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 **Pietro
Fiorentini**

X Workshop Italiano Sol-Gel

Padova, 15-16 Settembre 2025

Gasochromic Tungsten Oxide Thin films for Hydrogen Monitoring in Natural Gas

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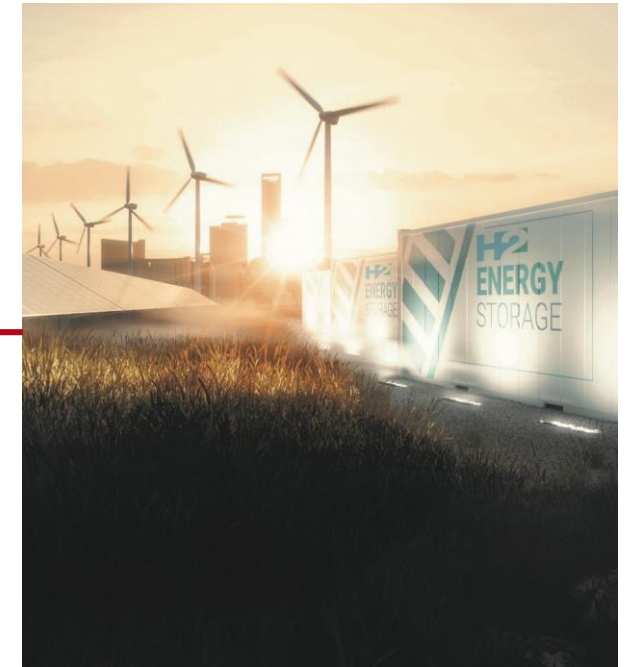


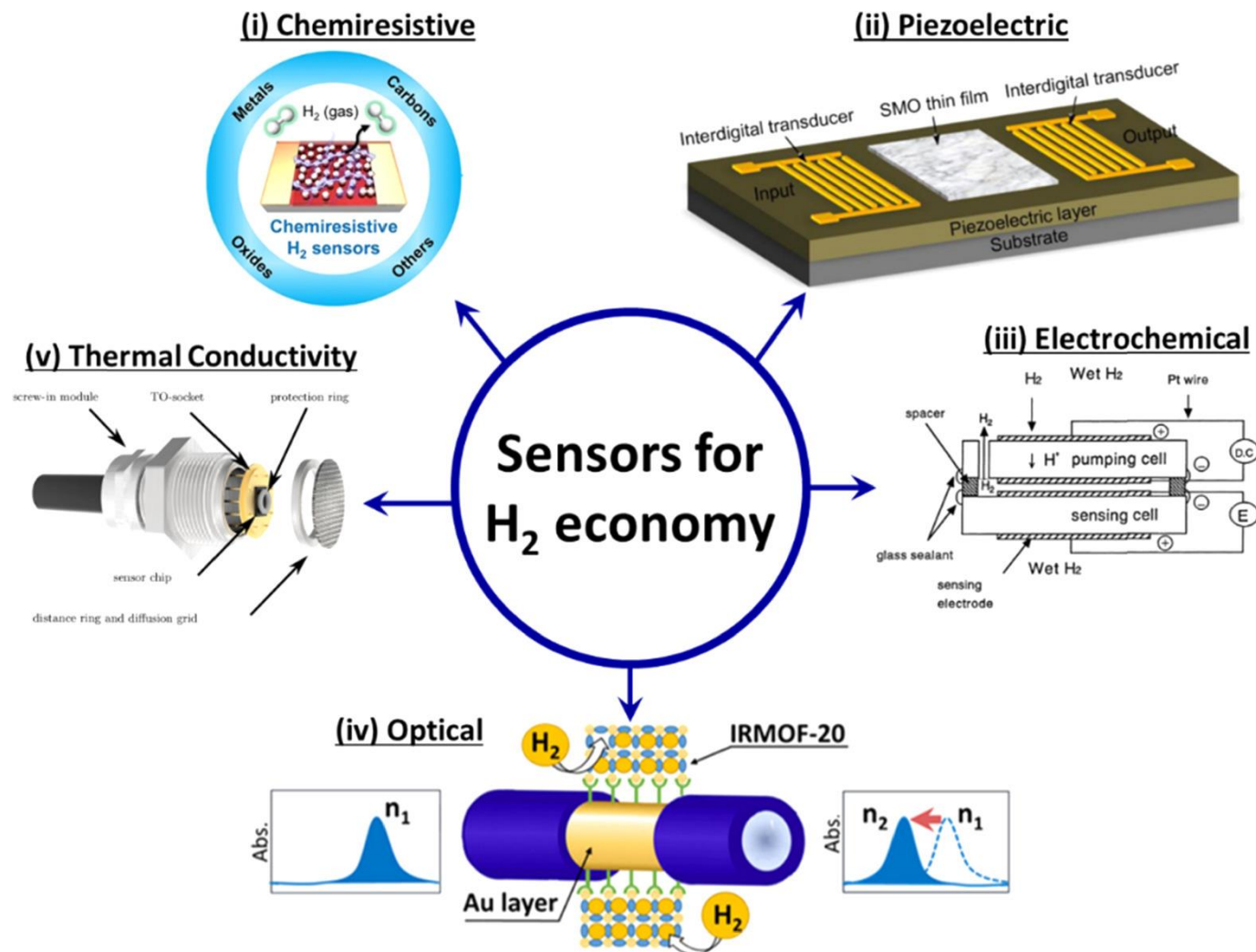
The present...

