

File 2003-01A

INIS-AT -- 0038



# Trends in Sample Preparation 2002

Development and Application

## BOOK of ABSTRACTS

organized by  
Institute for Analytical Chemistry, Micro- and Radiochemistry,  
Graz University of Technology  
Austria



30<sup>th</sup> June to 3<sup>rd</sup> July 2002

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### Scope and Themes

The organizers invite you to participate in the international conference "Trends in Sample Preparation 2002" to be held in the castle of Seggau/Styria from 30<sup>th</sup> June to 3<sup>d</sup> July 2002.

The Conference has the intention to provide a platform for the discussion of modern developments in sample preparation. The scientific program is dedicated to all chemists working on topics associated with sample decomposition, solvent extraction, solid phase extraction, derivatization techniques and uncertainty in sample preparation with special regards to systematic errors. In particular microwave assisted sample preparation techniques and equipments will be discussed extensively.



### International Scientific Committee:

**K.-S. Eoos**, Technical University of Munich, Germany

**I. Brenner**, Environmental Analytical Laboratory, Jerusalem, Israel

**O. Ponard**, University of Pau, Pau, France

**W. Goessler**, Karl-Franzens University of Graz, Graz, Austria

**H.M. Kingston**, Duquesne University, Pittsburgh, USA

**G. Knapp**, Graz University of Technology, Graz, Austria

**E.P. Lankmayr**, Graz University of Technology, Graz, Austria

**H. Matusiewicz**, Politechnika Poznanska, Poznan, Poland

**J. Nobrega**, Universidade Federal de Sao Carlos, Brazil

**P. Schramel**, National Research Center for Environment and Health, Munich, Germany

**Organizing Committee:**

T. Wenzl (Conference Secretary and Coordinator): mobile phone: ++43-(0)664-4323684

M. Eberl (Secretary): mobile phone: ++43-(0)664-4323806

M. Zischka

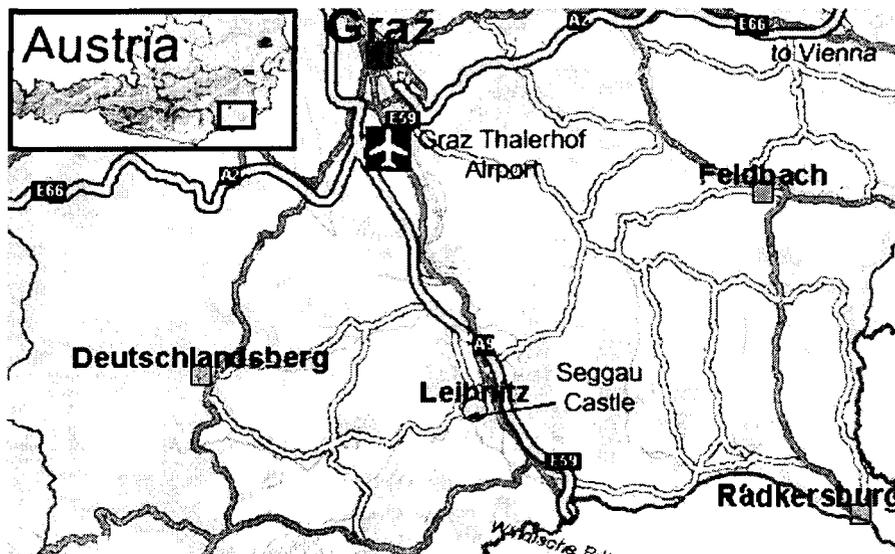
G. Knapp (Chairman)

**Location:**

The conference centre is to be reached over the highway A9 via the exit Leibnitz.

The next airport in Austria is Graz, which is located about 40 km from the conference venue.

Transportation from the airport and from the train station Graz to Seggau Castle will be organized.



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**Sunday, 30<sup>th</sup> June, 2002**

18:30 Welcome Cocktail and Buffet Dinner with Styrian Wine

**Monday, 01<sup>st</sup> July, 2002**

08:15 Opening Ceremony

**Trace Element Analysis**

Chair: G. Knapp

08:50 (M 1) State-of-the-Art Sample Preparation for Trace Analysis

Ramon Barnes

University Research Institute for Analytical Chemistry,

Amherst, Mass, USA

09:30 (M 2) A Critical Review of Sample Preparation Strategies for Multielement Analysis of Geoenvironmental Samples Using Inductively Coupled Plasma Atomic Emission and Massspectrometry

Joe Brenner

Environmental Analytical Laboratory Jerusalem

Jerusalem, Israel

10:00 Coffee Break

10:30 (M 3) A Universal Digestion Procedure for the Determination of Metals in Geological Matrices

Nicolas Bloom

Frontier Geoscience Research and Consulting

Seattle, WA, USA

10:50 (M 4) Ultrasonic Assisted Extraction - An Alternative for Sample Preparation

Francisco J. Krug

CENA, University of Sao Paulo

Piracicaba, Brazil

11:10 (M 5) Use of Tertiary Amines to Neutralize Hydrofluoric Acid Digests for ICP-OES Analysis of Silicon

Mark Tatro

Spectra Spectroscopy & Chromatography Specialists

Warwick, NY, USA

11:30 (M 6) Water-Soluble Tertiary Amines for Sample Preparation in Atomic Spectroscopy: An Overview

Joaquim Nobrega

University of Sao Carlos

Sao Carlos, Brazil

11:50 (M 7) Critical Evaluation of Pressurized Microwave-Assisted Digestion Efficiency Using Nitric Acid Oxidizing Systems  
Henryk Matusiewicz  
Department of Analytical Chemistry  
Poznan University of Technology  
Poznan, Poland

12:10 (M 8) Focused-Microwave-Assisted Sample Preparation  
Joaquim Nobrega  
University of Sao Carlos  
Sao Carlos, Brazil

12:30 Lunch

### Trace Analysis of Organic Compounds

Chair: E. Lankmayr

14:00 (M 10) State-of-the-Art in Solid Phase Extraction of Biofluids  
Karl Siegfried Boos  
Institute of Clinical Chemistry, University Hospital Grosshadern, Munich, Germany

14:40 (M 11) Evaluation of a Fast Sample Extraction Protocol for the Determination of the Oil Content of Pumpkin Seeds  
Thomas Wenzl  
Institute for Analytical Chemistry, Micro- and Radiochemistry,  
Graz University of Technology  
Graz, Austria

15:00 (M 12) Extraction of Pharmaceuticals from Waters by Use of Natural and Liquid-Membrane Systems  
Mahmoud Bataineh  
ISAS  
Dortmund, Germany

15:20 Coffee Break

15:50 (M 13) Direct Analysis of Basic Drugs in Rat Plasma Using Cation/Anion On-line SPE Sorbent by LC/MS/MS  
Claude Mallet  
Waters Corporation  
Milford, Mass, USA

16:10 (M 14) Comparative Study of Modern Extraction Techniques for the Determination of Environmental Samples  
Marion Gfrerer  
Institute for Analytical Chemistry, Micro- and Radiochemistry,  
Graz University of Technology

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- 16:30 (M 15) Microwave Accelerated Extraction of Fat from Food Samples without Co-Solvents  
Axel Schöner  
CEM GmbH  
Kamp-Lintfort, Germany
- 16:50 (M 16) Evaluation of Solid Phase Extraction Protocols for Isolation of Analgesic Compounds from Biological Fluids Prior to HPLC determination  
Victoria Samanidou  
University of Thessaloniki  
Thessaloniki, Greece
- 18:00 Departure for visiting a "Buschenschenke"

**Tuesday, 02<sup>nd</sup> July, 2002****High Performance Instrumentation in Sample Preparation - Part 1**

Chair: R. Barnes

- 08:50 (T 1) The Future of Microwave Chemistry  
Michael J. Collins  
CEM  
Matthews, NC, USA
- 9:30 (T 2) Integrated Microfabricated Biodevices: New Advances in Sample Preparation  
Andras Guttmann  
Torrey Mesa Research Institute  
San Diego, CA, USA
- 10:00 Coffee Break
- 10:30 (T 3) Coherent Synthesis with Dedicated Instrumentation for MW-Assisted Chemistry  
Martin Keil  
Personal Chemistry GmbH  
Konstanz, Germany
- 10:50 (T 4) Microwave Assisted UV-Digestion  
Michael Zischka  
Institute for Analytical Chemistry, Micro- and Radiochemistry  
Graz University of Technology  
Graz, Austria
- 11:10 (T 5) High Performance Flow Digestion Systems  
Günter Knapp  
Institute for Analytical Chemistry, Micro- and Radiochemistry  
Graz University of Technology  
Graz, Austria

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- 11:30 (T 6) Open Vessel Microwave Digestion of Food Matrices  
Axel Schöner  
CEM GmbH  
Kamp-Lintfort, Germany
- 11:50 (T 7) Improving Methodology in Open Vessel Digestion with a Graphite Heating Block  
Peter Kainrath  
S•prep GmbH  
Überlingen, Germany
- 12:10 (T 8) Simplified Polymer Characterization after Microwave Assisted Sample Preparation  
Markus Lafer  
Anton Paar Company  
Graz/Austria
- 12:30 Lunch
- High Performance Instrumentation in Sample Preparation –Part 2**  
Chair: J. Nobrega
- 14:00 (T 9) Trends and Applications of Integrated Automated Ultra-Trace Sample Handling and Analysis  
Skip Kingston  
Duquesne University Pittsburgh and Metara Inc.  
Sunnyvale, CA, USA
- 14:40 (T 10) A New High-Throughput Reactor for Improved Performance in Microwave Closed Vessel Acid Digestion  
Camillo Pirola  
Milestone  
Sorisole, Italy
- 15:00 (T 11) Open Vessel Digestion in a Closed System – New Developments for the HPA-System  
Peter Kettisch, Brigitte Maichin, Günter Knapp  
Anton Paar GmbH  
Graz University of Technology  
Graz, Austria
- 15:20 Coffee Break
- 15:50 (T 12) Accelerated Solvent Extraction (ASE) – a Fast and Automated Technique with Low Solvent Consumption for the Extraction of Solid Samples  
Frank Höfler  
Dionex Corporation Europe  
Olten, Switzerland

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- 16:10 (T 13) Comprehensive 2D Capillary LC as Front End Technique for MS in Proteomics  
Goran Mitulovic  
LC Packings-A Dionex Company  
Amsterdam, Holland
- 16:45 Guided Tour through the Castle
- 18:00 Dinner
- 19:30 Wine Tasting in an old Wine Cellar

**Wednesday, 03<sup>rd</sup> July, 2002**

**Speciation Analysis**

Chair: W. Goessler

- 08:50 (W 1) Advances in Modern Sample Preparation Techniques Using Microwaves Assisted Chemistry for Metal Species Determination

Olivier Donard

University of Pau

Pau, France

- 09:30 (W 2) Complete Extraction of Arsenic Species from Biological Samples – a Worthwhile Goal?

Kevin Francesconi

Institute of Chemistry-Analytical Chemistry

Karl-Franzens-University Graz

Graz, Austria

- 09:50 (W 3) Effects of Extraction and Derivatization Techniques on Recovery of Organotin Species in Environmental Matrices

Raimund Wahlen

LGC Limited

Teddington, Great Britain

10:10 Coffee Break

10:40 Poster Discussion

Chair: J. Brenner

12:30 Lunch

**General Topic**

Chair: P. Schramel

14:00 (W 4) Compromises Between Representativeness of Sample Preparation and Quality of Analytical Data as Exemplified by CRM Experience

Herbert Muntau

GSE

Munich, Germany

14:30 (W 5) Traceability and Measurement uncertainty in Sample Preparation

Wolfgang Wegscheider

Montanuniversität Leoben

Leoben, Austria

15:00 (W 6) Recovery of Total Hg from Geological Matrices: A Methodological Intercomparison Study

Nicolas Bloom

Frontier Geoscience Research and Consulting

Seattle, WA, USA

15:20 (W 7) So you've got your Sample in Solution: What Next?

Ian Brindle

Brock University

St. Catharines, Ontario, Canada

15:40 Closing Ceremony

16:00 Coffee Break

16:30 Departure for sight seeing in Graz

19:00 Bus Transfer to Seggau Castle

20:00 Conference Dinner in Seggau Castle

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Posters are displayed on Monday, Tuesday and Wednesday and are discussed in the lecture hall on Wednesday from 10:40 till 12:30.

**Posters:**

(P. 1) Sample Preparation with Microwave: Experiences in the Environmental- and Industrial Analytics Laboratory of Voestalpine

Andreas Schönauer, Klaus Hammerlitz, Gerhard-Christian Kastner  
Voestalpine Stahl GmbH  
Linz, Austria

(P. 2) Comparison of Two Microwave-Assisted Sample Digestions of Sediment and Soils for Trace Metals Using ICP-MS: HNO<sub>3</sub> and HNO<sub>3</sub>/HF

Crystal Rose Howard, A. Vandervort, Nicolas Bloom  
Frontier Geosciences  
Seattle, WA, USA

(P. 3) Analysis of Soil and Sewage Sludge by ICP-OES and the German Standard DIN 38414 Sample Preparation Techniques

Maria Edlung, Peter Heitland, Henk Visser,  
Spectro Analytical Instruments GmbH, Kleve  
Germany

(P. 4) Direct Analysis of Iron in Sand by Solid Sampling Electrothermal Atomic Absorption Spectrometry

Pedro Oliveira, Cintia Silva, Cassiana Nomura, Elisabeth de Oliveira, Joaquim Nobrega  
University of Sao Paulo  
Sao Paulo, Brazil

(P. 5) Combination of Sophisticated Sample Digestion Methods and Highly Accurate Determination Procedures Exemplified for Sulphur in Oil and Cadmium and Lead in Sediment

Gundel Riebe, Markus Ostermann, W. Pritzkow, J. Vogel  
Federal Institute for Materials Research and Testing  
Berlin, Germany

(P. 6) On the Determination of the Moisture content in Different Matrix Materials

Markus Ostermann, Nadine Janisch, Gundel Riebe, J. Vogel  
Federal Institute for Material Research and Testing  
Berlin, Germany

(P. 7) A Novel Rapid and Simple Method of Sample Preparation for LC/MS Analysis of Carbohydrates from Plasma

Eduard Rogatsky, Daniel Stein  
Albert Einstein College of Medicine  
New York, NY, USA

(P 8) Microwave Assisted Digestion of Organoarsenic Compounds for the Determination of total Arsenic in aqueous, biological, and sediment Samples using EL-HG-ETAAS

K. Boch, M. Schuster  
Technical University, Munich  
Germany

(P 9) Solid Phase Microextraction and Stir Bar Sorptive Extraction for Organotin Compounds – a Comparison

Sibylle Mothes, R. Wennrich  
UEZ Centre for Environmental Research  
Leipzig, Germany

(P 10) Determination of Polycyclic Aromatic Hydrocarbons from Pumpkin Seeds and Pumpkin Seed Oils by Microwave-Assisted Saponification

Marion Gfrerer, Ernst Lankmayr  
Graz University of Technology  
Graz, Austria

(P 11) Membrane extraction with a sorbent interface-gas-chromatography as effective and fast means for continuous monitoring of thermal degradation products of polymers.

