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**HJ**

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

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**Soil and sediment. Determination of sulfide. Methylene blue  
spectrophotometric method**

土壤和沉积物 硫化物的测定 亚甲基蓝分光光度法

**(English Translation)**

**Issued on: July 18, 2017**

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### Foreword

For the implementation of the "People's Republic of China Environmental Protection Law" to protect the environment, protect human health, regulate soil and sediment sulfide Determination method, the development of this standard.

This standard specifies the methylene blue spectrophotometric method for the determination of sulfides in soil and sediments.

This standard is the first release.

Appendix A to this standard is an informative appendix.

This standard is organized by the Environmental Monitoring Department of the Ministry of Environmental Protection, the Secretary for Science and Technology Standards.

The main drafting unit of this standard. Tianjin Environmental Monitoring Center.

The standard verification unit. Liaoning Province Environmental Monitoring Experimental Center, Inner Mongolia Environmental Monitoring Center Station, Fuzhou Environmental Monitoring Center Station, Lake South China Environmental Monitoring Center Station, Tianjin Binhai New Area Environmental Protection Monitoring Station, Yangzhou City Environmental Monitoring Center Station.

The Environmental Protection Department of this standard approved on July 18, 2017.

This standard has been implemented since September 1, 2017.

This standard is explained by the Ministry of Environmental Protection.

Determination of soil and sediments Sulfide - Methylene blue spectrophotometric method  
Warning. The sulfuric acid, hydrochloric acid, N, N-dimethyl-p-phenylenediamine

hydrochloride used in the experiment are corrosive.

Wear protective equipment as required to avoid direct contact with these chemicals. The sample pretreatment process should be operated in a fume hood.

## 1 Scope of application

This standard specifies the methylene blue spectrophotometric method for the determination of sulfides in soil and sediments.

This standard applies to the determination of sulfides in soil and sediments.

When the sampling amount is 20 g, the detection limit is 0.04 mg/kg and the determination limit is 0.16 mg/kg.

## 2 Normative reference documents

The contents of this standard refer to the following documents or their terms. For undated references, the valid version applies to this standard.

Specification for ocean monitoring - Part 3. Sample collection, storage and transport  
Specification for marine monitoring - Part 5. Sediment analysis Determination of Dry Matter and Moisture in Soil Technical specification for soil environmental monitoring

## 3 Terms and definitions

The following terms and definitions apply to this standard.

Sulfide

Refers to soil and sediments in the acid-soluble sulfide.

## 4 Principle of the method

The sulfide in the soil and the sediment is acidified to form hydrogen sulfide gas, and the hydrogen sulfide is blown out by heating the blowing or distillation apparatus, Sodium oxide solution absorption, the formation of sulfur ions in the presence of high iron ions in acidic solution with N, N-dimethyl-p-phenylenediamine reaction to produce methylene Base blue, measured at 665 nm wavelength of its absorbance, sulfide content and absorbance value is proportional to.

## 5 Interference and elimination

Under specified conditions, elemental sulfur does not interfere with the determination of sulfide. Determination of sulfide by sulfite, bisulfite and thiosulfate no interference. Nitrite can react with methylene blue to make the determination result low, when the nitrite

concentration (in N) is higher than 12.0 mg/kg , This method does not apply.

## 26 Reagents and materials

Unless otherwise stated, analytical reagents conforming to national standards are used in the analysis, and the test water is freshly prepared distilled water or deionized water.

6.1 Sulfuric acid.  $\rho(\text{H}_2\text{SO}_4) = 1.84 \text{ g/ml}$ .

6.2 hydrochloric acid.  $\rho(\text{HCl}) = 1.19 \text{ g/ml}$ , excellent grade pure.

6.3 Sodium hydroxide (NaOH).

6.4 N, N-dimethyl-p-phenylenediamine hydrochloride  $[\text{NH}_2\text{C}_6\text{H}_4\text{N}(\text{CH}_3)_2 \cdot 2\text{HCl}]$ .

6.5 Ammonium sulfate  $[\text{Fe}(\text{NH}_4)(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}]$ .

6.6 soluble starch  $[(\text{C}_6\text{H}_{10}\text{O}_5)_n]$ .

6.7 Zinc acetate  $[\text{Zn}(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}]$ .

6.8 Iodine ( $\text{I}_2$ ).

6.9 Potassium iodide (KI).

6.10 Sodium thiosulfate  $(\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O})$ .

6.11 anhydrous sodium carbonate  $(\text{Na}_2\text{CO}_3)$ .

6.12 Sodium sulfide  $(\text{Na}_2\text{S} \cdot 9\text{H}_2\text{O})$ .

6.13 ascorbic acid  $(\text{C}_6\text{H}_8\text{O}_6)$ .

