

BS ISO 14687-2:2012



BSI Standards Publication

# Hydrogen fuel — Product specification —

Part 2: Proton exchange membrane (PEM)  
fuel cell applications for road vehicles

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### National foreword

This British Standard is the UK implementation of ISO 14687-2:2012. It supersedes DD ISO/TS 14687-2:2008 which is withdrawn.

The UK participation in its preparation was entrusted by Technical Committee PVE/3, Gas containers, to Subcommittee PVE/3/8, Gas containers - Hydrogen technologies.

A list of organizations represented on this committee can be obtained on request to its secretary.

This publication does not purport to include all the necessary provisions of a contract. Users are responsible for its correct application.

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**Hydrogen fuel — Product  
specification —**

Part 2:  
**Proton exchange membrane (PEM)  
fuel cell applications for road vehicles**

*Carburant hydrogène — Spécification de produit —*

*Partie 2: Applications des piles à combustible à membrane à échange  
de protons (MEP) pour les véhicules routiers*



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## Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 14687-2 was prepared by Technical Committee ISO/TC 197, *Hydrogen technologies*.

This first edition of ISO 14687-2 cancels and replaces the first edition of ISO/TS 14687-2:2008.

ISO 14687 consists of the following parts, under the general title *Hydrogen fuel — Product specification*:

- *Part 1: All applications except proton exchange membrane (PEM) fuel cell for road vehicles*
- *Part 2: Proton exchange membrane (PEM) fuel cell applications for road vehicles*
- *Part 3: Proton exchange membrane (PEM) fuel cell applications for stationary appliances*

## Introduction

This part of ISO 14687 specifies two grades of hydrogen fuel, “Type I, grade D” and — Type II, grade D. These grades are intended to apply to the interim stage of proton exchange membrane (PEM) fuel cells for road vehicles (FCV) on a limited production scale.

It is also noted that this part of ISO 14687 has been prepared based on the research and development focusing on the following items:

- PEM catalyst and fuel cell components tolerance to hydrogen fuel contaminants;
- effects/mechanisms of contaminants on fuel cell systems and components;
- contaminant measurement techniques for laboratory, production, and in-field operations;
- onboard hydrogen storage technology;
- vehicle demonstration results.

Since the FCV and related technology are developing rapidly, this part of ISO 14687 needs to be revised according to technological progress as necessary. Technical Committee ISO/TC 197, *Hydrogen Technologies*, will monitor this technology trend.

# Hydrogen fuel — Product specification —

## Part 2:

# Proton exchange membrane (PEM) fuel cell applications for road vehicles

## 1 Scope

This part of ISO 14687 specifies the quality characteristics of hydrogen fuel in order to ensure uniformity of the hydrogen product as dispensed for utilization in proton exchange membrane (PEM) fuel cell road vehicle systems.

## 2 Normative references

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

ISO 6145 (all parts), *Gas analysis — Preparation of calibration gas mixtures using dynamic volumetric methods*

ISO 14687-1, *Hydrogen fuel — Product specification — Part 1: All applications except proton exchange membrane (PEM) fuel cell for road vehicles*

## 3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 14687-1 and the following apply.

### 3.1

#### **constituent**

component (or compound) found within a hydrogen fuel mixture

### 3.2

#### **contaminant**

impurity that adversely affects the components within the fuel cell system or the hydrogen storage system

NOTE An adverse effect can be reversible or irreversible.

### 3.3

#### **detection limit**

lowest quantity of a substance that can be distinguished from the absence of that substance with a stated confidence limit

### 3.4

#### **determination limit**

lowest quantity which can be measured at a given acceptable level of uncertainty

### 3.5

#### **fuel cell system**

power system used for the generation of electricity on a fuel cell vehicle, typically containing the following subsystems: fuel cell stack, air processing, fuel processing, thermal management and water management



**3.6**  
**hydrogen fuel index**

fraction or percentage of a fuel mixture that is hydrogen

**3.7**  
**irreversible effect**

effect, which results in a permanent degradation of the fuel cell power system performance that cannot be restored by practical changes of operational conditions and/or gas composition

**3.8**  
**on-site fuel supply**

hydrogen fuel supplying system with a hydrogen production system in the same site

**3.9**  
**off-site fuel supply**

hydrogen fuel supplying system without a hydrogen production system in the same site, receiving hydrogen fuel which is produced out of the site

