

# JOURNAL OF AOAC INTERNATIONAL

A Peer-Reviewed Journal Publishing Basic and Applied Research in the Analytical Sciences Related to Foods, Drugs, Agriculture, and the Environment

## EDITORIAL BOARD

Jim Ault, *Chair*  
Rodney Bushway  
Steven Dentali  
John Finley  
Larry Fosdick  
Gabriel Giancaspro  
Anthony Hitchins  
Milan Ilnat  
Jeanne I. Rader  
David L. Soderberg  
Hans P. van Egmond  
Edward Waysek  
Luann Wetzler  
Krystyna McIver, *Staff Liaison*

## JOURNAL EDITORS

**Agricultural Materials**  
W. Emmett Braselton

**Dietary Supplements**  
Albert E. Pohland

**Drug Formulations and Clinical Methods**  
Samir Wahab

**Food Biological Contaminants**  
Anthony D. Hitchins

**Food Chemical Contaminants**  
Albert E. Pohland

**Food Composition and Additives**  
Sarwar Gilani

**Microbiological Methods**  
Anthony Hitchins

**Residues and Trace Elements**  
Alexander J. Kryniak

**Statistical Analysis**  
George W. Latimer, Jr

**Veterinary Drug Residues**  
James D. MacNeil

**JOURNAL STAFF**

**Publisher:** E. James Bradford  
**Sr Director, Publications:** Brian K. Theil, CAE  
**Managing Editor:** Robert E. Rathbone  
**Production Manager:** Jennifer J. Diaz  
**Production Editor:** Tien L. Milor  
**Subscriptions:** Chris Bradley

## Advertising Sales:

AOAC INTERNATIONAL  
481 N. Frederick Ave, Suite 500  
Gaithersburg, MD 20877, USA  
Tel: +1-301-924-7077 • Fax: +1-301-924-7089  
E-mail: cbradley@aoac.org

## AOAC BOARD OF DIRECTORS

**President:** Robert E. Koertzer  
**President-Elect:** Samuel W. Page  
**Treasurer:** Jonathan W. DeVries  
**Secretary:** Darryl M. Sullivan  
**Immediate Past President:** Maire C. Walsh  
**Directors:** Michael H. Brodsky  
Russell S. Flowers  
James D. MacNeil  
Lee L. Pieta  
Frank P. Ross  
Barry Titlow  
**Directors-at-Large:** Douglas A. Hite  
Ronald L. Johnson

# Table of Contents

January/February 2007 VOL. 90, No. 1

JAINEE 90(1) 1-348 (2007)

8A	<b>Journal Information</b>
9A	<b>For Your Information</b>
15A	<b>A Field Guide to Instrumentation</b>
22A	<b>Cumulative Author Index</b>

## KEYNOTE ADDRESS

- 1 Food Safety Controls in Europe—Challenges and Opportunities  
*Colm Gaynor*

## AGRICULTURAL MATERIALS

- 6 Repeatability and Reproducibility of Determination of the Nitrogen Content of Fishmeal by the Combustion (Dumas) Method and Comparison with the Kjeldahl Method: Interlaboratory Study  
*Eric L. Miller, Anthony P. Bimbo, Stuart M. Barlow, and Berni Sheridan*

## DIETARY SUPPLEMENTS

- 21 Quantitative Estimation of Parthenolide in *Tanacetum parthenium* (L.) Schultz-Bip. Cultivated in Egypt  
*Ali M. El-Shamy, Seham S. El-Hawary, and Mostafa E.M. Rateb*
- 28 Determination of the Anthraquinones Aloe-Emodin and Aloin-A by Liquid Chromatography with Mass Spectrometric and Diode Array Detection  
*Mahmoud A. ElSohly, Waseem Gul, Bharathi Avula, and Ikhlas A. Khan*
- 43 Evaluation of a Method to Determine Flavonol Aglycones in *Ginkgo biloba* Dietary Supplement Crude Materials and Finished Products by High-Performance Liquid Chromatography: Collaborative Study  
*Dean Gray, Kerri LeVanseler, Meide Pan, and Edward H. Waysek*
- 54 A Novel Multielemental Scanning Thermal Analysis (MESTA) Method for the Identification and Characterization of Solid Substances  
*Yuch-Ping Hsieh*
- 60 Gas Chromatographic/Mass Spectrometric Analysis of the Essential Oil of *Houttuynia cordata* Thunb by Using On-Column Methylation with Tetramethylammonium Acetate  
*Muhammad Ishtiaq Ch, Yang F. Wen, and YiYu Cheng*

