

Article

Development and Validation of an Analytical Method for Determination of Al, Ca, Cd, Fe, Mg and P in Calcium-Rich Materials by ICP OES

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Abstract: Four procedures based on closed-vessel microwave-assisted wet digestion with different oxidative reagents, including HNO₃ (P1), HNO₃ + H₂O₂ (P2), aqua regia (P3) and *Lefort aqua regia* (P4), for preparation of calcium (Ca)-rich materials prior to determination of total concentrations of Al, Ca, Cd, Fe, Mg and P by inductively coupled optical emission spectrometry (ICP OES) were compared. It was found that digestion with *Lefort aqua regia* (P4) provided the best results for all examined elements, i.e., precision of 0.30–4.4%, trueness better than 2%, recoveries of added elements between 99.5–101.9%, and limits of detection within 0.08–1.8 ng g^{−1}. Reliability of this procedure was verified by analysis of relevant certified reference materials (CRMs), i.e., Natural Moroccan Phosphate Rock—Phosphorite (BCR-C02). Additionally, selection of appropriate analytical lines for measurements of element concentrations, linear dynamic ranges of calibration curves and matrix effects on the analyte response were extensively investigated. Finally, the selected procedure was successfully applied for routine analysis of other Ca-rich materials, i.e., CRMs such as NIST 1400 (Bone Ash), CTA-AC-1 (Apatite Concentrate Kola Peninsula) and NCS DC70308 (Carbonate Rock), and six natural samples, such as a dolomite, a phosphate rock, an enriched superphosphate fertilizer, pork bones, pork bones after incineration, and after steam gasification.

Keywords: calcium-rich materials; sample preparation; multi-element analysis; validation; ICP OES



Citation: Szymczycha-Madeja, A.; Welna, M.; Zabłocka-Malińska, M.; Pohl, P.; Szczepaniak, W. Development and Validation of an Analytical Method for Determination of Al, Ca, Cd, Fe, Mg and P in Calcium-Rich Materials by ICP OES. *Molecules* **2021**, *26*, 6269. <https://doi.org/10.3390/molecules26206269>

Academic Editor: José-Luis Todolí

Received: 20 September 2021

Accepted: 13 October 2021

Published: 16 October 2021

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1. Introduction

Calcium (Ca)-rich materials mainly include phosphorite, apatite, dolomite (sedimentary carbonate rock) and bones. Among them, the most important are those that contain phosphorus (P) in addition to Ca. Phosphorites (sedimentary rocks) and apatite rocks (volcanic sediments) are the two main raw materials used for production of P fertilizers [1,2]. Additionally, due to a high P content, the phosphorite is the most important source of this element for the European industry of phosphoric acid. The dolomite is mainly used for production of fertilizers, ceramic and refractory materials [3,4]. In the last years, utilization of bones as fertilizers represents a valuable recycling strategy [5,6]. Bones or other by-products obtained from their rendering industry may serve as an additional source of P. Each one can be divided into two main parts, i.e., inorganic and organic. The inorganic part contains mainly calcium phosphate (Ca₅(PO₄)₂), while the organic part is made of gelatin [7].

Due to the fact that Ca-rich materials are used in industry, elemental characterization is the first and essential step in evaluating their suitability for direct applications. In addition

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Practical Guide to ICP-MS and Other Atomic Spectroscopy Techniques Robert Thomas, 2023-09-29 Written by one of the very first practitioners of ICP MS Practical Guide to ICP MS and Other Atomic Spectroscopy Techniques A Tutorial for Beginners presents ICP MS in a completely novel and refreshing way By comparing it with other complementary atomic spectroscopy AS techniques it gives the trace element analysis user community a glimpse into why the technique was first developed and how the application landscape has defined its use today 40 years after it was first commercialized in 1983 What's new in the 4th edition Updated chapters on the fundamental principles and applications of ICP MS New chapters on complementary AS techniques including AA AF ICP OES MIP AES XRF XRD LIBS LALI TOFMS Strategies for reducing errors and contamination with plasma spectrochemical techniques Comparison of collision and reaction cells including triple multi quad systems Novel approaches to sample digestion Alternative sample introduction accessories Comprehensive glossary of terms used in AS New vendor contact information The book is not only suited to novices and beginners but also to more experienced analytical scientists who want to know more about recent ICP MS developments and where the technique might be heading in the future Furthermore it offers much needed guidance on how best to evaluate commercial AS instrumentation and what might be the best technique based on your lab's specific application demands I feel honored to have been asked to deliver the Foreword for this book which is suited not only for beginners but also for more experienced analytical scientists who want to know the advances in plasma spectrochemistry instrumentation and related future opportunities Dr Heidi Goenaga Infante LGC Science Fellow Chief Scientist National Measurement Laboratory Visiting Professor University of Strathclyde UK

