Overview

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- Analyzer Instrument at point of use
  - Continuous Wave Cavity Ring-Down Spectroscopy (CW CRDS) - up to four contaminants with one device
  - Field and lab measurement techniques
- Gather sample in field at point pf use for in-lab analysis
  - Concentrate contaminants to increase analyzer sensitivity
  - Collect concentrated samples for lab analysis
- HEMS – Analyzer Instrument at point of use
  - Describe how Pd-alloy microchannel membrane works to enhance contaminant concentration by 150 to 700 fold
  - Data demonstrating performance using residual gas MS analysis
- Outstanding issues, challenges
- Plans going forward/Conclusions

Technology for Measuring Contaminants Possibly at Distribution Site

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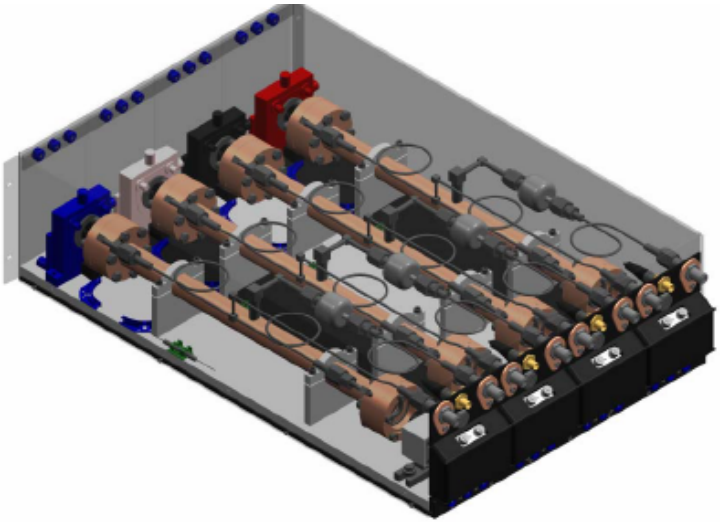
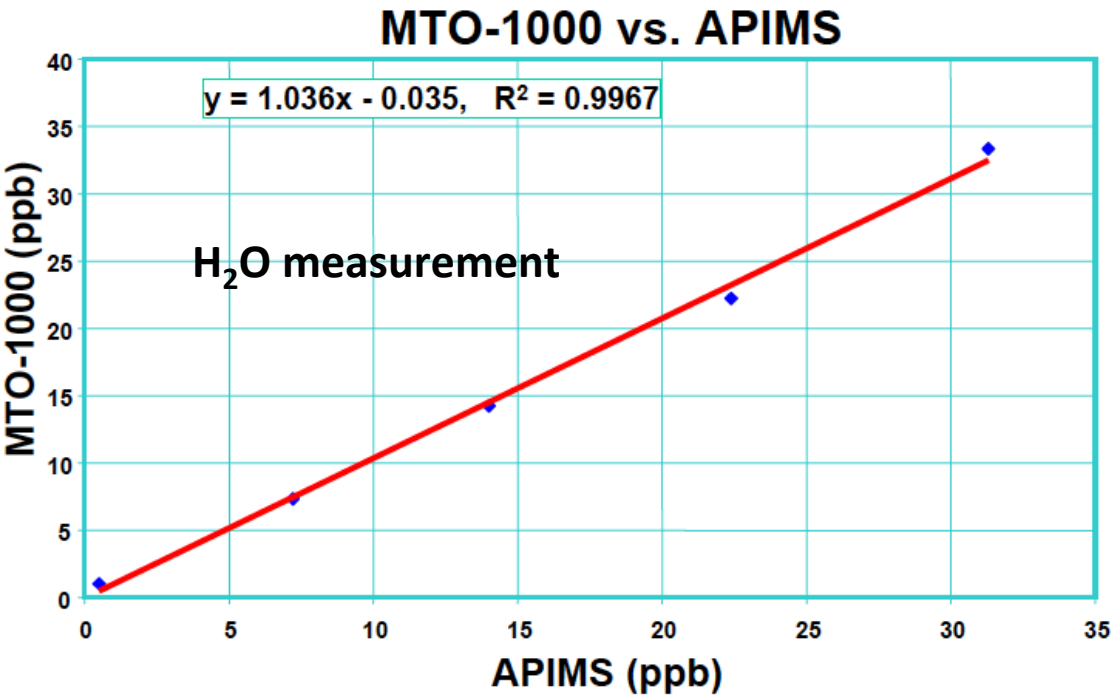
- Continuous Wave Cavity Ring-Down Spectroscopy (CW CRDS) can measure up to four specific contaminants in same device with ppm-ppt sensivity
  - Tiger Optics Prismatic 2 multi species analyzer can measure 4 species
  - Picarro Instruments similar idea
  - Others