**3.10**  
**particulate**

solid or aerosol particle that can be entrained somewhere in the delivery, storage, or transfer of the hydrogen fuel

**3.11**  
**reversible effect**

effect, which results in a temporary degradation of the fuel cell power system performance that can be restored by practical changes of operational conditions and/or gas composition

## **4 Requirements**

### **4.1 Classification**

Hydrogen fuel for PEM fuel cell applications for road vehicles shall be classified according to the following types and grade designations:

- a) Type I (grade D): Gaseous hydrogen
- b) Type II (grade D): Liquid hydrogen

### **4.2 Applications**

The following information characterizes representative applications of each type and grade of hydrogen fuel. It is noted that suppliers commonly transport hydrogen of a higher quality than some users may require.

Type I (grade D) Gaseous hydrogen fuel for PEM fuel cell road vehicle systems

Type II (grade D) Liquid hydrogen fuel for PEM fuel cell road vehicle systems

NOTE 1 Type I, grade A, B, C, Type II, grade C and Type III, which are applicable for all applications except PEM fuel cells applications, are defined in ISO 14687-1.

NOTE 2 There is no equivalent grade A and B for Type II fuels.

NOTE 3 Hydrogen fuel specifications applicable to PEM fuel cell applications for stationary appliances are addressed in ISO 14687-3.

### 4.3 Limiting characteristics

The fuel quality requirements at the dispenser nozzle applicable to the aforementioned grades of hydrogen fuel for PEM fuel cells in road vehicles shall meet the requirements of Table 1. The fuel specifications are not process or feed stock specific. Non-listed contaminants have no guarantee of being benign.

NOTE Annex A provides the rationale for the selection of the impurities specified in Table 1.

**Table 1 — Directory of limiting characteristics**

Characteristics (assay)	Type I, Type II
	Grade D
Hydrogen fuel index (minimum mole fraction) <sup>a</sup>	99,97 %
Total non-hydrogen gases	300 µmol/mol
<b>Maximum concentration of individual contaminants</b>	
Water (H <sub>2</sub> O)	5 µmol/mol
Total hydrocarbons <sup>b</sup> (Methane basis)	2 µmol/mol
Oxygen (O <sub>2</sub> )	5 µmol/mol
Helium (He)	300 µmol/mol
Total Nitrogen (N <sub>2</sub> ) and Argon (Ar) <sup>b</sup>	100 µmol/mol
Carbon dioxide (CO <sub>2</sub> )	2 µmol/mol
Carbon monoxide (CO)	0,2 µmol/mol
Total sulfur compounds <sup>c</sup> (H <sub>2</sub> S basis)	0,004 µmol/mol
Formaldehyde (HCHO)	0,01 µmol/mol
Formic acid (HCOOH)	0,2 µmol/mol
Ammonia (NH <sub>3</sub> )	0,1 µmol/mol
Total halogenated compounds <sup>d</sup> (Halogenate ion basis)	0,05 µmol/mol
Maximum particulates concentration	1 mg/kg
For the constituents that are additive, such as total hydrocarbons and total sulfur compounds, the sum of the constituents are to be less than or equal to the acceptable limit.	
<sup>a</sup> The hydrogen fuel index is determined by subtracting the “total non-hydrogen gases” in this table, expressed in mole percent, from 100 mole percent.	
<sup>b</sup> Total hydrocarbons include oxygenated organic species. Total hydrocarbons shall be measured on a carbon basis (µmolC/mol). Total hydrocarbons may exceed 2 µmol/mol due only to the presence of methane, in which case the summation of methane, nitrogen and argon shall not exceed 100 µmol/mol.	
<sup>c</sup> As a minimum, total sulphur compounds include H <sub>2</sub> S, COS, CS <sub>2</sub> and mercaptans, which are typically found in natural gas.	
<sup>d</sup> Total halogenated compounds include, for example, hydrogen bromide (HBr), hydrogen chloride (HCl), chlorine (Cl <sub>2</sub> ), and organic halides (R-X).	

## 5 Hydrogen fuel qualification test

### 5.1 General requirements

Quality verification requirements for the qualification tests shall be performed at the dispenser nozzle under applicable standardized sampling and analytical methods where available. Alternatively, the quality verification requirements may be performed at other locations or under other methods acceptable to the supplier and the customer.

