

ICS 73.040



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

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**Determination of mercury of coal
(English Translation)**

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1 Scope

This Document specifies the reagents and materials, instruments and equipment, test conditions, samples, test procedures, test data processing, precision and test reports for the determination of mercury in coal by cold atomic absorption spectrophotometry, atomic fluorescence spectrometry, mercury detector method and solid sampling direct mercury detector method.

This Document is applicable to the determination of mercury content in lignite, bituminous coal, anthracite and coal gangue.

2 Normative References

The provisions in following documents become the essential provisions of this Document through reference in 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 Document.

GB/T 213 Determination of calorific value of coal

GB/T 474 Preparation of coal sample

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

GB/T 6682 Water for analytical laboratory use - Specification and test methods

GB/T 19494.2 Mechanical sampling of coal - Part 2.Method for sample preparaion

3 Terms and Definitions

For the purposes of this Document, there are no terms and definitions apply.

4 Reagents and Materials

Unless otherwise specified, only reagents confirmed as analytically pure are used in the analysis;

and the used water is Class-II water or water of equivalent purity as specified in GB/T 6682.

4.1

Oxygen. in accordance with GB/T 213.

4.2

Argon. purity above 99.99%.

4.3

Nitric acid. guarantee reagent, $\rho=1.42\text{g/mL}$.

4.4

Sulfuric acid. guarantee reagent, $\rho=1.84\text{g/mL}$.

4.5

Hydrochloric acid. guarantee reagent, $\rho=1.19\text{g/mL}$.

4.6

Vanadium pentoxide.

4.7

Hydrogen peroxide. 30% (mass fraction).

4.8

Anhydrous ethanol.

4.9

Nitric acid solution. volume ratio is (1+9).

4.10

Nitric acid solution. volume ratio is (5+95).

4.13

Fixed solution. 0.5g/L.

Pipette 10.00mL of potassium dichromate solution (4.12) into a 1L volumetric flask; add appropriate amount of water; then add 50mL of nitric acid (4.3); dilute to scale with water; and make constant volume.

4.14

Potassium permanganate solution. 50g/L.

Weigh 5.0g of potassium permanganate and dissolve in 100mL of water; and store in a brown reagent bottle.

4.15

Hydroxylamine hydrochloride solution. 15g/L.

Weigh 1.5g of hydroxylamine hydrochloride and dissolve in 100mL of water.

4.16

Potassium hydroxide solution. 5.0g/L.

Weigh 5.0g of potassium hydroxide and dissolve it in 1L of water.

4.17

Potassium borohydride solution or sodium borohydride solution. 5.0g/L.

Weigh 5.0g of potassium borohydride (guarantee reagent) or sodium borohydride (guarantee reagent) and dissolve it in 1L of potassium hydroxide solution (4.16); prepare it before use.

5 Apparatus

5.1

Analytical balance. graduation value 0.1mg.

5.2 Atomic absorption spectrophotometer. equipped with a mercury hollow cathode lamp with

a wavelength of 253.7nm.

5.3 Atomic fluorescence spectrometer. equipped with a mercury hollow cathode lamp with a

wavelength of 253.7nm.

5.4 Mercury detector. cold atomic absorption mercury detector or cold atomic fluorescence

mercury detector.

5.5 Solid sampling direct mercury detector.

5.6 Electric heating plate. controllable in the range of 50°C~200°C.

5.7 Microwave digestion system. including microwave digestion instrument, digestion tank, etc.

5.8 Mercury vapor generation bottle. a conical bottle with a scale of 50mL and a tightly fitting plug, with inlet and outlet pipes on the plug and the end of the inlet pipe 5mm~10mm away from the bottom of the bottle.

6 Test Conditions

6.1

The test shall be carried out in a room with a temperature above 10°C.

6.2

All glassware used in the test must be soaked in nitric acid solution (4.10) for at least 24 h before use, and then washed by water for later use.

7.1 Specimens

The coal samples for the general analysis test shall be prepared according to GB/T 474 or GB/T 19494.2.

7.2.1 Wet digestion

Weigh about 0.1g (accurate to 0.0002g) of the coal sample for general analysis test into a mercury vapor generator bottle. Add 50mg of vanadium pentoxide (4.6) and a few drops of anhydrous ethanol (4.8) to moisten the specimen; then add 10mL of nitric acid (4.3);

immediately cover the mercury vapor generator bottle with a porcelain cover; leave it overnight.

Add 4mL of sulfuric acid (4.4) and place it on a electric hot plate; first heat at (120±10) °C for about 1.5h; then gradually increase the temperature to (160±10) °C; and continue heating until sulfur trioxide smoke just emerges.