Sensors in the hydrogen industry
Report number MetH4-A1.3.2

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Title

Protocol for testing hydrogen sensors

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Abstract

This report presents a protocol to test different metrics used to determine the performance of sensors: trueness, precision, accuracy, response time (T90)/recovery time (T10), stability and drift, selectivity or cross-sensitivity, limit of quantification, sensitivity, and linear range/measuring range/nominal range (saturation), resolution, hysteresis, reversibility, environmental effects and operation conditions (temperature, pressure, relative humidity, vibration). The tests proposed here are based on ISO 26142 which defines the performance requirements and test methods of hydrogen detection devices designed to measure and monitor hydrogen concentrations in stationary applications and on the standard hydrogen test protocols for the NREL sensor testing laboratory together with considerations from the Eurachem guide - the fitness for purpose of analytical methods in order to fulfill metrological assessment of the performances.

Key words

sensors, protocol, hydrogen, hydrogen-enriched natural gas, gas quality

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1 - Introduction

Clean hydrogen is currently enjoying unprecedented political and business momentum, with the number of policies and projects around the world expanding rapidly [1]. Hydrogen supply chain is therefore a prerequisite to achieve the political and business momentum.

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The supply chain for hydrogen comprises the processes which are used to produce, distribute, and dispense hydrogen gas. These processes rely on various measurements for their safety, their performance optimization or to comply with regulation and/or end-users' requirement. Such measurements may be done by analytical laboratories after sampling if they are complex or require specific analysis or realized online or onsite if the measurements are monitoring quick evolution (i.e., process parameter evolution as dryer performance) or unexpected events (i.e., leak, failure). The deployment of the online analysis depends on several points: the criticality of the measurement, the condition of the measurement (i.e., position in the system, pressure, flow), the frequency, the accuracy, and the accepted cost. These points will determine if the online analysis is realized by a sensor or by an online analyser.

The report will investigate two situations for sensors in hydrogen supply chain:

1) Safety case related to fugitive/emission of hydrogen gas (i.e., hydrogen leak): hydrogen is a colorless and odorless gas having a very broad flammability range (4 to 74 % in air), it is critical to avoid oxygen containing gas such as air from mixing with hydrogen, mostly in confined spaces. It is very important to ensure the safety of system, staff and the public. The main risk of hydrogen mixing with air in the supply chain is unexpected leak. Here, the measurement should be sensitive and rapid. Sensors have different main functions: they can be used to trigger alarms and activate ventilation or shut down systems to prevent hydrogen reaching flammable levels.
2) Ensuring system performance: in the supply chain, online analysis can be of high importance. For example, water sensors are used to monitor the gas quality during the production of hydrogen through electrolysis to ensure the system performance and the dryness of the gas. Due to the number of electrolysers to be rolled out and the hydrogen cost objective, sensors are considered the most suitable approach. In addition, sensors can contribute to ensure the lifetime of fuel cell electrical vehicles by monitoring some impurities in hydrogen [2] or to measure hydrogen in a mixture, such as blending of hydrogen with natural gas [3].

In these cases, sensors play a crucial role in either detect leak (safety sensors) or control hydrogen supply process (quality sensors).

The performances of sensors must be independently and metrologically assessed to ensure their reliable and accurate operation. Sensors has a large range of performances (i.e., working range, repeatability) or operational conditions (i.e., ambient pressure or high-pressure operation [2]).
Each sensor has its own advantages and disadvantages in terms of performance and operational conditions. Sensors need to be chosen for a specific application depending not only on the working conditions but also on the detection requirements and sensor performance capabilities which must be validated and documented.

To identify the optimum sensor technologies for a given application, and to understand the performance and limitations of the sensor technologies, sensors must be tested according to testing protocols, preferably standardized. Protocols define performance requirements and test methods to assess that the metrics fulfil the requirements. Without guidance or protocols in place, uncertainties arise regarding how well sensors perform, how to operate (e.g., calibrate) them, and how well they need to perform to be fit for a given purpose.

