METROLOGY for HYDROGEN VEHICLES

Inter-laboratory comparison

Dr Thomas Bacquart

Workshop on hydrogen quality and flow metering for hydrogen fuel cell vehicles
11-12 September 2019, Delft (NL)

EMPIR EURAMET FCH

Overview

- What will the audience learn in this session?
- Importance of inter-comparison
- Options for inter-comparison
- Challenges with inter-comparison in hydrogen fuel quality

METROLOGY for HYDROGEN VEHICLES

HYDRAITE

Laboratory inter-comparison

- What?
  - testing the “same samples” by different laboratories and comparing the results
- Why?
  - Check the ability of laboratories to deliver accurate testing results to their customers: **proficiency testing**
  - Find out whether a certain analytical method performs well and is fit for its intended purposes: **collaborative method validation study**

Laboratory equivalence

- HyCoRA studies

Table 9. Comparison between NPL and Smart Chemistry results. Additional analysis by SINTEF is also shown for sample #4.

	#1		#2		#4			#5		#6	
	SC	NPL	SC	NPL	SC	NPL	SINTEF	SC	NPL	SC	NPL
Water	< 1	2.93	< 1	2.51	1.3	13.2		< 1	3.44	< 1	1.38
Nitrogen	< 5	4.8	8.3	18.25	452	579		41.0	89.3	49.4	87.69
Argon	< 0.4	< 1	< 0.4	< 1	4.3	< 1		0.48	< 1	0.51	< 1
Helium	10	< 20	< 10	< 20	< 10	< 20		< 10	< 20	< 10	< 20
Oxygen	< 1	0.59	< 1	0.67	1.8	< 0.5		3.1	4.84	4.8	0.272
Carbon dioxide	< 0.1	< 0.02	< 0.1	< 0.02	0.37	0.316	0.250	< 0.1	0.0306	< 0.1	< 0.02
Carbon monoxide	0.0022	< 0.02	0.0010	< 0.02	0.0093	< 0.02		0.0030	< 0.02	0.0017	< 0.02
Methane	0.21	< 0.02	0.60	0.0491	17	14.28	12	0.22	0.242	< 0.2	< 0.02
Ethane					5.6	319	400				
Propane					8.7	0.117	1				
N-butane					15	0.46	1				
Total hydrocarbons	0.22	< 0.02	1.7	< 0.02	47	> 200		1.2	< 0.02	0.27	< 0.02
Total sulphur	0.000016	< 0.001	0.000010	< 0.001	0.0000042	< 0.001		0.000015	< 0.001	0.000016	< 0.001
Total halogenates	0.00067	< 0.052	0.0026	< 0.052	0.0062	< 0.052		0.0028	< 0.052	0.0035	< 0.052

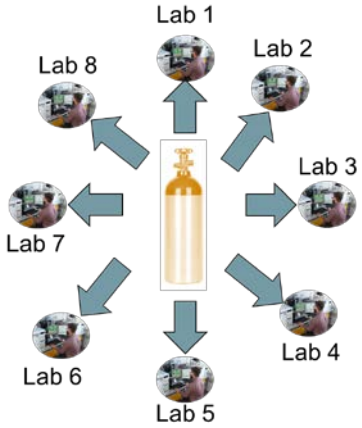
Reliability
Confidence

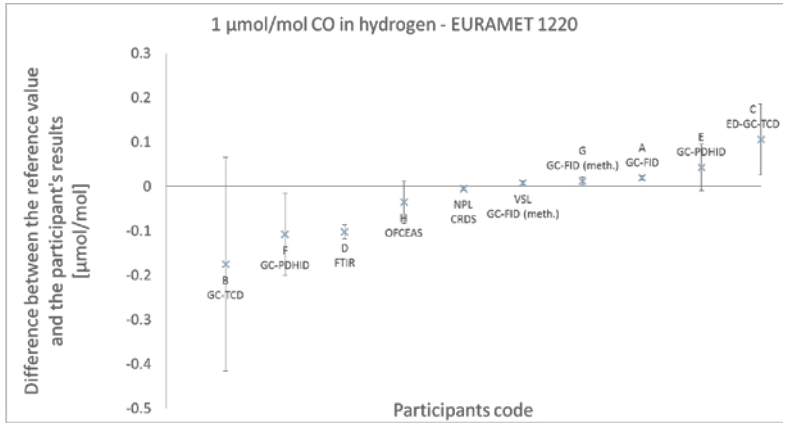
METROLOGY for HYDROGEN VEHICLES

HYDRAITE

Measurement challenge

- Inter-comparison challenges
  - Material (artefact, reference material)
    - Amount fraction
    - Stability
    - Volume and pressure
  - Number of participants: $n > 6$
  - Logistic (transport regulation)





