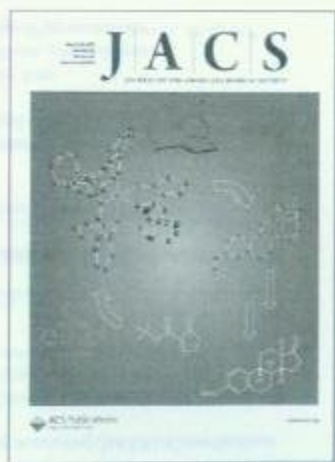




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ANALYTICAL METHODS

- 5213 **Validation of Selective Ion Flow Tube Mass Spectrometry for Fast Quantification of Volatile Bases Produced on Atlantic Cod (*Gadus morhua*)**
*Bert Noseda, Peter Ragaert, Danny Pauwels, Tom Anthierens, Herman Van Langenhove, Jo Dewulf, and Frank Devlieghere**
- 5220 **A Continuous, Quantitative Fluorescent Assay for Plant Caffeic Acid *O*-Methyltransferases**
*Nathan A. Palmer, Scott E. Sattler, Aaron J. Saathoff, and Gautam Sarath**
- 5227 **Stereospecific Analysis of Triacylglycerols as a Useful Means To Evaluate Genuineness of Pumpkin Seed Oils: Lesson from Virgin Olive Oil Analyses**
*Bojan Butinar, Milena Bučar-Miklavčič, Vasilij Valenčič, and Peter Razpor**
- 5235 **Development of a Cost-Effective Method for Nitrate and Nitrite Determination in Leafy Plants and Nitrate and Nitrite Contents of Some Green Leafy Vegetables Grown in the Aegean Region of Turkey**
Özgül Özdecan and Ali Üren*
- 5241 **Monoclonal Antibody-Based Enzyme-Linked Immunosorbent Assays for the Organophosphorus Insecticide *O*-Ethyl *O*-4-Nitrophenyl Phenylphosphonothioate (EPN)**
*Jee Youn Shim, Young Ah Kim, Yong Tae Lee, Bruce D. Hammock, and Hye-Sung Lee**
- 5248 **Multiresidue Method for Pesticides and Persistent Organic Pollutants (POPs) in Milk and Cream Using Comprehensive Two-Dimensional Capillary Gas Chromatography–Time-of-Flight Mass Spectrometry**
Douglas G. Hayward, Tamami S. Pisano, Jon W. Wong, and Richard J. Scudder*
- 5257 **Interaction of Paraquat with Calf Thymus DNA: A Terbium(III) Luminescent Probe and Multispectral Study**
Changlun Tong, Guanghong Xiang, and Yu Bai*
- 5263 **High-Performance Liquid Chromatography–Inductively Coupled Plasma Mass Spectrometry Based Method for the Determination of Organic Arsenic Feed Additives and Speciation of Anionic Arsenics in Animal Feed**
*Peilong Wang, Genlong Zhao, Jing Tian, and Xiaosu Su**
- 5271 **Raman Analysis of Caraway (*Carum carvi* L.) Single Fruits. Evaluation of Essential Oil Content and Its Composition**
K. Seidler-Lozykowska, M. Baranska*, R. Baranski, and D. Krol*

- 5276 **Assessment of the Quality Attributes of Cod Caviar Paste by Means of Front-Face Fluorescence Spectroscopy**
Diego Airado-Rodriguez, Josefina Skaret, and Jens Petter Wold*
- 5286 **Leather Structure Determination by Small-Angle X-ray Scattering (SAXS): Cross Sections of Ovine and Bovine Leather**
*Melissa M. Basil-Jones, Richard L. Edmonds, Timothy F. Allsop, Sue M. Cooper, Geoff Holmes, Gillian E. Norris, David J. Cookson, Nigel Kirby, and Richard G. Haverkamp**

