

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

January 14, 2015 8:00 am to 5:00 pm San Antonio, TX

## **COMMITTEE RECOMMENDATIONS:**

1. None

## **BOARD RECOMMENDATIONS:**

1. None

2.

### **ASSOCIATION ACTIONS:**

1. None

2.

# <u>Committee Participants: (C \*Committee members, \*\* Advisors)</u>

Aaron Price*	Canadian Food Inspection Agency	aaron.price@inspection.gc.ca	
Louise Ogden*	MN Dept. of Ag.	Louise.ogden@state.mn.us	
Andy Crawford**	Consultant	andy@crawford.org	
Jason Kong*	Ohio Dept. of Ag	jason.kong@agri.ohio.gov	
Sharon F. Webb*	UK Div. of Reg. Services	Sharon.webb@uky.edu	
Kristi McCallum*	Colorado Dept. of Ag.	Kristina.mccallum@state.co.us	
Ruiqing Pamboukian*	FDA / ORA	Ruiqing.Pamboukian@fda.hhs.gov	
Heidi Hickes*	Montana Dept. of Ag	Hhickes @MT.gov	
Nancy Thiex*	TLS / AAFCO Consultant	nancy.thiex@gmail.com	
Ken McManus*	Maryland Dept. of Ag.	Kenneth.mcmanus@maryland.gov	
Teresa Grant*	North Carolina Dept. of Ag	Teresa.grant@ncagr.gov	
Robert Sheridan*	New York Dept. of Ag	Robert.sheridan@agriculture.ny.gov	
Joshua Arbaugh*	West Virginia Dept. of Ag	jarbaugh@wvda.us	
Alex MacDonald**	Pharma Science	beemac201@aol.com	
Jennifer Mirabile*	New York Dept. of Ag	Jennifer.mirabile@agriculture.ny.gov	
Brenda Snodgrass*	Oklahoma Dept of Ag	Brenda. Snodgrass@ag.ok.gov	
Daniel Berg**	Covance Laboratories	Dan.berg@covance.com	
Greg Hoffman**	Zoetis	Gregory.a.hoffman@zoetis.com	
Melissa Miller**	Covance Laboratories	Melissa.Miller@covance.com	
Mark Coleman**	Elanco	mcoleman@elanco.com	
H. Dorota Inerowicz*	Office of Indiana State Chemist	inerowic@purdue.edu	
Dale Hill**	ADM Alliance Nutrition	Dale.Hill@adm.com	



Ken Riter**	Nestle Purina	ken.riter@purina.nestle.com	
Maria Ofitserova**	Pickering Labs	Maria_o@pickeringlabs.com	
Mark Stenske*	Michigan Dept of Ag and Rural Dev.	stenskem@michigan.gov	
Dennis Givens**	Cargill Corn Milling	Dennis_givens@cargill.com	
Michele Swarbrick*	Minnesota Dept of Ag	Michele.Swarbrick@state.mn.us	
Gale Hagood*	Mississippi State Chem Lab	ghagood@msd.msstate.edu	
Lei Tang*	FDA/CVM	Lei.Tang@fda.hhs.gov	
Faye Wei	FDA/CVM	Faye.wei@fda.hhs.gov	
Amanda Andersen	Kansas Dept of Ag	Amanda.andersen@kda.ks.gov	
Ametra Berry	Georgia Dept. of Ag	Ametra.berry@agr.georgia.gov	
Barbara James	Potash Corp. – Aurora	bjames@PCSphosphate.com	
Mark Empie	Empecor	empecor@outlook.com	
Richard McKinley	SPF	rmckinley@diana-petfood.com	
Becky Owen	FDA/CVM	Rebecca.owen@fda.hhs.gov	
Gina Clapper	AOCS	Gina.Clapper@AOCS.com	
Tom McKamey	Silliker Inc.	tom.mckamey@silliker.com	
David Levin	Covance Labs	David.Levin@covance.com	
Evelyn Cadman	FDALabels.com	ecadman@FDALABELS.com	
Chuck Ramsey	Envirostat	chuck@envirostat.org	
Yvonne Salfinger	AFDO consultant	yhale@aol.com	
Scott Bantam	Nebraska Dept of Ag	Scott.bantam@nebraska.gov	
Abigail Hiles	Romer Labs	Abigail.hiles@romerlabs.com	
Mary Koestner	Missouri Dept of Ag	mary.koestner@mda.mo.gov	
Jonathan Goodson	Eurofins Corp.	Jonathan.goodson@evonik.com	
Isaiah Isakson	FDA/ORA	Isaiah.isakson@fda.hhs.gov	
Jimmie Ward	Mars Petcare	Jimmie.ward@effem.com	
John Szpylka	Silliker	john.szpylka@silliker.com	
Jeff Ruffing	Mars Petcare	Jeffrey.ruffing@effem.com	
Ken Gilmurray	JBS USA	Ken.gilmurray@jbssa.com	
Karin Helen Ekborg	Mars Petcare	Karin.ekborg@effem.com	
Sharon Benz	FDA CVM	Sharon.benz@fda.hhs.gov	
Sara Williams	Office of the Texas State Chemist	smw@otsc.tamu.edu	
Paul Mostyn	Westway Feed Products LLC	Paulm@westwayfeed.com	
Ashli Brown	Mississippi State Chem Lab	abrown@mscl.msstate.edu	
Jessica Flowers	Georgia Dept of Ag	Jessica.flowers@agr.georgia.gov	
Keith Mizwicki	SEM Minerals	Keithmizwicki@mineralslp.com	
Patty Lucas	Florida Dept. of Ag. & Consumer Serv.	patricia.lucas@freshfromflorida.com	
Carolyn Siebecker	Hampshire Pet Products	csiebecker@hampshirepetproducts.com	