Analytical Techniques for Trace Elements in Geochemical Exploration

Raghav Saran, 2022-12-07 Trace element analysis plays a prominent role in various fields from mineralogy and geology to semiconductor manufacture and foods In geochemical exploration the analysis of trace elements assumes high significance due to the multifaceted role played by them The analyte is at the detection limit of many instrumental techniques This makes their determination difficult This book covers a wide spectrum of destructive and non destructive analytical techniques and recent developments in them used all over the world including developing countries for quantitation of trace elements With revolutionary progress in the last three to four decades in analytical techniques several ICP based techniques like ICP OES and ICP MS and other nuclear analytical techniques have enabled determination of trace elements at the ppb level However these methods require expensive instrumentation and cannot be made available everywhere The quality of analytical data is dependent on valid reference standards The book contains detailed sample preparation in varying matrices and an important chapter on statistical treatment of analytical data for the purpose of quality control and quality assurance Pulling together the book containing the work carried out by the author's group in India will be useful to analysts involved in geochemical explorations

Validation in Chemical Measurement

Paul De Bièvre, Helmut Günzler, 2005-01-12 The validation of

analytical methods is based on the characterisation of a measurement procedure selectivity sensitivity repeatability reproducibility This volume collects 31 outstanding papers on the topic mostly published in the period 2000 2003 in the journal Accreditation and Quality Assurance They provide the latest understanding and possibly the rationale why it is important to integrate the concept of validation into the standard procedures of every analytical laboratory In addition this anthology considers the benefits to both the analytical laboratory and the user of the measurement results

Automationslösungen in der analytischen Messtechnik Heidi Fleischer, Kerstin Thurow, 2019-10-28 Das Buch behandelt die Entwicklung und Systematisierung geeigneter Automatisierungssysteme in der analytischen Messtechnik Anwendungen aus verschiedenen Bereichen werden vorgestellt beispielsweise aus der Umweltmesstechnik Arzneimittelentwicklung und Qualitätssicherung

Advances in Analytical Techniques for Forensic Investigation Priyanka Chhabra, Divya Bajpai Tripathy, Anjali Gupta, Shruti Shukla, Rajeev Kumar, Kajol Bhati, 2024-08-06 This book is essential for anyone seeking to understand and apply the latest analytical techniques in forensic investigation saving time materials energy and manpower by providing guidance on the most appropriate techniques for different types of investigations Advances in Analytical Techniques for Forensic Investigation is aimed to describe the applicability of different types of analytical techniques used for the forensic investigation including FT IR chromatography mass spectroscopy NMR spectroscopy atomic absorption spectroscopy UV vis spectroscopy etc This book will focus on current and emerging developments in the latest analytical techniques and methods used in the forensic investigation and sample analysis of various physical chemical and biological samples in order to facilitate the smooth conduction of justice

Metals and Related Substances in Drinking Water Prosun Bhattacharya, Ingegerd Rosborg, Arifin Sandhi, Colin Hayes, Maria Joao Benoliel, 2011-11-25 Part of Metals and Related Substances in Drinking Water Set buy all five books together to save over 30% Metals and Related Substances in Drinking Water comprises the proceedings of COST Action 637 METEAU held in Kristianstad Sweden October 13 15 2010 This book collates the understanding of the various factors which control metals and related substances in drinking water with an aim to minimize environmental impacts Metals and Related Substances in Drinking Water Provides an overview of knowledge on metals and related substances in drinking water Promotes good practice in controlling metals and related substances in drinking water Helps to determining the environmental and socio economic impacts of control measures through public participation Introduces the importance of mineral balance in drinking water especially when choosing treatment methods Shares practitioner experience The proceedings of this international conference contain many state of the art presentations by leading researchers from across the world They are of interest to water sector practitioners regulators researchers and engineers

