Case No.: IPR2023-00208
U.S. Patent No. 11,013,250

JAFCAU

STEENBOCK LIBRARY
MAY 0 1 1995

APRIL 1995
VOLUME 43
NUMBER 4

Journal of Agricultural and Food Chemistry

Published by the
American Chemical Society

Motif Exhibit 1129, Page 1 of 11

Journal of Agricultural and Food Chemistry

EDITOR: IRVIN E. LIENER

Associate Editors: G. Wayne Ivie
Marshall Phillips

Manuscript Office: Marie Crunkleton Wiggins, Manager; Bryan D. Tweedy, Senior Associate Editor; Sharon A. Dixon, Editorial Assistant

ADVISORY BOARD

Terry E. Acree
Eric Block
John W. Finley
Paul Hedin
Chi-Tang Ho
R. McGorrin
Steven Nagy
R. Petroski
Russ Rousseff
Richard Scanlan
Allan E. Smith
Walter Wolf

Journal of Agricultural and Food Chemistry (ISSN 0021-8561) is published monthly by the American Chemical Society at 1155 16th St., N.W., Washington, DC 20036. Second-class postage paid at Washington, DC, and additional mailing offices. POSTMASTER: Send address changes to *Journal of Agricultural and Food Chemistry*, Member & Subscriber Services, P.O. Box 3337, Columbus, OH 43210. Canadian GST Reg. No. R127571347. Printed in the USA.

PUBLICATIONS DIVISION

Robert H. Marks, Director

Journals Department: Charles R. Bertsch, Head

Editorial Office: Mary E. Scanlan, Manager; Anne C. O'Melia, Journals Editing Manager; Eileen M. Richardson, Associate Editor

Advertising Office: Centcom, Ltd., 1599 Post Road East, P.O. Box 231, Westport, CT 06881.

© Copyright 1995 American Chemical Society. **Copyright permission**: Reprographic copying beyond that permitted by Section 107 or 108 of the U.S. Copyright Act is allowed, provided that the appropriate per-copy fee is paid directly to the Copyright Clearance Center, Inc., 222 Rosewood Dr., Danvers, MA 01923. Anyone may excerpt up to 200 words per article for noncommercial, scholarly purposes with appropriate credit to the source. Direct other reprint permission requests to ACS Copyright Office, Publications Division, at the ACS Washington address. For quotes and information on ordering bulk reprints, please call (202) 872-4539 or write to the Distribution Office at the ACS Washington address.

The paper used in this publication meets the minimum requirements of American National Standard for Information Sciences—Permanence of Paper for Printed Library Materials, ANSI Z39.48-1984.

Editorial Information

Guide for Authors and copyright transfer form appear in the first issue of each volume.

Manuscripts for publication should be submitted (six copies) to Manuscript Office, *Journal of Agricultural and Food Chemistry*, at the ACS Washington address.

Correspondence regarding **accepted papers and proofs** should be directed to the Journals Department, at the address below.

Page charges of $40.00 per page may be paid for papers published in this journal. Payment does not affect acceptance or scheduling of papers.

The American Chemical Society and its Editors assume no responsibility for the statements and opinions advanced by contributors.

Registered names and trademarks, etc., used in this publication, even without specific indication thereof, are not to be considered unprotected by law.

A document number appears after the bibliographic information for each paper. It is a nine character code unique to each manuscript and is assigned during editorial production. It serves as a link between the documents in the printed product and the electronic products (CJACS Plus Images and journals on CD-ROM) and makes it easy to retrieve the full text of the exact paper.

1995 Subscription and Ordering Information

Members may share/donate their personal subscriptions with/to libraries and the like but only after 5 years from publication.

	U.S.	Canada and Mexico	Europe*	All Other Countries*
Printed				
Members—1 yr	$ 35	$ 54	$ 77	$ 91
Nonmembers	$420	$439	$462	$476
Microfiche				
Members—1 yr	$ 35	$ 35	$ 46	$ 46
Nonmembers	$420	$420	$431	$431
Supplementary material (microfiche)	$ 15	$ 21	$ 21	$ 21

* Air service included.

Nonmember subscribers in Japan must enter subscription orders with Maruzen Company Ltd., 3-10 Nihonbashi 2-chome, Chuo-ku, Tokyo 103, Japan. Tel: (03) 272-7211.

Microforms and Back Issue orders should be sent to the Microforms & Back Issues Office at the ACS Washington address.

New and renewal subscriptions should be sent with payment to American Chemical Society, Department L-0011, Columbus, OH 43268-0011. Subscriptions are available only on a calendar year basis.

Claims for missing numbers will not be allowed if loss was due to failure of notice of change of address to be received in the time specified; if claim is dated (a) North America—more than 90 days beyond issue date, (b) all other foreign—more than 180 days beyond issue date. Claims are handled by Member & Subscriber Services.

Supplementary material is noted in the table of contents with a ■. It is available as photocopy ($12.00 for up to 3 pages and $1.50 per page for additional pages, plus $2.00 for foreign postage) or as 24× microfiche ($12.00, plus $1.00 for foreign postage). Canadian residents should add 7% GST. See supplementary material notice at end of journal article for number of pages. Orders *must* state whether for photocopy or for microfiche and give complete title of article, names of authors, journal, issue date, and page numbers. Prepayment is required and prices are subject to change. Order from Microforms & Back Issues Office at the ACS Washington address. Supplementary material except structure factors also appears in the microfilm edition.

American Chemical Society
1155 16th St., N.W.
Washington, DC 20036
(202) 872-4600
TDD (202) 872-8733
FAX (202) 872-4615

Journals Department
American Chemical Society
2540 Olentangy River Road
P.O. Box 3330
Columbus, OH 43210
(614) 447-3600, Ext. 3171
TELEX 6842086; FAX (614) 447-3745

Member & Subscriber Services
American Chemical Society
P.O. Box 3337
Columbus, OH 43210
(614) 447-3776; FAX (614) 447-3671
(800) 333-9511

JAFCAU 43(4) 855–1114 (1995)
ISSN 0021-8561

Registered in U.S. Patent and Trademark Office
Copyright 1995 by the American Chemical Society

Journal of Agricultural and Food Chemistry®

APRIL 1995

STEENBOCK LIBRARY
MAY 0 1 1995

SHORT COMMUNICATIONS

855 Irreversible Sorption of Nicosulfuron on Clay Minerals
Ljerka Ukrainczyk* and Naghmana Rashid

858 Sialyloligosaccharides from Egg Yolk as an Inhibitor of Rotaviral Infection
Mamoru Koketsu,* Teruhiko Nitoda, Lekh Raj Juneja, Mujo Kim, Naoki Kashimura, and Takehiko Yamamoto

