METHODS COMMITTEE REPORTS

Committee on Additives, Beverages, and Food Process-Related Analytes

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[1] Inés Cereijo passed away on March 29, 2006.

Committee Actions

The Committee recommends the appointment of Armen Mirzoian, Alcohol and Tobacco Tax and Trade Bureau (TTB), Alcohol and Tobacco Laboratory, 6000 Ammendale Rd, Ammendale, MD 20705, Tel: 240-264-1474, Fax: 240-264-1489, E-mail: armen.mirzoian@ttb.gov, to replace Abdul Mabud, TTB, Alcohol and Tobacco Laboratory, 6000 Ammendale Rd, Ammendale, MD 20705, Tel: 240-264-1402, Fax: 240-264-1489, E-mail: md.mabud@ttb.gov, as General Referee for Alcoholic Beverages.

(1) *Pesticide Residues in Wine:* There was a question whether this topic had been referred to Committee on Residues and Related Topics. Chair will determine the status.

(2) *Determination of Fat in Distilled Spirits (Cream Liqueurs):* The Committee endorses the appointment of Les Pfahl to the position of Study Director.

(3) *Malt Beverages and Brewing Materials:* Chuck Eckermann, AOAC Liaison to the American Society of Brewing Chemists, has agreed to act as Topic Advisor for Malt Beverages. There is no study currently associated with this topic.

The Committee concurs with the General Referee for Alcoholic Beverages for the following actions:

(1) *Analysis of Carbohydrate in Wine:* Pending the report of the Wine Institute on the first round of samples, continue study.

(2) *Determination of Cyanide in Stone Fruit Brandies and Liqueurs by IC with Amperometric Detection:* Discontinue study.

(3) *Thujone, Vanillin, Ethyl Vanillin, Coumarin, Maltol, and Ethyl Maltol in Beverage Alcohol Products:* This is an important topic. Continue study.

(4) *Determination of Site-Specific (D/H) Ratios in Vanillin by ^{2}H-NMR:* The manuscript was submitted to the Chair for review. In the absence of a General Referee for Flavors, John Casanova, General Referee for Food Additives, agreed to shepherd this study for the Committee. The manuscript has been reviewed by the General Referee and editing is complete. The manuscript is ready for Committee review.

The Committee concurs with the General Referee for Spices and Other Condiments for the following actions:

(1) *Moisture in Spices, Vacuum Oven Method, C-14*: There is no interest in pursuing this method. Discontinue study.

(2) *Water Activity of Spices*: Remove the Study Director. Discontinue study.

(3) *Moisture and Total Fat in Dressing, Sauces and Condiments by Rapid Microwave Drying and Automatic Solvents Extraction*: There is no interest in pursuing this method. Discontinue study.

(4) *Piperine in Black Pepper:* The Committee recognizes the safety concerns expressed by the General Referee are legitimate. They endorse the recommendation that a peer review study look into the change in extraction solvent to determine if this is a minor or a major change to the Official Method.

The Committee received the initial report of the Alcohol Beverage Working Group. They endorse the efforts of industry members to initiate and fund the development of methods for the nutritional analysis of alcohol beverage products. The working group is encouraged to work closely with the General Referee for Alcohol Beverage Products on this project.

General Referee Reports

Alcoholic Beverages, Abdul Mabud, Armen Mirzoian

Selected Study Director topics:

(1) *Analysis of Carbohydrate in Wine:* TTB is in the process of developing guidelines for the alcohol facts label in alcoholic beverage products. One of the ingredients in the wine facts label is the carbohydrate content. The Wine Institute, in collaboration with ETS Laboratory, has developed an HPLC method for the analysis of carbohydrates in wine. They would like to conduct a collaborative study through AOAC. The Wine Institute has approved the AOAC collaborative study fee. We are waiting for further communication with the Wine Institute. Continue study.

