Application Note: 41707

# Analysis of Directly Cast Metallurgical Slags

*ARL OPTIM'X WD-XRF sequential configuration*


Key Words

- ARL OPTIM'X
- Iron & Steel
- Slags
- WDXRF
- X-Ray Fluorescence


## Introduction

Slags originate from various stages in the iron & steel process, e.g. blast furnace, converter, basic oxygen furnace (BOF also referred to as BOS = basic oxygen steelmaking), electric arc furnace or ladle.

In the blast furnace, slag is formed from impurities in the iron ores (known as the gangue), the flux and coke ashes. It is a complex silicate of aluminum, calcium and magnesium containing small quantities of oxides of manganese and iron as well as calcium sulfide. Slag has a double role: it permits removal of the gangue thanks to its fusibility and fluidity, it also allows exchange reaction with the liquid metal and permits a control of the process in order for the desirable elements to stay in the melt while the others are removed. As an example, in an electric arc furnace the slag formation process can be controlled by adding oxygen, carbon and slag formers to the melt. This will promote formation of CO instead of MnO and FeO and help keep these elements in their metallic form in the melt. The basic slag formers like lime (CaO) and magnesia (MgO) will help neutralize the acidity of the slag in order to save the refractory bricks of the furnace.

## Instrumentation

The Thermo Scientific ARL OPTIM'X is a WD-XRF instrument designed for ease of use, minimal operating and maintenance costs. An ARL OPTIM'X XRF spectrometer configured as a sequential unit was used to obtain the results presented in this application note. The instrument was fitted with a SmartGonio™ covering elements from F (Z=9) to U (Z=92). The ARL OPTIM'X XRF spectrometers are fitted with a low power Rh anode X-ray tube and the geometry of the instrument is optimized to provide the highest sensitivity. The instrument does not require external or internal water cooling, and has superior resolution, precision, short and long term stability. It can analyse Na and Mg without problem or even F if necessary. Ease of operation is obtained through the state-of-the-art WinXRF software running under Windows® XP Professional environment.

## Sample preparation

While still hot the slag sample is directly cast into a steel ring using a ceramic mold specially designed for this purpose. This direct cast sample can be analyzed in the XRF instrument without further preparation (Figure 1).

Figure 1: Directly cast slags

## Calibration and results

Ten secondary slag standards were used for calibration of the ARL OPTIM'X. These standard samples allow the concentration ranges shown in Table 1 to be covered. A working curve is established for each element using the Multi-Variable-Regression incorporated in the WinXRF software package. The Standard Error of Estimate (SEE) is a measure of the accuracy of analysis. It is the average error between the certified concentration of the standard samples and the calibration curve of a given oxide.

| ELEMENTS/ OXIDES | ANALYTICAL DEVICE | CALIBRATION RANGES | SEE (%) |
|---|---|---|---|
| $Al_2O_3$ | SmartGonio™ | 5 % - 12 % | 0.074 % |
| MgO | SmartGonio™ | 2.4 % - 9 % | 0.14 % |
| $SiO_2$ | SmartGonio™ | 34 % - 38 % | 0.23 % |
| CaO | SmartGonio™ | 32 % - 47 % | 0.24 % |
| $Cr_2O_3$ | SmartGonio™ | 1.6 % - 7.8 % | 0.16 % |

Table 1: Summary of performance

SEE = Standard error of estimate : it is a measure of the accuracy

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## Stability tests

In order to show the excellent repeatability of the ARL OPTIM'X for the analysis of slags both in short and long term, stability tests were performed. For short term repeatability 10 consecutive measurements were performed on one of the cast slag samples. The repeatability test was done twice using counting times of 20s and 40s per element. Average concentration and standard deviations are shown in Table 2.

| ELEMENT/ OXIDE | ANALYTICAL DEVICE | AVERAGE CONCENTRATION % | COUNTING TIME | STD.DEV. % | COUNTING TIME | STD.DEV. % |
|---|---|---|---|---|---|---|
| $Al_2O_3$ | SmartGonio™ | 9.74 | 20 s | 0.034 | 40 s | 0.027 |
| CaO | SmartGonio™ | 44.1 | 20 s | 0.034 | 40 s | 0.033 |
| $Cr_2O_3$ | SmartGonio™ | 4.59 | 20 s | 0.018 | 40 s | 0.017 |
| MgO | SmartGonio™ | 3.17 | 20 s | 0.018 | 40 s | 0.013 |
| $SiO_2$ | SmartGonio™ | 36.2 | 20 s | 0.041 | 40 s | 0.026 |
| Total counting time | | | 100 s | | 200 s | |

Table 2: Results of a repeatability tests (10 runs) using counting times of 20s and 40s per element

For long term repeatability test, one of the standard specimen was analyzed every hour over 15 hours. The total counting time per run was set at 100s. Average concentration and standard deviations are shown in Table 3.

Table 3: Results of an overnight reproducibility test over 15 hours

## Conclusion

Various elements in directly cast slags can be succesfully analyzed using the ARL OPTIM'X low power WD-XRF instrument. The results obtained on cast slags are very similar to those obtained on slags prepared through grinding and pressing (see application notes nr. 41703 and 41704)

The limitation in term of accuracy is not due to the instrument but to the sample preparation and the variety of slags used in this test. In order to get the best accuracy with slags of different origins it is necessary to prepare the samples as fused beads with Lithium tetraborate (see application note nr. 41702) in order to avoid grain size effects and mineralogical effects.

Good repeatability and reproducibility is obtained with 100s of total counting time with the SmartGonio™ If better results are required for any element the counting time for that particular element can be increased. Additionally two fixed channels can be fitted alongside the SmartGonio™ of the ARL OPTIM'X in order to improve the performance for two specific elements when required.

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