

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

HJ 998-2018

**Soil and sediment - Determination of volatile phenolic
compounds - 4-AAP spectrophotometric method**

土壤和沉积物挥发酚的测定 4-氨基安替比林分光光度法

(English Translation)

Issued on: December 26, 2018

Implemented on: June 1, 2019

Issued by: Ministry of Ecology and Environment

Table of Contents

Foreword

1 Determination of volatile phenols in soil and sediment

2 Normative references

3 Terms and definitions

24 Principles of the method

48.2 Sample Collection and Storage

Foreword

To protect the "Environmental Protection Law of the People's Republic of China" and the Law of the People's Republic of China on Soil Pollution Prevention this standard is formulated to ensure human health and to regulate the determination of volatile phenols in soils and sediments.

This standard specifies the 4-aminoantipyrine spectrophotometric method for the determination of volatile phenols in soils and sediments.

Appendix A of this standard is an informative annex.

This standard is the first release.

This standard is formulated by the Department of Eco-Environmental Monitoring, the Department of Regulations and Standards of the Ministry of Ecology and Environment.

This standard was drafted. Dalian Environmental Monitoring Center.

This standard is verified by. Shenyang Environmental Monitoring Center Station, Anshan Environmental Monitoring Center Station, Jinzhou City Environmental Monitoring Xinzhe, Zibo Environmental Monitoring Station, Shaanxi Provincial Environmental Monitoring Center Station and Hunan Environmental Monitoring Center Station.

This standard is approved by the Ministry of Ecology and Environment on December 26, 2018.

This standard has been implemented since June 1, 2019.

This standard is explained by the Ministry of Ecology and Environment.

1 Determination of volatile phenols in soil and sediment

4-aminoantipyrine spectrophotometry Warning. Phenols are toxic substances, and solution preparation should be carried out in a fume hood; protective equipment should be worn as required during operation.

Avoid breathing into the respiratory tract or direct contact with skin and clothing.

1 Scope of application this standard specifies the 4-aminoantipyrine spectrophotometric method for the determination of volatile phenols in soils and sediments.

This standard applies to the determination of volatile phenols in soils and sediments.

When the sampling amount is 20 g and the detection optical path is 10 mm, the detection limit of the volatile phenol (based on phenol) is 0.3.

Mg/kg, the lower limit of determination is 1.2 mg/kg.

2 Normative references

This standard refers to the following documents or their terms. For undated references, the valid version applies to this standard.

GB 17378.3 Marine monitoring specification Part 3. Sample collection, storage and transport GB 17378.5 Marine monitoring specification Part 5. Sediment analysis HJ 494 Water Quality Sampling Technical Guidance HJ 503-2009 Determination of Volatile Phenols in Water - 4-Amino Antipyrine Spectrophotometric Method HJ 613 Determination of dry matter and moisture in soils - Gravimetric method HJ/T 91 Surface Water and Wastewater Monitoring Technical Specifications HJ/T 166 Technical Specifications for Soil Environmental Monitoring

3 Terms and definitions

The following terms and definitions apply to this standard.

Volatile phenolic compounds it can be extracted from soil and sediment under the conditions stipulated in this standard, can be distilled with water vapor and combined with 4-amino Antipyrine reacts to form volatile phenolic compounds of colored compounds, with the result being phenol.

Note: Volatile phenols measured under the conditions specified in this standard do not contain 4-cresol, 2,4-xyleneol, 3,4-xyleneol, 2,4-dinitrophenol, 4,6-A compound such as dinitro-2-cresol which does not undergo a color reaction with 4-aminoantipyrine.

24 Principles of the method

Extraction of phenolic compounds from soil and sediment with an alkaline solution, the extract is distilled under acidic conditions, and in the distillate Volatile phenol reacts with 4-aminoantipyrine in an alkaline solution in the presence of potassium ferricyanide catalyst to form orange-red indophenol. Tebulin was measured at a wavelength of 510 nm, and the volatile phenol content was proportional to the absorbance value over a range of concentrations.

5 interference and elimination Determination of volatile phenols in soils and sediments by aniline containing less than 750 mg/kg and oils of 1.5×10^4 mg/kg no interference.

