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NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

GB/T 223.89-2019

Iron, steel and alloy - Determination of tellurium content - Hydride generation-atomic fluorescence spectrometric method

钢铁及合金 碲含量的测定 氢化物发生-原子荧光光谱法

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1 Scope

GB/T 223.89-2019 is the Chinese national standard on iron, steel and alloy - determination of tellurium content - hydride generation-atomic fluorescence spectrometric method, in the field of metallurgy. The /T suffix marks it as a recommended standard: it is not compulsory by itself, but it becomes binding as soon as a contract, a tender or a customer specification calls it up - which in practice is how most foreign buyers meet it. It was issued on 30 August 2019 by the State Administration for Market Regulation; Standardization Administration of China, and has been in force since 1 July 2020. Classification: ICS 77.080.01, CCS H11. This page is published from the official record of the standard held by the Chinese standards administration: the identification, the dates, the classification and the issuing body are taken from there. The clause text, the tables and the numeric limits are in the document itself, which is delivered complete in English translation.

This Part of GB/T 223 specifies a method for the determination of tellurium content by hydride generation-atomic fluorescence spectrometric method. This Part applies to the determination of tellurium content in iron, steel and alloy. The determination range is 0.00001 % ~ 0.005 % of mass fraction.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

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 6682 Water for analytical laboratory use - Specification and test methods

GB/T 20066 Steel and iron - Sampling and preparation of samples for the determination of chemical composition WEIGH 10 g of potassium borohydride; DISSOLVE in potassium hydroxide solution (5 g/L) and DILUTE to 500 mL. Prepare before use.

4.12 Tellurium standard solution

4.12.1 Tellurium standard stock solution, 100 μg/mL WEIGH 0.1000 g of pure tellurium (the mass fraction of tellurium is not less than 99.99 %); PLACE in a 150 mL beaker; ADD 10 mL of nitric acid (see 4.6); COVER the surface dish; HEAT to complete dissolution at low temperature; TRANSFER to a 1000 mL volumetric flask; DILUTE to the mark with water; MIX well. 1 mL of this solution contains 100 μg of tellurium.

4.12.2 Tellurium standard solution A, 10.0 μg/mL PIPETTE

10.0 mL of tellurium standard stock solution (see 4.12.1); PLACE in a 100 mL volumetric flask; DILUTE to the mark with hydrochloric acid (1+9); MIX well. 1 mL of this solution contains 10.0 μg of tellurium.

4.12.3 Tellurium standard solution B, 1.0 μg/mL PIPETTE

10.0 mL of tellurium standard solution A (see 4.12.2); PLACE in a 100 mL volumetric flask; DILUTE to the mark with hydrochloric acid (1+9); MIX well. 1 mL of this solution contains 1.0 μg of tellurium.

4.12.4 Tellurium standard solution C, 0.10 μg/mL, prepare before use PIPETTE

10.0 mL of tellurium standard solution B (see 4.12.3); PLACE in a 100 mL volumetric flask; DILUTE to the mark with hydrochloric acid (1+9); MIX well. 1 mL of this solution contains 0.10 μg of tellurium.

5 Instruments

Non-dispersive atomic fluorescence spectrometer, fitted with hydride generator and special tellurium hollow cathode lamp. After optimization, the non-dispersive atomic fluorescence spectrometer used shall achieve the following indicators:

a) Short-term precision: Measure the fluorescence intensity of the tellurium 5 %, precipitate will occur at the bottom of the beaker. The precipitate may be crushed with a glass rod and then ammonium fluoride solution (see 4.9) is added. Table 2 -- Acid used to dissolve different types of samples Sample type Amount of hydrochloric acid and nitric acid used Medium and low alloy steels of which the mass fraction of pure iron, pure nickel and chromium is less than 5 % Add 5 mL of nitric acid (see 4.6) to decompose the sample, and then add

7.5 mL of hydrochloric acid (see 4.3) Stainless steel 10 mL of hydrochloric acid, nitric acid mixed acid (see 4.7) Superalloy 10 mL of hydrochloric acid, nitric acid mixed acid (see 4.8)

7.3.2 Treatment of test solution

7.3.2.1 Mass fraction of tellurium is not greater than 0.0005 % TRANSFER the test solution (see 7.3.1) to a 50 mL volumetric flask; ADD 15 mL of hydrochloric acid (see 4.3) and 15 mL of citric acid solution (see 4.10); COOL to room temperature; DILUTE to the mark with water; MIX well; PLACE for 40 min for determination.

7.3.2.2 Mass fraction of tellurium is greater than 0.0005 % TRANSFER the test solution (see 7.3.1) to a 50 mL volumetric flask; DILUTE to the mark with water; MIX well. PIPETTE

5.0 mL of test solution; PLACE in a 50 mL volumetric flask; ADD 24 mL of hydrochloric acid (see 4.3),

2.5 mL of ammonium fluoride solution (see 4.9), 15 mL of citric acid solution (see 4.10); COOL to room temperature; DILUTE with water to the mark; MIX well; PLACE for 40 min for determination.

7.3.3 Measurement of atomic fluorescence intensity START UP; SET the lamp current and negative high voltage; PREHEAT at least 20 min before measurement; OPTIMIZE the instrument according to the instrument specifications. PREPARE potassium borohydride solution (see 4.12) and test solution (see

7.3.2.1 or see 7.3.2.2); USE hydrochloric acid (see 4.4) as the carrier; MEASURE the atomic fluorescence intensity of tellurium in the test solution according to the set procedure. The atomic fluorescence intensity of tellurium in the test solution minus the atomic fluorescence intensity of tellurium in the blank solution of the sample is the net atomic fluorescence intensity.

7.4 Plotting of calibration curve

7.4.1 Mass fraction of tellurium is not greater than 0.0005 % micrograms per milliliter (μg/mL); f - the diluting factor; V - the volume of the test solution to be tested, in milliliters (mL); m - the value of the sample mass, in grams (g). The analysis results retain 2 significant figures.

9 Precision

The precision of this Part is determined by a joint test of 9 levels of tellurium in 11 laboratories. Each level is tested twice in each laboratory under the repeatability conditions specified in GB/T 6379.1. The samples used and the precision test results are listed in Annex A. The test results reported by each laboratory are statistically analyzed according to GB/T 6379.2. The correlation between the obtained tellurium content and the repeatability limit r and the reproducibility limit R is shown in Table 5. Table 5 -- Repeatability and reproducibility of tellurium Element Range/% Repeatability limit r Reproducibility limit R Tellurium 0.00001 ~

0.005 m in the formulas is the average value of the two measured values, expressed in % (mass fraction). The repeatability limit r and the reproducibility limit R are obtained according to the formulas given in Table 5. Under repeatability conditions, the absolute difference between the two independent test results is not greater than the repeatability limit r, on the premise that the repeatability limit r is greater than 5 %. Under reproducibility conditions, the absolute difference between the two independent test results is not greater than the reproducibility limit R, on the premise that the reproducibility limit R is greater than 5 %.

10 Test report

The test report shall include the following: