Application note

EDXRF analysis of cast iron composition with ARL QUANT'X Spectrometer

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Introduction

Cast iron is a class of iron alloys characterized by a carbon content of 1.8–4.0% w/w along with silicon at 1–3% w/w. The carbon content is critical for controlling the properties of the cast iron, and iron alloys with lower carbon content are instead classified as steel. Cast iron, like steel, is widely used in a broad range of applications and industries, making it essential that the alloy can be reliably produced and analyzed.

In the characterization of iron alloys, combustion analysis with infrared spectroscopy is currently the reference technique for the determination of carbon content. Recent improvements in detector windows, however, have also enabled carbon analysis with energy-dispersive X-ray fluorescence (EDXRF), which is a quick, non-destructive method for elemental analysis.

In this application note, the Thermo Scientific™ ARL QUANT'X™ Spectrometer is used to reliably and repeatably analyze the composition of a cast iron sample with a series of calibration curves.

Instrumentation

The ARL QUANT'X EDXRF Spectrometer is equipped with a 50-W, 50-kV rhodium (Rh) anode X-ray tube, a filter wheel with 9 selectable filter positions, and a state-of-the-art silicon drift detector (SDD500G) fitted with a 0.9-µm thick graphene window. This window is highly transparent to low-energy X-rays, like those emitted by carbon atoms, and allows for the detection of all elements in the periodic table from carbon (Z = 6) to americium (Z = 95). Additionally, a sample spinner, a 10- or 20-position sample changer, and the ability to analyze in either air, helium, or vacuum make the ARL QUANT'X Spectrometer a highly versatile analytical tool.

Calibration standards and sample preparation

Eight VASKUT cast iron certified reference materials (CRMs) were used to set up calibration curves for C, Si, P, Si, Cr, Mn, and Ni. Prior to measurement, the standards were surfaced using 120-grit Al_2O_3 sandpaper on a Herzog circular sander. Table 1 shows the certified elemental concentration ranges for these standards.

Element	Conc. range (% w/w)	RMSE (% w/w)	R^2
C	2.32–3.89	0.16	0.9197
Si	0.10–3.80	0.10	0.9943
P	0.03–0.80	0.019	0.9961
S	0.01–0.12	0.005	0.9920
Cr	0.03–0.54	0.006	0.9990
Mn	0.20–1.88	0.03	0.9982
Ni	0.02–0.64	0.003	0.9998

Table 1. Concentration ranges and calibration data.

Measurement conditions

Only two measurement conditions were needed to determine the elemental composition of the cast iron samples (Table 2). Light elements, including carbon, were measured using the Low Za condition, with an excitation voltage of 4 kV and no primary beam filter. To generate enough carbon intensity, a 600 s live time was used for the calibration. Heavier elements were measured with a second condition (Mid Za) which has an excitation voltage of 16 kV and uses a thin Pd primary beam filter. A 120 s measurement live time was used for this condition. All measurements were conducted in vacuum, which is mandatory for the detection of carbon X-ray fluorescence and generally improves sensitivity for light elements.

Carbon Kα signal

Figure 1A shows the spectrum for standard VASKUT 185, obtained with measurement condition Low Za. This standard contains 3.28% w/w carbon. Although well-defined, the carbon signal is still relatively small compared to other elemental lines in the spectrum. Figure 1B superimposes the Low Za spectra obtained for all standards, focusing on the energy range between 0–0.5 keV. As shown, the different carbon concentrations give rise to distinct carbon Kα intensities.

The region between 0.16–0.38 keV, which encompasses the carbon peak, was used to derive the net carbon Kα peak intensity. A straight line was used to correct for the background, and any remaining intensity was attributed entirely to the carbon Kα signal.

Condition	Filter	Voltage (kV)	Current	Atmosphere	Live time (s)	Analytes
Low Za	No filter	4	Auto	Vacuum	600	C, Si, P, S
Mid Za	Pd thin	16	Auto	Vacuum	120	Cr, Mn, Fe, Ni

Table 2. Measurement conditions used to determine cast iron elemental composition.