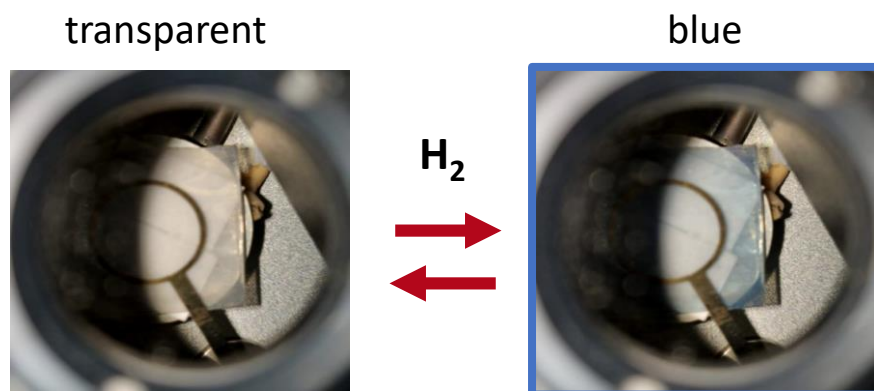
Many countries are pushing regulations to abate CO₂ emission and achieve a carbon neutral World by 2050.

.... And the future

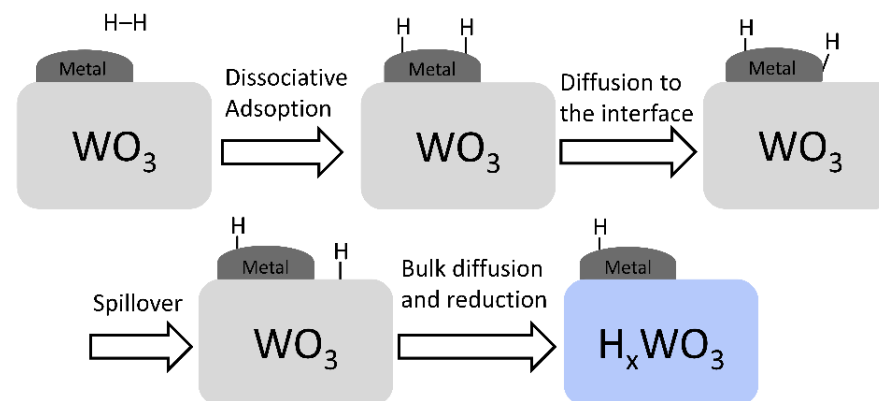
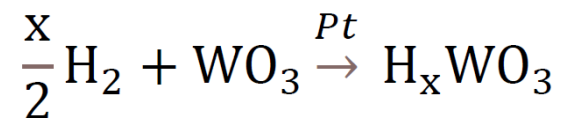
Blending hydrogen into existing natural gas pipelines is regarded as one of the most cost-effective approaches to reduce carbon emissions.



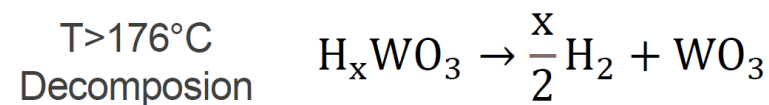
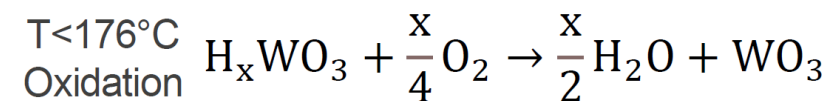




