

CCS Z 18

HJ

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

HJ 703-2014

Soil and sediment-Determination of phenolic compounds.

土壤和沉积物 酚类化合物的测定 气相色谱法

(English Translation)

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Table of Contents

Preface	.ii
1. Scope	
2 Normative references	
3 Principle of the method	
4 Reagents and materials	.1
5. Apparatus	.2
6 .3 Sample	
8 Represents the results of calculation and	
9 Precision and accuracy	
10 Quality Assurance and Quality Control	.7
11 Waste treatment	
12 Note	
Appendix A (normative) Determination of the detection limit and a lower limit	
Annex B (informative) of the title compound with a retention time and reference	.10
Annex C (informative) method precision and accuracy	.11
Annex D (normative) assisted qualitative reference chromatogram	.12

Foreword

To implement the "People's Republic of China Environmental Protection Law", protecting the environment, safeguarding human health, regulate soil and sediment Determination of phenolic compounds, the development of this standard.

This standard specifies the gas chromatographic determination of soil and sediment phenolic compounds.

This standard is the first release.

The Standard Appendix A normative annex, appendix B ~ 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 September 15, 2014 for approval.

This standard shall be December 1, 2014 implementation.

The standard explanation by the Ministry of Environmental Protection.

Gas chromatography of phenolic compounds in soil and sediment Warning. Reagents 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 specifies the gas chromatographic determination of soil and sediment phenolic compounds.

This standard applies to the determination of phenolic compounds in soil sediments and 21 species of other phenolic compounds if verified Also apply to this standard.

When the sample volume is 10.0 g, 21 kinds of phenolic compounds method detection limit of 0.02 ~ 0.08 mg/kg, detection limit is 0.08 ~ 0.32 mg/kg. 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.

GB 17378.3 specification for marine monitoring Part 3 sample collection, storage and transport
GB 17378.5 specification for marine monitoring Part 5 Analysis of Sediments
HJ/T 166 soil environmental monitoring technical specifications
HJ 613 Determination of dry matter and soil moisture weight method

3 PRINCIPLE OF THE METHOD

Soil or sediment with a suitable organic solvent extraction, the extract was purified acid distribution, phenolic compounds into the aqueous phase, the aqueous phase was adjusted to acidic water and extracted with a suitable organic solvent phase, the extract was dehydrated, concentrated, backward constant volume Gas Chromatography Separation, hydrogen flame detector. 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 or prepared by pure 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 solution. $c(\text{NaOH}) = 5 \text{ mol/L}$.

The solid weighed 20 g NaOH (4.1), cooled with water to dissolve 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 dichloromethane and 4.10 ethyl acetate. 4 1 (V/V).

A mixed solvent of dichloromethane and 4.11 hexane. 2 1 (V/V).

4.12

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

Can be purchased directly certified standard solution including all relevant analytes can

also be used pure reference material prepared. Include phenol, O - cresol, p - cresol, m - cresol, 2,4-xyleneol, 2-chlorophenol, 2,4-dichlorophenol, 2,6-dichlorophenol, 4-chloro-3-cresol, 2,4,6-trichlorophenol, 2,4,5-trichlorophenol, 2,3,4,6-tetra-chlorophenol, 2,3,4,5-chlorophenol, 2,3,5, 6-tetrafluoro, pentachlorophenol, 2-Nitrophenol, 4-nitrophenol, 2,4-dinitrophenol, 2-methyl-4,6-dinitrophenol, 2- (1-methyl - n-propyl) -4,6 dinitrophenol (ground Dinoseb), 2-cyclohexyl-4,6-dinitrophenol.

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 at 4 °C Refrigerator away from light, sealed save one month.

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.

5 Apparatus

5.1

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

5.2 Column. 30 m x 0.25 mm x 0.25 µm, 100% methyl silicone capillary column; or 30 m x 0.25 mm

x 0.25 µm, 50% 50% phenyl methyl silicone capillary column, capillary column, or other equivalent.

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

5.4 funnel. with polytetrafluoroethylene (PTFE) stoppers.