



Lab Report XRF 141

S8 TIGER Series 2

- Quick Analysis of Ladle Metallurgical Furnace (LMF) Slags

Introduction

Slag is an important agent in high temperature metallurgical processes and is tuned to an equilibrium condition for maximum action on the metal purity, least attack on the refractory lining, and optimal physical properties. Dosage of slag enhancing additives such as lime, dolomite, or other additives is performed based on the chemical composition of the slag. The driver to better cost efficiency and higher steel quality has been good slag practices in all primary and secondary steel making processes.

Ladle refining in steelmaking leads to better de-oxidation and alloying of the steel, improves the steel cleanliness and homogenizes the temperature and composition during steelmaking. A bad ladle slag contains large amounts of reducible Oxides MnO and FeO, which are causing Al, Si and Mn fuming. Wavelength-dispersive X-ray fluorescence (WDXRF) is mainly used to analyze the ladle slag composition in order to optimize the ladle refining step. Simple, fast sample preparation, ease of

use, quick results and robustness are the key factors to decide on the analytical instrumentation used in daily routine.

This lab report outlines the overall performance of the S8 TIGER Series 2 with 1 kW, including analytical quality, robustness, and ease-of-use, for the analysis of ladle slags.



Fig. 1: S8 TIGER Series 2 with TouchControl

Instrumentation

The S8 TIGER Series 2 spectrometer (WDXRF) with 1 kW is an ideal instrument for quick, robust process control to be placed close to production. Based on an intelligent water cooling system with an integrated water-to-air heat exchanger the S8 TIGER Series 2 with 1 kW can be easily placed in a process lab with little installation requirements. There is no need for an additional heat exchanger or water cooler. With 1 kW excitation, 20 kV excitation voltage for light elements and up to 50 kV for heavy elements it delivers the analytical performance one would need to get precise results for major and minor elements. With the default configuration of 2 collimators (fine and coarse) and three analyzer crystal (XS-55 from C to Mg, PET from Al to Cl, and LiF200 from K to Am) it covers the entire required element range and delivers even for low concentrations of fluorine excellent results.

The recommended configuration of the S8 TIGER Series 2 with 1 kW for slag analysis in steel works comes with SampleCare and TouchControl:

One important feature of SampleCare are the dust shields in front of the tube and the goniometer. When running pressed pellet samples loose particles may fall on the tube or into the goniometer. This is prevented with SampleCare and increases instrument uptime and reduces cost of operation dramatically.

The intuitive operation of the S8 TIGER Series 2 with TouchControl allows even unskilled or untrained personal to quickly and intuitively start samples and get results without accidentally changing calibrations, corrupting files or get lost on complicated software. Analyzing samples in daily routine becomes as easy as using a smartphone.

All four properties are combined in the S8 TIGER Series 2: Analytical performance (as shown in this lab report), robustness based on german Bruker made technology, ease-of-use, and little installation requirements are the main advantages of the S8 TIGER Series 2 with 1 kW for process control applications.

Sample Preparation

The slag samples were prepared as pressed powder pellets with the aid of a binding agent. After the crushing step remaining iron can be easily separated with a magnet before grinding. The slags are typically prepared with 15g of sample to 1g of grinding aid tablet, and were ground in a tungsten carbide grinding vessel (~3 min). The milled powder was then pressed in aluminum cups (40mm in diameter) using a minimum of 15 tons for 30 s. Such preparation can be performed with any commercially available grinder and pellet press.

Measurement Parameters

Measurements were performed on the S8 TIGER Series 2 with 1kW Rh excitation. This method is dedicated for the optimum determination of major and minor elements in slag samples. It provides optimized measurement parameters for tube voltage, crystals, and collimators. Total measurement time was about 5 minutes per sample.

Calibration

A set of 8 certified reference materials (CRMs) standards and 8 validated secondary standards were used to calibrate the instrument for F, MgO, Al_2O_3 , SiO_2 , P_2O_5 , S, CaO, TiO_2 , MnO, and FeO (See Table 2). The standards were prepared as pressed pellets using the same recipe as the unknown samples. Since they were prepared as pressed pellets a calibration must be made for each application (EAF, LMF, & BOF). The purpose of adding secondary standards is to help extend the cali-

Line Name	Tube [KV]	Tube [mA]	Filter	Collimator	Crystal	Detector
F K α 1	30	33	No Filter	0.46	XS-55	Flow Counter
Mg K α 1	30	33	Be 50um	0.46	XS-55	Flow Counter
Al K α 1	30	33	Be 50um	0.46	PET	Flow Counter
Si K α 1	30	33	Be 50um	0.46	PET	Flow Counter
P K α 1	30	33	Be 50um	0.46	PET	Flow Counter
S K α 1	30	33	Be 50um	0.46	PET	Flow Counter
Ca K α 1	50	20	Al 100um	0.23	LiF200	Flow Counter
Ti K α 1	50	20	Be 50um	0.46	LiF200	Flow Counter
Mn K α 1	50	20	Be 50um	0.23	LiF200	Scintillation Counter
Fe K α 1	50	20	Al 100um	0.23	LiF200	Scintillation Counter

Table 1: Measurement conditions of the S8 TIGER Series 2

bration ranges in which the certified reference materials do not cover and to compensate for the different particle size between the CRMs and unknown samples, especially for the light elements of F, MgO and Al₂O₃.

