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NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

GB/T 44242-2024

Hydrogen for proton exchange membrane fuel cell vehicles - Determination of inorganic halides and formic acid - Ion chromatography

质子交换膜燃料电池汽车用氢气 无机卤化物、甲酸的测定 离子色谱法

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1 Scope

GB/T 44242-2024 specifies the determination of inorganic halides and formic acid in the hydrogen used by fuel cell vehicles, by ion chromatography. Halides are among the most damaging impurities in the fuel: chloride attacks the catalyst and corrodes the bipolar plates and the effect is cumulative and irreversible, which is why the limit is set at a few parts per billion. Formic acid is limited for the same reason. Measuring either at that level in a gas means trapping it out of a large, precisely known volume of hydrogen into a small volume of water and then analysing that, and the standard covers the whole chain: the principle, the reagents and materials, the apparatus and the absorption system, the sampling and the volume measurement, the preparation of the standard solutions, the chromatographic conditions and the suppression, the calibration, the analytical procedure, the calculation back to a concentration in the gas, the precision and the limit of quantification. For a hydrogen supplier or a testing laboratory in China, this is the prescribed method.

This document describes the principle, reagents and materials, instruments and equipment, test procedures, test data processing, precision, quality assurance and control, and test report for the determination of inorganic halogenated compounds and formic acid in

hydrogen for proton exchange membrane fuel cell vehicles by ion chromatography. This document is applicable to the determination of inorganic halogenated compounds and formic acid in hydrogen for proton exchange membrane fuel cell vehicles. The determination of inorganic halogenated compounds and formic acid in hydrogen for other purposes shall be carried out in reference. The determination range of inorganic halogenated compounds and formic acid in hydrogen is 0.025 μmol/mol~0.5 μmol/mol. The detection limits of hydrogen chloride, chlorine, hydrogen bromide and formic acid components in hydrogen are 0.014 μmol/mol, 0.007 μmol/mol, 0.002 μmol/mol and 0.001 μmol/mol, respectively. NOTE. Inorganic halogenated compounds in this document refer to hydrogen chloride, chlorine and hydrogen bromide which may be present in hydrogen gas.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

GB/T 629, Chemical reagent. Sodium hydroxide

GB/T 637, Chemical reagent -- Sodium thiosulfate pentahydrate

GB/T 639, Chemical reagent -- Sodium carbonate anhydrous

GB/T 640, Chemical reagent -- Sodium hydrogen carbonate

GB/T 6682, Water for analytical laboratory use -- Specification and test methods

GB/T 14666, Terms for analytical chemistry

GB/T 43306, Gas analysis -- Sampling guidelines

GB/T 44262, Technical requirements for sampling of hydrogen for proton exchange membrane fuel cell vehicles

JJF 1342, General Requirements for the Competence of Reference Material Producers JJG 823, Ion Chromatographs

3 Terms and definitions

For the purposes of this document, the terms and definitions defined in GB/T 14666 apply.

4 Principle

Inorganic halogenated compounds and formic acid in the hydrogen sample are absorbed by the alkaline solution to generate halide ions and formate ions. They are separated by anion chromatography column and detected by conductivity detector. They are qualitatively determined by retention time and quantitatively determined by calibration curve.

5 Reagents and materials

5.1 Test water It shall comply with the requirements of GB/T 6682 for Grade 1 reagent water.

5.2 Reagents

5.2.1 Sodium thiosulfate pentahydrate (Na2S2O3·5H2O). in accordance with the requirements of GB/T 637 for guaranteed reagents.

5.2.2 Sodium hydroxide (NaOH). in accordance with the requirements of GB/T 629 for guaranteed reagents.

5.2.3 Sodium carbonate (Na2CO3). in accordance with the requirements of GB/T 639 for guaranteed reagents.

5.2.4 Sodium bicarbonate (NaHCO3). in accordance with the requirements of GB/T 640 for guaranteed reagents.

5.3 Solutions

5.3.1 Chloride stock solution. an aqueous solution with a chloride ion concentration of 1000 mg/L. A commercially available certified standard solution may be selected.

5.3.2 Bromide stock solution. aqueous solution with a bromide ion mass concentration of 1000 mg/L. A commercially available certified standard solution may be selected.

5.3.3 Formic acid stock solution. aqueous solution with a formate ion mass concentration of 1000 mg/L. A commercially available certified standard solution may be selected.

5.3.4 Alkaline solution. aqueous solution with NaOH and S2O32- mass concentrations of 200 mg/L and 5 mg/L respectively. Accurately weigh

0.200 g of sodium hydroxide and

0.011 g of sodium thiosulfate pentahydrate and dissolve in appropriate amount of water. Transfer to a 1000 mL volumetric flask. Add water to the set volume and mix. Prepare when needed.

5.3.5 Hydroxide eluent. generated online by an automatic eluent electrolytic generator.

5.3.6 Carbonate eluent. aqueous solution with Na2CO3 and NaHCO3 concentrations of

0.2 mmol/L respectively. Accurately weigh

0.530 g of sodium carbonate and

0.017 g of sodium bicarbonate. Dissolve in an appropriate amount of water. Transfer to a

1000 mL volumetric flask. Add water to the set volume and mix well.

5.4 Gas reference materials

5.4.1 The qualifications of the research and development (production) institutions of gas reference materials shall comply with the requirements of JJF 1342.Users shall give priority to the use of certified gas reference materials.

5.4.2 Hydrogen chloride gas standard substance. hydrogen chloride content is 0.05 μmol/mol. The balance gas is nitrogen or hydrogen. It can be prepared by diluting high-concentration gas reference material with a gas diluter.

5.4.3 Formic acid gas standard substance. formic acid content is 0.1 μmol/mol. The balance gas is nitrogen or hydrogen. It can be prepared by diluting high-concentration gas reference material with a gas diluter.

6.1 Instruments

6.1.1 Ion chromatograph. equipped with conductivity detector and anion suppressor. The main performance indicators of the conductivity detector shall meet the requirements of JJG 823.It is equipped with anion chromatographic column. The matrix is divinylbenzene/ethylvinylbenzene polymer or styrene/divinylbenzene polymer or polyvinyl alcohol. It is with quaternary ammonium functional group, or other equivalent anion chromatographic columns. The separation degree of the target ion is not less than 1.5.

6.1.2 Gas diluter. dilution ratio is 20 to 400 times. All pipeline inner walls and connecting parts must be inertized. Gas diluter shall be calibrated regularly by a qualified metrology agency.

6.1.3 Balance. the graduation value is 0.0001 g. The balance shall be calibrated regularly by a qualified metrology agency.

6.2 Equipment

6.2.1 Water purifier. water treatment equipment capable of producing water that meets the requirements of test water (5.1).

6.2.2 Connecting pipe. polytetrafluoroethylene hose or silicone rubber tube lined with polytetrafluoroethylene film.

6.2.3 Impact absorption bottle. capacity is 25 mL. Accurate to

0.1 mL. Material can be glass. Polytetrafluoroethylene material must be tested. The impact absorption bottle filled with 10 mL of water is left to stand for 4 h. The measured values of the target ions shall be less than

0.1 mg/L.