

METHODS COMMITTEE REPORTS**Committee on Feeds, Fertilizers, and Related Agricultural Topics****LUANN WETZLER, CHAIR**

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Committee Actions

The committee has not processed any new collaborative studies since the 2001 AOAC INTERNATIONAL Meeting and Exposition. Committee I completed the review on collaborative study I-29 that was in process at the 2003 meeting utilizing the only Committee I conference call. The study was adopted, with modifications, as First Action in November 2003 and assigned Official Method **2003.14**.

There are currently 2 collaborative study protocols that have been approved: Controlled Release Nutrient Extraction, I-25, and Monensin, Narasin, and Salinomycin in Premixes and Animal Feeds, Liquid Chromatographic Method Using Post-Column Derivatization, IA019. Notification of study approval was published in the ILM, May/June 2004 issue.

At the January meeting of the Control Officials (specifically the AAFCO and AAPCO, AAPFCO) held in Denver, CO, AOAC representatives of the Agricultural Materials Task Force, AAFCO, AAPFCO, and AAPCO members discussed the need for analytical methodology in the Agricultural Community. The Agricultural Materials Task Force is currently developing subgroups that will prioritize needed methodology, spearhead the method development, work to get the collaborative studies completed, and provide the Agricultural Community with needed validated methodology. The focus of Committee I will be to assist and supplement the work being carried out by the Agricultural Materials Task Force. This will be done in a manner similar to the other AOAC Task Forces currently in operation. The Agricultural Materials Task Force was well publicized with articles in the ILM (March/April and May/June 2004 issues) and the JAOAC (March/April issue).

The Committee Chair, with the help of Diana Hopkins, Tom Jensen, Agricultural Materials Task Force Chairman, and Nancy Theix expanded the scope and increased the number of Committee I members.

Two committee members will complete their terms of service at the end of this meeting. The Committee Chair and the members of Committee I would like to thank Previous Committee Chair, Mark Coleman for all his time and effort in the past 5 or more years. Coleman will become OMB Committee Chair at the end of the Business Meeting on September 22. The Committee also would like to thank George Latimer for his work as a member of Committee I. Latimer will become Chair of Committee F, Commodity Foods and Commodity Products. Máire Walsh, Safety Committee representative, also completes her term and will not be reappointed. Congratulations are in order as Walsh will become President-Elect of AOAC at the end of the AOAC Business Meeting on, September 22, 2004.

Additional committee members and a Secretary are yet to be identified.

Antibiotics in Feeds

- (1) *Apramycin in Feeds*.—Discontinue topic.
- (2) **993.29** *Bacitracin in Feeds*.—Topic Advisor Elizabeth Luigs for zinc and MD bacitracin, Alpharma Animal Health Division, 400 State St, Chicago Heights, IL 60411, Tel: 708-758-0111, Fax: 708-757-2510. Continue study.
- (3) *Neomycin in Feeds*.—Topic Advisor Scott Panagiotis, Pennfield Animal Health, 14040 Industrial Rd, Omaha, NE 68144, Tel: 402-330-6000, Fax: 402-330-6004, E-mail: Panagiotis@noria.net. Continue study.
- (4) *Low Level Chlortetracycline in Feeds*.—Topic Advisor Scott Panagiotis. Continue study.
- (5) *Tylosin in Premix and Animal Feeds*.—Topic Advisor Mark R. Coleman, Eli Lilly and Co., 2001 W. Main St, GL 36, Greenfield, IN 46140-0108, Tel: 317-277-4167, Fax: 317-277-4993, E-mail: coleman_mark_r@lilly.com. Continue study.
- (6) *Narasin*.—Topic Advisor Mark R. Coleman. Continue study of Microbiological method.
- (7) *Penicillin*.—Topic Advisor Steven Holmes, Alpharma, 400 State St, Chicago Heights, IL 60411, Tel: 708-757-2505, Fax 708-757-3510. Continue study.
- (8) *Chlortetracycline*.—Topic Advisor Steven Holmes. Continue study.
- (9) *Virginiamycin with Other Drugs*.—Topic Advisor Hussein Ragheb, Office of Indiana State Chemist, 1154 Biochemistry, Purdue University, West Lafayette, IN 47907, Tel: 765-494-1572, Fax: 765-496-6349, E-mail: Raghebh@purdue.edu. Discontinue topic.
- (10) *Bambermycin*.—Topic Advisor Denise Riley Moore, Woodson Tenent Laboratories, 345 Arbor Ave, Memphis, TN 38103, Tel: 901-521-4500, Fax: 901-521-4510, E-mail: Dmoore@wtlabs.com. Continue study.
- (11) *Direct Fed Microbiological Products and Silage Inoculants*.—Vacant.

