Lab Report XRF 149

S6 JAGUAR

- Highly Accurate Grade Control of Nickel Ores with WDXRF

Introduction

The rock type laterite is rich in iron and aluminum oxides. They developed through tropical weathering from parents rocks and forms the red soil in tropical countries. Laterite is commonly known as one source of aluminum ore, but with increasing commodity prices it is today an important source for nickel. This low grade mineral is mined in large open pit operations, therefore the grade control becomes vital for the optimization of the nickel production process. 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.

Sequential wavelength-dispersive X-ray fluorescence (WDXRF) spectrometry is today well established for the analysis of this mineral to access the grade in mining operations and at smelter plants for incoming inspection of raw materials. Especially the nickel grade is vital for the cost optimization when selling or buying nickel laterite and concentrates. This report demonstrates the outstanding analytical performance of the S6 JAGUAR regarding accuracy for the grade control of nickel ores.

Instrument

The S6 JAGUAR WDXRF spectrometer offers high analytical performance in compact size: Equipped with the HighSense goniometer, its closely coupled beam path and 400 W excitation power it offers outstanding sensitivity for every single element in this class of instruments. It covers the entire concentration range from the ppm up to 100% due to the HighSense detector, which is vital for elements such as Nickel in low and high grade ores. For reducing line overlaps the S6 JAGUAR can be equipped with a fourth crystal LiF 220, which enhances the spectral resolution of traces, such as Cr, Mn, Fe, Co and Ni.

Equipped with a 24 position EasyLoad sample magazine and TouchControl the S6 JAGUAR ensures optimal productivity. With its unique SampleCare technology and the added vacuum pump it offers lowest cost-of-operation and optimal instrument uptime. By adding TouchControl the S6 JAGUAR is easy to operate and ensures data integrity by its failsafe operation.

Preparation

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 g of wax binder for XRF (Licowax C Micropowder) and pressed for 15 sec. at 15 tons.

Measurement

Each element is analyzed with an optimal set of instrument parameters: Light elements are excited with low voltage at maximum power, while all heavy elements starting from Ca upwards are best excited with 50 kV. For Mg the XS 55 multilayer, for the elements Al and Si the PET crystal is used and for the element range Ca to Zn the LiF200 is applied. To answer the demand for better trace analysis and better line separation an LiF220 can be used for Mn, Cr, Ni, Cu and Co. This can be vital if traces are becoming important. A scan is shown in figure 1. The measurement parameters are shown in table 1.

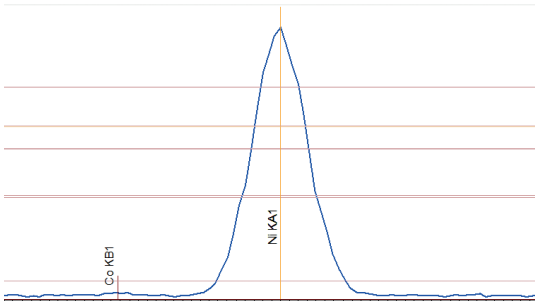

Figure 1: Scan of a Nickel laterite sample, resolving Co Kβ from Ni Kα by an LiF220

Table 1: Set of measurement parameters of the S6 JAGUAR for Nickel laterite

Name	Voltage (kV)	mA	Filter	Crystal	Detector	Peak
Mg KA1	30	13.3	No filter	XS-55	FlowCounter	20.379
Al KA1	30	13.3	No filter	PET	FlowCounter	144.811
Si KA1	30	13.3	No filter	PET	FlowCounter	109.064
Ca KA1	50	8	No filter	LiF (200)	FlowCounter	113.035
Ti KA1	50	8	No filter	LiF (200)	FlowCounter	86.079
Cr KA1	50	8	No filter	LiF (220)	HighSenseXE	107.023
Cr KA1/Alt-220	50	8	No filter	LiF (200)	HighSenseXE	69.324
Fe KA1	50	8	No filter	LiF (200)	HighSenseXE	57.488
Mn KA1	50	8	No filter	LiF (220)	HighSenseXE	95.127
Mn KA1/Alt-220	50	8	No filter	LiF (200)	HighSenseXE	62.944
Co KA1	50	8	No filter	LiF (200)	HighSenseXE	52.766
Co KA1	50	8	No filter	LiF (200)	HighSenseXE	52.766
Ni KA1	50	8	No filter	LiF (200)	HighSenseXE	48.638
Ni KA1/Alt-220	50	8	No filter	LiF (220)	HighSenseXE	71.204
Cu KA1	50	8	No filter	LiF (200)	HighSenseXE	44.997
Zn KA1	50	8	No filter	LiF (200)	HighSenseXE	41.768

Calibration

A set of 15 international certified reference materials (CRMs) were used to prepare the calibration for the 11 elements. Table 2 shows the concentration ranges of the different nickel laterite CRMs used to carry out the calibration. Two calibration curves, one for Ni and one for MnO, are shown in figure 2 and 3.

Table 2: Concentration ranges used for the nickel laterite calibration

	Minimum concentration	Maximum concentration	Squared correlation coefficient
MgO	0.74 %	27.4 %	0.99688
Al_2O_3	1.6 %	17.5 %	0.99249
SiO_2	22.8 %	47.7 %	0.98911
CaO	0.13 %	3.1 %	0.99902
TiO_2	0.02 %	1.36 %	0.99977
Cr_2O_3	0.17 %	1.75 %	0.99094
Fe_2O_3	12.7 %	46.0 %	0.99721
MnO	0.11 %	1.94 %	0.99939
Co	222 ppm	900 pm	0.99256
Ni	0.05 %	2.89 %	0.99912
Zn	73 ppm	327 ppm	0.982

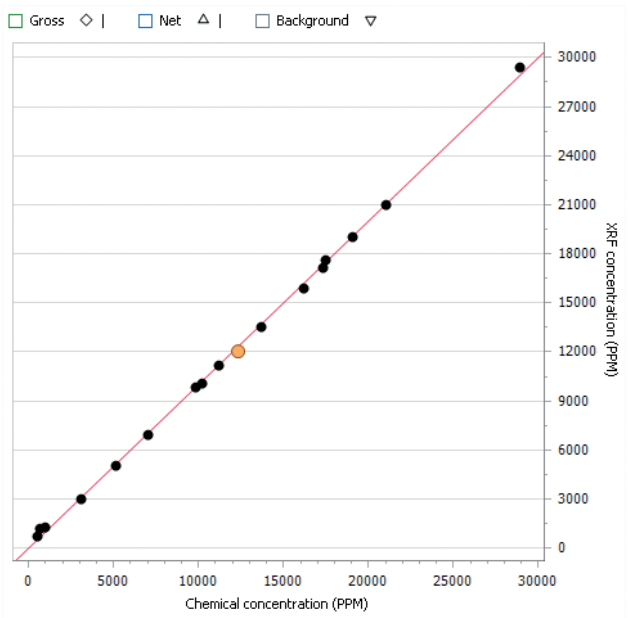

Figure 2: Calibration curve for Ni covering the range from 500 ppm to 2.89 %

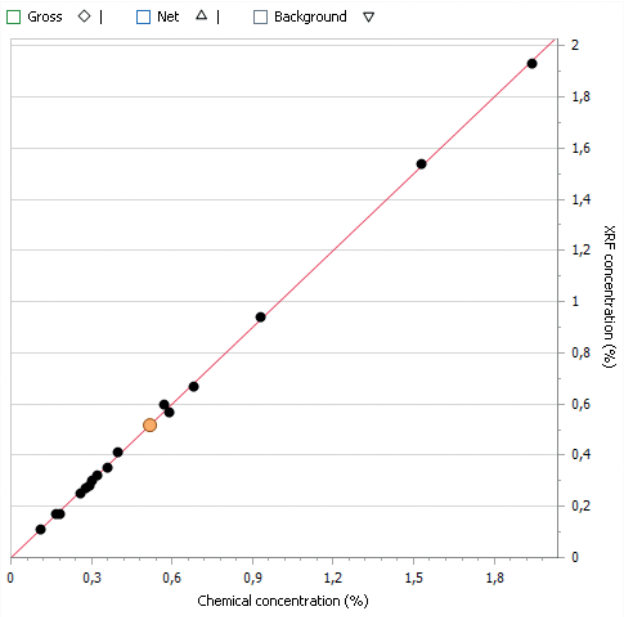

Figure 3: Calibration curve for MnO covering the range from 0,11 % to 1.94 %

Results

For successful quality control analytical precision is vital. The S6 JAGUAR shows its performance with a 20fold repetition test for one sample, shown in table 3. For the most important element Ni and majors the relative standard deviation is less than 0.3%. For trace element the precision is still excellent with a rel. standard deviation of less than 2 %. Even for light elements, such as Mg, the analytical performance of the S6 JAGUAR shows outstanding precision.

Table 3: Concentration ranges used for the nickel laterite calibration

	MgO (%)	Al_2O_3 (%)	SiO_2 (%)	CaO (%)	TiO_2 (%)	Cr_2O_3 (%)	MnO (%)	Fe_2O_3 (%)	Co (PPM)	Ni (%)	Zn (PPM)
1	18.65	2.87	43.36	0.34	0.04	0.94	0.29	18.56	477	2.97	297
2	18.63	2.88	43.21	0.35	0.03	0.93	0.29	18.57	488	2.98	297
	…	…	…	…	…	…	…	…	…	…	…
19	18.74	2.91	43.62	0.35	0.04	0.93	0.29	18.48	478	2.96	301
20	18.78	2.91	43.65	0.35	0.04	0.92	0.28	18.42	476	2.95	289
Mean [%]	**18.69**	**2.90**	**43.45**	**0.35**	**0.04**	**0.93**	**0.29**	**18.53**	**477**	**2.97**	**302**
Std. Dev. [%]	**0.05**	**0.01**	**0.15**	**0.005**	**0.004**	**0.005**	**0.005**	**0.04**	**7**	**0.01**	**5**
Rel Std. Dev.	**0.24**	**0.43**	**0.34**	**1.35**	**10.80**	**0.49**	**1.76**	**0.23**	**1.47**	**0.27**	**1.65**

Conclusion

For grade control in mining operations and smelter plants for nickel laterite the S6 JAGUAR is a perfect fit: With high spectral resolution it analyzes trace elements as good as major and minors. With the HighSense beam path and its 400 W it delivers an optimal analytical precision helping to achieve significant cost benefits. The simple sample preparation technique, TouchControl and SampleCare are making the S6 JAGUAR the ideal instrument for industry labs in mine operations: Easy-to-use, robust and reliable and with a short sample turn-around-time.

Bruker AXS GmbH

info.baxs@bruker.com

Worldwide offices

bruker.com/baxs-offices

Online information

bruker.com/s6jaguar

www.bruker.com