



MefHySto – Overview and Tangible Results

EMN Energy Gases "Measurement Solutions for Energy Gases" Workshop Lisbon, Protugal, March 22nd, 2023



The EMPIR initiative is co-funded by the European Union's Horizon 2020 research and innovation programme and the EMPIR Participating States

Michael Maiwald

- Duration 2020–2023
- EU Funding 2.3 M€
- Coordinator: BAM
- 14 Partners
- Power-to-Hydrogen
- Thermophysical properties
- Hydrogen-to-power
- Reversible storage
- Geological storage

- Ambitious new EU energy target of using 32 % → 40 % of renewable energy by 2030
- Cannot be reached without advanced energy storage solutions, e.g., H₂ storage
- Measurement science is critical for realisation of this commitment
- Financial concerns of amount of hydrogen generated – stored – back-converted



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Hydrogen as a Secondary Energy Carrier





- Conversion from thermal machines such as turbines and motors are limited by Carnot efficiency
- Hydrogen as new secondary energy carrier
- Can be produced from primary energy with much higher conversion efficiency
- Electrolysers, batteries, and FCs, which have theoretical efficiencies approaching 100 %

Sources

[1] IEA, Total primary energy supply by fuel, 1971 and 2019, IEA, Paris - https://www.iea.org/data-and-statistics/charts/total-primary-energy-supply-by-fuel-1971-and-2019 [2] IEA, World total final consumption by source, 1973-2018, IEA, Paris - https://www.iea.org/data-and-statistics/charts/world-total-final-consumption-by-source-1973-2018

WP1 Metrology for Hydrogen Quality from **Power-to-Hydrogen**



- Quality from Water Electrolysis
- **Online Gas Analysis**
- **Reference Analytics**

Tasks

- New metrology for realisation and . measurement of H₂ key impurities with fast response
- Testing and validation of instruments for measuring H₂ key impurities
- Trials of rapid response analysis of H₂ key contaminants from electrolysis in-situ

Water vapour and oxygen identified as key impurities.

Water vapour step change facility with a 4-way switching valve developed with traceability to NPL Primary Standard Humidity Generator.



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6 Impact

1 Power-to-

Hydrogen

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- Hygrometers with seven different measurement principles tested including: SAW, metal oxide dew-point probe, fibre optic, chilled-mirror, electrolytic and spectrometry.
- Fitting function to response time data developed to estimate t_{90} values with uncertainties.



data fit

Figure 2: *t*₉₀ response time analysis for seven hygrometer types.



Two laboratory electrolyser setups

transient profiles on the cell performances and on the quality of produced hydrogen to be studied with online sensors (including μ GC and GC-methaniser-FID)



Figure 4: PEMWE testing at single cell level test bench specifications

Figure 3: Rising series response time



WP3: Metrology for Hydrogen Quality from Electrical **Energy Storage by Hydrogen Back Conversion**



- Impact of Impurities on Fuel Cells
- **Reference Materials**

CEN/TC 234, ISO/TC 197, CEN/CLC/JTC 6 and CEN/CLC SFEM WG Energy Storage End users

Properties

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Air Quality Effects

Tasks

- Gas Reference Materials for fuel cell tests
- Impact of impurities on fuel cell performance; standards for H₂ quality
- Specify a process air quality sensor for . monitoring fuel cell performance and durability
- Validation of metrological chain for gas analysis: complete H₂ Storage Providers platform level and Manufacture Electrolyser and Eucl Cell Manufactures

Power

Fuel Cells

stry, energy sector, gas suppliers for distribution

Impact of Hydrogen and Air Impurities

- Literature review on previous/current work on hydrogen and air impurities to set specific compositions to be studied
- Synergy effects between NO_x and SO_y species has been highlighted in literature and will be further studied for air supply
- Development of analytical methods for VOC (Volatile Organo-halogenated Compounds) analysis in air and sorbents qualification is ongoing



Figure 1: Example of sorbents for VOCs sampling/enrichment



Figure 2: Fuel cell lab with analytics for FC stack and single cell (@CEA)

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6 Impact

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Metrology for Advanced **Hvdrogen Storage Solutions**

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Figure 3: FC-DLC automotive

load profile for ageing test

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WP3: Metrology for Hydrogen Quality from Electrical

