

HJ

INDUSTRY STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

HJ 714-2014

**Solid wastes. Determination of volatile halohydrocarbons.
Headspace gas chromatography mass method**

固体废物 挥发性卤代烃的测定 顶空气相色谱-质谱法

(English Translation)

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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 headspace/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.

Headspace 1 solid waste volatile halogenated hydrocarbons - 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 solid waste chloride and other volatile halogenated headspace/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 verified onset halogenated hydrocarbons can also be applied to this standard.

The solid waste sample amount of 2 g, 35 kinds of volatile halogenated hydrocarbons detection limit of 2 ~ 3 µg/kg, detection limit is 8 ~ 12 µg/kg; the solid waste leachate volume of 10.0 ml, the detection limit was 0.7 ~ 1.5 µg/L, detection limit of 2.8 to 6.0 µg/L. See Appendix A.

2 Normative references

This standard refers to the contents of the following documents or the terms. For undated references, the effective version suitable for 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

Under certain temperature conditions within the sample vial of volatile halogenated hydrocarbons to the headspace volatiles, have some steamed Pressure and achieve

liquid-solid three phase, vapor samples taken after entering gas chromatography with mass spectrometer detection. according to Retention time fragment ion mass to charge ratio and different ion abundance ratios of qualifier 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 goals or objectives Concentrations below the detection limit.

4.2

methanol (CH₃OH). pesticide grade, before use by testing to confirm no goal or target concentration below detection method the limit.

4.3

phosphoric acid. pure class distinctions.

4.4

Sodium chloride (NaCl). excellent pure, at 400 °C in a muffle furnace bake 4 h, the dryer to cool to room temperature and placed, Stored in a grinding mouth brown glass bottles sealed.

4.5

based modifier. phosphoric acid (4.3) dropwise to a 100 ml test water for adjusting the pH value of less than 2; then add 36 g of sodium chloride (4.4) were mixed. At 4 °C stored for up to 6 months.

4.6

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

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

24.7 standard solution. $\rho = 20 \text{ mg/L}$.

Take the right amount of stock standard solution (4.6) with methanol (4.2) appropriately diluted.

4.8

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.

4.9

internal standard solution. $\rho = 25 \text{ mg/L}$.

The proper amount of the internal standard stock solution (4.8), with methanol (4.2) appropriately diluted.

4.10

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

Selection dichloromethane -d₂,1,2- -d₄ dichlorobenzene as an alternative. Direct purchase of certified reference solution, also available standard Quality preparation.

4.11

alternatives to the use of liquid. $\rho = 25 \text{ mg/L}$.

The appropriate amount of stock solution substitute (4.10), methanol (4.2) appropriately diluted.

4.12 4-bromo-fluorobenzene (BFB) solution. $\rho = 25 \text{ mg/L}$.

It can be purchased directly certified standard solution can also be used in preparing a standard, diluted with methanol.

4.13

quartz sand. 20 to 50 mesh, before use by testing to confirm no goal or target concentration below the detection limit.

4.14

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 bottles. a Teflon liner 60 ml screw brown glass bottle.

5.3

Gas chromatography - mass spectrometry. EI ionization source.

5.4

Column. capillary column, length 30 m, an inner diameter of 0.25 mm, a thickness of 1.4 μm , the stationary phase is 6% nitrilpropionamide phenyl/94% Dimethyl polysiloxane may