Committee on Feeds Fertilizers and Related Agricultural Topics

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Committee Actions

No methods were due for First or Final Action this year.

Three collaborative studies are in progress. Studies for OMA-2004-Oct-017 *Oxytetracycline, LC Method,* OMA-2005-Jan-002 *Lasalocid*, and OMA 2006-Jun-050 *Determination of Decoquinate in Feeds by Liquid Chromatography* are underway. The driving force for new methods entering the collaborative study process in the past year continues to be the Agricultural Materials Community and its subgroups. A summary of the activities of the Community is available on the AOAC Website at http://www.aoac.org/Ag_Materials/community.htm. Information on the Fertilizer Subgroup is available at http://www.aoac.org/Ag_Materials/fertilizer/mission.htm and information on the Feed Additives and Contaminants Subgroup is available at http://www.aoac.org/Ag_Materials/additives/main.htm.

Antibiotics in Feeds, Hussein Ragheb

No reports regarding analysis of antibiotics in animal feeds were received from any of the study directors and no GR report was received.

Drugs in Feeds, Vacant (Harold Campbell) GR ***(IS THE POSITION VACANT OR IS IT HAROLD CAMPBELL?)***

(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. The topic advisor reports no progress.

(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. 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 dibutylamine acetate (DBAA) and acetonitrile. Both analytes exhibit excellent peak shape when using a C18 column that is both acid- and base-deactivated. 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. No work was reported in 2007.

(4) *Decoquinate, LC Method*: Study Director 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. The collaborative study of an LC method of analysis of decoquinate in feeds and mineral premixes has been approved by the Feed Additives and

Contaminants subgroup of the Agricultural Materials Community. Information will be posted on the AOAC Website. The interlaboratory study is being planned for late 2006 or early 2007.

(5) *Ivermectin*: Method Advisor James A. Whitehead, Merial Ltd, 3360 Maury Ave, St. Louis, MO 63116, Tel: 314-752-8053, Fax: 314-752-8061. The current Peer-Verified Method and interlaboratory study information is published, *J. AOAC Int.* (1994) **77**, 1353–1358.

(6) *Lasalocid*. Study Director 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. A collaborative study was conducted in September–October 2005, using 8 samples in blind duplicate covering a range of guarantees from the premix level (68 g/lb) to finished feed level (30 g/T). In addition, 2 sample pairs contaminated at the residue level were included as a component for residue testing. Data for the trace level samples indicated that the samples were not suitably homogeneous. Three new residue level samples were sent to participants as blind duplicates using the identical procedure as was used for the original sample distribution (but with revised LC standard solution concentrations), with the majority of work performed in April–May 2006. Results for these supplemental samples were much improved. The Study Director study report has been submitted to AOAC's Manuscript Central and is being peer reviewed.

(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 manuscript for the joint ISO/AOAC collaborative study was reviewed, revised, and accepted by AOAC Method **2006.01** Analysis of Monensin, Narasin and Salinomycin in Premixes and Animal Feeds by Liquid Chromatography and Post-Column Derivatization has been granted First Action status.

(9) *Monensin, LC Method*: Method 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. Method **997.04** Monensin in Premix and Animal Feeds has Final Action status.

(10) *Morantel Tartrate*: A Topic Advisor is required. An LC method is desired; method published by Goras and Gauthier, *J. Assoc. Off. Anal. Chem.* (1985) **68**, 598–601 is a promising possibility.

(11) *Narasin, LC Method*: Topic Advisor Mark R. Coleman. *See* (9).

(12) *Nicarbazin*: The topic advisor position is vacant. An interlaboratory study of an LC method was reported by de Jong et al. *J. AOAC Int*. (2004) **87**, 1269–1277. A LC method reported by Krabel et al. *J. AOAC Int*. (2000) **83**, 1027–1037 is also a viable method.

(13) *Oxytetracycline, LC Method*: Study Directors Richard Larson and Nancy Thiex, Olson Biochemistry Laboratories, SAS 133, Box 2170, South Dakota State University, Brookings, SD 57006. Richard Larson, Tel: 605-688-6706, Fax: 605-688-6295, E-mail: Richard.Larson@sdstate.edu. A liquid chromatographic (LC) method which utilizes the hydrochloric acid–methanol (1 + 50) extraction solution from AOAC Method **957.23** with mechanical shaking, centrifugation, dilution, and filtration was collaboratively studied in 2006–2007. Determination of oxytetracycline hydrochloride is by reversed-phase LC using a gradient elution with methanol and aqueous buffer containing disodium EDTA, calcium chloride, and sodium acetate and fluorescence detection with excitation of 390 nm and emission of 512 nm. Four familiarization samples and 28 study samples (14 blind duplicates) of animal and fish feeds, animal feed premixes and concentrates, and mineral premixes and animal remedies were sent to 17 collaborators. Levels of oxytetracycline hydrochloride in the study samples ranged from a few mg/kg to >400 000 mg/kg. Acceptable study results were received from 11 laboratories. For the medicated feeds, premixes, mineral premixes, and remedies, RSD_r values (within-laboratory repeatability) ranged from 1.3 to 4.0%, RSD_R values (among-laboratory reproducibility) ranged from 2.4 to 7.7%, and HorRat values ranged from 0.54 to 3.02. For the trace-level test samples, RSD_r values ranged from 9.2 to 10.0%, RSD_R values ranged from 12.9 to 17.3%, and HorRat values ranged from 1.20 to 1.36. The manuscript will be submitted prior to the 2007 Annual Meeting for peer review.

