

## METHODS COMMITTEE REPORTS

## Committee on Commodity Foods and Commodity Products

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### Committee Actions

The committee completed the review of 3 collaborative study reports and is in the final stages of reviewing a fourth. Two methods were adopted Official First Action: **2004.01** *Detection of  $^{13}C/^{12}C$  Ratio of Ethanol Derived from Fruit Juices and Maple Syrup by IRMS* and **2004.05** *Total Folate in Cereal Products, Microbiological Assay Using Trienzyme Extraction*. Two methods were approved as Official Final

Action: **2001.14** *Total Nitrogen (Protein) Content of Cheese by Kjeldahl Nitrogen Analysis* and **2000.18** *Fat Content of Raw and Pasteurized Whole Milk—Gerber Method by Weight*. The General Referee for the topic of Processed Vegetable Products resigned and the committee voted to discontinue the topic.

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

(1) **2004.05** *Total Folate in Cereal Products, Microbiological Assay Using Trienzyme Extraction*: Study Director Jonathan DeVries, Medallion Laboratories/General Mills, 9000 Plymouth Ave N., Minneapolis, MN 55427-3870, Tel: 763-764-2774, Fax: 763-764-7487, E-mail: jon.devries@genmills.com. The method was approved Official First Action as Method **2004.05** in May 2004. Method will be eligible for Final Action in 2006. Continue study.

(2) **2002.01** *Measurement of Alpha-Amylase Activity in White Wheat Flour, Milled Malt, and Microbial Enzyme Preparations Using the Ceralpha Assay*: 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 method was successfully subjected to interlaboratory evaluation and approved Official First Action in 2002. The method has been used for 10 years and is the standard procedure of the UK Flour Milling and Baking Association. Recommend method be approved Final Action.

(3) *Assessment of Starch Pasting Quality by Rapid Viscosity Analysis*: Study Director Mark Bason, Newport Scientific, Unit 1, 2 Apollo St, Warriewood, NSW, 2102, Australia, Tel: 61-2-9979 6992, Fax: 61-2-9979 6993, E-mail: mark@newport.com.au. The method to be assessed is a viscometric analysis of various types of starch pastes cooked under defined shear and temperature regimes. A protocol was evaluated by the committee in 2000 but not approved. Substantial revision of the protocol was requested but has not been acted upon. Recommend that topic be discontinued.

(4) **997.06** *Protein (Crude) in Wheat, Whole Grain Analysis, Near-Infrared Spectroscopic Method*: Study Director Stephen R. Delwiche, U.S. Department of Agriculture, ARS, Henry A. Wallace, Beltsville Agricultural Research Center, Building 303 BARC-East, 10300 Baltimore Ave, Beltsville, MD 20705-2350, Tel: 301-504-8450, Fax: 301-504-9466, E-mail: delwiche@ba.ars.usda.gov. The Study Director realized that the method had never been recommended for Final Action, but should be. The method is in use and no problems have been reported. Recommend Final Action approval.

*Chocolate and Cacao Products, Simon J. Freeman*

(1) *Shell in Cocoa Products*: Topic Advisor W. Jeffrey Hurst, Hershey Foods Co., Hershey Foods Technical Center, PO Box 805, Hershey, PA 17033, Tel: 717-534-5145, Fax: 717-534-6132, E-mail: whurst@hersheys.com. The primary published methods for determination of shell content in cocoa are still related to the determination of the 2 fatty acid tryptamides (lignoceric acid tryptamide [LAT] and behenic acid tryptamide [BAT]), although other approaches such as carbohydrate content or the use of near infrared spectroscopy have been proposed. Continue study.

(2) *Carbohydrates in Chocolate Products*: Topic Advisor W. Jeffrey Hurst (*see* 1). Method **930.13** (Fructose, Glucose, Sucrose, Maltose, and Lactose in Chocolate Products) continues to be widely used by the confectionery industry. However, many laboratories are now using HPAEC with pulsed amperometric detection (PAD) and a gold electrode as an alternative to the current method, although this has not yet been peer-verified. Such a study would be initiated once funding has been obtained. Continue study.

