
**Gas analysis — Analytical methods
for hydrogen fuel — Proton
exchange membrane (PEM) fuel cell
applications for road vehicles**

*Analyse des gaz — Méthodes analytiques pour carburant hydrogène
— Applications utilisant des piles à combustible à membrane
échangeuse de protons (MEP) pour véhicules routiers*

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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This document was prepared by Technical Committee ISO/TC 158, *Gas analysis* in collaboration with Technical Committee ISO/TC 197, *Hydrogen technologies*.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

The hydrogen supply infrastructure for fuel cell electric vehicles (FCVs) requires specifications and an operational protocol for maintaining the quality of the hydrogen used to fuel the vehicles. To fulfil these requirements, several documents have been written: ISO 14687 which sets forth the quality specifications of hydrogen, and ISO 19880-8 which specifies the quality assurance and control protocol for ensuring them. There was still a need for developing a standard on analytical methods to measure the level of contaminants found in the gaseous hydrogen fuel. The development and validation of these analytical protocols is necessary in order to assure the hydrogen quality required by ISO 14687 for permeating commercialized FCVs and hydrogen infrastructure in the market. This document sets criteria to validate the analytical methods used for the quality control at hydrogen distribution facilities.

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Gas analysis — Analytical methods for hydrogen fuel — Proton exchange membrane (PEM) fuel cell applications for road vehicles

1 Scope

This document specifies the validation protocol of analytical methods used for ensuring the quality of the gaseous hydrogen (H₂) at hydrogen distribution bases and hydrogen fuelling stations for road vehicles using proton exchange membrane (PEM) fuel cells. It also gives recommendations on the calculation of an uncertainty budget for the amount fraction.

This document is established mainly for analysis done in laboratories after the sampling of hydrogen either at hydrogen distribution bases or at hydrogen refuelling stations. The specific requirements for on-line monitoring are not covered by this document.

This document gives a list of suitable analytical techniques used to measure each impurity in hydrogen, according to the specification of hydrogen grade D defined by ISO 14687:—¹⁾.

Moreover, recommendations for keeping the integrity of the sample are also given in order to ensure the quality of the measurement. It also includes the requirements for reporting the analytical results.

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 edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 14687:—, *Hydrogen fuel quality — Product specification*

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <http://www.electropedia.org/>

4 Symbols

b	absolute bias
$b(\%)$	relative bias in %
k_Q	multiplier used in calculating limit of quantification
m	number of replicate observations used during the validation of analytical method
n	number of replicate observations averaged when reporting results

1) Under preparation. Stage at the time of publication: ISO/DIS 14687:2018.

R	relative recovery (apparent recovery) in %
R'	relative spike recovery in %
s	standard deviation
s_0	estimated standard deviation of single results at or near zero concentration
s'_0	standard deviation used for calculating a limit of detection (LOD) or limit of quantification (LOQ)
s_r	repeatability standard deviation
s_R	reproducibility standard deviation
u	standard uncertainty
x_{CRM}	certified property value of a certified reference material
x_{LOD}	value of the limit of detection
x_{LOQ}	value of the limit of quantification
$x_{threshold}$	value of the specification for each impurity listed in ISO 14687:— grade D
x_{mean}	mean value (arithmetic average)
x_{ref}	reference value
x'_{blank}	mean value of the unspiked sample in a recovery experiment
x'_{mean}	mean value of spiked sample in a recovery experiment
$x_{refproficiency}$	reference value assigned during a proficiency test
x_{spike}	added concentration in a recovery experiment

5 Quality characteristics of the fuel

The quality specifications of hydrogen fuel dispensed to PEM fuel cells for road vehicles are listed in ISO 14687:—. The fuel quality at the dispenser nozzle applicable to the hydrogen fuel for PEM fuel cells in road vehicles shall meet the requirements of grade D of ISO 14687:—.

