

Prepare and Characterization of a Soil Reference Material for Inorganic Nutrients and Contaminants

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The certified reference materials make an important role in the establishment of traceability in analytical chemistry, ensuring reliability and accuracy to the measurements and are fundamental for the validation of analytical methods. The demand for new certified reference materials increases in all areas of knowledge, with emphases in the agriculture. Basis for the production of farming products, the demand for soil analysis is great and the existence of a reference material of a typical Brazilian soil is important to ensure the quality of results. In this context, a reference material for Cr, Pb, Cd, and As soil contaminants and Ca, P, Mg, K, Cu, Fe, Mn, Zn, and Na macro and micronutrients was prepared and characterized.

The material was produced following the internationally accepted ISO Guides 30-35 norms considering the preparation and packaging, homogeneity, short and long term stability, material characterization and certification. A Typical Paleudult soil was previously artificially contaminated with 25 mmol L⁻¹ of As, Cd, Cr, and Pb. After 12 month of contamination, 65 kg of soil was dried at room temperature, ground (particles size < 390µm), homogenized, packaged and gamma ray irradiated (20 - 25 kGy) aiming to increase the shelf-life. The analyte soil extractions were performed by assisted microwave oven with the use of 14 mol L⁻¹ HNO₃, based on the USEPA 3051. The evaluation of the estimate minimum amount, homogeneity and stability of the material were carried out by using inductively coupled plasma optical emission spectroscopy (ICP OES) determination technique. The results were submitted to analysis of variance of single factor (ANOVA). The homogeneity and stability of the material presented means within a confidence interval of 95% for As, Cd, Pb, Cr, Ca, Cu, Fe, Na, K, Mg, Mn, and P. The chemical characterization of the material was performed through a collaborative test and the uncertainties related to each step of the material prepare were calculated to obtain the expanded uncertainty for each one of the analytes and to the final drafting of the control chart containing the results of the reference material.

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