



STAKEHOLDER PANEL ON INFANT FORMULA AND ADULT NUTRITIONALS (SPIFAN)

DRAFT PROCEEDINGS

**Meeting at the
Gaithersburg Marriott Washingtonian Center**
9751 Washingtonian Boulevard
Gaithersburg MD 20878, USA

Thursday, March 14, 2019

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MEETING HELD AT

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Gaithersburg MD 20878, USA

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STAKEHOLDER PANEL MEETING PROCEEDINGS

SPIFAN CHAIR

Darryl Sullivan, Eurofins Food Integrity & Innovation

Stakeholder Attendees:

Sean Austin, Nestlé

Lei Bao, Nestlé

Charles Barber, NIST

Jennifer Burgess, Scientialis Consulting

Esther Campos Gimenez, Nestlé

Chung Cho, US FDA

Bob Clifford, Shimadzu

Mark Collison, ADM

Tongtong Cu, US Pharmacopeia

David Dain, DSM Nutritional Products

Marcel de Vreeze, NEN - ISO

Aurelie Dubois, International Dairy Federation

Mathieu Dubois, Nestlé

Ping Feng, Wyeth Nutrition

Lauren Fleury, AsureQuality Ltd

Carlos Galera, Hygiena Diagnostica España

Don Gilliland, Abbott Nutrition

Tetsuhisa Goto, CSC JP

Philip Haselberger, Abbott Nutrition

Steve Holroyd, Fonterra

Greg Hostetler, Perrigo Nutritionals

Greg Jaudzems, Nestlé

George Joseph, AsureQuality

Estela Kneeteman, INTI

Mary Krogull, Eurofins

Paolo Lecchi, DSM

Soo Kwang Lee, US Food and Drug
Administration

Chengzhu Liang, SDCIQ

Kai Liu, Eurofins Nutrition Analysis Center

Sean McClure, Abbott Nutrition

Josh Messerly, Eurofins

Deepali Mohindra, Thermo

Melissa Phillips, NIST

Shay Phillips, RB/MJN

Cezary Poplawski, R-Biopharm Rhone

Robert Rankin, INCA

Joe Romano, Waters Corporation

Jana Rousova, Restek

Andre Santos, Agilent Technologies, Inc.

Mei Shotts, Abbott Nutrition

Véronique Spichtig, Nestec

Cynthia Srigley, US Food and Drug
Administration

Dustin Starkey, Abbott Nutrition

Hiroko Suzuki, Japan Food Research Laboratories

John Szpylka, Mérieux NutriSciences

Elizabeth Teigland, Agropur

Jeanice Thomas, NIST

Joseph Thompson, Abbott Nutrition

Marina Torres, LATU

Martine van Gool, FrieslandCampina

Jeroen van Soest, Eurofins

Wayne Wargo, Abbott Nutrition

Kenny Xie, US Pharmacopeia

Tongtong Xu, US Pharmacopeia

Jinchuan Yang, Waters Corporation

Jupiter Yeung, Nestlé

Yuichi Yotsuyanagi, Shimadzu

AOAC Staff in Attendance

David Schmidt (*Executive Director*)
Palmer Orlandi (*Deputy Executive Director/CSO*)
Delia Boyd
Scott Coates
Jennifer Diatz
Jonathan Goodwin (*Deputy Executive Director*)

Nora Marshall
Deborah McKenzie
Alicia Meiklejohn
Tien Milor
La'Kia Phillips
Bob Rathbone

I. WELCOME/INTRODUCTIONS

Darryl Sullivan (Eurofins) chair of the AOAC SPIFAN stakeholder panel welcomed participants to the eighteenth stakeholder meeting of AOAC Stakeholder Panel on Infant Formula and Adult Nutrition (SPIFAN) and led the introductions of attendees. The AOAC policy and procedures were also discussed.

II. GLOBAL ENGAGEMENT ACTIVITIES

a. *AOAC/ISO/IDF COOPERATIVE UPDATE*

The cooperative update was presented by Marcel de Vreeze (NEN & ISO) with collaboration from Erik Konings (Nestlé) and Aurelie Dubois (IDF). The update included the current status of methods within ISO/IDF with ISO/TC 34/WG 14 (Vitamins, carotenoids, and other nutrients) and ISO/TC 34/SC 5 | IDF (Milk & Milk products).

