



## S8 TIGER Series 2 and GEO-QUANT Iron Ore Accuracy and Precision for the Iron Ore Mining Industry – Grade Control of Iron Ores According to ISO 9516:2003

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### Introduction

Iron ore is, besides oil, the most important natural resource in our global economy: Almost 95% of all metals are iron and steel mainly used for construction, machinery and automobiles. Most valuable are the direct shipping ores with iron concentrations above 60% (hematite and magnetite), but the commercial value strongly depends also on trace elements, such as P, As or Cu, due to their bad influence on production costs and final steel quality. Australia, Brazil, China and India are the biggest iron ore producers, while China is using nearly half of the globally traded iron ore, followed by Japan and Korea.

The grade control of iron ore requires maximum accuracy and precision due to the big shipped volume of material. Vital for flawless global trade is the standardization of analytical methods: High performance WDXRF is the perfect choice for this analytical task, delivering optimal accuracy for majors, minors and traces with the shortest measurement times. Fused bead preparation has been proved to be the most suitable sample preparation technique for iron ore. It excludes mineralogical effects on data quality and supports, with the higher dilution compared to pressed pellets, the accurate analysis of high iron.

This lab report details the performance of Bruker's sequential WDXRF spectrometer **S8 TIGER Series 2** in combination with the turn-key solution **GEO-QUANT Iron Ore** for the fast grade control of iron ores.

### Instrumentation

The **S8 TIGER Series 2** with **GEO-QUANT Iron Ore** addresses the specific needs for fast grade control of iron ore in geochemical service labs, mine side labs or central labs at steel plants. It starts with the direct loading of samples into the measurement



Fig 1: The new S8 TIGER Series 2 sequential wavelength dispersive X-ray fluorescence (WDXRF) spectrometer.

position, eliminating mechanical errors from turret-based sample handling systems. A small detail with big influence on system precision is the absolute positioning of the sample, maintaining always the same distance between tube anode – sample surface – goniometer, independent of the different sample cups. It continues with the HighSense HV generator in combination with the X-ray tube, which delivers an outstandingly stable excitation. This outstanding precise sample positioning comes in handy with the efficient 4 kW excitation based on the HighSense X-ray tubes. The high precision

goniometer with the additional XS-100 multilayer optics and the high-resolution analyzer crystal LiF220 ensure optimal sensitivity, resolution and precision. Finally, detection is based on the new HighSense detectors and counting electronics delivering a maximum count rate of 4 Mcps for both detectors, flow and scintillation counters. The linear range covers almost 2 Mcps, which enables a wide-range calibration for low and high grade ores. The new **S8 TIGER Series 2** sequential wavelength dispersive X-ray spectrometer is shown above in Figure 1.

**Table 1: Calibration Standards for Iron Ore Analysis According to ISO 9516:2003**

	#1 *	#2 *	#3	#4	#5 *	#6	#7 *	#8 *	#9	#10	#11	#12	#13	#14
Na <sub>2</sub> O	T	T	T	T	T	T	T	0.07	T	T	T	T	T	T
MgO	T	T	T	T	T	T	T	5.00	T	T	T	T	T	T
Al <sub>2</sub> O <sub>3</sub>	T	T	T	T	T	T	T	5.00	T	T	T	T	T	T
SiO <sub>2</sub>	70.02	33.32	89.99	90.02	T	90.02	100.00	2.29	89.99	90.00	90.02	89.99	90.02	89.99
P <sub>2</sub> O <sub>5</sub>	T	T	T	T	T	T	T	2.66	T	T	T	T	T	T
SO <sub>3</sub>	T	T	T	T	T	T	T	9.49	T	T	T	T	T	T
K <sub>2</sub> O	T	T	T	T	T	T	T	1.77	T	T	T	T	T	T
CaO	T	T	T	T	T	T	T	4.31	T	T	T	T	T	10.00
TiO <sub>2</sub>	T	T	T	T	T	T	T	1.51	10.025	T	T	T	T	T
V <sub>2</sub> O <sub>5</sub>	T	T	T	T	T	T	T	0.20	T	10.02	T	T	T	T
Cr <sub>2</sub> O <sub>3</sub>	T	T	T	T	T	T	T	0.20	T	T	T	T	10.02	T
Mn <sub>3</sub> O <sub>4</sub>	T	T	T	T	T	10.34	T	2.05	T	T	T	T	T	T
Fe <sub>2</sub> O <sub>3</sub>	29.97	66.67	T	T	100.00	T	T	64.02	T	T	T	T	T	T
Co <sub>3</sub> O <sub>4</sub>	T	T	T	10.02	T	T	T	0.20	T	T	T	T	T	T
NiO	T	T	T	T	T	T	T	0.20	T	T	T	T	T	T
CuO	T	T	T	T	T	T	T	0.20	T	T	T	T	T	T
ZnO	T	T	T	T	T	T	T	0.20	T	T	9.99	T	T	T
As <sub>2</sub> O <sub>3</sub>	T	T	T	T	T	T	T	0.11	T	T	T	T	T	T
SnO <sub>2</sub>	T	T	T	T	T	T	T	0.20	T	T	T	T	T	T
BaO	T	T	10.01	T	T	T	T	0.20	T	T	T	T	T	T
PbO	T	T	T	T	T	T	T	0.20	T	T	T	9.99	T	T

\* Standards marked with \* were prepared in duplicate.