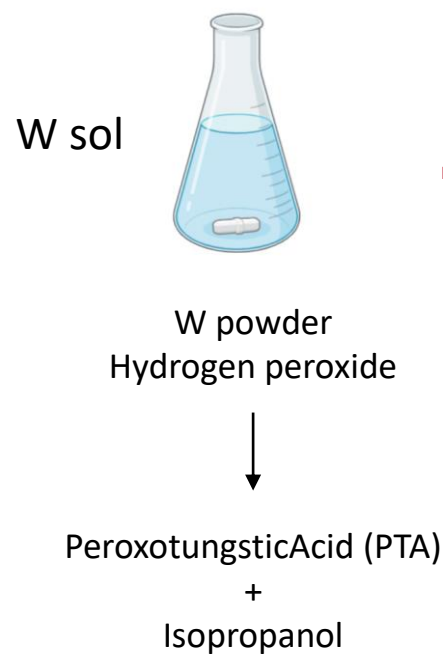
Coloring reaction



Bleaching reaction



Sol-gel synthesis



2h @ 60°C

Deposition and crystallization

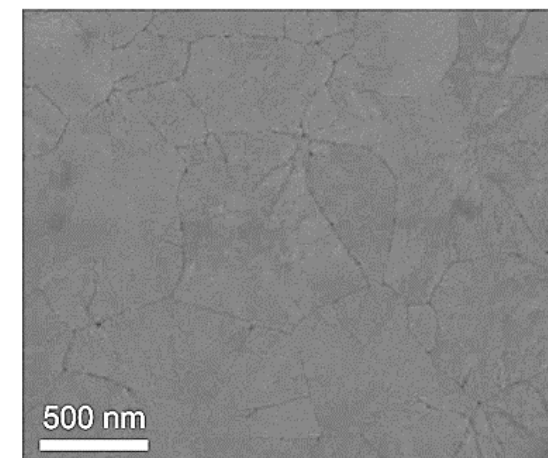
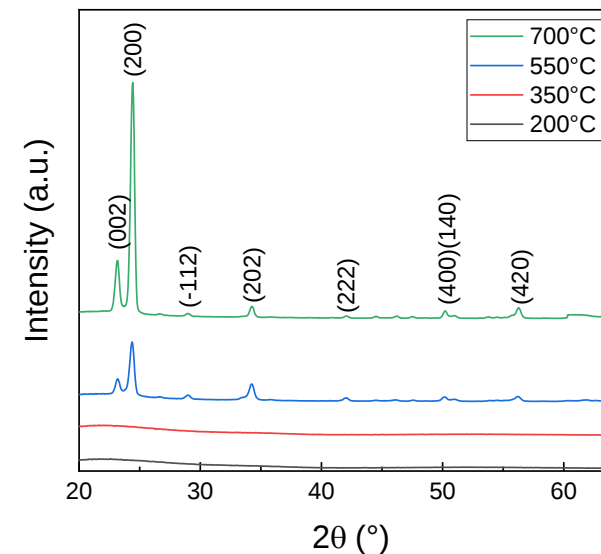


