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Committee Actions

The committee has been quite active since the 2001 AOAC Meeting and Exposition. Five collaboratives in various stages are progressing toward committee approval. First was the Alternative Methanol Extraction Procedure for Method 2001.12, on June 13 a conference call was held to discuss and ratify Method 2002.04, Gravimetric Determination of Amylase Treated Neutral Detergent Fiber in Feeds Using Refluxing Beakers or Crucibles. Currently the committee is processing the following collaboratives: IC-017, Crude Fat, Diethyl Ether Extraction in Feed, Cereal Grain, and Forage (Randall/Soxtec/Submersion Method); IC28 Crude Fat, Hexane Extraction in Feed, Cereal Grain, and Forage (Randall/Soxtec/Submersion Method); and IC29, Determination by Liquid Chromatography of Urea in Certain Liquid Fertilizers Containing Substantial Amounts of Water-Soluble Urea-Formaldehyde Reaction Products and in Aqueous Urea Solutions. The goal is to have all reviews completed, reviews by the 2 designated OMB members, and the committee vote set for September at the Committee meeting in Los Angeles.

Antibiotics in Feeds

(1) Apramycin in Feeds: Vacant.
(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 (see 3). Continue study.
(6) Narasin: Topic Advisor Mark R. Coleman (see 5). Continue study.
(8) Chlortetracycline: Topic Advisor Steven Holmes (see 7). Continue study.
(9) Virginiamycin with Other Drugs: Topic Advisor Hussein Raghbeb Office of the Indiana State Chemist, 1154 Biochemistry Bldg, Purdue University, 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.
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 an investigation of alternate LC separations has not yet produced any with a clear advantage over the current systems. Publications in 2001 include an LC method for amprolium and halofuginone in chicken muscle and egg (Yamamoto, Y., & Kondo, F., J. Assoc. Off. Anal. Chem. 84, 43–46[2001]); however, there were no methods published for amprolium in feeds. Continue study.


(3) Chlortetracycline, LC Method: Study Director Richard Larson, South Dakota State University, Oscar E. Olson Biochemistry Lab, 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)]. A presentation was made at the 2001 AOAC Forum on Methods for Antibiotics and Drugs in Feeds detailing changes made since the original publication. Continue study and submit to a collaborative 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.


(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. The Study Director is evaluating 2 reversed-phase LC methods. The method, Analyst 120, 2179–2180 (1995) with some modifications, is being compared to a method with a similar but more rigorous extraction procedure. The latter method appears to give higher results with less variability; however, data is considered to be preliminary. Continue study with the intent of a round robin 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. Continue study.

(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, is in progress.

(9) Monensin, 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: +1-317-277-4613, Fax: +1-317-277-4167, E-mail: Coleman_Mark_R@lilly.com. (See 8.)

(10) Morantel Tartrate: A new 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, Elanco Animal Health, A Division of Eli Lilly and Co., 2001 W. Main St, Greenfield, IN 46140, Tel: +1-317-277-4613, Fax: +1-317-277-4167, E-mail: Coleman_Mark_R@lilly.com. (See 8.)

(12) Nicarbazin: Topic Advisor Noah Miller, Koffolk (1949) Ltd., Hamagshimim St 7, Kiryat 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) Nifursol: Topic Advisor Ellen Jan de Vries, Solvay Pharmaceuticals BV, Fideliolaan 41, Amstelveen, 1183-PH, The Netherlands, Tel/Fax: +31-20-641-1211, E-mail: ej.devries@wxs.nl. Continue study.

(14) Oxytetracycline, LC Method: Topic Advisor 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: luannw@agr.state.ne.us. Continue study.

(15) Pyrantel Tartrate: A new Study Director is required. An LC method is desired.

(16) Roxarsone: Topic Advisor Margaret Pomeroy, Alpharma 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 arsionic acid) in feeds. An LC method is being sought; however, no advances have been made during the last year. Continue study.

(17) Sulfamethazine, LC Post-Column Method: Topic Advisor Kendrick Albert, Office of the Indiana State Chemist, Purdue University, 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 First Action status in 1999. Recommend adoption as Final Action.

(18) 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 was published, *J. AOAC Int.* **80**, 1156–1170 (1997).


(20) **New Topics**: Study Directors are solicited for other topics such as clopidol, decoquinate, 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


(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, Oscar E. Olson Biochemistry Labs, South Dakota State University, 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 340349, Fax: +46 42 361500 or +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/Sextec/Submersion Method, Ic-017**: Study Directors Nancy Thiex and Bryan Gildemeister, South Dakota State University, Brookings, SD, Tel: +1-605-688-5466, Fax: +1-605-688-6295, E-mail: nancy_thiex@sdstate.edu and bryan_gildemeister@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. 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/Sextec/Submersion Method, Ic-028**: Study Directors Nancy Thiex and Bryan Gildemeister, South Dakota State University, Brookings, SD, Tel: +1-605-688-5466, Fax: +1-605-688-6295, E-mail: nancy_thiex@sdstate.edu and bryan_gildemeister@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. 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, South Dakota State University, Brookings, SD, Tel: +1-605-688-5466, Fax: +1-605-688-6295, E-mail: nancy_thiex@sdstae.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: +593908 and Marcelle Eudaimon, Quality Manager-Customer’s Laboratory, Zone Industrielle Nord, Rue de Vaux, F-80884 AMIENS CEDEX 2 France, Tel: +(33)225470-11 or 17, Fax: +(33)22547038, E-mail: Johannes.Fontaine@degussa-huels.de. Method recommended for Final Action status. Discontinue study.

(7) **Microscopy**: Topic Advisor Mike Bucker, 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: mbucker@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, Brooksings, SD 57007-1217, Tel: +1-605-688-5466, Fax: +1-605-688-6295, E-mail: nancy_thiex@sdstate.edu and terri_vanerem@sdstate.edu. Method approved as AOAC Official 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**. 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.


(11) **Sample Preparation**: Topic Advisor George W. Latimer, Office of the Texas State Chemist, College Station, TX
continued study of the topic. Therefore, the General Referee is in the process of being appointed Study Director for Sampling, and recommends continuation. Thus, in addition to providing the GR to complete Caine’s work and submit a method modification to the methods committee for review. The Study Director recommends approval of the method.

(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: whall@imcglobal.com. Collaborative study data was received, and review of the data and comments indicates the need for additional modifications in the procedure. Data and an update presentation was submitted to the AAPFCO Slow Release Taskforce. Based on TF input and the author’s analysis it was determined that another set of samples should be sent to participants. However, in order to incorporate the modifications to the procedure, additional developmental time was needed to evaluate the effects of the changes (if any) to the method. This work has now been completed, and evaluation shows better accuracy, precision, and ease of use. It is anticipated a new study can begin with a presentation at the 2003 AOAC meeting. Continue study.

(4) Trace Elements in Fertilizers: Study Director David W. Averitt, IMC Global, PO Box 2000, 3095 CR640 W., Mulberry, FL 33860-1100, Tel: +1-863-428-2675, Fax: +1-863-428-1563, E-mail: dwa@imcglobal.com. In February of this year a Metals Forum was held in Lakeland, FL, with an attendance of about 70 representatives from state laboratories, fertilizer regulators, fertilizer industry representatives, and instrument manufacturers. In addition to presentations of the status of regulations, fertilizer methodology, and related issues, 4 ongoing work groups were formed: Digestion, Detection, Soil Extracts and Plant Uptake, and Regulations and Enforcement. As a result of the forum, an interlaboratory evaluation of various digestion techniques has been conducted, and additional objectives and goals established for each work group. An active dialogue between attendees has been established, and it is anticipated that work will lead to pursuit of AOAC collaboration of digestion methods and instrumental detection methods for trace elements. Continue 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: +1-520-791-2940, Fax: +1-520-625-8091, E-mail: mhojjatie@tkinet.com. Hojjatie has received General Referee, 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 recently been appointed Study Director, and will be conferring with the General Referee for the purpose of making an editorial change to the method. Continue study.

Nutrients in Soils

Recent developments regarding AOAC soil methods validation initiatives.—At the 2001 AOAC Meeting and Exposition, the AOAC volunteer membership was advised by Executive Director, James Bradford, of the severe financial difficulties faced by the association. Among the many reasons cited were dramatically reduced sales of OMA over the past decade, and the withdrawal of support by the FDA. Among the cost saving measures employed was a significant reduction in AOAC’s permanent staff, resulting in major backlog in the processing of analytical methods through the Official Methods Program. The Executive Director emphasized that in order for AOAC to survive financially, a new business model would have to be developed. According to the new business plan, a key change to the Official Methods Program would be for AOAC to “charge for the process, instead of for the product.” In other words, sales of OMA could no longer be counted upon to provide sufficient funding; instead a cost would be attached to submitting a study protocol to the Official Methods Program, as well as additional costs at various stages of the collaborative process, and for ultimate publication in the Journal of AOAC INTERNATIONAL. The total costs of validating and publishing a method were estimated to be as much as $30,000.
studies within the Official Methods Program were discussed by committee members in attendance, and later through e-mail correspondence. There was general agreement within the committee that the cost involved with conducting a full collaborative study through AOAC would be prohibitive, and alternatives for soil methods validations should be explored.

In addition, several AOAC-assigned Study Directors who are conducting collaborative studies for soil methods expressed concerns about submitting their study reports to AOAC for official validation. It is their belief that publication of these methods in OMA would bestow upon these methods a unique status, to the discredit of other, equally suitable, test procedures for the same soil analytes. Due to the anticipated costs attached to the submission of additional soil methods for AOAC validation, the group of soil methods published in OMA is likely to remain very small. The AOAC General Referee shares the concerns of the Study Directors, as do other S889 Committee members.

Due to the questions resulting from AOAC’s new business policy, all Study Directors assigned to soil methods investigations have suspended their activities with regard to AOAC soil methods validations. An alternative to AOAC’s Official Methods Program for soil methods validation has been proposed, and will be considered at the next annual joint meeting of the American Society of Agronomy and the Soil Science Society of America, to be held in November 2002, in Indianapolis, Indiana. As AOAC General Referee for Nutrients in Soils, Focht will attend SSSA subcommittee meetings at that conference, and will report to AOAC on any deliberations and decisions that are made regarding the future of AOAC soil methods validations.

(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. SD has completed the first draft of his study report.

(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. SD reports data has been received from 26 of the 29 collaborators who participated in the study. Twelve soils in blind duplicate were used in the study.

(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. SD reports data has been received from all 21 collaborators who participated in the study. Twelve soils in blind duplicate were used in the study.

(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. Continue study.

(5) Lime Requirement Determinations in 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. Continue study.