

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



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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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Calibration and Validation of Analytical Methods Mark Stauffer, 2018-04-25 This book seeks to introduce the reader to current methodologies in analytical calibration and validation This collection of contributed research articles and reviews addresses current developments in the calibration of analytical methods and techniques and their subsequent validation Section 1 Introduction contains the Introductory Chapter a broad overview of analytical calibration and validation and a brief synopsis of the following chapters Section 2 Calibration Approaches presents five chapters covering calibration schemes for some modern analytical methods and techniques The last chapter in this section provides a segue into Section 3 Validation Approaches which contains two chapters on validation procedures and parameters This book is a valuable source of scientific information for anyone interested in analytical calibration and validation *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 *Detection, Identification, and Monitoring of Food Contaminants* Ogwu, Matthew Chidozie, Izah, Sylvester Chibueze, 2025-09-17 The detection identification and mitigation of food contaminants are critical in food safety and public health Contaminants enter the food supply chain at several points from production and processing to storage and distribution Advances in analytical techniques such as spectroscopy chromatography and molecular diagnostics improve the ability to detect and identify harmful substances in food Once identified effective mitigation strategies including regulatory controls good manufacturing practices and advanced decontamination technologies are essential to reduce risk and prevent foodborne illnesses This approach plays a pivotal role in maintaining consumer confidence and safeguarding global food systems *Detection Identification and Monitoring of Food Contaminants* explores the latest and most critical developments in the detection identification and mitigation of food contaminants that have recently surfaced or have been under recognized It presents current research methodologies new technologies and innovative approaches for addressing food contamination and proposes strategies for improving food safety across the global food system This book covers topics such as genetics microplastics and water quality and is a useful resource for business owners engineers biologists academicians researchers and environmental scientists **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

Analytical Chemistry of Uranium Zeev Karpas, 2014-11-21 Accurate uranium analysis and particularly for isotope measurements is essential in many fields including environmental studies geology hydrogeology the nuclear industry health physics and homeland security Nevertheless only a few scientific books are dedicated to uranium in general and analytical chemistry aspects in particular *Analytical Chemistry of Uranium Environmental Forensic Nuclear and Toxicological Applications* covers the fascinating advances in the field of analytical chemistry of uranium Exploring a broad range of topics the book focuses on the analytical aspects of industrial processes that involve uranium its presence in the environment health and biological implications of exposure to uranium compounds and nuclear forensics Topics include Examples of procedures used to characterize uranium in environmental samples of soil sediments vegetation water and air Analytical methods used to examine the rigorous specifications of uranium and its compounds deployed in the nuclear fuel cycle Health aspects of exposure to uranium and the bioassays used for exposure assessment Up to date analytical techniques used in nuclear forensics for safeguards in support of non proliferation including single particle characterization Each chapter includes an overview of the topic and several examples to demonstrate the analytical procedures This is followed by sample preparation separation and purification techniques where necessary The book supplies readers with a solid understanding of the analytical chemistry approach used today for characterizing the different facets of uranium providing a good starting point for further investigation into this important element

Handbook of Dairy Foods Analysis Fidel Toldrá, Leo M.L. Nollet, 2021-03-29 Dairy foods account for a large portion of the Western diet but due to the potential diversity of their sources this food group often poses a challenge for food scientists and their research efforts Bringing together the foremost minds in dairy research *Handbook of Dairy Foods Analysis Second Edition* compiles the top dairy analysis techniques and methodologies from around the world into one well organized volume Exceptionally comprehensive in both its detailing of methods and the range of dairy products covered this handbook includes tools for analyzing chemical and biochemical compounds and also bioactive peptides prebiotics and probiotics It describes noninvasive chemical and

physical sensors and starter cultures used in quality control This second edition includes four brand new chapters covering the analytical techniques and methodologies for determining bioactive peptides preservatives activity of endogenous enzymes and sensory perception of dairy foods and all other chapters have been adapted to recent research All other chapters have been thoroughly updated Key Features Explains analytical tools available for the analysis of the chemistry and biochemistry of dairy foods Covers a variety of dairy foods including milk cheese butter yogurt and ice cream Analysis of nutritional quality includes prebiotics probiotics essential amino acids bioactive peptides and healthy vegetable origin compounds Includes a series of chapters on analyzing sensory qualities including color texture and flavor Covering the gamut of dairy analysis techniques the book discusses current methods for the analysis of chemical and nutritional compounds and the detection of microorganisms allergens contaminants and or other adulterations including those of environmental origin or introduced during processing Other methodologies used to evaluate color texture and flavor are also discussed Written by an international panel of distinguished contributors under the editorial guidance of renowned authorities Fidel Toldr and Leo M L Nollet this handbook is one of the few references that is completely devoted to dairy food analysis an extremely valuable reference for those in the dairy research processing and manufacturing industries Latex and Synthetic Polymer Dispersions 2013 Smithers Rapra,2013-11-13 The 8th Smithers Rapra conference on Latex and Synthetic Polymer Dispersions gave a very broad picture of the industry These proceedings cover all the presentations from the two day event which included The scientific principles underlying latex dipping were described by Professor C C Ho and Dr Aik Hwee Eng of Ansell spoke about a modern result of dipping the antimicrobial glove Very interesting observations about the allergenic potential of synthetic latex gloves compared to those dipped from natural rubber were made by Hardi Tamm of Korymbos The use of gamma radiation from the very start of the process as a means of prevulcanization to the end of the production process in sterilization was described by Dr Rosamma Alex of the Rubber Research Institute of India and Eric Beers of Nordion respectively The versatility of natural latex was demonstrated in a paper by Dr Azura of Universiti Sains Malaysia who showed us how it can be used for the cleaning of compression moulds Innovative polymer synthesis in the manufacture of latex dispersions was presented by Dr Joachim Storsberg of the Fraunhofer Institute and Dr Soeren Butz of Synthomer told how more clever chemistry could be used to e tailor makee pressure sensitive adhesives The environmental side of the industry was not forgotten with two presentations from the Malaysian Rubber Board Muhammad D Syraarani describing an environmentally friendly method for the analysis of magnesium in latex and Dr Devaraj Veerasamy presenting the use of ultrafiltration to process latex In a similar vein Prof Khairah Haji Badri of the Universiti Tun Abdul Rahman showed how natural resources such as palm oil can be used to create useful polymers David Hill of David Hill and Associates described how to carry out Process Validation of dipped condoms and gloves and the delegates were told how the newest latex for dipping synthetic polyisoprene compares with the oldest natural rubber by Dr Bert Krutzer of Kraton The conference ended