6 Requirements for analytical method validation and fit for purpose

6.1 General

Method validation of fit for purpose is basically the process of defining the application requirements, and confirming that the method under consideration has capabilities consistent with what the application requires. The method performance characteristics ([Table 1](#)) that are associated with method validation shall be evaluated as described in Eurachem guide [2]. Using the information produced during the validation, the laboratory shall determine if the method is suitable for the analysis of impurities in H₂. Criteria are defined for establishing the fit for purpose of the developed method. If the method doesn't fulfil these criteria, it shall not be used for the analysis of impurities in H₂; another method shall be used.

A laboratory may adopt a validated procedure which has been published as a standard, or buy a complete measuring system to be used for a specific application from a commercial manufacturer. In both of these cases, basic validation work has already been carried out but the laboratory shall confirm its ability to apply the method. This means that some experimental work shall be done to demonstrate

that the method works in the end-user's laboratory. The laboratory shall also determine if the method is suitable for the analysis of impurities in H₂.

6.2 Characteristics for analytical methods

6.2.1 List of main characteristics

The list of the main characteristics commonly evaluated during method validation is given in [Table 1](#).

Table 1 — Overview of characteristics for analytical methods

Performance characteristic	Clause/Subclause
Selectivity	6.2.2
Limit of detection (LOD) and limit of quantification (LOQ)	6.2.3
Working range	6.2.4
Trueness bias, recovery	6.2.5
Precision repeatability, intermediate precision and reproducibility	6.2.6
Measurement uncertainty	6.2.7
Ruggedness (robustness)	6.2.8

6.2.2 Selectivity

6.2.2.1 Definition and estimation

Analytical selectivity relates to “the extent to which the method can be used to determine particular analytes in mixtures or matrices without interferences from other components of similar behaviour” [3].

The selectivity of a method is usually investigated by studying its ability to measure the analyte of interest in samples to which specific interferences have been deliberately introduced (those thought likely to be present in samples).

6.2.2.2 Fit for purpose for H₂ analysis

The selectivity is acceptable if:

- the effect of interferences is examined by the analysis of test samples containing various suspected interferences in the presence of analytes of interest;
- the presence of suspected interferences doesn't inhibit detection or quantification of the analytes;
- the presence of suspected interferences doesn't increase the uncertainty of the measurements.

If detection or quantification is inhibited by interferences, further method development is required or another method shall be used.

In the case of impurities in hydrogen, the interferences with the other impurities specified in ISO 14687:— have to be investigated. If other impurities not specified could have an effect on the measurement, they shall also be studied.

6.2.3 Limit of detection and limit of quantification

6.2.3.1 Definition and calculation

It is necessary to distinguish between the instrument detection limit and the method detection limit (x_{LOD}). The instrument detection limit can be based on the analysis of a sample, often a blank, presented directly to the instrument (i.e. omitting any sample preparation steps), or on the signal to noise ratio in, for example, a chromatogram.

To obtain the method detection limit x_{LOD} , the calculation shall be based on the analysis of samples according to the whole measurement procedure (including sample preparation or preconcentration) and calculated with the same formula as for the test samples.

The determination limit or limit of quantification (x_{LOQ}) is the lowest amount of analyte in a sample that can be quantitatively determined with a stated acceptable precision and accuracy, under stated experimental conditions. Depending on the degree of rigour needed or the level of risk that can be tolerated, the x_{LOQ} is typically 4 to 20 times the standard deviation of a low level sample or blank measurement^[2].

The suitable samples for estimating x_{LOD} and x_{LOQ} should preferably be:

- a) blank samples, i.e. matrices containing no detectable analyte;
- b) test samples with concentrations of analyte close to expected x_{LOD} .

Blank samples work well for methods where a measurable signal is obtained for a blank, such as spectro-photometry and atomic spectroscopy. However, for techniques such as chromatography, which rely on detecting a peak above the noise, samples with concentration levels close to or above the x_{LOD} are required. These can be prepared by spiking a blank sample.

