Xenemetrix

The Power to Change Energy Into Information

Quantitative elemental analysis of soil samples

EDXRF Analyzer: X-Calibur

equipped with Silicon Drift Detector

By: Riham Mahroum

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Abstract

Qualitative and quantitative analysis of major oxides and trace elements in three different soil samples were performed with Xenemetrix EDXRF analyser, model X-Calibur equipped with a high performance Silicon Drift Detector (SDD).
Quantification of the elemental composition was performed using a Standard less Fundamental parameter (SLFP) method that does not need any calibration standards.

Objective

- Qualitative analysis to determine the elemental content
- Quantitative analysis using SLFP method to determine the concentration of each component in the samples.
- To evaluate the instrument performance and the method repeatability

Background

EDXRF is an ideal method for a quick and simple elemental analysis tests for industrial control purposes. This analytical technique is extremely quick, noninvasive, and requiring minimal sample preparation. It can easily be operated by non-skilled personnel in the production line.
The analysis by Fundamental Parameter software is based upon theoretical equations and fundamental-parameter database, such as all the absorption of X-rays and the inter-elements effects and FP equations to describe fluorescence and scattering effects resulting from x rays interaction with the sample. The concentration results obtained by this method can be significantly improved by using a single reference standard to calculate theoretical calibration coefficients used in the FP calculations.

Analytical Configuration

Table 1: Instrumental analytical configuration

Instrument	X-Calibur SDD Bench top system
Anode	Rh-Anode X-ray Tube, 50kV,50W
Detector	Silicon Drift Detector (SDD)
Environment	Vacuum
Type of analysis	Quantitative analysis
Analysis time	180 sec
Sample Preparation	No sample preparation

Experimental and Results

Three different soil samples in powder form were received for qualitative and quantitative analysis using a Standard less fundamental parameter method.

The spectra acquisition was performed in special X ray cups with special 4µm thick prolene thin film support, in vacuum environment. Vacuum is essential for the analysis of low molecular weight elements, since otherwise the oxygen in the X-ray beam path absorbs the low energy signal emitted by the light elements.

Qualitative analysis:
A Typical spectrum is shown in figures 1a and 1b in two parts to show the spectral peaks better.

Quantitative analysis
To determine the concentrations of the different elemental component found by the qualitative analysis, each of the three soil samples were measured 10 times consecutively without moving the samples in between acquisitions. The spectral data was processed using a Standardless fundamental parameter method.

The Static precision test performed on the three soil samples show the repeatability of the method and the performance of X-Calibur Analyzer. The results of the repeatability test; i.e. the measured average concentration±1 standard deviation are shown in table #2.

Figure 1a: typical spectrum of soil sample (part 0ne)

