HYDRAITE 1st OEM Workshop

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Quality Assurance to ensure H_2 quality at HRS

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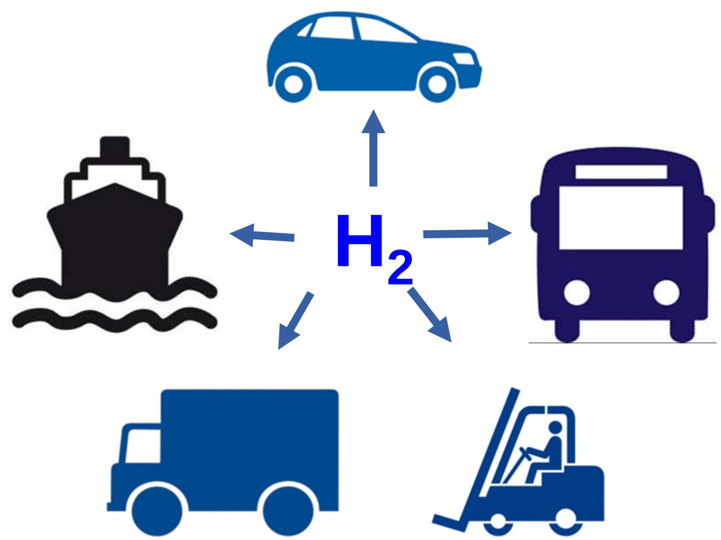
Outline

1. Regulation
2. Cost of H_2 analysis
3. Quality assurance
4. Example for SMR source
5. Conclusion

Air Liquide
creative oxygen

1- Regulation

- Alternative Fuel Infrastructure European Directive (AFI) is applicable since January 2018
- Each European country has to translate this directive in national regulation
- H2 quality for fuel cell vehicles shall be in agreement with ISO 14687-2 until EN 17124 is published
- **EN 17124: Hydrogen fuel — Product specification and quality assurance — Proton exchange membrane (PEM) fuel cell applications for road vehicles will be published by May - June 2018**



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1- Regulation

Component	ISO 14687 -2 µmol/mol	EN 17124 µmol/mol
Helium	300	300
Nitrogen	100	300
Argon	100	300
Methane	/	100
Oxygen	5	5
Carbon dioxide	2	2
Carbon monoxide	0.2	0.2
Water	5	5
Total Hydrocarbons	2	2
Total Sulfured compounds	0.004	0.004
Ammonia	0.1	0.1
Formaldehyde	0.01	0.2
Formic acid	0.2	0.2
Total halogenated compounds	0.05	0.05

