

METHODS COMMITTEE REPORTS

Committee on Feeds, Fertilizers, and Related Agricultural Topics

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Committee Actions

Three collaborative studies were granted First Action status: **2008.01** *Determination of Lasalocid Sodium in Animal Feeds and Premixes by Reversed-Phase Liquid Chromatography*; **2008.08** *Determination of Decoquinatate in Animal Feeds by Liquid Chromatography*; and **2008.09** *Determination of Oxytetracycline/Oxytretracycline Hydrochloride in Animal Feed, Fish Feed, and Veterinary Medicinal Products by Liquid Chromatography with Fluorescence Detection*.

Three First Action Methods were adopted Final Action: **2003.14** *Urea in Water-Soluble Urea-Formaldehyde Fertilizer Products—and in Aqueous Urea Solutions by Liquid Chromatography*; **2006.01** *Monensin, Narasin, and Salinomycin Mineral Premixes, Supplements, and Animal Feeds by Liquid Chromatography and Post-Column Derivatization*; and **2006.03** *Arsenic, Cadmium, Cobalt, Chromium, Lead, Molybdenum, Nickel, and Selenium in Fertilizers by Microwave Digestion and Inductively Coupled Plasma-Optical Emission Spectrometry*.

Continue the First Action status of Method **2000.12** *Phytase Activity in Feed, Colorimetric Enzymatic Method*.

Due to work commitments, committee member Dave Mertens resigned.

The driving force for new methods in the collaborative study process in the past year continues to be the Agricultural Materials Community and its subgroups. A study titled “*Sampling of Fertilizer Materials and Blends from Bulk Bags Designed to Hold Approximately 1 Ton of Material: Bag Probing Technique Compared to AOAC Fertilizer Stream Cutting Technique*” has been received and is under committee review.

After considerable discussion, the committee has decided that it will be in the best interests of the Agricultural Community to extend the existence of the Methods Committee on Feeds, Fertilizers, and Related Agricultural Products for at least another 6 months (to April 1, 2009). This should allow for a smooth transition to, and guidance for, ad hoc committees as they are formed in the coming year. It will also provide some training and transition for some of the new members who will eventually populate the ad hoc committees. Since the committee could easily review 6–10 methods in the upcoming year, extending the committee will ease the transition, maintain infrastructure, and facilitate communication until the ad hoc committees are up and the process for their establishment is working smoothly.

A complete list of members who have been vetted through the community groups and are willing and able to serve is appended.

A motion was made to extend the existence of the Methods Committee on Feeds, Fertilizers, and Related Agricultural Products for a minimum of 6 months (to April 1, 2009). The motion was seconded by Dennis McCurdy, and passed unanimously by vote of members present.

Antibiotics in Feeds, Hussein Ragheb

Study Directors: No reports were submitted to the General Referee.

Due to the curtailed use of antibiotics in feeds, the main effort in our laboratory was concerned with detection limits of residue of some antibiotics in agricultural products. For

example, detection of virginiamycin, monensin, and penicillin in distillers dried grain solubles (DDGs) were investigated. The technique of bioautography using *Micrococcus luteus* for virginiamycin and penicillin and *Bacillus subtilis* for monensin was most useful in detecting 0.20–0.50 ppm for virginiamycin and monensin. Our efforts will continue to improve the technique so that lower limits can be possible. Also, future experiments will focus on plate assay sensitivity to increase the limits of quantitation for several antibiotics used in agriculture.

Drugs in Feeds, General Referee Position Vacant

(1) *Amprolium, LC Method*.—Topic Advisor Fred Armstrong, Canadian Food Inspection Agency, Ottawa Laboratory (Carling), Bldg 22, Central Experimental Farm, 960 Carling Ave, Ottawa, ON K1A 0C6, Canada, Tel: 613-759-1299, Fax: 613-759-1260, E-mail: farmstrong@inspection.gc.ca.

