GB

NATIONAL STANDARD OF

THE PEOPLE'S REPUBLIC OF CHINA

GB/T 223.60-1997

Replacing GB 223.60-87

Methods for chemical analysis of iron, steel and alloy - The perchloric acid dehydration gravimetric method for the determination of silicon content

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Foreword

This Standard was revised based on GB/T 1.1-1993 “Directives for the work of standardization. Unit 1: Drafting and presentation of standards. Part 1: General rules for drafting standards”, GB 1.4-88 "Directives for the Work of Standardization-Rules for Drafting Chemical Analysis Standards" and GB 223.60-87 “Methods for chemical analysis of iron, steel and alloy - The perchloric acid dehydration gravimetric method for the determination of silicon content”.

According to the provisions of 4.2.3, 4.3.3 in Chapter 4 of GB/T 1.1-1993 and 6.10 of GB 1.4-88, "foreword", "Scope" in Chapter I, "Normative References" in Chapter Two, "sampling and sample preparation" in Chapter 6 and "test report" in Chapter 10.

The Appendix A and Appendix B of this Standard are informative.

This Standard replaces GB 223.60-87 when it comes into force.

This Standard was proposed by the Ministry of Metallurgical Industry of the People's Republic of China.

This Standard shall be under the jurisdiction of National Technical Committee on Iron and Steel of Standardization Administration of China.

The drafting organization of this Standard: Central Iron and Steel Research Institute, Ministry of Metallurgical Industry.

Main drafters of this Standard: Wang Yuxing and Cui Qiuhong.

This Standard was issued as GB 223.5 (1)-81 in 1981 for the first time, revised to GB 223.60-87 in 1987 and determined the method accuracy.

Methods for chemical analysis of iron, steel and alloy - The perchloric acid dehydration gravimetric method for the determination of silicon content

1 Scope

This Standard specifies the perchloric acid dehydration gravimetric method for the determination of silicon content in iron, steel, high temperature alloy and precious alloy.

This Standard is applicable to determination of 0.10%(m/m) ~ 6.00%(m/m) silicon content in iron, steel, high temperature alloy and precious alloy.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this Standard. As all the standards will be revised, each party using this Standard shall discuss the feasibility of using the latest edition of the following standards.

GB/T 222-84, *Method of Sampling Steel for Determination of Chemical Composition and Permissible Variations for Product Analysis*

GB 6379-86, *Precision of Test Methods: Determination of Repeatability and Reproducibility for a Standard Test Method by Interlaboratory Tests*

3 Method summary

Dissolve the specimen with hydrochloric acid and nitric acid dissolution, dehydrate silicic acid with perchloric acid smoking, filter and clean the silicic acid and then firing it into silicon dioxide. Handle it with sulfuric acid -hydrofluoric acid, make it produce silicon tetrafluoride which is volatilized and removed. Calculate the silicon contents according to the mass difference after the removal of silicon.

4 Reagents and materials

4.1 Hydrochloric acid (ρ1.19 g/mL).

7.3.2 Take it out and cool a while, moisten salt with 6 mL of hydrochloric acid, deoxidize them with sexavalent chrome, add 100 mL of hot water, stirring, and heat slightly to make the soluble salt dissolve. Add little pulp, filter immediately with medium-texture filter paper, wipe carefully out of the precipitation sticking on the wall of glass with policemen, clean the beaker with hot hydrochloric acid (4.2) and wash the precipitation until no ferric ion is available [check with ammonium thiocyanate solution (4.8)], and then wash with hot water for three times.

7.3.3 Shift filtered solution and washing liquid in the original beaker, heat and condense them until the perchloric acid smokes and then flow back for 15min ~ 25min. The following is carried out according to 7.3.2.

7.3.4 Place the precipitation got in 7.3.2 and 7.3.3 together with filter paper into platinum crucible, drying, ashing, cover part of crucible with the cover, firing in the high temperature furnace of 1000°C ~ 1050°C for 30min ~ 40min (firing time is determined according to the quantity of silicon dioxide and whether wolfram and molybdenum are available in the steel). Take it out and cool a while, place them in dryer, cool until room temperature, weigh them, firing repeatedly until constant quantity.

Add 4~5 drops of sulfuric acid (4.7) and 5 mL of hydrofluoric acid (4.5) along inner-wall of crucible, heat at low temperature until the sulfuric acid fume disappears, place platinum crucible into high temperature furnace of 1000°C ~1050°C for firing for 20 min, take it out and cool a while, place it into dryer, cool until room temperature, weigh, and firing repeatedly until the constant quantity.

Note:

1 For the samples containing niobium, tantalum, titanium and zirconium, firing them at the temperature of 1000°C ~ 1050°C,take them out and cool a while, add 1 mL ~ 1.5 mL of sulfuric acid (4.7), heat at low temperature until the sulfuric acid fume disappears and then firing at the temperature of 800°C for 10 min. Take them out again, place into dryer, cool until room temperature, weigh them, and firing repeatedly until the constant quantity. Add 1 mL of sulfuric acid (4.6) and 5 mL of hydrofluoric acid (4.5), heat at low temperature until the sulfuric acid fume disappears and then firing at the temperature of 800°C until constant quantity.

2 As for the samples containing high wolfram and molybdenum, in the process of firing precipitation, platinum crucible need to be taken out and precipitate them by mashing with platinum wire to speed volatilization of wolfram and molybdenum. hydrofluoric acid is fired at the temperature of 800°C until the constant quantity after the volatilization of silicon.

8 Analysis result and its expression

Silicon content expressed with mass percent is calculated according to

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