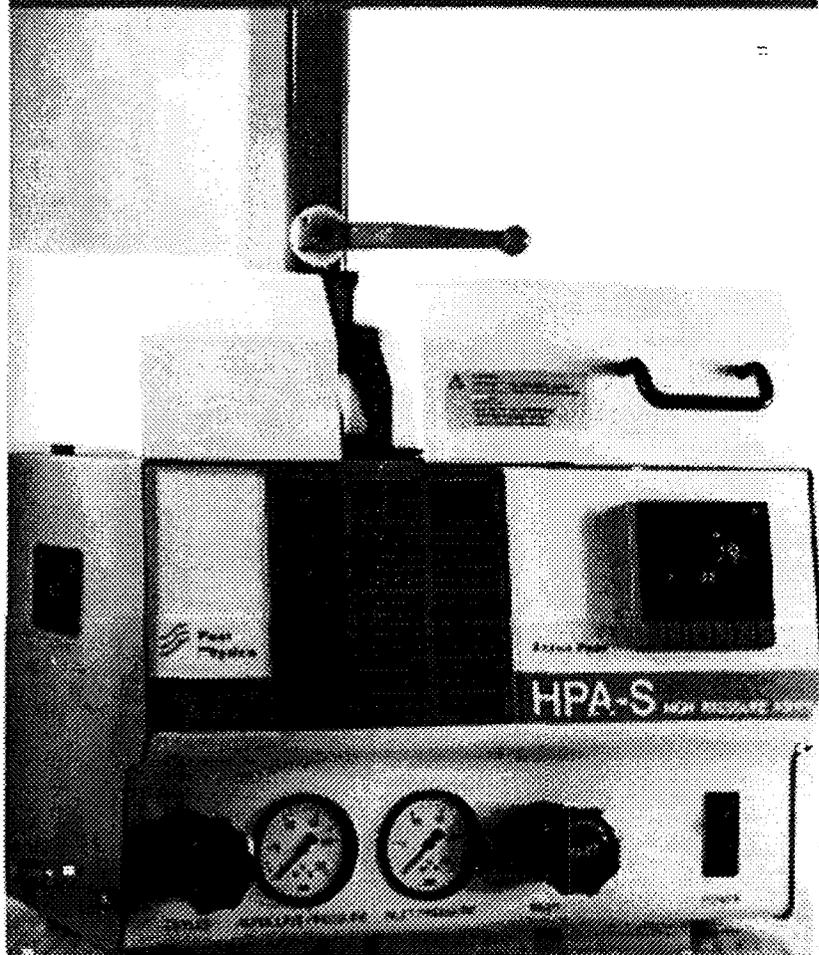
Kaykhail Massoud



**Anton Paar**

**HPA-S**

The High Pressure Asher



The High Pressure Asher HPA-S is the reference instrument for wet chemical pressure decomposition. The instrument is TÜV certified and can prepare even the most difficult samples for reliable AAS, ICP-OES, ICP-MS and voltammetric analysis.

Please e-mail us to: [info@anton-paar.com](mailto:info@anton-paar.com) or visit our homepage: [www.anton-paar.com](http://www.anton-paar.com) or send a fax to: 0043-316-257 9131

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ABSTRACTS of LECTURES

TRISP 2002

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**STATE OF THE ART IN SAMPLE PREPARATION FOR TRACE  
ELEMENT ANALYSIS (M 1)****Ramon M. Earnes***University Research Institute for Analytical Chemistry, 85 N. Whitney Street, Amherst, MA  
01002-1869*

The accelerated capabilities of modern trace element analysis techniques, especially inductively coupled plasma mass spectrometry (ICP-MS), have challenged the sample preparation competence of most laboratories. Exceptional analytical sensitivity, remarkable analysis speed, automated sample presentation, and intelligent sample sequencing of modern spectroscopic instrumentation have led to demanding requirements for appropriate sample preparation steps needed for ultratrace concentration and speciation measurements. Contamination control, reliable digestion and extraction techniques, preservation of chemical forms, sample matrix management, and intelligent sample processing available today are often inadequate for the most demanding measurements. Some commercial instrumentation provides convenient implementation of well-established contamination control measures, and reagent and container purity are steadily being improved. Direct sample introduction approaches offer alternatives to conventional solution samples, but achieving calibration reliability is difficult. Developing new sample preparation chemistry is especially arduous and rare, yet progress exists in characterizing microwave-assisted reactions.

This presentation will describe contemporary targets for modern sample preparation approaches for ultratrace elemental analysis and the likelihood that they can be reasonably achieved.

13. März 2003



**A CRITICAL REVIEW OF SAMPLE PREPARATION STRATEGIES  
FOR MULTIELEMENT ANALYSIS OF GEOENVIRONMENTAL  
SAMPLES USING INDUCTIVELY COUPLED PLASMA ATOMIC  
EMISSION AND MASS SPECTROMETRY (M 2)**

**I.P.BRENNER**

*Environmental Analytical Laboratory, BGU, 9 Dishon Street, Malkha, Jerusalem, Israel.  
Brenner@cc.huji.ac.il. Tel 972-2-6797-255, FAX 972-2-6797-145*

Inductively coupled plasma atomic emission and mass spectrometries (ICP-AES and ICP-MS) are now widely employed for multielement analysis of geoenvironmental samples. Reliable analyses of these complex materials depend on efficient sample preparation routines. Numerous approaches using mixed mineral acids in open and closed vessels, microwave and high-pressure devices have been documented. In general these procedures are characterized by low contamination but have low sample throughput. Furthermore, low recoveries for analytes accommodated in resistant mineral species have been reported. This chemical resistance has been overcome using alkali fusion and sinters. However blanks can be high and the final solutions have high salt contents that interfere in aerosol generation and transport, in the plasma and plasma-mass spectrometer interface.

While it is customary to judge the efficacy of sample preparation strategies in terms of recovery, throughput, minimum contamination, safety, and cost, the all-important effects of the final sample solution and reagent concomitants on basic instrumental performance should also be addressed.

In this presentation we will provide a brief overview of sample preparation approaches and then examine spectroscopic effects (spectral interferences and polyatomic ions) and nonspectroscopic effects such as those that occur in sample introduction systems, in the plasma and in the plasma-MS interface. Several real sample situations will be given using ICP-AES and MS and sample preparation will be appraised in terms of instrumental analytical figures of merit.

13. März 2003



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**A UNIVERSAL DIGESTION PROCEDURE FOR THE  
DETERMINATION OF METALS IN GEOLOGICAL MATRICES (M 3)****N.S. Bloom and C. Howard***Frontier Geosciences Inc. 414 Pontius North Seattle, WA 98109.*

Quantification of all metals in minerals often requires multiple digestion procedures. The lanthanides, most of the alkaline earths, Al and Th form insoluble fluorides, which render them difficult to recover by HF based digestions. Precipitated metal fluorides can also scavenge other metals, such as Mn and Cr, sporadically reducing their recoveries. Digestions containing HCl can result in low recoveries of Ag, due to precipitation of AgCl, while mixtures that do not contain HCl cannot quantitatively extract platinum group metals. Methods which bring the digest to dryness risk volatilizing Hg and Se, while fusion based methods have higher detection limits owing to high salt contents. We have optimized a single digestion procedure which overcomes these obstacles, and allows quantitative determination of virtually all trace metals by spectrometric means. Samples are pulverized, and 500 mg aliquots digested in Teflon bombs with a 15:7:3 mixture HNO<sub>3</sub> + HF + HCl. The bombs are digested overnight, in batches of 48, at 125 ± 5°C. After dilution to 100 mL, aliquots are taken for Hg and Se analysis by cold vapor techniques. The remaining digests are then evaporated to dryness in Teflon beakers on a hotplate held at 225°C in a vented clean hood. The residues are re-wetted with 25 mL of concentrated HNO<sub>3</sub>, brought to dryness again, and then redissolved in 5 mL of hot concentrated HNO<sub>3</sub>. After redissolution, the samples are diluted to 40 mL with 5% HNO<sub>3</sub>. The remaining metals are then analyzed after appropriate further dilution by ICP/MS or other technique.

13. März 2003



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## ULTRASONIC ASSISTED EXTRACTION – AN ALTERNATIVE FOR SAMPLE PREPARATION (M 4)

**Páριο Santos Júnior<sup>1</sup>, Fernando Barbosa Júnior<sup>1</sup>, Lilian Cristina TREVIZAN<sup>2</sup>,  
Joaquim A. Nóbrega<sup>2</sup>, Francisco José Krug<sup>1\*</sup>**

<sup>1</sup> *Centro de Energia Nuclear na Agricultura – USP, Av. Centenário 303, Piracicaba, SP,  
Brazil - Caixa Postal 96, CEP 13400-970, \* (fjkrug@cena.usp.br)*

<sup>2</sup> *Departamento de Química, Universidade Federal de São Carlos, São Carlos, SP - Brazil*

In the last years the ultrasound assisted metal extraction has been frequently proposed as a simple and inexpensive alternative for sample preparation of biological and inorganic samples. The extraction effect is considered as being caused by acoustic cavitation, that is, bubble formation and subsequent disruptive action. The collapse of bubbles created by sonication of solutions results in the generation of extremely high local temperature and pressure gradients, which may be regarded as localized “hot spots”. On a timescale of about  $10^{-10}$  s, effective local pressures and temperature of about  $10^5$  atm and about 5000 K, respectively, are generated under sonochemical conditions. Usually, this method uses a diluted acid medium decreasing blank values and reducing both reagents and time consumption compared to traditional wet digestion systems using conductive or microwave-assisted heating. Furthermore, sonication can also allow the preparation of samples directly within the sample container, thereby preventing sample losses and minimizing sample contamination. Although some controversial results concerning metals extraction behaviour have been reported, they could be explained by analyte-matrix interaction and the ability of the ultrasonic processor to generate ultrasound (*i. e.* the use of an ultrasonic bath or an ultrasonic probe at different power, frequency, and amplitude).

This contribution presents a review of ultrasound assisted metal extraction and recent performance data obtained in our laboratory for determination of elements in biological materials, soils and sediments by ICP-OES and ETAAS. The effect of extraction parameters, such as type and concentration of the leaching solution, sonication time and performance of ultrasonic processor (bath or probe) will be presented.

13. März 2003

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ABSTRACTS of LECTURES

TRISP 2002

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**USE OF TERTIARY AMINES TO NEUTRALIZE HYDROFLUORIC  
ACID FOR THE ICP-OES ANALYSIS OF SILICON (M 5)**

**Mark E. Tatro**

*Spectra Spectroscopy & Chromatography Specialists, Inc., P.O. Box 1126, Warwick, NY USA  
10990 -1126*

The ICP analysis of hydrofluoric acid digests is complicated by the need to remove free fluoride aqueous ions by either volatilization or complexation prior to aspiration through a quartz transport system. Volatilization of free fluoride requires a laborious and dangerous technique involving the use of perchloric acid with evaporation to near dryness which results in the loss of silicon. Complexation of free fluoride requires the use of boric acid which not only tends to clog quartz concentric nebulizers but also does not completely neutralize fluoride which results in variable blanks for silicon.

W.R. Grace's analytical method for the analysis of major and minor elements in zeolites, catalysts and clays calls for the volatilization of free fluoride which required that the concentration of silicon be calculated by difference. To increase accuracy, Grace sought a method to easily analyze for silicon.

The method chosen is one in which the silicate samples were dissolved in Teflon pressure digestion vessels using a combination of nitric – hydrochloric – hydrofluoric acids. The digestates were neutralized using the Spectrasol tertiary amine reagent which deactivates all free fluoride. The resulting samples were aspirated into an ICP using a quartz concentric nebulizer for analysis of silicon.

Data correlating the ICP results with more conventional methods will be presented and discussed.

13. Mar 2003



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**WATER-SOLUBLE TERTIARY AMINES FOR SAMPLE PREPARATION IN ATOMIC SPECTROSCOPY: AN OVERVIEW (M 5)**

**Joaquim A. Nóbrega<sup>1</sup>, Daniele M. Santos<sup>1</sup>, Ramon M. Barnes<sup>2</sup>, Poliana C. Aleixo<sup>1</sup>, Ana Rita A. Nogueira<sup>3</sup> and Mark Tatro<sup>4</sup>**

*Departamento de Química, Universidade Federal de São Carlos, São Carlos, SP, Brasil  
University Research Institute for Analytical Chemistry, 85 N. Whitney Street, Amherst, MA  
01002, USA*

*Embrapa Pecuária Sudeste, São Carlos, SP, Brasil  
Spectrasol Inc., Warwick, NY, USA*

Despite the predominance of wet acid digestion methods, would it not be more appropriate to carry out sample preparations in alkaline medium? Alkaline digestion procedures, however, will not become as general as acid digestions owing to their chemical properties, such as the cation hydrolysis at high pH's or for some easily hydrolysable cations even in less acidic solutions. Nevertheless, for some elements and samples, the use of an alkaline medium should be preferred for sample preparation, owing to solubility, matrix and volatility characteristics. This work focuses on the use of water-soluble tertiary amines for sample preparation for atomic spectroscopy. A commercial mixture of tertiary amines called CFA-C (Spectrosol, Warwick, NY) was initially proposed to neutralize hydrofluoric acid. Subsequently, its application has been extended in atomic spectroscopy. Beneficial effects on sample nebulization, isobaric interferences, and sensitivities are observed for inductively coupled plasma mass spectrometry (ICP-MS) and optical emission spectrometry (ICP-OES). Efficient extraction of elements in animal and vegetable tissues, dilution of milk samples, and improvement of automatic sampler action in graphite furnace atomic absorption spectrophotometry also are achieved. These spectroscopic applications are discussed and the main effects are critically reviewed.

13. März 2003



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**CRITICAL EVALUATION OF PRESSURIZED MICROWAVE-  
ASSISTED DIGESTION EFFICIENCY USING NITRIC ACID  
OXIDIZING SYSTEMS (M 7)**

**Henryk Matusiewicz**

*Politechnika Poznańska, Department of Analytical Chemistry, 60-965 Poznań, Poland*

The possibilities of enhancement of a medium-pressure microwave-assisted digestion system for sample preparation in trace element analysis of biological material was investigated. Based on optimal digestion conditions for oxidizing systems with nitric acid, different digestion procedures were examined to minimize residual carbon. The substitution of nitric acid and the addition of hydrogen peroxide and ozone to nitric acid was evaluated. The residual carbon content of the digestate was determined coulometrically. Addition of hydrogen peroxide during organic oxidation reactions does not lower the dissolved carbon in the solution. Ozone was tested as an additional, potentially non-contaminating, digestion/oxidation system to the nitric acid used in the sample preparation method.

05. Marz 2003

**FOCUSED-MICROWAVE-ASSISTED SAMPLE PREPARATION (M 8)**

**Joaquim A. Nóbrega<sup>1</sup>, Paniele M. Santos<sup>1</sup>, Lilian C. TREVIZAN<sup>1</sup>, Letícia M. Costa<sup>1</sup> and Ana Rita A. Nogueira<sup>2</sup>**

*Departamento de Química, Universidade Federal de São Carlos, São Carlos, SP, Brasil  
Embrapa Pecuária Sudeste, São Carlos, SP, Brasil*

Focused-microwave-assisted sample preparation is a suitable strategy when dealing with high masses of organic samples. However, the final acid concentration of the digestate can difficult routine analytical measurements using spectroscopic techniques. Acids could be evaporated, but this step could be slow even when using microwave-assisted heating and requires a scrubber system for acid vapor collection and neutralization. We are investigating two procedures to decrease the acid concentration of digestates. The first one is based on acid vapor phase digestion of samples contained in PTFE devices<sup>1</sup> inserted into the microwave flask. The acid solution is heated by absorption of microwave radiation, then the acid vapor partially condenses in the upper part of the reaction flask and it is partially collected in each sample container. Calcium, Fe, Mg, Mn, and Zn were quantitatively recovered in samples of animal and vegetable tissues. Better recoveries were attained when adding a small volume of sodium hypochlorite to the sample. This effect is probably related to the generation of chlorine in the sample container after collecting condensed acid. The second procedure developed is based on the gradual addition of liquid samples to a previously heated acid digestion mixture. This procedure was successfully applied for digestion of milk, fruit juices, and red wine. The main advantage is the possibility of digesting up to four-fold more sample using up to ten-fold lower amounts of concentrated acids. Results obtained using both digestion procedures and measurements by ICP-OES with axial view will be presented.

