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**Iron and Steel – Determination
of Multi-Element Contents – X-Ray
Fluorescence Spectrometry (Routine Method)**

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Foreword

Appendix A in this Part of GB/T 223 is normative.

This Part was proposed by China Iron and Steel Industry Association.

This Part shall be under the jurisdiction of National Technical Committee for Standardization of Steel.

Drafting organization of this Part: Wuhan Iron and Steel (Group) Corporation.

Chief drafting staffs of this Part: Shen Ke, Liu Xiang, Li Xiaojie, and Gu Fang.

Iron and Steel – Determination of Multi-Element Contents – X-Ray Fluorescence Spectrometry (Routine Method)

1 Scope

This Part of GB/T 223 specifies using x-ray fluorescence spectrometry to determine the contents of silicon, manganese, phosphorus, sulfur, copper, aluminum, nickel, chromium, molybdenum, vanadium, titanium, tungsten, and niobium.

This Part is applicable to cast iron, pig iron, non-alloy steel, low-alloy steel; the determination range of each element can refer to Table 1.

Table 1 Elements and Determination Range

Analytical Elements	Determination Range (Mass Fraction) / %
Si	0,002~4,00
Mn	0,002~4,00
P	0,001~0,70
S	0,001~0,20
Cu	0,002~2,00
Al	0,002~1,00
Ni	0,003~5,00
Cr	0,002~5,00
Mo	0,002~5,00
V	0,002~2,00
Ti	0,001~1,00
W	0,003~2,00
Nb	0,002~1,00

2 Normative References

The provisions in following documents become the provisions of this Part of GB/T 223 through reference in this Part. For dated references, the subsequent amendments (excluding corrigendum) or revisions do not apply to this Standard, however, parties who reach an agreement based on this Standard are encouraged to study if the latest versions of these documents are applicable. For undated references, the latest edition of the referenced document applies.

GB/T 6379.1 Accuracy (Trueness and Precision) of Measurement Methods and

Results - Part 1: General Principles and Definitions (GB/T 6379.1-2004, ISO 5725-1:1994, IDT)

GB/T 6379.2 Accuracy (Trueness and Precision) of Measurement Methods and Results - Part 2: Basic Method for the Determination of Repeatability and Reproducibility of a Standard Measurement Method (GB/T 6379.2-2004, ISO 5725-2:1994, IDT)

GB/T 16597 Analytical Methods of Metallurgical Products General Rule for X-Ray Fluorescence Spectrometric Methods

GB/T 20066 Steel and Iron - Sampling and Preparation of Samples for the Determination of Chemical Composition (GB/T 20066-2006, ISO 14284: 1996, IDT)

3 Principles

When the X-ray tube produced primary X-ray irradiates to the flat and smooth sample surface, after the crystal light splitting of the produced characteristic X-ray, the detector measures the X-ray fluorescence strength at the selected characteristic wavelength corresponding 2θ angle. According to calibration curve and measured X-ray fluorescence strength, calculate the mass fraction of silicon, manganese, phosphorus, sulfur, copper, aluminum, nickel, chromium, molybdenum, vanadium, titanium, tungsten, and niobium in the sample.

4 Reagents and Materials

4.1 P10 gas (mixed gas with 90% argon and 10% methane) used for flow-gas proportional counter.

4.2 Certified standard sample/standard material

When the certified standard sample/standard material is used for daily analyzing and plotting calibration curve, the contents of analytical elements in the selected serial certified standard sample/standard material shall cover the analytical range and have appropriate gradient; when performing drift correction against the apparatus, the selected certified standard sample/standard material shall have good uniformity, and is close to the upper and lower limit of the calibration curve.

5 Apparatus

5.1 Sample-preparing device

The cutting equipment adopts abrasive wheel cutting machine, specimen surface

7 Preparation of Apparatus

7.1 Working environment of apparatus

The working environment of apparatus shall conform to the provisions of GB/T 16597 *Analytical Methods of Metallurgical Products General Rule for X-Ray Fluorescence Spectrometric Methods*.

7.2 Working conditions of apparatus

The X-ray spectrometer shall, before measurement, optimize the working conditions as per the requirements of the manufacturer; and shall pre-heat at least 1h before measurement to stabilize the apparatus.

8 Analytical Procedures

8.1 Measuring conditions

According to the type of apparatus used, type of specimen, analytical elements, coexisting elements and change range of their contents, select appropriate measuring conditions:

- (1) The counting time of analytical elements depends on the content of quantitative elements and the analytical accuracy to be achieved; it generally is 5s~60s;
- (2) The counting rate shall generally not exceed 4×10^5 ;
- (3) The selection of light tube voltage and current shall consider the measuring lowest excitation voltage of spectral line and rated power of light tube;
- (4) When using several sample boxes, the sample box mask shall not significantly influence the analytical results; the diameter of sample box mask is generally about 20mm~35mm;
- (5) Recommend the rotation working mode of sample box;
- (6) The used analytical line, recommended used spectroscopic crystal, 2θ angle, light tube voltage and current, and possible interference elements are listed into Table 2.

be-tested elements in certified standard sample/standard material is confirmed; otherwise, if there is significant difference, find out the reasons, re-calibrate and confirm.

8.3 Unknown specimen analysis

8.3.1 Standardization of apparatus

Confirm and analyze the standardized sample regularly; when the apparatus occurs drift, perform drift correction against the apparatus through measuring the X-ray fluorescence intensity of the standardized sample.

Calculate as per the following formulas:

$$I_i = \alpha \times I'_i + \beta \quad \dots\dots\dots (5)$$

$$\alpha = \frac{I_h - I_l}{I'_h - I'_l} \quad \dots\dots\dots (6)$$

$$\beta = I_h - \alpha \times I'_h \quad \dots\dots\dots (7)$$

Where:

I_i – correction intensity of analytical element i ;

I'_i – measurement intensity of analytical element i ;

I'_h – measurement intensity of high-content standardized sample;

I'_l – measurement intensity of low-content standardized sample;

I_h – initial intensity of high-content standardized sample;

I_l – initial intensity of low-content standardized sample;

α, β – drift correction coefficient.

8.3.2 Confirmation of standardization

After drift correction, analyze the certified standard sample/standard material, and confirm that the analytical value conforms to the provisions of 8.2.2, or is within the accreditation range of the laboratory.

8.3.3 Measurement of unknown specimen

According to the working conditions selected in 8.1, use X-ray fluorescence spectrometer to measure the fluorescence intensity of analytical elements in the unknown samples, each sample shall be measured twice at least.

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