## 5.2 Report results

The detection and determination limits for analytical methods and instruments used shall be reported along with the results of each test as well as the employed analytical method, the employed sampling method and the amount of sample gas.

## 6 Sampling

### 6.1 Sample size

The quantity of hydrogen in a single sample container should be sufficient to perform the analyses for the limiting characteristics specified in Table 1. If a single sample does not contain a sufficient quantity of hydrogen to perform all of the analyses required to assess the quality level, additional samples from the same lot shall be taken under similar conditions.

### 6.2 Gaseous hydrogen

Gaseous hydrogen samples shall be representative of the dispensed hydrogen. The sampling location shall be in accordance with 5.1.

A sample from the dispenser nozzle shall be withdrawn through a suitable connection that does not contaminate the sample or compromise safety. Attention shall be paid to ensure that the sampled hydrogen is not contaminated with residual gases inside the sample container by repeated purge cycles. A validated sampling method should be used (see Annex B for guidance).

Clause 9 provides guidance relative to managing hazards associated with withdrawing samples from the high pressure hydrogen system.

### 6.3 Particulates in gaseous hydrogen

Particulates in hydrogen should be sampled from a dispenser nozzle. Samples shall be collected in a manner that does not compromise safety. Appropriate measures should be taken for the sample gas not to be contaminated by particulates coming from the connection device and/or the ambient air. When using a filter, samples should be collected if possible under the same conditions (pressure and flow rate) as employed in the actual refuelling operation. To avoid trapping particles or contaminating the sample, no regulator should be used between the dispenser nozzle and the particulate filter.

### 6.4 Liquid hydrogen

Vaporized liquid samples shall be representative of the liquid hydrogen supply. Samples shall be obtained in a manner that does not compromise safety. For example, one of the following procedures can be used to obtain samples:

- a) vaporizing, in the sampling line, liquid hydrogen from the supply container;
- b) flowing liquid hydrogen from the supply container into or through a suitable container in which a representative sample is collected and then vaporized.

## 7 Analytical methods

### 7.1 General

The analytical methods suitable for measuring characteristics listed in Table 1 are described below. Other analytical methods are acceptable if their performances, including safety of use are equivalent to those of the methods listed below.

## 7.2 Parameters of analysis

The parameters for analytical techniques contained in this clause are

- a) mole fraction, expressed as a percentage (%),
- b) number of micromoles per mole ( $\mu\text{mol}/\text{mol}$ ), and
- c) number of milligrams per kilogram of hydrogen ( $\text{mg}/\text{kg}$ ) (particulate concentration only).

The determination limits for the analytical methods listed should be less than or equal to the limiting characteristics of hydrogen for all constituents listed in Table 1.

If calibration gas standards which contain the applicable gaseous components at applicable concentrations and standardized dilution procedures are used to calibrate the analytical instruments used to determine the limiting characteristics of hydrogen, calibration gas mixtures shall be prepared in accordance with ISO 6145.

The calibration of measuring equipment should be traceable to a primary standard.

Analytical equipment shall be operated in accordance with the manufacturer's instructions and validated.

## 7.3 Water content

The water content can be determined using one of the following instruments:

- a) an electrostatic capacity type moisture meter;
- b) a fourier transform infrared spectrometer (FTIR) with suitable cell path length, scan wavelength and detector;
- c) a gas chromatograph-mass spectrometer (GC-MS) and jet pulse injection;
- d) a vibrating quartz analyser.

Alternatively, water content may be determined with a dew point analyser in which the temperature of a viewed surface is measured at the time moisture first begins to form.

## 7.4 Total hydrocarbon content

The total (volatile) hydrocarbon content (as methane) can be determined using one of the following instruments:

- a) a gas chromatograph with a flame ionization detector (GC/FID);
- b) a flame ionization detector (FID) based total hydrocarbon analyser;
- c) a fourier transform infrared spectrometer (FTIR) with suitable cell path length, scan wavelength and detector;
- d) a gas chromatograph-mass spectrometer (GC-MS) with a concentrating device.

## 7.5 Oxygen content

The oxygen content can be determined using one of the following instruments:

- a) a galvanic cell type oxygen analyser;
- b) a gas chromatograph-mass spectrometer (GC-MS) and jet pulse injection;
- c) a gas chromatograph with thermal conductivity detector (GC/TCD).