METROLOGY for HYDROGEN VEHICLES

HYDRAITE

Inter-comparison - Euramet 1220

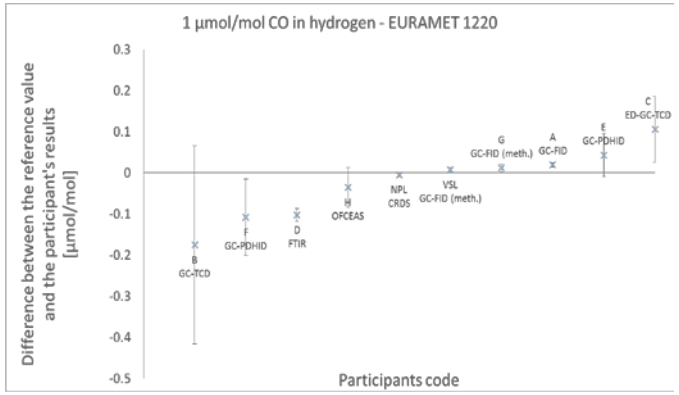
1st inter-comparison for Hydrogen purity

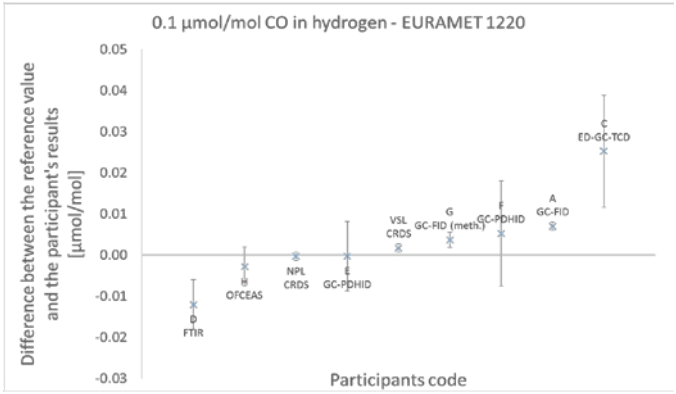
Validation of laboratory measurement:

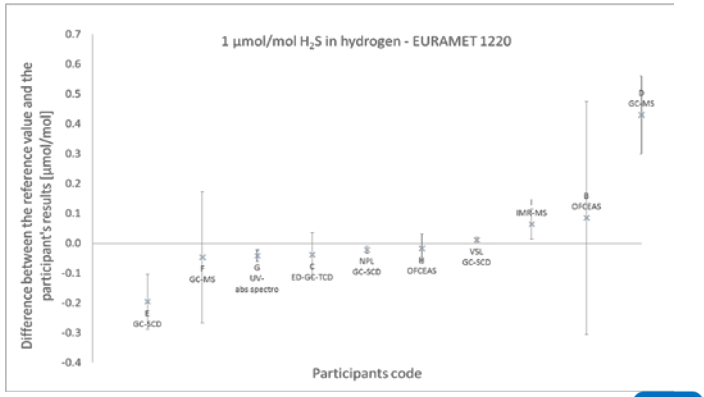
- Test method
- Calibrant
- Staff

CO and H_2S chosen for study

Industrial laboratories and NMI's







METROLOGY for HYDROGEN VEHICLES

HYDRAITE

MetroHyVe inter-comparison

	amount fraction [µmol/mol]
CO	0.1 – 1.0
H_2S	0.02 – 0.5
N_2	50 – 500
H_2O	1 – 10

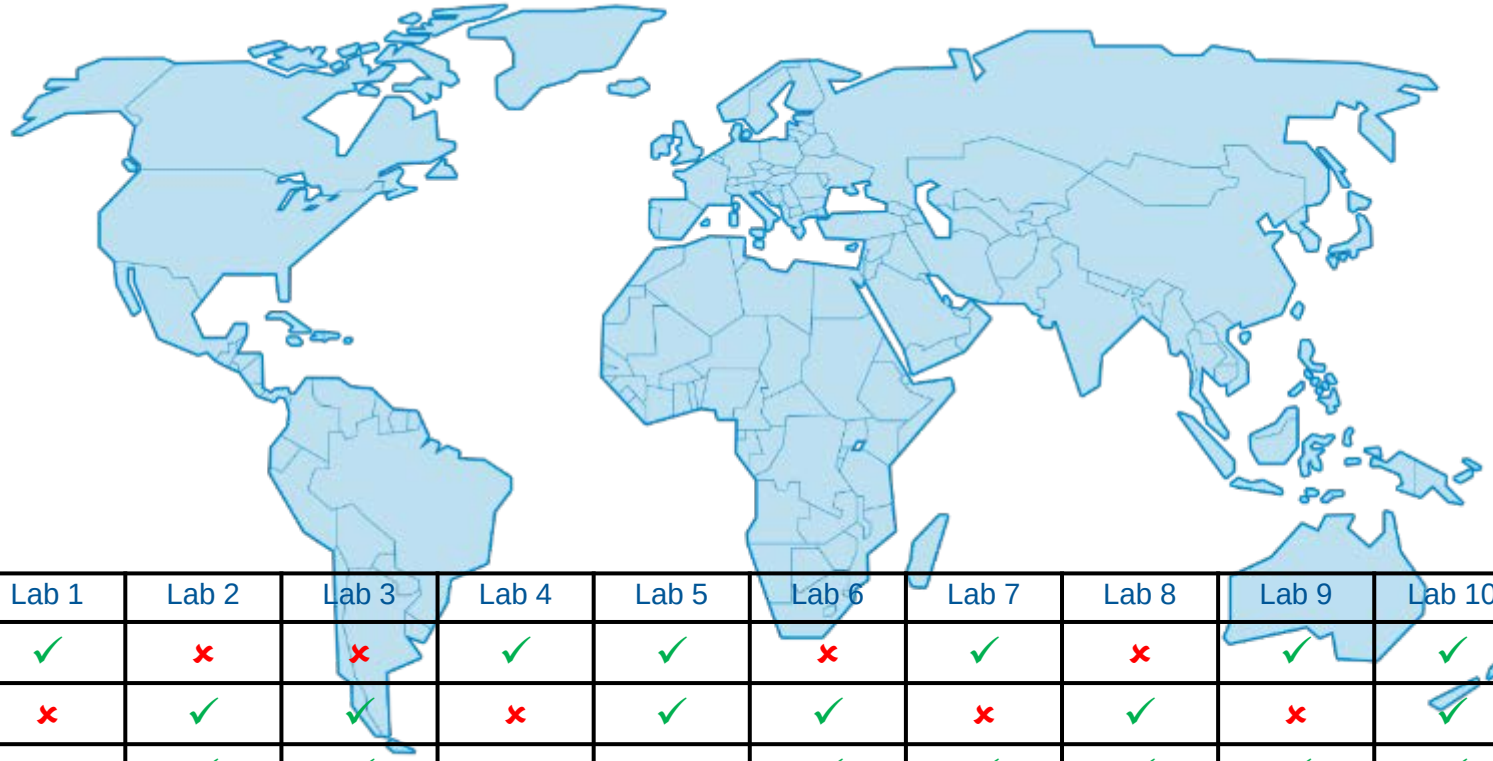
H2comparison@npl.co.uk

METROLOGY for HYDROGEN VEHICLES

HYDRAITE

Hydrogen Purity Analysis

International comparison for laboratory analysis



	amount fraction [µmol/mol]	Lab 1	Lab 2	Lab 3	Lab 4	Lab 5	Lab 6	Lab 7	Lab 8	Lab 9	Lab 10
CO	0.1 – 1.0	✓	✗	✗	✓	✓	✗	✓	✗	✓	✓
H_2S	0.02 – 0.5	✗	✓	✓	✗	✓	✓	✗	✓	✗	✓
N_2	50 – 500	✗	✓	✓	✗	✗	✓	✓	✓	✓	✓
H_2O	1 – 10	✗	✓	✓	✓	✗	✗	✗	✗	✓	✓