The standard deviation s_0 is normally obtained under repeatable conditions. It is important that this standard deviation is representative of the precision obtained for typical test samples, and that sufficient replicate measurements are made to give a reliable estimate. The number of replicates (m) should be at least 6 to give a representative value; 10 replicates are often recommended in validation protocols. The standard deviation s_0 is obtained by the following [Formula \(1\)](#).

$$s_0 = \sqrt{\sum (x_i - x_{mean})^2 / (m-1)} \tag{1}$$

where

- x_i is the measurement i ;
- x_{mean} is the average value;
- m is the number of replicates used during validation of the analytical method.

In many measurement procedures, the mean of n replicates is reported in the routine use of the method, where each replicate is obtained by following the entire measurement procedure. In this case,

the standard deviation of single results (s_0) should be calculated by dividing with the square root of n , where n is the number of replicates averaged in the routine use following [Formula \(2\)](#):

$$s'_0 = s_0 / \sqrt{n} \quad (2)$$

Both x_{LOD} and x_{LOQ} are normally calculated by multiplying the standard deviation s'_0 by a suitable factor^[4]. The x_{LOD} shall be calculated using [Formula \(3\)](#):

$$x_{LOD} = 3 \times s'_0 \quad (3)$$

x_{LOQ} is calculated using [Formula \(4\)](#):

$$x_{LOQ} = k_Q \times s'_0 \quad (4)$$

This will be sufficient if an estimate of the limit of detection and limit of quantification is required simply to demonstrate that the concentrations of samples will be well above these values. Where laboratory samples are expected to contain low concentrations of the analyte, both limit of detection and limit of quantification should be monitored on a regular basis. This could be the case for the impurities at the lowest quality specifications where the limit of quantification could be close to the specification value (for example sulphur compounds analysis).

6.2.3.2 Fit for purpose for H₂ analysis

The calculation of x_{LOQ} shall be done according to [Formula \(4\)](#) with the following rules:

- $k_Q = 10$ for specification values equal or above 1 $\mu\text{mol/mol}$
- $k_Q = 5$ for specification values less than 1 $\mu\text{mol/mol}$ and above 10 nmol/mol
- $k_Q = 3$ for specification values equal or less than 10 nmol/mol , meaning that the limit of quantification is equal to the limit of detection

The limit of quantification of the analytical method shall be below the specification value in order to be able to quantify the impurity in the sample. The criterion for acceptance shall meet the conditions specified in [Formula \(5\)](#):

$$x_{LOQ} + u_{LOQ} < x_{\text{threshold}} \quad (5)$$

where u_{LOQ} is the uncertainty at the x_{LOQ} value.

The limit of detection and limit of quantification should be determined only if they are close to the threshold value requested in ISO 14687 :— for H₂ grade D.

6.2.4 Working range

6.2.4.1 Definition and calculation

The “working range” is the interval over which the method provides results with an acceptable uncertainty^[2].

The lower end of the working range is generally bounded by the limit of quantification. This lower end can also be defined by the concentration of the lower calibration gas standard used during the validation of the method.

The upper end of the working range is defined by the concentration at which significant anomalies in the analytical sensitivity are observed (end of model linearity or saturation of the signal). This upper end should also be defined by the concentration of the upper calibration standard used during the validation of the method.

Between the lower and the upper end of the instrument working range, the response of the instrument obeys a known relationship, e.g. linear, curvilinear, etc. During validation it shall be necessary to:

- a) calibrate the analyser using calibration gas or liquid mixtures with defined uncertainties. For example, use calibration gas mixtures prepared by gravimetry (see Reference [5]) or by dynamic methods (see ISO 6145 series);
- b) establish this relationship.

6.2.4.2 Fit for purpose for H₂ analysis

In order to assess the method working range, the following shall be available:

- a) samples with known concentrations and sample blanks;
- b) the instrument shall have been calibrated by use of methods described in, e.g. ISO 12963 (single point bracketing), or ISO 6143 (multiple points). An alternative protocol using standard addition method is also described in ISO 19229.

For the analysis of impurities in H₂, the higher value of the working range shall be at least equal to 2 times the threshold value. The lower value of the working range (either limit of quantification or lowest calibration standard) shall meet the conditions specified in [Formula \(5\)](#).