## 7.6 Helium content

The helium content in hydrogen can be determined using a gas chromatograph with thermal conductivity detector (GC/TCD) or a gas chromatograph-mass spectrometer (GC-MS).

## 7.7 Argon and nitrogen contents

The argon and nitrogen contents can be determined using one of the following instruments:

- a) a gas chromatograph with thermal conductivity detector (GC/TCD) or a gas chromatograph with a pulsed discharge helium ionization detector (GC/PDHID);
- b) a gas chromatograph-mass spectrometer (GC-MS) and jet pulse injection.

## 7.8 Carbon dioxide content

The carbon dioxide content can be determined using one of the following instruments:

- a) a gas chromatograph-mass spectrometer (GC-MS) and jet pulse injection;
- b) a gas chromatograph equipped with a catalytic methanizer and a flame ionization detector (GC/FID with methanizer);
- c) a gas chromatograph with a pulsed discharge helium ionization detector (GC/PDHID);
- d) a fourier transform infrared spectrometer (FTIR) with suitable cell path length, scan wavelength and detector.

## 7.9 Carbon monoxide content

The carbon monoxide content can be determined using one of the following instruments:

- a) a gas chromatograph equipped with a catalytic methanizer and a flame ionization detector (GC/FID with methanizer);
- b) a gas chromatograph with a pulsed discharge helium ionization detector (GC/PDHID);
- c) a fourier transform infrared spectrometer (FTIR) with suitable cell path length, scan wavelength and detector.

## 7.10 Total sulfur content

The content of inorganic and organic sulfur compounds shall be determined using a gas chromatograph (GC) and a chemiluminescence detector with concentration device.

Alternatively, the total sulfur content may be determined using the following procedure. An oxygen-hydrogen flame, whose sulfur contents have been removed completely by absorption or by other suitable method, may be used to burn the sample at a high temperature. The combustion products are absorbed in hydrogen peroxide/water to oxidize the sulfur to sulfuric acid, after which the content is determined and calculated as sulfur dioxide. The sulfur content analysis can be conducted with an ion chromatograph (IC), capable of separating and detecting the desired component. Appropriate impurity-concentrating techniques may be used to attain the sensitivity.

## 7.11 Formaldehyde content

The formaldehyde content can be determined using one of the following instruments:

- a) a gas chromatograph with a flame ionization detector (GC/FID);
- b) a gas chromatograph with a pulsed discharge helium ionization detector (GC/PDHID);

- c) a fourier transform infrared spectrometer (FTIR) with suitable cell path length, scan wavelength and detector;
- d) a gas chromatograph-mass spectrometer (GC-MS) with concentration device.

Alternatively, the formaldehyde content may be determined using the following procedure. The formaldehyde is absorbed in a 2,4-Dinitrophenylhydrazine (DNPH) cartridge by flowing the sampled hydrogen through the cartridge and then extracted from the cartridge with solvent. The extraction liquid can be analysed with a high-performance liquid chromatography (HPLC) technique, capable of separating and detecting the desired component. Appropriate impurity-concentrating techniques may be used to attain the sensitivity.

### 7.12 Formic acid content

The formic acid content can be determined using one of the following instruments:

- a) a fourier transform infrared spectrometer (FTIR) with suitable cell path length, scan wavelength and detector;
- b) a gas chromatograph-mass spectrometer (GC-MS) with concentration device.

Alternatively, the formic acid content may be determined using the following procedure. The formic acid is absorbed in an appropriate solution in a series of impingers by flowing the sampled hydrogen through the impingers. The absorbing solution can be analysed with an ion chromatograph (IC).

### 7.13 Ammonia content

The ammonia content can be determined using a fourier transform infrared spectrometer (FTIR) with suitable cell path length, scan wavelength and detector. Alternatively, the ammonia content may be determined using the following procedure. The ammonia is absorbed in an appropriate solution and determined with an ion chromatograph (IC).

### 7.14 Total halogenated compounds content

The total halogenated compounds can be determined using the one of the following instruments:

- a) a gas chromatograph equipped with an electron capture detection (GC/ELCD) with concentration device for HBr, HCl, and Cl<sub>2</sub>;
- b) a gas chromatograph-mass spectrometer (GC-MS) with concentration device for organic halides;
- c) an ion chromatograph (IC) with a concentrator.

Alternatively, the halogenated compounds may be determined using the following procedure. The total halogenated compounds are absorbed in an appropriate solution and determined with ion chromatograph (IC).

