

# HJ

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

### HJ 713-2014

---

**Solid wastes. Determination of volatile halohydrocarbons.  
Purge and trap gas chromatography mass spectrometry**

固体废物 挥发性卤代烃的测定 吹扫捕集气相色谱-质谱法

**(English Translation)**

**Issued on: November 27, 2014**

**Implemented on: January 1, 2015**

---

**Issued by: Ministry of Ecology and Environment**

## Table of Contents

|                                      |
|--------------------------------------|
| Foreword                             |
| 1 Scope                              |
| 2 Normative references               |
| 3 PRINCIPLE OF THE METHOD            |
| 4 Reagents and materials             |
| 5 Apparatus                          |
| 6 Samples                            |
| 6.3 Preparation of the sample        |
| 6.4 Preparation of blank samples     |
| 7 Procedure                          |
| 47.1 Instrument Reference conditions |

### Foreword

To implement the "People's Republic of China Environmental Protection Law" and "People's Republic of China Solid Waste Pollution Prevention Law" Protecting the environment, safeguarding human health, specifications determination of volatile halogenated solid waste, the development of this standard.

This standard specifies the determination of solid waste chloride and other 35 kinds of volatile halogenated Purge and Trap - gas Chromatography/Mass Spectrometry.

This standard is the first release.

Appendix A of this standard is a normative appendix, Appendix B and Appendix C are informative appendices.

This standard is developed by the Ministry of Environmental Protection Science, Technology organization.

This standard drafting of the main unit. Harbin Municipal Environmental Monitoring Center.

The standard verification unit. Heilongjiang Provincial Environmental Monitoring Center, Dalian Municipal Environmental Monitoring Center, Changchun Municipal Environmental Monitoring Heart Station, Anshan Municipal Environmental Monitoring Center Station, Qiqihar Municipal Environmental Monitoring Center Station and the Daqing Municipal Environmental Monitoring Center.

This standard MEP November 27, 2014 for approval.

This standard since January 1, 2015 implementation.

The standard explanation by the Ministry of Environmental Protection.

1 Determination of volatile halogenated solid waste Purge and Trap - gas Chromatography/Mass Spectrometry WARNING. The internal standard used in the experiments, the alternatives and the standard solution are volatile, toxic chemicals, preparation process it should be performed in a hood; the preparation of the sample should be considered whether it is hazardous solid wastes; according to the provisions of Seeking to wear protective equipment to avoid contact with skin and clothing.

## 1 Scope

This standard specifies the determination of volatile halogenated solid waste purge and trap/gas chromatography - mass spectrometry.

This standard applies to the determination of solid waste and solid waste leaching solution of chloride and other 35 kinds of volatile halogenated hydrocarbons. Other play If validated onset halogenated also apply to this standard.

The solid waste sample amount of 5 g, 35 kinds of volatile halogenated hydrocarbons detection limit of 0.2 ~ 0.4 µg/kg, detection limit of 0.8 ~ 1.6 µg/kg. The solid waste leachate volume of 5.0 ml, the detection limit was 0.2 ~ 0.4 µg/L, measured at Limit of 0.8 ~ 1.6 µg/L. See Appendix A.

## 2 Normative references

The standard content of the following documents cited in the articles. For undated references, the effective version applies to this standard.

HJ/T 20 industrial solid waste sampling sample preparation Technical Specifications HJ/T 299 methods for leaching toxicity of solid waste sulfuric acid nitric acid method HJ/T 300 solid waste leaching toxicity Acetic acid buffer solution method

## 3 PRINCIPLE OF THE METHOD

Samples of volatile halogenated hydrocarbons purged with helium, adsorption trap tube, the collection tube and heated with helium blowback Trap tube volatile halogenated hydrocarbons are thermally desorbed component after entering gas chromatography with mass spectrometer detection. root According to the retention time, the fragment ions of different mass to charge ratio and ion abundance ratio qualitative and quantitative internal standard.

## 4 Reagents and materials

### 4.1

experimental water. secondary distilled water or pure water preparation equipment. Before using the subject blank check to confirm no object or Target concentrations below detection limit.

### 4.2

methanol (CH<sub>3</sub>OH). pesticide grade, before use by testing to confirm no goal or target concentration is below the detection method limit.

### 4.3

standard stock solution.  $\rho = 2000 \text{ mg/L}$ .

Direct purchase a commercially available certified reference solution below -10 °C from light, or by reference to the manufacturer's product description. Should be used Returned to room temperature and shake well.

### 4.4

standard solution.  $\rho = 2.5 \text{ mg/L}$ .

Take the right amount of stock standard solution (4.3), appropriately diluted with methanol (4.2).

24.5 internal standard stock solution.  $\rho = 2000 \text{ mg/L}$ .

Selection fluorobenzene, 1-chloro-2-bromo-propane, 4-bromo-fluorobenzene as an internal standard. Direct purchase of certified reference solution, also available standard Quality preparation.

### 4.6

internal standard solution.  $\rho = 2.5 \text{ mg/L}$ .

Proper amount of internal standard stock solution (4.5), with methanol (4.2) appropriately diluted.

### 4.7

alternatives stock solution.  $\rho = 2000 \text{ mg/L}$ .

Selection dichloromethane -d<sub>2</sub>,1,2- -d<sub>4</sub> dichlorobenzene as an alternative. Direct purchase of certified reference solution, also available standard Quality preparation.

### 4.8

was used as substitutes.  $\rho = 2.5 \text{ mg/L}$ .

Take appropriate alternatives stock solution (4.7), appropriately diluted with methanol (4.2).

4.9 4-bromo-fluorobenzene (BFB) solution.  $\rho = 25 \text{ mg/L}$ , can be purchased directly certified standard solution can also be used for preparing reference material, Diluted with methanol.

#### 4.10

quartz sand. 20 to 50 mesh. Before use by testing, confirming that no target or targets below the detection limit.

#### 4.11

Helium. purity  $\geq 99.999\%$ , by deoxidizer oxygen, molecular sieve dehydration.

### 5 Apparatus

#### 5.1

Sampling equipment. shovels and stainless steel spatula.

#### 5.2

Sampling bottle. PTFE silicone liner screw cap 60 ml wide-mouth glass jar.

#### 5.3

Vial. Teflon liner with screw cap 40 ml brown glass bottles and colorless.

#### 5.4

Gas chromatography - mass spectrometry. EI ionization source.

#### 5.5

Column. capillary column, length 30 m, an inner diameter of 0.25 mm, a thickness of 1.4  $\mu\text{m}$ , 6% stationary phase phenyl nitrilpropionamide/94 % Dimethyl polysiloxane may be used other equivalent capillary column.

#### 5.6

Purge and Trap Device. for the determination of solid samples and viscous liquid samples. Trap tube using 1/3 Tenax, 1/3 silica gel, 1/3 mixing activated carbon adsorbents or other equivalent adsorbent.

#### 5.7

micro syringe. 10  $\mu\text{l}$ , 25  $\mu\text{l}$ , 100  $\mu\text{l}$ , 250  $\mu\text{l}$ , 500  $\mu\text{l}$  and 1000  $\mu\text{l}$ .

#### 5.8

Balance. accuracy of 0.01 g.

#### 5.9

Reciprocating oscillator. oscillation frequency of 150 times/min, the vial can be fixed.