



Lab Report XRF 132 S2 PUMA

Fast and Reliable Analysis of Nickel Ore by EDXRF

Introduction

For a long time the main source for the nickel metal production was magmatic sulfide ore. But with the globally increased demand of nickel metal other sources become more needful. One of these nickel sources is laterite which is typically mined as lowgrade deposit in open pit mines. Due to their large dimensions these mines usually contain a significant amount of nickel and therefore they contribute more and more to the increasing nickel demand. In order to optimize the nickel production processes the monitoring of the chemical composition of nickel laterite and its associated materials is more than ever

essential. Besides the major elements such as Ni, Fe, and Si the elements Mg, Al, Ca, Ti, Cr, Mn, Co, Cu, and Zn typically occur in minor and trace amounts.

All these elements can be analyzed using the S2 PUMA energy-dispersive X-ray fluorescence (EDXRF) spectrometer. This instrument offers a fast and accurate control of both the mining process and the subsequent beneficiation processes. This lab report shows the performance of this instrument for the analysis of nickel laterites, prepared as pressed pellets and as fused beads.

Innovation with Integrity

Instrument

The S2 PUMA is a high-performing benchtop EDXRF spectrometer for a wide range of applications. Its HighSenseTM technology with the optimized beam path geometry and its latest XFlash[®] silicon drift detector (SDD) technology ensures high sample through-put in combination with best precision and accuracy.

Due to its ergonomic, easy-to-use TouchControlTM interface the instrument is perfectly equipped for harsh industrial environments which allows for independent routine operation without using an external PC for instrument control. With the dedicated instrument protection system SampleCareTM important system components such as the X-ray tube and the XFlash detector are additionally protected. This guarantees a high instrument uptime and ensures low cost of ownership.

For these measurements an S2 PUMA with standard XFlash detector and a Pd target X-ray tube was used.

Sample preparation: Pressed pellets for gangue material – Fused beads for best performance on Ni-ore

For fast process control the samples were prepared as pressed pellets. Due to the simple and straightforward sample preparation the analytical results are available within minutes after taking the sample. For pressed pellets 10.0 g of sample material has been mixed with 2.5 g of wax binder for XRF (Licowax C Micropowder) and pressed for 15 sec. at 15 tons. For this application some samples have also been prepared as fused beads. This eliminates any mineralogical effects and therefore allows measuring a wider range of samples from different open pit mines. For fused beads 0.80 g of sample material has been fused with 8.00 g of flux (Lithium Tetraborate 50 % + Lithium Metaboarate 50 %).

Measurement parameters

Two measurement regions were defined. The tube current was optimized and fixed in order to gain maximum count rate for the various elements. Table 1 shows the detailed measurement parameters.

Elements	Tube voltage [kV]	Tube current [µA]	Filter	Measurement time [s]
Mg, Al, Si	20	20	none	100
Ca, Ti, Cr, Fe, Mn, Ni,Co, Cu Zn	40	20	500 μm Al	100

Table 1: Measurement parameters for the different elements

The samples have been measured under vacuum which avoids the use of expensive helium as purge gas. This measurement condition minimizes dramatically the costs per sample and results in lowest running costs of the instrumentation.

Calibration

A set of 15 international certified reference materials (CRMs) were used to prepare the calibration for the 12 elements. Table 2 shows the concentration ranges of the different nickel laterite CRMs used to carry out the calibration. Figure 1 shows the excellent spectral resolution of the overlaid spectra for the Ni standards prepared as pressed pellets for standards in the concentration range from 0.003 % to 2.89 % Ni.

	Minimum Concentration	Maximum Concentration	Standard deviation of calibration		
MgO	0.42 %	27.4 %	0.4 %		
AI_2O_3	1.6 %	18.2 %	0.11 %		
SiO ₂	38.1 %	56.7 %	0.46 %		
CaO	0.089 %	0.72 %	0.022 %		
TiO ₂	0.02 %	1.93 %	0.02 %		
Cr ₂ O ₃	0.088 %	1.75 %	0.02 %		
Fe ₂ O ₃	9.77 %	39.4 %	0.16 %		
MnO	0.063 %	0.68 %	0.0062 %		
Ni	0.003 %	2.89 %	0.009 %		
Со	6 ppm	900 ppm	12.4 ppm		
Cu	21 ppm	709 ppm	8.7 ppm		
Zn	30 ppm	327 ppm	10.4 ppm		

Table 2: Concentration ranges used for the nickel laterite calibration



Figure 1: Overlaid spectra at the Ni K α peak between 7.2 and 7.7 keV

Figures 2 to 4 show the calibration curves for the major oxides Fe_2O_3 , CaO, and for Ni, respectively.