Zhirong Jiang	Ajinomoto	Jiangz@ajiusa.com	
Adela P. Ramic	Minnesota Dept of Ag	Adela.parganlija-ramic@state.mn.us	
Tai Ha	Ne Dept of Ag	Tai.ha@nebraska.gov	
Erika Weltzien	Rothsay	Erika.weltzien@rothsay.ca	
Jean Hobus	Central Garden & Pet	jhobus@central.com	

### Committee Report:

Committee Activities

**ACTION: Revised Agenda Approval** 

MOTION: "Motion to accept the revised agenda for the 2015 Annual Meeting of the Lab Methods and Services Committee" passes (all in favor)

#### Committee Minutes:

- Committee attendees introduced themselves.
- The Laboratory Methods and Services Committee (LM&SC) membership roster and industry advisor list was reviewed and A. Price indicated new appointments. People wishing to be added or eliminated from the list should contact Aaron Price.
- The new AAFCO website was presented to the group by Sharon Webb. The website has the same address (www.aafco.org). A media inquireies section has been added to the page. Only the laboratory portion was reviewed. Links to the Check Sample Program and PT reporting website are available, with all programs, pricings and schedules present. Working group links can be found as well as a document library and ISO accreditation information.
- FSMA Implementation Task Force (T. Grant): Labs have spent a lot of time getting ready for FSMA. Education and information have been a big part and not every state is involved. Those that aren't might not be aware of ISO information and what they need to do to be prepared. Question whether any new contaminants are going to be added? If so, what are the levels of concern? These may not be known, and more time is required by the labs to prepare information. J. Mirabile and R. Sheridan will also represent our committee on this task force along with T. Grant at future meetings.
- AAFCO Board Survey on Committee Structure (A. Price and N. Thiex): In 2010 AAFCO combined several committees and the Board has distributed a survey to ask questions about these changes. The changes made do not seem to have much impact on this committee. Some members commented that it makes sense to have the Finance sub-committee report to the AAFCO Board and not the Strategic Affairs committee. A response will be prepared by A. Price and sent to the committee for approval prior to submitting to the Board.
- Updates on collaborative studies:



- Dietary Starch (update provided by N. Thiex): Method has achieved First Action and will be published in EOMA and then the AOAC Journal.
- Fatty Acids AOCS (update provided by Gina Clapper). Method includes two direct methylation procedures and is a one-pot method. Choice is made (alkali only or acid alkali extraction) depending on the matrix. Acid alkali is needed for extruded products. Method numbers are Ce 2b-11 and 2c-11. Method validated for tallow, chocolate cake mix, evaporated milk, canned cat food, butter blend, DHA/EPA supplemented juice, etc (24 samples). In general, the AOAC food triangle was used. Direct methylation methods done between Cargill and Silliker labs. Both methods are published on AOCS website and available for purchase.