FOOD CHEMISTRY/BIOCHEMISTRY

862 Proteolysis of Kunitz Soybean Trypsin Inhibitor. Influence on Its Activity
Iosif A. Vaintraub* and Haram B. Yattara

867 Structure–Function Relationship of Acyl Amino Acid Surfactants: Surface Activity and Antimicrobial Properties
Jiding Xia, Yongmei Xia, and Ifendu A. Nnanna*

872 Nutritional and Antigenic Characterization of an Enzymatic Whey Protein Hydrolysate
Julio J. Boza, Olga Martínez-Augustin,* and Angel Gil

876 Flavonoid–DNA Interaction Studied with Flow Linear Dichroism Technique
Riccardo Solimani, Filippo Bayon, Ida Domini, Pier Giorgio Pifferi, Paolo E. Todesco, Giancarlo Marconi, and Bruno Samorì*

883 Hen Egg White Lysozyme–Metal Ion Interactions: Investigation by Electrospray Ionization Mass Spectrometry
Sabine Moreau, Abalo C. Awadé,* Daniel Mollé, Yvon Le Graet, and Gérard Brulé

890 Principal Phenolic Phytochemicals in Selected California Wines and Their Antioxidant Activity in Inhibiting Oxidation of Human Low-Density Lipoproteins
Edwin N. Frankel,* Andrew L. Waterhouse, and Pierre L. Teissedre

895 Immobilization of Biotinylated Transglutaminase by Bioselective Adsorption to Immobilized Avidin and Characterization of the Immobilized Activity
Xiaolin L. Huang, George L. Catignani, and Harold E. Swaisgood*

902 In Search of a New Vegetable Oil
A. Nag* and K. B. De

COMPOSITION OF FOODS/FEEDS

904 Studies on the Constituents of *Amaranthus caudatus* (Kiwicha) Seeds. Isolation and Characterization of Seven New Triterpene Saponins
Luca Rastrelli, Cosimo Pizza, Paola Saturnino, Oreste Schettino, and Antonio Dini*

Case No.: IPR2023-00208
U.S. Patent No. 11,013,250

910 Concentration of Total Al, Cr, Cu, Fe, Hg, Na, Pb, and Zn in Commercial Canned Seafood Determined by Atomic Spectrometric Means after Mineralization by Microwave Heating
Jorge E. Tahán, José M. Sánchez, Víctor A. Granadillo, Hernán S. Cubillán, and Romer A. Romero*

ANALYTICAL METHODS

916 Improved Methodology for the Quantitative Assessment of Tobacco-Specific *N*-Nitrosamines in Tobacco by Supercritical Fluid Extraction
Bogdan Prokopczyk,* Ming Wu, Jonathan E. Cox, Shantu Amin, Dhimant Desai, Ali M. Idris, and Dietrich Hoffmann

923 Evaluation of a Graphite Furnace Atomic Absorption Method Developed for the Determination of Lead in Sugars
N. J. Miller-Ihli*

928 Direct Determination of Styrene and Tetrachloroethylene in Olive Oil by Membrane Inlet Mass Spectrometry
Tapio Kotiaho,* Søren Gylling, Anita Lunding, and Frants R. Lauritsen

931 Rapid HPLC Determination of Tetracycline Antibiotics in Milk
William A. Moats* and Raida Harik-Khan

935 Quantification of Cholesterol Oxidation Products in Milk Powders Using [$^2H_7$]Cholesterol To Monitor Cholesterol Autoxidation Artifacts
Christine Rose-Sallin, Anthony C. Huggett, Jacques O. Bosset, Raffaele Tabacchi, and Laurent B. Fay*

942 Analysis of 3-(*N*-Oxalyl)-L-2,3-diaminopropanoic Acid and Its α-Isomer in Grass Pea (*Lathyrus sativus*) by Capillary Zone Electrophoresis
Anne Marie K. Arentoft and Brendon N. Greirson*

946 Sensitive Enzyme-Linked Immunosorbent Assay for the Mycotoxin Zearalenone in Barley and Job's-Tears
Touichi Tanaka,* Reiko Teshima, Hideharu Ikebuchi, Jun-ichi Sawada, and Masakatsu Ichinoe

951 Comparison of Capillary Electrophoresis, HPLC, and Enzyme Immunoassay for Terbuthylazine Detection in Water
Giovanni Dinelli,* Alberto Vicari, Alessandra Bonetti, and Pietro Catizone

956 Determination of Low Molecular Weight Dicarboxylic Acids in Root Exudates by Gas Chromatography
Anna M. Szmigielska,* Ken C. J. Van Rees, Grzegorz Cieslinski, Pan Ming Huang, and Doug R. Knott

960 Optimization of Analysis of Methyl Bromide on Charcoal Sampling Tubes
Jianying Gan,* Scott R. Yates, William F. Spencer, and Marylynn V. Yates

NUTRITION/TOXICOLOGY

967 Nitrate, Nitrite, and Volatile Nitrosamines in Whey-Containing Food Products
Claudia P. Oliveira, M. Beatriz A. Glória,* James F. Barbour, and Richard A. Scanlan*

970 *In Vitro* Binding of Benzo[*a*]pyrene by Extruded Potato Peels
Mary E. Camire,* Jianxin Zhao, Michael P. Dougherty, and Rodney J. Bushway

FLAVORS AND AROMAS

974 Synthesis of Glucosides Related to Grape and Wine Aroma Precursors
George K. Skouroumounis,* Ralph A. Massy-Westropp, Mark A. Sefton, and Patrick J. Williams

981 Test of Isotopic Fractionation during Liquid–Liquid Extraction of Volatile Components from Fruits
Sophie Dautraix,* Karin Gerola, Roger Guilluy, Jean-Louis Brazier, Anne Chateau, Elisabeth Guichard, and Patrick Etievant

984 Volatile Compounds from the Reaction of Cysteine, Ribose, and Phospholipid in Low-Moisture Systems
Donald S. Mottram* and Frank B. Whitfield

Case No.: IPR2023-00208
U.S. Patent No. 11,013,250

CHEMICAL CHANGES DURING PROCESSING/STORAGE

989 Physicochemical, Microbiological, and Akara-Making Properties of Hard-To-Cook Cowpeas
Yen-Con Hung,* Kay H. McWatters, Robert D. Phillips, Larry R. Beuchat, and Manjeet S. Chinnan

