Application Note WDXRF1120

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XRF1120 - Quantitative analysis of low alloy steel using the ZSX Primus III NEXT

Introduction

Alloy steels with up to 4 to 8% of alloying elements added are called low alloy steels. Low alloy steels are made by adding various elements intended to improve a specific characteristic of steel such as hardenability. Alloy steels are generally made in electric furnaces. The concentrations of elements in molten steel are adjusted during the process of steel making, so that rapid analysis of the elemental composition is required. As part of the control of the steel making process, analyses of slag and raw materials such as quicklime and ferroalloys are also required. X-ray fluorescence spectrometers are the most common analysis tools to analyze steel owing to rapid analysis and the ability to measure both bulk metal and powders. This application note describes low alloy steel analysis using the ZSX Primus III NEXT, which is optimized for process control.

Instrument

The ZSX Primus III NEXT has tube above optics, where the X-ray tube is placed above the sample. Tube above optics reduce the risk of instrument contamination or damage that can occur when pressed pellet samples break inside the spectrometer while being measured or transported to the measurement position. The ZSX Primus III NEXT is ideal for the steel industry where both bulk metal and powder samples are analyzed as part of the process control protocol during the manufacture of alloy steels. The system software is based on Rigaku's Flowbar interface that leads the user through a series of step by step procedures to optimize and execute measurements. "EZ analysis", another Rigaku software innovation greatly simplifies the analysis setup and sample measurement. In addition, the software has various kinds of statistical process control functions ideal for the steel industry.

Standard and sample preparation

The certified standard reference materials of low alloy steel provide by NIST and JSS (Japanese Iron and Steel Certified Reference Materials) were used to establish the calibration. The samples were polished with 80-grit corundum abrasive paper. 80-grit SiC abrasive paper was used for the analysis of aluminum to avoid contamination from the corundum paper.

Measurement and calibration method

Measurements were performed using the ZSX Primus III NEXT with a 3 kW Rh target X-ray tube. The Kα line was measured for all the elements at a counting time of 20 seconds. An LiF(200) analyzing crystal and the scintillation counter were used for all the heavy elements from V to Mo. A PET crystal was used for Si and Al, and an optional Ge was used to provide high resolution for the measurement of P and S.

A gas flow proportional counter was used for all the light elements.

The representative calibration curves obtained are shown in Figure 1 through Figure 9. Overlap correction is performed in the calibration of P and S to correct for overlapping of Mo as shown in Figures 3 and 4.