Multiple Channels



Low Detection Limit



Lab Analysis of Impurities in H2 Through Enrichment with Pd Membrane

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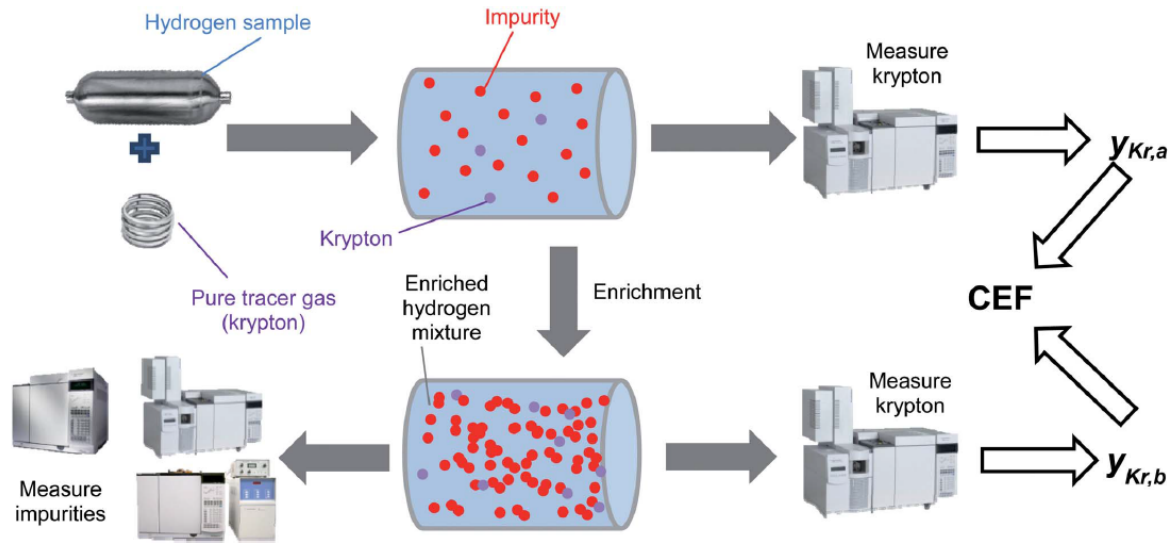

Fig. 3 Schematic diagram of the tracer enrichment method.

National Physics Lab UK, Arul Murugan* and Andrew S. Brown, Royal Society of Chemistry, March 6, 2014

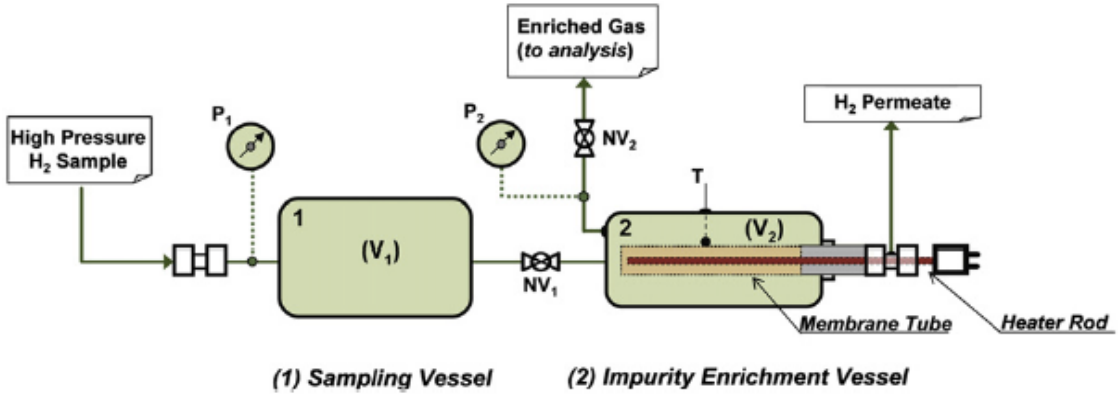

Fig. 1 – Schematic of the gas sampling and impurity enrichment device (GSIED).

