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NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

GB/T 11446.7-2013

Replacing GB/T 11446.7-1997

Test method for trace anion in electronic grade water by ion chromatography

电子级水中痕量阴离子的离子色谱测试方法

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Foreword

The expected structure of GB/T 11446 is as follows: - GB/T 11446.1 Electronic grade water; - GB/T 11446.2 (To be determined); - GB/T 11446.3 Generic rules for test methods of electronic grade water; - GB/T 11446.4 Test method for resistivity of electronic grade water; - GB/T 11446.5 Test method for measuring trace metals in electronic grade water by atomic absorption spectrophotometry; - GB/T 11446.6 Test method for SiO2 in electronic grade water by spectrophotometer; - GB/T 11446.7 Test method for trace anion in electronic grade water by ion chromatography; - GB/T 11446.8 Test method for total organic carbon in electronic grade water; - GB/T 11446.9 Test method for particles in electronic grade water by instrument; - GB/T 11446.10 Test method for total bacterial count in electronic grade water by membrane filters. This part is Part 7 of GB/T 11446. This part was drafted in accordance with the rules given in GB/T 1.1-2009. This part replaces GB/T 11446.7-1997 "Test method for trace anion in electronic grade water by ion chromatography". Compared with GB/T 11446.7-1997, this Part mainly has the following changes. - ADD the determination method of trace amounts of "fluoride ion", "nitrite ion", "bromide ion"; -

INCLUDE the reaction formulas of the working principle of ion chromatography, in Appendix A; - ADD the "Disturbing factors" (see Chapter 5); - DELETE the "Precautions" (see Chapter 10 of the 1997 edition). This part was proposed by the Ministry of Industry and Information Technology of the People's Republic of China. This part shall be under the jurisdiction of China Electronics Standardization Institute. Drafting organizations of this Part. Special Material Quality Supervision and Inspection Center of the Ministry of Information Industry, Institute of Semiconductors of Chinese Academy of Sciences, China Electronics Standardization Institute, 46th Research Institute of China Electronics Technology Group Corporation. The main drafters of this Part. Wang Yi, Chu Lianqing, He Xiukun, Duan Shuguang, Ti Liuwang, Liu Yun. This standard replaces the standard previously issued as follows: - GB/T 11446.7-1989, GB/T 11446.7-1997. Test method for trace anion in electronic grade water by ion chromatography

1 Scope

GB/T 11446.7-2013 is the Chinese national standard on test method for trace anion in electronic grade water by ion chromatography, in the field of electronics. The /T suffix marks it as a recommended standard: it is not compulsory by itself, but it becomes binding as soon as a contract, a tender or a customer specification calls it up - which in practice is how most foreign buyers meet it. It was issued on 31 December 2013 by the General Administration of Quality Supervision, Inspection and Quarantine of the People's Republic of China; Standardization Administration of China, and has been in force since 15 August 2014. Classification: ICS 31.030, CCS L90. This page is published from the official record of the standard held by the Chinese standards administration: the identification, the dates, the classification and the issuing body are taken from there. The clause text, the tables and the numeric limits are in the document itself, which is delivered complete in English translation.

This Part of GB/T 11446 specifies ion chromatography test method, for the trace amounts of fluoride ion (F-), chloride ion (Cl-), nitrite ion (NO2-), bromide ion (Br-), nitrate ion (NO3-), phosphate ion (PO43-), sulfate ion (SO42-), in electronic grade water. This Part is applicable to the determination of trace amounts of fluoride ion, chloride ion, nitrite ion, bromide ion, nitrate ion, phosphate ion, sulfate ion, in electronic grade water. The detection limit is 0.7 μg/L, 0.8 μg/L, 1 μg/L, 1 μg/L, 1 μg/L, 1 μg/L, 1 μg/L, respectively.

2 Normative references

The following documents are essential to the application of this document. For the dated documents, only the versions with the dates indicated are applicable to this document; for the undated documents, only the latest version (including all the amendments) is applicable to this standard.

GB/T 11446.1-2013 Electronic grade water

GB/T 11446.3-2013 Generic rules for test methods of electronic grade water

3 Terms and definitions

The terms and definitions as defined in GB/T 11446.3-2013, as well as the following terms and definitions, apply to this document.

4 Principles of the method

The ion chromatograph is composed of eluent reservoir, pump, injection valve, separation column, suppression column (or suppressor), conductivity detector, data processor, etc. When analyzing anions in water, the separation column is filled with cation exchange resin, which has low exchange capacity. The eluent is a dilute alkali solution. When the eluent and the sample flow through the separation column, the cations in the sample and the eluent pass smoothly, whilst the anion A (A. F-, Cl-, NO2-, Br-, NO3-, PO43-, SO42-, etc.) compete with the anions in the eluent, to replace the position of the hydroxide (OH-) on the resin (RN+ - OH-), in the separation column. See Appendix A, for the reaction formula.

5 Disturbing factors

5.1 Environmental conditions shall be strictly controlled. The instrument shall be placed in a temperature-adjustable ultra-clean environment.

5.2 When analyzing the specimen, every time the specimen is analyzed, it shall inject the blank water immediately.

5.3 The anion chromatogram changes with the instrument parameters and working conditions. The standard solution shall be calibrated, for each measurement, to determine the position of each anion chromatogram.

6 Reagents

6.1 Blank water EW-I electronic grade water, which is specified in GB/T 11446.1-2013.

6.2.1 Fluoride standard stock solution (1 mL solution contains

0.1 mg of F-) Accurately weigh 0.2211 g of sodium fluoride (excellent grade pure). Dissolve it in blank water. Dilute it to the mark, in a 1 L volumetric flask. Store in a polyethylene bottle.

6.2.5 Nitrate standard stock solution (1 mL solution contains

0.1 mg of NO3-) Accurately weigh 0.1630 g of potassium nitrate (excellent grade pure), which was baked at 120 °C ~ 130 °C to constant weight. Dissolve it in blank water. Dilute it to the mark, in a 1 L volumetric flask.

6.2.6 Sulfate standard stock solution (1 mL solution contains

0.1 mg of SO42-)

6.2.7 Phosphoric acid standard stock solution (1 mL solution contains

0.1 mg of PO43-) Accurately weigh 0.1727 g of sodium phosphate (excellent grade pure). Dissolve it in blank water. Dilute it to the mark, in a 1 L volumetric flask.

6.3 Eluent Weigh

6.30 g of sodium bicarbonate (excellent grade pure). Dissolve it in blank water. Then add

21.20 g of sodium carbonate (excellent grade pure). Mix well. Use blank water, to dilute it to 1 L, to prepare the stock solution for elution, which contains

0.075 mol/L sodium bicarbonate and

0.200 mol/L sodium carbonate. Use blank water to dilute it 100 times, before use.

7 Measuring instruments

7.1 High-efficiency ion chromatograph and accessories, such as high-efficiency anion separation column, trace analysis column, suppression column (or suppressor), guard column, conductivity detector, recording system, etc.

7.2 Analytical balance. The division is

0.1 mg.

8 Operation procedures

8.1 Preparation of standard series solutions Accurately measure an appropriate amount of standard stock solution. Dilute it step by step, to prepare a corresponding series of mixed standard solutions, which contain fluoride ion, chloride ion, nitrite ion, bromide ion, nitrate ion, phosphate ion, sulfate ion.

8.4 Drawing of working curve Under the best working conditions of the instrument, measure the standard series of solutions. Record the peak conductivity of each chromatogram. Use the concentration as the abscissa and the conductivity as the ordinate, to draw the working curve.

8.5 Analysis of water samples Under the same conditions as drawing the working curve, carry out analysis and determination of the water sample. From the measured conductivity, calculate the content of fluoride ion, chloride ion, nitrite ion, bromide ion, nitrate ion, phosphate ion, sulfate ion, in the water sample, from the standard working curve.