



Round Robin Testing of Commercial Hydrogen Sensor Performance—Observations and Results

Preprint

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*Presented at the 2010 National Hydrogen Association Hydrogen
Conference and Expo
Long Beach, California
May 3-6, 2010*

NREL is a national laboratory of the U.S. Department of Energy, Office of Energy Efficiency & Renewable Energy, operated by the Alliance for Sustainable Energy, LLC.

Conference Paper
NREL/CP-5600-48305
October 2010

Contract No. DE-AC36-08GO28308

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ROUND ROBIN TESTING OF COMMERCIAL HYDROGEN SENSOR PERFORMANCE—OBSERVATIONS AND RESULTS

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1. Introduction

Hydrogen sensors are essential to facilitate the detection of accidental hydrogen releases wherever hydrogen will be produced, distributed, stored, and used. Already, the use of hydrogen safety sensors is required for the indoor fueling of hydrogen powered forklifts (e.g., NFPA 52, Vehicular Gaseous Fuel Systems Code [1]). It is stipulated in NFPA 52 (e.g., 9.2.14, 9.2.14.1 and 9.2.14.2) that equipment used for the compression, processing, dispensing, storage, and generation of hydrogen shall be provided with gas detectors and flame detectors, such that fire and gas can be detected at any point on the equipment, and that these detectors shall be maintained and calibrated in accordance with the manufacturer's instructions on at least an annual basis or earlier if required by the manufacturer. NFPA 52 also requires that the station owner or operator shall maintain a record of detector maintenance and calibration in good condition and accessible to the inspector. Recognizing the importance of sensors for facilitating the development of the hydrogen infrastructure, DOE has published a list of target specifications for hydrogen safety sensors [2]. In addition, Codes and Standards for hydrogen detectors are being developed [3, 4] which, when adopted by authorities having jurisdiction, will impose mandatory analytical performance metrics. As a key enabling technology for safety monitoring of hydrogen applications, hydrogen sensors should be accurate, sensitive, specific, insensitive to other species, resistant to long term drift and environmental conditions, and should be capable of reliably alerting to the presence of hydrogen before explosive conditions are reached.

1.1 Sensor Performance Verification

Hydrogen sensor performance metrics are typically determined through a variety of test procedures performed in a laboratory on a custom built test apparatus. It is imperative that the resulting data should be representative of the expected performance of the sensor when deployed. Thus, quantitative assessment of sensor performance metrics should be independent of test site. However, owing to the sometimes subtle differences in test procedures, conditions, and hardware, variability in performance can often be observed between test laboratories for ostensibly identical sensors. This variability can arise from incomplete definition of test parameters or from fundamental variability

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between laboratory test systems. Sensor manufacturers will typically verify performance of their technology using in-house equipment and protocols. Oftentimes, however, end-users encounter sensor performance that deviates from that observed by suppliers. Independent measurement of sensor performance is critical to verifying that test results obtained by the manufacturer are repeatable and that test systems developed in different laboratories will produce comparable results under realistic and defined conditions. Variability in sensor performance metrics between laboratories leads to uncertainty of the device capability. To address this issue, the U.S. Department of Energy (DOE) has set up a sensor test facility at the National Renewable Energy Laboratory (NREL) in Golden, CO. Independently, the Joint Research Centre (JRC) of the European Commission has a sensor test facility at the Institute of Energy in Petten, the Netherlands.

The NREL and JRC sensor laboratories are both independent facilities dedicated to impartially evaluating and improving hydrogen sensor technologies for end-user applications. This is achieved by not only focusing on stakeholder requirements, but also by working directly with the sensor developers and manufacturers. Working with manufacturers will ensure that emerging commercial technologies meet end-user needs. Working with stakeholders will assist in the selection of the most suitable technology for a specific application.

1.2 Inter-laboratory Testing of Sensor Technologies

A large number of commercially available hydrogen safety sensors currently exist [5]. Sensor types include electrochemical, metal oxide, catalytic, thermal conductivity, optical, palladium-based, and other [6, 7]. Each sensor type has unique operating principles that will ultimately control its performance, and thus each technology has its advantages and limitations. No existing technology will be ideally suited for all applications. However, based upon specific requirements, one technology may be better suited for a particular application than others. The ultimate decision is the responsibility of the end-user. However, end-users need data in order to make informed decisions on monitoring technology; it is essential that this data represent the true capability of the sensing technology.

2. Sensor Testing Facilities

2.1 NREL Facility

NREL initiated sensor testing in June of 2008 using a sensor test apparatus developed at the Illinois Institute of Technology (IIT), which was specifically designed to address hydrogen sensor needs for PEM fuel cells [8]. The test apparatus had been operational at IIT since 2003. Evaluations subjected sensors to a variety of environmental stresses including elevated temperature (ambient to 200° C), elevated pressures (ambient to 5 atm), and relative humidity (dry to >95% at the test temperature). The tests performed at NREL were restricted to a maximum temperature of 80° C and 1.5 bar pressure. Gas composition and flow were controlled by four mass flow controllers

(MFC). A LabVIEW based data acquisition system logged the hydrogen sensor response and critical experimental parameters including temperature, pressure, humidity, and total gas flow. NREL has recently enhanced its sensor evaluation capability by the design and construction of an advanced chemical sensor test system, shown in Figure 1. The new NREL system was commissioned in the fourth quarter of 2009 and has a broader range of capabilities than the old IIT system, including parallel testing of multiple hydrogen sensors, sub-ambient to elevated temperature extremes (-30 to +80° C), sub-ambient to elevated pressure (0.7 to 1.4 bar), active humidity control (dry to 95% at test temperature and pressure), and improved control of gas parameters