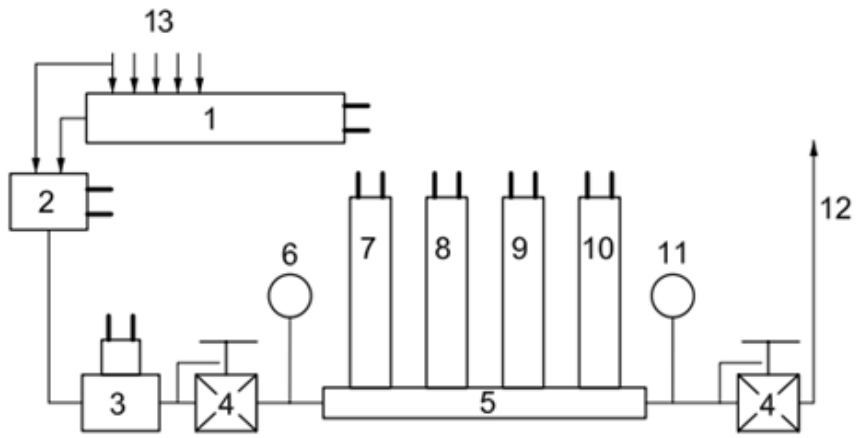
In this report, appropriate methods to measure the metrological metrics are described to support sensor validation in the laboratory. The main metrological criteria for sensors include trueness, precision, accuracy, response time (T90)/recovery time (T10), stability and drift, selectivity or cross-sensitivity, limit of quantification, sensitivity and linear range/measuring range/nominal range (saturation), resolution, hysteresis, reversibility, environmental effects and operation conditions (temperature, pressure, relative humidity, vibration).

2 – Preparation and equipment

The sensor to be tested will be prepared and mounted in a manner representative of the typical application, in accordance with the instruction or operation manual. There are at least two distinct methods to test sensors, the "flow-through test" method (more adapted to sensors for hydrogen quality assessment) and the "chamber test" method (more adapted to fugitive/emission measurement).

In the flow-through testing method, the interface of the sensors to the gas line is hermetically sealed to assure that the sensors are subjected to the proper gas composition without any leaks. With this method, several sensors can be tested simultaneously, in parallel or in series (some sensors consume the component they measure).

Examples of flow-through test set-up are shown on Figure 1 (ISO 26142) and on Figure 2 [4].



Key

1	test-gas selector	8	test sensor No. 1
2	switch	9	test sensor No. 2
3	mass flow controller (MFC)	10	reference sensor No. 2
4	back-pressure regulator (BPR)	11	manifold outlet sensors (temperature, pressure, humidity, etc.)
5	test manifold		
6	manifold inlet sensors (temperature, pressure, humidity, etc.)	12	line to vent
7	reference sensor No. 1	13	test gases

Figure 1 - Set-up for a flow-through test according to ISO26142

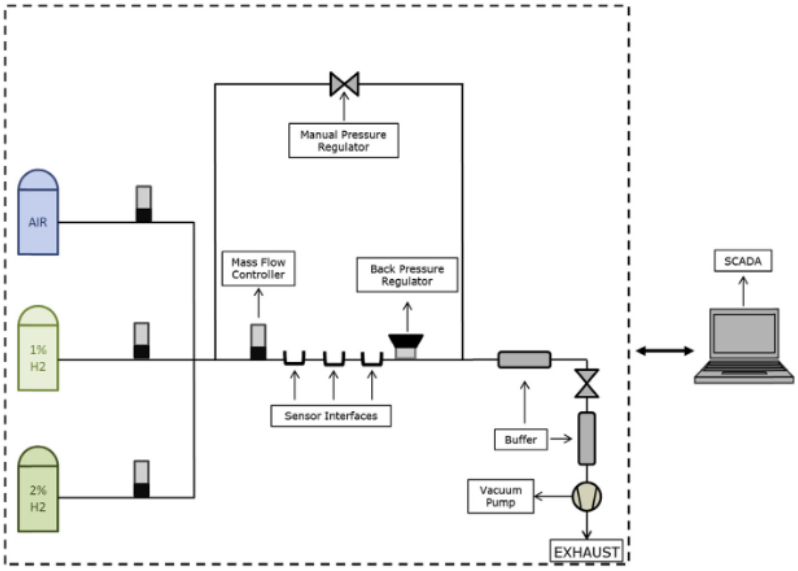


Figure 2 - Set-up for a flow-through test according to [4]

In the "chamber test" method, the sensors are placed in a micro-chamber where flow-through conditions are simulated. The environmental parameters can be easily controlled. The number of sensors that can be tested simultaneously depends upon the size of the chamber.

