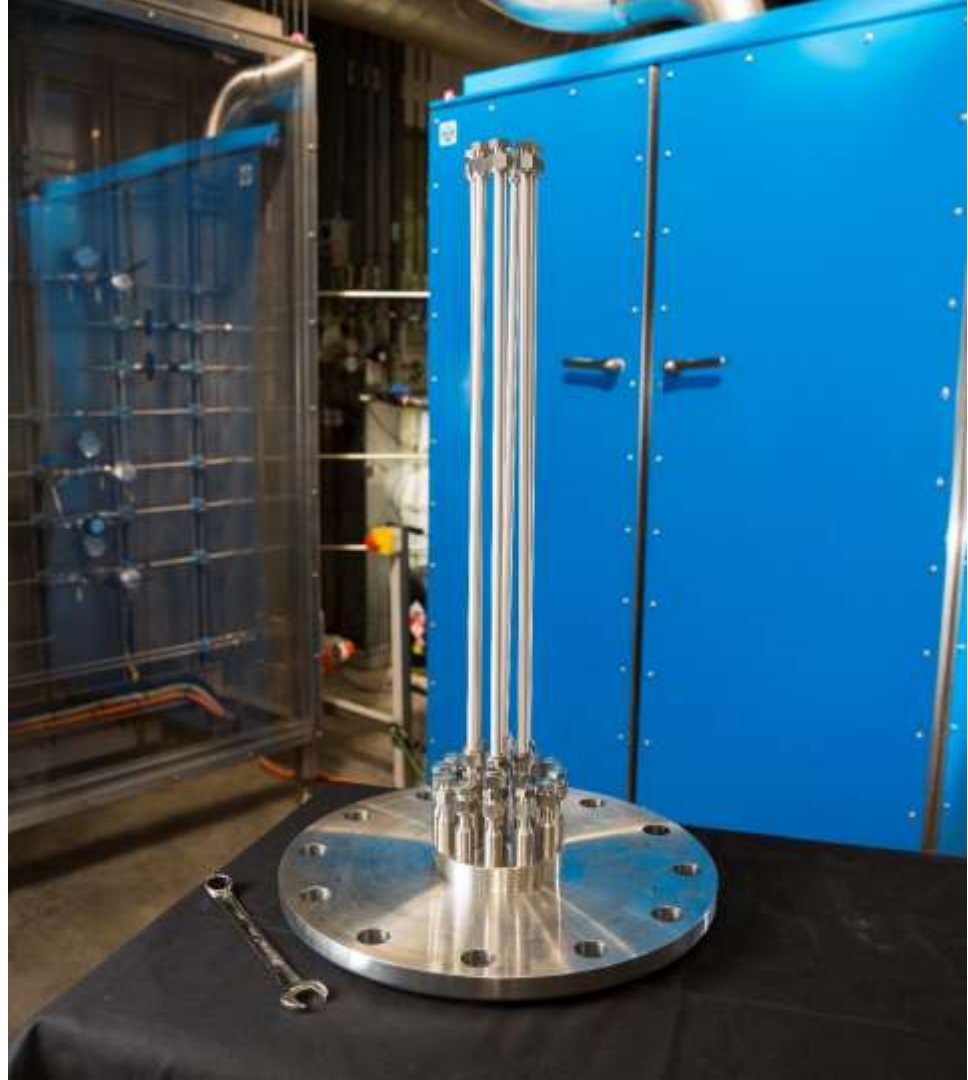




# CSIRO's Metal Membrane Technology (MMT) – *Technical Update*

Ammonia Energy Association, 5<sup>th</sup> APAC  
Conference | 16-18 August 2023 |  
Newcastle, Australia

