

METHODS COMMITTEE REPORTS

Committee on Feeds, Fertilizers, and Related Agricultural Topics

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Committee Actions

The committee continued to process several collaborative studies since the 2002 AOAC Annual Meeting and Exposition. During the first Committee I conference call, collaborative studies "Crude Fat, Hexanes Extraction, in Feed, Cereal Grain, and Forage (Randall/Soxtec/Submersion Method)," and "Crude Fat, Diethyl Ether Extraction, in Feed, Cereal Grain, and Forage (Randall/Soxtec/Submersion Method)" were adopted with modifications as First Action on May 8, 2003. They were assigned as Official Methods **2003.06** and **2003.05**, respectively. Another collaborative I-29, is laboriously moving through the process continuing toward committee action. The second committee conference call was held July 28, 2003. During this call the committee formed a consensus that collaborative study I-29 should be approved with some manuscript editing. After a review of the manuscript, the

chairman will forward the method on for First Action to Committee I.

At the August meeting of the Control Officials (specifically the AAFCO and AAPCO) held in Denver, CO, AOAC representatives, AAFCO, and AAPCO members discussed the possibility of targeting needed methodology and developing funding sources for the collaboratively studies for those targeted methods.

The Committee Chair has attempted to increase the number of Committee I members. Two committee members rotated off the committee and then in May 2003 another member resigned their position on the committee. Anthony T. Le was added to the committee in July. It has been suggested that members of AAFCO and AAPCO method services may provide additional committee members.

This was the last meeting with Mark Coleman as Committee Chair. Current secretary, Lu Wetzler, became the Chairman at the end of the 117th AOAC Annual Meeting and Exposition. Additional committee members and a secretary have not yet been identified.

Antibiotics in Feeds

(1) *Apramycin in Feeds*.—Vacant.

(2) **993.29** *Bacitracin in Feeds*.—Topic Advisor, Elizabeth Luigs, Alpharma, Animal Health Division, 400 State St, Chicago Heights, IL 60411, Tel: +1-708-758-0111, Fax: +1-708-757-2510. Topic Advisor for Zinc and MD Bacitracin. Continue study.

(3) *Neomycin in Feeds*.—Topic Advisor, Scott Panagiotis, Pennfield Animal Health, 14040 Industrial Rd, Omaha, NE 68144, Tel: +1-402-330-6000, Fax: +1-402-330-6004, 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: +1-317-277-463, Fax: +1-317-277-4993, E-mail: coleman_mark_r@lilly.com. Continue study.

(6) *Narasin*.—Topic Advisor, Mark R. Coleman. Microbiological method. Continue study.

(7) *Penicillin*.—Topic Advisor, Steven Holmes, Alpharma, 400 State St, Chicago Heights, IL 60411, Tel: +1-708-757-2505, Fax +1-708-757-3510. Continue study.

(8) *Chlortetracycline*.—Topic Advisor, Steven Holmes. Continue study.

(9) *Virginiamycin with Other Drugs*.—Topic Advisor, Hussein Ragheb, Purdue University, Office of Indiana State Chemist, 1154 Biochemistry, W. Lafayette, IN 47907, Tel: +1-765-494-1572, Fax: +1-765-496-6349, E-mail: Raghebh@purdue.edu. Continue study.

(10) *Bambermycin*.—Topic Advisor, Denise Riley Moore, Woodson Tenent Laboratories, 345 Arbor Ave, Memphis, TN 38103, Tel: +1-901-521-4500, Fax: +1-901-521-4510, 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: +1-613-759-1299, Fax: +1-613-759-1260, E-mail: farmstrong@inspection.gc.ca. The Study Director reports that LC separation on a phenyl-hexyl column using a mobile phase of methanol and water, containing acetic acid and ammonium hydroxide, is being investigated for use with both UV and LC/MS detection.

There are 2 papers of note in the literature for 2002. Furusawa determined amprolium in chicken tissues, to a limit of quantitation of 0.22 g/g, using a fast and simple LC/UV method (Furusawa, N. (2002) *J. Chromatogr. Sci.* **40**, 355–358). Hormazabal determined amprolium and 5 other coccidiostats in feeds using an LC/MS method. The quantitation range for amprolium was 20 to 150 g/g with an estimated detection limit of 2 g/g (Hormazabal, V., Yndestad, M., & Ostensvik, O. (2002) *J. Liq. Chrom. Rel. Technol.* **25** (17), 2655–2663). Continue study.

