MARCH 14, 2016

AOAC STAKEHOLDER PANEL on STRATEGIC FOOD ANALYTICAL METHODS (SPSFAM)

HEAVY METALS EXPERT REVIEW PANEL MEETING BOOK

SALON G
Gaithersburg Marriott Washingtonian Center
9751 Washingtonian Boulevard
Gaithersburg, Maryland, 20878

contacts: dfrazier@aoac.org or cdent@aoac.org

The Scientific Association Dedicated to Analytical Excellence®

AOAC Stakeholder Panel on Strategic Food Analytical Methods

Arsenic Speciation Expert Review Panel

Monday, March 14, 2016

8 :00 a.m. – 12 :00 p.m.

A G E N D A

EXPERT REVIEW PANEL CHAIR: Rick Reba, Nestlé

1. Welcome and Introductions
   Rick Reba, Nestlé (ERP Chair)

2. Review
   A. AOAC Volunteer Policies & ERP Proccess Overview and Guidelines
      Deborah McKenzie

3. Review of Methods
   For each method the assigned ERP members will present a review of the revised method manuscripts, after which the ERP will discuss the method and render a decision on the status for each method.

   A. ARS-01
   B. ARS-02
   C. ARS-03
   D. ARS-04
   E. ARS-05

4. Final Action Requirements for Approved Method(s)

5. Adjourn

SPSFAM ERP for Heavy Metals
As of: March 08, 2016

Rick Reba, Chair
Nestle USA, Inc.
6625 Eiterman Rd
Quality Assurance Center
Dublin, OH 43016
USA
Tel. +1(614) 526-5358 (O)
Fax. +1(480) 3796-6700
Email: rick.reba@us.nestle.com
Term: June 27, 2013 - June 26, 2016

Farzaneh Maniei, MS, Member
The Coca-Cola Company
One Coca Cola Plz NW
TEC 720
Atlanta, GA 30313
USA
Tel. +1(404) 676-9876 (O)
Fax. +1(404) 598-9876
Email: fmaniei@coca-cola.com
Term: June 27, 2013 - June 26, 2016

Sneh D. Bhandari, Ph.D, Member
Mérieux NutriSciences
3600 Eagle Nest Dr
Crete, IL 60417
USA
Tel. +1(708) 367-4628 (O)
Fax. +1(708) 756-0049
Email: sneh.bhandari@silliker.com
Term: June 27, 2013 - June 26, 2016

William Mindak, Member
FDA/CFSAN
5100 Paint Branch Pkwy
MS HFS-716
College Park, MD 20740
USA
Tel. +1(240) 402-2005 (O)
Fax. +1(301) 436-2632
Email: William.Mindak@fda.hhs.gov
Term: August 12, 2013 - June 26, 2016

Michelle Briscoe, Member
Brooks Applied Labs
18804 North Creek Pkwy
Suite 100
Bothell, WA 98011
USA
Tel. +1(206) 632-6206 (O)
Fax. +1(206) 632-6017
Email: michelle@brooksapplied.com
Term: June 27, 2013 - June 26, 2016

Mr. Cory J Murphy, Member
Canadian Food Inspection Agency
1992 Agency Dr
Dartmouth, NS B3B 1Y9
Canada
Tel. +(902) 426-0232 (O)
Fax. +(902) 426-0314
Email: cory.murphy@inspection.gc.ca
Term: August 1, 2013 - June 26, 2016

Min Huang, Member
Frontage Laboratories, Inc.
700 Pennsylvania Dr
Exton, PA 19341
USA
Tel. +1(484) 348-4869 (O)
Email: MHuang@frontagelab.com
Term: June 27, 2013 - June 26, 2016

Jenny Nelson, Member
Agilent Technologies, Inc.
3045 Deakin St Unit U
Berkeley, CA 94705
USA
Tel. +1(510) 517-6475 (O)
Email: jenny_nelson@agilent.com
Term: June 27, 2013 - June 26, 2016

Jenny Scifres, Member
USDA FSIS OPHS LQAD ALP
950 College Station Rd
Athens, GA 30605
USA
Tel. +1(706) 546-2337 (O)
Fax. +1(706) 546-4265
Email: jenny.scifres@fsis.usda.gov
Term: August 1, 2013 - June 26, 2016

Li Sheng, Member
EPL Bio Analytical Services
9095 W Harristown Blvd
Niantic, IL 62551
USA
Tel. +1(217) 963-2143 (O)
Email: li.victoria@gmail.com
Term: June 27, 2013 - June 26, 2016

Christopher Smith, Member
The Coca-Cola Company
1 Coca Cola Plz NW
TEC 933
Atlanta, GA 30313
USA
Tel. +1(404) 676-2360 (O)
Email: christopsmith@coca-cola.com
Term: June 27, 2013 - June 26, 2016

Mr. Darryl M. Sullivan, Member
Covance Laboratories
N2743 Butternut Rd
Poynette, WI 53955
USA
Tel. +1(608) 242-2711 (O)
Fax. +1(608) 242-7903
Email: darryl.sullivan@covance.com
Term: June 27, 2013 - June 26, 2016

Scott G. Coates, AOAC Staff Liaison
AOAC INTERNATIONAL
2275 Research Blvd Ste 300
Rockville, MD 20850-3250
USA
Tel. +1(301) 924-7077 x137 (O)
Fax. +1(301) 924-7089
Email: scoates@aoac.org
Term: June 27, 2013 - June 26, 2016

Christopher Dent, AOAC Staff Liaison
AOAC INTERNATIONAL
2275 Research Blvd Ste 300
Rockville, MD 20850-3250
USA
Tel. +1(301) 924-7077 x119 (O)
Fax. +1(301) 924-7089
Email: cdent@aoac.org
Term: June 7, 2014 - June 7, 2017

Dawn L. Frazier, AOAC Staff Liaison
AOAC INTERNATIONAL
2275 Research Blvd Ste 300
Rockville, MD 20850-3250
USA
Tel. +1(301) 924-7077 x117 (O)
Fax. +1(301) 924-7089
Email: dfrazier@aoac.org
Term: June 27, 2013 - June 26, 2016

Deborah McKenzie, AOAC Staff Liaison
AOAC INTERNATIONAL
2275 Research Blvd Ste 300
Rockville, MD 20850-3250
USA
Tel. +1(301) 927-7077 x157 (O)
Fax. +1(301) 924-7089
Email: dmckenzie@aoac.org
Term: June 27, 2013 - June 26, 2016

Review of Methods Purporting to Meet AOAC SMPR 2015.006,
Quantitation of Arsenic Species in Selected Food and Beverages
(http://www.aoac.org/imis15_prod/AOAC_Docs/SPSFAM/SMPR2015_006.pdf)

LIST OF REVIEWERS

Method	Primary Reviewer	Secondary Reviewer	Other Reviewers
ARS-01	Cory Murphy	Jenny Nelson	Sneh Bhandari
ARS-02	Farzaneh Maniei	Christopher Smith	Sneh Bhandari
ARS-03	Min Huang	Darryl Sullivan	Sneh Bhandari
ARS-04	Bill Mindak	Jenny Scifres	Sneh Bhandari
ARS-05	Michelle Briscoe	Li Sheng	Sneh Bhandari

AOAC Expert Review Panels

2016 ERP Meeting Logistics

ERP Meetings

- ERPs will meet in person at a minimum of twice a year and up to four times per year:
  - AOAC Mid-Year meeting (DC metro area)
  - AOAC Annual Meeting.
  - 2 additional designated times for proprietary method Organziational Affiliates
- At the ERP meeting:
  - Primary and secondary reviewers **or entire ERP** will present their reviews and makes a motion/recommendation to the ERP whether or not to adopt the method as First Action OMA.
  - ERP discusses the method.
  - ERP renders a decision on First Action status.
  - ERP renders decisions on modifications to First Action methods only.
- If the method is adopted
  - ERP decides on what additional information is needed to recommend the method for Final Action status

ERP MEETINGS

- MEETINGS ARE HELD IN-PERSON, HOSTED BY AOAC
- A QUORUM IS THE PRESENCE OF SEVEN (7) MEMBERS OR 2/3 OF THE TOTAL VETTED ERP, WHICHEVER IS GREATER.

IF NO QUORUM, THEN NO MEETING!

ERP MEETINGS

- REVIEWERS PRESENT THEIR REVIEWS AND MAY INITIATE A MOTION TO ADOPT THE METHOD IF THEY CHOOSE
  - Chair recognizes the reviewers
  - Primary and secondary / ERP reviews are presented.
    - If in favor, they may make and second a motion to adopt or not adopt the method
    - Chair can then entertain discussion on the method
    - Chair can call for a vote once deliberation is complete

ERP MEETING - Discussions

- In your collective judgment, is the method scientifically sound and can be followed as written?
- **In your collective judgment, does the method sufficiently meet the SMPR?** Are method claims supported (sole source methods)?
- In your collective judgment, what are the strengths and weaknesses of the method?
- In your collective judgment, do the weaknesses outweigh the strengths in your recommendation for the method?
- In your collective judgment, is the method safe and can it serve well the stakeholder community that will use the it?
- In your collective judgment, is additional information needed to before considering this method for Final Action OMA status?

ERP CONSENSUS

ERP CONSENSUS

- First Action Official Methods status is granted:
- Method must be adopted by unanimous decision of ERP on first ballot, if not unanimous, negative votes must delineate scientific reasons.
- Negative voter(s) can be overridden by 2/3 of voting ERP members after due consideration.
- Method becomes First Action on the date when ERP decision is made.

ERP CONSENSUS

- The ERP may then reach consensus on any additional information that it needs to review to be able to make a recommendation for Final Action *Official Methods* status.

- This is a separate motion.

Approval of Modifications

- If ERP approves a method modification including extensions, then the method begins a new two (2) year period.

- If the method modification is to correct an editorial error, then the method, then there is no change.

a ethod modificationă require substantiation of the modiɪication or extension with proof of method performance as deemed suitable by the EPR.

FIRST ACTION TO FINAL ACTION STATUS

ERP Tracking

- Between First Action and Final Action:
  - The primary and secondary reviewers track the methods on behalf of the ERP over this time period.
    - Based on information from method authors, laboratories using the method, general community feedback, additional laboratory work
    - Are ERP recommendations being fulfilled?
    - Is the method meeting the standard criteria more closely?
    - How well is community guidance and OMB guidance being reflected?
  - Updates on the method are given by the primary and secondary reviewers during the ERP meetings.
  - At the end of two years, ERP makes a recommendation to OMB for Final Action status, repeal, or continuance.

