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ENVIRONMENTAL PROTECTION STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

HJ 799-2016

Ambient air - Determination of the water soluble anions

(F⁻, Cl⁻, Br⁻, NO₂⁻, NO₃⁻, PO₄³⁻, SO₃²⁻, SO₄²⁻) from

atmospheric particles - Ion chromatography

环境空气 颗粒物中水溶性阴离子 (F⁻, Cl⁻, Br⁻, NO₂⁻, NO₃⁻, PO₄³⁻,
SO₃²⁻, SO₄²⁻) 的测定 离子色谱法

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Table of contents

Foreword.....	3
1 Scope	4
2 Normative references	4
3 Terms and definitions	5
4 Principles of the method	5
5 Interference and elimination	5
6 Reagents and materials	6
7 Instrument and equipment.....	10
8 Samples	11
9 Analysis steps	12
10 Results calculation and presentation.....	14
11 Precision and accuracy	15
12 Quality assurance and quality control.....	16
13 Waste disposal	17
14 Precautions	17
APPENDIX A (Normative) Method detection limit and lower measurement limit	18
APPENDIX B (Informative) Method precision and accuracy	19
Appendix C (Informative) Anion standard solution chromatogram	22

Foreword

This standard is formulated for the purpose of implementing the Law of the People's Republic of China on Environmental Protection and the Law of the People's Republic of China on the Prevention and Control of Atmospheric Pollution, protecting the environment, safeguarding human health, AND regulating the determination method of water-soluble anions in ambient airborne particles.

This standard specifies the ion chromatography method for the determination of eight water-soluble anions (F^- , Cl^- , Br^- , NO_2^- , NO_3^- , PO_4^{3-} , SO_3^{2-} , SO_4^{2-}) in the ambient airborne particles (TSP, PM_{10} , $PM_{2.5}$, dustfall, etc.).

This standard is firstly issued.

Appendix A of this standard is a normative appendix, AND Appendix B and Appendix C are informative appendixes.

This standard is formulated under the organization of the Department of Science, Technology and Standards of the Ministry of Environmental Protection.

The main drafting organizations of this standard: Gansu Provincial Environmental Monitoring Center Station.

The verification organizations of this standard: College of Chemistry and Chemical Engineering of Xiamen University, Center for Analysis and Testing of Guangzhou University, Environmental Research Center of Chinese Academy of Sciences, Jiayuguan City Environmental Monitoring Station, Environmental Monitoring Station of Hulunbeir of Inner Mongolia, Guangdong Polytechnic of Environmental Protection Engineering, Jingmen Environmental Protection Monitoring Station.

This standard was approved by the Ministry of Environmental Protection on May 13, 2016.

This standard has been implemented since August 1, 2016.

As for this standard, the Ministry of Environmental Protection is responsible for its interpretation.

Ambient air - Determination of the water soluble anions (F^- , Cl^- , Br^- , NO_2^- , NO_3^- , PO_4^{3-} , SO_3^{2-} , SO_4^{2-}) from atmospheric particles - Ion chromatography

Warning: The chemical reagents used in the experiment such as sodium fluoride and sodium nitrite are harmful to human health. **WEAR** protective equipment in accordance with the regulations, and **AVOID** contact with skin and clothing.

1 Scope

This standard specifies ion chromatography for the determination of water-soluble anions (F^- , Cl^- , Br^- , NO_2^- , NO_3^- , PO_4^{3-} , SO_3^{2-} , SO_4^{2-}) in ambient airborne particles.

This standard applies to the determination of 8 kinds of water-soluble anions (F^- , Cl^- , Br^- , NO_2^- , NO_3^- , PO_4^{3-} , SO_3^{2-} , SO_4^{2-}) in the ambient airborne particles (TSP, PM_{10} , $PM_{2.5}$, dustfall, etc.).

As for the filter sample of ambient airborne particles, when the sampling volume is 60 m^3 (standard state), the extract volume is 100 ml, AND the sample injection volume is 25 μl , the detection limit of this method is $0.010\text{ }\mu\text{g}/\text{m}^3 \sim 0.085\text{ }\mu\text{g}/\text{m}^3$, AND the lower detection limit is $0.040\text{ }\mu\text{g}/\text{m}^3 \sim 0.340\text{ }\mu\text{g}/\text{m}^3$. As for the dustfall sample, when the sample injection amount is 0.100 g, the extract volume is 100 ml, AND the sample injection volume is 25 μl , the detection limit of this method is $0.006\text{ }\mu\text{g}/\text{g} \sim 0.051\text{ }\mu\text{g}/\text{g}$, AND the lower detection limit is $0.024\text{ }\mu\text{g}/\text{g} \sim 0.204\text{ }\mu\text{g}/\text{g}$. As for details, SEE Appendix A.

Note 1: This method is also applicable to the analysis of samples collected from particles such as dust aerosols and dust storms in special weather conditions.

2 Normative references

The following documents or the terms thereof are referenced in this standard. For undated references, the valid version of the referenced document applies to this standard.

GB/T 15265 Ambient air - Determination of dustfall - Gravimetric method

GB/T 15432 Ambient air - Determination of total suspended particulates - Gravimetric method

HJ 93 Specifications and test procedures for PM₁₀ and PM_{2.5} sampler

HJ 618 Determination of atmospheric particles PM₁₀ and PM_{2.5} in ambient air by gravimetric method

HJ/T 194 Manual methods for ambient air quality monitoring

HJ/T 374 Technical requirement and test procedures for total suspended particulates sampler

HJ/T 375 Technical requirement and test procedures for ambient air sampler

3 Terms and definitions

The following terms and definitions apply to this standard.

3.1 Ambient air atmospheric particles

It refers to the ambient air atmospheric particles collected on the filter membrane the sampling device (including TSP, PM₁₀, PM_{2.5}, etc.), OR the ambient air dustfall collected through such collection devices as dust cylinder.

3.2 Water-soluble anions

It refers to the anions of the ambient air atmospheric particles which are dissolved in deionized water without adding chemical reagent. The water-soluble anions determined by this method include F⁻, Cl⁻, Br⁻, NO₂⁻, NO₃⁻, PO₄³⁻, SO₃²⁻, SO₄²⁻.

4 Principles of the method

The ambient air atmospheric particle samples collected is, after being subjected to ultrasonic extraction by deionized water AND exchange and separation by anion chromatographic column, detected by a suppressed conductivity detector. MAKE qualitative determination based on the retention time; and MAKE quantitative determination based on peak height of peak area.

5 Interference and elimination

5.1 For the 2 kinds of anion with similar retention time, when the concentration of the 2 kinds of anions is greatly different that it affects the determination of the

6.17 Nitrate ion standard stock solution: $\rho(\text{NO}_3^-) = 1000 \text{ mg/L}$.

Accurately WEIGH 1.6304 g of potassium nitrate (6.5); DISSOLVE it into appropriate amount of water; TRANSFER it into a 1000 ml volumetric flask; USE water to dilute it to the mark; MIX it uniformly. TRANSFER it into the polyethylene bottle; KEEP it sealed at below 4 °C in the dark; AND it can be preserved for 6 months. It may also purchase the commercially available certified standard materials.

6.18 Phosphate ion standard stock solution: $\rho(\text{PO}_4^{3-}) = 1000 \text{ mg/L}$.

Accurately WEIGH 1.4316 g of potassium dihydrogen phosphate (6.6); DISSOLVE it into appropriate amount of water; TRANSFER it into a 1000 ml volumetric flask; USE water to dilute it to the mark; MIX it uniformly. TRANSFER it into the polyethylene bottle; KEEP it sealed at below 4 °C in the dark; AND it can be preserved for 1 month. It may also purchase the commercially available certified standard materials.

6.19 Sulfite ion standard stock solution: $\rho(\text{SO}_3^{2-}) = 1000 \text{ mg/L}$.

Accurately WEIGH 1.5750 g sodium sulfite (6.7); DISSOLVE it into appropriate amount of water; TRANSFER it into a 1000 ml volumetric flask; ADD 1 ml of formaldehyde (6.8) to prevent oxidation of SO_3^{2-} ; USE water to dilute it to the mark; MIX it uniformly. TRANSFER it into the polyethylene bottle; KEEP it sealed at below 4 °C in the dark; AND it can be preserved for 1 month.

6.20 Sulfate ion standard stock solution: $\rho(\text{SO}_4^{2-}) = 1000 \text{ mg/L}$.

Accurately WEIGH 1.4792 g anhydrous sodium sulfate (6.9); DISSOLVE it into appropriate amount of water; TRANSFER it into a 1000 ml volumetric flask; USE water to dilute it to the mark; MIX it uniformly. TRANSFER it into the polyethylene bottle; KEEP it sealed at below 4 °C in the dark; AND it can be preserved for 6 months. It may also purchase the commercially available certified standard materials.

6.21 Mixed standard solution

Respectively PIPETTE 10.0 ml of fluoride ion standard stock solution (6.13), 100.0 ml of chloride ion standard stock solution (6.14), 10.0 ml of bromide ion standard stock solution (6.15), 10.0 ml of nitrite standard stock solution (6.16), 100.0 ml of nitrate standard stock solution (6.17), 50.0 ml of phosphate ion standard stock solution (6.18), 50.0 ml of sulphite ion standard stock solution (6.19), AND 200.0 ml of sulphate ion standard stock solution (6.20) into a 1000 ml volumetric flask; USE water to dilute it to the mark; MIX it uniformly. PREPARE it into the mixed standard solution containing 10 mg/L of F^- , 100

7 Instrument and equipment

7.1 Ambient air atmospheric particles sampler: the sampling unit consists of a sampling head, a sampling pump, and a flowmeter. The sampling head is equipped with different cutting devices to collect the particles of TSP, PM₁₀, and PM_{2.5}; the flowmeter is of medium range, with the range of 60 L/min ~ 125 L/min and flow rate indication error ≤ 2%; AND the other performance and technical indicators shall comply with the relevant provisions of HJ 93, HJ/T 194, HJ/T 374, and HJ/T 375.

7.2 Environmental air dustfall sampling cylinder.

7.3 Sampling filter: SELECT the high quality filter membrane made from glass fiber, quartz or other materials with low blank value, as well as the products which comply with the technical requirements of particle sampling products.

7.4 Ion chromatograph: It is an analytical system consisting of an ion chromatograph, operating software and required accessories.

7.4.1 Chromatograph column: it is composed of anion separation columns (the chromatograph column of polydivinylbenzene/ethylvinylbenzene/polyvinyl alcohol matrix, with alkyl quaternary ammonium or alkanol quaternary ammonium functional groups, hydrophilic, and high capacity) AND anion protection column. One sample injection can be used to determine 8 water-soluble anions as specified in this method; AND the peak separation is not less than 1.5.

7.4.2 Anion suppressors.

7.4.3 Conductivity detector.

7.5 Filter cartridge: Polytetrafluoroethylene (PTFE) or polystyrene (PS) material.

7.6 Vials: hard glass or polyethylene material, volume ≥ 100 ml, with screw cap.

7.7 Ultrasonic cleaner: frequency 40 KHz ~ 60 KHz.

7.8 Exhaust filter: with a pore size ≤ 0.45 μm acetate or polyethylene filter.

7.9 Sample tube: polypropylene (PP) or Teflon (PTFE), with screw cap.

7.10 Disposable water-based microporous membrane syringe filter: 0.45 μm pore size.

7.11 Disposable syringes: 1 ml to 10 ml.

7.12 Common laboratory instruments and equipment.

Particle filter lab blank test specimen: USE the blank filter of the same batch as the sampling and FOLLOW the procedures same as the particle filter test specimen preparation (8.4.1) to prepare the test specimen. The dustfall lab blank test specimen: DO not take dustfall sample; FOLLOW the procedures same as the dustfall test specimen preparation (8.4.2) to prepare the test specimen.

8.6 Preparation of whole procedure blank test specimen

TAKE the blank filter which is of the same batch as sampling to the sampling site; DO not take particle sample; FOLLOW the requirements for sample transport and preservation (8.3), to take it together with the sample back to the laboratory; FOLLOW the procedures same as the particle filter test specimen preparation (8.4.1) to conduct preparation.

9 Analysis steps

9.1 Reference conditions for ion chromatography analysis

Based on the instrument manual, OPTIMIZE the measurement conditions or parameters; AND it may follow the matrix and composition of the actual sample to optimize the eluent concentration. The conditions of ion chromatographic analysis given below are provided for reference.

9.1.1 Reference condition 1

Anion separation column (7.4.1): Carbonate eluent I (6.22.1), flow rate: 1.0 ml/min, suppressed conductivity detector, continuous self-recirculation suppressor; OR carbonate eluent II (6.22.2) ml/min, flow rate: 0.7 mL/min, suppressed conductivity detector, continuous self-recirculation suppressor, CO₂ suppressor. Sample injection volume: 25 µl. Under this reference conditions, the chromatogram of standard solution is shown in Figure C.1 and Figure C.2 in Appendix C.