Photo Credit: Robert Burgess, co-author
Figure 1: NREL Sensor Testing Facility

with six precision digital mass flow meters operating in parallel. A pressure controller is used to regulate test pressure, and sub-ambient pressures are obtained with a high-throughput vacuum pump. Test conditions (flow, T, P, RH) are monitored with traceable probes. The current limitations on test parameters (e.g., T and P) are restricted by in-house administrative requirements and not by the fundamental design of the test facility. Broader environmental ranges can be achieved through routine changes in operational protocols. Evaluations are carried out in the sensor chamber, which isolates the test sensor from the external environment. The test chamber (without external thermal insulation) is shown in Figure 2. In operation, the chamber is fully insulated to maintain a stable test temperature. Temperature control is achieved by circulating a thermally regulated heat transfer fluid through sealed channels machined into the walls of the chamber. The test chamber has eight side ports that accommodate pneumatic



Photo Credit: Matthew Post, co-author
Figure 2: The NREL sensor test chamber with viewing port on a load-lock lid and 8 side ports for pneumatic and electrical feedthroughs.

feedthroughs to allow gas flow and electric feedthroughs to provide communication and power to the test sensors and the probes for temperature, pressure and humidity measurements. Up to six DB9 electric feedthrough connectors can be mounted on the side ports. The test chamber top was designed with a load-lock door assembly with a view port that allows for easy access to the test chamber for rapid sensor deployment. The viewport feature allows for visual monitoring of the test

sensors during operation. Although the residual gas analyzer is not yet in operation, gas composition will be independently and continuously verified by mass spectrometric analysis which will provide near real-time analysis of the test gas as it leaves the test chamber. LabVIEW 2009 is used for data acquisition and to control and monitor all

critical test parameters. The system is fully automated for around-the-clock operation with remote control and monitoring capabilities via the internet. Strategically, the NREL Sensor Laboratory was developed to serve as an unbiased interface between end-user needs and sensor producers. In keeping with the NREL goal of facilitating the development of sensors that meet end-user requirements, NREL has been working closely with various manufacturers to provide critical data and expert analysis of the sensor performance [e.g., 6]. There is currently a waiting list for access to the facility.

2.2 JRC Facility

A similar sensor test facility has been deployed in the European Union by the Joint Research Centre at the Institute for Energy in Petten, The Netherlands, and has been previously described [7, 9-12]. As with the NREL sensor test fixture, the JRC facility is comprised of a test chamber, a gas handling system, a LabVIEW-based control and data acquisition system, a gas analyzer for independent verification of test gas concentrations, and systems for control of environmental parameters (e.g., T, P, RH). The LabVIEW-based control and data acquisition system controls test conditions and generates electronic data files for the hydrogen sensors and supplemental probes. The vessel, with an inner volume of 2.4 L, is internally coated with HALAR polymer for chemical resistance and is further contained inside another vessel, which can be closed and purged with an inert gas flow to isolate the main chamber and test vapors from the laboratory environment. The insulation improves thermal stability. Up to six sensor samples can be mounted in the chamber. All electrical signals, including sensors' inputs and outputs are transferred into and out of the chamber by means of two 25-pin feedthroughs. The test gas is introduced into the bottom of the test chamber and leaves from the top. A small fan inside the chamber ensures homogeneity of temperature distribution and gas composition. Moreover the gas piping is heated to prevent condensation of water vapor on the internal walls. A series of mass flow controllers are used to control the flow rates of up to four gases or gas mixtures into the system. Temperature in the chamber is controlled by circulating a thermostated heat transfer fluid between the walls of the double-walled sensor test chamber.

Temperature in the chamber is measured by means of three Pt100 thermometers connected to the LabVIEW data collecting system. Humidity is controlled by dynamic evaporation of water into the test gas by means of a Bronkhorst controlled evaporator mixer (CEM). Based on the water/gas ratio introduced into the CEM the relative humidity of the gas can be estimated. Nevertheless, the humidity is monitored during all tests using a chilled mirror dew point

meter. The gas flow and pressure control system have been configured to remove pressure and gas flow instabilities. Gases pass from the respective MFCs through a gas



Photo Credit: Lois Brett, co-author
Figure 3: JRC Sensor Test Facility

mixer where they are mixed thoroughly. The actual composition of the gas is continuously analyzed during tests by a calibrated compact gas chromatograph (GC).

2.3 Comparison of Facilities

The NREL and JRC sensor test facilities are similar, but having been developed independently, differences do exist between the respective designs and operational protocols. Control of environmental parameters (e.g., T, P, RH) uses different components, design elements, and protocols. For example, both facilities control humidity via a liquid delivery system that precisely regulates the rate of water injected into the sample gas stream to generate a controlled RH. For the JRC, a liquid mass flow control system is used, while NREL designed their system around a precision HPLC pump. Different probe designs are used to monitor the humidity of the test. NREL monitors the RH with a commercial relative humidity sensor with a NIST traceable calibration. The JRC facility uses a dew point probe certified to European standards. Differences exist in the temperature and pressure control systems as well. Data acquisition protocols are different; NREL designed its DAQ system to log at a fixed, constant frequency (typically 1 point/sec), whereas the JRC facility logs at a variable rate controlled by the rate of change of the sensor response such that the logging rate at steady state is slower than that during fast changes in the sensor output. NREL also over samples during data acquisition (e.g., each logged data point is the average of ten or more individual measurements) to minimize noise in the system, whereas the JRC system does not average the output signal. There are also subtle changes in pneumatic parameters such as chamber volume, total gas flow rate, mixing designs. Although both laboratories use certified gas standards for testing, they are obtained from different vendors. One other potentially significant difference in the operation of the laboratories is that the facility at the JRC performs analyses during normal business hours whereas the NREL facility was developed for “around-the-clock” operation, with remote integration capability via the internet. Sensors may have less chance to recover with continuous testing. Such differences, and especially more subtle unidentified design or operational parameters could impact in an unpredictable manner on the performance of a sensor. It is critical that sensor evaluations be independent of test site.

3. Round Robin Sensor Testing

3.1 Motivation

Recognizing the need for validating the respective test facilities and protocols for consistency, the NREL and JRC sensor laboratories committed to performing an inter-laboratory *round robin test* (RRT) of representative commercial hydrogen detectors. The JRC-NREL RRT has been named the *Sensor Interlaboratory Comparison* (SINTERCOM), and is a collaborative project that addresses the need for validating hydrogen sensor test systems and protocols for inter-laboratory consistency. This paper describes the SINTERCOM test protocols and results obtained to date. Direct comparison of the results from the RRT highlights factors which may influence the

results and improve comparability between laboratories. More importantly, such RRT testing benefits sensor developers and other stakeholders. The benefits of SINTERCOM include:

- Support towards Technology Development
 - Independent, unbiased assessment of performance
 - Expert analysis and feed back opportunities to the manufacturer
 - Exposure to international markets
- Test Laboratories
 - Opportunity to critically evaluate sensor test protocols
 - Validate competences of participating laboratories
- End-User benefits
 - Information on sensor performance
 - Importance of sensor use

The current policy for SINTERCOM is to not publically disclose performance obtained from a specific technology and to restrict the distribution of the obtained data. Although data and assessments will be published to summarize general performance capabilities of a sensor type, no specific vendor will be identified. Data and other assessments obtained within SINTERCOM will be shared with the manufacturer.

The term “sensor” refers to the discrete element which undergoes a change in electrical property upon exposure to hydrogen. The term “sensor” is also applied to instrumented systems that provided quantitative information on hydrogen levels. The user interface may be an electrical signal (e.g., voltage, current), digital output (e.g., counts), or a visual display (e.g., LED). “Detector” or “analyzer” is also used for an instrumented system with a user interface. In ISO/FDIS 26142, the active discrete element is defined as the “sensing element”. SINTERCOM testing focuses on instrumented systems (detectors) which provide quantitative information on hydrogen levels.

3.2 Test Protocol

The SINTERCOM test protocol stipulates three rounds of evaluations to be performed on each model type at each participating laboratory. The specific performance tests were designed to follow defined protocols consistent with but not necessarily identical to those described in international standards (e.g., ISO/FDIS 26142 “*Hydrogen Detection Apparatus for Stationary Applications*”). Representative samples of a selected sensor technology were tested by each laboratory and then exchanged before being retested by the other laboratory. SINTERCOM calls for six units of the same type to be acquired, with three units distributed to each participating laboratory for the first round of evaluations. Detectors employing different detection principles (e.g., different types of sensing elements) were chosen. Testing is currently restricted to mature technologies that are readily available commercially. Although developing technologies are evaluated by both laboratories, these evaluations are not performed under the auspices of SINTERCOM.

Round 1 of SINTERCOM focuses on the assessment of the analytical performance of the sensor. A variety of tests were designed to verify accuracy, measuring range, lower detection limit, short term stability and the impact of changes in environmental parameters on sensor response. The specific Round 1 protocols are illustrated in Figure 4 and summarized in Table 1. All tests were performed under regulated temperature, pressure and humidity. The standard test condition was set to 1.00 ± 0.05 bar pressure, $50.0 \pm 5.0\%$ RH, and $25.0 \pm 2.0^\circ$ C. Naturally, the test conditions are adjusted when measuring the impact of temperature, pressure, or humidity on the instrument, but typically only one parameter is varied for each test. Instruments were calibrated by the manufacturer prior to delivery and thus were tested as received from the manufacturer, unless specific recommendations were provided mandating user-calibrations. An *initial calibration* (validation) of the sensor system was performed to measure the response of the detector to certified standards of $1 \pm 0.05\%$ and 2 ± 0.05 vol% hydrogen in air under the standard conditions (Figure 4, left). This measurement serves as a reference response to track any long-term changes in instrument behaviour that may develop during SINTERCOM.

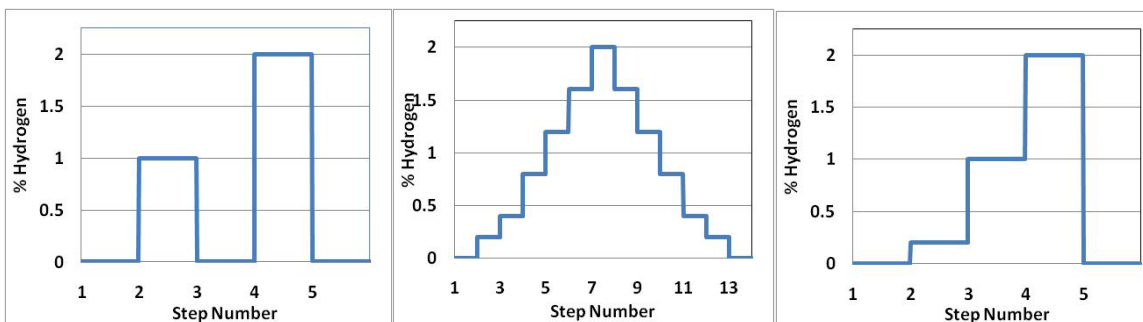


Figure 4: Hydrogen exposure profiles for sensor evaluations performed in SINTERCOM. (Left): Each sensor is exposed to two concentrations of a certified hydrogen in air standard at 1 vol% \pm 0.05% and 2 vol% \pm 0.05% with a constant nominal condition of 25° C, 1 bar, and 50% RH. The sensor is exposed to clean air with 0% hydrogen between steps (Center): The sensor is then exposed to seven hydrogen in air concentrations in the range 0 to 2 vol%. (Right): A four-concentration exposure profile consisting of 0, 0.2, 1.0 and 2.0 vol% hydrogen in air generated by the gas preparation systems of the NREL or JRC sensor test apparatus. This profile is used to assess short term stability (repeated nine times in 48 hours) and the impact of environmental parameters (performed once for each defined condition).

