

ICS 71.080.15

CCS G 18



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

GB/T 3208-2009

Replacing GB/T 3208-1982

**Standard test method for the total sulfur content in benzene --
Type products by oxidative microcoulometry**

苯类产品总硫含量的微库仑测定方法

(English Translation)

Issued on: July 8, 2009

Implemented on: April 1, 2010

**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

1	Scope
2	Normative references
3	Principles
4	Sample collection and preparation
5	Reagents
7	Test steps
7.1	Preparation of test equipment
7.2	Calibration
7.3	Determination of samples
8	Result calculation
9	Precision

1 Scope

1.1

This standard specifies the principles, reagents, equipment, test procedures, result calculation, precision, etc. for the determination of the total sulfur content of benzene products.

1.2

This standard applies to the determination of sulfur content in coking benzene, coking toluene and coking xylene. Measuring range. 1mg/kg~ 1000mg/kg.

1.3

For samples with nitrogen content exceeding 0.1% or chlorine content exceeding 1.0% per unit weight, there may be certain the error can be eliminated by adding an appropriate amount of sodium azide to the electrolyte of the titration vessel. This standard does not apply per unit weight Amounts of bromine and organometallic compounds content of more than 500mg/kg samples.

1.4

For samples with a sulfur content exceeding.200 mg/kg per unit weight, it can be diluted to about 50 mg/kg with isooctane or toluene.

Re-measure.

2 Normative references

The clauses in the following documents become clauses of this standard through reference in this standard. For dated references, all subsequent Amendments (excluding errata content) or revisions do not apply to this standard, however, parties to agreements based on this standard are encouraged to study is the latest version of these files available. For undated references, the latest edition applies to this standard.

GB/T 1999 Sampling method for coking oil products GB/T 8170 Numerical Rounding Rules and Representation and Judgment of Limit Values

3 Principles

The sample is mixed with oxygen in the combustion tube and burned, so that the sulfur in the sample is converted into sulfur dioxide, which is brought into the titration cell by the carrier gas. Dioxygen Sulfur reacts with I_3^- in the pool as follows.

$I_3^- + SO_2 + H_2O \rightarrow SO_3 + 3I^- + 2H^+$ when I_3^- is consumed, the indicator-reference electrode pair indicates this change and sends the signal to the microcoulomb counter amplifier, which outputs A corresponding current is passed to the electrolytic anode-electrolytic cathode electrode pair, where the following reactions occur at the electrolytic anode.

$3I^- - 2e^- \rightarrow I_3^-$

To supplement the I_3^- consumed by SO_2 , until the I_3^- produced by electrolysis restores the concentration of I_3^- in the titration cell to the concentration before the titration. Electrolysis produces the electricity consumed by I_3^- is the product of the digital display value of the microcoulomb counter and the selected electricity range. According to Faraday's law of electrolysis, through the standard sample Correction can calculate the sulfur content in the sample.

4 Sample collection and preparation

According to GB/T 1999 regulations.

5 Reagents

5.1

Reaction gas. oxygen, with a purity of not less than 99.99%.

5.2

Inert gas. argon or helium with a purity of not less than 99.99%.

5.3

Electrolyte. dissolve potassium iodide (KI) and sodium azide in 500 mL of distilled water according to the requirements in Table 1, and add glacial acetic acid.

(CH₃COOH), dilute the solution to 1000mL with distilled water.

GB/T 3208-2009

6.1 Combustion furnace. The inlet, central and outlet parts of the combustion tube can be individually heated and adjusted.

6.2 Combustion tube. made of quartz, which can burn the sample in the atmosphere of oxygen and inert gas.

6.3 Titration cell. a glass electrolyte cell with a magnetic stirrer and a built-in indicator-reference electrode pair, which is used to test the cell the concentration of I₃⁻; another electrolysis anode-electrolysis cathode electrode pair, its role is to maintain a constant concentration of I₃⁻. The platinum sheet of the indicator electrode is semi-electric the reference electrode was a platinum wire half-cell in an I₃-saturated electrolyte. The electrolytic anode is made of platinum sheet, and the electrolytic cathode is made of platinum wire. ginseng both the specific electrode and the electrolytic cathode communicate with the central chamber of the titration cell through a bundle of glass capillaries.

6.4 Microcoulomb counter. It measures the potential of the indicator-reference electrode pair, compares this potential with a given bias voltage, and informs the difference.

The signal is amplified to the corresponding current and then applied to the electrolytic anode-electrolytic cathode pair, so that the electrolysis produces I₃⁻.

6.5 Sulfur content recorder. Provide a recorder for converting the electricity of the generating electrode into sulfur content.

6.6 Micro syringe. 10 μL, 20 μL, 50 μL, 100 μL.

6.7 Sample bolt. quartz or platinum material.

7.1 Preparation of test equipment

7.1.1

Before carrying out the test, first confirm the combustion tube and gas duct as shown below.

7.1.1.1

Combustion tube. Check the combustion tube and quartz fiber for quartz aging and dirt, if not very clean, it needs to be cleaned or perform a sufficient air-burning after replacement.

7.1.1.2

Gas duct. Check the gas duct for aging or dirt, and clean it sufficiently.

7.1.2

After cleaning the inside of the titration cell with the electrolyte, add the electrolyte to it until the electrodes are completely submerged.

7.1.3

Indicating electrode. connect the respective terminals of the reference electrode and the generating electrode to the circuit of the microcoulomb counter.

Note: If a vertical test instrument is used, the end point potential needs to be set at this stage.

7.1.4

Connect the gas conduit of the titration cell to the top position of the outlet of the combustion tube, and energize the strip heater to make the gas conduit of the titration cell.

The temperature is kept above 100°C.

7.1.5

Set the flow rate of oxygen and inert gas, the temperature of the combustion furnace, and the microcoulomb counter to meet the measurement conditions. Typical Conditions for Assays shown in Table 2.

7.2 Calibration

7.2.1

According to the range of sulfur content, select the appropriate sulfur standard solution or standard material.

7.2.2

Select the injection volume of the sulfur standard solution from Table 3, then use a micro-syringe to inject the sample, and inject the fuel according to the following method.

Burn the tube.

7.2.2.1

When using the vertical instrument. insert the needle tip of the microinjector into the instrument through the sample filling port until it reaches the entrance of the combustion tube, then Then inject the sulfur standard solution into it at a speed of 1.0 $\mu\text{L/s}$ ~ 1.2 $\mu\text{L/s}$, accurately read the injected amount or weigh its mass (accurate to 0.1 mg).

7.2.2.2

When using the horizontal instrument. insert the needle tip of the micro syringe into the test instrument through the sample filling port, and then insert the needle into the sample bolt.