

INDUSTRY STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

HJ 999-2018

**Solid waste - Determination of fluoride - Alkali fusion - ion
selective electrode method**

固体废物 氟的测定 碱熔-离子选择电极法

(English Translation)

Issued on: December 26, 2018

Implemented on: June 1, 2019

Issued by: Ministry of Ecology and Environment

Table of Contents

Foreword

1 Determination of fluorine in solid wastes Alkali fusion-ion selective electrode method

2 Normative references

3 Terms and definitions

4 Principle of the method

5 Interference and elimination

26 Reagents and materials

Foreword

In order to implement the "Environmental Protection Law of the People's Republic of China" and the "Law of the People's Republic of this standard is formulated to protect the ecological environment, ensure human health, and regulate the determination of fluorine in solid waste.

This standard specifies the alkali fusion-ion selective electrode method for the determination of fluorine in solid waste.

This standard is the first release.

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

This standard was drafted. Tianjin Ecological Environment Monitoring Center.

The verification unit of this standard. Yunnan Environmental Monitoring Center Station, Hunan Environmental Monitoring Center Station, Shandong Province Environmental Monitoring Xinzhan, Nanjing Environmental Monitoring Center Station, Tianjin Huanke Testing Technology Co., Ltd. and Tianjin Municipal Domestic Waste Inspection heart.

This standard is approved by the Ministry of Ecology and Environment on December 26, 2018.

This standard has been implemented since June 1, 2019.

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

1 Determination of fluorine in solid wastes Alkali fusion-ion selective electrode method

Warning. The hydrochloric acid used in the experiment is highly volatile and corrosive, and the sample preparation process should be carried out in a fume hood;

Wear protective equipment as required to avoid direct contact with skin and clothing.

1 Scope of application this standard specifies the alkali fusion-ion selective electrode method for the determination of fluorine in solid waste.

This standard applies to the determination of fluorine in solid waste.

When the sample is solid or can be dried in a semi-solid state, the sample volume is 0.25 g, and when 10.0 ml of the digestion solution is taken, the side is measured.

The detection limit of the method for determining fluorine is 0.03 g/kg, and the lower limit of determination is 0.12 g/kg. When the sample is liquid or semi-solid without drying State, the sampling amount is 10 g, and when the 10.0 ml digestion solution is removed, the detection limit of the method for measuring fluorine is 0.5 mg/kg.

The lower limit of determination is 2.0 mg/kg.

2 Normative references

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

HJ/T 20 Industrial solid waste sampling and preparation technical specifications HJ/T 298 Hazardous Waste Identification Technical Specification

3 Terms and definitions

The following terms and definitions apply to this standard.

Fluoride

Refers to the fluoride (measured as F) measured after the sample has been extracted by sodium hydroxide under the conditions specified in this standard.

4 Principle of the method

The fluorine in the sample is extracted by high-temperature melting of sodium hydroxide, and is separated by fluorine at a certain pH range and total ionic strength.

The sub-selective electrode method determines that the logarithm of the fluoride ion concentration in the solution is linear with the electrode potential within a certain concentration range.

5 Interference and elimination

When the concentration of Al^{3+} in the sample is higher than 60 mg/L and the concentration of Fe^{3+} is higher than 150 mg/L, the determination of fluorine is negative.

Disturbance can be eliminated by dilution.

26 Reagents and materials

Analytically pure reagents in accordance with national standards were used for analysis, unless otherwise stated. The experimental water was deionized water.

6.1 Hydrochloric acid. $\rho(\text{HCl}) = 1.19 \text{ g/ml}$.

6.2 Sodium hydroxide (NaOH).

6.3 Sodium citrate ($\text{Na}_3\text{C}_6\text{H}_5\text{O}_7 \cdot 2\text{H}_2\text{O}$).

6.4 Citric acid ($\text{C}_6\text{H}_8\text{O}_7$).

6.5 Bromocresol purple ($\text{C}_{21}\text{H}_{16}\text{Br}_2\text{O}_5\text{S}$).

6.6 Sodium fluoride (NaF).

Bake in an oven at $105^\circ\text{C} \sim 110^\circ\text{C}$ for 2 h, placed in a desiccator, cooled to room temperature, and set aside.

6.7 Sodium hydroxide solution. $c(\text{NaOH}) = 5 \text{ mol/L}$.

Weigh 100 g of sodium hydroxide (6.2), dissolve in water, dilute to 500 ml, and mix.

6.8 Sodium hydroxide solution. $c(\text{NaOH}) = 0.2 \text{ mol/L}$.

Weigh 0.8 g of sodium hydroxide (6.2), dissolve in water, dilute to 100 ml, and mix.

6.9 Hydrochloric acid solution. 1 L.

Measure 100 ml of hydrochloric acid (6.1) and dilute to 200 ml with water.

6.10 Total ionic strength adjustment buffer solution (TISAB).

Weigh 294 g of sodium citrate (6.3) and 26 g of citric acid (6.4), add about 900 ml of water to dissolve, and dissolve with hydrochloric acid.

Adjust the pH to about 5.5 with liquid (6.9) or sodium hydroxide solution (6.7), dilute to 1000 ml, and mix. Stored in polyethylene the bottle is stored in a refrigerator at 4°C , taken out at the time of use, and used after being placed at room temperature.

6.11 Fluorine standard stock solution. $\rho(\text{F}) = 1000 \text{ mg/L}$.

Accurately weigh 0.22 g (accurate to 0.1 mg) of sodium fluoride (6.6), dissolve in water, and transfer to a 100 ml volumetric flask.

Dilute to the mark with water, mix and store in a polyethylene bottle. Commercially available certified reference materials are also available.

6.12 Fluorine standard use solution. $\rho(F) = 50.0 \text{ mg/L}$.

Accurately remove 5.00 ml of fluorine standard stock solution (6.11), place in a 100 ml polyethylene volumetric flask, and dilute to the standard with water.

Line, mix.

6.13 bromocresol purple indicator. $\rho(C_{21}H_{16}Br_2O_5S) = 0.4 \text{ g/L}$.

Weigh 0.10 g of bromocresol purple (6.5), dissolve in 10 ml of sodium hydroxide solution (6.8), and dilute to 250 ml with water.

6.14 Qualitative filter paper.

7 Instruments and equipment

7.1 Ion meter or acidity meter. resolution 0.1 mV.

7.2 Fluoride ion selective electrode and reference electrode (or fluoride ion composite electrode).

7.3 Magnetic stirrer and polyethylene magnetic stirrer.

7.4 Muffle furnace. room temperature $\sim 800^\circ \text{C}$.

7.5 Electric heating plate. It has temperature control function and can set the temperature not lower than 200°C .

7.6 Ultrasonic cleaner. frequency (40 kHz~60 kHz), temperature can be displayed.

7.7 Nickel. 50 ml or 100 ml with lid.

37.8 Sampling bottle. 250 ml polyethylene bottle.

7.9 Polyethylene beaker. 100 ml.

7.10 Balance. The sensitivity is 0.01 g.

7.11 Analytical balance. The sensitivity is 0.1 mg.

7.12 Experimental sieve. 0.15 mm in diameter (100 mesh).

7.13 Common instruments and equipment used in general laboratories.

8 samples

8.1 Sample collection and preservation Solid waste samples were collected in accordance with the relevant regulations of HJ/T 20 and HJ/T 298. The collected sample is in the sampling bottle Sealed in (7.8).

8.2 Preparation of samples