**Copyright**, 2007, by AOAC INTERNATIONAL, Inc. All rights reserved. The *Journal of AOAC INTERNATIONAL* (ISSN 1060-3271) is published bimonthly by AOAC INTERNATIONAL, 481 N. Frederick Ave, Suite 500, Gaithersburg, MD 20877, USA. **Subscriptions** for Vol. 90 (2007) are: Members, \$340.00 in North America, \$430.00 outside North America; Nonmembers, \$475.00 in North America, \$565.00 outside North America. Periodicals postage paid at Gaithersburg, Maryland, USA, and additional mailing offices. Abstracted or indexed in AGRICOLA, Analytical Abstracts, Biological Abstracts, Cambridge Scientific Abstracts, Chemical Abstracts, Current Advances in Ecological Sciences, Current Contents, and others. Printed in USA.

**POSTMASTER:** Send address changes to *Journal of AOAC INTERNATIONAL*, 481 N. Frederick Ave, Suite 500, Gaithersburg, MD 20877, USA.

- 68 Determination of Synephrine in Bitter Orange Raw Materials, Extracts, and Dietary Supplements by Liquid Chromatography with Ultraviolet Detection: Single-Laboratory Validation  
*Mark C. Roman, Joseph M. Betz, and Jana Hildreth*

#### DRUG FORMULATIONS AND CLINICAL METHODS

---

- 82 Simultaneous Determination of Paracetamol, Chlorzoxazone, and Related Impurities 4-Aminophenol, 4'-Chloroacetanilide, and *p*-Chlorophenol in Pharmaceutical Preparations by High-Performance Liquid Chromatography  
*Mohammed Shahid Ali, Syed Rafiuddin, Mohsin Ghori, and Aamer Roshanali Kahtri*
- 94 Determination of the Analgesic Components of *Spasmomigraine*<sup>®</sup> Tablet by Liquid Chromatography with Ultraviolet Detection  
*Fawzy A. Elbarbry, Mokhtar M. Mabrouk, and Mohamed A. El-Dawy*
- 102 Simultaneous Determination of Hyoscine Butylbromide and Ketoprofen in Pharmaceutical Preparations by Spectrophotometric and Liquid Chromatographic Methods  
*Yasser Shaker El-Saharty, Fadia H. Metwally, Mohamed Refaat, and Sonia Zaki El-Khateeb*
- 113 Development and Validation of Three Stability-Indicating Methods for Determination of Bisacodyl in Pure Form and Pharmaceutical Preparations  
*Fadia H. Metwally, Mohammed Abdelkawy, and Ibrahim A. Naguib*
- 128 Determination of Two Antibacterial Binary Mixtures by Chemometrics-Assisted Spectrophotometry  
*Abd El-Maaboud I. Mohamed, Osama H. Abdelmageed, and Ibrahim H. Refaat*
- 142 Simultaneous Estimation of Pantoprazole and Domperidone in Pure Powder and a Pharmaceutical Formulation by High-Performance Liquid Chromatography and High-Performance Thin-Layer Chromatography Methods  
*Bhavesh H. Patel, Bhanubhai N. Suhagia, Madhabhai M. Patel, and Jignesh R. Patel*
- 147 Phosphorated Calix[6]arene Derivatives as an Ionophore for Atropine-Selective Membrane Electrodes  
*Mohsen M. Zareh and Elizabeita Malinowska*