# • Working Group updates:

- CTC WG A. Price reported in Tom Phillips' absence: Kentucky sent 50 samples analyzed using plate assay method to Tom for testing using his HPLC method. Minnesota and Michigan would like to participate in this WG.
- Tylosin WG A. Price reported in Tom Phillips absence: Has had promising results using UPC2 instrument, but is waiting to get more instrument time. FDA is not opposed to MS/MS methods, but want to make sure that State labs have this equipment if the method is to become an official method. Many states are moving toward Triple Quad instruments which might be overkill for verifying drug levels. Sample can be diluted down, but accuracy/precision is sacrificed. Also, bench space is limited for this equipment. Often, triple quads shared with other sections do not want to dirty their instruments with feed samples.
- Fat Soluble vitamins WG K. Riter reported that they will be preparing and sending out samples to the participating labs in the next month or so. The results should be analyzed by the next meeting and will be reported. Dale Hill from ADM is providing most of the samples. Some labs have dropped off the participant list, but Ken clarified that since method performance is being tested, labs using duplicate methods were removed. Other labs can later join in on the full collab study.
- Sugars (mono- and disaccharide) WG D. Berg reported that the goal of this group is to develop a method appropriate for feeds and pet foods. Ideally to achieve quantitation of 6 mono and disaccharides down to 0.1% in difficult matrices (might not be achievable). Method being studied by 6 different laboratories uses 50% ethanol extraction and 3 different detection techniques (HPAEC-PAD, HPLC-PCD, HPLC-ELSD). Three labs are running samples in duplicate to generate method performance data. Results to be reviewed at the next meeting. The intent is to publish the method and ideally to seek AOAC Official Method status.
- Mycotoxin WG R. Sheridan reported that the group is looking for methods that work well. Addition of incurred samples available through the AAFCO CSP has helped this group. The data from the mycotoxin check sample program is being reviewed. Issues with T2 and HT2 since kits report these together however MS methods can separate.



Trilogy is the reference lab preparing the samples and they use a different extraction method for each. 84:16 acetonitrile/water works well for AFLA, DON, ZON, OCR-A, HT-2 and T-2, but FUM requires more water (50/50 ACN/water). The WG is still looking for other extraction methods, but the most ideal method is becoming obvious. Interferences (suppression/enhancement) can be solved with isotope labelled internal standards, but this is expensive. The group is focusing on AFLA right now because of the low detection limit. If you can get AFLA, you can get the rest of them. The group is looking to find the clean-up information from lab methods as well as the extraction protocols.

Best Practices WG – Sharon Webb reported that the group is currently focused on phosphorus. High P levels are the most challenging. Dry ashing makes P, Ca, Cu, Fe, Mn and Zn very insoluble in the acid solubilization method. Next steps are to update the matrices and method codes. P should be completed by August after which the group will start to tackle another difficult analyte. S. Webb is replacing L. Novotny as the chair of this group.

### • FDA Cooperative Agreement:

- o FDA/ORA/ORS and FERN network update by R. Pamboukian from the Office of Regulatory Affairs. The Office of Regulatory Science (ORS) has a Lab Accreditation Program and they coordinate field sample collection and testing. Roles include methods and cooperative agreement for Food Emergency Response Network (FERN). Programs include pesticides, dioxins, mycotoxins, colour additives, filth, chemotherapeutics, select radioisotopes and they interact with many centers such as CFSAN, CVM, etc. The FERN network was recently used to carry out high volume microbiological surveillance on avocados. Also, peanut butter from Peanut Butter Corp 2008. The ORA is involved with external collaboration and building a food/feed safety network. Under FSMA the activities will include sampling to help ID risks and figure out where preventive controls can be used.
- QA/QC Working Group ISO Accreditation Program/Quality Mgmt: Group presented on the following topics: internal audits (T. Grant), PT and QC for ISO 17025 (L. Ogden), reporting results (J. Kong), root cause investigations (L. Ogden). Group's presentation should be available on the AAFCO website.
- Sampling Working Group update given by N. Thiex. There will be a special section on sampling in the J of AOAC International in the March/April 2015 issue. This will be available for free download from the AAFCO website following publication. In August 2015, GOODSamples should be available. K. McManus presented results from his sample study. The objective was to define the variation in feed sample preparation. Fourteen labs processed unground feed samples and each sent them back to Maryland Dept of Ag for analysis. N. Thiex and C. Ramsey reported on the concept of minimum mass and the decision unit. At this point the group doesn't know what this is, but it is needed to represent the target material. It will be highly different for a textured feed vs



a pelleted feed, therefore there isn't a consistent amount for each type of material. Particle size is a key aspect in the theory of sampling. This working group will need to establish sampling protocols and processes to split out samples for retains, etc. If keeping a reserve sample is part of a lab's sample processing, then the original sample will need to be twice the minimum mass needed. Most committee members indicated that they're using rotary splitting in their labs. A working group should be formed soon to look at these problems and come up with procedures for labs to follow and ways to estimate the sampling error.

