

Final meeting

Metrology for sustainable hydrogen energy applications

Work package 1 Hydrogen purity measurements according to ISO 14687-2 and risk assessment for fuel cells

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21th May 2019



Work package 1



• All the partners involved in standardization work at national or international level

10 partners: 5 NMI + 3 industrial and research centres



In-kind support of stakeholders and industry

Background of work package 1



- 2015: Hydrogen quality Not standardised
 - No sufficient evidence on hydrogen quality (open access data)
 - HYCORA project Hydrogen quality at HRS
 - No data on production method only
 - No sufficient understanding on quality control planning (ISO 19880-8 as draft)
 - Guidelines but no examples
 - Contaminants impact and threshold values
 - HYCORA project leading (Formaldehyde, CO, formic acid)
 - Lack of knowledge on halogenated (i.e. HCl, C₄Cl₄F₆) and NH₃

Objectives of work package 1



Aims:

- understand the real risk of contaminants in hydrogen from different production methods
- Understand the negative impact of these impurities to a fuel cell system.

Final target:

 Provide technical evidences to the ISO TC 197 to justify the ISO 14687 revisions

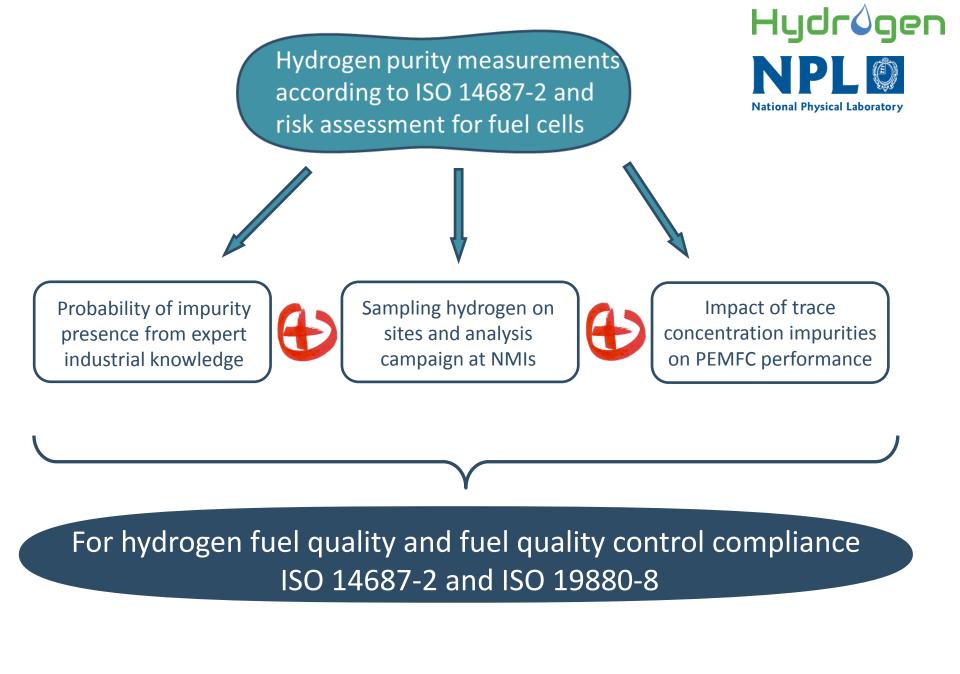
Deliverables of work package 1

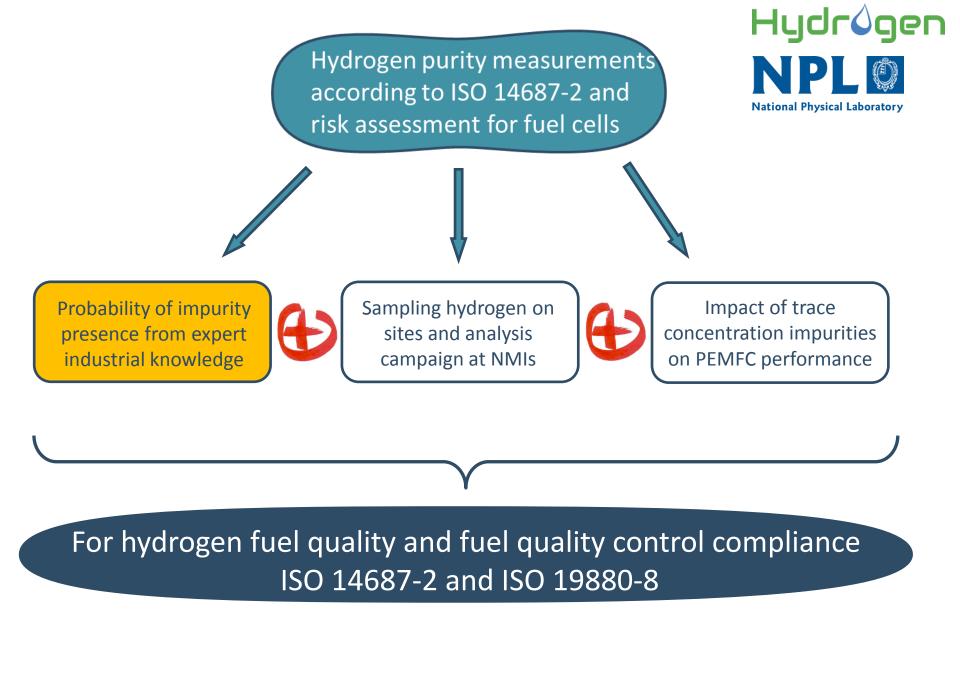


 D1 Report on risk assessment of impurities in hydrogen for fuel cells and recommendations on maximum concentration of individual compounds based on the new fuel cell degradation studies and on the probability of presence