Argonne National Lab D. Papadias, S.H.D. Lee, S. Ahmed, International Journal of Hydrogen Energy 35 (201) 12480-12490

Laboratory Analytical methods used on enriched samples: GC-PDHID, GC-MS, GC-SCD

P&E has Developed Novel Technology Platforms

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- **Based on:**
  - **Microchannel tube-in-tube technology**
  - **Palladium alloy micro-channel membranes for separation of H_2**
  - **Inside-out flow configuration**
  - **Robotic Manufacturing and Assembly**
- **Mission: "Enable the Hydrogen Economy and promote energy efficiency through the application of micro-channel technology"**

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How Palladium Micro-channel Works

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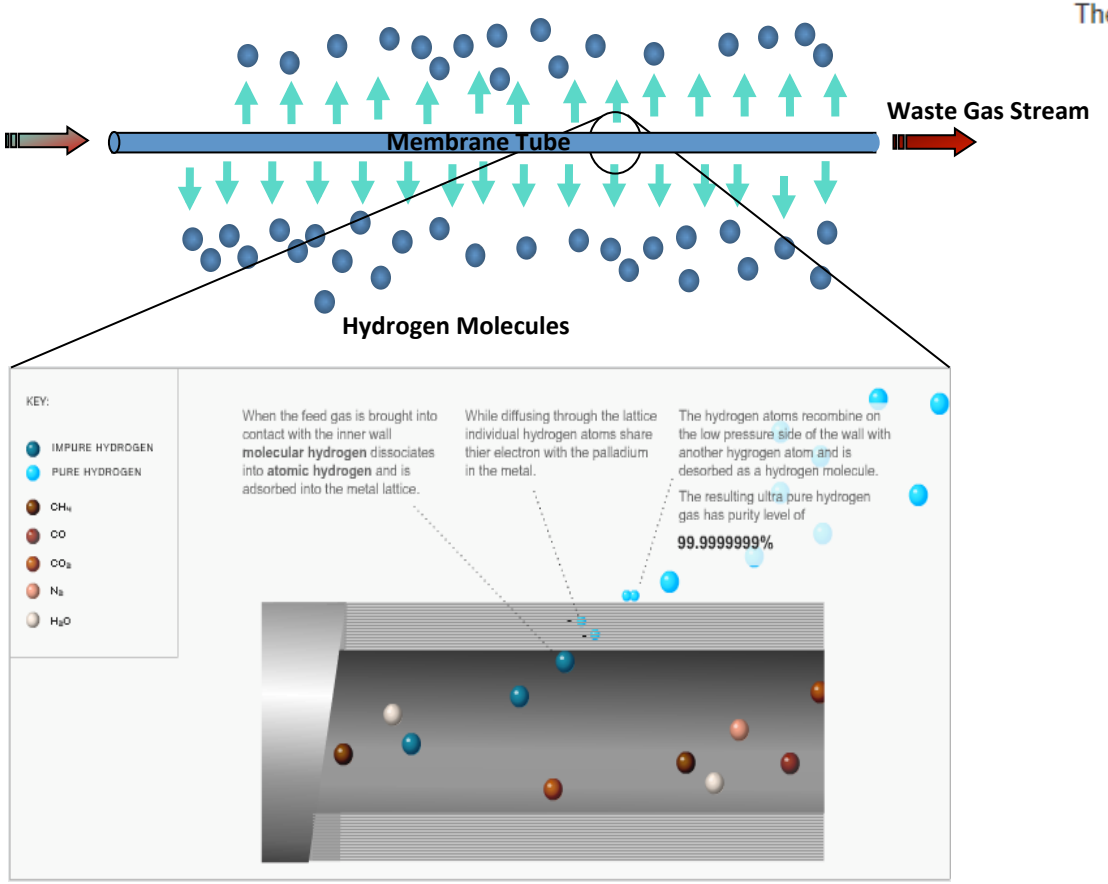

- **Palladium (Pd) separators work by dissolving H_2 into a Pd membrane as H atoms.**
- **Pressure across the membrane 'pushes' the H atoms through the membrane where they recombine. No other gas can pass through Pd.**
- **Pd separators operate at between 380 and 410 ^oC.**

Hydrogen Contamination Detection (HCD) for the FCEV market

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- Need an analyzer which can ensure hydrogen quality at point of use, verify compliance with the standards at hydrogen filling dispenser
- Protect vehicle's FC from contaminant caused degradation
- Analyzer economically viable
  - Drive Cost of analysis low
  - Limited or no use of consumables
  - "Push button", simple to operate
  - At dispenser, no need for operator