993 ■ Pesticides and Total Polychlorinated Biphenyls in Chinook Salmon and Carp Harvested from the Great Lakes: Effects of Skin-on and Skin-off Processing and Selected Cooking Methods
Mary E. Zabik,* Matthew J. Zabik, Al M. Booren, Miriam Nettles, Jeong-Hee Song, Robert Welch, and Harold Humphrey

1002 Suwari and Kamaboko Sardine Gels: Effect of Heat Treatment on Solubility of Networks
Mercedes Careche, Cristina Alvarez, and Margarita Tejada*

1011 Effect of Calcium Pretreatments on the Texture of Frozen Cherries. Role of Pectinesterase in the Changes in the Pectic Materials
Jesus Alonso,* Teresa Rodríguez, and Wenceslao Canet

1017 Antioxidative Activity of Volatile Browning Reaction Products and Related Compounds in a Hexanal/Hexanoic Acid System
Emad S. Shaker, M. Atef Ghazy, and Takayuki Shibamoto*

1023 Characterization of the Products Formed during Microwave Irradiation of the Nonenzymatic Browning Lysine/(*E*)-4,5-Epoxy-(*E*)-2-heptenal Model System
Francisco J. Hidalgo* and Rosario Zamora

1029 Influence of Irradiation Time, pH, and Lipid/Amino Acid Ratio on Pyrrole Production during Microwave Heating of a Lysine/(*E*)-4,5-Epoxy-(*E*)-2-heptenal Model System
Rosario Zamora* and Francisco J. Hidalgo

CHEMISTRY OF CROP AND ANIMAL PROTECTION

1034 ■ Preparation and Structure−Activity Relationships of 4-Substituted Amino-2-methylpyrido[3,4-*d*]pyrimidines as Cytokinin Analogs
Shiro Nishikawa,* Yoshio Nishikimi, Shinji Maki, Zenzaburo Kumazawa, and Naoki Kashimura

1039 Effects of a Supplemented Deltamethrin and Piperonyl Butoxide Diet on Levels of Residues in Products of Animal Origin. 2. Feeding Study in Poultry
Gilberte N. Marti-Mestres,* Jean-François M. Cooper, Jean-Paul M. Mestres, Georges De Wilde, and Nigel R. Wynn

1044 Toxicity of Pumiliotoxin 251D and Synthetic Analogs to the Cotton Pest *Heliothis virescens*
Thomas M. Bargar,* Renee M. Lett, Peter L. Johnson, James E. Hunter, C. P. Chang, Daniel J. Pernich, Mark R. Sabol, and Michael R. Dick

1052 Metabolism of the Insecticide Phoxim in Plants and Cell Suspension Cultures of Soybean
Hans-Ulrich Höhl and Wolfgang Barz*

1057 Preformed Antifungal Compounds of Lemon Fruit: Citral and Its Relation to Disease Resistance
Victor Rodov,* Shimshon Ben-Yehoshua, De Qiu Fang, Jong Jin Kim, and Rina Ashkenazi

1062 Preformed Antifungal Compounds of Citrus Fruit: Effect of Postharvest Treatments with Heat and Growth Regulators
Shimshon Ben-Yehoshua,* Victor Rodov, De Qiu Fang, and Jong Jin Kim

1067 Sorption of Monodisperse Alcohol Ethoxylates and Their Effects on the Mobility of 2,4-D in Isolated Plant Cuticles
Markus Riederer,* Markus Burghardt, Sabine Mayer, Hans Obermeier, and Jörg Schönherr

ENVIRONMENTAL CHEMISTRY

1076 Mobility and Dissipation of ^{14}C-Labeled Atrazine, Metolachlor, and Primisulfuron in Undisturbed Field Lysimeters of a Coastal Plain Soil
Kyle E. Keller* and Jerome B. Weber

1087 Distribution and Persistence of Diflubenzuron within Littoral Enclosure Mesocosms
Michael L. Knuth* and Larry J. Heinis

Motif Exhibit 1129, Page 5 of 11

Case No.: IPR2023-00208
U.S. Patent No. 11,013,250

BIOTECHNOLOGY

1098 Capillary Enzyme Immunoassay with Electrochemical Detection for the Determination of Atrazine in Water
Tongbo Jiang, H. Brian Halsall,* William R. Heineman,* Thomas Giersch, and Bertold Hock

1105 Purification and Characterization of Neomycin Phosphotransferase II from Genetically Modified Cottonseed (*Gossypum hirsutum*)
David C. Wood,* Linh V. Vu, Nancee M. Kimack, Glennon J. Rogan, Joel E. Ream, and Thomas E. Nickson

1110 Content of Free Terpenic Compounds in Cells and Musts during Vinification with Three *Saccharomyces cerevisiae* Races
Luis Zea, Juan Moreno, Jose M. Ortega, and Manuel Medina*

AUTHOR INDEX

Alonso, J., 1011
Alvarez, C., 1002
Amin, S., 916
Arentoft, A. M. K., 942
Ashkenazi, R., 1057
Awadé, A. C., 883

Barbour, J. F., 967
Bargar, T. M., 1044
Barz, W., 1052
Bayon, F., 876
Ben-Yehoshua, S., 1057, 1062
Beuchat, L. R., 989
Bonetti, A., 951
Booren, A. M., 993
Bosset, J. O., 935
Boza, J. J., 872
Brazier, J.-L., 981
Brulé, G., 883
Burghardt, M., 1067
Bushway, R. J., 970

Camire, M. E., 970
Canet, W., 1011
Careche, M., 1002
Catignani, G. L., 895
Catizone, P., 951
Chang, C. P., 1044
Chateau, A., 981
Chinnan, M. S., 989
Cieslinski, G., 956
Cooper, J.-F. M., 1039
Cox, J. E., 916
Cubillán, H. S., 910

Dautraix, S., 981
De, K. B., 902
Desai, D., 916
De Wilde, G., 1039
Dick, M. R., 1044
Dinelli, G., 951
Dini, A., 904
Domini, I., 876
Dougherty, M. P., 970

Etievant, P., 981

Fang, D. Q., 1057, 1062
Fay, L. B., 935
Frankel, E. N., 890

Gan, J., 960
Gerola, K., 981
Ghazy, M. A., 1017
Giersch, T., 1098
Gil, A., 872
Glória, M. B. A., 967
Granadillo, V. A., 910
Greirson, B. N., 942
Guichard, E., 981
Guilluy, R., 981
Gylling, S., 928

