

**METHODS COMMITTEE REPORTS****Committee on Drugs and Related Topics****LEENDERT A. VAN GINKEL, CHAIR**

National Institute of Public Health and the Environment,  
PO Box 1, 3720 AB Bilthoven, The Netherlands

**MARY C. CARSON, SECRETARY**

U.S. Food and Drug Administration, Center for Veterinary  
Medicine, 8401 Muirkirk Rd, Laurel, MD 20708

**CATHARINA Y.W. ANG**

U.S. Food and Drug Administration, National Center for  
Toxicological Research, 3900 NCTR Rd, Jefferson, AR  
72079

**CLYDE NORA CARDUCCI**

Facultad De Farmacia y Bioquímica, Universidad de  
Buenos Aires, Junín 956 (1113) Buenos Aires, Argentina

**JACK F. KAY**

Department for Environment, Food and Rural Affairs,  
Veterinary Medicines Directorate, Woodham Ln, New  
Haw Addlestone, Surrey, KT15-3LS, United Kingdom

**THOMAS P. LAYLOFF**

Management Sciences for Health, 5 Thomas Ct, PO Box  
286, Granite City, IL 62040

**MIN LI**

California Air Resources Board, PO Box 2815,  
Sacramento, CA 95812

**DAVID J. MAZZO**

Schering-Plough Research Institute, 2000 Galloping Hill  
Rd, Kenilworth, NJ 07033

**LINDA NG**

U.S. Food and Drug Administration, Center for Drug  
Evaluation and Research, HFD-550, 5600 Fishers Ln,  
Rockville, MD 20857

**GALEN W. RADEBAUGH (OUTGOING MEMBER)**

Schering-Plough Research Institute, 2000 Galloping Hill  
Rd, Kenilworth, NJ 07033

**VALERIE B. REEVES**

U.S. Food and Drug Administration, Center for Veterinary  
Medicine, 7500 Standish Pl, Rockville, MD 20850

**SUMIT SEN**

U.S. Food and Drug Administration, Office of Regulatory  
Affairs, Pacific Regional Laboratory SW, 1521 W. Pico  
Blvd, Los Angeles, CA 90015

**GEORGE F. PETERSON, SAFETY ADVISOR**

Bureau of Alcohol, Tobacco, and Firearms, Chief of the  
Alcohol Section, 35575 Conovan Ln, Fremont, CA 94536

**FOSTER D. MCCLURE, STATISTICAL ADVISOR**

U.S. Food and Drug Administration, Center for Food  
Safety and Applied Nutrition, 5100 Paint Branch Pkwy,  
College Park, MD 20740

**Committee Actions**

The committee decided at the September 2001 meeting to sponsor a symposium at the 2003 Annual Meeting. During the past year, the committee filled the following General Referee position: Drugs—María Inés Cereijo (replaces Elaine Bunch, who retired).

***Cosmetics, David Prantis***

Avon Products, Inc., Suffern, NY 10901, Tel:  
+1-845-369-2710, E-mail: david.pranitis@avon.com. The  
General Referee's laboratory is developing 4 analytical meth-  
ods for general use in the measurement of drug active ingredi-  
ents in cosmetics. They are focused on common drug ingredi-  
ents: aluminum chlorohydrate, zinc pyrithione, titanium  
dioxide, zinc oxide, and organic sunscreen agents.

*Sunscreen Agents:* A large range of cosmetic products are marketed with SPF claims. Aside from simple sunscreen products themselves, sun protection claims are attached to makeup products, insect repellent products, and skin-care products. Several sunscreen agents are in common use, and these are frequently used in combination with each other within a single formulation.

Organic sunscreen agents in cosmetic products can usually be readily determined by means of liquid chromatography (LC), using ultraviolet detection. LC provides efficient separation of the active ingredients from other formula components, and UV detection is an efficient "screen" for sunscreens because of their intense UV absorptivity.

