



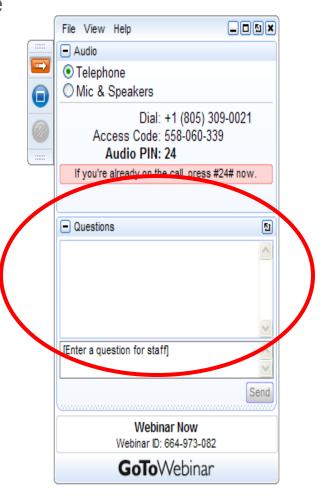
Presenter:

Bill Hoagland – Element One John Poplawski – Midsun Specialty Products

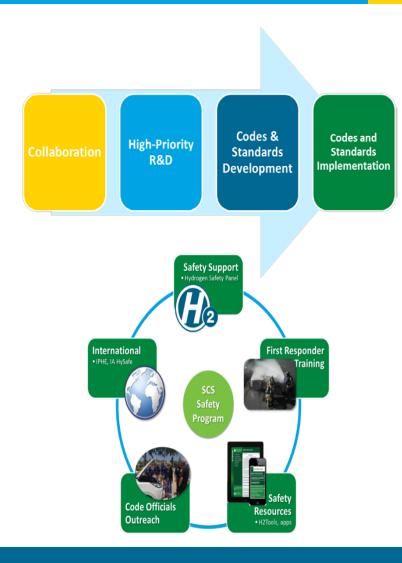
#### DOE Host:

Will James – Safety, Codes, and Standards Program Manager

U.S. Department of Energy Fuel Cell Technologies Office March 14<sup>th</sup>, 2016 Please type your questions into the question box



2



#### **Emphasis**

- R&D Activities: H<sub>2</sub> Behavior, Risk Assessment/Mitigation, Materials Compatibility, H<sub>2</sub> Fuel Quality, Metering, Sensors, Component Testing
- Safety Management & Resources: Hydrogen Safety Panel, Databases, and Training Props
- Outreach: Codes & Standards and Permitting, Continuous Codes and Standards Improvement, Resource Dissemination

Enable the widespread commercialization of hydrogen and fuel cell technologies through the timely development of codes and standards and dissemination of safety information

#### **NREL Sensor Testing Laboratory**



- Provide independent assessment of hydrogen sensor performance
- Interact with manufacturers to improve sensor performance to meet DOE 2012 targets
- Support deployment with information and expertise on sensor use and performance
- Test/validate new sensor R&D
- Support hydrogen sensor codes and standards development (national and international)
- Outreach and Education
  - Publications, presentations, consultations
  - Student Internships
- Client confidentiality





# Visual Leak Detectors for Hydrogen Equipment

Presented by











## **PRESENTERS**



**Bill Hoagland** | Element One 20+ Years in Hydrogen Research



John Poplawski | MSP 12+ Years in Silicone Manufacturing







## WEBINAR SUMMARY

- Company Backgrounds
- Leak Detection Summary
- Benefits of Low-Cost Visual Leak Detectors
- DetecTape<sup>™</sup> H<sub>2</sub> Technology and Performance
- Applications and Markets
- Availability and Upcoming Developments









## **ELEMENT ONE BACKGROUND**

- Incorporated in 2005 and began R&D of products based on core patent (U.S. Patent #6,895,805).
- Key members are veterans of NREL. Collaboration with NREL 2005 - present.
- Conduct thin film research in NREL's Hydrogen Sensor Laboratory.
- Funded contracts with DOE/NREL, NASA, Industry. Licensing agreements with industry. One technology license with NREL.
- Four patents, three pending.
- In 2015, partnered with MSP to create and market DetecTape™.

www.elem1.com









# **DOE** Recognition

March 2013

March 2012 August 2014



NREL: 2012 Industrial Growth Forum Presenting Company 2014 NREL Commercialization Assistance Program 2016 Small Business Voucher Award