Path to Final Action

Review of ERP Method Recommendations

What to Expect from AOAC Official Method Board (OMB)

Standard Method Performance Pathway

1. Standard Method Performance Requirements authored by Working Groups and established by Stakeholders
2. Expert Review Panel (ERP) vetted by OMB
3. ERP approves methods for First Action
4. Method reproducibility data collected
5. ERP monitors method performance
6. ERP recommendations sent to OMB within 2 years
   - Final Action, First Action continuation, or Repeal

OMB Liaison

- OMB member or designee is assigned to your ERP
- Liaison monitors First Action to Final Action process
- Monitors ERP's documentation of all items in OMB Guidance document (OMA Appendix G)

Method Applicability

- Determine how method meets stakeholder's needs
  - scope, accuracy, precision, etc.
- Are ERP recommendations & improvements implemented?
- Assess method limitations & concerns

Safety Concerns

- Safety review completed for First Action
  - Participation by Safety Committee
- All safety issues identified during 2 year review addressed
  - Participation by Safety Committee

Reference Materials

- Identification of potential reference materials (RM)
  - If none found, define alternative options
- RM performance expectations

Available resource is the AOAC Technical Division on Reference Materials (TDRM)

Single Laboratory Validation

Chemistry

- Linearity
- Accuracy
- Repeatability
- LOD / LOQ
- Matrix scope
- Selectivity

Microbiology

- Inclusivity/Exclusivity
- Robustness
- Repeatability
- POD or equivalent
- Matrix scope

AOAC Committee on Statistics is your resource

Quantitative Reproducibility/Uncertainty

- Experimental designs may vary
  - Collaborative study
  - Proficiency Testing data
  - Multi-lab study variations
- Committee on Statistics
  - is available to discuss new study design protocols
  - Formalized tools were presented at the 2013 Annual Meeting

Qualitative Reproducibility/Uncertainty

- Experimental designs may vary
- Committee on Statistics is available to discuss new study protocols designs

Compare to SMPR

- Method meets Performance Criteria
- Method does not meet Performance Criteria
  - Acceptable or not? List reasoning
- Document acceptability to Stakeholders

Feedback from Users

- Solicit and document user feedback
  - ERP Chair determines mechanism
  - May take form of
    - Proactive calls to users
    - Tally of incoming calls
    - Emails
    - Web surveys

Feedback from Users

- Method performance
- Safety Concerns
  - Warnings
  - Alternatives
- Equipment and supply availability
  - Readily available
  - Practicality
  - Suggested improvements
  - Failures
- Reference material availability

ERP SUMMARY FOR FIRST TO FINAL ACTION METHOD RECOMMENDATION

AOAC No.	NAME OF METHOD

GUIDANCE FOR AOAC ERPS - APPENDIX G[1]	**Considered?**	**Comments/Reference if applicable**
Method Applicability		
ERP First Action to Final Action recommendations & improvements		
Draft Final Action method reviewed by ERP		
Safety Concerns		
Reference Materials		
Single Laboratory Validation		
Reproducibility/Uncertainty and Probability of Detection		
Comparison to SMPR (SMPR criteria met?)		
Feedback from Users of Method		

DOCUMENTATION	**Available?**	**Comments**
Safety Evaluation		
Reference Materials		
SLV or PTM		
Approved Validation Protocols		
Statistics Review		
Method Published in OMA		
Method Performance vs SMPR criteria		
Feedback Information		
Additional Recognition(s)		
ERP Reports		
Manuscript(s) Published in JAOAC		
ERP Method Recommendation (Final Action/Repeal/Continuation)		

[1] *Official Methods of Analysis of AOAC INTERNATIONAL*, Appendix G: Procedures and Guidelines for the Use of AOAC Voluntary Consensus Standards to Evaluate Characteristics of a Method of Analysis, p.3 "*First Action to Final Action Methods: Guidance for AOAC Expert Review Panels.*"

ERP SUMMARY FOR FIRST TO FINAL ACTION METHOD RECOMMENDATION

AOAC 2012.25	**Residues of Three Triphenylmethane Dyes and Their Metabolites (Malachite Green, Leuco Malachite Green, Crystal Violet, and Brilliant Green) in Aquaculture Products** **Liquid Chromatography/Tandem Mass Spectrometry**

GUIDANCE FOR AOAC ERPS - APPENDIX G[1]	**Considered?**	**Comments/Reference if applicable**
Method Applicability	Yes	Triphenylmethane dyes as specified in applicability statement.
ERP First to Final Action recommendations & improvements implemented/addressed	Yes	
Draft Final Action method reviewed by ERP	Yes	
Safety Concerns	Yes	Completed and discussed during ERP meeting
Reference Materials	Yes	Currently no reference materials available for these types of drugs
Single Laboratory Validation	Yes	Hurtaud-Pessel et al., *J. AOAC Int*. **96**, 1152(2013) Andersen et al., *J. AOAC Int*. **98**, 636(2015) – modification – matrix extension
Reproducibility/Uncertainty and Probability of Detection	Yes	Schneider & Andersen *J. AOAC Int*. **98**, 658(2015)
Comparison to SMPR (SMPR criteria met?)	Yes	SMPR 2009.001 – SMPR for Quantitative Methods for Drug Residues in Shrimp, Tilapia, Catfish, and Salmon; SMPR criteria met according to ERP
Feedback from Users of Method	Yes	Discussed in ERP Meeting

DOCUMENTATION	**Available?**	**Comments**
Safety Evaluation	Yes	Completed; Discussed in ERP meeting
Reference Materials	No	None specified in SMPR; none available
SLV or PTMs	Yes	Hurtaud-Pessel et al., *J. AOAC Int*. **96**, 1152(2013) Andersen et al., *J. AOAC Int*. **98**, 636(2015)
Approved Validation Protocols	No	Used SMPR; OMA appendix D, and help from Chemical Contaminants Community subgroup
Statistics Review	Yes	Completed
Method Published in OMA	Yes	2012.25
Method Performance vs SMPR criteria	Yes	SMPR 2009.001 – SMPR for Quantitative Methods for Drug Residues in Shrimp, Tilapia, Catfish, and Salmon
Feedback Information	Yes	Discussed in ERP meeting
Additional Recognition(s)	No	
ERP Reports	Yes	10/2012; 12/2015
Manuscript(s) Published in JAOAC	Yes	Hurtaud-Pessel et al., *J. AOAC Int*. **96**, 1152(2013) Andersen et al., *J. AOAC Int*. **98**, 636(2015) Schneider & Andersen *J. AOAC Int*. **98**, 658(2015)

ERP Method Recommendation (Final Action/Repeal/Continuation)	**Final Action**	**Method scope expanded and the latest version of the method approved by ERP is in Collaborative Study Manuscript published in 2015 by Schneider and Andersen.**

ERP Recommendations

- Supply all documentation to AOAC by established deadline
  - Documentation includes ERP review details
- Representative from ERP present at OMB review meeting
- If method to be repealed, document reasoning

POST ERP MEETING

Post ERP Meeting

- An ERP report with the decisions of the ERP will be drafted
  - Review and approval by ERP chair
  - Posted on website within 15 business days after the ERP meeting
- AOAC staff will send notification to method authors/submitters regarding outcomes on specific methods

PUBLICATIONS

Publication of First Action Methods

- Any approved method(s) along with supporting manuscript(s) and documentation sent to AOAC Publications after the meeting.
  - AOAC Official Methods number assigned.
  - Method and method manuscript prepared for publication in the *Official Methods of Analysis of AOAC INTERNATIONAL* and in Journal of AOAC INTERNATIONAL
  - Updates on methods approved or status changes are published in the Inside Laboratory Management magazine and on the AOAC website

Format for AOAC *Official Methods of Analysis*

The language of the method should be concise and completely free from ambiguity. Conciseness is desirable, both to ensure clarity and to save space. Whenever there is a conflict between clarity and style, clarity is more important.

Present Tense and Imperative Mode

- Check sentences that do not begin with a verb and change them, if feasible, to the imperative mode (e.g. Pipet 10 mL..., Stir..., etc.). Exceptions are: use of adverb modifier ("Accurately weigh..."), prepositional clause ("For refined sugars, use..."), permissive statements ("Ferric hydroxide may be used..."), and statements in the "Principle" section.

Abbreviations

- Most abbreviations are the same as those used by Chemical Abstracts. Do not use abbreviations in titles and headings. See the *Definitions of Terms and Explanatory Notes*.

Repetition and Redundancy

- Eliminate repetition and redundancy as far as possible; use only for emphasis. Do not use "distilled" with water, "concentrated" with common acids, "95%" with alcohol, or "ACS" with reagents covered by ACS specifications. These are understood by definition.

Terminology, Formulae and Chemical Names

- For names of chemical compounds, use the spelling, hyphenation, and word division given in Chemical Abstracts. Use a national pharmacopeia for names for drugs. Use ISO nomenclature for pesticides and Codex nomenclature for names of food additives and color additives.

Consistency

- Watch for internal contradictions in the text: volumes that do not add up or that exceed the capacity of the container; too abrupt a transition from one operation to another (a line may be omitted); and impractical or impossible numbers (e.g., 100 g NaCl will not dissolve in 100 mL water).

Cross-references

- All new AOAC methods should be written as complete and self-contained as practical. Do not refer to other AOAC methods. If part of a procedure in an *Official Method*^{SM} is taken from material previously published elsewhere, incorporate those steps in the method rather than referring the analyst to another publication.

Definitions

- The section "Definition of Terms and Explanatory Notes," *Official Methods of Analysis of AOAC INTERNATIONAL*, is the basic guide to conventions and consistency.

Illustrations and Tables

- If symbols are used on the figure, include an explanation in the caption or text. Provide descriptive titles for tables. Explain any obscure headings in a footnote.

Bibliographic References

- Check all references for accuracy. Use standard Chemical Abstracts abbreviations for *Journal* titles. In general avoid references in method. Cite background references in the "Introduction" or "Discussion" section of the collaborative study manuscript -- not in the method. If part of a procedure in an *Official Method*^{SM} is taken from material previously published elsewhere, incorporate those steps in the method rather than referring the analyst to another publication.

Safety

- All methods must be reviewed for safety and potential hazards. Methods should automatically incorporate cross-references to the safety statement(s), or present questioned conditions to the attention of the Committee on Safety for resolution.
- Decisions regarding inclusion of safety statements should be practical, recognizing that overuse will be self-defeating.
- Methods that create toxic, obnoxious or environmentally hazardous fumes and wastes should contain practical directions for disposal.

Checking Edited Copy and Proofreading

- The author must review a copy of the original version and edited copy to ensure that there has been no change in meaning, to correct typographical errors, and to answer any questions posed by the editor. The author must review the typeset method for accuracy.

Revised October 2013

Online Technical Resources

Method Development, Optimization & Validation

- OMA - Appendix F - Guidelines for Standard Method Performance Requirements
- Homogeneity
- Guide for Writing Methods in AOAC Format
- Statistics Protocol Review Form
- OMA - Appendix D: Guidelines for Collaborative Study Procedures to Validate Characteristics of a Method of Analysis
- OMA - Appendix G: Procedures and Guidelines for the Use of AOAC Voluntary Consensus Standards to Evaluate Characteristics of a Method of Analysis
- OMA - Appendix I: AOAC INTERNATIONAL Methods Committee Guidelines for Validation of Biological Threat Agent Methods and/or Procedures
- OMA - Appendix J: AOAC INTERNATIONAL Methods Committee Guidelines for Validation of Microbiological Methods for Food and Environmental Surfaces
- OMA - Appendix K: Guidelines for Dietary Supplements and Botanicals
- OMA - Appendix L: AOAC Recommended Guidelines for Stakeholder Panel on Infant Formula and Adult Nutritionals (SPIFAN) Single-Laboratory Validation
- OMA - Appendix M - Validation Procedures for Quantitative Food Allergen ELISA Methods: Community Guidance and Best Practices
- Safety Checklist

Method Review

- Examples of Statistical Analysis
- Statistics Manuscript Review Form
- OMA - Appendix A: Standard Solutions and Reference Materials
- OMA - Appendix D: Guidelines for Collaborative Study Procedures to Validate Characteristics of a Method of Analysis
- OMA - Appendix H: Probability of Detection (POD) as a Statistical Model for the Validation of Qualitative Methods