An example of chamber test set-up is shown on Figure 3 [4].

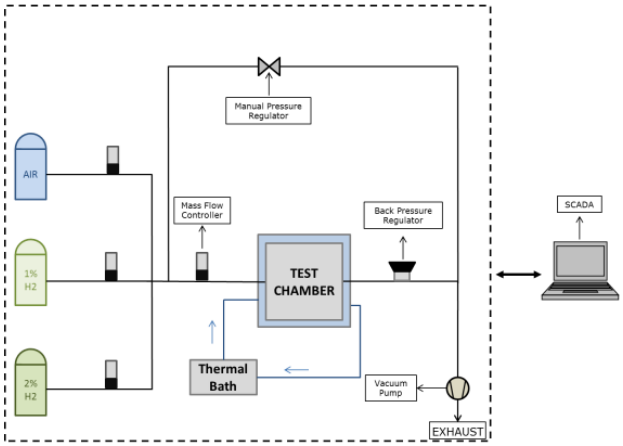


Figure 3 - Set-up for a chamber test according to [4]

Independently of the method chosen, the rig must include test gases, mass flow, temperature, pressure, and humidity controllers/monitors, control system and safe flow path including vent, pressure relief. All measurements with gases should be performed in well-ventilated areas, preferably located in a fume cupboard. If the test gases include reactive/corrosive gases (e.g. NH3, HCl, HF etc.), the testing area should be suitable to handle any release (i.e., suitable extraction, personal protective equipment, handheld guard sensor).

2.1 – Test gases

The two key parameters are the origin and the amount fraction of the test gas and number of test gas required.

All gases used should preferably be certified reference material (provided values with traceability links to units of the International System (SI)). Test gases can be generated from blending certified gas mixtures with synthetic air (safety sensors), hydrogen (quality control sensors) or methane (H2/NG blend sensors). In the protocol developed by NREL [5], the test gas concentrations are mixed within ±10% of the nominal concentration but are known to within ±2%. When blending certified gas mixtures with a pure gas (i.e., hydrogen), the purity of the pure gas needs to be determined to avoid any bias due to contaminant presence (i.e., presence of CO in the pure hydrogen gas while testing CO sensor would lead to an inaccurate response).

The working range of the sensor will provide indication regarding the amount fraction and number of test gas required:

- Safety sensors: ISO 26142 states that the tests shall be conducted using a single test gas per one order of magnitude in the measuring range with a hydrogen volume fraction at the midpoint of that order. If the measuring range is less than two orders of magnitude, then the test shall be conducted with a single test gas having a hydrogen volume fraction at the midpoint of the measuring range. If the measuring range is between two to three orders of magnitude, two test gases shall be used.

- Quality control sensors: the conditions stated above should also apply for quality control sensors. The tests shall be conducted using a single test gas per one order of magnitude in the measuring range with a volume fraction for the targeted compound at the midpoint of that order. If the measuring range is less than two orders of magnitude, then the test shall be conducted with a single test gas. If the measuring range is between two to three orders of magnitude, two test gases shall be used.

Change of gas: the rig to test sensors should be equipped with components to allow to effectively change the test gas. It can be systems a dilutor or a mixing device. The composition of the resulting gas should be verified using a reference analytical instrument.

2.2 – Mass flow controllers

The mass flow controllers (MFC) should be appropriate for the pressure and flow of the sensor (range of flow 10-100% of the full flow controller). The MFC should be calibrated.

2.3 – Temperature controllers

The temperature during any test should be controlled. A typical range would be from 0 to 50ºC. A temperature monitor should be implemented to record the gas temperature supplied to the sensor. The temperature monitor should have an accuracy of 1% of span.

The temperature controller and monitor may be the same instrument as long as the accuracy is suitable.

2.4 – Pressure controllers

The pressure during any test should be controlled. It could be realized using a pressure regulator or back pressure regulator. A typical range would be from 0.5 to 100 bar gauge. A pressure monitor (i.e., pressure transducer) should be implemented to record the gas pressure supplied to the sensor. The pressure monitor should have an accuracy of 1% of span.

Some flowmeters can measure mass flow and pressure simultaneously.

The pressure controller and monitor may be the same instrument as long as the accuracy is suitable.

2.5 – Humidity controllers

The humidity during any test should be controlled. It could be realized using dry gas or through humidification system. A humidity monitor (i.e., humidity transducer) should be implemented to record the gas humidity supplied to the sensor. The humidity monitor should have an accuracy of 1% of span.

The pressure humidity controller and monitor may be the same instrument as long as the accuracy is suitable.

2.6 – Control system

A control system is needed to collect data from the sensor.

The data-collecting interval should low enough to evaluate response time and recovery time.

2-7 – Line to vent

The vent line should be made of suitable material (chemically inert tubing, pressure rating suitable for the operating condition), should reach a safe release point (i.e., allowed to release flammable gas), and should include a non-return valve.

2.8 – Conditioning of the system (reaching zero condition) prior to testing

3 – Performance test methods

Many performance criteria are developed to define how a sensor performs: precision, trueness/accuracy, response time (T90)/recovery time (T10), stability and drift, selectivity or cross-sensitivity, limit of quantification, sensitivity and linear range/measuring range/nominal range (saturation), resolution, hysteresis, reversibility, environmental effects and operation conditions (temperature, pressure, relative humidity, vibration),. The definition of some of these metrics has been listed in a report done as part of EMPIR project MetroHyVe2 [6]. The definition of the metrics is given below:

3.1 – Precision

The precision describes the closeness of results to one another and is a measure of the standard deviation of results obtained by carrying replicate measurements. The precision can be expressed as **repeatability**.

In ISO26142, it is recommended to perform five consecutive tests of 150 s each with 300 s exposure to clean air in between each test. NREL´s protocol recommends performing consecutive cycles without clean air in between (0%, 0.2%, 1%, and 2% hydrogen at a constant flow rate of 1 l/min) for 1 hour. This is then performed nine times over a three-day period (short-term stability).

The EURACHEM guide; the fitness for purpose of analytical methods [7] recommends 6 to 15 replicates during a short timescale for the determination of the repeatability standard deviation.

What to do	Evaluation of results	Pressure conditions	Comments
6-15 replicates for at least 10 min during a short timescale using a single test gas having a volume fraction at the midpoint of the measuring range using a flow at the midpoint of the flow interval. Calculate the standard deviation	Calculate the standard deviation of the replicates	0.8 to 1.2 bar, kept constant within ±0.1 bar throughout the duration of the test 15°C and 25°C kept constant within ±2 °C throughout the duration of the test 20 % and 80 % within ±10 % throughout the duration of the test.	For sticky" impurities*, the duration of the test should be extended (to the time needed to obtain a stable signal). A reference analytical instrument can be used to confirm that the sensor is exposed to the amount of analyte present in the test gas

*General comment: this affects several other performance parameters.

3.3 – Trueness/Accuracy

Describes the closeness of agreement between the value (or the mean value of a series of measurements) and an accepted reference value or conventional true value and is a measure of the systematic error (also called bias) of measurement of an instrument. Trueness is often called **accuracy** when manufacturer list the specifications of sensors/analysers**.**

Accuracy is the closeness of agreement between a measured quantity and a true quantity value of a measurand. Measurement accuracy describes how close a single measurement result is to the true quantity value and therefore includes the effect of both precision and trueness.

In ISO 26142, the sensor is exposed in an ascending order to each hydrogen volume fraction of the test gas for 3 min without exposure to clean air (air that is free of flammable gases, interfering or contaminating substances, and dust) between the hydrogen volume fractions. Following the highest hydrogen volume fraction, the sensor shall be exposed to clean air for 10 min. This operation shall be carried out three times consecutively. For all measurements, the variation of the final indication from the hydrogen volume fraction of the test gas is compared to the indication of the sensor.

In the NREL protocol, the “accuracy of Response Test” (Linear Range Test) is performed by exposing the sensor to a gas mixture whose hydrogen concentration is changed stepwise between 0% and 2% hydrogen (maximum) by mixing with synthetic air – purity not specified (concentration established by independent gas analysis).

What to do	Evaluation of results	Pressure conditions	Comments
Expose the sensor 10 times to the test gas having a volume fraction at the midpoint of the measuring range. Other concentrations (for example, close to the limit of quantification) can be tested in the same manner	Calculate bias (b), relative bias, b(%) or the relative recovery R(%) (apparent recovery). b = $\bar{x} - x_{ref}$ b(%) = $\frac{\bar{x} - x_{ref}}{x_{ref}} . 100$ R(%)= $\frac{\bar{x}}{x_{ref}} . 100$	0.8 to 1.2 bar, kept constant within ±0.1 bar throughout the duration of the test 15°C and 25°C kept constant within ±2 °C throughout the duration of the test 20 % and 80 % within ±10 % throughout the duration of the test.	For sticky” impurities, see section 3.2

3.4 - Response time

The response time is defined as the speed of response to an input signal change and is often expressed in seconds. The response time is often also dependent upon test conditions, such as calibration gas flow rate and ambient temperature. Typically, the response time can be measured by changing the gas concentration and monitoring the sensor output as change of concentrations (increase and decrease) are introduced. The response time **T90** is

commonly used by the sensors industry and corresponds to the time taken to reach 90% of the applied target gas concentration or its stable reading. The recovery Time **T10** is defined as the time for a sensor to return to baseline value after the step removal of the measured variable, usually specified as time to fall to 10% of final value after step removal of measured variable.

According to ISO26142, the sensor shall be switched on in clean air and, after an interval corresponding to at least two times the warm-up time, the sensor shall be subjected to the standard test gas and from standard test gas to clean air.

What to do	Evaluation of results	Pressure conditions	Comments
6-15 replicates starting with clean air or hydrogen, expose the sensor to the standard test gas followed by clean air or clean hydrogen, let the sensor reach stability in each step,	Evaluate T90 and T10 as the average of the replicates	0.8 to 1.2 bar, kept constant within ±0.1 bar throughout the duration of the test 15°C and 25°C kept constant within ±2 °C throughout the duration of the test 20 % and 80 % within ±10 % throughout the duration of the test.	

3.5 - Stability and Drift

Drift is a temporal change in the response of an instrument to a constant concentration. Drift implies that the performance of a measuring instrument changes, and re-calibration must be performed. It is generally due to sensor´s aging, but it can also be caused by dust and variations of pressure, temperature, humidity.

What to do	Evaluation of results	Pressure conditions	Comments
Expose the sensor to three levels of concentration: midpoint of the working range, close to the lower limit of quantification, close to the upper limit of the working range after a period of time (ex: a month, three months, six months, a year)	Calculate bias and compare with the bias obtained when started testing the sensor. If the bias increases, the response of the sensor is not stable	0.8 to 1.2 bar, kept constant within ±0.1 bar throughout the duration of the test 15°C and 25°C kept constant within ±2 °C throughout the duration of the test 20 % and 80 % within ±10 % throughout the duration of the test.	

3.6 - Selectivity or cross-sensitivity

Sensors are designed to be selective to a specific compound or to a certain type of compounds. However, in the presence of some non-targeted compounds, a signal may be produced leading to errors in the measurement of the target compound; the signal of target compounds being affected (either higher or lower than predicted); this is called cross-sensitivity. The manufacturer can sometimes provide a list containing common gases and the typical effect they would have at a given concentration on the signal of sensors.