Element One, Inc., of Boulder, Colorado, developed and patented a unique family of hydrogen and gas sensing technologies that form the basis for a wide array of low cost and reliable gas detection systems, an effort earning them the runner-up position in DOE's "America's Next Top Energy Innovator" competition. These hydrogen detection systems are based on simple chemochromic chemistry that visually indicates the presence or absence of hydrogen. These low cost hydrogen sensors have the ability to indicate the presence of leaking hydrogen at concentrations as low as 0.04%, or 1/100th of the lower flammability limit. Element One has developed similar detection systems and prolotypes for several other hazardous gases, including hydrogen suffide, ammonia and chlorine.

The prolific use of low cost gas sensors, like those developed by Element One, will significantly reduce the potential for undetected releases of hazardous gases in industry and fuel cell energy systems. This technology has the potential to reduce costs while improving the ability to detect leaks wherever such gases are used, whether it is used as a feedstock, reagent or fuel.







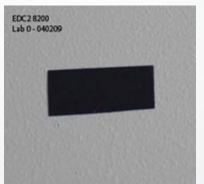


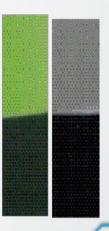


#### **DETECOAT® AND DETECTAPE™**



Thin films and "Smart" coatings for the detection of leaked or leaking hydrogen to achieve a **new level of reliability** and make possible a wide array of **very low cost** gas detection products.













## **APPLICATIONS**

- ✓ Tapes and wraps
- ✓ Leak detecting pipe covers
- ✓ Safety badges and warning s
- ✓ Leak detection coatings
- ✓ Pressure/reaction vessels and storage containers
- ✓ Sensors











#### HYDROGEN CHARACTERISTICS

- Odorless and colorless
- High propensity to leak
- Low ignition energy
- Invisible flame
- High energy content
- Lighter than air diffuses rapidly



Difficult to Detect Leaks









#### IMPORTANCE OF GAS LEAK DETECTION

- Gas detection is a critical element of the safe manufacture, handling and use of many industrial gases.
- The \$700+ million gas detection market, served by more than twenty-five OEMs and hundreds of maintenance contractors, is mature and stable with a projected annual growth in the 3 to 6% range.
- The global hydrogen market in 2015 was estimated at
   53 million metric tons with 12% in the merchant hydrogen market with a growth rate of 5-6% per year.









#### **CURRENT LEAK DETECTION METHODS**

- Bubble Testing Simple spot test method with no continuous monitoring at low pressures.
- Thermal Conductivity Works well in stable environments with minimal temperature change, background gas must have conductivity very different from hydrogen.
- Catalytic Combustion –
   Detects heat of combustion,
   works well for low
   concentrations. Not for pure
   hydrogen.
- Electrochemical Sensors use a liquid electrolyte, but varying temperature affects gas diffusion making the sensor unreliable.

- Mass Spectrometers –
   Extremely sensitive, expensive, require skilled operators and may have long response times.
- Gas Chromatographs Similar to mass spectrometers and have the same disadvantages.
- Ultrasonic Technology improving, but cannot determine exact location or whether combustible mixture is present.
- Semi-Conducting Oxide –
  Sensor relies on surface effects
  with a minimum oxygen
  concentration. Performance
  degrades at lower temperatures.









#### THE IDEAL LEAK DETECTOR

- ✓ Very Low-cost affordably used prolifically at potential leak sites;
- ✓ Positive Indication of both the presence and the absence of hydrogen;
- ✓ Simple, reliable, easily incorporated into the system, no power source;
- ✓ Hydrogen specific and sensitive;
- ✓ Quick response, but no false indications;
- ✓ Durable, long useful life requiring little or no maintenance; and
- ✓ Detectable by one or more of the human senses (sight, smell, sound).