A Practical Approach to Quantitative Metal Analysis of Organic Matrices Martin Brennan, 2008-09-15 There has been significant expansion in the application of atomic spectrographic techniques in recent years which has brought with it the need to provide more flexible methods to a wider range of samples particularly non aqueous samples This book compares

the traditional and improved methods in the analysis of non aqueous samples for elemental analyses by atomic emission spectroscopic methods whilst describing procedures that will attempt to improve sample preparation methods

Analytical Techniques for Clinical Chemistry Sergio Caroli, Gyula Záray, 2012-06-26 Discover how analytical chemistry supports the latest clinical research This book details the role played by analytical chemistry in fostering clinical research Readers will discover how a broad range of analytical techniques support all phases of clinical research from early stages to the implementation of practical applications Moreover the contributing authors careful step by step guidance enables readers to better understand standardized techniques and steer clear of everyday problems that can arise in the lab Analytical Techniques for Clinical Chemistry opens with an overview of the legal and regulatory framework governing clinical lab analysis Next it details the latest progress in instrumentation and applications in such fields as biomonitoring diagnostics food quality biomarkers pharmaceuticals and forensics Comprised of twenty five chapters divided into three sections exploring Fundamentals Selected Applications and Future Trends the book covers such critical topics as Uncertainty in clinical chemistry measurements Metal toxicology in clinical forensic and chemical pathology Role of analytical chemistry in the safety of drug therapy Atomic spectrometric techniques for the analysis of clinical samples Biosensors for drug analysis Use of X ray techniques in medical research Each chapter is written by one or more leading pioneers and experts in analytical chemistry Contributions are based on a thorough review and analysis of the current literature as well as the authors own firsthand experiences in the lab References at the end of each chapter serve as a gateway to the literature enabling readers to explore individual topics in greater depth Presenting the latest achievements and challenges in the field Analytical Techniques for Clinical Chemistry sets the foundation for future advances in laboratory research techniques

The Analytical Chemistry Laboratory Companion Michael D. Holloway, 2025-06-02 The Analytical Chemistry Laboratory Companion is essential for both students and professionals as it provides quick clear explanations on critical topics in analytical chemistry equipping you with the statistical tools necessary to ensure accurate and reliable data interpretation The Analytical Chemistry Laboratory Companion serves as a reference guide for students and professionals alike who need quick explanations on specific topics laboratory operations the structure of designing experiments and the use of statistics to gain increased accuracy precision repeatability and reproducibility of data This volume will also provide in depth and advanced studies and build the necessary background knowledge for success in the field This companion provides a concise examination of the various analytical tools used for chemistry and defines basic analytical instrument principles techniques and applications in addition to exploring statistical tools useful in data interpretation test result reporting and common root causes for faulty data with suggested remedies The introduction provides a concise guide on foundational topics such as developing standard operating procedures laboratory safety instrumental analytical methods and common statistical tools useful for data interpretation This companion covers both wet chemical and instrumental analysis including

their principles applications and pitfalls The Analytical Chemistry Laboratory Companion is a must have comprehensive guide in the field of analytical chemistry *Elemental Analysis* Frank Vanhaecke, José Luis Todolí, Gerhard Schlemmer, Michael W. Hinds, 2025-06-16 Elemental Analysis is an excellent guide introducing cutting edge methods for the qualitative and quantitative analysis of elements Each chapter gives an overview of a certain technique such as AAS AFS ICP OES MIP OES ICP MS and XRF Readers will benefit from a combination of theoretical basics operational principles of instruments and their applications New extended section on supply of liquid samples for AAS ICP OES and ICP MS

