

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

HJ 997-2018

**Soil and sediment - Determination of carbonyl compounds -
High performance liquid chromatography**

土壤和沉积物醛、酮类化合物的测定 高效液相色谱法

(English Translation)

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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 aldehydes and ketones in soils and sediments.

This standard specifies high performance liquid chromatography for the determination of aldehydes and ketones in soils and sediments.

Appendix A of this standard is a normative appendix, and Appendix B to Appendix D are informative appendices.

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. Tianjin Ecological Environment Monitoring Center.

This standard is verified by the National Environmental Analysis and Testing Center, Shanghai Environmental Monitoring Center, and Shenyang Environmental Monitoring Center.

Station, Qingdao Environmental Monitoring Center Station, Tianjin Product Quality Supervision and Inspection Technology Research Institute and Tianjin Binhai New Area Environmental Protection Protection 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 aldehydes and ketones in soils and sediments - High performance liquid chromatography Warning. The organic solvents and reference materials used in the experiment are toxic and hazardous substances, standard solution preparation and sample preparation.

The process should be carried out in a fume hood; protective equipment should be worn as required to avoid direct contact with skin and clothing.

1 Scope of application

This standard specifies high performance liquid chromatography for the determination of aldehydes and ketones in soils and sediments.

This standard applies to soil, sediment, formaldehyde, acetaldehyde, acrolein, acetone, propionaldehyde, crotonaldehyde, butyraldehyde, benzene Aldehyde, isovaleraldehyde, n-pentanal, o-methylbenzaldehyde, m-methylbenzaldehyde, p-methylbenzaldehyde, n-hexanal, 2,5-dimethyl Determination of 15 kinds of aldehydes and ketones such as benzaldehyde.

When the sample volume is 10 g and the volume is 10 ml, the detection limit of 15 aldehydes and ketones is 0.02.

Mg/kg~0.06 mg/kg, the lower limit of determination is 0.08 mg/kg~0.24 mg/kg. See Appendix A for details.

2 Normative references

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

In this standard.

GB 17378.3 Marine monitoring specification Part 3 Sample collection, storage and transportation GB 17378.5 Marine monitoring specification Part 5. Sediment analysis HJ 494 Water Quality Sampling Technical Guidance HJ 613 Determination of dry matter and moisture in soils - Gravimetric method HJ/T 166 Technical Specifications for Soil Environmental Monitoring

3 Principle of the method

Soil and sediment samples were extracted by shaking with acetic acid-sodium acetate solution. The aldehydes and ketones in the extract were at a certain temperature.

And derivatization with 2,4-dinitrophenylhydrazine (DNPH) at pH to form stable terpenoids, extracted After concentration, it is separated by high performance liquid chromatography, detected by a UV detector, and quantified according to the retention time and external standard method.

4 Reagents and materials

Unless otherwise stated, analytically pure reagents that meet national standards were used for the analysis. Experimental water is double distilled water or pure the water prepared by the water equipment shall be subjected to blank inspection before use to confirm that the target compound concentration is lower than the method detection limit.

4.1

Sodium chloride (NaCl).

After burning at 400 °C for 4 h, it is cooled slightly, placed in a desiccator, cooled to room temperature, transferred to a ground glass bottle, and kept in a desiccator.

Save.

4.2

anhydrous sodium sulfate (Na₂SO₄).

After burning at 400 °C for 4 h, it is cooled slightly, placed in a desiccator, cooled to room temperature, transferred to a ground glass bottle, and kept in a desiccator.

2 save.

4.3

Sodium acetate (CH₃COONa).

4.4

Glacial acetic acid (CH₃COOH). content >= 98%.

4.5

Citric acid (C₆H₈O₇).

4.6

Sodium citrate (C₆H₅Na₃O₇·2H₂O).

4.7 2,4-Dinitrophenylhydrazine [NH₂NHC₆H₃(NO₂)₂, DNPH]. purity >=99%.

4.8

Acetonitrile (CH₃CN). Pure by liquid chromatography.

4.9

Dichloromethane (CH₂Cl₂). Pure by liquid chromatography.

4.10

Sodium chloride solution. $\rho(\text{NaCl}) = 0.365 \text{ g/ml}$.

Weigh 36.5 g of sodium chloride (4.1) and dissolve to 100 ml with water.

4.11

Extractant. Acetic acid-sodium acetate solution.

Weigh 5.3 g of sodium acetate (4.3), dissolve in water, add 2.0 ml of glacial acetic acid (4.4), and dilute to 1 L with water.

4.12

Buffer solution. pH ~3.

84.0 g of citric acid (4.5) and 29.4 g of sodium citrate (4.6) were weighed and dissolved in water to a volume of 500 ml.

4.13

Derivatizing agent. $\rho(\text{DNPH}) = 3.00 \text{ mg/ml}$.

3.00 g of 2,4-dinitrophenylhydrazine (4.7) was weighed into acetonitrile (4.8) and dissolved to 1 L with acetonitrile.

4.14

Aldehyde, ketone-DNPH standard solution. $\rho = 100 \text{ } \mu\text{g/ml}$ (based on aldehydes, ketones).

Including formaldehyde-DNPH, acetaldehyde-DNPH, acrolein-DNPH, acetone-DNPH, propionaldehyde-DNPH, crotonaldehyde-DNPH, Butyraldehyde-DNPH, benzaldehyde-DNPH, isovaleraldehyde-DNPH, n-pentanal-DNPH, o-methylbenzaldehyde-DNPH, inter-Methyl benzaldehyde-DNPH, p-methylbenzaldehyde-DNPH, n-hexanal-DNPH, 2,5-dimethylbenzaldehyde-DNPH.

Dissolve the agent in acetonitrile (4.8). A commercially available certified standard solution can be purchased.

4.15

Standard stock solution of aldehyde and ketone. $\rho = 1000 \text{ } \mu\text{g/ml}$.

Including formaldehyde, acetaldehyde, acrolein, acetone, propionaldehyde, crotonaldehyde, butyraldehyde, benzaldehyde, isovaleraldehyde, n-pentanal, ortho-Methyl benzaldehyde, m-methylbenzaldehyde, p-methylbenzaldehyde, n-hexanal, 2,5-dimethylbenzaldehyde. The