### Ford/BASF-SE/UM Activities in Support of the Hydrogen Storage Engineering Center of Excellence

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### Overview



Min. Delivery Temp

Max Delivery Temp

Min. Delivery Pressure

Max. Operating Temp

#### Timeline

- Project Start: February 2009
- Project End: June 2015

### **Budget**

- Total Project Value: \$2,783K
  - Cost Share: \$643K
  - DOE Share: \$2,140K
- DOE Funding Spent\*: \$1,715K

\*as of 3/31/14

HSECoE

### **Barriers**

#### • All DOE System Targets\*\*

- Volumetric Density
- Gravimetric Density
- System Cost
- \*\*http://www1.eere.energy.gov/hydroge nandfuelcells/storage/pdfs/targets\_ onboard\_hydro\_storage.pdf

#### **Partners**

- Project Lead: Ford
- Subcontractors: BASF and U. Michigan
- Center Partners:







Gravimetric Density

100%-

Start Time to Full Flow (20°C)

Fill Time (5kg H2

Start Time to Full Flow (-20°C

Transient Respons

Adsorbent System Example



### **Relevance:** Technical





### Three Technical Tasks Contribute to the Overall HSECoE Mission

Task 1: Develop dynamic vehicle parameter model that interfaces with diverse storage system concepts Ford

Task 2: Development of robust cost projections for storage system concepts

Ford Task 3: Devise and develop system-focused strategies for processing and packing

framework-based sorbent hydrogen storage media

Materials Properties Vehicle Viability Vehicle Task 3 data supports the creation Viability Tasks 1 & 2 models support of sorbent bed models & aids in determination of overall vehicle cost tradeoffs analyses System Modeling and performance & Development **Bed Modeling** System Modeling Thermal Management & Bed Modeling Tasks 1 & 2 models enable storage Task 3 data supports the concepts to be exercised at the validation of sorbent bed and real-world vehicle level system models Materials Properties & Compaction **HSECoE** 2014 DOE Annual Merit Review Meeting 4

### Relevance: Organizational

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#### Ford project has many roles and responsibilities within the DoE Program Management HSECoE at both the executive and working levels. N. Stetson J. Adams R. Bowman **Center Coordinating Council** Key organizational functions: D. Anton, Center Director T. Motyka, Assistant Director As technical contributors, **DOE Program Liaisons Technology Area Leads** disseminate data & models **OEMs** Independent Projects Performance Cost & M. Cai, GM Energy Analysis across the HSECoE M. Veenstra, Ford T. Motyka M. Thornton Materials Operating As team leads, foster inter-Hydrogen Safety System Architects Requirements J. Khalil E. Rönnebro partner communication & MH System Transport Phenomena T. Motyka streamline & align research B. Hardy External Communications CH System Integrated Storage Act as liaisons between the T. Motyka T. Semelsberger System/Power Plant Modeling B. van Hassel A System HSECoE and the C&S and Leads adsorbent Enabling Technologies D. Siegel Storage Tech. Teams K. Simmons MOR team Subscale Prototype Lead adsorbent Provide an automotive Leads powerplant Construction, Testing, & Evaluation system architect modeling team T. Semelsberger perspective & context

- Core contribution areas of project outcomes [red]
- Ancillary contribution areas of project outcomes [green]

### Approach: System Architect and OEM perspective



#### System Architect Role (D. Siegel)

- Performed analysis for Phase 3 Go/No-go
- Coordinated design status within Adsorbent Team
- Identified and prioritized the research gaps
- Developed SMART milestones and GANTT chart
- Completed operating conditions downselection process
- Organized regular meetings with Adsorbent Team

#### **OEM Perspective Role (M. Veenstra)**

- Involved in the HSECoE framework model release
- Assisted in the system integration and cost analysis
- Coordinated design verification plan for FMEA
- Engaged in trade-offs for system optimization

HexCell

HSECoE



Adsorbent system has progressed significantly from Phase 1

### Approach: System Architect and OEM perspective



- Full tank: P = 100 bar, T = 80 K
- Empty tank: P = ~5 bar, T = ~140 K
- Single, Aluminum (6061-T6) Type 1
- LN<sub>2</sub> vessel wall chilling channels

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\*2013 AMR references ANL Project ID: ST001 SA Project ID: ST100

BAS

### Approach: Enhance MOF Performance Potential



#### Key Objectives of the HSECoE:

- Design, model, and test innovative material-based systems for gap analysis
- Define required materials properties to meet the system technical targets
- Validate models with sub-scale prototype system for predictive capability
- Develop and provide system models for further material research

#### **Reviewer Comments from 2013 AMR:**

"It is highly unlikely that a system based on MOF-5 will meet the DOE targets. *It would be helpful if a pathway to identifying an optimum adsorbent system could be provided*."