BIOACTIVE CONSTITUENTS

- 5292 **Fatty Acid Hydroxytyrosyl Esters: Structure/Antioxidant Activity Relationship by ABTS and in Cell-Culture DCF Assays**
Daniela Tofani, Valentina Balducci, Tecla Gasperi, Sandra Incerpi, and Augusto Gambacorta*
- 5300 **Diffusion of Active Proteins into Fish Meat To Minimize Proteolytic Degradation**
Patricio A. Carvajal-Rondanelli and Tyre C. Lanier*
- 5308 **An Onion Byproduct Affects Plasma Lipids in Healthy Rats**
*Eduvigis Roldán-Marín, Runa I. Jensen, Britta N. Krath, Mette Kristensen, Morten Poulsen, M. Pilar Cano, Concepción Sánchez-Moreno, and Lars O. Dragsted**
- 5315 **Group B Oleanane Triterpenoid Extract Containing Soyasaponins I and III from Soy Flour Induces Apoptosis in Hep-G2 Cells**
*Wei Zhang and David G. Popovich**
- 5320 **Gut Metabolites of Anthocyanins, Gallic Acid, 3-O-Methylgallic Acid, and 2,4,6-Trihydroxybenzaldehyde, Inhibit Cell Proliferation of Caco-2 Cells**
*Sarah C. Forester and Andrew L. Waterhouse**
- 5328 **Effect of Micronutrient-Enriched Sunflower Oils on Plasma Lipid Profile and Antioxidant Status in High-Fat-Fed Rats**
*R. Di Benedetto, L. Attorri, F. Chiarotti, A. Eusepi, A. Di Biase, and S. Salvati**
- 5334 **Consumption of Human Milk Oligosaccharides by Gut-Related Microbes**
Angela Marcolal, Mariana Barboza, John W. Froehlich, David E. Block, J. Bruce German, Carlito B. Lebrilla, and David A. Mills**
- 5341 **Bioactive Compounds in Cashew Nut (*Anacardium occidentale* L.) Kernels: Effect of Different Shelling Methods**
*Jennifer Trox, Veilingiri Vadivel, Walter Vetter, Wolfgang Stuetz, Veronika Scherbaum, Ute Gola, Donatus Nohr, and Hans Konrad Biesalski**
- 5347 **Profiles of Betacyanins in Epidermal Layers of Grafted and Light-Stressed Cacti Studied by LC-DAD-ESI-MS/MS**
Slawomir Wybraniec, Pawel Stalca, Aneta Sporna, and Yosef Mizrahi*
- 5355 **Inhibitory Effect of Gallic Acid and Its Esters on 2,2'-Azobis(2-amidinopropane)hydrochloride (AAPH)-Induced Hemolysis and Depletion of Intracellular Glutathione in Erythrocytes**
Valdecir F. Ximenes, Mariana G. Lopes, Maicon Segalla Petrônio, Luis Octavio Regasini, Dulce H. Siqueira Silva, and Luiz M. da Fonseca*
- 5363 **Flavonoids and Phenolic Compounds from *Rosmarinus officinalis***
Naisheng Bai, Kan He, Marc Roller, Ching-Shu Lai, Xi Shao, Min-Hsiung Pan, and Chi-Tung Ho*

- 5368 **Tyrosinase Inhibitory Constituents from the Roots of *Morus nigra*: A Structure-Activity Relationship Study**
*Zong-Ping Zheng, Ka-Wing Cheng, Qin Zhu, Xia-Chang Wang, Zhi-Xiu Lin, and Mingfu Wang**
- 5374 **Bioaccessibility, Uptake, and Transport of Carotenoids from Peppers (*Capsicum* Spp.) Using the Coupled *In Vitro* Digestion and Human Intestinal Caco-2 Cell Model**
*Laurie O'Sullivan, Marvin A. Jiwan, Trevor Daly, Nora M. O'Brien, and S. Aisling Aherne**