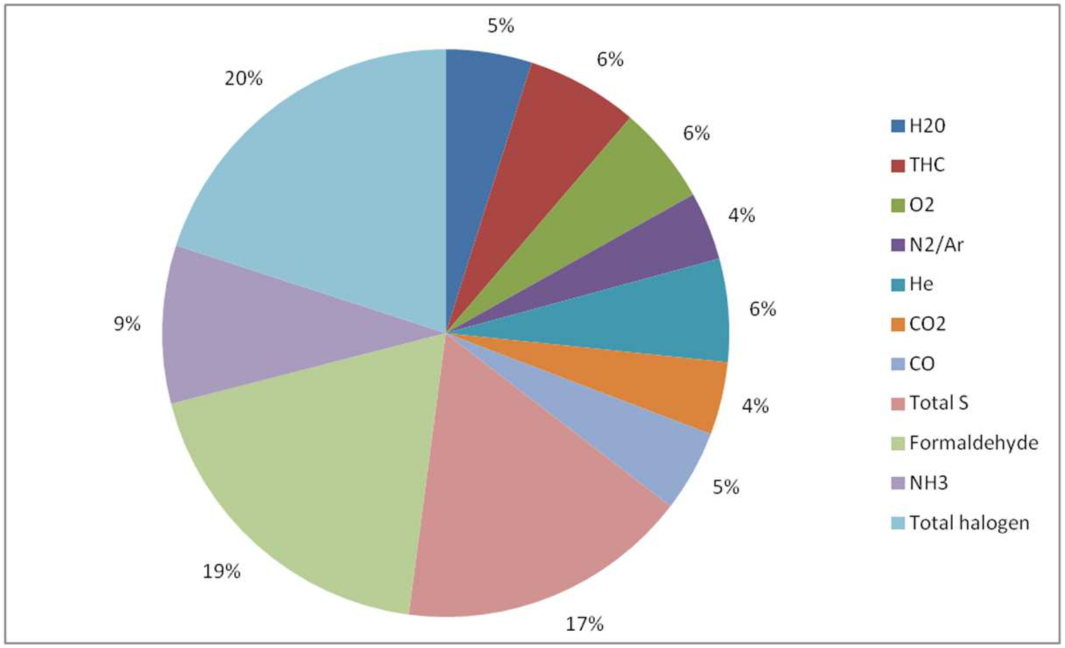
2 – Cost of analysis

- ❑ **Based on the different laboratories able to analyse H_2 to the ISO specifications the cost is :**
  - ❑ **Between 6500 € and 11 000 € for one sample**
  - ❑ **Cost for sampling could be added also (around 4000 €):**
    - ❑ **Man power**
    - ❑ **Sampling device cost**
    - ❑ **Transport from HRS to laboratory**
- ❑ **No analytical service lab is available today in Europe**

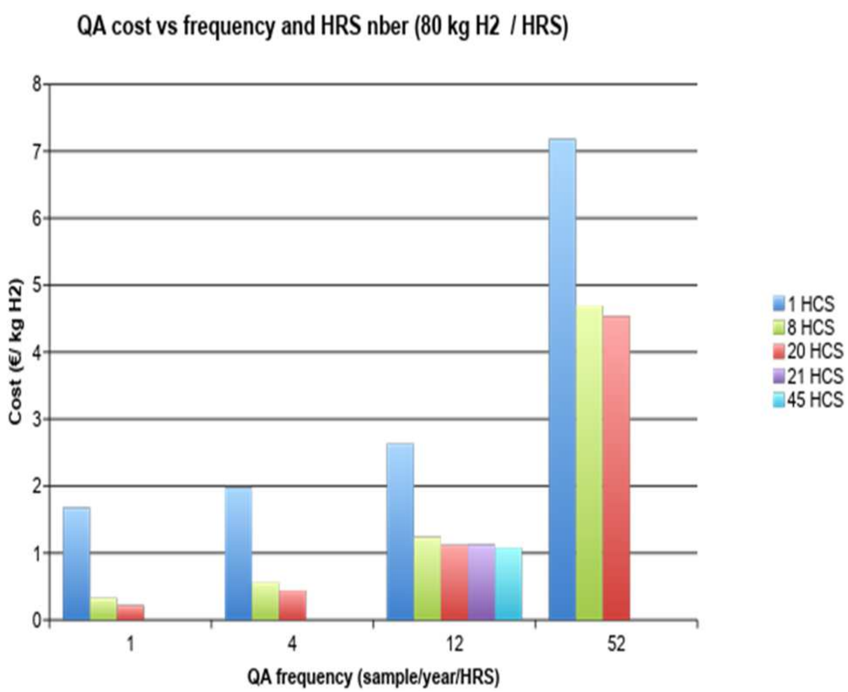
2 – Cost of analysis

Repartition per impurities

3 impurities represent more than 50 % of the total cost



2 – Cost of analysis



- QA cost is impacted by
  - Number of HRS and volume of H2 per HRS
  - Number of analysis per year and per HRS (QA frequency)
  - The number and type of elements to analyse
- Maintenance cost & CAPEX lower with high number of HCS and independent of the QA frequency
- Labour and other running costs for sampling and analysis is the most contributing part of the total QA cost (above 8 HRS)

3 – Quality Assurance

- FCEVs:
  - Make sure the H2 supplied will not damage the vehicle or affect its performance

- H2 providers:
  - Make sure the required quality can be guaranteed, at an acceptable cost.