6.2.5 Trueness

6.2.5.1 Definition and calculation

Measurement “trueness” is an expression of how close the mean of a large number of results (produced by the method) is to a reference value. We can make a practical assessment of the trueness by repeated measurements of a reference material. This assessment is normally expressed quantitatively in terms of “bias”, see References [4] and [10].

A practical determination of the trueness is based on the measurement of bias and relies on comparison of the mean of the results (x_{mean}) obtained from the method with a suitable reference value (x_{ref}). One of the three following approaches shall be used:

- a) analysis of certified reference materials

To determine the bias using a Certified Reference Material (CRM), the mean (x_{mean}) and standard deviation of a series of replicate measurements are determined and the results compared with the assigned property value of the CRM (x_{CRM}). Within the scope of this document, the ideal CRM is a certified gas mixture in hydrogen balance gas.

The bias, b , can be expressed in absolute terms according to [Formula \(6\)](#):

$$b = x_{\text{mean}} - x_{\text{CRM}} \tag{6}$$

or relative in percent according to [Formula \(7\)](#):

$$b(\%) = \left[\frac{(x_{\text{mean}} - x_{\text{CRM}})}{x_{\text{CRM}}} \right] \times 100 \tag{7}$$

- b) recovery experiments using spiked samples

In the absence of suitable CRMs, recovery studies using spiked and unspiked samples are used to give an indication of the likely level of bias. The spike addition to the sample shall be done using an accurate protocol. The analytical result of the spiked sample is compared to the expected spike concentration.

In this case, the bias can be expressed as the relative spike recovery according to [Formula \(8\)](#):

$$R'(\%) = \left[\frac{(x'_{\text{mean}} - x'_{\text{blank}})}{x_{\text{spike}}} \right] \times 100 \quad (8)$$

where

x'_{mean} is the mean value of the spiked sample;

x'_{blank} is the mean value of the unspiked sample;

x_{spike} is the added concentration to the sample.

c) participation in an interlaboratory comparison

It is also possible to assess bias by comparing results from the method used by the laboratory (x_{mean}) to the reference value assigned during a proficiency test ($x_{\text{refproficiency}}$). The bias of the laboratory will be measured by [Formula \(9\)](#):

$$b = x_{\text{mean}} - x_{\text{refproficiency}} \quad (9)$$

6.2.5.2 Fit for purpose for H₂ analysis

In order to assess the fit for purpose of the analytical method, the bias of the method shall be determined at concentrations close to the threshold value using one of the above three approaches.

This bias shall be small enough to have a relative combined standard uncertainty below 10 % of the concentration generally. An exception to this may be accepted for amount fractions equal or below 10 nmol/mol. In these cases, higher relative combined standard measurement uncertainties could be accepted but should not be higher than 50 % of the amount fraction.

6.2.6 Precision

6.2.6.1 Definition and calculation

Precision (measurement precision) is a measure of how close results are to one another^{[2][6]}. It is usually expressed by the standard deviation (or relative standard deviation), calculated from results obtained by carrying out replicate measurements on a suitable material under specified conditions.

“Measurement repeatability” and “measurement reproducibility” represent the two extreme measures of precision which can be obtained.

The repeatability, expected to give the smallest variation in results, is a measure of the variability in results when a measurement is performed by a single analyst using the same equipment over a short timescale.

The reproducibility, expected to give the largest variation in results, is a measure of the variability in results between laboratories.

Between these two extremes, “intermediate (measurement) precision” gives an estimate of the variation in results when measurements are made in a single laboratory but under conditions that are more variable than repeatability conditions. The exact conditions used should be stated in each case. The aim is to obtain a precision estimate that reflects all sources of variation that will occur in a single laboratory under routine conditions (different analysts, extended timescale, different pieces of equipment, etc.).

The precision is generally dependent on analyte concentration, and so should be determined at a number of concentrations across the range of interest. For the purpose of this document, the precision could be determined for samples with concentrations at the limits of the measuring interval. If relevant,

the relationship between precision and analyte concentration should be established. In cases where the measured concentration is well above the detection limit, the precision is often found to be proportional to analyte concentration. In such cases it may be more appropriate to express precision as a relative standard deviation since this is approximately constant over the range of interest.