Drugs in Feeds

- (1) *Amprolium, LC Method*.—Study Director 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. The Study Director reports that the search for a better LC separation continues. No publications dealing with amprolium determination were noted in 2003. Continue study.
- (2) *Carbadox*.—Study Director 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. No further activity was reported regarding the study of an LC method based on the method of Aerts and Werdmuller (1988) *J. Assoc. Off. Anal. Chem.* **71**, 484–489.

(3) *Chlortetracycline, LC Method*.—Study Director 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@sdsstate.edu. Work continues on optimizing the method of Houglum and Larson, *J. AOAC Int.* **80**, 961–965 (1997). Mobile phase pressure problems are being experienced by some laboratories, apparently related to insolubility of the buffer salts as the mobile phase gradient approaches 60% methanol. Flushing the column with 15% methanol for 5 minutes before equilibrating at 35% methanol seems to help eliminate the buildup of buffer salts in the system. The flow rate has also been reduced from 1.5 to 1.3 mL/min. These changes have added 5 min to the run time but do not negatively affect the chromatography. Validation of the method is ongoing. Continue study.

(4) *Ethopabate*.—Topic Advisor Joseph Hillebrandt, Cornell University Diagnostic Laboratory, Nutritional & Environmental Analytical Services, 777 Warren Rd, Ithaca, NY 14850, Tel: 607-257-2345, Fax: 607-257-5041, E-mail: jh119@cornell.edu. Continue study of LC method.

(5) *Ivermectin*.—Topic Advisor David W. Fink, Merial Ltd., 631 US Route 1 South, NB-500, North Brunswick, NJ 08902, Tel: 732-729-5722, Fax: 732-729-5761, E-mail: David.Fink@merial.com. There is no activity to report.

(6) *Lasalocid*.—Study Director Charles L. Focht, Nebraska Department of Agriculture, 3703 S. 14th St, Lincoln, NE 68502-5399, Tel: 402-471-2176, Fax: 402-471-0091, E-mail: chaslf@agr.state.ne.us. The Study Director is planning a collaborative study of the method, *Analyst* **120**, 2179–2180 (1995), but with a more rigorous extraction procedure that is required for mineral premixes. It was recently identified that the analysis of 4 homologues needs to be included in the method; chromatographic conditions and quantitation parameters are being finalized. Continue study.

(7) *Melengestrol Acetate in Feeds*.—Topic Advisor Dawn A. Merritt, Pharmacia Animal Health, MR 7926-300-409, 301 Henrietta St, Kalamazoo, MI 49007, Tel: 616-833-2382, Fax: 616-833-7721, E-mail: dawn.a.merritt@pfizer.com. There is no activity to report.

(8) *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. The Study Director reports that a joint ISO/AOAC collaborative study of an LC method using post-column derivatization, similar to AOAC Official Method **997.04**, has been completed. The report for AOAC has been submitted. The study was successful for all feeds (trace levels and medicated feeds and premixes), but failed for drug premixes.

(9) *Monensin, LC Method*.—Topic Advisor Mark R. Coleman. Method **997.04** “Monensin in Premix and Animal Feeds” has Final Action status.

(10) *Morantel Tartrate*.—A Study Director is required. An LC method is desired; method published by Goras and Gauthier, *JAOAC* **68**, 598–601 (1985), is a good possibility.