Report NPL, CEM, SP, VSL, AH2GEN, Air Liquide, CEA, FHA June 2018 (M25)

 D2 Letter from ISO/TC197 confirming that the documentary report D1 on risk assessment results has been received for a potential incorporation in an approved Technical Specification or in the revised version of ISO 14687
Letter from the Technical Committee
NPL, LNE, CEM, SP, VSL, AH2GEN, Air Liquide, CEA, FHA May 2019 (M36)





Work package 1 achievements: Probability of Hydrogen impurity presence from expert industrial knowledge NPL 👰

National Physical Laboratory

Rationale of impurities presence based on production process and following an approach in ISO 19880.

| Probability of impurity presence | Steam methane reforming with PSA | PEM water electrolysis process with TSA | Chlor-alkali process (membrane cell process) | |
|-------------------------------------|--|---|---|--|
| Frequent | CO None identified | | 0 ₂ | |
| Possible | N ₂ | None identified | None identified | |
| Rare | Ar, CH ₄ | N ₂ , O ₂ , H ₂ O | H ₂ O, N ₂ | |
| Very Rare | НСНО | CO ₂ | CO ₂ | |
| Unlikely | He, O ₂ , CO ₂ , HCOOH, NH ₃ , sulfur compounds, hydrocarbons (except CH ₄), halogenated compounds | He, Ar, CO, CH ₄ , HCHO, HCOOH, NH ₃ , sulfur compounds, hydrocarbons (except CH ₄), halogenated compounds | He, Ar, CO, CH ₄ , HCHO, HCOOH, NH ₃ , sulfur compounds, hydrocarbons (except CH ₄), halogenated compounds | |

Achievements:

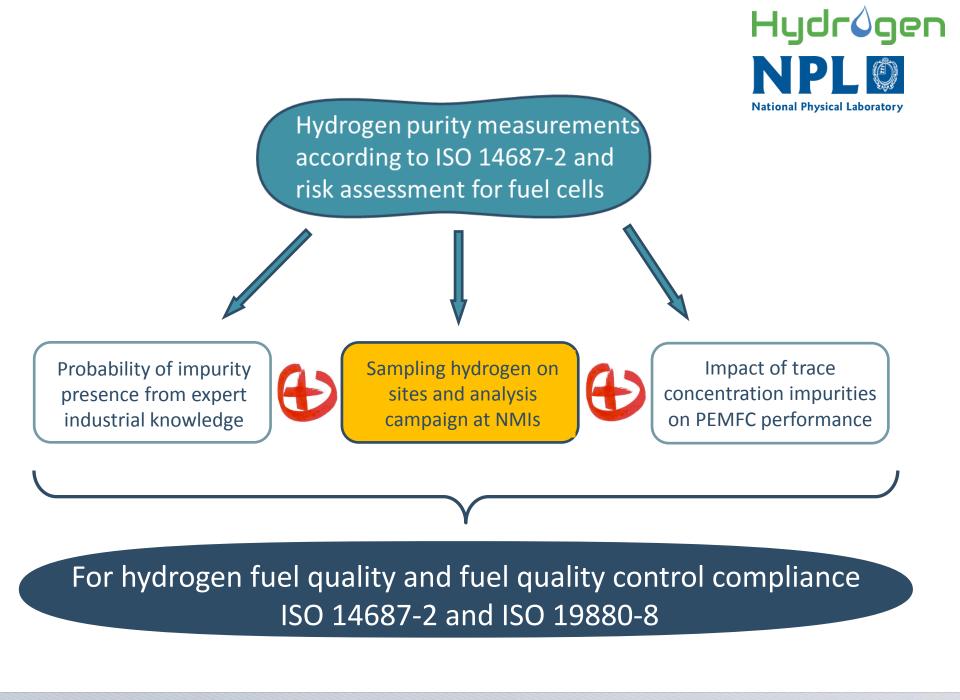
- 3 project reports

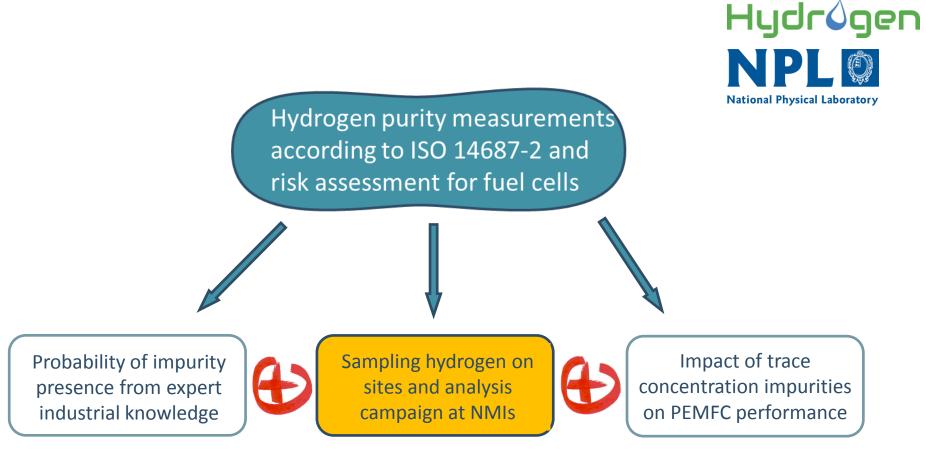
- Article published in International Journal of Hydrogen Energy:

Probability of occurrence of ISO 14687-2 contaminants in hydrogen: Principles and examples from steam methane reforming and electrolysis (water and chlor-alkali) production processes model, Thomas Bacquart, Arul Murugan, Martine Carré, Bruno Gozlan, Fabien Auprêtre, Frédérique Haloua, Thor A. Aarhaug. International Journal of Hydrogen Energy, 2018, 43 (11872-11883)









Aim:

- 10 SMR samples
- 10 PEM Water electrolyser samples
- 1 Chlor-alkali samples

Analysis scope

- ISO 14687-2
- National metrology institutes analysis

Analytical campaign for hydrogen production methods

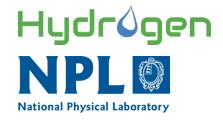


Analytical method used by National Metrology Institutes

| | | NATIONAL Physical Laboratory | VSL Dutch Metrology Institute | Rie SEE Preserch bestfuture of Sereden | CENTRO ESPAÑOL DE METROLOGÍA |
|-----------------------------|----------------------------------|---|--|---|--|
| Compounds | ISO 14687-2 threshold [µmol/mol] | NPL | VSL | RISE | CEM |
| Water H ₂ O | 5 | Quartz crystal microbalance (LOD: 0.5 µmol/mol) CRDS (LOD: 0.5 µmol/mol) | - | OFCEAS (LOD: 2 µmol/mol) | - |
| Methane CH₄ | 2 | GC-methaniser-FID (LOD: 0.01 µmol/mol) | - | GC-FID (LOD: 0.5 µmol/mol) | - |
| Non methane hydrocarbons | 2 | GC-methaniser-FID (LOD: 0.01 µmol/mol) | - | - | - |
| Oxygen O ₂ | 5 | GC-PDHID (LOD: 0.5 µmol/mol) | - | OFCEAS (LOD: 3 µmol/mol) GC-TCD (Ar+O ₂ LOD: 25 µmol/mol) | GC-PDHID (LOD: 5 µmol/mol) GC-TCD (LOD: 25 µmol/mol) |
| Helium He | 300 | - | - | - | GC-TCD (LOD: 5 μmol/mol) |
| Nitrogen N ₂ | 100 | GC-PDHID (LOD: 1 µmol/mol) | - | GC-TCD (LOD: 25 μmol/mol) | GC-TCD (LOD: 80 µmol/mol) GC-PDHID (LOD: 25 µmol/mol) |
| Argon Ar | 100 | GC-PDHID (LOD: 0.5 µmol/mol) | - | GC-TCD (Ar+O2 LOD: 25 µmol/mol) | GC-TCD (LOD: 50 µmol/mol) GC-PDHID (LOD: 25 µmol/mol) |
| Carbon dioxide CO_2 | 2 | GC-methaniser-FID (LOD: 0.01 µmol/mol) | - | OFCEAS (LOD: 0.1 µmol/mol) GC-TCD (LOD: 5 µmol/mol) | - |

TD: Thermo-Desorption
MS: Mass spectrometryCRDS: cavity ring down spectroscopy
TCD: Thermal Conductivity Detector
OFCEAS: Optical Feedback cavity enhanced absorption spectroscopyGC: gas chromatography
PDHID: Pulse discharge helium ionisation detectorFID: Thermal Conductivity Detector
OFCEAS: Optical Feedback cavity enhanced absorption spectroscopyFID: Flame ionisation detector

Analytical campaign for hydrogen production methods



DE METROLOGÍ

Analytical method used by National Metrology Institutes

VSL

RI.

| | | Hattonial Physical Caboratory | Institute | Research Institutes of Soundarn | |
|-------------------------|----------------------------------|---|-------------------------------|--------------------------------------|-----|
| Compounds | ISO 14687-2 threshold [µmol/mol] | NPL | VSL | RISE | CEM |
| Carbon monoxide CO | 0.2 | GC-methaniser-FID (LOD: 0.01 µmol/mol) | - | OFCEAS (LOD: 0.02 µmol/mol) | - |
| Total sulphur compounds | 0.004 | GC-SCD (LOD: 0.002 µmol/mol) | - | OFCEAS (LOD: 0.004 µmol/mol) | - |
| Formaldehyde HCHO | 0.01 | - | CRDS (LOD: 0.005 µmol/mol) | - | - |
| Formic acid HCOOH | 0.2 | - | CRDS (LOD: 0.1 µmol/mol) | - | - |
| Ammonia NH ₃ | 0.1 | - | CRDS (LOD: 0.1 µmol/mol) | - | - |
| Total halogenated (HCI) | 0.05 | - | CRDS (LOD: 0.005 µmol/mol) | - | - |
| C2 hydrocarbons | 2 | - | - | GC-FID (LOD: 0.5 µmol/mol) | - |
| C3 hydrocarbons | 2 | - | - | GC-FID (LOD: 0.5 µmol/mol) | - |
| C4 hydrocarbons | 2 | - | - | GC-FID (LOD: 1 µmol/mol) | - |
| C5 hydrocarbons | 2 | - | - | GC-FID (LOD: 1 µmol/mol) | - |
| C6 - C18 hydrocarbons | 2 | - | - | TD-GC-FID/MS (LOD: 0.05 µmol/mol) | - |

CRDS: cavity ring down spectroscopy TCD: Thermal Conductivity Detector OFCEAS: Optical Feedback cavity enhanced absorption spectroscopy

GC: gas chromatography PDHID: Pulse discharge helium ionisation detector FID: Flame ionisation detector TD: Thermo-Desorption MS: Mass spectrometry

Hydrogen fuel quality from production Hydrogen methods – Steam methane reforming with PSA NPL 💿