The timeline for meetings of WG 14 is July 4-5, 2018 in IMR, Bergen, Norway with CEN/TC 275/WG 9 – Vitamins and carotenoids and IDF/SC 5: ISO/IDF Analytical Week held June 21-25, 2019 in Prague.

The next set of methods to be submitted into the Codex process (CCMAS/CAC) will include: Vitamin K₁, Minerals & Trace elements by ICP-AES, and ICP-MS.

b. *AOAC INDIA SECTION*

Palmer Orlandi (AOAC) updated the stakeholders on the AOAC India Section meeting held February 2019, including any priorities or new opportunities of interest to AOAC SPIFAN.

c. *AOAC CHINA SECTION*

Cheng-zhu Liang (AOAC China Section) shared information on the progress on evaluation of GB standards for food safety. He provided background information, current status, achievement to date, and the 2019 workplan. China Society of Inspection and Quarantine (CSIQ) has been assigned to lead the program by the China's National Health Commission (NHC) along with the China National Center for Food Safety Risk Assessment (CFSA) to carry out the agreement signed in February 2018. The GB program consists of 256 analytical methods including chemical and microbiological. The program collects opinions and feedback from both authorities and industry, organizes working groups to review the information and provides a deliverable report from CSIQ with suggestions for future GB method formulation & revisions. CSIQ leads ten (10) working groups to review and evaluate comments with participation from 196 organizations with the majority from regulators & government laboratories and food enterprises. The workflow, which includes a submitted report to NHC & CFSA would then be revised & formulated into new GB standards based on CSIQ's report.

High-level meetings of GB authorities and ISO/IDF/AOAC, hosted by AOAC China Section met in June 2018 to discuss standards and methods harmonization. AOAC China Section coordinated with CSIQ, and SAC, NHC/CFSA, MOA and AOAC, ISO and IDF on proposed selected standards for milk and dairy. During the AOAC annual meeting in September, sessions were held between GB Authority and AOAC/ISO/IDF to discuss GB Evaluation Program hosted by AOAC China section. It was proposed to start with standards/methods for milk and dairy including infant formula and use the "comparison template: to compare standard as well as validate the template using the example of vitamin D and Aerobic Plate count.

Liang invited stakeholders to participate in the 2019 AOAC China Analytical Science & Standards Conference to be held May 13-May 15, 2019 in Shanghai.

d. **CODEX UPDATES**

i. **Status of SPIFAN Methods in Codex Process**

Robert Rankin (INCA) provided an update on the status of methods in the SPIFAN process. The goal of AOAC SPIFAN is to have the most updated dispute resolution methods (Type II) in the Codex Alimentarius. The SPIFAN Codex Strategy Group advocates for support/endorsement by the Codex committees with information including conference room documents and other materials on our position which includes joint AOAC/ISO/IDF method submissions.

The progress to date includes thirteen (13) AOAC SPIFAN methods in the Codex process as Type II dispute resolution methods:

- Vitamin A
- Vitamin E
- Vitamin D
- Vitamin C
- Vitamin B12
- Biotin
- Pantothenic Acid
- Myo-inositol
- Fatty Acid Profile
- Total Nucleotides
- Iodine
- Chloride
- Chromium/Molybdenum/Selenium

The Codex Committee on Nutrition and Foods for Special Dietary Uses (CCNFSDU) referred three (3) methods to Codex Committee on Methods of Analysis and Sampling (CCMAS) in November 2018 for technical review and typing: Minerals and Trace Elements (AOAC 2015.06/ISO 21424 | IDF 243), Vitamin K (AOAC 2015.09/ISO 21446), and Folic Acid/Folate (AOAC 2011.06).

During the next meeting of CCMAS' 40th session in Budapest (May 27-31, 2019), the group is slated to review SPIFAN methods for Minerals and Trace Elements (MTE), Vitamin K and Folic Acid/Folate for endorsement as Type II. SPIFAN recommended re-typing/revoking existing Type II/III methods for MTE and Folic Acid; SPIFAN will also introduce AOAC 2011.14 / ISO 15151 | IDF 229 (ICP-AES for MTE) for endorsement as Type III.

