



FINAL: 2016-03-18

**Laboratory Methods and Services Committee Report/Minutes**

AAFCO Mid Year Meeting  
 January 19, 2016  
 8:00 am to 5:00 pm  
 Isle of Palms, SC

**COMMITTEE RECOMMENDATIONS:**

1. None

**BOARD RECOMMENDATIONS:**

1. None
- 2.

**ASSOCIATION ACTIONS:**

1. None
- 2.

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Committee Report:

- *Committee Activities*  
**ACTION: Motion passes, all in favour**  
**MOTION: “Motion to accept the Best Practices working group’s phosphorus white paper and method matrix as complete.” (L. Novotny move, M. Stenske second)**

Committee Minutes:

- Committee attendees introduced themselves. Sign-up sheet was sent around and verified the webex was working.
- The Laboratory Methods and Services Committee (LM&SC) membership roster and industry advisor list was reviewed and A. Price indicated new appointments. A. Price reminded Committee Members that they should be active participants due to the size of the committee. People wishing to be added or eliminated from the list should contact A. Price.
- FSMA Implementation Task Force reps for our committee are Teresa Grant and Robert Sheridan. The task Force will be meeting on Wednesday, Jan 20. They will be looking at the needs and if or what methods need to be developed.
- Working Group updates:
  - Tylosin WG – Tom Phillips was absent, no update from him. Tom has started acting as the State Chemist for Maryland and no longer has time to devote to this work. He is looking for another lab to chair this working group, but he would still like to be involved. M. Miller from Covance Laboratories said that her lab has done some work on tylosin. Getting all homologues of tylosin (A, B, C, D and urea adduct) is the challenge. Residue methods need only identify tylosin A to be effective (CFIA method).
  - CTC WG – Similar to above, Tom Phillips no longer has time to move this project forward and is looking for someone else to chair the group. Concerns were raised that laboratories don't have access to UPC<sup>2</sup> and super critical fluid systems. Also, Greg Hoffman (Zoetis) raised concerns with the limits in FDA – Guide 135 that they are too tight down at drug levels seen in type C feeds. Leo Schilling (Eurofins) volunteered to chair the CTC working group going forward. A Price to provide him with T. Phillips contact info.
  - Fat Soluble vitamins WG – Chairs (K. Riter and D. Inerowicz) reported that 9 samples were sent out to 11 labs for Vitamin A, 8 labs for Vitamin E. The reproducibility and repeatability were very high on these results and wider than typical AAFCO results. They compared the results to the sample weight used for each lab and saw some correlation, but it wasn't definitive. The working group had a conference call in November to discuss where to go next. The group decided it would be a good idea to send more homogeneous samples to the labs, such as infant formula and cheese products (processed cheese slices). The working group would welcome suggestions on where to go next. Discussion from the committee agreed that an increased sample size would probably improve reproducibility. Also, some thought it might not be realistic for one method to cover all vitamin levels in feed. The working group will go forward with a more uniform sample and sample prep and try to have the labs analyze a uniform sample size.
  - Best Practices WG – Sharon Webb and Lawrence Novotny presented to the committee a white paper on best practices guidelines for phosphorus and a method matrix for

various sample types, which will be added to the LM&SC website. An article in JAOAC (50) pg 137, 1975, stated that dry ashing mineral mixes results in low recoveries, which may be why some of the higher concentration samples in the AAFCO check sample program have had issues. The information from this article will be added to the white paper and method matrix. The committee voted that the phosphorus best practices guidelines are complete and the working group should move on to another analysis. After discussion, the committee decided fiber (crude, ADF and NDF) would be next. There will be a call sent out by A. Price and S. Webb to the membership calling for experts to join the working group.

- Sugars (mono- and disaccharide) WG – J. Horst reported that the single laboratory validation was performed by Covance. They complete a paper that has been reviewed and is about to be published on the AOAC website through their Fast Track process. The working group is looking for a sponsor (preferably affiliated) to move the method through to a full collaborative study. Some of the committee raised concerns that not enough labs have the required technology. A solution to this could be to open the collaborative study up to food matrices.
- Mycotoxin WG – Robert Sheridan reported that the working group has sent out AAFCO mycotoxin check samples (201461, 201462, 201463, and 201464) to labs, but is still waiting for results. They are considering removing aflatoxin G1 and G2 from the study, since those can't be spiked into the samples. By next meeting they will have mycotoxin results from each lab using their own individual methods. R. Sheridan also reported that the European Commission has completed a collaborative study for mycotoxin in feed. The only lab that participated from our WG was Romer Labs. The method analyzes for 18 mycotoxins, 13 in positive mode and 5 in negative mode using a single extraction. They matrix match and use isotope labelled standards and can achieve >85% recovery for all mycotoxins. This method is similar to what New York State has been working on and is close to meeting the mycotoxin method needs statement, including matrices required. The working group needs to consider whether this method is fit for the group's needs, since it has already been through a collaborative study. Should this working group join with the European group?
- Quality Assurance WG – Brenda Snodgrass and Louise Ogden were not able to attend but sent in a brief update to Nancy. Currently, the group is working on clarifying recommendations on water conductivity testing frequency. It has been recommended that this working group be made a sub-committee under the LM&SC. The LM&SC chairs agree with this proposal, which will make the working group permanent. Both Brenda and Louise must resign as chairs of the working group since they will be taking on the quality management of the AAFCO check sample program. Srinu Chigurupati (FDA) volunteered to be a chair of the sub-committee and Teresa Grant from NC State volunteered to be a co-chair. Assisting State labs to become ISO accredited and other aspects of lab quality assurance will be the mandate of the new sub-committee.