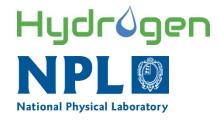
| Compounds | ISO 14687-2 threshold [µmol/mol] | <u>SMR with PSA (8 samples)</u> Results [µmol/mol] | Probability of occurrence [16] |
|--------------------------------|--|---|-----------------------------------|
| Water H ₂ O | 5 | < 2 | Unlikely (0) |
| Methane CH ₄ | 2 | < 0.02 to 0.05 | Rare (2) |
| Non methane hydrocarbons | 2 | < 0.05 | Unlikely (0) |
| Oxygen O ₂ | 5 | < 1.0 | Unlikely (0) |
| Helium He | 300 | < 54 | Unlikely (0) |
| Nitrogen N ₂ | 100 | < 1.2 to 11 | Possible (3) |
| Argon Ar | 100 | < 0.5 to 1.3 | Rare (2) |
| Carbon dioxide CO ₂ | 2 | < 0.02 to 0.45 | Unlikely (0) |
| Carbon monoxide CO | 0.2 | < 0.02 | Frequent (4) |
| Total sulphur compounds | 0.004 | < 0.0036 | Unlikely (0) |
| Formaldehyde HCHO | 0.01 | < 0.005 | Very rare (1) |
| Formic acid HCOOH | 0.2 | < 0.1 | Unlikely (0) |
| Ammonia NH ₃ | 0.1 | < 0.1 | Unlikely (0) |
| Total halogenated | 0.05 | < 0.005 | Unlikely (0) |
| C2 hydrocarbons | 2 | < 0.5 | Unlikely (0) |
| C3 hydrocarbons | 2 | < 1 | Unlikely (0) |
| C4 hydrocarbons | 2 | < 1 | Unlikely (0) |
| C5 hydrocarbons | 2 | < 1 | Unlikely (0) |
| C6 - C18 hydrocarbons | 2 | <0.05 | Unlikely (0) |



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Hydrogen fuel quality from production methods – PEMW electrolyser with TSA

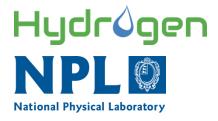


| Compounds | ISO 14687-2 threshold [µmol/mol] | PEM water electrolysis withTSA Results on 8 samples [µmol/mol] | Probability of occurrence |
|----------------------------------|--|---|---------------------------|
| Water H ₂ O | 5 | < 3 | Rare (2) |
| Methane CH ₄ | 2 | < 0.02 | Unlikely (0) |
| Non CH ₄ hydrocarbons | 2 | 0.08 to 0.2 | Unlikely (0) |
| Oxygen O ₂ | 5 | < 0.5 - 2 | Rare (2) |
| Helium He | 300 | < 9 to 45 | Unlikely (0) |
| Nitrogen N ₂ | 100 | < 1.0 to 4.6 | Rare (2) |
| Argon Ar | 100 | < 0.5 | Unlikely (0) |
| Carbon dioxide CO ₂ | 2 | < 0.02 to 0.25 | Very rare (1) |
| Carbon monoxide CO | 0.2 | < 0.02 | Unlikely (0) |
| Total sulphur compounds | 0.004 | < 0.0036 | Unlikely (0) |
| Formaldehyde HCHO | 0.01 | < 0.005 | Unlikely (0) |
| Formic acid HCOOH | 0.2 | < 0.1 | Unlikely (0) |
| Ammonia NH ₃ | 0.1 | < 0.1 | Unlikely (0) |
| Total halogenated | 0.05 | < 0.005 | Unlikely (0) |
| C2 hydrocarbons | 2 | < 0.5 | Unlikely (0) |
| C3 hydrocarbons | 2 | <1 | Unlikely (0) |
| C4 hydrocarbons | 2 | <1 | Unlikely (0) |
| C5 hydrocarbons | 2 | <1 | Unlikely (0) |
| C6 - C18 hydrocarbons | 2 | <0.05 | Unlikely (0) |



CENTRO ESPAÑOL DE METROLOGÍA

Hydrogen fuel quality from production methods - PEMW electrolyser



Analytical campaign – Summary – PEM water electrolysis

| Compounds | ISO 14687-2 threshold [µmol/mol] | <u>PEM water electrolysis</u> <u>with TSA</u> [µmol/mol] | <u>PEM water electrolysis</u> Results on 5 samples [µmol/mol] |
|----------------------------------|-------------------------------------|---|---|
| Water H ₂ O | 5 | < 3 | > 100 |
| Methane CH ₄ | 2 | < 0.02 | < 0.02 to 0.1 |
| Non CH ₄ hydrocarbons | 2 | 0.08 to 0.2 | < 0.02 to 0 .09 |
| Oxygen O ₂ | 5 | < 0.5 – 2 | 18- > 500 |
| Helium He | 300 | < 9 to 45 | < 9 |
| Nitrogen N ₂ | 100 | < 1.0 to 4.6 | 1.2 to 4.5 |
| Argon Ar | 100 | < 0.5 | < 0.5 |
| Carbon dioxide CO ₂ | 2 | < 0.02 to 0.25 | 0.2 to 5.4 |
| Carbon monoxide CO | 0.2 | < 0.02 | < 0.02 |
| Total sulphur compounds | 0.004 | < 0.0036 | < 0.0036 |
| Formaldehyde HCHO | 0.01 | < 0.005 | < 0.005 |
| Formic acid HCOOH | 0.2 | < 0.1 | < 0.1 |
| Ammonia NH ₃ | 0.1 | < 0.1 | < 0.1 |
| Total halogenated | 0.05 | < 0.005 | < 0.005 |
| C2 hydrocarbons | 2 | < 0.5 | < 0.5 |
| C3 hydrocarbons | 2 | <1 | <1 |
| C4 hydrocarbons | 2 | <1 | <1 |
| C5 hydrocarbons | 2 | <1 | <1 |
| C6 - C18 hydrocarbons | 2 | <0.05 | <0.05 |