The calibration was performed with SPECTRA^{plus} software operated from an external PC connected to the S8 TIGER Series 2. This integrated XRF spectroscopy package offers a wide range of matrix and interference corrections.

Measurement Accuracy & Precision

Measurement of one unknown LMF slag pellet was repeated 25 times against the calibration. The sample was unloaded and reloaded between measurements. Table 3 demonstrates the impressive accuracy of the results obtained with the S8 TIGER Series 2. The precision is well within the expected range for the analysis of slag material shown in table 4.

Element	LMF Concentration Range with Certified & Secondary Calibration Standards [Wt. %]
F	1 - 10
MgO	0.7 - 20
Al ₂ O ₃	6 - 40
SiO ₂	20 - 45
P ₂ O ₅	0 - 0.2
S	0 - 1.2
CaO	20 - 65
TiO ₂	0.1 - 0.9
MnO	0.3 - 10
FeO	0.2 - 10

Table 2: Calibrated Concentration Ranges

	F [%]	MgO [%]	Al ₂ O ₃ [%]	SiO ₂ [%]	P ₂ O ₅ [%]	S [%]	CaO [%]	TiO ₂ [%]	MnO [%]	FeO [%]	V-Ratio	B3-Ratio
XRF Conc	6.53	6.03	2.14	26.27	0.017	0.66	57.59	0.35	0.095	0.31	2.19	2.00
Expected Conc	6.12	6.10	1.98	26.39	0.015	0.68	57.79	0.31	0.103	0.35	2.19	2.01

Table 3: Accuracy of the Calibration

	F [%]	MgO [%]	Al ₂ O ₃ [%]	SiO ₂ [%]	P ₂ O ₅ [%]	S [%]	CaO [%]	TiO ₂ [%]	MnO [%]	FeO [%]	V-Ratio	B3-Ratio
Rep-01	6.62	6.03	2.14	26.28	0.018	0.659	5750	0.353	0.096	0.303	2.19	2.00
Rep-02	6.60	6.03	2.13	26.29	0.018	0.661	5753	0.353	0.094	0.305	2.19	2.00
Rep-03	6.57	6.02	2.14	26.18	0.019	0.658	5766	0.354	0.095	0.305	2.20	2.01
Rep-04	6.55	6.03	2.14	26.23	0.017	0.659	5762	0.353	0.094	0.306	2.20	2.01
Rep-05	6.57	6.02	2.15	26.25	0.017	0.659	5758	0.353	0.095	0.304	2.19	2.00
Rep-06-20
Rep-21	6.48	6.05	2.13	26.30	0.016	0.661	5760	0.353	0.095	0.306	2.19	2.00
Rep-22	6.49	6.05	2.14	26.25	0.016	0.660	5764	0.353	0.095	0.309	2.20	2.00
Rep-23	6.48	6.04	2.14	26.28	0.016	0.662	5762	0.354	0.094	0.307	2.19	2.00
Rep-24	6.47	6.04	2.14	26.29	0.016	0.660	5762	0.355	0.094	0.305	2.19	2.00
Rep-25	6.43	6.03	2.14	26.32	0.017	0.661	5765	0.355	0.095	0.305	2.19	2.00
Average	6.53	6.03	2.14	26.27	0.017	0.660	5759	0.354	0.095	0.305	2.19	2.00
Abs.Std.Dev	0.044	0.008	0.005	0.027	0.001	0.001	0.036	0.001	0.001	0.001
Rel.Std.Dev	0.67	0.13	0.23	0.10	5.40	0.15	0.06	0.19	0.61	0.45

Table 4: Precision of the measurements of main components in a LMF slag sample

LMF Viscosity (V) Ratio = CaO / SiO₂

LMF Basicity (B3) Ratio = CaO / (SiO₂ + Al₂O₃ + TiO₂)

Conclusions

The S8 TIGER Series 2 with 1 kW is the perfect analytical tool to be used in routine in steel plants for the analysis of slag samples and other process relevant materials. Its ease-of-use by TouchControl™ makes running routine samples very simple for the shift operator with minimal operator training.

The robustness, flexibility, and stability of the S8 TIGER Series 2 ensures achieving the analytical accuracy and precision required at the steel plant and enabling dramatic cost savings and improvements in efficiency and product quality.

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