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GB/T 2286-2008

Replacing GB/T 2286-1991

Coke - Determination of total sulfur

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

Standardization Administration of the People's Republic of China.

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Foreword

Consistency BETWEEN this Standard AND ISO 344:1992(E) “Consistency of this Standard and ISO 344:1992(E) and ISO 351:1996(E) “Solid Mineral Fuels - Determination of Total Sulphur - Eschka Method" and ISO 351:1996(E) “Solid Mineral Fuels - Determination of Total Sulphur - High-Temperature Combustion Method” is non-equivalent. This Standard replaces GB/T 2286-1991 “Coke - Determination of total Sulphur”.

Compared with GB/T 2286-1991, main changes of this Standard are as follows:

— Revise the writing format, terms and symbols;

— Change English name of the standard from “Coke - Determination of Total Sulphur” to “Coke - Determination of Total Sulfur”;

— Add the Clause “Foreword”;

— Do not divide the standard into chapters;

— Add the “test report” content;

— Do not list the “test preparation” as a separate chapter; move the “test preparation” content to “test procedures”;

— Add the blank test content according to the provisions of ISO 351:1996(E);

— Annex A Standard solution preparation and calibration are revised and improved according to GB/T 601-2002.

Annex A to this Standard is normative.

This Standard was proposed by China Iron and Steel Industry Association.

This Standard shall be under the jurisdiction of National Steel Standardization Technical Committee.

Main drafting organizations of this Standard: Sinosteel Anshan Research Institute of Thermo-energy Co., Ltd. AND China Metallurgical Information And Standardization Institute.

Main drafters of this Standard: Yang Jinxia, Wang Wei, Yu Yinping, Wang Xiong, Guo Faqing and Sun Wei.

This Standard was released for the first time in 1980 and revised for the first time in 1991.

Coke - Determination of Total Sulfur

1 Scope

This Standard specifies the determination principle of total sulfur, reagents and materials, instruments and equipment, sampling, test procedures, calculation of results and degree of precision.

This Standard applies to determination of total sulfur. Method I is Eschka method; method II is high-temperature combustion method. Eschka method shall be adopted in arbitration analysis.

2 Normative references

The articles contained in the following documents have become part of this document when they are quoted herein. For the dated documents so quoted, all subsequent modifications (including all corrections) or revisions made thereafter do not apply to this standard. However, the parties who reach an agreement according to this standard are encouraged to study whether the latest versions of these documents may be used. For the undated documents so quoted, the latest versions (including all modification sheets) apply to this document.

GB/T 601 Chemical reagent - Preparations of standard volumetric solutions

GB/T 1997 Coke - Sampling and preparation of samples

3 Method I (Eschka method)

3.1 Principle

Mix sample with Aldrin sufficiently; burn at a certain temperature; make sulfur in coke turn into sulfate; then make sulfate ion generate barium sulfate precipitation; calculate total sulfur content in sample according to the mass of barium sulfate.

3.2 Reagents and materials

Warning — Be careful when handling reagents of which many are toxic and corrosive.

Unless otherwise specified, only use the analytically-pure reagents AND distilled water or deionized water or water with equivalent purity.

3.2.1 Barium chloride.

3.2.2 MgO: Chemically pure.

3.2.3 Anhydrous sodium carbonate: Chemically pure.

3.2.4 Silver nitrate.

3.2.5 Aldrin: Weigh 2 mass-portions of magnesium oxide and 1 mass-portion of anhydrous sodium carbonate; grind to particles of which the size is less than 0.2mm; mix well and store in airtight container.

3.2.6 Hydrogen peroxide: Concentration of 30%.

3.2.7 Hydrochloric acid solution: Density of 1.19g/cm^3.

3.2.8 Nitrate: Density of 1.42 g/cm^3.

3.2.9 Barium chloride solution (100 g/L): Weigh 100 g of barium chloride; dissolve in water and dilute to 1 000 mL with water.

3.2.10 Hydrochloric acid solution: (1 + 1).

3.2.11 Silver nitrate solution (10 g/L): Weight 1 g of silver nitrate; dissolve in water and dilute to 100 mL with water; add a few drops of nitric acid; store in a dark bottle.

3.2.12 Methyl red indicator solution (1 g/L): Weight 0.1 g of methyl red; dissolve in 50 mL of ethanol; dilute to 100 mL with water.

3.2.13 Qualitative filter paper: Intermediate speed, *Φ*90 mm~*Φ*110 mm.

3.2.14 Quantitative filter paper: Intermediate speed, *Φ*90 mm~*Φ*110 mm.

3.3 Instruments and equipment

3.3.1 Analytical balance: Sensitivity is 0.000 1 g.

3.3.2 Counter balance: Sensitivity is 0.01 g.

3.3.3 Muffle furnace: With temperature measurement and temperature control devices that can maintain at temperature 800°C to 850 °C and accompanied by thermocouple and pyrometer. The position of small hole in rear wall of the furnace shall enable temperature measuring point of thermocouple to be in the middle of constant-temperature area and 20mm to 30 mm away from furnace bottom; there is a chimney for gas exhaust in the rear.

3.3.4 Dryer: With allochroic silicagel or granular anhydrous calcium chloride inside.

3.3.5 Beaker: 400 mL.

Note: Dilute with sulfuric acid solution [$c(1/2H_2SO_4)$ = 0.1 mol/L] prepared based on the Annex before using sulfuric acid solution.