*Dairy Chemistry, Robert L. Bradley, Jr*

No report received. The chair, however, is aware of the following:

(1) **2001.14** *Total Nitrogen (Protein) Content of Cheese by Kjeldahl Nitrogen Analysis*: Study Directors Joanna M. Lynch and David M. Barbano, Cornell University, 207 Stocking Hall, Ithaca, NY 14853, Tel: 607-273-6307, Fax: 607-254-4868, E-mail: JL72@cornell.edu, dmb37@cornell.edu. The method was approved Final Action in January 2004. Discontinue topic and appoint Study Directors Lynch and Barbano as Method Advisors.

(2) **972.16** *Infrared Spectroscopic Methods for the Determination of Milk Composition*: Topic Advisors Barbano (*see* 1) and Lynch (*see* 1) are planning to write a manuscript and revision of the precalibration quality assurance portions of Method **972.16**. During the past year, studies were conducted to compare the performance of modified liquid calibration samples and traditional commercial calibration samples. Compared to the traditional samples, the modified samples resulted in improved method performance and a more stable calibration. Manuscripts describing these studies are being prepared. An AOAC collaborative study of different calibration methods (e.g., MLR, fixed intercorrection factors, etc.) is being planned. Calibration methods in AOAC Method **972.16** have never been collaboratively studied. Method should stay at First Action. Continue study.

(3) *Lactose in Milk*: Topic Advisors Barbano (*see* 1) and Lynch (*see* 1) report work is continuing on a nonproprietary method for the enzymatic determination of the anhydrous lactose content of milk. The sample preparation procedure has been revised to eliminate the need for adjustment of pH of the filtrate from the Carrez solutions using NaOH. This has improved the filtration (faster and filtrates are more clear) and repeatability of the method. A collaborative study is planned for the fall/winter of 2004. Continue study.

(4) **2000.18** *Fat Content of Raw and Pasteurized Whole Milk, Gerber Method by Weight*: Study Director Martin Mitchell, Certified Laboratories, Inc., 200 Express St, Plainview, NY 11803, Tel: 516-576-1400, Fax: 516-576-1410, E-mail: corp@800certlab.com. The method was approved Final Action in January 2004. Discontinue topic.

(5) **2002.06** *Trans-Retinyl Palmitate (Vitamin A) in Fluid Milks by HPLC*: Study Director Douglas Hite, Tennessee Department of Agriculture, PO Box 40627, Melrose Station, Nashville, TN 37220, Tel: 615-837-5317, Fax: 615-837-5516, E-mail: dhite@mail.state.tn.us. The Study Director has identified that the regenerant solution described in the method is no longer available from the supplier. The method should be modified to reflect in-house preparation: 1 L LC grade hexane, 10 mL glacial acetic acid, and 8 mL 2,2-dimethoxy propane. The method is in use and no problems have been reported. Recommend the method be approved Final Action and be edited to include in-house preparation of the regeneration solution.

(6) **992.22** *Iodine (as Iodide) in Pasteurized Liquid Milk and Skim Milk Powder, Liquid Chromatographic Method*: There is currently no Study Director for this method. In the absence of Study Director, the General Referee recommended in 2002 that it be moved from First to Final Action. A member of the Official Methods Board (OMB), however, reported experiencing problems with the method and asked for more information. The chair made contact with one of the original collaborators, who said that the method worked well but was rarely used because alternative methods are available. The chair also contacted the original Study Director (David Sertl) who then corresponded with the OMB member's laboratory. Recommend that the method be approved Final Action and topic be discontinued.

(7) **998.04** *Neutral -Galactosidase Activity in Industrial Enzyme Preparations*: In the absence of a Study Director, the General Referee recommended in 2001 that the method be moved from First to Final Action. However, the OMB identified significant issues with the description of the evaluation of the ONPG reagent and removed consideration for Final Action pending clarification. P. Randsdorp was appointed as the new Study Director in 2002 but unfortunately he passed away in 2003. The chair has been in contact with the organization (DSM Food Specialties) where Randsdorp was employed to see if an individual could be identified who could address the method description. Although there was interest, it is not clear if anyone will specifically be identified to make the necessary modifications. The laboratory at DSM indicated that the ONPG evaluation itself might not be necessary anymore because of improvements in the quality of commercially available ONPG. Recommend the method be repealed.

