

DRAFT

MINUTES OF THE MEETING
AAFCO LABORATORY METHODS AND SERVICES COMMITTEE
Downtown Marriott Hotel, Memphis TN
January 23-24, 2005

Uncertainty method recommendation and presentation – An afternoon was used to cover excellent materials prepared and presented by Ed Moore. The major take home message from this time was “you probably already have the data” to make the uncertainty calculations.

ISO17025 requires testing laboratories to provide reasonable estimation of uncertainty, which may be based on previous experience and validation data as an example of knowledge of method performance and its measurement scope. Uncertainty determination may take two major approaches: bottom up by determining all individual component of uncertainty and deriving the total uncertainty by combining the components; or top down using measures such as inter-laboratory studies, collaborative studies, quality control, validation measures, etc. Materials presented were relatively comprehensive and accompanied by more than adequate reference materials to give laboratories a good start on understanding what might be done to determine uncertainty in their individual laboratory environments.

There was strong support at the end of the day among those attending the uncertainty session for holding a full day on the subject at the AAFCO August meeting in Orlando. This includes the opportunity to bring one’s laboratory data for use in calculating uncertainty in a workshop, following the morning seminar with examples as per Memphis. If you are thinking about 17025, this will be a do not miss event! Attendance ~ 15 interested listeners.

January 24, 2005

In attendance: see attached list

Nancy Thiex opened the meeting. Introductions were followed by approval and adoption of the agenda.

Minutes of the July 31-August 1, 2004 meeting – The minutes of the half and whole day sessions of the LM & SC from the AAFCO annual meeting were briefly reviewed and accepted.

Codex activities – The next meeting of the Codex Committee on Methods of Analysis and Sampling (CCMAS) is scheduled in Budapest for April 4-8, 2005 with an Ad Hoc Working Group on Endorsement of Methods on April 2 and a session of the InterAgency

Meeting (IAM) at the Hungarian Institute of Standards on April 1, 2005, preceding the formal CCMAS sessions. There are several topics of interest as evidenced in the attached agenda: Criteria for Evaluating Methods of Analysis; Review of Analytical Terminology for Codex Use in the Procedures Manual; The Use of Analytical Results: Sampling Plan, Relationship between the Analytical Results, Measurement Uncertainty, Recovery Factors and Provisions in Codex Standards; Criteria for the Methods of Detection and Identification of Foods Derived from Biotechnology, etc. LMSC has already provided input on the analytical terms topic through AOACI to the U.S. Head of Delegation for the CCMAS. Generally, the terms being considered are ISO based and acceptable to LMSC members responding with comments to materials provided on candidate terms. AAFCO may have further opportunity to comment on the terms and other topics before the CCMAS meetings, both domestically at a U.S. delegation session, Washington, D.C., March 15, 2005, and in Budapest during a meeting of the U.S. delegation. Alan Hanks will be in Washington, D.C., at an AAPCO meeting and will attend the March 15th meeting.

The work of the Codex Ad Hoc Task Force on Good Animal Feeding has concluded its work, but comments on various aspects of the code are still acceptable until March 1, 2005. Although not a specific issue open to comment except perhaps as it may relate to the open topic of a listing of negative substances (contaminants) in the code, AAFCO's LMSC could suggest methods for feeds, at least contaminants, be subject to review for endorsement by CCMAS for Codex purposes if there are associated health and safety concerns in international trade. By so doing we could exert long term influence on which methods Codex selects for endorsement.

National Food/Agricultural Laboratory Committee – Bill Krueger first gave an update with background on this group formed to provide a coordinated voice, representation and input of agricultural groups/associations like AAFCO into the Food Emergency Response Network (FERN). Further, it will provide a point of contact and information through a website (www.nfalc.org) for state laboratory capabilities, etc. Further, the visibility provided through NFALC should facilitate greater access of these laboratories to funding related to analytical capabilities and capacities for responding in food emergencies, disasters, terrorism and other events needing rapid health and safety related responses. Work on the website is ongoing with some laboratories doing prototype work adding information as has been found at the Ag Labs website for some time now. Security for information like state data will be included, while access to some data will be more generally available. Further discussion related to potential for educational experiences through the site, etc. were presented.