(11) *Narasin, LC Method*.—Topic Advisor Mark R. Coleman. See (8).

(12) *Nicarbazin*.—Topic Advisor Noah Miller, Koffolk (1949) Ltd., Hamagshimim St 7, Kirjat Matalon, Petach Tikva, Israel, Tel: 972-3-927-3139, Fax: 972-3-923-0341, E-mail: noah@koffolk.co.il. Continue study of an LC method reported by Krabel et al., *J. AOAC Int.* **83**, 1027–1037 (2000).

(13) *Oxytetracycline, LC Method*.—Study Director Luann Wetzler, Nebraska Department of Agriculture, 3703 S. 14th St, Lincoln, NE 68502-5399, Tel: 402-471-2176, Fax: 402-471-0091, E-mail: luanmw@agr.state.ne.us. A collaborative study of the method of Houghlum, Larson, Mutchler, and Wetzler, *J. AOAC Int.* **81**, 919–922 (1998), is being initiated by Richard Larson.

(14) *Pyrantel Tartrate*.—A new Study Director is required. An LC method is desired.

(15) *Roxarsone*.—Topic Advisor Margaret Pomeroy, AlphaPharma Inc., Animal Health Division, 400 State St, Chicago Heights, IL 60411-1242, Tel: 708-758-0111, Fax: 708-757-2510, E-mail: peggy.pomeroy@alpharma.com. AOAC Official Method **971.47** is the most commonly used procedure for roxarsone (3-nitro-4-hydroxyphenyl arsonic acid) in feeds. There was no activity on the development of an LC method. Continue study.

(16) *Sulfamethazine, LC Post-Column Method*.—Topic Advisor Kendrick Albert, Office of the Indiana State Chemist, Purdue University, 1154 Biochemistry Bldg, West Lafayette, IN 47907-1154, Tel: 765-496-3079, Fax: 765-494-4331, E-mail: albertk@purdue.edu. Method **999.16** received Final Action status in 2003. There is no activity to report.

(17) *Tilmicosin*.—The Topic Advisor position is vacant. The current method and interlaboratory study information is published, *J. AOAC Int.* **80**, 1156–1170 (1997).

(18) *Tylosin, LC Method*.—Topic Advisor Mark R. Coleman. There is no activity to report. Continue study.

(19) *New Topics*.—Study Directors are solicited for other topics such as clopidol, decoquinat, diclazuril, fenbendazole, halofuginone, levamisole, maduramicin, ormetoprim/sulfadimethoxine, robenidine, semduramicin, and tiamulin. Interested scientists or organizations are encouraged to contact the General Referee or AOAC INTERNATIONAL for more information.

Feeds

(1) **2002.04** *Amylase-Treated Neutral Detergent Fiber*.—Study Director David Mertens, U.S. Dairy Forage Research Center, Madison, WI, Tel: 608-264-5228, Fax: 608-264-5275, E-mail: davem@dfrc.dfr.wisc.edu. Approved as First Action Method **2002.04**, Gravimetric Determination of Amylase-Treated Neutral Detergent Fiber in Feeds Using Refluxing in Beaker or Crucibles. Method recommended for Final Action status. Discontinue study.

(2) **2001.11** *Crude Protein in Animal Feeds, Forages, Cereal Grains, and Oilseeds; Block Digestion, Steam*

Distillation Method.—Study Directors Nancy Thiex and Harold Manson, South Dakota State University, Oscar E. Olson Biochemistry Labs, Brookings, SD 57007, Tel: 605-688-5466, Fax: 605-688-6295, E-mail: nancy_thiex@sdstate.edu and harold_manson@sdstate.edu; Shirley Anderson, Foss North America, 7682 Executive Dr, Eden Prairie, MN 55344, Tel: 952-974-9892, ext. 161, Fax: 952-974-9823, E-mail: sanderson@fossnorthamerica.com; and Jan-Ake Persson, Foss Tecator, Tecator AB, Box 70, SE-263 21 Höganäs, Sweden, Tel: 46-42-361500 or 46-42-361638, Fax: 46-42-340349, E-mail: jan-ake.persson@foss.tecator.se. Approved as First Action Method **2001.11** Determination of Crude Protein in Animal Feed, Forage, Grain, and Oilseed, Block Digestion Using Copper Catalyst, Steam Distillation into Boric Acid. Method recommended for Final Action status. Discontinue study.

