

Annual Report
AAFCO LABORATORY METHODS AND SERVICES COMMITTEE
Nancy Thiex, Chair
John D. McCurdy, LuAnn Wetzler, Louise Ogden, Aaron Price, Vice Chairs

The Laboratory Methods and Services Committee met three times during the past year. The Committee met at the AAFCO Annual Meeting in Washington DC on August 2, 2009 with 34 people in attendance. The Committee also met at the AOAC International Meeting in Philadelphia, PA (September 15, 2009) with 28 people in attendance. The committee met last at the AAFCO Midyear Meeting in Redondo Beach, CA on January 20, 2010 with 31 people in attendance.

ELECTRONIC MAILING GROUP – Submitted by Aglabs Administrator Richard Larson. The AgLabs mailing group continues its success in facilitating communication and resource sharing among feed control laboratories. The AgLabs listserv is hosted at FoodShield.org.

As of June 21, 2010, there were a total of 428 subscribers, with 380 representing all states except Alaska and Arkansas. There are 11 from EPA, 15 from FDA, 10 from USDA, 1 from DHS and 11 from Ag Canada. An approximate breakdown of subscribers by states follows:

Alabama	5	Iowa	2	New Hampshire	1	Texas	20
Alaska	0	Kansas	15	New Jersey	1	Utah	5
Arizona	9	Kentucky	7	New Mexico	3	Vermont	4
Arkansas	0	Louisiana	8	New York	11	Virginia	15
California	13	Maine	1	No. Carolina	7	Washington	4
Colorado	3	Maryland	9	North Dakota	3	West Virginia	1
Connecticut	11	Mass.	3	Ohio	5	Wisconsin	16
Delaware	4	Michigan	16	Oklahoma	16	Wyoming	6
Florida	16	Minnesota	24	Oregon	3	DHS	1
Georgia	5	Mississippi	17	Pennsylvania	5	EPA	11
Hawaii	2	Missouri	6	Rhode Island	1	FDA	15
Idaho	5	Montana	13	So. Carolina	4	USDA	10
Illinois	8	Nebraska	11	South Dakota	12	Ag Canada	11
Indiana	9	Nevada	4	Tennessee	7		

COMMITTEE WEB SITES - Submitted by Vice Chair Luann Wetzler

The AAFCO Website was completely updated in 2010. The Home page displays a new contemporary look, warm colors, and easy to find drop down menus. The AAFCO Laboratory Methods and Services web site is located in the open access portion on the AAFCO home page under Portals.



With the continued help of Melinda Walsh, the AAFCO Laboratory Methods and Services web site is updated with information. As time permits the Web Site Sub-group is providing the same information found on the AAFCO web site to the FoodShield web site. The Laboratory Methods and Services Committee no longer provides AOAC International with information.

The Web site Committee had hoped to coordinate with the FoodShield staff and facilitate a training session to demonstrate the process required to update and in most instances correct the information for each state laboratory that is now found on the Foodshield State Laboratory information section. In addition it is our goal that all Aglabs listserv users would gain experience and become familiar with the many resources available on the Foodshield website. It is anticipated that this training will still take place before the end of 2010. This training will be offered to all the subscribers of the Aglabs listserv.

All AFFCO Laboratory Methods and Services information continues to be accessible to anyone with internet access. One need not be a registered AAFCO website user. All information from the Laboratory Methods and Services Committee web site is easily down loaded as a printable document file (pdf).

The Laboratory Methods and Services Committee information is accessible from the Homepage of the AFFCO Web site under the AAFCO News and Information heading.