(2) *Carbadox*.—Topic Advisor Alexander MacDonald, Pharma Science, Inc., 16 Cypress Ave, North Caldwell, NJ 07006, Tel: 973-228-2392, Fax: 973-228-3498, E-mail: BeeMac201@aol.com. Jane Sabbatini, Study Director, Covance Laboratories, 3301 Kinsman Blvd, Madison, WI, Tel: 608-395-3604, E-mail: jane.sabbatini@covance.com. The proposed method uses a water prewetting step to enhance extraction of pyrantel followed by extraction with acetonitrile–methanol (50 + 50). Sample extracts are filtered through a glass fiber filter and cleaned up using alumina solid-phase extraction (SPE) columns. Chromatography is accomplished using a C18 column with a gradient mobile phase of dibutylamine acetate (DBAA) and acetonitrile. Both analytes exhibit excellent peak shape when a C18 column that is both acid- and base-deactivated is used. Linearity has been established and initial recovery studies on medicated swine feeds are promising.

(3) *Chlortetracycline, LC Method*.—Topic Advisor Richard Larson, South Dakota State University, ASC 133, PO Box 2170, Brookings, SD 57007-1217, Tel: 605-688-6706, Fax: 605-688-6295, E-mail: Richard.Larson@sdstate.edu.

(4) *Decoquinatate, LC Method*.—Topic Advisor Anivis Sanchez, Canadian Food Inspection Agency, Ottawa Laboratory (Carling), Bldg 22, Central Experimental Farm, 960 Carling Ave, Ottawa, ON K1A 0C6, Canada, Tel: 613-759-1298, Fax: 613-759-1260, E-mail: Sanchezaa@inspection.gc.ca. Method granted First Action **2008.08**.

(5) *Lasalocid*.—Topic Advisor Charles L. Focht, Nebraska Department of Agriculture, 3703 South 14th St, Lincoln, NE 68502-5399, Tel: 402-471-2176, Fax: 402-471-0091, E-mail: cfocht@agr.ne.gov. Method granted First Action **2008.01**.

(6) *Monensin, Narasin, and Salinomycin, LC Method*.—Study Director Harold Campbell, Canadian Food Inspection Agency, Ottawa Laboratory (Carling), Bldg 22, Central Experimental Farm, 960 Carling Ave, Ottawa, ON K1A 0C6, Canada, Tel: 613-759-1227, Fax: 613 759-1260, E-mail: hcampbell@inspection.gc.ca. Method **2006.01**

Analysis of Monensin, Narasin, and Salinomycin in Premixes and Animal Feeds by Liquid Chromatography and Post-Column Derivatization to be granted Final Action status.

(7) *Neomycin by LC Method*.—Study Director Julee Driver, South Dakota State University, Olson Biochemistry Laboratories, SAS 133, Box 2170, Brookings, SD 57006. Tel: 605-688-5466, Fax: 605-688-6295, E-mail: Julee.Driver@sdstate.edu. Initial study and single-laboratory validation (SLV) to be published in *J. AOAC Int*.

(8) *Oxytetracycline, LC Method*.—Topic Advisors Richard Larson and Nancy Thiex, South Dakota State University, Olson Biochemistry Laboratories, SAS 133, Box 2170, Brookings, SD 57006. Richard Larson, Tel: 605-688-6706, Fax: 605-688-6295, E-mail: Richard.Larson@sdstate.edu. Nancy Thiex, Tel: 605-688-5466, Fax: 605-688-6295, E-mail: Nancy.Thiex@sdstate.edu. Method granted First Action **2008.09**.