Miscellaneous

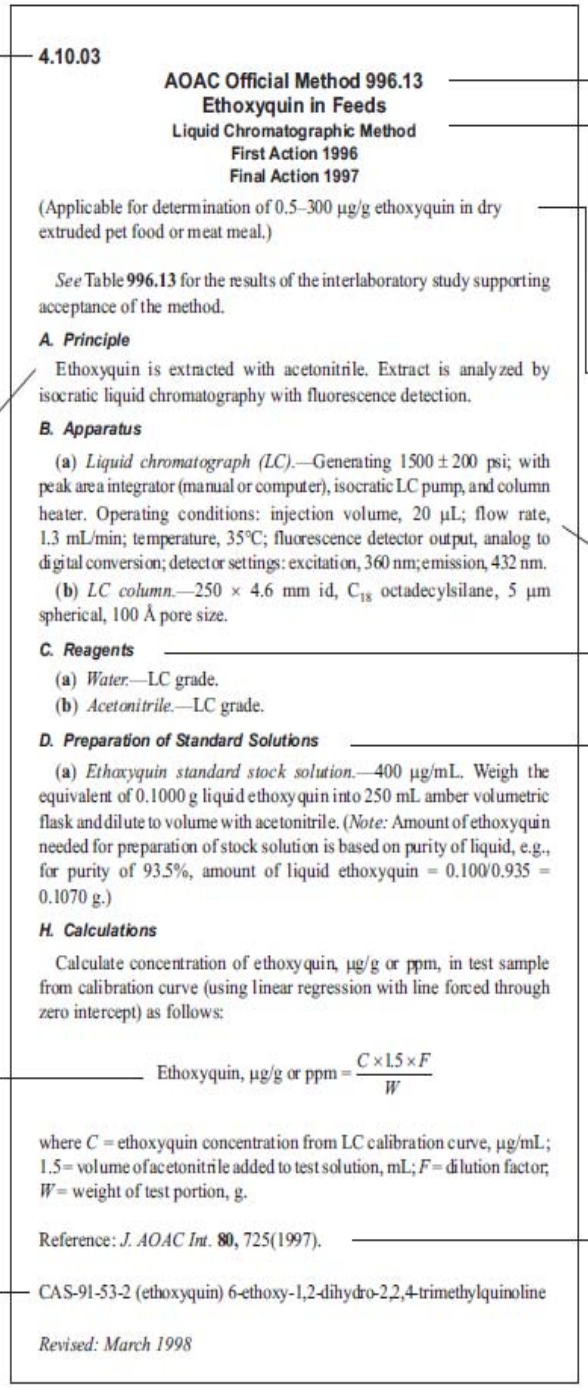
- Definition of Terms and Explanatory Notes
- OMA - Appendix B: Laboratory Safety
- OMA - Appendix E: Laboratory Quality Assurance
- OMA - Appendix C: Reference Tables

All resources are accessible at
http://www.aoac.org/vmeth/guidelines.htm

For questions, please contact:
P 301-924-7077 x157 E dmckenzie@aoac.org

Guide to Method Format

(Method shown is incomplete to allow space for description.)



4.10.03

AOAC Official Method 996.13
Ethoxyquin in Feeds
Liquid Chromatographic Method
First Action 1996
Final Action 1997

(Applicable for determination of 0.5–300 µg/g ethoxyquin in dry extruded pet food or meat meal.)

See Table **996.13** for the results of the interlaboratory study supporting acceptance of the method.

A. Principle

Ethoxyquin is extracted with acetonitrile. Extract is analyzed by isocratic liquid chromatography with fluorescence detection.

B. Apparatus

(a) *Liquid chromatograph (LC).*—Generating 1500 ± 200 psi; with peak area integrator (manual or computer), isocratic LC pump, and column heater. Operating conditions: injection volume, 20 µL; flow rate, 1.3 mL/min; temperature, 35°C; fluorescence detector output, analog to digital conversion; detector settings: excitation, 360 nm; emission, 432 nm.

(b) *LC column.*—250 × 4.6 mm id, C_{18} octadecylsilane, 5 µm spherical, 100 Å pore size.

C. Reagents

(a) *Water.*—LC grade.

(b) *Acetonitrile.*—LC grade.

D. Preparation of Standard Solutions

(a) *Ethoxyquin standard stock solution.*—400 µg/mL. Weigh the equivalent of 0.1000 g liquid ethoxyquin into 250 mL amber volumetric flask and dilute to volume with acetonitrile. (*Note:* Amount of ethoxyquin needed for preparation of stock solution is based on purity of liquid, e.g., for purity of 93.5%, amount of liquid ethoxyquin = 0.100/0.935 = 0.1070 g.)

H. Calculations

Calculate concentration of ethoxyquin, µg/g or ppm, in test sample from calibration curve (using linear regression with line forced through zero intercept) as follows:

$$\text{Ethoxyquin, µg/g or ppm} = \frac{C \times 1.5 \times F}{W}$$

where C = ethoxyquin concentration from LC calibration curve, µg/mL; 1.5 = volume of acetonitrile added to test solution, mL; F = dilution factor; W = weight of test portion, g.

Reference: *J. AOAC Int.* **80**, 725(1997).

CAS-91-53-2 (ethoxyquin) 6-ethoxy-1,2-dihydro-2,2,4-trimethylquinoline

Revised: March 1998

Locator number identifies method by chapter, subchapter, and sequence within the subchapter for easy cross referencing and access. 4 = chapter 4; .10 = subchapter 10; .03 = the third method found in Chapter 4, subchapter 10. The locator number is not the permanent number and is included only for convenient accessibility.

Chemical names of pesticides and drugs are given at end of pertinent chapter.

Calculation symbols are identified and show correct units.

Chemical Abstracts Service Registry Number. A unique identifier that may be used to search a number of data-retrieval systems.

Method number identifies method by year of adoption or first appearance in *Official Methods of Analysis of AOAC INTERNATIONAL*. 996 = First Action 1996; .13 = sequence of adoption in 1996.

Title may include analyte and matrix, type of method, and official status.

Applicability statement addresses utility and limitations on use of method or other information.

Specifications for necessary laboratory apparatus and reagent preparations. *See also Definition of Terms and Explanatory Notes.*

Method may be divided into several descriptive sections.

References direct the user to the published collaborative study and any subsequent revisions in the method. Other informative references may be included.

The AOAC style used for preparing methods for publication in the *Official Methods of Analysis of AOAC INTERNATIONAL* includes the following essentials:

- ✓ Standardized format that follows the order of laboratory operations.
- ✓ Use of the imperative mode.
- ✓ Cross-references to identical reagents, apparatus, and operations.
- ✓ Use of standardized definitions, terminology, and style.
- ✓ Use of accepted abbreviations and simplifications.
- ✓ Use of SI units
- ✓ Methods should be written as complete and self-contained as practical.
- ✓ Normality should be referred in terms of Molarity.
- ✓ ppm should be changed to mg/kg or mg/L
- ✓ ppb should be changed to ng/g or ng/mL
- ✓ ppt should be changed to pg/g or pg/mL

FORMAT OF AOAC® *OFFICIAL METHODS of ANALYSIS OF AOAC INTERNATIONAL*

Title:
- ❖ Includes analyte being determined, type of matrix (matrices), and analytical technique used for analysis.

Applicability:
- ❖ Includes list of matrix(es) along with specific matrix types and range or limits of determination or detection.

Precautions:
- ❖ Makes an analyst aware of hazardous materials used in analysis.

Data Collection:
- ❖ Table(s) that presents performance parameters including matrices tested in a collaborative study, levels of analyte(s), % recovery, RSD_r, RSD_R, s_r, s_R, HORRAT, number of observations, etc

Principle:
- ❖ Explains scientific premise on which the method is operates specifically the mechanism of the analysis.

Apparatus:
- ❖ Lists the equipment that requires assembly or that has specifications critical to the method performance. Describe equipment in terms of performance characteristics.

Reagents:
- ❖ List the reagents with amounts and appropriate units needed to conduct the analysis and describe the reagents in terms of performance characteristics.

Sample and Test Portion Preparation:
- ❖ Describe the preparation of samples and the test portion.

Determination:
- ❖ Describes the actual analysis.

Calculations:
- ❖ Section that explains how to calculate final results; presented in a form of equation or description.

Other sections as needed

REFERENCING *AOAC® OFFICIAL METHODS*℠

When referencing *AOAC® Official Methods*℠, only the method number should be used as seen in the following example:

(1) *Official Methods of Analysis of AOAC INTERNATIONAL* (2012) 19th Ed., AOAC INTERNATIONAL, Gaithersburg, MD, USA, Official Method **2008.01**

Revised October 2013

Publication of First Action Methods

NO OMA NUMBER ASSIGNED

UNTIL ALL DOCUMENTATION SUBMITTED

1. Method incorporating ERP revisions (preferably in AOAC Format)

2. Method Manuscript incorporating ERP revisions (in AOAC Format)

3. Signed AOAC Copyright Authorization form

DOCUMENTATION

Reports and Documentation

- AOAC staff or designee will capture the decisions and action items into an ERP report.
- The draft report will be sent back to the ERP Chair whose responsibility it is to sign off on the report once approved.
- The report is then distributed to the ERP.
- ERP is responsible for a drafting a written recommendation to the OMB for each method at a maximum of two years following adoption as First Action OMA
- Approved methods from the ERP meetings are published in the OMA and in the *Journal of AOAC INTERNATIONAL*.
- Meeting overviews are published in the AOAC *Inside Laboratory Management* magazine.

SUMMARY OF RESPONSIBILITIES

Roles and Responsibilities

- Expert Review Panel:
  - Review methods and meet in person to discuss and render decisions on methods for First Action *Official Methods* status.
  - Track First Action *Official Methods*
  - Modify First Action methods if necessary
  - Make recommendations on First Action methods no more than 2 years after adoption to OMB.
- Official Methods Board:
  - Vet and approve ERP membership
  - Assign OMB liaison to be a resource to the ERP
  - Review ERP recommendations and render decisions (*Final Action, Repeal or remain First Action*) on First Action OMAs
- AOAC Staff
  - Coordinate the ERP and meetings, facilitate reviews, document ERP actions/decisions.
  - Issue necessary calls for experts and methods

Expert Review Panels

The ERPs review and approve appropriate methods (as submitted or modified) for adoption as First Action Official Methods or for further validation. ERPs also make recommendations regarding Final Action Official Methods status.

Expert Review Panels

- Must be supported by relevant stakeholders.
- Constituted for the review of methods, not for Standard Method Performance Requirements (SMPR) purposes or as an extension of a Working Group.
- Consist of a minimum of seven (7) members representing a balance of expert stakeholders. **Quorum is a minimum of 7 members present or 2/3 of the total vetted members, whichever is greater.**
- ERP constituency must be approved by the Official Methods Board (OMB).
- Holds transparent public meetings only.
- Remains in force as long as method in First Action Status.

First Action Official Method Status decision

- Must be made by an ERP constituted or reinstated post 2011-03-28 for First Action Official Method Approval (FAOMA).
- Must be made by an ERP vetted for FAOMA purposes by OMB post 2011-03-28.
- Method adopted by ERP must perform adequately against the SMPR set forth by the stakeholders. Or demonstrate performance or characteristics that meet the scope, applicability and/or claims of the method.
- Method must be adopted by unanimous decision of ERP on first ballot, If not unanimous, negative votes must delineate scientific reasons.
- Negative voter(s) can be overridden by 2/3 of non-negative voting ERP members after due consideration
- Method becomes First Action Official Methods on date when ERP decision is made.
- Methods to be drafted into AOAC format by a knowledgeable AOAC staff member or designee in collaboration with the ERP and method author.
- Report of FAOMS decision complete with ERP report regarding decision including scientific background (references etc) to be published concurrently with method in traditional AOAC publication venues.