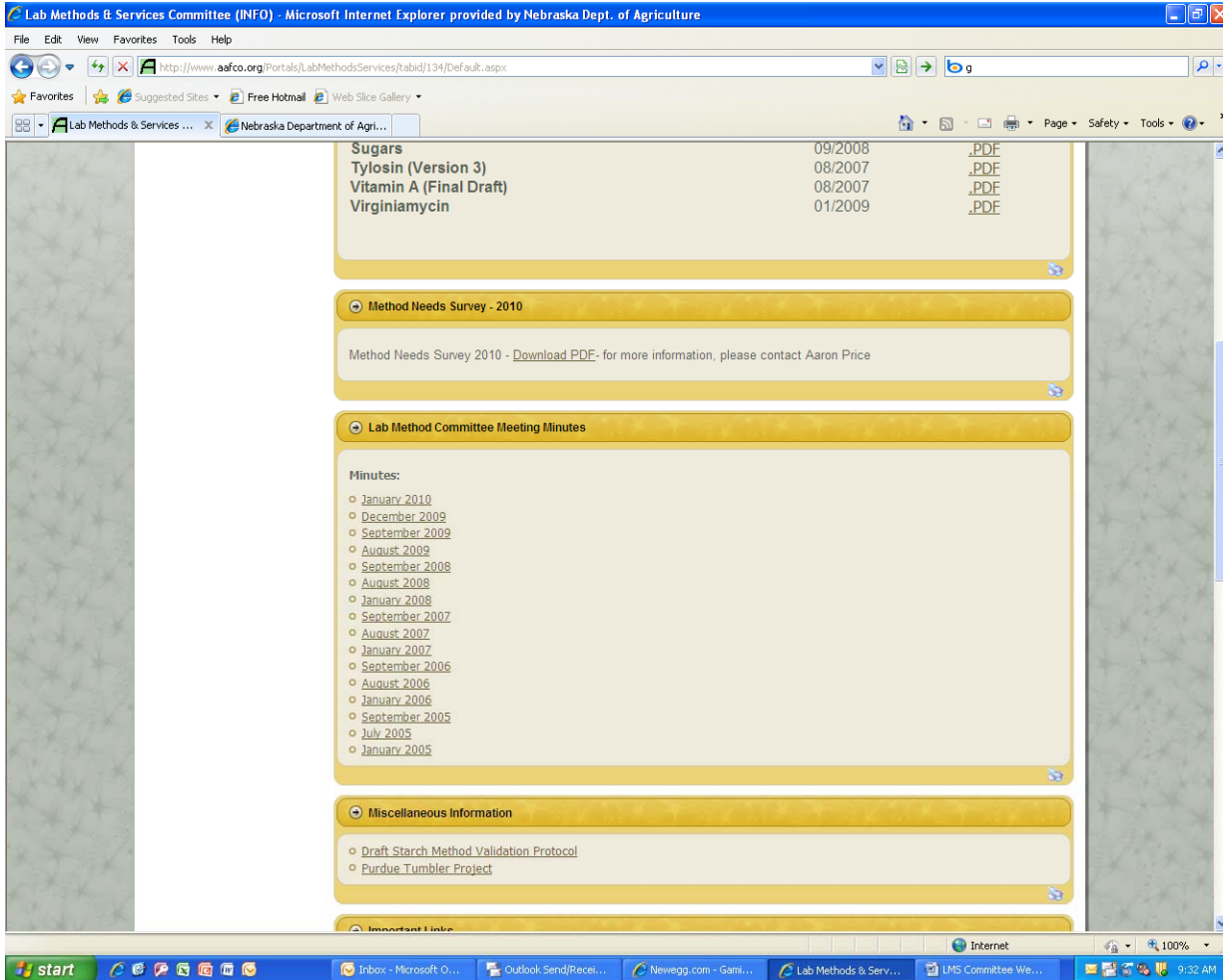
The Laboratory Methods and Services Committee information is found at this location:



One click of the mouse button brings you to the Laboratory Methods and Services Committee web site.



As you peruse down the page you will notice that we have all the committee minutes since September 2005.



Again if anyone is willing to become part of the AAFCO Lab Methods and Services Web Sites work group, please contact Lu Wetzler at luann.wetzler@nebraska.gov.

QUALITY SUBGROUP ANNUAL REPORT– Submitted by Vice Chair Louise Rhodes Ogden -

The quality subgroup was inactive in 2010.

CVM METHOD SUBMITTANCE BY AAFCO–Submitted on behalf of Vice Chair John Dennis McCurdy.

Yippee, Hurray!! In 2009, FDA CVM approved a method for lasalocid B and C submitted to AAFCO Veterinary Master File (VMF) 5919; a method for oxytetracycline B and C submitted to AAFCO VMF 5918; and a method for decoquinatate B and C submitted to AAFCO VMF 5920. So, we have three new regulatory methods for drugs in animal feed.