CHEMICAL ASPECTS OF BIOTECHNOLOGY/MOLECULAR BIOLOGY

- 5380 **Organophosphorus Hydrolase (OpdB) of *Lactobacillus brevis* WCP902 from Kimchi Is Able To Degrade Organophosphorus Pesticides**
*Shah Md. Asraful Islam, Renukaradhya K. Math, Kye Man Cho, Woo Jin Lim, Su Young Hong, Jong Min Kim, Myoung Geun Yun, Ji Joong Cho, and Han Dae Yun**
- 5387 **Selection of a Single Chain Variable Fragment Antibody against Ivermectin from a Phage Displayed Library**
Shuang Wen, Xiao Zhang, Yuan Liu, Qingqing Zhang, Xianjin Liu, and Jiunsheng Liang**
- 5392 **Construction of Chromosomally Located T7 Expression System for Production of Heterologous Secreted Proteins in *Bacillus subtilis***
Po Ting Chen, Jei-Fu Shau, Yun-Peng Chao, Yuan-Hua David Ho, and Su-May Yu**

BIOFUELS AND BIOPRODUCTS CHEMISTRY

- 5400 **Mechanical, Thermal, and Interfacial Properties of Green Composites with Ramie Fiber and Soy Resins**
*Jun Tae Kim and Anil N. Netravali**

CHEMICAL ASPECTS OF FOOD SAFETY

- 5408 **Distribution of Penicillin G Residues in Culled Dairy Cow Muscles: Implications for Residue Monitoring**
Marilyn J. Schneider, Katerina Mastovska, and Morse B. Solomon*
- 5414 **Tissue Deposition and Residue Depletion in Laying Hens Exposed to Melamine-Contaminated Diets**
Xue Bai, Fan Bai, Keying Zhang,* Xiaowen Lv, Yuchang Qin, Yun Li, Shiping Bai, and Shunquan Lin*

- 5421 **Inhibition of Biological Activity of Staphylococcal Enterotoxin A (SEA) by Apple Juice and Apple Polyphenols**
Reaven Rasooly, Paula M. Do, and Mendel Friedman*

CHEMICAL CHANGES INDUCED BY PROCESSING/STORAGE

- 5427 **Effect of Heating on the Stability of Quinolones in Milk**
*M. Roca, M. Castillo, P. Martí, R. L. Althaus, and M. P. Molina**
- 5432 **Effects of Storage Condition Factors on Fungal Invasion of *Radix Ophiopogonis***
*Hui Wang, Jin Qi, Tian Xu, Ji-Hua Liu, Min-Jian Qin, Dan-Ni Zhu, and Bo-Yang Yu**

- 5438 **Selective Removal of Monoterpenes from Bergamot Oil by Inclusion in Deoxycholic Acid**
Giancarlo Fanini, Marco Fogagnolo, Silvia Maletti, and Stefano Rosselli*
- 5444 **Crystallization Control of Thermal Stability and Morphology of Hen Egg White Lysozyme Crystals by Ionic Liquids**
*Zhanzhong Wang, Leping Dang, Ye Han, Pingping Jiang, and Hongyuan Wei**
- 5449 **A Pectin-Methylesterase-Inhibitor-Based Molecular Probe for *In Situ* Detection of Plant Pectin Methylesterase Activity**
*Ruben P. Jolie, Thomas Duvetter, Evelien Vandevenne, Sandy Van Buggenhout, Ann M. Van Loey, and Marc E. Hendrickx**
- 5457 **Functional Properties of Select Edible Oilseed Proteins**
*Girdhari M. Sharma, Mengna Su, Aditya U. Joshi, Kenneth H. Roux, and Shridhar K. Sathe**
- 5465 **Formation of Volatile Chemicals from Thermal Degradation of Less Volatile Coffee Components: Quinic Acid, Caffeic Acid, and Chlorogenic Acid**
*Joon-Kwan Moon and Takayuki Shibamoto**