1. Patent pending, INPI Brazil.

13. März 2003

**STATE-OF-THE-ART IN SOLID-PHASE EXTRACTION OF  
BIOFLUIDS (M 10)****Boos, K.-S., Fleischer, C.T.***Institute of Clinical Chemistry, University Hospital Grosshadern, D-81366 Munich, Germany*

A major drawback of many SPE protocols in bioanalytical LC is a rather unselective and/or time consuming clean-up procedure. In order to optimise SPE based sample processing with regard to selectivity and speed we investigated tailor-made SPE adsorbents, distinct SPE column hardware as well as a novel software (ChromSword<sup>®</sup>ADS, Merck KGaA) for LC(MS)-integrated SPE using column-switching.

Restricted Access Materials (RAM) are one family of such special SPE column packings. RAMs are characterized in that they have a defined size-selective exclusion barrier and a non-adsorptive outer particle surface towards macromolecular matrix components (e.g. proteins, nucleic acids, polysaccharides). Low-molecular compounds (e.g. drugs) have free access to the binding centres and thus can be extracted and enriched prior to their analytical separation and MS detection. A RAM-SPE column (e.g. LiChrospher<sup>®</sup>ADS, Merck KGaA, Germany) allows the direct, multiple injection and fractionation of crude, complex samples such as hemolysed blood, plasma, serum, milk, saliva, urine, fermentation broth and cell-culture or tissue homogenate supernatant [1].

In order to improve the selectivity, we extended this on-line sample clean-up protocol by coupling a reversed-phase modified RAM-SPE column with a second short column packed with a Molecularly Imprinted Polymeric (MIP) adsorbent. MIP-SPE adsorbents can be compared with immunoaffinity adsorbents. Besides selective/specific molecular recognition properties, these artificial antibodies exhibit a high stability with respect to pressure, pH, temperature as well as organic solvents. These innovative adsorbents are also attractive because of the ease, low-cost and high reproducibility of preparation [2,3]. Using MIPs in combination with RAMs we developed a generic SPE platform and protocol, respectively, for highly selective clean-up of complex (bio)fluids [4,5]. Finally, we succeeded in speeding up the RAM fractionation step (e.g. extraction of 50 µl of human plasma in less than 60 seconds) by applying an optimised column size and flow-rate, respectively [6]. In conclusion, tailor-made SPE-column packings and hardware allow a robust and rugged operation of on-line (high speed) SPE-(LC)-MS(MS).

- [1] Boos, K.-S., Grimm, C.-H., TrAC 18 (1999) 175
- [2] Sellergren, B., TrAC 16 (1997) 310
- [3] Takeuchi, T., Haginaka, J., J. Chromatogr. B 728 (1999) 1
- [4] Boos, K.-S., Fleischer, C.T., Fresenius J. Anal. Chem. 371 (2001) 16
- [5] Koeber, R. et al, Anal. Chem. 73 (2001) 2437
- [6] Boos, K.-S., Fleischer, C.T., Chimia 55 (2001) 42

13. März 2003

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**EVALUATION OF A FAST SAMPLE EXTRACTION PROTOCOL FOR  
THE DETERMINATION OF THE OIL CONTENT OF PUMPKIN  
SEEDS (M 11)**

**T. Wenzl, E.P. Lankmayr**

*Institute for Analytical Chemistry, Micro- and Radiochemistry, Graz University of  
Technology, Technikerstr. 4, 8010 Graz, Austria*

Pumpkin seed oil pressed from the seeds of *Cucurbita pepo L.* convar: *citrullina I.GREB.* var. *styriaca* is widespread used as salad oil in the south of Austria and the adjacent regions in Slovenia and Hungary. These country sides are the main cultivation areas of pumpkins in Europe too. More than two thousand Styrian farmers cultivate pumpkins and sell their seeds to professional oil mills. They are dried, milled and roasted there prior to the pressing step.

The amount of oil, which may be obtained from the seeds, is primarily determined by the degree of ripeness of the pumpkins. Secondly roasting parameters determine the quality and the yield of the oil. Even experienced oil millers have problems in estimating the exact oil content of the pumpkin seeds, which implies a residual financial risk in the purchase of the raw material.

Different methodologies may be applied for the determination of the oil content. The simplest method, regarding the equipment, is gravimetric analysis of the residue after Soxhlet extraction of the grinded seeds. This is also the standard method according to DGF, the German Society of Fat Research. The most specialized equipment is known as "FastFat HT", a supercritical carbon dioxide applying extraction system.

An alternative is the fluidized bed extractor fexIKA 200<sup>®</sup>, which combines fast extraction with low initial costs and wide range applicability. The results of analysis obtained with an optimized extraction protocol will be presented and compared to those which were gained by the standard method.

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**EXTRACTION OF PHARMACEUTICALS FROM WATERS BY USE OF  
NATURAL FLAT- AND LIQUID- MEMBRANE SYSTEMS (M 12)**

M. Eataineh<sup>1</sup>, M. Grote<sup>2</sup>, E. Haciosmanoglu<sup>2</sup>, J. Nolte<sup>1</sup>

<sup>1</sup> ISAS, Institute of Spectrochemistry and Applied Spectroscopy, 44139 Dortmund,  
Bunsen-Kirchhoff-Straße 11, FRG

<sup>2</sup> University of Paderborn, Analytical Chemistry, 33098 Paderborn, Warburgerstr. 100,  
FRG

Residues of pharmaceuticals and their metabolites from human and animal applications can be found in all aquatic compartments. Meanwhile, because of the enormous variety technical membranes have become a standard method for water cleaning processes. To improve these techniques for analytes in the lower molecular range an enrichment procedure by means of animal intestines with regard to the permeation characteristics and the separation mechanism should be developed. Thereby, the development should take into account both, the application for the technical water treatment and as an analytical sample enrichment device.

In this study devices for the enrichment of selected pharmaceuticals such as sulfamethoxazol, carbamazepine, diclofenac, and ibuprofen by means of natural intestine and liquid membranes were developed. Different types of intestine parts of cattle, sheep and mainly pig were applied in the original wet or dried form. The flat membranes were mounted in a window between two teflon chambers, one for the sample input and one for the permeate uptake. The solutions in the chambers are stirred. Parallel bulk-membrane experiments were carried out in a three-compartment transport cell equipped with an agitator. The liquid membrane system consists of a feed solution containing the analyte, a liquid membrane (organic solvent combined with a mobile carrier) and an aqueous stripping solution (e.g. mineral acids, salts or alkalies).

The first results presented raise hope that both systems will become proper devices for the separations of pharmaceuticals from aquatic compartments.

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**DIRECT ANALYSIS OF BASIC DRUGS IN RAT PLASMA USING  
CATION/ANION  
ON-LINE SPE SORBENT BY LC/MS/MS (M 13)**

**C. Mallet**

*Waters Corporation, Milford, Massachusetts, USA*

During the last decade, an increasing numbers of pharmaceutical companies upgraded their laboratories with automated hyphenated instrumentation (LC/MS/MS). This trend was sparked by the demands for faster analysis and lower detection limits for drug analysis in various matrices. Consequently, to keep a constant feed to an LC/MS/MS system, sample preparation protocols quickly became an overwhelming bottleneck. New techniques, such as 96 well plates, fast gradients or ultra-high-flow chromatography, alleviated this lack of speed, but are now being rapidly surpassed by on-line extraction techniques capable of breaching the one-thousand-analyses-per-day barrier. The on-line extraction technique is particularly well suited for high throughput analysis [1-3]. This method combines the extraction, clean up and analysis using automated events either in sequential or parallel mode, and is capable of reducing the analysis time to the one-minute range. However, to achieve this level of analysis speed, it may require using complex valve schemes. Recently, we studied several on-line extraction configurations using standalone pumps and 10-port switching valves [4]. The extraction of plasma samples was performed using cartridges filled with Oasis HLB sorbent. The first extraction step uses a 100 % aqueous mobile phase, set at high flow rate (i.e. 4 mL/min). This ensures the removal of macromolecules (i.e. proteins), but traps the smaller analytes on the head of the column. Next, the cartridge is eluted using a gradient or an isocratic mobile phase with a compatible mass spectrometer flow rate (0.4 mL/min). This approach is usually used for a fast turn around, consisting of a maximum of two steps processes (load and elution). For lower LOQs, additional washing steps can be added to achieve better levels of clean up. However, the use of mixed mode cation/anion sorbents (MCX/MAX) in-line with an Oasis HLB cartridge can yield better clean up. With this type of configuration, it is possible to use stronger mobile phase to clean the reverse phase portion of the sorbents, while the drugs are safely retained on the ion exchanger. A four valves configuration using a two extraction columns was used for the analysis of basic drugs in rat plasma. The on-line extraction was performed on an MCX and HLB column (2.1 x 20 mm, 25 µm) using four Waters 515 pumps, a Waters 2700 auto sampler and a Waters 2690 in gradient mode. The extracted analytes were back flushed onto an XTerra, µ column (2.1 x 30 mm, 3.5 µm), added to provide additional separation power. The drugs were quantified using a Micromass Ultima triple quadrupole mass spectrometer equipped with an electrospray source and set in multiple reaction monitoring (MRM)

- [1]- Y.Q. Xia, D.B. Whigan, M.L. Powell, M. Jemal, *Rapid Commun. Mass Spectrom.*, Vol. 14, p. 105, 2000 [2]- J. Ayrton, G.J. Dear, W.J. Leavens, D.N. Mallett, R.S. Plumb, *J. of Chromatogr. A*, Vol. 828, p. 199, 2000 [3]- N.V. Eeckhout, J.C. Perez, J. Clearboudt, R. Vandeputte, C. Van Peteghen, *Rapid Commun. Mass Spectrom.*, Vol. 14, p. 280, 2000 [4] C.R. Mallet, J. R. Mazzeo, U.D. Neue, *Rapid Commun. Mass Spectrom.*, Vol. 15, p. 1075, 2001



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**COMPARATIVE STUDY OF MODERN EXTRACTION TECHNIQUES  
FOR THE DETERMINATION OF ENVIRONMENTAL SAMPLES  
(M 14)**

**Marion Gfrerer, Ernst Lankmayr**

*Institute for Analytical Chemistry, Micro- and Radiochemistry,  
Graz University of Technology, Technikerstr. 4/P, A-8010 Graz*

The aim of any extraction method in analytical chemistry is, to effectively separate the analytes from the matrix. The whole step should be fast and quantitative with minimal solvent and time required. The classical Soxhlet extraction usually requires large volumes (up to 200 mL) of solvent to be refluxed through the solid samples for several hours. Therefore, in the last decades, alternatives for this extraction method have been presented and investigated such as ultrasonic extraction (UE), supercritical fluid extraction (SFE), accelerated solvent extraction (ASE), microwave-assisted extraction (MAE) and fluidized-bed extraction (FBE). The actual choice for analytical application is frequently the initial capital cost, operating costs, simplicity of operation, amount of organic solvent required and sample throughput.

Since sample preparation is a critical step in the analytical cycle, special care has to be taken for an accurate choice and optimization of extraction techniques and clean-up procedures. Therefore, MAE and FBE were investigated for their influential extraction parameters and these parameters were optimized for the extraction of organochlorine biocides [1], polychlorinated biphenyls [2] and polycyclic aromatic hydrocarbons [3] from environmental matrices like soil, sediment and sewage sludge. The extraction yields were compared with those obtained by Soxhlet extraction performed following DIN-methods. Finally, the optimized modern methods were validated by systematic experiments with certified reference materials.

[1] Martens D, Gfrerer M, Wenzl T, Zhang A, Gawlik BM, Schramm K-W, Lankmayr E, Kettup A. *Anal Bioanal Chem* 372 (2002) 562-568

[2] Gfrerer M, Stadlober M, Gawlik BM, Wenzl T, Lankmayr E. *Chromatographia* 53 7/8 (2001) 442-446

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3 April 2003

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**MICROWAVE ACCELERATED EXTRACTION OF FATS WITHOUT  
CO-SOLVENTS (M 15)****Axel Schoener**

*European Manager  
CEM Corporation  
P.O. Box 200  
Matthews, NC 28106 USA*

Microwave Accelerated Extraction has proven to shorten the time required extracting analytes from solid matrices. By using extraction solvents above their normal atmospheric boiling points, Microwave Accelerated Extraction drastically reduces the volume of solvent used from 150 – 500mL to 20 – 50mL per sample. This paper demonstrates the application of Microwave Accelerated Extraction, in coordination with a novel sample stirring mechanism and the newly improved GreenChem *Plus* Glass Vessel Polymer samples. The newly improved glass vessels now incorporate temperature and pressure monitoring during the extraction.

This presentation will review the methods optimization process when using a closed vessel microwave accelerated extraction technique for the extraction of fat from food matrices using only non-polar solvents. Derivatization reactions will also be examined. It will focus on extraction temperature and time. Recovery data from “real world” and quality control samples will be presented comparing the microwave technique to the conventional Soxhlet technique.

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**EVALUATION OF SOLID PHASE EXTRACTION PROTOCOLS FOR ISOLATION OF ANALGESIC COMPOUNDS FROM BIOLOGICAL FLUIDS PRIOR TO HPLC DETERMINATION (M 16)**

**V. F. Samanidou, I. P. Imamidou and I. N. Papadoyannis**

*Laboratory of Analytical Chemistry*

*Department of Chemistry.*

*Aristotle University of Thessaloniki*

*GR-54006 Thessaloniki, Greece.*

*<http://www.chem.auth.gr/english/analytical/analytical.html>*

A comparative study of various sorbents, reversed phase silica based C<sub>18</sub>, C<sub>8</sub>, and copolymeric hydrophilic-lipophilic balanced, from different manufacturers and various eluting solvents (methanol, acetonitrile and isopropanol) was conducted for optimization of isolating the constituents of multi-component analgesic mixtures by means of Solid Phase Extraction (SPE). Optimized SPE protocols were subsequently applied to human serum and urine samples. Traditional minicolumns and disc formats of C<sub>18</sub> sorbent were studied as well. The effect of sorbent bed conditioning was also investigated when using novel copolymeric sorbent materials such as OASIS and NEXUS as both claim to function under no conditioned sorbent bed as being water wettable.

An analgesic mixture containing: paracetamol, caffeine and codeine was selected as a model for this survey, since they very often co-exist in pharmaceutical formulations. Analytes were monitored at 240 nm, after isocratic elution from a C<sub>8</sub> Inertsil analytical column. The mobile phase was a mixture of methanol and ammonium acetate (0.05 M) at a volume ratio of 40:60. Statistical evaluation revealed satisfactory accuracy, repeatability and intermediate precision. Pharmaceutical formulation analysis yielded high recoveries ranging from 95.4 to 107.5 %. Various recovery rates were obtained when the different protocols were applied. Reversed phase C<sub>18</sub> sorbent yielded a 80-90% recovery, while copolymeric sorbents reached the 100 % of analyte concentration in optimizing extraction conditions concerning the activation step and the eluting solvent.

AT0300297



AT0300298

ABSTRACTS of LECTURES

TRISP 2002

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## THE FUTURE OF MICROWAVE CHEMISTRY (T 1)

**Michael J. Collins, PhD**

*President, CEO  
CEM Corporation  
P.O. Box 200  
Matthews, NC 28106 USA*

Microwave Chemistry has become a key enabling technology for sample preparation. It is now used and accepted in laboratories throughout the world for a wide range of applications including both inorganic and more recently organic analysis. Technological advancements have continued to play a major role in the expanding use of microwaves.

This presentation will provide a historical prospective on the development of this important field of technology. It will focus on recent key developments, which have had a major impact expanding and enhancing the capabilities of microwave sample preparation. Some of the newer more difficult application areas will be discussed.

Microwave synthesis which is the fastest growing segment of microwave chemistry will be discussed with particular emphasis on various analytical applications including hydrolysis and derivitization. Finally, future trends will be discussed focusing on some potential technology and hardware breakthroughs that are expected to provide exciting new opportunities in the future.