The evaluation of precision requires sufficient replicate measurements to be made on suitable materials. The materials should be representative of test samples in terms of matrix and analyte concentration, homogeneity and stability, but do not need to be Certified Reference Materials. The minimum number of replicates specified varies with different protocols, but a minimum of 6 is acceptable.

Typically between 6 and 15 replicates are recommended.

6.2.6.2 Fit for purpose for H₂ analysis

To assess the fit for purpose of the analytical method, the precision of the method shall be determined at least at amount fraction close to the threshold value. The precision for this amount fraction shall be small enough to have a relative combined standard measurement uncertainty below 10 % of the concentration generally.

An exception could be accepted for an amount fraction equal or below 10 nmol/mol. In these cases, a higher relative combined standard measurement uncertainty could be accepted but should not be higher than 50 % of the amount fraction.

6.2.7 Measurement uncertainty

6.2.7.1 Definition and calculation

The uncertainty of an analytical procedure evaluates its ability to produce accurate results. Thus, the uncertainty estimation is fundamental to assess the validity of a method. This uncertainty is mainly due to trueness (bias) and precision plus the impact of calibration or external parameters like temperature or pressure.

A full discussion of (measurement) uncertainty is beyond the scope of this document but detailed information can be found elsewhere. Uncertainty is associated with a measurement result which expresses the range of values that can reasonably be attributed to the quantity being measured. An uncertainty estimate should take account of all recognised effects operating on the result. The uncertainties associated with each effect are combined according to well-established procedures [11].

Several approaches to obtaining an uncertainty estimate for the results from chemical measurements are described [12][14][15][16]. These take into account:

- the overall, long-term precision of the method (i.e. the intermediate precision or reproducibility);
- bias and its uncertainty, including the statistical uncertainty involved in the bias measurements, and the uncertainty in the reference value, see References [17] to [21];
- equipment calibration: uncertainties associated with the calibration of analytical equipment;
- any significant effects operating in addition to the above. For example, temperature, pressure or time ranges permitted by the method may not be fully exercised in validation studies, and their effect may need to be added. Such effects can be usefully quantified by ruggedness studies or related studies which establish the size of a given effect on the result.

6.2.7.2 Fit for purpose for H₂ analysis

To assess the fitness for purpose of the analytical method, mixtures close to the threshold value should be prepared by spiked addition to pure hydrogen. The uncertainty should be determined for each impurity. The spiked samples should be measured using the developed analytical method. The relative combined standard measurement uncertainty for that concentration should be below 10 % of the concentration generally.

An exception could be accepted for an amount fraction equal or below 10 nmol/mol. In these cases, a higher relative combined standard measurement uncertainty could be accepted but should not be higher than 50 % of the amount fraction.

6.2.8 Ruggedness (Robustness)

6.2.8.1 Definition and calculation

In any method there will be certain stages which, if not carried out sufficiently carefully, will have a significant effect on method performance and may even result in the method not working at all. These stages should be identified, usually as part of method development, and if possible, their influence on method performance evaluated using a “ruggedness test” (robustness test).

A “ruggedness test” involves making deliberate changes to the method, and investigating the subsequent effect on performance. It is then possible to identify the variables in the method which have the most significant effect and ensure that, when using the method, they are closely controlled.

6.2.8.2 Fit for purpose for H₂ analysis

There is no specific requirement for the robustness but the critical parameters should be investigated to ensure that, when using the method, they are closely controlled.

6.3 Validation report

A validation report describing all the tests done for the evaluation of all the characteristics of the analytical methods should be produced. This report should be presented upon request.