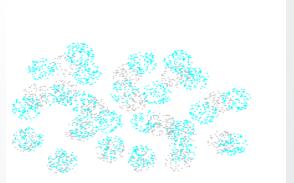
#### TECHNICAL APPROACHES

To meet varying requirements, Element One's research has taken two directions:

- 1. Thin films that both change color and resistance for use in low cost wireless sensors; and
- 2. Color changing pigments for coatings for visual indication.



Vacuum deposited multi-layer thin film



Synthesized nano-particles







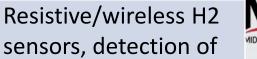


#### PIGMENTS VS. THIN FILMS

	Pigments	Thin Films
Response Speed	Slower	Faster
Manufacture	Easier to produce	Vacuum deposition process
Detection method	Visual only	Both visual and resistance change
Physical	Incorporated into	Multi-layer thin-film
Structure	carriers	devices
Material	More pigment per unit	Less pigment per unit
required	area	area
Durability	Extremely durable, resistant to contamination, sunlight (UV), acids, bases, moisture.	Less durable, requires selectively permeable protective coating.
Applications	Thick coatings, paints,	Resistive/wireless H2

inks, wraps, molded









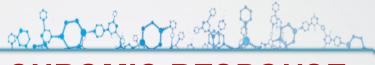
# Chemo-chromic chemical reactions: result of a partial reduction of a transition metal oxide

a partial oxidation of a catalyzed transition metal oxide pigment of < 20% effects a readily visible color change from light gray to virtually black.

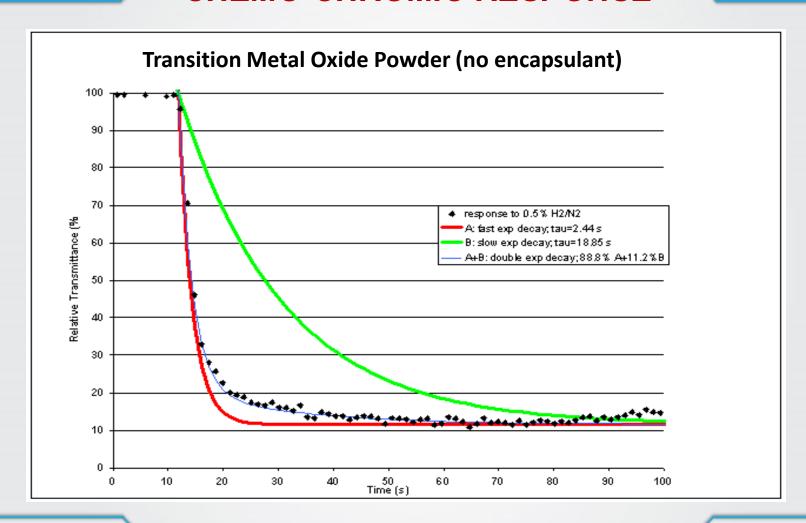








#### CHEMO-CHROMIC RESPONSE



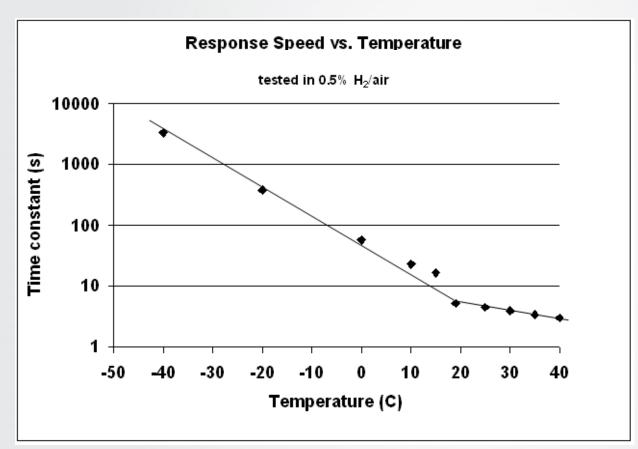








## RESPONSE VS. TEMPERATURE



✓ Responds more quickly at higher temperatures.

