

Translated English of Chinese Standard: DL/T2296-2021

www.ChineseStandard.net → Buy True-PDF → Auto-delivery.

Sales@ChineseStandard.net

DL

ELECTRIC POWER INDUSTRY STANDARD

OF THE PEOPLE'S REPUBLIC OF CHINA

ICS 27.100

CCS D 21

DL/T 2296-2021

**Determination of total mercury in coal and coal ash - Direct
combustion method**

煤和煤灰中总汞的测定方法 直接燃烧法

Issued on: April 26, 2021

Implemented on: October 26, 2021

Issued by: National Energy Administration

Table of Contents

Foreword.....	3
1 Scope	4
2 Normative references.....	4
3 Terms and definitions.....	4
4 Principles of the method	5
5 Reagents and materials	5
6 Instruments and equipment.....	5
7 Specimens	7
8 Test steps.....	7
9 Result calculation	10
10 Method precision.....	11
11 Requirements for test records and report	11
References.....	12

Determination of total mercury in coal and coal ash - Direct combustion method

1 Scope

This document specifies the direct combustion method for the determination of total mercury, in coal and coal ash.

This document applies to lignite, bituminous, anthracite, coal ash.

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 474 Method for preparation of coal sample

GB/T 475 Method for manual sampling of commercial coal

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

GB/T 14599 Pure oxygen and high purity oxygen and ultra pure oxygen

GB/T 19494.1 Mechanical sampling of coal - Part 1: Method for sampling

GB/T 19494.2 Mechanical sampling of coal - Part 2: Method for sample preparation

DL/T 567.1 Test methods of fuel in thermal power plants - Part 1: General rule

DL/T 567.3 Test methods of fuel in thermal power plants - Part 3: Sampling and preparation for fly ash and bottom ash

JB/T 5228 Specification for mercury determinator

3 Terms and definitions

The following terms and definitions apply to this document.

3.1

Amalgamation

The process of forming an alloy between mercury and metal powder, in a solid state.

3.2

Amalgamator

A device for amalgamation, between mercury vapor and gold powder.

4 Principles of the method

The specimen is dried, burnt, decomposed in an oxygen stream. The sulfur oxides, nitrogen oxides, halides in the combustion products are removed; the mercury of various valence states are catalytically reduced to elemental mercury. Elemental mercury is amalgamated in the amalgamator. After purged by oxygen, to remove the remaining residues, rapidly heat the amalgamator, to release gaseous mercury. Measure its absorbance, at the characteristic wavelength of 253.7 nm, to obtain the total mercury content in the specimen.

5 Reagents and materials

5.1 Oxygen: Pure oxygen in accordance with GB/T 14599.

5.2 Coal standard material (hereinafter referred to as "standard sample"): A certified coal standard material, which has the standard value of mercury content.

5.3 The specimen boat: It is made of nickel, quartz or ceramics, etc., of suitable shape and size; it does not react with the mercury during specimen pyrolysis.

6 Instruments and equipment

6.1 The mercury measuring instrument is mainly composed of sample injection unit, drying and combustion unit, catalytic unit, amalgamation unit, dehumidification unit, detection unit, signal acquisition and processing unit, tail gas disposal unit, as shown in Figure 1. The functional requirements of each part are as follows:

- h) Tail gas disposal unit: It contains adsorbent, which can adsorb the mercury in the tail gas.

6.2 The technical conditions of the measuring instrument shall meet the requirements of JB/T 5228.

6.3 Analytical balance, which has a division value of 0.1 mg.

7 Specimens

7.1 Collection and preparation of coal samples

Collect samples according to GB/T 475 or GB/T 19494.1. Prepare coal samples for general analysis test, according to the requirements of B/T 474 or GB/T 19494.2.

7.2 Collection and preparation of fly ash and slag samples

Collect and prepare fly ash and slag samples, according to the requirements specified in DL/T 567.3. Grind the specimen to a particle size of less than 0.1 mm.

8 Test steps

8.1 Calibration

8.1.1 Basic requirements

8.1.1.1 Measure the moisture content on the air-dried basis of the standard sample. Calculate the standard value of the mercury content, on the air-dried basis.

8.1.1.2 According to step 8.2, use the calibrated measuring instrument, to measure the mercury content in the standard sample. For each calibration point, carry out 4 repeated measurements. If the range of the four measurement results does not exceed $1.3r$ (r represents the method repeatability limit), THEN, the average value is taken as the measured value of the standard sample, at the calibration point. Otherwise, it shall find out the reason, handle it, before re-calibration. For multi-point calibration, it is necessary to convert the standard sample mass, at each calibration point, to a unified mass, to calculate the average value of the measured values.

8.1.2 Single point calibration

8.1.2.1 Estimate the range of mercury content in the specimen. Select a standard sample, which is suitable for the standard value of mercury content.

8.1.2.2 Weigh the mass range of the specimen, according to the specification of the mercury measuring instrument. Calculate the mass of the standard sample to be weighed, according to the formula (1), based on the mercury content in the specimen.

shall be less than the mass of mercury in the specimen. The maximum mass of the standard sample, which is actually weighed, shall be greater than its calculated value; the mass of mercury in the standard sample shall be greater than the mass of mercury in the specimen.