with Dr Siby Varghese of the Rubber Research Institute of India and Prof Sabu Thomas of the Mahatma Gandhi University describing recent advances and applications in the field of nanotechnology **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 *Handbook of Methods Used in Rhizosphere Research* Jörg Luster, Roger D. Finlay, 2006

Comprehensive Analytical Chemistry Cecil Leeburn Wilson, David Woodburn Wilson, 2003 **Analytical Applications of Nuclear Techniques**, 2004 The IAEA has compiled this overview of current applications of nuclear analytical techniques NATs The contributions included in this book describe a variety of nuclear techniques and applications such as those in the fields of environment and health industrial processes non destructive testing forensic and archaeological investigations cosmochemistry and method validation The techniques covered range from classical instrumental neutron activation analysis INAA its radiochemical derivative RNAA in beam methods such as prompt γ neutron activation analysis PGNA and accelerator mass spectrometry AMS to X ray fluorescence XRF and proton induced X ray emission PIXE spectroscopy Isotopic techniques to investigate element behaviour in biology and medicine and also to validate other non nuclear analytical techniques are described Destructive and non destructive approaches are presented along with their use to investigate very small and very large samples archaeological samples and extraterrestrial samples Several nuclear analytical applications in industry are described that have considerable socioeconomic impact wherever they can be implemented

Speciation Analysis of Arsenic, Chromium and Selenium in Aquatic Media International Atomic Energy Agency, 2007 This publication presents the results of a project analysing the speciation of As 3 As 5 Cr 3 Cr 6 and Se 4 Se 6 in water samples Total element concentration does generally not provide adequate information to understand the effects of trace and heavy metals observed in the environment and in living systems The toxicity bioavailability physiological and metabolic processes and the mobility and fate of elements are to a large extent dependent on the specific chemical form of an element Speciation analysis has yet to be developed to its full potential for biochemical clinical and environmental investigations and still more work is needed in the near future However nuclear techniques particularly the use of radioisotopes can help in method development optimization and stability tests of chemical species **Elements and their Compounds in the Environment** Ernest Merian, Manfred Anke, Milan Ihnat, Markus Stoepler, 2004 Since the last edition was published in 1991 the Merian has established itself as the standard reference on this topic and remains unmatched in the breadth of material covered This new edition has 20 % more pages while being more clearly and concisely structured New topical features are the focus on nutritional aspects and the coverage of further elements including non metals The newly added chapters cover more than 15 essential elements as well as some of environmental and nutritional importance While

many chapters of the previous edition have been practically rewritten to accommodate the rapid progress made in this field all the contributions have been revised and updated to present the current state of knowledge As such the handbook continues to provide detailed information on the environmental metals that influence the health of plants animals and humans with particular attention given to environmental and analytical chemistry bioavailability metabolic pathways and biological effects It also delineates the problems related to waste soils and wildlife as well as the risks caused by the increasing output of metals from industry and households It retains the proven features of the first edition such as the extensive bibliography numerous tables with useful data and a glossary of terms and once again all the contributions were not only written but also reviewed by acknowledged and experienced experts In total international experts from 15 countries have pooled their knowledge and experience to create this ultimate resource Essential information for chemists biologists geologists food scientists toxicologists and physiologists involved in environmental research and remediation risk assessment food research and industrial hygiene

Elements and Their Compounds in the Environment Manfred Anke, Milan Ihnat, Markus Stoeppler, 2004-03-26 Since the last edition was published in 1991 the Merian has established itself as the standard reference on this topic and remains unmatched in the breadth of material covered This new edition has 20 % more pages while being more clearly and concisely structured New topical features are the focus on nutritional aspects and the coverage of further elements including non metals The newly added chapters cover more than 15 essential elements as well as some of environmental and nutritional importance While many chapters of the previous edition have been practically rewritten to accommodate the rapid progress made in this field all the contributions have been revised and updated to present the current state of knowledge As such the handbook continues to provide detailed information on the environmental metals that influence the health of plants animals and humans with particular attention given to environmental and analytical chemistry bioavailability metabolic pathways and biological effects It also delineates the problems related to waste soils and wildlife as well as the risks caused by the increasing output of metals from industry and households It retains the proven features of the first edition such as the extensive bibliography numerous tables with useful data and a glossary of terms and once again all the contributions were not only written but also reviewed by acknowledged and experienced experts In total international experts from 15 countries have pooled their knowledge and experience to create this ultimate resource Essential information for chemists biologists geologists food scientists toxicologists and physiologists involved in environmental research and remediation risk assessment food research and industrial hygiene

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