A submission for AOAC Official Method 2006.01, for monensin, narasin and salinomycin in animal feed, was made on January 6, 2010 to AAFCO VMF 5968. Aaron Price retrieved data from the 2006 collaborative study and Nancy Thiex prepared the submission. A letter was received from CVM on June 9, 2010 requesting additional data which is currently being generated. A fifth submission to VMF 5886 for sulfamethazine is in preparation by Vicki Siegel.

METHOD NEEDS PRIORITIZATION AND METHOD NEEDS STATEMENTS - Submitted by Vice Chair Aaron Price.

The method prioritization process occurs every two years and is comprised of a survey that is sent out to AOAC and AAFCO members of the Feed Additives Committee and Laboratory Methods and Services group. The first part of the survey asks respondents to rank in order of importance six *areas of need* which have been previously identified as the major reasons why new analytical methods are developed. The second part of the survey asks participants to rate a number of different analytical methods with respect to each of the six *areas of need* according to their own organization's requirements. There is also a section for participants to add other methods, not included in this year's survey. It was decided at the 2010 mid-year meeting that methods for which there already exists a completed method needs statement would not appear on the survey. These methods are already considered to be in the method development "pipeline" and their priority has already been established.

Early in May the survey was sent to over 100 individuals who work at regulatory, academic, and private laboratories and they were asked to respond by early June. At this point a reminder message has been sent to those that received the survey and a total of approximately 10 responses have been collected. The survey has also been sent to the AgLabs community Listserv and it appears on the AAFCO website within the Lab Methods and Services portal. All surveys that are collected before the end of July will have their results tabulated and each method will receive a priority score. These scores will be presented at the Laboratory Methods and Services Committee meeting on August 1, 2010. The methods with high scores have priority for laboratory validation and collaborative study through AOAC.

Prior to a method being proposed for a collaborative study with AOAC, a method needs statement must be developed. A method needs statement outlines the performance requirements that a method should be able to attain. These performance requirements are determined based on the concentration of the analyte commonly found in commercial feed, the type of feed matrix, and the methodology (ie. technology used), etc. At the 2009 annual and 2010 midyear meetings, method needs statements for fructans and bacitracin were completed, while statements for a multi-element method, pesticides, fatty acids, vitamin D and vitamin E were reviewed and edited. A method needs template was reviewed and approved by the committee to aid in the creation of future method needs statements. The review and approval of method needs statements is an ongoing process carried out by the committee members and coordinated by the committee vice chair Aaron Price. Currently, committee members are awaiting the results of the 2010 method prioritization survey which should provide a number of new method needs for which statements will have to be generated.

AOACI AGRICULTURAL MATERIALS COMMUNITY, FEED ADDITIVE AND CONTAMINANT SUBGROUP - Submitted by Nancy Thiex. Work in progress on methods development and validations is summarized in the table below.

GROUP ACTIVITES & PROGRESS CHART

Methods	Method Needs Criteria	Call For Methods	SVL*	Project Leader*	AOAC Collaborative Study
Amino Acids		X		Yanhong Zhang	Planned
Amprolium	Completed	X	Needed	Fred Armstrong	Anticipated
Carbadox	Completed	X	In Progress	Jane Sabbatini Regina Wixon	Planned
Pyrantel	Completed	X	In Progress	Jane Sabbatini Regina Wixon	Planned
CTC	Completed	X	Needed	Tami Stoltzenbach	
MGA	Completed	X	In Progress	Johnson, Tinkey	Planned
Multi-element	Completed	X	Needed	Terry Field	
Multi-mycotoxin	Completed	X	Needed	Mclver	Anticipated
Neomycin	Completed	X	Needed	Needed	Anticipated
Prohibited protein	Draft		Needed	Needed	

Dietary Starch	Completed	X	Completed	Mary Beth Hall	Planned
Sugars	Completed	X	Needed	Needed	
Tylosin	Completed	X	In Progress	Needed	Anticipated
Virginiamycin	Draft		Needed	Needed	
Vitamin A	Completed	X	In Progress	Sub group	Anticipated
Bacitracin	Completed		Needed	Needed	
Vitamin D	Draft		Needed	Needed	
Vitamin E	Draft		Needed	Needed	
Pesticide multi-analyte	Assigned		Needed	Mclver	Anticipated
Fructans	Completed	X	Needed	Needed	
Fatty-acid analysis	Completed	X	Needed	Don Palmquist	Anticipated

Dietary Starch - Mary Beth Hall has completed the Single Laboratory Validation for Dietary Starch. The collaborative study protocol is nearly ready for submission to AOAC. A collaborative study is anticipated before the end of the calendar year. AOAC is reviewing this method at no charge.