5.2 Sulfide interferes with the determination of volatile phenols. Preparation process before sampling (8.1) and distillation of sample (8.4.2) the addition of copper sulfate pentahydrate (6.2) eliminates the interference of less than 3.0×10^4 mg/kg sulfide.

6 reagents and materials Unless otherwise stated, analytically pure reagents that meet national standards were used for the analysis.

6.1 Experimental water. no phenol water, should be stored in glass bottles, when used, should avoid with rubber products (rubber plug or latex tube) Etc.) Contact. The phenol-free water can be prepared in the following three ways.

Method 1. Newly prepared deionized water or distilled water, blank test is required before use, and the results should be in accordance with blank inspection.

(12.1) Requirements.

Method 2. Add 0.2 g of activated carbon powder activated at 200°C for 30 min per liter of water, shake well, and let stand overnight.

Filter with double layer medium speed filter paper.

Method 3. Add sodium hydroxide to make the water strong alkaline, and add potassium permanganate until the solution is purple-red, and transfer to full glass distillation.

The tube was heated and distilled, and the distillate was collected for use.

6.2 Copper sulfate pentahydrate ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$).

6.3 Ammonia. $\rho(\text{NH}_3 \cdot \text{H}_2\text{O}) = 0.90 \text{ g/ml}$.

6.4 Phosphoric acid. $\rho(\text{H}_3\text{PO}_4) = 1.87 \text{ g/ml}$.

6.5 sodium hydroxide (NaOH).

6.6 Phenol ($\text{C}_6\text{H}_5\text{OH}$).

6.7 Methyl orange ($\text{C}_{14}\text{H}_{14}\text{N}_3\text{SO}_3\text{Na}$).

6.8 Ammonium chloride (NH_4Cl).

6.9 4-Aminoantipyrine ($\text{C}_{11}\text{H}_{13}\text{N}_3\text{O}$).

6.10 Potassium ferricyanide ($\text{K}_3[\text{Fe}(\text{CN})_6]$).

6.11 Quartz sand.

After burning at 400°C for 4 h, cooled to room temperature, transferred to a ground glass bottle and stored in a desiccator.

6.12 Sodium hydroxide solution. $\rho(\text{NaOH}) = 10\text{ g/L}$.

Weigh sodium hydroxide (6.5) 10 g dissolved in water, dilute to 1 L, and store in a plastic reagent bottle.

6.13 Refined phenol.

Phenol (6.6) is taken in a distillation flask with an air condensing tube, heated and distilled, and a distillate portion of 182°C to 184°C is collected.

3 minutes, the fraction should be colorless crystals after cooling, stored in a brown bottle, sealed in a cool dark place, and re-refined after discoloration.

6.14 phenol standard stock solution. $\rho(\text{C}_6\text{H}_5\text{OH}) \sim 1.00\text{ g/L}$.

Weigh 0.1 g (accurate to 0.01 g) of refined phenol (6.13), dissolve in 100 ml of water, and store at 4°C or less in the dark.

It can be stored stably for one month, and needs to be reconstituted after discoloration. Before use, follow the method specified in Appendix A of HJ 503-2009.

set. Commercially available certified standard solutions can also be purchased.

6.15 Phenol standard use solution. $\rho(\text{C}_6\text{H}_5\text{OH}) = 10.0\text{ mg/L}$.

Take appropriate amount of phenol standard stock solution (6.14), dilute to 100 ml volumetric flask with water, shake well, and use it now.

6.16 methyl orange indicator solution. $\rho(\text{C}_{14}\text{H}_{14}\text{N}_3\text{SO}_3\text{Na}) = 0.5\text{ g/L}$.

Weigh 0.1 g of methyl orange (6.7) in 200 ml of water and store in a glass reagent bottle.

6.17 Buffer solution. $\text{pH} \sim 10.7$.

Weigh 20 g of ammonium chloride (6.8) dissolved in 100 ml of ammonia (6.3), and store in a plastic reagent bottle at 4°C or less.

6.18 4-aminoantipyrine solution 2 g of 4-aminoantipyrine (6.9) was weighed and dissolved in 100 ml of water. In accordance with the method specified in Appendix B of HJ 503-2009 Purification, refrigerated at 4°C or less can be stored for one week.

6.19 Potassium ferricyanide solution. $\rho(\text{K}_3[\text{Fe}(\text{CN})_6]) = 80\text{ g/L}$.