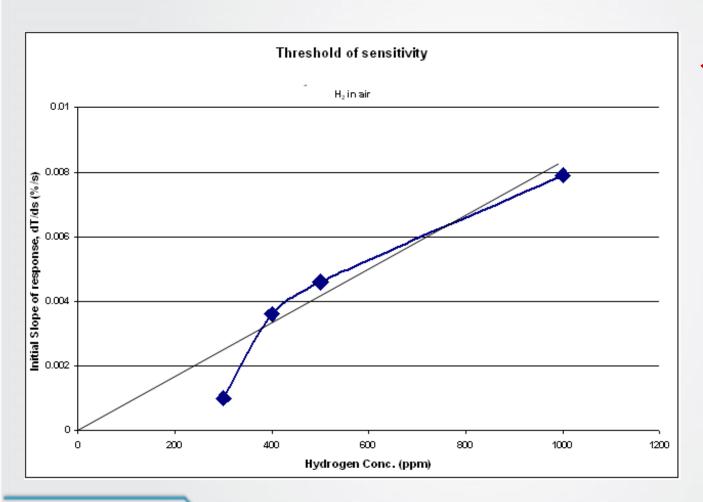








#### THRESHOLD of SENSITIVITY



✓ Thin film indication threshold in air is less than 400 ppm H₂

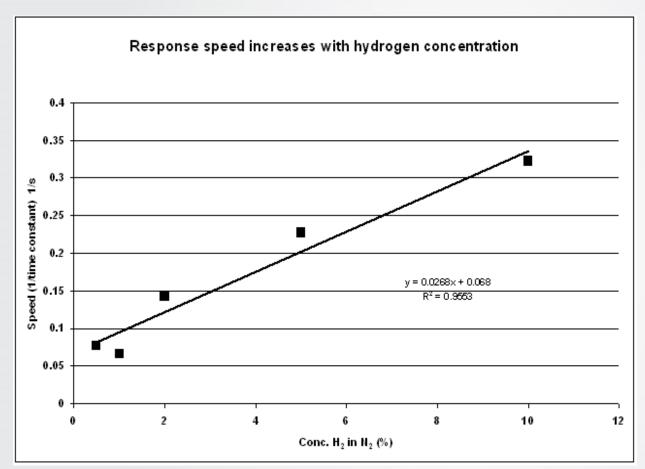








#### RESPONSE VS. CONCENTRATION



✓ Speed of Response is proportional to Hydrogen concentration.









# **DETECTAPE™** H<sub>2</sub>

(patent pending)



A chemo-chromic (color changing), self-fusing silicone tape designed to detect hydrogen gas (H<sub>2</sub>) leaks in storage, transmission and generation facilities.



