

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 (Ca₃(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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