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NATIONAL ENVIRONMENTAL PROTECTION

STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

HJ 780-2015

**Soil and Sediment – Determination of Inorganic Element –
Wavelength Dispersive X-Ray Fluorescence Spectrometry**

土壤和沉积物 无机元素的测定 波长色散 X 射线荧光光谱法

Issued on: December 14, 2015

Implemented on: February 01, 2016

Issued by: Ministry of Environmental Protection

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Soil and Sediment – Determination of Inorganic Element – Wavelength Dispersive X-Ray Fluorescence Spectrometry

1 Scope

This Standard specifies the wavelength dispersive X-ray fluorescence spectrometry method for the determination of 25 inorganic elements and 7 oxides in soil and sediment.

This Standard is application to the determination of 25 inorganic elements and 7 oxides in soil and sediment; including Arsenic (As), Barium (Ba), Bromine (Br), Cerium (Ce), Chlorine (Cl), Cobalt (Co), Chromium (Cr), Copper (Cu), Gallium (Ga), Hafnium (Hf), Lanthanum (La), Manganese (Mn), Nickel (Ni), Phosphorus (P), Lead (Pb), Rubidium (Rb), Sulfur (S), Scandium (Sc), Strontium (Sr), Thorium (Th), Titanium (Ti), Vanadium (V), Yttrium (Y), Zinc (Zn), Zirconium (Zr), Silicon Dioxide (SiO₂), Aluminum Oxide (Al₂O₃), Ferric Oxide (Fe₂O₃), Potassium Oxide (K₂O), Sodium Oxide (Na₂O), Calcium Oxide (CaO), Magnesium Oxide (MgO).

The detection limit of this method for 22 inorganic elements is 1.0mg/kg~50.0mg/kg; the lower limit for determination is 3.0mg/kg~150mg/kg. The detection limit for 7 oxides is 0.05%~0.27%; the determination lower limit is 0.15%~0.81%. See Appendix A for details.

2 Normative References

The content of this Standard refers to the following documents or their clauses. For undated references, their valid versions apply to this Standard.

GB 17378.3 The Specification for Marine Monitoring - Part 3: Sample Collection Storage and Transportation

GB 17378.5 The Specification for Marine Monitoring - Part 5: Sediment Analysis

HJ/T 166 Technical Specification for Soil Environmental Monitoring

3 Principle of Method

After the soil or sediment sample is pressed by a liner or an aluminum ring (or plastic ring), the atoms in the specimen are excited by appropriate high-energy radiation, and then the characteristic X-rays of the atoms are emitted; and the intensity of it is directly proportional to

the mass fraction of the element in the specimen. The mass fraction of each element in the specimen is quantitatively analyzed by measuring the intensity of characteristic X-rays.

4 Interference and Elimination

4.1 The X-ray fluorescence intensity value produced by the atoms of the element to be measured in the specimen after being excited by radiation is related to the mass fraction of the element and the mass absorption coefficient of the primary spectrum. The characteristic spectral line of an element is photoelectrically absorbed by another element in the matrix, which shall produce a matrix effect (that is, the inter-element absorption-enhancement effect). This matrix effect can be eliminated after accurate calculation and processing by the basic parameter method, the influence coefficient method or a combination of the two methods (i.e., the empirical coefficient method) (see Appendix B).

4.2 The uniformity and surface characteristics of the specimen shall affect the measurement intensity of the analytical line. If the particle size of the specimen is consistent with that of the standard sample, then these effects can be minimized or even negligible.

4.3 Use the interference correction coefficient to correct the overlapping interference of spectral lines (see Appendix B). Calculation method of overlapping interference correction coefficient: through element scanning, analyze the interference line related to the analysis line of the element to be measured; and determine the interference elements participating in the spectral line overlapping correction. Use the method of directly measuring the interference line to correct the X-ray intensity by the standard sample, and obtain the spectrum line overlapping correction factor.

5 Reagents and Materials

5.1 Boric acid (H_3BO_3): analytically pure.

5.2 High-density low-pressure polyethylene powder: analytically pure.

5.3 Standard samples: soil, sediment, commercially available certified standard substances or standard samples containing 25 inorganic elements and 7 oxides.

5.4 Plastic ring: inner diameter 34 mm.

5.5 Argon-methane gas: P10 gas, 90% argon + 10% methane.

6 Apparatus

6.1 X-ray fluorescence spectrometer: wavelength dispersion type, equipped with computer

control system, see Appendix C for targets and spectroscopic crystals.

6.2 Powder pressing machine: the pressure is $3.9 \times 10^5 \text{N}$.

6.3 Analytical balance: precision 1 mg.

6.4 Sieve: non-metallic sieve with a hole diameter of 0.075 mm, and 200 mesh.

7 Sample

7.1 Collection, preservation and pretreatment of sample

The collection and preservation of soil samples shall be carried out in accordance with HJ/T 166; and the collection and preservation of sediment samples shall be carried out in accordance with GB17378.3 and GB17378.5. Air-drying or oven-drying of the samples shall be carried out in accordance with the relevant provisions of HJ/T 166 and GB17378.5. After the samples are ground, pass through a 200-mesh sieve and dry at 105°C for later use.

7.2 Preparation of specimen

Use boric acid (5.1) or high-density low-pressure polyethylene powder (5.2) for putting at the bottom and trimming, or use plastic ring (5.4) for trimming; and press about 5 g of the sieved sample (7.1) on a pressing machine at a certain pressure to make it ≥ 7 mm thick slices. Determine the pressure and retention time according to the press machine and the trimming material.

8 Analysis Procedure

8.1 Establishing the measurement method

Refer to the instrument operating procedure to establish the measurement method. According to the determined measurement elements, the measurement spectral lines are selected from the database and corrected. Different models of instruments have different measurement conditions. Refer to the database provided by the instrument manufacturer to select the best working conditions, mainly including the high voltage and current of the X-ray tube, the analysis line of the element, the spectroscopic crystal, the collimator, the detector, the pulse height distribution (PHA), background correction. Appendix C gives the working conditions of some instrumental analysis.

8.2 Calibration curve

Following the same operation procedure as the sample preparation (7.2), press at least 20 standard samples (5.3) elements with different mass fraction into thin slices. See Appendix D for the mass fraction ranges of 25 inorganic elements and 7 oxides. Under the optimal working

10 Precision and Accuracy

10.1 Precision

Six laboratories analyzed and measured the national certified standard samples (soil, stream sediment) and actual samples (soil and bottom sediment). The intra-laboratory relative standard deviation is 0.0% ~ 15.7%; the inter-laboratory relative standard deviation is 0.0% ~ 22.8%; the repeatability limit is 0.00 mg/kg ~ 56.5 mg/kg; and the reproducibility limit is 0.08 mg/kg ~ 124 mg/kg. See Appendix E for summary precision data.

10.2 Accuracy

The six laboratories analyzed and measured the national certified standard samples (soil, stream sediment) and actual samples (soil and bottom sediment), and the relative error of the certified standard substance analysis is -70.2 % to 32.7 %. See Appendix E for summary accuracy data.

11 Quality Assurance and Quality Control

11.1 The measuring instrument shall be regularly drift-corrected, such as replacing the argon-methane gas, when the ambient temperature and humidity change greatly, and starting the instrument after it has been shut down for a long time, etc. The physical and chemical properties of the samples used for drift correction need to be stable. When the drift is too large, the standard curve needs to be redone; and standardized sample with high mass fraction can be used for calibration.

11.2 When analyzing each batch of samples, at least one national certified standard substance of soil or sediment shall be determined; and the relative error between the measured value and the certified standard substance is shown in Table 1.

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