CHEMICAL COMPOSITION OF FOODS/FEEDS

- 5471 **Profiling and Characterization by LC-MS² of the Chlorogenic Acids and Hydroxycinnamoylshikimate Esters in Maté (*Ilex paraguariensis*)**
*Rakesh Jaiswal, Tina Sovdat, Francesco Vivan, and Nikolai Kuhnert**
- 5485 **Variation in Content and Composition of Phenolic Compounds in Permanent Pastures According to Botanical Variation**
*Aurélie Reynaud, Didier Fraisse, Agnes Cornu, Anne Farruggia, Estelle Pujos-Guillot, Jean-Michel Besle, Bruno Martin, Jean-Louis Lamaison, Denis Paquet, Michel Doreau, and Benoît Graulet**
- 5495 **Soybean Tocopherol Concentrations Are Affected by Crop Management**
Philippe Seguin, Gilles Tremblay, Denis Pageau, and Wucheng Liu*
- 5502 **α -Solasonine and α -Solamargine Contents of Gboma (*Solanum macrocarpon* L.) and Scarlet (*Solanum arthiopicum* L.) Eggplants**
Maria-Cortez Sánchez-Mata, Wallace E. Yokoyama, Yun-Jeong Hong, and Jaime Prohens*

CROP AND ANIMAL PROTECTION CHEMISTRY

- 5509 **New Resistance-Related Saponins from the Insect-Resistant Crucifer *Barbarea vulgaris***
*Nikoline J. Nielsen, John Nielsen, and Dan Staerk**
- 5515 **Synthesis and Biological Activity of Some Novel Trifluoromethyl-Substituted 1,2,4-Triazole and Bis(1,2,4-Triazole) Mannich Bases Containing Piperazine Rings**
Bao-Lei Wang, Yan-Xia Shi, Yi Ma, Xing-Hai Liu, Yong-Hong Li, Hai-Bin Song, Bao-Ju Li, and Zheng-Ming Li**

ENVIRONMENTAL CHEMISTRY

- 5523 **Synthesis, Characterization, and Sorption Capacity of Layered Double Hydroxides and Their Complexes with Polymerin**
Marianna Iorio, Antonio De Martino, Antonio Violante, Massimo Pigna, and Renato Capasso*

- 5531 **Effects of Pesticides on the Bacterial Production of Pyrrolinitrin**
*Yuang Soo Keum, Hye Ri Lee, and Jeong-Han Kim**
- 5538 **Immobilization of Heavy Metal Ions (Cu^{II}, Cd^{II}, Ni^{II}, and Pb^{II}) by Broiler Litter-Derived Biochars in Water and Soil**
Minori Uchimiya, Isabel M. Lima, K. Thomas Klasson, Se-Chin Chang, Lynda H. Wartelle, and James E. Rodgers*
- 5545 **Purification and Characterization of a Peroxidase Isozyme from Indian Turnip Roots**
*Sohel Dalal and Munishwar Nath Gupta**
- 5553 **Hydrolysis and Speciation of Al Bound to Pectin and Plant Cell Wall Material and Its Reaction with the Dye Chrome Azurol S**
J. Bernhard Wehr, F. P. C. Blaney, J. V. Hama, P. M. Kopttke, G. L. Kerren, and N. W. Menzies*
- 5561 **Impact of Carbon Chain Length on Binding of Perfluoroalkyl Acids to Bovine Serum Albumin Determined by Spectroscopic Methods**
Pengfei Qin, Rutan Liu, Xingren Pan, Xiaoyan Fang, and Yue Mou*

FLAVORS AND AROMAS/CHEMOSENSORY PERCEPTION

- 5568 **Study on the Relevance Between Beany Flavor and Main Bioactive Components in Radix Astragalii**
*Hailfeng Sun, Daosheng Xie, Xiaoping Guo, Lizeng Zhang, Zhenyu Li, Bin Wu, and Xuenen Qin**
- 5574 **Effects of the Nonvolatile Matrix on the Aroma Perception of Wine**
*Maria-Pilar Sáenz-Navajas, Eva Campo, Laura Culleré, Purificación Fernández-Zurbano, Dominique Valentin, and Vicente Ferreira**

