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NATIONAL STANDARD

OF THE PEOPLE'S REPUBLIC OF CHINA

GB/T 16659-2008

Replacing GB/T 16659-1996

Determination of mercury in coal

煤中汞的测定方法

(ISO 15237: 2003, Solid mineral fuels –

Determination of total mercury content of coal, MOD)

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Foreword

This Standard modifies and adopts ISO 15237:2003 *Solid Mineral Fuels-Determination of Total Mercury Content of Coal* (English version).

This Standard is redrafted on the basis of ISO 15237:2003. Annex A provides a cross-reference table of chapter numbers between this Standard and ISO 15237:2003.

Considering China's actual conditions, this Standard makes some alterations to ISO 15237:2003. The related technical differences have been integrated into the text and the relevant articles involved have been marked with a vertical single line in the margin. Annex B provides a table for the technical differences and their reasons, for reference.

This Standard replaces GB/T 16659-1996 *Determination of Mercury in Coal*.

Compared with GB/T 16659-1996, main changes of this Standard are as follows:

- Move the "determination method - cold atomic absorption spectrophotometry" from the annex of previous standard to main body of this Standard.

Annex A and Annex B in this Standard are informative.

This Standard was proposed by the National Coal Standardization Technical Committee of China.

This Standard shall be under the jurisdiction of the National Coal Standardization Technical Committee of China.

Drafting organizations of this Standard: Chongqing Institute of Geology and Mineral Resources, and Lianyungang Entry-Exit Inspection and Quarantine Bureau.

Main drafters of this Standard: Zhu Zhenzhong, Wu Yang, Yang Jie, and Li Jingpin.

The previous version that is replaced by this Standard is as follows:

- GB/T 16659-1996.

Determination of Mercury in Coal

1 Scope

This Standard specifies the cold atomic absorption spectrophotometry AND mercury analyzer method that is based on the principle of determination of atomic fluorescence absorbance, for determination of mercury in coal.

This Standard applies to lignite, bituminous coal and anthracite.

2 Normative references

The articles contained in the following documents have become this Standard when they are quoted herein. For the dated documents so quoted, all the modifications (excluding corrections) or revisions made thereafter shall not be applicable to this Standard. For the undated documents so quoted, the latest editions shall be applicable to this Standard.

GB/T 6682 Water for analytical laboratory use - Specification and test methods (GB/T 6682-2008, ISO 3696: 1987, Water for analytical use - Specification and test methods, MOD)

3 Cold atomic absorption spectrophotometry

3.1 Principle

Use vanadium pentoxide as catalyst. Coal samples are decomposed by nitric acid - sulfuric acid, so as to transform the mercury in the coal into bivalent mercury ions. The mercury ions are then reduced to mercury atomic vapour. Use cold atomic absorption spectrophotometer to determine the mercury content.

3.2 Reagent

3.2.1 The water referred to in this Standard is the distilled water in conformity with Grade II water requirements as specified in GB/T 6682 or the deionized water of the same purity.

3.2.2 Nitric acid: GR (guaranteed reagent), relative density is 1.42.

3.2.3 Sulfuric acid: GR, relative density is 1.84.

3.2.4 Hydrochloric acid: GR, relative density is 1.19.

Mercury analyzer: sensitivity is not less than 0.1µg/L.

4.4 Analytical Procedures

4.4.1 Instrument Adjustment

Start the mercury analyzer. Preheat for 1h~2h. Add 20mL of water in the mercury vapour generating bottle. Plug the stopper. Connect the mercury vapour generating bottle and the flowmeter to the mercury analyzer's gas circuit. Open the pump switch and adjust the air flow-speed to be about 800mL/min. Adjust the mercury analyzer's parameters such as zero point.

4.4.2 Coal sample treatment

Follow 3.4.2 to deal with coal samples.

4.4.3 Determination

4.4.3.1 Determination conditions

The same as 3.4.3.1.

4.4.3.2 Working curve plotting

4.4.3.2.1 Prepare standard series solutions in mercury vapour generation bottles according to 3.4.3.2.1. Shake them up and leave them placed for a moment. Connect the mercury vapour generation bottle to mercury analyzer. Determine and record the maximum absorption value.

4.4.3.2.1 Use the absorption value of standard solution as the ordinate; and its corresponding mercury mass as the abscissa, plot the working curve. Each batch of determinations shall come with plotting of working curves.

4.4.3.3 Coal sample determination

Use the coal sample treatment solution from disposal under 3.4.2.2. Follow the procedures specified in 4.4.3.2.1 to determine and record the maximum absorption value, with exception of no addition of standard mercury solution. Look for mercury mass from the working curve.

4.4.3.4 Blank test

The same as 3.4.3.4.

4.5 Result calculation

The same as 3.5.

4.6 Precision

	of standard stock solution and standard solution	while the national standard adopts GR mercury bichloride, so there are differences in sampling weight and preparation process.
3.3.3, 3.3.4	Add requirements to apparatuses for analytical use	Put forward clear requirements for special appliance used in analysis.
-	Delete 5.2 and 5.3 in ISO 15237:2003 (E)	The test posterior to chemical decomposition of coal samples does not need the utensils specified in the two sections.
3.4.1	Add requirements for gas circuit connection and instrument adjustment	The requirements for testing gas circuit connection and instrument adjustment are more clear and definite.
3.4.2	Delete the method of resolving coal samples by oxygen-bomb combustion; add the chemical method of resolving coal samples	The chemical decomposition for coal samples are more common domestically.
3.4.3.2	Increase the number of standard preparation for standard series from 3 specified in international standards to 5 specified in national standards	The increase in the number of standard preparation is much more favourable to the practical application of standard curve.

Chapter Number of this Standard	Technical Differences	Reasons
3.6	Adopt the precision requirements specified in the original national standard.	The determination of precision specified in the original national standard has testing data to support.
4	Add methods of determining mercury in coal by mercury analyzer.	The method is common domestically and is also among the original standard methods.
-	Delete 12 in ISO 15237:2003 (E).	There are requirements for test reports in regulatory standards.

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