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

HJ 1054-2019

**Soil and sediment -- Determination of dithiocarbamate
pesticides -- Headspace gas chromatography**

土壤和沉积物 二硫代氨基甲酸酯（盐）类农药总量的测定 顶空气相色谱法
(English Translation)

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Table of Contents

Foreword	
1 Scope	
2 Normative references	
3 Terms and definitions	
4 Methodology	
5 Interference and cancellation	
6 Reagents and materials	
7 Instruments and equipment	
8 Sample	
9 Analysis steps	
10 Calculation and Representation of Results	
11 Precision and accuracy	
12 Quality Assurance and Quality Control	
13 Waste disposal	
Appendix A (informative) Precision and accuracy of the method	

Foreword

In order to implement the "Environmental Protection Law of the People's Republic of China" Environment, protect human health, standardize the determination method for the total amount of dithiocarbamate (salt) pesticides in soil and sediment Law to formulate this standard.

This standard specifies the headspace/gas phase color of total dithiocarbamate (salt) pesticides in soil and sediment.

Spectrum method.

Appendix A of this standard is an informative appendix.

This standard is issued for the first time.

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

This standard was drafted. Hainan Environmental Monitoring Center Station.

Verification units of this standard. Chongqing Ecological Environment Monitoring Center,

Hubei Province Environmental Monitoring Center Station, Hainan Entry-Exit Inspection and Quarantine Technology Center of the Quarantine Bureau, Haikou Environmental Protection Monitoring Station, Sanya Environmental Monitoring Station, and Haikou Disease Prevention and Control System center.

This standard was approved by the Ministry of Ecology and Environment on October 24, 2019.

This standard will be implemented from April 24, 2020.

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

Determination of total dithiocarbamate (salt) pesticides in soil and sediment Headspace/gas chromatography Warning. The chemical reagents used in the experiment are toxic and corrosive, and should be worn and protected according to regulations when operating Appliances to avoid contact with skin and clothing.

1 Scope

This standard specifies the headspace/gas phase color of total dithiocarbamate (salt) pesticides in soil and sediment.

Spectrum method.

This standard applies to the determination of total dithiocarbamate (salt) pesticides in soils and sediments.

Or carbon disulfide meter.

This standard does not apply to the determination of samples of soils and sediments containing hydroxanthic acid and its salts.

When the sample size is 2 g, the time limit is measured with mancozeb, the detection limit of the method is 0.05 mg/kg, and the lower limit of determination is 0.20 mg/kg when carbon disulfide was used, the detection limit of the method was 0.03 mg/kg, and the lower limit of determination was 0.12 mg/kg.

2 Normative references

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

GB 17378.3 Marine Monitoring Code Part 3. Sample Collection, Storage and Transportation
GB 17378.5 Marine Monitoring Code Part 5. Sediment Analysis HJ 494 Water Quality Sampling Technical Guide HJ 495 Water Quality Sampling Scheme Design Technical Regulations HJ 613 Soil dry matter and moisture determination by gravimetric method HJ/T 91 Technical specifications for surface water and sewage monitoring HJ/T 166 Technical

Specifications for Soil Environmental Monitoring

3 Terms and definitions

The following terms and definitions apply to this standard.

Total dithiocarbamate pesticides Refers to pesticides containing dithiocarbamoyl in the molecular structure under the conditions specified in this standard Fumei Zinc, Fumei Iron, Daisen United, Daisen Manganese, Daisen Zinc, Daisen Sodium, Propyl Zinc, etc.) Based on mancozeb or carbon disulfide.

4 Method principle

At a certain temperature, dithiocarbamate (salt) pesticides in the soil or sediment in the headspace bottle the reaction of tin in the inorganic acid medium generates carbon disulfide. When the carbon disulfide reaches the thermodynamic equilibrium in the gas-liquid-solid three-phase, Carbon disulfide in the gas phase is separated by gas chromatography and detected by an electron capture detector (ECD). According to the meter of carbon disulfide Calculate the total amount of dithiocarbamate (salt) pesticides.

5 Interference and cancellation

Carbon disulfide in the sample will positively interfere with the determination of dithiocarbamate (salt) pesticides.

Subtract the measurement result from the unacidified sample.

6 Reagents and materials

Unless otherwise specified, analytical reagents that comply with national standards are used in the analysis. The experimental water is freshly prepared pure water or Carbon Disulfide Free.

6.1

Sulfuric acid (H_2SO_4). $\rho = 1.84 \text{ g/ml}$, excellent purity.

6.2

stannous chloride ($\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$).

6.3

Sodium chloride (NaCl). excellent grade pure.

Bake at 400°C for 4 h in a muffle furnace, cool in a desiccator to room temperature, and transfer to a sealed glass bottle for storage.

6.4

Disodium ethylenediamine tetraacetate ($\text{C}_{10}\text{H}_{14}\text{N}_2\text{O}_8\text{Na}_2 \cdot 2\text{H}_2\text{O}$).

6.5

L-cysteine hydrochloride ($\text{C}_3\text{H}_7\text{NO}_2\text{S} \cdot \text{HCl} \cdot \text{H}_2\text{O}$).

6.6

Sodium hydroxide (NaOH).

6.7

Sodium hydroxide solution. $c(\text{NaOH}) = 10 \text{ mol/L}$.

Weigh 20 g of sodium hydroxide (6.6) in a small amount of experimental water and dilute to 50 ml.

6.8

Sulfuric acid solution. $c(\text{H}_2\text{SO}_4) = 3 \text{ mol/L}$.

Take 81 ml of sulfuric acid (6.1), slowly pour it into experimental water, and dilute to volume to 500 ml.

6.9

EDTA-cysteine alkaline solution.

Weigh 12.5 g of disodium ethylenediamine tetraacetate (6.4) and 12.5 g of L-cysteine hydrochloride (6.5) and add 800 ml Dissolve and mix the experiment water with sodium hydroxide solution (6.7) to adjust the pH to 9.0 ~ 10.0.

6.10

stannous chloride-sulfuric acid solution. $\rho(\text{SnCl}_2 \cdot 2\text{H}_2\text{O}) = 15 \text{ g/L}$.

Weigh 7.5 g of stannous chloride (6.2), slowly add to 500 ml of sulfuric acid solution (6.8), dissolve and mix.

6.11

Mancozeb ($\text{C}_4\text{H}_6\text{N}_2\text{S}_4\text{MnZn}$). purity $\geq 97.5\%$.

6.12

Mancozeb standard stock solution. $\rho = 100 \mu\text{g/ml}$.

Accurately weigh 50 mg (accurate to 0.1 mg) mancozeb (6.11), transfer to a 500 ml brown volumetric flask, add EDTA-cysteine alkaline solution (6.9) is dissolved and made up to volume.

For 2 months.