

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

HJ 711-2014

**Solid waste-Determination of phenolic compounds. Gas
chromatography**

固体废物 酚类化合物的测定 气相色谱法

(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, standardized monitoring method of solid waste in phenolic compounds, the development of this standard.

This standard provides the solid waste Phenols in Gas Chromatography.

This standard is the first release.

Appendix A of this standard is a normative appendix; Appendix B, Appendix C and Appendix D is an informative annex.

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

This standard is mainly drafted by. Nanjing Environmental Monitoring Center.

The standard verification. Jiangsu Provincial Environmental Monitoring Center, Changzhou Municipal Environmental Monitoring Center, Taizhou City Environment Monitoring Central station, Zhenjiang Municipal Environmental Monitoring Center, Ningbo Municipal Environmental Monitoring Center Station and the physical and chemical testing center in Jiangsu Province.

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 Gas Chromatography, solid waste phenolic compounds

Warning. Solvent and standard solutions used in the experiment on human health hazards, the operation should be carried out in a hood, Prescribed wear protective equipment to avoid skin contact.

1 Scope

This standard provides solid waste and solid waste leachate phenolic compounds was measured by gas chromatography.

This standard applies to the determination of solid waste leachate phenolic compounds in solid waste and 21 species of other phenolic compounds If the validation is also applicable to this standard.

When the sampling of solid waste was 10.0 g, 21 kinds of phenolic compounds method detection limit of 0.02 ~ 0.33 mg/kg, Detection limit of 0.08 ~ 1.32 mg/kg, see Appendix A.

When the sampling of solid waste leachate is 100 ml, 21 kinds of phenolic compounds method detection limit of 0.002 to 0.006 mg/L, detection limit of 0.008 ~ 0.024 mg/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 298 Identification of Hazardous Waste 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 HJ 557 solid waste leaching toxicity level oscillation method

3 PRINCIPLE OF THE METHOD

Solid waste or solid waste leachate extraction with a suitable organic solvent, the extract was purified acid distribution, phenolic compounds Substance into the aqueous phase, the aqueous phase was adjusted to acidic phase, extract was dehydrated, concentrated organic solvent extraction with a suitable water volume Backward by gas chromatography, FID measurement, retention time qualitative, external standard.

4 Reagents and materials

Unless otherwise indicated, the use of chemical reagents analytical line with national standards of analysis, the experiment doubly distilled water 2 prepared by water or water

equipment.

4.1

Sodium hydroxide (NaOH).

4.2

hydrochloric acid (HCl). $\rho = 1.19 \text{ g/ml}$.

4.3

anhydrous sodium sulfate (Na_2SO_4). baking at 400°C for 4 h, cooled to room temperature and placed in a desiccator, was transferred to a glass grinding mouth Glass bottle and stored in a desiccator.

4.4

Sodium hydroxide (NaOH) solution. $c(\text{NaOH}) = 5 \text{ mol/L}$.

Weigh 20 g of sodium hydroxide (4.1), dissolved in water after cooling and dilute to 100 ml.

4.5

hydrochloric acid solution. $c(\text{HCl}) = 3 \text{ mol/L}$.

Measure 125 ml of hydrochloric acid (4.2), diluted to 500 ml.

4.6

dichloromethane (CH_2Cl_2). chromatography.

4.7

ethyl acetate ($\text{CH}_3\text{COOC}_2\text{H}_5$). chromatography.

4.8

methanol (CH_3OH). chromatography.

4.9

n-hexane (C_6H_{14}). chromatography.

A mixed solvent of ethyl acetate and dichloromethane 4.10. 41.

4.11

methylene chloride mixed solvent of n-hexane. 21.

4.12

Standard stock solution. $\rho = 1000 \text{ mg/L}$.

It can be purchased directly commercially available certified reference solution, also prepared using standard materials. Include phenol, o - cresol, p - cresol, Room - cresol, 2,4-xyleneol, 2-chlorophenol, 2,4-dichlorophenol, 2,6-dichlorophenol, 4-chloro-3-cresol,

2,4,6-tris chlorophenol, 2,4,5 Trichlorophenol, 2,3,4,6-tetra-chlorophenol, chlorophenol 2,3,4,5, 2,3,5,6, pentachlorophenol, 2-nitro-phenol, 4-nitrophenol, 2,4 Dinitrophenol, 2-methyl-4,6-dinitrophenol, 2- (1-methyl - n-propyl) -4,6-dinitrophenol (dinoseb), 2-cyclohexyl 4,6 two Nitrophenol.

4.13 Standard solution. rho = 100 mg/L

With methanol (4.8) diluted stock standard solution (4.12), formulated at a concentration of 100 mg/L of standard solution, 4 °C to Stored in the dark, it can be stored for months.

Quartz sand 4.14 (0.84 ~ 0.297 mm, 20 ~ 50 mesh). Baking 4 h at 400 °C, placed in a desiccator to cool to room Temperature, transferred to a grinding mouth glass bottles, stored in a desiccator.

Celite 4.15 (0.15 ~ 0.038 mm, 100 ~ 400 mesh). baking at 400 °C for 4 h, placed in a desiccator and cooled to Room temperature, transferred to a grinding mouth glass bottles, stored in a desiccator.

4.16 Nitrogen (N₂). purity >= 99.999%;

4.17 Hydrogen (H₂). purity >= 99.999%;

4.18 paper sleeve with Soxhlet apparatus supporting. Before use, check the residual amount of phenolic compounds, to avoid interference.

35 Instruments and equipment

5.1 Gas chromatograph. with a split/splitless inlet, with a hydrogen flame detector (FID).

5.2 Flip oscillating means.

5.3 Column. 30 m x 0.25 mm x 0.25 m, 100% methyl silicone capillary column; or 30 m x 0.25 mm x 0.25 m, 50% 50% phenyl methyl silicone capillary column, capillary column, or other equivalent.

5.4 Extraction equipment. Soxhlet extraction apparatus, the probe can also be used ultrasonic extractor, microwave or pressurized fluid extraction device Extracting means.

5.5 Extraction bottles. 2 L with screw cap and an inner lid of the jar, you can use glass or polytetrafluoroethylene (PTFE) bottle.

5.6 concentration device. a rotary evaporator means or KD concentrators, nitrogen blowing instrument, such as concentrated a considerable performance devices.

5.7 funnel. with polytetrafluoroethylene (PTFE) stoppers.

5.8 micro syringe. 10 µl, 25 µl, 100 µl, 250 µl, 500 µl and 1000 µl.

5.9 mortar. made of glass, agate or other non-interfering substance material.