Method in First Action Status and Transitioning to Final Action Status

- Further data indicative of adequate method reproducibility (between laboratory) performance to be collected. Data may be collected via a collaborative study or by proficiency or other testing data of similar magnitude.
- Two years maximum transition time (additional year(s) if ERP determines a relevant collaborative study or proficiency or other data collection is in progress).
- Method removed from First Action Official Methods and OMA if no evidence of method use available at the end of the transition time.
- Method removed from First Action Official Methods and OMA if no data indicative of adequate method reproducibility is forthcoming as outlined above at the end of the transition time.
- ERP to recommend Method to Official Final Action Status to the OMB.
- OMB decision on First to Final Action Status

Revised October 2013

Online Technical Resources

Method Development, Optimization & Validation

- OMA - Appendix F - Guidelines for Standard Method Performance Requirements
- Homogeneity
- Guide for Writing Methods in AOAC Format
- Statistics Protocol Review Form
- OMA - Appendix D: Guidelines for Collaborative Study Procedures to Validate Characteristics of a Method of Analysis
- OMA - Appendix G: Procedures and Guidelines for the Use of AOAC Voluntary Consensus Standards to Evaluate Characteristics of a Method of Analysis
- OMA - Appendix I: AOAC INTERNATIONAL Methods Committee Guidelines for Validation of Biological Threat Agent
- Methods and/or Procedures
- OMA - Appendix J: AOAC INTERNATIONAL Methods Committee Guidelines for Validation of Microbiological Methods for Food and Environmental Surfaces
- OMA - Appendix K: Guidelines for Dietary Supplements and Botanicals
- OMA - Appendix L: AOAC Recommended Guidelines for Stakeholder Panel on Infant Formula and Adult Nutritionals (SPIFAN) Single-Laboratory Validation
- OMA - Appendix M - Validation Procedures for Quantitative Food Allergen ELISA Methods: Community Guidance and Best Practices
- Safety Checklist

Method Review

- Examples of Statistical Analysis
- Statistics Manuscript Review Form
- OMA - Appendix A: Standard Solutions and Reference Materials
- OMA - Appendix D: Guidelines for Collaborative Study Procedures to Validate Characteristics of a Method of Analysis
- OMA - Appendix H: Probability of Detection (POD) as a Statistical Model for the Validation of Qualitative Methods

Miscellaneous

- Definition of Terms and Explanatory Notes
- OMA - Appendix B: Laboratory Safety
- OMA - Appendix E: Laboratory Quality Assurance
- OMA - Appendix C: Reference Tables

All resources are accessible at
http://www.aoac.org/vmeth/guidelines.htm

For questions, please contact:
P 301-924-7077 x157 E dmckenzie@aoac.org

About Expert Review Panels (ERPs)

ERP OVERVIEW:

An Expert Review Panel (ERP) is assembled to review and adopt methods as Official First Action. ERPs will track Official Methods for two years or until such time as reproducibility has been demonstrated and cumulative feedback on method use and performance are obtained. ERPs will make a recommendation regarding Final Action method status for all OMAs to the Official Methods Board (OMB).

All ERP members are expected to serve with the highest integrity and without direct or indirect conflicts of interest. A method assignment can last two years. All members of the ERP are expected to actively participate in ERP meetings and to perform duties and reviews in timely fashion. All members should maintain strict adherence to review timelines and deadlines. AOAC staff documents ERP deliberations.

ESTABLISHING AN EXPERT REVIEW PANEL:

- AOAC staff issues a Call for Experts:
  - Based on voluntary consensus standards and methods submitted to AOAC INTERNATIONAL that may meet the standards.
  - Proprietary and sole source method developers submit individual methods to the AOAC Research Institute.
  - Candidates are asked to submit a CV or information that demonstrates expertise to AOAC staff if not already part of a recognized pool of experts.
- AOAC Chief Scientific Officer (CSO) reviews the documentation for the candidates and make recommends a slate for an expert review panel including the chair to the Official Methods Board.
- The candidate list and supporting documentation are forwarded to the Chair of the OMB who will assign the review to at least two OMB members.
- The OMB reviewers will review the candidates for expertise and perceived conflicts of interest and the OMB may then approve the members of the ERP. A Chair for the ERP is also approved.

EXPERT REVIEW PANEL (ERP):

- Review, discuss and demonstrate consensus on methods for Official First Action method status.
- Participate in the publications process of First Action methods.
- Track and discuss feedback all First Action methods for two years.
- Reach and demonstrate consensus on recommendations for Final Action method status.
- Actively participate in the broader stakeholder effort.

ERP CHAIR:

- Lead ERP discussions in the review and adoption of methods for First Action Official Methods.
- Participate in stakeholder panel activities.
- Review and approve ERP report.
- Work with AOAC staff, working groups and other stakeholder panels to ensure a thorough understanding of the standard method performance requirements and the methods to be assessed.
- Implement the OMB First Action to Final Action Guidelines with the ERP members.
- Advise and review First Action methods and post First Action publications.
- Represent the ERP in presenting the ERPs recommendation to the Official Methods Board regarding Final Action method status.

MECHANICS OF AN AOAC EXPERT REVIEW PANEL

- AOAC CSO assigns methods for review to the expert review panel members.
- For each method, 2 ERP members are assigned as primary and secondary reviewers and present at the ERP meeting.
- All members are expected to actively participate and review methods for First Action Official Method status - conducting thorough and prompt review of methods and being prepared to speak on assigned methods at ERP meetings
- The ERP chair and the 2 reviewers for each method are expected to participate in the publications peer review process for First Action methods.
- ERP reviewers track assigned methods that were adopted as First Action Official Methods and update ERP on method use during two year period between First Action and Final Action
  ERP members are expected to participant in the stakeholder panel activities and/or community at large .

- *ERPs can work with topic advisors (aka, subject matter experts)*
- *OMB can recognize a pool of experts from which ERP members can be selected*

Eligibility Criteria for Expert Reviewers

Be a key expert and/or thought leader of the method or priority under consideration.

- Demonstrated knowledge in the appropriate scientific disciplines.
- Demonstrated knowledge regarding data relevant to adequate method performance.
- Demonstrated knowledge of practical application of analytical methods to bona fide diagnostic requirements.

Be approved by the Official Methods Board

- Qualifications must be clearly described and submitted to AOAC headquarters.

Duties of Expert Reviewers

Members of the Pool of Experts will be called upon to serve on ERPs as needed and to review documents .These documents may include:

- Procedural documents on how methods will be selected and how single laboratory validation studies will be done;
- Methods submitted for consideration as First Action Official Methods;
- Methods submitted for selection for further validation studies;
- Protocols to be used for single laboratory validation studies;
- Selection of methods to be considered for full collaborative studies; and
- Validation study reports

Revised October 2013

Questions?

Thank you.

ARS-01

Method Title: Analytical Method for the Determination of Various Arsenic Species in Rice, Rice Food Products, Apple Juice, and Other Juices by Ion Chromatography-Inductively Coupled Plasma/Mass Spectrometry

Submitted by: David Ellingson

Submitted by Email: david.ellingson@covance.com

Enclosures: 0

Submitter notes:

Covance method that was recently published for arsenic speciation in rice/rice products/juice.

Reviewers: Cory Murphy, Jenny Nelson, Sneh Bhandari

Link to Method:
http://www.aoac.org/iMIS15_Prod/AOAC_Member/SH/SPSFAMCF/SPSFAM_ERPHM.aspx

The Scientific Association Dedicated to Analytical Excellence®

AOAC INTERNATIONAL Standards Development:
First Action Method Review Form

Name of Reviewer:

Cory Murphy

Title of Method:

Analytical Method for the Determination of Varie

AOAC Candidate Method Number:

ARS-01

Applicable SMPR:

2015.001

I. Summary of Method

The method as described seems relatively simple and quick. It uses nitric acid acid to extract arsenic species (As(+3), As(+5), DMA, MMA, AsC). The publication was easy to read and understand and included supporting data to shows it applicability in rice and rice based matrices as well as fruit juice. The method also separates both inorganic arsenic species which I feel is important and useful as opposed to only a total inorganic arsenic value.

II. Review of the Method Only

1. Does the applicability of the method support the applicability of the SMPR? If not, please explain what is missing.

The method showed applicability in rice, rice based foods, and fruit juices. It did not show applicability in seafood matrices, as described in the SMPR. The analytes discussed in the method also match the SMPR.

2. Does the analytical technique(s) used in the method meet the SMPR? If not, please specify how it differs from what is stated in the SMPR.

Yes. The method uses IC-ICP-MS which matches the SMPR preferred analytical technique.

3. Are definitions specified in the SMPR used and applied appropriately in the method? If not, please indicate how the terms are used.

Yes.

4. Does the method, as written, contain all appropriate precautions and warnings related to the method's reagents, components, instrumentation, or method steps that may be hazardous? If not, please suggest options.

The supplied method was a JAOAC publication, therefore, it did not have much detail on safety related information as this information is not typically required for such a publication. It this method is to be accepted as First Action status, the authors may need to expand into some of the safety requirements for use of concentrated acids, ICP-MS safety information, etc.

III. Review of Information in Support of the Method

1. Are the definitions specified in the SMPR used and applied appropriately in the supporting documentation (manuscripts, method studies, etc...)? If no, please explain differences and if the method is impacted by the difference.

Yes.

2. Is there information demonstrating that the method meets the SMPR Method Performance Requirements table? If not. for any of the parameters in the SMPR Method Performance Requirements table, then please explain what is missing and the impact on performance of the method.

I feel the method meets the SMPR Method Performance Requirements for rice and rice based foods as well as juice, but not seafood products. There was no reproducibility data presented in the publication, however, this is reasonable as it was a single lab experiment. I do have a question on the analytical range....I didn't really notice anything in the publication stating the analytical range and how it meets the SMPR. Can the authors clarify this?

3. Is there information demonstrating that the method performs within the SMPR Method Performance Requirements using the Reference Materials stated in the SMPR? If not, then specify what is missing and how this impacts demonstration of performance of the method.

Yes, use of reference materials was noted in the method and shown that the method works.

4. Is there information demonstrating that the method performs within the SMPR Method Performance Requirements table specification for all analytes in the SMPR applicability statement? If not, please specify what is missing and whether or not the method's applicability should be modified.

Yes.

IV. General Submission Package

1. Based on the supporting information, were there any additional steps in the evaluation of the method that indicated the need for any additional precautionary statements in the method?

No.

2. Does the method contain system suitability tests or controls as specified by the SMPR? If no, please indicate if there is a need for such tests or controls and which ones.

I do not believe the method had a "0 concentration" standard included in the calibration curve. Also, I do not recall the method discussing a reagent blank taken through the method. I could have missed this information, but for the most part the method meets this criteria.

3. Is there information demonstrating that the method system suitability tests and controls as specified in the SMPR worked appropriately and as expected? If not, please specify.

No.

4. Based on the supporting information, is the method written clearly and concisely? If not, please specify the needed revisions.

Yes, it was very clear and concise.

IV. General Submission Package (continued)

5. Based on the supporting information, what are pros/strengths of the method?

Pros: Quick, simple extraction method, speciates the analytes of concern including the inorganic species, chromatography appears to work well, ammonium carbonate mobile phase is best for running a large volume of samples with respect to instrument maintenance, method used CRMs, method directly measures the analytes of concern.

6. Based on the supporting information, what are the cons/weaknesses of the method?

Cons: The method discusses some conversion of the inorganic species which is a bit of problem for all scientists working on speciated arsenic methods, didn't try the method in seafood.

7. General comments about the submission:

I feel this is a good method and has merit. I would vote for this method as first action for the applicable matrices discussed.

V. Recommendation for the Method

Do you recommend this method be adopted as a First Action and published in the Official Methods of Analysis of AOAC INTERNATIONAL? *If not, please specify rationale.*

Yes for the matrices discussed in the publication only.

The Scientific Association Dedicated to Analytical Excellence®

AOAC INTERNATIONAL Standards Development:
First Action Method Review Form

Name of Reviewer:

Jenny Nelson

Title of Method:

Analytical Method for the Determination of Vario

AOAC Candidate Method Number:

ARS01

Applicable SMPR:

SPSFAM

I. Summary of Method

This method uses IC-ICP-MS for the detection of various Arsenic species (AsB, AsC, MMA, DMA, AsIII, AsV) in Rice, Rice products, and juices. This method stresses the importance of seperating the 2 inorganic As peaks AsIII and AsV. This method does not combine the inorganic As peaks like some other common methods.