(9) *Pyrantel Tartrate*.—Study Director Jane Sabbatini, Covance Laboratories, 3301 Kinsman Blvd, Madison, WI, Tel: 608-395-3604, E-mail: jane.sabbatini@covance.com. The proposed method uses a water prewetting step to enhance extraction of pyrantel followed by extraction with acetonitrile–methanol (50 + 50). Sample extracts are filtered through a glass fiber filter and cleaned up using alumina SPE columns. Chromatography is accomplished using a C18 column with a gradient mobile phase of DBAA and acetonitrile. Both analytes exhibit excellent peak shape when a C18 column that is both acid- and base-deactivated is used. Linearity has been established and initial recovery studies on medicated swine feeds are promising. SLV will be published in *J. AOAC Int*.

(10) *Tylosin, LC Method*.—Topic Advisor Mark R. Coleman, Elanco Animal Health, A Division of Eli Lilly and Co., 2001 W. Main St, Greenfield, IN 46140, Tel: 317-277-4613, Fax: 317-277-4167, E-mail: Coleman_Mark_R@lilly.com. Tom Phillips, Maryland Department of Agriculture, Proximate and Drug Analysis Group, State Chemist Section, 50 Harry S. Truman Pkwy, Annapolis, MD 21401, Tel: 410-841-2721, Fax: 410-841-2740 E-mail: PhilliTD@mda.state.md.us is working on the SLV.

Feeds, General Referee Position Vacant

Methods of interest and activity for the Feed Additives and Contaminants subgroup are summarized in Table 1.

(1) *Inorganic Elemental Constituents of Feed Samples, Microwave Digestion, ICP-OES Detection*.—Topic Advisor Terrance Field, CFIA/ACIA Feed and Fertilizer Section, Ottawa Lab (Carling), 960 Carling Ave, Bldg 22 Central Experimental Farm, Ottawa, ON, K1A 0C6, Canada, Tel: 613-759-1497, Fax: 613-759-1260, E-mail: fieldt@inspection.gc.ca.

(2) *Microscopy*.—Topic Advisor Mike Buckner, Division of Consolidated Lab Services (DCLS), 1 N. 14th St, Rm 127, Richmond, VA 23219, Tel: 804-225-4070, Fax: 804-796-7795, E-mail: mbucker@dgs.state.va.us.

Table 1. Progress chart for Feed Additives and Contaminants subgroup method projects

Methods	Method needs criteria	Call for methods issued	SLV	Project leader	AOAC collaborative study
Amprolium	Completed	X	Needed	Vicki Siegel	
Carbadox	Completed	X	In progress	Jane Sabbatini	Planned
Pyrantel	Completed	X	In progress	Jane Sabbatini	Planned
CTC ^a	Completed	X	Needed	Needed	
MGA ^b	Completed	X	Needed	Kurt Johnson	
Multi-element	Completed	X	Needed	Terrance Field	
Neomycin	Completed	X	Needed	Julee Driver	Planned
Prohibited protein	Draft	X	Needed	Needed	
Starch	Completed	X	In progress	Mary Beth Hall	Planned
Sugars	Completed	X	Needed	Needed	
Tylosin	Completed	X	Needed	Tom Phillips	
Virginiamycin	Draft	X	Needed	Needed	
Vitamin A	Completed	X	Needed	Wixon?	
Bacitracin	Draft	X	Needed	Needed	
Vitamin D	Assigned	X	Needed	Needed	
Vitamin E	Assigned		Needed	Needed	
Pesticide multi-analyte	Assigned		Needed	Needed	
Fructans	Assigned		Needed	Needed	
Fatty acids	Assigned		Needed	Don Palmquist	

^a CTC = Chlortetracycline.

^b MGA = Malengestrol acetate.

(3) *Neutral Detergent Fiber, Acid Detergent Fiber, and Lignin Using Filter Bag Technology.* Topic Advisor Andrew Komarek, ANKOM Technology Corp., Fairport, NY 14450, Tel: 716-425-3940, Fax: 716-425-3941, E-mail: akomarek@ankom.com. Continue topic.

(4) *Vitamin A, LC Method.*—Continue topic.