8.1.3.3 There shall be no less than 6 calibration points; the number of repeated measurements, at each calibration point, shall be not less than 4. The weighed sample mass of each calibration point shall be in or close to the equidistant sequence.

8.1.3.4 Take the calculated mass of mercury, in the standard sample, as the abscissa; take the mass of mercury, in the standard sample, which is measured by the measuring instrument, as the ordinate, to establish a linear regression equation, as shown in formula (3):

$$y = fx + a \dots\dots\dots (3)$$

Where:

f - The correction factor (slope);

y - The mass of mercury in the standard sample, which is measured by the mercury measuring instrument, in micrograms (μg);

x - The calculated mass of mercury in the standard sample, in micrograms (μg);

α - Intercept, in micrograms (μg).

8.1.3.5 The linear correlation factor of the calibration curve of the mercury measuring instrument shall not be less than 0.995; otherwise, it shall find the cause and handle it, before re-calibration.

8.1.4 Determination of calibration validity

8.1.4.1 Take 1 ~ 3 standard samples (quality control samples), that are not used for the calibration of the mercury measuring instrument, with appropriate content. Use the calibrated mercury measuring instrument, to measure the mercury content. If the difference between the measured value and its standard value (control value) is within the uncertainty range of its standard value (control value), the calibration is valid. Otherwise, it shall find the cause and handle it, before re-calibration.

8.1.4.2 If the reagents or materials are replaced, it shall re-calibrate the mercury measuring instrument.

8.2 Measurement

8.2.1 Start the instrument. Set the drying temperature to be 200 °C ~ 300 °C; set the combustion temperature to be 750 °C ~ 850 °C; set the catalytic temperature to be

450 °C ~ 600 °C; set the oxygen flow rate to be 0.4 L/h; set the catalytic time to be 300 s.

8.2.2 Carry out 2 blank tests. The measurement results shall not exceed the detection limit of the mercury measuring instrument.

8.2.3 Check the tail gas disposal unit, to confirm that the adsorbent has not failed.

8.2.4 Use a specimen boat, that is calcined and cooled to constant weight, to weigh 0.09 g ~ 0.11 g of specimen, accurate to 0.0002 g. When the mercury content of specimen is significantly higher or lower, the weighed sample mass can be appropriately reduced or increased; however, the sample weight should be within the range of 0.05 g ~ 0.15 g.

8.2.5 Put the specimen boat on the sample feeding unit of the mercury measuring instrument. Input the specimen mass. Start the sample feeding procedure, to automatically feed the specimen into the drying and combustion unit. Under the control of the prescribed program, the mercury measuring instrument starts to measure automatically. When the measurement process is over, record the measured value of the mercury measuring instrument.

8.2.6 After the specimen measurement is completed, take out the specimen boat. Turn off the power of the instrument. Stop the gas supply.

8.2.7 Before and at the end of the specimen test, measure the standard sample (quality control sample), which has a mercury content close to the specimen. The difference, between the measured value and its standard value (control value), is within the uncertainty range of its standard value (control value). Otherwise, it shall find the cause and handle it, before re-measurement.

9 Result calculation

9.1 Use formula (2) or formula (3), to calculate the mass of mercury in the specimen. Use the formula (4), to calculate the total mercury content of the specimen.

$$\omega(\text{Hg}) = \frac{m_1}{m} \dots\dots\dots (4)$$

Where:

$\omega(\text{Hg})$ - The total mercury content of the specimen, in micrograms per gram ($\mu\text{g/g}$);

m_1 - The mass of mercury in the specimen, in micrograms (μg);

m - The mass of the specimen weighed, in grams (g).

9.2 The measured value and the reported value are rounded to three decimal places, in microgram per gram ($\mu\text{g/g}$). The benchmark conversion is calculated, according to

This is an excerpt of the PDF (Some pages are marked off intentionally)

Full-copy PDF can be purchased from 1 of 2 websites:

1. <https://www.ChineseStandard.us>

- SEARCH the standard ID, such as GB 4943.1-2022.
- Select your country (currency), for example: USA (USD); Germany (Euro).
- Full-copy of PDF (text-editable, true-PDF) can be downloaded in 9 seconds.
- Tax invoice can be downloaded in 9 seconds.
- Receiving emails in 9 seconds (with download links).

2. <https://www.ChineseStandard.net>

- SEARCH the standard ID, such as GB 4943.1-2022.
- Add to cart. Only accept USD (other currencies - <https://www.ChineseStandard.us>).
- Full-copy of PDF (text-editable, true-PDF) can be downloaded in 9 seconds.
- Receiving emails in 9 seconds (with PDFs attached, invoice and download links).

Translated by: Field Test Asia Pte. Ltd. (Incorporated & taxed in Singapore. Tax ID: 201302277C)

About Us (Goodwill, Policies, Fair Trading...): <https://www.chinesestandard.net/AboutUs.aspx>

Contact: Wayne Zheng, Sales@ChineseStandard.net

Linkin: <https://www.linkedin.com/in/waynezhengwenrui/>

----- The End -----