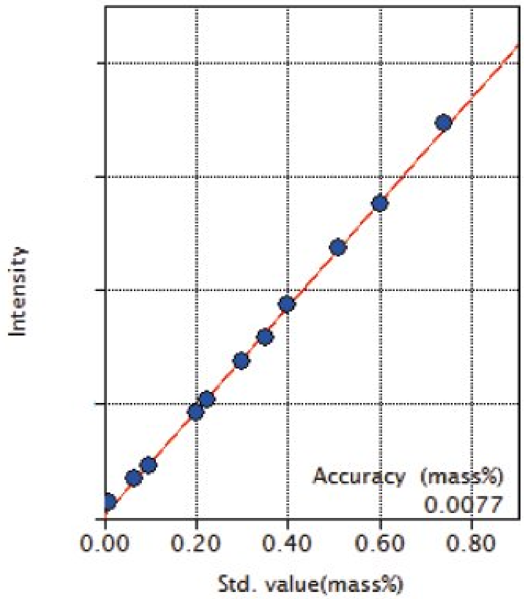

Figure 1: Calibration curve of Si

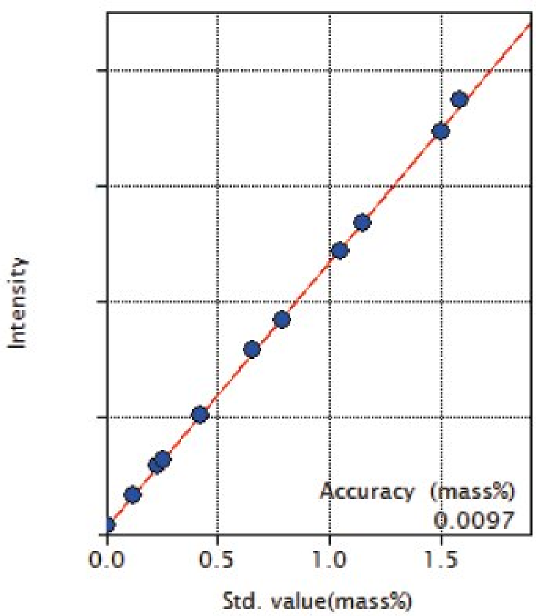

Figure 2: Calibration curve of Mn

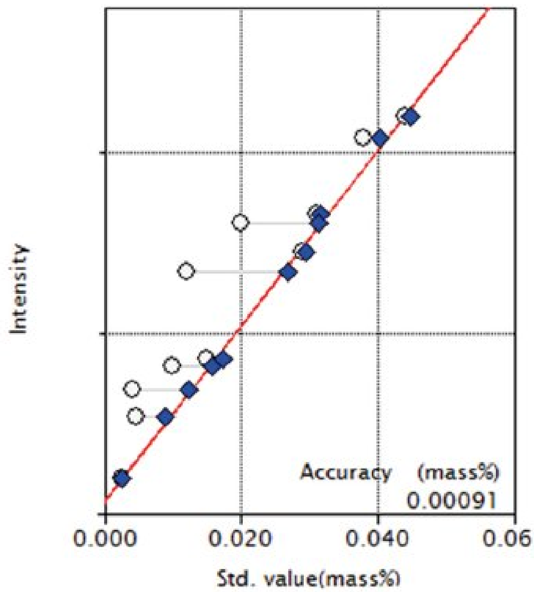

Figure 3: Calibration curve of P

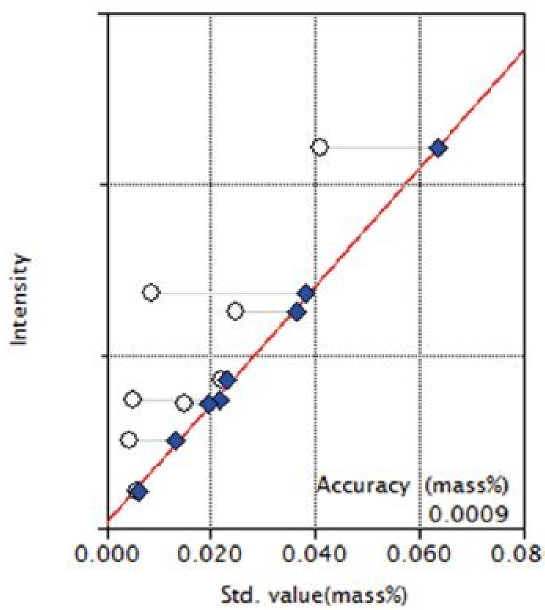

Figure 4: Calibration curve of S

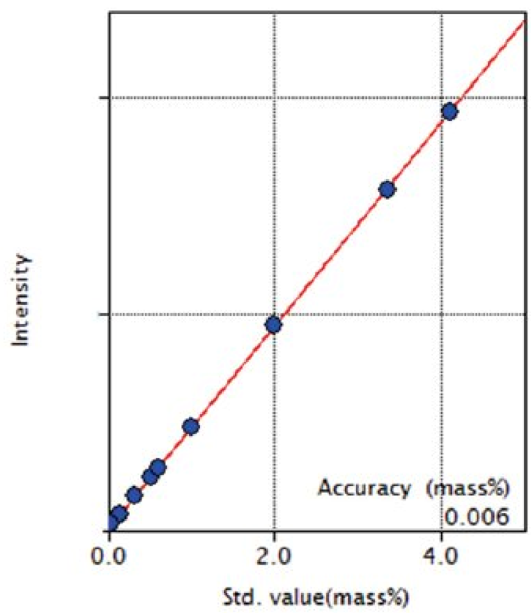

Figure 5: Calibration curve of Ni

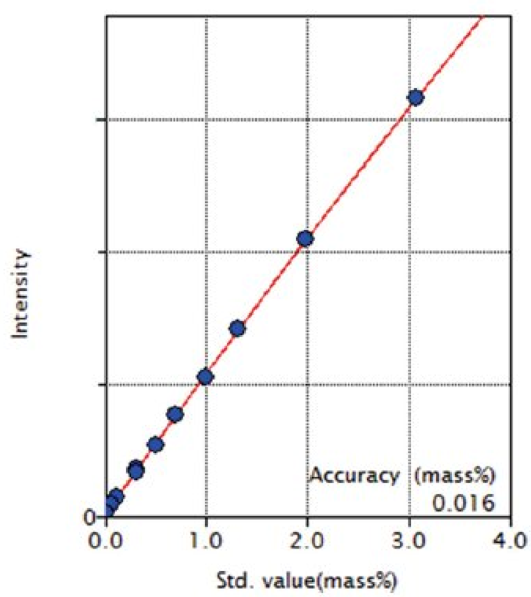

Figure 6: Calibration curve of Cr

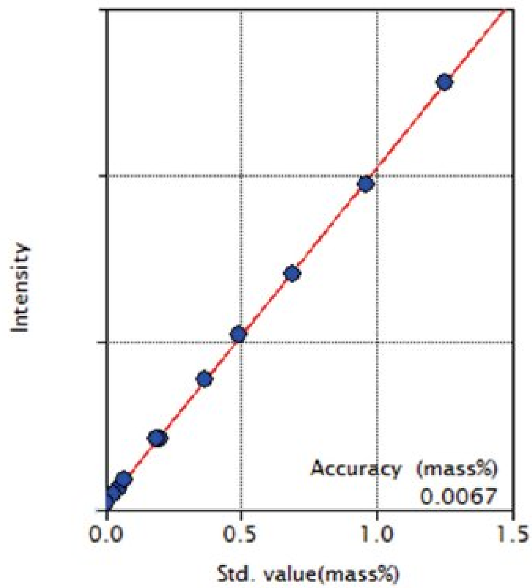

Figure 7: Calibration curve of Mo

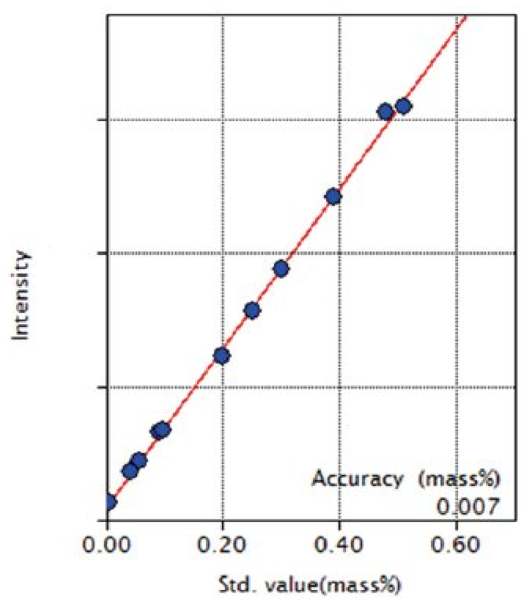

Figure 8: Calibration curve of Cu

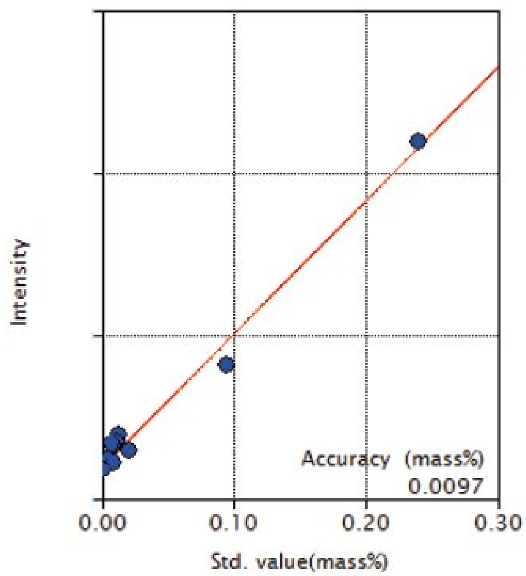

Figure 9: Calibration curve of Al

Results

The accuracies of the calibration curves are listed in Table 1 and the repeatability of the test results (10 times) are indicated in Table 2.

Table 1: Accuracy of calibration curves (unit : mass%)

Component	Concentration range	Accuracy
Si	0.008 - 0.732	0.0077
Mn	0.0057-1.59	0.0097
P	0.0025 - 0.044	0.0009