13. März 2003



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**INTEGRATED MICROFABRICATED BIODEVICES: NEW  
ADVANCES IN SAMPLE PREPARATION (T 2)****Guttman, Andras***Torrey Mesa Research Institute, San Diego, CA 92121, USA*

Interdisciplinary science and technologies have converged in the past few years to create exciting challenges and opportunities, which involve novel, integrated microfabricated systems, facilitating large-scale analytical applications. These new devices are referred to as lab-on-a-chip or micro Total Analysis Systems (uTAS). Their development involves both established and evolving technologies, which include microlithography, micromachining, micro-electromechanical systems (MEMS) technology, microfluidics and nanotechnology. The advent of this extremely powerful and rapid analysis technique opens up new horizons in analytical chemistry and molecular biology, capable of revealing global changes in gene expression levels by enabling genome, proteome and metabolome analysis on microchips. This presentation will provide an overview of the key device subject areas and the basic interdisciplinary technologies. It will also give a better understanding of how to utilize these miniaturized technologies as well as to provide appropriate technical solutions to problems perceived as being more fundamental. Theoretical and practical aspects of integrating sample preparation/purification and analysis units with chemical and biochemical reactors in monolithic microdevices are going to be thoroughly discussed. Important applications for this novel "synergized" technology in high throughput analysis of biologically important molecules will also be addressed.

13. Marz 2003



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**COHERENT SYNTHESIS WITH DEDICATED INSTRUMENTATION  
FOR MW-ASSISTED CHEMISTRY (T 3)****Erwin Martin Keil***Personal Chemistry GmbH, Konstanz/Germany*

Microwave (MW) assisted organic chemistry is a still new and exciting field in organic synthesis. The streamlining power of this type of methodology is typically characterized by an increase in yield and a decrease in reaction times. Within quite a short time all major companies which need to synthesize new compounds efficiently and successfully has realized these advantages and have invested in this technology.

The instruments presented here are specifically designed for organic synthesis. They incorporate Single Mode Resonator (SRM) and Dynamic Field Tuning designs. This latest developments in modern microwave technology is used due to the high demand for reproducible control and the wide variety of reaction types used in organic synthesis. In Coherent Synthesis, microwave assisted organic reactions can be performed under extreme temperature and pressure conditions. It is therefore essential to have sensitive control and advanced, adjustable energy-steering systems. The Dynamic Field Tuning system is based upon proprietary technology involving improved software and hardware. The system is capable of detecting the absorbance characteristics of the reaction mixture and optimises the coupling and quantity of energy delivered. It provides optimal efficiency and even temperature distribution in the reaction mixture regardless of involved materials, as e.g. a wide range of solvents or reagents.

Built-in temperature and pressure sensors allow real time monitoring and control of each individual reaction. The Workflow Manager software is a platform from where to plan and perform experiments, monitor reactions and document all results.

The chemical reactions are performed in uniquely designed process vials which allow even energy distribution throughout the entire reaction volume. The vials are manufactured from microwave-immune materials that are free from contaminants. The closure design is optimised for a complete seal allowing safe working pressures up to 20 bars. The reaction volumes fit excellent to the demands of reaction methods development and sample preparation needs.

All together the instrumentation has been developed to ensure excellent reproducible reactions and short reaction times under highly safe conditions in a very convenient manner.

13. März 2003



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**MICROWAVE ASSISTED UV-DIGESTION****M. Zischka, H. Motter, P. Maichin and G. Knapp (T4)**

*Institute for Analytical Chemistry, Micro- and Radiochemistry, Graz University of  
Technology, Technikerstrasse 4, A-8010 Graz, Austria*

A novel, microwave assisted, high temperature UV-digestion for accelerated decomposition of dissolved organic compounds or slurries was developed. UV irradiation is generated by immersed electrodeless Cd discharge lamps (228 nm) operated by the microwave field in the oven cavity. The high pressure – high temperature digestion vessels of the new MULTIWAVE 3000 offer ideal conditions for this decomposition technique at temperature up to 250 – 280 °C. Compared to open UV digestion devices, decomposition time is reduced by a factor of five. Up to 0.1 g powdered organic material are digested with just 1 mL H<sub>2</sub>O<sub>2</sub>, 5 mL H<sub>2</sub>O and 0.1 mL HNO<sub>3</sub>. The remaining organic carbon content is about 5-10 %. With the unique design of the new MULTIWAVE 3000 the digestion vessels can be additionally loaded with oxygen. The digestion efficiency is improved significantly and the residual organic carbon drops down to 1-2 %.

This new technique is ideal for extreme trace analysis due to the low blank values and low acid concentration. In addition this digestion method can be used for the determination of nonmetals like halogens by ion chromatography.

3. März 2003



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**HIGH PERFORMANCE FLOW DIGESTION SYSTEMS (T 5)****G. Knapp, H. Motter, E. Maichin and M. Zischka***Institute for Analytical Chemistry, Micro- and Radiochemistry, Graz University of  
Technology, Technikerstrasse 4, A-8010 Graz, Austria*

Many different designs of flow digestion systems have been published [1], but very few meet the prerequisites for high performance sample decomposition. In principle there is the same situation as with sample digestion in open and closed vessels. For powerful sample digestion nitric acid and high temperatures are the goal. In this context only two flow digestion techniques fulfil this postulate. One is the high temperature / high pressure flow digestion system in Pt-capillaries with conductive heating from H. Berndt [2]. Temperatures up to 360 °C are possible. The other technique has been developed in our institute. Applying a novel pressure equilibration system the digestion temperature in a Teflon tube can be risen up to 250 °C with a pressure of 40 bar [3].

Results of off- and online analysis of different organic materials show the capability of this new instrumentation (Autoflow).

**Literature:**

- [1] M. Burguera, J.L. Burguera; "Microwave-assisted sample decomposition in flow analysis", *Anal. Chim. Acta* 366 (1998) 63-80
- [2] S. Haiber, H. Berndt; "A novel high-temperature (360 °C)/high-pressure (30 Mpa) flow system for online sample digestion applied to ICP spectrometry", *Fresenius J. Anal. Chem.* 368 (2000) 52-58
- [3] U. Pichler, A. Haase, M. Michaelis, G. Knapp; „Microwave-enhanced flow system for high-temperature digestion of resistant organic materials“, *Anal. Chem.* 71 (1999) 4050-4055

13. März 2003

AT0300302



AT0300303

ABSTRACTS of LECTURES

TRISP 2002

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**OPEN VESSEL MICROWAVE DIGESTION OF FOOD MATRICES  
(T 5)**

**Leslie Rhodes and Greg LeElanc**

*CEM Corporation, POB 200, Matthews, NC 28106-0200*

Advancements in the field of open vessel microwave digestion continue to provide solutions for industries requiring acid digestion of large sample sizes. Those interested in digesting food matrices are particularly interested in working with large amounts of sample and then diluting small final volumes.

This paper will show the advantages of instantaneous reagent addition and post-digestion evaporation when performing an open vessel digestion. The digestion and evaporation methods for various food matrices will be presented along with analyte recovery data.

06. März 2003

## IMPROVING METHODOLOGY IN OPEN VESSEL DIGESTION WITH A GRAPHITE HEATING BLOCK (T 7)

Peter Kainrath\*, Bernhard Conrads\* and Art Ross\*\*

\* S-prep GmbH, Im Amann 7, D-88662 Ueberlingen

\*\* SCP SCIENCE, 21800 Clark Graham, Baie d'Urfé, QC H9X 4B6

Open block digestion systems have been very popular in environmental analysis over the past decades, but have consistently suffered from the major drawback of their sensitivity against corrosion and the subsequent risk of contamination. Therefore block digestion systems have not been considered state-of-the-art technology in trace and ultra trace sample preparation.

Graphite block digestion systems are well established in North America and are recently becoming more frequently considered in Europe. These systems overcome the deficiencies of the traditional systems, made from stainless steel or aluminum, because the block is manufactured from graphite and typically coated with a fluoro-polymer to prevent the possibility metallic contamination from the surface of the system during the handling of the samples. Graphite block systems present an alternative to the current mainstream technology of open and closed vessel microwave assisted digestion systems, as they allow large numbers of samples to be digested simultaneously, thus overcoming one of the major weaknesses of closed vessel systems.

More recently a number of improvements in the technology has been developed for graphite block digestion systems and studies have been performed to evaluate the effects of such improvements.

The paper presented will deal with the technological improvements:

monitoring and control of sample temperature vs. monitoring of block temperature (Figure 1)

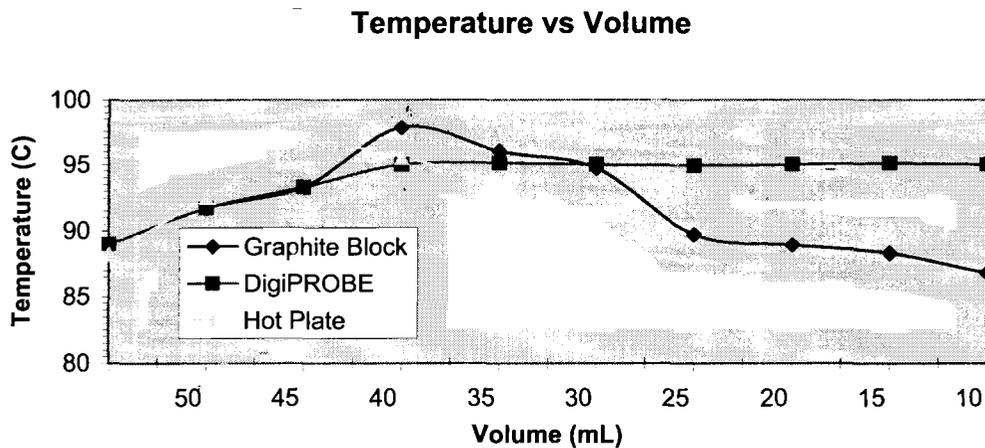
elimination of cross contamination effects during open vessel block digestion

evaporation of samples for pre-concentration or multiple digestion steps

addressing the needs of various labs and applications for block digesters

The effects of those developments will be discussed; application examples and finally a outlook into possible future trends for graphite block digestion systems will be given.

Figure 1: Temperature vs Volume of the three experiments



13. März 2003



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**SIMPLIFIED POLYMER CHARACTERIZATION AFTER  
MICROWAVE ASSISTED SAMPLE PREPARATION (T9)****M. Lafer\*, M. Gfrerrer\*\*, P. Kettisch\****\* Anton Paar GmbH, Anton-Paar-Strasse 20, A-8054 Graz, Austria**\*\* Institute for Analytical Chemistry, Micro- and Radiochemistry, Graz University of  
Technology, Techniker Strasse 4, A-8010 Graz, Austria*

Beside the determination of fillers and heavy metals in polymers after decomposition more often stabilizers, fire inhibitors and antistatic additive agents are measured after using fast microwave accelerated solvent extraction.

Determination of heavy metal traces for example in food packaging materials needs high sample weight to detect small amounts of impurities. High sample weight is also needed for plastic waste providing the homogeneity for representative analysis. Due to the high concentration of organic carbon and the fact that the materials swim on the acid surface, closed vessel digestion had limits concerning sample weight. A new vessel insert in combination with extremely fast reaction control allows now to double or triple usual sample weights.

High performance vessels can also be used to decompose polymers filled with TiO<sub>2</sub>, talcum, fibres or similar within short one or two step procedures gaining solutions without precipitates. Additional filtration or sample treatment is not necessary.

For the determination of organic components more and more classical, but time consuming methods are replaced by microwave assisted solvent extraction. Instead of hours or even half days using Soxhlet extraction samples can be extracted within minutes using vessels and rotors similar to those used for decomposition. The dual use of one basic microwave instrument for both, analysis of inorganic as well as organic parameters will help to increase efficiency by reduced costs.

3. März 2003



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**TRENDS AND APPLICATIONS OF INTEGRATED AUTOMATED  
ULTRA-TRACE SAMPLE HANDLING AND ANALYSIS (T 9)****H. M. Skip Kingston<sup>a,b</sup>, Ye Han<sup>b</sup>, Dirk Link<sup>a</sup>, Larry Stewart<sup>b</sup>**<sup>a</sup> *Duquesne University, 600 Forbes Ave, Pittsburgh PA 15282*<sup>b</sup> *Metara Inc. 1225 East Arques Ave, Sunnyvale CA 94085*

Automated analysis, sub-ppt detection limits, and the trend toward speciated analysis (rather than just elemental analysis) force the innovation of sophisticated and integrated sample preparation and analysis techniques. Traditionally, the ability to handle samples at ppt and sub-ppt levels has been limited to clean laboratories and special sample handling techniques and equipment. The world of sample handling has passed a threshold where older or "old fashioned" traditional techniques no longer provide the ability to see the sample due to the influence of the analytical blank and the fragile nature of the analyte. When samples require decomposition, extraction, separation and manipulation, application of newer more sophisticated sample handling systems are emerging that enable ultra-trace analysis and species manipulation. In addition, new instrumentation has emerged which integrate sample preparation and analysis to enable on-line near real-time analysis. Examples of these newer sample-handling methods will be discussed and current examples provided as alternatives to traditional sample handling.

Two new techniques applying ultra-trace microwave energy enhanced sample handling have been developed that permit sample separation and refinement while performing species manipulation during decomposition. A demonstration, that applies to semiconductor materials, will be presented<sup>1</sup>. Next, a new approach to the old problem of sample evaporation without losses will be demonstrated that is capable of retaining all elements and species tested<sup>2</sup>. Both of these methods require microwave energy manipulation in specialized systems and are not accessible through convection, conduction, or other traditional energy applications.

A new automated integrated method for handling samples for ultra-trace analysis has been developed. An on-line near real-time measurement system will be described that enables many new automated sample handling and measurement capabilities. This new approach has been developed for the semiconductor industry; however, as with most new technologies its applicability extends to many other areas as well including environmental, pharmaceutical, clinical and industrial chemical processing. This instrumental system represents a fundamentally new approach. Sample preparation has been integrated as a key system element to enable automation of the instrument system. It has long been believed that an automated fully integrated system was not feasible if a powerful MS system were included. This application demonstrates one of the first fully automated and integrated sample preparation and mass spectrometric instrumental analyses systems applied to practical use<sup>3</sup>. The system is also a broad and ambitious mass based analyzer capable not only for elements but also for direct speciated analysis. The complete analytical suite covering inorganic, organic, organo-metallic and speciated analytes is being applied for critical contamination control of semiconductor processes. As with new paradigms technology it will now extend from its current use into those other applications needing real-time fully automated multi-component analysis.

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Dirk Link, H.M. Kingston. "Use of Microwave-Assisted Evaporation for the Complete

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13 März 2003

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**A NEW HIGH-THROUGHPUT ROTOR FOR IMPROVED  
PERFORMANCE IN MICROWAVE CLOSED VESSEL ACID  
DIGESTION (T10)**

**C. Pirola**

*Milestone Srl, Via Fatebenefratelli, 1/5 – 24010 Sorisole (BG) Italy  
Email [c.pirola@milestonesrl.com](mailto:c.pirola@milestonesrl.com)*

The use of microwave systems for closed vessel acid digestion has been widely spread in the last decade and this technology is now days the state of the art in sample preparation for AA and ICP.

The heart of the microwave system is represented by the reaction vessel, and different approaches have been taken to safely process samples under high temperature and pressure.

Most systems use “non re-closing” type safety devices, in which the potential overpressure is released through a rupture disk or a membrane.

However in the last few years self-resealing/auto-venting valves have also been employed, as this technique may offer both performance and safety advantages.

The major drawbacks are represented by the fact that these rotors have limited sample throughput and often a bulky construction.

The aim of this paper is to present a new rotor, which combines the advantages of the self-resealing/auto-venting technology with a much higher sample throughput.