The Codex Alimentarius Commission's (CAC) 42nd Session will be held July 8-12, 2019 in Geneva with recommendations to adopt SPIFAN methods for MTE, Vitamin K and Folic Acid/Folate as Type II (bringing the total to 16).

ii. **Potential Enhancements to SPIFAN/Codex Process**

Douglas Balentine (US Delegate to CCNFSDU/US FDA) discussed the challenges with the existing process for introducing methods for CCNFSDU consideration and explored process improvements and enhancements by providing a roadmap and timeline for updating methods in Codex. AOAC SPIFAN methods to be introduced at CCNFSDU41 should be submitted to CCMAS Delegate for review by February 2019. If endorsed, the methods will be sent to CAC for adoption during their May 27, 2019 meeting. If no endorsement is provided, the method will be sent to CCNFSDU for matter referred (form of Vit D, Cr, Mo, Se data). AOAC SPIFAN methods and the draft of the paper should be submitted by July 8, 2019 with the paper due to Codex Secretariat by August 2019 and if CAC adopts the method, then CCMAS updates Codex Stan 234, otherwise the method is sent to CCNFSDU as a matter for information/referral. The goal of the physical working group (pWG), which meets November 25, 2019, is to get the committee's agreement to support the methods or answer matter referred.

Greg Noonan (US Delegate to CCMAS/US FDA) provided the stakeholders information on how methods are introduced, reviewed, endorsed and adopted in CCMAS. Items to consider includes, does the method performance meet the Codex Specifications, can the units be expressed to simplify comparison, and is the provision in agreement with the Codex Standard? Other factors that slows the process is timing (which would include final publications), has the method been finalized/updated, and are documents provided to the Secretariat in a timely manner. While reviewing the process, it was also reiterated that Standard Method Performance Requirements (SMPRs) are not referenced in Codex.

iii. **Update on CODEX STAN 234-1999 Revisions to Dairy Methods**

Greg Noonan (US Delegate to CCMAS/US FDA) provided an update on the status of the review of dairy methods in CODEX STAN 234-1999 by the electronic working group of CCMAS. Noonan included information on the submission process into Codex as well as how and when to provide information. Prior to submission, the methods should be reviewed, and information provided to ensure the most viable methods are included into Codex including the correct typing.

III. UPDATE ON THE NIST STANDARD REFERENCE MATERIALS (SRM)

Melissa Phillips (NIST) updated the stakeholder panel on the status of the NIST SRM. The current SRM (1849a) based on current sales, is projected to be out of stock by the end of 2019. New material (SRM 1849b) is being procured now and will have the same composition of SRM 1849a but will contain additional nutrients compared to SRM 1849a (Carotenoids, Vitamin D₂ and D₃, GOS and Retinyl acetate and palmitate). The projected release date is fall 2021.

IV. UPDATE ON PROFICIENCY TESTING

Shane Flynn (AOAC) provided an update on the AOAC Proficiency Testing (PT) program. The prime objective of the AOAC Proficiency Testing program is to evaluate and improve analytical performance by providing an independent measure of the quality of the data.

Currently, the PT program consists of both water-soluble and oil-soluble vitamins programs. Other competing programs may require enrollment in one or two additional and separate programs. Participants have the ability to submit results for AOAC SPIFAN methods as well as other methods. The program meets the requirements for accreditation and supports method validation. Current challenges in the program includes participation, obtaining future supply of homogeneous stable samples, and to ensure that nutrients meet laboratory needs.

The infant formula program has shipped materials in March and September 2018 and February 2019. The next shipment will be sent in June 2019 with 50% sent internationally.

V. UPDATE ON PROFICIENCY TESTING NUTRIENT WORKING GROUP UPDATES

a. **Amino Acids***

The working group co-chairs, Ping Feng (Wyeth/Nestlé)/Philip Haselberger (Abbott) presented that there be no revisions to the current Standard Method Performance Requirements (SMPR®) for the Determination of Selected Amino Acids in Infant and Adult/Pediatric Nutritional Formula (AOAC SMPR 2014.013). Background and timeline information included the development of the SMPR in September 2014, revision and approval by the Stakeholders in August 2018. During the public comment period, one comment was received and distributed to working group co-chairs in December 2018. During the stakeholder panel discussion, the working group co-chairs provided the response to the comment.