2500 rpm, 30 s
SiO₂ glass

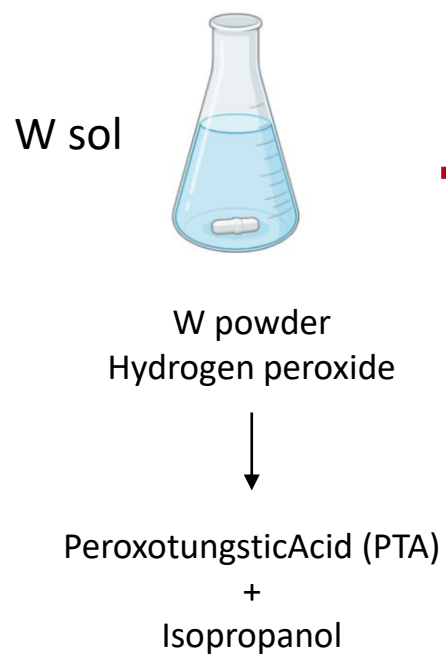
Thermal treatment

Monoclinic phase of WO₃
Film thickness: 40 ± 3 nm

JCPDS card no. 71-2141

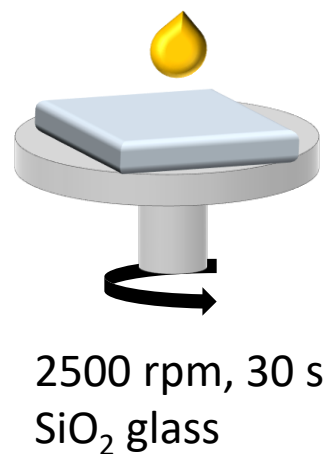


Sol-gel synthesis



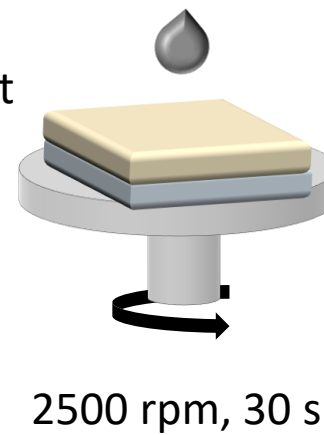
2h @ 60°C

Deposition and crystallization

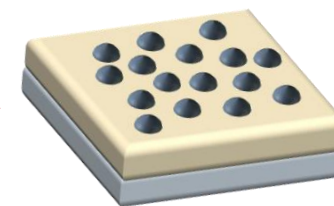


Thermal treatment

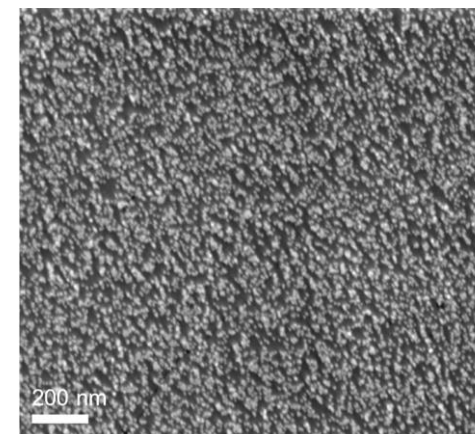
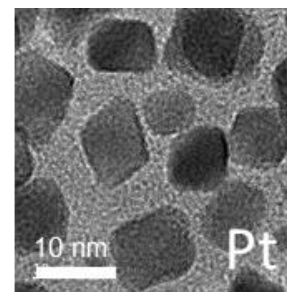
550°C

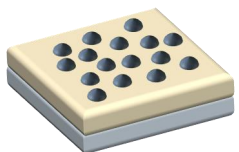


Pt nanoparticles

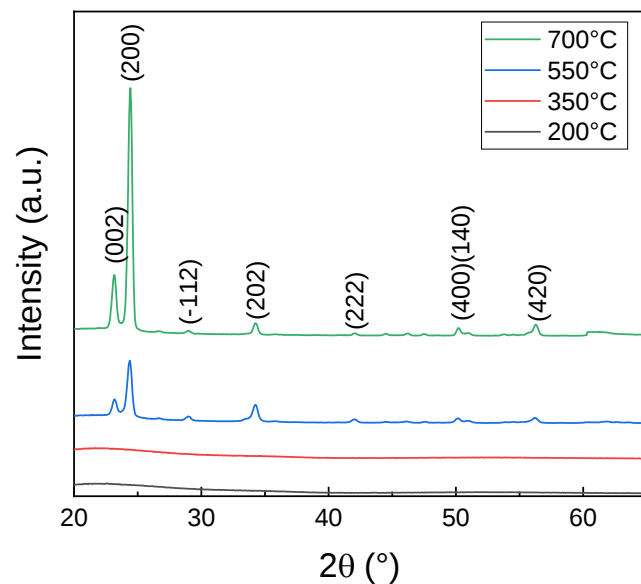


Pt NPs (polyol method, 10 nm)