The working principle of the safety valve is illustrated, and the major features of this new rotor documented, such as the maximum operating pressure and temperature, as well as cooling time.

Furthermore, examples of digestion procedures are shown, to highlight the possible range of applications of this new accessory for microwave sample preparation.



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**PRESSURIZED WET DIGESTION IN OPEN VESSELS (T 11)****P. Kettisch, E. Maichin, M. Zischka and G. Knapp***\* Anton Paar GmbH, Anton-Paar-Strasse 20, A-8054 Graz, Austria**\*\* Institute for Analytical Chemistry, Micro- and Radiochemistry, Graz University of Technology, Techniker Strasse 4, A-8010 Graz, Austria*

Pressurized wet digestion in closed vessels, microwave assisted or with conventional conductive heating, is the most important sample preparation technique for digestion or leaching procedures in element analysis. In comparison to open vessel digestion closed vessel digestion methods have many advantages, but there is one disadvantage – complex and expensive vessel designs.

A new technique -- pressurized wet digestion in open vessels – combine the advantages of closed vessel sample digestion with the application of simple and cheap open vessels made of quartz or PFA. The vessels are placed in a High Pressure Asher HPA, which is adapted with a Teflon liner and filled partly with water. The analytical results with 30 mL quartz vessels, 22 mL PFA vessels and 1.5 mL PFA auto sampler cups will be shown. In principle every dimensions of vessels can be used. The vessels are loaded with sample material (max. 1.5 g with quartz vessels, max. 0.5 g with PFA vessels and 50 mg with auto sampler cups) and digestion reagent. Afterwards the vessels are simply covered with PTFE stoppers and not sealed. The vessels are transferred into a special adapted HPA and digested at temperatures up to 270°C. The digestion time is 90 min. and cooling down to room temperature 30 min.

The analytical results of CRM's are within the certified values and no cross contamination and losses of volatile elements could be observed.

Marz-2003

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ABSTRACTS of LECTURES

TRISP 2002

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**ACCELERATED SOLVENT EXTRACTION (ASE) - A FAST AND  
AUTOMATED TECHNIQUE WITH LOW SOLVENT CONSUMPTION  
FOR THE EXTRACTION OF SOLID SAMPLES (T 12)**

**Frank Höfler**

*Dionex Corporation Europe  
Solothurnerstrasse 259  
CH-4600 Olten*

Accelerated Solvent Extraction (ASE) is a modern extraction technique that significantly streamlines sample preparation. A common organic solvent as well as water is used as extraction solvent at elevated temperature and pressure to increase extraction speed and efficiency. The entire extraction process is fully automated and performed within 15 minutes with a solvent consumption of 18 mL for a 10 g sample. For many matrices and for a variety of solutes, ASE has proven to be equivalent or superior to sonication, Soxhlet, and reflux extraction techniques while requiring less time, solvent and labor.

First ASE has been applied for the extraction of environmental hazards from solid matrices. Within a very short time ASE was approved by the U.S. EPA for the extraction of BNAs, PAHs, PCBs, pesticides, herbicides, TPH, and dioxins from solid samples in method 3545. Especially for the extraction of dioxins the extraction time with ASE is reduced to 20 minutes in comparison to 18 h using Soxhlet.

In food analysis ASE is used for the extraction of pesticide and mycotoxin residues from fruits and vegetables, the fat determination and extraction of vitamins.

Time consuming and solvent intensive methods for the extraction of additives from polymers as well as for the extraction of marker compounds from herbal supplements can be performed with higher efficiencies using ASE.

For the analysis of chemical weapons the extraction process and sample clean-up including derivatisation can be automated and combined with GC-MS using an online ASE-APEC-GC system.

13. März 2003

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**COMPREHENSIVE 2D CAPILLARY LC AS FRONT END TECHNIQUE  
FOR MS IN PROTEOMICS (T 13)**

**M. Smoluch, G. Mitulovic, R. Swart, J.P.Chervet**

*LC Packings - A Dionex Company, Amsterdam, The Netherlands*

Two-dimensional polyacrylamide gel electrophoresis (2D-PAGE) followed by mass spectrometry (MS) is the most widely used method for protein separation, quantification and identification. However, proteins with extremes in pI and molecular weight, low abundance proteins and membrane associated proteins are rarely seen in 2D-PAGE studies. Therefore, alternative high resolution analytical techniques are needed to analyze complex proteinaceous samples.

One of the promising techniques is the combination of strong cation exchange- (SCX) and reversed phase (RP) chromatography (2D-LC). Proteins are digested in a single step. The resulting peptide mixture is injected onto a SCX column and fractionated with salt steps. The main advantage of the method is the increased peak capacity by which 2D-PAGE can be omitted.

In addition, 2D-LC can be performed fully automated and directly coupled to MS.

In this work we have focused on several variables that affect the overall performance of 2D-LC. These variables include the size of the SCX column, the type of SCX stationary phase, the elution buffer and the arrangement of the SCX and RP column.

All these factors were found to influence the recovery and elution of peptides. Optimized conditions which were used for the identification of proteins in (complex) mixture will be discussed.



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**ADVANCES IN MODERN SAMPLE PREPARATION TECHNIQUES  
USING MICROWAVES ASSISTED CHEMISTRY FOR METAL  
SPECIES DETERMINATION (W 1)**

**O.F.X. Donard**

*Laboratoire de Chimie Analytique Bio-Inorganique et Environnement, CNRS UMR 5034,  
Pau, (France)*

Sample preparation has long been the bottleneck of environmental analysis for both total and species specific analysis. Digestion, extraction and preparation of the analytes are relying on a series of chemical reactions. The introduction of microwave assisted sample preparation has first been viewed as a mean to accelerate the kinetics of digestion of the matrix for total elements and fast samples preparation procedures. However, the extensive development and success of microwave digestion procedures in total elemental analysis has now allowed to have a larger insight of the perspectives offered by this technique.

Microwave technologies now offer to have a precise control of the temperature and indirectly control the reaction kinetics taking place during the sample preparation procedures. Microwave assisted chemistry permits to perform simultaneously the fundamental steps required for metal species extraction and derivatization. The number of sample preparation steps used for organotin or organomercury species have been reduced to one and the total time of sample preparation brought down for a few hours to some minutes. Further, the developments of GC/ICP/MS techniques allow to routinely use Speciated Isotopic Dilution Methods has internal probe of the chemical reactions. These new approaches allow us to use the addition of the labeled species for isotopic dilution as a mean to evaluate and follow the chemical processes taking place during the extraction procedure.

These procedures will help us to understand and check for the stability of the analytes during the chemistry of the sample preparation procedure and bring some insights of the chemistry taking place during the extraction.

Understanding the different mechanisms involved in the sample preparation steps will allow us in return to further improve all these procedures and bring us to the horizon of "on-line sample preparation and detection".

13. März 2003



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**COMPLETE EXTRACTION OF ARSENIC SPECIES FROM  
BIOLOGICAL SAMPLES-A WORTHWHILE GOAL? (W 2)****Kevin Francesconi<sup>1</sup> and Anne-Porthe Madsen<sup>2</sup>**<sup>1</sup>*Institute of Chemistry-Analytical Chemistry, Karl-Franzens-University, 8010 Graz, Austria*<sup>2</sup>*Institute of Biology, University of Southern Denmark, 5320 Odense Denmark*

A major reason cited for carrying out arsenic speciation work is that the various species have different toxicities. In accordance with this is the fact, that they also have very different chemical and physical properties including solubility. The polarity of naturally-occurring arsenic species range from highly polar arsenate to arseno-lipids of low polarity. It thus seems an impossible task to find extraction conditions suitable for all such arsenic species. And yet researchers are prepared to try. This lecture reports a brief history of the reported extraction procedures for arsenic for speciation analysis, and the methods commonly used today. The rationale behind the methods and their applicability to quantitative analysis are also discussed.

10. März 2003

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ABSTRACTS of LECTURES

TRISP 2002

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**EFFECTS OF EXTRACTION AND DERIVATISATION TECHNIQUES  
ON RECOVERY OF ORGANOTIN SPECIES IN ENVIRONMENTAL  
MATRICES (W 3)**

**Raimund Wahlen and Tim Catterick**

*LGC Limited, Queens Road, Teddington, Middlesex, TW11 0LY, Great Britain.  
Email: raimund.wahlen@lgc.co.uk*

Organotin (OT) contamination is still persistent in most coastal environments and estuaries and can be monitored accurately by analysis of sediments and sedentary aquatic animal populations. In addition to human exposure via contaminated seafood, there is increasing concern about OT compounds in man-made consumer products.

Organotin analysis involves the extraction from the matrix, derivatisation (for GC analysis), separation of the different species and detection.

Commonly used extraction techniques are mechanical shaking, ultrasonic agitation and microwave leaching (open or closed vessel). Accelerated solvent extraction (ASE) has also been reported.

In this study, closed-vessel microwave extraction and ASE methods are compared for OT analysis in environmental tissues using separation by gas chromatography (GC) and detection by inductively coupled plasma mass-spectrometry (ICP-MS). The efficiency of an ethylation procedure with sodiumtetraethylborate (NABEt<sub>4</sub>) using different matrices and derivatisation durations is monitored by comparing data obtained by external calibration with results by species-specific isotope dilution analysis (SS-IDMS) of tributyltin (TBT) and dibutyltin (DBT) enriched with <sup>117</sup>Sn. Uncertainty estimates for both quantification techniques will be provided.

13. März 2003



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**COMPROMISES BETWEEN REPRESENTATIVENESS OF SAMPLE  
PREPARATION AND QUALITY OF ANALYTICAL DATA AS  
EXAMPLIFIED BY CRM EXPERIENCE (W4)**

**Herbert Muntau**

*GSF-Forschungszentrum, Munich, Germany*

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AT0300312

ABSTRACTS of LECTURES

TRISP 2002

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## TRACEABILITY AND MEASUREMENT UNCERTAINTY IN SAMPLE PREPARATION (W 5)

**Wolfhard Wegscheider, Ulrike Walner and Johann Moser**

*Institute for General and Analytical Chemistry, University of Leoben  
A-8700-Leoben/Austria, e-mail: [wegschei@unileoben.ac.at](mailto:wegschei@unileoben.ac.at)*

Very few chemical measurements are being made directly on the object of interest and sample preparation is thus the rule rather than the exception in daily practice. Unfortunately the operations undertaken in the course of sample preparation are prone to rendering a sample useless for the purpose of interpreting a measurement performed on it, as it might not represent the original and relevant status any longer. Sample preparation along with sampling itself constitutes therefore a procedure that leads to a loss of representation of the original specimen or population.

On the other hand it is also not sufficient to confine aspects of traceability and measurement uncertainty to the ultimate measurement, as the key purpose of measuring is to supply adequate data for some kind of decision, be it in production, in health, in the environment, or indeed in any other circumstance. These considerations have led to severe confusion in the community as to what traceability really means in chemistry.

CITAC and EURACHEM have only recently issued a preliminary document that clarifies these issues and gives a firm handle on the future development of quality assurance in analytical chemistry.

In this talk it will be attempted to outline the general ideas and procedures that lead to traceability of analytical chemical results accompanied by valid statements of their uncertainty. It will be argued that the central element in achieving these goals is a well-designed validation study that frequently goes beyond those requirements currently laid out in official documents.

13. März 2003

AT0300313



AT0300313 ✓

**RECOVERY OF TOTAL Hg FROM GEOLOGICAL MATRICES: A  
METHODOLOGICAL INTERCOMPARISON STUDY (W 5)****N.S. Bloom,<sup>1</sup> E.J. Vanderveest,<sup>1</sup> E.M. Preus,<sup>1</sup> M. Horvat,<sup>2</sup> and N. Prosenč,<sup>2</sup> S. Kingston,<sup>3</sup>  
S. Apte,<sup>4</sup> and C. Peobbs,<sup>5</sup> <sup>1</sup>Frontier Geosciences, Seattle, USA, <sup>2</sup>J Stefan***Institut, Ljubljana, Slovenia, <sup>3</sup>Duquesne University, Pittsburgh, USA, <sup>4</sup>CSIRO, Sidney,  
Australia, <sup>5</sup>Alcoa of Australia, Kwinana, Australia.*

Aqua regia is commonly used for the extraction of Hg from soils prior to cold vapor spectrometry, but mass balance studies suggested it is not sufficiently robust to recover all Hg from more recalcitrant materials such as crystalline ores. As a result, an intercomparison exercise was undertaken between the following methods: (a) aqua regia digestion (EPA 7471) followed by SnCl<sub>2</sub> reduction, gold trapping and CVAFS detection, (b) Teflon bomb digestion at 110°C with a 10:5:2 HNO<sub>3</sub> + HF + HCl mixture followed by SnCl<sub>2</sub> reduction, gold trapping and CVAFS, (c) 800°C pyrolysis with gold trapping and CVAAS detection (EPA 7473), and (d) NAA followed by radiochemical separations using oxidative combustion and gold trapping. All methods gave quantitative recoveries from soil and sediment CRMs, but only the HF/HCl/HNO<sub>3</sub> digestion gave quantitative recoveries in all media. Pyrolysis resulted in approximately 88 ± 10% recovery, while aqua regia digestion resulted in only about 61 ± 24% recovery in bauxite ores and residues. RNAA generally gave the best agreement with the HF based digestion, although in a significant number of samples, particularly bauxite ores, recoveries were as low as 50%. Based on these findings, we recommend that bomb digestions which include the use HF be applied to the analysis of Hg in geological solids, unless the matrix is documented to be fully extractable by a less rigorous procedure. We suggest a reference value for Hg in bauxite reference material (NIST-600) of 79.0 ± 2.0 ng/g, based on the pooled results of three laboratories.

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**SO YOU'VE GOT YOUR SAMPLE IN SOLUTION: WHAT NEXT?  
(W 7)**

**Ian P. Erindle**

*Brock University, St. Catharines, Ontario, Canada*

Several factors must be considered after the sample is prepared for analysis. These factors include memory, oxidation state of analyte, presence of interfering elements, etc. Some elements are "sticky" and exhibit prodigious memory effects. For elements like mercury, gold and boron, memory effects make it difficult to determine elemental concentrations in samples that vary widely in concentration. When selenium is determined by hydride generation, the selenium cannot be in the VI oxidation state, since borohydride will not reduce this oxidation state. Different treatments must be used. The treatment of organometallics may require, in addition, the presence of reagents to improve the yield of derivitized species that are to be determined. Interfering elements must sometimes be masked or removed before determination of the analyte can proceed. In this presentation, these various issues will be discussed. Solutions to some of the problems, from the analytical chemistry laboratories at Brock University, will be presented. In addition, options for the simultaneous determination of elements by vapour generation and nebulization will be discussed, based on recent work in the Brock laboratories.