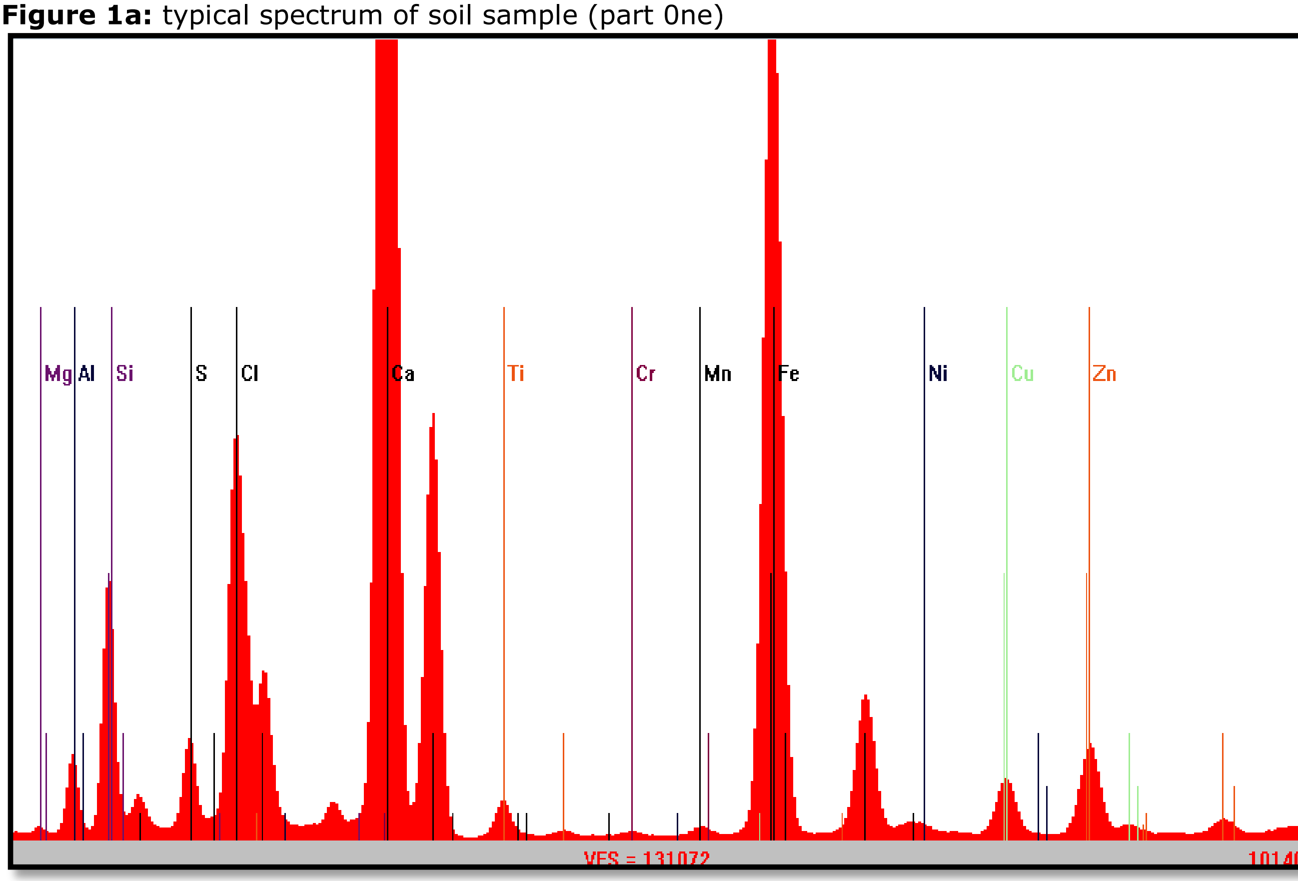

Figure 1b: typical spectrum of soil sample (part two)