Following the initial calibration, the detectors were subjected to a series of tests designed to provide a quantitative assessment of the analytical specifications. The “linear range” test subjects each detector to seven different hydrogen concentrations (0 to 2 vol%) in ascending and descending levels (Figure 4, center). The various test concentrations were prepared by dynamic blending of hydrogen in the gas generation systems from a single reference cylinder of hydrogen in air. The time for each step was independently defined by the respective test laboratory. The “repeatability” test (or “short-range stability” test) subjects the sensor to nine exposure sequences of four concentrations of hydrogen (0, 0.2, 1.0 and 2.0 vol%) in air. This sequence is illustrated in Figure 4, right. This exposure sequence is also used for assessing the impact of pressure, temperature, and humidity, however, the exposure sequence is performed only a single time for each environmental condition. The final test of the Round 1 evaluations is a repeat of the calibration protocol (final calibration) with the certified gas standards. The SINTERCOM Round 1 protocols are summarized in Table 1.

Table 1: The SINTERCOM Round 1 (and Round 2) Test Protocols

Test Name	Hydrogen Profile (in air)	T (°C)	P (bar)	%RH
Initial Calibration	0.0, 1.0, 2.0, 0.0	25.0 ± 2.0	1.00 ± 0.05	50.0 ± 5.0
Linear Range	0.0, 0.2, 0.4, 0.8, 1.2, 1.6, 2.0, 1.6, 1.2, 0.8, 0.4, 0.2, 0.0%	25.0 ± 2.0	1.00 ± 0.05	50.0 ± 5.0
Repeatability ¹	0.0, 0.2, 1.0, 2.0, 0.0,	25.0 ± 2.0	1.00 ± 0.05	50.0 ± 5.0
Pressure ²	0.0, 0.2, 1.0, 2.0, 0.0,	25.0 ± 2.0	0.80 ± 0.05 1.00 ± 0.05 1.20 ± 0.05	50.0 ± 5.0
Humidity ²	0.0, 0.2, 1.0, 2.0, 0.0,	25.0 ± 2.0	1.00 ± 0.05	< 5.0% (dry) 25.0 ± 5.0 50.0 ± 5.0 85.0 ± 5.0
Temperature ²	0.0, 0.2, 1.0, 2.0, 0.0,	-20.0 ± 2.0 0.0 ± 2.0 25.0 ± 2.0 50.0 ± 2.0 85.0 ± 2.0	1.00 ± 0.05	< 5.0% (dry)
Final Calibration	0.0, 1.0, 2.0, 0.0	25.0 ± 2.0	1.00 ± 0.05	50.0 ± 5.0

¹ The hydrogen profile is performed nine (9) times for the repeatability test.

² The hydrogen profile is performed once for each condition.

The results from each laboratory are shared and used to evaluate the performance of the sensors. It should be noted that NREL also shares data with and provides an analysis of the sensor performance to the sensor manufacturer. Upon completion of Round 1, the units are exchanged between laboratories and the sensors are then subjected to the Round 2 protocol. The test protocols for Round 2 are identical to Round 1, including the initial and final calibrations with certified standards. Thus, both the robustness of a hydrogen detector to maintain performance and the ability of the respective laboratories to produce comparable results are tested. Round 3 tests, which have not yet been initiated, will focus on the long-term stability (three months or longer with validation challenges performed every two weeks) of the sensor under standard conditions. Round 3 will also investigate the impact of gas interferants (e.g., carbon monoxide, carbon dioxide, nitrogen oxide, ammonia) and potential poisons (e.g., silicon compounds, sulphur compounds) on the performance and lifetime of the detectors.

4. SINTERCOM Results

As of the 2010 National Hydrogen Association conference, NREL and the JRC sensor testing laboratories have completed Round 1 on the initial detector technology selected for SINTERCOM. Preliminary data workup has been completed and reviewed with the manufacturer. Round 2 is initiating, as well as the addition of numerous other

technologies for incorporation into SINTERCOM. Round 3 testing will commence upon completion of Round 2 for each sensor type. Technologies already selected include electrochemical, metal oxide, semiconducting, and thermal conductivity. Additional technologies, including duplicate designs of similar types are to be tested within SINTERCOM. The following analysis summarizes the status of SINTERCOM as of May 2010. It is stressed that the results shown are illustrative of the types of tests that have been performed in Round 1. It is further emphasized that since the SINTERCOM policy is to restrict data distribution, the technology type will not be identified in the following assessment.

4.1 Round 1 Sensor Performance

Representative data from the JRC and NREL facilities are shown in Figure 5. Specifically, a single run (out of nine) for the Repeatability test is illustrated. It should be stressed that the kinetics of the sensor response indicated in Figure 5 are a manifestation of the design of the sensor test facilities at JRC and NREL, since it takes a finite time to purge the sensor chamber with the test gas and for the transient test gas concentration to reach the desired level. Figure 5 shows the response from two separate instruments of the same model type. A total of six units have been tested. Comparable results were obtained with all six units indicating a good unit to unit repeatability. As stated above, three units were tested at NREL and three at JRC. Slightly different protocols were used at each laboratory. At NREL each step was 60 minutes in length compared to 45 minutes at JRC. NREL used the standard exposure sequence of 0.0, 0.2, 1.0, and 2.0 vol% hydrogen in air while the JRC adjusted the lowest hydrogen concentration to 0.6 vol%. NREL recorded 1 data point every second whereas JRC used a variable logging frequency. The instrument whose performance is illustrated in Figure 5 was designed to null out (set to zero) any hydrogen concentration below 0.4 vol%. Thus, no response was observed at NREL for the 0.2 vol% hydrogen test. Because of this design feature, JRC chose to adjust the protocol from 0.2 to 0.6 vol% hydrogen. The following analyses compare the final stable indication of the sensor response between Unit 1 and Unit 2. The final indication corresponds to the steady state signal obtained for a specific gas concentration. For the analyses described below, this value was obtained from the sensor response measured in the last 60 seconds of exposure. The measured hydrogen concentration was derived from analog output signals (e.g., current, voltage) stored in the data files generated by the DAQ using calibration data and protocols provided by the manufacturer; in some instruments digital output via serial communications provides a direct readout of the hydrogen concentration.

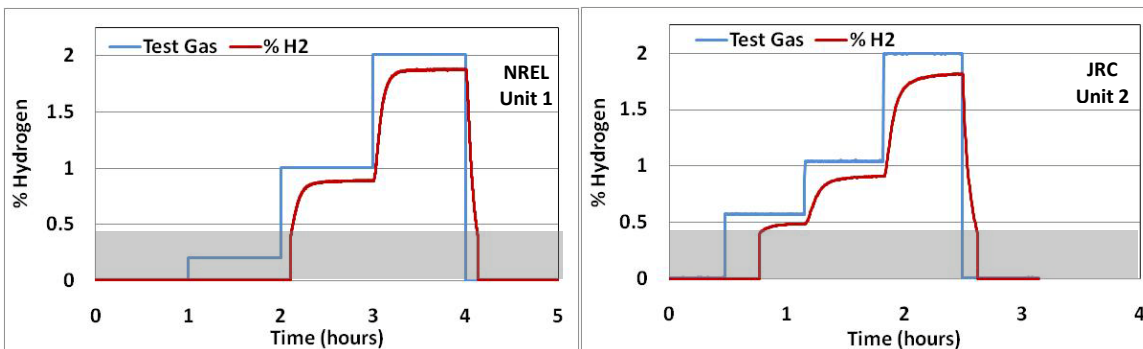


Figure 5: Hydrogen detector measurements performed at NREL for Unit 1 (left) and JRC for Unit 2 (right). Unit 1 and Unit 2 were two samples of the same model type. Nominal conditions were 25° C, 1 bar, and 50% RH. The hydrogen concentration obtained from each unit (—) is plotted against the concentration of the test gas (—). The detector was designed to null out any response corresponding to 0.4% hydrogen or less, as indicated by the shaded area in the plots.

4.2 Analytical Characterizations

The detector response to hydrogen was evaluated in detail as part of SINTERCOM. Tests were developed to allow for quantitative analysis of critical sensor performance parameters. The Linear Range test subjects the detector to seven concentrations of hydrogen in ascending and descending order, as illustrated in Figure 4 (center). The response of one of six detectors subjected to this test is illustrated in Figure 6, in which the measured hydrogen concentration is plotted along with the test gas concentration. The final indication of the detector to each concentration, as measured in the last 60 seconds of each step, was linear (Figure 6, right) at least for the range of concentrations that were in excess of 0.4%. Again, the design of this technology would null out any response that corresponded to hydrogen concentrations less than 0.4%. Although a linear response was observed, the measured concentration of hydrogen was less than the concentration of the test vapor. The measured concentration was however, within the manufacturer’s specifications for accuracy. The deviation of the measured hydrogen concentration from the test gas was investigated in detail in the Repeatability test protocol.

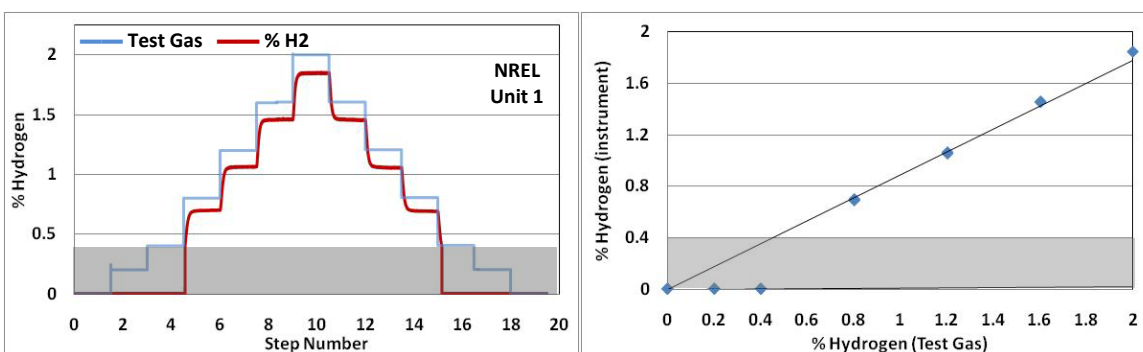


Figure 6: The Linear Range Test. (Left): Hydrogen detectors are exposed to seven concentrations of hydrogen in the range of 0.0 to 2.0 vol% in ascending and descending order (—). The output of the detector is monitored (—). (Right): Plot of the final indication of the detector vs. the input hydrogen concentration. The detector was designed to null out any response corresponding to 0.4% hydrogen or less, as indicated by the shaded area in the plots.

In the Repeatability test (also called the Short Term Stability test), the hydrogen detector is subjected to a series of repeated exposures to four concentrations of hydrogen (0.0, 0.2, 1.0, and 2.0 vol% in air). The specific sequence is illustrated in Figure 4, right, and this sequence is repeated nine times. This test focused on the ability of a detector to provide predictable, repeatable signals. The results for two detectors, one tested at NREL and one tested at the JRC, are shown in Figure 7. The results for the other four detectors were comparable. Draft international standards [4] stipulate that the final indication signal for short-term stability shall be within $\pm 10\%$ of the test gas concentration. However, it should be stressed that specifications are somewhat arbitrary. Although a standard requires accuracy to within $\pm 10\%$ of the test gas, the manufacturer specification is less stringent. In Figure 7, the manufacturer's accuracy specification is shown as $\pm 20\%$ of the test gas concentration. It is clear from figure 7 that the detector showed good stability, and that all measurements were within the manufacturer specification. To meet the draft ISO standards it would be necessary to recalibrate the instrument and to show that following signal adjustment (recalibration) the sensor reliably measures hydrogen concentration. This appeared to be the case for this detector, as shown in Figure 7, right.

