



AOAC INTERNATIONAL

Stakeholder Panel for Infant Formula and Adult Nutritionals (SPIFAN)

Gaithersburg Marriott Washingtonian Center

9751 Washingtonian Boulevard

Gaithersburg MD 20878, USA

Thursday, March 16, 2017 - 8:30am (Eastern US)

REPORT OF THE NUTRIENTS EXPERT REVIEW PANEL (ERP) PROCEEDINGS

Expert Review Panel Members (in attendance):

Darryl Sullivan

John Austad
Sneh Bhandari
Esther Campos-Giménez
Scott Christiansen
Hans Cruijisen/Martine van Gool
Brendon Gill
Don Gilliland/Karen Schimpf
Estela Kneeteman
Bill Mindak
Melissa Phillips/ Kate Rimmer
Shay Phillips
David Woollard
Jinchuan Yang

Covance Labs (Chair)

Covance Labs
Mérieux NutriSciences
Nestlé
Perrigo Nutritionals
FrieslandCampina
Fonterra Cooperative
Abbott Nutrition
INTI
FDA CFSAN
NIST
Mead Johnson Nutrition
Eurofins (formerly Hill Labs)
Waters Corp.

Expert Review Panel Members (unable to attend):

Sean Austin
Adrienne McMahon
Harvey Indyk
Jon DeVries
Maria Ofitserova
Günther Raffler

Nestlé Research Centre
Wyeth
Fonterra Cooperative
DeVries & Associates
Pickering Labs, Inc.
Eurofins/CLF

AOAC Staff Includes:

Delia Boyd
Scott Coates (CSO)
Chris Dent
Arlene Fox
Dawn Frazier
Jonathan Goodwin (Interim Executive Director)

Nora Marshall
Deborah McKenzie
Alicia Meiklejohn
Tien Milor
Bob Rathbone

Observers:

John, Austad, *Covance*
Charles Barber, *NIST*
Sneh Bhandari, *Mérieux NutriSciences*
Esther Campos Giménez, *Nestlé*
Marti Cenky, *Abbott Nutrition*
Scott Christiansen, *Perrigo*
Hans Cruijssen, *FrieslandCampina*
Marcel de Vreeze, *NEN ISO*
Jean-Luc Deborde, *SCL*
Jennifer Donelson, *VUV Analytics*
Jaap Evers, *IDF*
Ping Feng, *Wyeth/Nestlé*
Christophe Fuerer, *Nestlé Research Center*
Brendon Gill, *Fonterra Co-operative Group*
Donald Gilliland, *Abbott Nutrition*
Philip Haselberger, *Abbott*
Melissa Holskey, *Abbott Nutrition*
Greg Jaudzems, *Nestlé*
George Joseph, *AsureQuality*
Estela Kneeteman, *INTI*
Sookwang Lee, *FDA*
Alex Liu, *SCIEX*
Vicky Manti, *Danone*
Elaine Marley, *R-Biopharm Rhone Ltd*
Mary McBride, *Agilent*
Josh Messerly, *Eurofins*
Bill Mindak, *FDA*

Eystein Oveland, *National Institute of nutrition
and seafood research*
Lawrence Pacquette, *Abbott Nutrition*
Shay Phillips, *MJN*
Melissa Phillips, *NIST*
Annemieke Pustjens, *RIKILT*
Robert Rankin, *INCA*
Rick Reba, *Nestlé*
Andre Santos, *Agilent Technologies*
Karen Schimpf, *Abbott Nutrition*
Florian Schweigert, *BioAnalyt GmbH*
Maurice Seegers, *Mead Johnson Nutrition*
Aniko Solyom, *GAAS Analytical*
Sidney Sudberg, *Alkemist Labs*
Joe Thompson, *Abbott Nutrition*
Marina Torres, *LATU*
Martine van Gool, *FrieslandCampina*
Martijn Vermeulen, *Ausnutria Hyproca*
Wayne Wargo, *Abbott*
Laura Wood, *NIST*
David Woollard, *Eurofins NZ Laboratories*
Jinchuan Yang, *Waters*
Xionghai Yi, *Shanghai CIQ*
Wei Zhu, *Danone*
Joyce Zhu, *Jamieson Laboratories*
Jerry Zweigenbaum, *Agilent Technologies*

I. WELCOME AND INTRODUCTIONS

Darryl Sullivan welcomed all participants to the ERP meeting and introduced the ERP members.

