



Passive Leak Detection Using Commercial Hydrogen Colorimetric Indicator

Kevin Hartmann, William Buttner,
Robert Burgess, and Carl Rivkin

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September 2016

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List of Acronyms

atm	atmosphere
dp	dew point
ESIF	Energy Systems Integration Facility
H ₂	hydrogen
H ₂ O	water
NREL	National Renewable Energy Laboratory
ppm _v	parts per million by volume
PRD	pressure release device
RH	relative humidity
T	temperature
vol%	percent by volume

Executive Summary

Element One, Inc., a small business in Boulder, Colorado, has been developing hydrogen detection technology based upon a highly selective colorimetric indicator. The indicator pigment is initially pale gray in color, but becomes black upon exposure to hydrogen, a color transition that is readily observable by the naked eye. Recently, the colorimetric indicator was integrated into a pliable, self-adhesive tape that can be securely stretch-wrapped around pneumatic fittings to serve as a hydrogen leak detector. A prototype version of the indicator tape was tested in an operational National Renewable Energy Laboratory (NREL) hydrogen system and successfully identified the unexpected presence of a small leak. The tape has subsequently been configured into 15-foot rolls as a product prototype and named DetecTape®. The NREL Sensor Laboratory deployed DetecTape and evaluated its performance at several NREL hydrogen facilities, including outdoor operations associated with the NREL Hydrogen Fueling Station and several indoor operations. The investigation also involved evaluation of the indicator under controlled laboratory conditions. This work was performed under the auspices of Memorandum of Understanding Number-14-334 between NREL and Element One and with support from the U.S. Department of Energy Fuel Cell Technologies Office, Hydrogen Safety, Codes, and Standards Program, and with support from the NREL Commercialization Assistance Program.

In the course of the initial deployment study, over 50 pneumatic components were fitted with the indicator, encompassing over 60 individual DetecTape deployments. DetecTape identified numerous leaks that would have otherwise gone undetected. Nine clear indications were observed, identifying the existence and precise location of a hydrogen release. Although the majority of the identified leaks were ultimately identified as “normal,” three indications were identified as “out-of-normal,” and thus required corrective maintenance. The out-of-normal indications guided maintenance procedures, which ranged from re-torqueing the offending fitting on the pneumatic system to rebuilding or replacing suspect components. Although the identified out-of-normal leaks were small and did not pose a hazard, they were not supposed to be present. The ability of DetecTape to identify unexpected hydrogen releases was demonstrated in the deployment study. The identification of such leaks facilitated implementation of appropriate corrective action before the potential occurrence of major adverse events. DetecTape was also shown to be robust and immune to environmental extremes as evidenced by its survival in the outdoor deployments with significant weather extremes as experienced in Golden, Colorado, from July 2015 through May 2016. No unequivocal false indications were observed.

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Background

The Fuel Cell Technologies Office [1] in the U.S. Department of Energy Office of Energy Efficiency and Renewable Energy [2] is committed to the development of hydrogen as an energy carrier. The Fuel Cell Technologies Office supports programs facilitating the implementation of fuel cell technology in the U.S. marketplace. Hydrogen and fuel cell safety is under the auspices of the Hydrogen Safety, Codes, and Standards Program [3]. The mission of the Safety, Codes, and Standards Program is to assure safe implementation of hydrogen infrastructure through the development of national hydrogen codes and standards harmonized to international standards, outreach programs designed to educate stakeholders on the regulations and best practices for the safe handling of hydrogen, and technical programs that directly support hydrogen safety. One element of a hydrogen safety system is the use of sensors to detect unexpected releases. To assure the availability of hydrogen sensors, the NREL Hydrogen Sensor Test Laboratory [4] was established in 2008 to provide stakeholders (e.g., standard development organizations/code development organizations, sensor developers and manufacturers, end users, and code enforcement officials) with a resource for the independent, unbiased assessment of hydrogen sensors and guidance on their use. A main focus of the sensor laboratory is to assess the ability of commercial and developing sensor technologies to meet critical performance metrics as defined by national [5] and international [6] standards, as well as critical performance metrics as identified by Department of Energy [7]. The NREL Sensor Laboratory program encompasses the full range of issues related to safety sensors, including the development of advanced sensor platforms through collaboration with sensor manufacturers and research institutions, development and implementation of sensor-related code and standards, and outreach to stakeholders to facilitate deployment. The unbiased testing of sensor technology for both sensor providers and end users is a core mission of the NREL Sensor Laboratory.

Element One, Inc. (www.elem1.com), a small business in Boulder, Colorado, has been developing hydrogen detection technology based upon a highly selective colorimetric indicator (U.S. Patent 6,895,805) [8]. In its native state, the indicator pigment is a pale gray color, but becomes black upon exposure to hydrogen. The colorimetric change can be readily observed by the naked eye without the need for supplemental electronics or other hardware. Recently, the colorimetric indicator was integrated into a pliable, self-adhesive tape that can readily wrap around pneumatic fittings to serve as a hydrogen leak detector (Patent pending) [9]. A prototype version of the Element One indicator tape was tested in an NREL hydrogen system and successfully identified the unexpected presence of a small leak. A summary document for this case study is presented in Appendix A. The tape was subsequently configured into 15-foot rolls as a product prototype that has just recently been commercialized and marketed under the trade name DetecTape.¹ Figure 1 shows the commercial version of DetecTape along with an indicator sample in its native state and one that had been exposed to hydrogen. DetecTape is a self-fusing, silicone-based tape impregnated with a proprietary hydrogen-sensitive indicator. A length of the tape can be cut from the roll and stretched by a factor of two or three times around a fitting. Due to the self-adhesive property of the tape, this provides a tight seal around the fitting. The seal is not hermetic and is not intended to prevent the release of a leaking gas. However, a portion of the hydrogen leaking from a wrapped fitting will pass through the tape and react with the active indicator impregnated in the tape, thereby inducing blackening.

¹ DetecTape is a registered trademark of Element One, Inc.