Several other observations can be made. The units behaved in a nearly identical manner. This illustrates not only the unit to unit repeatability but it demonstrates that the JRC and NREL test facilities will produce comparable data. A second observation is that although the signals were quite reproducible between runs, there appeared to be a systematic decrease, albeit small, in the measured hydrogen concentration due to repeat exposures. Thus, although the measured response was within the manufacturer specification, signal drift with continued exposure may ultimately lead to inaccuracy. This effect was minimized when longer delay times between tests were used. JRC would stop testing at the end of the work day, thus there would be the opportunity for an overnight recovery leading to a smaller decrease in the detector response due to repetitive hydrogen exposure. This is observed in the JRC data in which the loss of signal from repeat testing is slightly less than that induced by the NREL protocol in which the stability tests were performed "around-the-clock". The observed loss of signal between sequential exposures was small and its effect would not impact the ability of the detector to respond to a spurious hydrogen event.

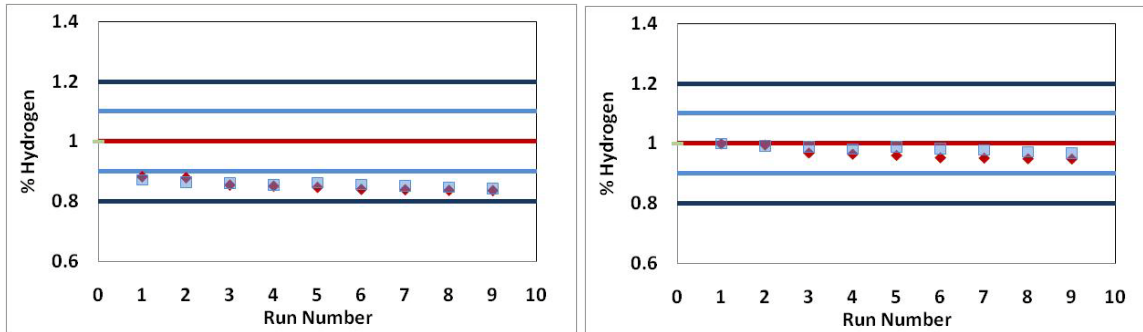


Figure 7: The SINTERCOM Repeatability test. Final indication of the hydrogen detector to the nine sequential exposures of hydrogen in air under nominal conditions of 25° C, 1 bar, and 50% RH. The 1% hydrogen results are shown (Left): Response obtained from Unit 1 (♦) tested at NREL and Unit 2 tested at JRC (□). The ISO draft specification for repeatability (—) and the manufacturer specification for overall accuracy (—) are plotted for comparison. Although the instrument did not meet the ISO specification, it did meet the manufacturer specification. (RIGHT): Same data, but concentrations recalculated according to a new calibration factor (the first data point for the repeatability test). Upon calibration, the instrument met both the ISO and manufacturer specification.

4.3 Impact of Environmental Parameters

The ability of a hydrogen detector to accurately measure hydrogen concentrations under standard conditions represents only a preliminary but critical assessment of a device performance. Real world deployment will not necessarily have controlled environmental conditions. Temperature, pressure, and humidity levels are likely to fluctuate, especially in an outdoor deployment. Accordingly, the detectors were subjected to a pressure test (0.8 to 1.2 Bar, to mimic likely barometric pressure conditions), a temperature test (-20 to +50° C), and a humidity test (RH at 25° C of <5% to 85%); see Table 1 for specific conditions. Figure 8 summarizes the impact of fluctuations in environmental parameters as measured at NREL and the JRC. Excellent repeatability between laboratories was observed. Again, the results are plotted against the draft international standard for the various parameters. Typically, the draft ISO standard allows deviations of up to $\pm 30\%$ of the test gas, whereas the manufacturer specification remains $\pm 20\%$ of the test gas. As with the repeatability test, the consistency between laboratories was quite high. The detector did show a positive pressure dependency, that is, its output increased with pressure. This is common with many sensing elements, because as pressure of the test gas mixture increases, the actual partial pressure of hydrogen in the mixture will also increase proportionally. The pressure variance can be easily compensated by an independent measurement of the pressure and adjusting the hydrogen concentration via a proportional scaling relative to the pressure that prevailed during calibration. Some instrument systems do this internally. Conversely, the detector showed a strong negative temperature effect, in which the measured hydrogen concentration increased with decreasing temperature. However, the device remained within specification except for the lowest temperature (-20° C). Again, since the data indicates comparable trends among all instruments tested, the temperature dependency is fairly predictable and can be compensated via incorporation of additional design features within the instrument itself (electronic or microprocessor controls) or by a temperature-controlled scaling of the measured hydrogen concentration. The humidity effect on this particular instrument was quite small.

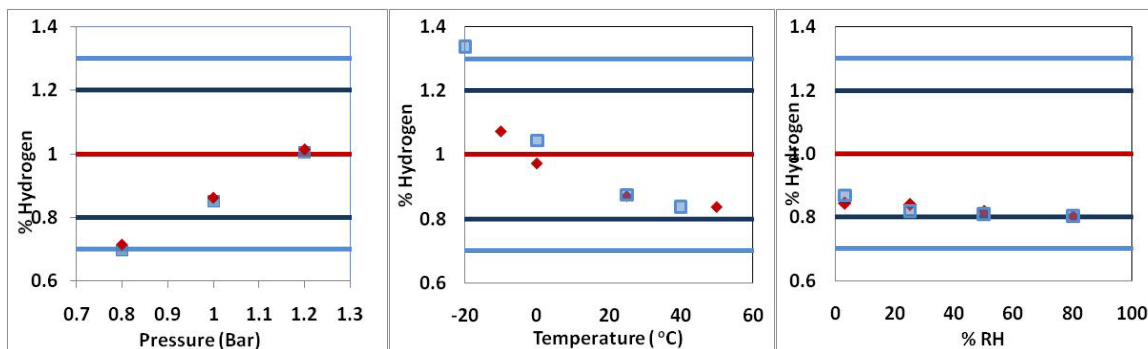


Figure 8: The SINTERCOM pressure, temperature and humidity test. Final indication of the hydrogen detector for each condition is plotted. The 1 vol% hydrogen results are shown (Left): Pressure dependence obtained from Unit 1 (♦) tested at NREL and Unit 2 tested at JRC (□). (Middle): Temperature dependence obtained from Unit 1 (♦) tested at NREL and Unit 2 tested at JRC (□). (Right): Humidity dependence obtained from Unit 1 (♦) tested at NREL and Unit 2 tested at JRC (□). Draft ISO specification for impact of the specific environmental parameter (—) and the manufacturer specification for overall accuracy (—) are plotted for comparison.

5. Summary

The round robin testing between the NREL and JRC sensor test facilities has recently been initiated. Round 1 testing of a selected hydrogen detector has been completed and preliminary analyses performed. The analysis was shared with the manufacturer. The six units tested showed comparable performance, indicating a satisfactory unit to unit repeatability. Testing was performed at two facilities and the results compared for analysis. The excellent inter-laboratory consistency on performance measurements affirms the accuracy and capability of the respective sensor test laboratories. Round 2 testing of the initial technology has commenced, which as per the SINTERCOM protocol requires that the participating laboratories exchange the three units and repeat the Round 1 testing. Round 2 focuses on instrument stability and ability to perform predictably in different environments. Round 3, which will commence upon completion of Round 2, will focus on stresses that a technology may experience in deployed conditions, including long-term stability and robustness against potential interferants and poisons. The scope of SINTERCOM is expanding. Additional technologies have been either acquired or identified for incorporation into the test plan. The selected technologies represent different sensing element platforms and will ultimately include multiple samples of all the major sensor types including metal oxide, electrochemical, combustible gas sensors, thermal conductivity, palladium devices and optical technologies.

SINTERCOM is not an academic exercise. It is critical that test laboratories provide accurate assessment of performance. This requires at a minimum a consistency between laboratories. SINTERCOM will specifically validate the inter-laboratory consistency between the NREL and JRC sensor laboratories. Additional test laboratories can be identified and will be included as appropriate. Inter-laboratory testing also provides the opportunity to test protocols used to assess sensor performance.

SINTERCOM was explicitly developed to serve the needs of end-users of hydrogen sensors. As a critical element in the design of safety systems for hydrogen operation, end-users must have an understanding of the performance specifications that can be expected from a given sensor technology. SINTERCOM was developed to provide stakeholders with the data that they need to make informed decisions on sensor technology. Finally, to ensure that technologies exist that meet the needs of the end-user, the SINTERCOM actively interacts with sensor manufacturers, including sharing of data and an unbiased, expert analysis of performance. Topical discussions with vendors may also include specific applications and the associated requirements for such applications. Requirements for an application may include those mandated by code [4] or by technical issues associated with the application.

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William Buttner, Ph.D.

Senior Scientist I, National Renewable Energy Laboratory

Dr. William Buttner is a staff scientist in the Hydrogen Technologies and System Center of the National Renewable Energy Laboratory (NREL) in Golden, CO. At NREL, his work focuses on the safe implementation of alternative fuels for vehicle applications, with an emphasis on hydrogen safety, codes and standards. He has been active in the design and construction of the Hydrogen Sensor Laboratory at NREL. Dr. Buttner serves on a variety of Standards Development Organizations that pertain to the safe utilization of hydrogen, including ISO, IEEE, and UL. Dr. Buttner serves on the Steering Committee for the Sensor Division of the Electrochemical Society. Prior to joining NREL, Dr. Buttner has over 25 years experience in chemical and physical sensor technology, field analytical chemistry, and electrochemical processes. Just prior to joining NREL, Dr. Buttner was at the Illinois Institute of Technology as a Research Professor in Chemistry and Director of the International Center for Sensor Science and Engineering. Dr. Buttner has over 45 publications in the area of chemical sensors and analytical methods for sensor and instrument-based on-site chemical analyses. Dr. Buttner received his Ph.D. in chemistry from Michigan State University.

Robert Burgess

Senior Engineer, National Renewable Energy Laboratory

Robert Burgess is a senior engineer, joining the National Renewable Energy Laboratory in the spring of 2007. Robert brings over 20 years of high technology industry experience to the Hydrogen Technologies and Systems Center at NREL. Prior to his current position he was a project manager responsible for the development of a super critical cryogenic hydrogen system supplied to Oak Ridge National Laboratory for use in their Spallation Neutron Source accelerator. A new world record was set by accelerating a proton beam to 1.01 GeV in a linear accelerator. He had the opportunity to engineer products as diverse as space shuttle main engine turbomachinery at Rocketdyne, high speed centrifugal equipment for the oil and gas industry while working at United Technologies, first generation F-22 Raptor jet aircraft engine fuel systems at a division of Colt Technologies and advanced automation equipment for disc drive manufacturing at Seagate Technologies. He was recognized with a best paper award at the 21st Joint Propulsion Conference of the AIAA for his work on small centrifugal pumps for low thrust rocket engines. His current work in hydrogen safety codes and standards draws

from his industry experience and includes testing hydrogen safety sensors, testing of high pressure hydrogen storage/fueling components and direct support of codes and standards development through membership on NFPA and SAE committees.

