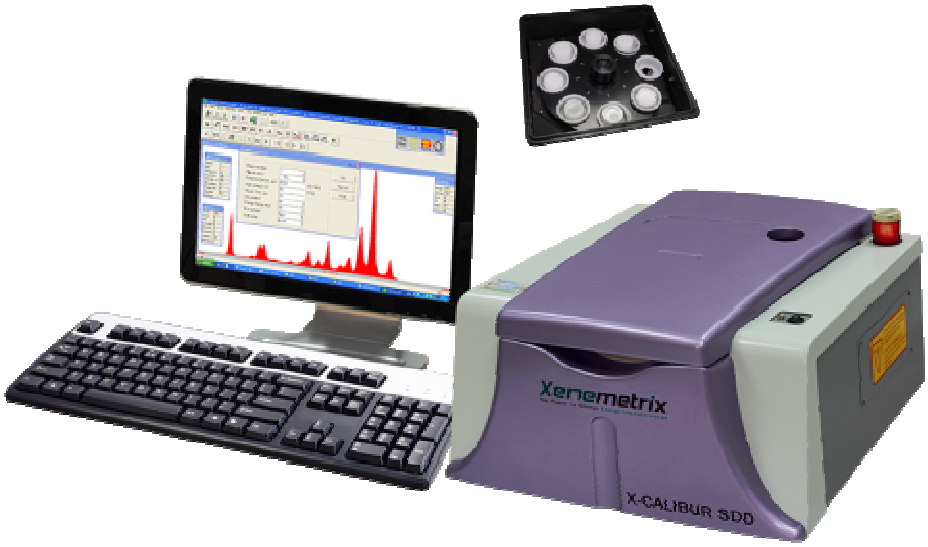
Elemental analysis and quantitative analysis of geological powder

With X-Calibur SDD Analyzer



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ABSTRACT
Qualitative analysis of main and trace elements in geological samples was performed. Standard-less Fundamental parameter program was used to analyze the elemental composition of the geological sample. Static precision was performed with 10 consecutive acquisition of the same sample.

OBJECTIVE

1. Qualitative analysis of main and trace elements in the geological powder sample
2. Standard-less quantitative analysis of elemental composition of main elements
3. Static precision to evaluate instrument performance

BACKGROUND
Energy Dispersive X-ray Fluorescence (EDXRF) is a fast and non-destructive, non invasive, quick, technique that can quantify any type of sample solid, powder and liquid from. EDXRF is an ideal method for a quick and simple elemental analysis for industrial control purposes offering the following advantages: 1.) Fast and minimal sample preparation, 2.) Automated analysis process, 3.) Limited or no exposure to corrosive reagents used by other analytical techniques, 4.) Ease of use for operation by non-technical or non-specialized personnel. These advantages have made XRF as the method of choice among diverse industries including mineral and mine sectors.

ANALYTICAL CONFIGURATIO N

Table 1: Analytical Configuration

Instrument	X-Calibur SDD EDXRF Bench top Spectrometer System.
Excitation	Rh-Anode X-ray Tube, 300W
Detector	High Performance Silicon Drift Detector with Light Element Optimized (LEO)window
Analysis Time	300 second
Type of analysis	Qualitative and SLFP (fundamental parameters) quantitative analysis
Environment	vacuum
Sample preparation	The samples was crushed into powder and analyzed as is in X-ray cups with X ray-film support.

EXPERIMENTALS

One Geological powder standard was provided by the customer. The spectra was acquired with different acquisitions conditions in order to enhance the instrument sensitivity for group of elements at given spectral regions: 10KeV low energy range in vacuum was used to stress the low Z elements and 40KeV energy range for the rest of the elements.

Analysis was performed in X ray cups with special prolene thin film support followed by slight tapping of the cup in order to compact the soil and eliminate the air voids.

