MET4H2

Sensors in the hydrogen industry
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Title

Test of the protocol developed in A1.3.2 using the rig developed in A1.3.3 for two different sensors

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Abstract

This report presents the tests done on two selected sensors to evaluate their metrics using the protocol developed in A1.3.2. The goal of these tests is to demonstrate the applicability of the protocol and if deemed necessary, to improve the protocol. The tests done also allow to conclude that these sensors performed well. As the results of these tests, some improvements of the protocol (A1.3.2) are suggested

Key words

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

Acknowledgement

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Contents

1 - Introduction

Hydrogen is one of the clean, secure and affordable future energy. Over the past few years, global spending on hydrogen energy research, development and demonstration by national governments has significantly risen.

The supply chain for hydrogen comprises the processes necessary to produce, distribute, and dispense the hydrogen. The competitiveness of these processes depends directly on their safety and the safety of the facilities where they are used. Chemical sensors respond to a particular analyte in a selective and reversible way. Chemical sensors exist for a wide variety of components including hydrogen. The sensors can be used to trigger alarms and activate ventilation or shut down systems to prevent hydrogen reaching flammable levels. Considering the future widespread use of hydrogen sensors, it is important to independently and metrologically assess their performance to ensure their reliable and accurate measurement. Each sensor has its own advantages and disadvantages in terms of performance and operational conditions; therefore, sensor needs to be chosen for a specific application depending not only on the ambient working conditions but also on the detection requirements and sensor performance capabilities. In each application, a sensor’s ability to perform the measurements must meet the end-user needs which must be identified and documented.

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). As part of activity A1.3.2, Met4H2 consortium developed a protocol defining performance requirements and test methods to assess that the metrics fulfil the requirements. In this report, we used the protocol to assess the performance of two sensors using the rigs developed in A1.3.3 and proposed, when necessary, changes to the protocol.

2 – Chosen sensors for testing

Two sensors from NevadaNano were chosen to test the protocol developed in A1.3.2:

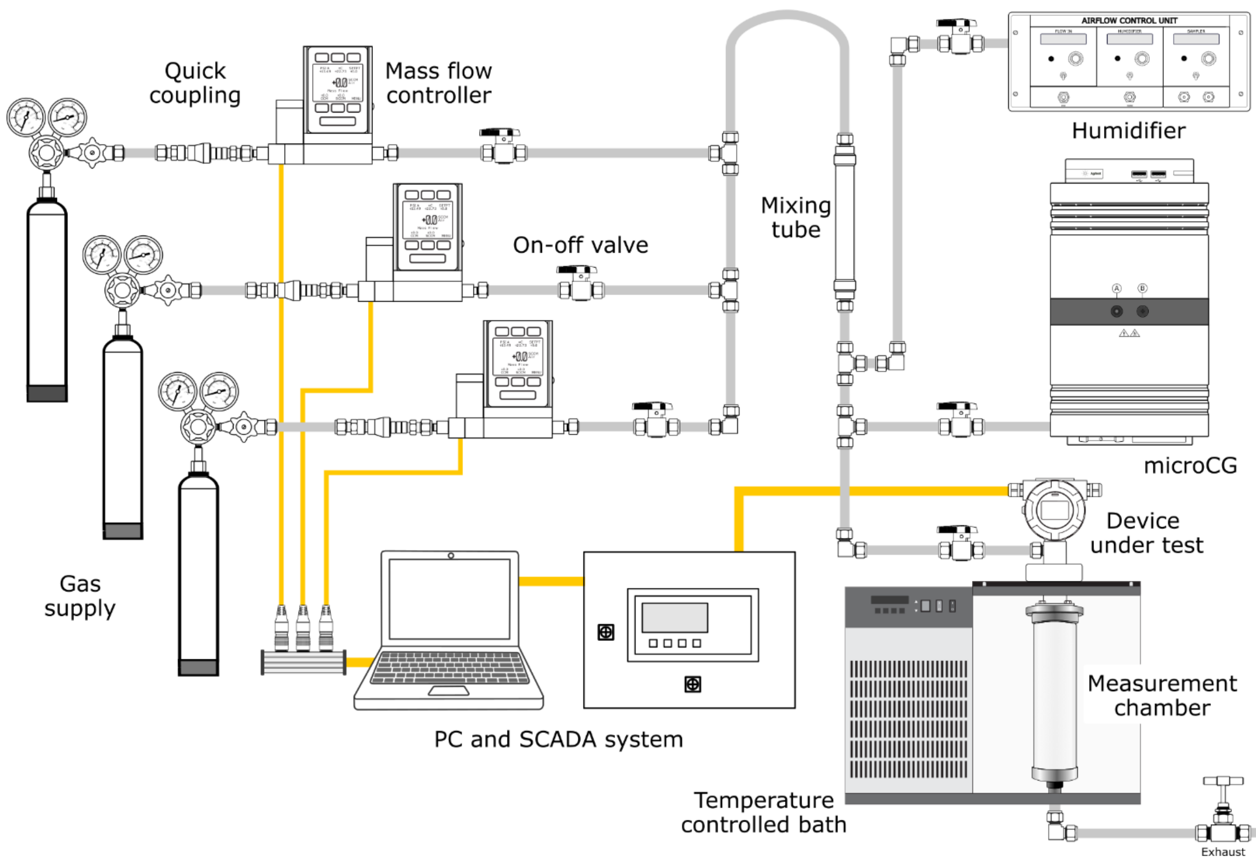
- Reference number: B224051055 will be referred to as Sensor 1 in the report
- Reference number: B324050006 will be referred to as Sensor 2 in the report

The two NevadaNano sensors are part of the company's molecular property spectrometer (MPS) flammable gas sensor family. Both sensors utilize NevadaNano's MPS™ technology which employs micro-electro-mechanical systems to measure the molecular properties of gases. According to the manufacturer, this technology allows the sensor to detect and quantify multiple flammable gases using a single sensor configuration, offering real-time measurement of gases from 0 to 199 % Lower Explosive Limit (LEL). The sensors achieve this with no need for calibration. According to the manufacturer, changes in background gas concentration during the test according to the protocol may impact the sensor´s output but for hydrogen, these impacts are negligible. For other gases, tests should be done without changing the background gas concentration drastically.

3 – Test rig

In a previous project, a sensor rig for sensor testing was developed. The rig is equipped with three gas supply lines, each with flow controllers (0 – 100 ml/min, 0- 500 ml/min, 0-2000 ml/min), that are connected to a mixing tube to ensure good mixing of gases. Pressure is controlled during testing using regulators and gas bottles, with readings displayed on flowmeters that can measure mass flow and pressure simultaneously. The rig is designed for testing sensors at atmospheric pressure and features a cylindrical glass chamber with a volume of 250 ml, which can be removed for flow-through testing. Analytical instruments, such as a micro GC/TCD, or a OFCEAS, are available for confirming the gas composition. A control system is also in place to collect data from the sensor.

Each line is built with chemically inert tubing, pressure rating suitable for the operating conditions and an on-off valve for safety reasons.



4 – Test parameters

The protocol developed in A1.3.2 described two methods to test sensors: using the "flow-through test" method or the "chamber test" method. In this report, we used only the first method, the interface of the sensors to the gas line was sealed to assure that the sensors are subjected to the proper gas composition without any leaks.

3.1 – Test gases

Ideally, a reference gas containing 2 vol-% of hydrogen in synthetic air would have been the best alternative. In this study, three reference gases from Air Liquide were used instead. The first gas with cylinder number N266WX2 contained 40 ± 0.4 vol-% of hydrogen in nitrogen, the second gas cylinder with number N1XC7RD contained 4 ± 0.08 vol-% of hydrogen in nitrogen and the third gas cylinder with number N1Y1640 contained 1 ± 0.02 vol-% of hydrogen in nitrogen. Other test gases were generated from blending this gas with synthetic air.

Change of gas during a series of tests (if necessary) was done effectively using fast connections.

3.2 – Mass flow controllers

The three lines for the supply of gases were each equipped with a calibrated Laminar Flow Element (LFE) flow controller (MC Series, Alicat, Tucson, USA), for which the volumetric flow rate is a function of the pressure difference and the viscosity of the gas to be measured. Flow range: 0 – 2 L/min, 0- 500 ml/min, 0-100 ml/min. The flow controllers were connected with fast connections so they can easily be moved from one line to the other depending on the gases and the required gas composition.

3.3 – Temperature controllers

All tests were performed at ambient temperature without temperature controllers. The sensors have a sensing device to measure the temperature, and a thermometer was placed near the sensor to control the readings from the sensor. According to the sensors, the temperature ranges from 21 to 26 °C which was confirmed by the thermometer. Figure 1 shows the variation of temperature recorded by sensor 2 during the tests done to evaluate response time, precision and trueness. After a very short period of time (100 s), the temperature stabilizes to 25.0 ± 0.2°C.