Importance of purification in quality control monitoring

Steam methane reforming with pressure swing adsorption - Analysis results

Hydrôgen NPL @ National Physical Laboratory

| | Sample 1 | Sample 2 | Sample 3 | Sample 4 | Sample 5 | Sample 6 | Sample 7 | Sample 8 |
|-----------------------------|-------------------|-------------|----------|----------|----------|-----------------|-----------------|-----------------|
| CO (1) | < 0.053 | < 0.053 | < 0.01 | < 0.01 | < 0.01 | < 0.02 | < 0.02 | < 0.02 |
| CO (2) | < 0.2 | < 0.2 | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. |
| CO ₂ (2) | < 0.1 | < 0.1 | < 5 | < 5 | < 5 | < 0.5 | n.a. | n.a. |
| CO ₂ (1) | 0.042 ± 0.016 | < 0.02 | < 0.01 | < 0.01 | < 0.01 | < 0.02 | < 0.02 | < 0.02 |
| CH ₄ (1) | 0.044 ± 0.007 | < 0.02 | < 0.01 | < 0.01 | < 0.01 | < 0.02 | < 0.02 | < 0.02 |
| CH ₄ (3) | n.a. | n.a. | ~ 0.01 | ~ 0.01 | ~ 0.01 | n.a. | n.a. | n.a. |
| CH ₄ (2) | < 0.5 | < 0.5 | < 0.5 | < 0.5 | < 0.5 | < 0.5 | < 0.5 | < 0.5 |
| Non CH4 hydrocarbons (1) | < 0.05 | < 0.05 | < 0.01 | < 0.01 | < 0.01 | < 0.02 | < 0.02 | < 0.02 |
| H ₂ O (1) | < 0.6 | < 0.6 | < 0.5 | < 0.5 | < 0.5 | < 1.8 | < 1.5 | < 1.2 |
| H ₂ O (2) | n.a | n.a. | n.a. | n.a. | n.a. | < 2 | n.a. | n.a. |
| Total sulphur compounds (1) | < 0.002 | < 0.002 | < 0.0036 | < 0.0036 | < 0.0036 | < 0.002 | < 0.002 | < 0.002 |
| H ₂ S (2) | < 0.04 | < 0.04 | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. |
| O ₂ (4) | n.a. | < 5 | n.a. | < 5 | < 5 | < 25 | < 25 | < 25 |
| O ₂ + Ar (2) | n.a. | n.a. | n.a. | n.a. | n.a. | < 50 | < 50 | < 50 |
| O ₂ (1) | 0.39 ± 0.13 | 0.39 ± 0.13 | < 0.5 | < 0.5 | < 0.5 | < 0.5 | < 1.0 | < 0.5 |
| N ₂ (4) | n.a. | n.a. | < 100 | < 50 | < 60 | < 50 | < 50 | < 50 |
| N ₂ (2) | n.a. | < 25 | n.a. | n.a. | < 80 | < 25 | < 25 | < 25 |
| N ₂ (1) | 1.5 ± 0.6 | < 1.0 | < 1.2 | < 1.2 | < 1.2 | 5.2 ± 0.6 | 10.4 ± 1.1 | 5.5 ± 0.6 |
| Ar (2) | n.a. | n.a. | < 30 | < 30 | < 30 | n.a. | n.a. | n.a. |
| Ar (4) | n.a. | < 25 | n.a. | n.a. | < 80 | < 25 | < 25 | < 25 |
| Ar (1) | 2.8 ± 0.1 | < 0.5 | < 0.5 | < 0.5 | < 0.5 | 1.00 ± 0.10 | 1.30 ± 0.10 | 1.11 ± 0.10 |
| Total halogenated (HCl) (3) | < 0.005 | < 0.005 | < 0.005 | < 0.005 | < 0.005 | < 0.005 | < 0.005 | < 0.005 |
| CH ₂ O (3) | < 0.005 | < 0.005 | < 0.005 | < 0.005 | < 0.005 | < 0.005 | < 0.005 | < 0.005 |
| $CH_2O_2(3)$ | < 0.1 | < 0.1 | < 0.1 | < 0.1 | < 0.1 | < 0.1 | < 0.1 | < 0.1 |
| NH ₃ (3) | < 0.1 | < 0.1 | n.a. | < 0.1 | < 0.1 | < 0.1 | < 0.1 | < 0.1 |
| He (4) | 20 ± 4 | 12 ± 5 | n.a. | n.a. | < 50 | 44 ± 10 | 43 ± 10 | 43 ± 8 |
| C2 hydrocarbons (2) | < 0.5 | < 0.5 | < 0.5 | < 0.5 | < 0.5 | < 0.5 | < 0.5 | < 0.5 |
| C3-hydrocarbons (2) | <1 | <1 | <1 | <1 | <1 | <1 | <1 | <1 |
| C4-hydrocarbons (2) | <1 | <1 | <1 | <1 | <1 | <1 | <1 | <1 |
| C5-hydrocarbons (2) | <1 | <1 | <1 | <1 | <1 | <1 | <1 | <1 |
| C6 – C18 hydrocarbons (2) | <0.050 | <0.050 | <0.050 | <0.050 | <0.050 | <0.050 | <0.050 | <0.050 |