H2comparison@npl.co.uk

METROLOGY for HYDROGEN VEHICLES

HYDRAITE

HYDRAITE inter-comparison on real samples

Sample number - vessel identifier		***Lab 1 - Hydr 02***	***Lab 2 - Hydr 02***	***Lab 3 - Hydr 02***	***Lab 1 - Hydr 08***	***Lab 2 - Hydr 08***	***Lab 3 - Hydr 08***
measured species	***Limit ISO 14687:2018***	***Result +/- Uncertainty*** uncertainty of calibration gas, instrument and measurement taken into account with k =2					
	in ppmv	*in ppmv*			*in ppmv*	*in ppmv*	*in ppmv*
H_2O	5	3 ± 0,2	2.63 ± 0.15	-	3 ± 0,2	0.67 ± 0.10	-
Total non methane Hydrocarbons	2	< 0,1	< 0.10	0.11	< 0,1	< 0.10	0.58
CH4	100	< 0,1	< 0.020	0.0073	< 0,1	< 0.020	0.069
O_2	5	1,1 ± 0,1	< 0.30	-	< 0,1	< 0.30	-
He	300	< 10	< 30	-	< 10	< 30	-
Ar	300	< 0,1	< 0.30	-	< 0,1	< 0.30	-
N_2	300	9,8 ± 0,7	2.52 ± 0.33	-	9,9 ± 0,6	6.45 ± 0.34	7.1
CO_2	2	< 0,9	< 0.020	-	< 0,9	< 0.020	-
CO	0,2	< 0,003	< 0.030	0.0016	< 0,003	< 0.030	0.0014
Total sulfur compounds	0,004	< 0,001	< 0.0010	0.00013	< 0,001	< 0.0010	0.000011
HCOH	0,2	< 0,003	< 0.018	-	< 0,003	< 0.018	-
HCOOH	0,2	< 0,003	< 0.013	-	< 0,003	< 0.013	-
NH3	0,1	< 0,01	< 0.020	-	< 0,01	< 0.020	-
H2S	subset of TS	< 0,007	-	-	< 0,007	-	-
COS	subset of TS	< 0,003	-	-	< 0,003	-	-
Halogenated compounds	0,05	< 0,001	< 0.020	0.0064	< 0,001	< 0.020	0.0124

METROLOGY for HYDROGEN VEHICLES

HYDRAITE

Inter-laboratory comparison summary

- Few inter-comparison on Hydrogen fuel (Euramet 1220, MetroHyVe)
- Two designs:
  - 1-2 gas **cylinders shared** between participants
    - Simple preparation
    - Complex logistic (shipment) and long term stability needed
  - **Batch** of cylinders (i.e. 1 per participant)
    - Complex preparation (preparation and homogeneity of the batch)
    - Simple logistic (1 shipment) and short term stability

- Two type of material/artefact:
  - Gas standard (few compounds in gas cylinder)
  - Real samples (difficulty to have measurable amount fraction of contaminant)
- Identify equivalence and difference (propose corrective action plan)

New challenges

- Needs of inter-comparison scheme for H2 quality according to ISO 14687, EN17124:2018 and ISO 21087:2019

Get involved!

- Artefact / Reference materials:
  - Real sample: almost no contaminant present
  - Preparation of fake sample: not technically available

- Inter-comparison organisation:
  - preparation cost supported by EU project (after 2019, potentially EMPIR MetroHyVe 2 and EMPIR Decarb (Grade A)
  - Number of participant
  - Cylinder transport (stability / regulation)

METROLOGY for HYDROGEN VEHICLES

HYDRAITE

Available resources

- Reports EURAMET 1220
- ISO 17034:2016
- In 2020 - MetroHyVe inter-comparison report

METROLOGY for HYDROGEN VEHICLES

HYDRAITE

This project has received funding from the Fuel Cells and Hydrogen 2 Joint Undertaking under grant agreement No 779475. This Joint Undertaking receives support from the European Union's Horizon 2020 research and innovation programme and Hydrogen Europe and Hydrogen Europe Research.

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

THANK YOU

METROLOGY for HYDROGEN VEHICLES

metrohyve.eu

HYDRAITE

hydraite.eu