FOOD CHEMISTRY/BIOCHEMISTRY

- 5586 **Virgin Olive Oil Authentication by Multivariate Analyses of ¹H NMR Fingerprints and δ^{13} C and δ^2 H Data**
Rosa M. Alonso-Salces, José M. Moreno-Rojas, Margaret V. Holland, Fabiano Reniero, Claude Guillou, and Károly Héberger*
- 5597 **Coffee and Caffeine Ameliorate Hyperglycemia, Fatty Liver, and Inflammatory Adipocytokine Expression in Spontaneously Diabetic KK-^A Mice**
*Rie Yamazaki, Misato Kobayashi, Yuji Matsuda, Makoto Ojika, Shigeru Shigeoka, Yuko Yamamoto, Yoshie Tou, Takashi Inoue, Takao Katagiri, Atsushi Murai, and Fumihiko Horio**
- 5604 **6-Dehydrogingerolone Sensitizes Human Hepatoblastoma Hep G2 Cells to TRAIL-Induced Apoptosis via Reactive Oxygen Species-Mediated Increase of DR5**
Chung-Yi Chen, Cheng-Jeng Tai, Jiu-Tsuey Cheng, Juan-Juan Zheng, Ying-Zong Chen, Tsan-Zun Liu, Shuenn-Jiun Yün, and Chi-Liang Chern**
- 5612 **Characterization of *Lactobacillus reuteri* Interaction with Milk Fat Globule Membrane Components in Dairy Products**
*Guillaume Brisson, Hannah F. Payken, John P. Sharpe, and Rafael Jiménez-Flores**
- 5620 **Interaction between Amylose and β -Cyclodextrin Investigated by Complexing with Conjugated Linoleic Acid**
*Ying Yang, Zhengbiao Gu, Hui Xu, Fengwei Li, and Genyi Zhang**