Future workshops – Mark Lee presented information on California's plan to alternate workshops between pesticide residues and other agricultural areas like feeds and fertilizers with an every other year frequency. Currently a fertilizer seminar/workshop is scheduled for February 9-10, 2005 in Sacramento. Label review/registration, terminology, metals and ISO17025 are included in the program. A pesticide training

conference with hands-on experience is scheduled for April 25-29, 2005, also in Sacramento. FDA technology transfer, Dow AgroSciences new pesticides and USEPA technology transfer are among items on the agenda for these sessions. In 2006, feed topics will be on the agenda. Heidi Hickes from Montana will also be organizing a future workshop.

Videotaping of some workshop sessions in the future to develop training elements for AAFCO and the NFALC website was discussed. The tapes could be made available to states unable to attend and should be useful when in need of satisfying ISO17025 training requirements. A study group of Dennis McCurdy, Mark Lee, Jim Balthrop and Bill Krueger was formed and asked to report back at the August meeting on the potential for development of training materials like videos for AAFCO and the NFALC website. Mark accepted leadership for the group.

Amino acids – Ken Riter introduced the topic. George Latimer appears to have provided AVs for amino acids from use of past data for various AA containing feed check samples. This led to a discussion on review of AVs in the AAFCO Official Publication with particular interest in seeing if revisions would be appropriate. Ed Moore observed that changes in technology used for salt determination has reduced variance in the analysis. Further discussion of the AAFCO AVs was carried forward to the next day and the AAFCO Check Sample Committee meeting.

Report on ISO and CEN activities – Ed Moore reported on the November 24, 2004, meeting of ISO TC-34, SC-10 and the November 25-26, 2004, meeting of CEN TC 327, Animal Feeding Stuffs. Sixteen countries participated, but the time of the meeting was not such that a U.S. representative/observer could attend. The scope of an ISO aflatoxin project was limited to EU's needs (B₁ only vs B₁, B₂, G₁ and G₂). The methods for protein/nitrogen by Kjeldahl Part 1 & 2, tryptophan, amino acids and monensin – narasin – salinomycin (a Canadian CFIA project leader), were reported to require editorial changes before going to the next ISO stage. The three drug method will be presented at CEN and accepted as a CEN standard. Five methods were subject to the ISO five year review process. Ed indicated that the quality of the work is improving with more attention being given to collaborative study protocols and data is being published. They are short on study directors and have no real team approach, but there may be a willingness to change, which might help resolve some existing duplication of effort. The AOAC Agricultural Community concept with multi stakeholder participation in method prioritization and selection through forums appeared to be ideas of interest, perhaps for development.

At the CEN Meeting, work on heavy metals in feed minerals was presented. Much of what has been found in the AAPFCO work on a metals method might be of value here, but the CEN metals project may not be recognizing/seeing that they are treading ground already trod by others. Work by the Enzyme Manufacturers Association is trying to harmonize methods for phytase in feeds. The effort is at the feeding level in feeds, while

it might be good to first look at ingredient product first. A pesticide residue method for organochlorine and PCBs is in the plans for 2005. Work on decoquinatone is at the method identification stage. There appears to be higher interest in greater communication and coordination with AOACI at CEN as well.

d-Ca-Panthenate – At our last meeting, efforts were initiated to include a code for an HPLC method with range and CV in the AAFCO Official Publication. George Latimer committed to lead the process, and it has been completed by George and Nancy Thiex.

Letter to Dr. Vaughn, FDA/CVM – A letter has been written (Tom Jensen) and it was reviewed at this meeting and slight revisions made. This letter is a response to prior correspondence with Dr. Vaughn on having non-sponsor drug methods that have been fully validated in an AOACI collaborative study recognized for regulatory purposes by FDA/CVM. The letter was finalized for signature of Phil Petry, AAFCO President. Recent messages indicate it is being signed and sent to FDA/CVM.