Halsall, H. B., 1098
Harik-Khan, R., 931
Heineman, W. R., 1098
Heinis, L. J., 1087
Hidalgo, F. J., 1023, 1029
Hock, B., 1098
Hoffmann, D., 916
Höhl, H.-U., 1052
Huang, P. M., 956
Huang, X. L., 895
Huggett, A. C., 935
Humphrey, H., 993
Hung, Y.-C., 989
Hunter, J. E., 1044

Ichinoe, M., 946
Idris, A. M., 916
Ikebuchi, H., 946

Jiang, T., 1098
Johnson, P. L., 1044
Juneja, L. R., 858

Kashimura, N., 858, 1034
Keller, K. E., 1076
Kim, J. J., 1057, 1062
Kim, M., 858
Kimack, N. M., 1105
Knott, D. R., 956
Knuth, M. L., 1087
Koketsu, M., 858
Kotiaho, T., 928
Kumazawa, Z., 1034

Lauritsen, F. R., 928
Le Graet, Y., 883
Lett, R. M., 1044

Lunding, A., 928

Maki, S., 1034
Marconi, G., 876
Marti-Mestres, G. N., 1039
Martínez-Augustin, O., 872
Massy-Westropp, R. A., 974
Mayer, S., 1067
McWatters, K. H., 989
Medina, M., 1110
Mestres, J.-P. M., 1039
Miller-Ihli, N. J., 923
Moats, W. A., 931
Mollé, D., 883
Moreau, S., 883
Moreno, J., 1110
Mottram, D. S., 984

Nag, A., 902
Nettles, M., 993
Nickson, T. E., 1105
Nishikawa, S., 1034
Nishikimi, Y., 1034
Nitoda, T., 858
Nnanna, I. A., 867

Obermeier, H., 1067
Oliveira, C. P., 967
Ortega, J. M., 1110

Pernich, D. J., 1044
Phillips, R. D., 989
Pifferi, P. G., 876
Pizza, C., 904
Prokopczyk, B., 916

Rashid, N., 855
Rastrelli, L., 904
Ream, J. E., 1105
Riederer, M., 1067
Rodov, V., 1057, 1062
Rodríguez, T., 1011
Rogan, G. J., 1105
Romero, R. A., 910
Rose-Sallin, C., 935

Sabol, M. R., 1044
Samorì, B., 876

Sánchez, J. M., 910
Saturnino, P., 904
Sawada, J., 946
Scanlan, R. A., 967
Schettino, O., 904
Schönherr, J., 1067
Sefton, M. A., 974
Shaker, E. S., 1017
Shibamoto, T., 1017
Skouroumounis, G. K., 974
Solimani, R., 876
Song, J.-H., 993
Spencer, W. F., 960
Swaisgood, H. E., 895
Szmigielska, A. M., 956

Tabacchi, R., 935
Tahán, J. E., 910
Tanaka, T., 946
Teissedre, P. L., 890
Tejada, M., 1002
Teshima, R., 946
Todesco, P. E., 876

Ukrainczyk, L., 855

Vaintraub, I. A., 862
Van Rees, K. C. J., 956
Vicari, A., 951
Vu, L. V., 1105

Waterhouse, A. L., 890
Weber, J. B., 1076
Welch, R., 993
Whitfield, F. B., 984
Williams, P. J., 974
Wood, D. C., 1105
Wu, M., 916
Wynn, N. R., 1039

Xia, J., 867
Xia, Y., 867

Yamamoto, T., 858
Yates, M. V., 960
Yates, S. R., 960
Yattara, H. B., 862

Zabik, M. E., 993
Zabik, M. J., 993
Zamora, R., 1023, 1029
Zea, L., 1110
Zhao, J., 970

■ Supplementary material for this paper is available separately (consult the masthead page for ordering information); all supplementary material except structure factor tables will also appear following the paper in the microfilm edition of this journal.

* In papers with more than one author, the asterisk indicates the name of the author to whom inquiries about the paper should be addressed.

Motif Exhibit 1129, Page 6 of 11

Case No.: IPR2023-00208
U.S. Patent No. 11,013,250

This material may be protected by Copyright law (Title 17 U.S. Code)

Volatile Compounds from the Reaction of Cysteine, Ribose, and Phospholipid in Low-Moisture Systems

Donald S. Mottram*

Department of Food Science and Technology, The University of Reading, Whiteknights, Reading RG6 2AP, United Kingdom

Frank B. Whitfield

Food Research Laboratory, CSIRO Division of Food Science and Technology, P.O. Box 52, North Ryde, NSW 2113, Australia

The headspace volatiles from Maillard reaction mixtures of cysteine and ribose heated at 185 °C with or without the addition of phosphatidylcholine, either dry or in the presence of a small quantity of water, were analyzed by GC–MS. The major products of the reaction were 3,5-dimethyl-1,2,4-trithiolane, 3-methyl-1,2,4-trithiane, 3,6-dimethyl-1,2,4,5-tetrathiane, and thieno[2,3-*b*]thiophene. Other products included a number of thiazoles and small amounts of some furanthiols and disulfides. Water had a significant effect on the relative amounts of volatiles produced; in particular, 1-(2-furanylmethyl)-1*H*-pyrrole, two other 1-(2-furanylmethyl)alkyl-1*H*-pyrroles, and two bis(furan) compounds were only produced to any extent in systems without water. The addition of lipid had only a small effect on the volatile profile, with small amounts of lipid degradation products and lipid/Maillard interaction products formed. These results differ greatly from previous studies on the volatiles formed in reaction systems carried out in the presence of large quantities of water.

Keywords: *Volatiles; Maillard reaction; moisture content; phospholipid*

INTRODUCTION

The two most important series of reactions for the formation of volatile aroma compounds in cooked and thermally processed foods are the Maillard reaction (between amino compounds and reducing sugars) and lipid degradation (thermal and oxidative). A number of early patents noted that lipid could influence the flavor of amino acid–sugar mixtures in reaction product flavorings (MacLeod and Seyyedain-Ardebili, 1981). However, the significance of Maillard/lipid interactions in the production of cooked food flavors was not fully recognized until the late 1980s. In a series of articles Whitfield, Mottram, and Farmer showed that a range of flavor compounds, particularly those with heterocyclic nuclei, were formed in interactions between amino acids, the pentose sugar, ribose, and phospholipids (Farmer and Mottram, 1990a; Farmer et al., 1989; Salter et al., 1988; Whitfield et al., 1988). All of these reactions were performed in aqueous solutions with the concentrations of the reactants similar to those found in raw meat. In a recent review of this work (Whitfield, 1992) it was suggested that future studies in this field should examine Maillard/lipid interaction in nonaqueous systems such as those encountered in roasting and extrusion.