A simple template for a validation report could, for example, consist of the following Clauses^{[2][25]}:

- **Title:** This section should identify the method and when and who is performing the work. Brief information about the method scope and a short description of the method should be given, as well as details of the status of the method (e.g. an international standard, a method developed in-house, etc.), the analyte, measurement unit, types of sample and the intended use. Sampling can be part of the measurement procedure and shall, in those cases, be validated. Even if these steps are performed elsewhere, it is useful to include information about them in the validation plan/report.
- **Planning:** This section should outline the purpose, e.g. full validation of a new method, verification of performance of a standardized method, extension to method scope, etc. The extent of the validation work should be indicated, i.e. the performance characteristics which will be investigated and any associated requirements.
- **Performance characteristics:** This section should give a brief explanation of the performance characteristic, repeat any specific requirements, outline the experiments which will be done and how the results are to be evaluated. Results and conclusions from the experiments should be stated. Separate sections are used for each performance characteristic.
- **Summary:** The last section summarizes the validation work and the results. Implications concerning routine use, and internal and external quality control, can be given. Most importantly, a concluding statement as to whether the method is fit for purpose shall be given.

6.4 Quality control of the analytical method

Method validation gives an idea of a method’s capabilities and limitations which may be experienced in routine use while the method is in control. Specific controls shall be applied to the method to verify that it remains in control, i.e. is performing in the way expected^[2].

During the validation stage the method was largely applied to samples of known content. Once the method is in routine use it is used for samples of unknown content. Suitable internal quality control shall be applied by continuing to measure stable test samples, thus allowing the analyst to decide

whether the variety of answers obtained truly reflects the diversity of samples analysed or whether unexpected and unwanted changes are occurring in the method performance. In practice, these known samples should be measured with every batch of samples as part of the quality control process.

7 Analytical techniques

[Table 2](#) lists analytical methods that can be suitable for measurement of impurities in hydrogen as required by ISO 14687:— . Each laboratory shall verify the performance of the method against the fitness for purpose acceptance criteria of this document before introducing them.

[Table 2](#) reflects the existing state of the art analytical techniques but others can be applied if fully validated according to the previous protocol.

Table 2 — Analytical methods suitable for measurement of impurities in hydrogen

Impurity	ISO 14687:— limit for grade D ($\mu\text{mol mol}^{-1}$)	Analytical technique	Considerations	Reference
Water	5	Chilled mirror hygrometer (Dew point meter)	A	JIS K0512 <i>Murugan and Brown</i> [23]
		Quartz crystal microbalance	A	<i>Murugan and Brown</i> [23]
		CRDS	A	<i>NPL Report AS 64</i>
		Capacitance	A	JIS K0512
		Continuous wave CRDS	A	ASTM D7941
		GC-MS		<i>NPL Report AS 64</i>
		GC-MS with jet pulse injection		ASTM D7649
		FTIR	A	ASTM D7653 JIS K0512
Considerations: A: Analysis may require a volume of gas greater than 0,5 l. B: Analytes may be lost during the enrichment or the bubbling steps. C: A non-retaining column shall be used. D: Helium carrier gas cannot be used. E: Only organic compounds are detected.				

Table 2 (continued)

Impurity	ISO 14687:— limit for grade D ($\mu\text{mol mol}^{-1}$)	Analytical technique	Considerations	Reference
Total hydrocarbon content (THC)	2	GC-FID	C	ASTM D7675 JIS B 7956
		Methaniser-GC-FID		NPL Report AS 64
		GC-MS (with pre-concentrator)	A, B	ASTM D7892 Murugan and Brown ^[23]
		FTIR	A	ASTM D7653
Oxygen	5	Electrochemical sensor	A	ASTM D7607
		GC-MS with jet pulse injection		ASTM D7649
		GC-TCD		NPL Report AS 64
		GC-PDHID		NPL Report AS 64
		Continuous wave CRDS	A	ASTM D7941
Helium	300	GC-TCD	D	NPL Report AS 64
Nitrogen	300	GC-TCD		NPL Report AS 64 JIS K 0512
		GC-PDHID		NPL Report AS 64
		GC-MS with jet pulse injection		ASTM D7649
Argon	300	GC-TCD		NPL Report AS 64
		GC-PDHID		NPL Report AS 64
		GC-MS with jet pulse injection		ASTM D7649
Considerations: A: Analysis may require a volume of gas greater than 0,5 l. B: Analytes may be lost during the enrichment or the bubbling steps. C: A non-retaining column shall be used. D: Helium carrier gas cannot be used. E: Only organic compounds are detected.				