(2) *Carbadox*.—Study Director, Alexander MacDonald, Pharma Science, Inc., 16 Cypress Ave, North Caldwell, NJ 07006, Tel: +1-973-228-2392, Fax: +1-973-228-3498, E-mail: BeeMac201@aol.com. An LC method based on the method of Aerts and Werdmuller [*J. Assoc. Off. Anal. Chem.* **71**, 484–489 (1988)] will be studied in conjunction with FDA to meet the *Peer-Verified Methods*SM Program requirements.

(3) *Chlortetracycline, LC Method*.—Study Director, Richard Larson, South Dakota State University, ASC 133, PO Box 2170, Brookings, SD 57007-1217, Tel: +1-605-688-6706, Fax: +1-605-688-6295, E-mail: richard_larson@sdstate.edu. Work continues on optimizing and disseminating the method of Houglum and Larson, *J. AOAC Int.* **80**, 961–965 (1997). Some laboratories using low pressure mixing of the mobile phase are experiencing precipitation problems with the mobile phase buffer when the methanol content approaches 60%. A change in the mobile phase buffer to improve the solubility is being investigated. Continue study.

(4) *Ethopabate*.—Topic Advisor, Joseph Hillebrandt, Cornell University, Diagnostic Laboratory, Nutritional & Environmental Analytical Services, 777 Warren Rd, Ithaca, NY 14850, Tel: +1-607-257-2345, Fax: +1-607-257-5041, E-mail: jh119@cornell.edu. Continue study of LC method.

(5) *Ivermectin*.—Topic Advisor, David W. Fink, Merial Ltd., 631 U.S. Route 1 South, NB-500, North Brunswick, NJ 08902, Tel: +1-732-729-5722, Fax: +1-732-729-5761, E-mail: David.Fink@merial.com.

(6) *Lasalocid*.—Study Director, Charles L. Focht, Nebraska Department of Agriculture, 3703 S. 14th St, Lincoln, NE 68502-5399, Tel: +1-402-471-2176, Fax: +1-402-471-0091, E-mail: chaslf@agr.state.ne.us. Study Director is intending to conduct an interlaboratory study of the method, *Analyst* **120**, 2179–2180 (1995), but with a more rigorous extraction procedure. The revised extraction procedure is required for mineral premixes. 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: +1-616-833-2382, Fax: +1-616-833-7721, E-mail: dawn.a.merritt@pharmacia.com.

(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: +1-613-759-1227, Fax: +1-613-759-1260, 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 remains to be completed.

(9) *Monensin, LC Method*.—Topic Advisor, Mark R. Coleman. Method **997.04** “Monensin in Premix and Animal Feeds” was approved for First Action in 1997. The method is recommended for Final Action.

(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.

(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: +1-402-471-2176, Fax: +1-402-471-0091, E-mail: luannmw@agr.state.ne.us. Continue study of the method of Houglum, Larson, Mutchler, and Wetzler, *J. AOAC Int.* **81**, 919–922 (1998).

(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: +1-708-758-0111, Fax: +1-708-757-2510, 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. An LC method is being sought; however, no advances have been made during the last year. Continue study.

(16) *Sulfamethazine, LC Post-Column Method*.—Topic Advisor, Kendrick Albert, Purdue University, Office of the Indiana State Chemist, 1154 Biochemistry Bldg, W. Lafayette, IN 47907-1154, Tel: +1-765-496-3079, Fax:

+1-765-494-4331, E-mail: albertk@purdue.edu. Method **999.16** received Final Action status in 2003.

(17) *Tilmicosin*.—Topic Advisor, Robin S. Readnour, Elanco Animal Health, A Division of Eli Lilly and Co., 2001 W. Main St, Greenfield, IN 46140, Tel: +1-317-277-5063, Fax: +1-317-277-4993, E-mail: R.S.Readnour@lilly.com. 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. Work continues slowly on the development of an LC method. Continue study.

(19) *New Topics*.—Study Directors are solicited for other topics such as clopidol, decoquinat, diclazuril, halofuginone, maduramicin, ormetoprim/sulfadimethoxine, 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: +1-608-264-5228, Fax: +1-608-264-5275; E-mail: davem@dfrc.dfrc.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. Continue study.

(2) **2001.11** *Crude Protein in Animal Feeds, Forages, Cereal Grains, and Oilseeds, Block Digestion, Steam Distillation Method, I-3*.—Study Directors, Nancy Thiex and Harold Manson, South Dakota State University, Oscar E. Olson Biochemistry Labs, Brookings, SD 57007, Tel: +1-605-688-5466, Fax: +1-605-688-6295, E-mail: nancy_thiex@sdstate.edu and harold_manson@sdstate.edu; Shirley Anderson, Foss North America, 7682 Executive Dr, Eden Prairie, MN 55344, Tel: +1-952-974-9892, ext. 161, Fax: +1-952-974-9823, 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). The method is under consideration by ISO and CEN. Continue study.