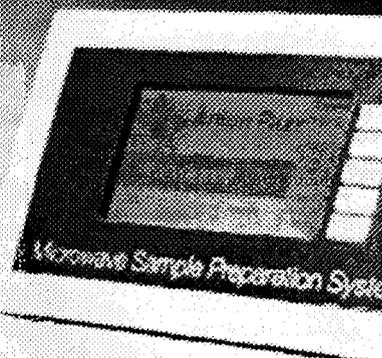
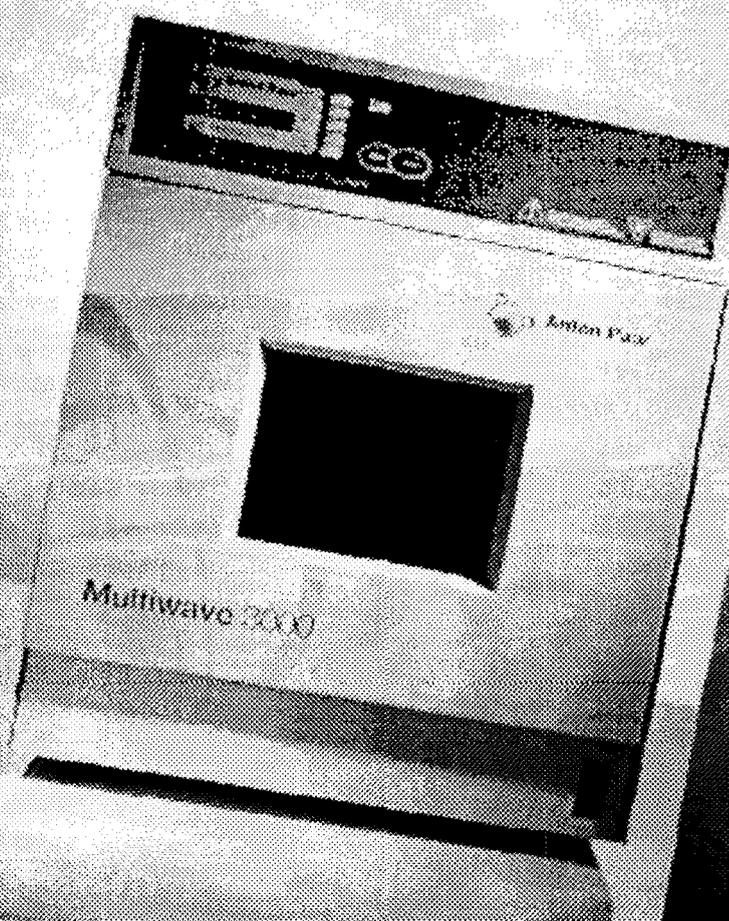
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**SAMPLE PREPARATION WITH MICROWAVE: EXPERIENCES IN  
THE ENVIRONMENTAL- AND INDUSTRIAL ANALYTICS  
LABORATORY OF VOESTALPINE (P 1)**

**Andreas Schoenauer, Klaus Hammerlitz, Gerhard-Christian Kastner**

*Voest Alpine Stahl GmbH, Linz, Austria*

Since around one and a half year two microwave sample preparation units tested and used in the environmental- and industrial analytics laboratory of voestalpine. On basis of the experiences the technique offered good results for the specific applications in a steel company.

In comparison with the traditional sample preparation of iron oxides for ICP-OES determination through a open vessel wet-chemical acid pulping sample preparation with closed vessel microwave digestion got a large quantity of advantages. The problem with simultaneously sample preparation and determination of silica and other compounds in ultra-pure iron oxides could be solved. We obtained an excellent recovery and reproducibility with microwave pulping.

In the range of environmental analytics the possibilities of microwave sample preparation to prepare typical dusts, landfill wastes, process and waste water of a steel company was analysed. The microwave sample preparation showed good reproducibility to the conventional techniques, e.g. pulping and soxleth-extraction. Someone of them are already replaced by the new method. Here also the microwave technique possess a large potential for more uses.

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**COMPARISON OF TWO MICROWAVE-ASSISTED SAMPLE DIGESTIONS OF SEDIMENT AND SOILS FOR TRACE METALS USING ICP-MS: HNO<sub>3</sub> AND HNO<sub>3</sub>/HF (P 2)**

**C. Howard, A. Vandervort, and N.S. Bloom**

*Frontier Geosciences Inc., 414 Pontius Ave. N., Seattle WA 98109 USA*

Our goal was to develop an accurate and dependable digestion method for extracting trace metals from soil and sediment samples. This method would be applied to analytes at low concentrations in various sediments, including soil, contaminated soil, and marine sediment. Analysis was performed for a wide spectrum of trace metals (Ag, Al, As, Cd, Ce, Co, Cr, Cu, Eu, Fe, Hf, Ho, La, Mg, Mn, Mo, Nd, Ni, Pb, Th, Ti, Tl, Sb, Se, Sm, Sn, Yb, and Zn) using an Elan 6000 ICP-MS (Perkin-Elmer).

Two sample preparation procedures were adjusted and applied to three certified reference materials (CRM): NIST2709, NIST2710, and MESS-3. The first procedure involved digestion with 10 mL nitric acid (HNO<sub>3</sub>) at 180 °C for 25 min in a microwave (Milestone-ETHOS plus). The process included four acid blanks, one blank spike, three replicates of each CRM, and one matrix spike on each of the CRM. The second procedure involved digestion with 8 mL HNO<sub>3</sub>, and 4 mL hydrofluoric acid (HF) at 180 °C for 25 min in the microwave. Digestion included four acid blanks, one blank spike, three reps of each CRM, and one matrix spike on each of the CRM.

Each method proved to be robust and accurate for different analytes. Rare earth elements worked particularly well using the HNO<sub>3</sub> digest. By utilizing both digestions, all trace metals examined were successfully and completely extracted.

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**ANALYSIS OF SOIL AND SEWAGE SLUDGE BY ICP-OES AND THE  
GERMAN STANDARD DIN 38414 SAMPLE PREPARATION  
TECHNIQUE (P 3)**

**Maria Edlund, Peter Heitland, Henk Visser**

*Spectro Analytical Instruments GmbH, Boschstr. 10, D-47533 Kleve, Germany*

The elemental analyses of soil and sewage sludge has developed to become one of the main applications for ICP Optical Emission Spectrometry (ICP-OES) and is described in many official procedures.

These methods include different acid mixtures and digestion techniques. Even though the German standard DIN 38414 Part 7 and the Dutch NEN 6465 do not guarantee complete recoveries for all elements, they are widely accepted in Europe. This paper describes sample preparation, line selection and investigates precision, accuracy and limits of detection.

The SPECTRO CIROSCCD EOP with axial plasma observation and the SPECTRO CIROSCCD SOP with radial observation were compared and evaluated for the analyses of soil and sewage sludge. Accuracy was investigated using the certified reference materials CRM-141 R, CRM-143 R and GSD 11.

Both instruments show excellent performance in terms of speed, precision, accuracy and detection limits for the determination of trace metals in soil and sewage sludge.

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**DIRECT ANALYSIS OF IRON IN SAND BY SOLID SAMPLING  
ELECTROTHERMAL ATOMIC ABSORPTION SPECTROMETRY (P 4)**

**Cíntia S. Silva<sup>a</sup>, Cassiana S. Nomura, Elisabeth de Oliveira, Joaquim A. Nóbrega<sup>a</sup>,  
Pedro V. Oliveira\***

*Instituto de Química, Universidade de São Paulo, C.P. 26077, 05513-970, São Paulo, SP,  
Brasil, \*E-mail: pvolivei@quim.iq.usp.br*

<sup>a</sup>Departamento de Química, Universidade Federal de São Carlos, São Paulo, SP, Brazil

Direct solid sampling electrothermal atomic absorption spectrometry (SS-ETAAS) has deserved attention for trace and ultra-trace elements determination in technological, biological and environmental samples [1,2]. The reduced sample requirements, the possibility to carry out the sample decomposition inside the graphite furnace, the simplicity and high sensitivity are the main attributes of the SS-ETAAS. In this work the direct determination of iron in sand by using SS-ETAAS was investigated. The sand samples analyzed are used as raw material for glass industry. Each sand sample was directly weighed onto a boat-type platform using microbalance, after then the platform was inserted into the furnace with a manual solid sampling. Samples with different particle sizes were analyzed. Calibration was performed by increasing weighed masses (0.030 – 0.120 mg). Three-field background corrector was used to diminish the sensitivity of the ETAAS, allowing the weigh of high masses. This helps to reach proper representativity. By using one sample with known iron concentration it was possible to perform the ETAAS calibration. Due to the delayed heating rate of the platform boat-type, it was observed that the matrix vaporized after the analyte. However, this temporal separation was not so effective for high masses of sand (> 0,120 mg). Good agreement (95% confidence level) was obtained for iron determination by comparing this direct method and a reference method. The relative standard deviation was lower than 16% (n=5).

[1] U. Kurfürst, *Solid Sample Analysis. Direct and Slurry Analysis Using GF-AAS and ETV-ICP*, Springer-Verlag, Berlin, 1998.

[2] K.W. Jackson ed., *Electrothermal Atomization for Analytical Atomic Spectrometry*, John Wiley & Sons, New York, 1999.

13. März 2003



**COMBINATION OF SOPHISTICATED SAMPLE DIGESTION  
METHODS AND HIGHLY ACCURATE DETERMINATION  
PROCEDURES EXEMPLIFIED FOR SULPHUR IN OIL AND  
CADMIUM AND LEAD IN SEDIMENT (P 5)**

**G. Riebe, M. Ostermann, W. Pritzkow, J. Vogl**

*Laboratory I.42  
Isotope Dilution and Nuclear Fuel Analysis  
Federal Institute for Material Research and - Testing (BAM)  
Unter den Eichen 87  
D-12205 Berlin*

New measurement techniques and instruments have improved the analytical precision impressively over the last years. On the other hand, the control of the quality and completeness of the chemical treatment has fallen in some cases far behind.

The aim of this study is to present elaborated varieties of modern decomposition methods combined with application of classical analytical chemistry used for the determination of trace elements in difficult matrix sample materials at highest metrological level. The analytical technique used is isotope dilution mass spectrometry, which offers the possibility to perform checks on the quality of the chemical treatment.

Herein the sample pre-treatment for two different materials; gas oil and sediment is described.

The gas oils were weighed into quartz vessels together with the decomposing reagents for digestion in a High Pressure Asher (HPA-S) followed by a wet chemical reduction of the sulphate to  $H_2S$ . This was carried out using a mixture of conc. HCl, conc. HI and  $H_3PO_2$ . The solution was refluxed in a stream of  $N_2$  and the  $H_2S$  formed was trapped in an aqueous  $NH_3$  solution and precipitated as  $As_2S_3$ .

The total decomposition of the sediment samples was carried out in a three step procedure. The sample was digested with HCl/ $HNO_3$  at  $220^\circ C$  using a microwave oven. After removal of the silicates using HF, the insoluble residue was decomposed by melting with soda. Then cadmium and lead were separated from the combined digestion solution.

The measurements for both sample materials were carried out on thermal ionisation mass spectrometers (TIMS).

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**ON THE DETERMINATION OF THE MOISTURE CONTENT IN  
DIFFERENT MATRIX MATERIALS (P 6)****M. Ostermann, P. Becker, N. Janisch, G. Riebe, J. Vogl**

*Laboratory I.42  
Isotope Dilution and Nuclear Fuel Analysis  
Federal Institute for Material Research and - Testing (BAM)  
Unter den Eichen 87  
D-12205 Berlin*

The knowledge of an accurate and reproducible moisture content is essential for the minimization of possible systematic errors in the quantitative determination of the analyte measured. Erroneous dry mass corrections influence directly the obtained result and its uncertainty budget. Moreover, the moisture content of a commercial product must be well known in order to ascertain its quality. It is known that there are different binding forms of water. The dry-mass correction used must therefore be chosen in order to account for these different binding forms. An additional problem can be that the sample material picks up extra moisture from the environment because of its highly hydroscopic character. Numerous investigations showed that drying methods are not capable of distinguishing between different binding forms of water. Only the total water content, measured by Karl Fisher titration, provides results which can be attributed to a defined physical property. For this investigation three different matrix materials (rice, sediment and fly ash) were taken and tested for their moisture content using various drying methods. The drying methods are oven drying at 85°C, or 105°C respectively, with and without previous equilibration of the sample with air humidity, Karl-Fischer titration and a commercial balance equipped with an infrared drying device. One result reveals that the water adsorption of two samples caused by humid air during the weighing procedure could be source of severe errors. A model calculation of a worst-case scenario will be presented and the results of all the different drying methods will be compared.

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**A NOVEL RAPID AND SIMPLE METHOD OF SAMPLE  
PREPARATION  
FOR LC/MS ANALYSIS OF CARBOHYDRATES FROM PLASMA (P 7)**

**Eduard Rogatsky and Daniel Stein**

*Albert Einstein College of Medicine  
New York, NY*

Sample preparation for mass spectrometry analysis (including pre-column clean up) is frequently a prolonged and multi-step process. Sample preparation for large batches, which frequently occurs in the clinical research setting, is the most important rate limiting step for sample throughput, and thus is both costly and labor intensive. For example, GC/MS analysis of carbohydrates requires 5 different chemical steps including protein precipitation, purification, derivitization, and extraction prior to analysis.

Our new method of carbohydrate analysis from plasma developed at Albert Einstein College of Medicine is free from routine multi-step manipulation. The method is based upon selective protein denaturation and co-precipitation (step 1, clarification) by acetone/acetonitrile/water. The second step is performed directly in autosampler vials with direct sample extraction/concentration based upon extraction of hydrophobic contaminants in a supernatant hexane layer with simultaneous preconcentration of the polar analyte (such as carbohydrates) in a lower (polar) phase which is picked up by the auto-sampler needle. This simple method allows the generation of hundreds of samples of high purity sufficient for stable LC separation of carbohydrates on microbore columns prior to sensitive measurement by electrospray mass spectrometry in a mass range of 40 to 700 ng.



**MICROWAVE ASSISTED DIGESTION OF ORGANOARSENIC COMPOUNDS FOR THE DETERMINATION OF TOTAL ARSENIC IN AQUEOUS, BIOLOGICAL, AND SEDIMENT SAMPLES USING FI-HG-ETAAS (P 8)**

**K. Eoch, M. Schuster**

*Technical University of Munich, Munich, Germany*

A microwave assisted wet digestion method for organoarsenic compounds and subsequent determination of total arsenic in aqueous, biological and sediment samples by means of flow injection hydride generation electrothermal atomic absorption spectrometry is described. Sodium persulfate, sodium fluoride and nitric acid serve as digestion reagents, which allow a quantitative transformation of organoarsenic compounds to hydride forming species in a commercial microwave sample preparation system.

Arsenic recovery from aqueous testing solutions of dimethylarsinic acid, phenylarsonic acid and tetraphenylarsonium chloride at initial concentrations of  $100 \mu\text{g l}^{-1}$  and  $10 \mu\text{g l}^{-1}$  is complete, even in the presence of an excess of organic carbon or fatty acids.

Arsenic recovery from aqueous arsenobetaine solutions with the same initial concentrations is also complete if high pressure vessels and a higher concentration of fluoride ions are used, whereas the addition of organic carbon leads to a decrease in arsenic recovery of about 2% to 5%. In all cases, residual carbon contents are close to the limit of detection for the applied analytical method ( $15 \text{ mg l}^{-1}$ ).

Results of arsenic analysis in reference standard materials revealed a significant dependence on the material's nature. Sediment samples and plant materials show recoveries for arsenic around 100% after a single-step digestion in medium pressure (30 bar) tetrafluorometoxil liners. Seafood usually require either the use of high pressure vessels (75 bar) or a second digestion step if medium pressure vessels are used.

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**SOLID PHASE MICROEXTRACTION AND STIR BAR SORPTIVE  
EXTRACTION FOR ORGANOTIN  
COMPOUNDS – A COMPARISON (P 9)**

**Mothes.S., Wennrich. R.**

*UFZ Centre for Environmental Research Leipzig-Halle, Department of Analytical Chemistry,  
Permoserstrasse 15, D-04318 Leipzig*

Organotin compounds have been largely used in agricultural and industrial applications. Hyphenated techniques were developed for the sensitive and selective determination of such species. For this task GC has been coupled with atomic emission detection.

Derivatization to transform the Sn-compounds into sufficiently volatile compounds was necessary and carried out using sodium tetrapropylborate.

For sample preparation the application of solid phase microextraction (SPME) give recent advances in comparison to classical liquid-liquid extraction (LEE). A problem in the usage of SPME exists however in the small volume of the PDMS coating for enrichment the analytes. For improvement of both sample enrichment and extraction of the organotin compounds Stir Bar Sorptive Extraction (SBSE) [1] was applied. It base on the application of stir bars coated with PDMS. Here the extraction yield is substantially higher.