Energy Storage by Hydrogen Back Conversion

Testing Facilities and Test Results

- Fuel cell single cell and stack technologies were set up and are now available
- First reference tests under both reference "pure" H_2 and air were conducted
- Preparation of Gas Mixture for fuel cell tests following the H_2 ISO 14687-2 with all contaminants at their maximum respective levels
- Fuel cell tests (performance and durability) planned with this H_2 composition

Figure 4: Cell voltage evolution during reference durability tests for 500 h using FC-DLC in 25 cm² single cell



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- Thermophysical Properties of H₂ and H₂ enriched natural gas
- Equations of State

Tasks

- Develop and test lab techniques to provide reference mixtures of humid H₂/ H₂enriched natural gas
- Influence of H₂ content on the saturation curve of H₂-enriched natural gas mixtures
- Develop reference equations of state (EOS) for H₂-enriched natural gas mixtures and H₂ under geological storage conditions



Equations of State

- European Gas Research Group (GERG)-2008 equation of state (EoS) is the approved ISO standard (ISO 20765-2)
- For increased H₂ content of NG, the GERG-2008 EoS needs to be extended to application range for diversification
- As part of this, high-precision experimental pressure, mass density and temperature (*ρ*, *ρ*, *T*) data for gravimetrically prepared synthetic natural gas mixtures are needed.



Figure 1: GERG-2008 Reference Equation of State Compound Matrix [1]

Reference

[1] O. Kunz; W. Wagner; J. Chem. Eng. Data 57, 3032—3091, 2012. DOI: 10.1021/je300655b © 2012 Am. Chem. Soc.

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WP2 Deep Dive Gravimetric Preparation of Gas Mixtures



 Preparation of gas mixtures that qualify as reference materials (according to ISO 6142-1)

- Pre-treatment of cylinders > filling station direct filling, small cylinder, liquid injection
 > use of pre-mixtures > fine weighing
- Mixture validation by process-GC
- Bracketing method according to ISO 12963



Table 1: Proposed gas mixtures within MefHySto





Figure 2: Experimental stand for filling gas cylinders, liquid injection



Figure 3: mechanical balance Voland HCE 25: 25 kg ± 15 mg



Figure 4: Multichannel process GC (12 TC detectors, one single isothermal method (t = const. = 60 °C), 40 min run)

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(0.95 H2, 0.05 C3H8) from AGA8-DC92 EoS calculated values

WP2 Deep Dive Gravimetric Preparation of Gas Mixtures

- A new spherical microwave resonator has been built for high precision measurements of thermophysical properties of hydrogen
- A reference humidity generator and reference hygrometer were set up
- Density measurements over a large *p*,*T*-region using a single-sinker densimeter with magnetic suspension coupling





Figure 5: Magnetic suspension coupling allows the measurement of the apparent sinker mass without any contact between the balance and the sinker. This ensures high accuracy





WP 4: Metrology for Reversible Hydrogen Storage Technologies – (a) Metal Hydride Storage



- Dynamic Methods for Adsorption Capacity
- Storage Key Performance Indicators . Tasks
- Assess and harmonize key performance indicators for H₂ adsorption using dynamic methods
- Develop dynamic methods for H₂ ad/absorption capacity and influence of pollutants
- Develop reference materials and reference methods for H₂ ad/absorption capacity



Storage Key Performance Indicators

- The dynamic methods for H₂ adsorption need to be harmonised, e.g., using reference materials.
- Metal hydride AB5 alloy was purchased and verified
- Interlaboratory comparisons ongoing

Effect of Impurities

- The selection of pollutants was based on ISO 14687-2-2019 (PEM fuel cell specs)
- Most important impurities for metal hydride hydrogen compression process are O_2 and water vapour from electrolysis, or traces of CO₂ and CO from carbonaceous feedstock



Figure 1: Metal Hydride Storage Container @MAHYTEC



Figure 2: Experimental bench on the effect of some pollutant in hydrogen on

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WP 4: Metrology for Reversible Hydrogen Storage Technologies – (b) Cryo Storage in Metal-Organic Frameworks



- Dynamic Methods for Adsorption Capacity
- Storage Key Performance Indicators Tasks
- Assess and harmonize key performance indicators for H₂ adsorption using dynamic methods
- Develop dynamic methods for H₂ ad/absorption capacity and influence of pollutants
- Develop reference materials and reference methods for H₂ ad/absorption capacity