P&E's Hydrogen Elimination Mass Spectrometer (HEMS) Analyzer

HEMS: Principle of operation

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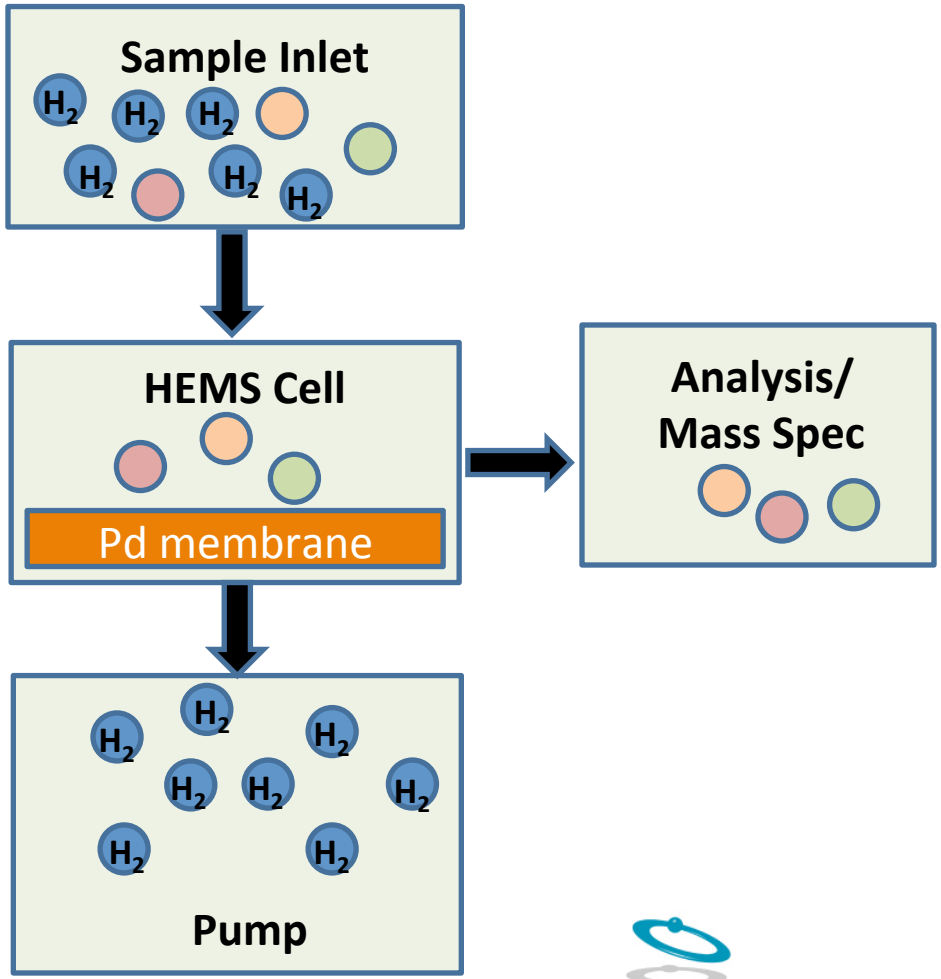
- **HEMS Concentrates Impurities in Hydrogen using Pd-Alloy µchannel Membrane and Performs Analysis in Field**
  - **By increasing the concentrations of impurities it is possible to use lower cost analysis with higher sensitivity**
  - **All surfaces are protected with coatings**
  - **Impurities are measured with a quadrupole MS**
  - **An additional method of separation has been implemented to resolve isobaric interferences**



