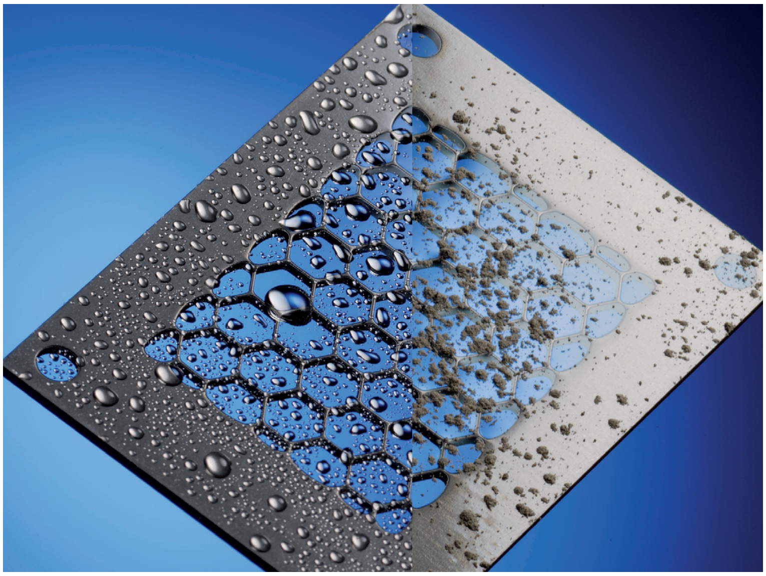
X-RAY FLUORESCENCE

Analysis of Ferrosilicon with a Benchtop WDXRF Spectrometer

Lab Report 185

Introduction

Ferrosilicon (FeSi) is an alloy containing two major elements of Silicon (Si) and Iron (Fe) and can contain minor and trace elements of Aluminum (Al), Phosphorus (P), Sulfur (S), Calcium (Ca), Titanium (Ti), Zirconium (Zr) and Lead (Pb). Ferrosilicon is added during the production of steel to adjust the properties of the melt, and helps with deoxidizing steel and preventing the loss of Carbon (C). Using X-ray Fluorescence (XRF) is an easy and quick way to accurately monitor and verify the Ferrosilicon composition, which can save cost during the production of steel.



Instrumentation

Using the S6 JAGUAR for Ferrosilicon analysis offers the full power of a Wavelength Dispersive XRF (WDXRF) system, that fits on a standard lab bench, as well as the advantages of having better resolution (meaning less spectral overlaps) and more intensity (lower limits of detection and quantification (LLD & LOQ) compared to Energy Dispersive XRF (EDXRF) (Figure 1).

In contrast to other methods like AAS (Atomic Absorption Spectroscopy) and ICP-OES (Inductively Coupled Plasma – Optical Emission Spectroscopy), a simple one-time calibration makes the S6 JAGUAR ready for your application.

This application is then checked daily by running a control sample / daily check to verify that the instrument and application are working correctly. There is no need for an expert operator after the calibration is set up, due to the intuitive software with TouchControl™ and customizable interface for each type of operator, from expert lab manager to less trained lab technician.

Sample Preparation

The calibration standards and unknown samples were weighed out (10 g of samples and 1.0 g of Copolywax® binder) and pulverized using a Tungsten-Carbide (W-C) ring and puck mill for 3 minutes. The samples were then pressed at 30 tons for 30 seconds using a 40 mm die.

The result are stable pellets that can be measured in vacuum mode for best detection, see Figure 2. This preparation can be performed with any commercially available grinder and pellet press.

Table 1
Technical specification for S6 JAGUAR for this application.

Instrument	S6 JAGUAR XY Autochanger Benchtop WDXRF
HighSense™ X-ray power	400 W; 50 kV max.; 17 mA max
Collimator Mask	34 mm
Primary Beam Filter	Automatic 5-position beam filter changer (optional)
Analyzer Crystals	XS-55 for F - Mg; PET for Al - Cl; LiF200 for K - Am
Detectors	Proportional counter for F - Sc; HighSense XE™ for Ti - Am
Measurement Mode	Vacuum
Power Supply	100 - 240 V, 50/60 Hz; max. 1 kVA
Dimensions	80 cm x 67 cm x 74 cm; 147 kg (H x W x L; Weight)

