

# EMEC21

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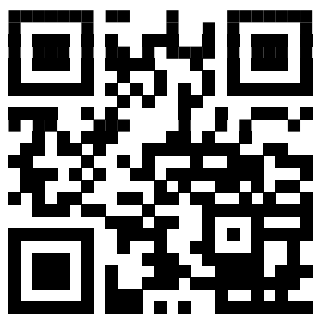
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## BOOK OF ABSTRACTS





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## Effect of Sample Preparation on Portable X-Ray Fluorescence Spectrometry Analysis of Contaminated Soils

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Toxic metals in soil are routinely determined by several analytical spectroscopic techniques (Atomic Absorption Spectrometry AAS, Inductively Coupled Plasma Optical Emission Spectrometry ICP-OES, and Inductively Coupled Plasma Mass Spectrometry ICP-MS)[1]. Those techniques measure metals from aqueous samples. Procedures of sample dissolution or extraction typically involve a lengthy process which requires the use of harsh conditions. Sample preparation procedures make these routinely used techniques generally time-consuming and too expensive [2]. On the other side, the need for reliable, economical, and environmental friendly technique for soil composition measuring has been growing in the environmental field, so has the demand for time and cost-efficient analytical methods for soil analysis [3].

X-ray fluorescence spectrometry (XRF) is a multi-element analytical technique for direct, non-destructive analysis of various materials (including soils) with minimal sample preparation. The most attractive advantage of XRF is the wide dynamic range (from mg kg<sup>-1</sup> to 100%). A portable X-ray fluorescence spectrometer (PXRF) is also capable of in-situ analysis in a short time (30–120 s) [4].

In situ PXRF analysis provides flexibility and allows rapid collection of data for a large number of samples, and produces real-time data that can be used for rapid decision making. It is well-known that the physical characteristics of the sample play an important role in obtaining accurate results when it comes to XRF methods. Therefore it is important to determine how reliable in situ PXRF results are.

Analytical accuracy and precision could be generally improved if adequate sample preparation procedure is applied compared to in situ measurements.

The aim of this research was to determine in what extent sample preparation procedure changes measured concentrations of elements and is that change the same

for all investigated elements. Does soil sample homogenization or further pressing into the compact pellet systematically affect measured concentrations?

Soil samples from 32 industrial, potentially contaminated sites were collected from a depth of 10 cm, 30 cm, and 50 cm. Such soils provide wide concentration range of different elements. Samples were first directly analyzed in the field, without any sample preparation using the Thermo Scientific™ Niton™ XL3t GOLDD<sup>+</sup> PXRF Analyzer. The second PXRF analysis was performed in the laboratory on the dry, ground, and homogenized soil powder sample. One aliquot of soil powder was digested for AAS analysis, while another aliquot was pressed into a 32 mm diameter pellet and analyzed using PXRF.

The quality control program involves comparison of the results with AAS reference technique. Additionally, certified reference materials of stream sediment (STSD-3) and soil (NCS DC 77301) are analyzed with different sample preparation procedures.

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