Aim of this study was to compare the capabilities of GC-AED in combination with SPME and SBSE. After optimisation of the experimental parameters it was possible to reach detection limits in the pg / l – level.

A comparison of both methods shows the expected results. By application of SBSE it was possible to increase the detection limits one order of magnitude.

With SPME the reproducibility of the analytical results (in the 1 ng / l concentration range) was found to be between 10 and 15%, it could be enhanced to 5-8 % by application of SBSE.

These low limits of detection and the good reproducibility allowed the determination of organotin compounds according required regulations.

[1] E. Baltussen, P. Sandra, F. David, C. Cramers, J. Microcolumn Sep. 1999, 11 (10), 737

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**DETERMINATION OF POLYCYCLIC AROMATIC HYDROCARBONS  
FROM PUMPKIN SEEDS AND PUMPKIN SEED OILS BY  
MICROWAVE-ASSISTED SAPONIFICATION (P 10)**

**Marion Gfrerer, Ernst Lankmayr**

*Institute for Analytical Chemistry, Micro- and Radiochemistry,  
University of Technology Graz, Technikerstr. 4/P, A-8010 Graz*

Polycyclic aromatic hydrocarbons (PAHs) are widespread environmental contaminants of both anthropogenic and natural origin. Human health hazards associated with these compounds are owing to their persistence, their hydrophobic character, their bioaccumulation and the carcinogenic properties of several individual polyarenes. Human exposure to PAH occurs principally by (1) direct inhalation of polluted air and tobacco smoke; (2) dietary intake of smoked and other foodstuffs and polluted water; and (3) dermal contact with soot, tars and polluted soils.

The production of styrian pumpkin seed oil requires a roasting of the pumpkin seeds, generally higher than 60 °C. The roasting at elevated temperatures produces the typical taste of the pumpkin seed oil but may also be the source for a contamination with PAHs. Since norms and legal limits for hot-pressed oils are missing the legal limits of 25 µg/kg for the sum of 16 PAHs and 5 µg/kg for the heavy fraction for refined and cold-pressed oils, such as olive oil and others, have to be applied.

Separation of hydrophobic analytes from oily matrices can be performed by GPC, dialysis, caffeine-complex formation or saponification with alcoholic potassium hydroxide. Traditional saponification methods need at least 40 min, microwave-assisted saponification is completed within 10 min. Therefore, sample preparation was performed by microwave-assisted saponification with 1.5 M methanolic potassium hydroxide using a Multiwave 3000 from Anton Paar GmbH (Graz, Austria) followed by a liquid-liquid extraction. Further purification was achieved by extraction with sulfuric acid (76 %) and a final clean up procedure by adsorption chromatography on activated silica gel combined with a preparative Bondesil-cyano phase. For a selective measurement of the individual analytes, gas chromatography combined with mass spectroscopy was used in single ion monitoring mode [1].

The whole analytical procedure was validated by systematic recovery experiments.

[1] Gfrerer M, Serschen M, Wenzl T, Gawlik BM, Lankmayr E. *Chromatographia* 55 7/8 (2002) 467-473

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**MEMBRANE EXTRACTION WITH A SORBENT INTERFACE-GAS-  
CHROMATOGRAPHY AS EFFECTIVE AND FAST MEANS FOR  
CONTINUOUS MONITORING OF THERMAL DEGRADATION  
PRODUCTS OF POLYMERS**

**Kaykhaii Massoud**

Francisca ACEVEDO  
Montanuniversität Leoben  
Bachgasse 3/6  
A-8700 Leoben  
[face\\_72@yahoo.com](mailto:face_72@yahoo.com)

Rasheed ALAWODE  
S and R Company  
174 Broad Street Lagos Island  
234 Ibadan  
phone: +234 1 266 9023  
fax: +234 1 266 9023  
[waliyiat@yahoo.com](mailto:waliyiat@yahoo.com)

Mohammed ASIE  
King Saud University  
Dept. Food Science & Nutrition  
P.O. Box 2460  
11451 Riyadh  
phone: +966-1-4678717  
fax: +966-1-4678394  
[masifa@ksu.edu.sa](mailto:masifa@ksu.edu.sa)

Ramon BARNES  
University Research Institute for Analytical  
Chemistry  
85 N. Withney Street  
01003-1869 Amherst  
Massachusetts  
USA  
phone: +413 256 8942  
fax: +413 256 3746  
[rbarnes@chem.umass.edu](mailto:rbarnes@chem.umass.edu)

Mahmoud BATAINEH  
ISAS  
Bunsen-Kirchhoff-Straße 11  
D-44139 Dortmund  
phone: +231 1392212  
fax: +231 1392165  
[bataineh@isas-dortmund.de](mailto:bataineh@isas-dortmund.de)

Michael BICKEL  
EC – JRC – IRMM  
Retieseweg  
B-2400 Geel  
phone: +32-14 57 1734  
fax: +32-14-584273  
[Michael.bicke@irmm.jrc.be](mailto:Michael.bicke@irmm.jrc.be)

Nicolas BLOOM  
Frontier Geosciences Research and  
Consulting  
414 Pontius North  
98109 Seattle  
Washington  
USA  
phone: +206-622-6960  
fax: +206-622-6870  
[nicolasb@frontiergeosciences.com](mailto:nicolasb@frontiergeosciences.com)

Kerstin BOCH  
TU München  
Lichtenbergstr. 4  
D-85747 Garching  
phone: +49-89-289-13136  
[Kerstin.Boch@rz.tu-muenchen.de](mailto:Kerstin.Boch@rz.tu-muenchen.de)

Beatrice BOCCA  
Istituto Superiore di Sanita  
Viale Regina Elena 299  
00161 Italy  
phone: +39-0649902011  
fax: +39-0649387068  
[b.bocca@iss.it](mailto:b.bocca@iss.it)

Karl Siegfried BOOS  
Institute of Clinical Chemistry,  
University Hospital Grosshadern  
D-81366 Munich  
Germany  
phone: +49-89-70954732  
[boos@kch.med.uni-muenchen.de](mailto:boos@kch.med.uni-muenchen.de)

Joe BRENNER  
Environmental Analytical Laboratory  
BGU.9 Dishon Street, Malkha  
Jerusalem  
Israel  
phone: 972-2-6797-255  
fax: +972-2-6797-145  
[Brenner@cc.huji.ac.il](mailto:Brenner@cc.huji.ac.il)

Jan BRINDLE  
Brock University  
500 Glenridge Avenue  
St. Catharines  
L2S 3A1 Ontario  
Canada  
phone: +905-688-5550 ext. 3421  
fax: +905-641-0406  
[ibrindle@brocku.ca](mailto:ibrindle@brocku.ca)

Karl BUDNA  
Institute for Analytical Chemistry, Micro-  
and Radiochemistry  
Graz University of Technology  
Technikerstraße 4  
A-8010 Graz  
Austria  
phone: +43-316-873-8800  
fax: +43-316-873-8304  
[kbudna@analytchem.tu-graz.ac.at](mailto:kbudna@analytchem.tu-graz.ac.at)

Maurizio CASATI  
Pharmacia  
Via L. Pasteur 10  
I-20014 Nerviano  
phone: +39- 2-4838-5535  
fax: +39-2-4838-3012  
[Maurizio.casati@pharmacia.com](mailto:Maurizio.casati@pharmacia.com)

Michael J. COLLINS  
CEM Corporation  
3100 Smith Farm Road  
28106 Matthews  
North Carolina  
USA  
phone: 704-821-7015  
fax: 704-821-7894  
[Mike.Collins@cem.com](mailto:Mike.Collins@cem.com)

Sonia D'LLIO  
Istituto Superiore di Sanita  
Viale regina Elena, 299  
I-00161 Rome  
Phone: +39-0649902349  
Fax: +39-064-9902492  
[sdillio@iss.it](mailto:sdillio@iss.it)

Olivier DONARD  
Laboratoire de Chimie  
Analytique Bio-Inorganique et  
Environnement  
CNRS UMR 5034 Pau  
France  
[donard@messv2.univ-pau.fr](mailto:donard@messv2.univ-pau.fr)

Manuela EBERL  
Institute for Analytical Chemistry, Micro-  
and Radiochemistry  
Graz University of Technology  
Technikerstraße 4  
A-8010 Graz  
Austria  
phone: +43-316-873-8301  
fax: +43-316-873-8304  
[eberl@analytchem.tu-graz.ac.at](mailto:eberl@analytchem.tu-graz.ac.at)

Abiodun Christiana EDU  
Bro Edu Institute  
T2 Office 132 Obafemi Awolowo Way Oke  
Ado  
234 Ibadan  
Nigeria  
[broedu@onebox.com](mailto:broedu@onebox.com)

Mavro Rocha EVANGELHO  
CENPES-Petrobras  
AV 1 Quadra 7 Pça do Fundao  
21949-900 Rio de Janeiro  
phone: 55-21-3865-6157  
fax: 55-21-3865-6296  
[mre@cenpes.petrobras.com.br](mailto:mre@cenpes.petrobras.com.br)

Babar Dar FAISAL  
NIL  
44000 Islamabad 72  
phone: +92-51-2279899  
[faisaldar2000@yahoo.com](mailto:faisaldar2000@yahoo.com)

Gisela FAULER  
Institute for Analytical Chemistry, Micro-  
and Radiochemistry  
Graz University of Technology  
Technikerstraße 4  
A-8010 Graz  
Austria  
phone: +43-316-873-8802  
fax: +43-316-873-8304

Guenther FELLNER  
Institute for Analytical Chemistry, Micro-  
and Radiochemistry  
Graz University of Technology  
Technikerstraße 4  
A-8010 Graz  
Austria  
phone: +43-316-873-8801  
fax: +43-316-873-8304  
[fellner@analytchem.tu-graz.ac.at](mailto:fellner@analytchem.tu-graz.ac.at)

Kevin FRANCESCONI  
Institute of Chemistry-Analytical Chemistry  
Karl-Franzens-University  
Universitätsplatz 1  
A- 8010 Graz  
Austria  
phone: +43-316-380-5301  
[Kevin.francesconi@kfunigraz.ac.at](mailto:Kevin.francesconi@kfunigraz.ac.at)

Herbert FRUHMANN  
Institute for Analytical Chemistry, Micro-  
and Radiochemistry  
Graz University of Technology  
Technikerstraße 4  
A-8010 Graz  
Austria  
phone: +43-316-873-8802  
fax: +43-316-873-8304  
[fruhmann@analytchem.tu-graz.ac.at](mailto:fruhmann@analytchem.tu-graz.ac.at)

Jochen GERLACH  
Institute for Analytical Chemistry, Micro-  
and Radiochemistry  
Graz University of Technology  
Stremayrgasse 16/III  
A-8010 Graz  
Austria  
phone: +43-316-873-4326  
[gerlach@analytchem.tu-graz.ac.at](mailto:gerlach@analytchem.tu-graz.ac.at)

Marion GERERER  
Institute for Analytical Chemistry, Micro-  
and Radiochemistry  
Graz University of Technology  
Technikerstraße 4  
A-8010 Graz  
Austria  
phone: +43-316-873-8307  
fax: +43-316-873-8304  
[gfrerer@analytchem.tu-graz.ac.at](mailto:gfrerer@analytchem.tu-graz.ac.at)

Walter GOESSLER  
Institut für Chemie, Analytische Chemie  
Karl-Franzens Universität Graz  
Universitätsplatz 1  
A-8010 Graz  
phone: 0043-316-380-5302  
fax: 0043-316-380-9845  
[walter.goessler@uni-graz.at](mailto:walter.goessler@uni-graz.at)

Veronika GRUNAU  
Merck KGaA  
Frankfurter Straße 250  
D-64293 Darmstadt  
Germany  
phone: +49-61-51-72-56-77  
fax: +49-61-51-72-62-86  
[Veronika.Grunau@Merck.de](mailto:Veronika.Grunau@Merck.de)

Andras GUTTMAN  
Torrey Mesa Research Institute  
3115 Merryfield Row  
CA-92121 San Diego  
USA  
phone: +1-858-812-1085  
fax: +1-858-812-1097  
[andras.guttman@syngenta.com](mailto:andras.guttman@syngenta.com)

Qaisar Nisar HASHIMI  
Pharmagen Beximco Ltd  
Sialkot Road  
B/169 Cheema Colony  
Wazirabad  
Pakistan  
phone: +91-437-601040  
[hashimi.786@hotmail.com](mailto:hashimi.786@hotmail.com)

Frank HOEFLER  
Dionex Corporation Europe  
Solothurnerstrasse 259  
CH-4600 Olten  
Switzerland  
phone: +41-622049999  
fax: +41-622059960  
[frank.hoefler@dionex.ch](mailto:frank.hoefler@dionex.ch)

Simon HOLLAS  
Institute for Trans Uranium elements (ITU)  
Forschungszentrum Karlsruhe  
D-76125 Karlsruhe  
phone: 0049-7247-951423  
[simon.hollas@itv.fzk.de](mailto:simon.hollas@itv.fzk.de)

Leigh HOLMES  
EC-JRC-IRMM  
Retieseweg  
2440 Geel  
phone: +32-14-571804  
fax: +32-14-584273  
[leigh.holmes@irmm.jrc.be](mailto:leigh.holmes@irmm.jrc.be)

Crystal Rose HOWARD  
Frontier Geoscience  
414 Pontius Avenue North  
98109-5461 Seattle  
Washington  
USA  
phone: 206-622-6960  
fax: 206-622-6870  
[Crystalh@Frontier.wa.com](mailto:Crystalh@Frontier.wa.com)

Nadine JANISCH  
Bundesanstalt für Materialforschung und –  
prüfung  
Unter den Eichen 87  
D-12205 Berlin  
Germany  
phone: +49-30-8104-4119  
fax: +49-30-8104-1147  
[Nadine.janisch@bam.de](mailto:Nadine.janisch@bam.de)

Elisabeth KAHR  
General and Analytical Chemistry  
University Leoben  
Franz-Josef-Straße 18  
A-8700 Leoben  
phone: +43-3842-402-346  
fax: +43-3842-402-543  
[elisabeth.kahr@notes.unileoben.ac.at](mailto:elisabeth.kahr@notes.unileoben.ac.at)

Peter KAINRATH  
Soprep GmbH  
Im Amann 7  
D-88662 Überlingen  
Germany  
[Peter.Kainrath@t-online.de](mailto:Peter.Kainrath@t-online.de)

Bernd KAPP  
Berghof Laborprodukte GmbH  
Harretstraße 1  
D-72800 Eningen  
phone: 0049-70719878-20  
fax: 0049-7071-9878-88  
[B.Kapp@institut-berghof.de](mailto:B.Kapp@institut-berghof.de)

Massoud KAYKHAH  
Erdowsi University  
Department of Chemistry  
Faculty of Science  
91779 Mashhad/Iran  
fax: +98-511-8438032  
[kaykhah@science1.um.ac.ir](mailto:kaykhah@science1.um.ac.ir)

Martin KEIL  
Personal Chemistry GmbH  
Gottlieb-Daimler-Str. 5  
D-78467 Konstanz  
phone: +49-7531-9423460  
fax: +49-7531-9423470  
[martin.keil@personalchemistry.com](mailto:martin.keil@personalchemistry.com)

Andreas KERSCHBAUMER  
Fresenius Kabi GmbH  
Hafnerstraße 36  
8055 Graz  
Austria  
Phone: +43-316-249-654  
Fax: +43-316-249-505  
[Andreas.kerschbaumer@fresenius-kabi.com](mailto:Andreas.kerschbaumer@fresenius-kabi.com)

Peter KETTISCH  
Fa. Anton Paar  
Kärntnerstraße 322  
A-8054 Graz  
phone: +43-316-257-0  
fax: +43-316-257-257  
[kettisch@anton-paar.com](mailto:kettisch@anton-paar.com)

