

METHODS COMMITTEE REPORTS

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

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Committee Actions

The Committee has one study that will be recommended for First Action status in the near future this study was "grand fathered" under the new business model and is exempt from those funding requirements: *Moisture in Spices, Vacuum Oven Method, C-14*.—Study Director Louis Sanna, Gilroy Foods, 1350 Pacheco Pass Hwy, PO Box 1088, Gilroy, CA 95020-1088, Tel: 408-846-3452, Fax: 408-846-3152, E-mail: lsanna@gilroyfoods.com.

Committee C has identified 3 studies currently under investigation that have significant health and/or safety consequences and recommends that AOAC fund these important studies.

(1) *Use of Griess Reagents Containing Vanadium (III) for the Post-Column Derivatization and Simultaneous Determination of Nitrite and Nitrate in Baby Food*.—Study Directors John A. Casanova, Lois K. Gross, Sarah E. McMullen, and Frank J. Schenck, U.S. Food and Drug Administration, 60 8th St, Atlanta, GA 30309.

(2) *The Quantitative Analysis of Color Additives in Food*.—Study Director Sneh D. Bhandari, Silliker Laboratory, Chemistry, 1304 Halsted St, Chicago Heights, IL 60411, Tel: 708-756-3211 (ext. 342), Fax: 708-756-0049, E-mail: sneh.bhandari@silliker.com.

(3) *Pesticide Residues in Wine*.—Proposed Study Director Michael Webster, Alcohol and Tobacco Tax and Trade Bureau (TTB), Alcohol and Tobacco Laboratory, 6000 Ammendale Rd, Beltsville, MD 20705, Tel: 240-264-1402, Fax: 240-264-1489, E-mail: michael.webster@ttb.gov.

Committee General Referee Reports

Alcoholic Beverages

General Referee Abdul Mabud, Alcohol and Tobacco Tax and Trade Bureau (TTB), Alcohol and Tobacco Laboratory, 6000 Ammendale Rd, Beltsville, MD 20705, Tel: 240-264-1402, Fax: 240-264-1489, E-mail: md.mabud@ttb.gov.

(1) *Analysis of Carbohydrate in Wine*.—The U.S. Alcohol and Tobacco Tax and Trade Bureau (TTB) is in the process of

developing guidelines for alcohol facts label in alcoholic beverage products. One of the ingredients in the wine facts label would be carbohydrate content. The Wine Institute, in collaboration with ETS Laboratory, has developed an HPLC method for the analysis of carbohydrates in wine and would like to conduct a collaborative study through AOAC INTERNATIONAL. Norma Hill, Secretary of AOAC Methods Committee C, has informed GR that The Wine Institute may fund the collaborative study.

Committee recommends that when the Wine Institute officially approaches the GR, GR will appoint a Study Director for the collaborative.

(2) *Pesticide Residues in Wine*.—Pesticide residue in wine has health consequences, thus AOAC has an interest in initiating collaborative study for pesticide residue in wine. The National Laboratory Center of Alcohol and Tobacco Tax and Trade Bureau (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 the Study Director for a collaborative study for the method. The issue that is yet to be resolved is the \$30K fee for the collaborative study. Committee recommends the GR appoint an SD, work with AOAC to find sponsors, and initiate the search for 10–12 laboratories for the collaborative.

(3) *Determination of Cyanide in Stone Fruit Brandies and Liqueurs by IC with Amperometric Detection*.—Study Director Norma Hill, Bureau of Alcohol, Tobacco and Firearms, Laboratory Services, 355 N. Wiget Ln, Walnut Creek, CA 94598, Tel: 925-280-3642, Fax: 925-280-3601. Hydrogen cyanide (prussic acid) is a naturally occurring contaminant in stone fruit brandies and liqueurs. The levels of free cyanide and the associated toxic cyanohydrins have health consequences. The existing AOAC Method (973.19) is a qualitative method for the detection of cyanide. Thus, a method for the quantitative determination was desired. 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 OMB regarding the cost and procedure to incorporate it as an SLV/Regulatory Method in e-CAM. Norma Hill reports that her laboratory has begun work to update and simplify this method using current technology. SD recommends dropping this topic. When the new method has undergone SLV, it will be submitted separately.

Committee recommends dropping the topic at this time.

