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**High alloy steel - Determination of multi-element contents
- X-ray fluorescence spectrometry (routine method)**

高合金钢 多元素含量的测定

X 射线荧光光谱法（常规法）

(ISO 17054:2010, Routine method for analysis of high-alloy steel by
x-ray fluorescence spectrometry (XRF) by using a near-by technique, MOD)

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Foreword

This Standard is drafted in accordance with the rules given in GB/T 1.1-2009.

This Standard uses redraft law to use by modification ISO 17054:2010 "Routine method for analysis of high-alloy steel by x-ray fluorescence spectrometry (XRF) by using a near-by technique".

The technical differences between this Standard and ISO 17054:2010 are as follows:

- DELETE the contents and formulas related to accuracy and uncertainty after Table A.12 in Appendix A;
- DELETE Appendix B.

Chinese documents which have a consistent correspondence with the international documents normatively referenced in this Standard are as follows:

- GB 223.84-2009 Steel and iron - Determination of titanium content - Diantipyrylmethane spectrophotometric method (ISO 10280:1991, IDT)
- GB/T 223.64-2008 Iron, Steel and Alloy - Determination of Manganese Content - Flame Atomic Absorption Spectrometric Method (ISO 10700:1994, IDT)
- GB/T 20066-2006 Steel and Iron - Sampling and Preparation of Samples for the Determination of Chemical Composition (ISO 14284:1996, IDT)
- GB/T 223.5-2008 Steel and iron - Determination of acid-soluble silicon and total silicon content - Reduced molybdosilicate spectrophotometric method (ISO 4929-1:1986 and ISO 4929-2:1988, MOD)
- GB/T 223.11-2008 Iron, Steel and Alloy - Determination of Chromium Content - Visual Titration or Potentiometric Titration Method (ISO 4937:1986, MOD)
- GB/T 223.65-2012 Steel, iron and alloy - Determination of cobalt content - Flame atomic absorption spectrometric method (ISO 11652:1997, IDT)
- YB/T 4395-2014 Steel Determination of molybdenum, niobium and tungsten contents - Inductively coupled plasma atomic emission spectrometric method (all parts of ISO 13899, MOD)

This Standard was proposed by China Iron and Steel Association.

High alloy steel - Determination of multi-element contents

- X-ray fluorescence spectrometry (routine method)

1 Scope

This Standard specifies a routine method for improving the analysis performance of X-ray fluorescence spectrometry, by using a “near-by technique”.

The “near-by technique” requires at least one target sample [preferable a certified reference material/reference sample (CRM)] of a similar composition as the unknown sample.

This Standard is applicable to the determination of silicon, manganese, phosphorus, chromium, nickel, molybdenum, copper, cobalt, vanadium, titanium, and niobium contents in high alloy steel. The determination range of each element is shown in Table 1.

Table 1 -- Content range

Elements	Content range (mass fraction) ^a /%
Si	0,05~1,5
Mn	0,05~5,0
P	0,005~0,035
Cr	10~25
Ni	0,1~30
Mo	0,1~6,5
Cu	0,02~1,5
Co	0,015~0,30
V	0,015~0,15
Ti	0,015~0,50
Nb	0,05~1,0
^a A	

Keys:

- The specified content range indicates that a precision test has
A been performed. Those outside the range may be applicable, but need to be validated by laboratory.

This Standard applies to the analysis of either chill-cast or wrought samples having a diameter of at least 25 mm and with a carbon content of less than 0.3% (SEE Note). Other elements shall have a content below 0.2%.

4.4 Pure ethanol.

5 Instruments

5.1 Sample preparation equipment

The grinding equipment can be a grinder, a sandpaper grinding disc, or an abrasive belt grinder, or a milling machine, a lathe, or the like.

For the final sample preparation, USE 180-grit (0.080 mm) or finer aluminium oxide (SEE Note) of abrasive belts or grinding wheels. Other methods (such as turning) can also be used. However, in any case, CRM, RM, and samples shall be prepared under the same conditions.

Note: The use of silicon carbide sandpaper and zirconia sandpaper affects the determination of silicon and zirconium, respectively. The determination of aluminium is often contaminated by aluminium oxide.