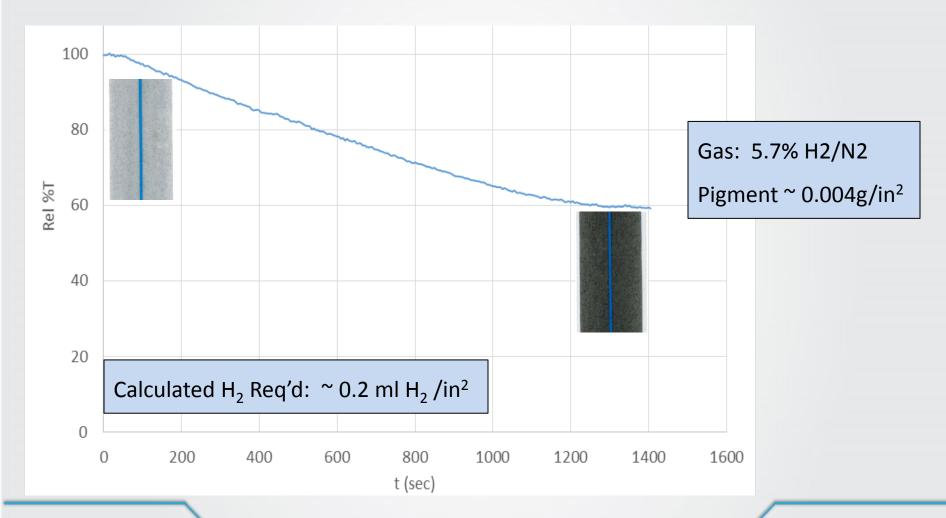








## DETECTAPE<sup>TM</sup> $H_2$ RESPONSE TEST







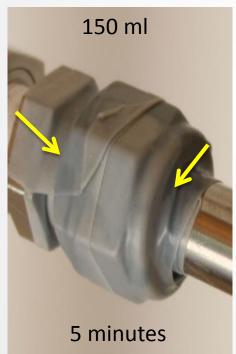


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# E1 DETECTAPE H<sub>2</sub> TESTS

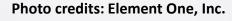
Controlled leak at ~ 0.5 ml/sec, 15 psi







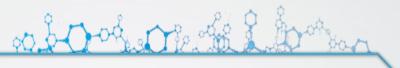












So...

If a typical wrap of DetecTape™ H<sub>2</sub> is 6 in<sup>2</sup>; and if it takes only .2 ml to change 1-in<sup>2</sup>....

Why does it take a leak of >300ml to completely change color?

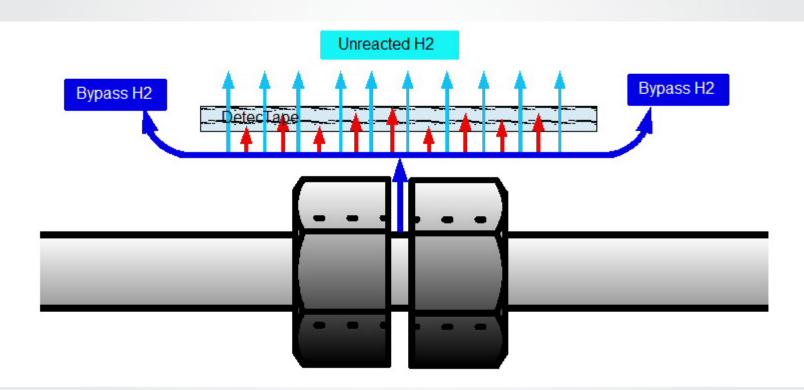








# Not all leaked gas reacts with the DetecTape™ H<sub>2</sub> pigment











# E1 DETECTAPE EXPOSURE TESTS (60 days and counting)

- √ 67° C under water
- ✓ 113° C in air
- ✓ 126° C in air
  - ? 195° C in air

- ✓ south-facing outdoors (sun)
- ✓ south-facing outdoors (shade)
- ✓ under NaOH solution pH 10
- ✓ under HCl solution pH 2

When exposed to hydrogen, all samples except the one exposed to 195° +/-5° C turned dark within 2 minutes. A significant darkening was apparent within 1 minute.









## NREL TESTING OF DETECTAPE™ H<sub>2</sub>

- Test duration: 8 months (on-going)
- 55 applications of DetecTape™ H<sub>2</sub> at NREL H<sub>2</sub> compressor and dispensing station
- Nine clear and verified leaks were indicated
- Four were "out-of-normal" requiring repair; four were "normal" (e.g., valve weep holes); one was noted for further monitoring









# NREL TESTING OF DETECTAPE™ H<sub>2</sub>

The deployment study was initiated with laboratory exposure testing

- Temperature cycling (-7° C to 44° C)
- Humidity cycling at ambient temperature (dry to 94% rh)
- Humidity cycling at elevated temperatures (56% and 75% relative humidity at 43° C)
- Hydrogen exposures after the temperature and humidity cycling, the indicator response was verified using 5.7 vol.% H<sub>2</sub> in N<sub>2</sub>.
   Result: Temperature and humidity cycling did not affect the functionality of the indicators.