CSIRO Energy – Dr David S. Wong  
Fortescue – Dr Michael Dolan





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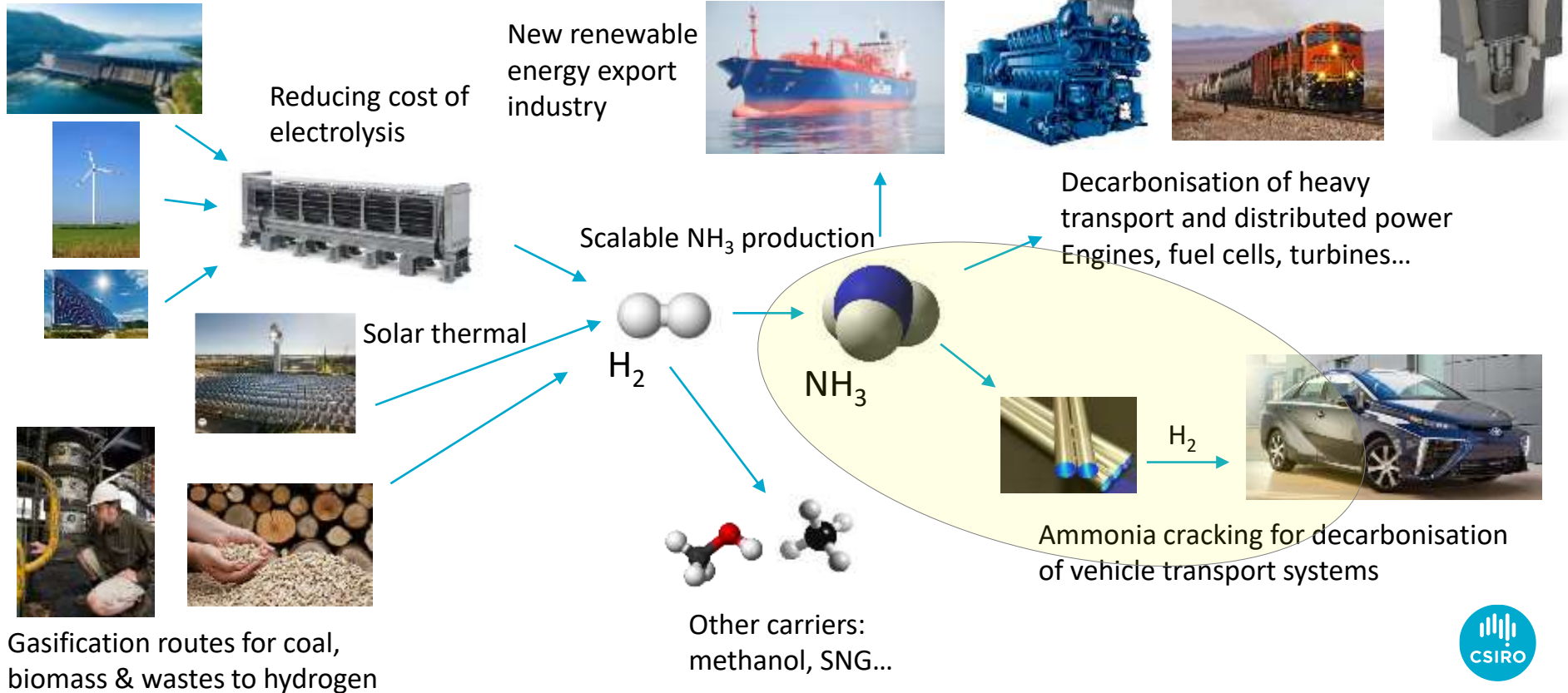
## CSIRO – MMT technical update

- Background to the technology and 2018 demonstration
- Scaling up of manufacturing since then
- Preserving H<sub>2</sub> product purity, using latest seal technology
- 40 kg/day scale pilot – 2022
- 200 kg/day scale pilot – current focus

