BRUKER

Lab Report XRF 167

S6 JAGUAR

● Quick quality control of iron ore grades with powerful benchtop WDXRF S6 JAGUAR

Iron Ore is an important mineral and finds its way into many of the products we use on a day to day basis. There are four main iron ore minerals: Hematite Fe_2O_3 (Fe at 70.0 %), Magnetite at Fe_3O_4 (Fe 72.4%), Limonite (2 $Fe_2O_3 \cdot 3H_2O$ (Fe at 59.8%) and Siderite $FeCO_3$ (Fe at 48.2%). Additionally, Goethite (FeO(OH) is often found with Hematite.

High quality ores with concentrations of more than 60% Fe are often called direct shipping ores. For ores of lower grade beneficiation may be performed near the mine site.

This includes

- Gravity Separation
- Magnetic Separation
- Flotation Separation
- Screen and screen classifiers

The beneficiation process increases the Fe content but also significantly reduces Si, Al, P and S. These elements will either have deleterious effects on the steel or the operation of the smelter.

Innovation with Integrity

XRF

Why use fused beads for ore samples?

In a comparison of the calibration of SiO_2 for both preparation forms, beads and pressed pellet, there is a difference in the standard deviation. The calibration curves are given below:
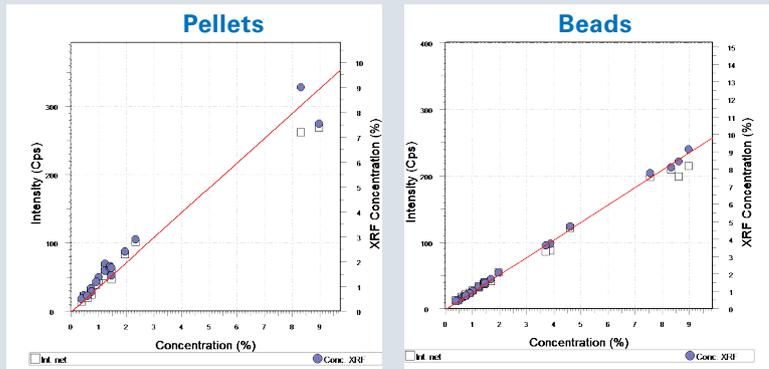
SiO$_2$ is distributed in the sample as shown on the following picture. Although the sample was ground for 2 minutes, the sample still appears inhomogeneous.

If one atom of Si is excited by the x-ray electrons coming from the tube, the fluorescence radiation emitted by Si will be absorbed on the way back to the surface. The same happens for the atoms of Fe. At a given depth, the absorption is large enough that the spectrometer does not "see" elements beyond this depth. This depth is called the saturation depth. Since the fluorescence radiation of both elements is different (1.7keV for Si vs 6.4 for Fe), the saturation depth of Si is lower than for Fe. The picture shows the saturation depth of Si (1 μm) and Fe (60 μm). The sample looks then more "homogeneous" for Fe than for Si.

In case of totally homogeneous samples like fused beads, there are no mineralogical effects. We expect that this preparation method provides the most accurate results. This preparation method is our reference method. However, because of the higher dilution, the limit of detection is then higher for fused beads as for pressed pellets. Wide ranging calibrations may result in larger deviations from the calibration curve. This is due to the different mineralogy's that are present in the samples. Calibrations for final product can also be implemented and results calculated against similar matrix.

A fused bead calibration allows the analysis of materials with different mineralogy from different mines.

In larger mines the iron ore is then loaded into train carts carrying more than 100 tons of ore. Trains may have more than 200 carts. Samples for testing are typically sent with the train to be analyzed in the central laboratory often using procedures based on the XRF method ISO 9516. This method is for up to 22 elements in iron ore. The samples are prepared as fused beads, in bigger ports in fully automated laboratories. The delay in returning results, taking typically more than 36 hours, can result in issues in the beneficiation plant continuing for many hours before the problem is spotted.

Can the compact bench top WDXRF instrument S6 JAGUAR meet the process control requirements? And provides quick and simple pressed pellet preparation sufficient accuracy, possibly in an automated laboratory at the production site?

