# MINUTES FROM THE MEETINGS OF AAFCO'S LABORATORY METHODS AND SERVICES COMMITTEE

January 19, 2011; 8:00- 5:00

at

# Tradewinds Hotel, St. Pete's Beach, FL

## Present were:

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Nancy Thiex opened the meeting. After introductions the following issues were covered:

Approval of Agenda – The agenda was approved. (attached (#1)).

Approval of Minutes from the September Meeting – The Minutes from the meeting held on September 28, 2010 in Orlando, FL were approved (attached (#2))

Status of FDA Submissions of Methods for Medicated Feed Additives and other Drug related Projects – Thiex reported that submissions of drug methods were on track -

- Sulfamethazine Siegel reported that the data had been collected but the package needed a "Table of Content" before it was ready for submission to FDA.
- Monensin, Narasin and Salinomycin An amendment to the submission has been submitted providing recovery data and interference data. FDA CVM approved the method for narasin. The review for salinomycin is underway. It wasn't until the approval for narasin was obtained by Elanco that AAFCO they had not requested approval for monensin simultaneously. Elanco is submitting a request for review for monensin and FDA will review the "monensin" part of the method after receiving the request.
- CTC Tami Stolzenbach has finalized the chromatographic conditions for the CTC feed residue method but while testing the method with some customer samples she saw some small possible interferences. At this point she wants to identify these possible interferences and she is running LC/MS confirmatory testing prior to beginning the validation work. Note that Tami will likely not being able to continue working on this subject due to restructuring activities within her company
- MGA Kurt Johnson was not present to provide an update.
- Tylosin Tommy Phillips still working on issue. No news/no recent activity.
- Antibiotic in DDG Survey McCurdy provided a short statement (attached (#3))
- State Survey of LC-MS capabilities. 20 States responded so far. Additional responses should be sent to Mark Coleman. Deadline of Jan 31.2011. Mark Coleman will report on findings at our next meeting. Example survey attached (#9).

Starch & Fructans Methods – Thiex reported that samples had been prepped for the starch collab and that the study protocol was in the final stages of editing before submission to AOAC. Mary Beth Hall is also starting work on a fructan method.

Relationship w/ISO – Cantrill updated everyone on the activities of ISO especially the lack of US involvement with ISO Committee TC34/SC10 covering Animal Feeds. Iran has been awarded the Secretariat for this Committee. In general USA is not much involved in this organization even though more and more countries are subscribing to its standards and incorporating them into their legislation. Discussion followed on the need for US to get more involved. AOCS can assist with some of the leg work but require some funding to offset cost.

It was agreed to try to develop White Paper on this issue (task force consisting of Reimann, Cantrill, Coleman and Hall). Input should be sought as well from Scott Coates (AOAC CSO) as well as Atcheson and Flowers (AOAC BOD members). Lars to update w/ help from Amy and AOCS.

Fatty Acid Profile Method Validation – Amy Johnson reported that a collaborative study covering 34 samples (mainly food matrices) and involving "single pot" and "double pot" procedures was underway. Not all results are in yet and some labs dropped out. Don Palmquist's method for feed ingredients (mainly forages) is almost ready for collaboration. AOCS looking for collaborators – hope to send samples out mid-February. For further information contact Amy Johnson w/ AOCS.

Vitamin A Work Group—Thiex reported that a Foodshield Working Group Internet site has been set up for the Vit A Working Group. The working group currently consists of Regina Wixon, Mary Koestner, Jennifer Kraus, Rajan Gupta, Dale Hill, Nancy Thiex, Ken Riter and Vicki Siegel. People interested in joining should contact Nancy Thiex. It was noted that all Vitamin A in feed is added. Reimann noted that AOAC has formed a working group for Vitamin A in infant formula chaired by Jon DeVries, General Mills.

Vitamin D – Thiex reported that Larry Thompson was not able to attend. Study status unknown. Need to form a Vit D Work Group. People interested in joining such a work group should contact Thiex. An ELISA based method is under review. Reimann noted that AOAC has formed a working group for Vitamin D in infant formula chaired by Don Gilliland, Abbott.