1: NPL; 2: RISE; 3: VSL; 4: CEM

PEM water electrolyser with temperature swing adsorption - Analysis results

Hydrôgen NPL @ National Physical Laboratory

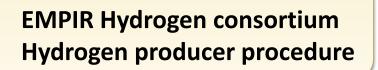
| Compounds | Sample 1 | Sample 2 | Sample 3 | Sample 4 | Sample 5 | Sample 6 | Sample 7 | Sample 8 |
|--------------------------------------|-------------------|---------------|------------------|----------|----------|-----------------|-------------------|-------------------|
| CO (1) | < 0.053 | < 0.02 | < 0.02 | < 0.02 | < 0.02 | < 0.02 | < 0.02 | < 0.02 |
| CO (2) | < 0.2 | n.a. | < 0.02 | < 0.02 | < 0.02 | n.a. | n.a. | n.a. |
| CO ₂ (2) | < 0.1 | < 5 | < 5 | < 0.4 | < 0.4 | < 5 | n.a. | < 5 |
| CO ₂ (1) | 0.443 ± 0.010 | 0.245 ± 0.010 | 0.229 ± 0.08 | < 0.02 | < 0.02 | < 0.01 | < 0.01 | < 0.01 |
| CH ₄ (1) | 0.031 ± 0.006 | < 0.01 | < 0.01 | < 0.02 | < 0.02 | < 0.01 | < 0.01 | < 0.01 |
| Non CH ₄ hydrocarbons (1) | < 0.05 | < 0.02 | < 0.02 | < 0.02 | < 0.02 | 0.156 ± 0.030 | 0.126 ± 0.026 | 0.111 ± 0.024 |
| CH ₄ (2) | < 0.5 | < 0.5 | < 0.5 | < 0.5 | < 0.5 | n.a. | n.a. | n.a. |
| H ₂ O (1) | < 0.6 | < 0.8 | < 1.4 | < 3 | < 3 | < 0.8 | < 1.2 | < 3 |
| H ₂ O (2) | n.a. | n.a. | n.a. | < 3 | < 5 | n.a. | n.a. | n.a. |
| Total sulphur compounds (1) | < 0.002 | < 0.002 | < 0.002 | < 0.002 | < 0.002 | < 0.0030 | < 0.0030 | < 0.0030 |
| H ₂ S (2) | < 0.04 | - | - | < 0.004 | < 0.004 | n.a. | n.a. | n.a. |
| O ₂ (4) | < 5 | < 5 | < 5 | < 5 | < 5 | < 5 | n.m. | < 5 |
| O ₂ + Ar (2) | n.a. | < 5 | < 11 | < 5 | < 3 | < 25 | n.a. | < 25 |
| O ₂ (1) | 0.45 ± 0.13 | < 0.5 | < 0.5 | < 0.6 | < 0.6 | 1.39 ± 0.36 | < 0.5 | 1.59 ± 0.45 |
| N ₂ (4) | n.a. | < 40 | < 70 | - | - | < 100 | n.a. | < 100 |
| N ₂ (2) | < 25 | < 50 | < 50 | < 50 | < 50 | < 80 | n.m. | n.m. |
| N ₂ (1) | 2.0 ± 0.5 | 4.6 ± 0.3 | 4.2 ± 0.4 | < 1.5 | < 1.5 | 1.51 ± 0.2 | < 1.0 | 1.86 ± 0.2 |
| Ar (4) | < 25 | < 50 | < 50 | < 50 | < 50 | < 80 | n.m. | n.m. |
| Ar (1) | < 0.5 | < 0.5 | < 0.5 | < 0.5 | < 0.5 | < 0.5 | < 0.5 | < 0.5 |
| Total halogenated (HCI) (3) | < 0.005 | < 0.005 | < 0.005 | < 0.005 | < 0.005 | n.a. | < 0.005 | < 0.005 |
| CH ₂ O (3) | < 0.005 | < 0.005 | < 0.005 | < 0.005 | < 0.005 | < 0.005 | < 0.005 | < 0.005 |
| CH ₂ O ₂ (3) | < 0.1 | < 0.1 | < 0.1 | < 0.1 | < 0.1 | < 0.1 | < 0.1 | < 0.1 |
| NH ₃ (3) | < 0.1 | < 0.1 | < 0.1 | < 0.1 | < 0.1 | n.a. | n.a. | n.a. |
| He (4) | 34 ± 5 | < 5 | < 5 | 15 - 45 | < 5 | < 9 | < 9 | < 9 |
| C2 hydrocarbons (2) | < 0.5 | < 0.5 | < 0.5 | < 0.5 | < 0.5 | n.a. | n.a. | n.a. |
| C3-hydrocarbons (2) | <1 | <1 | <1 | <1 | <1 | n.a. | n.a. | n.a. |
| C4-hydrocarbons (2) | <1 | <1 | <1 | <1 | <1 | n.a. | n.a. | n.a. |
| C5-hydrocarbons (2) | <1 | <1 | <1 | <1 | <1 | n.a. | n.a. | n.a. |
| C6 – C18 hydrocarbons (2) | <0.050 | <0.05 | <0.05 | <0.05 | <0.05 | n.a. | n.a. | n.a. |

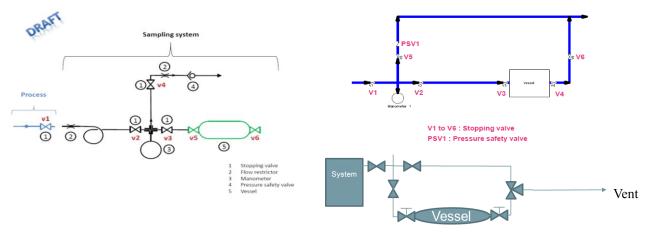
1: NPL; 2: RISE; 3: VSL; 4: CEM

Additional challenges - Sampling procedure



- Development of sampling strategy (Air Liquide, AREVA H2Gen, NPL)
 - Pressure (14 100 bar)
 - Flow
 - Location of sampling (as close as hydrogen production process)
 - Sampling material
 - Sampling procedure