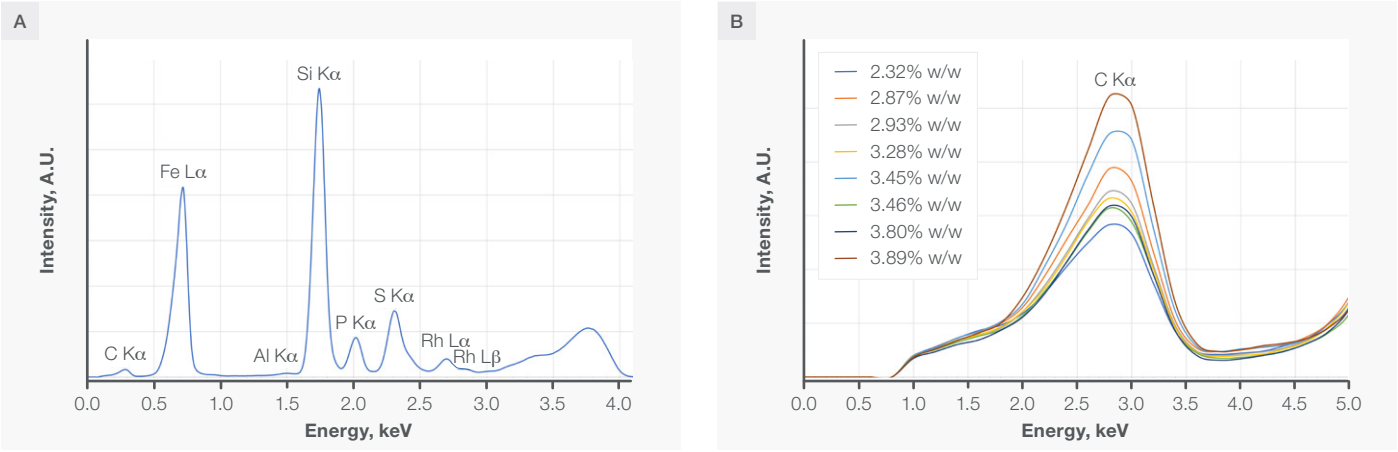

Figure 1. A) Full spectrum for standard VASKUT 185. B) An overlay of the spectra obtained for all eight VASKUT standards, zoomed in on the carbon peak. All spectra were obtained with condition Low Za (4 kV, no filter).

Calibration results

Calibration curves for the main alloying elements are shown in Figure 2. For most of the elements, no matrix correction is required, and the elemental line intensity is linear with respect to concentration. Si and Ni require one influence coefficient to correct for matrix effects. For these elements, the matrix corrected intensity is a linear function of the concentration. Table 1 shows the root mean squared error (RMSE) as well as the correlation coefficient (R^2) for every element calibration.

Generally speaking, satisfactory results are obtained for all elements. The calibration curve for carbon (C) clearly shows that quantification of carbon in cast iron is possible with the ARL QUANT'X Spectrometer, when equipped with the SDD500G detector with its ultra-thin graphene window.

