

Reliable Grade and Purity Control of Geological Materials by EDXRF

F. Portala, K. Behrens

Energy dispersive X-ray Fluorescence Spectrometry (EDXRF) instrumentation is the best suited analytical technology for the on-site control of mining operations and the determination of final product grade. The technique requires only a very simple and straightforward sample preparation which makes it a practical, fast and reliable method for multi-element determination in geological materials.

Until recently the sensitivity was poor for light elements such as sodium and magnesium due to thick detector windows which absorbed nearly all radiation from light elements. However, recent advantages of modern Silicon Drift Detector (SDD) technology combined with high power direct excitation and thin stabilized entrance window make the instrument discussed far more suitable for such elemental analysis compared to other instruments.

The article covers the recent advances in modern EDXRF instrumentation and the impact on the elemental analysis of geological materials. Data are shown to demonstrate the analytical performance, precision and long-term stability of such instrumentation for grade and purity control at mining sites and in control laboratories.



Fig. 1 Work at a mining site

1 Introduction

Traditionally, elemental analysis of geological materials is performed by analytical techniques such as Atomic Absorption Spectrometry (AAS) or Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES). A major disadvantage of these techniques is that geological materials have to be digested as these techniques cannot directly measure solid samples. This means prior to analysis, the geological samples have to be digested using substances like sulfuric, nitric or hydrofluoric acid, often in high concentrations which can present obvious hazards. Such additional sample preparation steps are costly, time-consuming and reduce the sample throughput of such systems.

In comparison, X-ray Fluorescence Analysis (XRF) [1, 2] does not suffer from such drawbacks. Compared with ICP-OES and AAS,

sample preparation for XRF is very simple and straightforward. Samples are ground to a fine powder of approximately uniform particle size and then, with the help of a binder either pressed to pressed pellets or with a flux melted to fused beads. These sample preparation steps are fast compared with sample digestions and can be fully automated, even at installations near to the exploration site.

Energy dispersive X-ray fluorescence spectrometers are often used for the elemental analysis of geological materials, especially to control mining operations and to determine final product grade [3]. Benefits of EDXRF instruments include its ease of use and the advanced analytical performance of modern instruments. EDXRF systems are typically very compact, normally benchtop and offer a very low cost of ownership which makes them attractive for dedicated analytical tasks.

2 Instrumentation

The EDXRF instrument S2 RANGER from Bruker was equipped with a Silicon Drift Detector (SDD). The detector used (XFlash® LE) is equipped with an ultrathin, high transmis-

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Tab. 1 Elemental concentration range covered by the calibration

Element	Low Range [%]	High Range [%]
Na ₂ O	0,02	11,0
MgO	0,02	100,0
Al ₂ O ₃	0,04	90,0
SiO ₂	0,40	100,0
P ₂ O ₅	0,01	20,0
SO ₃	0,05	55,0
K ₂ O	0,05	15,0
CaO	0,02	100,0
TiO ₂	0,01	8,00
Mn ₂ O ₃	0,01	0,80
Fe ₂ O ₃	0,01	40,0

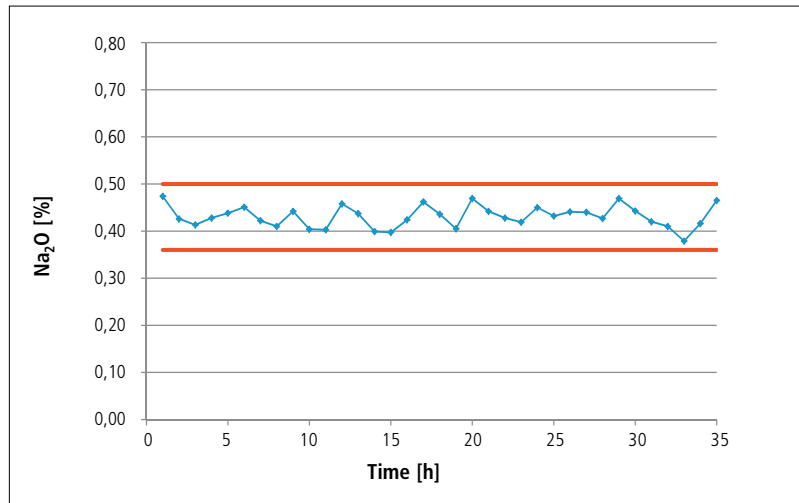


Fig. 2 Process control chart for Na₂O over 35 h

sion entrance window which enhances sensitivity for Na and Mg. In order to obtain optimum results for these elements, an Ag target X-ray tube was used instead of the

standard Pd target X-ray tube which avoids overlaps with tube lines. Two measurement regions with tube voltages of 10 kV and 40 kV were defined, each with a measuring

time of 100 s. The overall processing time per sample, including sample handling, evacuating the sample chamber and actual counting time for the measurement was

Tab. 2 Accuracy and precision test for a clay sample over 35 h. The sample was alternated with another sample

