

METHODS COMMITTEE REPORTS

Committee on Commodity Foods and Commodity Products

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Committee Actions

The committee was very busy this past year as evidenced by the number of recommendations submitted by the General Referees. The committee has reviewed or is currently reviewing 4 collaborative study reports and 1 collaborative study protocol. Two committee members resigned and 5 new members have been appointed.

Cereals and Cereal Products, General Referee,
Stephen R. Delwiche

(1) *F1, Total folate in cereal products, microbiological assay using trienzyme extraction.*—Study Director, Jonathan DeVries, Medallion Laboratories/General Mills, 9000 Plym-

outh Ave N., Minneapolis, MN 55427-3870, Tel: +1-763-764-2774, Fax: +1-763-764-7487, E-mail: jon.devries@genmills.com. This collaborative study is well along in the approval process. The report was submitted and reviewed by the General Referee, the Committee Statistician, the Safety Officer, and the Committee Chair. Some modifications were necessary for the determination of repeatability. The Study Director is in the process of making these changes, particularly with respect to the original Youden Pair analysis. Continue study.

(2) *2002.01 Measurement of alpha-amylase activity in white wheat flour, milled malt, and microbial 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 method "Measurement of alpha-amylase, using the Ceralpha assay" was successfully subjected to interlaboratory evaluation and accepted First Action by AOAC as Method **2002.01** and will be eligible for Final Action in 2004. A report was published in *J. AOAC Int.* Continue study.

(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. Substantial revision of the method has been requested by AOAC, including some contentious issues regarding equipment and supply. The study has been placed on hold pending resolution of this matter. Continue study.

(4) *Phytic acid, LC method.*—Method Advisor, 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. In a preliminary study, 8 unknown food samples (cereals) were sent to 6 collaborators. Results indicated that a high level of disagreement occurred among laboratories despite very good repeatability within the host laboratory. Additional work is underway. Continue study.

Chocolate and Cacao Products, General Referee,
Simon J. Freeman

Awaiting GR report. GR gave report to secretary at meeting. Chair is waiting for report.

Dairy Chemistry, General Referee, Robert L. Bradley, Jr

Awaiting GR report. The chair, however, is aware of the following:

(1) **2001.14** *Total Nitrogen (Protein) Content of Cheese*.—Study Directors, Joanna M. Lynch and David M. Barbano, Cornell University, 207 Stocking Hall, Ithaca, NY 14853, Tel: +1-607-273-6307, Fax: +1-607-254-4868, E-mail: JL72@cornell.edu, dmb37@cornell.edu. No problems have been reported with this method. Recommend Final Action. *Note*: At the committee meeting, GR did not initially recommend for Final Action in his report because it had not been 3 years since approval. After being informed that the waiting period was 2 (not 3 years), he agreed with the recommendation.

(2) **972.16** *Infrared Spectroscopic Methods for the Determination of Milk Composition*.—Topic Advisors D.M. Barbano (*see* 1) and Joanna M. Lynch (*see* 1) are planning to write a manuscript on precalibration quality assurance and submit a collaborative study proposal to characterize the performance of different calibration approaches. Continue study.

(3) *Lactose in Milk*.—Topic Advisors D.M. Barbano (*see* 1) and Joanna M. Lynch (*see* 1) report that the first phase of a study of a spectrophotometric lactose measurement method by an enzymatic procedure was completed and some additional work is planned. A poster summary of the work to date will be presented at the 2003 Annual Meeting. 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: +1-516-576-1400, Fax: +1-516-576-1410, E-mail: corp@800certlab.com. Method is eligible for Final Action. No reports of problems with the method have been received. The chair recommends the method be moved to Final Action and the topic discontinued. *Note*: At committee meeting, GR said the Official Method did not have “by weight” in the title and thus could not be recommended for Final Action. The chair checked in OMA after the meeting and the term “by weight” is in the title. Chair needs follow up with GR. Final Action should not be delayed until next year.

(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: +1-615-837-5317, Fax: +1-615-837-5516, E-mail: dhite@mail.state.tn.us. The method was adopted as Official First Action in 2002 and will be eligible for Final Action Status in 2004. The method report has been published in *J. AOAC Int.* and a poster will be presented at the 2003 meeting. Continue study.

(6) **992.22** and **998.04**—As far as the chair is aware, issues that prevented methods **992.22** (iodine in milk; brought up in 2002) and **998.04** (Neutral β -Galactosidase activity in industrial enzyme preparations; brought up in 2001) from moving to Final Action have not been resolved. GR handed in report at committee meeting. A number of issues need to be addressed. GR will hand in revised report by September 19,

2003. Committee recommends that **992.22** be surplused (instead of going to Final Action) since GR has been unable to contact any laboratories that use the method and one laboratory reported problems. GR says he will follow up on **998.04**.

Fruit and Fruit Products, General Referee, David Hammond

(1) **FE20**, *Determination of total monomeric anthocyanin pigment content, by the pH differential method for fruit juices, beverages, natural colorants, and wines*.—Study Director, Jungmin Lee, Oregon State University, Department of Food Science and Technology, Corvallis, OR 97331, Tel: +1-541-737-6490 or 3591, Fax +1-541-737-1877, E-mail: leeju@onid.orst.edu. The collaborative study for the quantitative analysis of monomeric anthocyanin pigments in fruit juices, beverages, natural colorants, and wines by a differential pH technique has been completed. The results from this study looked good with excellent performance characteristics for the materials: RSD_r ca 3%, RSD_R ca 7%, and HORRAT values ≤ 1.33 . However, the anthocyanin standard, a commercial sample of cyanidin-3-glucoside chloride, which was specifically included in the study to examine the accuracy of the procedure, failed to give acceptable results. The SD is presently examining this standard and others to determine the reason. If an acceptable cause for this apparent low recovery can be found, the GR would recommend that the method be put forward for formal approval as First Action. 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: +1-301-436-1958, E-mail: Micahel.mclaughlin@cfsan.fda.gov. The collaborative study has been completed and a report submitted for evaluation. The actual 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_r) for the 2 adulterants (between 0.9733 and 0.9867) and a low false negative rate (pf; 0.0133–0.0267) for the controls and between the adulterants. These data would indicate that the method is capable of detecting both HIS and HFCS in apple juices in the region of 7 to 8%. However, the committee did not recommend that 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 materials. The committee recommends that either the SD appeal the decision or submit a protocol for a new collaborative.