➔Define a QA scheme acceptable by all parties:

- Use the risk assessment for quality assurance of H2
- According to ISO:IEC Guide 73 the definition of risk assessment is Combination of the probability of an event and its consequences

3 – Quality Assurance

- Event : Be above the threshold value defined by ISO 14687-2 (for each impurity)
- probability of occurrence of this event for a given supply chain (production mode+ delivery + HRS)

Occurrence class	Class name	Occurrence or frequency	Occurrence or frequency
0	Very unlikely (Practically impossible)	Contaminant above threshold never been observed for this type of source in the industry	Never
1	Very rare	Known to occur in the Industry for the type of source/ Supply chain considered	1 per 1 000 000 refueling
2	Rare	Has happened more than once/year in the Industry	1 per 100 000 refueling
3	Possible	Has happened repeatedly for this type of source at a specific location	1 out of 10 000 refueling
4	Frequent	Happens on a regular basis	Often

Table 2 — Occurrence classes for an impurity (EN 17124)

3 – Quality Assurance

- Evaluation of severity (risk) : level of damage for the vehicle (table 3 EN 17124)

Severity class	FCEV Performance impact or damage	Impact categories		
		Performance impact	Hardware impact temporary	Hardware impact permanent
0	— No impact	No	No	No
1	— Minor impact — Temporary loss of power — No impact on hardware — Car still operates	Yes	No	No
2	— Reversible damage — Requires specific light maintenance procedure ,— Car still operates	Yes or No	Yes	No
3	— Reversible damage — Requires specific immediate maintenance procedure . Gradual power loss that does not compromise safety	Yes	Yes	No
4a	— Irreversible damage — Requires major repair (e.g. stack change) — Power loss or Car Stop that compromises safety	Yes	Yes	Yes or No

a Any damage, whether permanent or non-permanent, which compromises safety will be categorized as 4, otherwise non-permanent damage will be categorized as 1, 2 or 3.

3 – Quality Assurance

→ Acceptability table (table 5 of EN 17124)

		Severity				
		0	1	2	3	4
Occurrence as the combined probabilities of occurrence along the whole supply chain	4	green	red	red	red	red
	3	green	yellow	red	red	red
	2	green	green	yellow	red	red
	1	green	green	green	yellow	red
	0	green	green	green	green	green
	Unacceptable risk ; additional control or barriers are required (red)		Further investigations are needed: existing barriers or control may not be enough (yellow)		Acceptable risk area Existing controls acceptable (green)	

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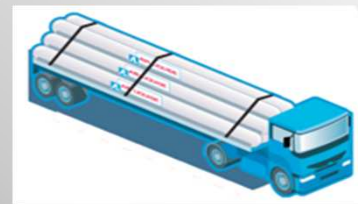
4 – example from SMR source

- **Production site**
  - SMR*
  - Electrolysis



- **Transport**
  - GH2
  - LH2

- **Point of use**
  - Automotive and
  - Stationnary applications

Purity analysis

4 – example from SMR source

- **Step 1 : evaluation of probability of occurrence for (done for each facility by a team of experts or people having the knowledge of each part of process):**
  - *source of H2 production : SMR*
  - *delivery mode of H2 : pipeline*
  - *Hydrogen Refueling Station*
- **Step 2: Combined with defined severity impact on vehicle :**
- **Step 3: Establish the acceptability level for this specific case**
- **Step 4.1: define the barrier to reduce the risk level to acceptable level**
- **Step 4.2: define the critical impurities to follow according to the results of the risk assessment**

