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

GB 3653.3-1983

Replacing GB/T 3653.3-1988

**Ferroboron - Determination of silicon content - Perchloric acid
dehydration-gravimetric method
(English Translation)**

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Foreword

This document complies with the provisions of GB/T 1.1-2020 "Standardization Work Guidelines Part 1: Structure and Drafting Rules of Standardization Documents" Drafting:

This document is Part 3 of GB/T 3653: GB/T 3653 has published the following parts:

---Chemical analysis method of ferroboron Determination of boron content by alkali titration method;

---Chemical analysis method of ferroboron Determination of carbon content by gas volumetric method;

---Determination of ferrosilicon content by perchloric acid dehydration gravimetric method;

---Determination of boron, iron and aluminum content by EDTA titration method;

---Chemical analysis method of ferroboron, chromatographic separation, barium sulfate gravimetric method, determination of sulfur content;

---Chemical analysis method of ferroboron Antimony phosphorus molybdenum blue photometry method to determine phosphorus content;

---Determination of boron, iron, and sulfur content by infrared absorption method:

This document replaces GB/T 3653.3-1988 "Methods for chemical analysis of ferric boron - Determination of silicon content by perchloric acid dehydration gravimetric method": and Compared with GB/T 3653.3-1988, in addition to structural adjustments and editorial changes, the main technical changes are as follows:

- a) Changed the content of the principle (see Chapter 4, Chapter 2 of the 1988 edition);
- b) Changed "Reagents and Materials" (see Chapter 5, Chapter 3 of the 1988 edition);

- c) Added "instruments and equipment" (see Chapter 6);
- d) Added "number of measurements" (see 8.1);
- e) The sample fusion method was changed from "acid fusion first and then alkali fusion" to "direct alkali fusion" (see 8.4:1, 5:3:1 of the 1988 edition);
- f) Changed the amount of perchloric acid added, changing "add 30mL perchloric acid" to "add 40mL perchloric acid" (see 8.4:2, 1988 version 5:3:2);
- g) Changed the precipitation ignition temperature, changing "1000°C" to "1050°C" (see 8.4:6, 5:3:6 of the 1988 edition);
- h) Changed "Calculation and Expression of Analysis Results" (see Chapter 9, Chapter 6 of the 1988 edition);
- i) Changed "permissible difference" to "precision" (see Chapter 10, Chapter 7 of the 1988 edition);
- j) Added test report (see Chapter 11);
- k) Added "Flowchart of Sample Analysis Results Acceptance Procedure" (see Appendix A):

Please note that some content in this document may be subject to patents: The publisher of this document assumes no responsibility for identifying patents:

Introduction

Since there are many detection elements involved in the ferroboron detection process, the applicable scope and applicable methods of the elements are different: To ensure boron the iron detection standards are convenient and accurate, and a national standard system has been established to support the detection of ferroboron based on the analysis methods of different elements of ferroboron:

The GB/T 3653 ferroboron series analysis method is the basic standard for ferroboron detection in my country and consists of the following seven parts:

---Chemical analysis method of ferroboron Determination of boron content by alkali titration method: The purpose is to measure the boron content in ferroboron, using alkali titration:

---Chemical analysis method of ferroboron: Determination of carbon content by gas volumetric method: The purpose is to measure the carbon content in ferroboron, using the gas volumetric method:

---Determination of ferrosilicon content by perchloric acid dehydration gravimetric method: The purpose is to measure the silicon content in ferroboron and use perchloric acid to dehydrate it:

gravimetric method:

---Determination of boron, iron and aluminum content by EDTA titration: The purpose is to measure the aluminum content in ferroboron, using the EDTA titration method:

---Chemical analysis method of ferroboron Chromatographic separation Barium sulfate gravimetric method Determination of sulfur content: The purpose is to measure the sulfur content in ferroboron, using Chromatographic separation with barium sulfate gravimetric method:

---Chemical analysis method of ferroboron: Determination of phosphorus content by antimony phosphorus molybdenum blue photometry: The purpose is to measure the phosphorus content in ferroboron, using antimony phosphorus molybdenum Blue photometry:

---Determination of boron, iron, and sulfur content by infrared absorption method: The purpose is to measure the sulfur content in ferroboron, using infrared absorption method:

Determination of ferrosilicon content Perchloric acid dehydration gravimetric method
Warning---Personnel using this document should have regular laboratory work experience:
This document does not address all possible security issues question: Users are responsible for taking appropriate safety and health measures and ensuring compliance with the conditions stipulated in relevant national regulations:

1 Scope

This document describes a method for the determination of the silicon content in ferroborates using the perchloric acid dehydration gravimetric method:

This document is applicable to the determination of silicon content in ferroboron: The determination range (mass fraction) is: 0.50%~16.00%:

2 Normative reference documents

The contents of the following documents constitute essential provisions of this document through normative references in the text: Dated citations files, only the version corresponding to that date applies to this document: For undated referenced documents, the latest version (including all amendments) applies to this document:

GB/T 4010 Collection and preparation of samples for chemical analysis of iron alloys GB/T 6379.1 Accuracy of measurement methods and results (correctness and precision) Part 1: General principles and definitions GB/T 6379.2 Accuracy of measurement methods and results (correctness and precision) Part 2: Determination of repeatability of standard measurement methods Basic methods of sex and reproducibility GB/T 6682 Specifications and test methods for water used in analytical laboratories GB/T 8170 Numerical rounding rules and expression and determination of limit values

3 Terms and definitions

There are no terms or definitions to be defined in this document:

4 Principles

The sample is melted with sodium carbonate and sodium peroxide to convert the silicon into silicate, acidified with hydrochloric acid, and dehydrated with perchloric acid for secondary smoking:

Filter and wash, burn the precipitate at 1050°C and then cool it: Use methanol to volatilize the boron at low temperature, burn to a constant amount, and use hydrofluoric acid to tetrafluoride the silicon:

The silicon is volatilized and removed, and then burned to a constant amount: The silicon content is calculated based on the mass difference before and after hydrofluoric acid treatment:

5 Reagents and materials

Unless otherwise stated, only approved analytical grade reagents and grade three or above evaporators that comply with GB/T 6682 are used in the analysis:

Distilled or deionized water or water of equivalent purity:

5.1

Anhydrous sodium carbonate, solid:

5.2

Sodium peroxide, solid:

5.3

Mix flux, 2 parts sodium peroxide and 1 part anhydrous sodium carbonate, mix well: