

Translated English of Chinese Standard: GB/T2286-2017

www.ChineseStandard.net → Buy True-PDF → Auto-delivery.

Sales@ChineseStandard.net

GB

NATIONAL STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA

ICS 75.160.10

H 32

GB/T 2286-2017

Replacing GB/T 2286-2008

Determination of total sulfur composition of coke

焦炭全硫含量的测定方法

Issued on: October 14, 2017

Implemented on: May 01, 2018

**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.

Table of Contents

Foreword.....	3
1 Scope	4
2 Normative references	4
3 Eschka method (arbitration method)	4
4 High temperature combustion method.....	8
5 Coulometric titration method	12
6 Infrared spectroscopy method	16
7 Result expression.....	19
8 Test report.....	19

Determination of total sulfur composition of coke

WARNING -- The use of this Standard involves certain hazardous materials, operations and equipment, but does not make recommendations on all safety issues related thereto. Users are responsible for taking appropriate safety and protective measures before using this standard, and ensuring compliance with the conditions stipulated in relevant national regulations.

1 Scope

This Standard specifies the method principle, reagents and materials, instruments and equipment, specimen collection, test steps, result calculation and precision for the determination of total sulfur composition in coke by Eschka method, high temperature combustion method, coulometric titration and infrared spectroscopy.

This Standard applies to coke and semi-coke (blue charcoal).

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 601, *Chemical reagent -- Preparations of reference titration solutions*

GB/T 1997, *Coke -- Sampling and preparation of samples*

GB/T 2001, *Coke -- Determination of proximate analysis*

GB/T 8170, *Rules of rounding off for numerical values and expression and judgement of limiting values*

3 Eschka method (arbitration method)

3.1 Principle

Thoroughly mix the specimen with Eschka reagent. Burn at a certain temperature to convert the sulfur in the specimen into sulfate. The sulfate ions are then allowed to form barium sulfate precipitates. Calculate the total sulfur composition in the specimen based on the mass of barium sulfate.

3.2 Reagents and materials

Unless otherwise stated, only reagents confirmed to be analytically pure and distilled or deionized water or water of equivalent purity are used in the analysis.

3.2.1 Magnesium oxide: chemically pure.

3.2.2 Anhydrous sodium carbonate: chemically pure.

3.2.3 Eschka reagent: Weigh 2 portions by mass of magnesium oxide and 1 portion by mass of anhydrous sodium carbonate. Grind until the particle size is less than 0.2 mm. Mix evenly and store in an airtight container.

3.2.4 Hydrogen peroxide: The concentration is 30%.

3.2.5 Hydrochloric acid solution: The density is 1.19 g/cm³.

3.2.6 Nitric acid: The density is 1.42 g/cm³.

3.2.7 Barium chloride solution (100 g/L): Weigh 100 g of barium chloride and dissolve in water. Dilute to 1000 mL with water.

3.2.8 Hydrochloric acid solution (1+1): 1 volume of hydrochloric acid plus 1 volume of water and mix well.

3.2.9 Silver nitrate solution (10 g/L): Weigh 1 g of silver nitrate and dissolve it in water. Dilute to 100 mL with water. Add a few drops of nitric acid, and store in a dark bottle.

3.2.10 Methyl red indicator solution (1 g/L): Weigh 0.1 g of methyl red and dissolve it in 50 mL of ethanol. Dilute to 100 mL with water.

3.2.11 Qualitative filter paper: medium speed; ϕ 90 mm~ ϕ 110 mm.

3.2.12 Quantitative filter paper: medium speed; ϕ 90 mm~ ϕ 110 mm.

3.3 Instruments and equipment

3.3.1 Analytical balance: sensitive to 0.0001 g.

3.3.2 Pallet balance: sensitive to 0.01 g.

3.3.3 Muffle furnace: equipped with temperature control device; can maintain the temperature between 800°C~850°C; the temperature can be adjusted; the furnace can be ventilated.

