

ICS 77.080.01

CCS H11



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

GB/T 223.72-2008

Replacing GB/T 223.72-1991

**Iron, steel and alloy - Determination of sulfur content -
Gravimetric method**

钢铁及合金 硫含量的测定 重量法

Issued on: May 13, 2008

Implemented on: November 1, 2008

**Issued by: General Administration of Quality Supervision, Inspection and
Quarantine of the People's Republic of China;
Standardization Administration of China**

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Foreword

In this revision, this part of GB/T 223 is changed to "Iron steel and alloy - Determination of sulfur content - Gravimetric method" and has two analysis methods. --Method 1. gravimetric method; --Method 2. aluminum oxide chromatographic separation - barium sulfate gravimetric method. Method 1 in this Part is identical to ISO 4934. 2003(E). To be convenient for use, the following editing revisions are made in this part. --"This international standard" is changed into "this Part"; --The comma "," acting as decimal point was replaced by the decimal point "."; --The foreword of the international standard is canceled; --In "normative reference", the international documents or specific chapter/article are consistent with domestic documents and replaced with the domestic documents. This part replaces GB/T 333.72-1991 "Methods for chemical analysis of iron, steel and alloy - The alumina chromatographic separation - barium sulfate gravimetric method for the determination of sulphur content". Compared with GB/T 223.72-1991. method 1 of this Part has the following revisions. --In sample dissolution, the conical flask containing test portion and agent is connected with a condenser, to control the dissolving speed of the sample better and avoid the sample dissolving too fast; a small amount of sulfur is oxidized to generate sulfate radical and escapes by the way of hydrogen sulfide. --For precipitant adding, firstly add an

approximate amount of BaCl₂ required depositing all -24SO, stand for 1h, and add about 25 mg of BaCl₂; control the S content in the test portion within 1.5~

5.5 mg and exceed precipitant BaCl₂ about (0.7~3) times; but in GB/T 223.72-1991. add 500 mg of BaCl₂ at a time and exceed precipitant BaCl₂ about (15 ~ 30) times. Compared with GB/T 223.72-1991. method 2 of this Part has the following revisions. --The description content for reagents and water in the analysis is added, and the expression method for solution concentration is revised; --The expression of quantity in result calculation formulae and quantity units in formulae is revised; --The explanation for precision function formula is specified. Appendix A and Appendix B of this part are informative. This Part was proposed by China Iron and Steel Association. This Part is under the jurisdiction of the National Technical Committee on Iron and Steel of Standardization Administration of China. Chief Drafting Organization. China Iron & Steel Research Institute Group Chief drafting staffs. Fan Xiaoyun, Ke Ruihua, Fan Chunqi All previously replaced editions hereof are as. --GB/T 223.72-1981. GB/T 223.73-1991.

1 Scope

GB/T 223.72-2008 is the Chinese national standard on iron, steel and alloy - determination of sulfur content - gravimetric 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 13 May 2008 by the General Administration of Quality Supervision, Inspection and Quarantine of the People's Republic of China; Standardization Administration of China, and has been in force since 1 November 2008. 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 gravimetric method for the determination of the sulfur content in steels and iron, excluding steels containing selenium. The method is particularly suitable as a reference method for the standardization of samples on which certified standard values are to be established. Method 1 is applicable to a sulfur content between 0.003 % (mass fraction) and 0.35 % (mass fraction). Method 2 is applicable to a sulfur content between 0.003 % (mass fraction) and 0.20 % (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 6379.1-2004, 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 20066 Steel and Iron - Sampling and Preparation of Samples for the Determination of Chemical Composition (GB/T 20066-2006, ISO 14284. 1996, IDT)

ISO 565 Test sieves - Metal wire cloth, perforated metal plate and electroformed sheet - Nominal sizes of openings

ISO 3696 Water for analytical laboratory use - Specification and test methods ISO 5725-

3 Accuracy (trueness and precision) of measurement methods and results - Part 3. Intermediate measures of the precision of a standard measurement method

3 Method 1. Gravimetric Method

3.1 Principle Dissolution of a test portion in dilute nitric acid in the presence of bromine, or in the mixed acid of nitric acid and hydrochloric acid in the presence of bromine (with the aid of an appropriate device to prevent sulfur losses). Addition of perchloric acid and evaporation of the solution until white fumes of perchloric acid are evolved. Filtration of the solution and removal of the dehydrates of silicon, tungsten, niobium, etc. Addition of a determined quantity of sulfate ions to aid precipitation. Chromatographic separation of the sulfate ions from the test solution by adsorption on an alumina column, and elution using an ammonium hydroxide solution. Precipitation of the sulfate ions as barium sulfate under controlled conditions and filtering, washing, heating and weighing.

3.2 Reagents and material During the analysis, use only reagents of recognized analytical grade and only grade 2 water as specified in ISO 3696.

3.2.1 Aluminium oxide (alumina, Al_2O_3), prepared for chromatography, of particle size corresponding to a sieve mesh size of 75 (μm) to 150 (μm) (supplementary series R 40/3 of

ISO 565) Alumina designated alkaline, neutral or acid may be used. Place approximately 200 g of the dry alumina in a 400 ml beaker containing 300 ml of water and place the beaker in a sink. Insert a glass tube having a bore of 5 mm, such that it extends to the bottom of the beaker and connect the tube to a water supply. Adjust the water flow so that the suspended fine material overflows the rim of the beaker. Continue this treatment until all the fine material, which does not settle within 1 min of stopping the water flow, is removed. Pour off the supernatant liquid from the coarser material, add the hydrochloric acid (3.2.5) in an amount sufficient to cover the alumina; stir and allow to stand for not less than 12 h. Pour off the hydrochloric acid and wash the alumina with water as described in the second

paragraph. For preparation of the column, make a slurry of the washed alumina and the hydrochloric acid solution (3.2.8).

3.2.4 Nitric acid (HNO₃), rho = approximately

1.40 g/ml, diluted 1 + 1

3.2.6 Hydrochloric acid (HCl), rho = approximately

1.19 g/ml, diluted 1 + 1

3.2.7 Hydrochloric acid (HCl), rho = approximately

1.19 g/ml, diluted 1 + 9

3.2.8 Hydrochloric acid (HCl), rho = approximately

1.19 g/ml, diluted 1 + 19

3.2.9 Perchloric acid (HClO₄), rho = approximately

Note. If this reagent is shown to have a high sulfate content, this may be removed by passing the reagent through the adsorption column (3.3.4).

3.2.10 Perchloric acid (HClO₄), rho = approximately

1.54 g/ml, diluted 1 + 49

3.2.11 Acid mixture Mix hydrochloric acid (4.5) and nitric acid (4.3) in proportions liable to ensure total solubility of the samples. This mixture should be freshly prepared as required.

Note. Generally, aqua regia can be used. For some samples, the proportion of hydrochloric acid and nitric acid can be adjusted.

3.2.12 Ammonium hydroxide (NH₄OH), rho = approximately

0.90 g/ml

3.2.13 Ammonium hydroxide (NH₄OH), rho = approximately

0.90 g/ml, dilut...