## Sample Preparation

Calibration standards for this method were made up to the recipes described in ISO 9516:2003 and fused according to the protocol using the flux described as “Flux A”, a mixture of 66% lithium tetraborate and 34% lithium metaborate. Beads were made using 0.8 g dried sample, 8.2 g “Flux A” and 0.5 g  $\text{NaNO}_3$  as an oxidizing agent.

The concentrations of the calibration standards are described in Table 1. For the purposes of this report, the concentrations shown here have all been expressed to two decimal places. However, in the software, the correct number of decimal places, as described in the Norm, were used.

As described in the Norm, the standards marked with \* were prepared in duplicate. This means a total of 19 standards were used for the calibration. When using  $\text{NaNO}_3$  as an oxidizing agent, the accurate analysis of traces of Na is not possible. If Na becomes important, one needs to change to  $\text{LiNO}_3$  or  $\text{NH}_4\text{NO}_3$ . The samples are shown in Figure 2.



Figure 2: Fused bead samples with different iron concentrations

## Measurement and Method Parameters

The **S8 TIGER Series 2** is equipped with Bruker’s brand new HighSense counting electronics. This new generation technology enables the instrument to access a much larger linear dynamic range up to 4 Mcps, meaning that higher count rates can be processed without increasing the dead time for a measurement.

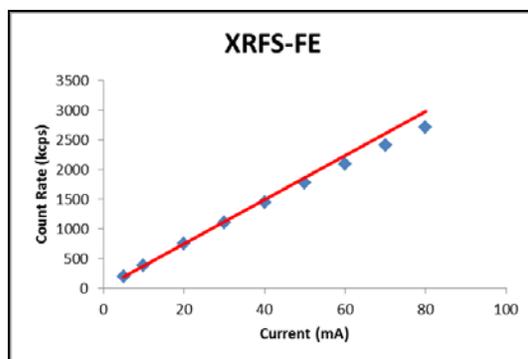


Figure 3: Linearity of the flow counter.

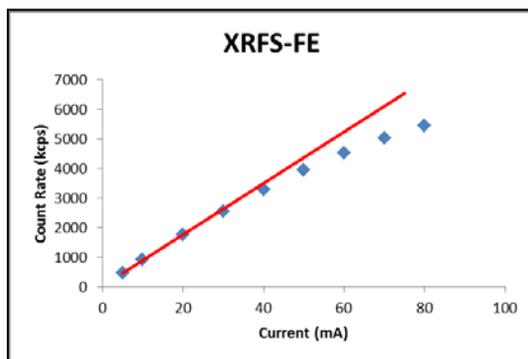


Figure 4: Linearity of the scintillation counter.

To demonstrate the linearity of the system, a test was performed using the Iron Ore monitor from XRF Scientifics (Australia). For this test, the Fe-K $\alpha$  1 line was measured at 50 kV with variable current from 5 mA to 80 mA. The measured intensity was plotted, and is shown above for each detector, shown in Figures 3 and 4.

The red line in the plots shows the expected intensities for perfectly linear behavior up to 2 Mcps. For both detectors, the non-linearity for 5, 10 and 20 mA was calculated to be less than 0.02%.

## Instrument Precision Test

A stability repetition test was performed using the Iron Ore drift monitor from XRF Scientific (Australia). This monitor has been specifically designed to produce compatible intensities for drift correcting methods, but more importantly, it contains all the target elements.

Although the certificate provided for these monitors states that there can be variations in the

final glass from batch to batch, the stability of this sample makes it an ideal one to use for determining the stability of the instrument.

For this test, the sample was measured 10 times, reloading the sample each time so as to simulate a “proper” measurement.

As can be seen from the low standard deviation and RSD, the **S8 TIGER Series 2** with **GEO-QUANT Iron Ore** is capable of delivering extremely stable as well as extremely accurate results.

