

HJ

INDUSTRY STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

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**Soil and sediment Determination of polychlorinated
dibenzo-p-dioxins (PCDDs) and polychlorinated dibenzofurans
(PCDFs) Isotope dilution HRGC-LRMS**

土壤、沉积物 二噁英类的测定 同位素稀释／高分辨气相色谱－低分辨质谱法
(English Translation)

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5.13 Sodium hydroxide alkaline silica gel. $\omega = 33\%$

5.14 Silicate Sulfuric Acid. $\omega = 44\%$

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5.19 Dichloromethane-n-hexane solution. 3% (v/v)

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Foreword

To protect the environment and protect the human body in order to implement the Environmental Protection Law of the People's Republic of China and the Law of the People's Republic of China on Water Pollution Prevention and Control this standard is developed for high-resolution gas chromatography-low resolution mass spectrometry monitoring of dioxin-like contaminants in soils and sediments.

This standard specifies high resolution gas chromatography-low resolution mass spectrometry for dioxins in soils and sediments.

This standard is the first release.

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

This standard was formulated by the Science and Technology Standards Department of the Ministry of Environmental Protection.

This standard is mainly drafted by. Zhejiang Environmental Monitoring Center, Environmental Protection Standards Institute of the Ministry of Environmental Protection.

This standard is verified by. National Environmental Analysis and Testing Center, Liaoning Provincial Environmental Monitoring Center, Hubei Environmental Monitoring Center, Ningbo City Ring Environmental monitoring center this standard was approved by the Ministry of Environmental Protection on June 3, 2013.

This standard has been implemented since September 1, 2013.

This standard is explained by the Ministry of Environmental Protection.

Determination of soil, sediment dioxin class isotope dilution /High resolution gas chromatography-low resolution mass spectrometry Warning. The standard and organic reagents used in the experiment are all toxic compounds, and their solution preparation, pretreatment, etc.

It should be carried out in a fume hood, and protective equipment should be worn as required to avoid contact with skin and clothing.

1 Scope of application

This standard specifies the determination of polychlorinated dibenzodioxins and polychlorinated dibenzofurans in soils and sediments.

Resolution gas chromatography-low resolution mass spectrometry.

This standard applies to the preliminary screening of dioxins in soils and sediments, mainly including polychlorinated from tetrachloro to octachloro.

Determination of dibenzodioxin and dibenzofuran by high resolution gas chromatography/low resolution mass spectrometry. Accident arbitration, construction Project evaluation and acceptance are recommended using high resolution mass spectrometry methods such as HJ 77.4.

The detection limits of this standard method vary with the sensitivity of the instrument, the concentration of dioxin in the sample, and the level of interference. Earth when the soil sample volume is 20g, the detection limit for 2,3,7,8-T4CDD should be less than 1.0 ng/kg, see Table 1.

2 Normative references

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

Used in this standard.

GB/T 8170 Numerical Rounding Rules and Representation and Determination of Limit Values GB 17378.3 Marine monitoring specification Part 3. Sample collection, storage and transport HJ 77.4 Determination of Dioxins in Soils and Sediments Isotope Dilution High Resolution Gas Chromatography-High Resolution Mass Spectrometry HJ/T 166 Technical Specifications for Soil Environmental Monitoring HJ 613 Determination of dry matter and moisture in soils - Gravimetric method

3 Terms and definitions, symbols and abbreviations

The following terms and definitions apply to this standard.

3.1.1 Dichlorinated dibenzodioxins (PCDDs) and Polychlorinated dibenzofurans (PCDFs)

A collective term for polychlorinated dibenzo-p-dioxins (PCDDs) and polychlorinated dibenzofurans (PCDFs).

3.1.2 Congener

All compounds of dioxins are similar to each other. There are 210 congeners in the dioxin class.

3.1.3 2,3,7,8-chlorinated dioxins PCDDs/PCDFs isomer substituted at 2,3,7,8-positions
Refers to dioxin-like congeners with chlorine atoms at the 2, 3, 7, and 8 positions. Among them, there are 7 kinds of polychlorinated dibenzo-p-dioxins.