### 7.15 Particulates concentration

The concentration of particulates can be determined using the following procedure. The particles sampled with a filter as described in 6.3 are weighed. The concentration of particulates is calculated from the mass and the total volume of sample hydrogen flowed through the filter.

## 8 Detection limit and determination limit

Generally, detection limit and determination (quantification) limit are based on the ratio of the signal to the noise (S/N ratio) of analytical instruments and on standard deviation (sigma) of the data. The effects of concentrating techniques should be considered when required. In cases where analysis results are less than the detection limit, they shall be reported as "below detection limit".

To establish detection and determination limits, the detection and determination limits provided by the hardware specification of analytical equipment shall be referred to and confirmed.

In cases where no detection and determination numbers are provided by the hardware specification of analytical equipment, 3 sigma and 10 sigma shall be employed for the detection and determination limits, respectively.

NOTE Annex B provides the detection and determination limits for the analytical methods described in Clause 7.

## **9 Quality assurance**

### **9.1 On-site fuel supply**

In the case of hydrogen production using steam methane reforming (SMR) in combination with a pressure swing adsorption (PSA) purification system, Annex C provides an example of one common practice for quality assurance.

In other hydrogen production processes of on-site supply, the supplier should specify quality assurance requirements for production similar to Annex C.

### **9.2 Off-site fuel supply**

The supplier (primary distributor) should specify analytical requirements for qualification tests and lot acceptance requirements.

## **10 Safety**

The sampling, transportation and testing of hydrogen may be hazardous. For example

- a) hydrogen is flammable and can be an asphyxiant,
- b) exposure to liquid hydrogen can cause severe injury, and
- c) sampling is typically performed from pressurized system so there is a risk of burst if the sampling equipment and containers are not capable of withstanding the pressure.

Guidance for the safe use of hydrogen in its gaseous and liquid forms can be found in ISO/TR 15916. This report describes the hazards associated with the use and presence of hydrogen, discusses the properties of hydrogen relevant to safety, and provides a general discussion of approaches taken to mitigate hydrogen hazards.

The sample container and sampling system shall have a rated service pressure at least equal to the maximum allowable working pressure of the system from which the sample is taken or utilize pressure relief devices (PRDs) or other suitable counter-measures to prevent over-pressure of the sampling system.

## Annex A (informative)

### Rationale for the selection of hydrogen contaminants

#### A.1 Water content

Water (H<sub>2</sub>O) generally does not affect the function of a fuel cell, however; it provides a transport mechanism for water-soluble contaminants such as K<sup>+</sup> and Na<sup>+</sup> when present as an aerosol. Both K<sup>+</sup> and Na<sup>+</sup> are recommended not to exceed 0,05 µmol/mol. In addition, water may pose a concern including ice formation for onboard vehicle fuel and hydrogen dispensing systems under certain conditions. Water should remain gaseous throughout the operating conditions of systems.

#### A.2 Total hydrocarbon content

Different hydrocarbons have different effects on fuel cell performance. Generally aromatic hydrocarbons adsorb more strongly on the catalyst surface than other hydrocarbons inhibiting access to hydrogen. Methane (CH<sub>4</sub>) is considered an inert constituent since its effect on fuel cell performance is to dilute the hydrogen fuel stream.

#### A.3 Oxygen content

Oxygen (O<sub>2</sub>) in low concentrations does not adversely affect the function of the fuel cell system; however, it may be a concern for some onboard vehicle storage systems, for example, by reaction with metal hydride storage materials.

#### A.4 Helium, nitrogen and argon contents

Inert constituents, such as helium (He), nitrogen (N<sub>2</sub>) and argon (Ar) do not adversely affect the function of fuel cell components or a fuel cell system. However, they dilute the hydrogen gas. N<sub>2</sub> and Ar especially can affect system operation and efficiency and can also affect the accuracy of mass metering instruments for hydrogen dispensing.

#### A.5 Carbon dioxide content

Carbon dioxide (CO<sub>2</sub>) does not typically affect the function of fuel cells. However, CO<sub>2</sub> may adversely affect onboard hydrogen storage systems using metal hydride alloys. With CO<sub>2</sub>, at levels very much higher than the specification, a reverse water gas shift reaction can occur under certain conditions in fuel cell systems to create carbon monoxide.