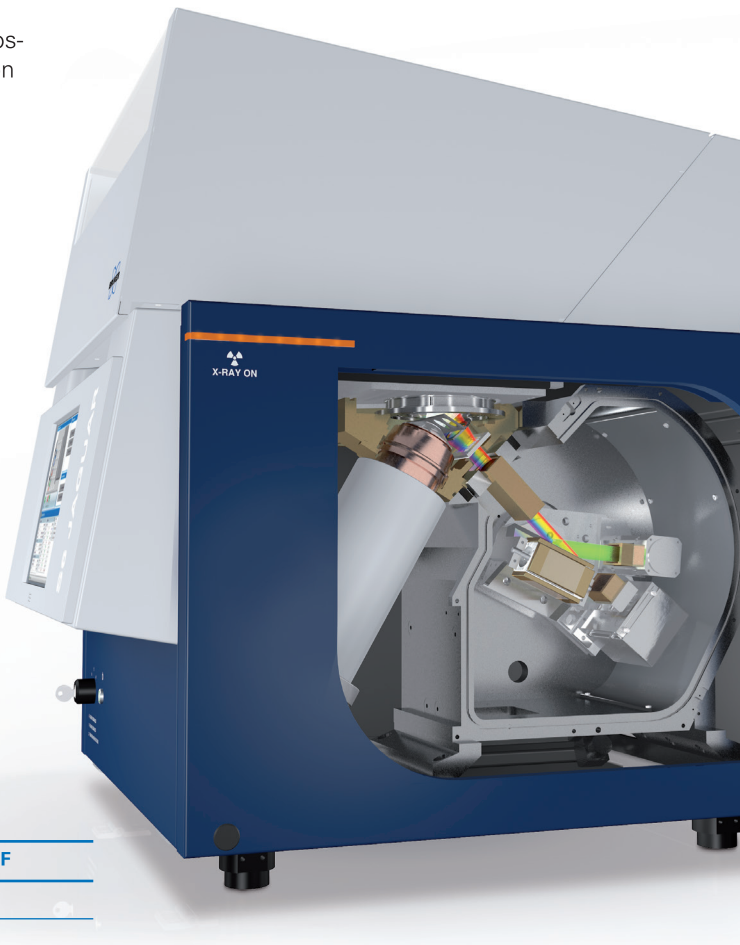


Figure 1
HighSense™ Technology.

Figure 2
Example for finished pressed pellets.

Measurement Parameters

The measurements were performed on the S6 JAGUAR with 400 W Rh excitation. This method is dedicated for the optimal determination of high amounts of Fe and Si as well as minor or trace levels of Al, P, Ca, Ti, Zr and Pb. It defines ideal measurement parameters for tube voltage, tube current, and analyzer crystals. The total measurement time was approximately 8 minutes per sample.

Table 2
Measurement parameters.

Element	Voltage [kV]	Current [mA]	Filter	Crystal	Detector	Collimator	Peak Time [s]	BG Time [s]
Al	30	13.3	None	PET	Flow Counter	0.66°	20	20
Si	30	13.3	None	PET	Flow Counter	0.66°	20	20
P	30	13.3	None	PET	Flow Counter	0.66°	60	60
Ca	50	8	None	LiF200	Flow Counter	0.66°	20	20
Ti	50	8	None	LiF200	Flow Counter	0.66°	20	20
Fe	50	8	None	LiF200	HighSense XE™	0.66°	20	20
Zr	50	8	None	LiF200	HighSense XE™	0.66°	60	60
Pb	50	8	None	LiF200	HighSense XE™	0.66°	60	60

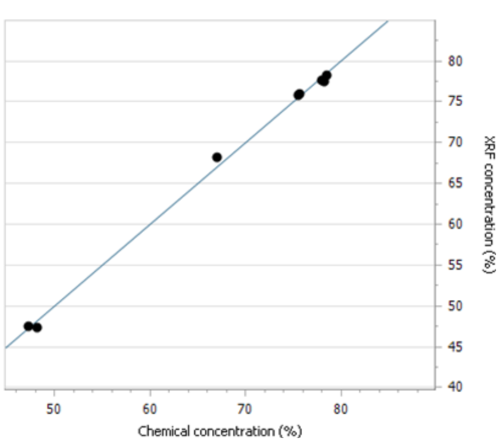
Calibration Details

Ten calibration standards were used - a combination of certified reference materials (CRMs) standards and validated secondary standards were used for building calibration for each element presented in this report (Si, Fe, Al, P, Ca, Ti, Zr, and Pb). The purpose of adding secondary standards is to help extend the calibration ranges to where the certifed reference materials do not cover, and to compensate for the difference in particle size between the CRMs and unknown samples.

