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HJ

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

HJ 873-2017

**Soil. Determination of water soluble fluoride and total fluoride.
Ion selective electrode method**

土壤 水溶性氟化物和总氟化物的测定 离子选择电极法
(English Translation)

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Foreword

In order to carry out "Environmental Protection Law of the People's Republic of China", protect the environment, protect human health, standardize the soil water-soluble fluorine Determination of total fluoride and chemical compounds, the development of this standard.

This standard specifies the soil water-soluble fluoride and fluoride total ion selective electrode method.

Appendix A of this standard is an informative annex.

This standard is released for the first time.

This standard by the Environmental Protection Department of Environmental Monitoring Division and Science and Technology Standards Division to develop.

This standard was drafted. Nanjing Environmental Monitoring Center Station.

This standard verification unit. Taizhou Environmental Monitoring Center Station, Wuxi City Environmental Monitoring Center Station, Nantong City Environmental Monitoring Heart Station, Jiangsu Province Environmental Monitoring Center, Jiangsu Provincial Institute of Geological Survey and Physical and Chemical Testing Center of Jiangsu Province.

This standard MEP approved on November 28, 2017.

This standard since January 1, 2018 come into operation.

This standard is interpreted by the MEP.

1 Soil water-soluble fluoride and total fluoride determination Ion-selective electrode method

Warning. Hydrochloric acid used in the experiment has strong volatility and corrosivity, the process should be carried out in a fume hood, the operation should be as required, wear protective equipment to avoid contact with skin and clothing.

1 scope of application this standard specifies the determination of soil water-soluble fluoride and fluoride ion selective electrode method.

This standard applies to soil water-soluble fluoride and total fluoride determination.

When the sample amount of 5.0 g, sample removal amount of 40.0 ml, the standard method for determination of water-soluble fluoride detection limit of 0.7 mg/kg, the lower limit of determination is 2.8 mg/kg and the upper limit of determination is 125 mg/kg. When the sample weight is 0.2 g and the sample removal amount is 20.0 ml, The detection limit of total fluoride was 63 mg/kg, the lower limit of determination was 252 mg/kg and the upper limit of determination was 1.25×10^4 mg/kg.

2 Normative references

This standard references the following documents or the terms of them. For undated references, the effective version applies in this standard.

HJ 613 Determination of soil dry matter and moisture Gravimetric method HJ/T 166 Soil Environmental Monitoring Technical Specifications

3 Terms and definitions

The following terms and definitions apply to this standard.

Water-soluble fluoride water soluble fluoride Under the experimental conditions specified in this standard, water was extracted from soil samples measured fluoride (fluoride).

Total fluoride

Under the experimental conditions specified in this standard, the fluorides (in terms of fluorine) of the soil samples are extracted by alkali high temperature melting.

4 Method principle

The water-soluble fluoride in the soil is extracted with water and the total fluoride is extracted with an alkali-melting method. The total ionic strength Adjusting the buffer solution, using fluoride ion selective electrode method, the fluoride ion activity in the logarithm of the electrode potential was linear.

25 Interference and elimination

Al^3 , Fe^3 , Ca^{2+} , Mg^{2+} and other metal ions easily form a complex with fluoride ions, resulting in negative interference, the interference range the degree depends on the type of metal ions, the concentration and pH of the solution. Under the experimental conditions specified in this standard, add the total ion strong Degree adjustment buffer solution to eliminate interference.

6 Reagents and materials Unless otherwise specified, the analysis of the use of analytical standards in line with national standards of chemical reagents, experimental water resistivity $\geq 18 \text{ Mohm} \cdot \text{cm}$ (25°C) deionized water.

6.1 Sodium hydroxide (NaOH).

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

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

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

6.5 Sodium fluoride (NaF). superior grade pure, dried at $105^\circ \text{C} \sim 110^\circ \text{C}$ 2 h, placed in a desiccator cooled standby.

6.6 hydrochloric acid solution. 1 1.

Measure 50 ml hydrochloric acid (6.2) and dilute to 100 ml with water.

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

Weigh 0.80 g sodium hydroxide (6.1), dissolved in water and diluted to 100 ml.

6.8 bromocresol purple indicator. $w(\text{C}_{21}\text{H}_{16}\text{Br}_2\text{O}_5\text{S}) = 0.04\%$.

Weigh 0.10 g bromocresol purple (6.3), dissolved in 10 ml of sodium hydroxide solution (6.7), diluted with water to 250 ml.

6.9 Total ionic strength adjustment buffer solution (TISAB). 1.0 mol/L trisodium citrate buffer solution.

Weigh 294 g of trisodium citrate (6.4) in a 1000 ml beaker, add about 900 ml of water to dissolve, use hydrochloric acid solution (6.6) Adjust pH to 6.0 $\sim$ 7.0, diluted to 1000 ml, stored in polyethylene bottles.

6.10 Fluorine standard stock solution. $\rho(\text{F}^-) = 500 \text{ mg/L}$.

Weigh 1.1050 g sodium fluoride (6.5), dissolved in water, transferred to a 1000 ml volumetric flask, the volume of water to mark, Shake, stored in polyethylene bottles, refrigerated below 4°C , light-sealed can be stored for 6 months; or purchase a certified public standard solution.

6.11 fluorine standard solution. $\rho(F^-) = 50.0 \text{ mg/L}$.

Pipette 10.00 ml fluorine standard stock solution (6.10), transferred to 100 ml volumetric flask, diluted with water to the mark, shake.

Pro use now with.

7 instruments and equipment

7.1 Ion Meter. Resolution 0.1 mV.

7.2 fluoride ion composite electrode or fluoride ion selective electrode and saturated calomel/silver - silver chloride electrode.

7.3 ultrasonic cleaner. frequency (40 kHz ~ 60 kHz), the temperature can be displayed.

37.4 muffle furnace. room temperature ~ 800 °C.

7.5 Centrifuge. maximum speed of not less than 4000 r/min, with polyethylene/polypropylene centrifuge tube.

7.6 Extraction bottle. polyethylene/polypropylene bottle, 100 ml with lid.

7.7 Beaker. Polyethylene/polypropylene, 100 ml.

7.8 Nickel crucible. 50 ml with lid.

7.9 General laboratory equipment and equipment.

8 samples

8.1 Collection and preservation Collect and store soil samples according to HJ/T 166 requirements.

8.2 Sample Preparation the soil samples were placed in a dry dish, flattened into 2 cm ~ 3 cm thick layer, first remove the plant, insects, stones and other debris, Crush clods with wooden sticks, flip several times a day and air dry naturally.

Take a well-mixed, air-dried sample by quartering and grind over a 2 mm (10 mesh) soil sieve. Take coarse grinding samples, over 0.149 mm (100 mesh) soil sieve into a sample bag or polyethylene vial.

8.3 Determination of dry matter content the dry matter content of soil samples was determined according to the relevant requirements of HJ 613.

8.4 Sample Preparation

8.4.1 Water-soluble fluoride Accurately weigh 5 g (accurate to 0.01 g) of soil sample of 0.149 mm (100 mesh) sieve in extraction flask (7.6), add 50.0 ml of water, shake with a lid, at $25 \text{ }^{\circ}\text{C} \pm 5 \text{ }^{\circ}\text{C}$ water bath temperature ultrasonic extraction 30 min, allowed to stand a few minutes, transferred to a centrifuge tube (7.5), centrifugation 5 min ~ 10 min (speed 4 000