The current study was undertaken to observe the effect that the absence of water had on the volatile products formed in the Maillard reaction between cysteine and ribose and on the Maillard/lipid interaction products.

EXPERIMENTAL PROCEDURES

Materials. L-Cysteine, D-(−)-ribose, and lecithin (L-α-phosphatidylcholine, type X-E from dried egg yolk) were purchased from Sigma Chemical Co. Phosphate buffer (0.5 M, pH 5.6) was prepared from disodium hydrogen phosphate and sodium dihydrogen phosphate (BDH Chemicals Ltd.) in glass-distilled water. Authentic samples of volatile compounds were purchased from a range of laboratory chemical suppliers or were obtained as gifts from flavor laboratories.

Preparation of Reaction Mixtures. Mixtures of cysteine (10 mg) and ribose (10 mg) were prepared by grinding to fine powder in a mortar and pestle. The mixtures were transferred to small glass reaction tubes. Lecithin (40 mg) was added to one sample, water (100 μL, buffered with phosphate at pH 5.6) was added to another, and both water and lecithin were added to a third, while a fourth sample contained only cysteine and ribose. The contents of the tubes were thoroughly mixed, and the tubes were flame sealed and then heated in an aluminum block at 185 °C for 20 min. Reactions of cysteine alone and cysteine with water were also carried out.

Isolation of Volatile Maillard Reaction Products. After cooling, each reaction mixture was transferred to a 250 mL conical flask containing 20 mL of 0.5 M phosphate buffer (pH 5.6) and a magnetic stirrer bar. The reaction tube was rinsed twice with buffer solution (2 mL), and these washings were added to the conical flask. The flask was fitted with a Dreschel head, and a glass-lined stainless steel tube (115 mm long × 0.75 mm i.d.) packed with Tenax GC (Scientific Glass Engineering Pty Ltd., Australia) was attached by a stainless steel reducing union to the head outlet. During the collection of the volatile components, the dilute reaction solution was stirred slowly and maintained at 60 °C in a water bath, while the Tenax trap was maintained at room temperature. The volatiles were swept from the flask to the adsorbent in the trap using a flow of oxygen-free nitrogen (60 mL/min), and the collection was continued for 1 h. At the end of this time, the flask was removed and the trap was connected directly to the nitrogen supply for 5 min to remove moisture. An internal standard, 1,2-dichlorobenzene (65 ng, in 1 μL of diethyl ether) was added to the front end of the trap just before GC–MS analysis.

Gas Chromatography–Mass Spectrometry. The gas chromatograph was a Hewlett-Packard HP5890 gas chromatograph, equipped with a "Unijector" (Scientific Glass Engineer-

* Author to whom correspondence should be addressed.

0021-8561/95/1443-0984$09.00/0 © 1995 American Chemical Society

Case No.: IPR2023-00208
U.S. Patent No. 11,013,250

ing Pty Ltd., Australia) set in the concentrator-headspace mode and fitted with a fused silica capillary column (50 m long × 0.32 mm i.d.) coated with DB-5 (Scientific Glass Engineering Pty Ltd., Australia). The gas chromatograph was directly coupled to a Hewlett-Packard HP5988A mass spectrometer. The helium carrier gas was set at a flow of 1 mL/min. The trapped volatile components were thermally desorbed onto the GC column by heating the trap at 250 °C for 5 min while the oven was cooled at 0 °C using the subambient facility. By directing the subambient cooling gas at the front of the column, efficient cryofocusing of the desorbed volatiles was achieved. After removal of the coolant, the column temperature was increased to 60 °C at a rate of 60 °C/min, held at that temperature for 5 min, and then increased to 250 °C at a rate of 4 °C/min. GC–MS of C_6–C_{25} *n*-alkanes (100 ng of each in 1 μL of diethyl ether added to the trap), analyzed under the above conditions, were used as external standard references for the calculation of linear retention indices of the volatile reaction products.

The mass spectrometer was operated in the electron impact mode with an electron energy of 70 eV and an ion source temperature of 250 °C. A continuous scan mode was employed with a scan time of 1 s over a mass range of 20–400 amu. Compounds were tentatively identified by comparing their mass spectra with those contained in the NIST/EPA/MSDC and Wiley Mass Spectral Databases, in collections of mass spectra of flavor compounds (ten Noever de Brauw et al., 1980) or in previously published literature. Where possible, confirmation of identifications was carried by comparing of linear retention indices with those of authentic compounds.

RESULTS AND DISCUSSION

The volatiles from the reaction mixtures were dominated by sulfur-containing heterocyclics, with thiazoles, trithiolanes, and trithianes the most abundant. Table 1 lists 52 compounds identified from the cysteine/ribose reaction systems with or without phospholipid, carried out in wet or dry conditions. Thirty-nine of these compounds contained sulfur, and 10 compounds were only present in the lipid-containing systems and were derived from lipid degradation or lipid/Maillard interactions. To obtain some measure of the relative concentrations of these compounds in the different reaction mixtures, peak areas in each total ion chromatogram were compared with that of the dichlorobenzene internal standard (65 ng). Peaks that were >150% of the internal standard were defined as present in extra large quantities (++++), those between 50 and 150%, large quantities (+++), those between 10 and 50%, medium quantities (++), those between 1 and 10%, small quantities (+), and those <1%, trace quantities. A number of components remain unidentified, but none of these showed major differences between the reaction systems.

Cysteine was thermally stable when heated alone under dry conditions; however, heating cysteine on its own in the presence of a small amount of water led to the formation of a number of the above compounds, including most of the thiazoles, the thienothiophenes, and the polysulfur heterocyclics found in the cysteine/ribose systems. The quantities were significantly lower, as might be expected, since the breakdown of amino acids in the Maillard reaction occurs more readily than thermal degradation in the absence of a sugar. Recently, Zhang et al. (1988) observed the formation of trithiolanes, trithianes, and tetrathianes in the thermal degradation of cysteine. However, they reported very few thiazoles but significant quantities of thiadiazines and dithiazines. None of these latter compounds were found in the present reaction of cysteine alone; however, the pH was lower and the water content was much less in the present work, and these variations in conditions may explain the differences between the two systems.