(5) *Starch.*—Topic Advisor Mary Beth Hall, U.S. Department of Agriculture, Agricultural Research Service, U.S. Dairy Forage Research Center, 1925 Linden Dr West, Madison, WI 53706, Tel: 608-890-0078, Fax: 608-890-0076, E-mail: mbhall@wisc.edu.

(6) *Mono- and Disaccharides.*—Method needs statement written; various techniques being evaluated. Study Director needed.

Fertilizer and Agricultural Liming Materials, William Hall, General Referee

The Fertilizer subgroup has been gathering momentum over the last year and has one method ready for Study Director appointment and protocol review. The groups has several other high-priority methods and hopes to have protocols ready within the next 6 months. Funding is a major concern and was the focus of a presentation given to the AOAC Board of Directors in July 2008. The Subgroup (The Fertilizer Institute) hopes to enter into a contract with AOAC sometime following this presentation that will allow several methods to be

accepted as funded and move quickly through the AOAC administrative and validation channels. The group also hopes to clarify how method modifications will be made within the new ad hoc committee structure. However, the community has discussed options other than paying to go through the AOAC process to make validated methods available to its members. Discussions with Association of American Plant Food Control Officials (AAPFCO) will continue for the next few months, taking into account the responses of the AOAC Board of Directors and Executive Director to the proposal.

Functional process and activities.—Because of the nature of the fertilizer industry, member travel to multiple meetings has been limited. Although the fortunes of the industry have recently turned around, travel and budget restrictions remain in place for many at the technical level in both regulatory and industry roles. In an effort to reach the community, functions have been scheduled where industry and regulatory participants already meet. The group has worked to share a portion of each meeting of the fertilizer laboratory services committee of AAPFCO with the chair. This has worked out well and raised the awareness of the community and its activities to a larger audience. The primary means of communication and interaction has been via the following routes:

(1) Two community meetings take place annually with the AAPFCO Lab Services Committee.

Table 2.

Proposed new Associate Referee and Study Directors	Method/Topic	Funding possible	Timeframe
James Bartos	Determination of ammonium citrate–EDTA soluble P and K in commercial inorganic fertilizer materials by inductively coupled plasma-optical emission spectrometry (ICP-OES)	Yes	Commitment in place
Sanford Siegle	TP ₂ O ₅ & K ₂ O by ICP	Possible by suppliers	6–12 Months
Lawrence Datnoff	Soluble silica	Possible by suppliers	6–12 Months
Robert Richter	Hot block trace metals digestion	Perhaps by vendors	6–12 Months
Dave Pfeil	Hg in fertilizers	Perhaps by vendors	6–12 Months
Carolina Medina	Slow release nutrient extraction	Possible suppliers	6–12 Months
John Peterson	Differentiating phosphite from phosphate in commercial fertilizers	Doubtful	1–2 Years

(2) *Methods Forum*.—Gives members an opportunity to form consensus-based method validation plans.

(3) *Method Validation and Protocol Review Training*.—Initiated this year with 27 registrants.

(4) *Priority Ranking*.—Was completed via e-mail in early 2008 with 32 methods prioritized.

Top method priorities and development summary.—Table 2 lists proposed new Associate Referee and Study Directors active method investigations, and development with some detail as to their history, timeline, and funding.

(1) OMA-2004-Nov-023 *Arsenic, Cadmium, Cobalt, Chromium, Lead, Molybdenum, Nickel, and Selenium in Fertilizer by Microwave Digestion and ICP-OES Detection*.—Topic Advisor William Hall, The Mosaic Co., PO Box 2000, 3095 County Rd, 640 W, Mulberry, FL 33860-1100, Tel: 863-428-7161, Fax: 863-428-7398, E-mail: Bill.Hall@mosaicco.com. Method **2006.03** to be granted Final Action status.

(2) **2003.14** *Determination of Urea in Fertilizers by LC*.—Topic Advisor Michael Hojjatie, Tessengerlo Kerley Inc., 2480 Twin Buttes, Sahuarita, AZ 85629, Tel: 520-791-2940, Fax: 520-625-8091, E-mail: mhoggiatie@tkinet.com. Method **2003.14** to be granted Final Action status.