#### A.6 Carbon monoxide content

Carbon monoxide (CO) is a severe catalyst poison that adversely affects fuel cell performance and needs to be kept at very low levels in hydrogen fuel. Although its effect can be reversed through mitigating strategies, such as material selection of membrane electrode assembly (MEA), system design and operation, the life time effects of CO on performance is a strong concern. Lower catalyst loadings are particularly susceptible to catalyst poisoning contaminants.



### **A.7 Total sulfur compounds contents**

Sulfur containing compounds are severe catalyst poisons that at even very low levels can cause irreversible degradation of fuel cell performance. The specific sulfur compounds that are addressed are in particular: hydrogen sulfide ( $\text{H}_2\text{S}$ ), carbonyl sulfide ( $\text{COS}$ ), carbon disulfide ( $\text{CS}_2$ ), methyl mercaptan ( $\text{CH}_3\text{SH}$ ). Lower catalyst loadings are particularly susceptible to catalyst poisoning contaminants.

### **A.8 Formaldehyde and formic acid contents**

Formaldehyde ( $\text{HCHO}$ ) and formic acid ( $\text{HCOOH}$ ) have a similar effect on fuel cell performance as  $\text{CO}$  and are thus considered as reversible contaminants. The effect of  $\text{HCHO}$  and  $\text{HCOOH}$  on fuel cell performance can be more severe than that of  $\text{CO}$  due to slower recovery kinetics and their specifications are lower than that for  $\text{CO}$ . Lower catalyst loadings are particularly susceptible to catalyst poisoning contaminants.

### **A.9 Ammonia content**

Ammonia ( $\text{NH}_3$ ) causes some irreversible fuel cell performance degradation by affecting the ion exchange capacity of the ionomer of the proton exchange membrane and/or electrode.

### **A.10 Total halogenated compounds contents**

Halogenated compounds cause irreversible performance degradation. Potential sources include chlor-alkali production processes, refrigerants used in processing, and cleaning agents.

### **A.11 Particulates**

A maximum particulate concentration is specified to ensure that filters are not clogged and/or particulates do not enter the fuel system and affect operation of valves and fuel cell stacks. A maximum particulate size diameter is not specified but should be addressed in fuelling station and/or component standards. Particulate sizes should be kept as small as possible. It is noted that a specific threshold for particulate size which causes degradation has not been made clear and it is influenced by the particulate in ambient air while sampling and refuelling process.

## Annex B (informative)

### Suggested analytical and sampling methods with detection and determination limits

#### B.1 General

This annex, as informative, is intended to provide a list of suggested analytical and sampling methods with their detection and determination limits (see Clause 7). Detection limits should be at least three times lower than the specifications listed in Table 1, and the uncertainty of measurements should meet adequate threshold levels. The information in this table is likely to change as analysis technologies and equipment continue to develop.

#### B.2 Analytical methods

**Table B.1 — Suggested analytical methods with detection and determination limits**

Impurities	Analytical methods	Detection limit μmol/mol (unless otherwise noted)	Determination limit μmol/mol (unless otherwise noted)	Example of test methods that could be used
Water (H <sub>2</sub> O)	Dewpoint analyser	0,5	1,7	JIS K0225
	GC-MS with jet pulse injection	1	3	ASTM D7649-10 JIS K0123
	GC-MS with direct injection	0,8	2,4	NPL Report AS 64
	Vibrating quartz analyser	0,02	0,07	JIS K0225
	Electrostatic capacity type moisture meter	0,04	0,1	JIS K0225
	FTIR	0,12 1	0,4 3	ASTM D7653-10 JIS K0117
	Cavity ring-down spectroscopy	0,01	0,03	NPL Report AS 64
Total hydrocarbons (C1 basis)	FID	0,1	0,3	ASTM D7675-11
	GC/FID	0,01 - 0,1	0,03 - 1,0	JIS K0114
	FTIR	0,01	0,03	JIS K0117
Oxygen (O <sub>2</sub> )	Galvanic cell O <sub>2</sub> meter	0,01	0,03	JIS K0225
	GC-MS with jet pulse injection	1	3	ASTM D7649-10
	GC/PDHID	0,006	0,018	NPL Report AS 64
	GC/TCD	3	9	NPL Report AS 64
	Electrochemical Sensor	0,1	0,3	ASTM D7607-11
Helium (He)	GC/TCD	3 - 5	10 - 15	ASTM D1945-03 JIS K0114
	GC-MS	10	30	JIS K0123