(4) *Vanillin, Ethyl Vanillin, Coumarin, Maltol and Ethyl Maltol in Beverage Alcohol Products*.—Study Director, vacant.

Committee recommends dropping the topic.

(5) *Malt Beverages and Brewing Materials*.—Study Director, vacant. Charles Eckermann reports that there are a number of topics of interest to the brewing industry. He is confident that he will be able to solicit a GR.

Committee recommends continuing the topic.

Color Additives

GR Sneh D. Bhandari, Silliker Laboratory, 1304 Halsted St, Chicago Heights, IL 60411, Tel: 708-756-3211 (ext. 342), Fax: 708-756-0049, E-mail sneh.bhandari@silliker.com.

FDA regulates color additives used in foods, drugs, cosmetics and medical devices in the United States. FDA conducts a certification program for batches of additives that are required to be certified before sale. Public Health Security and Bioterrorism Preparedness and Response Act of 2002 requires domestic and foreign manufacturers of the color additives used as ingredients in food to register with FDA by December 12, 2003. FDA requires declaration of all the color additives including certified colors in food and dietary supplements. If undeclared certifiable colors are found in a sample, it can be considered adulterated/misbranded. Color additives-related food recall accounted for about 19% of all the recalls reported by FDA in the United States in 2003. The use of color additives and their concentrations in food are closely regulated in many other countries including Europe.

The current AOAC Method 988.13 for FD&C color additives in food, is based on isolation of different colors from food extract using a C18 solid phase (Sep-Pak) and application of a scheme of different amounts and concentrations of isopropanol. The colors are identified by spectrophotometry and confirmed by thin-layer chromatography. This is a laborious and tedious semiquantitative method. The spectrophotometric methods often prove inadequate in the analysis of food samples due to the overlapping of the spectral absorption maxima. There is an urgent need for validation/development of a simple, rapid, accurate and precise method for quantitation of FD&C colors in food and beverage samples. Regulatory agencies, food industry and independent laboratories routinely analyze vast numbers of food samples for FD&C colors for compliance and other purposes. GR has identified a method, which is simple and rapid. This is an HPLC-based method using a specific gradient program for elution of the colors. The detection is in the visible range. This is a single laboratory validated method. A study was undertaken by GR recently in collaboration with the Beverage Alcohol Laboratory of the Alcohol and Tobacco Tax and Trade Bureau for analysis of synthetic colors in certain beverage and candy samples. The analysis was performed by the HPLC method as well as by a modified AOAC TLC Method 988.13 (Dugar et al., 1994). The purpose of the study was to compare the performance of the HPLC method with a standard (conventional) TLC method. The data was presented at the 118th AOAC annual meeting in St. Louis.

One of the conventional methods to purify and concentrate colors from the foods and beverages is to acid-fix the dyes to virgin wool. The dyes can be eluted out selectively with ammonia and other solutions. The ATF Methods of Analysis Manual (1991) describes this method for synthetic dyes analysis. A recent preliminary study by John Casanova at FDA Analytical Laboratories in Atlanta evaluated the use of the virgin wool against SPE columns in the process of

concentration and purification of colors from a beverage sample with very low concentration of the dye. The SPE columns worked far better compared to the virgin wool to recover, purify, and concentrate synthetic colors from the sample.

Recommendations

The GR recommends that the HPLC method continue to be further studied collaboratively using the AOAC protocols. Collaborators are solicited to study this HPLC method for FD&C colors, specifically, Yellow 5 and 6, Red 2, 3 and 40, Blue 1 and 2, and Green 3. Those who are interested please contact the GR.

Committee recommends moving this topic as a study under Food Additives. SD has volunteered to continue under those circumstances.

Filth and Extraneous Materials in Foods and Drugs

GR has retired. No report. In October 2003, Mickey Anderson of the FDA expressed interest in succeeding to the position of GR. Repeated attempts to contact Anderson on this point have met with no response.

Alan Olsen has volunteered to continue as GR, if the committee is unable to recruit as successor. Ben Canas reports that FDA is actively moving to recruit a successor to Olsen.

The Committee thanks Olsen for his offer and recommends the GR position be held open pending the result of the FDA recruitment effort.

Flavors

GR Janet M. Scalse, Bureau of Alcohol, Tobacco and Firearms, National Laboratory Center, 1401 Research Blvd, Rockville, MD 20850, Tel: 301-217-5763, Fax: 301-413-9463, E-mail: jmscalese@atfhq.atf.treas.gov.

