NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

GB/T 3058-2019

Determination of arsenic in coal

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Foreword

This standard was drafted in accordance with the rules given in GB/T 1.1-2009.

This standard replaces GB/T 3058-2008 "Method for the determination of arsenic in coal". As compared with GB/T 3058-2008, except for editorial changes, the main technical changes are as follows.

- In the method summary, CHANGE that "use hydrochloric acid to dissolve the burnt" into that "use sulfuric acid and hydrochloric acid to dissolve the burnt" (see 3.1; 3.1 of the 2008 edition);
- MODIFY the requirements for arsenic-free zinc particles (see 3.2.1; 3.2.1 of the 2008 edition);
- ADD the placement position of lead acetate cotton to the diagram of the arsenic determination device (see Figure 1; Figure 1 of the 2008 edition);
- ADD the calculation method of the recovery rate of the arsenic determination device by the arsenic molybdenum blue spectrophotometric method (see Appendix A).

This standard is prepared by using the redrafting method with reference to ISO **11723.2016 "Solid mineral fuels - Determination of arsenic and selenium -** Eschka's mixture and hydride generation method". The degree of consistency

with ISO 11723.2016 is nonequivalent.

This standard was proposed by the China Coal Industry Association.

This standard shall be under the jurisdiction of the National Coal Standardization Technical Committee (SAC/TC 42).

Drafting organization of this standard. Testing Branch of Coal Science and Technology Research Institute Co., Ltd.

Drafters of this standard. Chen Huizhu, Fu Kun, Yang Guang, Yang Huayu.

This standard replaces the standard previously issued as follows.

- GB/T 3058-1982, GB/T 3058-1996, GB/T 3058-2008.

Determination of arsenic in coal

1 Scope

This standard specifies the method summary, reagents and materials, instruments and equipment, samples, test procedures, calculation and presentation of results, the precision of the method for the determination of arsenic in coal by arsenic molybdenum blue spectrophotometry and hydride generation-atomic absorption method. Arsenic molybdenum blue spectrophotometry is the arbitration method.

This standard applies to lignite, bituminous coal, anthracite.

2 Normative references

The following documents are essential to the application of this document. For the dated documents, only the versions with the dates indicated are applicable to this document; for the undated documents, only the latest version (including all the amendments) is applicable to this standard.

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

GB/T 2304 Chemical reagent - Zinc granular free from arsenic

3.1 Method summary

The coal sample is mixed with Eschka reagent and burned. The burned material

is dissolved with hydrochloric acid and sulfuric acid. A reducing agent is added to reduce pentavalent arsenic to trivalent arsenic. Add zinc particles to form and release arsenic hydride gas. Use iodine solution to absorb and oxidize it to arsenic acid. Add ammonium molybdate-hydrazine sulfate solution to produce arsenic molybdenum blue. Then use a spectrophotometer to determine it.

3.2 Reagents and materials

Unless otherwise specified, only use reagents and distilled or deionized water or equivalent purity water confirmed to be analytically pure in the analysis.

3.2.1 Arsenic-free zinc metal. In accordance with GB/T 2304, screen out zinc particles with a particle size of not less than 3 mm for use.

3.2.2 Eschka reagent (hereinafter referred to as Aldrin). Commercially available, or 2 parts by mass of light magnesium oxide and 1 part by mass of anhydrous sodium carbonate are mixed and ground to a particle size of less than 0.2 mm, then stored in a closed container.

3.2.4 Hydrochloric acid solution. c(HCl) = 6 mol/L. Mix 1 volume of hydrochloric acid with 1 volume of water evenly.

3.2.5 Sulfuric acid solution. c(1/2 H2SO4) = 6 mol/L. Measure 167 mL of sulfuric acid with a relative density of 1.84 and slowly add it to an appropriate amount of water. Stir it while adding. Then use water to dilute it to 1 L.

3.2.6 Sulfuric acid solution. c (1/2 H2SO4) = 5 mol/L. Measure 139 mL of sulfuric acid with a relative density of 1.84 and slowly add it to an appropriate amount of water. Stir it while adding. Then use water to dilute it to 1 L.

3.2.7 Potassium iodide solution. 176.5 g/L. Dissolve 3.0 g of potassium iodide in 17 mL water and prepare it before use.

3.2.8 Stannous chloride hydrochloric acid solution. 666.7 g/L. Dissolve 8.0 g of stannous chloride in 12 mL of hydrochloric acid (3.2.3) and prepare it before

use.

3.2.10 Ammonium molybdate solution. 10 g/L. Dissolve 10.0 g of ammonium molybdate in 1 L sulfuric acid solution (3.2.6).

3.2.12 Ammonium molybdate-hydrazine sulfate mixed solution. Mix 1 volume of ammonium molybdate solution (3.2.10) and 1 volume of hydrazine sulfate solution (3.2.11) evenly. Prepare it before use.

3.2.17 Lead acetate cotton. It is replaced each time of the experiment. The absorbent cotton is fully soaked in a lead acetate solution which has a mass concentration of 400 g/L. Take it out and twist it dry. Dry it at 80 °C ~100 °C. Store it in a desiccator for later use.

3.2.18 Porcelain crucible. The capacity is 30 mL. The enamel on the inner surface is intact.

3.5.1.1 Respectively use the single marking line pipette to take 0.00 mL, 1.00 mL, 2.00 mL, 3.00 mL, 4.00 mL, 5.00 mL of arsenic standard working solution (3.2.16). Place it into the round flask of the arsenic determination device. First add 10 mL of sulfuric acid solution (3.2.5). Then add 20 mL of hydrochloric acid solution (3.2.4). Use water to dilute it to 50 mL.

3.5.1.2 Add 2 mL of potassium iodide solution (3.2.7), 1 mL of stannous chloride solution (3.2.8). Shake it uniformly. Place it at room temperature for 15 min.

3.5.1.3 Use a pipette to add 3 mL of iodine solution (3.2.9), 1 mL of sodium bicarbonate solution (3.2.13), 6 mL of water to the absorber of the arsenic determination device. Insert the absorber into the absorber casing which contains lead acetate cotton (3.2.17).

abscissa and the corresponding absorbance as the ordinate. Proceed it simultaneously with the analysis of the coal sample.