

MINIPAL 4

Analysis of major, minor and trace elements in cast iron



Introduction

This data sheet shows that the MiniPal 4 – a high performance bench top energy dispersive X-ray fluorescence spectrometer – is fully capable of analyzing a wide range of major, minor and trace elements in cast iron samples.

Application background

The term cast iron usually refers to gray cast iron, but can mean any of a group of iron-based alloys containing more than 2% carbon (alloys with less carbon are carbon steel by definition). It is made by re-melting iron, often together with substantial quantities of scrap iron and scrap steel, and by taking various steps to remove undesirable contaminants such as phosphorus and sulfur, which weaken the material. The effect of sulfur is balanced by adding manganese to the melt. The silicon content is adjusted to the desired level from 1 to 3% depending on the application. Also other elements are added to the melt before casting produces the final form. In general, cast iron samples may be regarded as being composed of a variety of major, minor and trace elements such as Al, As, Co, Cr, Cu, Fe, Mn, Mo, Nb, Ni, P, Pb, S, Si, Sn, Ti, V, W, and Zr. In this study, majors, minors and traces were measured to show the capabilities of the MiniPal 4.

Instrumentation

Measurements were performed using a PANalytical MiniPal 4 EDXRF spectrometer, equipped with a 30 kV rhodium anode tube, 5 filters, a helium purge facility, a high-resolution Silicon Drift Detector, a spinner and a 12-position removable sample changer.

Simple sample preparation

The cast iron standards and samples were prepared for analysis by polishing slightly with 120 grit sand paper.

Measurement Procedure

Six standards from Brammer Standard Company (Houston, TX, USA) were used to set up calibrations for the 19 major, minor and trace elements listed. Three different measurement conditions were used, each one optimizing the excitation of a group of elements. These measurement conditions are given in Table 1. The total measurement time per sample was less than five minutes. Matrix corrections were made by using either a ratio to the Compton peak or using theoretical alpha coefficients.

Condition	kV	μA	Measurement time (s)	Medium	Filter	Element
1	7	1000	120	Helium	None	Al, Si, P, S
2	14	200	80	Helium	Al Thin	Ti, V, Cr, Mn, Sn
3	30	300	80	Helium	Ag	Fe, Co, Ni, Cu, As, Zr, Nb, Mo, W, Pb