The sample acquisition was in vacuum in order to eliminate the oxygen in the X-ray beam path since otherwise the oxygen absorbs the low energy signal emitted by the light elements

In the lack of standards, quantitative analysis of main elements was using Fundamental Parameter software that compares ratio of the elemental peaks in the spectra and uses theoretical considerations to calculate the absorption of X-rays and the inter-elements effects without the need of certified calibration standards, the results are shown in table 2.

In order to show the repeatability of the X-CaliburSDD, the geological calibration standard underwent a static precision study. It was analyzed 10 times without moving it between the acquisitions. The measured mean result together with standard deviation and relative standard deviation for Fe are presented in table 3.

RESULTS and DISCUSSIONS

The main elements in the geological samples are shown in figures 1-3. Here various modes of acquisitions were used to enhance the sensitivity for group of elements.

Figure 1: Geological sample acquired at 45KV in Vacuum

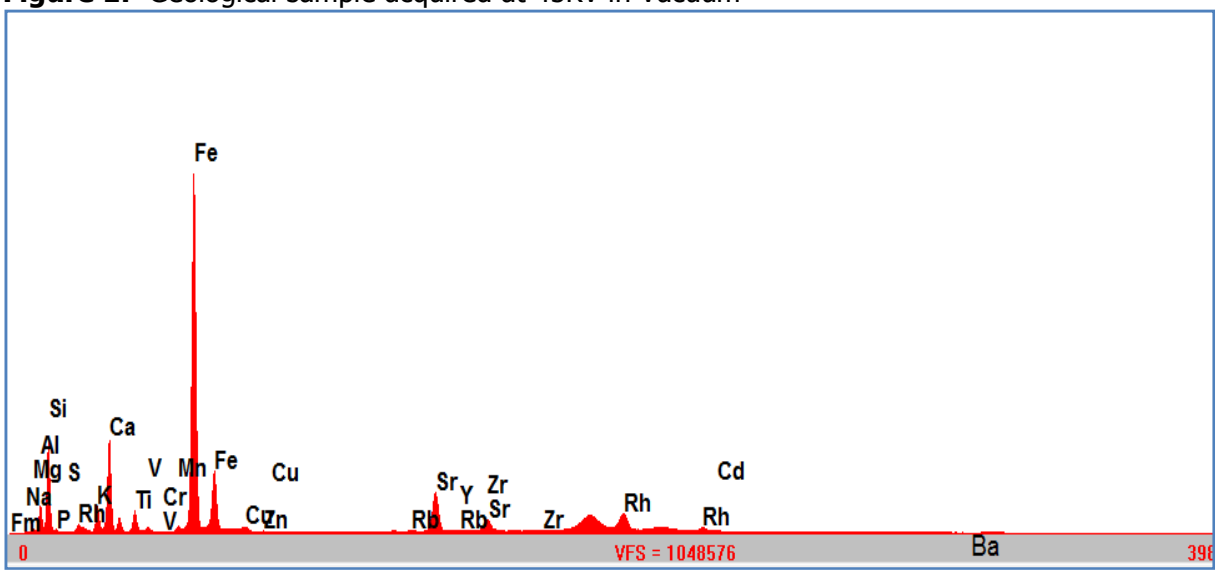
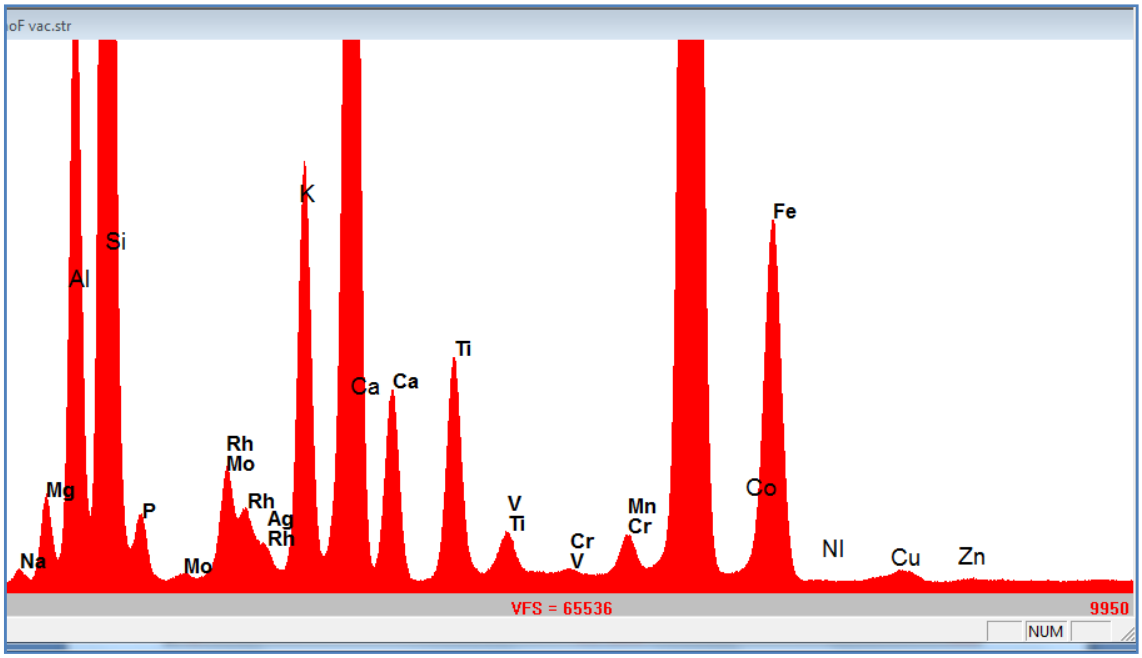
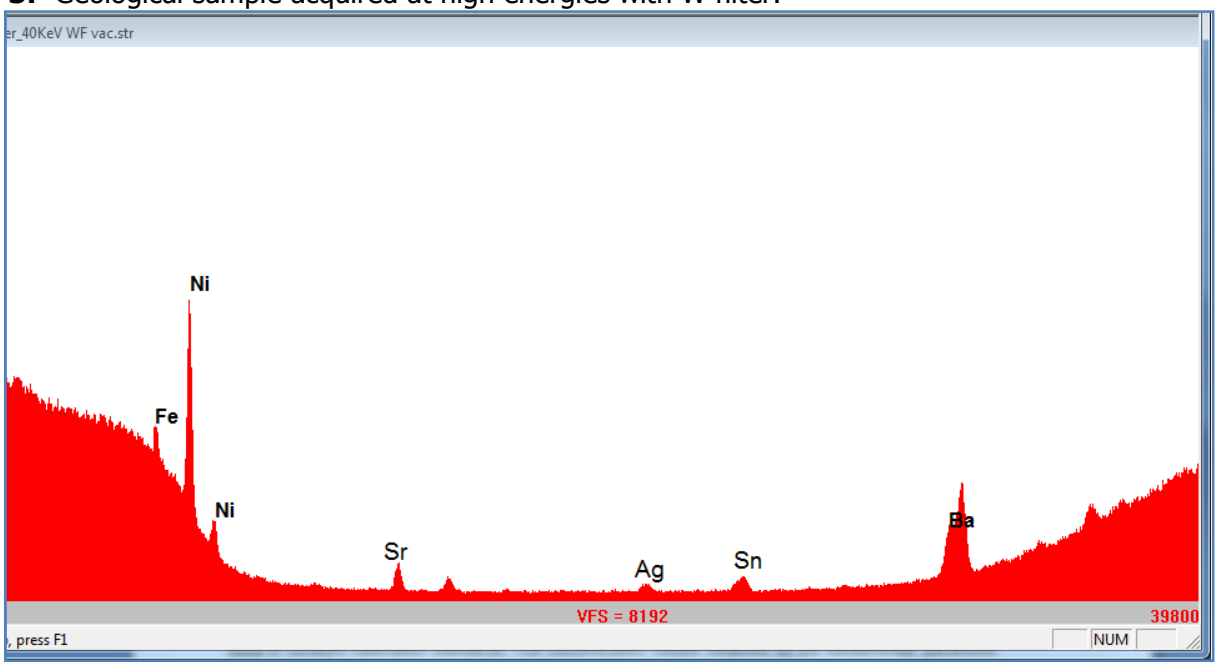