Under the auspices of a Memorandum of Understanding with Element One, Inc. [10] and with support from the NREL Commercialization Assistance Program [11], the NREL Sensor Laboratory evaluated the effectiveness of DetecTape as a hydrogen leak detector. The evaluations included both laboratory testing and field deployments on several active NREL hydrogen operations. This report covers the results of the NREL evaluation of DetecTape, including field deployments from July 2015 to May 2016. Both indoor and outdoor hydrogen operations were included in the investigation.

The term *indication* will be used whenever a colorimetric change was observed (i.e., the tape *indicated* a hydrogen leak). All indicator inspections, including both laboratory testing and field deployments, were done visually. Because an indication was accompanied by an obvious color change, no verification method other than visual inspection was necessary.



Figure 1. The commercial version of DetecTape. When exposed to hydrogen, the indicator transforms from a pale gray color to black. The color change is not reversible.

Photo by Kevin Hartmann, NREL

Methods

Laboratory Assessments

Verification of Indicator Functionality

Laboratory testing of DetecTape was performed to demonstrate basic functionality, i.e., to verify the transformation from a pale gray color to black upon exposure to hydrogen. Functionality testing was first performed on new indicators to verify that the laboratory protocol was valid. Subsequently, the functionality test was then performed on indicators that had been subjected to various pretreatments, including environmental stresses in a controlled laboratory environment chamber and following actual deployments on active hydrogen systems. Functionality was demonstrated by placing samples of the indicator inside a transparent, sealed hydrogen exposure chamber (Figure 2) and subjecting the samples to a multi-step exposure sequence. Although specific details (e.g., exposure time, flow rate) varied from test to test, the basic exposure sequence consisted of the following steps:

1. Purge the hydrogen exposure chamber with air at 2 standard liters per minute for 30 to 45 minutes.

2. Purge the hydrogen exposure chamber with 5.7 percent by volume (vol%) hydrogen (H₂) in nitrogen at 2 standard liters per minute for 1 hour
3. Continue the hydrogen exposure under static (no flow) conditions for up to 2 hours.
4. Purge the hydrogen exposure chamber with air at 2 standard liters per minute for 30 to 45 minutes.

Following the prescribed hydrogen exposure, the indicators were removed from the test chamber and visually inspected to verify their sensitivity to hydrogen. A color change was readily observed when active indicators were subjected to this exposure sequence. Digital photographs were also taken of the indicator to record the impact of hydrogen exposure. Typically, the color change could be observed within 45 minutes following initiation of the hydrogen exposure by visual inspection through the transparent walls of the exposure chamber.

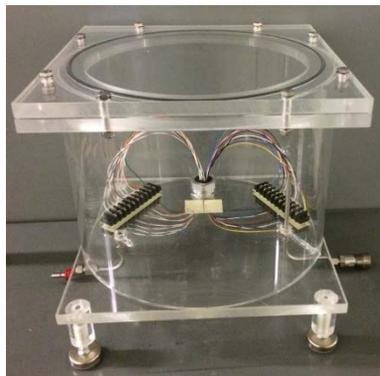


Figure 2. Hydrogen Exposure Chamber.
(The electrical feedthroughs were not needed for DetecTape.)

Photo by Kevin Hartmann, NREL

It is noted that this test protocol was used to verify that the indicator would show a visible color change when exposed to hydrogen. The test was not intended to be either a sensitivity/detection limit study or a kinetic study. The 5.7 vol% H₂ in nitrogen test gas is nonflammable and was used in the laboratory study for safety purposes. In another test using 10 vol% H₂ in nitrogen, a color change was observable but not fully developed within 10 minutes of the hydrogen exposure. Gas leaking from hydrogen pneumatic systems would be essentially pure hydrogen, which would react much faster on the indicator because the kinetics of the indicator reaction is dependent on the concentration of hydrogen.

Impact of Environmental Stresses on Indicator Functionality

Functionality testing was performed on new indicators and on indicators that were subjected to various pretreatments. Pretreatments included subjecting the indicator to the Environmental Stress Test protocol illustrated in Figure 3 and summarized in Table 1. The Environmental Stress Test was performed to elucidate the impact of short-term temperature and humidity fluctuations on the stability of the indicators. This testing consisted of three segments: temperature cycling, humidity cycling, and elevated temperature and humidity exposure. Indicator test samples (TS) were removed following each sequence and checked for activity. Humidity is plotted as the dew point (dp) with the corresponding parts per million volume (ppm_v) water vapor (H₂O), and

relative humidity (RH) labeled in the plot. Dew point and ppmv H₂O were calculated from the measured relative humidity (RH) and temperature (T) using an on-line conversion tool [12].

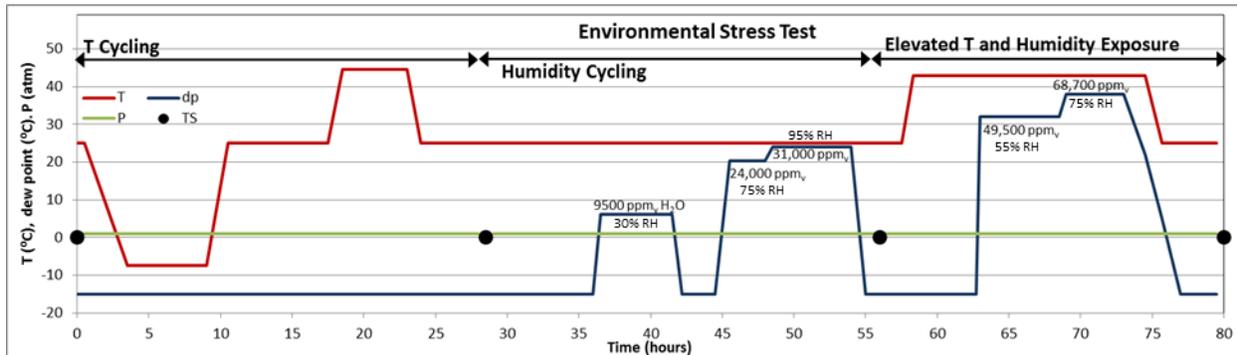


Figure 3. Illustration of the environmental stress test performed on DetecTape using environmental control features available in the NREL Sensor Test Apparatus [4].

