Enzyme-Linked Immunosorbent Assay" to AOAC for review. Continue study.

(13) **Weight Determination Methods for Quick Frozen Shrimp:** Study Director Jane E. Fox-Dobson (*see* 3). No comments received. A proposed collaborative study was approved by AOAC in October 1998. In 2000, the Study Director reported that the study is on hold awaiting the approval of funding. Continue study.

(14) Methods removed to surplus status per recommendations made in 2000: **954.04 Histamine in Seafood, Biological Method;** **957.07 Histamine in Seafood, Chemical Method;** and **967.14 Identification of Fish Species, Acrylamide Disc Electrophoresis Method.** Discontinue topics.

**Chocolate and Cacao Products, Simon J. Freeman**

(1) *Carbohydrates in Chocolate Products*: Study Directors W. Jeffrey Hurst, Hershey Foods Corp., Technical Center, PO Box 805, Hershey, PA 17033-0805, Tel: +1-717-534-5145, Fax: +1-717-534-6132, E-mail: whurst@hersheys.com; and John C. Robbins, 318 W. Caracas Ave, Hershey, PA 17033, Tel: +1-717-533-9270. General Referee will contact Study Directors to determine if topics should be continued.

(2) *Shell in Cacao Products: Study Directors W. Jeffrey Hurst (see 1) and John C. Robbins (see 1)*. No report received. General Referee will contact Study Directors to determine if topics should be continued.

**Fruits and Fruit Products, Carla Barry**

(1) *Sugars in Fruit Juices, Carbon-13 Ethanol Method*: Study Director Gilles Martin, Eurofins Scientific Laboratories, Rue Pierre Adolph Bobiere, BP 42301, Nantes, 44323 France, Tel: +33-2-5183-2100, Fax: +33-2-51832111, E-mail: gillesmartin@eurofins.com. Evaluate results of the collaborative study and submit report to AOAC for approval.

(2) *Added Invert Sugars, LC/Pulsed Amperometric Detection*: Study Director Nicholas H. Low, University of Saskatchewan, 3E 08 Agriculture Bldg 51, Campus Dr, Saskatoon, SK S7N 5AB, Canada, Tel: +1-306-966-5037, Fax: +1-306-966-8898, E-mail: nicholas.low@usask.ca. Continue study.

(3) *Anthocyanin Pigment Analysis of Fruit Juices*: Study Director Ronald E. Wrolstad, Oregon State University, Department of Food Science and Technology, Wiegand Hall, Corvallis, OR 97331-6602, Tel: +1-541-737-3591, Fax: +1-541-737-1877, E-mail: wrolstar@bcc.orst.edu. Collaborative study proposal for the topic "Determination of Total Monomeric Anthocyanin Pigments by the pH Differential Method" submitted to AOAC in August 2001. Evaluate protocol.

(4) *Fruit Acids*: Study Director Elia Coppola, Ocean Spray Cranberries, Inc., 1 Ocean Spray Dr, Lakeville, MA 02349, Tel: +1-508-946-1000, Fax: +1-508-946-7704. Modify topic to reflect the area of interest, that is, fruit acids by capillary electrophoresis. Continue study.

(5) *Hydrolyzed Inulin Syrup and High Fructose Corn Syrup in Apple Juice, Capillary GC Method*: Study Director Michael McLaughlin, U.S. Food and Drug Administration, HFS-347, 200 C St SW, Washington, DC 20204, Tel: +1-202-205-5180, Fax: +1-202-205-4422, E-mail: mmclaugh@bangate.fda.gov. The first draft of the paper describing the study and its results is under review. Evaluate results and submit for approval.

(6) *Invert Sugar in Apple Juice, Capillary GC Method of Low*: Study Director David Hammond, Reading Scientific Services, Whiteknights University, Lord Zuckerman Research Centre, Reading, R96-6LA, United Kingdom, Tel: +1-44-118-986-8541, Fax: +44-118-986-8932, E-mail: david.a.hammond@rssl.sprint.co. Organize another small ring test of the modified method using the peer-verified approach and submit to AOAC. Continue study.

(7) *Isotope Methods in Fruit Products*: Study Director Dana A. Krueger, Krueger Food Laboratories, Inc., 24 Blackstone St, Cambridge, MA 02139, Tel: +1-617-876-9118, Fax: +1-617-876-0572, E-mail: dkrueger@kfl.com. Due to continued interest in this topic, continue study.

(8) *Major Metals in Orange Juice*: Study Director Allan R. Brause, Analytical Chemical Services of Columbia, Inc., 9110 Red Branch Rd, Ste K, Columbia, MD 21045-2011, Tel: +1-410-730-7782, Fax: +1-410-730-8340, E-mail: accsinc@clark.net. Continue study.

(9) *Separation of Sugars in Orange Juice, LC Method*: Study Director Allan R. Brause (see 8). Continue study.

(10) **999.05** *Naringin and Neophesperidin in Orange Juice for Detection of Adulteration with Grapefruit Juice*: Study Director Wilbur Widmer, State of Florida Department of Citrus, 700 Experiment Station Rd, Lake Alfred, FL 33050, Tel: +1-813-956-1151. Method adopted First Action in 1999. Continue study.

(11) *Polyphenolic Acid Analysis of Fruit Juices*: Study Director position vacant. Ask Technical Committee for Juice and Juice Products (TCJJP) to recommend a new Study Director.

(12) *Pulpwash*: Study Director Paul Cancalon, Florida Department of Citrus, 700 Experiment Station Rd, Lake Alfred, FL 33850-2299, Tel: +1-863-325-8056, Fax: +1-863-295-5920, E-mail: pcancalo@citrus.state.fl.us. Method should be studied through the peer review process. Continue study.

(13) **995.17** *Beet Sugar in Fruit Juices by Site-Specific Natural Isotope Fractionation - Nuclear Magnetic Resonance (SNIF-NMR)*: Study Director Gilles Martin (see 1). Continue study.

(14) *Determination of Limonin in Citrus Juices and Concentrates*: Study Director Jonathan Hache, Laboratory Services Division, Canadian Food Inspection Agency, 960 Carling Ave, Ottawa, Ontario, K1A 0C6, Canada, Tel: +1-613-759-1266, Fax: +1-613-759-1260, E-mail: hachej@em.agr.ca. Protocol was submitted but was not approved by the committee in its original form. We believe the collaborative study was conducted without committee approval of the protocol. Submit collaborative study results to AOAC for evaluation.

(15) **940.26** *Ash of Fruits and Fruit Products*: This method cross-references methods **900.02A** and **900.02B**, which have surplus status. Since method **940.26** will be included in the list of methods for the CODEX juice standard, recommend that Methods **900.02A** and **900.02B** be changed from surplus to active status.

(16) *Capillary Electrophoresis of Organic Acids in Juices*: Appoint Steve Kupina, Canandaigua Wine Co., 12667 Rd 24, Madera, CA 93639, Tel: +1-209-673-7071, Fax: +1-209-673-0334, E-mail: steve\_kupina@winea.com, as Study Director.

**Meat and Meat Products, James G. Hess**

General Referee reports no activity in this topic. The General Referee's term has expired and he does not wish to continue. Methods Committee will seek new General Referee because of continued interest in this topic.

**Egg and Egg Products**

Topic discontinued.

**Nuts and Nut Products**

Topic discontinued.