Figure 2: Geological sample acquired at 15KV in vacuum at low energy range and higher resolution



From above spectra it can be seen that most elements are clearly observed. In order to stress other elements, preset at lower concentrations, the X-Calibur analyzer provides filters such as Rd, W, Ti, Fe and Cu often used to improve the signal to noise ration and thus the detection and sensitivity towards group of elements. Example for the use of W filter specific for Sr, Sn, and Ag is shown below (fig 3)

Figure 3: Geological sample acquired at high energies with W filter.



Quantitative analysis

Quantitative analysis in EDXRF is best done with regression analysis based on certified calibration standards (samples of known concentration and of the same matrix as the samples to be analyzed) and building calibration curves per element of interest. When calibration standards are not available the quantitative analysis can be performed using Fundamental Parameter software that compares ratio of the elemental peaks in the spectra and uses theoretical considerations to calculate the absorption of X-rays and the inter-elements effects without the need of certified calibration standards. However the use of at least one standard may improve the results. The results for the geological standard, used as sample, is shown below.

Table 2: Standardless calculation of main elements in the geological sample. The concentrations are in % W/W.

Layer Table ==

#	Thick	Type	Error	Units	Density	Norm.	Total
1	0.00	Bulk	0.00	ug/cm2	0.00	On	105.43

Sample Table ==

Layer	Component	Concn.	Error	Units
1	SiO2	40.284	0.049	wt.%
1	TiO2	0.835	0.003	wt.%
1	Al2O3	23.222	0.053	wt.%
1	Fe2O3	18.230	0.000	wt.%
1	MnO	0.089	0.001	wt.%
1	CaO	5.878	0.008	wt.%
1	MgO	8.539	0.058	wt.%
1	Na2O	5.555	0.134	wt.%
1	K2O	1.957	0.005	wt.%
1	P2O5	0.758	0.006	wt.%
1	Ni	0.001	0.001	wt.%
1	V	0.031	0.001	wt.%
1	Zn	0.009	0.001	wt.%
1	Cu	0.036	0.001	wt.%
1	Cr	0.005	0.001	wt.%

Since, such calculations carry ~10% estimated error, it is advised to use the SLFP calculations with the use of at least one standard. This dramatically improves the results and accounts for the matrix component in the samples; feature known to influence the XRF spectrum. Yet, since only one sample standard was provided, analysis without standards became the only available option.

STATIC PRECISION

Ten repeated acquisitions were performed for the Geological sample without moving the sample between the consecutive acquisitions. The intensity of each element extracted from spectral data. The precision was calculated as the relative standard deviation in %, % standard error (at one sigma) for Fe. The results demonstrate good repeatability of the X-CaliburSDD device.

Element:	Fe (both FeO and Fe_2O_3)
Mean value:	18.23%
Standard deviation:	0.018
% standard error:	0.1

CONCLUSIONS

This report shows that X-Calibur SDD EDXRF analyzer is excellent in performance when it comes to quick, easy and reliable qualitative analysis of powder and samples. In a very short time it is possible to get an overview of different elemental components in different types of samples without any sample preparation. The main peaks of all elements were seen and the sensitivity towards other elements can be specifically enhanced with the use of a set of filter inbuilt into the X-Calibur.
Fundamental Parameter software is based on theoretical considerations and calculations without the need of certified calibration standards. However sometimes, as in this case, it is advised to add one standard to optimize this calculation. This considerably improves the quantitative results.
Static precision of the instrument was made for Fe with one sample calibrations and shown that the system is robust and can provide repeatable results.
Based on this report X-Calibur EDXRF provided the necessary tolls for analysis of Geological samples.