## Fortescue – MMT commercial update

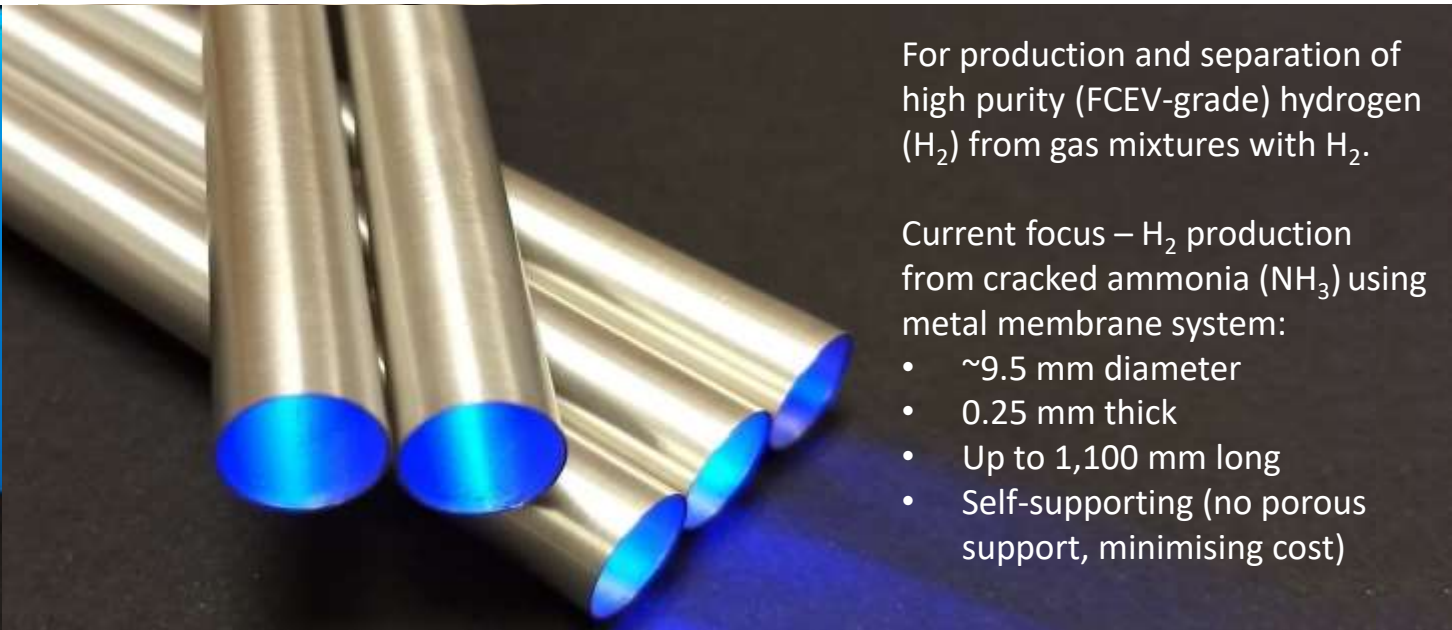
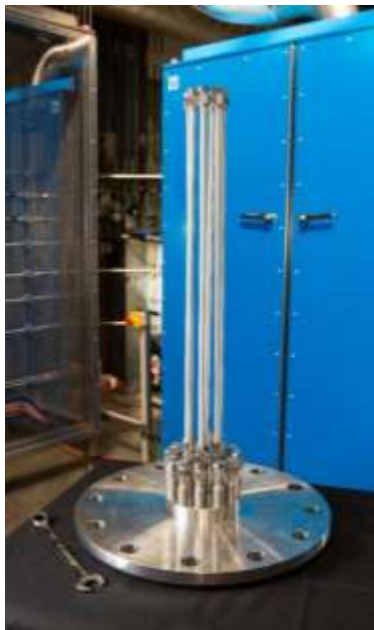
# Ammonia as a hydrogen carrier

Building on existing industries and infrastructure





# CSIRO's Metal Membrane Technology (MMT)



For production and separation of high purity (FCEV-grade) hydrogen ( $H_2$ ) from gas mixtures with  $H_2$ .

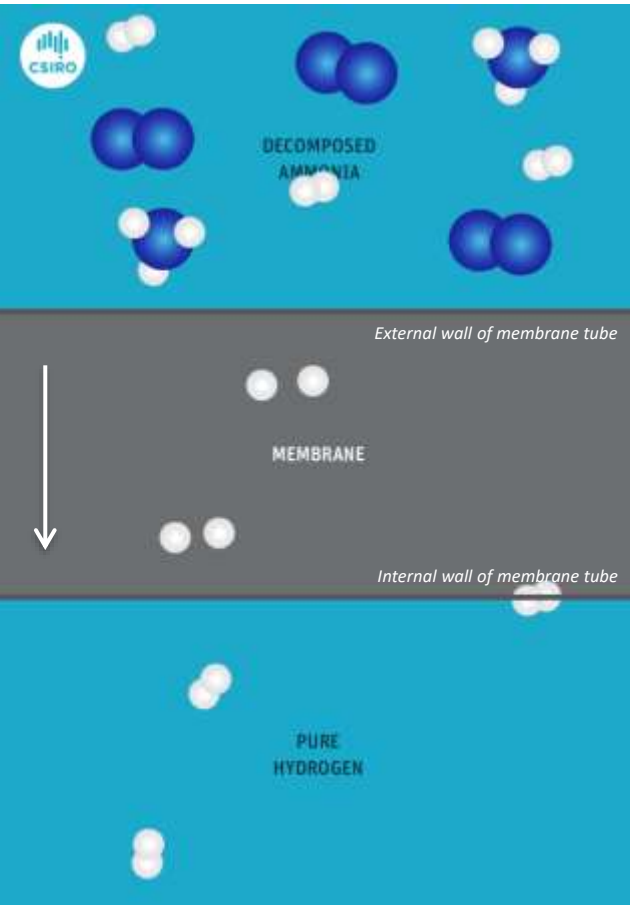
Current focus –  $H_2$  production from cracked ammonia ( $NH_3$ ) using metal membrane system:

- ~9.5 mm diameter
- 0.25 mm thick
- Up to 1,100 mm long
- Self-supporting (no porous support, minimising cost)

