



## Lab Report XRF 123

# S2 PUMA

- Fast and Reliable Analysis of Iron Ore as Pressed Pellets by EDXRF

### Introduction

Iron ore is one of the most important commodities in our society due to its high demand in the steel industry. However, some of the components in iron ore can interfere with the production process or have a negative impact on the material properties of the final product. Therefore monitoring the chemical composition of iron ores is not only important for the control of the mining process but also to optimize the feeding process of melting furnaces during steel production.

A number of elements must be monitored in iron ore samples. Beside the major element Fe, other elements of interest are Si, Ti, Ca, Mg, Mn, Al, and P. Even though some of these elements are only present in low concentrations, they have a significant impact on the steel manufacturing process and the final product quality.

Controlling the mining process often requires a fast feedback on the chemical composition. Therefore the time between collecting a sample and obtaining the analytical result should be as short as pos-

sible. For this reason, the iron ore samples measured for this report have been prepared as pressed pellets. Compared to samples prepared as fused beads, this method simplifies and shortens the sample preparation time considerably, meaning that results are available sooner after the sample has been collected.

### Instrumentation

Energy-dispersive X-ray fluorescence spectrometry (EDXRF) is the perfect analysis technique for this type of mining applications. The EDXRF S2 PUMA used for these measurements is a benchtop system with an easy-to-use TouchControl™ interface. The system is equipped with an XFlash® silicon drift detector (SDD) and a Pd target X-ray tube.

Samples prepared as pressed pellets can be measured under different analytical conditions. Therefore the measurement chamber is either flushed with air or helium gas or is evacuated by the optional vacuum pump. If light elements have to be measured, such as Mg or P in the case of this application, air absorbs much of the low energy radiation and therefore should not be used. Helium overcomes this disadvantage; however the increasing worldwide price of helium and potential local availability issues mean that the measurement cost per sample is much higher, even though only small volumes of helium are used. So the measurements under air or helium have some major disadvantages. Measurements under vacuum provide the best measurement conditions for all elements and mean lowest operating costs per sample. The S2 PUMA can be equipped with a vacuum pump and therefore this instrument provides the best analytical results at lowest costs of ownership.

### Sample preparation

The major and minor elements of interest were determined in this application and allow a fast and simple control of the mining process. The iron ore samples were prepared as pressed pellets by using 10 g sample with a binder in a manual press at a pressure of 150 kN.

### 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. All measurements were performed under vacuum.

Elements	Tube voltage [kV]	Tube current [µA]	Filter	Measurement time [s]
Mg, Al, Si, P, Cl	20	70	None	100
Ca, Ti, Mn, Fe	40	110	500 µm Al	100

Table 1: Measurement parameters for the different elements

### Calibration

A set of 18 in-house standards were used to perform the calibration for Fe, P, Mn, Si, Al, Ti, Ca, Mg, and Cl. The chemical composition of the standards has been verified by an independent analytical technique. Table 2 shows the concentration ranges of the different iron ore standards used to carry out the calibration.

	Minimum concentration [%]	Maximum concentration [%]	Standard deviation of the calibration [%]
Fe	39.97	68.74	0.18
P	0.01	0.18	0.004
Mn	0.02	6.88	0.01
SiO <sub>2</sub>	0.39	42.11	0.3
Al <sub>2</sub> O <sub>3</sub>	0.15	2.46	0.04
TiO <sub>2</sub>	0.01	0.24	0.01
CaO	0.01	0.04	0.003
MgO	0.02	5.13	0.07
Cl	0.02	0.037	0.001

Table 2: Concentration ranges used for the iron ore calibration

Table 2 also shows the standard deviation for the calibration curve obtained for the different elements. The standard deviation (SD) of the calibration curve is a measure for the accuracy of the calibration. The low SD values shown in Table 2 demonstrate the high performance of the system.

Figure 1 and 2 show the calibration curves for Fe and Mn, respectively.