Storage Key Performance Indicators

- Despite two decades of research, the dynamic methods for H₂ adsorption need to be harmonised, e.g., using reference materials
- Metal-organic framework (MOF) material ZIF-8 (Zn and 2methylimidazole) was selected, purchased and verified [1]

[1] R. Balderas-Xicohtencatl, J. A. Villajos, et al., ZIF-8 Pellets as a Robust Material for Hydrogen Cryo-

Adsorption Tanks, ACS Applied Energy Materials - DOI: 10.1021/acsaem.2c03719

 Interlaboratory comparisons almost completed



Figure 1: Cryo-adsorption labs @BAM and @MPI



Figure 2: First gram scale batches of MOF Reference Material produced



- WP 4 Deep Dive Cryo Storage in MOFs – ZIF-8 Reference Material [1]
- ZIF-8 was mechanochemically synthesized using a reactive extrusion process (Fig. 3) allowing the scale of the synthesis of the material to large quantities (Fig. 4)
- PXRD confirms that the powders and pellets are composed completely of ZIF-8 crystalline phase
- The pellets maintain the ZIF-8 phase with high crystallinity
- The pellets show higher uptake at p/p0 values ca. 0.01 compared to powder, indicating a slightly higher BET area (ca. 2%, see Table 1).

Sample	$A_{\rm BET}/{ m m}^2~{ m g}^{-1}$	$V_{\rm p}^{\ a}/{\rm cm}^3 {\rm g}^{-1}$	С	Table 1: Textural
ZIF-8 Powder	1115	0.54	3634	Properties of the Powder and
ZIF-8 Pellets	1142	0.55	3550	Pelletized ZIF-8
Cycled ZIF-8 Pellets	1128	0.54	3553	
^{<i>a</i>} Total pore volume at <i>p</i>	$p/p_0 = 0.9.$			[1] R. Baldera

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Figure 3: Lab Twin-Screw Extruder @BAM

Figure 5: X-ray diffraction of the material RM-1 in powder, compared to the calculated X-ray diffraction pattern for the theoretical crystalline framework

pelletised MOF RM [1]



Adsorption Tanks, ACS Applied Energy Materials - DOI: 10.1021/acsaem.2c03719





340 360 6 320 STP 300 320 / cm³

- The H₂ adsorption uptake at 77 K was measured with high accuracy considering the amount of analyzed sample, gas purity, activation conditions, and calibration procedures.
- The relative standard deviation (RSD) of 14 independent H₂ measurements is < 2 % at any pressure (up to 100 bar), indicating excellent reproducibility between the used analyzers.
- The operation stability of the material was demonstrated after 47 adsorption-desorption cycles



0.6

0.01

P/P_o

0.4

Powder ZIF-8

0.1

- Cycled Pellet ZIF-8

0.8

1.0

Pellet ZIF-8



Figure 7: Usable capacity of pelletized material ZIF-8 calculated for a TSA cycle from 77 K -100 bar and 117 K - 5 bar

> iterlaboratory comparison of hydroge adsorption of a reference mate

> > Protocol and Report Form

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Michael Maintald, Jose A. Vilhio

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30 Ŀ gH_2 Total uptake / 20 — 77 K 🗕 87 K 🗕 97 K - 107 K 🔶 117 K Ω 20 40 60 80 100 Pressure / bar

ZIF-8 material characterisation for protocol

100

360

280

260

240

220

0.0

280

240

0.2

0.001

WP 4 Deep Dive Cryo Storage in MOFs – Reference Material and Comparisons



WP5 Large-Scale Storage of Gases in Geological Storage Facilities



- Measurement Technology
- **CFD** Simulations
- Flow Metering

Tasks

- Determination of the requirements for the . measurement technology
- Measurement of storage-relevant . impurities (blanket, glycols) and traces of hydrocarbons in hydrogen
- Flow metering in gas mixtures (20/80; 50/50; 80/20)



- Course of the changeover of UGS from natural gas to hydrogen varies depending on the type of the storage
- In caverns a changeover to high H₂-contents can be achieved quickly
- Pore storage tanks must be converted over long periods of time
- Analytical requirements compiled through expert statement by underground storage operators





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WP5 Deep Dive Type of Underground Gas Storages (UGS) – Salt Caverns

- Caverns are artificial chambers created (drilled)
- within salt domes or bedded salt deposits
- in a thick cylindrical pit by controlled freshwater injection from the surface down to the deposits
- AKA Solution mining

Salt cavern schematic showing different dissolution setups [1, 2]



 Muhammed, N. S., et al. A review on underground hydrogen storage: Insight into geological sites, influencing factors and future outlook, Energy Reports 8 (2022) 461–499 https://doi.org/10.1016/j.egyr.2021.12.002
 Wallace, R.L., Cai, Z., Zhang, H., Zhang, K., Guo, C., 2021. Utility-scale subsurface hydrogen storage: UK perspectives and technology. Int. J. Hydrogen Energy 46, 25137–25159. <u>http://dx.doi.org/10.1016/j.ijhydene.2021.05.034</u>.



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Sources

 Muhammed, N. S., et al. A review on underground hydrogen storage: Insight into geological sites, influencing factors and future outlook, Energy Reports 8 (2022) 461–499 https://doi.org/10.1016/j.egyr.2021.12.002
 Wallace, R.L., Cai, Z., Zhang, H., Zhang, K., Guo, C., 2021. Utility-scale subsurface hydrogen storage: UK perspectives and technology. Int. J. Hydrogen Energy 46, 25137–25159. http://dx.doi.org/10.1016/j.ijhydene.2021.05.034. Aquifer schematic (a) before H_2 injection, and (b) after H_2 injection [1, 2]

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- An aquifer is a subsurface layer of permeable and porous rock
- It is filled with fresh or saline water
- Often hundreds of feet deep
- In the absence of caverns and depleted hydrocarbon reservoirs, they are primarily utilized for UHS



WP5 Deep Dive Type of UGS – Depleted Oil and Gas Reservoirs



- Depleted reservoir is the conventional storage means for natural gas
- Porous and permeable hydrocarbon reservoir
- Located thousands of feet beneath the subsurface
- Can be considered as a portion of an aquifer (geological traps)

An overview of a depleted reservoir condition (a), microscopic image of reservoir pores (b, c) [1, 2



Sources

 Muhammed, N. S., et al. A review on underground hydrogen storage: Insight into geological sites, influencing factors and future outlook, Energy Reports 8 (2022) 461–499 https://doi.org/10.1016/j.egyr.2021.12.002
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- Globally, successful H₂ storage in underground geological formations is limited to the caverns and aquifers
- Sites become interesting in a favorable area near renewable energy generation plants

Salt caverns					
Field name (s)	Country	Depth (m)	Volume (m ³)	Operating conditions	H ₂ (%)
Teesside	UK	400	$3 \times 70,000$ each	45 bars	95
Clemens	USA	1,000	580,000	70–137 bars	95
Moss Bluff	USA	1,200	566,000	55–152 bars	95
Kiel	Germany	_	32,000	100 bars	60
Aquifers					
Field name (s)	Country	Depth (m)	Volume (m ³)	Operating conditions	H ₂ (%)
Lobodice	Czech Republic	400-500	_	90 bars/34 °C	54
Beynes	France	430	3.3×10^{8}	-	50
Ketzin	Germany	200-250	-	-	62
				Table:	Worldwide

Sources

Muhammed, N. S., et al. A review on underground hydrogen storage: Insight into geological sites, influencing factors and future outlook, Energy Reports 8 (2022) 461–499 https://doi.org/10.1016/j.egyr.2021.12.002
 Liebscher, A., Wackerl, J., Streibel, M., 2016. Geologic storage of hydrogen - fundamentals, processing, and projects. In: Hydrog. Sci. Eng. Mater. Process. Syst. Technol., Vol. 2. Wiley-VCH Verlag GmbH & Co. KGaA, Weinheim, Germany, pp. 629–658.

http://dx.doi.org/10.1002/9783527674268.ch26..

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successful H_2 storage sites [1, 3]

WP5 Deep Dive Capacities and H₂ Storage Requirements

- Storage capacity in UGS in Germany approx.
 24 Bn. m³ of gas
- H₂ only 16,5 Bn. m³
 - approx. 7 Bn. m³ of pore storage facilities
 - approx. 9.5 Bn. m³ caverns
- Extrapolated capacity for H₂ with 3.54 kWh/m³
 - 57 TWh
 - approx. 34 TWh in caverns
- H₂ storage demands
 - BMWK (2021) 2050: 47–73 TWh
 - Guidehouse (2021) 2050: 110 TWh

https://erdgasspeicher.de/erdgasspeicher/gasspeicherkapazitaeten/





Hinweis: Die Angaben der Kapazitäten beziehen sich auf das in Betrieb befindliche Arbeitsgasvolumen der Gasspeicher.