Skip KINGSTON  
Duquesne University and Metara Inc.  
1225 East Arques Ave  
94085  
Sunnyvale CA  
USA  
Phone: 408-331-5215  
Fax: 408-523-0945  
[Kingson@dug.edu](mailto:Kingson@dug.edu)

Günter KNAPP  
Institute for Analytical Chemistry, Micro-  
and Radiochemistry  
Graz University of Technology  
Technikerstraße 4  
A-8010 Graz  
Austria  
Phone: +43-316-873-8300  
Fax: +43-316-873-8304  
[knapp@analytchem.tu-graz.ac.at](mailto:knapp@analytchem.tu-graz.ac.at)

Matheus KÖNING  
Wattgasse  
A-1160 Vienna  
Austria  
[matheus@yahoo.com](mailto:matheus@yahoo.com)

Ewa KOWALCZYK  
PanaLytica  
Rydygiera 8  
01-793 Warszawa  
Poland  
phone: +48-22-633-91-86  
fax: +48-22-669-39-67  
[EWA.KOWALCZYK@PANALYTICA.PL](mailto:EWA.KOWALCZYK@PANALYTICA.PL)

Francisco Jose KRUG  
CENA  
University of Sao Paulo  
13400-970 Piracicaba  
Avenida Centenario 303  
Brazil  
phone: +55-19-34294648  
fax: +55-19-34294610 or 34294647  
[fjkrug@cena.usp.br](mailto:fjkrug@cena.usp.br)

Doris KUEHNELT  
Institut für Chemie, Analytische Chemie  
Karl-Franzens-Universität Graz  
Universitätsplatz 1  
A-8010 Graz  
phone: 0043-316-380-8871  
fax: 0043-316-380-9845  
[doris.kuehnelt@uni-graz.at](mailto:doris.kuehnelt@uni-graz.at)

Markus LAFER  
Ea. Anton Paar  
Kärntnerstraße 322  
A-8054 Graz  
phone: +43-316-257-0  
fax: +43-316-257-257  
[lafer@anton-paar.com](mailto:lafer@anton-paar.com)

Ernst LANKMAYR  
Institute for Analytical Chemistry, Micro-  
and Radiochemistry  
Graz University of Technology  
Technikerstraße 4  
A-8010 Graz  
Austria  
phone: +43-316-873-8305  
fax: +43-316-873-8304  
[lankmayr@analytchem.tu-graz.ac.at](mailto:lankmayr@analytchem.tu-graz.ac.at)

Anita LEGENSTEIN  
Ea. Anton Paar  
Kärntnerstraße 322  
A-8054 Graz  
phone: +43-316-257-0  
fax: +43-316-257-257  
[legenstein@anton-paar.com](mailto:legenstein@anton-paar.com)

Sabine LONGIN  
ASA  
Schörgelgasse 53  
8010 Graz  
phone: +43-316-82-51-11  
fax: +43-316-81-09-26

Michael LUEBKE  
BASE AG  
Carl-Bosch-Straße 38 GKA – M320  
D-67056 Ludwigshafen  
Germany  
phone: +49-621-60-79788  
fax: +49-621-60-79460  
[michael.luebke@basf-ag.de](mailto:michael.luebke@basf-ag.de)

Brigitte MAICHIN  
Institute for Analytical Chemistry, Micro-  
and Radiochemistry  
Graz University of Technology  
Technikerstraße 4  
A-8010 Graz  
Austria  
phone: +43-316-873-8308  
fax: +43-316-873-8304  
[maichin@analytchem.tu-graz.ac.at](mailto:maichin@analytchem.tu-graz.ac.at)

Eveline MAJER  
PBI  
Körösisstraße 19  
A-8010 Graz  
phone: +43-316-671464  
fax: +43-316-671331-23

Claude MALLET  
Waters Corporation  
34 Maple St.  
01757-3696 Milford  
Massachusetts  
USA  
phone: 508-482-3045  
fax: 508-482-3100  
[claudemallet@waters.com](mailto:claudemallet@waters.com)

Henryk MATUSIEWICZ  
Politechnika Poznanska  
Dep. of Anal. Chem.  
Piotrowo 3  
60-965 Poland  
phone: 48-61-665-2312  
fax: 48-61-665-2571  
[Henryk.Matusiewicz@put.poznan.pl](mailto:Henryk.Matusiewicz@put.poznan.pl)

Goran MITULOVIC  
LC Packings-A Dionex Comp.  
Abberdaan 114  
1046 AA  
Amsterdam  
phone: +31-20-683-9768  
fax: +31-20-6853452  
[Goran.Mitulovic@lcpackings.nl](mailto:Goran.Mitulovic@lcpackings.nl)

Christoph MOSER  
Institute for Analytical Chemistry, Micro-  
and Radiochemistry  
Graz University of Technology  
Stremayrgasse 16/III  
A-8010 Graz  
Austria  
phone: +43-316-873-4323  
[moser@analytchem.tu-graz.ac.at](mailto:moser@analytchem.tu-graz.ac.at)

Sibylle MOTHE  
UFZ Centre for Environmental research  
Permoserstr. 15  
04318 Leipzig  
Germany  
phone: +49-341-235-2111  
fax: +49-341-235-2625  
[motbes@ana.vfz.de](mailto:motbes@ana.vfz.de)

Herbert MOTTER  
Institute for Analytical Chemistry, Micro-  
and Radiochemistry  
Graz University of Technology  
Technikerstraße 4  
A-8010 Graz  
Austria  
phone: +43-316-873-8303  
fax: +43-316-873-8304  
[motter@analytchem.tu-graz.ac.at](mailto:motter@analytchem.tu-graz.ac.at)

Herbert MUNTAU  
[Muntau@libero.it](mailto:Muntau@libero.it)

Joaquim NOBREGA  
Dept. Chemistry  
University Federal of Sao Carlos  
P.O. Box 676  
13565-905 Sao Carlos – SP  
phone: +55-16-260-8208  
fax: +55-16-260-8350  
[djan@terra.com.br](mailto:djan@terra.com.br)

Obiora OKOYE  
Okye Agriultura! firm  
G PO Box 18608 Dugbe  
Ibadan Oyo State  
234 Ibadan  
Nigeria  
[waliyiat@yahoo.com](mailto:waliyiat@yahoo.com)

Pedro Vitoriano OLIVEIRA  
Instituto de Quimica  
Universidade de Sao Paulo  
Av. Prof. Lineu Prestes, 748, B&S, Sala  
857.  
05508-900 Sao Paulo  
Brazil  
phone: +55-011-3091-3837  
fax: +55-011-3815-5579  
[pvoivei@quim.iq.usp.br](mailto:pvoivei@quim.iq.usp.br)

Taiwo Joseph OLUSEGUN  
Twince Chemical Firm  
P.O. Box 18608 Dugbe Ibadan  
234 Ibadan  
phone: 234-2-2318374  
fax: 234-2-2318374  
[broedu@yahoo.com](mailto:broedu@yahoo.com)

Markus OSTERMANN  
Isotopenverdünnungs- und  
Kernbrennstoffanalyse  
Bundesanstalt für Materialforschung und –  
prüfung  
Unter den Eichen 87  
D-12205 Berlin  
phone: +49-30-8104-4142  
fax: +49-30-8104-1147  
[markus.ostermann@bam.de](mailto:markus.ostermann@bam.de)

Dietmar PAAR

Ioannis PAPANAYANNIS  
University of Thessaloniki  
Lab. Analytical Chemistry  
Chemistry Department  
GR-54006 Thessaloniki  
phone: +30310-997793  
fax: +30310-997719  
[papadoya@chem.auth.gr](mailto:papadoya@chem.auth.gr)

Camillo PIROLA  
Milestone Srl  
Via Fatebenefratelli 1/5  
24010 Sorisole  
Italy  
Phone: +39-035-573857  
Fax: +39-035-575498  
[c.pirola@milestonesr.com](mailto:c.pirola@milestonesr.com)

Michael REIHER  
Berghof Laborprodukte GmbH  
Harretstraße 1  
D-72800 Eningen  
phone: 0049-7121-894-309  
fax: 0049-7071-9878-100  
[Reiher@berghof.com](mailto:Reiher@berghof.com)

Werner REPETSCHNIGG  
Ökopharm Forschungs- u. Entwicklungs-  
GmbH  
Mossham 29  
5580 Unternberg  
phone: +43-6476-805-0  
fax: +43-6476-805-555  
[wr@oekopharm.com](mailto:wr@oekopharm.com)

Gundel RIEBE  
Federal Institute of Materials Research and  
Testing  
Lab. I 42, Isotope Dilution and Nuclear  
Event Analysis  
Unter den Eichen 87  
D-12205 Berlin  
phone: +49-30-8104-4108  
fax: +49-30-8104-1147  
[gundel.riebe@bam.de](mailto:gundel.riebe@bam.de)

Eduard ROGATSKY  
Albert Einstein College of Medicine  
1300 Morris Park Ave., Rm. G47  
10461 New York  
USA  
phone: 718-430-3391  
fax: 718-430-8998  
[rogatsky@aecom.yu.edu](mailto:rogatsky@aecom.yu.edu)

Victoria SAMANIDOU  
University of Thessaloniki  
Laboratory of Analytical Chemistry  
GR-54006 Thessaloniki  
phone: +30310-997698  
fax: +30310-997719  
[samanidu@chem.auth.gr](mailto:samanidu@chem.auth.gr)

Dario SANTOS  
University of Sao Paulo  
Avenida Centenario 303  
13400-970 Piracicaba  
Brazil  
phone: +55-19-34294648  
fax: +55-19-34294610 or 34294647  
[dario@cena.usp.br](mailto:dario@cena.usp.br)

Volker SCHAIBLE  
CEM GmbH  
Carl-Friedrich-Gauss-Str. 9  
D-47475 Kamp-Lintfort  
Germany  
phone: +49-711-426991  
fax: +49-711-4201126  
[Volker.schaible@cem.com](mailto:Volker.schaible@cem.com)

Andreas SCHÖNAUER  
Voestalpine Stahl GmbH  
Stahlstr. 3  
A-4031 Linz  
Austria  
phone: +43-7-6585-3913  
fax: +43-70-6890-3496  
[andreas.schoenauer@voestalpine.com](mailto:andreas.schoenauer@voestalpine.com)

Axel SCHÖNER  
European Sales Office  
CEM International  
Amse!str. 6  
D-75239 Eisingen  
Germany  
phone: +49-7232-3830-28  
fax: +49-7232-3830-29  
[Axel.Schoener@cem.com](mailto:Axel.Schoener@cem.com)

Peter SCHRAMEL  
GSF-Forschungszentrum  
Institut für Ökologische Chemie  
AG Spurenelementanalytik und  
Metallspeziation  
Postfach 1129  
D-85758 Neuherberg  
Germany  
phone: +49-89-3187-4062  
fax: +49-89-3187-3348  
[peter.schramel@gsf.de](mailto:peter.schramel@gsf.de)

Sigma-Aldrich HandelsgesmbH  
Favoritengewerberg 10  
1100 Wien  
phone: 0043-1-605-81-14  
[Jbergstr@evnotes.sial.com](mailto:Jbergstr@evnotes.sial.com)

Kurt STUBENRAUCH  
Institute for Analytical Chemistry, Micro-  
and Radiochemistry  
Graz University of Technology  
Technikerstraße 4  
A-8010 Graz  
Austria  
phone: +43-316-873-8802  
fax: +43-316-873-8304

Steffen STRASSNIG  
Institute for Analytical Chemistry, Micro-  
and Radiochemistry  
Graz University of Technology  
Technikerstraße 4  
A-8010 Graz  
Austria  
Phone: +43-316-873-8307  
Fax: +43-316-873-8304  
[strossnig@analytchem.tu-graz.ac.at](mailto:strossnig@analytchem.tu-graz.ac.at)

Mark TATRO  
Spectrasol Inc.  
P.O. Box 1126  
10990 Warwick  
NY, USA  
phone: 845-987-1300  
fax: 845-987-1302  
[spectra9@optonline.net](mailto:spectra9@optonline.net)

Edith THOMSEN  
Elsam A/S  
Elsborgvej 185  
DK-6200 Aabenraa  
phone: +45-74-31-43-41  
fax: +45-74-31-41-59  
[eat@elsam.com](mailto:eat@elsam.com)

Klaudia TLACHAC  
Dionex Austria GmbH  
Laxenburgerstr. 220  
A-1230 Vienna  
Austria  
phone: 01-616-51-25  
fax: 01-616-51-25-55  
[klaudia.tlachac@dionex.at](mailto:klaudia.tlachac@dionex.at)

Cornelia WAGNER  
Fa. Anton Paar  
Kärntnerstraße 322  
A-8054 Graz  
Austria  
phone: +43-316-257-0  
fax: +43-316-257-257  
[wagner@anton-paar.com](mailto:wagner@anton-paar.com)

Raimund WAHLEN  
LGC Limited  
Queens Road  
TW11 0LY Teddington  
United Kingdom  
phone: +44-208-943-7661  
fax: +44-208-943-2767  
[Raimund.wahlen@lgc.co.uk](mailto:Raimund.wahlen@lgc.co.uk)

Silke WEBER  
TU-München  
Lichtenbergstr. 4  
D-84747 Garching  
Germany  
phone: +49-89-289-13136  
[Silke.Weber@lrz.tu-muenchen.de](mailto:Silke.Weber@lrz.tu-muenchen.de)

Wolfgang WEGSCHEIDER  
Montanuniversität Leoben  
Inst. f. Allg. u. Analyt. Chemie  
Franz-Josef-Str. 18  
A-8700 Leoben  
Austria  
phone: +43-3842-402340  
fax: +43-3842-402543  
[wegschei@uni-leoben.ac.at](mailto:wegschei@uni-leoben.ac.at)

Thomas WENZL  
Institute for Analytical Chemistry, Micro-  
and Radiochemistry  
Graz University of Technology  
Technikerstraße 4  
A-8010 Graz  
Austria  
phone: +43-316-873-8808  
fax: +43-316-873-8304  
[wenzl@analytchem.tu-graz.ac.at](mailto:wenzl@analytchem.tu-graz.ac.at)

Gerlinda WITA  
Perkin Elmer VertriebsgmbH  
Brunnerstraße 59/42  
A-1235-Vienna  
Austria  
phone: 088-111933  
Fax: +43-1-470-36-78  
[Gerlinda.wita@perkinelmer.com](mailto:Gerlinda.wita@perkinelmer.com)

Ursula WOLF  
Waters GmbH  
Hietzinger Hauptstraße 145  
A-1130 Vienna  
Austria  
phone: +43-1-877-1807  
fax: +43-1-877-18-08  
[ursula\\_wolf@waters.com](mailto:ursula_wolf@waters.com)

Karin WÖLKART  
Karl-Franzens-University of Graz  
Institute for Pharmacognosie  
Universitätsplatz 4  
A-8010 Graz  
Austria  
phone: +43-316-380-5534  
fax: +43-316-380-9860  
[karin.woelkart@ufanet.at](mailto:karin.woelkart@ufanet.at)

Mehdi YEKEHTAZ  
45194-159 Zanjan  
Iran  
phone: +98-241-424-9872  
fax: +98-241-424-9023  
[yekehtaz@iasbs.ac.ir](mailto:yekehtaz@iasbs.ac.ir)

**O!ga ZABUGA**

The International Slavic University

Kharkov Ukra

PO Box 10403

61002 Kharkov

phone: +380 50 588 1023

fax: +380 572 361637

[v!adpet@ukr.net](mailto:v!adpet@ukr.net)

**Michael ZISCHKA**

Institute for Analytical Chemistry, Micro-  
and Radiochemistry

Graz University of Technology

Technikerstraße 4

A-8010 Graz

Austria

phone: +43-316-873-8302

fax: +43-316-873-8304

[zischka@analytchem.tu-graz.ac.at](mailto:zischka@analytchem.tu-graz.ac.at)

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