II. REVIEW OF METHODS BY EXPERT REVIEW PANEL (ERP) FOR FIRST ACTION *OFFICIAL METHOD*SM STATUS – AOAC SPIFAN II

For each method, the ERP/Working Group Co-Chairs discussed methods submitted. Three (3) methods were received and reviewed and two (2) method received First Action *Official Method*SM status.

Method	Method Title	Reviewer(s)	Vote	Comments
Amino Acids	<i>Amino-01 - Single Laboratory Validation Report for Total Amino Acids by UHPLC-UV in Infant Formulas and Adult Nutritionals</i>	Maria Ofitserova (via reviewer form) Shay Phillips	<p>ERP voted to take no action with comments to submitter</p> <p>Scott Christiansen (moved) Sneh Bhandari (second) Yes-12/ No-0/Abstain-1</p> <p>Method not recommended for First Action <i>Official Method</i>SM status</p> <hr/> <p>Convene the working group to review SMPR®</p> <p>Esther Campos Giménez (moved) Shay Phillips (second) Yes-12/ No-1/Abstain-0</p>	<ul style="list-style-type: none"> ▪ May need to revise SMPR® for reproducibility ▪ Nitrogen blanket step prior to hydrolysis ▪ Investigate optimizing chromatography (peak resolution criteria) cysteine methionine ▪ Include system suitability step ▪ Proprietary solvents/reagents vs generic (see data comparing) ▪ Include data with alternative column ▪ Include data from proteins rather than free amino acids for accuracy ▪ Comparison data with and without SH protection <p>SMPR® (stakeholders)</p> <ul style="list-style-type: none"> ▪ Revisit the reproducibility & repeatability
	<i>Amino-02 - HPLC Determination of Total Tryptophan in Infant Formula and Adult/Pediatric Nutritional Formula Following Enzymatic Hydrolysis</i>	Adrienne McMahon (via reviewer form) Estela Kneeteman	<p>Estela Kneeteman (moved) David Woollard (second) Yes-12/ No-0/Abstain-1</p> <p>Method recommended for First Action <i>Official Method</i>SM status</p>	<ul style="list-style-type: none"> ▪ See reviewer form
Carotenoids	<i>Carot-03 - Determination of beta-Carotene, Lutein and Lycopene in Infant and Adult/Pediatric Nutritional Formula Using High Performance Liquid Chromatography With UV Detection</i>	Jon DeVries (via reviewer form) Adrienne McMahon John Austad	<p>John Austad (moved) Bill Mindak (second) Yes-12/ No-0/Abstain-1</p> <hr/> <p>Method recommended for First Action <i>Official Method</i>SM status</p>	<ul style="list-style-type: none"> ▪ Use 3 separate evaluations: <ol style="list-style-type: none"> 1. Alpha & Beta 2. Lutein 3. Lycopene ▪ Chromatography (should take account in the calculations) ▪ Alpha & beta separation needed ▪ Provide clarity

III. REVIEW OF METHODS BY EXPERT REVIEW PANEL (ERP) FOR FINAL ACTION *OFFICIAL METHOD*SM STATUS

The Expert Review Panel (ERP) reviewed four (4) methods for Final Action *Official Methods*SM status. Two (2) methods were recommended to the Official Methods Board (OMB) for Final Action *Official Method*SM consideration.