Amino Acids Project: – Yan Zhang summarized the result of the study of the instrumental techniques and provided a quick update as to the status of the study focusing on digestion techniques. The latter study involved 8 labs assaying 5 samples in triplicate. To date 4 labs had returned digestates to AOCS. A summary of project to date attached (attachment (#7).

Dried Distillers Grain Particle Size Project: – Yan Zhang summarized the result of a study of the impact of particle size on methods for analysis of dried distillers' grain. Project summary attached (#9).

Sugar methodologies – Jeff Horst, Jeremy Shia and Lazlo Torma each presented their data. All were based on HPLC-ELSD. Reimann to compile the results. The need for reference materials was discussed as well as the possibility of separating extraction from quantitation and including an internal standard. It was agreed that a conference call in mid February among the participants would be the next step.

Mycotoxin Work Group – Siegel reported that the Mycotoxin Work Group headed by Lu Wetzler with 9 members and several more "pending" has a site on FoodShield. Focus on multi-mycotoxin screening methods. Anyone interested in joining should contact Siegel. Currently methods from Romer, FDA, State of NY and State of TX are under review.

Karen Stephani reported that her lab got a grant from FDA to among other things develop/validate a multi-residue method for mycotoxins. Currently the method is based on a modified QUECHERS extraction for most mycotoxins, water/methanol for ochratoxin A & fumonisin B with two injections on LC-MS/MS. Summary attached (attachments #4 and #4b)

Vickie Siegel reviewed the 2010 mycotoxin data from 9 states covering aflatoxin, fumonisins, vomitoxin and zearalenone.

Christy Brewe presented Romer Labs' method based on two extractions, two injections using LC-MS/MS and isotope dilution to reduce interferences. Uses the Phenominex Gemini column. Method to be published. Will provide application brief. May start collab study with other people in the working group. A comparison of different methods is attached (#5)

Quality – The new FDA regulations may result in regulatory labs having to be accredited and AAFCO having to prepare general operational procedures

Grinder – MacDonald described a tumbler for mixing samples after grinding prepared by Purdue University's machine shop and successfully used by MacDonald. He is interested in having others join him in having Purdue making several units. For further information contact Alex MacDonald.

Method Needs Statements (MNS) – Thiex reported that the most updated versions of each method needs statement on the AAFCO web site. A draft template for preparing such documents is posted there as well.

- Fatty Acids –Price to send current version of MNS for fatty acids to Amy at AOCS.
- Total Metals completed Price reported the method for metals by ICP-OES developed by CFIA. The improved method did not meet the MNS standards in terms of LOQ especially for the toxic metals.
- Pesticides Stephani presented the first draft of a MNS for pesticide. Price to incorporate Canadian limits. An updated MNS will be posted for review on the AAFCO web site and comments should be sent to Stephani. Attached (#6).
- Amprolium completed –looking for person.
- CTC completed looking for person to head up collaborative study activities including SLV,
- Mycotoxins completed see summary of Christine Nochetto's presentation later
- Bacitracin / Bacitracin MD completed looking for person to head up collaborative study activities including SLV.
- Vitamin D Draft completed needs to be approved and posted on web site.
- Vitamin E Draft completed needs to be approved and posted on web site.

#### **Upcoming Meetings**

Annual AOAC Meeting – Thiex reported that proposed symposium was not accepted. The process requires that an outline be submitted almost 1 year in advance. We need to advertise / promote our sessions better.

Annual Mid-West AOAC Meeting – Scheduled for June 6-9 in Lincoln, Nebraska. Very active program covering among other things testing for endocrine disruptors, mycotoxins, food allergens, fatty acids and dietary starch as well as feed/fertilizer sample prep and pesticide formulation sample collection.

Respectfully submitted,

#### **Lars Reimann**

Eurofins Scientific, Inc.

## Attachments:

- 1. Agenda January, 2011
- 2. Meeting Minutes September 2010
- 3. FDA DDG Survey update
- 4. and 4b Mycotoxins NY Dept of Ag
- 5. Mycotoxin Methods Comparison
- 6. Pesticide Method Needs Statement Draft
- 7. Amino Acid Project Summary
- 8. DDG Particle Size study
- 9. LC-MS/MS Survey