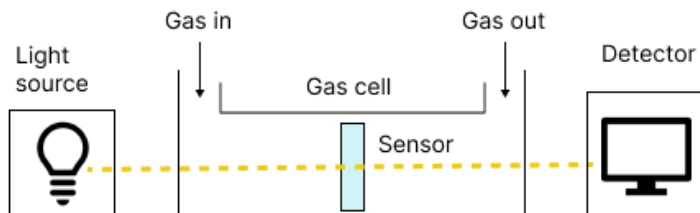


Pt/WO₃

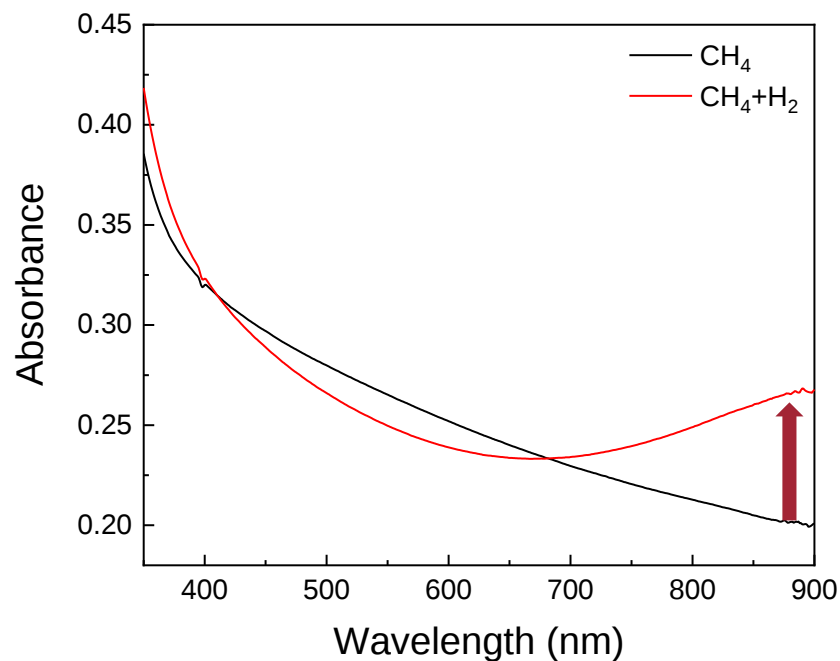


Sample	OT [°C]	Abs _{max}	τ_{color} [s]	τ_{bleach} [s] in Air	τ_{bleach} [s] in Ar	RP [%], in Air	RP [%], in Ar
Pt-200WO ₃	20	0.031	985	1178	/	90	25.7
	100	0.075	707	275	/	98	67.5
	250	0.207	490	/	/	83	22.7
Pt-350WO ₃	20	/	/	/	/	/	/
	100	0.049	816	/	/	74.5	20.0
	250	0.22	664	/	/	57.1	23.3
Pt-550WO ₃	20	0.016	1028	/	/	67	48.4
	100	0.040	584	567	/	94.5	64.6
	250	0.055	11	7	58	100	100
Pt-700WO ₃	20	0.011	998	/	/	87.6	57.4
	100	0.102	526	110	/	100	76.2
	250	0.050	20	6	57	100	100

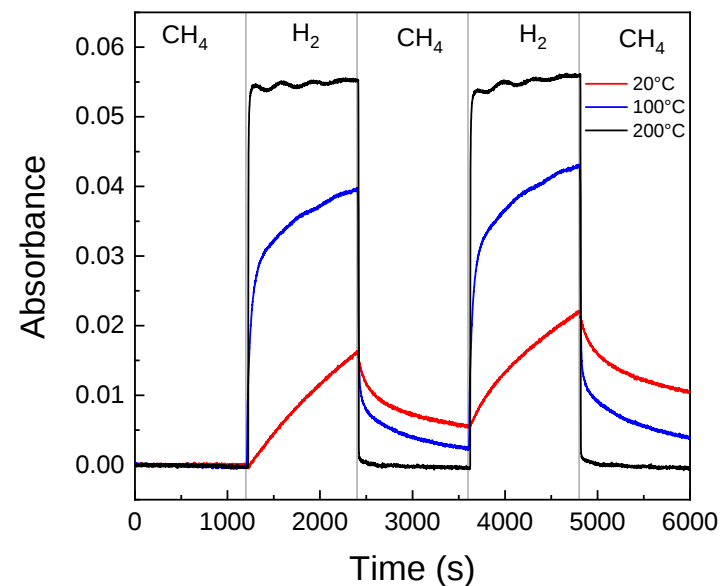
H₂ SENSING TEST



- UV-VIS Spectrophotometer
- Customize cell
- Gas flux, operating temperature (OT)



5% H₂ in CH₄; $\lambda=850\text{nm}$



ISO 26142:2010 –hydrogen detection apparatus

The International Standard sets out the requirements applicable to a sensor for hydrogen, such as precision, response time, stability, selectivity and poisoning.

$\tau_{\text{response}} < 30\text{s}$; $\tau_{\text{recovery}} < 60\text{ s}$

Sensing Performance

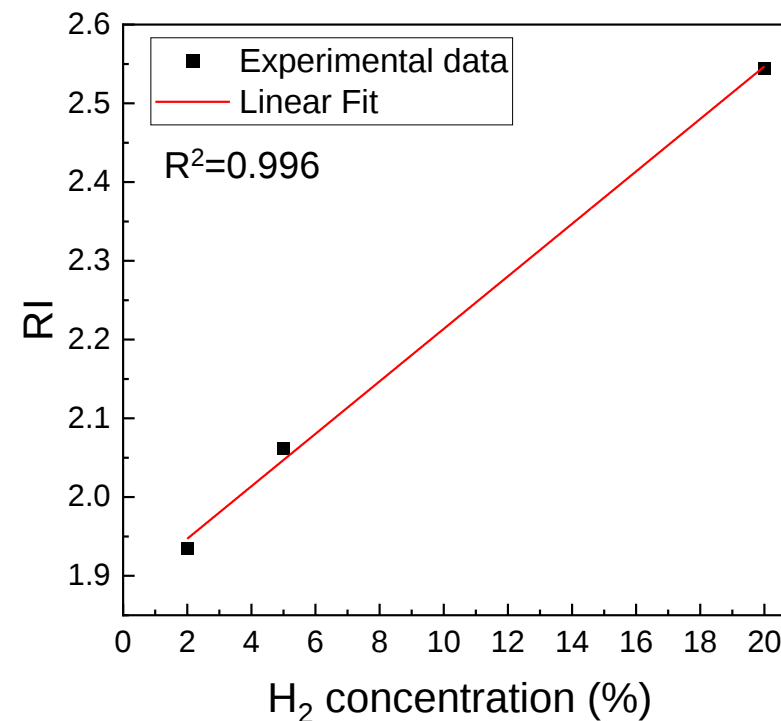
- Operating temperature 200°C
- Coloring time **11s**
Time to reach 90% of absorbance change
- Bleaching time **58s**
Time to return to within 10% of baseline

ISO 26142:2010 –hydrogen detection apparatus

The International Standard sets out the requirements applicable to a sensor for hydrogen, such as precision, response time, stability, selectivity and poisoning.

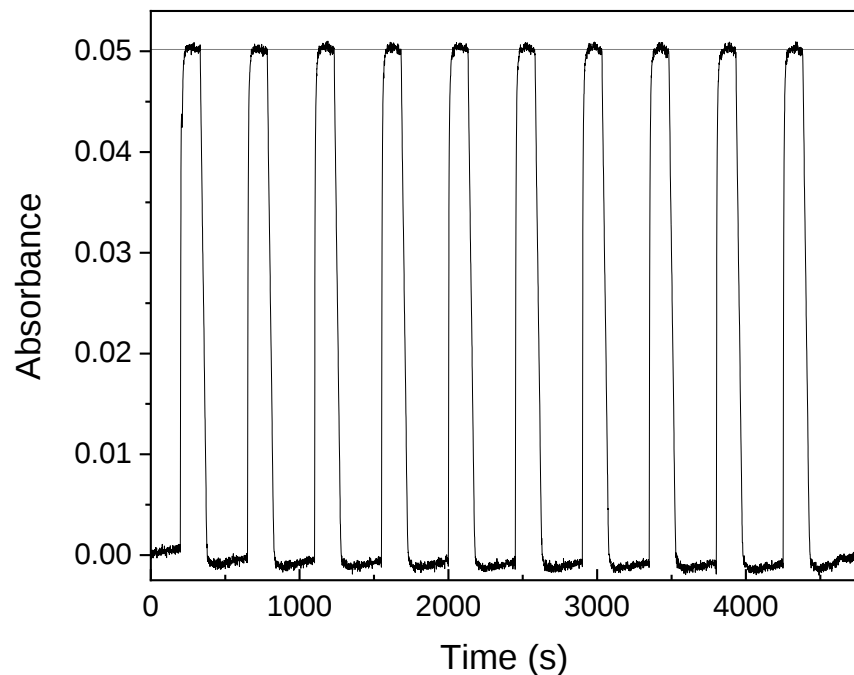
$\tau_{\text{response}} < 30\text{s}$; $\tau_{\text{recovery}} < 60\text{s}$