II. Review of the Method Only

1. Does the applicability of the method support the applicability of the SMPR? If not, please explain what is missing.

This method is applicable to the SMPR. However, I have issues with the AsB detection in this method, as well as the As5 background. I also feel that this method is not as well laid out as the FDA method, and other As speciation methods submitted.

2. Does the analytical technique(s) used in the method meet the SMPR? If not, please specify how it differs from what is stated in the SMPR.

anaylitcal techqnies are fine.

3. Are definitions specified in the SMPR used and applied appropriately in the method? If not, please indicate how the terms are used.

they are fine

4. Does the method, as written, contain all appropriate precautions and warnings related to the method's reagents, components, instrumentation, or method steps that may be hazardous? If not, please suggest options.

The method states that they had consistant contamination level of As 5. This worries me. I would have figured out the source of this contamination and then proceeded with method development. This is not something that I have seen doing this type of method. Where all starting materials checked?

III. Review of Information in Support of the Method

1. Are the definitions specified in the SMPR used and applied appropriately in the supporting documentation (manuscripts, method studies, etc...)? If no, please explain differences and if the method is impacted by the difference.

they are fine

2. Is there information demonstrating that the method meets the SMPR Method Performance Requirements table? If not. for any of the parameters in the SMPR Method Performance Requirements table, then please explain what is missing and the impact on performance of the method.

They used a blank to determine the LOD, did they also do this with a low level standard?

3. Is there information demonstrating that the method performs within the SMPR Method Performance Requirements using the Reference Materials stated in the SMPR? If not, then specify what is missing and how this impacts demonstration of performance of the method.

this is fine

4. Is there information demonstrating that the method performs within the SMPR Method Performance Requirements table specification for all analytes in the SMPR applicability statement? If not, please specify what is missing and whether or not the method's applicability should be modified.

this is fine

IV. General Submission Package

1. Based on the supporting information, were there any additional steps in the evaluation of the method that indicated the need for any additional precautionary statements in the method?

As stated in II.1

2. Does the method contain system suitability tests or controls as specified by the SMPR? If no, please indicate if there is a need for such tests or controls and which ones.

this is fine

3. Is there information demonstrating that the method system suitability tests and controls as specified in the SMPR worked appropriately and as expected? If not, please specify.

this is fine

4. Based on the supporting information, is the method written clearly and concisely? If not, please specify the needed revisions.

Yes, this method is very well written, very easy to follow.

IV. General Submission Package (continued)

5. Based on the supporting information, what are pros/strengths of the method?

Strength- both rice and juice are run on the same method, do not need 2 different calibrations and methods

6. Based on the supporting information, what are the cons/weaknesses of the method?

AsB peak is split. This needs to be resolved. As5 Background not acceptable.

7. General comments about the submission:

First, the entire method uses the acryonom ICP/MS. That is wrong, it should be ICP-MS. I am surprised this was published like that?

V. Recommendation for the Method

Do you recommend this method be adopted as a First Action and published in the Official Methods of Analysis of AOAC INTERNATIONAL? *If not, please specify rationale.*

NO, they need to figure out the AsV contamination and run again. The authors does not provide the BEC for As(V) in the blank. Since their calibration is forced through the blank, the blank is subtracted from the sample peak areas. There needs to be more explanation into what QC measures are in place if the blank concentrations change throughout the run. Also, there are no internal standards used.

The Scientific Association Dedicated to Analytical Excellence®

AOAC INTERNATIONAL Standards Development:
First Action Method Review Form

Name of Reviewer:

Jenny Nelson

Title of Method:

Analytical Method for the Determination of Vario

AOAC Candidate Method Number:

ARS01

Applicable SMPR:

SPSFAM

I. Summary of Method

This method uses IC-ICP-MS for the detection of various Arsenic species (AsB, AsC, MMA, DMA, AsIII, AsV) in Rice, Rice products, and juices. This method stresses the importance of seperating the 2 inorganic As peaks AsIII and AsV. This method does not combine the inorganic As peaks like some other common methods.

II. Review of the Method Only

1. Does the applicability of the method support the applicability of the SMPR? If not, please explain what is missing.

This method is applicable to the SMPR. However, I have issues with the AsB detection in this method, as well as the As5 background. I also feel that this method is not as well laid out as the FDA method, and other As speciation methods submitted.

2. Does the analytical technique(s) used in the method meet the SMPR? If not, please specify how it differs from what is stated in the SMPR.

anaylitcal techqnies are fine.

3. Are definitions specified in the SMPR used and applied appropriately in the method? If not, please indicate how the terms are used.

they are fine

4. Does the method, as written, contain all appropriate precautions and warnings related to the method's reagents, components, instrumentation, or method steps that may be hazardous? If not, please suggest options.

The method states that they had consistant contamination level of As 5. This worries me. I would have figured out the source of this contamination and then proceeded with method development. This is not something that I have seen doing this type of method. Where all starting materials checked?

III. Review of Information in Support of the Method

1. Are the definitions specified in the SMPR used and applied appropriately in the supporting documentation (manuscripts, method studies, etc...)? If no, please explain differences and if the method is impacted by the difference.

they are fine

2. Is there information demonstrating that the method meets the SMPR Method Performance Requirements table? If not. for any of the parameters in the SMPR Method Performance Requirements table, then please explain what is missing and the impact on performance of the method.

They used a blank to determine the LOD, did they also do this with a low level standard?

3. Is there information demonstrating that the method performs within the SMPR Method Performance Requirements using the Reference Materials stated in the SMPR? If not, then specify what is missing and how this impacts demonstration of performance of the method.

this is fine

4. Is there information demonstrating that the method performs within the SMPR Method Performance Requirements table specification for all analytes in the SMPR applicability statement? If not, please specify what is missing and whether or not the method's applicability should be modified.

this is fine

IV. General Submission Package

1. Based on the supporting information, were there any additional steps in the evaluation of the method that indicated the need for any additional precautionary statements in the method?

As stated in II.1

2. Does the method contain system suitability tests or controls as specified by the SMPR? If no, please indicate if there is a need for such tests or controls and which ones.

this is fine

3. Is there information demonstrating that the method system suitability tests and controls as specified in the SMPR worked appropriately and as expected? If not, please specify.

this is fine

4. Based on the supporting information, is the method written clearly and concisely? If not, please specify the needed revisions.

Yes, this method is very well written, very easy to follow.

IV. General Submission Package (continued)

5. Based on the supporting information, what are pros/strengths of the method?

Strength- both rice and juice are run on the same method, do not need 2 different calibrations and methods

6. Based on the supporting information, what are the cons/weaknesses of the method?

AsB peak is split. This needs to be resolved. As5 Background not acceptable.

7. General comments about the submission:

First, the entire method uses the acryonom ICP/MS. That is wrong, it should be ICP-MS. I am surprised this was published like that?

V. Recommendation for the Method

Do you recommend this method be adopted as a First Action and published in the Official Methods of Analysis of AOAC INTERNATIONAL? *If not, please specify rationale.*

NO, they need to figure out the AsV contamination and run again. The authors does not provide the BEC for As(V) in the blank. Since their calibration is forced through the blank, the blank is subtracted from the sample peak areas. There needs to be more explanation into what QC measures are in place if the blank concentrations change throughout the run. Also, there are no internal standards used.

ARS-02

Method Title: High Performance Liquid Chromatography-Inductively Coupled Plasma-Mass Spectrometric Determination of Four Arsenic Species in Fruit Juice

Submitted by: Kevin Kubachka, FDA

Submitted by Email: Kevin.Kubachka@fda.hhs.gov

Enclosures: 2

Submitter notes:

I have attached a method and a multi-lab validation package for arsenic speciation in juice – which is the US FDA's EAM 4.10 method.

Reviewers: Farzaneh Maniei, Christopher Smith, Sneh Bhandari

Link to Method:
http://www.aoac.org/iMIS15_Prod/AOAC_Member/SH/SPSFAMCF/SPSFAM_ERPHM.aspx

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AOAC INTERNATIONAL Standards Development:
First Action Method Review Form

Name of Reviewer:

Jenny Nelson

Title of Method:

Analytical Method for the Determination of Vario

AOAC Candidate Method Number:

ARS01

Applicable SMPR:

SPSFAM

I. Summary of Method

This method uses IC-ICP-MS for the detection of various Arsenic species (AsB, AsC, MMA, DMA, AsIII, AsV) in Rice, Rice products, and juices. This method stresses the importance of seperating the 2 inorganic As peaks AsIII and AsV. This method does not combine the inorganic As peaks like some other common methods.

II. Review of the Method Only

1. Does the applicability of the method support the applicability of the SMPR? If not, please explain what is missing.

This method is applicable to the SMPR. However, I have issues with the AsB detection in this method, as well as the As5 background. I also feel that this method is not as well laid out as the FDA method, and other As speciation methods submitted.

2. Does the analytical technique(s) used in the method meet the SMPR? If not, please specify how it differs from what is stated in the SMPR.

anaylitcal techqnies are fine.

3. Are definitions specified in the SMPR used and applied appropriately in the method? If not, please indicate how the terms are used.

they are fine

4. Does the method, as written, contain all appropriate precautions and warnings related to the method's reagents, components, instrumentation, or method steps that may be hazardous? If not, please suggest options.

The method states that they had consistant contamination level of As 5. This worries me. I would have figured out the source of this contamination and then proceeded with method development. This is not something that I have seen doing this type of method. Where all starting materials checked?

III. Review of Information in Support of the Method

1. Are the definitions specified in the SMPR used and applied appropriately in the supporting documentation (manuscripts, method studies, etc...)? If no, please explain differences and if the method is impacted by the difference.

they are fine

2. Is there information demonstrating that the method meets the SMPR Method Performance Requirements table? If not. for any of the parameters in the SMPR Method Performance Requirements table, then please explain what is missing and the impact on performance of the method.

They used a blank to determine the LOD, did they also do this with a low level standard?

3. Is there information demonstrating that the method performs within the SMPR Method Performance Requirements using the Reference Materials stated in the SMPR? If not, then specify what is missing and how this impacts demonstration of performance of the method.

this is fine

4. Is there information demonstrating that the method performs within the SMPR Method Performance Requirements table specification for all analytes in the SMPR applicability statement? If not, please specify what is missing and whether or not the method's applicability should be modified.

this is fine

IV. General Submission Package

1. Based on the supporting information, were there any additional steps in the evaluation of the method that indicated the need for any additional precautionary statements in the method?

As stated in II.1

2. Does the method contain system suitability tests or controls as specified by the SMPR? If no, please indicate if there is a need for such tests or controls and which ones.

this is fine

3. Is there information demonstrating that the method system suitability tests and controls as specified in the SMPR worked appropriately and as expected? If not, please specify.

this is fine

4. Based on the supporting information, is the method written clearly and concisely? If not, please specify the needed revisions.

Yes, this method is very well written, very easy to follow.

IV. General Submission Package (continued)

5. Based on the supporting information, what are pros/strengths of the method?

Strength- both rice and juice are run on the same method, do not need 2 different calibrations and methods

6. Based on the supporting information, what are the cons/weaknesses of the method?

AsB peak is split. This needs to be resolved. As5 Background not acceptable.

7. General comments about the submission:

First, the entire method uses the acryonom ICP/MS. That is wrong, it should be ICP-MS. I am surprised this was published like that?