6.14 Ethylenediamine tetraacetic acid disodium  $(\text{C}_{10}\text{H}_{14}\text{O}_8\text{N}_2\text{Na}_2 \cdot 2\text{H}_2\text{O})$ .

6.15 Potassium Dichromate  $(\text{K}_2\text{Cr}_2\text{O}_7)$ . Reference reagent.

Take appropriate amount of potassium dichromate in the weighing bottle, drying at  $105^\circ\text{C} \pm 1^\circ\text{C}$  2 h, placed in the dryer cooling, spare.

6.16 Sulfuric acid solution. 1 5 (V/V).

Measure 20 ml of sulfuric acid (6.1) slowly into 100 ml of water, cooling.

6.17 hydrochloric acid solution. 1 1 (V/V).

Measure 250 ml of hydrochloric acid (6.2) slowly into 250 ml of water, cooling.

6.18 Antioxidant solution.

Weigh 2.0 g of ascorbic acid (6.13), 0.1 g of disodium ethylenediaminetetraacetate (6.14), 0.5 g of sodium hydroxide (6.3) Water, shake and store in a brown reagent bottle. Pro is now available.

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

Weigh 10.0 g of sodium hydroxide (6.3) dissolved in 1000 ml of water, shake.

6.20 N, N-dimethyl-p-phenylenediamine solution.  $\rho[\text{NH}_2\text{C}_6\text{H}_4\text{N}(\text{CH}_3)_2 \cdot 2\text{HCl}] = 2 \text{ g/L}$ .

Weigh 2.0 g of N, N-dimethyl-p-phenylenediamine hydrochloride (6.4) in 700 ml of water, slowly add 200 ml of sulfuric acid (6.1), cold but then diluted with water to 1000 ml, shake. The solution was stored in a sealed brown bottle at room temperature for three months.

6.21 ammonium ferric sulfate solution.  $\rho[\text{Fe}(\text{NH}_4)(\text{SO}_4)_2] = 100 \text{ g/L}$ .

Weigh 25.0 g of ammonium ferric sulfate (6.5) in 100 ml of water, slowly add 5.0 ml of sulfuric acid (6.1), and then diluted with water to 250 ml, shake well. Solution such as the emergence of insoluble, should be used after filtration.

6.22 Starch solution.  $\rho[(\text{C}_6\text{H}_{10}\text{O}_5)_n] = 10 \text{ g/L}$ .

3 Weigh 1.0 g of soluble starch (6.6), with a small amount of water into a paste, slowly pour 50 ml of boiling water, continue to boil to the solution to clarify, Set to 100 ml, cooled and stored in the reagent bottle. Pro is now available.

6.23 Zinc acetate solution.  $\rho[\text{Zn}(\text{CH}_3\text{COO})_2] = 1 \text{ g/L}$  Weigh 1.20 g of zinc acetate (6.7), dissolve in a small amount of water and dilute to 1000 ml.

6.24 potassium dichromate standard solution.  $c(1/6\text{K}_2\text{Cr}_2\text{O}_7) = 0.1000 \text{ mol/L}$ .

Accurately weighed 4.9032 g potassium dichromate (6.15) dissolved in 100 ml of water, transferred to 1000 ml volumetric flask, diluted to the mark, shake.

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6.25 iodine standard solution.  $c(1/2\text{I}_2) \sim 0.01 \text{ mol/L}$ .

Accurately weighed 1.27 g of iodine (6.8) dissolved in 100 ml of water, then add 10.0 g of potassium iodide (6.9), dissolved and transferred to 1000 ml Brown capacity bottle, diluted to the mark, shake. Pro is now available.

6.26 sodium thiosulfate standard solution.  $c(\text{Na}_2\text{S}_2\text{O}_3) \sim 0.1 \text{ mol/L}$ .

Weigh 24.8 g sodium thiosulfate (6.10) in 100 ml of water, add 1.0 g of anhydrous sodium carbonate (6.11), dissolve and transfer to 1000 ml brown volumetric flask, diluted with water to the mark, shake. Stored in a brown glass reagent bottle, protected from light for 6 months. Pro is now available Standard If the solution appears turbid, you have to filter and use after calibration. You can also purchase commercially available certified materials.

Calibration method. In a 250 ml iodine flask, add 1.0 g of potassium iodide (6.9), 50 ml of water and 10.00 ml of potassium dichromate Solution (6.24), shaken until completely dissolved, then add 5.0 ml of sulfuric acid solution (6.16), immediately dense packing, in the dark place for 5 min.