Source:

Quelle: LBEG





- Conversion from natural gas to H₂ without any problems for new caverns
- All planned projects with pure H₂ in Germany are cavern projects
- Primarily caverns that have not previously been used for natural gas
- Brine is directly displaced by H₂
- Impurities
 - Water
 - Blankett (traces of hydrocarbons)
 - Sulphur compounds
- Can be achieved quickly

- Conversion of used caverns Traces of gas could then be detectable in the hydrogen
- Microbiological processes are possible in brine, but not safe – metrological monitoring

Source	new cavern	converted cavern (from oil/gas)
H ₂ S (Microbiology)	Х	Х
CO ₂ (Microbiology)	Х	х
H ₂ O	Х	Х
Blankett (e.g. Diesel)	(X)	Х
Rests of oil / gas	-	Х

WP5 Deep Dive Pore Storages (Former Oil and Gas Fields, Aquifers) – Conversion

- Pore storage widely used in Germany and other European countries
- Problem: filled with natural gas
- New storage facilities conceivable, but not very likely
- Contaminants
 - Methane and other hydrocarbons (residual natural gas)
 - Water
 - Sulphur compounds (e.g., odorants in French storage facilities)
- Gradual changeover over long periods of time expected
- H₂ cavern possibly in 2026 (relevant order of magnitude)



Understanding of Geological Hydrogen Storage

Ind. Eng. Chem. Res. 2022, 61, 9, 3233-3253,



Metrology for Advanced

WP5 Deep Dive Measurement Requiremets

Component / substance group	measureme fo	ent relevant or	limit*					
	NG/H₂ - mixtures	pure hydrogen	natural gas	Mixture	Hydrogen 1	Hydrogen 2		
					≥98 Mol%	≥99,97 Mol%		
Methanol	(x)	(x)						
higher Hydrocarbon Blanket		х						
higher Hydrocarbon Compressor		x				2 ppm		
Glycol		x						
O ₂	x	x	10 ppm	10 ppm	10 ppm	5 ppm		
L	×	Y	0 - 20 Mol	1 - 98 Mol	2 - 20 Mol	Measurement		
112	^	^	%	%	%	rest		
H ₂ O	x	x	50 mg/m ³	50 mg/m ³	50 mg/m ³	5 ppm		
S total		х	6 mg/m ³	6 mg/m ³	6 mg/m ³	4 ppb		
H₂S + COS	x	x	5 mg/m ³	5 mg/m ³	5 mg/m ³	4 ppb		
Mercaptans	x	x	6 mg/m ³	6 mg/m ³	6 mg/m ³	4 ppb		
HC Downsint	v		<-2°C (0 - 70	<-2°C (0 - 70				
HC Dewpoint	x		bara)	bara)				
Hydrocarbon (trace)		x				2 ppm		
Methane (trace)		x				100 ppm		
СО		(x)				0,2 ppm		
NH _{3 /} Amins	(x)		$< 10 \text{ mg/m}^3$	< 10 mg/m ³	< 10 mg/m ³	0,1 ppm		
Halogens		(x) ?			≤ 0,05 ppm	≤ 0,05 ppm		
Dust,			technical free	technical free	technical free	1 mg/m³		

Hydrogen Qualities currently discussed (*based on expert discussion)

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Metrology for Advanced Hydrogen Storage Solutions

Technical requirements	9	arresian							cardinume's			22 ppm	20урт		S paper
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More Information

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Summary



- EU renewable energy target cannot be reached without advanced energy storage solutions
- Hydrogen as new secondary energy carrier
- Development of swift analytical methods
- Development of certified reference materials
- Thermophysical Properties of H₂ and H₂ enriched natural gas → extending Equations of State
- Save the Date: Final Conference and Workshops:
 03.–05. July 2023, Berlin/Germany
- MefHySto.eu









Thank you

Michael Maiwald, BAM and the Consortium



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EMPIR 19ENG03



MefHySto – Introduction Project Landscape Towards H₂ Readiness and a Rapid Market Launch

Metrology for Advanced Hydrogen Storage Solutions

MefHySt



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