V. Recommendation for the Method

Do you recommend this method be adopted as a First Action and published in the Official Methods of Analysis of AOAC INTERNATIONAL? *If not, please specify rationale.*

NO, they need to figure out the AsV contamination and run again. The authors does not provide the BEC for As(V) in the blank. Since their calibration is forced through the blank, the blank is subtracted from the sample peak areas. There needs to be more explanation into what QC measures are in place if the blank concentrations change throughout the run. Also, there are no internal standards used.

The Scientific Association Dedicated to Analytical Excellence®

AOAC INTERNATIONAL Standards Development:
First Action Method Review Form

Name of Reviewer:

Jenny Nelson

Title of Method:

Analytical Method for the Determination of Vario

AOAC Candidate Method Number:

ARS01

Applicable SMPR:

SPSFAM

I. Summary of Method

This method uses IC-ICP-MS for the detection of various Arsenic species (AsB, AsC, MMA, DMA, AsIII, AsV) in Rice, Rice products, and juices. This method stresses the importance of seperating the 2 inorganic As peaks AsIII and AsV. This method does not combine the inorganic As peaks like some other common methods.

II. Review of the Method Only

1. Does the applicability of the method support the applicability of the SMPR? If not, please explain what is missing.

This method is applicable to the SMPR. However, I have issues with the AsB detection in this method, as well as the As5 background. I also feel that this method is not as well laid out as the FDA method, and other As speciation methods submitted.

2. Does the analytical technique(s) used in the method meet the SMPR? If not, please specify how it differs from what is stated in the SMPR.

anaylitcal techqnies are fine.

3. Are definitions specified in the SMPR used and applied appropriately in the method? If not, please indicate how the terms are used.

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The method states that they had consistant contamination level of As 5. This worries me. I would have figured out the source of this contamination and then proceeded with method development. This is not something that I have seen doing this type of method. Where all starting materials checked?

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they are fine

2. Is there information demonstrating that the method meets the SMPR Method Performance Requirements table? If not. for any of the parameters in the SMPR Method Performance Requirements table, then please explain what is missing and the impact on performance of the method.

They used a blank to determine the LOD, did they also do this with a low level standard?

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Yes, this method is very well written, very easy to follow.

IV. General Submission Package (continued)

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Strength- both rice and juice are run on the same method, do not need 2 different calibrations and methods

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AsB peak is split. This needs to be resolved. As5 Background not acceptable.

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V. Recommendation for the Method

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The Scientific Association Dedicated to Analytical Excellence®

AOAC INTERNATIONAL Standards Development:
First Action Method Review Form

Name of Reviewer:

Jenny Nelson

Title of Method:

Analytical Method for the Determination of Vario

AOAC Candidate Method Number:

ARS01

Applicable SMPR:

SPSFAM

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ARS-03

Speciation and Determination of Inorganic Arsenic in Rice Using Liquid Chromatography-Inductively Coupled Plasma/Mass Spectrometry: Collaborative Study

Submitted by: Takanori Ukena

Submitted by Email: ukenataka@gmail.com

Enclosures: 3

Submitter notes:

In response to the call for methods for the quantification of arsenic species in selected foods and beverages, I would like to submit our method internationally validated for speciation and determination of inorganic arsenic in rice (husked rice and polished rice of both indica and japonica subspecies) using liquid chromatography with inductively coupled plasma-mass spectrometry.
Please find attached four electronic files: the report of collaborative study of this method published in the Journal of AOAC INTERNATIONAL, Vol. 97, No. 3(2014): 946 – 955; the SOP of this method; the archive file list; and the analytical results obtained during the collaborative study presented in the AOAC Interlaboratory Study Workbook format.
We developed the method with specific emphasis on rice as consumed in Asia, not on rice products, because: (1) in the Codex Alimentarius Commission, the source directed measures, management of environmental contaminants in upper food chain, are considered to be more effective than the control of processed products; and (2) rice is mostly consumed in the form of polished rice after cooking in water or steaming in Asia; and (3) rice is one of the most traded agricultural produce in the world. For the second reason, we involved some Asian countries in international validation of this method.
The international validation was successfully completed with sixteen laboratories participating from 4 countries, Indonesia, Singapore Thailand and Japan for analysis of arsenite (As(III)), arsenate (As(V)), monomethylarsonic acid (MMA) and dimethyl arsenic acid (DMA) in husked rice and polished rice of both japonica and indica subspecies.
Applicability of the method to both subspecies and to both husked and polished rice was confirmed. For analysis of total inorganic arsenic (sum of As(III) and As(V)), limit of quantification was estimated to be 0.02 mg/kg, reproducibility relative standard deviation (RSD_R) was in a range of 10 to 36% at five concentrations between 0.03 and 0.68 mg/kg.
We believe that this validated method for speciation and determination of inorganic arsenic in rice will be useful for the government authorities in the world, in particular in Asia, and will be sufficient for an AOAC First Action method.

Reviewers: Min Huang, Darryl Sullivan, Sneh Bhandari

Link to Method: http://www.aoac.org/iMIS15_Prod/AOAC_Member/SH/SPSFAMCF/SPSFAM_ERPHM.aspx

The Scientific Association Dedicated to Analytical Excellence®

AOAC INTERNATIONAL Standards Development:
First Action Method Review Form

Name of Reviewer:

Jenny Nelson

Title of Method:

Analytical Method for the Determination of Vario

AOAC Candidate Method Number:

ARS01

Applicable SMPR:

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ARS-04

Arsenic Speciation Analysis in Beverages and Rice Based Products
Ion Chromatography-Inductively Coupled Plasma–Mass Spectrometry

Submitted by: Russ Gerads

Submitted by Email: Russ@brooksapplied.com

Enclosures: 1

Submitter notes:

Attached is my response to the SMPR_2015_006 associated with arsenic speciation analysis.

Reviewers: Bill Mindak, Jenny Scifres, Sneh Bhandari

Link to Method:
http://www.aoac.org/iMIS15_Prod/AOAC_Member/SH/SPSFAMCF/SPSFAM_ERPHM.aspx

Expert Review Panel – Method Review Form

Evaluation of Method # ARS--04

Title: Arsenic Speciation Analysis in Beverages and Rice Based Products Ion Chromatography-Inductively Coupled Plasma–Mass Spectrometry

Author: Russ Gerads

Summary of Method: TFA extraction (rice and rice products) followed by isocratic LC-ICPMS using phosphate mobile phase and an anion exchange column. Juices are diluted without any further sample preparation. Method was developed using an Agilent 7700 ICPMS.

Method Scope/Applicability: Applicable for the determination of arsenite, arsenate, total inorganic arsenic, monomethylarsonic acid, dimethylarsinic acid and possibly arsenobetaine. No standard curve was constructed for arsenobetaine. Rather, response of other species wee used. Validated matrices include fruit juice, fruit juice concentrate, rice, infant formula, rice snacks, baby-food cereals, and cereals, using dilution (beverages) or acidic extraction and LC-ICPMS.

General comments about the method: General approach is sound and is well supported in the literature. Method uses the popular Hamilton PRP X-100 anion exchange column. Dilute trifluoracetic (TFA) acid is used to extract species in rice and rice products. Hotblock is used to heat samples during extraction. 82Se is added to all extracts and used an an internal standard.

Method Clarity: Method is clear but lacking in detail.

Pros/Strengths: Extensive safety precautions mentioned. Much guidance on contamination control. This is important for trace level work. Uses very popular column (PRP-X100) and instrument (Agilent 7700). Uses a small magnetic stir bar in each extraction tube to aid in the extraction.

Cons/Weaknesses:

1. There were several several mistakes and inconsistencies: "Concentrated" methanol listed in reagent section. Concentrated is not usually associated with solvent. "Digestion" used in a few places instead of "extraction". One is instructed to prepare standards in 1mM TMAH but THMH is not included in the list of reagents. Should this be TFA and not TMAH? The amount of arsenobetaine specified to use in preparing the 1000 ppm stock standard was incorrect. The amount specified would result in a 100 ppm solution.
2. Detailed instruction missing in some places. For example, the pH of the eluent "must be checked and verified that it is 7." However, no tolerance is specified such as pH 6.9-7.1. No guidance is given about what to do if the pH is not exactly pH7. Should the solution be remade or can it be pH adjusted?
3. Collision-reaction cell settings of 10 ml/min He and an energy discrimination voltage of 0 V is specified. The 10 mL/min is about twice what is normally used. Zero volts of energy discrimination means there is no discrimination.

4. Juice concentrates are diluted far more than regular juices even when considering the concentration aspect. I would suggest diluting juice concentrates to ready to drink brix and then proceeding as a regular juice.
5. No mention is made of pulpy juices like apricot nectar and what modification might be necessary for the preparation.
6. Weighted linear calibration curve mentioned but the type of weighting factor is not specified. Instrument software offers several choices of weighting factors.
7. "MS" and MSD" are used but defined.
8. In Table 2015.01F under "Acceptance Criteria" recovery limits are listed for calibration standards. It does not make sense to have recovery criteria for the calibration standards.
9. In the Method Performance section, it is stated that the LOD and LOQ were calculated from the analysis of 23 method blanks. Data are presented in Table 2015.01G. However, the data in the table appear to be the result of analyzing 0.02 ppb standards and not method blanks. This would probably result in a lower and inaccurate estimate of the LOD and LOQ.
10. Table 2015.01H lists identical LOQs for rice and juice. This conflicts with the different preparation dilution factors listed in section F. Juice is diluted by a factor of 25. Rice is diluted 0.3g to 20 ml and then again by a factor of 5 for a total dilution factor of 333. Additionally, juice concentrates are listed with an LOQ 1/10 of what is listed for juice. This conflicts with the instructions listed in section F (Sample prep) where juice is diluted by a factor of 25 and juice concentrate is diluted by 10 and then by 10 again for a factor of 100.
11. No criteria was listed for mass balance of all the species versus a total As analysis.
12. I don't think you can list "rice snacks" in the applicability statement when only one type of this class of food was evaluated. The applicability statement needs to be more specific or more types of rice based snack foods need to be evaluated with the method. There might be problems with snacks high in sugar and/or protein.
13. There are no instructions on what to do when an analyte peak overlaps with a peak from an unknown compound.
14. Rice is hydroscopic. The percent moisture can vary from about 5% to 12% depending on the humidity. This was not addressed and can bias results between labs and even within a lab over a timeframe. One way to address this problem is to require that rice grain samples be dried and report results on a dry weight basis.
15. Method determines inorganic species as well as total inorganic arsenic. However, there was no mention of verification of the lack of interconversion between arsenite and arsenate.

Supporting Data

General Comment: There was a general lack of details in the supporting data. For example, Table 2015.01I lists recovery for TIAs, MMAs and DMAs with no mention on how many replicates were analyzed to arrive at the means. In Appendix A, Repeatability Study for NIST 1548a Table, I would like to see the actual values (spiked and unspiked) for the species not just the RSDs. Also was SRM 1548a (Typical Diet) used or is that a typo error and 1568a (Rice Flour) was used?

In the Matrix Specific Repeatability, Reproducibility and Recovery Studies table for Grape Juice, the values listed for arsenite are <LOQ according to the LOQs listed in Table 2015.01H. The values listed for MMAs are < 0.068 µg/kg. This conflicts with the LOQ listed in Table 2015.01H of 3.1 µg/kg. This is almost 2 orders of magnitude difference. In the same table but for Baby Cereal, 4 out of 8 sections of data have < LOQ reported and, hence, no statistics. Samples with species > LOQ should have been used for the SLV.

There were no example chromatograms. An analyst not familiar with the method should know what order the peaks elute and what an example chromatogram should look like.

There was no accuracy data for arsenite and arsenate but rather only for total inorganic arsenic. Therefore arseneite and arsenate should be removed from the applicability statement.

There was no data for juice concentrate in the Matrix Specific Repeatability, Reproducibility and Recovery Studies section. Therefore juice concentrate should be removed from the applicability statement.

