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National Standard of the People's Republic of China

GB/T 3558-2014

Replacing GB/T 3558-1996

Determination of chlorine in coal

(ISO 587:1997, Solid mineral fuels -

Determination of chlorine using Eschka mixture, NEQ)

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Foreword

This Standard is drafted according to the rules given by GB/T 1.1-2009.

This Standard replaces GB/T 3558-1996 *Determination of chlorine in coal*. Compared with GB/T 3558-1996, except the editorial modifications, the main changes are as follows:

- Change the preparation concentration (see 3.2.2, or 2.2.2 in 1996 version) and adding amount (see 3.4.2, or 2.4.2 in 1996 version) of the sulfuric acid solution;
- Change the concentration of the silver nitrate standard solution (see 3.2.6 or 2.2.9 in 1996 version);
- Add a potentiometric titration assembly, using the silver-silver chloride reference electrodes available on the market (see 3.3.2).

Using re-drafting method, this Standard is compiled with reference to ISO 587:1997 *Solid mineral fuels – Determination of chlorine using Eschka mixture*. This Standard is not equivalent to ISO 587:1997.

Determination of chlorine in coal

1. Scope

This Standard specifies the summary of method, reagents and materials, apparatus, test procedure, calculation of results and precision for the determination of total chlorine content in coal using the high temperature combustion hydrolysis - potentiometric titration and Aldrin melt sample - potassium hydrogen sulfate titration.

This Standard is applicable to brown, bituminous coal and anthracite.

2. Normative references

The following documents are indispensable for the application of this Standard. For dated reference, the versions dated apply to this Standard. For undated references, the latest edition of the normative document (including all modifications) referred to applies.

GB/T 483 General rules for analytical and testing methods of coal

3. Method A: High temperature combustion hydrolysis - potentiometric titration

3.1 Summary of method

A coal sample is combusted and hydrolyzed in the mixed gas flow of oxygen and water vapor. All chlorines contained in the coal are converted to chlorides and dissolved in the water quantitatively. Titrate the concentration of chlorine ions in the condensate directly by silver nitrate potentiometric method. Use the silver as an indicator electrode and the silver-silver chloride as a reference electrode. Calculate the total chlorine content in the coal sample according to the consumed volume of silver nitrate standard solution.

3.2 Reagents and materials

During analysis, unless otherwise stated, use only reagents of recognized analytical grade and only distilled water or deionized water or water of equivalent purity.

3.2.1 Absolute ethyl alcohol: C_2H_5OH , $\rho_{20^\circ C}=0.79$ g/mL.

3.2.2 Sulfuric acid solution: (1 + 23) ($V_1 + V_2$), add 1 volume of concentrated sulfuric acid of reagent grade to 23 volumes of water slowly, and mix uniformly.

3.2.3 Sodium hydroxide solution: 10 g/L, dissolve 1 g of reagent grade sodium hydroxide in 100 mL of water.

and hold for 15 min.

3.4.2.2 After completion of combustion-hydrolysis, take off the absorber; cut off the supply of oxygen and water vapor; remove the sample injection rod; and withdraw the porcelain boat by using the nichrome wire with a hook.

3.4.2.3 Pour the sample solution from absorption flasks to a 200 mL beaker. Wash the flasks and gas tubing with distilled water. The washing lotion is transferred to the beaker directly. Add the distilled water to (140 ± 10) mL. Then add 3 drops of the bromocresol green indicator (see 3.2.7) to the beaker. Neutralize the solution with sodium hydroxide solution (see 3.2.3) till the indicator becomes light blue. Then, add 1 mL of the sulfuric acid solution (see 3.2.2), 3 mL of the saturated potassium nitrate solution (see 3.2.4), and 5 mL of the sodium chloride standard solution (see 3.2.5).