"Experimental and modeling *analysis should be performed on a promising physisorption material* that is different from MOF-5."



### **Progress:** Enhance MOF Performance Potential



### Additional gains in H<sub>2</sub> capacity may be realized using known MOFs



- Performed a comprehensive assessment of the theoretical capacities of several thousand known MOFs
  - Accomplished by mining the 600,000+ entry Cambridge Structured Database
  - Automated routines for structure cleanup and analysis
- Identified several MOF with the opportunity of having both high gravimetric and volumetric H<sub>2</sub> density
- Relationship between gravimetric and volumetric density is concave downward:
  - Optimal MOFs for H<sub>2</sub> have a surface area in the range of  $3,100 4,800 \text{ m}^2/\text{g}$ ; density ~ 0.55 g/cm<sup>3</sup>

40 g/L

20

Higher surface area can compromise volumetric performance

10

DUT-10.11.12

CN-610/NU-100

CMOF-L4

IMP-9





Goldsmith, Wong-Foy, Cafarella, and Siegel, Chem. Mater., 25, 3373 (2013)

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### Approach: Enhance MOF Performance Potential



- Four MOF Targets of Opportunity were identified
- Exhibit high gravimetric and volumetric densities simultaneously
- Overlooked compounds: no/limited experimental evaluation

#### Can these be synthesized in a robust form?

(No retained solvent, no pore collapse)

35 bar & 77 K Modeled Values (measured)	EPOTAF (SNU-21)	DIDDOK	LURGEL (TO-MOF)	ENITAX (IMP-9)	MOF-5
Total Grav. (wt. %)	11	10.2	9.7	9.3	<mark>8.2</mark> (8.4)
Total Volumetric (g/L)	71	60	57	59	<mark>52</mark> (54)
Crystal Density (g/cm <sup>3</sup> )	0.58	0.53	0.53	0.57	0.59
BET Surface Area (m <sup>2</sup> /g)	<b>5208</b> (700-900)	4651	<b>4386</b> (680)	4162	<b>3660</b> (3800)
Notes	Best performer. H <sub>2</sub> uptake measured previously: 5 wt. %	No measurements	CO <sub>2</sub> uptake measured	No measurements	For reference purposes

Goldsmith, Wong-Foy, Cafarella, and Siegel, Chem. Mater., 25, 3373 (2013)

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Potential system improvements are 34% gravimetric & 37% volumetric



### Approach: Phase 3 SMART Milestones and Tasks



Component	Partner	Proposed SMART Milestones for Phase 3	Due Date
Adsorbent Media	Ford/UM/BASF	<b>Conduct a scale-up of the MOF-5 manufacturing process</b> to deliver > 9 kg of material while maintaining performance, as measured by surface area and particle size, to within 10% of lab-scale procedure.	12/31/2013
Adsorbent Media	Ford/UM/BASF	Evaluate MOF-5 degradation beyond 300 cycles based on maximum allowable impurity levels as stated in SAE J2719 and report on the ability to mitigate to less than 10%.	9/30/2014
Adsorbent Media	Ford/UM/BASF	<b>Complete the failure mode and effects analysis (FMEA)</b> associated with real-world operating conditions for a MOF-5-based system, for both HexCell and MATI concepts <u>based on the Phase 3 test results</u> . Report on the ability to <u>reduce the risk priority numbers (RPN)</u> from the phase 2 peak/mean and identify key failure modes.	6/30/2015
System Modeling	NREL/SRNL/ PNNL/Ford/ UTRC	<b>Update the cryo-adsorbent system model</b> with Phase 3 performance data, integrate into the framework; document and <u>release models to</u> <u>the public.</u>	9/30/2014
Addition	al task:	Explore approaches to maximize the MOF-5 "real-world" material properties: advance thermal conductivity, mass transport, and safety	

Project approach based on collaborative HSECoE SMART milestones

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Milestone Task: Conduct a scale-up of the MOF-5 manufacturing process to deliver > 9 kg of material while maintaining performance, as measured by surface area and particle size, to within 10% of lab-scale procedure.