The calibration was performed with the SPECTRA.ELEMENTS software, which can be operated from either the touchscreen and / or an external PC connected to the S6 JAGUAR. This integrated XRF spectroscopy package offers a wide range of matrix and interference corrections (theoretical, empirical, fundamental parameters).

Table 3
Calibration range of the FeSi standards.

Element	Calibration Range [wt %]
Si	45 - 90
Fe	10 - 50
Al	0.05 - 1.5
P	0.002 - 0.3
Ca	0.002 - 0.3
Ti	0.02 - 0.15
Zr	0.001 - 0.015
Pb	0.001 - 0.015



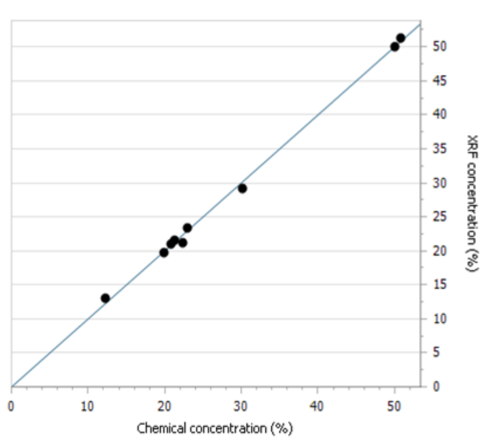


Figure 3 *(left)*
Calibration curve for Si.

Figure 4 *(right)*
Calibration curve for Fe.

Precision and Accuracy Testing

One certifed FeSi calibration standard (NBS 58A) was measured 10 times. The sample was unloaded and reloaded between each measurements. Table 4 shows the impressive accuracy of the results obtained with the S6 JAGUAR in comparison with the certified values. For all measured elements the absolute deviation is < 20 ppm.

Table 4
S6 JAGUAR FeSi data for precision test.

CRM NBS 58A	Al [%]	Si [%]	P [%]	Ca [%]	Ti [%]	Fe [%]	Zr [%]	Pb [%]
Rep. 01	0.950	73.04	0.010	0.266	0.050	25.15	0.002	0.002
Rep. 02	0.953	73.06	0.010	0.263	0.050	26.16	0.002	0.002
Rep. 03	0.948	73.11	0.010	0.265	0.050	25.16	0.002	0.002
Rep. 04	0.952	73.09	0.010	0.266	0.049	25.17	0.002	0.002
Rep. 05	0.953	73.09	0.010	0.266	0.050	25.17	0.002	0.002
Rep. 06	0.948	73.06	0.010	0.267	0.051	25.18	0.002	0.002
Rep. 07	0.951	73.12	0.010	0.266	0.050	25.19	0.002	0.002
Rep. 08	0.954	73.10	0.010	0.262	0.050	25.18	0.002	0.003
Rep. 09	0.954	73.12	0.011	0.263	0.050	25.18	0.002	0.002
Rep. 10	0.955	73.19	0.010	0.265	0.049	25.19	0.002	0.002
Certified	**[0.953]**	**73.13**	**[0.0105]**	**[0.271]**	**[0.051]**	**25.24**	**< 0.005**	**0.001**
Average	**0.952**	**73.10**	**0.0105**	**0.265**	**0.050**	**25.17**	**0.002**	**0.002**
Min.	0.948	73.04	0.010	0.262	0.049	25.15	0.002	0.002
Max.	0.955	73.19	0.011	0.267	0.051	25.19	0.002	0.003
Rel. Std. Dev.	0.27 %	0.05 %	3.30 %	0.65 %	1.20 %	0.05 %	3.92 %	15.97 %

Conclusion

The S6 JAGUAR is the most powerful benchtop WDXRF spectrometer on the market. Equipped with modern software solutions and state-of-the-art hardware, the S6 JAGUAR is ideally configured to enable best-in-its-class analytical performance. High system uptime – even in dusty environments like mining or cement operations – is ensured by the SampleCare™ technology, dedicated high-duty air filters, sturdy design, and overall high quality components.

The application presented here could also be set up with powders. This flexibility paired with the intuitive setup makes the S6 JAGUAR a great asset for any industrial or research lab.

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