# MINUTES FROM THE MEETINGS OF AAFCO'S LABORATORY METHODS AND SERVICES COMMITTEE August 2, 2009 8:00-5:30 in Washington, DC

#### Present were:

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Yan Zhang	yzhang@ethanolresearch.com	Natl. Corn-to-Ethanol Res Ctr

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

**Approval of Agenda** – The agenda (attached).was approved following the addition of vomitoxin (DON) and amino acid methods report from Dr. Zhang

**Approval of Minutes from the January Meeting** –The Minutes from the meeting held on January 21, 2009 in Tucson, AZ were approved. (attached)

**Annual Report** – Thiex mentioned that an Annual Report had been submitted to the AAFCO Board. (attached).

## FDA Submissions -

**Lasalocid** (VFM 005-918) – Thiex reported that FDA had approved the method and this project therefore was completed.

**Oxytetracycline (VFM 005-919)** - Thiex reported that the method has been submitted to FDA. McCurdy reported that it had not reached his desk yet and promised to check on status.

**Decoquinate (VFM 005-920)** – Price reported that submission was held up pending collection of product labels. There is time pressure to complete the package since Alpharma has already put FDA on notice that the package is on its way.

**Sulfamethazine (VFM 005-886)** – Siegel reported that she had been able to locate the original study documents and would put together a package for submission to FDA.

## Communications -

**LabDir (foodshield.org)** – Wetzler reported that a web training session would be scheduled in the near future to help States getting the most out of the site including updating their listings. Industry has limited access but must register first. Currently the most up-to date information regarding the AAFCO methods and Services Committee can be found on the AAFCO website

AAFCO Website (aafco.org) - The link for accessing the most recent lab committee related

information and be found on http://www.aafco.org/AAFCONewsInformation/LabMethodsServicesCommitteeINFO/tabid/134/Default. aspx.

## <u>Methods -</u>

**Fatty Acids** – Dr. Palmquist gave a basic update on the quantitation of fat and fatty acids recommending replacing the analysis and labeling of crude fat with fatty acids as these were far more nutritionally relevant. He was advocating his "one pot" method. As a source for further information about fatty acid analysis he recommended <u>http://www.lipidlibrary.co.uk/analysis.html</u>

Clapper summarized AOCS position on fatty analyses – AOCS has two methods ready for collaboration (AOCS Ce 1k-09 and AOCS 1j-07). Steve Hansen w/ Cargill is the study director. The plan is to have the studies completed by years end.

• A comparison of the two methods led to the following questions:

- Does the hexane/heptane mixture extract all the lipids or is it too non-polar?
- Has the need for adding an antioxidant been demonstrated?
- What are the benefits of the BF3 catalyst relative to a H2SO4 catalyst?
- Is refluxing needed?

The method needs statement would be forwarded to Clapper and Palmquist for their comments. Discussion followed concerning making a quick comparison of the two methods using samples such as full fat oilseed meal, corn, pelleted alfalfa, liquid sweet feed, pet food etc. Palmquist thought he could find 3 labs and Reimann and Thiex also tentatively volunteered. The two Fatty Acid presentations are attached.

Antimicrobials in distiller's grain – Dr. de Alwis reported that her LC-MS/MS (iontrap) method would be published within the next three months in FDA's LIB publication together with spike recovery data. The method covers 13 antibiotics. It uses the presence of Virginiamycin M as an indicator of the presence of virginiamycin but does not \check for the Virginiamycin S factor or any of the other factors present in the pure drug. She will also evaluate the merits of using a LC- MS/MS (triplequad). She was firm that no survey data would be published as part of this project. Alex MacDonald was unable to attend and provide an update on Phibro's success due to recent hospitalization. In a recent note he stated that some issues were still being worked out.

**Tylosin** – Phillips reported on the status of the method. The method should cover all stages of the drug from premixes to residues. He is currently collecting samples and is especially in need of samples representing combinations. In the meantime he is working on further developing the method. Residue levels in feeds down to 1PPB a challenge while slightly higher levels look good.

