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

SN/T 3365-2012

**Determination of lead, iron, titanium, copper, manganese, zinc,
chromium, aluminium content in quartz sand. Inductively
couple plasma atomic emission spectrometric method
(English Translation)**

Issued on: May 12, 2017

Implemented on: December 1, 2017

**Issued by: General Administration of Quality Supervision, Inspection and
Quarantine of the People's Republic of China**

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1 Scope

This Standard specifies the method for directly injecting samples to determine the mercury content in coal using a mercury analyzer.

This Standard is applicable to the determination of mercury content in lignite, bituminous coal, anthracite and coal gangue. The measuring range is: $0.800 \times 10^{-3} \mu\text{g/g}$ ~10.000 $\mu\text{g/g}$.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

GB/T 474, Method for preparation of coal sample

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

GB/T 602, Chemical reagent -- Preparations of standard solutions for impurity

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 preparation

3 Method summary

The test coal sample is directly sent to the mercury analyzer. After drying, decomposition, and oxidation, various forms of mercury are released in the gaseous state. After amalgamation occurs in the amalgamation tube and is enriched, mercury atoms are released through rapid heating. The released mercury is measured using an atomic absorption spectrometer at 253.7 nm.

4 Reagents and materials

Unless otherwise stated, all reagents used are guaranteed reagents, and the experimental water is grade two water specified in GB/T 6682.

4.1

Nitric acid ($\rho=1.42$ g/mL).

4.2

Nitric acid

(5+95).

4.3

Certified reference materials for coal

Mercury content has been determined.

4.4 Mercury standard stock solution (1000 $\mu\text{g/mL}$): use national certified standard solution or prepare according to GB/T 602.

4.5 Mercury standard intermediate solution (100 $\mu\text{g/mL}$): accurately pipette 10 mL of

mercury standard stock solution (4.4) into a 100 mL volumetric flask. Add 5 mL of nitric acid (4.1). Add water to dilute to volume. Shake well.

4.6 Mercury standard working solution series 1: absorb mercury standard intermediate

solution (4.5) to prepare: 0 $\mu\text{g/mL}$, 0.020 $\mu\text{g/mL}$, 0.050 $\mu\text{g/mL}$, 0.100 $\mu\text{g/mL}$, 0.250 $\mu\text{g/mL}$, 0.500 $\mu\text{g/mL}$ mercury standard working solution. The solvent is nitric acid (4.2). Prepare when needed.

4.7 Mercury standard working solution series 2: absorb mercury standard intermediate

solution (4.5) to prepare: 0 µg/mL, 0.50 µg/mL, 1.00 µg/mL, 2.00 µg/mL, 5.00 µg/mL, 10.00 µg/mL mercury standard working solution. The solvent is nitric acid (4.2).

Prepare when needed.

5.1 Mercury analyzer: equipped with a powder direct sampling device, a thermal decomposition device, an amalgamation device and an atomic absorption device (see Annex A).

5.2 Analytical balance: division is 0.1 mg.

5.3 Volumetric flask: grade A, 100 mL.

5.4 Pipette tubes: grade A, 1.0 mL, 5.0 mL, 10.0 mL.

5.5 Pipette: 0 µL~100 µL.

5.6 Sample boat: made of quartz or nickel, suitable for instrument use. The volume is not less than 150 µL.

5.7 Agate mortar.

5.8 Standard sieve: 116 µm.

5.9 High purity oxygen: purity $\geq 99.999\%$.

6 Sample preparation

The general analysis test coal samples prepared according to GB/T 474 or GB/T 19494.2 are ground with an agate mortar until all of them pass the 116 µm standard sieve, and are set aside.

7.1 Blank test

Do a blank test along with the specimen.

7.2 Sample determination

Accurately weigh 0.09 ± 0.01 g of the general analysis test coal sample. Weighing is accurate to 0.0001 g. Place in the sample boat. Refer to the working conditions in Annex B for measurement.

7.3.1 Working curve 1

Adjust the instrument conditions to appropriate working parameters according to the instrument instructions (see Annex B). In order from low concentration to high concentration, pipette 100 μL of mercury standard working solution series 1 (4.6) and add it to the sample boat for measurement. Draw the working curve 1 with the mercury content in the standard working solution as the abscissa and the absorbance as the ordinate.

7.3.2 Working curve 2

Determine the mercury standard working solution series 2 (4.7) according to steps 7.3.1.

Draw working curve 2.

7.4 Verification of standard curve

Measure the standard materials with similar contents at the upper and lower ends of each standard curve, respectively. If the measurement result is within the uncertainty range of the standard value, the standard curve is valid; otherwise, the cause should be found and the standard curve should be redrawn.

8 Result expression

The mercury content in general analytical test coal samples is calculated according to formula (1):

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