According to NREL´s protocol, the sensor shall be subjected to two exposure cycles of the interferent gas at the test concentration. Following the second interferent exposure and recovery in air, the sensor is exposed continuously to 1% hydrogen while the interferent exposure cycles are repeated two or more times. The concentration of the interferent is not changed. Following the second interferent exposure, the sensor is allowed to stabilize in 1% hydrogen followed by a recovery time in air. Following the recovery, the sensor response to 1% hydrogen in air is measured twice. Some of the proposed interferents may be poisons (i.e., vapors that induce irreversible effects on sensor behavior). The proposed interferents are carbon monoxide (50 µmol/mol), nitrogen dioxide (5 µmol/mol), hydrogen sulfide (20 µmol/mol), methane (1 vol-%), ammonia (50 µmol/mol), and carbon dioxide (5000 µmol/mol).

According to ISO 26142, the sensor shall be exposed to methane (500 µmol/mol), isooctane (500 µmol/mol), and carbon monoxide (500 µmol/mol) and other species enhanced by the sensor´s manufacturer, individually in air.

What to do	Evaluation of the results	Pressure conditions	Comments
List suspected interferences and adequate test concentrations. Analyse test gases containing suspected interferences individually at least 3 times each.	Examine effect of interferences. Is the Interference causing a bias by increasing or decreasing the signal?	0.8 to 1.2 bar, kept constant within ±0.1 bar throughout the duration of the test 15°C and 25°C kept constant within ±2 °C throughout the duration of the test 20 % and 80 % within ±10 % throughout the duration of the test.	

3.7 – Limit of quantification

According to UIPAC [8], the limit of detection is derived from the smallest measure that can be detected with reasonable certainty. The limit of quantification (LOQ) is derived from the lowest signal that can be quantified with reasonable certainty for a given analytical procedure.

3.8 – Sensitivity, nominal range, saturation

Sensitivity refers to the sensor output signal per µmol/mol of the target gas. The **nominal range** is also often a specification for sensor and corresponds to the range where the gas sensor outputs show the best linearity. This can be measured by successively increasing the concentration from the lowest detectable level and recording the outputs.

Saturation is a state in which the signal that needs to be measured is larger than the dynamic range of the sensor. In that case, the output of the sensor becomes the limiting value of the sensor range. This induces error between the true and estimated values.

What to do	Evaluation of the results	Measuring conditions	Comments
Expose the sensor to increasing concentration from the lowest detectable level to at least the upper level of the working range indicated by the sensor´s manufacturer	Record the outputs. Saturation is reached when the bias between the true value and the output from the sensor differ by more than X%	0.8 to 1.2 bar, kept constant within ±0.1 bar throughout the duration of the test 15°C and 25°C kept constant within ±2 °C throughout the duration of the test 20 % and 80 % within ±10 % throughout the duration of the test.	

3.9 – Resolution

This resolution is the smallest detectable incremental change of input parameter that can be detected in the output signal. Resolution can be expressed either as a proportion of the reading (or the full-scale reading) or in absolute terms.

What to do	Evaluation of the results	Measuring conditions	Comments
		0.8 to 1.2 bar, kept constant within ±0.1 bar throughout the duration of the test 15°C and 25°C kept constant within ±2 °C throughout the duration of the test 20 % and 80 % within ±10 % throughout the duration of the test.	

3.10 - Hysteresis

A sensor should be capable of following the changes of the input parameter regardless of which direction the change is made; hysteresis is the measure of this property.

What to do	Evaluation of the results	Measuring conditions	Comments
Expose the sensor to increasing amounts of the measurand (6-10 concentrations evenly	Plot results and study if the signals overlap (no hysteresis) or differ (hysteresis)	0.8 to 1.2 bar, kept constant within ±0.1 bar throughout the duration of the test	

spaced across the linear range). Expose the sensor to decreasing amounts of the measurand		15°C and 25°C kept constant within ±2 °C throughout the duration of the test 20 % and 80 % within ±10 % throughout the duration of the test.	

3.11 - Reversibility

Reversibility is the ability of a sensor to recover, or return to its original background/baseline condition, after exposure to a target gas (an analyte).