<b>'7 L scale' synthesis:</b> Becher Laboratory stirrer Yield <sub>Terephtalic acid</sub> : 81 mol% Surface area: 2680 m²/g Batch Amount: >0.1 kg	<b>´60 L scale´ syr</b> Steal reactor Plant stirrer Yield <sub>Terephtalic</sub> a Surface area: 2 Batch Amount:	nthesis: <b>´200 L sca</b> Steal rea Plant s acid: 81 mol% Yield <sub>Ten</sub> 2905 m²/g Surface : > 1 kg Batch Am STY: >150	ale´ synthesis: actor tirrer <sub>ephtalic acid</sub> : 81 mol% area: 2937 m²/g nount: > 3 kg kg/m³/d	
Crystallizing	Filtering Washing	Drying	Milling	MOF-5 Powder
Reactor	Filter	Oven	Mill	Drum
Successfu	I Phase 3 MOF	-5 scale-up and o	lelivery of a 9.3	kg drum
HSECOE	2014 DOE	Annual Merit Review	/ Meeting	12



Milestone Task: Conduct a scale-up of the MOF-5 manufacturing process to deliver > 9 kg of material while maintaining performance, as measured by surface area and particle size, to within 10% of lab-scale procedure.

Batch Code	Reactor Size [L]	Amount [kg]	BET [m²/g]	LSA [m²/g]	Zn [wt%]	C [wt%]	Crystal size [µm]	Particle size [mm]
GP0372	200	3.1	2937	3838	32	37	0.2-2.0	
GP0374	200	3.5	2870	3794	34	37	0.2-2.0	
GP0375	200	3.2	2955	3896	34	37	0.2-2.0	
GP0378	Mix	9.3	2937	3877	30	37	0.2-2.6	0.1-1.3
GP0326	60	1	2905	3891	34	37	0.2-3.0	0.1-1.4
Sca	ale-Up Di	fference:	1%	.4%				7%
Reference GW0116	7	.14	2680	3547			0.2-2.0	

MOF-5 scale-up material achieved target of 10% of lab-scale synthesis

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Milestone Task: Conduct a scale-up of the MOF-5 manufacturing process to deliver > 9 kg

Crystal size SEM microscopy comparison analysis - magnification 5000:1

200 L Batch

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MOF-5 scale-up material has comparable crystal size with lab-scale

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60 L Batch

# Concernant BASE The Channel Company

GP0375

### Progress: MOF-5 Manufacturing Scale-up

#### Milestone Task: Conduct a scale-up of the MOF-5 manufacturing process to deliver > 9 kg

#### Crystal size SEM microscopy comparison analysis - magnification 5000:1

**GP0374** 



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Crystal surface roughness variation can occur as a result of different washing times during the solvent filtering step.

MOF-5 scale-up material has repeatable crystal size between batches





MOF-5 scale-up material has consistent particle size as lab-scale

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*Milestone Task: Conduct a scale-up of the MOF-5 manufacturing process to deliver > 9 kg of material while maintaining performance, as measured by surface area and particle size, to within 10% of lab-scale procedure.* 



MOF-5 scale-up material has equivalent performance as lab-scale

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### **Progress:** MOF-5 Robustness to H<sub>2</sub> Impurity

# Milestone Task: Evaluate MOF-5 degradation beyond 300 cycles based on maximum allowable impurity levels as stated in SAE J2719 and report on the ability to mitigate to less than 10%.