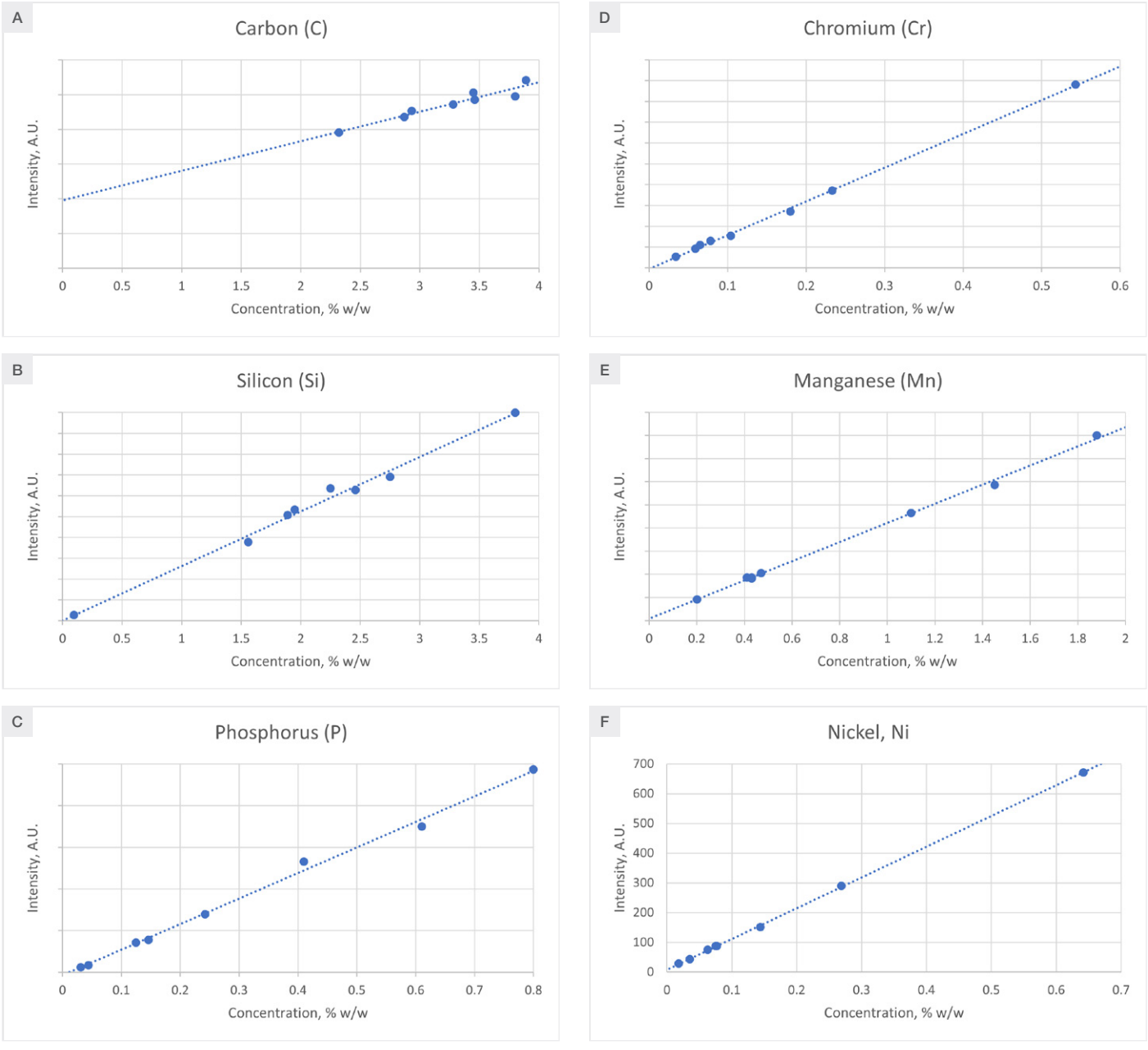

Figure 2. Calibration curves for elements found in cast iron.

Repeatability

To estimate the short-term repeatability (or precision) of the analysis, standard VASKUT 185 was measured 10 times consecutively. Table 3 compares the average concentration of these measurements to the certified concentration for each element. Overall, precision appears to depend on the measurement time. In addition to the measurement times used in the calibration (i.e., 120 and 600 s), data was also collected at 400 and 80 s, 200 and 40 s, as well as 100 and 20 s live times. Even at the shortest measurement times, the results remain sufficiently precise for a reliable analysis.

Conclusions

This application note demonstrates that the ARL QUANT'X EDXRF Spectrometer can quantify a range of elements in cast iron, including C, Si, P, S, Cr, Mn, and Ni. Simple calibration curves, requiring few matrix corrections, were easily generated for all elements. Repeatability data shows excellent accuracy and precision, even at short measurement times. These results highlight the applicability of EDXRF for the determination of cast iron composition. The ARL QUANT'X Spectrometer, fitted with the latest detector window technology, clearly expands the capabilities of EDXRF analysis.

Result (% w/w)	Live time (s)	C	Si	P	S	Live time (s)	Cr	Mn	Ni
Certified		3.28	1.89	0.146	0.062		0.233	0.43	0.269
Average	**600**	3.37	1.966	0.1440	0.1255	**120**	0.2364	0.435	0.281
1-sigma		0.04	0.0017	0.0003	0.0002		0.0014	0.004	0.003
Average	**400**	3.19	1.899	0.1337	0.1212	**80**	0.2302	0.433	0.2721
1-sigma		0.03	0.0017	0.0004	0.0004		0.0016	0.003	0.003
Average	**200**	3.09	1.870	0.1301	0.1193	**40**	0.2281	0.436	0.269
1-sigma		0.06	0.0017	0.0002	0.0002		0.0019	0.005	0.003
Average	**100**	3.34	1.992	0.1485	0.1277	**20**	0.241	0.438	0.283
1-sigma		0.09	0.006	0.0013	0.0005		0.004	0.007	0.003

Table 3. Repeatability results for the VASKUT 185 cast iron standard at different live times.

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