3.5 Potentiometric titration

3.5.1 Preparation

Place the beaker containing 150 mL of the distilled water on a titration set. Insert an indicator electrode. Connect the titration assembly (self-made silver-silver chloride reference electrode) according to Figure 4. Connect to the self-made silver-silver chloride reference electrode with a salt bridge; or connect the titration assembly (silver-silver chloride reference electrode available on the market) according to Figure 5. Connect the indicator electrode lead and reference electrode lead to measuring terminal of a millivolt ammeter. Put the stirring bars inside. Start up the stirrer. At this point, the ammeter should indicate the potential difference ($\pm m$ V); otherwise, it shall verify if the connection of measuring circuit is correct.

3.5.2 Potentiometric calibration of end point

3.5.2.1 Preparation of blank solution: Same as 3.4.2, except that no coal sample is added.

3.5.2.2 Potentiometric calibration of titration end point: Place a beaker containing the blank solution on a titration set. Drip the silver nitrate standard solution that has a definite dripping quantity at a rate of 0.03 mL/s. Record the potential value at this point. Carry out the potential determinations twice as above. The difference between the measured values shall not exceed ± 3 mV. Use the mean as the potential of titration end point. For the first determination or change of chemical reagents, draw the differential curve of titration according to Annex A to determine the consumed volume (V_1) of silver nitrate standard solution when the potential of end point is calibrated.

3.5.3 Titration for sample solution

Place a beaker containing the sample solution on a titration set. Firstly, drip the silver nitrate standard solution at a rate of 0.05 mL/s; observe the reading of millivolt ammeter. When it is close to calibrated potential of end point, make titration at a rate of 0.02 mL/s till the calibrated potential of end point is reached. After 1 min of stirring, write down the

4.4.1 Weigh (1 ± 0.1) g of the coal sample (accurate to 0.000 2 g) for general analytic test. Place in a crucible in where 3 g (to the nearest 0.1 g) of the Eschka mixture is contained. Mix thoroughly. Then, cover with a further 2 g of the Eschka mixture. Place the crucible in the muffle furnace. Keep the furnace door half-opened. Raise the temperature to $(680 \pm 20)^{\circ}\text{C}$. Maintain this temperature for 3 h.

4.4.2 Withdraw the crucible from the muffle furnace. Cool to room temperature. Transfer the incinerated mixture to a 250 mL beaker. Wash the inner wall of crucible with 50 mL ~ 60 mL of hot water. Add the washing lotion to the beaker.

4.4.3 Filter the solution through a qualitative filter paper by decantation method. Wash the residues with hot water once or twice. Then transfer the residues to a funnel. Wash the residues and paper thoroughly with hot water till there is no chlorine ions (without turbidity if it is examined with the silver nitrate solution). In the process of filtration and residues washing, control the filtrate to a final volume of about 110 mL.

4.4.4 Add 1 drop of the phenolphthalein indicator to filtrate. Regulate with the nitric acid until the red colour disappears, with an extra addition of 5 mL; add 5 mL of the sodium chloride standard solution and 10 mL of the silver nitrate standard solution accurately with a one-mark pipette. Stand over a period of 2 min ~ 3 min. Measure 3 mL of hexanol; cover with a watch glass. Place the beaker on an electromagnetic stirrer and stir for 1 min rapidly. Then add 1 mL of the ammonium iron sulfate solution. Titrate with the potassium hydrogen sulfate standard solution. The end point is reached when the solution changes to light orange from cream white. Write down the volume (V_3) of consumed potassium hydrogen sulfate standard solution.

4.4.5 Determination of blank value

Determine each lot of coal samples. Carry out two or more blank determinations by using the same procedure as described in 4.4.1 ~ 4.4.4. Take the mean as the blank value (V_4).

4.5 Calculation of results

The chlorine content of the coal is calculated according to Formula (3) and is recorded with the mean of two repeated determinations by rounding off to the third decimal places in according with GB/T 483.

$$Cl_{ad} = \frac{(V_3 - V_4) \times c \times M_{Cl}}{m} \times 100 \quad \dots\dots\dots (3)$$

Where:

Cl_{ad} – is the chlorine content of the air-dried coal sample, expressed as a percentage (%) by mass;

V_3 – is the volume, in millilitres, of the potassium hydrogen sulfate standard solution used in the determination;

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