Table 1. Measurement conditions

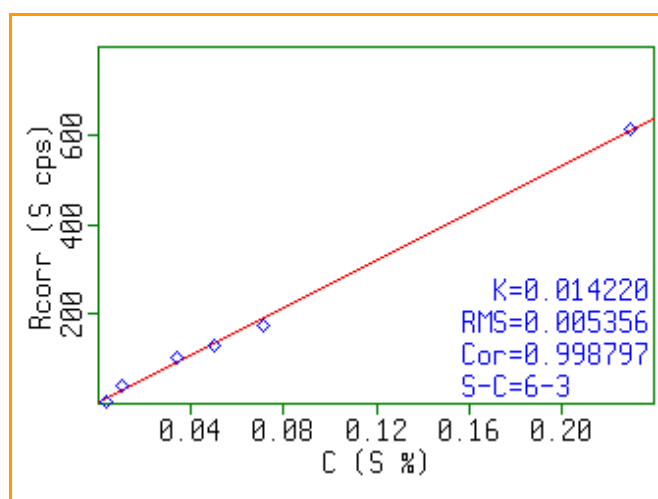


Figure 1. Calibration plot for sulfur in cast iron standards

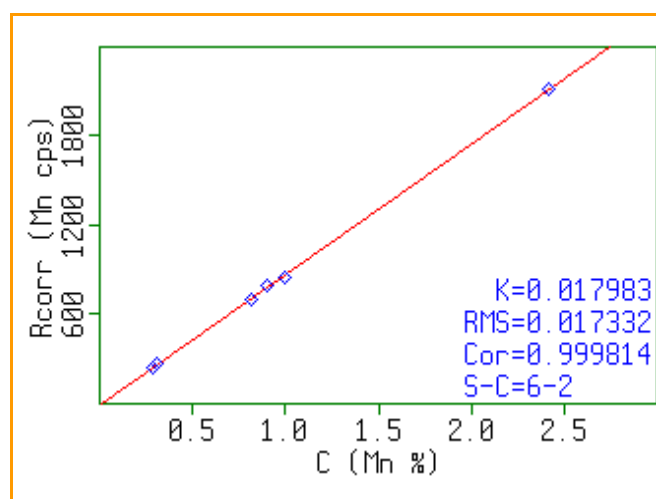


Figure 2. Calibration plot for manganese in cast iron standards

Element	Concentration range (wt%)	RMS (wt%)	K value	Correlation	LLD (wt%)
Al	0 - 0.11	0.0041	0.0120	0.9968	0.006
As	0 - 0.07	0.0024	0.0071	0.9977	0.002
Co	0 - 0.20	0.0080	0.0237	0.9976	0.010
Cr	0 - 2.11	0.0030	0.0076	0.9999	0.003
Cu	0 - 0.51	0.0105	0.0201	0.9993	0.003
Fe	84 - 94	0.3970	0.0413	0.9960	-
Mn	0.3 - 2.42	0.0173	0.0180	0.9998	0.006
Mo	0 - 1.60	0.0089	0.0141	0.9999	0.002
Nb	0 - 0.04	0.0009	0.0027	0.9990	0.002
Ni	0 - 5.27	0.0444	0.0307	0.9999	0.009
P	0 - 0.57	0.0119	0.0283	0.9992	0.003
Pb	0 - 0.005	0.0013	0.0039	0.8840	0.003
S	0 - 0.23	0.0054	0.0142	0.9988	0.003
Si	0.3 - 3.04	0.1570	0.1546	0.9956	0.003
Sn	0 - 0.10	0.0149	0.0388	0.9552	0.009
Ti	0 - 0.16	0.0037	0.0112	0.9988	0.002
V	0 - 0.21	0.0046	0.0134	0.9986	0.003
W	0.01 - 0.04	0.0043	0.0115	0.9730	0.004
Zr	0 - 0.10	0.0054	0.0142	0.9909	0.001

Table 2. Calibration results and detection limits

Accurate calibration results

Figures 1 and 2 show the calibration plots for sulfur and manganese defined by the cast iron standards. The calibration plots show good correlations between the certified concentrations and the intensities. Detailed calibration results are shown in Table 2, together with the Lower Limits of Detection (LLD), which are based on the measurement times given in Table 1.

Precision

One cast iron standard was measured ten times consecutively, using the conditions given in Table 1. The certified and average concentration, the RMS error (1 sigma), the relative RMS error and the relative CSE (Counting Statistical Error) of the repeated measurements are expressed in Table 3. Except for the relatively low concentrations of aluminum, phosphorus and titanium in the standard, all elements show precisions of 1 – 6 % relative. The concentrations of cobalt, lead, sulfur and vanadium in the sample were lower than the detection limit and therefore not reported. Tungsten is not certified in this standard.

Conclusion

Accurate results have been obtained for 6 major, 7 minor and 6 trace elements in cast iron standards. The measurement program only took less than five minutes and resulted in relatively low detection limits. The accurate analysis of Si, P and S can be largely attributed to the excellent resolution of the detector and the powerful software correction models. Finally, the results obtained with the MiniPal 4 are very reproducible.

Element	Certified conc. (wt%)	Average conc. (wt%)	RMS (wt%)	Rel. RMS (%)	Rel. CSE (%)
Al	0.020	0.020	0.002	10	4
As	0.038	0.037	0.001	2.3	2.0
Co	0.007	< LLD	-	-	-
Cr	0.028	0.028	0.002	6.8	1.1
Cu	0.510	0.503	0.005	1.1	1.0
Fe	91.379	90.974	0.063	0.07	0.06
Mn	0.900	0.917	0.006	0.7	0.4
Mo	0.550	0.561	0.003	0.6	0.5
Nb	0.032	0.032	0.001	2.0	1.9
Ni	1.760	1.721	0.021	1.2	0.8
P	0.010	0.012	0.001	8	4
Pb	0.003	< LLD	-	-	-
S	0.003	< LLD	-	-	-
Si	1.320	1.284	0.014	1.1	0.3
Sn	0.003	< LLD	-	-	-
Ti	0.025	0.028	0.002	7	2
V	0.002	< LLD	-	-	-
W	No certified concentration				
Zr	0.016	0.014	0.001	4.7	3.3

Table 3. Results of the repeatability test

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