Constituent	Chemical Formula	Limits	Laboratory Test Methods to Consider and Under Development <sup>e</sup>	_		
Hydrogen fuel index	H <sub>2</sub>	> 99.97%		l Ir	nnurity	degradation projections
Total allowable non-hydrogen, non- helium, non-particulate constituents listed below		100			npunty	
Acceptable limit of each individual constituent			Impurity per cycle [g]	Impurity in 300 cycles	Estimated effect on MOF-5	
Water <sup>a</sup>	$H_20$	5	ASTM D7653-10, ASTM D7649-10	0.031	9.3 g	≤2% destruction of surface area
Total hydrocarbons <sup>ь</sup> (C <sub>1</sub> basis)		2	ASTM D7675-11	0.0124	3.7 g	<0.1% surface area blocking
Oxygen	O <sub>2</sub>	5	ASTM D7649-10	0.031		no effect
Helium		300	ASTM D1945-03	1.86		no effect
Nitrogen, Argon	N <sub>2</sub> , Ar	100	ASTM D7649-10	0.62		no effect
Carbon dioxide	CO <sub>2</sub>	2	ASTM D7649-10, ASTM D7653-10	0.0124	3.7 g	<0.1% surface area blocking
Carbon monoxide	СО	0.2	ASTM D7653-10	0.00124	0.4 g	<0.1% surface area blocking
Total sulfur <sup>c</sup>		0.004	ASTM D7652-11	0.0000248	0.01 g	potential damage -not detectable at impurity level
Formaldehyde	HCHO	0.01	ASTM D7653-10	0.000062		no effect
Formic acid	НСООН	0.2	ASTM D7550-09, ASTM D7653-10	0.00124	0.4 g	potential damage -not detectable at impurity level
Ammonia	NH <sub>3</sub>	0.1	ASTM D7653-10	0.00062	0.2 g	potential damage -not detectable at impurity level
Total halogenates <sup>d</sup>		0.05	(Work Item 23815)	0.00031	0.1 g	potential damage -not detectable at impurity level
Particulate Concentration		1 mg/kg	ASTM D7650-10 , ASTM D7651-10			

See SAE J2719 for original reference

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Hypothesis: MOF-5 will only have minor effects during impurity testing



### **Progress:** FMEA - Failure Mode Reduction



#### FMEA = Failure Mode and Effects Analysis (industry tool per SAE J1739)

- Identifies and evaluates the potential failure of a product and its effects
- Documents the risk and helps prioritize the key actions to reduce failures

#### Top Failure Modes for Adsorbent System at phase 2 with Risk Priority Number (RPN) >300

- 1. Material release rate insufficient due to non-homogenous materials or bed
- 2. Material release rate insufficient due to impurities (from station at single time or lifetime)
- 3. Tank incompatible with adsorbent or in-service activation
- 4. Material release rate insufficient due degradation in heat transfer in bed and to the thermal management system





**Explore approaches to advance thermal conductivity, mass transport, and safety** <u>MOF-5 with random ENG</u>



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#### MOF-5 with ENG layers



The pellet was formed by filling the die with alternating layers of MOF-5 and ENG. When all the layers were filled the pellet was pressed. The ENG appears to form one connected layer across the pellet. (Ford Patent Pending)



MOF thermal conductivity break-through using aligned ENG



#### Explore approaches to advance thermal conductivity, mass transport, and safety

#### **MOF-5 formation with pins**

Formed the MOF-5 bed around the pins to increase conduction enhancement.



Two (solid & hollow) aluminum pin configurations were formed that had .1 cm diameter with roughly a 1 cm height (depth into the MOF5 bed) with spacing of about 1 cm



W1

Η1

Average:

Std Dev:

23.43

0.059

#### **MATI Puck formation**





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Pucks: ø5 cm x 1.5 cm



rightepealability with forming					
	<u>Weight</u> (g)	<u>Density</u> (g/cc)	<u>Avg</u> <u>Height</u> (mm)	<u>D1</u> (mm)	<u>W1</u> (mm)
А	23.39	0.40	15.14	50.23	96.33
В	23.42	0.40	15.28	50.23	96.35
С	23.35	0.40	15.08	50.25	96.35
D	23.41	0.41	14.93	50.22	96.38
Е	23.46	0.40	15.27	50.26	96.39
-	00 50	0.40	45.05	50.04	00 07

0.40

0.003

High repeatability with forming

Puck formation offers additional enhancements to thermal conductivity

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50.24

0.015 0.022

0.139

96.36

Explore approaches to advance thermal conductivity, mass transport, and safety

MicroCT analysis: the density difference within a puck at .40 g/cc and 10 wt.% ENG density. The scan confirmed an average density of 0.41g/cc (density: red > green > blue)



Sample Section Distributions Mean density: 0.443g/cc Standard deviation: 0.123g/cc