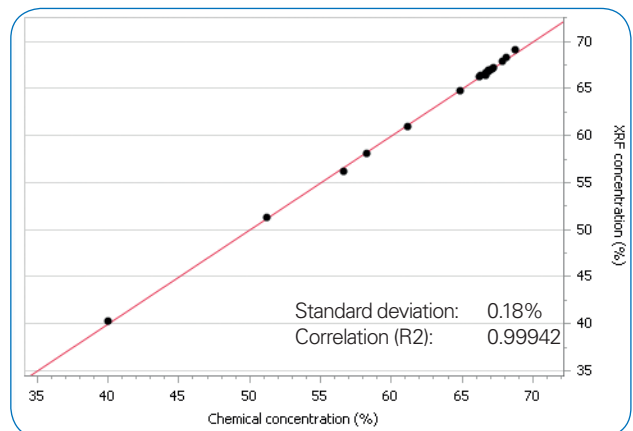


Figure 1: Calibration curve for Fe

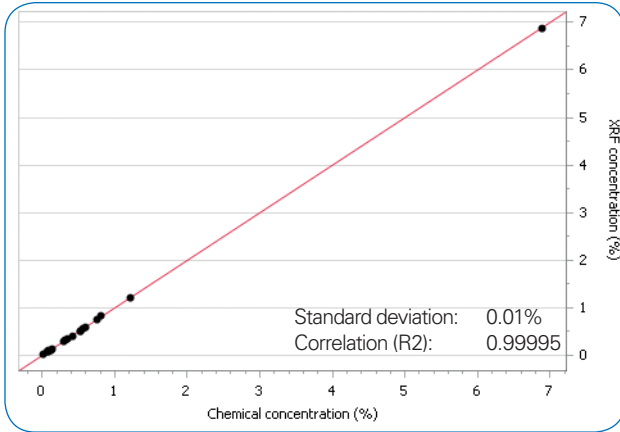


Figure 2: Calibration curve for Mn

In order to get the best results, standards should be chosen to match the mineralogy of the material that has to be analyzed. If no suitable standards exist, secondary standards can be prepared by analyzing mine site samples using a fusion calibration. Pressed pellets of this material can then become the calibration standards for regular analysis at the mine site.

### Results

The precision of the system has been proven by 10 repetitive measurements of the same iron ore sample. For each measurement the sample was loaded into and un-loaded from the measurement chamber. Table 3

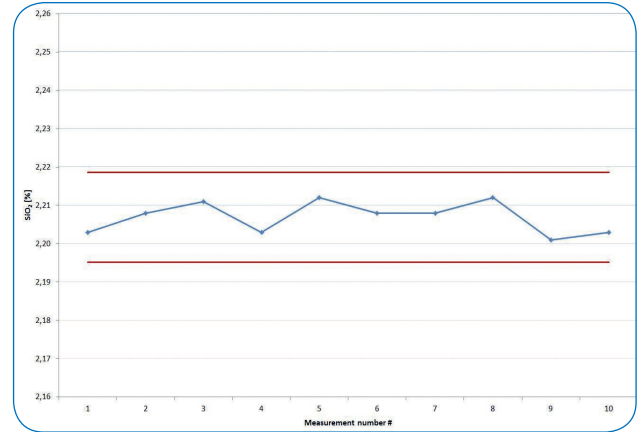


Figure 3: Repeatability for a typical iron ore sample shown for SiO<sub>2</sub>

shows some typical results obtained for such 10 measurements and the achieved precision and accuracy on the iron ore sample.

Figure 3 graphically shows the repeatability of SiO<sub>2</sub> measurements for an iron ore sample. The red lines show three standard deviations of the measurements. Threshold values can be defined for each element within the instrument software, which indicate immediately if the result for a particular sample is out of specification. This frees up work time of the operator and allows him to concentrate on process control instead.

Measurement	Fe [%]	P [%]	Mn [%]	SiO <sub>2</sub> [%]	Al <sub>2</sub> O <sub>3</sub> [%]	TiO <sub>2</sub> [%]	CaO [%]	MgO [%]	Cl [%]
Rep_01	67.60	0.019	0.299	1.56	0.34	0.033	0.036	0.38	0.021
Rep_02	67.64	0.019	0.295	1.56	0.32	0.028	0.033	0.41	0.021
Rep_03	67.59	0.019	0.293	1.53	0.35	0.029	0.029	0.40	0.021
Rep_04	67.60	0.021	0.299	1.52	0.33	0.025	0.029	0.37	0.021
Rep_05	67.56	0.018	0.292	1.56	0.33	0.033	0.031	0.38	0.021
Rep_06	67.61	0.020	0.293	1.52	0.34	0.028	0.030	0.37	0.021
Rep_07	67.65	0.019	0.292	1.52	0.34	0.030	0.030	0.42	0.022
Rep_08	67.75	0.020	0.296	1.54	0.33	0.030	0.033	0.40	0.022
Rep_09	67.51	0.019	0.293	1.54	0.34	0.030	0.035	0.38	0.022
Rep_10	67.56	0.018	0.298	1.52	0.32	0.033	0.028	0.41	0.021
Mean value	67.61	0.019	0.295	1.54	0.33	0.030	0.031	0.39	0.021
Abs. standard deviation	0.065	0.001	0.003	0.02	0.01	0.003	0.003	0.02	0.000
Reference value	67.8	0.02	0.29	1.44	0.31	0.03	0.03	0.34	0.021
Difference to ref. value	0.19	0.00	0.01	0.10	0.02	0.00	0.00	0.05	0.00

Table 3: Precision and accuracy test of ten repetitive measurements of an iron ore sample

## Conclusion

The data shown prove the high performance of the EDXRF spectrometer S2 PUMA with XFlash® detector. With a set of 18 standards, the 9 most important elements in iron ores can be determined. The samples were prepared as pressed pellets which makes the sample preparation simple, fast, and straight forward. Hence, any time-consuming preparation steps can be avoided which results in an immediate analytical feedback to mining process control. The achieved high accuracy and precision demonstrate the ideal suitability of the S2 PUMA to monitor the chemical composition of iron ores.

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