Analysis of Ferrous Base with Thermo Scientific ARL PERFORM'X 4200 W Advanced WDXRF Spectrometer

Keywords
ARL PERFORM'X, XRF, WDXRF, ferrous base, X-ray fluorescence

ARL PERFORM'X Series Advanced X-ray Fluorescence Spectrometer

Goal

Show the analytical performance of ARL PERFORM'X in iron matrix.

Introduction

Ferrous base materials are important products in metals industry as they are the foundation in many applications such as building, automotive and many manufacturing processes. It is therefore important to be able to accurately analyze these materials to confirm compliance with their chemical specifications and allow for high quality and efficient production.

There are several kinds of irons which are distinguished by their composition and use. They belong to two main categories:

- Pig irons (hot metal), forming the basic production for the manufacture of steel
- Cast irons, used for the production of semimanufactured products

From a metallographic point of view, a distinction can be made between white cast iron with a cementite structure and grey cast iron which contains free graphite either in the form of laminae or nodules. These make grey cast iron inhomogeneous and therefore difficult to analyze. Alloy cast irons also exist where alloying elements such as nickel, chromium, manganese, copper, etc. are added to improve hardness, corrosion resistance or engineering properties.

Low alloy steels

This category covers steels which are destined for a wide variety of uses, such as the production of:

- Steel castings, rails, axles, boiler and ship plates, automobile bodies
- Girders, all kinds of bridge and structural sections
- Wires, nuts, bolts and forgings of almost any description
- Springs, cutting steels

From a compositional point of view, these steels can be distinguished by the fact that the alloying elements generally total less than 5-7 %. Typically, the main alloying elements are present at less than the following concentrations:

Mn 2 %; Cr 3 %; Ni 5 %; Cu 1.5 %; Mo 1.5 %; V 1 %.

High alloy steels

High alloy steels contain, in addition to iron and carbon, notable quantities of one or more of the following elements: nickel, chromium, manganese, silicon, cobalt, tungsten, molybdenum and vanadium. Included under this heading are:

Stainless steels of types such as 18/8, austenitic, maraging, martensitic and all types of special stainless steels

- Tool steels
- High speed steels
- High manganese steels

Instrument parameters and conditions

The Thermo Scientific ARL PERFORM'X 4200 XRF spectrometer can be calibrated using commercially available standards or well analyzed samples from the user. It should be stressed that an XRF spectrometer is a very accurate comparator, but the accuracy of the final analysis is entirely dependent on the quality of the standards used for calibration.

Typical performance in low alloy steel samples

Table 1 gives a summary of analytical results obtained using a set of international standards with best parameters in regards to crystal, detector, collimator and power. Limits of detection (LoD) were determined using 100 seconds per element counting times.

Element	Line	LoD (ppm)
Al	Kα	3.2
Si	Kα	1.9
P	Kα	1.1
S	Kα	0.7
Ti	Kα	0.9
V	Kα	0.9
Cr	Kα	1
Mn	Kα	1.6
Co	Kα	4
Ni	Kα	2.4
Cu	Kα	2
Ta	Lβ	5.7
W	Lα	4
As	Kβ	7.2
Pb	Lβ	3
Zr	Kα	1.3
Nb	Kα	1.2
Mo	Kα	1.3
Sn	Kα	7.3
Sb	Kα	8.7

Table 1. Typical limits of detection in 100 s in ferrous matrix.

Typical stability test

A stability test consisting of several runs on a typical sample over 12 days was performed. The stability of an instrument reflects the precision that can be obtained. It should be noted that the accuracy of the instrument is dependent upon the accuracy of the standards used to calibrate the instrument. The stability for each element using 10 s and 100 s counting time is shown in Table 2 along with the concentration of the analyte in our test sample.

Element	Line	Concentration (ppm)	Std. Dev. (ppm in 100s)	Std. Dev. (ppm in 10s)
P	Kα	432	1.1	3.5
S	Kα	340	0.9	2.8
Al	Kα	840	2.6	8.2
Ti	Kα	959	1	3.2
Cr	Kα	3034	1.6	5.1
Ni	Kα	6164	4.1	13
Cu	Kα	5243	3.5	11.1
As	Kα	980	4.1	13
Nb	Kα	3082	1.9	6
Mo	Kα	795	1.1	3.5
Sb	Kα	179	3	9.5
W	Lα	2139	3.8	12
Pb	Lβ	55	1.2	3.8
Ta	Lβ	2207	4.8	15.2

Table 2. Long term stability over 12 days with different counting times.

UniQuant standard-less analysis

As in many real life cases, obtaining any or enough standards to create a calibration is not always possible. Or you may receive a sample that is not flat and/or does not encompass the entire diameter of the sample cassette. In such situations, we can offer the most comprehensive standard-less software package on the market to solve these unique issues: Thermo Scientific UniQuant package.

The UniQuant package is a factory calibration based on 64 pure element standards that allows for concentration determination of unknown samples in any matrix, size or shape by using complex mathematical algorithms. These algorithms correct for matrix effects as well as interelemental effects to provide an accurate result of a totally unknown sample.

As can be seen in Table 3 the samples can be measured either as bulk metallic piece (with a maximum diameter of 52 mm) or as drillings (see picture). UniQuant is capable of good quantification in both instances.

Element	Chemical %	UniQuant as bulk sample %	UniQuant as 159 mg drillings %
W	14.2	14.04	13.99
Cr	3.56	3.73	3.79
V	0.52	0.524	0.507
Mn	0.29	0.3	0.32
Mo	0.22	0.223	0.298
Co	0.21	0.194	Not found
Ni	(0.19)	0.163	0.133
Cu	(0.1)	0.0975	0.022
Sn	(0.035)	0.049	0.027
P	0.021	0.015	0.077

Table 3. UniQuant analysis on bulk sample and drillings – parenthesis indicate non certified values.

Conclusion

It is seen that analysis of irons and steels can be performed with ease using the ARL PERFORM'X sequential XRF spectrometer. The precision and accuracy are excellent in these matrix types for routine or R&D analysis.

Furthermore, operation is made easy through the new state-of-theart OXSAS software which operates with the latest Microsoft Windows® 10 package.

Learn more at **thermofisher.com/performx**

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