#### FOOD CHEMICAL CONTAMINANTS

---

- 153 Development of Extraction and Gas Chromatography Analytical Methodology for Cyanogenic Glycosides in Flaxseed (*Linum usitatissimum*)  
*Ray Bacala and Véronique Barthet*
- 162 Determination of Patulin in Apple Juice: Comparative Evaluation of Four Analytical Methods  
*David R. Katerere, Sonja Stockenström, Gabriele Balducci, and Gordon S. Shephard*
- 167 Determination of Patulin in Apple and Hawthorn Beverages by Solid-Phase Filtration Column and Liquid Chromatography  
*Fengqin Li, Shan Zhao, Leejiuan Chin, Yuwei Li, Duoqia Wu, Xi Zhao, Chunhui Han, Hongyuan Zhang, and Rong Ji*

- 173 An Electrochemiluminescence-Based Competitive Displacement Immunoassay for the Type-2 Brevetoxins in Oyster Extracts  
*Mark A. Poli, Victor R. Rivera, Dwayne D. Neal, Daniel G. Baden, Shawn A. Messer, Steven M. Plakas, Robert W. Dickey, Kathleen El Said, Leanne Flewelling, David Green, and Jill White*

#### FOOD COMPOSITION AND ADDITIVES

---

- 179 Analysis of Mitochondrial DNA for Authentication of Meats from Chamois (*Rupicapra rupicapra*), Pyrenean Ibex (*Capra pyrenaica*), and Mouflon (*Ovis ammon*) by Polymerase Chain Reaction-Restriction Fragment Length Polymorphism  
*Violeta Fajardo, Isabel González, Inés López-Calleja, Irene Martín, María Rojas, Miguel Ángel Pavón, Pablo E. Hernández, Teresa García, and Rosario Martín*
- 187 Determination of Site-Specific (Deuterium/Hydrogen) Ratios in Vanillin by <sup>2</sup>H-Nuclear Magnetic Resonance Spectrometry: Collaborative Study  
*Eric Jamin, Frédérique Martin, and Gilles G. Martin*
- 196 Determination of the Lactose Content of Fluid Milk by Spectrophotometric Enzymatic Analysis Using Weight Additions and Path Length Adjustment: Collaborative Study  
*Joanna M. Lynch, David M. Barbano, and J. Richard Fleming*
- 217 A Simplified Modification of the AOAC Official Method for Determination of Total Dietary Fiber Using Newly Developed Enzymes  
*Shusaku Tada and Satoshi Inami*
- 225 A Simplified Modification of the AOAC Official Method for Determination of Total Dietary Fiber Using Newly Developed Enzymes: Preliminary Interlaboratory Study  
*Kenichiro Kanaya, Shusaku Tada, Bunpei Mori, Rie Takahashi, Sachie Ikegami, Shin'ichi Kurasawa, Masami Okuzaki, Yoko Mori, and Satoshi Inami*
- 238 A Novel Method for Determination of Protein in Human Serum Utilizing the Trihydroxyphenylfluorone-Molybdenum(VI) Complex as a Probe by Fluorescence Spectrometry  
*Qin Wei, Hongmin Ma, Caihong Duan, Jin Wang, Shuyuan Liu, and Bin Du*
- 244 Optimized Determination Method for *trans*-10-Hydroxy-2-Decenoic Acid Content in Royal Jelly by High-Performance Liquid Chromatography with an Internal Standard  
*Jinhui Zhou, Xiaofeng Xue, Yi Li, Jinzhen Zhang, and Jing Zhao*

#### MICROBIOLOGICAL METHODS

---

- 250 Quantitative Recovery of *Listeria monocytogenes* and Select *Salmonella* Serotypes from Environmental Sample Media  
*Michael C. Bazaco, Joseph D. Eifert, Robert C. Williams, and Sophia Kathariou*

#### RESIDUES AND TRACE ELEMENTS

---

- 258 Direct Inductively Coupled Plasma-Atomic Emission Spectrometry Analysis of Trace Elements in Muscle Tissues of Hairtail Using Electrothermal Vaporization with Slurry Sampling  
*Shizhong Chen, Dengbo Lu, and Zhixiong Hu*
- 263 Pesticide Residue Analysis of Vegetables by Gas Chromatography with Electron-Capture Detection  
*José Fenoll, Pilar Hellin, Carmen M. Martínez, and Pilar Flores*