# NREL DEPLOYMENT STUDY





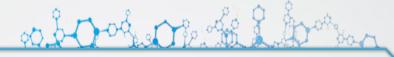






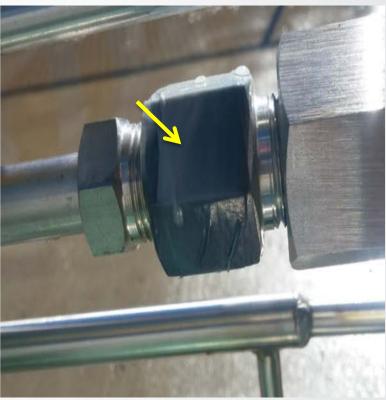






# NREL Deployment tests: Clear indication of a leak













# NREL TESTING DATA







Photo credits: K. Hartman/W. Buttner, NREL









## NREL TESTING DATA

 This indication was seen as the initiation of a slight darkening of the pigment.
 Since the color change was not as obvious as other indications, this indicator will continue to be monitored.

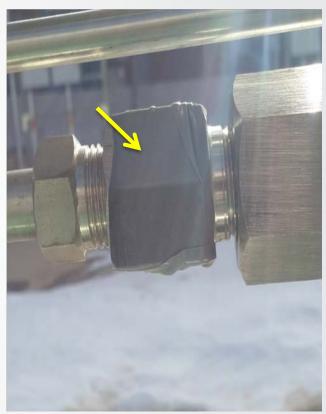


Photo credits: K. Hartman/W. Buttner, NREL









# Conclusions drawn from data by Element One

- DetecTape™ H₂ was shown to reliably identify unintended hydrogen releases from complex pneumatic systems.
- 2. DetecTape™ H₂ was verified as a viable technology to identify small hydrogen leaks which would otherwise go undetected. The identification of such leaks facilitates implementation of appropriate corrective action before the potential occurrence of major adverse events.







# DETECTAPE™ H<sub>2</sub> PRODUCTION AND MARKETING

# MSP, Inc. John Poplawski Executive VP / COO









#### **MSP BACKGROUND**

- 1997 Established as "Tommy Tape Manufacturing"
- 2000 Acquired by Midsun Group (est. 1992 Southington, CT), a leading provider of animal mitigation and corrosion coating products designed for electric utilities
- 2006 Renamed to Midsun Specialty Products, Inc.
- 2009 Moved operations to Berlin, CT
- 40 employees
- Manufacturer and distributor of self-fusing silicone tapes, corrosion protection, and specialty products worldwide

http://www.midsunsp.com









#### **CHANGES COLOR UPON DETECTION**



Hydrogen Leak Detected

Upon hydrogen detection, tape changes color from light grey to dark black.

- Noticeable change within 3 minutes.
- Significant change within 30 minutes.







## oo it Dodge Other I it is not one

## **SELF-FUSING SILICONE**

- Cross-linking silicone bonds to itself on contact.
- Does not use adhesives, leaves no residue on substrate.
- Naturally resists UV and most corrosive chemicals.
- Protected by a special removable liner before installation.
- Potential for vibration dampening.
- Conforms smoothly when wrapped around irregular shapes.









### FEATURED IN

DOX visit to MSP Detectape.

Satyapal, Director of Fuel

Cell Technologies at the

US Department of Energy.

emphasised the importance

of new technologies like these

that improve facility safety

of the nation's growing

hydrogen infrastructure.

www.gasworld.com/technology

and strengthen the reliability



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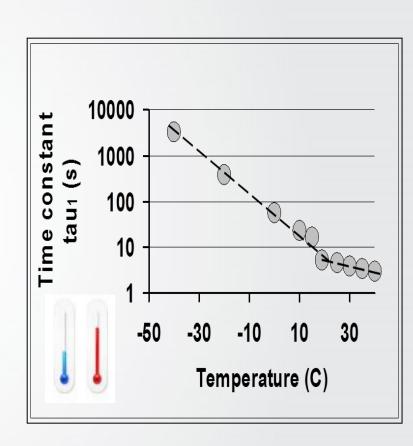






#### TEMPERATURE RANGE

- Extreme Temperature Range
- As low as 0°F (-17°C)
- As high as 350°F (176°C)
- Detection reaction time may be reduced or improved depending on temperature.







<sup>\*\*</sup> Temperature data still being collected. Subject to change \*\*



# 

## HYDROGEN REACTION

 Silicone is a hydrogen permeable carrier for chemo-chromic pigment that changes color when exposed to hydrogen.











#### HYDROGEN SAFETY INTEREST

- Very large quantities of hydrogen are safely used in industry today, but it's hard to contain and difficult to detect leaks.
- Hydrogen is a very large industrial commodity with a growing market.
- The introduction of hydrogen as a consumer fuel has caused heightened concerns over its safety with a corresponding increased interest in hydrogen sensors.









#### HYDROGEN REACTION

- Leak Rate and Concentration Determine Speed of Color Change. \*Formula in Development\*
- Cumulative Effect –
   Slow or small leaks can lead to
   significant color change over a long
   period of time.
- Reaction is permanent after hydrogen is detected, tape replacement is recommended after repair.











#### **EASY TO USE**

- Can be easily installed by on-site maintenance, inspection teams or equipment operators.
   No certified training required.
- Does not require power to operate.
- Can be used in unison with existing leak detection tools including sniffers and ultrasonic detectors.











#### INTENDED USAGE

DetecTape<sup>™</sup> H<sub>2</sub> can be wrapped on most objects including:

- Connections, Fittings and Reducers
- Swagelok and NPT
- Rigid Pipeline
- Flexible Tubing
- Flanges
- Welded Seams and Joints









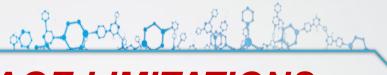
#### INTENDED USAGE

- Initially wrap DetecTape™ H<sub>2</sub> on all hydrogen connections that may be leak prone or susceptible to vibration damage.
- Leave DetecTape™ H<sub>2</sub> on connections permanently or until a leak is detected and a repair needs to be made.
- Can be easily removed with a blade or scissor.
- Once repair is complete and connection is in working order, wrap with DetecTape™ H₂ to monitor for future leaks.









#### **USAGE LIMITATIONS**

DetecTape<sup>™</sup> H<sub>2</sub> is **not** intended to:

- be used to stop nor slow down a leak.
- be used to detect anything other than hydrogen gas.
- prevent other gases from mixing with hydrogen.







#### **COUNTRY OF ORIGIN**

- All components of DetecTape<sup>™</sup> H<sub>2</sub> are manufactured in the United States.
- Tape core denotes Country of Origin.
- Hydrogen reactive pigment synthesized in Boulder, Colorado USA.
- Tape manufactured in Berlin, Connecticut USA.











#### **DOCUMENTATION**

- New Product Announcement Sheet
- Product Data Sheet
- Safety Data Sheet
- Instruction Sheet
- Videos (Coming Soon)
- NREL Deployment Study Coming Soon









#### TAPE ROLL SPEC

- Thickness 10 mil (0.01 inch / 0.254mm) thick
- Profile Rectangular / 10 mil across
- Width 1 inch (25mm) wide
- Length 15 feet (4.57m) long
- Color No leak detected : Light grey
   Leak Detected : Cumulative dark black blotches
- Guideline Blue to denote Hydrogen Gas Detector
- Current Perforation 4 inch (10cm) strips







#### **PERFORATION**

Strips perforated to specific lengths depending on object diameter.

- 1/16" 1/8" (1mm 4mm) diameter 4" strip (10cm)
   40-45 applications per roll NOW AVAILABLE
- 1/4" 1/2" (6mm 12mm) diameter 8" strip (20cm)
   25-30 applications per roll \* Coming Soon \*
- 1" 4" (25mm 100mm) diameter 12" strip (30cm)
   10-12 applications \* Coming Soon \*
- Multiple strips can be used on large diameters.