Multiple indicator samples were installed in the NREL Sensor Test Apparatus [4], which served as an Environmental Test Chamber. As a control, two indicators were not subjected to the temperature or humidity cycling prescribed by the Environmental Stress Test (Samples 2 and 3) before being subjected to the functionality test. The remaining indicator samples were placed inside the Environmental Test Chamber and subjected to the temperature cycling test, which exposed the indicators to temperatures ranging from approximately -7°C to $+45^{\circ}\text{C}$ at a pressure of 1 atm and a dry RH corresponding to a dew point less than -15°C . Following the temperature cycling test, two indicators (Samples 3 and 4) were removed from the Environmental Test Chamber and transferred to the Hydrogen Exposure Chamber and subjected to the functionality test described above. The indicators remaining in the Environmental Test Chamber were then subjected to the humidity cycling test, which ranged from $<1\%$ RH ($dp < -15^{\circ}\text{C}$) up to approximately 95% RH ($dp = 24.1^{\circ}\text{C}$) at a temperature of 25°C and a pressure of 1 atm. Following the humidity cycling test, two indicators (Samples 6 and 7) were removed from the Environmental Test Chamber and transferred to the Hydrogen Exposure Chamber and subjected to the functionality test. The indicators remaining in the Environmental Test Chamber were then subjected to an elevated T and RH exposure test performed at a temperature of 40°C and humidity up to 75% ($dp = 38^{\circ}\text{C}$) and a pressure of 1 atm. Following the elevated temperature and humidity exposure test, the remaining indicators (Samples 8 and 9) were removed from the Environmental Test Chamber, transferred to the Hydrogen Exposure Chamber, and subjected to the functionality test described above. Table 1 summarizes the indicator pretreatments. Photographs of the indicators following respective sequences of the environmental stress test and the functionality test are shown in Figure 4, which clearly show that the functionality of the indicator was not impacted by short-term exposures to the temperature and humidity extremes prescribed by the Environmental Stress Test.

Table 1. Impact of Environmental Cycling on DetecTape Hydrogen Indicators (Laboratory Assessment)

Sample Number	Pretreatment	Hydrogen Exposure	Functionality
Sample 1	Ambient only ^a	None	n.a.
Sample 2 Sample 3	Ambient only	Standard ^b	Active
Sample 4 Sample 5	Ambient, temperature cycling	Standard	Active
Sample 6 Sample 7	Ambient, temperature cycling, and humidity cycling	Standard	Active
Sample 8 Sample 9	Ambient, temperature cycling, humidity cycling, and elevated temperature and humidity cycling	Standard	Active

Pretreatment Performed in the Environmental Test Chamber under controlled T, P, RH.

Hydrogen Exposure performed in separate test fixture at ambient T, P, and 50% RH.

^a Ambient conditions were external to the environmental chamber (approximately 23±2°C, 10 to 20% RH, 0.82 atm).

^b Following the indicated pretreatment, samples were transferred from the Environmental Test Chamber to the Hydrogen Exposure Chamber. Testing consisted of exposing the indicator to 5.7 vol% H₂ in nitrogen.



Figure 4. Images of DetectTape samples that had been exposed to hydrogen following the indicated portion of the Environmental Stress Test. For comparison, an unexposed indicator sample (center top row) is shown. The samples on the right and left are duplicate samples.

Photo by Kevin Hartmann, NREL

DetecTape Deployment in the Open Air

DetecTape will readily respond to hydrogen leaking through pneumatic components and when stored in a closed chamber with a uniformly mixed hydrogen atmosphere (e.g., such as those used in the functionality test). However, it was found that DetecTape is not an efficient detector for hydrogen released into the open air. This can be rationalized by the need for hydrogen to partition from the surrounding air into the layered silicone structure in order to access the active indicator chemical. In an open system, the proclivity of hydrogen to diffuse into the open atmosphere would prevent significant adsorption into the solid substrate. Even when the flow from a hydrogen gas cylinder was directed onto the surface of DetecTape in the open air, no color change was observed. While it is likely that eventually a color change would take place with such direct exposure to hydrogen, the partition into the silicone membrane is a slow process in open air, and the most likely fate of the hydrogen would be to simply diffuse away. This is illustrated in Figure 5. No color change was observed after 10 vol% H₂ in nitrogen was flowed directly onto the DetecTape surface for 10 minutes in an open system (Figure 5A, middle photograph). Conversely, DetecTape will undergo a color change in a closed system that was continuously purged with 10 vol% H₂ in nitrogen for 10 minutes (Figure 5A, bottom photograph). Although not fully developed, the color change was clearly visible. The rationale for this behavior is illustrated in Figure 5B. In an open environment, hydrogen will tend to diffuse away from the tape rather than be adsorbed. In a closed environment, the hydrogen will be uniformly mixed, thus minimizing the driving force for dispersion, and as a result will more readily partition into the silicone substrate to react with the indicator. When used over a leaking fitting, released hydrogen will necessarily diffuse directly into and through the permeable silicone tape. Thus, DetecTape will work well as a leak detector when applied over pneumatic components, but not as an area monitor for unintended hydrogen releases. DetecTape will not readily react to background levels of hydrogen in open air. Verification of its potential as a passive leak detector was demonstrated in the NREL field deployment study, described in the next section.

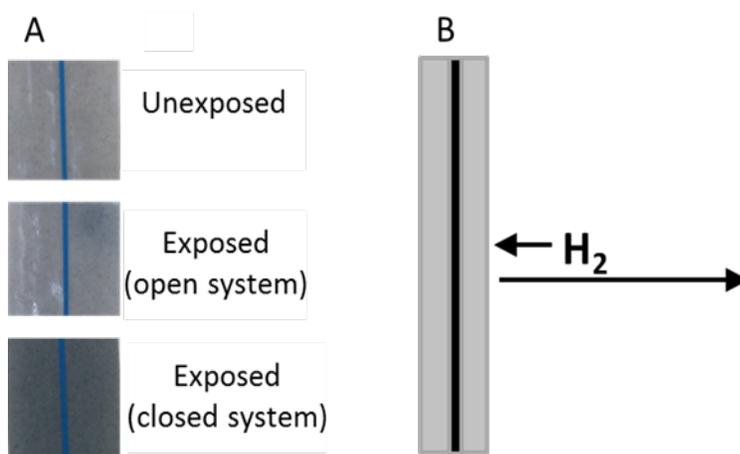


Figure 5. A: Comparison of unexposed and exposed DetecTape under different deployment conditions. B: Illustration of the layered silicone structure of DetecTape and its interaction with hydrogen. The hydrogen will diffuse away from the surface of the tape in an open system.