The goal is to establish standard analytical conditions for time-efficient LC determination of common sunscreen agents in typical cosmetic products. By establishing one or 2 such methods in the AOAC compendium, a "starting point" can be provided for analysts who need to develop particular methods for proprietary formulas. This would also provide a reference method for use by analysts when challenged by regulatory agencies for validity of their sunscreen chromatographic methods.

Development efforts have led to an LC method capable of determining 6 separate sunscreen active ingredients in a single formulation. These 6 are octylmethoxycinnamate, benzophenone-3, butylmethoxydibenzoylmethane, homomenthylsalicylate, octocrylene, and octylsalicylate. Validation data (accuracy, linearity, specificity, etc.) on this method have

been collected, and preparations for cross-testing the method by other interested laboratories are underway.

(1) *Zinc Oxide/Titanium Dioxide, X-Ray Fluorescence Method*: Inorganic sunblocking agents in cosmetic products pose an analytical challenge. These ingredients are typically analyzed by classical means (colorimetry, titration). The final determination is not difficult but the measurement requires elimination of the cosmetic matrix. This sample preparation can be time-consuming and sometimes hazardous, requiring use of solvents and/or concentrated hot acids. Precision of such methods is usually not as great as would be desired, due to the lengthy sample preparation steps.

Inorganic elemental determinations such as these can be accomplished with less intense preparation by means of x-ray fluorescence (XRF). This instrumental technique is well suited to the analysis of complex matrixes because it requires no sample preparation. Cosmetic products merely need to be placed in a reproducible orientation relative to an x-ray beam for reliable measurements to be made. Quantitation is achieved by comparison with standard samples of the same matrix.

Standard XRF conditions for the measurement of ZnO and TiO<sub>2</sub> in cosmetic products are being established. Cross-laboratory evaluation of these methods will be required for further evaluation/validation, because the capabilities of various XRF instruments will need to be considered.

(2) *Zinc Pyrithione, Polarigraphic Method*: This drug active ingredient finds prominent use in antidandruff hair-care products. Classical analytical methods for its determination are based on matrix separation followed by colorimetric measurement. The classical method is time consuming and requires organic solvents.

An alternative method that is by no means new, but should nevertheless be revived, is polarographic measurement of the pyrithione species. This technique requires minimal sample preparation and is therefore much more time-efficient than classical means. The General Referee's laboratory is in the process of revalidating this method in the context of modern hair-care formulas with the goal of making it available for interlaboratory review.

Polarography is historically performed through the use of liquid mercury electrodes. As part of this development, alternatives such as carbon electrodes and film-coated carbon electrodes will be reviewed. This would remove the safety considerations surrounding the use of mercury in the analytical laboratory.

(3) *Aluminum Chlorohydrate*: The General Referee's laboratory is investigating possible new analytical methods for the determination of this drug-active ingredient. Although not typically used in "cosmetic" products, it finds wide use in antiperspirant products and therefore may fall within the purview of this committee as a "personal-care" item of interest.

(4) *Interlaboratory Participation*: These method development/validation activities are pursued with the goal of making contributions to the AOAC methods compendium. As such, they will require participation of collaborating laboratories to establish suitability and reliability. The General Referee in-

vites all interested parties to contact him at the earliest opportunity in order to establish these interlaboratory activities.

Some of these methods require instrumentation (XRF, LC, electrochemical analyzer) that may not be available to all laboratories. Therefore it is critical for any user of this equipment, whether specifically active in cosmetics or not, to consider participation in interlaboratory collaboration so as to widen the participant base as much as possible.

#### ***Drug Residues in Animal Tissues, James MacNeil***

(1) *Residues of Triclabendazole in Liver, LC Method*: Study Director Herbert Koch, Swiss Federal Veterinary Office, Schwarzenburgstrasse 161, Liebefeld 3097-CH, Switzerland, Tel: +41-31-323-8522, Fax: +41-31-323-8522, E-mail: Herbert.Koch@bvet.admin.ch. Discontinue topic.