4.2.8 Preparation of methyl red-methylene blue mixed indicator:

4.2.8.1 Mix methylene blue ethanol solution (1 g/L) with methyl red ethanol solution (1 g/L) based on a 1+2 volume ratio.

4.2.8.2 Weigh 0.125 g of methyl re; dissolve in 50 mL of ethanol; dilute to 100 mL with water; weigh 0.083 g of methylene blue; dissolve in 50 mL of ethanol; dilute to 100 mL with water; respectively store in brown bottles. Mix based on a 1 + 1 volume ratio before use; store in brown dropping bottle.

Note: Above two types of indicator solutions are both optional; validity period of indicator solution after mixing is 7 days.

4.3 Instruments and equipment

4.3.1 High-temperature tube furnace: Heat through silicon carbide rod or silicon carbon tube, with temperature regulating device, which can maintain furnace temperature at 1 250°C±10°C.

4.3.2 Combustion tube: Made of high-temperature porcelain, corundum or quartz. Total tube length is about 750 mm; one end has an outer diameter of 22 mm, an inner diameter of 19mm and a length of 690 mm; the other end has an outer diameter of 10 mm, an inner diameter of 7 mm and a length of 60 mm.

4.3.3 Combustion boat: Made of high-temperature porcelain or corundum, length of 77 mm, upper length of 12 mm, lower length of 9 mm and height of 8 mm.

4.3.4 Absorption bottle: Erlenmeyer flask, with a volume of 250mL.

4.3.5 Nickel-chromium wire hook: Diameter of about 2 mm, length of 650 mm, one end bent into a small hook.

4.3.6 Silicone rubber tube: Outer diameter of 11 mm, inner diameter of 8 mm, length of about 80 mm.

4.4 Sample preparation

Conduct according to the provisions of GB/T 1997.

4.5 Test procedures

4.5.1 Test preparation

4.5.1.1 Connect the instruments for standby application based on the sequence as shown in Figure 1.

Where,

M_{ad} — Moisture content of analytical sample, mass fraction (%).

4.7 Degree of precision

Repeatability: Not greater than 0.05%.

Reproducibility: Not greater than 0.1%.

4.8 Test report

Test report shall contain following information:

a) Test sample:

b) Basis standard:

c) Application method;

d) Calculation of results;

e) Deviation from standard;

f) Anomalies observed in test;

g) Test date.

Annex A

(Normative)

Standard Solution Preparation and Calibration

A.1 Sodium hydroxide standard solution (4.2.6) [c(NaOH)=0.1 mol/L]

A.1.1 Preparation

Weigh 110 g of sodium hydroxide; dissolve in 100 mL of water and shake well; inject into polyethylene container; keep airtight until the solution becomes clear. Use plastic pipe to siphon 5.4 mL of supernatant; inject into 1 000 mL of carbon dioxide-free water and shake well.

A.1.2 Calibration

Weigh 0.75 g of working standard reagent potassium hydrogen phthalate that has been dried in electric stove at 105°C~110°C to a constant weight; dissolve in 50 mL of carbon dioxide-free water; add 2 drops of phenolphthalein ((10 g/L) indicator solution; use prepared sodium hydroxide solution for titration of solution to pink; and keep for 30 s; make blank test simultaneously.

Concentration value of sodium hydroxide standard titration solution [c(NaOH)] is in moles per liter ((mol/L), calculated according to Formula (A.1):

$$c(\mathrm{NaOH}) = \frac{m}{(V_1 - V_2) \times 0.204\ 2} \qquad \text{(A.1)}$$

Where,

m — Exact value of potassium hydrogen phthalate mass, in grams (g);

V_1 — Value of sodium hydroxide solution volume, in millimeters (mL);

V_2 — Value of blank sodium hydroxide solution for test, in milliliters (mL);

0.204 2 — Value of mmol mass of potassium hydrogen phthalate, in grams per mmol (g/mmol).

A.2 Sulfuric acid standard solution (4.2.7)[c(1/2H_2SO_4) =0.1 mol/L]

A.2.1 Preparation

Measure 3 mL of sulfuric acid; slowly inject into 1 000 mL of water; cool and shake well.

Note: Preparation of bromocresol green-methyl red indicator solution.

Solution I: Weigh 0.1 g of bromocresol green; dissolve in ethanol and dilute to 100 mL with ethanol (95%).

Solution II: Weigh 0.28 of methyl red; dissolve in ethanol (95%) and dilute to 100 mL with ethanol (95%).

Take 30mL of solution I and 10mL of solution II; mix well.

A.2.2 Calibration

Weight 0.2 g of working standard reagent anhydrous sodium carbonate that has been ignited to constant weight in 270 °C~300°C high temperature furnace. Dissolve in 50 mL of water; add 10 drops of bromocresol green-methyl red indicator solution; use prepared sulfuric acid solution for titration of solution from green to dark red; boil for 2 min; continue the titration of solution to dark red again. Make a blank test simultaneously.

Concentration value of sulfuric acid standard titration solution [$c(1/2H_2SO_4)$] is in moles per liter ((mol/L), calculated according to Formula (A.2):

$$c(1/2H_2SO_4) = \frac{m}{(V_1 - V_2) \times 0.052\ 99} \quad \text{(A.2)}$$

Where,

m — Exact value of anhydrous sodium carbonate mass, in grams (g);

V_1 — Value of sulfuric acid solution volume, in millimeters (mL);

V_2 — Value of blank sulfuric acid solution volume for test, in milliliters (mL);

0.052 99 — Value of mmol mass of anhydrous sodium carbonate, in grams per mmol (g/mmol).

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