Calibration test

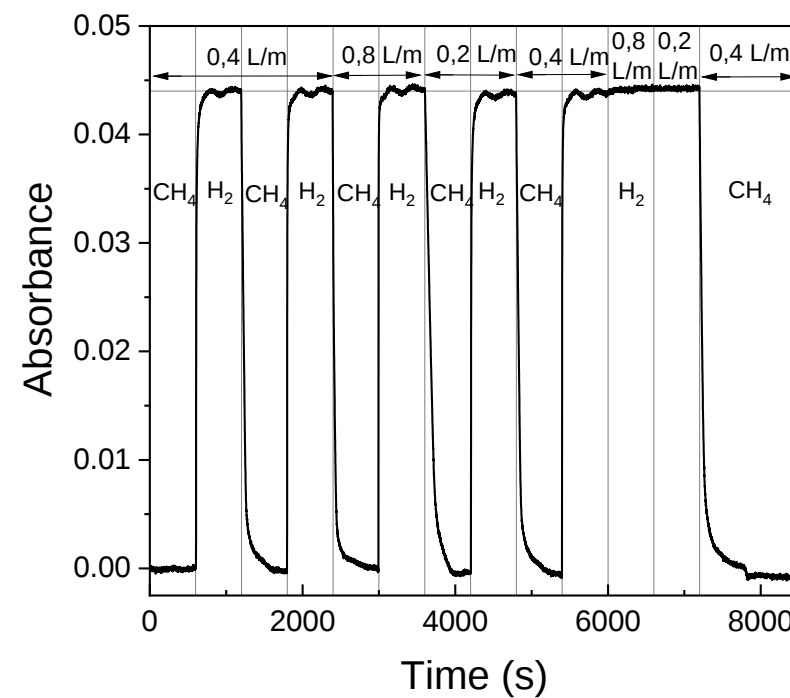


$$RI = \left| 1 - \frac{Abs_{gas}}{Abs_{CH_4}} \right|$$

ISO 26142:2010 – Repeatability and flow rate test



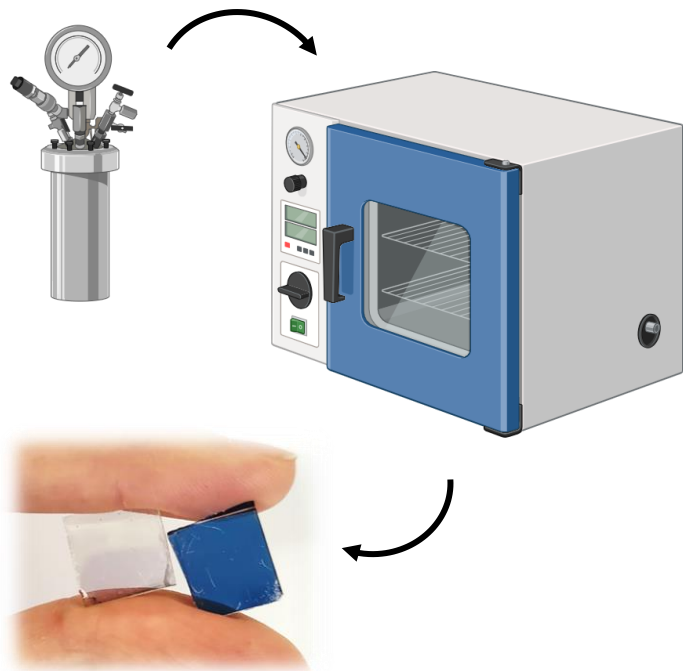
5% H_2 in CH_4 ; $\lambda=850\text{nm}$; $T=200^\circ\text{C}$



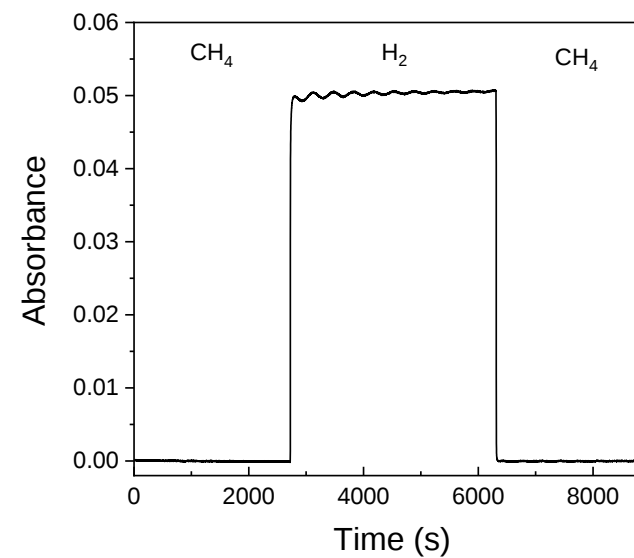
20% H_2 in CH_4 ; $\lambda=850\text{nm}$; $T=200^\circ\text{C}$

Long term stability

1 month in H_2 in CH_4 (20vol%) @120°C



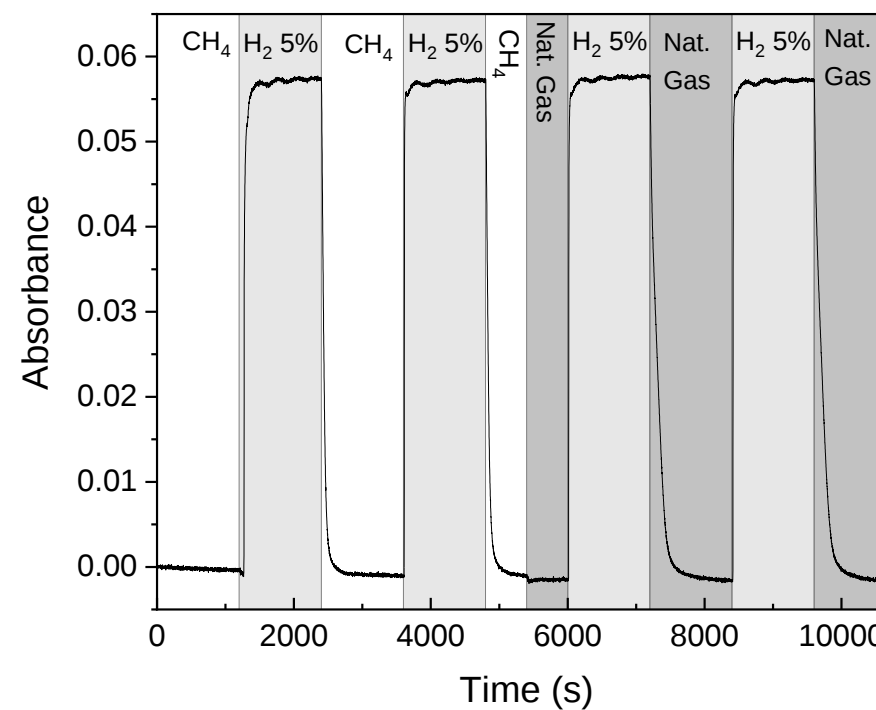
5% H_2 in CH_4 ; $\lambda=850\text{nm}$; $T=200^\circ\text{C}$