There was only one reference material to judge accuracy. Two or more CRMs would have been better.

There was no information on how the Reproducibility and Recovery Study data was obtained such as how many analysts and instruments were involved, over how many days were the data collected and were the days consecutive.

There was no ruggedness data.

Method Optimization: No data

Performance Characteristics

Analytical Range:

Juice - 0.5 ppb to 0.5 ppm Fails SMPR upper range of 1 ppm

Juice concentrate - 2 ppb to 2 ppm

Rice and rice Products - 6.7 ppb to 6.7 ppm

LOQ: Difficult to tell what is the LOQ. Table 2015.01H lists values but the values are the same for juice and rice even though these sample types have different prep dilutions.

Accuracy/Recovery: Accuracy based on SRM 1568b was excellent for TIAs (99%), MMAs (99%) and DMAs (100%). Note that there was no data for arsenite and arsenate individually.

Precision (RSD_r): Generally in the 1% to 5% range for the 4 matrix types. Exceptions outside of SMPR requirements were baby cereal and rice for arsenate (both at 25%).

Reproducibility (RDS_R): Ranged 1% to 17%. However, there were several missing data sets because the results were <LOQ. For example baby cereal matrix was missing data for arsenate, MMAs and DMAs.

System suitability:

Recommendations

Do you recommend that the ERP adopt this method as an AOAC Official Methods of Analysis (First Action status)?

No. The method has potential and is based on sound science and a popular column but is missing too much detail and data. See items listed above.

Reviewer Name: William Mindak

Date: March 7, 2016

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AOAC INTERNATIONAL Standards Development:
First Action Method Review Form

Name of Reviewer:

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Applicable SMPR:

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This method is applicable to the SMPR. However, I have issues with the AsB detection in this method, as well as the As5 background. I also feel that this method is not as well laid out as the FDA method, and other As speciation methods submitted.

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The method states that they had consistant contamination level of As 5. This worries me. I would have figured out the source of this contamination and then proceeded with method development. This is not something that I have seen doing this type of method. Where all starting materials checked?

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1. Based on the supporting information, were there any additional steps in the evaluation of the method that indicated the need for any additional precautionary statements in the method?

As stated in II.1

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Attachment

First Action Method Review For ARS-04

Prepared by Jenny Scifres

Continuation of discussion:

III. 1) There is no substantial discussion on how the Matrix Specific Repeatability, Reproducibility, and Recovery Studies were conducted. It would be very beneficial to have a discussion section that includes how the reproducibility studies were done and spiking levels in order to better understand the tables in Appendix A. It appears that arsenate (As(V)) fails the SMPR for repeatability in the lowest range for grape juice and baby cereal. MMA is not detected in grape juice and baby cereal, but a spike recovery of 98% is present for baby cereal. It is unclear if the mean spike recovery is based on any of the repeatability or reproducibility data or if it is a stand-alone study.

III. 2) Although the method does not appear to demonstrate that it meets the LOQ requirements for dry matrices, the reviewer does not have enough information to definitively make this determination. A quick estimated calculation of the existing data shows that the LOQs are very close to meeting the SMPRs. The estimate was done by calculating the LOQ based on the data in the method, including the blank value in the calculation, and multiplying the result by 250 (a rough estimate from the method of the adjustment between blanks and dry matrices). The method mentions several dilution steps. If any of these can be eliminated, the method would probably meet the LOQ. Additionally, labware are not further cleaned, which may help improve LOQs. Additional information should be provided by the authors to prove that the method meets the LOQ in the SMPR.

The percent RSD for arsenate is also very close to meeting the SMPR for baby cereal and rice. These matrices present the lowest levels of arsenate in the dry foods tested in this method. This might be improved upon with further investigation since only three reps are presented.

IV. 4) The reviewer believes that overall the method is written clearly and concisely. In the reviewer's opinion, the method would be greatly enhanced by a little more discussion and information. For example, some information about the chromatographic details such as order of elution, retention time, and any potential problems such as co-elution would be helpful. However, this is just a recommendation and personal preference of the reviewer.

There are some very minor issues with clarity.

> In the second paragraph of section A, the method refers to reaction/collision cell. The abbreviation CRC appears in section D.(d)(3) without previous mention. If these are the

same, “reaction/collision cell” should be changed to “collision/reaction cell (CRC)” in section A.

In the sections listing equipment and reagents, there is inconsistent use between “--“ and “–“. Also with italicized and non-italicized fonts.

In section E.(j), instructions should be added for adjusting the pH of the solution if it is not 7.

In section H.(c), “element” should probably be “species”. Section H.(e) states, “ … calibration standard analyzed in quadruplicate …”. Should this be “…calibration standard prepared and analyzed in quadruplicate..”? Are these prepared the same as samples and the method blanks?

MDL is used a couple of times in the document without definition. Is this the same as LOD in the method?

Blank	As3	As5	MMA	DMA	
1	0.0257	0.0251	0.0234	0.0221	
2	0.0236	0.0246	0.0192	0.026	
3	0.0256	0.0198	0.022	0.0242	
4	0.0273	0.021	0.0197	0.0229	
5	0.0218	0.0171	0.0249	0.0263	
6	0.0269	0.0174	0.0207	0.0237	
7	0.0257	0.0211	0.025	0.0238	
8	0.0198	0.0194	0.0218	0.0234	
9	0.0228	0.0242	0.0254	0.0233	
10	0.0231	0.0213	0.0253	0.0238	
11	0.0215	0.0213	0.0243	0.0226	
12	0.0223	0.0231	0.0244	0.0225	
13	0.022	0.0206	0.021	0.0253	
14	0.0205	0.0204	0.0206	0.0248	
15	0.0228	0.0222	0.0226	0.0254	
16	0.0228	0.021	0.0241	0.0236	
17	0.0211	0.0226	0.0236	0.0259	
18	0.0232	0.0241	0.0266	0.0254	
19	0.0234	0.0247	0.0237	0.024	
20	0.0184	0.0214	0.0193	0.0198	
21	0.0195	0.0227	0.0199	0.0194	
22	0.0205	0.0197	0.0208	0.02	
23	0.0196	0.0211	0.0194	0.0198	
	========	========	========	========	
x =	0.022604	0.021561	0.022509	0.023391	
SD =	0.002418	0.002154	0.002296	0.00206	
LOD =	0.029858	0.028024	0.029398	0.02957	
LOQ =	0.059717	0.056048	0.058796	0.05914	2 x LOD
LOQ_{10} =	0.046785	0.043105	0.045473	0.043987	10 x SD
Appx LOQ Dry	11.69613	10.7763	11.36823	10.9967	

RSD Check
7.539
6.516
10.47
2.052292
8.175
25.10448

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AOAC INTERNATIONAL Standards Development:
First Action Method Review Form

Name of Reviewer:

Jenny Nelson

Title of Method:

Analytical Method for the Determination of Vario

AOAC Candidate Method Number:

ARS01

Applicable SMPR:

SPSFAM

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ARS-05

Method Title: Arsenic Speciation in Rice and Rice Products Using High Performance Liquid Chromatography-Inductively Coupled Plasma-Mass Spectrometric Determination

Submitted by: Kevin Kubachka, FDA

Submitted by Email: Kevin.Kubachka@fda.hhs.gov

Enclosures: 2

Submitter notes:

Here is our documentation for our method EAM 4.11, for determination of arsenic species in rice and rice products. I included our method protocol (available to the public), a presentation summarizing our multilaboratory validations of this method and a supplemental information document to address the SMPR specifics.

Reviewers: Michelle Briscoe, Li Sheng, Sneh Bhandari

Link to Method:
http://www.aoac.org/iMIS15_Prod/AOAC_Member/SH/SPSFAMCF/SPSFAM_ERPHM.aspx

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AOAC INTERNATIONAL Standards Development:
First Action Method Review Form

Name of Reviewer:

Jenny Nelson

Title of Method:

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AOAC Candidate Method Number:

ARS01

Applicable SMPR:

SPSFAM

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AOAC INTERNATIONAL Standards Development: First Action Method Review Form

Name of Reviewer: Michelle Briscoe

Title of Method: Arsenic Speciation in Rice and Rice Products Using High Performance Liquid Chromatography-Inductively Coupled Plasma-Mass Spectrometric Determination

AOAC Candidate Method Number: ARS-05

Applicable SMPR: 2015.006

Section IV, Question 6: *Based on the supporting information, what are the cons/weaknesses of the method?*

1. The method's calibration range is not sufficiently low to achieve the SMPR range and LOQ requirements. A lower calibration standard (0.2 ppb or lower) should be used.

2. The method should be updated with validation data for NIST 1568b. Validation data is not presented that shows RM results that meet SMPR.

3. The method does not allow for independent quantification of As(III) and As(V).

4. The extracted samples are diluted into a basic solution to adjust the pH, while all standards are in DIW. Matrix matched calibration standards would produce more accurate data.

5. During HNO3 extraction, other elements such as Ca, Mg, Fe, and Mn can also be solubilized. These elements precipitate at high pH and As(V) is known to adsorb onto these precipitates. This could result in a low bias for inorganic As sample results.

6. Working standards are only verified for total arsenic (section 4.11.6). The purity of the standards are not verified.

7. Methods that use an ammonium phosphate mobile phase typically have a pretty high background, making it hard to achieve low detection limits and LOQs. Although the background concentration isn't discussed in the method, it's assumed it's significant as section 4.11.9, parts 4a-4e, describe how to test for an ammonium phosphate reagent that is too high to use. In addition, the method described for testing the ammonium phosphate (evaluating the ratio of mobile phase response to that of DIW) may not necessarily provide a good indication of the cleanliness of the reagent. The background equivalent concentration (BEC) should be calculated and referenced, since the response of the instrument could be different on different days. This is critical because the performance of the method at low levels relies on the cleanliness of the mobile phase. An appropriate BEC should be suggested by the authors, and that will help with replication of their method and achieving similar LODs.

8. The sensitivities reported in Table 4 are troubling. The sensitivity for As(III) is ~15% lower than that of MMAs and As(V), and the sensitivity for DMAs is 10% higher than that of MMAs and As(V). This strongly suggests that there are either impurities in standards (possible) or that the integrations are not correct (more likely). The sensitivities for all species should be identical, and this is an important QA measure to control. When this method was published on the FDA website, there were some chromatogram posted as well, and they showed some As(III) tailing into the DMAs, and the integration for DMAs was being biased, causing high-bias for DMA. Based on the sensitivities listed in Table 4, this could be happening with this method.

The Scientific Association Dedicated to Analytical Excellence®

AOAC INTERNATIONAL Standards Development:
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Appendix W

POLICY AND PROCEDURES ON VOLUNTEER CONFLICT OF INTEREST

Statement of Policy

While it is not the intention of AOAC INTERNATIONAL (AOAC) to restrict the personal, professional, or proprietary activities of AOAC members nor to preclude or restrict participation in Association affairs solely by reason of such activities, it is the sense of AOAC that conflicts of interest or even the appearance of conflicts of interest on the part of AOAC volunteers should be avoided. Where this is not possible or practical under the circumstances, there shall be written disclosure by the volunteers of actual or potential conflicts of interest in order to ensure the credibility and integrity of AOAC. Such written disclosure shall be made to any individual or group within the Association which is reviewing a recommendation which the volunteer had a part in formulating and in which the volunteer has a material interest causing an actual or potential conflict of interest.

AOAC requires disclosure of actual or potential conflicts of interest as a condition of active participation in the business of the Association. The burden of disclosure of conflicts of interest or the appearance of conflicts of interest falls upon the volunteer.

A disclosed conflict of interest will not in itself bar an AOAC member from participation in Association activities, but a three-fourths majority of the AOAC group reviewing the issue presenting the conflict must concur by secret ballot that the volunteer's continued participation is necessary and will not unreasonably jeopardize the integrity of the decision-making process.