It is particularly interesting to compare the data for the present cysteine/ribose reactions with those obtained from earlier studies that used dilute aqueous solutions of cysteine and ribose heated at 140 °C (Farmer and Mottram, 1990a, 1992; Farmer et al., 1989; Whitfield et al., 1988). The present work was carried out either in the absence of water or with a relatively low moisture content (5:1, water:solids) and was performed at a higher temperature (185 °C). The conditions chosen were similar to those used for roasting coffee or those attained on the surface of grilled meat. There was a marked difference in the results obtained from these systems, i.e. containing no water or low water concentrations, compared with the aqueous systems investigated previously.

In reactions between cysteine, ribose, and lecithin under aqueous conditions a total of 152 compounds were identified, including many present in trace quantities. Only 34 of these were among the 52 compounds identified in the current study. Thus, 18 compounds, principally heterocyclic compounds containing sulfur, were not formed under aqueous conditions. Larger quantities of reactants were used in the aqueous studies, which may have accounted for the large numbers of trace components, but the absence of so many of the components that have now been found in the low-moisture systems illustrates the importance of reaction conditions on the formation of volatile products in the Maillard reaction.

In the previously reported aqueous systems, in the absence of lipid, the major volatile components were thiols such as 2-methyl-3-furanthiol, 2-furanmethanethiol, 2-thiophenethiol, 2-methyl-3-thiophenethiol, 2-mercapto-3-butanone, and two mercaptopentanones. Other major components were thienothiophenes, several thiophenones, and other oxygenated thiophenes. In the corresponding dry or low-moisture systems these compounds were not detected or were found at only trace levels, the only exception being the thienothiophenes, which were present in high concentrations. Other major components of the dry and low-moisture systems were 3,5-dimethyl-1,2,4-trithiolane, 3-methyl-1,2,4-trithiane, 3,6-dimethyl-1,2,4,5-tetrathiane, 1-(2-furanylmethyl)-1*H*-pyrrole, and 5-ethyl-4-methylthiazole. Although most of these were reported in the aqueous systems in significant amounts, they were not the dominant components. These current findings partly explain the differences in flavor observed between meats and other foods cooked under dry (e.g. grilled) or moist (e.g. boiled) conditions.

The addition of phospholipid to the dry and low-moisture system produced some equally unexpected results. Under the aqueous conditions of the previous work, large quantities of Maillard/lipid interaction products, including 2-pentylthiophene, 2-hexylthiophene, 2-pentylthiapyran, and 2-pentylpyridine, were produced together with large amounts of lipid oxidation products such as pentylfuran, alkanals, and alkanols. The low-moisture and dry systems reported in the present work contained only small amounts of these Maillard/lipid interaction products, and the only significant lipid oxidation products were a series of 2-alkanones. Clearly, when water is present in a very large excess, the volatile products of the Maillard reaction differ greatly from those of low-moisture systems. The conditions also affect the formation of lipid oxidation products, the dry

Case No.: IPR2023-00208
U.S. Patent No. 11,013,250

Table 1. Volatile Compounds Obtained from Reactions between Cysteine and Ribose in the Presence and Absence of Phospholipid and Water [a]