## $NH_3$ -to- $H_2$ System Demonstrations

- 5 kg/day proof-of-concept scale – demonstrated in Brisbane, 2018
- 40 kg/day pilot scale – completed 2022
- 200 kg/day pilot demonstration scale up – current focus, UK 2023-24

# Vanadium-based membranes for H<sub>2</sub> purification



**High pressure +  
Mixed Gas Stream**  
(NH<sub>3</sub>, N<sub>2</sub>, H<sub>2</sub>)

← **Feed-side catalyst (Pd)**

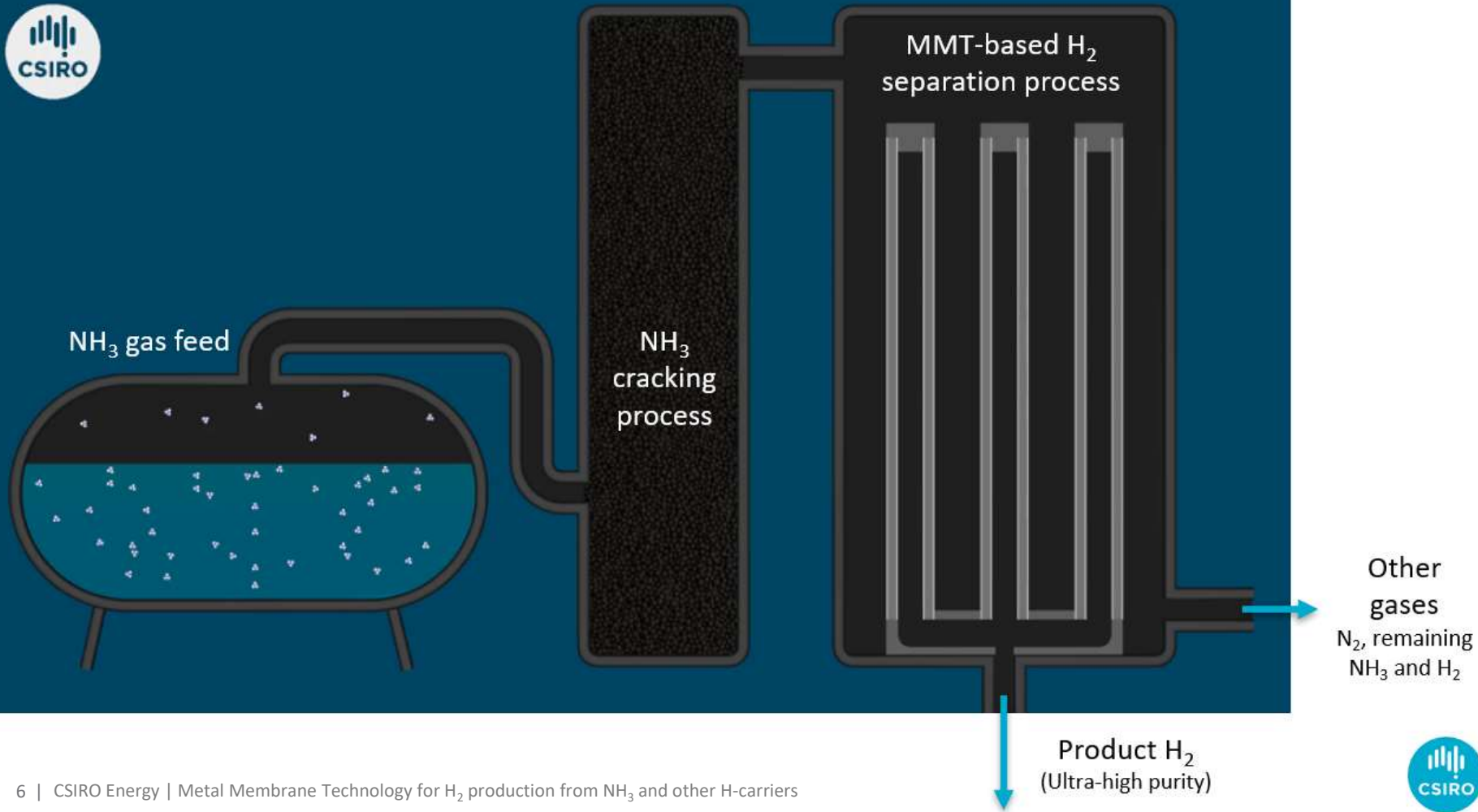
**V or V-alloy core**

← **Permeate-side catalyst (Pd)**

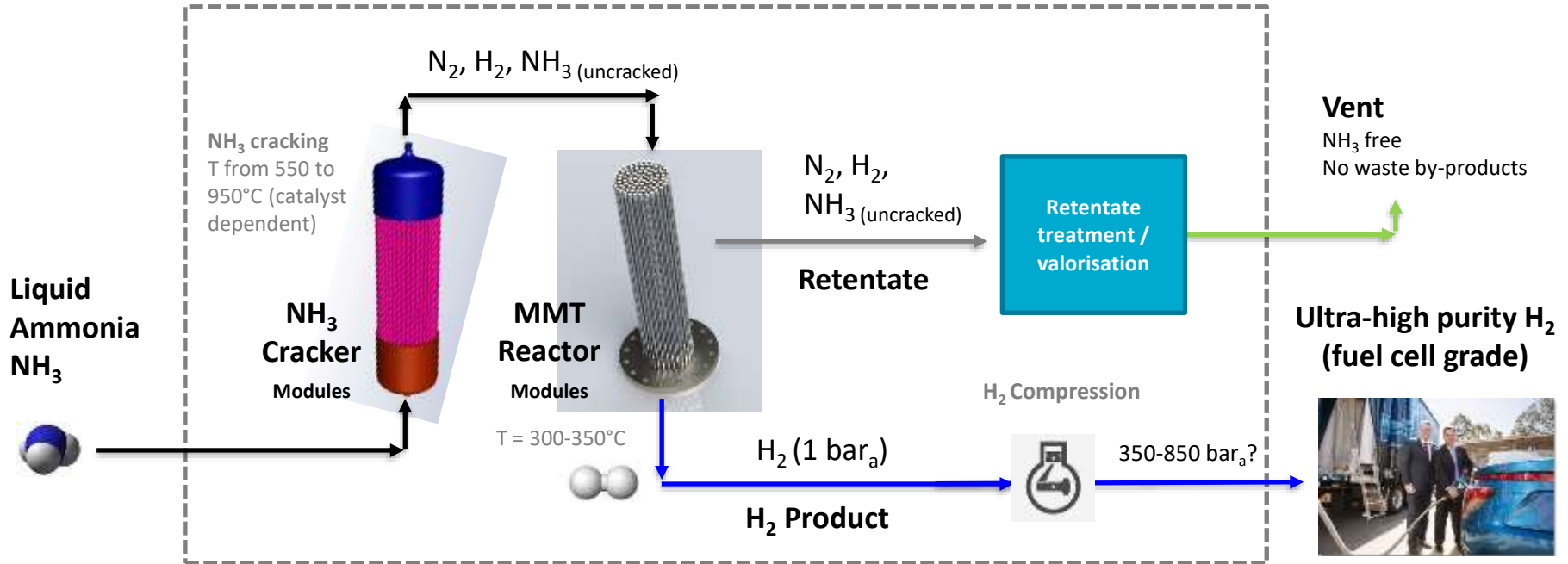
**Low pressure +  
Ultra-Pure H<sub>2</sub>**

Our design philosophy:

- Minimise materials costs (minimise use of palladium)
- Use scalable manufacturing techniques (metal tube extrusion and electroplating)
- Prioritise purity over flux (to meet ISO-14687 for PEM fuel cells)



# Example MMT-based NH<sub>3</sub>-to-H<sub>2</sub> System



# Brisbane 2018 Proof-of-Concept Demonstration



World-first demonstration of FCEV-refuelling using  $\text{NH}_3$ -derived  $\text{H}_2$  in 2018



2018 proof-of-concept  $\text{NH}_3$ -cracking and  $\text{H}_2$  separation system