(3) **2003.05** *Crude Fat, Diethyl Ether Extraction, in Feeds, Cereal Grains, and Forages by Randall/Soxtec/Submersion Method*.—Study Directors Nancy Thiex, Bryan Gildemeister, South Dakota State University, Brookings, SD, Tel: 605-688-5466, Fax: 605-688-6295, E-mail: bryan_gildemeister@sdstate.edu, and Shirley Anderson. Approved as First Action Method **2003.05**, Crude Fat, Diethyl Ether Extraction, in Feeds, Cereal Grains, and Forages by Randall/Soxtec/Submersion Method. Method recommended for Final Action status. Discontinue study.

(4) **2003.06** *Crude Fat, Hexanes Extraction, in Feeds, Cereal Grains, and Forages by Randall/Soxtec/Submersion Method*.—Study Directors Nancy Thiex, Bryan Gildemeister, and Shirley Anderson. Approved as First Action Method **2003.06** Crude Fat, Hexanes Extraction, in Feeds, Cereal Grains, and Forages by Randall/Soxtec/Submersion Method. Method recommended for Final Action status. Discontinue study.

(5) *Inorganic Elemental Constituents of Plant Samples, Microwave Digestion*.—Topic Advisor Robert Miller, Colorado State University, Fort Collins, CO, Tel: 970-493-4382, Fax: 970-416-5820, E-mail: rmiller@lamar.colostate.edu and Nancy Thiex. Continue topic.

(6) *Microscopy*.—Topic Advisor Mike Bucker, Division of Consolidated Lab Services (DCLS), 1 N 14th St, Room 127, Richmond, VA 23219, Tel: 804-225-4070, Fax: 804-796-7795, E-mail: mbucker@dgs.state.va.us. No activity during the past year. Continue topic.

(7) **2001.12** *Water (Moisture)/Dry Matter in Animal Feed, Grain and Forage (Plant Tissue) by Karl Fischer Titration*.—Study Directors Nancy Thiex and Terri Van Erem, SD State University, Oscar E. Olson Biochemistry Labs, PO Box 2170, Brookings, SD 57007-1217, Tel: 605-688-5466, Fax: 605-688-6295, E-mail: terri_vanerem@sdstate.edu. Method approved as First Action Method **2001.12** Determination of Water (Moisture)/Dry Matter in Animal Feed, Grain, and Forage (Plant Tissue), Karl Fischer Titration Methods. The Alternative Extraction Procedure for AOAC Method **2001.12** was also accepted as First Action and

incorporated into Method **2001.12**. Method recommended for Final Action status. Discontinue study.

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

(9) **2000.12** *Phytase Activity in Feeds*.—Study Director, SDM Food Specialties, Dept. ISL/SP pp 010-0585, PO Box 1, Delft, 2613-MA, The Netherlands, Tel: 31-15-2793543, Fax: 31-15-2792357. Method has Official First Action Status. Continue study.

(10) *Sample Preparation*.—Topic Advisor George W. Latimer, Office of the Texas State Chemist, College Station, TX 77841-3160, Tel: 979-845-1121, Fax: 979-845-1389, E-mail: GWL@otsc.tamu.edu. Topic Advisor retiring. Discontinue topic.

(11) *Vitamin A, LC Method*.—Find new Study Director/Topic Advisor. Continue topic.