Employees of AOAC are governed by the provision of the AOAC policy on conflict of interest by staff. If that policy is in disagreement with or mute on matters covered by this policy, the provisions of this policy shall prevail and apply to staff as well.

Illustrations of Conflicts of Interest

1. A volunteer who is serving as a committee member or referee engaged in the evaluation of a method or device; who is also an employee of or receiving a fee from the firm which is manufacturing or distributing the method or device or is an employee of or receiving a fee from a competing firm.

2. A volunteer who is requested to evaluate a proposed method or a related collaborative study in which data are presented that appear detrimental (or favorable) to a product distributed or a position supported by the volunteer's employer.

3. A referee who is conducting a study and evaluating the results of an instrument, a kit, or a piece of equipment which will be provided gratis by the manufacturer or distributor to one or more of the participating laboratories, including his or her own laboratory, at the conclusion of the study.

4. Sponsorship of a collaborative study by an interest (which may include the referee) which stands to profit from the results; such sponsorship usually involving the privilege granted by the investigator to permit the sponsor to review and comment upon the results prior to AOAC evaluation.

5. A volunteer asked to review a manuscript submitted for publication when the manuscript contains information which is critical of a proprietary or other interest of the reviewer.

The foregoing are intended as illustrative and should not be interpreted to be all-inclusive examples of conflicts of interest AOAC volunteers may find themselves involved in.

Do's and Don't's

Do avoid the appearance as well as the fact of a conflict of interest.

Do make written disclosure of any material interest which may constitute a conflict of interest or the appearance of a conflict of interest.

Do not accept payment or gifts for services rendered as a volunteer of the Association without disclosing such payment or gifts.

Do not vote on any issue before an AOAC decision-making body where you have the appearance of or an actual conflict of interest regarding the recommendation or decision before that body.

Do not participate in an AOAC decision-making body without written disclosure of actual or potential conflicts of interest in the issues before that body.

Do not accept a position of responsibility as an AOAC volunteer, without disclosure, where the discharge of the accepted responsibility will be or may appear to be influenced by proprietary or other conflicting interests.

Procedures

Each volunteer elected or appointed to an AOAC position of responsibility shall be sent, at the time of election or appointment, a copy of this policy and shall be advised of the requirement to adhere to the provisions herein as a condition for active participation in the business of the Association. Each volunteer, at the time of his or her election or appointment, shall indicate, in writing, on a form provided for this purpose by AOAC, that he or she has read and accepts this policy.

Each year, at the spring meeting of the AOAC Board of Directors, the Executive Director shall submit a report certifying the requirements of this policy have been met; including the names and positions of any elected or appointed volunteers who have not at that time indicated in writing that they have accepted the policy.

Anyone with knowledge of specific instances in which the provisions of this policy have not been complied with shall report these instances to the Board of Directors, via the Office of the Executive Director, as soon as discovered.

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Adopted: March 2, 1989
Revised: March 28, 1990
Revised: October 1996
Reviewed by outside counsel March 2000 (Fran Dwornik) and found to be current and relevant

Appendix U

ANTITRUST POLICY STATEMENT AND GUIDELINES

Introduction

It is the policy of AOAC INTERNATIONAL (AOAC) and its members to comply strictly with all laws applicable to AOAC activities. Because AOAC activities frequently involve cooperative undertakings and meetings where competitors may be present, it is important to emphasize the on-going commitment of our members and the Association to full compliance with national and other antitrust laws. This statement is a reminder of that commitment and should be used as a general guide for AOAC and related individual activities and meetings.

Responsibility for Antitrust Compliance

The Association's structure is fashioned and its programs are carried out in conformance with antitrust standards. However, an equal responsibility for antitrust compliance -- which includes avoidance of even an appearance of improper activity -- belongs to the individual. Even the appearance of improper activity must be avoided because the courts have taken the position that actual proof of misconduct is not required under the law. All that is required is whether misconduct can be inferred from the individual's activities.

Employers and AOAC depend on individual good judgment to avoid all discussions and activities which may involve improper subject matter and improper procedures. AOAC staff members work conscientiously to avoid subject matter or discussion which may have unintended implications, and counsel for the Association can provide guidance with regard to these matters. It is important for the individual to realize, however, that the competitive significance of a particular conduct or communication probably is evident only to the individual who is directly involved in such matters.

Antitrust Guidelines

In general, the U.S. antitrust laws seek to preserve a free, competitive economy and trade in the United States and in commerce with foreign countries. Laws in other countries have similar objectives. Competitors (including individuals) may not restrain competition among themselves with reference to the price, quality, or distribution of their products, and they may not act in concert to restrict the competitive capabilities or opportunities of competitors, suppliers, or customers.

Although the Justice Department and Federal Trade Commission generally enforce the U.S. antitrust laws, private parties can bring their own lawsuits. Penalties for violating the U.S. and other antitrust laws are severe: corporations are subject to heavy fines and injunctive decrees, and may have to pay substantial damage judgments to injured competitors, suppliers, or customers. Individuals are subject to criminal prosecution, and will be punished by fines and imprisonment. Under current U.S. federal sentencing guidelines, individuals found guilty of bid rigging, price fixing, or market allocation must be sent to jail for at least 4 to 10 months and must pay substantial minimum fines.

Since the individual has an important responsibility in ensuring antitrust compliance in AOAC activities, everyone should read and heed the following guidelines.

1. Don't make any effort to bring about or prevent the standardization of any method or product for the purpose or intent of preventing the manufacture or sale of any method or product not conforming to a specified standard
2. Don't discuss with competitors your own or the competitors' prices, or anything that might

affect prices such as costs, discounts, terms of sale, distribution, volume of production, profit margins, territories, or customers.

3. Don't make announcements or statements at AOAC functions, outside leased exhibit space, about your own prices or those of competitors.

4. Don't disclose to others at meetings or otherwise any competitively sensitive information.

5. Don't attempt to use the Association to restrict the economic activities of any firm or any individual.

6. Don't stay at a meeting where any such price or anti-competitive talk occurs.

7. Do conduct all AOAC business meetings in accordance with AOAC rules. These rules require that an AOAC staff member be present or available, the meeting be conducted by a knowledgeable chair, the agenda be followed, and minutes be kept.

8. Do confer with counsel before raising any topic or making any statement with competitive ramifications.

9. Do send copies of meeting minutes and all AOAC-related correspondence to the staff member involved in the activity.

10. Do alert the AOAC staff to any inaccuracies in proposed or existing methods and statements issued, or to be issued, by AOAC and to any conduct not in conformance with these guidelines.

Conclusion

Compliance with these guidelines involves not only avoidance of antitrust violations, but avoidance of any behavior which might be so construed. Bear in mind, however, that the above antitrust laws are stated in general terms, and that this statement is not a summary of applicable laws. It is intended only to highlight and emphasize the principal antitrust standards which are relevant to AOAC programs. You must, therefore, seek the guidance of either AOAC counsel or your own counsel if antitrust questions arise.

Adopted by the AOAC Board of Directors: September 24, 1989
Revised: March 11, 1991
Revised October 1996

Appendix V

POLICY ON THE USE OF THE ASSOCIATION NAME, INITIALS, IDENTIFYING INSIGNIA, LETTERHEAD, AND BUSINESS CARDS

Introduction

The following policy and guidelines for the use of the name, initials, and other identifying insignia of AOAC INTERNATIONAL have been developed in order to protect the reputation, image, legal integrity and property of the Association.

The name of the Association, as stated in its bylaws, is "AOAC INTERNATIONAL". The Association is also known by its initials, AOAC, and by its logo, illustrated below, which incorporates the Association name and a representation of a microscope, book, and flask. The AOAC logo is owned by the Association and is registered with the U.S. Patent and Trademark Office.

The full Association insignia, illustrated below, is comprised of the logo and the tagline, "The Scientific Association Dedicated to Analytical Excellence," shown below. The typeface used is Largo. The AOAC tagline is owned by the Association and is registered with the U.S. Patent and Trademark office.

The Scientific Association Dedicated to Analytical Excellence®

Policy

Policy on the use of the Association's name and logo is established by the AOAC Board of Directors as follows:

> "The Board approves and encourages reference to the Association by name, either as AOAC INTERNATIONAL or as AOAC; or reference to our registered trademark, AOAC®, in appropriate settings to describe our programs, products, etc., in scientific literature and other instances so long as the reference is fair, accurate, complete and truthful and does not indicate or imply unauthorized endorsement of any kind.
>
> The insignia (logo) of AOAC INTERNATIONAL is a registered trade and service mark and shall not be reproduced or used by any person or organization other than the Association, its elected and appointed officers, sections, or committees, without the prior written permission of the Association. Those authorized to use the AOAC INTERNATIONAL insignia shall use it only for

the purposes for which permission has been specifically granted.

The name and insignia of the Association shall not be used by any person or organization in any way which indicates, tends to indicate, or implies AOAC official endorsement of any product, service, program, company, organization, event or person, endorsement of which, has not been authorized by the Association, or which suggests that membership in the Association is available to any organization."

The Executive Director, in accordance with the above stated policy, is authorized to process, approve, fix rules, and make available materials containing the Association name and insignia.

It should be noted that neither the Association's name nor its insignia nor part of its insignia may be incorporated into any personal, company, organization, or any other stationery other than that of the Association; nor may any statement be included in the printed portion of such stationery which states or implies that an individual, company, or other organization is a member of the Association.

Instructions

1. Reproduction or use of the Association name or insignia requires prior approval by the Executive Director or his designate.

2. Association insignia should not be altered in any manner without approval of the Executive Director or his designate, except to be enlarged or reduced in their entirety.

3. Artwork for reproducing the Association name or insignia, including those incorporating approved alterations, will be provided on request to those authorized to use them (make such requests to the AOAC Marketing Department). Examples of the types of alterations that would be approved are inclusion of a section name in or the addition of an officer's name and address to the letterhead insignia.

4. When the Association name is used without other text as a heading, it should, when possible, be set in the Largo typeface.

5. Although other colors may be used, AOAC blue, PMS 287, is the preferred color when printing the AOAC insignia, especially in formal and official documents. It is, of course, often necessary and acceptable to reproduce the insignia in black.

6. Do not print one part of the logo or insignia in one color and other parts in another color.

7. The letterhead of AOAC INTERNATIONAL shall not be used by any person or organization other than the Association, elected and appointed officers, staff, sections, or committees; except by special permission.

   Correspondence of AOAC official business should be conducted using AOAC letterhead. However, those authorized to use AOAC letterhead shall use it for official AOAC business only.

   Copies of all correspondence using AOAC letterhead or conducting AOAC official business,

whether on AOAC letterhead or not, must be sent to the appropriate office at AOAC headquarters.

8. AOAC INTERNATIONAL business cards shall not be used by any person or organization other than the Association, its staff, and elected officials, except by special permission.

   Those authorized to use AOAC business cards shall use them for official AOAC business only and shall not represent themselves as having authority to bind the Association beyond that authorized.

Sanctions

1. Upon learning of any violation of the above policy, the Executive Director or a designate will notify the individual or organization that they are in violation of AOAC policy and will ask them to refrain from further misuse of the AOAC name or insignia.

2. If the misuse is by an Individual Member or Sustaining Member of the Association, and the misuse continues after notification, the Board of Directors will take appropriate action.

3. If continued misuse is by a nonmember of the Association or if a member continues misuse in spite of notification and Board action, ultimately, the Association will take legal action to protect its property, legal integrity, reputation, and image.

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Adopted by the AOAC Board of Directors: September 24, 1989
Revised: June 13, 1991; February 26, 1992; March 21, 1995; October 1996