compound	LRI[b]	relative concentration[c] water absent C + R	C + R + L	water present C + R	C + R + L	C alone	method of identification[d]	MS data, m/z (rel intens), or literature reference[e]
5-methylthiazole[f]	846	tr	–	tr	–	+	MS	ten Noever de Brauw et al. (1980)
2,4-dimethylthiazole[f]	892	tr	–	tr	–	–	MS + LRI	
4,5-dimethylthiazole	933	tr	–	tr	–	tr	MS + LRI	
5-ethylthiazole[f]	940	+	tr	–	–	tr	MS	ten Noever de Brauw et al. (1980)
2-propylthiazole[f]	981	tr	–	–	–	tr	MS	
trimethylthiazole	1000	+	+	+	+	+	MS + LRI	
5-ethyl-2-methylthiazole	1015	+++	++	+	++	+	MS	Farmer et al. (1989)
5-ethyl-4-methylthiazole	1039	tr	–	tr	tr	tr	MS + LRI	
2,5-dimethyl-4-ethylthiazole[f]	1066	+	tr	+	+	–	MS + LRI	
4-ethyl-5-methylthiazole	1068	tr	–	tr	–	–	MS	
2,4-dimethyl-5-ethylthiazole	1078	+	+	+	+	–	MS + LRI	
4,5-dimethyl-2-ethylthiazole[f]	1105	tr	tr	–	tr	–	MS + LRI	
4,5-dimethyl-2-propylthiazole[f]	1180	tr	+	tr	tr	tr	MS + LRI	
4(or 5)-ethyl-2-propylthiazole[f]	1190	+	+	tr	+	+	ms	127 (100), 140 (31), 71 (25), 112 (20), 85 (16), 45 (15), 126 (13), 39 (13), 128 (10), **155** (8), 154 (7)
1-(2-furanylmethyl)-1*H*-pyrrole	1192	++	+++	–	tr	–	MS + LRI	
1-(2-furanylmethyl)methyl-1*H*-pyrrole[f]	1276	tr	+	–	–	–	ms	81 (100), 53 (14), **161** (8), 82 (6), 27 (3), 97 (2), 117 (2)
1-(2-furanylmethyl)dimethyl-1*H*-pyrrole[f]	1361	–	tr	–	–	–	ms	81 (100), **175** (22), 97 (7), 82 (6)
2,2′-methylenebis(furan)[f]	1086	tr	+	–	–	–	MS	
2,2′-(1,2-ethylenediyl)bis(furan)[f]	1336	++	++	–	tr	–	MS	
4,5-dihydro-2-methyl-3(2*H*)-thiophenone	989	–	–	++	tr	–	MS + LRI	
2-acetylthiophene	1086	tr	–	tr	tr	tr	MS + LRI	
1-(2-thienyl)-1-propanone	1188	tr	–	tr	tr	–	MS + LRI	
thieno[2,3-*b*]thiophene	1212	++	++	+++	++++	++	MS	Farmer et al. (1989)
thieno[3,2-*b*]thiophene	1214	+	tr	+	+	tr	MS	ten Noever de Brauw et al. (1980)
3,5-dimethyl-1,2,4-trithiolane (*E* or *Z*)	1136	++	++	+++	++++	+	MS + LRI	
3,5-dimethyl-1,2,4-trithiolane (*E* or *Z*)	1148	++	++	+++	++++	+	MS + LRI	
3-methyl-1,2,4-trithiane	1243	++	++	++	++	+	MS + LRI	**152** (100), 60 (94), 59 (87), 92 (53), 64 (44), 45 (36), 58 (13), 154 (12), 61 (11)
3,6-dimethyl-1,2,4,5-tetrathiane (*E* or *Z*)[f]	1388	++	++	+++	+++	++	MS	Zhang et al. (1988)
3,6-dimethyl-1,2,4,5-tetrathiane (*E* or *Z*)[f]	1406	+	+	++	++	+	MS	Zhang et al. (1988)
4,6-dimethyl-1,2,3,5-tetrathiane[f]	1442	+	–	+	+	+	MS	Zhang et al. (1988)
2-methyl-3-furanthiol	870	tr	–	tr	tr	–	MS + LRI	Evers et al. (1976)
2-furanmethanethiol	913	+	+	tr	–	–	MS + LRI	
2-methyl-3-thiophenethiol	1058	–	–	tr	–	–	MS	van den Ouweland and Peer (1975)
2-methyl-3-(methylthio)furan	947	–	+	–	–	–	MS	MacLeod and Ames (1986)
3-(methylthio)thiophene[f]	1082	–	++	–	tr	–	MS	ten Noever de Brauw et al. (1980)
2-methyl-3-(methylthio)thiophene[f]	1144	–	+	–	–	–	ms	129 (100), **144** (91), 45 (16), 85 (15), 97 (14), 131 (9), 69 (9), 146 (8)
bis(2-methyl-3-furanyl) disulfide	1547	+	–	++	tr	–	MS + LRI	Mottram et al. (1994)
3-(2-methyl-3-furanyldithio)pentan-2-one	1580	–	–	tr	–	–	MS + LRI	Mottram et al. (1994)
2-(2-methyl-3-furanyldithio)pentan-3-one	1593	–	–	+	–	–	MS + LRI	Mottram et al. (1994)
2-methyl-3-(2-furanylmethyldithio)furan	1649	–	–	+	–	–	MS + LRI	Mottram et al. (1994)
bis(2-furanylmethyl) disulfide	1701	–	–	tr	–	–	MS + LRI	Mottram et al. (1994)
2-methyl-3-(2-methyl-3-furanyldithio)thiophene	1727	tr	–	+	tr	–	MS	Werkhoff et al. (1990)
2-heptanone	890	–	+	–	tr	–	MS + LRI	
2-octanone	993	–	+	–	tr	–	MS + LRI	
2-nonanone	1095	–	++	–	tr	–	MS + LRI	
2-decanone	1198	–	++	–	–	–	MS + LRI	
2-undecanone[f]	1297	–	+	–	–	–	MS + LRI	
1-octen-3-ol	981	–	++	–	tr	–	MS + LRI	
2-pentylfuran	991	–	+	–	–	–	MS + LRI	
2-pentylthiophene	1164	–	+	–	–	–	MS + LRI	
2-pentylthiapyran	1317	–	++	–	–	–	ms	97 (100), 98 (6), **168** (4), 45 (4), 111 (2)
2-pentylpyridine	1202	–	tr	–	–	–	MS + LRI	

[a] C, cysteine; R, ribose; L, phospholipid (lecithin). [b] Linear retention index. [c] Relative size of peaks in GC–MS chromatogram: ++++, very large; +++, large; ++, medium; +, small; tr, trace; –, not detected. [d] MS + LRI, mass spectrum and LRI agree with those of authentic compound; MS, mass spectrum agrees with literature spectrum; ms, interpretation of mass spectrum. [e] Where neither mass spectrum nor a reference is given, the reference spectrum can be found in NIST/EPA/MSDC or Wiley Mass Spectral Databases. [f] Compound not found in previous work involving reaction in dilute aqueous solution.

Motif Exhibit 1129, Page 9 of 11

Case No.: IPR2023-00208
U.S. Patent No. 11,013,250

and low-moisture systems producing many fewer lipid-derived volatiles.

A number of studies have investigated the role of moisture in the oxidation of lipids in food systems and, in general, oxidation decreases with decreasing water activity, although at activities below a_w 0.2–0.3 oxidation is higher than at the intermediate levels (Nelson and Labuza, 1992). The food matrix plays a significant role in determining these effects, and the ability of water to mobilize reactants and catalysts may be important. Other effects of water, such as hydrogen bonding to lipid hydroperoxides and the reaction with free radical oxidation intermediates, are also believed to be important. In the systems reported in this paper the mobility of reactants, especially the polar phospholipid, and intermediates in the aqueous systems may play an important role in determining the amount and type of lipid oxidation products.

A comparison within the systems used in the present study further demonstrates the importance of water in determining the nature of the volatiles produced in the Maillard reaction. The dry cysteine/ribose systems showed more thiazoles than the other systems and quantities were higher. In the absence of water, 1-(2-furanylmethyl)-1*H*-pyrrole was a major volatile component, the concentration of which was increased by the presence of lipid. Small amounts of other 1-(furanylmethyl)alkyl-1*H*-pyrroles were also found. However, with the addition of a small amount of water these compounds were almost eliminated from the volatiles. Similarly, the two bisfuran compounds were associated with the dry Maillard systems. Thiophenes and thienothiophenes were more readily formed in the presence of water, as were the trithiolanes, trithianes, and tetrathianes.

Among the most important contributors to the characteristic aroma of cooked meat are 2-methyl-3-furanthiol, the corresponding thiophene, and disulfides derived from these thiols (Mottram, 1991). These compounds have extremely low odor threshold values, and consequently only trace amounts are needed for meatlike aromas; indeed, at higher concentrations many of the compounds become unpleasantly sulfurous in character. Such thiols were found in trace quantities in the four reaction systems, and disulfides were only found to any extent in the water-containing system without phospholipid. Interestingly, the three methyl sulfides, 2-methyl-3-(methylthio)furan, 2-methyl-3-(methylthio)-thiophene, and 3-(methylthio)thiophene, were only formed in systems containing phospholipid. Other reports have discussed the importance of pH and lipid in the formation of thiols and disulfides in meatlike model systems (Farmer and Mottram, 1990b; Mottram and Madruga, 1994; Mottram and Whitfield, 1994). The present work demonstrates that other conditions can influence their formation.

