

HJ

INDUSTRY STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

HJ 779-2015

**Ambient air. Determination of hexavalent chromium(?). Ion
chromatography with post-column method**

环境空气 六价铬的测定 柱后衍生离子色谱法 含2018年第1号修改单

(English Translation)

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Foreword

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 the Prevention and Control of Atmospheric Pollution, Protection of human health, regulate the ambient air particles in the determination of hexavalent chromium, the development of this standard.

This standard specifies the determination of hexavalent chromium in ambient air particulate matter by ion chromatography.

This standard is the first release.

This standard is organized by the Ministry of Environmental Protection Science and Technology Standards Division.

The main drafting of this standard. China Environmental Monitoring Station, the National Environmental Analysis and Testing Center.

The standard verification unit. Beijing Environmental Protection Monitoring Center, Beijing Chaoyang District Environmental Protection Monitoring Station, Tianjin Ring Environmental Monitoring Center, Jiangsu Province Environmental Monitoring Center, Hunan Provincial Environmental Monitoring Center Station, Chongqing Environmental Monitoring Center, Sichuan Province Environmental monitoring terminus.

This standard is approved by the Ministry of Environmental Protection on December 4, 2015.

This standard has been implemented since January 1, 2016.

This standard is explained by the Ministry of Environmental Protection.

Determination of hexavalent chromium in ambient air - post - column derivatization ion chromatography WARNING. Potassium dichromate and diphenylcarbonylate used in this method are highly toxic and should be avoided during contact with skin and clothing.

1 Scope of application

This standard specifies the determination of hexavalent chromium in ambient air particulate matter by ion chromatography.

This standard is applicable to the determination of hexavalent chromium in ambient air particulate matter.

When the sampling volume is 21 m³ (standard state), the volume of the extract is 10 ml and the injection volume is 1 ml.

The limit is 0.005 ng/m³ and the lower limit of determination is 0.020 ng/m³.

2 Normative reference documents

The contents of this standard refer to the following documents or their terms. Those who do not specify the date of the reference file, the effective version of the appropriate for this standard.

Technical specification for manual monitoring of ambient air quality Technical Requirements and Test Methods for HJ 93 Environmental Air Particulate (PM₁₀ and PM_{2.5}) Sampler Determination of ambient air PM₁₀ and PM_{2.5} by HJ 618

3 Principle of the method

In the ambient air particulate matter, hexavalent chromium present in the form of chromate and dichromate is collected into the finely treated fibers Filter, under alkaline conditions by ultrasonic extraction, anion column separation, and the developer diphenyl carbonyl hydrazine formation the absorbance, and the concentration of hexavalent chromium in the sample were measured at 530 nm with a UV-visible detector is proportional to it.

4 Interference and elimination

When the ambient air of ferrous iron, nitrite, thiosulfate and other strong reducing ion concentration is high, with hexavalent chromium the occurrence of redox reaction, resulting in negative interference. A higher content of soluble ferric ions may also cause negative interference to the assay.

The extraction and determination in the alkaline environment can inhibit the conversion of hexavalent chromium, and can avoid the interference of ferric ions.

5 Reagents and materials

Unless otherwise stated, analytical pure chemical reagents are used in accordance with national standards. Experimental water for the resistivity ≥ 18.2 Mohm · cm of deionized

water.

5.1

sodium bicarbonate (NaHCO_3). excellent grade pure.

5.2

ammonium sulfate. $\rho [(\text{NH}_4)_2\text{SO}_4] = 1.77 \text{ g/ml}$, excellent grade pure.

5.3

ammonia. $\rho (\text{NH}_3 \cdot \text{H}_2\text{O}) = 0.91 \text{ g/ml}$, excellent grade pure.

5.4

Diphenylcarbazide ($\text{C}_{13}\text{H}_{14}\text{N}_4\text{O}$).

5.5

Methanol. $\rho (\text{CH}_3\text{OH}) = 0.79 \text{ g/ml}$, HPLC grade, purity greater than 99.9%.

5.6

Sulfuric acid. $\rho (\text{H}_2\text{SO}_4) = 1.84 \text{ g/ml}$, excellent grade pure.

5.7

potassium dichromate ($\text{K}_2\text{Cr}_2\text{O}_7$). reference reagent or excellent grade pure.

Weigh 5.0 g of potassium dichromate in a porcelain crucible, placed in an oven at 105°C for 2 h, cooled to room temperature and placed in a dryer. Save, spare.

5.8

Sodium bicarbonate solution. $c (\text{NaHCO}_3) = 0.12 \text{ mol/L}$.

Weigh 5.0 g of sodium bicarbonate (5.1), dissolve in water and dilute to 500 ml.

5.9

Sodium bicarbonate solution. $c (\text{NaHCO}_3) = 20 \text{ mmol/L}$.

3.36 g of sodium bicarbonate (5.1) was weighed and dissolved in water and diluted to 2000 ml.

5.10

potassium dichromate stock solution. $\rho (\text{Cr (VI)}) = 1000 \text{ mg/L}$.

Accurately weighed 0.2829 g potassium dichromate (5.7) dissolved in water, transferred to 100 ml volumetric flask, dilute the volume to the line.

You can also purchase a commercially available standard material or a certified standard solution.

5.11

Potassium dichromate solution. $\rho(\text{Cr(VI)}) = 10 \text{ mg/L}$.

Accurately remove 0.5 ml of potassium dichromate stock solution (5.10) in 50 ml volumetric flask, dilute with sodium bicarbonate solution (5.9) Release the line.

5.12

potassium dichromate standard use of liquid. $\rho(\text{Cr(VI)}) = 100 \text{ ng/ml}$.

Accurately remove 0.5 ml of potassium dichromate intermediate solution (5.11) in a 50 ml volumetric flask with sodium bicarbonate solution (5.9) Dilute the volume to the mark.

5.13

eluent. 250 mmol/L ammonium sulfate 100 mmol/L ammonium hydroxide.

Weigh 66 g of ammonium sulfate (5.2) dissolved in water, add 14 ml of ammonia (5.3), shake, dilute the volume to 2000 ml.

Immediately transferred to the eluent bottle, can be added nitrogen protection, to slow down the alkaline eluent to absorb CO_2 in the air and failure.

5.14 Derivative reagents

Weigh 0.50 g of diphenylcarbazide (5.4), dissolved in methanol (5.5) and diluted to 100 ml with methanol (5.5).

Add 28 ml of concentrated sulfuric acid (5.6) slowly to 300 ml of water and dilute to 500 ml. Transfer the diphenylcarbazide solution to methanol Transferred to sulfuric acid aqueous solution, diluted with water to 1000 ml, and transferred to the derivative reagent bottle, can add nitrogen, pressure 6 psi ~ 9 Psi (1 psi = $6.89475 \times 10^3 \text{ Pa}$) to prevent air bubbles from occurring. Need to save the light, save time should not exceed 3 d.

5.15 sampling filter. cellulose material, 47 mm.

5.16 Water-based microporous membrane. pore size $0.22 \mu\text{m}$.

5.17 filter box. Teflon (PTFE) or polystyrene (PS) material.

5.18 Nitrogen. Purity $\geq 99.999\%$.

6 Instruments and equipment

6.1

ambient air particulate sampling equipment. sampling device from the sampling head, sampling pump and flow meter components. Sampling head is equipped with different Cutter can collect TSP, PM₁₀ and PM_{2.5} particles; flow meter for small flow, range 7 samples