Field Deployment

Concurrent laboratory testing, the deployment of over 60 samples of DetecTape on various active hydrogen systems in and around the NREL Energy Systems Integration Facility (ESIF) was initiated. The deployment locations included both outdoor sites and indoor facilities. The outdoor sites were mainly associated with the NREL hydrogen fueling station, while the indoor operations included experimental systems within the ESIF high-pressure bays or the indoor water electrolysis system in the Energy Systems Integration Laboratory. The various systems selected for the DetecTape field deployment study are described below.

Hydrogen Storage Tanks

The NREL onsite hydrogen storage facility consists of three separate hydrogen cylinder storage banks pressurized to 200 bar, 400 bar, and 875 bar. During a cascade fill, hydrogen is drawn from separate storage tanks to better match the pressure of the vehicle tank; this is done to minimize the cost and complexity of pressurization during the fill and is controlled through the Gas Management Panel. The storage tanks and support pneumatic system, shown in Figure 6, are outside and thus exposed to prevailing weather conditions, including rain, snow, and sun, along with the daily and long-term temperature fluctuations.



Figure 6. Support systems for the NREL hydrogen dispenser, including (from left to right) the compressor, the gas management panel, and the hydrogen storage tanks. DetecTape was deployed on pneumatic components for each system.

Photo by Kevin Hartmann, NREL

Gas Management Panel

The Gas Management Panel is the interface between the hydrogen storage tanks and the compressor. During a fill, hydrogen is drawn from the appropriate storage tank as controlled by the Gas Management Panel and Dispenser. The Gas Management Panel, shown in more detail in Figure 7, is enclosed within a gray metal cabinet. Access is through two doors, which open to

expose the Gas Management Panel pneumatic system. The cabinet is fully enclosed, thereby protecting tubing and valves from direct exposure to sun, rain, and snow. The cabinet, however, is not thermally regulated, and thus is subjected to daily and seasonal temperature fluctuations.

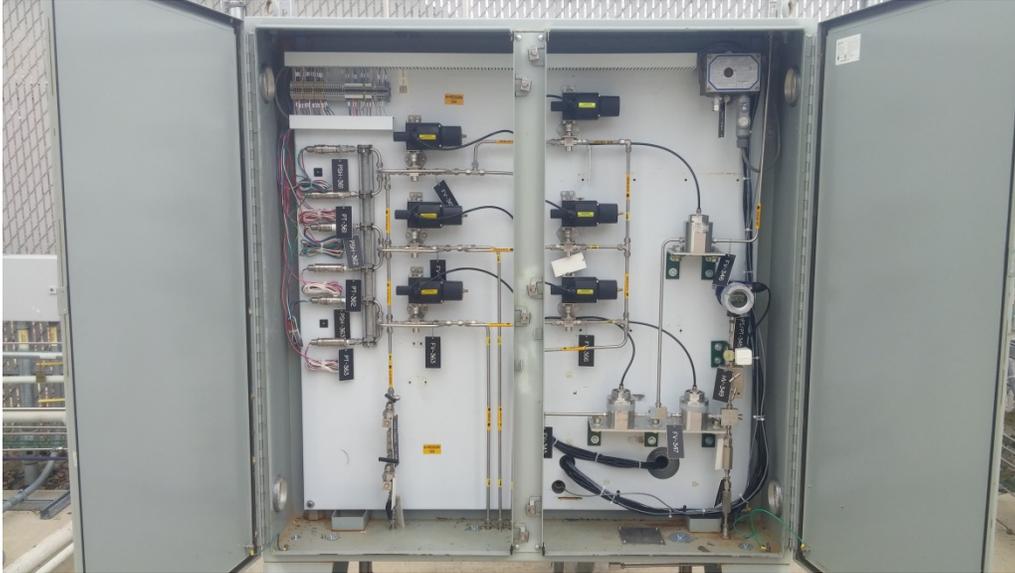


Figure 7. An internal view of the Gas Management Panel. DetecTape was deployed on numerous fittings within the Gas Management Panel.

Photo by Kevin Hartmann, NREL

Fuel Station Compressor

The compressor for the NREL hydrogen fueling station is shown in Figure 8. Components in the compressor cycle between high and low temperatures and pressures. Vibrations are also present when the compressor is running. Vibrations, pressure, and temperature fluctuations can stress the integrity of pneumatic fittings and thus can be prone to induce leaks. Furthermore, as an outdoor system, the compressor is in direct contact with environmental conditions such as rain, snow, and sun, as well as daily and seasonal temperature fluctuations.



Figure 8. Hydrogen fuel station compressor

Photo by Kevin Hartmann, NREL

Dispenser

The dispenser is shown in Figure 9. It consists of a roofless cabinet with two external fueling hoses and internal plumbing. The dispenser is the interface that delivers hydrogen from the storage tank, through the compressor, and into the vehicle. The dispenser transfers gas that is pressurized up to 875 bar and cooled to temperatures as low as -40°C . The tubing and components in the dispenser cabinet would be exposed to precipitation and the sun, as well as daily and seasonal temperature fluctuations.



Figure 9. Fuel station dispenser. The internal pneumatic system is partially visible through the open panel.