Date	Na ₂ O [%]	MgO [%]	Al ₂ O ₃ [%]	SiO ₂ [%]	P ₂ O ₅ [%]	SO ₃ [%]	K ₂ O [%]	CaO [%]	TiO ₂ [%]	Mn ₂ O ₃ [%]	Fe ₂ O ₃ [%]
20.02.2012 14:48	0,47	0,36	35,68	57,95	0,09	0,32	2,58	0,23	1,27	0,01	0,89
20.02.2012 15:45	0,43	0,38	35,77	58,07	0,09	0,32	2,49	0,24	1,25	0,01	0,89
20.02.2012 16:45	0,41	0,34	35,72	57,93	0,08	0,33	2,52	0,20	1,24	0,00	0,89
20.02.2012 17:45	0,43	0,37	35,64	57,86	0,08	0,33	2,52	0,27	1,25	0,01	0,89
20.02.2012 18:45	0,44	0,36	35,71	58,02	0,08	0,32	2,53	0,23	1,26	0,00	0,90
20.02.2012 19:45	0,45	0,36	35,71	57,91	0,08	0,32	2,52	0,22	1,26	0,00	0,88
...
21.02.2012 02:45	0,44	0,40	35,71	57,98	0,09	0,32	2,47	0,25	1,26	0,00	0,89
21.02.2012 03:45	0,40	0,36	35,53	57,84	0,09	0,31	2,40	0,21	1,26	0,01	0,89
21.02.2012 04:45	0,40	0,37	35,77	58,09	0,08	0,32	2,52	0,22	1,26	0,00	0,90
21.02.2012 05:45	0,42	0,36	35,58	57,87	0,08	0,32	2,42	0,27	1,26	0,01	0,90
...
21.02.2012 12:45	0,42	0,35	35,63	57,88	0,09	0,32	2,53	0,25	1,27	0,01	0,89
21.02.2012 13:45	0,45	0,38	35,69	57,78	0,09	0,33	2,56	0,25	1,24	0,01	0,89
21.02.2012 14:45	0,43	0,38	35,56	57,83	0,08	0,33	2,50	0,19	1,25	0,00	0,89
21.02.2012 15:45	0,44	0,34	35,68	57,97	0,09	0,32	2,52	0,24	1,24	0,00	0,89
...
21.02.2012 21:45	0,41	0,38	35,61	57,87	0,08	0,32	2,62	0,25	1,26	0,01	0,91
21.02.2012 22:45	0,38	0,37	35,76	57,92	0,08	0,33	2,50	0,24	1,28	0,00	0,90
21.02.2012 23:45	0,42	0,39	35,62	57,79	0,08	0,33	2,38	0,24	1,26	0,01	0,89
22.02.2012 00:45	0,47	0,35	35,65	57,86	0,08	0,32	2,47	0,22	1,25	0,01	0,89
Mean value	0,43	0,37	35,66	57,91	0,08	0,32	2,51	0,23	1,25	0,00	0,89
Abs. std. dev.	0,02	0,02	0,07	0,08	0,00	0,00	0,06	0,02	0,01	0,00	0,00
Rel. std.	5,34	4,24	0,20	0,14	4,87	1,14	2,43	9,84	1,19	53,97	0,49
NIST CRM	0,39	0,34	35,83	57,94	0,08	0,28	2,53	0,19	1,22	T	

5 min. In order to measure geological materials from different sites and with different chemical compositions the samples were prepared as fused beads. This sample preparation technique eliminates any interfering mineralogical effects.

In order to obtain best accuracy for different geological samples the instrument has been calibrated with a set of 20 certified reference materials (CRM). The CRMs consist of different geological materials such as clays, feldspars, limestones, dolomites, rocks and silicas and are chosen in order to cover a wide concentration range of the various elements to be determined. The chosen set of standards (GEO-QUANT M) used for this application cover the 11 most important elements for the analysis of major and minor elements as oxides in geological materials. The elements and the concentration ranges covered with this calibration are shown in Tab. 1.

3 Results

Different geological samples prepared as fused beads were measured against the established calibration curves. For precision and long-term stability tests these samples were analyzed over a period of 35 h. No intermediate drift correction was performed during this period. As an example for the instrument stability of the system used, the precision data for the clay sample are shown as process control chart in Fig. 2. The red lines shown in the graph indicate the three times of standard deviation of the measurements. Such threshold values can be easily defined within the instrument software for each element which helps the user to identify "out-of-spec" samples.

Typical accuracy and precision data achieved for selected elements for a clay sample are



Fig. 3 EDXRF spectrometer S2 RANGER from Bruker used for the analysis of the geological samples

shown in Tab. 2. The results prove the repeatability of the system and demonstrate the good agreement with the certified concentrations of the geological reference material.

4 Summary

The data shown proves the performance of the used EDXRF system (Fig. 3) with modern SDD technology. With a well selected set of 20 standard reference materials, the 11 most important major and minor elements in geological materials can be determined. Samples were prepared as fused beads in order to overcome any mineralogical effects due to differing mineral compositions between samples from different mining sites. In comparison to other spectroscopic techniques such as AAS, the sample preparation required for this method is fast and does not require any chemical sample

digestion steps. Due to the ultrathin, high transmission window of the silicon drift detector and the chosen anode material, lower ppm levels of light elements can be accurately determined. The high degree of accuracy and precision demonstrate the suitability of this instrument for reliable grade and purity control of geological materials by EDXRF.

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