- Cycling purge
- Purge through cylinder
- Cleanliness of cylinder
- Absence of leak

Example of contaminants observed in project samples



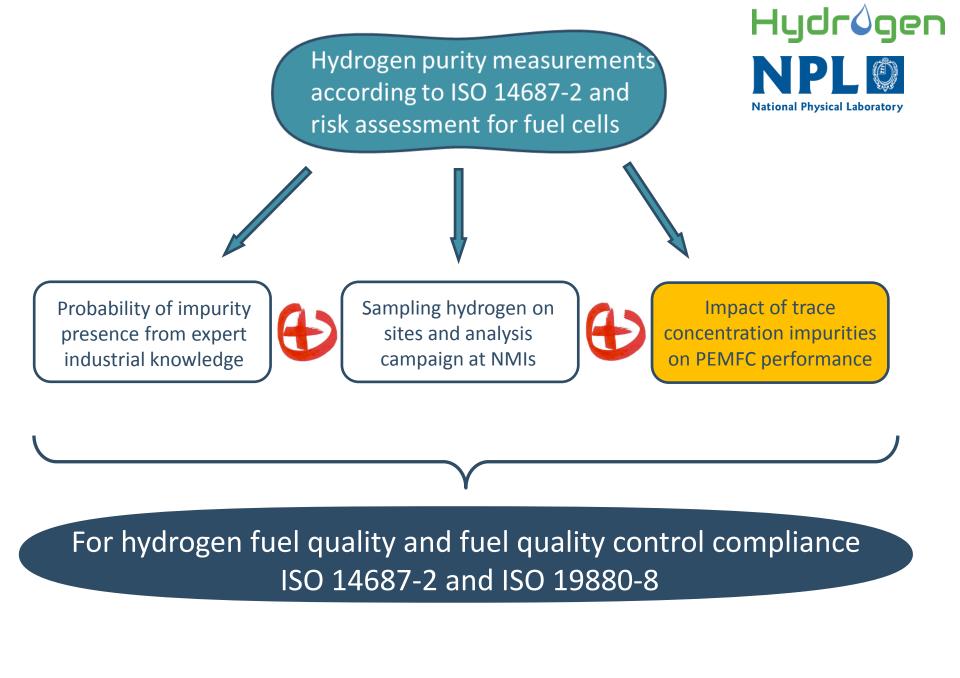
| | ISO 14687-2 | <u>Contaminated sample</u> <u>1</u> | <u>Contaminated sample</u> <u>2</u> | <u>Contaminated sample</u> <u>3</u> |
|----------------------------------|-------------------------|---|--|--|
| Compounds | threshold [µmol/mol] | <u>Chlor-alkali membrane</u> <u>electrolysis feedstock</u> | SMR feedstock | SMR feedstock |
| | | Results [µmol/mol] | Results [µmol/mol] | Results [µmol/mol] |
| Water H ₂ O | 5 | 13.2 ± 1.7 | 2.48 ± 0.25 | 17.1 ± 3.5 |
| Methane CH ₄ | 2 | 14.28 ± 0.07 | < 0.02 | 0.038 ± 0.004 |
| Non CH ₄ hydrocarbons | 2 | > 200 | < 0.05 | < 0.040 |
| Oxygen O ₂ | 5 | < 0.5 | 35 ± 2 | 1.35 ± 0.07 |
| Helium He | 300 | < 20 | 12 ± 5 | n.a. |
| Nitrogen N ₂ | 100 | 579 ± 23 | 134 ± 2 | 14.6 ± 0.8 |
| Argon Ar | 100 | < 1.0 | 1.43 ± 0.10 | 4.2 ± 0.3 |
| Carbon dioxide CO ₂ | 2 | 0.316 ± 0.007 | 0.101 ± 0.004 | < 0.04 |
| Carbon monoxide CO | 0.2 | < 0.02 | < 0.053 | < 0.02 |
| Total sulphur compounds | 0.004 | < 0.0036 | < 0.002 | <0.002 |
| Formaldehyde HCHO | 0.01 | < 0.005 | < 0.005 | < 0.005 |
| Formic acid HCOOH | 0.2 | < 0.1 | < 0.1 | < 0.1 |
| Ammonia NH ₃ | 0.1 | < 0.1 | < 0.1 | n.a. |
| Total halogenated | 0.05 | < 0.005 | < 0.005 | < 0.005 |
| | | HRS maintenance contamination suspected | Improper purging (air) | Improper purging (humidity) 19 |