4 – example from SMR source

ISO spec		Supply Chain								Residual		
Contaminant	Threshold	Production SMR	Pipeline Distribution	HRS	Compounded probability	Severity	Criticality	Additional risk reduction measures	Severity reduction measures	P	S	C
Inert gases : N2	100	3	1	2	3	1		Systematic N2 analysis after shutdown before resuming operation Or specific purging procedure		1	1	
Inert Gas Ar		2	0	0	2	1				2	1	
Oxygen	5	0	1	2	2	0				2	0	
Carbon dioxide	2	0	0	0	0	1				0	1	
Carbon monoxide	0,2	4	0	0	4	2		CO adsorber at HRS design margin 100% + Operation procedure for replacement when H2 quantity purified = 50% of design capacity.		1	2	
Methane (CH4)	100	2	0	0	2	1				2	1	
Water	5	0	0	1	1	4		Check water at commissioning and after maintenance involving opening of vessels or piping. Measurement shall be done at appropriate location downstream of the considered		0	4	
Total sulphured components	0,004	0	0	1	1	4		Check sulfurs at commissioning and after maintenance involving parts modification (piping, valves, seals, gaskets). Not required for part replaced by identical component.		0	4	
Ammonia	0,1	0	0	0	0	4				0	4	
Total hydrocarbons	2	0	0	2	2	4		Oil/grease cleaning at commissioning and after maintenance. Compressor surveillance depending on compressor technology (coalescing filter?) THC analysis or commissioning and after maintenance		0	4	
Formaldehyde	0,01	1	0	0	1	2				1	2	
Formic acid	0,2	0	0	0	0	2				0	2	
Halogenated compounds	0,05	0	0	1	1	4		Halogenated analysis at commissioning (species must be defined) or after maintenance		0	4	
Helium	300	0	0	0	0	1				0	1	

4 – example from SMR source

❑ When the result is green, the control of this impurity is acceptable. No additional barrier is necessary and this impurity has no reason to be controlled at the HRS nozzle.

❑ When the result is yellow, which is the case for nitrogen, the conclusion is to further investigate the means to decrease occurrence probability:

- In addition of the existing analysis at SMR plant, it is necessary to measure N2 at the commissioning of the HRS and after each maintenance where some parts of the system are open to air.
- Or applying specific purge procedure at the HRS which guarantees to reach a value within specification.

4 – example from SMR source

- When the result is red, it is necessary to reduce the probability of occurrence or to decrease the severity to bring back the risk at an acceptable limit. Additional barriers must be added. These barriers are studied case by case.

- For CO the conclusion in this example of risk assessment may be:
  - to add a purifier with proper capacity of purification and to define a strict procedure for the purifier replacement.
  - Or to add a continuous analysis of CO and a shutoff valve at the inlet of the HRS (at pipeline connection)

- For H_2O the conclusion is to measure H_2O at the commissioning of the HRS and after each maintenance involving opening of vessels or piping or replacement of one of them. This analysis could be done at low pressure to have more sensitivity.

Air Liquide creative oxygen

Hydrogen

- **Duration: 36 months, start date: 1 June 2016**
- **Consortium:** **5 National Metrology Institutes**
  5 key industrial and research partners in fuel cells, storage devices and hydrogen-related technologies
- **Coordinator: LNE**

- **2 collaborators**

	Unit	Sample 1-1	Sample 1-2	Sample 1-3	NMIs
Methane	µmol/mol	< 0.5	< 0.5	< 0.5	SP
C2 hydrocarbons	µmol/mol	< 0.5	< 0.5	< 0.5	SP
C3-hydrocarbons	µmol/mol	<1	<1	<1	SP
C4-hydrocarbons	µmol/mol	<1	<1	<1	SP
C5-hydrocarbons	µmol/mol	<1	<1	<1	SP
C6 – C18 hydrocarbons	µmol/mol	<0.050	<0.050	<0.050	SP

No unexpected compounds or C6-C12 hydrocarbons (SP)

