

CCS Z 15

HJ

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

HJ 685-2014

**Stationary source emission. Determination of lead. Flame
atomic absorption spectrophotometric method**

固定污染源废气 铅的测定 火焰原子吸收分光光度法

(English Translation)

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Table of Contents

Preface	.II
1. Scope	.1
2 Normative references	
3 .1 The principle of the method	
4 And eliminate interference	.1
5 Reagents and materials	.1
6 .2 instruments and equipment	
Sample	2

Foreword

To implement the "People's Republic of China Environmental Protection Law", "People's Republic of China Air Pollution Prevention Law," to protect the environment, Protection of human health, standardized method for measuring the exhaust gas from stationary sources of lead in the development of this standard.

This standard specifies the determination of the exhaust particulate matter from stationary sources of lead by flame atomic absorption spectrophotometry.

This standard is the first release.

This standard is developed by the Ministry of Environmental Protection Science, Technology organization.

This standard is mainly drafted by. China National Environmental Monitoring Center.

The standard verification unit. Anhui Provincial Environmental Monitoring Center Station, Henan Province Environmental Monitoring Center, Zhejiang Environmental Monitoring Heart, Chongqing Municipal Environmental Monitoring Center, Jiangsu Provincial Environment Monitoring Center, Nanjing Environmental Monitoring Center.

This standard MEP January 13, 2014 for approval.

This standard since April 1, 2014 implementation.

The standard explanation by the Ministry of Environmental Protection.

Stationary source emission flame atomic absorption spectrophotometry lead WARNING. Lead and its compounds belong to toxic substances, the test should be carried out in a well-ventilated environment.

1 Scope

This standard specifies the determination of the exhaust particulate matter from stationary sources of lead by flame atomic absorption spectrophotometry.

This standard applies to the determination of the exhaust particulate matter from stationary sources of lead.

When the sample size is 0.5 m³, volume of 50.0 ml, the detection limit of this method was the standard 1.0 x 10⁻² mg/m³, Detection limit of 4.0 x 10⁻² mg/m³.

2 Normative references

This standard refers to the contents of the following documents or the terms. For undated references, the latest valid version this is suitable for this standard.

GB/T 16157 Stationary source emission-Determination of particulates and gaseous pollutants sampling methods HJ/T 373 from stationary sources to monitor quality assurance and quality control techniques (Trial) HJ/T 48 dust sampling technology

3 PRINCIPLE OF THE METHOD

Quartz fiber cartridge collection stationary source emissions of particulate matter, nitric acid - hydrogen peroxide digestion to prepare a solution.

Solution of lead in the air - acetylene flame atomic ground state atoms of lead hollow cathode lamp emission characteristic lines (283.3 nm) of laser light. Within a certain range, the absorbance and mass concentration is linear.

4 and elimination of interference 500mg/L of iron, aluminum, bismuth, chromium, cadmium, copper, zinc, cobalt, tin, manganese, magnesium, silver, sodium, potassium, and calcium silicate the determination of no significant interference.

5 Reagents and materials

The reagents used in this standard, unless otherwise noted, are in line with national standards for the use of analytical reagent analysis, test water Deionized water or equivalent purity.

5.1

nitric acid. $\rho(\text{HNO}_3) = 1.42 \text{ g/ml}$, pure class distinctions.

5.2

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

5.3

nitric acid solution, 199.

5.4

nitric acid solution, 11.

5.5 Lead standard stock solution. $\rho(\text{Pb}) = 1000 \text{ mg/L}$

Accurately weigh 1.0000 g spectroscopically pure metallic lead placed in 100ml beaker, add nitric acid in a fume hood (5.1) 15 ml, Slowly heated on a hot plate until completely dissolved, nitric acid solution (5.3) and dilute to 1000ml. Into polyethylene plastic Bottle, stored in the refrigerator, at least stable for one month. It can also be purchased commercially available certified reference materials/substances.

5.6 Lead standard solution. $\rho(\text{Pb}) = 100 \text{ mg/L}$

Imbibe lead standard stock solution (5.5) 10.00 ml to 100 ml volumetric flask, dilute to the mark with a solution of nitric acid (5.3), Mix well.

6 Instruments and equipment

Unless otherwise indicated, all using the national standard of Class A glassware analysis.

6.1 Flame atomic absorption spectrophotometer

6.2 Lead hollow cathode lamp.

6.3 dust sampler, sampling flow rate (5 ~ 80) L/min, performance and other technical indicators in line with the provisions of HJ/T 48's.

6.4 Quartz fiber cartridge

Lead content of not more than 5 μg . Barrier efficiency particle size greater than 3 μm is not less than 99.9%.

6.5 thermostat electric hot plate, (40 ~ 200) $^{\circ}\text{C}$.

6.6 filtration device, 0.45 μm cellulose acetate material or similar properties of the membrane.

6.7 Teflon scissors.

6.8 General Commonly used laboratory instruments and equipment.

7 Sample

Samples collected 7.1 GB 16157 in accordance with the relevant requirements, sample collection using the isokinetic sampling method.

7.2 Field blank

With each sample batch taking at least two cartridges, sampling site to site as a blank sample.

Save 7.3 sample

Sampling is completed, carefully remove the cartridge seal inwardly folded back into the original cartridge box vertically, back to the lab for analysis.

7.4 Preparation of the sample

PTFE scissors Shredded cartridge, into 250 ml Erlenmeyer flask, moistened with a little water, add 50 ml Nitric acid solution (5.4), 15 ml of hydrogen peroxide (5.2), inserted into the small funnel, a hot plate heated to a boil, held 2 h, to be cooled and then carefully added dropwise 5 ml of hydrogen peroxide (5.2), can be supplemented with a small amount of water, if necessary, continue to boil for half an hour, cooling After filtration. The filtrate was transferred to a beaker, the above solution of nitric acid (5.3) washing the flask, residues and suction flask three times. wash After the wash liquid and the filtrate were combined, evaporated on a hot plate to boil nearly dry. Then add 2 ml of nitric acid solution (5.4), heating residue Residue is dissolved, all transferred to a 50 ml volumetric flask with water to volume (V), shake test.

7.5 Preparation of the laboratory sample blank

Take the same batch number two blank cartridge, 7.4 reference laboratory method for preparing a blank sample.

Analysis Step 8

8.1 Reference measurement instrument conditions

Reference instrument conditions were as follows.

Wavelength. 283.3 nm; slit width. 0.5 nm; the flame type. air - acetylene; burner height. 8 mm.

8.2 Calibration

Take seven 100 ml flask, were added to the lead standard solution (5.6) 0.00,0.50,1.00,2.00,4.00, 8.00 and 10.00 ml, then diluted to the mark with a solution of