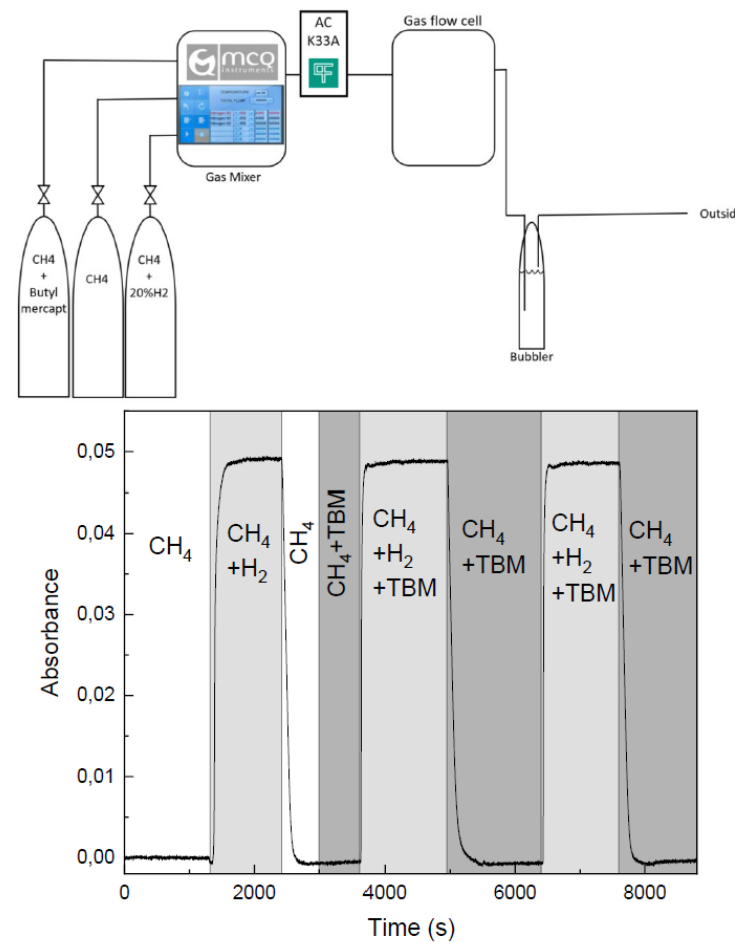
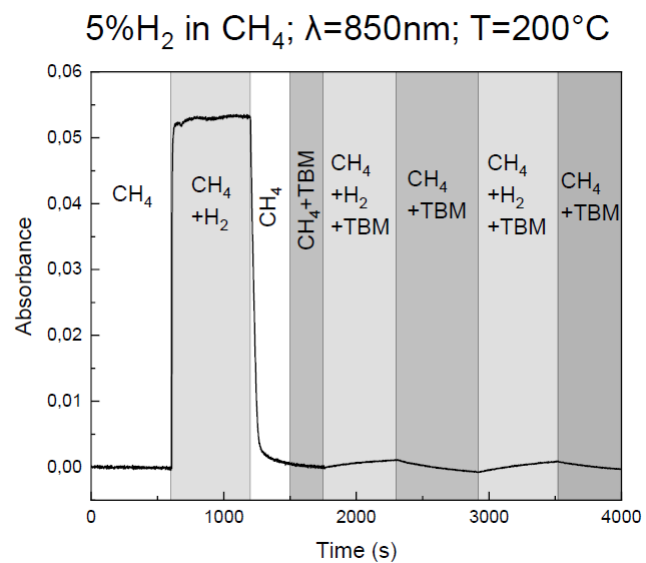
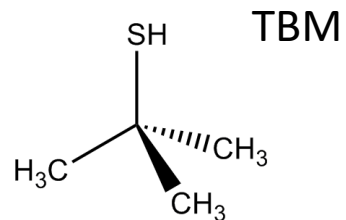
Natural gas mixture containing:

- Methane (primary component)
- Ethane (4.17mol%)
- Propane (0.82mol%)
- Nitrogen (1.30mol%)
- CO₂ (0.65mol%)
- Other hydrocarbons (<0.5mol%)

$\lambda=850\text{nm}$; $T=200^\circ\text{C}$



Odorants



Pt coated WO₃ sensor tested in hydrogen-blended natural gas



Fast response and complete recovery



Excellent repeatability ($\leq 1\%$ variation)



Long-term stability



Flow-rate independency



No cross sensitivity in natural gas

**.... prototype under
development**

Thank you for the attention!



Acknowledgments



A. Longato

NanoENG

Nanomaterials Engineering Group

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