Mean density: 0.439g/cc Standard deviation: 0.145g/cc

Mean density: 0.420g/cc Standard deviation: 0.134g/cc



Mean density=0.411g/cc



Mean density=0.406g/cc





#### Explore approaches to advance thermal conductivity, mass transport, and safety



Major increase in mass transport permeability with low density powder

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Explore approaches to advance thermal conductivity, mass transport, and safety Explosion Severity of Dust Cloud, K<sub>st</sub> (ASTM E 1226)

- Test provides an indication of the severity of a dust cloud explosion
- Data produced:
  - Maximum developed pressure, P<sub>max</sub>
  - Maximum rate of pressure rise, (dP/dt)<sub>max</sub>
- Deflagration index (explosion severity) K<sub>st</sub>

 $K_{st} = (dP/dt_{max}) V^{1/3}$  [bar.m/s] where V is the volume of the test vessel

Used for the design of deflagration protection

Based on test data using  $1m^3$  and 20L Vessels and 10KJ Ignition Source

Dust Explosion Class	K <sub>st</sub> (bar * m/s)	Characterization	<b>TEST RESULTS FOR MOF-5</b> Maximum explosion pressure: 6.3 bar abs
St 0	0	Non-explosible	Deflagration index (K <sub>st</sub> ) value: 48 bar * m / s
St 1	0 < K <sub>st</sub> < 200	Weak to moderately explosible	Dust explosion class: St 1
St 2	200 < K <sub>st</sub> < 300	Strongly explosible	
St 3	K <sub>st</sub> > 300	Very strongly explosible	

MOF-5 testing resulted in a low explosion class and deflagration index



## Summary: Phase 3 SMART Milestones and Tasks



SMART Milestone Tasks	<u>Status</u>
Conduct a scale-up of the MOF-5 manufacturing process > 9 kg	<ul> <li>Delivered 9.3 kg of MOF-5 for Phase 3 to HSECoE partners within 10% of lab-scale synthesis material</li> </ul>
Evaluate MOF-5 degradation cycles using impurity levels as stated in SAE J2719	<ul> <li>Degradation projections completed and initial cycling has started with ammonia impurity without degradation</li> </ul>
Complete the failure mode and effects analysis (FMEA) based on the Phase 3	<ul> <li>Initiated design verification plan (DVP) to align the FMEA action items with the Phase 3 test results</li> </ul>
Support system model release and validation with Phase 3 performance results	<ul> <li>Provided fuel cell model to Simulink framework based on validated data and participated in modeling group</li> </ul>
Additional Tasks	<u>Status</u>
Additional Tasks Enhance thermal conductivity	<ul> <li><u>Status</u></li> <li>✓ Demonstrated significant improvements (20x) in thermal conductivity with anisotropic ENG layering</li> </ul>
Additional Tasks Enhance thermal conductivity Conduct compaction puck formation	Status         ✓       Demonstrated significant improvements (20x) in thermal conductivity with anisotropic ENG layering         ✓       Formulated the MATI half pucks with embedded thermocouples with high consistency
Additional TasksEnhance thermal conductivityConduct compaction puck formationExtend permeation flow evaluation	Status         ✓       Demonstrated significant improvements (20x) in thermal conductivity with anisotropic ENG layering         ✓       Formulated the MATI half pucks with embedded thermocouples with high consistency         ✓       Tested flow hydrogen flow parameter through powders



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# Image: Construction of the construc

### Future Work: Complete Phase 3 Tasks

- □ Complete MOF-5 degradation cycle testing based on impurity levels as stated in SAE J2719 and report on the ability to mitigate to less than 10%.
- □ Complete the failure mode and effects analysis (FMEA) associated with real-world operating conditions for a MOF-5-based system, for both HexCell and MATI concepts based on the Phase 3 test results. Reduce the risk priority numbers (RPN) from the phase 2 peak/mean and identify key failure modes.
- Complete the optimization approaches to enhance thermal conductivity, mass transport, and density variations in formed pucks.
- Support the modeling validation using Phase 3 test data, further integration of the system BOP components for the cost analysis, and prepare for HSECoE project summary documentation to guide material researchers.



