

HJ

INDUSTRY STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

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**Solid waste. Extraction procedure for leaching toxicity.
Sulphuric acid and nitric acid method
(English Translation)**

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1 Scope

This standard specifies the leaching procedures and quality assurance measures for the leaching toxicity of solid waste.

This standard applies to the identification of leaching toxicity of organic and inorganic substances in solid waste and its recycled products, as well as soil samples. Samples containing non-water-soluble liquids are not applicable to this standard.

2 Terms and definitions

The following terms and definitions apply to this standard.

2.1

leaching

The process by which soluble components dissolve and change from the solid phase to the liquid phase.

2.2

leaching toxicity

When solid waste comes into contact with water, the leached harmful substances migrate and transform, polluting the environment. This hazardous characteristic is called leaching toxicity.

2.3

initial liquid phase

For samples where both liquid and solid phases are evident, the liquid that is obtained through filtration prior to the leaching steps.

3 Principle

This method uses a nitric acid/sulfuric acid mixed solution as an extractant to simulate the process in which harmful components in waste are leached from the waste and enter the environment under the influence of acid precipitation when the waste is disposed of through irregular landfill or storage, or when the waste is reused in the land after non-hazardous treatment.

4 Reagents

4.1

Reagent water

Use pure water that meets the requirements of the analytical method standard of the substance to be tested.

4.2

Sulfuric acid $\rho(\text{H}_2\text{SO}_4) = 1.84 \text{ g/ml}$, guaranteed reagent.

4.3

Nitric acid

$\rho(\text{HNO}_3) = 1.42 \text{ g/ml}$, guaranteed reagent.

4.4 1% nitric acid solution: Prepare by diluting concentrated nitric acid (4.3).

4.5.1 Extractant 1#: Add a mixture of concentrated sulfuric acid and concentrated nitric

acid in a mass ratio of 2:1 to reagent water (about 2 drops of the mixture per 1 L of water) to adjust the pH value to 3.20 ± 0.05 . This extractant is used to determine the leaching toxicity of heavy metals and semi-volatile organic compounds in samples.

4.5.2 Extractant 2#: Reagent water, used to determine the leaching toxicity of cyanide

and volatile organic compounds.

5 Instruments and equipment

5.1

Oscillation equipment

A rotary oscillator with a rotation speed of (30 ± 2) r/min.

5.2.1 Zero-Headspace Extraction Vessel (ZHE): 500~600 ml, a special device used to

leach volatile substances from samples.

5.2.2 Extraction bottle: 2 L wide-mouth bottle with a screw cap and inner cap, used to leach non-volatile and semi-volatile substances in the sample. The extraction bottle shall be made of an inert material that cannot leach or absorb the components contained in the sample. When inorganic substances are analyzed, glass bottles or polyethylene (PE) bottles can be used; when organic substances are analyzed, glass bottles or polytetrafluoroethylene (PTFE) bottles can be used.

5.3.1 Zero-Headspace Extraction Vessel (ZHE): Analyze the volatile substances in the

sample, and use ZHE for filtration.

5.3.2 Vacuum filter or positive pressure filter: with a volume of ≥ 1 L.

5.3.3 Filter membrane: glass fiber filter membrane or microporous filter membrane, tests.

7.2 Sample crushing

The sample particles shall be able to pass through a sieve with an aperture of 9.5 mm.

Large particles can be reduced in size by crushing, cutting or milling.

When determining the volatile organic compounds in a sample, a ruler shall be used to measure the particle size to avoid loss of the components to be measured during sieving; the sample and the tools used to reduce the particle size shall be cooled, and exposure of the sample to the air shall be avoided as much as possible.

7.3 Leaching steps for volatile organic compounds

7.3.1

Cool the sample to 4 °C, weigh 40~50 g of the sample on a dry basis, and quickly transfer it to the ZHE (5.3.1). Install the ZHE and slowly pressurize it to remove the headspace.

7.3.2 When the sample contains an initial liquid phase, connect the leachate collection

device (5.5) to the ZHE, slowly increase the pressure until no more filtrate flows out, collect the initial liquid phase, and store it in a refrigerator.

7.3.3 If the dry solid percentage in the sample is less than or equal to 9%, the initial liquid phase obtained is the leachate and is analyzed directly; if the dry solid percentage is greater than 9% of the total sample volume, continue with the following leaching steps and mix the obtained leachate with the initial liquid phase for analysis.

7.3.4 Calculate the required volume of extractant based on the water content of the sample and the liquid-to-solid ratio of 10:1 (L/kg). Add extractant 2# using the extractant transfer device (5.6), install the ZHE, and slowly pressurize to remove the headspace. Close all valves.

7.3.5 Fix the ZHE on the rotary oscillator (5.1), adjust the speed to (30 ± 2) r/min, and oscillate at (23 ± 2) °C for (18 ± 2) hours. After the oscillation stops, remove the ZHE and check whether the device is leaking (if the ZHE device is leaking, resample for leaching), collect the leachate with the same leachate collection device (5.5) that collected the initial liquid phase, and store it in a refrigerator for analysis.

7.4 Leaching steps for substances other than volatile organic compounds

7.4.1

If the sample contains an initial liquid phase, filter the sample using a pressure filter (5.3.2) and a filter membrane (5.3.3). If the dry solid percentage is less than or equal to 9%, the initial liquid phase obtained is the leachate and is analyzed directly; if the dry solid percentage is greater than 9%, leach from the filter residue according to

7.4.2, and mix the initial liquid phase with the leachate for analysis.

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