(2) *Pesticide Residues in Wine:* Pesticide residue in wine has health consequences, thus AOAC has an interest in initiating a collaborative study for pesticide residue in wine. The National Laboratory Center of the TTB has developed a GC/MS method for the analysis of pesticide residues in wine. Michael Webster, a chemist of TTB, has expressed an interest in serving as Study Director for a collaborative study for the method. The issue that is yet to be resolved is the AOAC fee for the collaborative study. Continue study.

(3) *Determination of Fat in Distilled Spirits (Cream Liqueurs):* Study Director vacant. Continue study.

(4) *Determination of Cyanide in Stone Fruit Brandies and Liqueurs by IC with Amperometric Detection:* The Study Director has completed the method development and validation but did not find any collaborators for the study. The study is currently on hold until guidance is received from the Offical Methods Board regarding the cost and procedure to incorporate it as an SLV/Regulatory Method in e-CAM. Discontinue study.

(5) *Thujone, Vanillin, Ethyl Vanillin, Coumarin, Maltol, and Ethyl Maltol in Beverage Alcohol Products:* Study Director vacant. TTB is continuing working in this area. Continue study.

(6) *Malt Beverages and Brewing Materials:* Study Director vacant. Continue study.

Color Additives, Vacant

Flavors, Vacant

Food Additives, John Casanova

(1) *Use of Griess Reagents Containing Vanadium (III) for the Post-Column Derivatization and Simultaneous Determination of Nitrite and Nitrate in Baby Food:* John A. Casanova, Lois K. Gross, Sarah E. McMullen, and Frank J. Schenck, Food and Drug Administration, 60 8th Street, Atlanta, GA 30309. This publication has been completed and has been submitted to *JAOAC*. We are awaiting word from AOAC as to whether it has been accepted for publication. A follow-up using a reverse-phase chromatographic method may be submitted if the time is available this coming year.

(2) *Determination of Site-Specific (D/H) Ratios in Vanillin by ^{2}H-NMR: Collaborative Study:* Publication was reviewed and was suggested for submission to Committee for recommendation as an Official Method. Study Directors Eric Jamin, Frédérique Martin, Gilles G. Martin.

In addition, there are a number of projects underway.

(3) *Determination of N-methyl Carbamate Residues in High Moisture Food Matrixes*: The number of *N*-methyl carbamates presently analyzed in the agency has been increased from 11 to around 37 compounds. All 37 have been run on at least 3 columns of different polarity and other polar columns are presently being investigated. Further work will expand the extraction procedures to dry and fatty commodities (e.g., animal feeds, grains, etc.) in order to increase the scope of regulatory products analyzed. We have arranged to work with at least 2 other laboratories: FDA (Jefferson, AR) with Robert Mobley and Leonard Collins of the Mississippi State Laboratory (tentative Study Director for this project). There is also a possibility that our FDA/CFSAN laboratory will participate (John Wong). Collaboration with these laboratories will hopefully proceed later this year and into next year as we work to modify the Quechers Method for analysis of *N*-methyl carbamates from these difficult matrixes.

In addition, a GC/MS confirmatory procedure is in development for the *N*-methyl carbamates in the FDA (Atlanta) laboratory.

Other projects of great interest that we would like to develop include the following that were mentioned in last year's report:

(1) *Analysis of Carboxylic Acids in Food*: The lack of suitable procedures for acids in seafood has prompted some of us in the agency to find new extraction and analysis methods. Confirmatory methods are also being explored using GC/MS derivatization but we are not at the point where the methods are publishable. Improvement and fine-tuning of the method is needed. We are also interested in the acid extraction in general to simplify the extraction of samples for analysis of glyphosate and its metabolites for routine analysis.

(2) *Analysis of Color Additives in a Variety of Matrixes*: Eventually, we would like to collaborate with other

laboratories (e.g., Silliker Labs) with a method to analyze color additives in a variety of food matrixes; special emphasis is being placed on sample preparation because this area has traditionally caused the most difficulties. Some work has been performed using solid-phase extraction (SPE) with very promising results. However, not enough colors/food additives were explored with the method, and therefore different types of SPE materials will need to be tested depending on the color additives (acidic, basic, etc). We hope to at least try to work on this before the next year's GR report.