Figure 3: Calibration curve for CaO for standards prepared as pressed pellets



Figure 4: Calibration curve for Ni for standards prepared as fused beads

Results

The precision of the S2 PUMA is demonstrated by 10 repetitive measurements of the same nickel ore sample. For each measurement the sample was loaded into and un-loaded from the measurement chamber. Figure 5 graphically shows the repeatability of CaO measurements for a sample prepared as pressed pellet while Figure 6 shows the repeatability of Fe_2O_3 for a fused bead sample. The red lines show three standard deviations of the measurements. For process control such threshold values can be defined for each element within the instrument software SPECTRA.ELEMENTS. These values indicate immediately if the result for a particular sample is out of specification.



Figure 5: Repeatability for CaO for a typical nickel ore sample prepared as pressed pellet



Figure 6: Repeatability for Fe₂O₃ for a typical nickel ore sample prepared as fused bead

	MgO [%]	Al ₂ O ₃ [%]	SiO ₂ [%]	CaO [%]	TiO ₂ [%]	Cr ₂ O ₃ [%]	Fe ₂ O ₃ [%]	MnO [%]	Ni [%]	Co [ppm]	Cu [ppm]	Zn [ppm]
Rep_01	4.85	4.73	48.8	0.59	0.09	1.5	33.89	0.54	1.30	747	46	266
Rep_02	4.81	4.78	48.8	0.61	0.10	1.5	34.06	0.54	1.30	792	39	270
Rep_03	4.82	4.71	48.7	0.59	0.08	1.5	34.02	0.53	1.30	814	42	270
Rep_04	4.84	4.67	48.6	0.58	0.10	1.5	33.96	0.53	1.28	741	47	274
Rep_05	4.77	4.68	48.4	0.58	0.09	1.48	34.01	0.53	1.29	726	47	269
Rep_06	4.74	4.69	48.4	0.57	0.09	1.5	34.02	0.54	1.29	826	47	275
Rep_07	4.73	4.66	48.2	0.61	0.09	1.5	33.98	0.53	1.28	732	43	279
Rep_08	4.74	4.71	48.4	0.59	0.09	1.5	33.93	0.53	1.30	765	40	270
Rep_09	4.79	4.66	48.2	0.59	0.09	1.5	33.97	0.52	1.29	746	49	267
Rep_10	4.64	4.62	48.0	0.59	0.09	1.5	34.12	0.53	1.3	722	46	275
Mean meas- ured value	4.77	4.69	48.44	0.59	0.091	1.50	34.00	0.532	1.293	761	44.6	271.5
Abs. standard deviation	0.060	0.042	0.248	0.012	0.005	0.006	0.062	0.006	0.008	35.24	3.200	3.879
Rel. standard deviation	1.26	0.90	0.51	2.01	5.92	0.40	0.18	1.13	0.60	4.63	7.18	1.43
Certified value	4.82	5.11	46.24	0.564	0.07	1.41	31.72	0.519	1.22	680	57	276

The precision of the system has been proven by 10 repetitive measurements of the standard reference material OREAS 186. Table 4 shows the results obtained for these 10 measurements and compares it to the certified values of the CRM.

Table 4: Precision test for fused beads of ten repetitive measurements of OREAS 186

Conclusion

The data shown prove the high performance of the EDXRF spectrometer S2 PUMA with HighSense technology and XFlash detector. With a set of 15 standards the 12 most important elements in nickel ore samples can be determined. For fast process control samples were prepared as pressed pellets while for the analysis of samples from different open pit mines they were prepared as fused beads. In comparison to other spectroscopic techniques such as atomic absorption spectroscopy (AAS) or inductively coupled plasma optical emission spectrometry (ICP-OES) the sample preparation required for XRF is not time consuming and does not require any chemical sample digestion steps. The achieved high accuracy and precision demonstrate the excellent suitability of the S2 PUMA to determine the elemental composition of nickel ores.



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