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**INDUSTRY STANDARD OF THE  
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**Soil and sediment. Digestion of total metal elements. Microwave  
assisted acid digestion method**

土壤和沉积物 金属元素总量的消解 微波消解法

**(English Translation)**

**Issued on: July 18, 2017**

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## Table of Contents

Preface

1 Scope of application

2 Normative reference documents

3 Principle of the method

4 Reagents and materials

5 Instruments and equipment

6 Samples

7 Sample digestion .2

8 Precision and accuracy

9 Quality assurance and quality control

10 Precautions

Appendix A (informative) method of precision and accuracy

## Foreword

For the implementation of the "People's Republic of China Environmental Protection Law" to protect the environment, protect human health, regulate soil and sediment Metal element digestion method, the development of this standard.

This standard specifies the microwave digestion of the total amount of 17 metal elements in soil and sediments.

This standard is the first release.

Appendix A to this standard is an informative appendix.

This standard is organized by the Environmental Monitoring Department of the Ministry of Environmental Protection, the Secretary for Science and Technology Standards.

The main drafting unit of this standard. Sichuan Province Environmental Monitoring Station.

The standard verification unit. Nanjing Environmental Monitoring Center Station, Xinxiang City Environmental Protection Monitoring Station, Ningbo City Environmental Monitoring Heart, Panzhihua City Environmental Monitoring Center Station, Chengdu Environmental Monitoring Center Station and Hunan Province Environmental Monitoring Center Station.

The Environmental Protection Department of this standard approved on July 18, 2017.

This standard has been implemented since September 1, 2017.

This standard is explained by the Ministry of Environmental Protection.

Soil and Sediment Metals Total Digestion Microwave Digestion WARNING. The operation of the microwave acid digestion should be carried out in a fume hood and should be covered with protective equipment to avoid contact with the skin and clothing.

## 1 Scope of application

This standard specifies the microwave digestion of the total amount of metal elements in soil and sediments.

The method is applicable to arsenic (As), barium (Ba), beryllium (Be), bismuth (Bi), cadmium (Cd), cobalt (Co) Chromium (Cr), copper (Cu), mercury (Hg), manganese (Mn), nickel (Ni), lead (Pb), antimony (Sb), selenium (Se) Vanadium (V) and zinc (Zn) and other 17 kinds of metal elements of the digestion. If verified, this method also applies to other metal elements the digestion of the total amount of prime.

## 2 Normative reference documents

The contents of this standard refer to the following documents or their terms. Those who do not specify the date of the reference file, the effective version of the appropriate for this standard.

GB 17378.3 Marine monitoring specification Part III. Sample collection, storage and transportation Technical specification for soil environmental monitoring

## 3 Principle of the method

Soil or sediment samples and acid mixture of microwave energy absorption, the acid oxidation activity increased at high temperatures, the metal elements in the sample are released into the solution under high pressure conditions.

## 4 Reagents and materials

Unless otherwise noted, the use of the use of national standards in accordance with the use of excellent pure chemical reagents.

### 4.1

Experimental water. Newly prepared secondary deionized water or sub-boiling distilled water, resistivity  $\geq 18 \text{ Mohm} \cdot \text{cm}$  (25 °C).

### 4.2

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

#### 4.3

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

#### 4.4

hydrofluoric acid.  $\rho(\text{HF}) = 1.16 \text{ g/ml}$ .

#### 4.5

perchloric acid.  $\rho(\text{HClO}_4) = 1.67 \text{ g/ml}$ .

#### 4.6

nitric acid solution. 1 99, with nitric acid (4.2) preparation.

#### 4.7

nitric acid solution. 1 1, with nitric acid (4.2) preparation.

### 5.1 Microwave digestion device

Using a sealed microwave digestion device, can simultaneously carry out a number of sample pretreatment. General power of 400 ~ 1600 W, induction Temperature control accuracy of  $\pm 2.5 \text{ }^\circ\text{C}$ , equipped with microwave digestion tank.

**Note:** The magnetron should be maintained on a regular basis.

5.2 Analysis of the balance. the accuracy of 0.0001 g.

5.3 temperature control heating equipment. temperature control accuracy of  $\pm 5 \text{ }^\circ\text{C}$ .

5.4 General laboratory equipment and equipment commonly used.

### 6.1 Collection and storage of samples

With reference to the relevant provisions of HJ/T 166 for soil samples collected and preserved; with reference to the relevant provisions of GB 17378.3 Collection and storage of sediment samples.

### 6.2 Preparation of samples

According to HJ/T 166 and GB 17378.3, the collected samples were air-dried in the laboratory, broken, 0.15 mm (100 Mesh) nylon sieve, save.

### 7.1 Digestion method one

#### 7.1.1

Suitable for the digestion of thallium, beryllium, barium, manganese, copper, lead, zinc, cadmium, chromium, nickel, cobalt and vanadium.

### 7.1.2 Digestion steps

Weigh the powder, sieve the sample 0.25 ~ 0.5 g (accurate to 0.0001 g) placed in the digestion tank, with a small amount of experimental water (4.1) Wetting. In the acid-proof fume hood, 6 ml of nitric acid (4.2), 3 ml of hydrochloric acid (4.3) and 2 ml of hydrofluoric acid (4.4) were added in this order the sample and digestion solution are well mixed. If there is a strong chemical reaction, until the end of the reaction and then tighten the cover. Digest the digestion tank into digestion After the tank holder is placed in the furnace cavity of the microwave digestion device, it is confirmed that the temperature sensor and the pressure sensor are working properly. According to Table 1 the temperature program is microwave digested and cooled after the end of the program. After the temperature in the tank drops to room temperature, remove the digestion in the anti-acid fume hood Cans, slowly depressurization, open the digestion tank cover.

Table 1 Microwave digestion procedure

Temperature rise time Decomposition temperature holding time 7 min at room temperature 120 °C 3 min 5 min 120 160 °C 3 min 5 min 160 190 °C for 25 min the solution in the digestion tank was transferred to a polytetrafluoroethylene crucible and the digestion tank and lid were washed with a little experimental water (4.1) and then into the crucible. The crucible is placed on a temperature-controlled heating apparatus (5.3) for conducting acid in a slightly boiling state. Until the liquid into a sticky Thick, take a little cold, with a small amount of nitric acid dropper (4.6) rinse the crucible inner wall, the use of residual temperature dissolved in the crucible wall and then transferred to a 25 ml volumetric flask, and then a small amount of nitric acid (4.6) was pipetted to repeat the above procedure. Wash the liquid and transferred into the volumetric flask, and then use nitric acid (4.6) constant volume to the mark, mix, put it aside for 60 min to take the supernatant to be measured.

**Note 1:** If the black residue after microwave digestion, indicating that the carbide is not completely digested. Add 2 ml to the crucible on the thermo-heating device (5.3) Nitric acid (4.2), 1 ml hydrofluoric acid (4.4) and 1 ml perchloric acid (4.5) were added and the reaction was carried out for 30 min in a slightly boiling state.

Continued heating to the perchloric acid white smoke exhausted, the liquid into a sticky shape. The above process is repeated until the black carbide disappears.

**Note 2:** Due to the large amount of organic matter in soil and sediment samples, the amount of nitric acid, hydrochloric acid and hydrofluoric acid used in microwave digestion can be determined according to the actual situation is increased as appropriate.

**Note 3:** When the measured element content in the sample is low, the sample weighed can be increased to 1 g (accurate to 0.0001 g), microwave digestion of nitric acid, hydrochloric acid and the amount of hydrofluoric acid should also be increased in proportion to the actual situation, or increase the number of digestion.