S	0.0045 - 0.041	0.0009
Ni	0.041 - 4.1	0.0060
Cr	0.0072 - 3.08	0.016
Mo	0.005 - 1.25	0.0067
Cu	0.0058 - 0.51	0.007
V	0.0006 - 0.4	0.0031
Al	0.0007 - 0.24	0.0097

Table 2: Result of repeatability NIST1261

Component	Average	Std dev.	RSD%
Si	0.22	0.00077	0.35

Mn	0.67	0.00075	0.11
P	0.0144	0.00026	1.8
S	0.0173	0.00016	1.00
Ni	2.01	0.0022	0.11
Cr	0.70	0.00094	0.13
Mo	0.194	0.00038	0.19
Cu	0.045	0.00041	0.90
V	0.0111	0.00015	1.4
Al	0.0118	0.00054	4.5

Conclusions

The results show that high precision and accurate analysis of the elements in low alloy steel can rapidly be performed using the ZSX Primus III NEXT. It is also possible to analyze high alloys such as stainless steel and nickel alloy with excellent precision using the ZSX Primus III NEXT, which is optimized for X-ray spectrometer for process control of steelmaking including the analyses of slag and ferroalloys.

Related products

ZSX Primus III NEXT

Affordable, high-end, tube-above Industrial WDXRF for the a nalysis of solid samples