REPORT - Minutes of Panel Discussion Session 1

What are the next steps?
Session 1: *Flow Metering*
VSL, Delft - 11 September 2019

Chair: Frank Schramm (Heinrichs)

Participants: Marc de Huu (METAS), Marc McDonald (NEL), Remy Maury (LNE-LADG/Cesame Exadebit SA), Sam Bartlett (NPL).

Frank Schramm spoke about the challenges manufacturers of H_2 flow meters encounter with regard to certification. As a first hurdle, he named different EMC requirement for the OIML R 137 and R 139, which causes dual costs and responsibilities for the manufacturer. Further, he noted that environmental requirements were higher in the R137. Environmental tests are expensive, can take quite some time, and are sometimes impossible for a lab to conduct. This could be solved with simulations, which are very close to real conditions, but unfortunately not always accepted. Another challenge is a difference in accuracy tests, with different Qmin and Qmax values. Further, Mr Schramm stated that the R 139 is applied to dispenser stations, which his company also has in the US where a different national standard is obliged with deviating MMQ values. Different standards may require different flow meters, which is costly. Another issue mentioned was material use. We are obliged to use HB160 metal for parts. In Japan the 360 TI is used. Changing requirements and standards is costly and takes much time. As a last challenge, Mr Schramm noted that there is no test possibly for rig for flow meter manufacturers.

Conclusion: National standards should be harmonized. Starting in Europe.

<u>Panel discussion:</u>

Q 1: Remy Maury: you said that setting up an operation station takes three days, which is quite long. Does it take less time in a busier station?
A: Setting up takes 2 hours, refueling 5 minutes, however, venting the tank takes ages. We need to find new ways to do this. Type 4 tanks have to be drained very slowly, to avoid collapse in the liner. These gravimetric measurements are done seven times a day for three consecutive days, so the overall timeframe needed is long.
Marc de Huu: If you only do OIML R139, it takes less time. We plan to work on this topic in MetroHyVe 2 to find solutions.

Q 2a: Are there plans to verify T- and p-dependencies of the Coriolis flowmeters in the field?
A: Marc McDonald: No. First comes verification of reproducibility under controlled lab environment.

Q 2: Do you look in your next project at gas grid flow metering for the calibration of H2 metering, just like water calibration?
A: McDonald: Transfer of NG gas grid metering to H_2 metering was not considered.
A: de Huu: There is no point in doing that, because water calibration is the favored approach.

Q 3: Do impurities like argon in H_2 affect the gravimetric analysis?
A: Marc de Huu: Maybe it would break the car by damaging the fuel cell.

Q 4: What if you have 100 ppm of, e.g., argon?
A: Ole Kjos: Impurities in H_2 would have an impact on gravimetric standards due to their different molar mass.
A: Marc de Huu: What you are comparing is what the flowmeter shows and what you gravimetrically measure. It will probably influence the range of the car, but the issue is what you have bought: a customer is agreeing to pay for hydrogen of ISO quality, not perfectly clean hydrogen.

Q 5: Would it affect the Coriolis flowmeter?
A: That is a tricky one, it just measures the average density, and I am not sure how gas behaves at 700 bar, and its flow speed. **FrankSchramm**: the Coriolis flowmeter does not care about the density. It directly measures the mass.

Q 6: Did you consider using helium?
A: Marc de Huu: It is just about the money. First, helium is an expensive gas. Second, you need special equipment, as flow meters and most systems are not well adjusted to helium. Transitioning to helium would need additional qualification of methods and setups. Further, producers are looking for water or nitrogen comparison calibration, because the measurement methods are already qualified for these fluids.

Q 7: Frank Schramm for representative from Shell: we talk a lot about energy density. I heard that there might be a 10% difference in energy density depending on where you buy your gasoline. Do you know more about this?
A: I am sure Shell knows very well which quality they offer at which refueling station. We can discuss the numbers in the break.

Comment Marc de Huu: Harmonization of US [0.5 kg] and European [1 kg] MMQ is highly recommended. Adoption of the NIST value of 0.5 kg will - at current state - pose a problem for gravimetric systems.

Comment Frank Schramm: The lack of harmonization is a hindrance for approval of stations and their components across multiple states.