		Results with expanded uncertainty (k=2)			
Compounds	Unit	Sample 2-1	Sample 2-2	Sample 2-3	NMIs
CO	µmol/mol	< 0.01	< 0.01	< 0.01	NPL
CO_2	µmol/mol	< 5	< 5	< 5	SP
CO_2	µmol/mol	< 0.01	< 0.01	< 0.01	NPL
CH_4	µmol/mol	< 0.01	< 0.01	< 0.01	NPL
CH_4	µmol/mol	~ 0.01	~ 0.01	~ 0.01	VSL
Non methane hydrocarbons	µmol/mol	< 0.01	< 0.01	< 0.01	NPL
H_2O	µmol/mol	< 0.5	< 0.5	< 0.5	NPL
Total sulphur compounds	µmol/mol	< 0.0036	< 0.0036	< 0.0036	NPL
O_2	µmol/mol	Not measured	< 5	< 5	CEM
O_2	µmol/mol	< 0.5	< 0.5	< 0.5	NPL
N_2	µmol/mol	< 100	< 50	< 60	SP
N_2	µmol/mol	Not measured	Not measured	< 80	CEM
N_2	µmol/mol	< 1.2	< 1.2	< 1.2	NPL
Ar	µmol/mol	< 30	< 30	< 30	SP
Ar	µmol/mol	Not measured	Not measured	< 80	CEM
Ar	µmol/mol	< 0.5	< 0.5	< 0.5	NPL
Total halogenated (HCl)	µmol/mol	< 0.005	< 0.005	< 0.005	VSL
CH2O	µmol/mol	< 0.005	< 0.005	< 0.005	VSL
CH2O2	µmol/mol	< 0.1	< 0.1	< 0.1	VSL
NH3	µmol/mol	Not measured	< 0.1	< 0.1	VSL
He	µmol/mol	Not measured	Not measured	< 50	CEM

4 – example from SMR source

In conclusion

- ❑ For commissioning : at least measure N2, CO, H2O, THC, total sulfur, and halogenated products
- ❑ After maintenance: measure N2, H2O, THC
- ❑ The analysis of other impurities is not necessary as soon as there is analysis of CO and N2 at production site.

5 – Conclusion

Quality Assurance is necessary to:

- ❑ Reduce the cost of analytical control of Hydrogen quality
- ❑ Maintain a high level of guarantee for Hydrogen car manufacturers and users.
- ❑ Improve the supply chain by addition of barriers to avoid introduction of impurities.

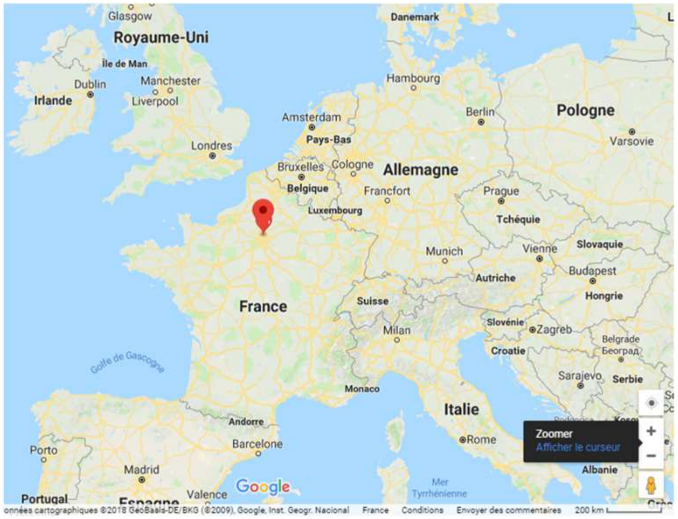
Analytical laboratories are necessary for :

- Making the analysis required for commissioning
- Making analysis according to the quality assurance plan
- Improve the supply chain by addition of barriers to avoid introduction of impurities.
- Building a database of analytical results according to the supply chain in order to improve the quality assurance plan and to revise the quality standards

5 – Conclusion

European laboratories able to analyze all impurities given in EN 17124 at lowest cost are necessary

- Air Liquide will offer new analytical service for the 13 impurities in hydrogen
- New dedicated facility for European market located near Paris.
- Global offer including sampling vessels, sampling service and analysis



Thank you for your attention

4 – example from SMR source