**MGA** – Thiex shared some recent data indicating that R-Biopharm's test kits may work well. The method initially provided by R-Biopharm used very small sample sizes that in Thiex' hands led to highly variable results. However, when she increased the sample size the results got much better. Thiex will continue to further explore the method.

**Amino Acid Analysis** – Tom Wheat presented his data showing the results from analyzing a variety of samples using precolumn derivatization (AccuTag) followed by UPLC combined with a UV/Vis or fluorescence detector. The data indicated a run time of 10.5 min between shots and cv's in the range of 1%. Yanhong Zhang presented the data from ring trials sponsored by her company, Corn-to-Ethanol Research Foundation, as well as AOCS/USB. In both trials amino acid digestates were sent to the participating labs for analysis. The data indicated that the between lab variability among labs supposedly using same analytical processes was so large that no statistically sound assessment of the relative bias and other performance characteristics specific to the individual methodologies. It was agreed to continue study to better determine the optimal methodology for determining the amino acid composition of digestates. Several labs indicated that the study had highlighted weaknesses within their processes and expressed interest in participating again with hopefully a better outcome. People interested in participating should contact Yanhong Zhang. The two amino acid analysis presentations are attached

**Starch** – Thiex reported that Mary Beth Hall's method was ready for collaboration and would distribute the method to labs interested in being collaborators. Discussion followed about how and where to get and manage the funds needed to compensate AOAC for getting the method through the AOAC validation process. Bill Hall explained how he had been successful in raising \$220,000+ to cover over the next three years AOAC costs for validating methods of interest to the fertilizer community. Reimann offered to contact any of the following trade associations - PFI, AFIA, NGFA and AAFCO – to see if they would facilitate the funds transfers without any administrative charge. (Update – AFIA willing to support this).

**Crude Fiber, Ankom Method –** Thiex reported that she had not heard from Brian Layton for quite some time.

## Method Needs Statement Updates:

**Minerals** – Thiex reported that the study director, Terrence Field w/ CFIA, was updating the needs statement.

**Fructans** – Fructans are mainly an issue in forage and grain based feeds although it is used as an additive to some pet foods. Price/Thiex will send the current needs statement out for feedback with a 14 day response period. The needs statement is attached.

**Vitamin D** – It was agreed that it is important for a method to be able to quantitate both D2 and D3 even though D3 is almost exclusively used to fortifying feeds. However D2 is naturally occurring in some plant materials. It was agreed that the complexity of the issue required the formation of a committee to address issue. Hill and Thiex will work on this issue.

**Vitamin E** – It was agreed that the complexity of the issue required the formation of a committee to address issue. Hill (ADM manufactures Vit E products) and Thiex will work on this issue. It was agreed that all results should be reported in mg Vit E rather than IU due to varying factors used to correlate the two.

**Pesticides** – Tom Phillips offered to help with the project. Main issue is to identify the pesticides such a method should cover. It was suggested we look to FDA for such information. Currently not a lot of momentum.

**Prohibited Animal Protein –** No update from Dr. Herrman. Mike Myers with FDA has validated a PCR method. It was agreed to discontinue this subject.

Method Needs Template - Price offered to update and distribute a draft template.

## **Business Issues:**

**AACO Laboratory Accreditation Program** – Discussion centered around an inexpensive way to facilitate the accreditation of State Feed Labs to standards similar to ISO. A committee consisting of Thiex, Phillips and Webb would review the options and present an update at the next meeting.

Respectfully submitted,

#### Lars Reimann

#### Eurofins Scientific, Inc.

Attachments: Agenda August 2009, 2009 Lab Committee Annual Report, AAFCO Lab Minutes 012109, Tom Phillips' Tylosin presentation, Yanhong Zhang's amino acid presentation Tom Wheat's amino acid presentation, Fat Analysis – AOAC, Fat Analysis – Palmquist Fructans Need Statement