

ICS 13.030.10
CCS Z 13

HJ

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

HJ 874-2017

**Solid waste. Determination of acrolein, acrylonitrile and
acetonitrile. Headspace gas chromatography method**

固体废物 丙烯醛、丙烯腈和乙腈的测定 顶空-气相色谱法

(English Translation)

Issued on: November 28, 2017

Implemented on: January 1, 2018

Issued by: Ministry of Ecology and Environment

Table of Contents

Foreword

1 Scope of application

2 Normative references

3 Method principle

4 Reagents and materials

5 Instruments and equipment

36 Samples

Foreword

In order to carry out the "Law of the People's Republic of China on Environmental Protection" and the "Law of the People's Republic of China on Prevention and Control of Environmental Pollution by Solid Waste," protect the environment, Protection of human health, regulate solid waste and leachate acrolein, acrylonitrile and acetonitrile determination, the development of this standard.

This standard specifies the determination of solid waste and leachate acrolein, acrylonitrile and acetonitrile by headspace - gas chromatography.

Appendix A of this standard is an informative annex.

This standard is released for the first time.

This standard by the Environmental Protection Department of Environmental Monitoring Division and Science and Technology Standards Division to develop.

This standard was drafted. Yangzhou City Environmental Monitoring Center Station, Nanjing Environmental Monitoring Center Station.

This standard verification unit. Zhenjiang Environmental Monitoring Center Station, Jiangsu ?? Environmental Monitoring Co., Ltd., Changzhou City Environmental Monitoring Center, Jiang Yin City Environmental Monitoring Station, Wuxi City Environmental Monitoring Center Station and Taizhou City Environmental Monitoring Center Station.

This standard MEP approved on November 28, 2017.

This standard since January 1, 2018 come into operation.

This standard is interpreted by the MEP.

Solid waste - Determination of acrolein, acrylonitrile and acetonitrile - Headspace - Gas

chromatography Warning. reagents and standard solutions used in the experiment are volatile toxic compounds, the preparation process should be carried out in a fume hood; operation when required, wear protective equipment as required to avoid contact with skin and clothing.

1 Scope of application

This standard specifies the determination of solid waste and leachate acrolein, acrylonitrile and acetonitrile by headspace - gas chromatography.

This standard applies to solid waste and leachate acrolein, acrylonitrile and acetonitrile determination.

The detection limit of acrolein and acrylonitrile was 0.3 mg/kg when the sample of solid wastes was 2.0 g, and the lower limit of determination was 1.2 mg/kg.

The detection limit of acetonitrile was 0.4 mg/kg, and the lower limit of determination was 1.6 mg/kg. Solid waste leachate volume of 10 ml, acrolein, the method detection limit of nitrile and acetonitrile was 0.05 mg/L, and the lower limit of determination was 0.20 mg/L.

2 Normative references

This standard references the following documents or the terms of them. For undated references, the effective version applies to this standard.

HJ/T 20 Industrial solid waste sampling Sampling specifications HJ/T 298 Hazardous Waste Identification Technical Specifications Solid waste extraction toxicity Toxicity method HNO₃ method HJ/T 300 solid waste leaching toxic leaching method acetic acid buffer solution method

3 Method principle

At a certain temperature, the volatile components in the sample in the headspace vial volatilize to the liquid space to generate the vapor pressure. In the gas-liquid-solid three-phase (or gas Liquid two-phase) to reach the thermodynamic equilibrium, the volatile components in the gas phase separated by gas chromatography, flame ionization detector detection. To Retention time Qualitative, peak height or peak area quantification.

4 Reagents and materials

Unless otherwise specified, analytical reagents are used in accordance with national standards for analytical purposes. The experimental water is a freshly prepared, organo-free Water.

4.1

Acrolein (C₃H₄O). Chromatographic purity.

4.2

Acrylonitrile (C₃H₃N). Chromatographic purity.

Acetonitrile (C₂H₃N). Chromatographic purity.

4.4

Methanol (CH₃OH). Chromatographic purity.

24.5 Sodium chloride (NaCl). excellent grade pure. Bake at 400 °C 4 h, remove possible interference substances, sealed in a sealed glass jar after cooling Save.

4.6

Phosphoric acid. ρ (H₃PO₄) = 1.874 g/ml.

4.7

matrix modifier. Take 500 ml of water, add a few drops of phosphoric acid (4.6) adjust pH $\leq$ 2, add 180 g of sodium chloride (4.5), dissolved Mix well.

4.8

Acrolein, acrylonitrile and acetonitrile mixed standard solution. ρ = .2000 mg/L.

Weigh 0.20 g (accurate to 0.1 mg) acrolein (4.1), acrylonitrile (4.2) and acetonitrile (4.3) in a small amount of water, using experimental water Set the volume to 100 ml. Or direct purchase of certified public standard solution. Standard solution at 0 °C ~ 4 °C dark place. After unsealing with dense bottles to avoid Light preservation, valid for 1 month.

4.9

Quartz sand (SiO₂). 270 μ m ~ 830 μ m (20 mesh ~ 50 mesh). Before use to go through a blank test to confirm that no target compound or the target compound concentration is below the method detection limit.

4.10

Carrier gas. high purity nitrogen ($\geq$ 99.999%).

4.11

Gas. High purity hydrogen ($\geq$ 99.999%).

4.12

help gas. air, dehydration organic matter.

5 Instruments and equipment

5.1

Gas Chromatograph. Capillary Split/Splitless Inlet, Programmable Temperature, Flame Ionization Detector (FID).

5.2

Capillary column. 1.30 m x 0.32 mm x 0.50 μ m, 100% polyethylene glycol solution, other equivalent capillary column can also be used.

Column 2.30 m x 0.32 mm x 0.25 μ m, 50% diphenyl 50% dimethylsiloxane fixative, other equivalent capillary columns are also available.

5.3

Headspace Sampler. headspace bottles, gaskets (Teflon/siloxane or Teflon/butyl rubber), sealed caps (screw caps or one-time gland).

5.4

reciprocating oscillator. oscillation frequency of 150 times/min, headspace bottle can be fixed.

5.5

Balance. a sense of the amount of 0.01 g, 0.0001 g.

5.6

Micro syringe. 10 μ l, 100 μ l.

5.7

Sampling equipment. shovels and stainless steel medicine spoon.

5.8

Vials. 60 ml or 250 ml, threaded brown glass with Teflon septa.

5.9

Brown glass bottle. 2 ml with Teflon liner and solid screw cap.

5.10

Portable Reefer. Volume 20 L, temperature below 4 °C.

5.11

disposable Pasteur glass pipette.

5.12

common laboratory equipment and equipment.