Photo by Kevin Hartmann, NREL

Indoor Deployment: Component Testing in the ESIF High Pressure Test Bays

A core capability of ESIF is the High Pressure Test Bay, which supports experiments and testing of components at elevated pressures. Leaks are a critical concern for high-pressure testing of hydrogen components. The High Pressure Test Bay is an enclosed room with moderate environmental controls to maintain the temperature and relative humidity to around 22°C and 10% to 20%, respectively. ESIF has two High Pressure Test Bays available. One test bay was used for reliability testing of a pressure release device (PRD) [13]. DetecTape was placed on pneumatic fittings and valves that were subjected to repeated elevated but controlled pressure variations. The second High Pressure Test Bay was used for the Hose Reliability Test Project [14], which subjected commercial dispenser hoses to repetitive pressurizations to 12,500 psi and thermal swings to -40°C. DetecTape was placed on specific fittings and plastic-metal interfaces that had been identified as potential inconsistent leak locations.

Indoor Deployment: Hydrogen Production in the Energy Systems Integration Laboratory

DetecTape was deployed on two components associated with the electrolysis hydrogen production system operating in the ESIF Energy Systems Integration Laboratory. These included a fitting installed on hydrogen transfer line downstream from the cathode, and near the seal of the water storage tank. As an indoor deployment, these indicators were tested under controlled environmental conditions.

Results of Field Deployments

Deployment was performed by cutting a short length of the tape and “stretch” wrapping it around a pneumatic component; the actual length was determined by the size of the component (e.g., a small standard ¼-inch tube fitting required approximately a 2-inch length of tape, while longer lengths would be required for larger fittings). Typically, the indicator was wrapped two or three times around a fitting or valve to assure proper coverage. Table 2 lists the deployments of DetecTape in this project. The deployments were monitored by periodic visual inspections. Typically, the first inspection was within 24 hours of deployment, followed by weekly or monthly inspections.

Each deployed indicator was assigned a unique identification code that consisted of the date of deployment and a sequential number, as shown in Table 2. Table 2 also includes the location of each individual deployment and whether an indication was observed. Because an indication was accompanied by an obvious color change, no verification method other than visual inspection was necessary, although a sniffer (either the TIF Model TIF8800A Combustible Gas Detector or the Bacharach Model Informant 2 Leak Detector) was sometimes used for verification. In the event of an indication, a replacement sample of DetecTape with a new identifier code was installed on the fitting. Indicators that were decommissioned after over 8 months of deployment are also identified in Table 2. These decommissioned samples were subjected to laboratory analysis (e.g., the functionality test) to verify if they were still active. The results are described in the following section.

Several hydrogen releases were identified by DetecTape during the field deployments. In all cases, the fitting was inspected, and, if warranted, subjected to corrective maintenance, which ranged from adjusting the mounting torque to rebuilding or replacing failed components. Following the necessary maintenance, a new indicator would be applied over the component. During the deployment study, nine clear indications were seen and verified as hydrogen releases. Not all of the releases were identified as a “leak” (e.g., a hydrogen release arising from an out-of-normal situation). In some cases, the indication was in fact a response to a normal phenomenon, although this was not always realized at the time the indication was observed. Other times, the indication was clearly in response to an out-of-normal event and represented a real, but usually small leak. In these cases, the leak would be independently verified by a sniffer, and then the fitting would be subjected to maintenance to assure leak-free operation. The sniffer was also used to verify the effectiveness of maintenance procedures protocols performed following the identification of a leak. An overview of the nine indications is presented below.

Indication #1: ID# 2015-07-08-008 – This indication formed as a small, well-defined dot on DetecTape applied over the weep hole of a cone and thread fitting on a valve mounted in the Gas Management Panel. The valve was rebuilt and reinstalled into the Gas Management Panel. A replacement indicator (ID# 2015-11-13-043) was applied to this valve. Figure 10 shows DetecTape mounted on the valve before and after the indication. A function of the weep hole is to allow controlled venting of hydrogen that is released during normal operation of the component, thus this indication represents a normal phenomenon e.g., not a leak.



Figure 10. Indication 1: DetecTape indicator mounted on a valve (ID# 2015-07-08-008) showing before (left photo) and after (right photo) an indication.

Photo by Kevin Hartmann, NREL

Indication #2: ID# 2015-10-16-039 – This was a large and splotchy indication that formed over the weep hole of a cone and thread fitting mounted on the compressor. Figure 11 shows the DetecTape mounted on the valve before and after the indication. This indication was considered out-of-normal, and the leak was verified with a sniffer. The fitting was remounted on the pneumatic line, and a replacement indicator (ID# 2015-11-16-047) was applied to this fitting. The replacement indicator showed some coloration and is described below (Indication # 7), but this second indication was somewhat ambiguous and not clearly indicative of an out-of-normal hydrogen release.



Figure 11. Indication 2: DetecTape mounted on a cone and thread fitting (ID# 2015-10-16-039) before (left photo) and after (right photo) indication.

Photo by Kevin Hartmann, NREL

Indication #3: ID# 2015-10-16-040 – This indication formed over the weep hole of a check valve with a cone and thread fitting mounted on the compressor. This fitting and indication are shown in Figure 12. This indication was considered out-of-normal and was verified with a sniffer. The fitting was re-torqued, and a replacement indicator (ID# 2015-11-16-048) was applied to this fitting. The replacement indicator showed some coloration and is described below (Indication # 8), but this second indication was somewhat ambiguous and not clearly indicative of an out-of-normal hydrogen release.



Figure 12. Indication 3: DetecTape mounted over the weep hole of a check valve with a cone and thread fitting on the compressor at the NREL hydrogen dispenser (ID# 2015-10-16-040), showing before (left photo) and after (right photo) the indication.

Photo by Kevin Hartmann, NREL

Indication #4: ID# 2015-11-13-043 – This indicator replaced indicator ID# 2015-07-08-008 (Indication #1). Although the two indications observed on this valve were ultimately considered to be normal, the valve was rebuilt following the first indication but prior to the application of the replacement indicator. The second indication formed again as a small confined dot over the weep hole of a cone and thread fitting on a valve located in the Gas Management Panel, as shown in Figure 13.



Figure 13. Indication 4: Indication formed over a weep hole of a cone and thread fitting (ID# 2015-11-13-043). This was ultimately determined to be a normal occurrence.

Photo by Kevin Hartmann, NREL

Indication #5: ID# 2015-11-16-045 – This indication was formed as a small confined dot over the weep hole of a cone and thread fitting on a valve installed near the fuel storage tanks. This indication was considered to be normal. In this case, the indicator was left in place, and the valve was not rebuilt or re-torqued. This indication is shown in Figure 14.



Figure 14. Indication 5: Before (left photo) and after (right photo) the indication (ID# 2015-11-16-045). This was considered to be a normal event.

Photo by Kevin Hartmann, NREL

Indication #6: ID# 2015-11-16-046 – This was a large and splotchy indication over the weep hole of a cone and thread fitting on a valve near the fuel storage tanks. Compared to normal indications on other fittings observed in this study, this indication was much larger, spreading out over the DetecTape. This indication clearly represented an out-of-normal hydrogen release that required the valve to be rebuilt. This indication is shown in Figure 15. Following valve maintenance, indicator ID# 2015-12-04-051 was applied to this fitting. No further out-of-normal indications were observed for this specific fitting.



Figure 15. Indication 6: Indication formed on a valve (ID# 2015-11-16-046). This was an out-of-normal occurrence caused by a valve failure, resulting in maintenance of the valve.

Photo by Kevin Hartmann, NREL

Indication #7: ID# 2015-11-16-047 – This indicator replaced ID# 2015-10-16-039 (Indication #2). This indication was observed over the weep hole of a cone and thread fitting mounted on the compressor (Figure 16). Unlike the normal releases associated with other cone and thread fittings (e.g., Indication #5, Figure 14), it could not be unequivocally determined that this indication arose from a normal release or from a small but out-of-normal event. Because the color change was not as obvious as indications associated with out-of-normal releases (e.g., Indication #3, Figure 12), this indicator will continue to be monitored to determine if it transforms into an unambiguous indication and then it will be ascertained whether it is a normal or out-of-normal indication.

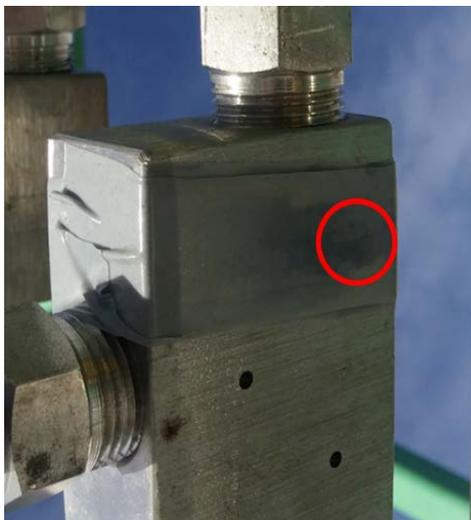


Figure 16. Indication 7: Slight coloration of the indicator (ID# 2015-11-16-047). This was the second indication observed on this fitting (Indication 2, corresponding to ID# 2015-10-16-039 shown in Figure 11). This was viewed as an ambiguous indication requiring continued monitoring to differentiate between normal and out-of-normal hydrogen releases.

Photo by Kevin Hartmann, NREL

Indication #8: ID# 2015-11-16-048 – This indicator replaced ID# 2015-10-16-040 (Indication #3). The possible indication was seen as the initiation of a slight darkening of the pigment over the weep hole of a check valve with a cone and thread fitting mounted in the compressor (Figure 17). Although more pronounced than the indication associated with the weep hole on other cone and thread fittings (e.g., Indication # 5, Figure 14), the color change was not as obvious as other out-of-normal indications. This indicator will continue to be monitored to determine if it transforms into an unambiguous indication and then it will be ascertained whether it is a normal or out-of-normal indication.



Figure 17. Indication 8: Slight coloration of the indicator (ID# 2015-11-16-048). This was the second indication observed on this fitting (Indication 3, corresponding to ID# 2015-10-16-040, shown in Figure 12). This is an ambiguous indication requiring further monitoring to verify if it is a normal or out-of-normal hydrogen release.

Photo by Kevin Hartmann, NREL

Indication #9: ID# 2015-11-16-050 –This indication was formed as a small confined dot on the weep hole of a cone and thread fitting on a valve in the high pressure test bay (PRD testing). This indication is considered to be normal; therefore, the indicator was left in place, and the valve was not rebuilt or re-torqued. This indication is shown in Figure 18.



Figure 18. Indication 9: DetecTape mounted on a valve installed on a hydrogen system in the ESIF High Pressure Test Bay, showing before (left) and after (right) a normal indication (ID# 2015-11-16-050).

Photo by Kevin Hartmann, NREL

A total of nine indications were observed. Of these, Indications 1, 4, 5, and 9 were considered normal.² These were seen from weep holes of cone and thread fittings (e.g., Figure 12). Most of the valve indications were due to very small releases of hydrogen seen as black dots on the indicator tape mounted over the weep holes. The hydrogen releases through the weep hole were miniscule and normal for this valve and did not pose a risk. In one case, however, the DetecTape clearly identified a hydrogen release on a valve that was out-of-normal (Indication #6). For this valve, the indication was much larger than those observed for a low-level release deemed acceptable (normal) for a cone and thread fitting weep hole. Overall, there were three clear “out-of-normal” indications (2, 3, and 6). There were two additional indications (Indications 7 and 8) that are probably associated with normal hydrogen releases that are being monitored to verify this assertion.

Stability of DetecTape

The first indicator was deployed within this project on July 8, 2015, and additional samples were deployed over the duration of the project. The initial deployments remained in place until April 2016, when some of the initially deployed indicators were removed to verify functionality; this was done by subjecting the decommissioned samples to the Functionality Test. In outdoor deployments, the indicators were subjected to variable weather conditions, including dry, hot, sunny weather; extreme cold; rain; and snow. Table 2 identifies several indicators that were decommissioned (removed from deployment) to allow for laboratory testing. For each decommissioned indicator, a new indicator was applied over the fitting and assigned a new identification number, which are also listed in Table 2. The decommissioned indicators were all still active, as verified by the Functionality Test. Figure 19 shows a comparison of a decommissioned sample exposed to hydrogen (ID# 2015-07-08-013) to an unexposed sample (ID# 2015-07-08-004). Both samples were subjected to deployments of comparable duration and environmental conditions. The color change of a fresh sample is included in Figure 19. Thus, extended deployment and exposure to environmental extremes associated with the outdoor environment had no apparent adverse impact on functionality.