Carl Rivkin, P.E.

Senior Project Leader II, National Renewable Energy Laboratory

Mr. Rivkin is the project leader for the Codes and Standards Project team at the National Renewable Energy Laboratory (NREL) in Golden, CO. The Codes and Standards Project at NREL has responsibility for implementing the U.S. National Template of Codes and Standards for alternative fuels for vehicles. This implementation effort runs the gamut from running a sensor test laboratory to evaluate the performance of chemical sensors to conducting codes and standards seminars to assist code officials in permitting facilities. Mr. Rivkin has over twenty five years of experience in safety and environmental engineering including work at a regulatory agency. Prior to joining NREL, Mr. Rivkin worked for the National Fire Protection Association on alternative energy code projects. Mr. Rivkin was also the editor of *The NFPA Guide to Gas Safety*, published in 2005, which has several chapters devoted to hydrogen and flammable gas safety. Mr. Rivkin has a bachelor's degree in chemical engineering from the University of Michigan and an MBA from the University of Baltimore. He is a licensed Professional Engineer (P.E.) and Certified Safety Professional (CSP).

Matthew Post

Senior Research Technician, National Renewable Energy Laboratory

Matt Post was hired at NREL in April of 2008. His main project involves providing specialized expertise on process and instrumentation for the Hydrogen Sensor Testing Laboratory. This work involves design and construction of systems, instrument setup and computer programming. As well as the technical aspect of his work, he also provides input on experiment design and is involved in sensor evaluation. He also contributes technical assistance for many projects throughout the Hydrogen Technologies and Systems Center. Prior to starting at NREL, he worked as a Research Associate at Eltron Research and Development Inc in Boulder, CO from 2001 to 2008. While at Eltron, he provided technical work and analytical input on projects for which the lab received funding. Projects that he worked on were supported both by private industry and by government grants including DOE, DOD, NASA and USDA. Before Eltron, he was a chemist at AAT Labs Inc in Grand Rapids, MI from 1998 to 2001. While at AAT Labs, he coordinated sample procurement with clients, performed wet chemistry analysis, and assisted on special projects. He graduated in 1999 from Calvin College in Grand Rapids, MI with a B.S. in Environmental Science – Chemistry Emphasis.

Lois Brett, Ph.D.

Scientific Officer, European Commission DG-JRC, Institute for Energy

Lois Brett obtained her PhD in Combustion Chemistry from the National University of Ireland, Galway in 1999. The topic of her thesis was “Re-commissioning of a Rapid Compression Machine and Computer Modeling of Hydrogen and Methane Autoignition.” Having worked 2 years as a publishing officer at the International Flame Research Foundation she initially joined the JRC Institute for Energy in 2002 as a postdoctoral

fellow. At this time she was involved in testing of high pressure hydrogen storage systems and subsequently designing a hydrogen safety sensor testing facility. Following this she joined the Rocket Technology Group of the Dutch National Research Organization for a period of over 2 years to investigate the application of their solid gas generator technology used in space applications to irreversibly store hydrogen. Lois rejoined the JRC in April 2007 to continue the work on hydrogen safety sensor performance testing.

Grainne Black, Ph.D.

European Commission DG-JRC, Institute for Energy

Grainne Black gained an MSc in Science Communication in 2004 from Dublin City University, Ireland. This was followed by a PhD in 2009 from the National University of Ireland, Galway, entitled “The Combustion Chemistry of Oxygenates: A Computational, Modelling and Experimental Study.” Part of this work was carried out at the CNRS laboratory in Orleans, France. As of the start of 2009, she has been working in the Institute for Energy of the JRC carrying out test method development and performance testing of hydrogen safety sensors. She is the author and co-author of 7 scientific publications.

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1. REPORT DATE (DD-MM-YYYY) October 2010		2. REPORT TYPE Conference Paper		3. DATES COVERED (From - To)		
4. TITLE AND SUBTITLE Round Robin Testing of Commercial Hydrogen Sensor Performance-Observations and Results			5a. CONTRACT NUMBER DE-AC36-08GO28308			
			5b. GRANT NUMBER			
			5c. PROGRAM ELEMENT NUMBER			
6. AUTHOR(S) W. Buttner, R. Burgess, C. Rivkin, and M. B. Post (NREL) L. Boon-Bret, G. Black, F. Harkshamp, and P. Moretto (European Commission, DG Joint Research Centre)			5d. PROJECT NUMBER NREL/CP-5600-48305			
			5e. TASK NUMBER FC087750			
			5f. WORK UNIT NUMBER			
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) National Renewable Energy Laboratory 1617 Cole Blvd. Golden, CO 80401-3393				8. PERFORMING ORGANIZATION REPORT NUMBER NREL/CP-5600-48305		
9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)				10. SPONSOR/MONITOR'S ACRONYM(S) NREL		
				11. SPONSORING/MONITORING AGENCY REPORT NUMBER		
12. DISTRIBUTION AVAILABILITY STATEMENT National Technical Information Service U.S. Department of Commerce 5285 Port Royal Road Springfield, VA 22161						
13. SUPPLEMENTARY NOTES						
14. ABSTRACT (Maximum 200 Words) This paper presented observations and results from round robin testing of commercial hydrogen sensor performance.						
15. SUBJECT TERMS hydrogen sensors; sensor testing; round robin sensor testing						
16. SECURITY CLASSIFICATION OF:			17. LIMITATION OF ABSTRACT UL	18. NUMBER OF PAGES	19a. NAME OF RESPONSIBLE PERSON	
a. REPORT Unclassified	b. ABSTRACT Unclassified	c. THIS PAGE Unclassified			19b. TELEPHONE NUMBER (Include area code)	

Standard Form 298 (Rev. 8/98)
Prescribed by ANSI Std. Z39.18