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**NATIONAL STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA**

GB/T 32548-2016

**Steel and iron -- Determination of tin, antimony, cerium, lead
and bismuth -- Inductively coupled plasma mass spectrometric
method**

钢铁 锡、锑、铈、铅和铋的测定 电感耦合等离子体质谱法

(English Translation)

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Foreword

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

Revision of this standard ISO 16918-1.:2009 "Determination of nine elements in steel inductively coupled plasma mass spectrometry Part 1.

Tin, antimony, cerium, lead and bismuth. " the main technical differences between this standard and ISO 16918-1.:2009 are as follows.

- the references in the ISO 5725-1, ISO 5725-2 and ISO 5725-3 are included in the "2 normative reference document" Reference section to facilitate the use of standards;

--- in accordance with GB/T 20001.4 preparation requirements, adjust the order of reagents, remove the international standard "4.4 nitric acid", and the international standard in the "10 standard solution preparation" and "11 internal standard solution preparation" adjusted to the standard "4 reagent", some of which explanatory text the word is adjusted to note 1 of the "7 analysis step"

--- 5.1 added a "single-line suction tube", improve the standard preparation;

--- in accordance with the requirements of GB/T 20001.4, the international standard "6 measurement performance requirements" in the content adjusted to the standard "5 instrument";

- the international standard "7 sample" adjusted to the standard "6 sample", improve the standard preparation;
 - the international standard "9 steps" to adjust the standard "7 analysis steps" to improve the preparation of the standard;
 - the international standard "8 cleaning", "13 inductively coupled plasma mass spectrometry prepared" merger, adjusted for the standard "7.5 instrument preparation" in line with the preparation of GB/T 20001.4 requirements for ease of use;
 - the international standard "12 calibration solution preparation" adjusted to this standard "7.4 calibration solution preparation", improve the standard preparation;
 - the international standard "14 inductively coupled plasma mass spectrometry" adjusted to this standard "7.6 measurement", improve the standard preparation;
 - the international standard "15 drawing calibration curve" adjusted to this standard "7.7 to draw the calibration curve", improve the standard preparation;
 - the international standard "16 results that" adjusted to the standard "8 results that", improve the standard preparation;
 - the international standard "17 test report" adjusted to this standard "9 test report", improve the standard preparation;
 - the international standard "PTFE high pressure tank" to "fluorine plastic high pressure cans", the expression is more clear, in line with the standard preparation requirements;
 - delete the international standard in Table 1 ~ Table 4, because the text can be clearly stated, the follow-up form serial number, improve the standard preparation;
 - delete the international standard Chapter 12 in the three suspension section, improve the standard preparation;
 - the international standard "10.2 cerium standard solution preparation" specific, so that the method easy to operate;
 - the international standard in the 12.1.2 note into the text, improve the standard preparation;
 - in Appendix A to increase the measured element mass fraction w and repeatability r , laboratory reproducibility limit R_w , inter-laboratory reproduction the relationship between the law of the relationship between the degree of R to facilitate the application of the standard;
- In the standard solution of 7.3.1.2 and 7.3.2.2.
- increase the sample in the carbon content of more than 1% when the sample dissolved method, more in line with the use of our country;

- After taking the perchloric acid smoke, the method of dissolving the salt with dilute water is added to make the method more perfect.

- In 7.3.2.2, the note is added to illustrate the precautions for the dissolution of the sample when the sample contains tungsten and niobium, making the method easy to operate.

Fixed bug that ^{117}Sn and ^{122}Sn interfere with isotopes in Appendix B.

This standard is proposed by China Iron and Steel Industry Association.

This standard is under the jurisdiction of the National Steel Standardization Technical Committee (SAC/TC183).

The drafting unit of this standard. steel research institute, steel research nanke detection technology limited company.

1 Scope

This standard specifies the determination of trace tin, antimony, cerium, lead and bismuth in steel by inductively coupled plasma mass spectrometry (ICP-MS) method.

This standard applies to the following mass fraction ($\mu\text{g/g}$) range of trace elements. tin $5\mu\text{g/g} \sim 200\mu\text{g/g}$, antimony $1\mu\text{g/g} \sim 200\mu\text{g/g}$ of cerium, $10\mu\text{g/g}$ to $1000\mu\text{g/g}$ of cerium, $0.5\mu\text{g/g}$ to $100\mu\text{g/g}$ of lead, $0.3\mu\text{g/g}$ to $30\mu\text{g/g}$ of bismuth.

Determination of trace element interference by inductively coupled plasma mass spectrometry is given in Appendix B.

2 Normative reference documents

The following documents are indispensable for the application of this document. For dated references, only the dated edition applies to this article Pieces. For undated references, the latest edition (including all modifications) applies to this document.

GB/T 6379.1 Accuracy (accuracy and precision) of measurement methods and results Part 1. General and definitions (GB/T 6379.1-2004, ISO 5725-1.:1994, IDT) GB/T 6379.2 Accuracy of measurement methods and results (accuracy and precision) - Part 2. Determination of standard measurement methods the basic method of sex and reproducibility (GB/T 6379.2-2004, ISO 5725-2.:1994, IDT) GB/T 6379.3 Accuracy of measurement methods and results (accuracy and precision) - Part 3. Standard measurement method - Precision Intermediate metric (GB/T 6379.3-2012, ISO 5725-3.:1994, IDT) GB/T 12806 laboratory glass instrument single standard line capacity bottle (GB/T 12806-2011, ISO 1042.:1998, NEQ) GB/T 12808 Laboratory glass instruments Single line pipette (GB/T 12808-1991, eqvISO 648. 1977) GB/T 20066 Sampling and sample preparation methods for the determination of chemical constituents of steels and iron (GB/T 20066-2006, ISO 14284.

1996, IDT)

3 Principle

The sample was dissolved in a mixed acid of hydrochloric acid, nitric acid and hydrofluoric acid using a microwave digestion system or a hot plate. Dilute the sample solution through the peristaltic pump Inductively coupled plasma mass spectrometer. Simultaneous measurement of the mass spectra of the elements with an inductively coupled plasma mass spectrometer.

The calibration blanks and calibration solutions match the main elements in the steel with the wet decomposition of the matrix. Use internal standard compensation instrument drift.

4 Reagent

Unless otherwise stated, only high purity reagents or equivalent purity of less than 0.0001% of each of the elements to be tested are used in the analysis Reagents.

4.1

high purity iron, the measured element mass fraction is less than 0.0001%.

4.2

ultra-pure water, produced by the water purification system, the resistivity of not less than 18Mohm/cm.

4.3

hydrochloric acid, rho about 1.19g/mL.