(2) *Improved Analysis of Tetracycline Residues in Swine Tissues Using Polymer-Based Extraction Cartridges*: Study Director Jan Zmudzki, National Veterinary Research Institute, Al Partyzantow 57, Pulawy 24100, Poland, Tel: +48-81-866-3051, Fax: +48-81-866-2595, E-mail: zmudzki@piwet.pulawy.pl. The General Referee considers that the method as published, involving a study by 5 laboratories, will fit in the proposed e-CAM<sup>®</sup> database in the category of "multilaboratory validated methods." No further work is planned. Discontinue topic.

(3) *Determination of Clopidol in Chicken Tissues*: Study Director Guo-Fang Pang, Qinhuaingdao Entry-Exit Inspection and Quarantine Bureau of P.R. China, No. 39 Haibin Rd, Qinhuaingdao 06600-2, People's Republic of China, Tel/Fax: +86-335-340-7608, E-mail: panggfciq@pang.com.cn. The report is under review by the Committee and the method will be considered for Official First Action upon completion of the review. Continue study.

(4) *Beta-Lactam Antibiotics in Milk, LC Method*: Vacant. Continue study. Seeking new Study Director. Any scientist or organization interested in participating in this topic is asked to contact the General Referee or AOAC INTERNATIONAL.

(5) *Determination of Ivermectin in Animal Tissues*: Vacant. Continue study. Seeking new Study Director. Any scientist or organization interested in participating in this topic is asked to contact the General Referee or AOAC INTERNATIONAL.

(6) *Application of Optical Immunobiosensor Technology to the Determination of Veterinary Drug Residues in Foods*: Establishment of a new topic and appointment of a Topic Advisor is recommended. Any scientist or organization interested in participating in this topic is asked to contact the General Referee or AOAC INTERNATIONAL.

(7) *Oxytetracycline in Shrimp, LC Method*: Topic Advisor Phil Kijak, FDA, Center for Veterinary Medicine, 8401 Muirkirk Rd, Laurel, MD 20708, Tel: +1-301-827-8166, E-mail: pkijak@cvm.fda.gov. This topic is being transferred from the Seafood Methods Committee. A multilaboratory validation study has already been conducted. Phil Kijak is reviewing the data and writing the report.

(8) *Aminoglycosides in Edible Tissues, LC-MS/MS Confirmation Method*: Topic Advisor Mary Carson, FDA, Center for

Veterinary Medicine, 8401 Muirkirk Rd, Laurel, MD 20708, Tel: +1-301-827-8169, E-mail: mcarson@cvm.fda.gov.

(9) *Chloramphenicol in Foods, LC-MS/MS Confirmation Methods*: Topic Advisor Joanne M. Cook, Florida Department of Agriculture and Consumer Services, 3125 Conner Blvd, Lab 3, Tallahassee, FL 32399, E-mail: cookj@doacs.state.fl.us.

***Drugs, Maria Ines Cereijo***

(1) *Atenolone in Combination with Chlorthalidone*: Topic Advisor Everett H. Jefferson. The FDA has no interest in pursuing this study. Discontinue study.

(2) *Dexamethasone in Tablets*: Topic Advisor Elaine A. Bunch. The Topic Advisor has retired from the FDA, and the FDA has no interest in pursuing this study. Discontinue study.

(3) *Reserpine and Rescinnamin in Rauwolfia Serpentina Powders and Tablets, LC Method*: Topic Advisor Ugo Cieri. The FDA has no interest in pursuing this study. Discontinue study.

***Drug Residues in Foods, Diagnostics and Test Kits, Joe Boison***

The General Referee did not report on any activity this past year.

***Forensic Sciences, Robert Bianchi***

The General Referee summarized the field of forensic sciences and submitted the following recommendation last year: Recommended that AOAC INTERNATIONAL no longer pursue forensic sciences area of study.