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REPORT – Minutes of Panel Discussion Session 2

What are the next steps?
Session 2: *Hydrogen Purity*
VSL, Delft - 11 September 2019

Chair: Hidenori Tomioka (ISO/TC 197/WG27 ´Hydrogen fuel quality´)

Participants: Janneke van Wijk (VSL) and Thomas Bacquart (NPL)

Hidenori Tomioka gave a presentation about standardization activities in the field of hydrogen. He provided insight in the work of ISO/TC 197 *Hydrogen Technologies,* and specifically for WG 27 *Hydrogen fuel quality,* and WG 28 *Hydrogen quality control*. He gave an introduction about the responsibilities of these WG's, the progress of their standardization activities, and provided insight in their standards, the ISO 14687 (WG27) and ISO 19880-8 (WG 28), and discussed how those standards relate to each other.

- **ISO 14687 -** *Hydrogen fuel quality:* This standard is currently in the progress of being revised. He emphasized that H2 quality standards need to be mature enough to meet the large hydrogen and FCV market. Further, he argued that that standards need to be prepared for the 'bad guy' in the market.
- **ISO 19880-8** *Hydrogen quality control:* This standard will be revised right after the ISO 14687 publication. He noted that the standard provides two ways of quality control for the whole supply chain: 1) prescriptive methodology and 2) risk assessment.

Panel discussion:

Q 1: Merit Bodner [Nedstack]: Were there any studies on the cost impact of analysis. For example, how all quality analysis is translated to €/kg of H_2?
A: Janneke van Wijk: We focused more on the technical aspect. It is costly to do analysis, between the 4-8k€ per analysis. Multiplying this with the number of analyses per ton hydrogen, you can see the impact on the cost of hydrogen.
Comment Martine Carré: AirLiquide did a study of contribution to costs per contaminant per analysis: total sulfur, total halogenated and formaldehyde together account for 60% of current costs. A separation of total compounds into particular species might reduce complexity of analysis and thus cost.
Comment Thomas Bacquart: In Europe, a full analysis is about 4 – 8 k€. Currently, it is up to the H_2 distributors [HRS holders] to judge how often they need or want to do a full analysis. This way the amount of on top cost on hydrogen due to analysis can be steered / controlled.

Q 2: Thomas Bacquart: We need to gather information on how every lab quantifies the total compounds [HCs, halogenates, sulfur compounds] to get the same information independent of the laboratory that is conducting the measurement. For the next revision of the H2 fuel standard, we need to specify clearly the compounds to be measured instead of 'totals'.
A: Hidenori Tomioka: It is very difficult to specify. [For the codes and standards] it is hard to keep up with the real world progress. Previously, the technology was lagging behind the C&S, now it is the other way around. We will try to improve on this during the next revision.
A: Martine Carré [AL]: Risk assessment could provide a tool for species identification. For example if you are using a halogenated cleaning agent, you need to know. It is both: analytical data and risk assessment for what you need.

A: Hidenori Tomioka: It is a combination of [the] two [methods].
A: Jari Ihonen [VTT]: Halogenated species have a short-term and a long-term effect. The short-term effect is adsorption. The long-term effect is that they are accelerating the particle dissolution. If you are shutting down the system, the anode potential stays low. So that the chloride effect is not that big on the platinum. It has been a topic over the years.
Comment: Hidenori Tomioka: Nevertheless we need to take care about that, halogenates, to prevent cars from breaking down.

Comment: public (Toyota): We are moving in a phase of cost reduction, and we are also learning new things. We have seen sulfur. We really believe risk assessment is a good approach, but also look at new things. If there is a move on the fuel side, it might cost more on the care side, e.g. platinum.

Comment: Thomas Bacquart: I am really happy that we have a real collaboration between EU/US/Asia. I think what is important from a laboratory point of view is to define different things to be measured. It is not restricted to the standard. It is what you want; if there is something to be changed from a laboratory point of view, it can be the standard.

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REPORT – Minutes of the Panel Discussion Session 3

What are the next steps?
Session 3: *Impurity Risk Analysis*
VSL, Delft - 12 September 2019

Chair: Martine Carré (Air Liquide)

Participants: Jari Ihonen (VTT), Sam Bartlett (NPL), Christian Spitta (ZBT)

Martine Carré first gave a presentation. For better insight and general overview, she showed the interrelations between these topics, as well as the whole chain of hydrogen (production-application). The aspects 'where' and 'when/how many' measurements are considered to be of importance (to know whether these are feasible, also money-wise).

Furthermore, she gave an insight into how to define the quality assurance plan and how to select the laboratory(s) for analysis.

Slide 14 about 'Industrial needs' provided very useful hints when dealing with risk assessment. She shared her view for the need of more data (for example via a network of NMI's).

REMARK: Martine also clarified that, in the ISO standards, it is **not** mandatory to do *online measurements*. Though measurements are mandatory, online measurement is just one of the options mentioned in the standard.

The panel discussion followed the needs and future work of NMI's:
Q 1: Vincent Mattelaer (Toyota): Do you advise for a new station to do a full analysis or to first develop the methods/knowledge and do the risk assessment [to limit the species to check for]?
A: Martine Carré: This is currently under discussion. Also because to eventually evaluate not only 1 rule/method, but to keep more options open for different approaches. For the time being, the recommendation is to do a full analysis and then readjust the time frame of the recurring full analysis.

Q 2: Ole Kjos: Splitting up the analyzers, would that be an option (different stages in the process, different types of measurements), also to lower the costs?
A: This was agreed [at Air Liquide]. Therefore, more knowledge of impurities at the production process is needed first.

- First, more knowledge about the processes (production, transportation, HRS technology) is needed.
  - A short discussion followed about thresholds to be set.
- Further knowledge behind the actual analytic requirement [at each stage in between] is needed. For a supplier, it is unimportant to exactly know how far below a threshold an impurity is, but just that it is below the threshold.
- This simplified approach is needed because suppliers are usually asked to take the costs for analyzers alone, but in the end, this could be too expensive for the supplier [if simplification is not accepted].
- And it was added that different thresholds for different production systems could be used

Q 3: Peter Bout (AirProducts): Key is the price of *good* hydrogen. Is not that up to the customer's needs?
A: The balance between reducing costs and increasing quality is the key. The 'purity' is what it is about (containing 'impurity x' with a max. and 'impurity y' with a max.). So we should move away from the discussion about 'quality' [defined in the standards by the hydrogen index].

Q 4: Martine Carré: The presented online particulate measurement devices are counting the particles and are measuring the size [distribution]. How does the mass calculation work (asked to Sam Bartlett), because the ISO standard explicitly asks for a result in mg/kg.
A: Sam Bartlett: The answer has to follow, because he is not the technical expert (very detailed). This will be asked to his NPL colleagues involved. (information on this will be provided afterwards)

Q 5: Thomas Bacquart: In the USA, the data [of contamination incidents] is collected [anonymized in a database], in the EU there is data, but it is not shared yet. Should not we think about how to get this information shared at whatever level available (including funding)?
A: Martine Carré mentions the European network of Gas Analysis – It could be good to try to create a project around this topic together with this network.

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REPORT – Minutes of the Panel Discussion Session 4

What are the next steps?
Session 4: *Sampling*
VSL, Delft - 12 September 2019

Chair: Ward Storms (Toyota Motor Europe)

Participants: Ole Kjos (SINTEF), Ward Storms (Toyota Motor Europe), Thomas Bacquart (NPL)

Ward Storms from Toyota Motor Europe held a presentation, to give share a point of view from (his part of) the industry.

Panel discussion:

Q 1: Have there been any studies of the materials in tanks, where hydrogen is stored [at the station], compared to sampling vessels (and could these tanks absorb different contaminants than uncoated sampling vessels)? Do they have an impact on supplied H_2?
A: Thomas Bacquart: As the sampling vessels are coated for the very specific objective of analytic tests, they are expected to behave differently from what is currently used for storage at the station. In addition, data is not available about what kind of material is used at the stations.
Comment: Ole Kjos: The stations might not be worried about details of adsorption properties (as long as at the station contamination limits are not exceeded) and that they are in the first place focused on safety aspects. Of course, secondly, it is considered important that the overall system impurity levels are not exceeded.

Q 2: Christian Spitta [ZBT]: The overpressure release solution of the Linde qualitizer would most likely not pass today's safety requirements in Germany and German HRS holders do not like any sources of hydrogen release. How is the situation in other countries?
A: Ole Kjos [SINTEF]: During the HYDRAITE sampling campaign the minor release of hydrogen during and after sampling was not an issue with the station owners. Nevertheless, a connection to a vent would be good.

Q 2: The redesign of overpressure release path (currently simple release valve on the Linde qualitizer) was mentioned as an option for new sampling systems, so could the design of the venting method and path be standardized?