Determination of site-specific (D/H) ratios in vanillin by ²H-NMR: Study Director Eric Jamin, Eurofins Scientific, Rue Bobierre BP 42301, F-44323 Nantes cedex 3, E-mail: EricJamin@Eurofins.com. According to the design of the study, a familiarization sample was circulated to the participants in the beginning of 2001. Eight laboratories performed the analyses. This pretrial was successful and allowed us to provide some additional advice to some of the laboratories regarding data treatment. At this stage it can be foreseen that reasonably reproducible values can be achieved by the selected group of laboratories. Ten laboratories have agreed to participate in the final trial (2 of them will have to analyze the familiarization samples first).

The samples of the proper trial have already been prepared, tested for homogeneity, and dispatched to the 10 participating laboratories. Although the deadline for reporting results is now passed, only 8 out of 10 laboratories have reported results. There are several reasons for this but the main one is obviously the amount of NMR spectrometer time required. However we are still confident that at least 9 laboratories can send their results. We are following up the progress and hope to send a report by the end of this year.

Committee recommends continuation of this study.

The GR has accepted the position of Committee Member. The Committee is actively searching for a new GR for this topic.

Food Additives

GR John Casanova, U.S. Food and Drug Administration, 60 Eighth St, Atlanta, GA 30309, Tel: 404-253-1200 ext. 5342, E-mail: jcasanov@ora.fda.gov.

Use of Griess Reagents Containing Vanadium (III) for the Post-Column Derivatization and Simultaneous Determination of Nitrite and Nitrate in Baby Food: Study Directors John A. Casanova, Lois K. Gross, Sarah E. McMullen, and Frank J. Schenck, U.S. Food and Drug Administration, 60 8th St, Atlanta, GA 30309.

This publication has been completed and will be submitted to *JAOAC* in the very near future (August 2004).

The Committee recommends submission of this topic to the Committee and to OMB for official review.

In addition, there are a number of projects underway:

(1) *Analysis of carboxylic acids in seafood:* 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 presently being explored.

The Committee recommends the appointment of an SD for this topic.

(2) *Quantitative analysis of color additive in Food:* Analysis of color additives in a variety of matrixes: the effort this fiscal year will be 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 the sample preparation because this area has traditionally caused the most difficulties.

The Committee recommends consolidation of this study with the GR for Colors.

(3) *Determination of N-methyl carbamate residues in high moisture food matrixes:* Work has been underway to expand the number of N-methyl carbamates presently analyzed in the agency from 11 to around 37 or more in current use around the world. All 37 have been run on at least 3 columns of different polarity to assess performance of the separation in general. Further work will expand the extraction procedures to dry commodities (e.g., animal feeds, grains, etc.) in order to increase the scope of regulatory products analyzed.

The Committee recommends the appointment of an SD for this topic.

Nonalcoholic Beverages

No GR. No report.

The FDA has expressed an interest in appointing a GR for this important topic.

The Committee recommends keeping this topic.

Spices and Other Condiments

GR Roman Grypa, McCormick & Co., Inc., 202 Wight Ave, Hunt Valley, MD 21031, Tel: 410-527-6531, Fax: 410-527-6527, E-mail: roman_grypa@mccormick.com.

Recommendations

Moisture in Spices, Vacuum Oven Method, C-14.—Study Director Louis Sanna, Gilroy Foods, 1350 Pacheco Pass Hwy, PO Box 1088, Gilroy, CA 95020-1088, Tel: 408-846-3452, Fax: 408-846-3152, E-mail: lsanna@gilroyfoods.com. Final study manuscript has been submitted for review. Recommend expediting process to move this method to First Action Official Method adoption or discontinue this topic.

The Committee is in the process of consolidating the paperwork in order to move this study to First Action status.

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. No report received. Continue study.

The Committee recommends the SD find a sponsor for this study.

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. Collaborative study protocol has been submitted for review. There is no interest from industrial contacts to fund this collaborative study. Recommend continuation of this study moving forward, if AOAC funds this study.

This topic does not have regulatory, health, or safety consequences. It is unlikely that AOAC will fund this study. The Committee recommends the SD find a sponsor for this study.

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.

The Committee recommends that an SD be appointed.