Tylosin-Tommy Phillips: A report from Tommy Phillips was presented at the Annual meeting on progress with method development and validation of a method for tylosin in feed.

Melengesterol Acetate (MGA) – Ian Schuetz reported on r-Biopharm and SDSU studies of the applicability of the Ridascreen Melengesterol acetate kit for quantifying the melengesterol acetate (MGA) content in feeds and premixes at the midyear (January) Meeting. The kit was originally developed for MGA in serum and urine.

Fatty Acids – At the 2009 Annual Meeting (August) 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 <http://www.lipidlibrary.co.uk/analysis.html>. 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 project is slowly moving forward. The protocol for the comparability study between two one pot methods on materials such as full fat oilseed meal, corn, pelleted alfalfa, liquid sweet feed, and pet food is in review. Questions to be answered are: 1) Does the hexane/heptane mixture extract all the lipids or is it too non-polar? 2) Has the need for adding an antioxidant been demonstrated? 3) What are the benefits of the BF₃ catalyst relative to a H₂SO₄ catalyst? 4) Is refluxing needed?

Neomycin –Jay Ghandi reported at the Midyear (January meeting) on preliminary data showing neomycin B and neomycin C separations by ion chromatography with electro-chemical detections. The limit of determination was around 1 ppb and the potential method also separated tetracyclines (sensitivity around 1 ppm) and tylosin. He reported that the CTC solutions were photosensitive. He will continue work on the methods. A collaborative study on the post column method will be delayed until data on the alternative method is available.

Virginiamycin in DDG— Alex MacDonald explained that virginiamycin (VM) consists of two major factors and several minor factors that interact in a synergistic fashion and therefore should be assayed using a biological activity based assay. VM is manufactured and distributed exclusively by Phibro. Phibro’s Ethanol Process lab in St. Paul, MN together with a commercial lab has validated a method that provides good recoveries in the range of 0.5-1.5ppm. It may be possible to drive the LOQ down to 0.2PPM. The resulting extracts are fairly stable. The method may be obtained from Phibro (QA@Phibro.com). FDA and CFIA both use HPLC-MS/MS based methodologies focusing on the M1 factor. Dr. de Alwis reported that her LC-MS/MS (iontrap) method would be published 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.

Amino Acid Related Studies – Yanhong Zhang and Amy Johnson reported on the status of the most recent of the amino acid round robins. A full report is expected at the 2010 Annual Meeting. Zhang also reported on current work involving the use of microwave digestion for preparing hydrolysates for tryptophan determination by LC-MS/MS and HPLC-UV. The correlation of the amino acid content in DDGS with the amino acid content in corn was also reported based on two recent studies and more detailed information can be obtained from <http://www.valueadded.org/renewableEnergy/ethanol/ddgs/>) and http://www.ddgs.umn.edu/profiles/us_profile_comparison_march_2009.pdf. Finally Dr. Zhang reported on the issues surrounding protein availability in swine and the use of furosine as an indicator of lysine availability.

Sugars – A presentation was provided by Waters; however, the presentation did not contain any findings as to the concentration of individual sugars in the feed sample set provided. Ian Schuetz reported on R-Biopharm's results when using their test kits on the feed sample set. To cover the profile 3 different test kits were used on each sample. Duplication was good (less than 5% CV between duplicates. Lars Reimann compiled a comparison of the two data sets and results by the two methods were not similar, so continued work is needed.

Vitamin A – Michael Stevenson, Regina Wixon, Ken Riter, Jen Kraus, Nancy Thiex and Mary Koestner may start working on updating the method for Vitamin A.

OTHER TOPICS

MEDICATED FEED MIXER – Alex MacDonald and Vickie Siegel reported on the separation issues experienced following the grinding of some medicated feeds. Purdue's machine shop had developed a mixer that seemed to be able to quickly make homogeneous powders of dried ground samples. Schematics for the tumbler were distributed. Orders were taken from feed laboratories and placed as a group with the Purdue machine shop for the tumbler mixer with an estimated price tag around \$2,500.