AOCS Collaborative Study on Fat – In the study only petroleum ether is used to extract a sample in a “bag” vs a thimble. Before the formation of the Feed Additives and Contaminants Subgroup, Andrew Komarek requested AAFCO’s feedback (support) for the study relative to AAFCO or AOACI adoption. Since the two solvents used with feeds, hexane and diethylether, and an important step used with pet foods (acid hydrolysis), were not included, it was hard to make conclusions beyond the apparent usefulness of the bag, which was acceptable in place of the thimble. As an official method per se for feeds, the AOCS studied method would present difficulties for AAFCO members without data for the commonly used solvents and acid hydrolysis step applicable to feeds.

AOACI Feed Additive and Contaminant Website –The Agricultural Materials Community, Feed Additives Subgroup Website is now up and running. It serves as a repository for all information related to the activities of the group including a statement of mission, overviews, and meeting agendas. The basis and categories for method prioritization are covered along with a layout for the program of work for this sub-community. Methods given priority for 2004-2005 period are identified on the site with respective method needs statements. Later in the LMSC meeting it was concluded priorities should be reviewed on a periodic basis.

Collaborative study protocol for multi-mycotoxin method – The first draft of a method needs statement and validation criteria were presented by Laszlo Torma for the project team including Gary Lombart, Harold Campbell and Chris Maragos. The team selected aflatoxins, deoxynivalenol, ochratoxin, fumonisins, T-2 toxin and zearalenone for this study. The method validation criteria were accepted by the LMSC. Method needs input will be sought from regulators and ISO TC-34, SC-10 (Nancy Thiex), plus FDA/CVM (Dennis McCurdy). A call for methods is the next step (collect and evaluate) with various postings at the NFALC website, in AOAC/ILM, from ISO/CEN, etc. Also, contact will be made with experts like FDA/CFSAN’s Mary Trucksess and the recent IUPAC international

meeting on mycotoxins reviewed for potential methods. Collection of potential methods should be completed by the August meeting.

Collaborative study protocol for lasalocid – The protocol has been a good example of how the process of community, needs gathering and method callouts can work. Charles Foacht is the project leader. Input from Alpharma indicates all homologs should be included in the analysis since they are all active drug components. Thus, the project leader has revised the method and protocol to accommodate the lasalocid homologs. The revisions are ready for expert review.

Collaborative study protocol for oxytetracycline – Richard Larson and Nancy Thiex are the project leaders/study directors on oxytetracycline. A methods needs statement has been accepted and all reviewer comments have been addressed. Some feeding levels need to be reviewed. The collaborative study protocol has been reviewed by AOAC. Reviewers comments have been addressed and the study directors have the go ahead to proceed.

Virginiamycin – A statement of needs was circulated for comment. Review by other stakeholders is needed (Phibro, AHI). If multi components are important is unknown. If multi components exist is not known as are HPLC methods not known. Project leaders are needed.

Neomycin needs statement – This drug is not used in Canada in animals, but Ed Moore indicated potential development of a needs statement if provided with the U.S. Compendium data. Further, he will take on those drugs previously established as needing methods needs statement from the list established in the recent past for which coverage has not been initiated.

Chlortetracycline validation data – The methods need statement is complete. Comparative data for microbiological results vs a potential HPLC method was provided for ADM products. Input from industry and a project leader are needed.

Prohibited materials workshop – Mark Lee reported on a seminar/workshop meeting organized in Sacramento during the first week of December 2004. Mike Meyer and Dragan Momcilovic of the FDA/CVM participated. There were both lecture and hands on sessions for training and testing of ELISA kits, DNA amplifier kits and PCR products. One kit gave many false positives (SDI), but no false negatives. This kit was more sensitive than another giving some false negatives and positives (Neogen). The timing of reading results for these kits was critical and has an effect on results. Manufacturers have been informed of this problem. One has already changed its instructions insert. The FDA validated PCR method gave a sensitivity of around 0.5%, but under different circumstances, in a well controlled environment, 0.01% may be achievable. The University of California at Davis ran their primer for a cytochrome and basic PCR (vs real

time PCR). It appears a gel run post real time replication monitoring would be a good final step for an approach for checking for ruminant tissues.