Fertilizer and Agricultural Liming Materials

(1) **929.01** *Sampling*.—Study Director Peter Kane, Office of the Indiana State Chemist, Purdue University, Biochemistry Building, 175 South University St, West Lafayette, IN 47907-2063, E-mail: kanep@purdue.edu. Evaluation of a procedure to sample fertilizer from 1 ton mini-bulk fertilizer bags has been conducted by former Study Director Douglas Caine. It is the intention of the GR to complete Caine's work and submit a method modification, pending appropriate determination of priority status by the Agricultural Materials Task Force. Therefore, the Study Director recommends continued study.

(2) **983.02** *Potassium, Flame Photometric Detection*.—Study Director Natalie Newlon, Office of the Indiana State Chemist, Purdue University, Biochemistry Bldg, 175 South University St, West Lafayette, IN 47907-2063, Tel: 765-494-1563, Fax: 765-494-4331, E-mail: newlonn@purdue.edu. Newlon recommended, and AOAC approved, an alternative extraction procedure for fertilizer potassium, **983.02B(c)**. The journal article summarizing the study and recommendations is now published, *JAOAC*, Vol. **86**, No. 4, 2003. In order to monitor performance of the method, the Study Director recommends continued study.

(3) *Controlled Release Nutrient Extraction*.—Study Director William Hall, IMC Global, PO Box 2000, 3095 County Rd, 640 W, Mulberry, FL 33860-1100, Tel: 863-428-7161, Fax: 863-428-7398, E-mail: wlhall@imcglobal.com. Precollaborative study data was received, and a review of the data and comments indicated the need for additional modifications in the procedure. The procedure has been revised and is ready for a second study.

Data and an update presentation were submitted to the AAPFCO Slow Release Task force in August 2004. All revisions have now been completed, and the single-laboratory evaluations show better accuracy, precision, and ease of use. Additionally, a reference incubation method for testing products in a biologically active soil setting has been tested and is complete. This allows correlation of the laboratory method to data generated by an agronomic (reference) method. Correlations of the 2 methods is high, >.90. It is anticipated a new collaborative study can begin in October of 2004 that will develop correlated data with the reference method. The final study and method will be submitted to the Journal and presented at the 2005 AOAC meeting. The Study Director recommends continued study.

(4) *Trace Elements in Fertilizers*.—Study Director David W. Averitt, IMC Global, PO Box 2000, Mulberry, FL 33860, Tel: 863-428-2500 ext. 4436, Fax: 863-428-7374, E-mail: dwaveritt@imcglobal.com. A third Metals Forum was held in Tampa, FL, February 18–19, 2004. The forum has recommended a microwave digestion of 1 g samples and 10 mL HNO₃, at 200 C. A hot block variation of the digestion procedure is still being investigated. Details of an instrumentation detection procedure are still being developed. A collaborative study is being planned for sometime this fall. Suitable reference materials with certified trace element content are being developed in collaboration with NIST for use in the collaborative study. The Study Director recommends continued study.

(5) *Determination of Urea in Fertilizers by LC, I-29*.—Study Director Michael Hojjatie, Tessenderlo Kerley Inc., 2480 Twin Buttes, Sahuarita, AZ 85629, Tel: 520-791-2940, Fax: 520-625-8091, E-mail: mhhojjatie@tkinet.com. Liquid chromatographic determination of urea in water soluble urea-formaldehyde fertilizer products and in aqueous urea solutions was given First Action status in November 2003, and was assigned as Method **2003.14**. The method was published in the *J. AOAC Int.* **87**, 346 (2004). The Study Director recommends continued study.

(6) **982.01** *Boron in Fertilizers*.—Study Director Wesley Hsu, U.S. Borax Inc., 26877 Tournay Rd, Valencia, CA 91355-1847, Tel: 661-287-5400, Fax: 661-287-5495, E-mail: Wesley.hsu@borax.com. Hsu submitted a revision of AOAC **982.01** in September 2002 to the Methods Committee on Feeds, Fertilizers, and Related Topics. Currently the proposal is awaiting assignment of priority status in the workload of the committee. The Study Director recommends continued study.

Nutrients in Soils

Tobacco

Veterinary Analytical Toxicology