- This could be a showstopper (as it is expensive, and as no hydrogen would be allowed to be released).
- **Comment: Christian Spitta [ZBT]:** It is not yet standardized, but already possible in sampling tenders in Germany. The new sampling systems [currently in development in HYLAB project] have safety installations through which hydrogen is vented to either a mobile chimney or - if permitted by the station owner and CEP – to the HRS chimney. This may be a long-term solution.

A: Other back-up safety measurements are possible (ASTM-based).

Q 3: Ward Storms [Toyota]: Lastly, an elaboration followed on capturing light and reactive contaminants like formaldehyde on sorption tubes.
A: Thomas Bacquart [NPL]: Development efforts may already exist. Karine Arrhenius (MetroHyVe WP 4 'Sampling' leader) possibly has more information on this.

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REPORT – Minutes of the Panel Discussion Session 5

What are the next steps?
Session 5: ***Impact on Fuel Cells***
VSL, Delft - 12 September 2019

Chair: Merit Bodner (Nedstack)

Participants: Merit Bodner (Nedstack), Jari Ihonen (VTT), Sylvie Escribano (CEA), Ulrich Misz (ZBT)

Merit Bodner (Nedstack) held an informative presentation about hydrogen supply, with the challenges and opportunities, based on Nedstack's experiences. She elaborated on lessons learned, for example using existing infrastructure (natural gas piping), with implications on resulting purity after extraction, etc.). She mentioned that it is not a 'perfect world' outside.

Panel discussion:

Q 1: Jaco Reijerkerk: What happens with permeating oxygen in the absence of CO?
A: Jari Ihonen: Typically, all the oxygen is consumed at the catalyst layer by direct reaction with hydrogen. In the presence of CO, oxidation of hydrogen is lowered - the more oxygen can be detected in the loop. This increased O_2 content at the anode inlet - in a recirculation scenario - may in turn help mitigate CO poisoning: it works as an internal air bleed, which can additionally be enhanced by catalyst formulation.

Q 2: Jaco Reijerkerk: Hydrogen soak seems to be the current trend in shut down procedures. Valves at the cathode inlet and outlet were recommended. However, what are the alternatives if there are not yet any valves?

Comment: Freezing conditions mean leakages in the long run because of potential ice formation. You should also dry it [the overall system] first and then do the closing. Non-freezing conditions are not a problem. It seems OEMs are positive about this.

Q 3: How do these impurities influence lifetime?
In this project, the lifetime aspect is not included (for example related to 'chlorine growth'). However, it is considered important, as the effect of contaminant can grow slowly. Our scope [in the HYDRAITE project] is are short-term effects at stack level comprising max. 2, 3, 4 days.

Q 4: Ward Storms: With regard to the varying impact of sulfur on CO tolerance in the first tests, are there already any more explanations?
A: Ulrich Misz: The observed irregularity in CO tolerance after sulfur contamination is a new and unexpected effect. One considered possibility is a subsequent conversion to carbonyl sulfide (COS) after prior contamination, but there may also be other explanations.

> **Jari Ihonen**: To our best knowledge, this is the first time sulfur contamination was studied with an anode gas recirculation setup. An intensive discussion is going on with a lot of speculation. Sulfur contamination has effects on CO tolerance, but we [HYDRAITE members] still need to find out the reasons.

Q 5: Martine Carré: Will there be recommendations about CO and sulfur available to the ISO TC for the revision of the ISO standards, which is starting soon? Sulfur seems more difficult.

A: Sylvie Escribano: Information will be made available, within the project's life time. Results will be exchanged/shared once conclusive data and explanations are gathered.

Q 6: What contaminants do you see coming through the membrane [from cathode to anode]?
A: Jari Ihonen: Anything with a high permeability (all (types of) contaminants, even sulfur).

Q 7a: What happens if there is no CO_2 in the fuel?
A: This depends, but a few hundred PPM will always appear on the catalyst side of the anode due to permeation from the cathode.

- It was added that carbon dioxide then reversibly poisons it [the anode catalyst] by adsorption and consecutive CO formation.

Q 7b: With CO_2 coming from the cathode anyways, does it make sense to have the [low] threshold level of 2 ppm?
A: Jari Ihonen: Yes, because the higher the content in the H2 fuel, the faster enrichment to levels poisoning the catalyst happens due to recirculation mode.
Comment: Hidenori Tomioka: The set threshold level in the ISO is primarily historical. It came from requirements of storage of hydrogen in metal hydride tanks. The same goes for the 5 ppm threshold level of water.