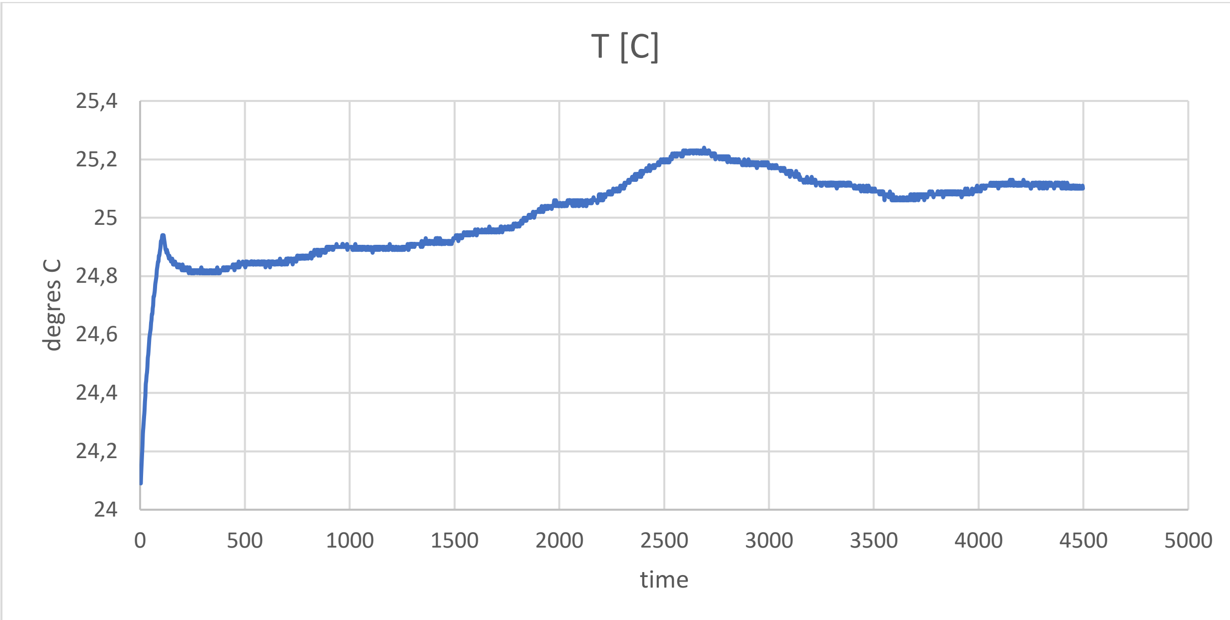


Figure 1. Temperature recorded by the sensor during a series of test.

3.4 – Pressure controllers

The flowmeters measured mass flow and pressure simultaneously (varies from 1002 to 1017 mbar). However, all tests were performed at ambient pressure as the outlet of the sensors was opened to the atmosphere. The sensors have a sensing device to measure the pressure, and the readings show a good agreement with atmospheric pressure recorded on the days of the testing (1012 mbar): Figure 2 shows the variation of pressure recorded by sensor 2 during the tests done to evaluate response time, precision and trueness. The difference between the reading from the sensor and the atmospheric pressure is less than 1 mbar. The pressure ($P_{recorded}$ 1011.25 ± 0.25) was stable almost immediately.

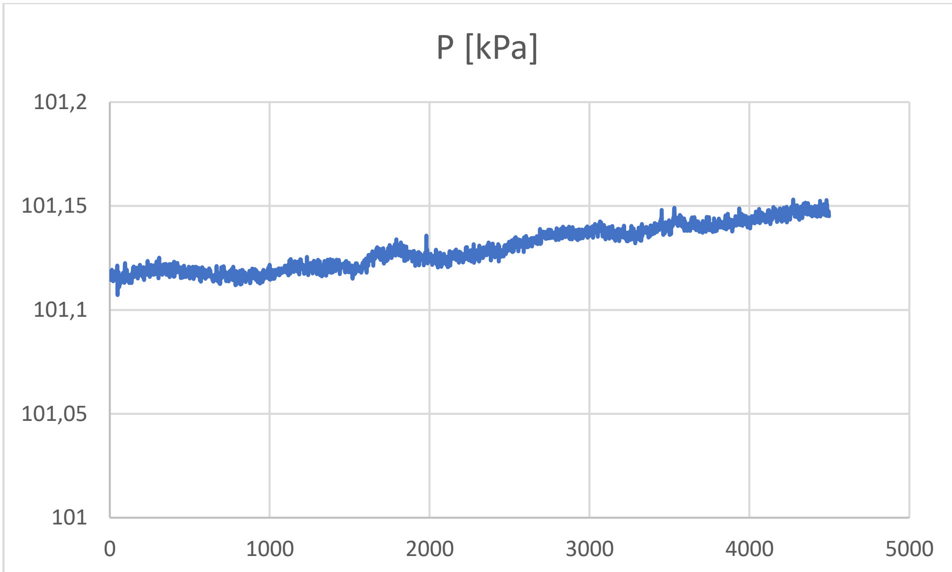


Figure 2. Pressure recorded by the sensor during a series of test.

3.5 – Humidity controllers

Contrary to what is indicated in the protocol, the tests were done at 0% RH as the manufacturer indicated that the sensors did not need humidity to function. This was the case for all tests performed in this study.

3.6 – Control system

The control system used to collect the data was supplied by the sensors supplier. The software used was the "MPS™ sensor interface V1.8.0.3" from NevadaNano. The data-collecting interval was 2 seconds.

4 – Test of the protocol

The protocol developed in A1.3.2 to test different metrics was 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, and reversibility. After each test, some improvement of the protocol is proposed when necessary.

4.1 – Precision

To evaluate the precision, the protocol recommends performing the following tests:

What was done	Evaluation of results	Experimental 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 of the replicates	1012 ± 2 mbar, 23.8 ± 0.6 °C (sensor 1), 25.4 ± 0.8 °C (sensor 2) throughout the duration of the test 0 % RH throughout the duration of the test.	-

For the tests, the test gas containing 40 vol-% hydrogen in nitrogen was used. This gas was diluted with synthetic air (around 500 ml/min air and 27 ml/min of the 40% hydrogen gas). The resulting mixture contained 2.153 vol-% of hydrogen which correspond to a LEL of 53.8), 19.9% oxygen and 78.0 % nitrogen. The response of the sensor being in the order of magnitude of seconds, each replicates were measure during 3 minutes instead of 10.

The first measurement was discarded to ensure that the sensors were fully stabilized and operational. The results presented in table 1 were evaluated by calculating the average readings from minute 1 to minute 2.5 (an average of 45 values). The standard deviation obtained is also indicated in Table 1.

Table 1. Evaluation of the precision for sensor 1 and sensor 2

Tests	Sensor 1	Sensor 2
1	53.432	53.453
2	53.566	53.112
3	53.569	53.064
4	53.525	53.063
5	53.534	53.118
6	53.540	52.057
7	53.498	53.089
8	53.554	53.132
9	51.875	53.105
10	52.665	53.116
11	52.768	53.003

Std deviation (%) with outliner	1.044	0.645
Std deviation without outliner	0.646	0.228

The results are also presented in Figure 3.

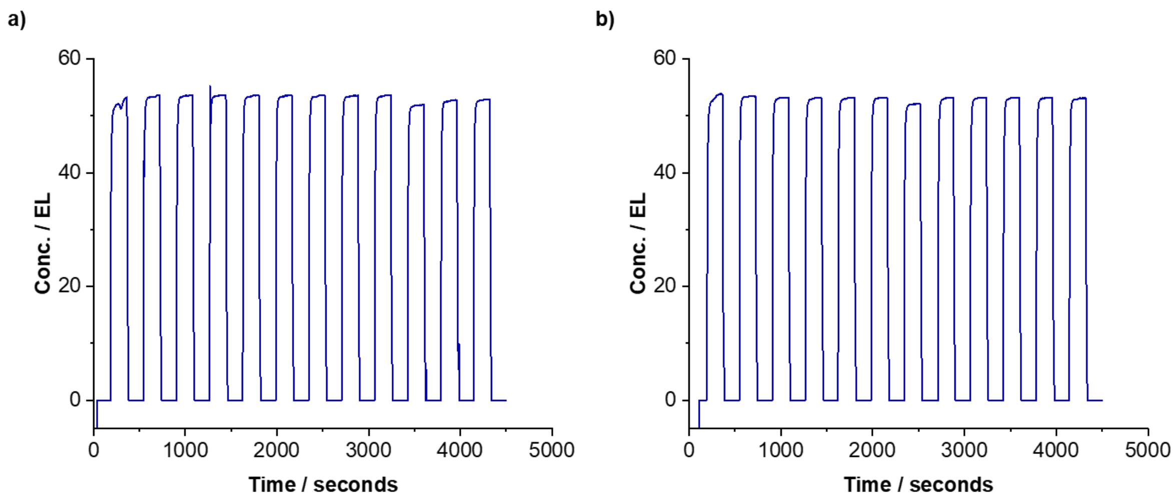


Figure 3. Replicates at 2.153 vol-% (a: sensor 1, b: sensor 2)

<u>Comments on the protocol:</u>

In the preliminary version of the protocol, the testing time was set to 10 minutes, with 6 to 15 replicates, the time needed to perform the testing is relatively long. This time can be changed to “at least 10 times the response time”.

New recommendation:

What to do	Evaluation of results	Experimental conditions	Comments
6-15 replicates for a duration of at least 10 times the response time 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

4.2 – Trueness/Accuracy

To evaluate the trueness/accuracy, the protocol recommends performing the following tests:

What was done	Evaluation of results	Experimental conditions	Comments
Expose the sensor 12 times to the test gas having a volume fraction of 2.153 ± 0.003 vol-% Hydrogen in synthetic air (with a small dilution with nitrogen, ca 3 vol-%) (midpoint of the measuring range is 2 vol%). 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(%)= $\frac{\bar{x}}{x_{ref}} . 100$	1012 ± 2 mbar, 23.8 ± 0.6 °C (sensor 1), 25.4 ± 0.8 °C (sensor 2) throughout the duration of the test 0 % RH throughout the duration of the test.	

As for the precision (same data), the sensors were exposed 12 times to a gas having a volume fraction of 2.153 ± 0.003 vol-% hydrogen in synthetic air (the resulting mixture also contained 19.9% oxygen and 78.0 % nitrogen), which is close to the midpoint of the measuring range, LEL 50. The first measurement was also discarded to ensure that the sensors were fully stabilized and operational. The results presented in table 2 were evaluated by calculating the average readings from minute 1 to minute 2.5 (average of 45 values).

Table 2. Evaluation of the trueness for sensor 1 and sensor 2

Tests	Sensor 1				Sensor 2			
Tests	Readings LEL	Reference value LEL	Bias LEL	Rel. bias %	Readings LEL	Reference value LEL	Bias LEL	Rel. bias %
1	53.43	52.63	0.81	1.53	53.45	52.65	0.80	1.52
2	53.57	52.58	0.99	1.88	53.11	52.73	0.39	0.73
3	53.57	52.63	0.94	1.79	53.06	52.60	0.47	0.89
4	53.52	52.61	0.92	1.75	53.06	52.62	0.44	0.83
5	53.53	52.60	0.94	1.80	53.12	52.63	0.49	0.93
6	53.54	52.62	0.92	1.75	52.06	52.64	-0.58	-1.11
7	53.50	52.60	0.89	1.70	53.09	52.64	0.45	0.86
8	53.55	52.72	0.83	1.58	53.13	52.64	0.50	0.94
9	51.87	52.73	-0.85	-1.61	53.11	52.69	0.42	0.79
10	52.66	52.70	-0.04	-0.07	53.12	52.69	0.43	0.81
11	52.77	52.68	0.09	0.16	53.00	52.72	0.29	0.55
Average			**0.59**	**1.11**			**0.37**	**0.71**

<u>Comments on the protocol:</u>

In the preliminary version of the protocol, no testing time was set for this parameter. We recommend adding an indication of the testing time: "at least 10 times the response time".

What to do	Evaluation of results	Experimental conditions	Comments
Expose the sensor 10 times for a duration of at least 10 times the response time 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

4.3 - Response time

To evaluate the response time, the protocol recommends performing the following tests:

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.	

As for the precision and trueness (calculations based on the same data set), the sensors were exposed 12 times to the test gas having a volume fraction of 2.153 ± 0.003 vol-% hydrogen (equal to 53.8% LEL). The mixture also contained 19.9% oxygen and 78.0 % nitrogen. T90 corresponds to the time to reach 90% of the applied target gas concentration or its stable reading. The recovery time T10 is defined as the time to fall to 10% of final value after step removal of measured variable.

Sensor 1

The results of the assessment of the response time of the sensors are presented in Figures 4 for sensor 1 and Figure 5 for sensor 2.

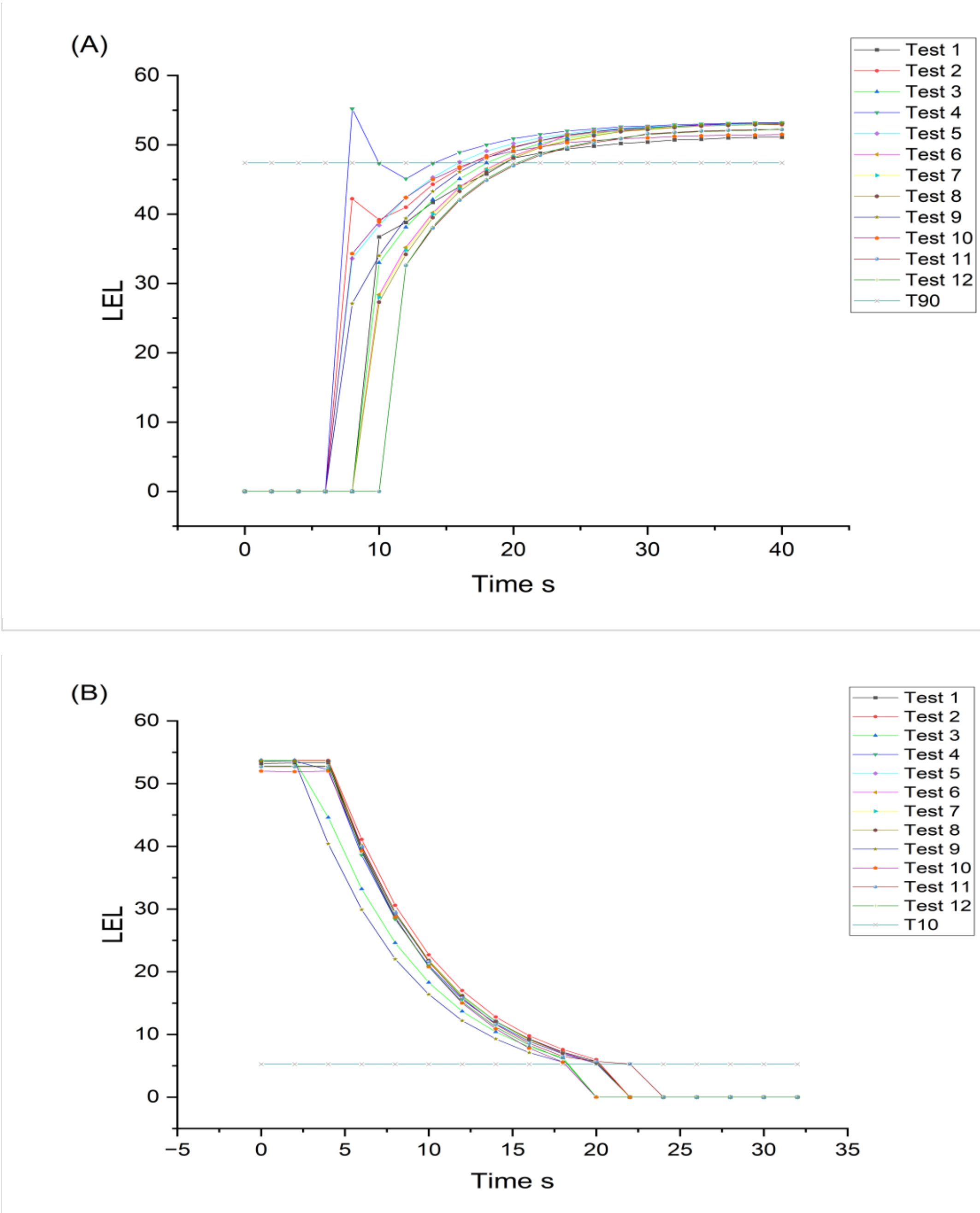
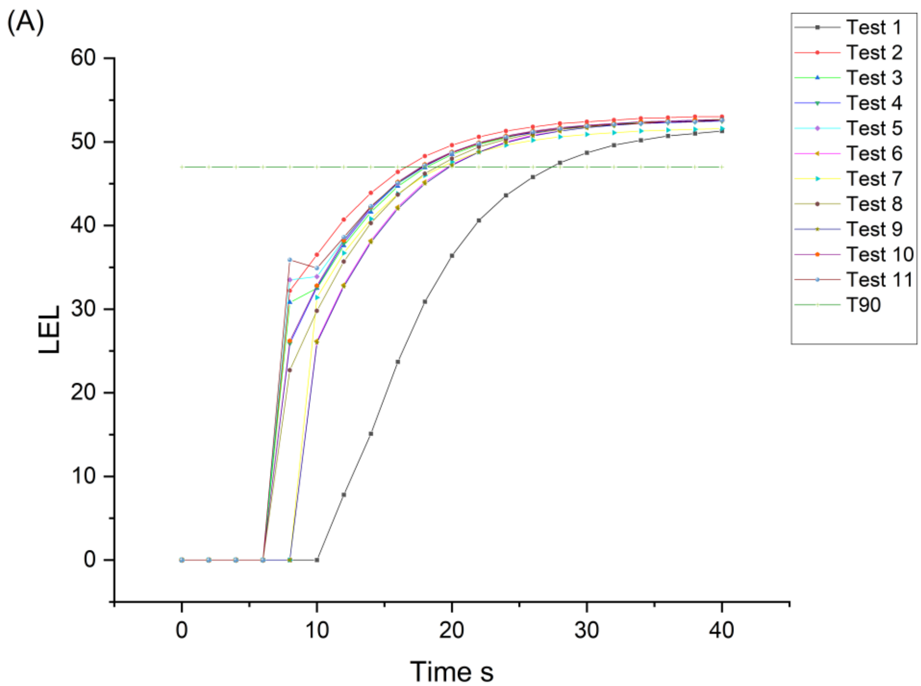


Figure 4. Response time; T90 (A), and recovery time; T10 (B) assessment for sensor 1 at LEL ca 50

Sensor 2


(A)
LEL
Time s
0
10
20
30
40
50
60
Test 1
Test 2
Test 3
Test 4
Test 5
Test 6
Test 7
Test 8
Test 9
Test 10
Test 11
T90

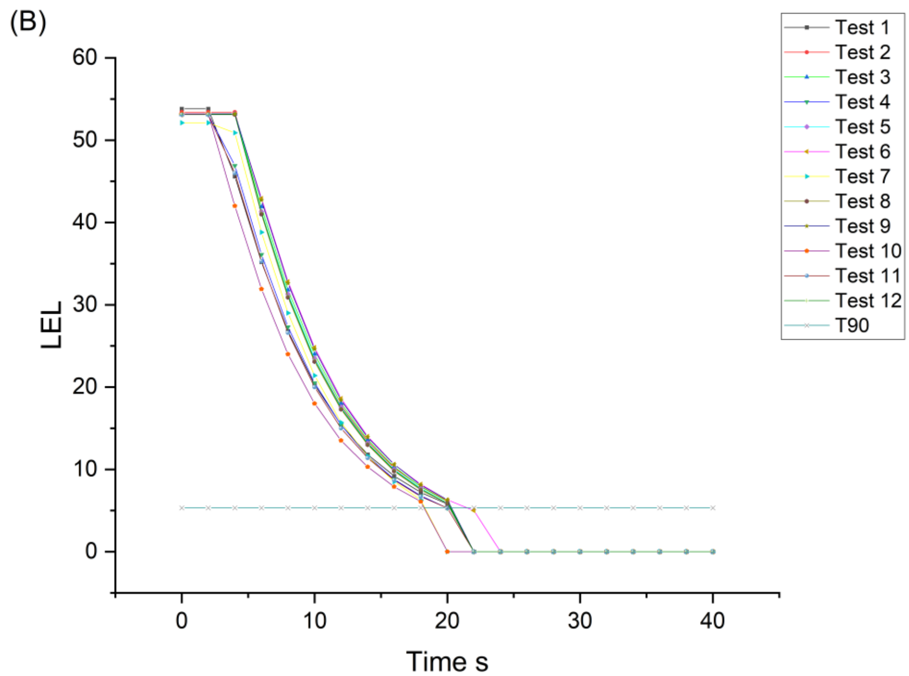


Figure 5. Response time; T90 (A), and recovery time; T10 (B) assessment for sensor 2 at LEL ca 50

The determination of T10 and T90 for sensors with response time of some seconds is limited due to the rig itself: when the change of gas from clean air to the test gas is done using the mixing device, some length of tubing needs to be filled with the test gas before reaching the sensor. The time needed to do so is difficult to evaluate. But tests to measure the time needed to detect a flow when a change of gas occurs (here from no gas to the test gas) showed that this time was negligible. The T90 was evaluated to be 17 ± 3 for sensor 1 and 19 ± 2 seconds for sensor 2 (series 1 is excluded as the sensor was probably not ready), the difference obtained from different series can be explained by the data-collecting interval which is 2 seconds. The T10 was evaluated to be from 19 ± 2 seconds for sensor 1 and 21 ± 2 seconds for sensor 2, the difference obtained from different series can be explained here again by the data-collecting interval of 2 seconds.

<u>Comments on the protocol</u>

No concentration was given in the description of the method to evaluate the response time. However, sensor´s response time may be different at different concentrations. Therefore, we recommend amending the test to include this aspect.

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 at the midpoint of the measuring range followed by clean air or clean hydrogen. Let the sensor reach stability in each step. Other concentrations (relevant for the application) can be tested in the same manner to evaluate if the response time depends on the concentration of the measurand	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.	

To demonstrate the importance of testing response time at different concentrations, the response time T90 was measured at LEL 20 and LEL 72 for both sensors. Figure 6 shows results for sensor 1 and Figure 7 for sensor 2.

Sensor 1

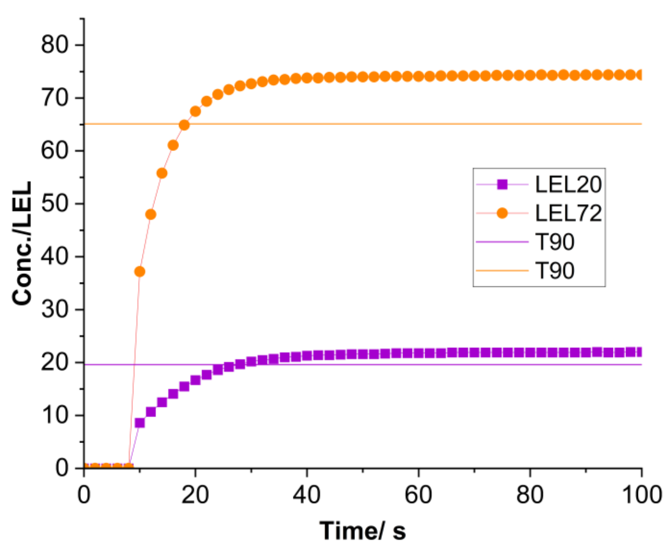


Figure 6 . Response time; T90 assessment for sensor 1 at LEL 20 (purple) and LEL 72 (orange).

The T90 response time was found to be 18 seconds at LEL 72 (similar to the T90 at LEL 50) and slightly higher at LEL 20 confirming the dependency of the response time on concentrations.

Sensor 2

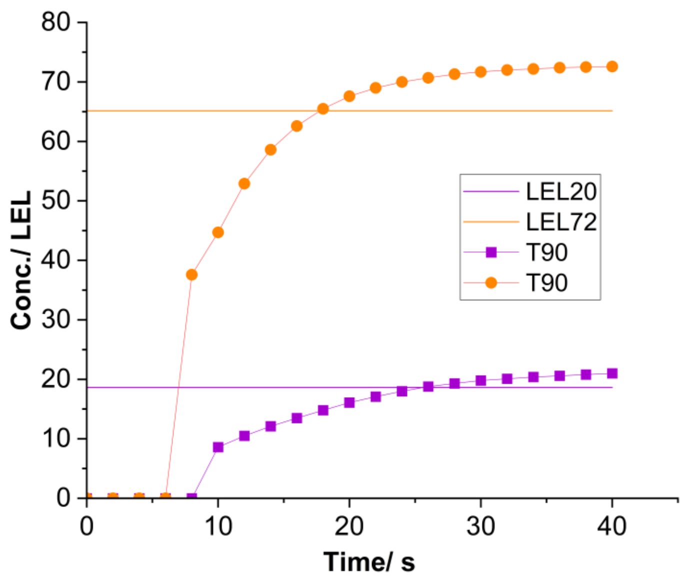


Figure 7. Response time; T90 assessment for sensor 2 at LEL 20 (purple) and LEL 72 (orange)

The T90 and was found to be 25 seconds at the lower concentration (LEL 20) and 18 seconds at the mid-point of the measuring range (LEL 72), therefore dependent upon the concentration.

4.4 - Stability and Drift

To evaluate the stability, the protocol recommends performing the following tests:

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.	

The sensors were exposed to three levels of hydrogen concentrations (0.86 vol-%; 25.1 LEL, 2.08 vol-%; 52.1 LEL, 3.24 vol-%; LEL 80.9) in June 2024, the same measurements were repeated in December 2024. For these tests, a test gas containing 40 vol-% hydrogen in nitrogen was used. This gas was diluted with synthetic air (ca 500 ml/min air and 11; 27.5 respective 43.9 ml/min hydrogen gas). The results are shown in Figure 8 for sensor 1 and Figure 9 for sensor 2. The signals obtained in December 2024 overlap with the signals obtained in June 2024, showing that the response of the sensors was stable for at least six months. The results are also shown in Table 3 where biases were calculated. Biases have increased slightly at LEL 20 and LEL 80 so it would be interesting to redo tests in six months to see if it is a trend.

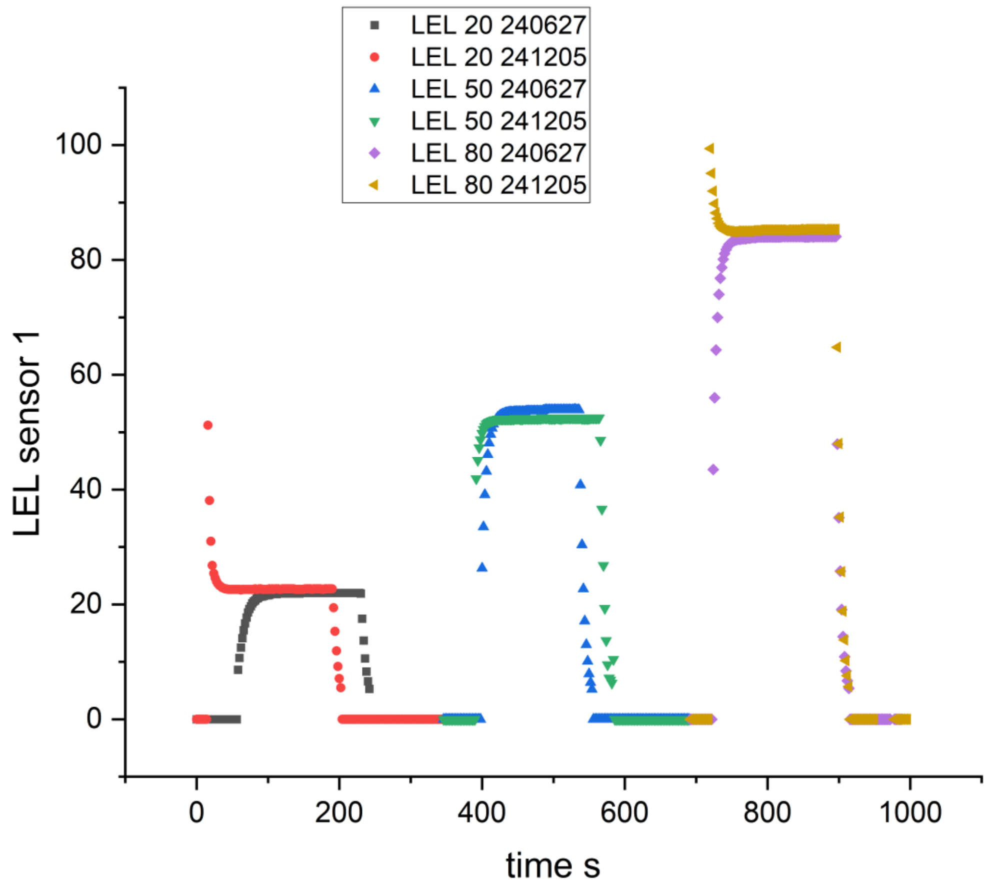


Figure 8. Stability test for sensor 1 with superposition of signals measured for LEL around 20, 50 and 80 in June 2024 (grey, blue and purple symbols) and December 2024 (red, green and yellow symbols).

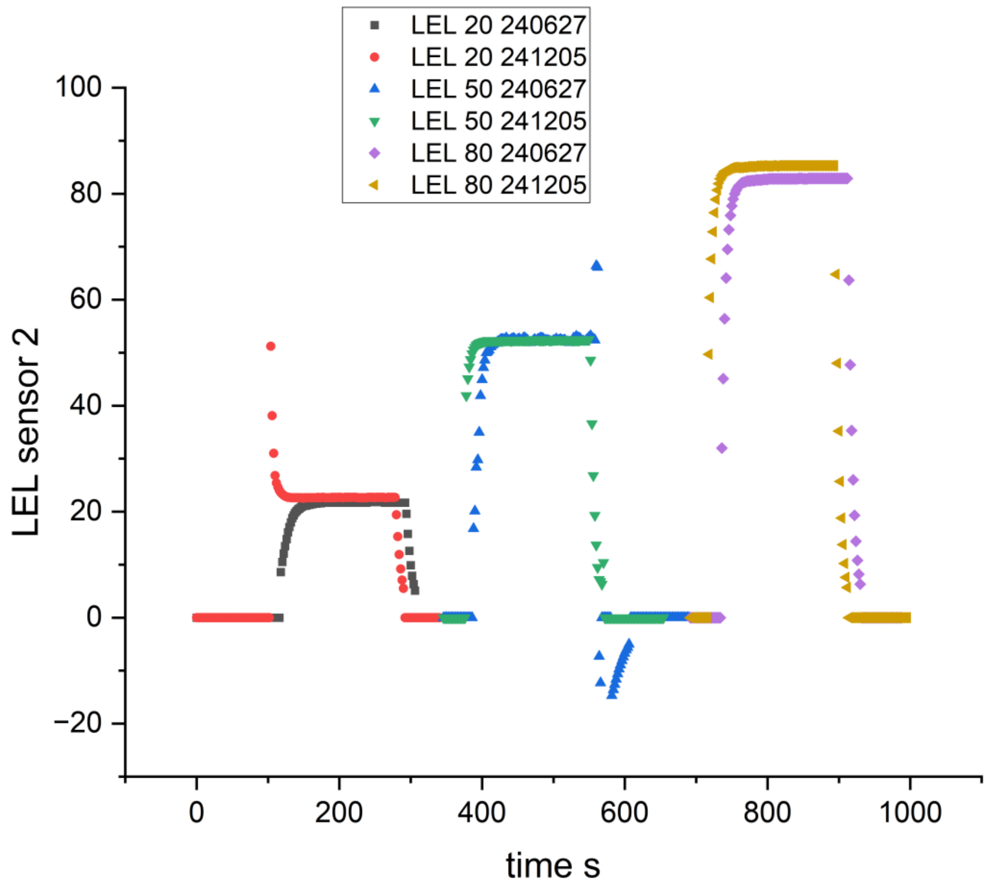


Figure 9. Stability test for sensor 2 with superposition of signals measured for LEL around 20, 50 and 80 in June 2024 (grey, blue and purple symbols) and December 2024 (red, green and yellow symbols).

Table 3. Compared biases at LEL 20, 50 and 80 in June and December 2024

	Sensor 1		Sensor 2	
Bias	Bias rel. / abs. June 2024	Bias rel. / abs. December 2024	Bias rel. / abs. June 2024	Bias rel. / abs. December 2024
LEL 20	0.6 / 2.8%	1.2 / 5.6%	0.2 / 2.8%	1.1 / 5.1%
LEL 50	0.9 / 1.8 %	0.3 / 0.8%	0.7 / 1.3%	0.4 / 0.8%
LEL 80	2.6 / 3.2%	4.3 / 5.3%	1.3 / 1.6%	4.4 / 5.4 %

4.5 - Selectivity or cross-sensitivity

To evaluate the cross-sensitivity, the protocol recommends performing the following tests:

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.	

According to the manufacturer, due to the nature of the measuring principle, CO_2 concentrations above 0.5 to 1 vol-% should start to show in the sensor as a flammable gas concentration. Outside of CO_2, most gases will cause no issues in terms of cross interference.

Sensor 2 was exposed to a gas containing hydrogen by mixing around 440 ml/min of dry air with around 440 ml/min of a dry gas containing 4 vol-% of hydrogen in nitrogen. To this mixture, an increasing flow of pure carbon dioxide was added (from 8.9 ml/min to 43 ml/min resulting in concentrations of 0.97 to 4.77 vol-% of CO_2 in the mixture with increment of 0.3 – 0.6 vol-%). The same tests were then performed by decreasing the concentration of CO_2 from 4.77 to 0 vol-%). As the flow of air and hydrogen containing gas were set to be constant, the LEL slightly decreased when adding the CO_2 flow (from 49.96 to 47.68). Therefore, to simplify the interpretation of the results, the LEL measured was first normalized to correspond to a theoretical value of 50 LEL for all measurements.

However, when performing the tests, the LEL measured without any addition of CO_2 was lower than the expected LEL (43.2 compared to 50) as it can be seen in Figure 10 where the difference LEL theoretical – LEL measured is plotted against the CO_2 concentration.

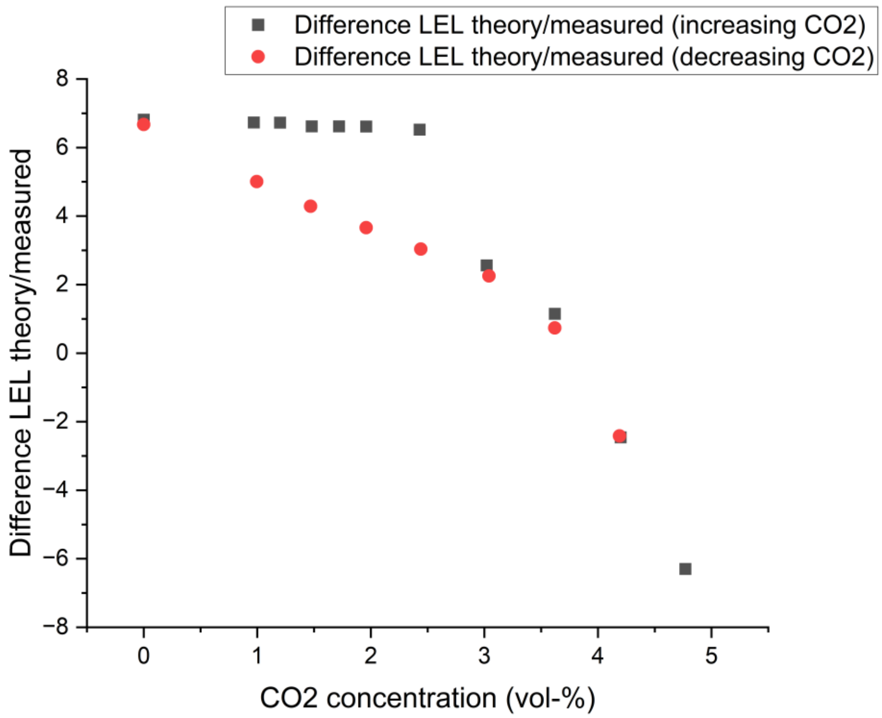


Figure 10. Effect of increasing (in black) and then decreasing (in red) amount of CO_2 of the LEL at the midpoint of the measuring range (sensor 2).

The results indicated that up to 2.5 vol-%, the signal of the sensor remained unaffected by CO_2. Above this concentration, the LEL measured when the sensor was exposed to the same amount of hydrogen, started to increase from 43 up to 56 when the gas containing 4.8 vol-% of CO_2. The LEL measured remained affected even when the sensor was exposed to decreasing amount fractions of CO_2 at all levels of CO_2 down to 0 where the signal came back to 43 as measured initially. The results confirm the statement from the manufacturer that CO_2 behaves as a flammable gas (thus causing a bias by increase of the signal) but from 2.5 vol-% compared to 0.5-1 vol-% stated by the manufacturer.

Comment on the protocol

The recommended tests did not include recommendation to vary the concentrations to determine from which concentrations the interferences are observed. We also propose to add tests where the concentration of the interference is decreased to examine if the sensor recovers after exposition to interference.

What to do	Evaluation of the results	Pressure conditions	Comments
List suspected interferences and adequate test concentrations.	Examine effect of interferences. Is the Interference causing a bias	0.8 to 1.2 bar. kept constant within ±0.1 bar throughout the duration of the test	

Analyse test gases containing suspected interferences at increasing concentrations (repeat the test with decreasing concentrations).	by increasing or decreasing the signal? Can the sensor recover when not exposed anymore to the interference	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.	

4.6 – Limit of quantification

To evaluate the limit of quantification, the protocol recommends performing the following tests:

What to do	Evaluation of the results	Measuring conditions	Comments
Expose the sensor to decreasing concentration starting from (for example) half the volume fraction at the midpoint of the measuring range followed by clean air or clean hydrogen until no signal can be recorded. Increase this concentration slowly until the signal is detected again Option 2 for the starting point: Use information provided by the sensor´s developer regarding the LOQ and start testing at 2 times this value	Record the outputs. LOQ is obtained at the lowest concentration tested that gives a signal with acceptable bias	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.	

The sensor was exposed to decreasing concentration from half the volume fraction at the midpoint of the measuring range (LEL 25) by increments of 5 LEL until no signal was recorded around 5%LEL, then the concentration was increased again with a smaller increment until the signal is detected again (Figure 11).

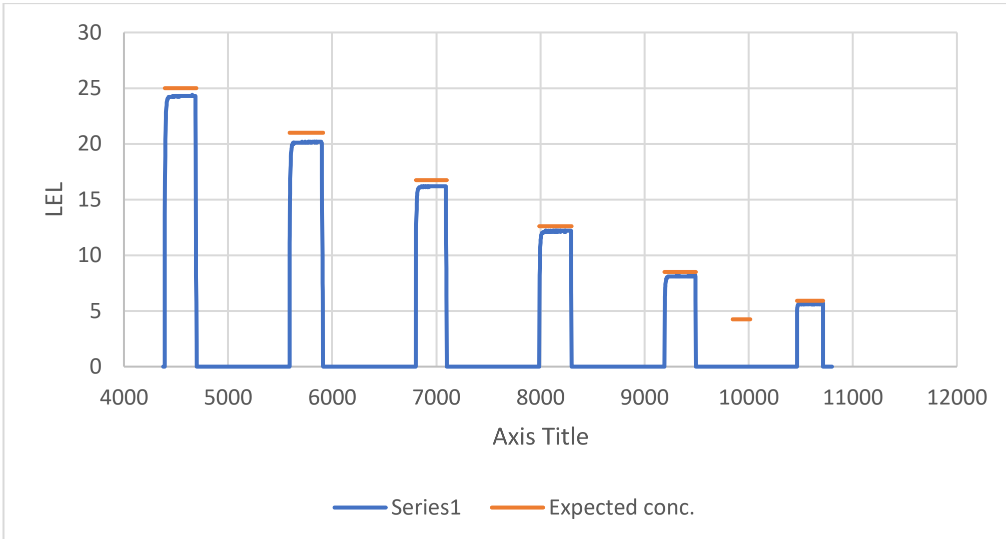


Figure 11. Evaluation of the limit of quantification (sensor 2). The measured concentration is shown in blue and the expected concentration in orange.

The limit of quantification was determined to be at 5.1 % LEL. The signal at LEL 5 appears and disappears (as it can be seen in Figure 12) as the concentration fluctuates slightly but the signal is stable if the sensor is exposed to concentration of hydrogen yielded a LEL of at least 5.1.

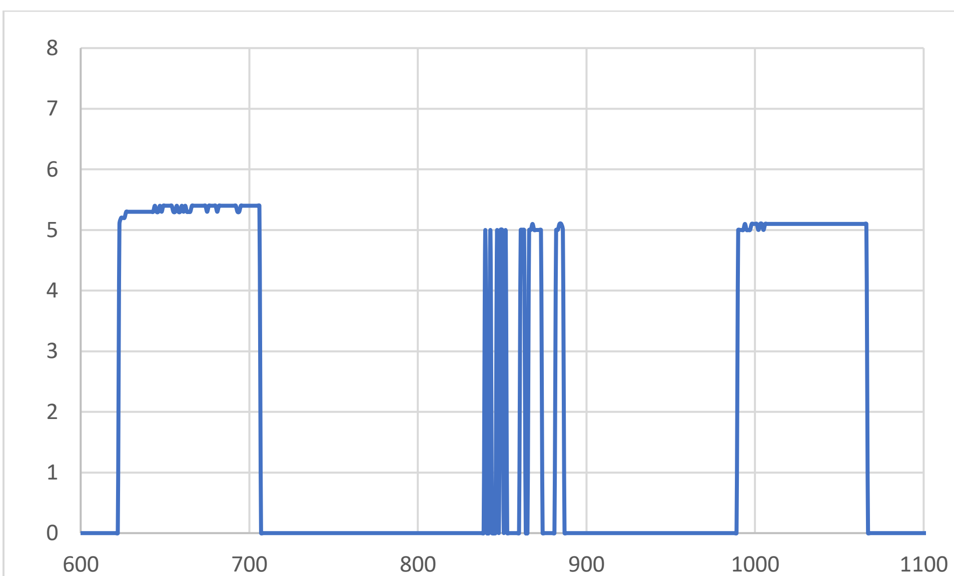


Figure 12. Signal measured for sensor 2 at around 5% LEL.

4.7 – Nominal range, saturation

Saturation is the point beyond which the sensor cannot measure higher values and nominal range is the span of input values the sensor can measure accurately.

To evaluate the saturation, the protocol recommends performing the following tests:

What was done	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.	

The tests were started at LEL 90 followed by increment of 10. The results are presented in Table 4 and in Figure 13 for sensor 2.

Table 4. Results of the tests done to evaluate the saturation of sensor 2

	Series 1	Series 2	Series 3	Series 4
Readings/outputs (LEL)	92.38	101.96	110.00	110.00
Reference values (LEL)	90.89	100.17	108.85	117.71
Bias (LEL)	1.49	1.79	1.15	-7.71
Rel. Bias	1.64	1.79	1.06	-6.55

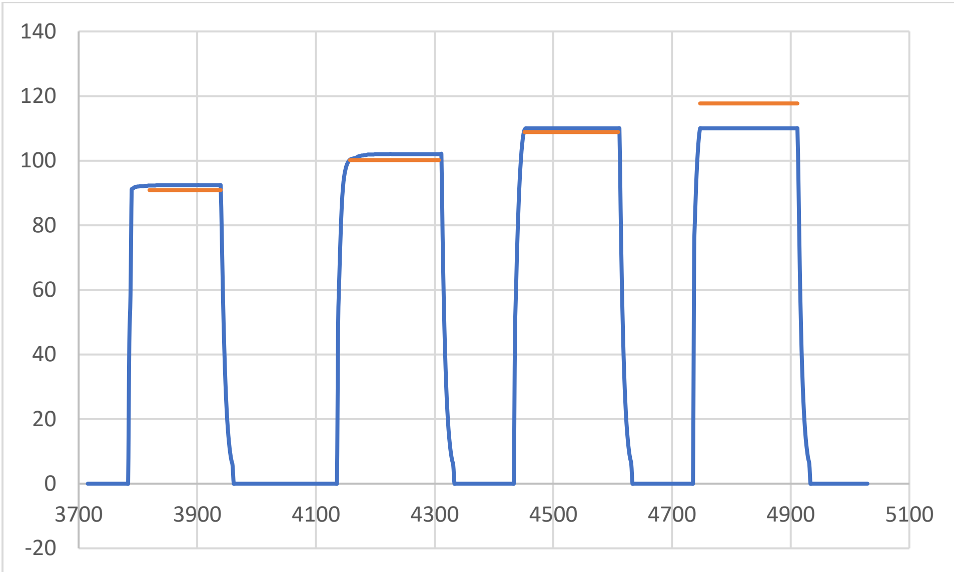


Figure 13. Saturation tests for sensor 2

The results show that the saturation occurs at LEL 110.

Using the limit of quantification (LEL 5) and saturation (110), the nominal range is 5-110.

Comments on the protocol

In the first version of the protocol, no value was given to guide the evaluation of the results of saturation testing. If only saturation is evaluated, it is enough to start the tests from the upper level of the working range. We propose to rephrase as:

What was done	Evaluation of the results	Measuring conditions	Comments
Expose the sensor to increasing concentration from the upper level of the working range indicated by the sensor´s manufacturer to evaluate the saturation.	Record the outputs. Saturation is reached when the bias between the true value and the output from the sensor differ by more than 2 times the bias at lower range	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.	

4.8 – Resolution

The 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.

To evaluate the resolution, the current protocol recommends performing the following tests (the column "what to do" has been completed afterwards:

What to do	Evaluation of the results	Measuring conditions	Comments
Expose the sensor to smaller and smaller changes of concentration around the mid-point or start close to the value provided by the software	Record the outputs	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.	

The reading´s resolution of the sensor is 0.1 LEL, as seen in the software. When the LEL is in between two readings, the signal fluctuates between these two values.

To test if the sensor actually responded to change of LEL of 0.1 LEL, the sensor was exposed to decreasing amounts of hydrogen with 0.03 LEL targeted increments for every step (with return to 0 between each test by exposing the sensor to synthetic air, this is not shown in the theoretical increment curve shown in Figure 16 in orange). For the tests, the test gas containing 40 vol-% hydrogen in nitrogen. This gas was diluted with synthetic air (ca 1200 ml/min air and 60 ml/min hydrogen gas). Due to the way the flow meters work (the actual flow is affected by the temperature and the pressure in the element), it is difficult to change the LEL with an exact increment. In that case, the flow of air was increased with 1 ml/min for every measurement, which resulted in increments of 0.02 (only 1-2 measurements) to 0.04 LEL (majority of the measurements), see Figure 14 that shows the evolution of the flow rate of air during the tests. It is also very important to wait until the temperature in the element has stabilized to ensure that increments will be as stable as possible. Figure 15 shows the variation of the temperature in the flowmeters used for a series of test. The figure clearly shows that the flowmeters require ca 50 minutes to reach a stable temperature.

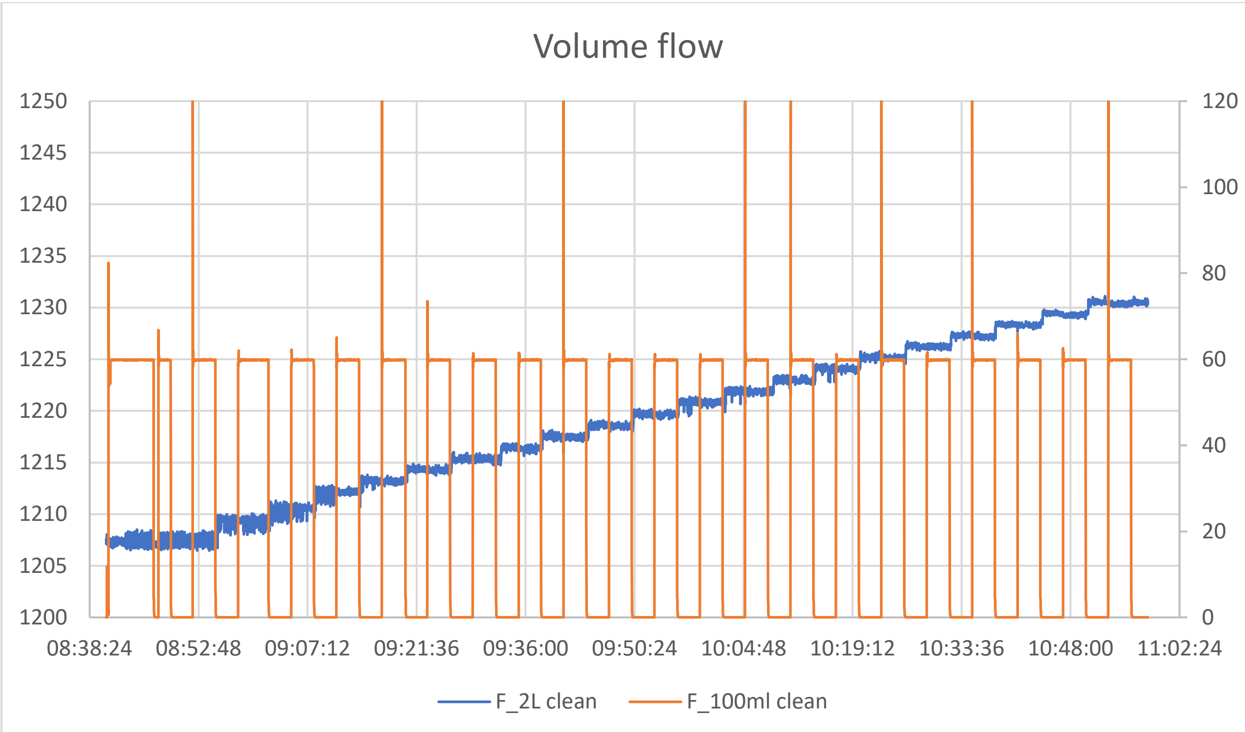


Figure 14. Flow of hydrogen (in red, set constant) and flow of air (increased by 1 ml/min every 3 minutes)

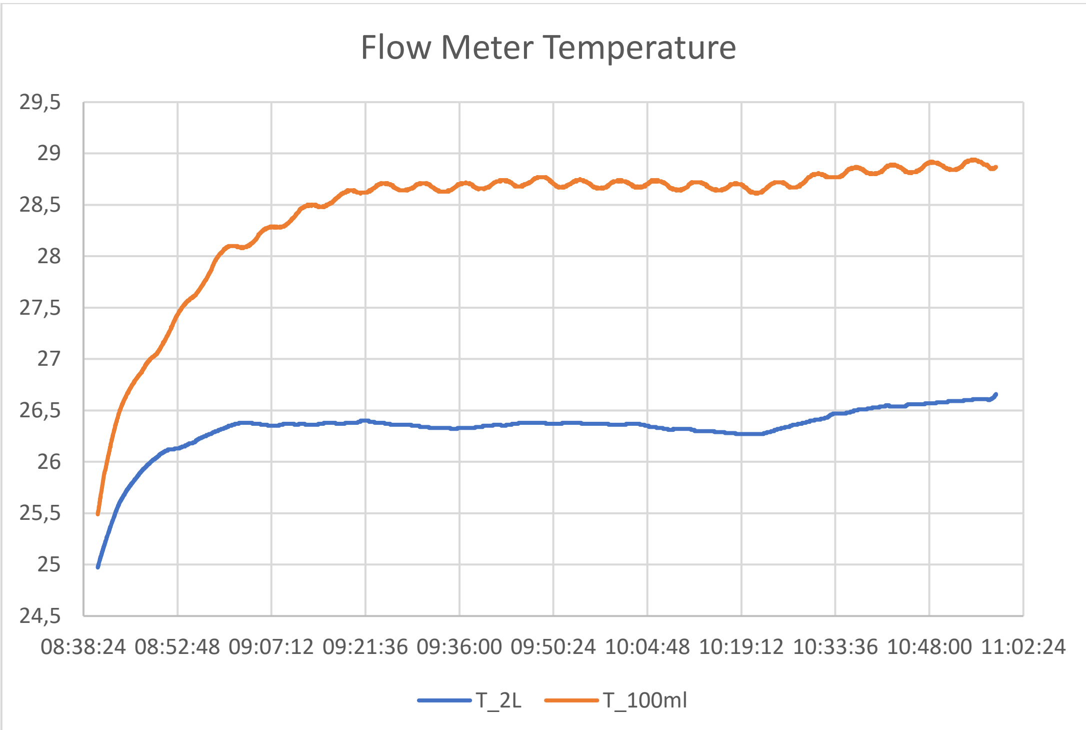


Figure 15. Evolution of the temperature inside the flowmeters used for the tests to evaluate the resolution

As it can be seen in Figure 16, the average of sensor´s response is similar for around three tests in a row (effective change of LEL 0.09-0.12) confirming the resolution of 0.1 LEL for this sensor.

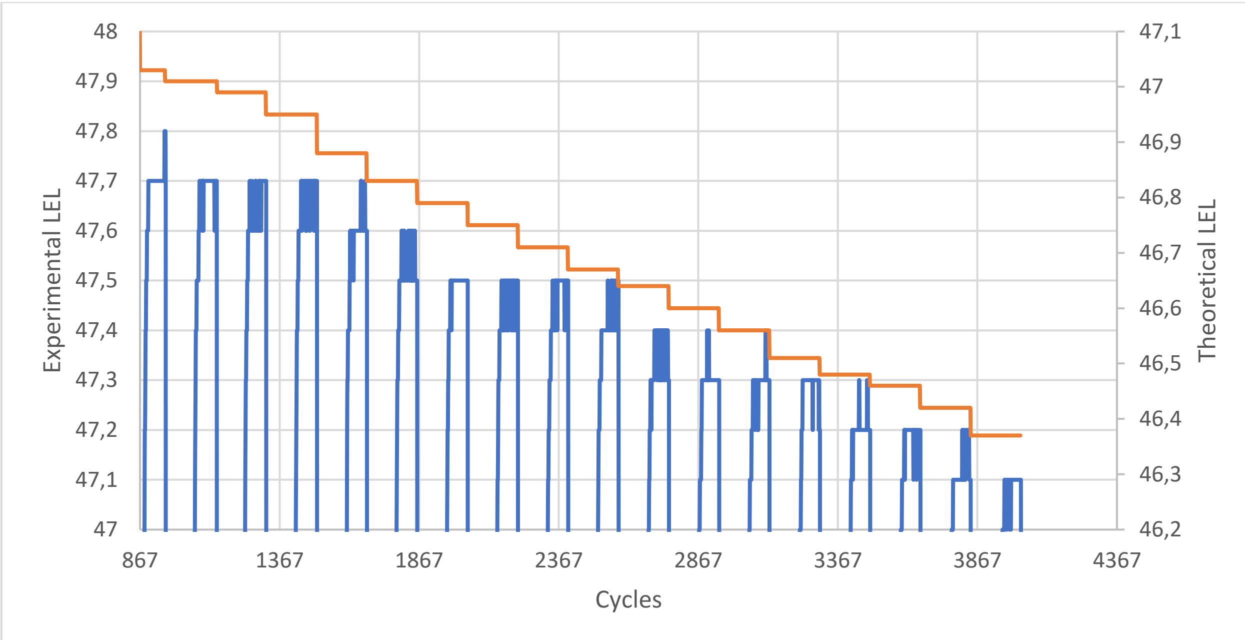


Figure 16. Sensor response (in blue) to decreasing LEL with increment of 0.02 to 0.04 LEL (in red)

In Figure 17, the theoretical LEL is compared to the measured LEL during the test. The results show that while the theoretical LEL follows a linear decrease, the measured LEL follows a staircase function with larger steps, where each step represents the resolution.