(3) *Crude Fat, Diethyl Ether Extraction, in Feeds, Cereal Grains, and Forages by Randall/Soxtec/Submersion Method, Ic-017*.—Study Directors, Nancy Thiex and Bryan Gildemeister, South Dakota State University, Brookings, SD, E-mail: bryan_gildemeister@sdstate.edu; Shirley Anderson. Collaborative study manuscript has been submitted to AOAC INTERNATIONAL and is in the review process at the Methods Committee level. Continue study.

(4) *Crude Fat, Hexanes Extraction, in Feeds, Cereal Grains, and Forages by Randall/Soxtec/Submersion Method, Ic-028*.—Study Directors, Nancy Thiex, Bryan Gildemeister, and Shirley Anderson, E-mail: sanderson@fossnorthamerica.com. Collaborative study manuscript

has been submitted to AOAC INTERNATIONAL and is in the review process at the Methods Committee level. Continue study.

(5) *Inorganic Elemental Constituents of Plant Samples, Microwave Digestion*.—Topic Advisors, Robert Miller, Colorado State University, Fort Collins, CO, Tel: +1-970-493-4382, Fax: +1-970-416-5820, E-mail: rmiller@lamar.colostate.edu and Nancy Thiex, E-mail: nancy_thiex@sdstate.edu. No activity during the past year. Continue topic.

(6) **999.13** *Lysine, Methionine, and Threonine in Pure Amino Acids (Feed Grade) and Premixes*.—Study Directors, Johannes Fontaine, Degussa AG Hanau, Germany, Tel: +49 6181 593259, Fax: +49 6181 593908 and Marcelle Eudaimon, Customer's Laboratory, Zone Industrielle Nord, Rue de Vaux, F-80884 AMIENS CEDEX 2 France, Tel: +33 225470-11 or 17, Fax: +3 22547038, E-mail: Johannes.Fontaine@degussa-huels.de. Method recommended for Final Action status. Discontinue study.

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

(8) **2001.12** *Water (Moisture)/Dry Matter in Animal Feed, Grain, and Forage (Plant Tissue) by Karl Fischer Titration, I-28*.—Study Directors Nancy Thiex and Terri Van Erem, South Dakota State University, Oscar E. Olson Biochemistry Labs, PO Box 2170, Brookings, SD 57007-1217, Tel: +1-605-688-5466, Fax: +1-605-688-6295, 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 Official First Action and incorporated into Method **2001.12**. Continue study.

(9) *Neutral Detergent Fiber, Acid Detergent Fiber, and Lignin Using Filter Bag Technology*.—Study Director, Andrew Komarek, ANKOM Technology Corp., Fairport, NY, 14450, Tel: +1-716-425-3940, Fax: +1-716-425-3941, E-mail: akomarek@ankom.com. Continue study.

(10) **2000.12** *Phytase Activity in Feeds*.—Study Director, Peter. H.G. Randsdorp, 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, E-mail: Peter.Randsdorp@dsm-group.com. Method has Official First Action status. Continue study.

(11) *Sample Preparation*.—Topic Advisor, George W. Latimer, Office of the Texas State Chemist, College Station, TX, 77841-3160, Tel: +1-979-845-1121, Fax: +1-979-845-1389, E-mail: GWL@otsc.tamu.edu. Continue topic.

(12) **999.12** *Taurine in Pet Foods, Liquid Chromatographic Method*.—Study Director, Claudia Hischenhuber, NESTEC Ltd Research Centre, Quality & Safety Assurance Department, Vers-Chez-Les-Blanc, Lausanne 26, 1000-CH, Switzerland, Tel:

+41-21-7858352, Fax: +41-21-7858553, E-mail: claudia.hischenhuber@rdls.nestle.com. Method is recommended for Final Action status. Discontinue study.

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

Fertilizer and Agricultural Liming Materials

(1) **929.01 Sampling**.—Study Director, Peter Kane. Kane has been appointed SD in order to complete the work of former SD Douglas Caine, a method modification to the bulk sampling procedure, to address the need of sampling from 1 ton mini-bulk fertilizer bags. It is the intention of the GR to complete Caine's work and submit a method modification. Therefore, the Study Director recommends continued study.