3.3.4 Desiccator: with built-in color-changing silica gel or granular anhydrous calcium chloride.

3.3.5 Beaker.

3.3.6 Porcelain crucibles: 30 mL and 20 mL.

3.4 Specimen preparation

Carry out in accordance with the provisions of GB/T 1997.

3.5 Test steps

3.5.1 Weigh 2 g (accurate to 0.1 g) of Eschka reagent (see 3.2.3) and 1 g (accurate to 0.0001 g) of the specimen with a particle size less than 0.2 mm in a 30 mL porcelain crucible (see 3.3.6). Use nichrome wire to mix evenly. Then cover the specimen with 1 g (accurate to 0.1 g) of Eschka reagent (see 3.2.3).

3.5.2 Move the crucible containing the specimen into the muffle furnace (see 3.3.3). Gradually increase the furnace temperature to 800°C~850°C within 1 h~1.5 h. Heat at this temperature for 1 h~1.5 h.

3.5.3 Remove the crucible from the muffle furnace. After cooling to room temperature, use a glass rod to loosen the burning material (if any unburned specimen particles are found, continue burning at 800°C~850°C for 0.5 h). Transfer it to a beaker. Carefully rinse the inside of the crucible with hot water. Add the rinse solution to the beaker. Then add 100 mL~150 mL of hot water. Crush the burning object with a glass rod (if unburned specimen particles are found at this time, the test will be invalid).

3.5.4 Add 1 mL of hydrogen peroxide (see 3.2.4) into the beaker. Heat it to 80°C and keep it for 30 min.

3.5.5 Filter with qualitative filter paper (see 3.2.11). Rinse the burn onto the filter paper with hot distilled water. Continue to rinse the burned material from the filter paper carefully with hot distilled water. The number of times shall not be less than 10 times.

3.5.6 Boil the filtrate for 2~3 min. Drain excess hydrogen peroxide. Add 2~3 drops of methyl red indicator solution (see 3.2.10) to the filtrate to indicate whether the elimination is complete. Add hydrochloric acid solution (see 3.2.8) dropwise until the color turns red. Add another 1 mL. Boil for 5 min to remove carbon dioxide. The volume of the solution at this time is approximately 200 mL.

3.5.7 Cover the beaker with a watch glass. Reduce heat until solution stops boiling. Remove the watch glass. Slowly drop 10 mL of barium chloride solution (see 3.2.7) into the hot solution. Stir the solution simultaneously. Cover the watch glass. Keep the solution at a temperature just below the boiling point for 30 min.

3.5.8 After the solution is cooled, filter it with quantitative filter paper (see 3.2.12). Wash with hot water until there is no chloride ion [use silver nitrate solution (see 3.2.9) to check that there is no turbidity].

3.5.9 Move the precipitate together with the filter paper into a 20 mL porcelain crucible of known mass (see 3.3.6). Ash the filter paper at low temperature first. Then move it into a muffle furnace with a temperature of 800°C~850°C and burn for 20 min. Take

NOTE: You can choose any one of the above two indicator solutions. The mixed indicator solution is valid for 7 days.

4.3 Instruments and equipment

4.3.1 High-temperature tube furnace: heated with silicon carbon rods or silicon carbon tubes, with a temperature control device to maintain the furnace temperature within the range of $(1250\pm 10)^{\circ}\text{C}$.

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

4.3.3 Combustion boat: made of high-temperature porcelain or corundum; the length is 77 mm, the top width is 12 mm, the bottom width is 9 mm, and the height is 8 mm.

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

4.3.5 Nickel-chromium wire hook: the diameter is about 2 mm; the length is 650 mm; one end is bent into a small hook.

4.3.6 Silicone rubber tube: the outer diameter 11 mm; the inner diameter 8 mm; the length is approximately 80 mm.

4.3.7 Acid burettes: 25 mL and 10 mL.

4.3.8 Alkali burettes: 25 mL and 10 mL.

4.3.9 Ear wash ball.

4.3.10 Measuring cylinder.

4.3.11 Analytical balance: sensitive to 0.0001 g.

4.4 Specimen preparation

Carry out in accordance with the provisions of GB/T 1997.