**Table 2: S8 TIGER Series 2 Instrument Precision Test**

	MIN	MAX	MEAN	ST.DEV	%RSD
<b>Na<sub>2</sub>O (%)</b>	0.096	0.096	0.096	0.000	0.15%
<b>MgO (%)</b>	38.994	39.213	39.092	0.059	0.15%
<b>Al<sub>2</sub>O<sub>3</sub> (%)</b>	44.673	44.820	44.751	0.041	0.09%
<b>SiO<sub>2</sub> (%)</b>	94.534	94.819	94.664	0.090	0.10%
<b>P<sub>2</sub>O<sub>5</sub> (%)</b>	5.217	5.237	5.225	0.005	0.10%
<b>SO<sub>3</sub> (%)</b>	38.003	38.153	38.092	0.052	0.14%
<b>K<sub>2</sub>O (%)</b>	3.634	3.651	3.640	0.006	0.16%
<b>CaO (%)</b>	6.271	6.299	6.289	0.008	0.12%
<b>TiO<sub>2</sub> (%)</b>	14.782	14.856	14.832	0.026	0.17%
<b>V<sub>2</sub>O<sub>5</sub> (%)</b>	5.449	5.483	5.466	0.011	0.20%
<b>Cr<sub>2</sub>O<sub>3</sub> (%)</b>	5.097	5.126	5.112	0.009	0.17%
<b>Mn<sub>3</sub>O<sub>4</sub> (%)</b>	5.205	5.225	5.211	0.007	0.13%
<b>Fe<sub>2</sub>O<sub>3</sub> (%)</b>	92.561	92.844	92.668	0.092	0.10%
<b>CoO (%)</b>	4.283	4.293	4.290	0.003	0.07%
<b>NiO (%)</b>	1.492	1.496	1.494	0.001	0.08%
<b>CuO (%)</b>	1.989	1.995	1.992	0.002	0.10%
<b>ZnO (%)</b>	3.574	3.587	3.578	0.004	0.11%
<b>As<sub>2</sub>O<sub>3</sub> (%)</b>	1.415	1.420	1.417	0.002	0.13%
<b>SnO<sub>2</sub> (%)</b>	2.014	2.035	2.023	0.007	0.32%
<b>BaO (%)</b>	5.105	5.152	5.129	0.018	0.35%
<b>PbO (%)</b>	4.338	4.361	4.351	0.007	0.17%

## Results

In order to verify and validate that the calibration was correct, a real iron ore CRM material was fused to the same recipe and measured using the method. The results from these validation measurements are

shown below in Table 3, with comparisons to the certified values. Only values for which a certified concentration was provided on the certificate are reported.

**Table 3: Accuracy Test Based on ECRM 698-1 Iron Ore from Sweden**

	MIN	MAX	MEAN	CERT	ST.DEV
<b>MgO (%)</b>	1.736	1.759	1.747	1.625	0.007
<b>Al<sub>2</sub>O<sub>3</sub> (%)</b>	2.209	2.230	2.220	2.240	0.006
<b>SiO<sub>2</sub> (%)</b>	11.498	11.572	11.541	11.900	0.026
<b>P<sub>2</sub>O<sub>5</sub> (%)</b>	0.215	0.234	0.223	0.162	0.006
<b>K<sub>2</sub>O (%)</b>	0.626	0.631	0.628	0.557	0.002
<b>CaO (%)</b>	1.523	1.540	1.531	1.655	0.005
<b>TiO<sub>2</sub> (%)</b>	0.457	0.465	0.461	0.544	0.003
<b>V<sub>2</sub>O<sub>5</sub> (%)</b>	0.113	0.118	0.115	0.182	0.002
<b>Fe<sub>2</sub>O<sub>3</sub> (%)</b>	81.462	81.899	81.729	81.616	0.138
<b>CoO (%)</b>	0.133	0.136	0.135	0.014	0.001
<b>NiO (%)</b>	0.074	0.075	0.074	0.025	0.000
<b>CuO (%)</b>	0.063	0.064	0.064	0.009	0.000
<b>ZnO (%)</b>	0.008	0.009	0.008	0.005	0.000

### Summary

The **S8 TIGER Series 2** sequential WDXRF spectrometer delivers extremely accurate and precise analytical results for the analysis of iron ore samples, in accordance with ISO 9516:2003. Its new HighSense counting electronics allow for extremely high intensities to be used, meaning that measurements happen even faster without compromising precision.

The excellent agreement between the observed values for the components in the CRMs analysed demonstrates the excellent analytical capability of the instrument. The extremely low standard

deviation values for the repetition test demonstrate the excellent stability.

The calibration set used in this report can be purchased as a pre-calibrated method **GEO-QUANT Iron Ore**. The set comes with *Ready-to-Fuse* glass powder standards, 2x original CRM QC samples, the Drift Monitor from XRF Scientifics and a memory stick containing the calibration parameters.

**For new systems:** order part number  
A1001A21-INST

**For systems already installed:** order part number  
A1001A21-SET