What to do	Evaluation of the results	Measuring conditions	Comments
Expose the sensor to increasing amounts of the measurand (6-10 concentrations evenly spaced across the linear range). Expose the sensor to decreasing amounts of the measurand (same as above).	Plot results and study if the signal measured during the descending series differs from the signal measured during the ascending series when the sensor is exposed to no measurand	0.8 to 1.2 bar, kept constant within ±0.1 bar throughout the duration of the test 15°C and 25°C kept constant within ±2 °C throughout the duration of the test 20 % and 80 % within ±10 % throughout the duration of the test.	For the exposure time, see comment in 3.1

3.12 – Environmental effects and operational conditions

The sensor response and/or the interpretation of the sensor response may depend on many environmental parameters, such as temperature, flow rate and pressure. Moreover, sensors only work effectively under specific conditions of temperature, pressure and flow rate.

3.12.1 – Temperature

It is the normal operating temperature or temperatures range. Operating gas sensors in a lower and higher temperature environment than the operational temperatures may result in slower (or faster) response time. It also may damage the sensors permanently. Some gas sensors may have a transient response to sudden temperature changes, and it may result in false alarming for a short time on the instrument using such sensors.
In between this range, the sensor output can be dependent upon the temperature. In this case, the signal is corrected for the average temperature dependence.

3.12.2 – Pressure

It is the normal operating pressure or pressures range for the gas sensors. Some gas sensors may have a transient response to sudden pressure changes, and it may result in false alarming for a short time on the instrument using such sensors. In addition, there are few sensors which are sensitive to pressure change (typically working at ambient pressure). Any changes cause pressure elevation will lead to wrong value in impurity measurement. Therefore, it is crucial to control and monitor the pressure in sensor performance evaluation.

3.12.3 – Flow range

The flow rate should be low enough to avoid damaging the sensor without being so low as to extend the system response time to an unacceptable level. To ensure that the conditions are in the correct ranges, sensors can be completed with a dedicated sample system.

3.12.4 – Vibration

Sensors shall be constructed to withstand the vibrations expected in its use.

3.13 – Compliance to ATEX requirements

Additional parameters such as compliance to ATEX requirements for H2 specific applications need to be taken into consideration. However, this is not a metric of the sensor.

General: Non-ATEX areas installations preferable

If there is no possibility to install the sensors in non-ATEX designated areas then the sensors has to comply to a certain ATEX -related requirements.

4 – Discussions

Depending on the intended utilization for the sensor, some performance characteristics will be more important that others and testing all performance characteristics may not be needed. This should be a discussion between the manufacturer and the end users in order to define how the testing shall be conducted. Example of relevant questions to be discussed:

- Manufacturer to provide results of testing / end users to verify or end users to define testing. The end-users can for example define acceptance tests they plan to perform to verify some of the performances stated by the manufacturer
- Measuring environment such as pressure/temperature/flow

- Lab validation, site verification or instrument development

The results of the discussion will allow to define who bears the responsibility of the testing and what to do if the sensor doesn´t fulfill the requirements. The following table needs to be agreed upon beforehand:

Parameters	Provided by the manufacturer	Required by the end-users	Lab verification	Site verification	Measuring environment
Precision					
Trueness					
Response time					
Stability					
Cross-sensitivity					
Limit of quantification					
Nominal range					
Resolution					
Hysteresis					
Reversibility					

5 – Conclusions

This report presents a protocol to test different metrics used to determine the performance of sensors: trueness, precision, accuracy, response time (T90)/recovery time (T10), stability and drift, selectivity or cross-sensitivity, limit of quantification, sensitivity, and linear range/measuring range/nominal range (saturation), resolution, hysteresis, reversibility, environmental effects and operation conditions (temperature, pressure, relative humidity, vibration). The tests proposed here are based on ISO 26142 which defines the performance requirements and test methods of hydrogen detection devices designed to measure and monitor hydrogen concentrations in stationary applications and on the standard hydrogen test protocols for the NREL sensor testing laboratory together with considerations from the Eurachem guide - the fitness for purpose of analytical methods in order to fulfill metrological assessment of the performances. The two sources were exclusively from hydrogen sensors used in safety applications whereas the protocol proposed here also applies to other types of sensors such as gas quality sensors (both aiming at measuring impurities in hydrogen and hydrogen in natural gas).

The protocol will be used by partners in the project Met4H2 (A1.3.4) and depending on the outcomes, optimized if necessary.

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