(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: +1-404-253-2249, Fax: +1-404-253-1209, E-mail: gware@ora.fda.gov. This procedure is an LC method for the analysis of hypoglycin-A in canned Ackee fruit. Hypoglycin-A is

a toxic compound that is found in unripe Ackee fruit but is 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 SD has conducted further work in this area and has recently published a procedure to isolate and characterize the toxin from Ackee fruit. The SD will revise the protocol and submit it for committee review. Continue study.

(4) *FE3, 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. SD submitted a report of the collaborative trial of the assessment of the carbon 13 isotope ratio in the ethanol produced from sugars. The SD sent back a revised manuscript based on the comments of the committee. The revised manuscript is currently under review. Change "Detection" in the title of the method to "Determination" and continue study.

(5) *Detection of components associated with pulp wash in orange juice.*—Method Advisor, Paul Concalon, Florida Department of Citrus, 1115 E Memorial Blvd, Lakeland, FL 33802-0148, Tel: +1-941-499-2490, E-mail: pconcalo@citrus.state.fl.us. The SD has drawn up 2 separate methods to check for the presence of 3 markers (phlorin, narirutin, and dydimim), which are associated with pulp wash/water extractable soluble solids (WESOS) or peel extracts. The first of these procedures uses capillary electrophoresis and UV detection. The second method is a conventional LC procedure linked with UV detection. The protocols for the methods and the collaborative study are in the final stages of preparation. It is proposed that the 2 methods be assessed via the AOAC peer validation approach that will involve 3 to 5 laboratories per method. Continue study.

(6) *Capillary Electrophoresis of Organic Acids in Juices.*—Method Advisor, Michael S. McCroan, Minute Maid/Coca-Cola North America, 2631 Orange Ave, Apopka, FL 32703, Tel: +1-407-814-2811, Fax: +1-407-814-9875, E-mail: mmccroan@minutemaids.com. This was an outstanding topic on the AOAC books where an initial collaborative study for a PVM was organized but failed to return acceptable results. The GR identified another scientist who is willing to take this procedure forward through the PV process. Recommend a protocol be submitted.

Meat and Meat Products, General Referee, William A. Trujillo

PVM 1:2003 The Rapid Determination of Fat and Moisture in Meats by Microwave and NMR Analysis.—Method Advisor, Cindy Moser, CEM Corp., 3100 Smith Farm Rd, Matthews, NC 28105, Tel: +1-704-821-7015, Fax: +1-704-821-7896, E-mail: cindy.moser@cem.com. This method has been accepted for *Peer-Verified Methods*SM status. Continue study.

Processed Vegetable Products, General Referee, Michael Jantschke

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. Study Director is no longer following the topic and has no feedback from users of the method for several years. Attempt to contact equipment manufacturer to ascertain interest in updating the method was not successful. Move to surplus status.

Seafoods, General Referee, Steve Winters

(1) **996.07 Putrescence in Canned Tuna and Cadaverine in Canned Tuna and Mahimahi, Gas Chromatographic 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: Patricia.Rogers@cfsan.fda.gov. The Study Director modified the method to extend its application to the determination of putrescine and cadaverine in shrimp. The sample extraction solvent was changed by the addition of potassium chloride and dilute hydrochloric acid. The chromatographic separation of the derivatized diamines has been significantly improved with a change from a cyanopropyl methyl phenyl methyl silicone megabore column to a more polar crosslinked polyethylene glycol megabore column. A paper on the modified method has been submitted for publication in the *Journal of AOAC INTERNATIONAL*. Recommend a collaborative study be planned and a protocol be submitted.

(2) **996.15 Fish Flesh Content in Frozen Coated Fishery Products by End-Scrape Method.**—Study Director, Jane E. Fox-Dobson, c/o MA Division of Marine Fisheries, 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 for First Action in 1996. The Study Director recommends adjustment factors for some seafood products. The General Referee recommends the adjustment factors be held in abeyance until a full interlaboratory study can be performed. Continue study.

(3) **999.01 Volatile bases in fish-ammonia ion selective electrode method.**—Study Director, Chris Ellis, Rhode Island Department of Health Laboratories, 20 Orms St, Providence, RI 02904, Tel: +1-401-222-5589, Fax: +1-401-222-6985, E-mail: ChrisE@doh.state.ri.us. The Study Director continues to work with the ammonia electrode and related projects pertaining to fish quality assessment. Continue study.

(4) **Histamine Determination by Flow Injection Analysis.**—Method Advisor, James M. Hungerford, U.S. Food and Drug Administration, Seafood Products Research Center (SPRC), 22201 23rd Dr, SE, Bothell, WA 98021, Tel: +1-425-483-4894, Fax: +1-425-483-4996, E-mail: jhungerf@ora.fda.gov. The flow injection method for histamine continues to be used in screening mode in 3 FDA laboratories. Recently the flow injection method has also been applied to the study of fish products treated with carbon monoxide. Rapid cleanup procedures are being investigated to remove positive interferences in specialized and/or ethnic products such as dried bonito powder. Continue study.