

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

HJ 538-2009

**Stationary source emission. Determination of lead. Flame
atomic absorption spectrometry**

固定污染源废气 铅的测定 火焰原子吸收分光光度法(暂行)

(English Translation)

Issued on: December 30, 2009

Implemented on: April 1, 2010

Issued by: Ministry of Ecology and Environment

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Foreword

To protect the environment and protect people in order to implement the Environmental Protection Law of the People's Republic of China and the Law of the People's Republic of China on the Prevention and Control of Air Pollution this standard is formulated for the health monitoring and monitoring methods for the determination of lead in waste gas from fixed sources.

This standard specifies the flame atomic absorption spectrophotometric method for the determination of lead in exhaust gas from stationary sources.

This standard was formulated by the Science and Technology Standards Department of the Ministry of Environmental Protection.

This standard was drafted. Beijing Environmental Protection Monitoring Center.

This standard was approved by the Ministry of Environmental Protection on December 30, 2009.

This standard has been implemented since April 1, 2010.

This standard is explained by the Ministry of Environmental Protection.

Determination of lead in fixed pollution source Flame atomic absorption spectrophotometry (provisional)

1 Scope of application

This standard specifies the flame atomic absorption spectrophotometric method for the determination of lead in exhaust gas from stationary sources.

This standard applies to the determination of lead in fixed source pollution.

The detection limit of the method is 5 µg/50 ml sample solution. When the sampling volume is 400 L, the detection limit is 0.013 mg/m³, and the lower limit of determination is 0.052 mg/m³.

2 Normative references

The contents of this standard refer to the following documents or their terms. For undated references, the valid version applies to this standard.

GB/T 16157 Determination of particulate matter in fixed pollution source exhaust gas and sampling method of gaseous pollutants HJ/T 373 Technical Specifications for Quality Assurance and Quality Control of Fixed Pollution Source Monitoring (Trial) GB/T 6682 Analytical laboratory water specifications and test methods

3 Principle of the method

The exhaust gas sample is collected by a quartz fiber filter cartridge, digested to prepare a sample solution, and the lead solution in the sample solution is determined by an atomic absorption spectrophotometer.

concentration.

4 Interference and elimination

Fe³⁺, Al³⁺, Be³⁺, Cr³⁺, Cd²⁺, Cu²⁺, Zn²⁺, Co²⁺, Hg²⁺, Sn²⁺, Mn²⁺, Mg²⁺, Ag over 100 times lead the plasma does not interfere with the measurement and SiO₃²⁻ is slightly disturbed. Na, K, and Ca²⁺ are slightly sensitized. When the concentration is high, the dilution method can be used.

Eliminate interference.

5 Reagents and materials

Unless otherwise stated, analytically pure reagents that meet national standards were used for the analysis; experimental water, GB/T 6682, grade 2.

5.1

Nitric acid. $\rho(\text{HNO}_3) = 1.42 \text{ g/ml}$, excellent grade pure.

5.2

Perchloric acid. $\rho(\text{HClO}_4) = 1.67 \text{ g/ml}$, excellent grade pure.

5.3

Hydrogen peroxide. $w(\text{H}_2\text{O}_2) = 30\%$, excellent grade pure.

5.4

Nitric acid solution. $\phi(\text{HNO}_3) = 1\%$.

Formulated with nitric acid (5.1).

5.5

Nitric acid solution. 1 1.

Formulated with nitric acid (5.1).

5.6

Lead standard stock solution. $\rho(\text{Pb}) = 1.00 \text{ mg/ml}$.

Weigh 0.500 0 g of metal lead (99.99%) in a 100 ml beaker and dissolve it with 15 ml of (1 1) nitric acid solution (5.5). After cooling, Transfer to a 500 ml volumetric flask and dilute to the mark with water.

Commercially available certified standard solutions can also be used as lead standard stock solutions.

5.7

Lead standard use solution. $\rho(\text{Pb}) = 100 \mu\text{g/ml}$.

The lead standard stock solution (5.6) was diluted with 1% HNO_3 (5.4) to a standard stock solution containing 100 μg lead per ml.

5.8

Acetylene. The purity is not less than 99.6%.

6 Instruments and equipment

Unless otherwise stated, Class A glassware in accordance with national standards was used for the analysis.

6.1

Smoke sampler. The flow range is 5~50 L/min.

6.2

Quartz fiber filter cartridge.

Note: If the filter cartridge blank value is high, it can be soaked with hot (1 1) nitric acid solution (5.5) for about 3 h before use (can not be boiled, so as not to damage the filter cartridge). From acid After taking out, soak it in water for 10 min, remove it with water and wash it to near neutrality. After drying, it can be used.

6.3

Atomic absorption spectrophotometer.

6.4

Electric heating plate.

7.1 Sample collection

Sample collection was performed in accordance with GB/T 16157. When the temperature is below 400 °C, it is sampled at the same speed in the pipeline. When the temperature is higher than 400 °C, lead in the presence of gas, the exhaust gas should be led out of the pipeline to lower the temperature to below 400 °C, and sample at a constant flow of 20 L/min for 10 to 30 minutes.

7.2 Preservation of samples

After the sample of the filter cartridge is collected, the seal is folded inward, placed vertically in the original sampling box, and placed in a desiccator for storage.

7.3 Preparation of samples

Cut the filter cartridge into small pieces, place in a 250 ml Erlenmeyer flask, add (1 1) nitric acid solution (5.5) 50 ml and 30% hydrogen peroxide (5.3) 15 ml immersion sample filter cartridge, the bottle mouth was inserted into a small funnel, heated to a slight boiling on the hot plate, and kept slightly boiling for 2 h. Carefully add 30% after cooling Hydrogen peroxide (5.3) 5 ml, if necessary, add a small amount of water, then heat on a hot plate to a slight boiling, keep boiling for 1 h. Filter after cooling, the filtrate was filtered into a beaker, and the conical flask and the filter residue were washed three times with water, and the washing liquid was combined with the filtrate. Place the beaker containing the filtrate on the