(14) *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 dibutylamine acetate (DBAA) and acetonitrile. Both analytes exhibit excellent peak shape when using a C18 column that is both acid- and base-deactivated. Linearity has been established and initial recovery studies on medicated swine feeds are promising.

(15) *Roxarsone*: Topic Advisor Margaret Pomeroy, Alpharma 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.

(16) *Sulfamethazine, LC Post-Column Method*: Method 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** Liquid Chromatographic–Post-Column Derivatization Method' has Final Action status. There is no information to report on either suggested changes, or reported problems or difficulties.

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

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

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

Feeds, Vacant

(1) *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. No activity during the past year. Investigate applicability to feed sample matrixes. Continue topic.

(2) *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.

(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.

Fertilizer and Agricultural Liming Materials, William Hall, GR

The fertilizer sub-group of the agricultural material community has been quite active in recent months, identifying several new method topics. This increase in activity will likely lead to the appointment of additional Study Directors, as well as several new study topics not listed below.

(1) **929.01** *Sampling*: Study Director Peter Kane, Office of the Indiana State Chemist, Purdue University, Biochemistry Bldg, 175 South University St, West Lafayette, IN 47907-2063, Tel: 765-494-1560, Fax: 765-494-4331. E-mail: Kanep@purdue.edu. The Sampling of fertilizer Bulk Bags manuscript is essentially complete with a recommendation that the sampling procedure be approved First Action. The study was initiated 7–8 years ago, the effort was on hold for a number of years while AOAC reworked its business plan, but now is back on track with a high priority with the Fertilizer Subcommittee of the Agricultural Community. The Study Director vacant, but the GR will make a recommendation for a new SD shortly. The GR 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. While there has been little activity this area is now again an area of interest due to the simultaneous determination of P_2O_5, K_2O and possibly N. The GR recommends continued study.

(3) *Controlled Release Nutrient Extraction*: Study Director 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. Preliminary data was submitted to the AAPFCO Slow Release Committee in 2004. Additionally, a reference incubation method for testing products in a biologically active soil system has been tested and is nearing a protocol submission. This allows correlation of the laboratory method to data generated by an agronomic (reference) method. Correlations of the 2 methods is high (>.90). Data is being generated to develop additional correlations with both methods. A new Study Sirector will be recommended in the near future since the current SD has assumed the GR position. The Study Director recommends continued study.

(4) *OMA-2004-Nov-023 Arsenic, Cadmium, Cobalt, Chromium, Lead, Molybdenum, Nickel, and Selenium in Fertilizer by Microwave Digestion and ICP-OES Detection*: Study Directors Peter Kane and William Hall. The collaborative study manuscript was published in the *J. AOAC Int*. (2006) **89**, **(PLEASE SUPPLY PAGE NUMBERS)** The method received Official First Action status. However there are several related activities to this method and its development that are ongoing. These activities will yield additional methods for trace metals analysis in fertilizer material in the coming months. The GR recommends continued study.

(5) **2003.14** *Determination of Urea in Fertilizers by LC*: Study Director Michael Hojjatie, Tessenderlo Kerley Inc., 2480 Twin Buttes, Sahuarita, AZ 85629, Tel: 520-791-2940, Fax: 520-625-8091, E-mail: mhojjatie@tkinet.com. No report. The General Referee recommends continued study.

(6) **982.01** *Boron in Fertilizers*: Study Director Wesley Hsu, U.S. Borax Inc., 26877 Tourney Rd, Valencia, CA 91355-1847, Tel: 661-287-5400, Fax: 661-287-5495, E-mail: Wesley.hsu@borax.com. The method was revised in 2006 and is now current. No additional information on additional work is available. The General Referee recommends continued study.

Nutrients in Soils, Charles Focht

(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.

AOAC Soil Methods Validation Status: There are currently 3 pending collaborative study reports in the area of nutrients in soils, as cited above. Study data for *Plant Available Zinc, Manganese, Iron, and Copper in Soils, DTPA Method* is expected to be submitted to reviewers in the near future. The study report manuscript is being cowritten by Study Director Don Horneck and Ed Hanlon, Chair of the Soils Subcommittee of AOAC's Task Force on Agricultural Materials. Study Director Maurice Watson has submitted the study report manuscript on *Available Potassium in Soils, Ammonium Acetate Method* to Charles Focht, General Referee for Nutrients in Soils. Statistical results are being reviewed before the report is forwarded on for official AOAC evaluation. Committee S889 of the Soil Science Society of America (SSSA) is responsible for proposing new soil testing methods for official validation: The committee is awaiting submission of the *Available Phosphorus in Soils* study report in order to conduct an initial review before submitting it to AOAC for consideration. Hailin Zhang of the Oklahoma State University, Department of Plant and Soil Sciences, has been appointed by Committee S889 to conduct an in-house study of *Avaliable Phosphorus in Soils, Mehlich III Method*. It is anticipated that a full collaborative study of the procedure will ultimately be conducted by Zhang.

Veterinary Analytical Toxicology, Merl Raisbeck, Acting GR

The GR 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 AAVLD has adopted the 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.