**Table B.1** (continued)

Impurities	Analytical methods	Detection limit μmol/mol (unless otherwise noted)	Determination limit μmol/mol (unless oth- erwise noted)	Example of test methods that could be used
Nitrogen (N <sub>2</sub> ), Argon (Ar)	GC-MS with jet pulse injection	5 (N <sub>2</sub> ), 1 (Ar) 0,03	15 (N <sub>2</sub> ), 3 (Ar) 0,1	ASTM D7649-10 JIS K0123
	GC/TCD	1 – 3	3 – 10	JIS K0114
	GC/PDHID	0,001	0,01	JIS K0114
Carbon dioxide (CO <sub>2</sub> )	GC-MS with jet pulse injection	0,5 0,01	1,5 0,03	ASTM D7649-10 JIS K0123
	GC/FID with methanizer	0,01	0,03	JIS K0114
	GC/PDHID	0,001	0,01	JIS K0114
	FTIR	0,01 0,02	0,03 0,06	ASTM D7653-10 JIS K0117
Carbon monoxide (CO)	GC/FID with methanizer	0,01	0,03	JIS K0114
	FTIR	0,01 0,1	0,03 0,3	ASTM D7653-10 JIS K0117
	GC/PDHID	0,001	0,01	JIS K0114
Total sulfur com- pounds	IC with concentrator	0,0001 – 0,001	0,0003 – 0,004	JIS K0127
	GC/SCD (Sulfur Chemi- luminescence Detector) with concentrator	0,00002 0,001	0,00006 0,003	ASTM D7652-11 JIS K0114
	GC/SCD without pre- concentration	0,001	0,003	NPL Report AS 64
Formaldehyde (HCHO)	DNPH/HPLC	0,002 – 0,01	0,006 – 0,03	JIS K0124
	GC/PDHID	0,01	0,03	JIS K0114
	FTIR	0,02 0,01	0,06 0,03	ASTM D7653-10 JIS K0117
Formic acid (HCOOH)	IC	0,001 – 1 0,002 – 0,01	0,003 – 3 0,006 – 0,03	ASTM D7550-09 JIS K0127
	FTIR	0,02 0,01	0,06 0,03	ASTM D7653-10 JIS K0117
Ammonia (NH <sub>3</sub> )	IC with concentrator	0,001 – 0,01	0,003 – 0,03	JIS K0127
	FTIR	0,02 0,01	0,06 0,03	ASTM D7653-10 JIS K0117
Total halogenated compounds	IC with concentrator	0,05	0,17	JIS K0101 K0127
Particulate concentra- tion	Gravimetric	0,005 mg/kg	0,015 mg/kg	ASTM D7651-10 JIS Z8813

### B.3 Sampling methods

ASTM D7606-11 and ASTM D7650-10 are examples of sampling methods that can be used for the sampling of hydrogen.

## Annex C (informative)

### One common practice of quality assurance for hydrogen production processes that utilize reforming processes associated with pressure swing adsorption (PSA) purification

#### C.1 Purpose

This annex, as informative, is intended to provide one common practice of quality assurance for hydrogen production processes based on reforming process combined with pressure swing adsorption (PSA) purification. The purpose of this recommended practice is to provide a guideline that fuelling station operators can implement in their routine work basis to demonstrate that hydrogen fuel meets the specifications listed in Table 1. The quality assurance practice recommended here was prepared to reflect the experience and learning from hydrogen production and supply to fuel cell vehicles (FCV) in worldwide demonstration activities. Process simulation and other analyses also provide theoretical and technical support for this recommended practice.

#### C.2 Process description

Hydrogen production from hydrocarbon feedstock (natural gas, naphtha, gasoline, kerosene, methanol, etc.) based on steam methane reforming with pressure swing adsorption (SMR-PSA) is expected to be the prevalent pathway for hydrogen production and supply for FCV from the initial market entry to full market penetration. This pathway is facilitated by the high availability of such feedstock from the natural gas pipeline network and the existing fuel supply infrastructure, economy of production and well established technology of on-site reformers.