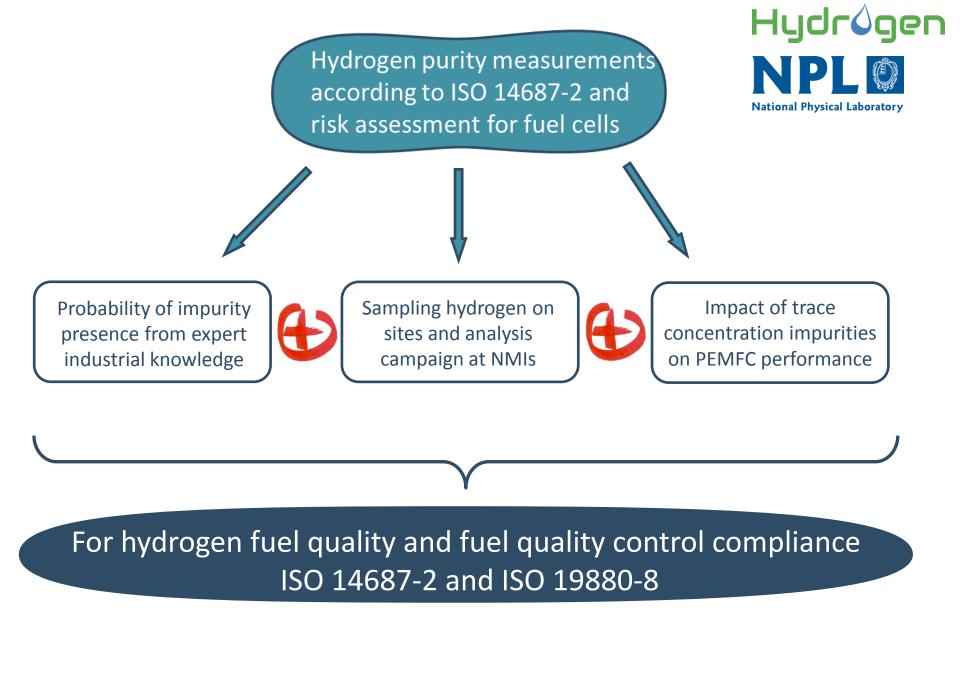
Work package 1 – sampling campaign achievements



• 27 hydrogen samples analysed according to ISO 14687-2 (Target: 21)

- 8 / 11 sample analysed of SMR with PSA
 - Reason: 3 samples contaminated during sampling by third party
- 8 / 15 sample analysed of PEMW electrolyser with TSA
 - Reason: 5 samples from PEMW electrolyser (no purification)
- 0 / 1 sample analysed of chlor-alkali membrane electrolysis process
 - Reason: 1 sample contaminated by HRS infrastructure/operation
- No contaminants observed above ISO 14687-2 threshold
 - 8 different samples from steam methane reformers with PSA
 - 8 different samples from PEM water electrolyser with TSA
- Difference in process expert evaluation and sampling campaign
 - Contamination within short time line (spike) Difficult to spot with point sampling
 - Conservative approach from expert group due to lack of analytical measurement





Deliverables of work package 1



 D1 Report on risk assessment of impurities in hydrogen for fuel cells and recommendations on maximum concentration of individual compounds based on the new fuel cell degradation studies and on the probability of presence

Report

NPL, CEM, SP, VSL, AH2GEN, Air Liquide, CEA, FHA

June 2018 (M25) → March 2019

 D2 Letter from ISO/TC197 confirming that the documentary report D1 on risk assessment results has been received for a potential incorporation in an approved Technical Specification or in the revised version of ISO 14687

- Letter from the Technical Committee
- NPL, LNE, CEM, SP, VSL, AH2GEN, Air Liquide, CEA, FHA May 2019 (M36) → April 2019





- Involvement of Hydrogen community
 - 6 institutes and industries provided in-kind support / samples
- Training on quality control and ISO 19880-8
 - More than 20 attendees

Open access data

- 4 reports
- 1 peer-review article (2 peer-review articles in draft)
- Communication to standardisation committees
 - Presentations at ISO TC 197 meetings
- Demonstration of new capabilities
 - New PRM gas standards, analytical capabilities

Recommendations and futures perspectives



- Quality control for hydrogen ISO 19880-8
 - Study other production methods (i.e. alkaline electrolyser, chlor-alkali, biomass, NH3 feedstock)
 - Study other part of supply chain (i.e. HRS infrastructure, pipeline)
 - Provide training and guidance on the implementation of probability of presence of contaminants according to ISO 19880-8
 - Establish the hydrogen quality monitoring plan on real case scenario and its benefit in term of cost and reliability of the HRS
- Hydrogen specification ISO 14687
 - Investigate new contaminants (as C₄Cl₄F₆) to allow the industry to evaluate its real impact
 - Propose revision of the three contaminants threshold or further studies

Sampling and analysis of hydrogen

- Standardise or provide guideline on sampling
- Evaluate stability of contaminant over time to define if there is an impact of late analysis.
- Analytical methods → Improvement needed (LOD close to ISO threshold)

ISO meeting TC197 WG27 / WG28, 7-8th October 2018, AFNOR, Paris (FRANCE)

Hydrogen

Hydrøgen

Hydrogen, as an energy source, is a clean and storable solution



hydrogen-energy sector that meet the requirements of the European Directive on the deployment of Alternative Fuels Infrastructure 2014/94/EU in order to bring forward the standardization in R&D related to metrology.

The project aims at supplementing the revision of two ISO standards that generic to enable a sustainable implementation in the fast emer at contributing to the elaboration of two new standards

Revisions of these two ISO standards (ISO Part 2: Proton exchange membra 16111:2008 Developing tran metal hvariae) CEN/TC 268

The two new s are ISO 21087 applications for

frederique.haloua@Ine.fr Analytical methods - Proton exchange membrane (PEM) fuel cell

a vehicles and ISO 19880-8 Gaseous hydrogen – Fueling stations – Part 8: Fuel quality control.

The EMPIR project Hydrogen runs from 1 June 2016 to 31 May 2019.

A workshop related to the project is planned in November 2018. More info here!







ISO meeting TC197 WG27 / WG28, 7-8th October 2018, AFNOR, Paris (FRANCE)



research and innovation programme and the EMPIR Participating States.

NEWS

Workshop at Air Liquide R&D Centre: November 7 & 8, 2018

Hydrogen quality: publication in International Journal of Hydrogen Energy, April 2018

Upcoming events

Past events