Instrument

The S6 JAGUAR is a compact benchtop WDXRF spectrometer with 400 W excitation power. It is equipped with the HighSense goniometer with a closely coupled beam path and 3 analyzer crystals and two detectors, the flow counter for light elements and the HighSense Detector for medium and heavy elements offering ultimate linearity for major elements, such as Fe. The S6 JAGUAR offers outstanding sensitivity for every single element compared to other instruments in this class, which lead to its high analytical precision. It covers the entire concentration range from the ppm up to 100% due to the HighSense detector, which is vital for elements such as iron in iron ore.

For industrial process and quality control labs with high sample throughput an online system with conveyor belt and 22 position EasyLoad sample magazine is available. With its unique SampleCare technology and the added vacuum pump it offers in addition lowest cost-of-operation and optimal instrument uptime.

Preparation

For fast process control the samples can be prepared as pressed pellets using automated system. In this report, the samples were prepared manually. Due to the simple and straightforward sample preparation, the analytical results are available within minutes after taking the sample. For pressed pellets 15.0 g of sample material has been mixed with 3 g of wax binder for XRF (Licowax C Micropowder) and pressed for 15 sec. at 15 tons.

Measurement and Calibration

Representative samples from the mine are prepared as fused beads and then evaluated using a calibration built with CRMs or synthetic standards following ISO 9516. As a second step the "certified" results are used to create a calibration based on pressed pellets. The samples for this new calibration are called Secondary Standards. The calibration may be wide ranging greater than 50% Fe_2O_3 or focused at final product eg > 90% Fe_2O_3. With this workflow the influence of the mineralogy on the accuracy is reduced and calibrations are created adapted to the type of ores at the mine site.

As this will be a process control system a restricted range of elements are of interest Mg, Al, Si, P, S, K, Ca, Mn and Fe. Of these elements, the critical elements are Si, Al, P, S and Fe. Ideally a measurement time under 10 minutes is required so that changes to the beneficiation plant could be done in real time.

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. The measurement parameters are listed in table 1.

Table 1: Measurement parameters for iron ore with S6 JAGUAR

Elements	Voltage [kV]	Current [mA]	Analyzer crystal	Filter	Detector
Mg	30	13.3	XS-55	none	FlowCounter
Al, Si	30	13.3	PET(002)	none	FlowCounter
P, S	30	13.3	Ge(111)	none	FlowCounter
K, Ca	50	8	LiF(200)	none	FlowCounter
Mn, Fe	50	8	LiF(200)	none	HighSense XE

An exciting addition to the S6 JAGUAR is the HighSense XE detector. This detector replaces the old scintillation counter. It cannot be saturated due to the countrate capability and ultimate high linearity in the S6 Jaguar. Interelement effects are significantly reduced because its resolution is half compared to conventional scintillation counters. The precision is in a class of its own. The calibration curve for Fe is shown in graph 1, the scans of the pressed pellets with up to 1.7 Mcps are shown in fig. 1.
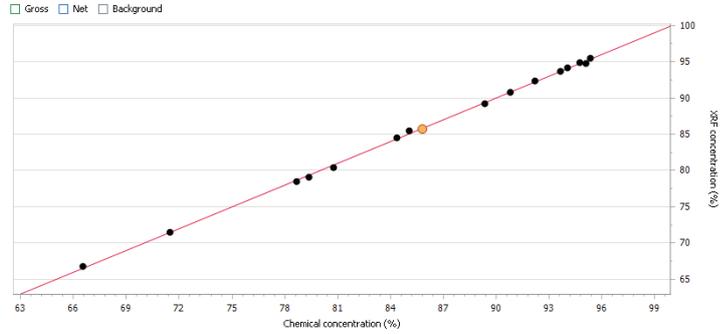
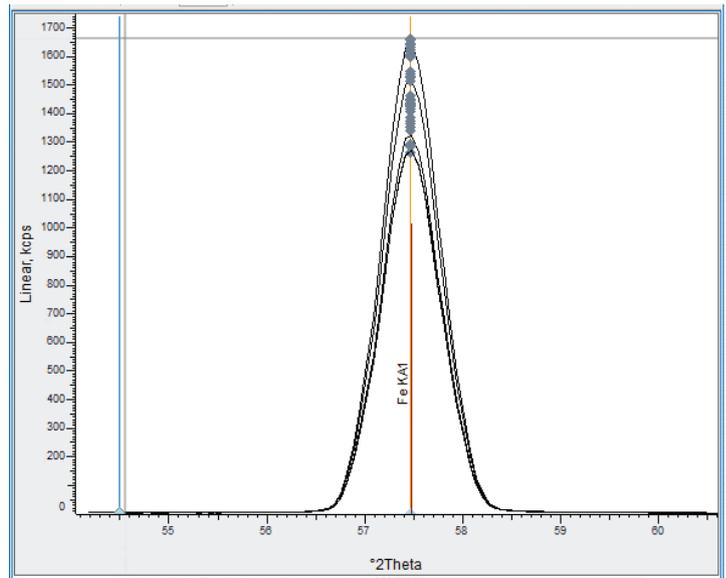Graph 1: Fe_2O_3 in Iron Ore Calibration > 60% Fe_2O_3