*Fruit and Fruit Products, David Hammond*

(1) **FE20** *Determination of Total Monomeric Anthocyanin Pigment Content of Fruit Juices, Beverages, Natural Colorants, and Wines by the pH Differential Method*: Study

Director Jungmin Lee, Oregon State University, Department of Food Science and Technology, Corvallis, Oregon 97331, Tel: 541-737-6490 or 3591, Fax 541-737-1877, E-mail: leeu@onid.orst.edu. The pH differential method is a rapid and simple spectrophotometric method based on the anthocyanin structural transformation that occurs with a change in pH. A protocol was approved in 2002, the collaborative study was conducted in 2003, and a report was submitted. Seven Youden pair materials (representing fruit juices, beverages, natural colorants, and wines) were tested. The RSD<sub>r</sub> ranged from 1.06 to 4.16% and the RSD<sub>R</sub> ranged from 2.69 to 10.12%. HORRAT values were well within the acceptable range (0.30–1.33). These features showed that the method was described well and returned reliable results. Issues associated with recovery of the standard were addressed. The collaborative study report is currently being reviewed by the committee. Continue study.

(2) **FE12** *Hydrolyzed Inulin Syrup and High Fructose Corn Syrup Detection in Apple Juice by GC*: Study Director Michael McLaughlin, U.S. Food and Drug Administration, Center for Food Safety and Applied Nutrition, HFS-347, 5100 Paint Branch Pkwy, College Park, MD 20740-3835, Tel: 301-436-1958, E-mail: michael.mclaughlin@cfsan.fda.gov. A collaborative study was conducted. Results obtained for the detection of high fructose corn syrup (HFCS) and hydrolyzed inulin syrup (HIS) were good and the method showed a high sensitivity ( $p_{+}$ ; between 0.9733 and 0.9867) and a low false negative rate ( $p_{f}$ ; 0.0133–0.0267). However, the committee could not recommend the method be adopted as Official First Action because only one material was used in the study: a single apple juice concentrate spiked at 2 levels with HFCS and at 2 levels with HIS. Although this material was specifically selected because it contained a moderate level of complex oligosaccharides, the criteria for acceptance of a method based on the AOAC/Harmonized Guidelines requires a minimum of 5 independent representative materials. A new protocol is being prepared and the Study Director and General Referee will discuss this during the AOAC Annual Meeting in St. Louis. Continue study.

(3) **FE19** *Determination of Hypoglycin-A in Canned Ackee Fruit by Liquid Chromatography*: Study Director George Ware, U.S. Food and Drug Administration, 60 8th St NE, Atlanta, GA 30309, Tel: 404-253-2249, Fax: 404-253-1209, E-mail: gware@ora.fda.gov. Hypoglycin-A is a toxic compound found in unripe Ackee fruit but not present at significant concentrations in the ripe material. There is a regulatory need to have a collaboratively tested procedure for hypoglycin-A analysis in canned fruit. During the initial review of the protocol, a major shortcoming was identified, which was the lack of a commercial source of pure hypoglycin-A. The Study Director has since identified a potentially feasible manufacturing process. The FDA Atlanta laboratory, with the help of a local university chemistry department, is working on this synthesis and the HG-A should be available early in the fall. Commercial distributors have expressed the willingness to make it available for purchase.

Recommend that a revised protocol be submitted and continue study.

(4) **2004.01** *Detection of  $^{13}C/^{12}C$  Ratio of Ethanol Derived from Fruit Juices and Maple Syrup by IRMS*: Study Directors Eric Jamin and Gilles Martin, Eurofins, Site De La Geraudiere, Rue Bobierre, BP42301, F-44323 Nantes, Cedex 03, France, Tel: 33-2-51-83-21-00, Fax: 33-2-51-83-21-11, E-mail: EricJamin@Eurofins.com. The method was approved Official First Action in 2004 as Method **2004.01**. The method will be eligible for Final Action in 2006. Continue study.

(5) *Detection of Components Associated with Pulpwash in Orange Juice*: Method Advisor Paul Concalon, Florida Department of Citrus, 1115 E. Memorial Blvd, Lakeland, FL 33802-0148, Tel: 941-499-2490, E-mail: pconcalo@citrus.state.fl.us. The Study Director has drawn up 2 separate methods to check for the presence of 3 markers (phlorin, narirutin, and didymin) which are associated with water extractable soluble solids (WESOS/pulpwash) or peel extracts. The first of these procedures uses capillary zone electrophoresis and UV detection. The second method is a conventional HPLC procedure linked with UV detection. The 2 methods were assessed via the AOAC peer validation approach and involved 3 to 6 laboratories per method. The data returned were, however, poor. Only one laboratory returned data using the CZE approach due to time pressures and procedural problems with the method on their equipment. The protocols will be reviewed by the Study Director and General Referee in St. Louis to see if the cause for the poor results can be ascertained. Continue study.