² Indications 1 and 4 were from the same fitting. Originally Indication #1 was viewed as out-of-normal, but was subsequently identified as being associated with a normal hydrogen release.

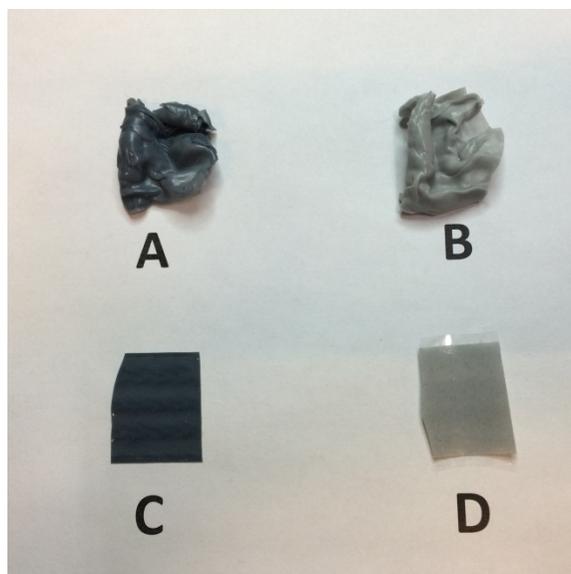


Figure 19: DetecTape samples deployed on the Gas Management Panel of the NREL Hydrogen Dispenser (A and B) compared to fresh samples (C and D). The deployment was from July 2015 until April 1, 2016. Samples A and C were exposed to 5.7 vol% H₂ in nitrogen under laboratory conditions, while Samples B and D are shown prior to laboratory hydrogen exposures. Sample A corresponds to deployment number 2015-07-08-013, and B corresponds to deployment number 2015-07-08-004.

Photo by Kevin Hartmann, NREL

Table 2. DetecTape Deployments

Date and ID#	Location	Roll	Date of Indication	Supplemental Actions	ID# for Replacement Indicator
2015-07-08-001	Electrolyzer Stack Test Bed	1			
2015-07-08-002	DI Water Tank	1			
2015-07-08-003	Gas Management Panel	1			
2015-07-08-004	Gas Management Panel	1		Removed for lab testing	2016-04-01-061
2015-07-08-005	Gas Management Panel	1			
2015-07-08-006	Gas Management Panel	1			
2015-07-08-007	Gas Management Panel	1			
2015-07-08-008	Gas Management Panel	1	7/22/2015		2015-11-13-043

Date and ID#	Location	Roll	Date of Indication	Supplemental Actions	ID# for Replacement Indicator
2015-07-08-009	Gas Management Panel	1			
2015-07-08-010	Gas Management Panel	1			
2015-07-08-011	Gas Management Panel	1			
2015-07-08-012	Gas Management Panel	1		Removed for lab testing	2016-03-28-056
2015-07-08-013	Gas Management Panel	1		Removed for lab testing	2016-03-28-057
2015-07-08-014	Valve Test Stand	1		Removed	
2015-07-16-015	Fuel Station Compressor	1			
2015-07-16-016	Fuel Station Compressor	2			
2015-07-16-017	Fuel Station Compressor	2			
2015-07-16-018	Fuel Station Compressor	2		Removed for lab testing	2016-03-28-058
2015-07-16-019	Fuel Station Compressor	2		Removed for lab testing	2016-03-28-059
2015-07-16-020	Fuel Station Compressor	2		Removed for lab testing	2016-03-28-060
2015-07-16-021	Fuel Station Compressor	2		Removed for lab testing	2016-04-01-062
2015-07-16-022	Fuel Station Compressor	2			
2015-07-17-023	High Pressure Bay (PRD)	2			
2015-07-17-024	High Pressure Bay (PRD)	2			
2015-07-17-025	High Pressure Bay (PRD)	2			
2015-07-17-026	High Pressure Bay (PRD)	2			
2015-07-17-027	High Pressure Bay (PRD)	2			
2015-07-17-028	High Pressure Bay (PRD)	2			

Date and ID#	Location	Roll	Date of Indication	Supplemental Actions	ID# for Replacement Indicator
2015-07-17-029	High Pressure Bay (PRD)	2			
2015-07-17-030	High Pressure Bay (PRD)	2			
2015-10-12-031	Dispenser	3			
2015-10-12-032	Dispenser	3			
2015-10-12-033	Dispenser	3			
2015-10-12-034	Dispenser	3			
2015-10-12-035	Dispenser	3			
2015-10-12-036	Dispenser	3			
2015-10-12-037	Dispenser	3			
2015-10-12-038	Dispenser	3			
2015-10-16-039	Fuel Station Compressor	3	11/16/2015		2015-11-16-047
2015-10-16-040	Fuel Station Compressor	3	11/16/2015		2015-11-16-048
2015-11-13-041	Fuel Station Compressor	3			
2015-11-13-042	Fuel Station Compressor	3			
2015-11-13-043	Gas Management Panel	3	11/23/2015		
2015-11-16-044	Gas Management Panel	3			
2015-11-16-045	Storage Tanks	3	12/4/2015		
2015-11-16-046	Storage Tanks	3	12/4/2015		2015-12-4-051
2015-11-16-047	Fuel Station Compressor	3	12/4/2015		
2015-11-16-048	Fuel Station Compressor	3	12/4/2015		