Nutrients in Soils, Charles Focht, General Referee

Focht is not currently officially serving as General Referee for Nutrients in Soils, as he was notified that his appointment had expired. He submitted a Curriculum Vitae and resumé for reappointment early in 2008 but never received notification of reappointment. So the position is officially vacant, although he is informally trying to keep on top of what limited activities are ongoing in this topic area.

In February 2008, Focht posted on Manuscript Central the collaborative study report on “Determination of Plant Available Potassium in Soils—Ammonium Acetate Method.” The study had been approved a number of years ago by the Methods Committee on Feeds, Fertilizers, and Related Agricultural Products and conducted by Study Director

Maurice Watson. Because Focht was actively involved with this study from its inception, Watson cited him as a co-author. Watson has since retired and gave consent for Focht to be the submitting author in order to facilitate committee review, etc. Focht also submitted a Curriculum Vitae for appointment as Study Director for this study. Because he could not serve as both General Referee and author, a request was made to AOAC that Ed Hanlon be appointed as a temporary General Referee for the review of this study report. A VAC and CV were submitted to AOAC by Hanlon in February 2008. Also in February 2008, AOAC Agricultural Materials Task Force Chair Tom Jensen submitted a letter to AOAC Executive Director James Bradford in support of these volunteer appointments.

In addition to the aforementioned study report, there are 2 other soil study reports pending for studies that were earlier approved by the Methods Committee on Feeds, Fertilizers, and Related Agricultural Products. Hanlon is assigned to be the submitting author for the study report on “Micronutrients in Soils—DTPA Extraction,” a study that was originally conducted by Don Horneck. The second study on “Plant Available Phosphorus for Noncalcareous Soils—Bray P-1 Method” was conducted by Bryan Hopkins. Due to the delay and uncertainty regarding the appointments required to proceed with the review of the Available Potassium in Soils study, further action for submission of the remaining 2 soil study reports has, of necessity, been put on hold.

Very recently, the Chair of the Methods Committee on Feeds, Fertilizers, and Related Agricultural Products again requested of AOAC information regarding the required appointments for the potassium study report so that the committee will be able to proceed with the review of this study.

(1) *Available Potassium in Soils, Ammonium Acetate Method*.—Study Director Maurice Watson, Ohio State University, Department of Natural Resources, Wooster, OH 44691-4096, Tel: 330-263-3755, E-mail: Watson.8@osu.edu.

(2) *Plant Available Zinc, Manganese, Iron, and Copper in Soils, DTPA Method*.—Study Director Donald Horneck,

Oregon State University Extension Service, Hermiston Agricultural Research and Extension Center, PO Box 105, Hermiston, OR 97838, Tel: 541-567-8321, Fax: 541-567-2240, E-mail: don.horneck@orst.edu.

(3) *Available Phosphorus in Soils, Bray P1 Method.*— Study Director Bryan Hopkins, University of Idaho, 1776 Science Center Dr, Idaho Falls, ID 83402-1575, Tel: 208-529-8376, E-mail: bhopkins@uidaho.edu.

*Veterinary Analytical Toxicology, Merl Raisbeck,
Acting General Referee*

The General Referee solicited candidate methods for multilaboratory validation via the VETTOX and ABVT listserves, direct e-mail, and personal phone calls. While there was general agreement about the need for improved

standardization of results between laboratories, no one volunteered a method for the process. The most common reason given for nonparticipation was insufficient time and resources. Another was the fact that the American Association of Veterinary Laboratory Diagnosticians has adopted the World Organization for Animal Health (OIE) Quality Standard and Guidelines for Veterinary Laboratories as the standard for accreditation, and several people indicated that they could not see the “payback” for involvement with the AOAC validation process.

Recommendations

Continue study of all topics.