- 5625 **Value-Added Processing of Peanut Meal: Aflatoxin Sequestration during Protein Extraction**
Lauren E. Seifert, Jack P. Davis, Joe W. Dorner, William F. Jaynes, Richard E. Zarman, and Timothy H. Sanders*
- 5633 **Variations in Starch Physicochemical Properties from a Generation-Means Analysis Study Using Amylomaize V and VII Parents**
Hongxin Jiang, Jay-lin Jane, Diana Acevedo, Andrew Green, George Shinn, Denyse Schrenker, Sathaporn Srichuwong, Mark Campbell, and Yusheng Wu*
- 5640 **Egg White Ovalbumin Digestion Mimicking Physiological Conditions**
*Gustavo Martos, Patricia Contreras, Elena Molina, and Rosina López-Fundición**
- 5649 **Isolation, Expression, and Characterization of a 13-Hydroperoxide Lyase Gene from Olive Fruit Related to the Biosynthesis of the Main Virgin Olive Oil Aroma Compounds**
María N. Padilla, M. Luisa Hernández, Ana G. Pérez, Carlos Sanz, and José M. Martínez-Rivas*
- 5658 **Development of a Mab-Based Heterologous Immunoassay for the Broad-Selective Determination of Organophosphorus Pesticides**
*Chunmei Wang, Xianbin Li, Yihua Liu, Yirong Guo, Rong Xie, Wenjin Gu, and Guonian Zhu**
- 5664 **Isolation and Structural Characterization of Anthocyanin-furfuryl Pigments**
*André Sousa, Nuno Mateus, Artur Manuel Soares Silva, Nicolas Vivas, Marie-Françoise Nonier, Isabelle Pianet, and Victor de Freitas**
- 5670 **Superior Anticarcinogenic Activity of *trans,trans*-Conjugated Linoleic Acid in *N*-Methyl-*N*-nitrosourea-Induced Rat Mammary Tumorigenesis**
*Mohammad A. Islam, Young S. Kim, Tae W. Oh, Gon S. Kim, Chung K. Won, Hoon G. Kim, Myung S. Choi, Jeong O. Kim, and Yeong L. Ha**
- 5679 **Effects of Chitosan and Rosmarinate Esters on the Physical and Oxidative Stability of Liposomes**
*Aitkorn Panya, Mickael Laguerre, Jerome Lecante, Pierre Villeneuve, Joehen Weiss, D. Julian McClements, and Eric A. Decker**
- 5685 **Oxygen-Dependent Fragmentation Reactions during the Degradation of 1-Deoxy-*D*-erythro-hexo-2,3-dulose**
*Michael Voigt, Mareen Smuda, Christoph Pfahler, and Marcus A. Glomb**
- 5692 **Directing the Oligomer Size Distribution of Peroxidase-Mediated Cross-Linked Bovine α -Lactalbumin**
*Walter H. Hejnis, Peter A. Wierenga, Willem J. H. van Berkel, and Harry Gruppen**
- 5696 **Differences in the Triticale (X Triticosecale Wittmack) Flag Leaf 2-DE Protein Profile between Varieties and Nitrogen Fertilization Levels**
Mari Ángeles Castillejo, Hristofor K. Kirchev, and Jesus V. Jorrián*
- 5708 **Overexpression of a Cell Wall Enzyme Reduces Xyloglucan Depolymerization and Softening of Transgenic Tomato Fruits**
Eva Miedes, Karin Herbers, Uwe Sonnwald, and Ester P. Laurences*
- 5714 **Purification and Biochemical Characterization of Brazil Nut (*Bertholletia excelsa* L.) Seed Storage Proteins**
*Girdhari M. Sharma, Claudius Mundoma, Margaret Seavy, Kenneth H. Roux, and Shridhar K. Sathu**
- 5724 **Effects of Lactate on Bovine Heart Mitochondria-Mediated Metmyoglobin Reduction**
Ranjith Ramanathan, Richard A. Mancini, and Naveena B. Maheswarappa*
- 5730 **Purification and Characterization of a Gelatinolytic Metalloproteinase from the Skeletal Muscle of Red Sea Bream (*Pagrus major*)**
*Guo-Ping Wu, Min-Jie Cao, Su-Hua Chen, Wu-Yin Weng, Qiu-Feng Cai, and Wen-Jin Su**
- 5737 **Bioproperties of Potent Nattokinase from *Bacillus subtilis* YJ1**
*Li-Jung Yin, Hsin-Hung Lin, and Shann-Tzong Jiang**
- 5743 **Fractionation of Alkali-Solubilized Hemicelluloses from Delignified *Populus gansuensis*: Structure and Properties**
Feng Peng, Jun-Li Ren, Feng Xu, Jing Bian, Pai Peng, and Run-Cang Sun**
- 5751 **Changes in the Main Nutrients, Phytochemicals, and Antioxidant Activity in Yellow Corn Grain during Maturation**
Jian-Guo Xu, Qing-Ping Hu, Xiang-Dong Wang, Ji-Yang Luo, Yu Liu, and Cheng-Rui Tian*
- 5757 **Effect of the Complex of Zinc(II) and Cerium(IV) with Chitosan on the Preservation Quality and Degradation of Organophosphorus Pesticides in Chinese Jujube (*Zizyphus jujuba* Mill. cv. Dongzao)**
Hao Wu, Dongfeng Wang, John Shi,* Sophia Xue, and Meiling Gao*
- 5763 **Biochemical Properties of Anionic Trypsin Acting at High Concentration of NaCl Purified from the Intestine of a Carnivorous Fish: Smooth Hound (*Mustelus mustelus*)**
Ali Bougatef, Rafik Balti, Rim Nasri, Kemel Jelloail, Nabil Souissi, and Moneef Nasri*
- 5770 **Characterization of Metabolism and *in Vitro* Permeability Study of Notoginsenoside R1 from *Radix Notoginseng***
Jian-Qing Ruan, Weng-Im Leong, Ru Yan, and Yi-Tao Wang*
- 5777 **Inhibitory Effect of Magnolol on TPA-Induced Skin Inflammation and Tumor Promotion in Mice**
Daih-Huang Kuo, You-Syuan Lai, Chih-Yu Lo, An-Chin Cheng, Hou Wu, and Min-Hsiung Pan**
- 5784 **Carotenoids in Clams, *Ruditapes philippinarum* and *Meretrix petechialis***
Takashi Maoka, Naoshige Akimoto, Michiaki Murakoshi, Keikichi Sugiyama, and Hoyoku Nishino*
- 5789 **Digestive Stability and Transport of Norbixin, a 24-Carbon Carotenoid, across Monolayers of Caco-2 Cells**
*Karina Polar-Cabrera, Tianyao Huo, Steven J. Schwartz, and Mark L. Failla**
- 5795 **Chitosan Reduces Gluconeogenesis and Increases Glucose Uptake in Skeletal Muscle in Streptozotocin-Induced Diabetic Rats**
*Shing-Hwa Liu, Yu-Han Chang, and Meng-Tsan Chiang**
- 5801 **Effect of BTH on Anthocyanin Content and Activities of Related Enzymes in Strawberry after Harvest**
Shifeng Cao, Zhichao Hu, Yonghua Zheng, and Binhong Lu*