Date and ID#	Location	Roll	Date of Indication	Supplemental Actions	ID# for Replacement Indicator
2015-11-16-049	High Pressure Test Bay (hose test)	4			
2015-11-16-050	High Pressure Test Bay (hose test)	4	11/23/2015		
2015-12-4-051	Storage Tanks	4			
2015-12-4-052	Gas Management Panel	4			
2015-12-4-053	Gas Management Panel	4			
2015-12-4-054	High Pressure Test Bay	4			
2015-12-4-055	High Pressure Test Bay	4			
2016-03-28-056	Gas Management Panel	5			
2016-03-28-057	Gas Management Panel	5			
2016-03-28-058	Fuel Station Compressor	5			
2016-03-28-059	Fuel Station Compressor	5			
2016-03-28-060	Fuel Station Compressor	5			
2016-04-01-061	Gas Management Panel	5			
2016-04-01-062	Fuel Station Compressor	5			

Summary

DetecTape was designed to be deployed directly on individual components of a hydrogen pneumatic system to identify leaks and other release sources. DetecTape is a sophisticated system consisting of a proprietary hydrogen-selective chemo-chromic chemical agent embedded in a silicone tape. However, from the end-user perspective, DetecTape is a low-cost and apparent “low-tech” hydrogen detection technology. Deployment consists simply of stretch-wrapping an appropriate length around specific fittings, coupled with periodic visual inspections. Blackening of the indicator is a direct verification of a hydrogen release.

To verify the functionality and reliability of DetecTape as a hydrogen leak detector, the NREL sensor laboratory initiated a case study involving both laboratory assessments and actual deployments. Between July 2015 and May 2016, over 60 individual deployments of DetecTape were implemented on various NREL hydrogen systems, including both indoor and outdoor operations. Nine locations of hydrogen releases were identified. In all cases, the releases were small and did not pose a safety hazard. In some cases, a hydrogen sniffer verified the hydrogen release, but the sniffer had to be placed directly at the suspect hydrogen release point (dispersion precluded hydrogen detection more than a few inches from the source). Of the nine indications, three were identified as “out-of-normal” (Indication #2, 3, and 6). The identification of these releases guided implementation of maintenance procedures that ranged from adjustment of the mounting torque to rebuilding the suspect component. Four indications were identified (or ultimately identified) as normal (Indication #1, 4, 5, and 9). These were all indications of hydrogen releases through a weep hole and manifested as a small, well-defined black spot on the indicator tape. Two indications (Indication #7 and 8), corresponded to a weep hole release that could be a normal release, but the coloring was more dispersed than that observed for the unambiguous normal releases. These indicators will be monitored to determine if the color change will grow, suggesting an “out-of-normal” release, or will remain unchanged, suggesting that the hydrogen release is normal. Throughout the course of the study, there was no evidence of a false indication.

In the NREL case study, the ability of DetecTape to identify unexpected hydrogen releases was demonstrated. The identification of such leaks facilitated implementation of appropriate corrective action before the potential occurrence of major adverse events. We found that DetecTape to be robust and immune to environmental extremes as evidenced by its survival in the outdoor deployments with significant weather extremes as experienced in Golden, Colorado, for July through May.

However, it is important to note that DetecTape has limitations:

- DetecTape is nonresponsive in an open environment, and thus is not an effective area monitor for hydrogen. However, Element One, Inc. has developed other sensor configurations based on the active chemical agent that does respond to hydrogen in an open environment [15].
- DetecTape will respond to any hydrogen release and will not differentiate between normal and out-of-normal release events. However, it was observed that a normal release usually manifests as a small, well-defined indication while the out-of-normal releases were large and broadly distributed throughout the DetecTape. The end user should be

familiar with the distinction between normal releases and out-of-normal behavior for his system.

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Appendix A: Case Study

Identification of a Small Hydrogen Leak in an NREL Existing Hydrogen Operation Using a Colorimetric Hydrogen Indicator



Colorimetric Hydrogen Indicator Tape for Leak Detection¹

Case Study: Identification of a small leak in an existing NREL hydrogen operation

Background

Element One has been developing cost effective, reliable visual indicators for hydrogen gas with particular interest in fuel cell and hydrogen energy applications. Due to the safety concerns with hydrogen leaks associated with pneumatic lines, the indicator has been configured into a tape compatible with wrapping around fittings, seals, valves and other pneumatic components. A prototype indicator tape sample is shown in Figure 1, but through a partnership with MSP, Inc. (Berlin, CT), the indicator tape has been produced in prototype 10 foot length rolls. The tape, based upon a hydrogen-permeable, self-fusing silicone tape, will be available soon.



Figure 1: The Element One H₂ Indicator impregnated into a 6-inch strip of a self-fusing silicone. 10 foot rolls are now available. The strips are available with a yellow or clear background.

Deployment Study

Under an MOU with Element One, the NREL sensor laboratory deployed samples of the prototype tape with the irreversible form of indicator at multiple locations within an operational H₂ test fixture. A discoloration of the indicator was observed, but at only one deployment location, shown in Figure 2. It is to be noted that the leak was small and did not pose a safety hazard (there is an operational hydrogen sensor for safety monitoring within the system). Although previously undetected, subsequent inspection with a sensitive electronic H₂ leak detector confirmed the existence of the leak. The fitting was replaced and the system remains operational.



Figure 2: The Element One H₂ indicator deployed in a hydrogen test fixture. Left: Freshly deployed. Center: After approximately 1 week (note dark color). Right: Indicator removed, clearly showing discoloration.

Future Work

Deployment of the indicators is on-going and will be expanded to large-scale NREL hydrogen operations (e.g., the Fueling Station) to serve as a tool to verify integrity of the pneumatic lines. On-going research within Element One is working to optimize the performance of the indicator strips and address the following focus areas:

- Are there any effects of the indicator pigment on the silicone curing process?
- Will the pigment decrease the long term shelf-life of the product?
- What is the optimum pigment loading for cost and performance?
- What is the optimum thickness of the tape to maintain good permeability to hydrogen while preserving its mechanical strength?
- What sensitivity (H₂ exposure) is optimum for typical applications? Indoors? Outdoors?

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¹Developed in part through DOE support under NREL Subcontract LXH 4 42308 (*Low Cost Visual and Resistive Sensors*), and evaluated by the NREL Sensor Laboratory under the auspices of MOU Number: NREL-05-097