Figure 1: Fe Ka1 on LiF200 – Scan on selection of secondary standards (setup mode)

Results

To demonstrate the performance of the S6 JAGUAR for process control tasks in an iron ore beneficiation plant two samples were tested for 21 times, a low- and a high-grade ore. The observed analytical precision for Fe_2O_3 is excellent with an relative standard deviation of less than 1 %, but also the analysis of penalty elements such as Mg, P, and S is excellent and enables a close monitoring of the product quality of the iron ore concentrates. The results are shown in table 2 and 3.

Tab 2: Analytical precision for important elements of the S6 JAGUAR for a low-grade ore.

	Al_2O_3 (%)	SiO_2 (%)	MgO (%)	MnO (%)	Fe_2O_3 (%)	TiO_2 (%)	P (%)	S (%)
Rep-1	15.91	15.72	0.26	0.02	62.46	0.48	0.118	0.01
Rep-2	15.89	15.70	0.28	0.02	62.68	0.48	0.117	0.01
Rep-3	15.88	15.71	0.25	0.02	62.68	0.48	0.118	0.01
…	…	…	…	…	…	…	…	…
Rep-19	15.88	15.70	0.26	0.02	62.76	0.48	0.118	0.02
Rep-20	15.85	15.73	0.27	0.02	62.75	0.48	0.116	0.02
Rep-21	15.88	15.72	0.28	0.02	62.61	0.48	0.118	0.02
Average	**15.89**	**15.71**	**0.27**	**0.02**	**62.57**	**0.48**	**0.12**	**0.02**
Std. Dev.	**0.077**	**0.032**	**0.008**	**0.000**	**0.379**	**0.002**	**0.001**	**0.004**
RSD	**0.48**	**0.20**	**2.95**	**0.00**	**0.61**	**0.46**	**0.55**	**19.31**

Tab 3: Analytical precision for important elements of the S6 JAGUAR for a high-grade ore.

	Al_2O_3 (%)	SiO_2 (%)	MgO (%)	MnO (%)	Fe_2O_3 (%)	TiO_2 (%)	P (%)	S (%)
Rep-1	1.39	1.01	0.03	0.08	96.62	0.05	0.056	0.01
Rep-2	1.38	1.01	0.03	0.08	96.98	0.05	0.056	0.01
Rep-3	1.39	1.01	0.03	0.08	96.96	0.05	0.056	0.01
…	…	…	…	…	…	…	...	…
Rep-19	1.39	1.01	0.03	0.07	96.89	0.05	0.056	0.01
Rep-20	1.39	1.01	0.02	0.08	96.95	0.05	0.056	0.01
Rep-21	1.39	1.01	0.02	0.08	97.04	0.05	0.056	0.01
Average	**1.39**	**1.01**	**0.03**	**0.08**	**96.85**	**0.05**	**0.06**	**0.01**
Std. Dev.	**0.006**	**0.006**	**0.007**	**0.002**	**0.285**	**0.004**	**0.000**	**0.000**
RSD	**0.46**	**0.57**	**24.69**	**2.74**	**0.29**	**7.38**	**0.78**	**0.00**

Conclusion

The S6 JAGUAR is a benchtop WDXRF system, which can compete with many full-size WDXRF systems. The high performance in a compact size stems from a smart combination of cutting-edge technical components and innovative geometric design; including the HighSense XE detector, the closely coupled beam path, and the 400 W X-ray tube. This report highlights the outstanding capabilities of the S6 JAGUAR for iron ore analysis. The S6 JAGUAR enables tight process control via accurate monitoring of the iron ore composition. This allows to enhance the beneficiation process using the S6 JAGUAR as an process control tool at site with ease of use and little installation requirements, but still offering a path into fully automated laboratories. In combination, with fast time-to-result, the S6 JAGUAR adds real values to the beneficiation plant and pays back rapidly!

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