Analytical Techniques in Forensic Science Rosalind Wolstenholme, Sue Jickells, Shari Forbes, 2021-01-26 An in depth text that explores the interface between analytical chemistry and trace evidence Analytical Techniques in Forensic Science is a comprehensive guide written in accessible terms that examines the interface between analytical chemistry and trace evidence in forensic science With contributions from noted experts on the topic the text features a detailed introduction analysis in forensic science and then subsequent chapters explore the laboratory techniques grouped by shared operating principles For each technique the authors incorporate specific theory application to forensic analytics interpretation forensic specific developments and illustrative case studies Forensic techniques covered include UV Vis and vibrational spectroscopy mass spectrometry and gas and liquid chromatography The applications reviewed include evidence types such as fibers paint drugs and explosives The authors highlight data collection subsequent analysis what information has been obtained and what this means in the context of a case The text shows how analytical chemistry and trace evidence can problem solve the nature of much of forensic analysis This important text Puts the focus on trace evidence and analytical science Contains case studies that illustrate theory in practice Includes contributions from experts on the topics of instrumentation theory and case examples Explores novel and future applications for analytical techniques Written for undergraduate and graduate students in forensic chemistry and forensic practitioners and researchers Analytical Techniques in Forensic Science offers a text that bridges the gap between introductory textbooks and professional level literature *X-Ray Fluorescence in Biological Sciences* Vivek K. Singh, Jun Kawai, Durgesh K. Tripathi, 2022-03-28 X Ray Fluorescence in Biological Sciences Discover a comprehensive exploration of X ray fluorescence in chemical biology and the clinical and plant sciences In X Ray Fluorescence in Biological Sciences Principles Instrumentation and Applications a team of accomplished researchers delivers extensive coverage of the application of X ray fluorescence XRF in the biological sciences including chemical biology clinical science and plant science The book also explores recent advances in XRF imaging techniques in these fields The authors focus on understanding and investigating the intercellular structures and metals in plant cells with advanced discussions of recently developed micro analytical methods like energy dispersive X ray fluorescence spectrometry EDXRF total reflection X ray fluorescence spectrometry TXRF micro proton induced X ray emission micro PIXE electron probe X ray microanalysis EPXMA synchrotron based X ray fluorescence microscopy SXRF SRIXE or micro XRF and secondary ion mass spectrometry

SIMS With thorough descriptions of protocols and practical approaches the book also includes A thorough introduction to the historical background and fundamentals of X ray fluorescence as well as recent developments in X ray fluorescence analysis Comprehensive explorations of the general properties production and detection of X rays and the preparation of samples for X ray fluorescence analysis Practical discussions of the quantification of prepared samples observed under X ray fluorescence and the relation between precision and beam size and sample amount In depth examinations of wavelength dispersive X ray fluorescence and living materials Perfect for students and researchers studying the natural and chemical sciences medical biology plant physiology agriculture and botany X Ray Fluorescence in Biological Sciences Principles Instrumentation and Applications will also earn a place in the libraries of researchers at biotechnology companies

Soil and Environmental Analysis Keith A. Smith, Malcolm S. Cresser, 2003-10-15 Evaluating traditional and recent analytical methods according to speed sensitivity and cost efficiency this reference supports specialists in the selection of effective analytical techniques and equipment for the study of soils soil contaminants and environmental samples Updated and revised this Third Edition illustrates the advantages limitations range and challenges of the major analytical approaches utilized in modern research laboratories It includes new chapters and expanded discussions of the measurement of organic pollutants in the environment and gas fluxes between the land surface and atmosphere and an extensive range of environmental materials

Advances in Portable X-ray Fluorescence Spectrometry B Lee Drake, Brandi L MacDonald, 2022-10-19 Over the last two decades advances in the design miniaturization and analytical capabilities of portable X ray fluorescence pXRF instrumentation have led to its rapid and widespread adoption in a remarkably diverse range of applications in research and industrial fields The impetus for this volume was that as pXRF continues to grow into mainstream use analysts should be increasingly empowered with the right information to safely and effectively employ pXRF as part of their analytical toolkit This volume provides introductory and advanced level users alike with readings on topics ranging from basic principles of pXRF and qualitative and quantitative approaches through to machine learning and artificial intelligence for enhanced applications It also includes fundamental guidance on calibrations the mathematics of calculating uncertainties and an extensive reference index of all elements and their interactions with X rays Contributing authors have provided a wealth of information and case studies in industry specific chapters These sections delve into detail on current standard practices in industry and research including examples from agricultural and geo exploration sectors research in art and archaeology and metals industrial and regulatory applications As pXRF continues to grow in use in industrial and academic settings it is essential that practitioners continue to learn share and implement informed and effective use of this technique This volume serves as an accessible guidebook and go to reference manual for new and experienced users in pXRF to achieve this goal