Q 8: Could more case differentiation be included in the standards?
A: Yes. Sulfur, has primarily a long-term effect; other contaminants have a long-term and/or a short-term effect (such as chlorine compounds). The impact of single contaminants is covered by the standards, but there is hardly any consideration of combined effects.

- It was mentioned that all constituents containing halogenates (e.g., freons) could be considered poisonous if not proven otherwise.
- This assumption resulted in some discussion: it was mentioned that, vice versa, you could state that everything is safe until it is proven unsafe).
- It was also mentioned that additional research might be needed, and some research on the effect of freons might be invested in HYDRAITE's "New Impurities" work package.

Q 9: Do systems (e.g., as the Nedstack systems) contain iridium besides platinum?
A: In general, it was answered that systems could contain iridium.
A: In the HYDRAITE test stacks, Pt catalysts with CRT additives are included in part of the project stacks. Iridium oxide is one of the most-likely used additives, but there is no public information on what the automotive manufacturers use in their stacks.

- They [CRS] are used, but we are speaking of extremely low loading when compared to platinum content.

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Annex 1 Final Program

REMARK Due to circumstances, some speakers were replaced by other speakers (what was highly appreciated).

WORKSHOP ON HYDROGEN QUALITY AND FLOW METERING FOR HYDROGEN FUEL CELL VEHICLES

Day 1: Wednesday 11th September 2019	*Chaired by Sylvie Escribano (CEA) and Jaana Viitakangas (VTT)*
9.00	Registration and coffee (VSL reception)
9.30	**Welcome** Janneke van Wijk (VSL)
10.00	**Introduction to the MetroHyVe and HYDRAITE projects** Janneke van Wijk (VSL) and Jaana Viitakangas (VTT)
10.30	Coffee break
Session 1: Flow Metering	
11.00	**Introduction to hydrogen flow metering** Marc de Huu (METAS)
11.25	**Laboratory calibrations using gas** Marc McDonald (NEL)
11.40	**Laboratory calibrations using liquids** Marc de Huu (METAS)
11.55	**Gravimetric systems and field results** Remy Maury (CESAME)
12.15	**What are the next steps? (Panel discussion)** Frank Schramm (Heinrichs)
13.00	Lunch
Session 2: Hydrogen Purity	
14.00	**Introduction to hydrogen purity** Thor Aarhaug (SINTEF)
14.30	**Analytical methods for hydrogen purity** Thomas Bacquart (NPL)
15.00	**Primary reference materials** Janneke van Wijk (VSL)
15.15	**Dynamic reference standards** Janneke van Wijk (VSL)
15.30	**Laboratory comparisons** Thomas Bacquart (NPL)
15.45	**What are the next steps? (Panel discussion)** Hidenori Tomioka (ISO TC 197 WG27)
16.30	Exhibition, Posters and Social event with BBQ (free for participants)

Day 2: Thursday 12th September 2019	*Chaired by Sylvie Escribano (CEA) and Joana Viitakangas (VTT)*
Session 3: Impurity Risk Analysis	
9.00	**Introduction to impurity risk analysis** Thor Aarhaug (SINTEF)
9.15	**Impurity risk assessment model** Jari Ihonen (VTT)
9.30	**Online gas and particle analysers** Sam Bartlett (NPL)
10:00	**Impurities beyond the scope of ISO 14687** Christian Spitta (ZBT) and Thor Aarhaug (SINTEF)
10.15	**What are the next steps? (Panel discussion)** Martine Carre (Air Liquide)
11.00	Coffee break
Session 4: Sampling	
11.30	**Gas sampling procedures** Thor Aarhaug (SINTEF)
11.45	**Sampling vessels and filters** Sam Bartlett (NPL)
12.00	**Particle sampling procedures** Jordan Tompkins (NPL)
12.15	**Lessons learnt with hydrogen sampling** Ole Kjos (SINTEF)
12.45	**What are the next steps? (Panel discussion)** Toyota Motor Europe
13.30	Lunch
Session 5: Impact on Fuel Cells	
14.30	**Fuel cell impurity measurements** Jari Ihonen (VTT)
15.00	**Reversible impurities** Jari Ihonen (VTT) and Sylvie Escribano (CEA)
15.30	**Irreversible impurities** Ulrich Misz (ZBT)
15.45	**What are the next steps? (Panel discussion)** Merit Bodner (Nedstack)
16.30	End