We would be interested to know if anyone from the Committee has an interest in any of these projects or else is familiar with persons or laboratories who might have the same analyses of concern. Any suggestions/recommendations would be welcomed.

Spices and Other Condiments, Roman Grypa

Roman Grypa, McCormick & Co., Inc., 202 Wight Ave, Hunt Valley, MD 21031, Tel: 410-527-6531, Fax: 410-5276527, E-mail: roman grypa@mccormick.com

(1) *Moisture in Spices, Vacuum Oven Method, C-14:* Study Director Louis Sanna, current address is unavailable. If there are continued discussions regarding the validity of this method, then my recommendation is to discontinue this method and initiate a new study, provided it is funded through AOAC.

(2) *Water Activity of Spices:* Study Director Pierre Metra, Lareal, PO Box 234, Vannes Cedex 56006, France, Tel: 33-2-9-754-5455, Fax: 33-2-9-754-5464, E-mail: pmetra@lareal.com. There has been no contact or reports submitted by this Study Director. Discontinue topic.

(3) *Moisture and Total Fat in Dressing, Sauces, and Condiments by Rapid Microwave Drying and Automatic Solvents Extraction, Cg013:* Study Director John Brill, McCormick & Co. Inc., 204 Wight Ave, Hunt Valley, MD 21031, Tel: 410-771-7975, Fax: 410-527-8071, E-mail: John_Brill@McCormick.com. Study Director has stated that there has not been any interest in this topic and the manufacturer has changed the fat extraction equipment. Recommendation is to discontinue topic, unless it is funded by AOAC.

(4) *Piperine in Black Pepper (AOAC Official Method **987.07**)*: Recommend changing the extraction and optical solvent from ethylene dichloride to ethanol. The American Spice Trade Association (ASTA Method 12.1) and the International Organization for Standardization (ISO Method 5564:1982) extract piperine from black and white pepper with ethanol. Changing the extraction solvent in AOAC Method **987.07** would make these 3 analytical methods identical. A peer review study can be performed to document differences in piperine content in black pepper that occur by switching extraction solvents.

Filth and Extraneous Materials in Food and Drugs, L. Mickey Anderson

L. Mickey Anderson, U.S. Food and Drug Administration, 60 80th St NE, Atlanta, GA 30309, Tel: 404-253-1200 ext. 5233, Fax: 404-253-1208, E-mail: manderso@ora.fda.gov. Recently appointed as General Referee. There is no formal report to submit. Future actions are to review recommendations and establish priorities from individuals with professional interest in filth and extraneous materials in foods and drugs. Future activity may include (pending funding approval) a study with collaborative efforts for multiple products with multiple filth elements. Emphasis should be placed on detecting filth that is associated with potential physical, chemical, or biological hazards and filth from vector insects or other pests.

(1) No changes are recommended to official analytical methods.

(2) We solicit suggestions and comments from interested individuals or laboratories within the agency or from outside sources. Interested party or parties should contact the General Referee.

Working Group Report

Nutritional Analysis for the Labeling of Alcoholic Beverages

Chair Les Pfahl, E-mail: lpfahl@bacardi.com. A working group has been formed to investigate the nutritional analysis for the labeling of alcohol containing beverages. The working group will consist of interested members of the brewers, vintners, and distillers. The mission of the group will be to explore areas of interest to the industry regarding nutritional analysis of alcohol containing beverages. The group consists of Les Pfahl, Assistant Vice President New Product Development, Jenny Bairo, Manager Research and Innovation Support, Brown Forman, and Steve Kupina, Manager of Technical Services, Constellation Wines. Initial areas of interest are the analysis of fat in cream liqueurs and the analysis of solids in cream liqueurs.