There are 10 kinds of chlorodibenzofurans, and there are 17 kinds, see Table 1.

3.1.4 Dioxin internal standard Internal standard for PCDDs/PCDFs analysis

See Table 2 for the dioxin-like standard substances labeled with known concentrations of isotope (^{13}C or ^{37}Cl). Including sample preparation process Added purified internal standard and injection internal standard.

3.1.5 Toxicity equivalency factor, referred to as TEF

Refers to the ratio of dioxin-like congeners to 2,3,7,8-tetrachlorodibenzo-p-dioxin to aromatic hydrocarbon (Ah) receptor affinity.

3.1.6 Toxic equivalent quantity, referred to as TEQ

The mass fraction of each dioxin-like congener is converted to the equivalent mass of 2,3,7,8-tetrachlorodibenzo-p-dioxin Fraction, toxic equivalent mass fraction is the product of the measured mass fraction and the toxic equivalent factor of the isomer.

Table 1 Detection limits of 2,3,7,8-chlorodioxins and methods

Serial number	isomer name	short name	detection limit (ng/kg)	1
2,3,7,8-tetrachlorodibenzo-p-dioxin	2,3,7,8-T4CDD	0.3	2	
1,2,3,7,8-pentachlorodibenzo-p-dioxin class	1,2,3,7,8-P5CDD	0.5	3	
1,2,3,4,7,8-hexachlorodibenzo-p-dioxin class	1,2,3,4,7,8-H6CDD	0.6	4	
1,2,3,6,7,8-hexachlorodibenzo-p-dioxin class	1,2,3,6,7,8-H6CDD	0.6	5	
1,2,3,7,8,9-hexachlorodibenzo-p-dioxin class	1,2,3,7,8,9-H6CDD	0.6	6	
1,2,3,4,6,7,8-heptachlorodibenzo-p-dioxin class	1,2,3,4,6,7,8-H7CDD	1.2	7	
octachlorodibenzo-p-dioxin	OCDD	1.7	8	
2,3,7,8-tetrachlorodibenzofuran	2,3,7,8-T4CDF	0.2		
1,2,3,7,8-pentachlorodibenzofuran	1,2,3,7,8-P5CDF	0.3	10	
2,3,4,7,8-pentachlorodibenzofuran	2,3,4,7,8-P5CDF	0.3	11	
1,2,3,4,7,8-hexachlorodibenzofuran	1,2,3,4,7,8-H6CDF	0.4	12	
1,2,3,6,7,8-hexachlorodibenzofuran	1,2,3,6,7,8-H6CDF	0.4	13	
1,2,3,7,8,9-hexachlorodibenzofuran	1,2,3,7,8,9-H6CDF	0.4	14	
2,3,4,6,7,8-hexachlorodibenzofuran	2,3,4,6,7,8-H6CDF	0.4	15	
1,2,3,4,6,7,8-heptachlorodibenzofuran	1,2,3,4,6,7,8-H7CDF	1.4	16	
1,2,3,4,7,8,9-heptachlorodibenzofuran	1,2,3,4,7,8,9-H7CDF	1.4	17	
octachlorodibenzofuran	OCDF	1.4		

Table 2 Available Dioxin Internal Standards

Replace the number of chlorine atoms	PCDDs	PCDFs	Tetrachloro	^{37}Cl	4-2,3,7,8-T4CDD	
^{13}C	12-2,3,7,8-T4CDD	^{13}C	12-1, 2, 3, 4-T4CDD	^{13}C	12-1, 2, 7, 8-T4CDF	
^{13}C	12-2,3,7,8-T4CDF	Pentachloro	^{13}C	12-1,2,3,7,8-P5CDD	^{13}C	12-2,3,4,7,8-P5CDF
^{13}C	12-1,2,3,7,8-P5CDF	Hexachloro	^{13}C	12-1, 2, 3, 7, 8, 9-H6CDD	^{13}C	12-1, 2, 3, 4, 7, 8-H6CDD
^{13}C	12-1,2,3,6,7,8-H6CDD	^{13}C	12-2,3,4,6,7,8-H6CDF	^{13}C	12-1,2,3,6,7,8-H6CDF	