- Sampling WG – The group (~12 members) is revising the AAFCO Sample Preparation Guidelines for laboratories to be in line with the recently published GOODSamples document. Areas such as sample splitting, explanation of minimum mass, sample quality criteria, etc. will be a total re-write from the previous version. The group hopes to have a draft out about a year from now. An early draft without examples may be available in August. There will be a workshop of sample preparation at the 2016 Mid-West AOAC meeting in Sioux Falls, SD. There will be a hands-on portion to the workshop and vendor equipment to try. The working group will contact the Inspection Sampling Committee to try to create some linkages and start a dialogue. This will be a challenge since the two groups do not speak the same language.
- Multi-Element Validation – R. Sheridan presented the validation data for toxic levels of minerals in pet and livestock food by ICP-MS using microwave digestion. Samples tested were wheat flour, tomato leaves, feed scratch, poultry feed, medicated sheep concentrate. The sample size was 0.25 to 0.5 grams with 2 mL of acid used. Some on the committee expressed concerns that this was quite a small sample size and could be the reason for some reproducibility values being larger than what might be expected. The lab is looking into cryogrinding 10 grams down to 0.5 grams. Sharon Webb shared with R. Sheridan her lab's digestion procedure. The next steps will be to start a working group that could work towards completing a collaborative study, and to review the method needs statement. Some other matrices that could be tested are rice flour and fish meal. Also, it was noted that a multi-element method was published as AOAC 2015.01.
- Upcoming Collaborative Studies and New Method Needs  
There are several methods and method needs that are in the queue. 2012 was the last time that a survey was sent to the Ag community requesting a method needs ranking. Currently, there are more methods in the pipeline than volunteers to take them on. The FSMA task force will eventually identify priority method needs. Fructans will need to be worked on after the sugars method is complete. A vitamin D LC-MS/MS method exists and it can be compared against the method needs statement. If anyone is interested in reviewing the list of method needs, it is posted on the AAFCO website, under the Laboratory tab as follows:  
<http://www.aafco.org/Regulatory/Committees/Laboratory-Methods-and-Services#method-needs>
- Laboratory Curriculum Framework  
Craig Kaml presented on the laboratory curriculum framework for State feed and food labs. The framework is organized into levels: entry, mid, expert, supervisor/manager, and senior manager. It can be used as a career ladder or path, identifying the competencies required to move between levels. A conceptual overview of the framework can be found at:



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<http://incs.ifpti.org/frameworks/Home>. The framework will have links to courses and ways to evaluate training. More information can be found at <http://incs.ifpti.org>.

- FDA Cooperative Agreement:
  - Update from Teresa Bills: There are 24 openings and 20 States have applied for grant funding. The goal is to get all labs ISO accredited and to continue to expand the program.
  - Ruiqing Pamboukian (FDA) presented on the Office of Regulatory Science (ORS) and the FERN network. She outlined the benefits of accreditation as 1) reliable and defensible data, 2) provides decision makers with confidence, and 3) establishing and implementing national standards. The ORS has a State mentor/mentee program which has many benefits such as strong collaboration among partners, enhanced state to state relationships, increased number of accredited food/feed testing laboratories, and building a strong national integrated food/feed safety system. The vision for the future is to promote lab accreditation and best practices, improve MFRPS and AFRPS, maintain a National Surveillance Program, use State data for regulatory action, and faster and more effective response to food safety events.
  - Robyn Pyle presented on APHL activities. She indicated there are 31 labs working on ISO Accreditation. APHL is looking to expand to fertilizer, FIFRA and radiation labs. Currently, APHL has 12 working groups with over 70 members. They are also looking at how to sustain the program after the funding sunsets. A lot of resources on training and discussion forums can be found at [www.APHL.org](http://www.APHL.org). There is a public site as well as a member site. The member site requires a login and has usable examples of documents from labs.
  