- 5806 **Polyphenol-Rich Extracts from *Solanum nigrum* Attenuated PKC α -Mediated Migration and Invasion of Hepatocellular Carcinoma Cells**
*Mon-Yuan Yang, Li-Sung Hsu, Chung-Huei Peng, Yuan-Shong Shi, Cheng-Hsun Wu, and Chau-Jung Wang**
- MOLECULAR NUTRITION
- 5815 **Protective Effect of Quercetin against Arsenite-Induced COX-2 Expression by Targeting PI3K in Rat Liver Epithelial Cells**
Kyung Mi Lee, Mun Kyung Hwang, Dong Eun Lee, Ki Won Lee, and Hyong Joo Lee**
- 5821 **Effect of Genistein and Daidzein on the Proliferation and Differentiation of Human Preadipocyte Cell Line**
Kaori Hirota, Keiko Morikawa, Haruka Hanada, Mitsuho Nomaka, Yuri Nakajima, Maiko Kobayashi, and Rie Nakajima*
- 5828 **Effects of Chlorogenic Acid and Bovine Serum Albumin on the Oxidative Stability of Low Density Lipoproteins *in Vitro***
Michael H. Gordon and Karl Wehbart*
- 5834 **Exopolysaccharide-Forming *Weissella* Strains as Starter Cultures for Sorghum and Wheat Sourdoughs**
Sandra Galle, Clarissa Schwab, Elke Arendt, and Michael Gänzle*
- 5842 **Feruloyl Dioleoylglycerol Antioxidant Capacity in Phospholipid Vesicles**
Joseph A. Luzzo, Kervin O. Evans, Karl E. Vermillion, and Michael Appell*
- 5851 **Frequent Cholesterol Intake Up-regulates Intestinal NPC1L1, ACAT2, and MTP**
*Rui Jiao, Lei Guan, Nan Yang, Cheng Peng, Yintong Liang, Ka Ying Ma, Yu Huang, and Zhen-Yu Chen**

ADDITIONS AND CORRECTIONS

- 5858 **Correction to Antihypertensive Effect of Peptide-Enriched Soy Sauce-Like Seasoning and Identification of Its Angiotensin I-Converting Enzyme Inhibitory Substances**
Takeharu Nakahara, Aisashi Sano, Hitomi Yamaguchi, Katsutoshi Sugimoto, Hiroyuki Chikata, Emiko Kinoshita, and Riehiro Uchida*
- 5858 **Correction to Calculating Radiation Exposures during Use of ^{14}C -Labeled Nutrients, Food Components, and Biopharmaceuticals To Quantify Metabolic Behavior in Humans**
*Seung-Hyun Kim Peter B. Kelly and Andrew J. Clifford**