- An update on the Microscopy Training Seminar was given by J. Arbough. He indicated
  that the course held in Manhattan was quite useful and that the old AAFM microscopy
  manual was used. Not enough time to get into minerals or spot testing. Results from
  the course survey were not available for review at this time.
- The National User Group is working with GNSI on upgrades around ELEXNET and increasing outreach efforts. Working on a proposal and roadmap for training sessions in 2015 for up to 10 labs.
- The Training Workgroup Update was given by Mark Stenske. They are working on the training competencies outlined by IFPTI for feed laboratories. Once competencies are identified, training courses and modules will become available. The group is looking for subject matter experts to identify tasks and needs. A survey should be produced by the group to determine what kind of training is needed. The APHL (Association of Public Health Laboratories) website can be found from the AAFCO webpage and it contains a lot of training information, extending beyond ISO requirements.
- The APHL update was given by K. McCallum (presenting for Yvonne Salfinger) who
   reported on activities and initiatives carried out by the group in support of the grant.
- FDA BSE Feed Safety Enhancement Grant: Laboratory Presentations
  - Sharon Webb presented on work at Kentucky looking at heavy metal analysis in feeds.
     Presentation to be posted on the LM&SC page of the AAFCO website.
  - Kristi McCallum presented on work in Colorado looking at salmonella analysis in pet food and treats. Presentation to be posted on the LM&SC page of the AAFCO website.
  - Abe Brown discussed accomplishments of the grant up to this point. The grant is now 10 years old and was put in place to enhance feed safety programs to comply with BSE rules. In 2010 grant was renewed and 12 grantees were awarded out of 17 (total of \$2.3 M awarded). The program was expanded to other commodities other than prohibited risk materials in 2010. 5000 7000 inspections conducted and 1000's of samples collected and analyzed.

## • Other topics of interest

 K. McCallum presented on her lab's amino acid analyzer. She mentioned that she would like help from someone in the group to determine other possible applications. MS
 Powerpoint presentation to be posted on LM&SC page.



- A. Price presented on method development and validation efforts at the CFIA to lower the LOQ for ractopamine in feed. The current method limit is 1 ppm and the new limit is 50 ppb. MS Powerpoint presentation to be posted on LM&SC page.
- O H. Hickes reported that the Midwest AOAC would be jointly hosted by Montana, North and South Dakota, in Bozeman, MT in June 2015. They are soliciting speakers and symposia chairs. Areas covered will be pesticides, paraquot, mycotoxins, ISO 17025, method validation and measurement uncertainty. Feed, forages and fertilizers will also be a focus. Covance and Silliker Labs will be participating and they are trying to organize a workshop on ICP operation and troubleshooting.
- The Association of Southern Feed, Fertilizer, and Pesticide Control Officials (ASFFPCO) will meet June 12-15, 2015 in Indianapolis, IN. Program details not available right now but a brochure will be created. Dorota Inerowicz is the contact and T. Grant will be chairing a session.
- The Animal Feed Regulatory Program Standards (AFRPS) were discussed with Tim Weigner. Challenges are seen since every state is different. Should move toward common QA/QC practices and AAFCO QA/QC Guide will probably be recognized for accreditation purposes. FDA wants to be able to rely on state lab results. 12 states have signed contracts to participate in AFRPS. This does not require a state to changes its laws and regulations.

#### • Round-table topics

- A Price asked the group whether the monthly conference calls of the committee were working well. People commented that they were valuable and should continue.
   Anyone interested in chairing them, please let Aaron know.
- Dr. Sharon Benz from the CVM enquired about whether ingredient definitions can include the analytical methodology submitted as part of registration. What would be the best way to distribute these methods? Should these methods be distributed widely or are they for regulators only? S. Webb and the Best Practices Working Group will look into this.
- Becky Owen from CVM asked labs to please continue participating in medicated feed assay drug transfer methods. The state lab input is important since these will be the users. They need to know if labs have the equipment and capabilities, etc. This could be something added to the AgLabs Listserv.



# Action Item Table:

Responsible	Item	Action	Timing / Status
All	Reviewing Committee Roster	Review the committee roster and let Nancy or Aaron know if you would like to be added to or removed from the committee.	Prior to next meeting
A Price	AAFCO Board Survey	Prepare response to survey and send to committee for approval. After approved, send to AAFCO Board.	Mid to late February
Fat Soluble vitamins WG chairs	Method performance study	Send out samples and instructions to participating labs.	Early to mid-February
Sugars WG chair	Method performance study	Compile lab participant results and present at the next annual meeting.	Early August (for next AAFCO meeting)
Mycotoxin WG participants	Method performance review	Working group members to send R. Sheridan and his group the extraction and clean-up details of their mycotoxin method(s), if not already done.	Mid to late March
S. Webb and Best Practices working group	Methods submitted as part of ingredient registrations	S. Webb and her group to look into what could be done to distribute the methods submitted to CVM as part of registrations.	Early August (for next AAFCO meeting)
A Price	LM&SC conference calls	Schedule next conference call of the LM&SC with members and advisors.	Late February to early March