There are many different routes to the formation of aroma compounds in the Maillard and associated reactions, few of which have been fully elucidated. The results reported here clearly indicate that certain reactions are more favored in dry conditions, whereas others occur more readily in the presence of large amounts of water. For example, furan- and thiophenethiols, mercaptoketones, and other oxygenated sulfur heterocylics, such as dithiolanones, are formed from the action of hydrogen sulfide on ribose-derived Maillard reaction products (Zhang and Ho, 1991; Mottram and Whitfield, 1994). On the other hand, 3,5-dimethyl-1,2,4-trithiolane, 3-methyl-1,2,4-trithiane, and 3,6-dimethyl-1,2,4,5-tetrathiane can be formed from the interaction of acetaldehyde, hydrogen sulfide, and mercaptoacetaldehyde, all of which are produced by the breakdown of cysteine at high temperature. At low moisture levels the thermal degradation of cysteine is much more prominent than reactions of ribose with hydrogen sulfide, which requires relatively high water content.

ACKNOWLEDGMENT

We thank Steve Elmore and Marta Madruga for assistance in the operation of the GC–MS.

LITERATURE CITED

Evers, W. J.; Heinsohn, H. H.; Mayers, B. J.; Sanderson, A. Furans substituted in the three position with sulfur. In *Phenolic, Sulfur and Nitrogen Compounds in Food Flavors*; Charalambous, G., Katz, I., Eds.; American Chemical Society: Washington, DC, 1976; pp 184–193.

Farmer, L. J.; Mottram, D. S. Interaction of lipid in the Maillard reaction between cysteine and ribose: effect of a triglyceride and three phospholipids on the volatile products. *J. Sci. Food Agric.* **1990a**, *53*, 505–525.

Farmer, L. J.; Mottram, D. S. Recent studies on the formation of meat-like aroma compounds. In *Flavour Science and Technology*; Bessiere, Y., Thomas, A. F., Eds.; Wiley: Chichester, U.K., 1990b; pp 113–116.

Farmer, L. J.; Mottram, D. S. Effect of cysteine and ribose on the volatile thermal degradation products of a triglyceride and three phospholipids. *J. Sci. Food Agric.* **1992**, *60*, 489–497.

Farmer, L. J.; Mottram, D. S.; Whitfield, F. B. Volatile compounds produced in Maillard reactions involving cysteine, ribose and phospholipid. *J. Sci. Food Agric.* **1989**, *49*, 347–368.

MacLeod, G.; Ames, J. M. 2-Methyl-3-(methylthio)furan: a meaty character impact aroma compound identified from cooked beef. *Chem. Ind. (London)* **1986**, 175–176.

MacLeod, G. M.; Seyyedain-Ardebili, M. Natural and simulated meat flavors (with particular reference to beef). *CRC Crit. Rev. Food Sci. Nutr.* **1981**, *14*, 309–437.

Mottram, D. S. Meat. In *Volatile Compounds in Foods and Beverages*; Maarse, H., Ed.; Dekker: New York, 1991; pp 107–177.

Mottram, D. S.; Madruga, M. S. Important sulfur-containing aroma volatiles in meat. In *Sulfur Compounds in Foods*; Mussinan, C. J., Keelan, M. E., Eds.; ACS Symposium Series 564; American Chemical Society: Washington, DC, 1994; pp 180–187.

Mottram, D. S.; Whitfield, F. B. Aroma volatiles from meatlike Maillard systems. In *Thermally Generated Flavors: Maillard, Microwave, and Extrusion Processes*; Parliment, T. H., Morello, M. J., McGorrin, R. J., Eds.; ACS Symposium Series 543; American Chemical Society: Washington, DC, 1994; pp 180–191.

Mottram, D. S.; Madruga, M. S.; Whitfield, F. B. Some novel meatlike aroma compounds from the reactions of alkanediones with hydrogen sulfide and furanthiols. *J. Agric. Food Chem.* **1995**, *43*, 189–193.

Nelson, K. A.; Labuza, T. P. Relationship between water and lipid oxidation rates. In *Lipid Oxidation in Food*; St.Angelo, A. J., Ed.; ACS Symposium Series 500; American Chemical Society: Washington, DC, 1992; pp 93–103.

Salter, L. J.; Mottram, D. S.; Whitfield, F. B. Volatile compounds produced in Maillard reactions involving glycine, ribose and phospholipid. *J. Sci. Food Agric.* **1988**, *46*, 227–242.

ten Noever de Brauw, M. C.; Bouwman, J.; Tas, A. C.; La Vas, G. F. *Compilation of Mass Spectra of Volatile Compounds in Food*; Institute for Nutrition and Food Research TNO: Zeist, The Netherlands, 1980.

Case No.: IPR2023-00208
U.S. Patent No. 11,013,250

van den Ouweland, G. A. M.; Peer, H. G. Components contributing to beef flavor. Volatile compounds produced by the reaction of 4-hydroxy-5-methyl-3(2*H*)-furanone and its thio analog with hydrogen sulfide. *J. Agric. Food Chem.* **1975**, *23*, 501–505.

Werkhoff, P.; Bruning, J.; Emberger, R.; Guntert, M.; Kopsel, M.; Kuhn, W.; Surburg, H. Isolation and characterisation of volatile sulfur-containing meat flavor components in model systems. *J. Agric. Food Chem.* **1990**, *38*, 777–791.

Whitfield, F. B. Volatiles from interactions of Maillard reactions and lipids. *Crit. Rev. Food Sci. Nutr.* **1992**, *31*, 1–58.

Whitfield, F. B.; Mottram, D. S.; Brock, S.; Puckey, D. J.; Salter, L. J. The effect of phospholipid on the formation of volatile heterocyclic compounds in heated aqueous solutionsof amino acids and ribose. *J. Sci. Food Agric.* **1988**, *42*, 261–272.

Zhang, Y.; Ho, C.-T. Formation of meatlike aroma compounds from thermal reaction inosine 5′-monophosphate with cysteine and glutathione. *J. Agric. Food Chem.* **1991**, *39*, 1145–8.

Zhang, Y.; Chien, M.; Ho, C.-T. Comparison of the volatile compounds obtained from the thermal reaction of cysteine and glutathione. *J. Agric. Food Chem.* **1988**, *36*, 992–996.

Received for review September 23, 1994. Accepted February 6, 1995.[⊗]

JF940539E

[⊗] Abstract published in *Advance ACS Abstracts,* March 15, 1995.

Motif Exhibit 1129, Page 11 of 11