4.5 Test steps

4.5.1 Test preparation

4.5.1.1 Connect the instruments according to the sequence in Figure 1 for later use.

5.2.3 Combustion boat: bisque or corundum product; the length of the sample loading part is about 60 mm; the temperature resistance is above 1200°C.

5.3 Instruments and equipment

5.3.1 Coulomb sulfur meter: According to the principle of coulomb analysis, the titration process is detected and controlled through double platinum indicator electrodes. Use a microcomputer to control the temperature of the combustion furnace and the test process in real time and perform data processing. Automatically calculate and display the milligrams or mass fraction of sulfur in the specimen. Coulomb sulfur meter mainly consists of the following parts.

5.3.1.1 Combustion furnace: equipped with a temperature measurement and control device, capable of raising the temperature to above 1200°C, and a high-temperature constant temperature zone of at least 70 mm long. The furnace is equipped with a combustion tube with a different diameter that can withstand a temperature of above 1300°C.

5.3.1.2 Sample delivery controller: the specimen can be automatically sent into the combustion tube; the specimen injection speed and stroke can be freely adjusted; the specimen can be flexibly moved forward and backward according to prescribed procedures.

5.3.1.3 Electrolytic cell: the electrolytic cell is divided into two parts: the upper cover and the cell body. The upper cover of the electrolytic cell is fixed with an electrolytic electrode pair and an indicating electrode pair, with a capacity of not less than 400 mL.

5.3.1.4 Electromagnetic stirrer: the electromagnetic stirrer drives the magnetic stirrer in the electrolytic cell to rotate through the stirrer to stir the electrolyte. The stirring rate can be adjusted through the stirring adjustment knob on the control panel.

5.3.1.5 Air supply and purification device: composed of electromagnetic pump and purification pipe. The gas supply volume is about 1500 mL/min; the gas extraction volume is about 1000 mL/min; the purification tube is filled with color-changing silica gel.

5.3.2 Analytical balance: sensitive to 0.0001 g.

5.4 Test steps

5.4.1 Test preparation

5.4.1.1 Turn on and debug the equipment according to the instrument instruction manual. Put it in a normal test state. Raise the temperature of the combustion furnace to 1150°C.

5.4.1.2 Fill the outlet of the combustion tube with clean and dry glass fiber wool.

5.4.1.3 Start the air extraction and supply pumps. Adjust the suction flow to 1000 mL/min. Then close the piston between the electrolytic cell and the combustion tube. If the air pumping volume can be reduced to less than 300 mL/min, it proves that the air tightness of each component and interface of the instrument is good and the measurement can be carried out. Otherwise, check the various components of the instrument and their interfaces. Recheck air tightness.

5.4.2 Instrument calibration

5.4.2.1 Calibration method

Use certified standard samples and perform instrument calibration according to one of the following methods.

5.4.2.1.1 Multi-point calibration method: Calibrate with at least 3 certified standard samples whose sulfur content can cover the sulfur content range of the sample being tested.

5.4.2.1.2 Single-point calibration method: Use a standard sample with a sulfur content similar to that of the sample to be measured for calibration.

5.4.2.2 Calibration procedure

5.4.2.2.1 Determine the air-dry basis moisture of the standard sample according to GB/T 2001. Calculate the standard value of total sulfur $S_{t,ad}$ on an air-dry basis.

5.4.2.2.2 According to the steps in 5.4.3, use the calibrated instrument to measure the sulfur content of the standard sample. Each standard sample is measured at least three times. The average value of three measured values is the measured sulfur value of the standard sample.

5.4.2.2.3 Input the sulfur measured value of the standard sample and the air-dry basis standard value into the sulfur meter (or the instrument automatically reads it) to generate a correction coefficient.