5.2 X-ray fluorescence analyzer

A commercially available simultaneous or sequential wavelength dispersive spectrometer.

5.3 X-ray tube

A light tube which contains high-purity element target. It is recommended to use rhodium target for the analysis of steel.

5.4 Analyzing crystals

To cover all the elements specified in this Standard, flat or curved crystals made of LiF (200) and PE (for light elements, atom no. approximately < 22) are required. Crystals made of LiF (220) and Ge or other crystals optimized for individual elements may also be used.

5.5 Collimators

For sequential instruments, a two collimator system is necessary: a coarse collimator for light elements (atom no. approximately < 22) and a fine collimator for heavy elements.

5.6 Detectors

One scintillation detector for heavy elements and one gas-flow proportional detector for light elements (atom no. approximately < 22). Sealed proportional detectors may also be used. Whether the detector uses single mode or dual

avoid any influence from the sample preparation. When not in use, they shall be stored in a desiccator. Before measurement, CLEAN the surface with pure ethanol (4.4).

9 Analytical procedures

9.1 Preparation of instruments

PREPARE the instrument for operation according to the manufacturer's instructions. Since most of the instruments are used for routine analyzes, it is assumed that they are running, and in most cases they also already are calibrated for steel. If the instrument has been turned off for a long period (several hours), ensure that the conditions have stabilized before starting the measurements, e.g., temperature and vacuum. VERIFY the calibration status by analyzing the SPC samples or CRMs.

9.2 Measurements

SELECT a certified reference material/reference sample (CRM) with a composition close to the composition of the unknown sample to be measured. The requirement for closeness in composition of the CRM shall be in accordance with 13.2. According to the instrument user's manual, LOAD and MEASURE the unknown sample and the CRM. BE sure that the sample is properly fixed in the sample cup.

To avoid effects from grinding striations, the instrument shall be equipped with a sample spinner. If there is no such device, it shall orient the sample in such a way that the relation between the X-ray beam and the grinding striations are always the same from measurement to measurement.

CALCULATE the contents according to Clause 14.

10 Calibration

The calibration is done when the instrument is installed, after major repairs are made, or as indicated by SPC data. To compensate for the instrumental day-to-day drift, standardization or recalibration procedures are used.

If a new calibration is going to be prepared, FOLLOW the calibration strategy according to CR 10299. Typical operating conditions for analysis of steel are given in Table 2.

MEASURE the calibration samples together with the standardization samples according to the instrument user's manual.

and further actions (start measuring unknowns, standardization or recalibration) are based on comparison with the specified control limits.

The control limits, upper and lower warning limits ($\pm 2 s$) and upper and lower action limits ($\pm 3 s$), are calculated from a set of measurements (>25) of the check samples. The whole measurement process shall be performed under reproducibility conditions within laboratory (same instrument, same sample, different sample cups, different operators, and different time).

Control charts for checking method performance are built up in the same way but the check samples shall be treated as unknown samples, e.g., sample preparation is included.

13 “Near-by technique” method

13.1 Principles

Since this Standard is a routine method, it is assumed that most users have their instruments already calibrated for steel (often optimized for in-house purposes). The content range given in Clause 1 represents a wide range of different steels. In order to eliminate the effect of not having a calibration which covers all different kinds of steel or to improve (minimize) the uncertainty in the reported values, a “near-by technique” method can be used.

By measuring the unknown sample together with a sample with known content (of the same steel grade), a correction of the calculated contents for the unknown sample can be done.

Note: Be careful when the sample content exceeds the calibrated range but is still within the range given in Clause 1. As an example, if the routine method is calibrated for a Mo content up to 3% and an unknown sample with a Mo content of 5% is going to be measured, ensure that the detector is not saturated due to high intensities. If that is the case, a new calibration shall be done covering that range.

13.2 Principles for selecting CRMs

The selection of CRM shall follow the following principles:

- a) USE only CRMs from qualified suppliers.

Note: A complete set of internationally recognized CRMs covering all steel grades are not available. Other CRMs can be used (e.g., in-house control sample) if they are certified by referee procedures based on SI units, for those elements of interest.

- b) If possible, these CRMs shall have the same metallurgical history (micro

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