California has distilled down these meeting and workshop experiences for their purposes to the following sequence for ruminant tissue detection:

Screen with SDI kit so as not to miss potential positive results

Run real time PCR on SDI positives

Run conventional gel on real time PCR positives

Run positive gel samples with second PCR primer (gel basic PCR gel confirmation)

Do microscopy on alternate primer PCR positives

Fish out tissue, bone and hair found by microscopy for final PCR on positive microscopy.

This six step process has not been necessary so far since nothing has had a positive hit in the first PCR test. With time and experience, it is believed that the process can be shortened.

Sadly it appears we are a long way from good tests at the needed level of sensitivity necessary to monitor for ruminant tissue in the field. The best defense against BSE is, and may remain for some time if not always, prevention, but a little monitoring would help.

Non-nutritive metals – Perhaps the methodology being developed for fertilizers can be adapted to feeds. It may in fact be nearly ideal for mineral feed ingredients and mixtures there of, since it is designed to work in a high mineral mixture.

Multi-pesticide residue method – The QuEChERS method approach for multiple pesticide residue appears to hold much promise, but there is potential for problems for incurred residue in real world samples. A multi-pesticide residue method for feeds needs a needs statement.

Mid-West AOACI Meeting – Nancy provided the meeting and technical program information for the May 23-26, 2005, Mid-West AOACI Meeting in Kansas City, MO, at the Hyatt Regency Crown Center.

AAFCO – LMSC – MIDYEAR

<u>Name</u>	<u>Organization</u>	<u>Email</u>
Rob Jeppsen	Albion Advanced Nutrition	rjeppsen@albion-an.com
Alan R. Hanks	Office of Indiana State Chemist	hanksa@purdue.edu
Jim Balthrop	OTSC	jim-balthrop@tamu.edu
David Fairfield	NGFA	dfairfield@worldnet.att.net
Bruce Arentson	Kent Feeds bruce_arentson@kentfeeds.com	
Roy Schulze	PFI	rschulze@purina.com
Bill Krueger	MDA	william.krueger@state.mn.us
Charlie Staff	Distillers Grains Tech Council	chstaf01@louisville.edu
Laszlo Torma	Pickering Labs	laszlo@pickeringlabs.com
Mark Lee	CDFA	mlee@cdfa.ca.gov
Mark Coleman	Elanco	mcoleman@lilly.com
John D. McCurdy	CVM/FDA	JMcCurdy@cvm.fda.gov
Eric W. Sespico	FL Dept. of Ag	sespice@doacs.state.fl.us
Weldon Collier	FL Dept. of Ag	colliew@doacs.state.fl.us
Ken Riter	OISC	riterk@purdue.edu
Melton Bryant	U of KY	mbryant@uky.edu
Ed Moore	CFIA	emoore@inspection.gc.ca
Jane Sabbatini	Eurofins Scientific	janesabbatini@eurofinsus.com
Tim Herrman	OTSC	tjh@otsc.tamu.edu
Kevin Armbrust	Office Mississippi State Chemist	armbrust@mscl.msstate.edu
Jim Larkin	IL Dept. Ag	jlarkin@ag.state.il.us
Sharon Benz	CVM/FDA	sbenz@cvm.fda.gov
Paul Kahlich	3D Corpsol	paul@3dcorpsol.com
Larry Whitlock	Texas Feed & Fert	1-whitlock@tamu.edu
Sondra Flick	Alpharma Inc.	sandy.flick@alpharma.com
Nancy Thiex	SDSU	thiex@sdstate.edu