Metal membrane assembly



# Brisbane 2018 Proof-of-Concept Demonstration



SCIENCE AND  
INDUSTRY  
ENDOWMENT  
FUND

Supported by BOC, Toyota, Hyundai

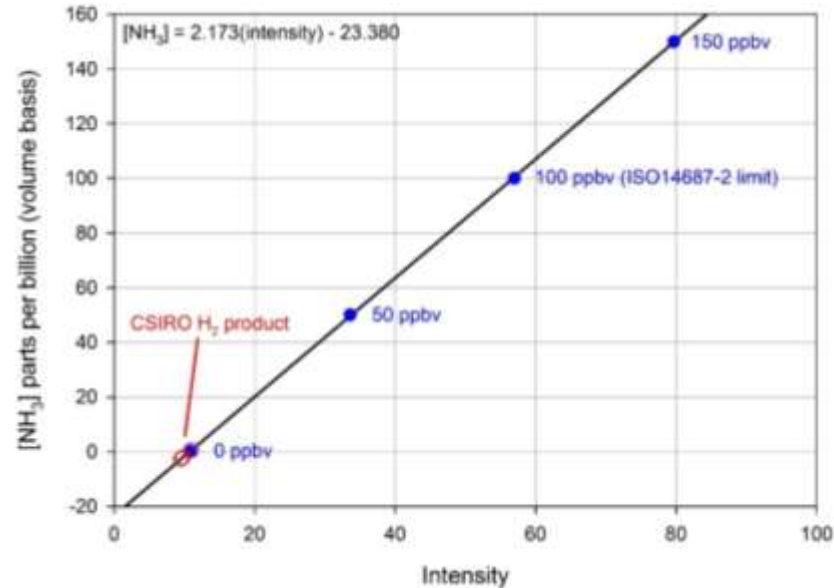


# Hydrogen purity analysis

H<sub>2</sub> purity for FCEV use  
specified by ISO 14687-02



Trailer-mounted SYFT Voice 200  
Secondary Ion Flow Tube Mass  
Spectrometer (SIFT-MS)

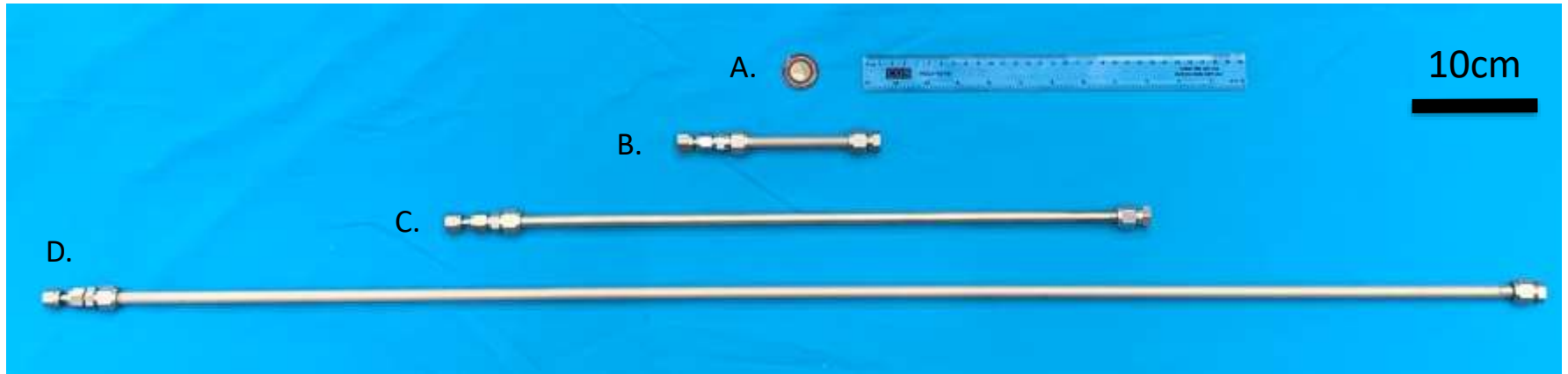


- Calibrated against 4.7ppmv certified NH<sub>3</sub>-in-H<sub>2</sub> mixture supplied by BOC, diluted on-demand with UHP-H<sub>2</sub>
- Detection limit << 10 ppbv
- SIFT-MS not suitable for online field applications: FTIR is best option



# Technology Updates since 2018

# Scaling up the manufacturing – form factor, quantity and quality



A. 2012-13 – planar membranes, 20c sized coin

B. 2013-17 – 100mm tubular membranes

C. 2017-18 – 350-500mm tubular membranes

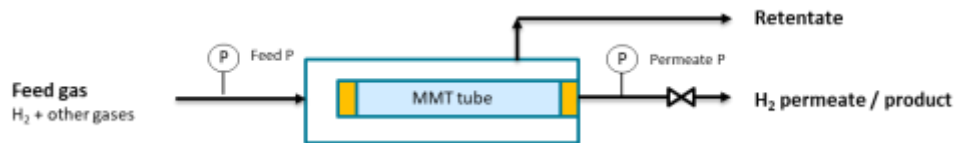
D. 2019-current – 1100mm tubular membranes, with greater scale and quality control

} ‘Bespoke’ manufacturing

→ Automated manufacturing

# Quality control

- Automation providing greater reproducibility, repeatability and quality control
- Monitoring of performance (e.g. H<sub>2</sub> permeability) over production batches