Method	Method Title	Reviewer(s)	Vote	Comments
Biotin	OMA# 2016.02 (Bio-02) - <i>Determination of biotin by liquid chromatography coupled with immunoaffinity column clean-up extraction</i>	Scott Christiansen Karen Schimpf Estela Kneeteman	Scott Christiansen (moved) Karen Schimpf (second) Yes-12/ No-0/Abstain-1 <hr/> Method recommended for Final Action <i>Official Method</i> SM status	<ul style="list-style-type: none"> ▪ See reviewer form(s) ▪ .12 in the sample with back to back flushing; information not clear in the original, but changed in the current version ▪ Method was easily applied ▪ In the cartridge (address in final version) ▪ Method contains unknown content ▪ Clarity around the column usage ▪ Stability of the column ▪ System suitability ▪ Ability to use other IA columns <p>1. Any immunoaffinity column could be used (data can be found in the SLV report)</p>
Chloride	OMA# 2016.03 (Chlor-02/04) – <i>Multi Laboratory Test – AOAC First Action Method 2016.03 Chloride in Infant Formula and Adult/Pediatric Nutritionals</i>	Shay Phillips Bill Mindak Karen Schimpf	<ul style="list-style-type: none"> ▪ First Round Voting: Shay Phillips (moved) Bill Mindak (second) Yes-8/ No-4/Abstain-1 ▪ Second Round Voting: Karen Schimpf (moved) Jinchuan Yang (second) Yes-8/ No-4/Abstain-1 <hr/> Method not recommended for Final Action <i>Official Method</i> SM status	<ul style="list-style-type: none"> ○ Questions were raise on how outliers were calculated 1. How the report was written with the outliers (outliers should be identified) ○ Method may have shelf life issues ○ Include outlier data tables ○ Recalculate prior to final action review
MTE	OMA# 2011.14 (MTE-01/03) – <i>Infant formula, Adult nutrition Milk and milk products — Determination of calcium, copper, iron, magnesium, manganese, phosphorus, potassium, sodium and zinc contents - Inductive coupled plasma atomic emission spectrometric method (ICP-AES)</i>	Bill Mindak Jinchuan Yang	Bill Mindak (Moved) Jinchuan Yang (Second) Motion withdrawn due to the incorrect version of method reviewed by some ERP members E-ballot to be sent to ERP members for voting <hr/> Method not recommended for Final Action <i>Official Method</i> SM status	<ul style="list-style-type: none"> ▪ See reviewer form ▪ Method needs clear scope and should change the title ▪ Review current version dated January 2017 ▪ Change title to reflect matrices that would give performance to match SMPR® ▪ Range of concentration applicable to various elements ▪ Cu/Mn/Fe/Zn

Vitamin D	OMA# 2016.05 (VitD-18) – <i>Analysis of Vitamin D2 and Vitamin D3 by LC MS/MS in Milk Powders, Infant Formulas, and Adult Nutritionals</i>	Don Gilliland Sneh Bhandari Jinchuan Yang	Don Gilliland (Moved) Sneh Bhandari (Second) Yes-12/ No-0/Abstain-1 <hr/> Method recommended for Final Action <i>Official Method</i> SM status	<ul style="list-style-type: none"> ▪ Comments received from ISO
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IV. REVIEW OF METHODS BY EXPERT REVIEW PANEL (ERP) FOR REPRODUCIBILITY TESTING

The ERP collectively discussed the methods and select a single method to move forward through the AOAC SPIFAN process for reproducibility testing.

Method Evaluation Forms - Methods processed via the voting/numbering system:

Method	Method Title	Vote	Comments
Amino Acid <i>(Tryptophan only)</i>	<i>Amino-02 - HPLC Determination of Total Tryptophan in Infant Formula and Adult/Pediatric Nutritional Formula Following Enzymatic Hydrolysis</i>	John Austad (Moved) Sneh Bhandari (Second) Yes-12/No-0/Abstain-1	

V. NEXT STEPS/FEEDBACK FROM EXPERT REVIEW PANEL

Darryl Sullivan provided next steps including Call for Methods for both Carotenoids and Folate (Folic Acid) and suggestions from the Expert Review Panel requesting that the SMPR® for Amino Acids be revisited by the working group.

❖ **To view the ERP Reviewer forms please [click here](#) or <https://cld.bz/6FelpGo>**