The SMR-PSA system is typically composed of four subsystems – desulfurizer, steam reformer, shift converter and PSA – and auxiliary components. The desulfurizer is placed most up-stream in the process gas flow and removes sulfur compounds in the feedstock to several parts per billion levels or less. The desulfurized feedstock is preheated and mixed with steam and introduced into the steam reformer where hydrocarbons catalytically decompose and react with steam to produce syngas whose main components are H<sub>2</sub>, CO, CO<sub>2</sub>, CH<sub>4</sub> and H<sub>2</sub>O. The main reaction called “steam reforming” (most simply, CH<sub>4</sub> + H<sub>2</sub>O = CO + 3H<sub>2</sub>) is largely endothermic, and so the reactor has to be externally heated by burning fuels. The high-temperature syngas is cooled and introduced into the shift converter(s) where most of CO catalytically reacts with steam and is converted into H<sub>2</sub> and CO<sub>2</sub>. The resulting hydrogen-rich reformed gas is cooled to ambient temperature to condense the steam into drain water. Thermodynamic calculations for typical natural gas and naphtha compositions predict no presence of oxygen and hydrocarbons other than methane in the reformed gas. HCHO and HCOOH might be present at the concentration levels of 10 to hundred parts per billion. Sulfur compounds are removed to parts to billion levels in the desulfurizer and captured by the catalysts in the steam reformer and shift converter because of the strong interactions between the sulfur compounds and active metals in the catalysts. No presence of sulfur is predicted in the reformed gas. Although ammonia in the gas phase can be present at levels of hundreds to thousands of parts per million, depending on the nitrogen content in the feedstock, it is dissolved and mostly removed in the drain water.

The dry reformed gas is then passed to the PSA purification system. The PSA system has multiple columns, typically 3 to 6, which contain selected adsorbents to capture impurity gases other than hydrogen. After a certain period of purification operation at elevated pressures, the adsorbents are saturated by the impurities. The process gas flow is then switched to another adsorbent column, and the saturated adsorbent is regenerated by desorption of impurities at lower pressures followed by cleaning through flowing purified hydrogen over the column. This adsorption, desorption and cleaning cycles in

which the operation pressures are “swinged” are repeated to produce high purity hydrogen. The “off-gases” from the regeneration process, are usually recycled as fuel for the reformer. The proportion of product high-purity hydrogen to the incoming dry reformed gas is called hydrogen recovery rate which determines the purification process efficiency.

Gases having molecular polarity such as H<sub>2</sub>O, NH<sub>3</sub>, HCHO and HCOOH are strongly adsorbed and easily removed from the product gas stream. Suitable adsorbents can be selected and used to adsorb other non-polar or polar gases such as CH<sub>4</sub>, CO<sub>2</sub>, CO and N<sub>2</sub>. The approximate order of adsorption capability of these gases is CO<sub>2</sub> > CH<sub>4</sub> > N<sub>2</sub> = > CO, which leads to the highest probability that CO will break out first. Inert gases such as He and Ar have limited adsorption capability and to the most part remain in the product hydrogen.

### C.3 Canary species

A canary species can serve as an indicator of the presence of other chemical constituents because it has the highest probability of presence in a fuel produced by a given process. In the case of SMR-PSA production and purification, CO can serve as a canary species for the presence of other impurities listed in Table 1. Confirmation that CO content is less than its specified limit indicates that other impurities, except water and inert gases, are present at less than their specified limits.

In-line monitoring of water content can be done using commercially available instruments, such as dew point meters.

The maximum content of inert gases in the product hydrogen can be estimated by using the maximum content of inert gases in the feedstock specified by the supplier and the flow increase in the SMR system and the flow decrease in the PSA system. The flow increase in the SMR system and the flow decrease in the PSA system can be calculated from the feedstock composition, steam to carbon ratio, and the hydrogen conversion rate.

### C.4 In-line monitoring of the canary species

In-line monitoring of CO is strongly recommended to show that its content in the hydrogen fuel is less than the specification on real-time basis, which indicates that other contaminants are less than their specifications on real-time basis. For this purpose, commercially available infrared CO analyzers can be used. The analyser should be placed just after the SMR-PSA system to avoid contamination of the equipment downstream.

### C.5 Batch analysis

For back-up of in-line monitoring of CO content, batch sampling of product hydrogen and laboratory analyses of all impurities species as listed in Table 1 are also recommended. The batch sample should be taken at the dispenser nozzle. The frequency of sampling and analysis is determined by the hydrogen supplier. The analytical methods as described in Clauses 6 and 7 of this part of ISO 14687 should be applied.

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