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GB/T 44243-2024

Hydrogen for proton exchange membrane fuel cell vehicles - Determination of sulfur compounds, formaldehyde and organic halides - Gas chromatography

质子交换膜燃料电池汽车用氢气 含硫化合物、甲醛和有机卤化物的测定 气相色谱法

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1 Scope

GB/T 44243-2024 specifies the determination of sulfur compounds, formaldehyde and organic halides in the hydrogen used by fuel cell vehicles, by gas chromatography. Sulfur is the impurity that matters most: hydrogen sulfide, carbonyl sulfide and the mercaptans poison the platinum catalyst permanently at concentrations of a few parts per billion, and since most fuel cell hydrogen comes from reforming natural gas that is odorised with sulfur compounds, the risk is real rather than theoretical. Formaldehyde and organic halides are limited for related reasons. The standard covers the principle, the interferences and the adsorption losses that dominate trace sulfur analysis, the reagents and calibration gases, the apparatus and the chromatographic conditions with sulfur chemiluminescence or the equivalent detection, the sample introduction and any preconcentration, the calibration, the analytical procedure, the calculation of results, the precision and the limit of quantification for each analyte. For a hydrogen supplier, a station operator or a laboratory in China, this is the prescribed method.

This document describes the principle, reagents and materials, instruments and equipment, system performance verification, test procedures, test data processing, precision and accuracy, quality assurance and control, and test report for the determination of sulfur compounds, formaldehyde and organic halides in hydrogen for proton exchange membrane fuel cell vehicles using the pre-concentration-gas chromatography-sulfur chemiluminescence detector/mass spectrometry detector method. This document applies to the determination of sulfur compounds, formaldehyde and organic halides in hydrogen for proton exchange membrane fuel cell vehicles; it serves as a reference for the determination of sulfur compounds, formaldehyde and organic halides in hydrogen for other purposes within the range specified in this document. The determination range of sulfur compounds in this document is

0.10 nmol/mol ~

10.0 nmol/mol, and the detection limit of hydrogen sulfide is

0.01 nmol/mol. The determination range of formaldehyde is

1.0 nmol/mol ~ 400.0 nmol/mol, and the detection limit is

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0.1 nmol/mol. The determination range of organic halides is

1.0 nmol/mol ~ 100.0 nmol/mol, and the detection limit of methyl chloride is

0.5 nmol/mol.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the version corresponding to that date is applicable to this document; for undated references, the latest version (including all amendments) is applicable to this document.

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

3 Terms and definitions

No terms and definitions need to be defined in this document.

4 Principle

Pass the sample into a gas concentrator to capture and desorb the target compound. Introduce the desorbed sample into a gas chromatograph equipped with a non-polar capillary column. The components to be tested separated by the column pass through a splitter assembly, one path enters a sulfur chemiluminescence detector for sulfur compound detection, and the other path enters a mass spectrometer for formaldehyde and organic halide detection. Use the external standard method for quantification.

5.1 Gas

5.1.1 Carrier gas. helium, with a purity of not less than 99.999%. Desulfurization traps as well as dehydration, dehydrocarbonization and deoxygenation filters shall be installed; the concentration of sulfur compounds in the purified gas shall not interfere with sample detection.

5.1.2 Fuel gas. hydrogen, with a purity of not less than 99.999%. Desulfurization traps as well as dehydration and dehydrocarbonization filters shall be installed; the concentration of sulfur compounds in the purified gas shall not interfere with sample detection.

5.1.3 Gas for ozone generator. oxygen, with a purity of not less than 99.999%. Desulfurization traps as well as dehydration and dehydrocarbonization filters shall be installed; the concentration of sulfur compounds in the purified gas shall not interfere with

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sample detection.

5.1.4 Combustion-supporting gas. air, dry air that meets the requirements for use of gas chromatograph. An air generator with a purifier can be used. Desulfurization traps as well as dehydration and dehydrocarbonization filters shall be installed; the concentration of sulfur compounds in the purified gas shall not interfere with sample detection.

5.1.5 Liquid nitrogen. used as the refrigerant in liquid nitrogen refrigeration concentrators.

5.2 Gas reference materials

5.2.1 The qualifications of institutions for gas reference material production (development) shall comply with the requirements of JJF 1342.Users shall give priority to certified gas reference materials.

5.2.2 The mixed reference materials of sulfur compounds should contain the following components. hydrogen sulfide, carbonyl sulfide, ethyl mercaptan, dimethyl sulfide, carbon disulfide, thiophene, dimethyl disulfide; the balance gas shall be nitrogen, helium or hydrogen; the preparation scheme can also be designed according to needs. For newly purchased gas reference materials, their standard values should be confirmed according to 7.2.2.

5.2.3 The mixed reference materials of organic halides should contain the following components. methane chloride, dichloromethane, trichloromethane, dichloroethylene, tetrachloroethylene, chlorobenzene, trichlorofluoromethane, methyl bromide; the balance gas shall be nitrogen, helium or hydrogen; the preparation scheme can also be designed according to the needs.

5.2.4 The balance gas for formaldehyde reference material should be nitrogen or helium.

5.2.5 Based on the preparation quality of various target compounds by the gas reference material production (research and development) organization, it is advisable to purchase gas reference materials with target values as close as possible to the technical indicator limits of sulfur compounds, formaldehyde and organic halides required by GB/T 37244 or other hydrogen uses. For example, the preparation target value of each component can be set at 1.00x10-6 mol/mol.

6 Instruments and equipment

6.1 System The system consists of a gas concentrator, and a gas chromatograph equipped with a sulfur chemiluminescence detector and a mass spectrometer. The recommended system configuration diagram is shown in Figure

1.The system can also be composed of a gas chromatograph equipped with a sulfur chemiluminescence detector AND a gas chromatography-mass spectrometer. Use a capillary column coated with 100% dimethylpolysiloxane on the inner wall or other

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