NOTE: Some instruments may require manual calculation of correction coefficients before entering them into the instrument.

5.4.2.3 Calibration validity verification

Select another 1 ~ 2 standard samples or other control samples. Use a calibrated sulfur meter to measure the total sulfur content according to step 5.4.3. If the difference between the measured value and the standard value (control value) is within the uncertainty range (control limit) of the standard value (control value), the calibration is valid. Otherwise, the cause shall be identified and recalibrated.

5.4.3 Determination steps

Use certified standard samples to calibrate the sulfur meter according to one of the following methods.

6.4.2.1.1 Multi-point calibration method: Calibrate with at least 3 certified standard samples whose sulfur content can cover the sulfur content range of the sample being tested.

6.4.2.1.2 Single-point calibration method: Use a standard sample with a sulfur content similar to that of the sample to be measured for calibration.

6.4.2.2 Calibration procedure

6.4.2.2.1 Determine the air-dry basis moisture of the standard sample according to GB/T 2001. Calculate the standard value of total sulfur $S_{t,ad}$ on an air dry basis.

6.4.2.2.2 According to the steps in 6.4.3, use the calibrated instrument to measure the sulfur content of the standard sample. Measure each standard sample at least three times. The average value of the three measured values is the sulfur measured value of the standard sample.

6.4.2.2.3 Input the sulfur measured value of the certified standard sample and the air-drying standard value into the sulfur meter (or the instrument automatically reads it) to generate a working curve or correction coefficient.

NOTE: Some instruments may require manual calculation of correction coefficients before entering them into the instrument.

6.4.2.3 Calibration validity verification

Select another 1 ~ 2 standard samples or other control samples. Use a calibrated sulfur meter to measure the total sulfur composition. If the difference between the measured value and the standard value (or control value) is within the uncertainty range (or control limit) of the standard value (or control value), the calibration is valid. Otherwise, the cause shall be identified and recalibrated.

6.4.3 Determination steps

6.4.3.1 After heating the high-temperature furnace to 1250°C, introduce oxygen. Adjust the flow regulation system. Control the oxygen flow at 3.0 L/min.

6.4.3.2 Weigh (0.3±0.01) g of air-dried specimen with a particle size less than 0.2 mm (accurate to 0.0001) in the combustion boat. Place the combustion boat on the setting device. Follow the instrument instruction manual, start the program, and perform the test. The system will automatically analyze the sulfur content in the sample. After the specimen is completely burned, the instrument displays and prints out the mass fraction of sulfur in the specimen.

6.4.4 Calibration check

This is an excerpt of the PDF (Some pages are marked off intentionally)

Full-copy PDF can be purchased from 1 of 2 websites:

1. <https://www.ChineseStandard.us>

- SEARCH the standard ID, such as GB 4943.1-2022.
- Select your country (currency), for example: USA (USD); Germany (Euro).
- Full-copy of PDF (text-editable, true-PDF) can be downloaded in 9 seconds.
- Tax invoice can be downloaded in 9 seconds.
- Receiving emails in 9 seconds (with download links).

2. <https://www.ChineseStandard.net>

- SEARCH the standard ID, such as GB 4943.1-2022.
- Add to cart. Only accept USD (other currencies - <https://www.ChineseStandard.us>).
- Full-copy of PDF (text-editable, true-PDF) can be downloaded in 9 seconds.
- Receiving emails in 9 seconds (with PDFs attached, invoice and download links).

Translated by: Field Test Asia Pte. Ltd. (Incorporated & taxed in Singapore. Tax ID: 201302277C)

About Us (Goodwill, Policies, Fair Trading...): <https://www.chinesestandard.net/AboutUs.aspx>

Contact: Wayne Zheng, Sales@ChineseStandard.net

Linkin: <https://www.linkedin.com/in/waynezhengwenrui/>

----- The End -----