Plastics Additives Jan C. J. Bart, 2006 Contains an outline of the principles and characteristics of relevant instrumental techniques provides an overview of various aspects of direct additive analysis by focusing on an array of applications in R ampD production quality control and

technical service **Advances in Microwave Chemistry** Bimal K Banik, Debasish Bandyopadhyay, 2018-12-07 Advances in Microwave Chemistry discusses the novel bond formation methodologies synergistic effects of microwaves with other entities sample preparation including digestion combustion and extraction techniques as well as selectivity in chemical processes Recent updates are provided on microwave assisted syntheses of pharmacologically significant aza oxo and other heterocycles including lactams nucleosides bile acids and sterols the preparation of nanomaterials composites and absorber layer materials for thin film This book also incorporates comparative discussions involving microwave irradiation with conventional methods in different aspects of organic inorganic medicinal and green chemistry Key Features Provides a comparative discussion on microwave irradiation with conventional methods in different aspects of organic inorganic medicinal and green chemistry Presents recent applications of microwave radiation in biocatalysis Offers a complete package correlating various aspects of microwaves in organic syntheses the biological impact of products formed in reactions pharmacological features and environmental sustainability of the procedures Explains microwave induced reactions on structurally complex bile acids and sterols Stands as a valuable and unique addition to the well established book series New Directions in Organic and Biological Chemistry Glow Discharge Plasmas in Analytical Spectroscopy R. Kenneth Marcus, José A. C. Broekaert, 2003-05-27 This multi author edited volume includes chapters which deal with both basic and highly complex applications Glow discharge devices are now being used in very novel ways for the analysis of liquids and gases including molecular species detection and identification an area that was beyond the perceived scope of applicability just ten years ago It is expected that the next decade will see a growth in the interest and application of glow discharge devices far surpassing the expectations of the last century Responding to the rapid growth in the field Includes both GD MS and GD AES In depth coverage of applications Co edited by the top names in the field in Europe and US with high calibre contributions from around the world Manual of Standard Operating Procedures for Selected Chemical Residue and Contaminant Analysis Food and Agriculture Organization of the United Nations, International Atomic Energy Agency, 2021-09-15 Food safety is an important global public health and trade matter with chemical hazards occupying centre stage due to associated acute and chronic health outcomes There is also an increasing need to address antimicrobial resistance concerns While food remains a major vehicle for exposure to these hazards related matrices cannot be ignored Animal feed for instance may contain drug or pesticide residues as well as mycotoxins that could carry over to food either as parent compounds or their metabolites of toxicological relevance Contaminated water is also another medium of potential exposure to food hazards A concerted effort is required to address the need for a safe food supply and one critical stakeholder is the testing laboratory While this requires trained and capable analysts as well as reliable instrumentation analytical methods are a major need Development and validation to ensure fitness of purpose and availability of these methods is a necessity This manual consisting of several Standard Operating Procedures SOPs presents another opportunity

for laboratories to address gaps in analytical methods and or expand their options The manual contains techniques for analyzing certain mycotoxins such as aflatoxins fumonisin and ochratoxin in matrices that include milk edible vegetable oil and animal feed etc A range of veterinary drug residues including permitted and prohibited substances in animal matrices including fish are also addressed Several pesticide residues in cereals fruits and vegetables are also covered A couple of methods for analysis of selected metals are also presented

MODERN PHARMACEUTICAL ANALYTICAL

TECHNIQUES Mrs. Mitali Yogesh Patil, Mr. Biprojit Paul, Mr. Mohit Chadha, Dr. Saroj Kumar Raul, In the dynamic realm of pharmaceutical sciences this project explores Modern Pharmaceutical Analytical Techniques delving into cutting edge methodologies crucial for ensuring the quality and efficacy of drugs From spectroscopy to advanced technologies like metabolomics each chapter demystifies the application and significance of these techniques Bridging academia and industry this work aims to be a practical guide underlining the realworld implications of these tools Gratitude is extended to mentors colleagues and institutions as this concise exploration seeks to serve students researchers and professionals navigating the ever evolving landscape of pharmaceutical analysis

Handbook of Food Analysis - Two Volume Set Leo M.L.

Nollet, Fidel Toldra, 2015-06-10 Updated to reflect changes in the industry during the last ten years The Handbook of Food Analysis Third Edition covers the new analysis systems optimization of existing techniques and automation and miniaturization methods Under the editorial guidance of food science pioneer Leo M L Nollet and new editor Fidel Toldra the chapters take an in

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