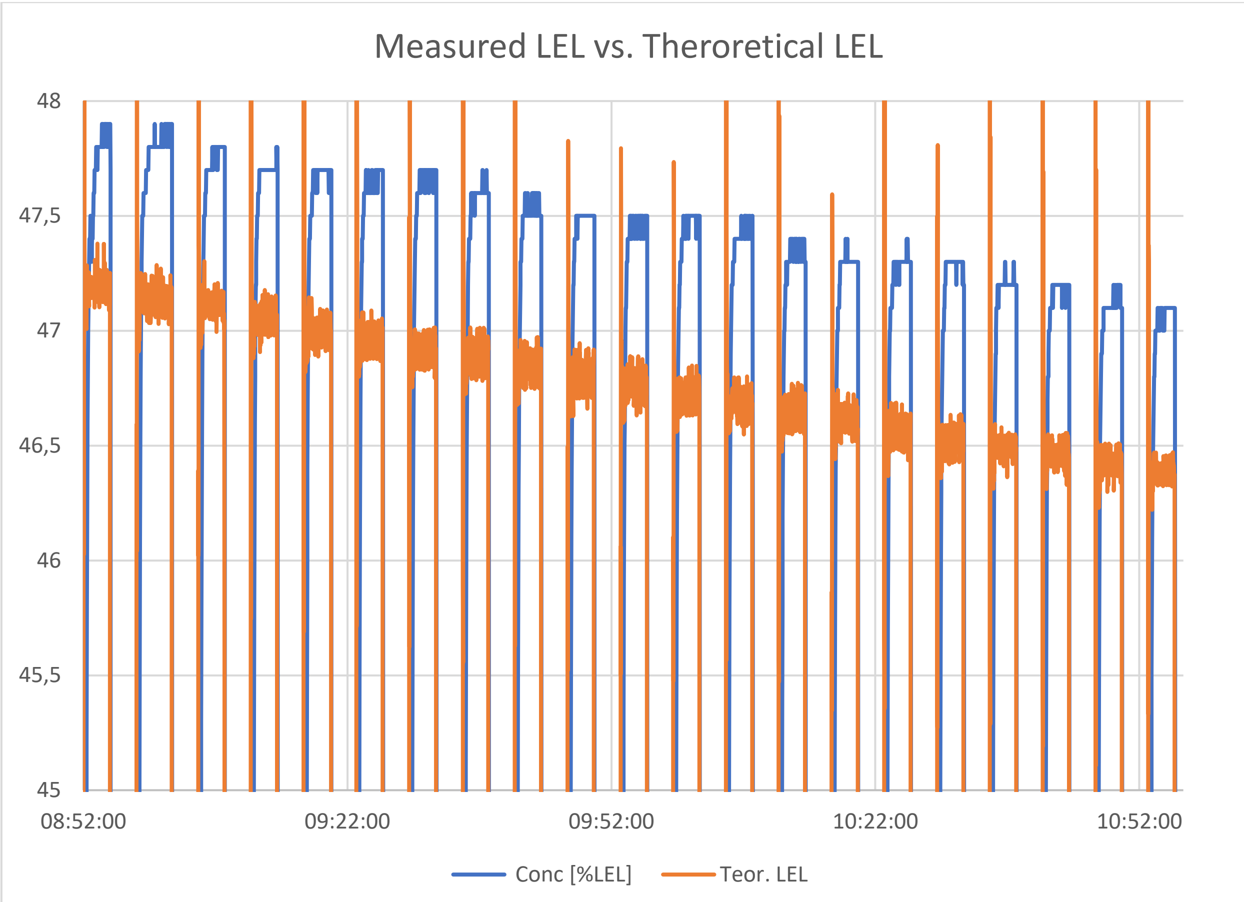


Figure 17. Comparison of the theoretical LEL and the measured LEL while exposing the sensor 2 to decreasing 0.02-0.4 LEL increments

In the series of tests performed, each increment of LEL was followed by exposing the sensor to air. The tests could also be performed by introducing increments continuously.

<u>Comment to the protocol</u>

<u>A suggestion on how to evaluate the results is added</u>

What to do	Evaluation of the results	Measuring conditions	Comments
Expose the sensor to smaller and smaller changes of concentration around the mid-point or start close to the value provided by the software	Record the outputs, the resolution is the value when the sensor actually reacts to a small change in concentration	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.	

4.9 - 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.

To evaluate the hysteresis, the current protocol recommends performing the following tests:

What to do	Evaluation of the results	Measuring conditions	Comments
Expose the sensor to 9 (or 10) increasing amounts of the measurand concentrations evenly spaced across the linear range). Expose the sensor to decreasing amounts of the measurand	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 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.	

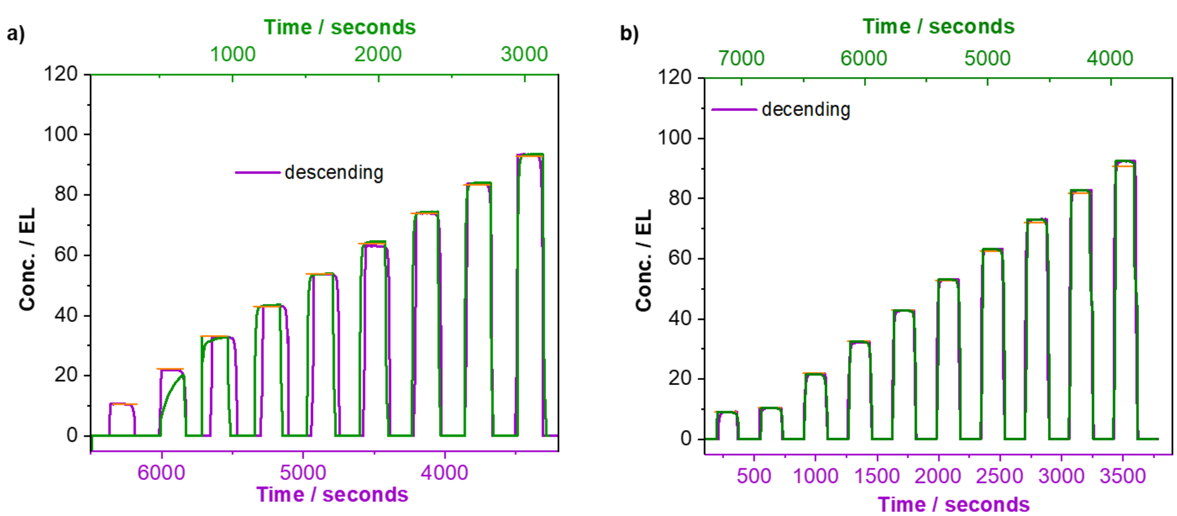


Figure 18. Evaluation of the hysteresis for sensor 1 (on the left) and sensor 2 (on the right) with descending concentration in purple and ascending concentration in green.

For the tests, the test gas containing 40 vol-% hydrogen in nitrogen was used. This gas was diluted with synthetic air (ca 500 ml/min air and from 10 to 50 ml/min hydrogen gas with increments of 5 ml/min, for low LEL, air flow was ca 1200 and then 1000 ml/min with 10 ml/min hydrogen gas on both occasions). The signals obtained when the sensor is exposed to increasing and then decreasing amounts of hydrogen is shown in Figure 18, the result show that the signals measured overlap for both sensors, showing that these sensors have no hysteresis. This is very clearly for sensor 2 while small differences are observed for sensor 1. For the measurement around 20% LEL recorded during the increasing series (performed first), it could be due to a warming-up effect of the sensor 1 (first test of the series). The lack of overlap of signal for the concentration around 40% LEL is due to different exposition times (4 min instead of 3min).

Comments on the protocol:

No comment

4.10 - Reversibility

Reversibility is the sensor's ability to return to its original state after the removal of the measured quantity. To evaluate the reversibility, the current protocol recommends performing the following tests:

What was done	Evaluation of the results	Measuring conditions	Comments
Expose the sensor to 9 (or 10) increasing amounts of the measurand 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

The same series of tests was used for hysteresis and reversibility. As it can be seen in Figure 19, the signal measured during the ascending and descending series when the sensor is exposed to no measurand is the same and equal to 0, showing that these sensors have good reversibility.

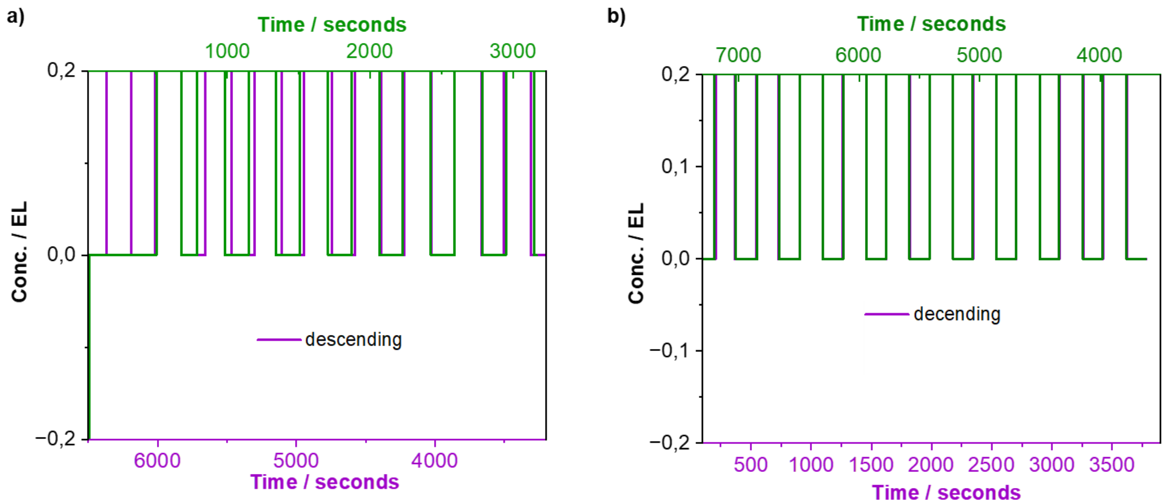


Figure 19. Evaluation of the reversibility for sensor 1 (on the left) and sensor 2 (on the right)

<u>Comments on the protocol:</u>

No comment

6 – Conclusions

This report presents the tests done on two selected sensors to evaluate their metrics using the protocol developed in A1.3.2. The goal of these tests is to demonstrate the applicability of the protocol and if deemed necessary, to improve the protocol. The tests done also allow to conclude that these sensors performed well.

As the results of these tests, some improvements of the protocol are suggested. For example, it is suggested to align this time with the response time (6-15 replicates for a duration of at least 10 times the response time, instead of a defined time of 10 min), which for the sensors having quick response time implies a shortened testing time and in turn, less testing gas is needed. When information was missing, new recommendations are now made. The outcomes of this activity will be used to write a guideline on validation, calibration and verification of hydrogen sensors used within (along) the hydrogen supply chain for quality control.