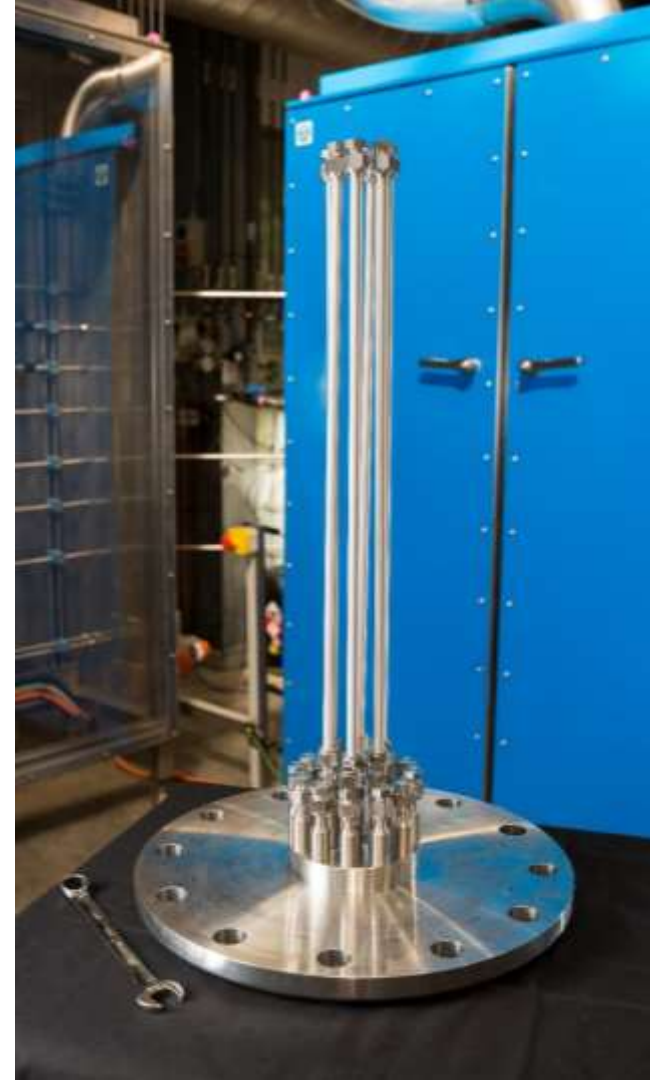
Membrane performance (H<sub>2</sub> permeability) vs. membrane no.





# Advances in robustness

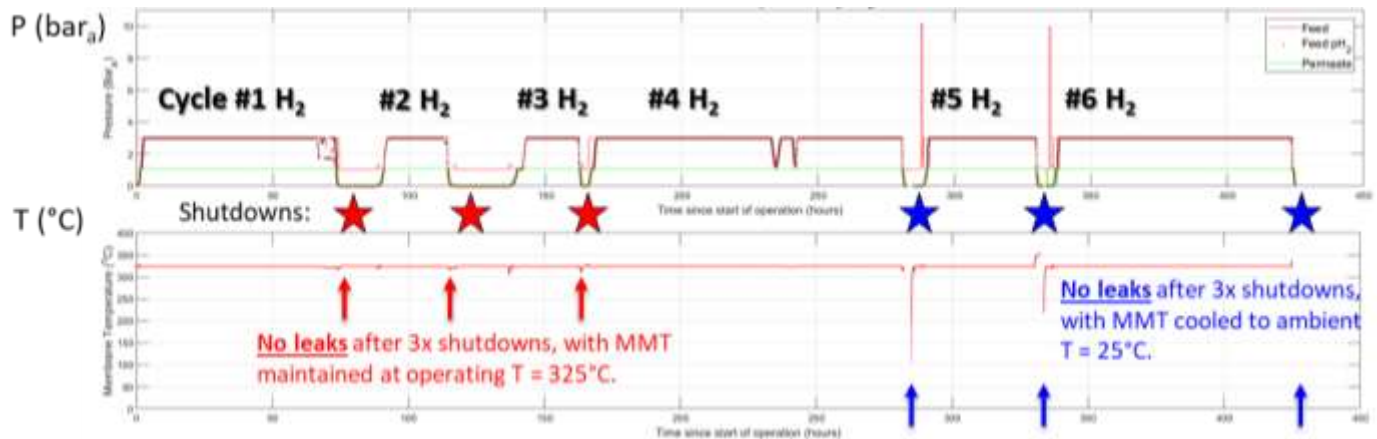
- Previous compression seals a challenge
- Latest seal technology (patents pending) provide additional robustness to the technology – importantly, preserving H<sub>2</sub> purity over cycles of operation



# Advances in robustness

Seal integrity maintained – vacuum pressure held on membrane permeate side – after multiple cycles of H<sub>2</sub> operations, including shutdown + cooling to ambient.

