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HJ

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

HJ 688-2019

**Stationary source emission--Determination of hydrogen
fluoride--Ion chromatography**

固定污染源废气 氟化氢的测定 离子色谱法

(English Translation)

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Foreword

In order to implement the "Environmental Protection Law of the People's Republic of China" Environment, protect human health, standardize the determination method of hydrogen fluoride in exhaust gas from fixed pollution sources, and formulate this standard.

This standard specifies ion chromatography for the determination of hydrogen fluoride in exhaust gas from stationary sources.

This standard is for the determination of hydrogen fluoride in exhaust gas from stationary sources by ion chromatography (Interim) (HJ 688-2013) Revision.

This standard was first published in 2013. The original drafting unit was Dongying City Environmental Monitoring Station. This is the first revision.

The main contents of the amendment are as follows.

- The scope of application was modified, and the method detection limit and determination lower limit were changed;
- Revised normative references;
- Improved sample collection, processing and storage methods;
- Refine the chromatographic reference conditions and increase the ion chromatogram of the target component;
- Revised the result expression formula and result expression requirements;
- Improved the terms of quality assurance and quality control and precautions;
- Added warnings, terms and definitions, interference and elimination, precision and accuracy clauses.

From the date of implementation of this standard, the original standard "Determination of hydrogen fluoride in stationary pollution sources by ion chromatography (interim)" (HJ

688-2013) Repealed.

This standard is formulated by the Department of Eco-Environmental Monitoring, Laws and Standards Department of the Ministry of Ecology and Environment.

This standard was drafted. Shandong Dongying Ecological Environment Monitoring Center, China University of Petroleum (East China).

Verification units of this standard. Shandong Ecological Environment Monitoring Center, Shandong Tai'an Ecological Environment Monitoring Center, Shandong Province Bo Ecological Environment Monitoring Center, Shandong Weifang Ecological Environment Monitoring Center, China University of Petroleum (East China) Dongying Zhongshi University Petroleum and Petrochemical Testing and Evaluation Center and Qingdao Institute of Bioenergy and Process, Chinese Academy of Sciences.

This standard was approved by the Ministry of Ecology and Environment on December 31, 2019.

This standard will be implemented from June 30, 2020.

This standard is explained by the Ministry of Ecology and Environment.

1 Determination of hydrogen fluoride in exhaust gas from stationary sources Ion chromatography Warning. Hydrogen fluoride is harmful to the human body. Take precautions when taking samples to avoid inhalation or contact with skin and eyes. Make use of the sodium hydroxide and potassium hydroxide used are strongly corrosive. Reagent preparation and sample preparation should avoid contact with skin and Clothing.

1 Scope

This standard specifies ion chromatography for the determination of hydrogen fluoride in exhaust gas from stationary sources.

This standard applies to the determination of hydrogen fluoride in exhaust gas from stationary pollution sources.

When the sampling volume is 20 L (standard state) and the constant volume is 100 ml, the method detection limit is 0.08 mg/m³.

The lower limit of determination is 0.32 mg/m³.

2 Normative references

This standard refers to the following documents or clauses therein. For undated references, the valid version applies to this standard.

GB/T 16157 Determination of particulate matter and sampling of gaseous pollutants in exhaust from stationary pollution sources HJ/T 47 Technical Specifications of Flue Gas

Sampler HJ/T 48 Soot Sampler Technical Specifications HJ/T 397 Fixed source exhaust gas monitoring technical specifications

3 Terms and definitions

The following terms and definitions apply to this standard.

Hydrogen fluoride

The hydrogen fluoride measured in this standard refers to inorganic fluorides such as hydrogen fluoride and silicon tetrafluoride (as fluorine Hydrogen meter).

4 Method principle

The heated sampling tube is used to collect the exhaust gas sample, and the particulate matter, gaseous hydrogen fluoride and gasified hydrogen fluoride are removed by a filtering membrane.

The droplets are absorbed by the alkaline absorbent to generate fluoride ions. The sample is injected into the ion chromatograph for separation and detection.