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Validation of Selective Ion Flow Tube Mass Spectrometry for Fast Quantification of Volatile Bases Produced on Atlantic Cod (*Gadus morhua*)

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Selective ion flow tube mass spectrometry (SIFT-MS) is a direct mass spectrometric technique that allows qualitative and quantitative analysis of a large number of volatile organic compounds. Because of its speed and ease of use, this nondestructive technique could be considered as a practical tool for quality control. This research focuses on the possibilities of direct headspace sampling by SIFT-MS for the quantification of the volatile basic nitrogen content (TVB-N) of fish filets. These volatile bases [trimethylamine (TMA), dimethylamine (DMA), and ammonia] give additional information in conjunction with the sensory scoring and microbiological analysis about the quality of the fish filets. This research validates in a first part the SIFT-MS method for the quantification of the volatile bases in mixed cod samples. With regard to the investigated linearity, repeatability, reproducibility, recovery, limit of detection, and limit of quantification, SIFT-MS appeared to be an adequate technique for measuring volatile bases spiked on cod. In the second part of this research, the technique was validated for the analysis of volatile bases on cod filets during a storage experiment under ice. A good correlation was obtained between the proposed direct headspace sampling and traditional methods. The sensitivity of the SIFT-MS method can be improved when cod filets are made more alkaline (pH > 11) during sampling.

KEYWORDS: SIFT-MS; Atlantic cod (*Gadus morhua*); TVB-N; TMA-N

INTRODUCTION

Quality estimation of raw fishery products is nowadays a necessity for good functioning quality systems in fish-processing companies. Fish and fishery products are very susceptible to irreversible quality losses during storage due to chemical, but mainly microbiological, degradation. To assess the degree of spoilage of fish and fishery products, fast, accurate, objective, and nonambiguous measurements are needed. An often used parameter to determine fish spoilage, next to sensory scoring, is the quantification of typical odorous basic nitrogen compounds present in the fish tissue. $(\text{CH}_3)_3\text{N}$ (trimethylamine or TMA), $(\text{CH}_3)_2\text{NH}$ (dimethylamine or DMA), and NH_3 (ammonia) are products of autolytic and microbiological degradation and are collectively known as the total volatile basic nitrogen (TVB-N) fraction.

Facultative anaerobic bacteria such as *Shewanella putrefaciens*, *Acinetobacter*, and *Photobacterium phosphoreum*, recovered on iced fish, are able to reduce trimethylamine-N-oxide (TMAO) to TMA (*1*). TMAO has an osmoregulatory function in marine fish species, and the amount of TMAO present in the muscle tissue depends on the species, season, and marine environment (*2*).

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TMA is known to have a typical "fishy" odor. Some gaded fish species are able to reduce TMAO in an autolytic pathway to DMA and formaldehyde (*3, 4*). When bacterial growth is reduced, as under freezing conditions, production of DMA is an important factor involving quality losses and is considered as a frozen storage index (*5*). Ammonia is generally a bacterial degradation product, produced by the decarboxylation of amino acids in the fish muscle tissues (*1*). Ammonia is also released in Elasmobranch fish species, even in the early stages of storage, because of a fast enzymatic ammonia production originating from ureum (*6*).

The determination of the TVB-N in a fish sample is known to be the most common chemical parameter applied, because of its simplicity to evaluate the microbiological spoilage degree of fish and meat products. According to European directive 95/149/EEC, this indicator can be used if sensorial methods raise doubts about the freshness of the food product. Critical limits have been set for groups of seafood species and are expressed in milligrams of TVB-N per 100 g of tissue, for example, 35 mg of TVB-N/100 g of tissue and 15 mg of TMA-N/100 g of tissue for cod stored under ice (*1, 7*). Fresh cod normally has <20 mg of N/100 g of TVB and 3 mg of N/100 g of TMA (*1*). Generally, the most common methods for TVB-N estimation are based on steam distillation of an alkalinized sample (*8–10*). TMA analysis is