*Multiple cycles of H<sub>2</sub> operations on lab-scale system, with latest seals (patent pending)*





# Supply chain

An important factor for all new technologies

COVID-19

Ukraine conflict early 2022



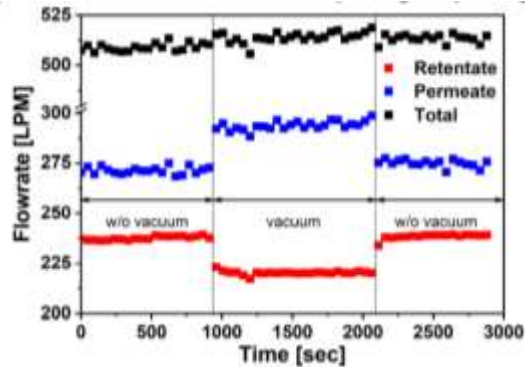
<https://www.kitco.com/charts/livepalladium.html>





# 40 kg/day pilot trial

- Consortium with CSIRO, FFI and partners
- Successful trial in 2022 with 40kg/day pilot ammonia cracker + MMT purification module
- H<sub>2</sub> produced with no N<sub>2</sub> / NH<sub>3</sub> leaks (per GC / FTIR analysis)

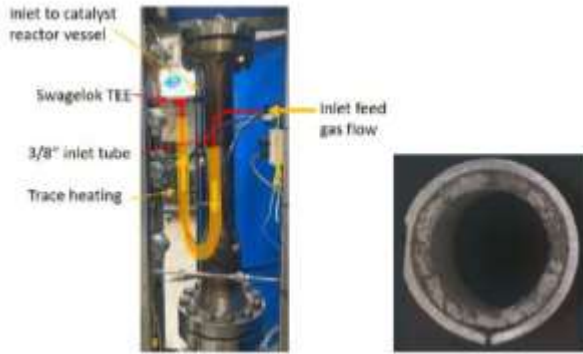


# Siemens Energy pilot – 200 kg/day

- Siemens Energy led consortium – including Geopura, Fortescue and CSIRO
- Ammonia cracker system prototype, designed to produce fuel-cell grade  $H_2$  from green  $NH_3$  via MMT purification module
- Testing two 100 kg/day modules
- Commissioning end-2023 / early-2024



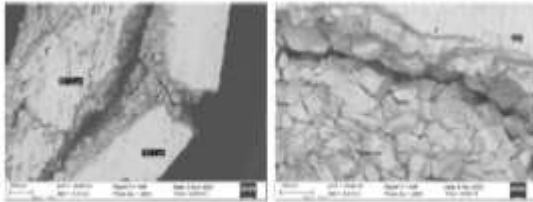
# Safety Share – Nitriding by high temperature NH<sub>3</sub>



Failed tube location



Cross-section



SEM imagery at failure

## What happened?

- In 2022, minor NH<sub>3</sub> leak (ppm levels) in a ventilated ammonia-to-hydrogen pilot system. Triggered gas detection systems and safe shutdown of the plant. No exposures/injuries.
- **SS316 tube failed due to severe nitriding attack** after 2000 hours operation, under high temperature (>450°C) and ammonia partial pressures (pure NH<sub>3</sub>, 10 bar<sub>g</sub>)

## What we learnt?

- **Carbon steel and stainless steels highly susceptible to nitride attack > 300°C, and particularly with NH<sub>3</sub> gas concentrations >30%**, worsened through thermal cycles.
- Nitriding can cause embrittlement and tube failure if severe enough. Rate of nitriding here was ~2-3 mm/year, higher than expected in literature (likely due to thermal cycling).
- Ammonia industry manages this with appropriate metallurgy / materials selection, plant design, condition monitoring + risk based inspection, and can achieve >20y service lifetimes.

## Recommendations – particularly to R&D community in the ammonia space:

- Avoid nitriding conditions in plant design, and avoid thermal cycling / shock
- In severe nitriding conditions (>30% NH<sub>3</sub>, T >300°C), use >50% Ni (e.g. Alloy600) or Ni-Co alloys
- Best practice – engage industry practitioners (ammonia industry) on materials selection
- Perform regular inspections of areas at risk of nitriding

# Towards commercialisation



Fortescue™



- Fortescue is the commercialisation partner and exclusive licensee of CSIRO's Metal Membrane Technology (MMT).
- Next scale up at 200 kg/day pilot demonstration
- Whilst focus is on FCEV-grade H<sub>2</sub> from cracked ammonia, MMT can be used for H<sub>2</sub> separation from other mixed gas feeds (\*noting limitations with specific impurities)



# Thank you

**CSIRO Energy**

Dr David S. Wong  
Team Leader & Project Manager  
Energy Technologies

David.Wong@csiro.au  
[csiro.au/energy](http://csiro.au/energy)