9.1.2 Reference condition 2

Anion separation column (7.4.1): hydroxyl eluent (6.22.3), flow rate: 1.2 ml/min. The gradient elution conditions are shown in Table 1; suppressed conductivity detector, continuous self-recirculation suppressor. Sample injection volume: 25 µl. Under this reference conditions, the chromatogram of standard solution is shown in Figure C.3 in Appendix C.

Note 2: If the concentration of anions to be measured exceeds the standard curve, the test specimen and the laboratory blank shall be measured after diluted to the same times; RECORD the dilution factor (D).

9.4 Measurement of laboratory blank test specimens

In accordance with the chromatographic conditions (9.1) and procedures(9.2) same as those used for drawing the standard curve, INJECT the laboratory blank test specimen (8.5) into the ion chromatograph to determine the anion concentration; USE the retention time for qualitative AND the instrument response value for quantification.

9.5 Measurement of whole procedure blank test specimen

In accordance with the chromatographic conditions (9.1) and procedures(9.2) same as those used for drawing the standard curve, INJECT the whole procedure blank test specimen (8.6) into the ion chromatograph to determine the anion concentration; USE the retention time for qualitative AND the instrument response value for quantification.

10 Results calculation and presentation

10.1 Calculation of water-soluble anion content of ambient air atmospheric particle (filter sample)

The mass concentration (ρ , $\mu\text{g}/\text{m}^3$) of the water-soluble anions (F^- , Cl^- , Br^- , NO_2^- , NO_3^- , PO_4^{3-} , SO_3^{2-} , SO_4^{2-}) in the filter sample is calculated in accordance with the equation (1):

$$\rho = \frac{(\rho_1 - \rho_0) \times V \times N \times D}{V_{\text{nd}}} \quad (1)$$

Where:

ρ - Mass concentration of anion in filter sample, $\mu\text{g}/\text{m}^3$;

ρ_1 - Mass concentration of anion in the test specimen, mg/L ;

ρ_0 - the average concentration of anion in the filter laboratory blank test specimen, mg/L ;

V - Extract volume, 100.0 ml;

the inner-lab relative standard deviation range is 0.1% ~ 5.7%, AND the inter-lab relative standard deviation range is 1.1% ~ 5.8%. As for the method precision measurement results, SEE Table B.1 in Appendix B.

11.2 Accuracy

Seven labs conducted the recovery measurement of the marked ambient air atmospheric particle sample of different types, AND the marked recovery range is from 81.7% to 118.3%. As for the method precision measurement results, SEE Table B.2 in Appendix B.

12 Quality assurance and quality control

12.1 As for the ambient air atmospheric particle sampling equipment (7.1), it shall, before each time of sampling, be subjected to flow rate calibration and air tightness inspection; AND the other quality assurance and quality control measures shall follow the relevant requirements of HJ/T 194.

12.2 Each batch (≤ 20) particle filter samples or dustfall samples shall be subjected to at least 2 lab blanks, the measurement result of lab blank shall be lower than the method detection lower limit, AND the relative deviation of the blank parallel sample measurement value shall be $\leq 20\%$.

12.3 Each batch (≤ 20) particle filter samples shall be subjected to at least 1 whole procedure blank, AND the measurement results of the whole procedure blank shall be lower than the method detection lower limit. Otherwise it shall find the causes AND re-sample for analysis, until it is qualified, before conducting sample measurement.

12.4 The correlation coefficient of the standard curve shall be ≥ 0.995 . Each batch (≤ 20) particle filter samples or dustfall samples shall be subjected to one standard solution analysis for which the concentration is located at the middle of the standard curve, AND the relative error between the measurement result and the point concentration at the standard curve shall be $\leq 10\%$. Otherwise, it shall re-draw the standard curve.

12.5 Each batch (≤ 20) particle filter samples or dustfall samples shall be subjected to 10% of the parallel double samples; when the sample quantity is less than 10, it shall measure at least one parallel double sample. AND the relative deviation of the measurement results of the parallel double sample shall be $\leq 20\%$.

12.6 Each batch (≤ 20) particle filter samples or dustfall samples shall be subjected to one marked recovery measurement, AND the actual sample marked recovery shall be controlled between 80% and 120%.

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