No further modifications are required for the SMPR based on the following considerations:

- Existing published AOAC methodology for total taurine (999.12)
- Literature evidence of bound taurine

b. GOS

Sean Austin (Nestlé) and Martine van Gool (FrieslandCampina) updated the stakeholders on a workshop that took place in December 2018 in the Netherlands to discuss GOS technologies. Information was provided on methods previously presented to the AOAC SPIFAN ERP for GOS analysis. The workshop was organized as a brainstorming session to find possible ways to move forward and the goal was to identify the most promising approaches for the determination of GOS in infant formula & adult nutritionals. Participants included those with interest in GOS analysis. A poster presentation with ideas for GOS determination was a requirement for participation and the group voted for the top three (3) ideas. The group was divided into teams to discuss and refine the top 3 ideas which included selective resin, labelling, and enzymatic removal of lactose.

c. Fluoride

Darryl Sullivan (Eurofins) discussed the lack of fluoride method submissions. Background information included the working groups development and approval of the SMPR (AOAC 2014.016). In 2014, a call for methods was issued, but yielded no methods. In 2015, one method was submitted, but did not meet the SMPR. Other methods that could meet the SMPR are available, but not currently applied to infant formula. At this time, no suitable methods are able to quantify at LOQ of 5µg/100g after the first call for methods. In 2018, the working group revised the SMPR with the following:

- LOQ adjusted from 5µg/100g to 30µg/100g
- Lower range band [for recovery, RSD_r and RSD_R] adjusted from 5-25 µg/100g to 30-100µg/100g
- Upper range band [for recovery, RSD_r and RSD_R] adjusted from 100 µg/100g to 200µg/100g

A call for methods was issued using the revised SMPR, but no method was received. In February 2019, one method was submitted for review by ERP.

The requirement for fluoride in the Codex STAN is < 100g/100 kcal (<65ug/100g* w=reconstituted infant formula) and in Australia and New Zealand the requirement is 50ug /100g in powder (corresponding to current LOQ = 5ug/100g in SMPR). The recommended options are to revise the SMPR or issue a call for methods with ISE, IC or GC with specific sample preparation (e.g., microwave digestion or specific buffer extraction).

VI. UPDATE ON EXPERT REVIEW PANEL (ERP) STATUS

Darryl Sullivan (Eurofins) provided an update on the methods submitted for review, the status of methods in MLT, the AOAC SPIFAN methods still in First Action since the 2018 AOAC Annual Meeting in Toronto. The ERP was convened on January 23, 2019 during a virtual meeting to review AOAC 2015.10 (Carnitine & Choline). The method was originally recommended for Final Action for Carnitine only, but after new data was submitted, the ERP reviewed method the for Choline with a recommendation for Final Action with some revisions. Since the method was reviewed for Choline only during the meeting, the ERP will again review the new data for Carnitine.

The AOAC Official Methods Board approved the following methods to Final Action status: AOAC # 2011.06 (folate), AOAC # 2017.03 (tryptophan), and AOAC # 2011.14 (minerals).

During the upcoming meeting, the ERP will review the following methods for Final Action Recommendations:

- AOAC # 2016.13 – Carotenoids
- AOAC # 2016.14 – FOS
- AOAC # 2015.10 – Carnitine/Choline

A Fluoride method was submitted for First Action review by Metrohm USA and a proposal was received from the AOAC India Section to extend a Final Action method (AOAC # 2015.06 – minerals). The ERP will review both and provide a recommendation.

The AOAC SPIFAN achievements to date include an agreement with the Infant Nutrition Council of America (INCA) since 2010. Other agreements between AOAC and ISO have also been established along with IDF. Currently, thirty-two (32) SMPRs have been established with thirty (30) for nutrients, one (1) for Compound 1080, and one (1) for MCPD. First Action methods are still needed for Fluoride and GOS, as well as laboratories required for multi-lab testing.

VII. TIMELINES/DEADLINES/WRAP-UP (Sullivan)

Darryl Sullivan (Eurofins) provided next steps including a timeline of AOAC SPIFAN activities including upcoming deadlines, review of any action items, and additional questions.