### **Collaborations:** HSECoE Partners





- SRNL (federal lab collaborator): team lead for sorbent (bed) transport phenomena, adsorbent system modeling, and center management
- Universite du Quebec a Trois-Rivieres (university collaborator): adsorption system test bench and MOF-5 isotherm validation
- GM (industrial collaborator): sorbent materials operating parameters, sorbent system modeling, and helical coil heat exchanger development
- Oregon State University (university collaborator): development of microchannel internal bed heat exchanger and combustors
- Hexagon Lincoln (industrial collaborator): pressure vessel development for hydrogen storage system concepts
- PNNL (federal lab collaborator): team lead for cost modeling, bill of materials, and materials operating requirements
- UTRC (industrial collaborator): material particulate testing, MOF-5 thermal conductivity measurements, and on-board system modeling
- NREL (federal lab collaborator): vehicle level modeling, wells-to-wheels analysis, MOF-5 isotherm validation, and low temperature isotherms
- JPL (federal lab collaborator): insulation development and cryogenic parameter evaluation

Interactions include monthly team meetings (sorbent system, material operating req., system modeling), regular data and information exchanges, and <u>ten</u> HSECoE face-to-face meetings



# **Technical Back-up Slides**



### General FMEA Overview and Approach

#### The FMEA is based on the required system functions from the technical targets.

Table 1. Technical System Targets: Onboard Hydrogen Storage for Light-Duty Fuel Cell Vehicles <sup>a</sup>					
Storage Parameter	Units	2017		Ultimate	
System Gravimetric Capacity: Usable, specific-energy from H <sub>2</sub> (net useful energy/max system mass) <sup>b</sup>	kWh/kg (kg H <sub>2</sub> /kg system)	1.8 (0.055)	)	2.5 (0.075)	
System Volumetric Capacity: Usable energy density from H <sub>2</sub> (net useful energy/max system volume) <sup>b</sup>	kWh/L (kg H <sub>2</sub> /L system)	1.3 (0.040)	)	2.3 (0.070)	
Storage System Cost: • Fuel cost <sup>c</sup>	\$/kWh net (\$/kg H <sub>2</sub> ) \$/gge at pump	12 400 2-4		8 266 2-4	
Durability/Operability:         • Operating ambient temperature <sup>d</sup> • Min/max delivery temperature         • Operational cycle life (1/4 tank to full)         • Min delivery pressure from storage system         • Max delivery pressure from storage system         • Onboard efficiency <sup>e</sup> • "Well" to powerplant efficiency <sup>e</sup> • Charging/Discharging Rates:         • System fill time (5 kg)         • Minimum full flow rate         • Start time to full flow (20°C)         • Start time to full flow (-20°C)	°C °C Cycles bar (abs) bar (abs) % % % % min (kg H <sub>2</sub> /min) (g/s)/kW s s	-40/60 (s -40/85 1,500 5 12 90 60 3.3 (1.5) 0.02 5 15	un)	-40/60 (sun) -40/85 1,500 3 12 90 60 2.5 (2.0) 0.02 5 15	
Transient response at operating temperature 10-90% and 90-0%	s	0.75		0.75	
Fuel Quality (H2 from storage): *         Environmental Health & Safety:         • Permeation & leakage g         • Toxicity         • Safety         Loss of Useable H : *	% H <sub>2</sub>	(99.97% drv basis) Meets or exceeds applicable standards		v basis) s applicable ds	
LUSS OF USCADIC 112.	(g/II)/Kg II2 stored	0.05		0.05	

Cost of Ownership (Provide a competitive system)

Accept Fuel (Fill storage system)

Deliver Fuel (Supply H<sub>2</sub> from storage system)

Store Fuel (Manage H<sub>2</sub> in the system)



### General FMEA Overview and Approach

X

ouverity				
Effect	Ranking			
Hazardous without warning	10			
Hazardous with warning	9			
Very High	8			
High	7			
Moderate	6			
Low	5			
Very Low	4			
Minor	3			
Very Minor	2			
None	1			

Sovarity

#### Occurrence Ranking Probability of Failure Very High: 10 Persistent 9 Failures High: 8 Frequent 7 Failures Moderate: 6 Occasional 5 Failures 4 Low: 3 Relatively 2 **Few Failures**

Remote:

Failure is Unlikely

Likelihood of Detection	Ranking
Absolute Uncertainty	10
Very Remote	9
Remote	8
Very Low	7
Low	6
Moderate	5
Moderately High	4
High	3
Very High	2
Almost Certain	1

Detection

X

RPN

Risk

=

Priority Number



1