(2) **983.02 Potassium, Flame Photometric Detection**.—Study Director, Natalie Newlon, Purdue University, Office of the Indiana State Chemist, Biochemistry Bldg, 175 S. University St, W. Lafayette, IN 47907-2063, Tel: +1-765494-1563, Fax: +1-765-494-4331, E-mail: newlonn@purdue.edu. Last year the Study Director recommended, and AOAC approved, an alternative extraction procedure for fertilizer potassium, **983.02B(c)**. The journal article summarizing the study and recommendations is now at the proof stage, and soon will be published. 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: +1-863-428-7161, Fax: +1-863-428-7398, E-mail: wlhall@imcglobal.com. Collaborative study data was received, and 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 was submitted to the AAPFCO Slow Release Task Force in August 2003. All revisions have now been completed, and in-laboratory evaluation shows better accuracy, precision, and ease of use. Additionally, a reference incubation method for testing products in a biologically active soil setting has been devised. This will allow correlation of laboratory method data to true, yet controlled setting data generated by the reference method. It is anticipated a new study can begin in October of 2003 that will develop data that will be correlated to the reference method, with method presentation at the 2004 AOAC Annual 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: +1-863-428-2500 ext. 4436, Fax: +1-863-428-7374, E-mail: dwaveritt@imcglobal.com. A second Metals Forum was held in February of this year in Sacramento, CA, with representation from state laboratories, fertilizer regulators, fertilizer industry representatives, and instrument manufacturers. An active dialogue between attendees is ongoing, and method development work has led to establishment of a proposed microwave digestion procedure. Parallel work is well in process to evaluate an alternative hot block digestion procedure. Attention will soon focus on the instrumentation portion of the

method. It is anticipated the work will lead to pursuit of AOAC collaboration of digestion methods and instrumental detection methods for trace elements. The Study Director recommends continued study.

(5) *Determination of Urea in Fertilizers by LC, I-29*.—Study Director, Michael Hojjatie, Tessengerlo Kerley Inc., 2480 Twin Buttes, Sahuarita, AZ 85629, Tel: +1-520-791-2940, Fax: +1-520-625-8091, E-mail: mhhojjatie@tkinet.com. Hojjatie has received GR, Safety Committee, and statistical approval, and conducted a collaborative study. A summary paper has been submitted to the methods committee for review. The Study Director recommends approval of the method.

(6) **982.01 Boron in Fertilizers**.—Study Director, Wesley Hsu, U.S. Borax Inc., 26877 Tourney Rd, Valencia, CA 91355-1847, Tel: +1-661-287-5400, Fax: +1-661-287-5495, E-mail: Wesley.hsu@borax.com. Hsu has been appointed Study Director. Plans for minor revision of the method are on hold, pending resolution of AOAC's current difficulties. The General Referee recommends continued study.

Nutrients in Soils—AOAC Soil Methods Validation Status

All Study Directors assigned to soil methods investigations have suspended their activities with regard to AOAC soil methods validations, due to the costs now attached to the submission of AOAC collaborative study protocols. An alternative to AOAC's Official Methods Program for soil methods validation has been proposed and is under consideration by the following committees of the Soil Science Society of America (SSSA): S889, the Committee for Coordination of Official Methods of Soil Analysis, S890, the North American Proficiency Testing Program Oversight Committee, and S877, the Soil and Plant Analysis Committee. As AOAC General Referee for Nutrients in Soils, and AOAC Liaison to SSSA, Charles Focht will attend these committee meetings at the 2003 joint conference of the American Society of Agronomy and the Soil Science Society of America, to be held November 2–6, 2003 in Denver, CO. He will report to AOAC on any deliberations and decisions that are made regarding the future of AOAC, or other, soil methods validations initiatives.

(1) *Available Phosphorus in Soils, Bray P1 Method*.—Study Director, Bryan Hopkins, University of Idaho, 1776 Science Center Dr, Idaho Falls, ID 83402-1575, Tel: +1-208-529-8376, E-mail: bhopkins@uidaho.edu.

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

(3) *Plant Available Zinc, Manganese, Iron and Copper in Soils, DTPA Method*.—Study Director, Donald Horneck, Oregon State University Extension Service, Hermiston Agricultural Research & Extension Center, PO Box 105, Hermiston, OR 97838, Tel: +1-541-567-8321, Fax: +1-541-567-2240, E-mail: don.horneck@orst.edu.

(4) *Available Phosphorus in Soils-Olsen Bicarbonate Method*.—Study Director, Brian Shreve, Servi-Tech Laboratories, Inc., 1602 Park W. Dr, Hastings, NE 68901, Tel:

+1-800-468-5411, Fax: +1- 402-463-8132, E-mail: brians@servi-techinc.com.

(5) *Lime Requirement Determinations Soils*.—Study Director, Steve Harrold, Servi-Tech Laboratories, Inc., 1816 E. Wyatt Earp, Dodge City, KS 67801, Tel: +1-800 557-7509, Fax: +1-316-227-2047, E-mail: steveh@servi-techinc.com. Veterinary Toxicology and Tobacco General Referee reports were not submitted this year.