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

GB/T 3558-2014

**Determination of Chlorine in Coal
(English Translation)**

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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

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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

3.1 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.

3.3.1.4 Water vapor generator. It is consisted of a 500 mL flat bottom flask and an adjustable electric disc furnace (0.5 kW, AC 0 V ~ 220 V, adjustable continuously).

3.3.1.5 Absorber. It is consisted of two 250 mL conical flasks, glass tubes and rubber stopper.

3.3.1.6 Oxygen flow meter. 1 000 mL/min at full scale, having the minimum division value of 10 mL/min.

3.3.2 Potentiometric titration assembly. as shown in Figure 4, using the self-made silver-

silver chloride reference electrodes. The silver-silver chloride reference electrodes available on the market may also be used, and the potentiometric titration assembly is shown in Figure 5.

3.4.1 Preparation of apparatus

Assemble the apparatus as shown in Figure 1. Stuff a little of refractory wool into the gas outlet of combustion tube. Connect the electric circuit, gas circuit and condensate water. Add about 30 mL of distilled water to No.1 absorption flask, and about 20 mL to No.2 absorption flask. Raise the high temperature furnace to 1 100°C. Mark the 300°C, 600°C and 800°C zones of the combustion tube. Plug up the rubber stopper of sample injection rod. Regulate the flow rate of oxygen to 500 mL/min and the evaporation capacity of water vapor generator to about 2 mL/min. Check if there is gas leakage.

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.

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;

3.6 Calculation of results

The chlorine content of the coal is calculated according to Formula (1), and is recorded with the mean of two repeated determinations by rounding off to the third decimal places in according with GB/T 483.

3.7 Precision of this method

The precision of this method for the determination of chlorine in coal is shown in Table 1.

4.1 Summary of method

Mix the coal sample and Eschka mixture. Melt them in a muffle furnace. Convert the chlorine to chlorides.

4.2 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.

4.2.1 Eschka mixture. Weigh two portions of mass of magnesium oxide AND one portion

of mass of anhydrous sodium carbonate. Grind to the granularity less than 0.2 mm. Mix well. Or, the Eschka mixture available on the market may be used.

4.2.4 Saturated ammonium iron sulfate solution. add the ammonium iron sulfate to a

certain amount of water till it does not dissolve anymore, and there is certain amount of solid ammonium iron sulfate in the solution. Add several milliliters of nitric acid to remove the brown colour. Take the supernatant liquid for use.

4.2.5 Silver nitrate solution. 10 g/L. Weigh out 1 g of the silver nitrate and dissolve in 100

mL of water, and add several milliliters of nitric acid.

4.2.6 Silver nitrate standard solution. $c(\text{AgNO}_3) = 0.025\ 00\ \text{mol/L}$. Weigh out accurately

4.247 2 g of reference reagent silver nitrate that has been pre-dried at 110°C for 1 h and cooled to room temperature in a drier. Dissolve in a little of water. Then transfer to a 1 000 mL brown measuring flask. Dilute to scale with water.