(6) *Capillary Electrophoresis of Organic Acids in Juices*: Tentative Method Advisor Michael S. McCroan, Minute Maid/Coco-Cola North America, 2631 Orange Ave, Apopka, FL 32703, Tel: 407-814-2811, Fax: 407-814-9875, E-mail: mmccroan@minutemaid.com. Some years ago a peer validation of a method for the analysis of organic acids was conducted using capillary zone electrophoresis (CZE). However, the data returned from this were unacceptable and the original Study Director did not want to continue with this study. The General Referee has identified McCroan to take over the project and recommends that he be appointed as Method Advisor. Continue study.

#### *Meat and Meat Products, William A. Trujillo*

**PVM 1:2003** *The Rapid Determination of Fat and Moisture in Meats by Microwave and NMR Analysis*: Method Advisor Cindy Moser, Project Manager, CEM Corp., 3100 Smith Farm Rd, Matthews, NC 28105, Tel: 704-821-7015, Fax: 704-821-7896, E-mail: cindy.moser@cem.com. Method was adopted as Peer Verified Methods<sup>SM</sup>.

#### *Processed Vegetable Products, Vacant*

(1) **985.26** *Solids (Total) in Processed Tomato Products, Microwave Oven Drying Method*: Study Director Henry Chin, National Food Processors Association, 6363 Clark Ave, Dublin, CA 94568, Tel: 925-551-4234, Fax: 925-833-8795, E-mail: hchin@nfpa-food.org. The Study Director is no longer following the topic and has no feedback from users of

the method for several years. Attempts to contact the equipment manufacturer to ascertain interest in updating the method were unsuccessful. Method was moved to Surplus status in January 2004. Later on in 2004, however, the OMB voted to eliminate the "Surplus" category from the OMA and return all Surplus methods to full active status. Thus, the committee's attempts to deal with this method appear to have been an exercise in futility. Discontinue topic.

*Seafoods, Steve Winters*

(1) **999.01** *Volatile Bases in Fish by Ammonia Ion Selective Electrode*: Study Director Chris Ellis, Rhode Island Department of Health Laboratories, 20 Orms St, Providence, RI 02904, Tel: 401-222-5589, Fax: 401-222-6985, E-mail: ChrisE@doh.state.ri.us. Work to date indicates limitations of the electrode in evaluating the quality of long-term frozen tuna. Rancidity factors are not picked up by the electrode. The test still serves as a good comparative indicator between 2 treatments in terms of their statistical similarity. Recommend Final Action approval.

(2) *Histamine Determination by Flow Injection Analysis*: Method Advisor James M. Hungerford, U.S. Food and Drug Administration, Seafood Products Research Center, 22201

23rd Dr, SE, Bothell, WA 98021, Tel: 425-483-4894, Fax: 425-483-4996, E-mail: jhungerf@ora.fda.gov. This method continues to be used for rapid screening of histamine in finfish. A peer-level validation of the method will be conducted this year. Prior to this, SAX clean-up of samples will be evaluated. Choice of clean-up procedure will emphasize rapidity since flow injection analysis is able to process 60 injections per hour and produce a calibration curve in 10 min. Continue study.

(3) **996.15** *Fish Flesh Content in Frozen Coated Fishery Products by End-Scrape Method*: Study Director Jane E. Fox-Dobson, c/o Massachusetts Division of Marine Fisheries, 30 Emerson Ave, Gloucester, MA 01930, Tel: 978-282-7463, Fax: 978-282-7463, E-mail: Jane.E.Fox-Dobson@noaa.gov. No progress has been made since last year's report. The Study Director recommended adding recovery factors for some of the products studied. However, the position of AOAC is that recovery factors are technically not part of method performance and are not endorsed. Furthermore, the collaborative study design is not the appropriate venue for generating accurate and reliable factors. Thus, the evaluation and approval of recovery factors is outside the charge of the committee. Discontinue study.