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Stainless Steel – Determination of Manganese, Nickle, Chromium, Molybdenum, Copper and Titanium – Handheld Energy Dispersive X-Ray Fluorescence Spectrometric Method (Semiquantitative Method)

不锈钢 锰、镍、铬、钼、铜和钛含量的测定

手持式能量色散 X 射线荧光光谱法（半定量法）

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Stainless Steel – Determination of Manganese, Nickle, Chromium, Molybdenum, Copper and Titanium – Handheld Energy Dispersive X-Ray Fluorescence Spectrometric Method (Semiquantitative Method)

1 Scope

This Standard specifies using handheld energy dispersive X-ray fluorescence spectrometric method to determine the contents of manganese, nickel, chromium, molybdenum, copper and titanium.

This Standard is applicable to the semiquantitative analysis of the manganese, nickel, chromium, molybdenum, copper and titanium in the block-shaped stainless-steel sample; the determination range of each element can refer to Table 1.

Table 1 – Elements and Determination Ranges

Names of elements	Determination range [a] (mass fraction) / %
Manganese	0.5~7.9
Nickel	0.2~20
Chromium	10~25
Molybdenum	0.3~6.2
Copper	0.2~3.4
Titanium	0.2~0.6

[a] The specified content range indicates that a precision test has been performed; it may also be applicable exceeding this range, but it needs to be confirmed by the laboratory.

2 Normative References

The following documents are essential to the application of this document. For the dated documents, only the versions with the dates indicated are applicable to this document; for the undated documents, only the latest version (including all the amendments) are applicable to this document.

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

Results - Part 1: General Principles and Definitions

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 31364 Test Methods for Main Performance of Energy Dispersive X-Ray Fluorescence Spectrometer

3 Principle

The sample is excited by high-energy X-ray; each element in the sample emits a characteristic X-ray fluorescence spectrum. The preliminary contents of manganese, nickel, chromium, molybdenum, copper and titanium in the sample shall be calculated from the calibration curve by measuring the characteristic X-ray intensity produced. Finally, the test results are corrected by testing the similar certified standard substance/standard sample and using the deviation between the certified value of the element in the certified standard sample/standard substance and the measured value.

4 Reagents and Materials

A set of certified standard substance/standard sample. When using for daily analysis and drawing calibration curve, the content of each measured element in the selected series of certified standard substance/standard sample shall cover the analysis range and have a gradient; when using for correcting the drift of the instrument, the selected standard sample/standard substance does not necessarily have certificate, but shall have good uniformity.

5 Apparatus

Handled energy dispersive X-ray fluorescence spectrometer. X-ray tube power ≥ 2W; the resolution ≤ 180eV.

6 Sampling and Sample Preparation

The diameter of the effective to-be-tested area of the sample to be tested shall be larger than the diameter of the measurement area selected by the instrument; it is recommended that the diameter of the to-be-tested area shall be no less than 10mm.

The certified standard substance/standard sample, analytical sample shall be ground and cleaned under the same conditions. The surface of the sample shall be smooth,

point shall be no less than 30s; take the average value. If necessary, select the standard sample/standard substance with the similar compositions to correct the measurement result of the to-be-tested sample. According to experience, the proximity of between selected certified standard substance/standard sample and the to-be-tested sample shall refer to the following requirements:

a) Main elements (≥1%): the content difference between certified standard substance/standard sample and to-be-tested sample shall not exceed ±10%.

b) Trace elements (<1%): the content difference between certified standard substance/standard sample and to-be-tested sample shall not exceed ±50%.

According to the instructions requirements of the instrument, check the instrument periodically; so that correct the daily drift of the instrument.

9 Calculation of Results

According to the fluorescence intensity of the tested element in the sample, calculate the content of the to-be-tested element through the calibration curve.

The analysis results of the tested elements shall be within the content range covered by the calibration curve.

The measurement results shall be reported in the form of mass fraction (%); in general, when the analytical content in the calculation results is greater than or equal to 1%, retain two digits after the decimal point; when it is less than 1%, retain three digits after the decimal point.

10 Precision

The precision test of this Standard is carried out by 10 labs for the contents of 4 levels of manganese, 6 levels of nickel, 4 levels of chromium, 4 levels of molybdenum, 5 levels of copper and 3 levels of titanium; each lab shall test 3 times against each level of element under the repeatability conditions specified in GB/T 6379.1. The participating labs pass and analyze 29 to-be-tested samples and standard samples with similar contents; correct and calculate the results of the to-be-tested sample; the correcting mode can refer to Table 8. The samples used for the precision test can refer to Appendix A. The original data shall be statistically analyzed according to GB/T 6379.2; the precision test results of each element shall refer to Table 2 ~ Table 7.

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