\*\* Configurations still in trial stages.

Subject to change based on user feedback \*\*





#### **CONTAINER**

- DetecTape<sup>™</sup> H<sub>2</sub> 15 foot rolls are shipped in a re-usable clarified polypropylene container to keep roll clean and dry while not in use.
- Portable enough to fit a roll in pocket, tool belt or toolbox.











## **USER INSTRUCTIONS**

- Every roll comes with user installation and detection instructions.
- More information and documentation (SDS, PDS, FAQ) is available to users on http://www.detectape.com













- Use on connections inside cabinets that are inspected on a routine schedule.
- Quickly identify hydrogen leaks during inspections.











- Use on hydrogen supply connection points in fueling stations.
- Attendants and maintenance crews can react quickly with a visual alert that there is a hydrogen leak present.











**Gas Refinery** 

- Use on all hydrogen network connections that are within visual range of inspection routes.
- Prioritize leak repair based on quantity of alerts in a zone to maximize efficiency.











Research Laboratory

- Use on hydrogen-related research equipment to improve laboratory safety.
- Once a leak is detected, equipment operator can safely shut down equipment for repair.











- Useful in conjunction with other gas detection alarm systems.
- Expedite repairs by visually identifying localized connections.









### OTHER APPLICATIONS

- On-Site Hydrogen Generation
- Manufacturing (Semiconductor, Aerospace, Etc.)
- Power Plants (Using Hydrogen Coolant)
- Gas Processing and Production
- Metallurgy and Welding
- Portable Power Units
- Pharmaceutical
- And More







#### **AVAILABILITY**

- Samples available upon request through website.
- Limited trial rolls available at introductory rate in exchange for user feedback and data.
- Case pack 36 rolls / case
- Single rolls available with 5 roll MOQ
- Available for Purchase









#### **UPCOMING DEVELOPMENTS**

 H<sub>2</sub> Reversible Tape - Hydrogen reaction is reversible. Designed for tightly enclosed spaces where in-air hydrogen may be a concern over time.



 H<sub>2</sub>S Tape – Hydrogen Sulfide leak detection for use as a marker for natural gas pipeline leaks.











## **ACKNOWLEDGEMENTS**

- We would like to thank the Office of Energy
   Efficiency and Renewable Energy's Fuel Cell
   Technologies Office for their continued support.
- We would like to thank Dr. William Buttner and NREL for the cooperation and assistance provided to Element One, Inc.







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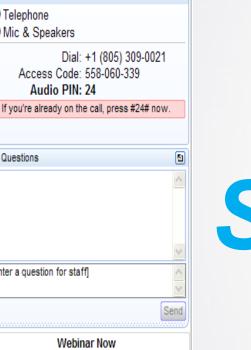
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Questions



## THANK YOU FOR WATCHING



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http://www.detectape.com



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## Thank You

#### Presenters:

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  - whoagland@elem1.com
- •John Poplawski MSP
  - info@detectape.com

#### DOE Host:

- •Will James Safety, Codes, and Standards Program Manager
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#### FREQUENTLY ASKED QUESTIONS

- 1. What range of hydrogen concentration has the tape been measured / proven with?
- 2. What other components have been included in the testing for Question 1?
- 3. What is the repeatability of the tape's performance, i.e. does the same size leak produce color at the same rate?
- 4. Is there a correlation of leak amount versus color change rate?
- 5. What is the impact on temperature on the color change?







#### FREQUENTLY ASKED QUESTIONS

- 6. What is the longest that the tape is been in use for any of the tests?
- 7. How can we tell if the tape is still working after it has been in service for a time? (eliminate false negatives)
- 8. Are there any other components that may cause false positives?
- 9. Are there any other components that may interfere with the hydrogen response? (eliminate false negatives)
- 10. Is the tape sample we received the same as will be sold for commercial use?