- Promotion of Resources for Labs:
  - Nancy Thiex informed the committee that there is a presentation that provides a good overview of AAFCO and the Check Sample Program available in the Feed Bin website. This presentation can be used to promote AAFCO and its lab relevant activities to other groups and committees.
  - Nancy Thiex reported on the completed GOODSamples document that is a free download from the AAFCO website. The objective of GOODSamples is to improve analytical data equivalency amongst laboratories by outlining a scientific and systematic approach to the sampling process. There are several outreach efforts that the GOODSamples working group is involved in, such as webinars, training events, presentations (at AOAC, BERM, MFRPA, Southwest Nutrition conference, etc), and papers. A four day training event will take place in early April, location TBD and possibly in other States at a later time. Also, the Midwest AOAC will host a 1 day sampling seminar.

- Other Topics:
  - Laboratory Centres of Expertise: A. Price and N. Thiex informed the committee on having a network of laboratories with specific expertise in certain chemistry testing areas so that the full range of FSMA contaminants can be covered more efficiently and cost-effectively. Many private laboratories are set up this way. State labs often use this system informally, if they can find a lab to do a test that they can't. The proposal is to formalize the system with some sort of agreement. Many details need to be worked out. The committee co-chairs will be presenting a white paper outlining this concept to the AAFCO BOD on Jan. 20, 2016. The white paper will ask the board to set up a working group comprised of AAFCO members from several different committees, including the LM&SC. The working group would examine the feasibility of setting up this sort of agreement and what it might look like. Initial discussions with the BOD on Jan 17 were positive.
  - Education and Training committee liaison update: Mark Stenske reported is our committee liaison and reported on this activity. Currently, it is felt that the relationship between our committee and Education and Training is under-utilized. There will be a survey sent around to identify what are the training needs. Once some training needs have been identified, the Education and Training committee could help facilitate this.
  - CFIA Method Update on Organic Arsenicals: Lise-Anne Prescott (CFIA) presented her lab's new method to determine arsanillic acid, roxarsone and nitarsone residues in feed. There were several challenges encountered along the way, e.g. dirty extracts, co-elution of interferences, and IS linearity. Their current method uses LC-MS/MS in negative ionization mode. The mobile phase and extraction solutions have been optimized and method validation has been completed.
  - Measurement uncertainty (MU) in Protein Combustion method: Heidi Hikes presented her lab's uncertainty budget for their protein analysis method. They used AAFCO check sample data points from over a year of testing, on different instruments with different analysts to obtain the analytical MU. They also attempted to estimate the sample preparation MU by performing replicate splits and replicate weighings for each split. This gave a robust uncertainty estimate for their method and outlined a good approach to incorporate sample prep uncertainty.
  - Multi-mycotoxin LC-MS/MS method by Romer Labs: Michael Prinster from Romer Labs presented their LC-MS/MS method for analyzing mycotoxins. Their method uses isotope labelled internal standards (IS) to correct for suppression and enhancement. For compounds that don't have internal standards, they used one that closely matched. For example, use DON IS for 3-ADON and 15-ADON. This way, all compounds had >85% recovery on 18 analytes. They also showed how the use of the isotope labelled IS can be minimized, which reduces the overall cost of the method. Romer used this method to participate in the EU collaborative study mentioned above. That study had an option to use or not use internal standards.

- Roundtable Discussion:
  - FDA has formed a working group to review the Animal and Food Standards (Standard 10). Several members of the LM&SC will be working on that. Comments need to be in ASAP and the draft will be completed by April. The current standards expire in 2017. The next version needs to be vetted prior to the current version's expiration. This document is on a 3 year review cycle.
  - G. Hoffman from Zoetis is requesting assistance from a laboratory to perform some testing on 30 to 40 samples. This will be needed near the end of the year. Please contact Greg Hoffman if you are interested.

Action Item Table:

Responsible	Item	Action	Timing / Status
All	Reviewing Committee Roster	Review the committee roster and let Aaron Price know if you would like to be removed or know someone who should be added to the committee.	Prior to next meeting
A. Price	CTC working group	Provide Leo Schilling with the contact info for Tom Phillips.	ASAP
D. Inerowicz and K. Riter	Fat-soluble vitamins working group	Decide on and send out more homogenous samples to labs for vitamin A and vitamin E analysis. These might be sliced cheese or infant formula.	Prior to next meeting
A. Price and S. Webb	Best Practices working group	Send out a call to membership looking for experts on fiber analysis methods.	Prior to next meeting
R. Sheridan and A. Price	Multi-element working group	Start a new working group to review the method needs statement and move towards a collaborative study.	Prior to next meeting
Education and training comm. liaison	Survey of training priorities	Send a survey to the committee to identify priority training needs.	Prior to next meeting
All	Analytical help to Zoetis	Contact Greg Hoffman from Zoetis if your lab is interested in helping them with some analyses.	ASAP
A. Price	LM&SC conference calls	Schedule next conference calls of the LM&SC with members and advisors.	Mid-March and Mid-May of 2016.