

HJ

INDUSTRY STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

HJ 1210-2021

**Soil and sediment - Determination of 13 aniline compounds and
2 benzidine compounds - Liquid chromatography-triple
quadrupole mass spectrometry**

土壤和沉积物 13种苯胺类和2种联苯胺类化合物的测定 液相色谱-三重四极杆质谱法

(English Translation)

Issued on: January 1, 1970

Implemented on: January 1, 1970

Issued by: Ministry of Ecology and Environment

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1 Scope of application

This standard specifies the liquid chromatography-triple quadrupole mass spectrometry method for the determination of anilines and benzidine compounds in soil and sediments.

This standard applies to benzidine, aniline, 4-methylaniline, 2-methoxyaniline, 3-methylaniline, 2-methylbenzene in soil and sediment Amine, 2,4-dimethylaniline, 4-nitroaniline, 3-nitroaniline, 4-chloroaniline, 2-naphthylamine, 2,6-dimethylaniline, 3-chloroaniline, 3,3 '-Determination of dichlorobenzidine and N-nitroso diphenylamine in total of 13 anilines and 2 benzidine compounds.

When the sampling volume is 5.0 g and the constant volume is 1.0 ml, the method detection limits for 13 anilines and 2 benzidine compounds are 2 µg/kg~4 µg/kg, the lower limit of determination is 8 µg/kg~16 µg/kg. See Appendix A for details.

2 Normative references

This standard refers to the following documents or clauses thereof. For dated references, only the dated version applies to this standard.

For undated references, the latest edition (including all amendments) applies to 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/T 166 Technical Specification for Soil Environment Monitoring HJ 494 Water Quality Sampling Technical Guide HJ 613 Gravimetric Method for Determination of Soil Dry Matter and Moisture

3 Principles of the method

Aniline and benzidine target compounds in soil or sediment are extracted under alkaline conditions, purified, concentrated and fixed to volume, and then used in liquid Phase chromatography-triple quadrupole mass spectrometer separation and detection. According to retention time and characteristic ion identification, internal standard method for quantification.

4 Interference and cancellation

When there is matrix effect interference in the sample, it can be reduced by cleaning the sample, optimizing the chromatographic conditions, reducing the sample volume or injection volume, etc.

Low or eliminate interference.

5 Reagents and Materials

Unless otherwise stated, analytical reagents that meet national standards are used in the analysis, and the experimental water is freshly prepared and does not contain target compounds.

of pure water.

5.1

Dichloromethane (CH_2Cl_2). chromatographically pure.

5.2

n-hexane (C_6H_{14}). chromatographically pure.

5.3

Acetone (CH_3COCH_3). chromatographically pure.

5.4

Methanol (CH_3OH). chromatographically pure.

5.5

Formic acid (HCOOH). chromatographically pure.

5.6

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

5.7

Ammonia ($\text{NH}_3 \cdot \text{H}_2\text{O}$). $w(\text{NH}_3 \cdot \text{H}_2\text{O}) = 25\% \sim 28\%$.

5.8

n-hexane-acetone mixed solvent.

Mix with n-hexane (5.2) and acetone (5.3) in a 1.1 volume ratio.

5.9

Formic acid-methanol solution.

Pipette 100 μl of formic acid (5.5) into 1000 ml of methanol (5.4).

5.10

Formic acid aqueous solution.

Pipette 100 μl of formic acid (5.5) into 1000 ml of water.

5.11

Standard stock solution of aniline and benzidine compounds. $\rho = 100 \text{ mg/L} \sim 200 \text{ mg/L}$.

It can be prepared with standard material, the purity of the standard material is more than 99%, dissolved in methanol (5.4), frozen below -18°C , sealed and protected from light. Save it for 3 months. You can also directly purchase certified standard solutions and store them with reference to the product instructions.

5.12

Standard intermediate solution for aniline and benzidine compounds. $\rho = 5.00 \text{ mg/L} \sim 10.0 \text{ mg/L}$.

Dilute the standard stock solution (5.11) of aniline and benzidine compounds with methanol (5.4), the concentration of 3-nitroaniline is 10.0 mg/L, its the concentration of the remaining compounds was 5.00 mg/L. The intermediate solution should be frozen, sealed, and protected from light below -18°C , and N-nitroso diphenylamine can be stored for 1 months,

the remaining compounds can be stored for 3 months.

5.13

Internal standard stock solution. $\rho=1000$ mg/L.

Recommended internal standards are benzidine-d8, aniline-d5, 3,3'-dichlorobenzidine-d6 and N-nitrosodiphenylamine-d6, and isotopic substitutions are also available for other aniline compounds, see Appendix B. It can be prepared with standard material, the purity of the standard material is more than 99%, dissolved in methanol (5.4), Freeze, seal, and protect from light below -18°C for 3 months. The standard solution can also be purchased directly and stored with reference to the product manual.

5.14

Internal standard intermediate solution. $\rho=20.0$ mg/L.

Dilute the internal standard stock solution (5.13) with methanol (5.4), and the concentration of each internal standard is 20.0 mg/L. The intermediate solution is frozen below -18°C , Sealed and protected from light, N-nitrosodiphenylamine-d6 can be stored for 1 month, and other internal standards can be stored for 3 months.

5.15

Internal standard solution I. $\rho=2.00$ mg/L.

Dilute the internal standard intermediate solution (5.14) with methanol (5.4), the concentration of each internal standard is 2.00 mg/L. The solution should be frozen below -18°C , Sealed and protected from light, it can be stored for 1 month.

5.16

Internal standard solution II. $\rho=50.0$ $\mu\text{g/L}$.

Dilute the internal standard intermediate solution (5.14) with water, and add an appropriate amount of methanol (5.4), the concentration of each internal standard is 50.0 $\mu\text{g/L}$, and the methanol the product ratio is 25%, and it is ready for immediate use.

5.17

Standard solution for aniline and benzidine compounds. $\rho=1.00$ mg/L~2.00 mg/L.

Dilute the standard intermediate solution (5.12) of aniline and benzidine compounds with water, and add an appropriate amount of internal standard solution I (5.15) and methanol (5.4), the concentration of 3-nitroaniline was 2.00 mg/L, the concentration of other compounds was 1.00 mg/L, the concentration of internal standard was 50.0 $\mu\text{g/L}$, and the the volume ratio of alcohol is 25%, and it is prepared for immediate use.

5.18

Quartz sand. particle size of 0.150 mm to 0.450 mm (100 mesh to 40 mesh).