

HJ

INDUSTRY STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

HJ 1211-2021

**Solid waste - Determination of inorganic elements - Wavelength
dispersive X-ray fluorescence spectrometry**

固体废物 无机元素的测定 波长色散 X 射线荧光光谱法

(English Translation)

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1 Scope of application

This standard specifies 16 categories for the determination of sludge, polluted soil, fly ash, soot, tailings waste rock and smelting slag in solid waste.

Wavelength Dispersive X-ray Fluorescence Spectroscopy of Inorganic Elements and 7 Oxides.

This standard applies to 16 types of inorganic solid wastes, such as sludge, polluted soil, fly ash, soot, tailings waste rock and smelting slag.

Determination of elements and 7 oxides including arsenic (As), barium (Ba), chlorine (Cl), cobalt (Co), chromium (Cr), copper (Cu), manganese (Mn), Nickel (Ni), Phosphorus (P),

Lead (Pb), Sulfur (S), Strontium (Sr), Titanium (Ti), Vanadium (V), Zinc (Zn), Zirconium (Zr), Silicon Dioxide (SiO₂), Aluminum oxide (Al₂O₃), iron oxide (Fe₂O₃), potassium oxide (K₂O), sodium oxide (Na₂O), calcium oxide (CaO), oxide Magnesium (MgO).

This standard adopts the molten glass tablet method or powder compression method to prepare solid waste samples. The molten glass sheet method is suitable for sludge, polluted soil, Preparation of solid waste samples such as fly ash, smoke dust, tailings waste rock, smelting slag, etc., but not suitable for the determination of chlorine (Cl) content;

The powder tableting method is suitable for the preparation of solid waste samples such as sludge, polluted soil, fly ash, and soot.

The detection limit of the method for the preparation and determination of 15 inorganic elements by the molten glass sheet method is 5 mg/kg~70 mg/kg, and the lower determination limit is 20 mg/kg~280 mg/kg; the detection limit of 7 oxides is 0.01%~0.03%, and the lower limit of determination is 0.04%~0.12%. powder the detection limit of the method for the preparation and determination of 16 inorganic elements by the uncompressed tablet method is 2 mg/kg~30 mg/kg, and the lower determination limit is 8 mg/kg~120 mg/kg;

The detection limit of the method for 7 oxides is 0.01%-0.03%, and the lower limit of determination is 0.04%-0.12%. See Appendix A for details.

2 Normative references

This standard refers to the following documents or clauses thereof. For dated references, only the dated version applies to this standard.

For undated references, the latest edition (including all amendments) applies to this standard.

HJ/T 20 Technical Specification for Sampling and Sample Preparation of Industrial Solid Waste
HJ 298 Technical Specification for Hazardous Waste Identification

3 Principles of the method

The samples were prepared by the molten glass plate method or the powder pressing method, and each element atom in the sample was excited in a wavelength dispersive X-ray fluorescence spectrometer.

It emits characteristic X-ray spectral lines, whose intensity is proportional to the mass fraction of the element in the sample after the overlap and matrix effect correction.

By measuring the characteristic X-ray intensity of the target in the sample, the mass fraction of each element in the sample can be quantitatively analyzed.

4.1 Matrix interference

The matrix interference in the sample includes the absorption and enhancement effect of the matrix element on the X-ray spectral intensity of the target element. through the empirical coefficient method or Mathematical analysis methods such as the basic parameter method can reduce the influence of this matrix effect after calculation and processing. See Appendix B for common matrix effect correction methods.

4.2 Spectral line overlap interference

During sample analysis, the analytical lines of the target element may be interfered by the lines of other elements in the matrix. Select target elemental analysis the interference of the spectral lines of other elements in the matrix should be avoided when the spectral lines are used, and the spectral line overlap interference correction can also be calculated by analyzing the measurement results of multiple standard samples.

Positive coefficient for noise cancellation. See Appendix B for common spectral line interference and spectral line correction methods.

4.3 Particle effect

When the sample is prepared by the powder compression method, the particle size, inhomogeneity and surface structure of the sample will affect the characteristic X-ray of the target element.

The intensity of spectral lines has an influence, and these factors should be controlled. The particle size of the measured sample should be consistent with that of the standard sample. reduce or eliminate these effects.

5 Reagents and Materials

Unless otherwise stated, analytical reagents that meet national standards were used in the analysis, and the experimental water was freshly prepared deionized water or distilled water.

5.1

Anhydrous lithium tetraborate ($\text{Li}_2\text{B}_4\text{O}_7$). excellent grade pure.

5.2

Anhydrous lithium metaborate (LiBO_2). excellent grade.

5.3

Lithium nitrate (LiNO_3).

5.4

Lithium Bromide (LiBr).

5.5

Boric acid (H_3BO_3) or high density low pressure polyethylene powder.

5.6

Lithium nitrate solution. $\rho(LiNO_3)=220\text{ g/L}$.

Weigh 22.0 g of lithium nitrate (5.3) and dissolve it in an appropriate amount of water. After dissolving, add water to make up to 100 ml, and shake well.

5.7

Lithium bromide solution. $\rho(LiBr)=60\text{ g/L}$.

Weigh 6.0 g of lithium bromide (5.4) and dissolve it in an appropriate amount of water. After dissolving, add water to make up to 100 ml, and shake well.

5.8

Standard samples. commercially available certified standard samples. Including but not limited to soil composition analysis standard material series, water system sediment composition analysis Reference material series, coal ash reference material series, iron ore reference material series, slag reference material series.

5.9

Plastic Rings.

5.10

Argon gas-methane gas. $\phi(Ar)=90\%$, $\phi(CH_4)=10\%$, in terms of volume fraction.

6 Instruments and equipment

6.1

X-ray fluorescence spectrometer. wavelength dispersion type, with computer control system.

6.2

Powder pressing machine. $\text{pressure} \geq 3.9 \times 10^5 \text{ N}$.

6.3

Muffle furnace. can be heated to $800\text{ }^\circ\text{C}$.

6.4

Fusion sample maker. can be heated to $1200\text{ }^\circ\text{C}$.