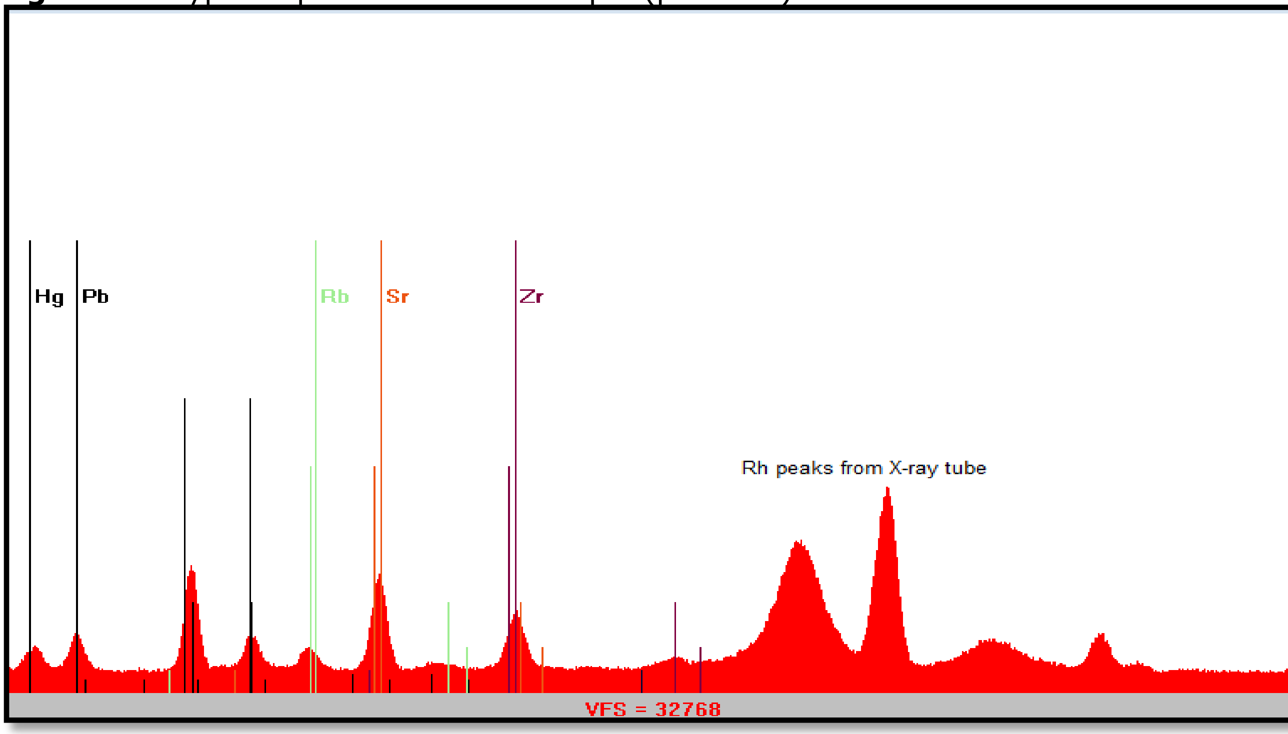

Table 2: Statistic precision test results

Element/Oxide	Sample 1	Sample 2	Sample 3
	Measured mean ± Std [wt.%]	Measured mean ± Std [wt.%]	Measured mean ± Std [wt.%]
MgO	3.481 ± 0.164	3.833 ± 0.441	3.542 ± 0.107
Al_2O_3	10.131 ± 0.066	8.688 ± 0.047	8.587 ± 0.049
SiO_2	15.711 ± 0.048	17.503 ± 0.036	15.464 ± 0.049
SO_3	1.955 ± 0.018	4.033 ± 0.045	1.991 ± 0.066
Cl	3.986 ± 0.018	1.195 ± 0.009	3.523 ± 0.012
CaO	55.509 ± 0.127	58.144 ± 0.324	57.749 ± 0.097
TiO_2	0.839 ± 0.006	0.822 ± 0.014	1.057 ± 0.006
Cr	0.014 ± 0.001	0.012 ± 0.001	0.011 ± 0.001
Mn_2O_3	0.096 ± 0.001	0.094 ± 0.002	0.105 ± 0.003
Fe_2O_3	7.263 ± 0.032	5.067 ± 0.046	7.364 ± 0.021
Ni	0.021 ± 0.002	0.007 ± 0.001	0.006 ± 0.001
Cu	0.24 ± 0.002	0.081 ± 0.002	0.092 ± 0.001
Zn	0.318 ± 0.001	0.242 ± 0.003	0.166 ± 0.001
Br	0.103 ± 0.002	0.0042 ± 0.0004	0.012 ± 0.001
Rb	0.008 ± 0.002	0.008 ± 0.001	0.007 ± 0.001
Sr	0.111 ± 0.002	0.11 ± 0.002	0.162 ± 0.002
Zr	0.047 ± 0.001	0.04 ± 0.001	0.033 ± 0.002
Hg	0.126 ± 0.002	0.051 ± 0.001	0.076 ± 0.001
Pb	0.108 ± 0.002	0.057 ± 0.001	0.067 ± 0.002

Conclusion

This application report shows how simple and rapid an elemental quantitative analysis of soil samples can be performed using Xenemetrix X-Calibur SDD analyzer combined with Fundamental Parameter software. In the absence of calibration standards, Standard less fundamental parameter method is a good solution to determine the concentration of elements in unknown sample. However, since the accuracy of this method is about 10% sometimes the result of SLFP calculations can be improved by including one type standard (materials with known concentration and with the same matrix as the sample to be analyzed).