Quantitatively, peak area or peak height.

25 Interference and cancellation

5.1 Particulate fluoride interferes with the measurement. Use a filter to remove it during sampling.

5.2 Acetate ions interfere with the determination of fluoride. You can adjust the eluent concentration and flow rate, and replace the high-efficiency special chromatography.

Column and other methods to eliminate and reduce its interference.

6 Reagents and materials Unless otherwise stated, all analytical reagents in accordance with national standards are used in the analysis. The resistivity of experimental water is $\geq$

18 Mohm · cm deionized water.

6.1 Potassium hydroxide (KOH). excellent grade pure.

6.2 Sodium hydroxide (NaOH). excellent grade pure.

6.3 Sodium carbonate (Na_2CO_3). Excellent grade. It should be dried at $105^\circ\text{C} \pm 5^\circ\text{C}$ and constant weight before use and stored in a desiccator.

6.4 Sodium bicarbonate (NaHCO_3). Excellent grade. It should be placed in a desiccator for 24 h before use.

6.5 Sodium fluoride (NaF). Excellent grade. It should be dried at $105^\circ\text{C} \pm 5^\circ\text{C}$ before use

and kept in a desiccator.

6.6 Aqueous solution absorption solution. $c(\text{KOH}) = 30 \text{ mmol/L}$ or $c(\text{NaOH}) = 30 \text{ mmol/L}$.

Weigh 1.68 g of potassium hydroxide (6.1) or 1.20 g of sodium hydroxide (6.2), dissolve in an appropriate amount of water and transfer to 1000 ml in a volumetric flask, dilute to volume with water, mix well, and transfer to a polyethylene bottle. Provisional use.

6.7 Eluent. Prepare according to the instrument model and the operating conditions of the column manual. The eluent conditions given below are provided reference.

6.7.1 Carbonate eluent. $c(\text{Na}_2\text{CO}_3) = 3.2 \text{ mmol/L}$, $c(\text{NaHCO}_3) = 1.0 \text{ mmol/L}$.

Accurately weigh 0.6784 g of sodium carbonate (6.3) and 0.1680 g of sodium bicarbonate (6.4), respectively, and dissolve them in an appropriate amount of water.

Transfer the volume to a 2000 ml volumetric flask, dilute to volume with water, and mix.

6.7.2 Hydroxide eluent. Generated by the eluent automatic electrolytic generator online.

Note: The eluent should be degassed before use to prevent air bubbles from entering the ion chromatography system.

6.8 Fluoride stock solution. $\rho(\text{F}^-) = 500 \text{ mg/L}$.

Accurately weigh 1.1053 g of sodium fluoride (6.5), dissolve it in an appropriate amount of water, transfer it to a 1000 ml volumetric flask, and dilute with water. Make up to volume, mix well, transfer to a polyethylene bottle, and refrigerate at 4°C for a month.

They can also be formulated using certified standard solutions.

6.9 Fluoride standard solution. $\rho(\text{F}^-) = 50 \text{ mg/L}$.

Pipette 10.00 ml of sodium fluoride stock solution (6.8), transfer to a 100 ml volumetric flask, dilute to volume with water, mix well, Store at room temperature for 14 days.

6.10 Filter membrane. Teflon material, the retention efficiency for particles larger than $0.3 \mu\text{m}$ is not less than 99.9%.

7 instruments and equipment

7.1 Constant temperature heating sampling tube A filter membrane (6.10) is installed at the end of the constant temperature heating sampling tube. The heating temperature is $120^\circ \text{C} \pm 5^\circ \text{C}$ and the temperature control accuracy is 1°C . Sampling tube 3 is made of polytetrafluoroethylene or titanium alloy, and the inner surface is smooth.

7.2 Flue gas sampler the flue gas sampler shall meet the technical requirements of HJ/T 47.

7.3 Smoke Sampler

The dust sampler should meet the technical requirements of HJ/T 48 and be equipped with