- 271 Determination of Organophosphorus Pesticides in *Lycium barbarum* by Gas Chromatography with Flame Photometric Detection  
*Li Li, Fengmao Liu, Chuanfan Qian, Shuren Jiang, Zhiqiang Zhou, and Canping Pan*
- 277 Determination of Butylated Hydroxytoluene in Food Samples by High-Performance Liquid Chromatography with Ultraviolet Detection and Gas Chromatography/Mass Spectrometry  
*Ana Sanches-Silva, José M. Cruz, Raquel Sendón-García, and Perfecto Paseiro-Losada*
- 284 Determination of Inorganic Arsenic(III) and Arsenic(V) in Water Samples by Ion Chromatography/Inductively Coupled Plasma-Mass Spectrometry  
*Yiu-Chung Yip, Hei-Shing Chu, Chung-Fai Yuen, and Wing-Cheong Sham*

#### **STATISTICAL ANALYSIS**

---

- 291 Computational Models for Structure-Hydrophobicity Relationships of 4-Carboxyl-2,6-Dinitrophenyl Azo Hydroxynaphthalenes  
*Olakunle S. Idowu, Olajire A. Adegoke, Abiola Idowu, and Ajibola A. Olaniyi*

#### **TECHNICAL COMMUNICATIONS**

---

- 299 Standard Practice for Bulk Sample Collection and Swab Sample Collection of Visible Powders Suspected of Being Biological Agents from Nonporous Surfaces: Collaborative Study  
*Laurie E. Locascio, Bruce Harper, and Matthew Robinson*

#### **VETERINARY DRUG RESIDUES**

---

- 334 Stable Competitive Enzyme-Linked Immunosorbent Assay Kit for Rapid Measurement of 11 Active Beta-Lactams in Milk, Tissue, Urine, and Serum  
*Stephen P. FitzGerald, Nuala O'Loan, Robert I. McConnell, El Ouard Benchikh, and Nicola E. Kane*
- 343 Simultaneous Determination of 17 Sulfonamides and the Potentiators Ormetoprim and Trimethoprim in Salmon Muscle by Liquid Chromatography with Tandem Mass Spectrometry Detection  
*Ross A. Potter, B. Garth Burns, Jeffrey M. van de Riet, David H. North, and Rozina Darvesh*

#### **GENERAL REFERENCE REPORTS—AVAILABLE ONLINE ONLY**

---

Available online only: [http://www.aoac.org/pubs/JOURNAL/committee\\_rpts.htm](http://www.aoac.org/pubs/JOURNAL/committee_rpts.htm) or  
<http://www.atypon-link.com/AOAC/loi/jaoi>

##### **Committee on Natural Toxins and Food Allergens**

Marine and Freshwater Toxins  
Mycotoxins

##### **Committee on Food Nutrition**

Fat-Soluble Vitamins  
Water-Soluble Vitamins

##### **Committee on Residues and Related Topics**

Pesticides and Other Chemical Contaminants

##### **Committee on Microbiology and Extraneous Materials**

Food Microbiology, Nondairy

**METHODS COMMITTEE REPORTS—AVAILABLE ONLINE ONLY**

---

Available online only: [http://www.aoac.org/pubs/JOURNAL/committee\\_rpts.htm](http://www.aoac.org/pubs/JOURNAL/committee_rpts.htm) or  
<http://www.atypon-link.com/AOAC/loi/jaoi>

- Committee on Pesticides and Disinfectant Formulations
- Committee on Drugs and Related Topics
- Committee on Additives, Beverages, and Food Process-Related Analytes
- Committee on Natural Toxins and Food Allergens
- Committee on Food Nutrition
- Committee on Commodity Foods and Commodity Products
- Committee on Residues and Related Topics
- Committee on Microbiology and Extraneous Materials
- Committee on Feeds, Fertilizers, and Related Agricultural Topics
- Committee on Environmental Quality
- Committee on Dietary Supplements

**TECHNICAL COMMITTEE REPORTS—AVAILABLE ONLINE ONLY**

---

Available online only: [http://www.aoac.org/pubs/JOURNAL/committee\\_rpts.htm](http://www.aoac.org/pubs/JOURNAL/committee_rpts.htm) or  
<http://www.atypon-link.com/AOAC/loi/jaoi>

- Safety Committee
- Statistics Committee