Table 2 (continued)

Impurity	ISO 14687:— limit for grade D ($\mu\text{mol mol}^{-1}$)	Analytical technique	Considerations	Reference
Carbon dioxide	2	Methaniser-GC-FID		NPL Report AS 64 JIS K 0114
		GC-PDHID		NPL Report AS 64
		GC-MS with jet pulse injection		ASTM D7649
		FTIR	A	ASTM D7653
		Continuous wave CRDS	A	ASTM D7941
Carbon monoxide	0,2	GC-PDHID		NPL Report AS 64
		Methaniser-GC-FID		NPL Report AS 64 JIS K 0114
		FTIR	A	ASTM D7653
		Continuous wave CRDS	A	ASTM D7941
Total sulphur compounds	0,004	GC-SCD (with pre-concentrator)	A, B	ASTM D7652
		GC-FPD (with pre-concentrator)	A	JIS K 0512
		GC-SCD (without pre-concentrator)	C	Murugan and Brown

Considerations:

- A: Analysis may require a volume of gas greater than 0,5 l.
 B: Analytes may be lost during the enrichment or the bubbling steps.
 C: A non-retaining column shall be used.
 D: Helium carrier gas cannot be used.
 E: Only organic compounds are detected.

Table 2 (continued)

Impurity	ISO 14687:— limit for grade D ($\mu\text{mol mol}^{-1}$)	Analytical technique	Considerations	Reference
Formaldehyde	0,2	GC-MS (with pre-concentrator)	A, B	ASTM D7892
		GC-MS (without pre-concentrator)		<i>NPL Report AS 64</i>
		Continuous wave CRDS	A	ASTM D7941
		HPLC (with DNPH cartridge)	A, B	JIS K0303
		FTIR	A	ASTM D7653
Formic acid	0,2	FTIR	A	ASTM D7653
		IC with impinger sampling device	A, B	JIS K0127
Considerations: A: Analysis may require a volume of gas greater than 0,5 l. B: Analytes may be lost during the enrichment or the bubbling steps. C: A non-retaining column shall be used. D: Helium carrier gas cannot be used. E: Only organic compounds are detected.				

Table 2 (continued)

Impurity	ISO 14687:— limit for grade D ($\mu\text{mol mol}^{-1}$)	Analytical technique	Considerations	Reference
Ammonia	0,1	GC-MS		NPL Report AS 64
		FTIR	A	ASTM D7653
		IC	A, B	
		IC with impinger sampling device	A, B	JIS K0127
		Continuous wave CRDS	A	ASTM D7941
Halogenated compounds	0,05	IC with impinger sampling device	A, B	JIS K0127
		GC-MS (with pre-concentrator)	A, B, E	ASTM D7892
Considerations: A: Analysis may require a volume of gas greater than 0,5 l. B: Analytes may be lost during the enrichment or the bubbling steps. C: A non-retaining column shall be used. D: Helium carrier gas cannot be used. E: Only organic compounds are detected.				

8 Sampling

8.1 Sampling strategy

Established sampling strategies for hydrogen fuel quality control are documented by ISO 19880-1:—²⁾, Annex I. Samples should be collected at the HRS (Hydrogen Refuelling Station) nozzle and be representative of the duration of the refuelling protocol.

The purging requirements for the various sampling devices depend on parameters like internal volume, materials and passivation thereof. Purging of the device with UHP hydrogen in order to avoid cross contamination of samples should be performed. The ratio of the sampling device internal volume to the sample volume to be collected gives an indication of the minimum purging requirement of the sampling device.

In the event of simultaneous sampling of gases and particulates, gases should be sampled upstream of any particulate filter in order to avoid condensation and other losses of impurities onto the filter.

2) Under preparation. Stage at the time of publication: ISO DIS 19880-1:2018.