HEMS Concentrates Impurities in Hydrogen from 150 to 700 times

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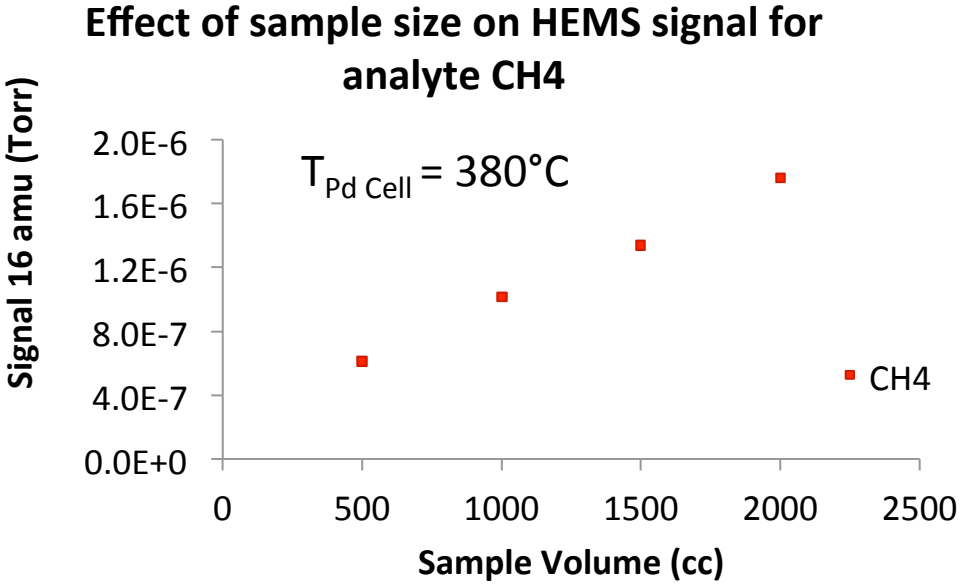
- HEMS concentration and sensitivity depends on sample volume
- The concentration effect can be increased by larger sample volume of gas
  - 500cc is normally used
  - The volume of gas collected is monitored using a mass flow meter and is accurate within 2%
- Palladium cell operates at 380°C
  - This maximizes hydrogen flow rate through the palladium membrane, while minimizing reactivity of the species at the palladium interface



- The volume of the palladium cell (where the impurities are collected) is approximately 3 cc
- Data demonstrates ability to increase concentration of contaminant and thus sensitivity by increasing sampled volume

Analysis Approach Includes Separation of Overlapping Mass Peaks

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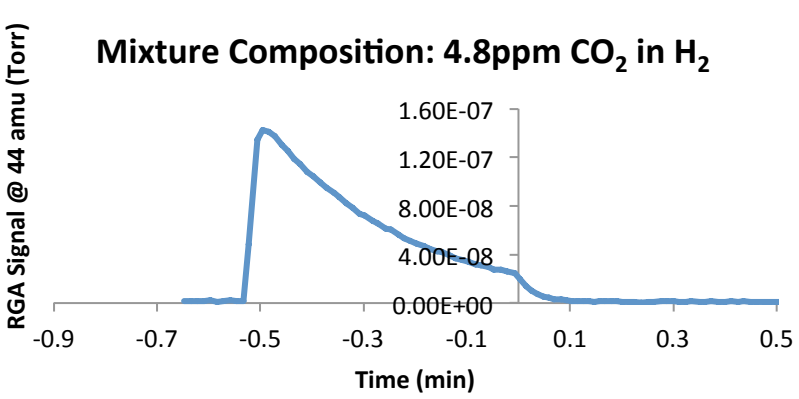
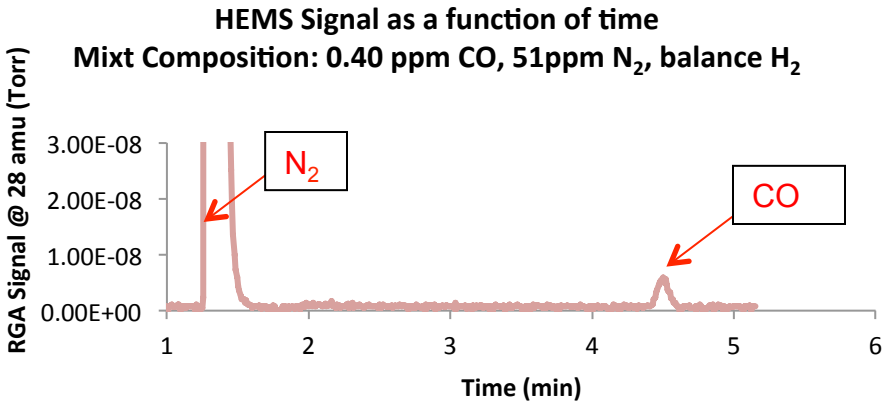
- The species concentrated with HEMS are detected by a Quadrupole MS
  - Species are introduced directly into the MS vacuum chamber
  - A restricted flow orifice limits the maximum signal observed, to meet the dynamic range of the Mass Spec's Faraday cup
- Some species require an additional method of separation in time since they have the same mass within the mass resolution of the detector
  - This is done using additional Pd membranes, without the need of additional consumables making this technology ideally suited for deployment at point of use





At t<0 direct injection of impurities in quadrupole vacuum chamber

At t>0 Additional method of separation

Signal Response for CO

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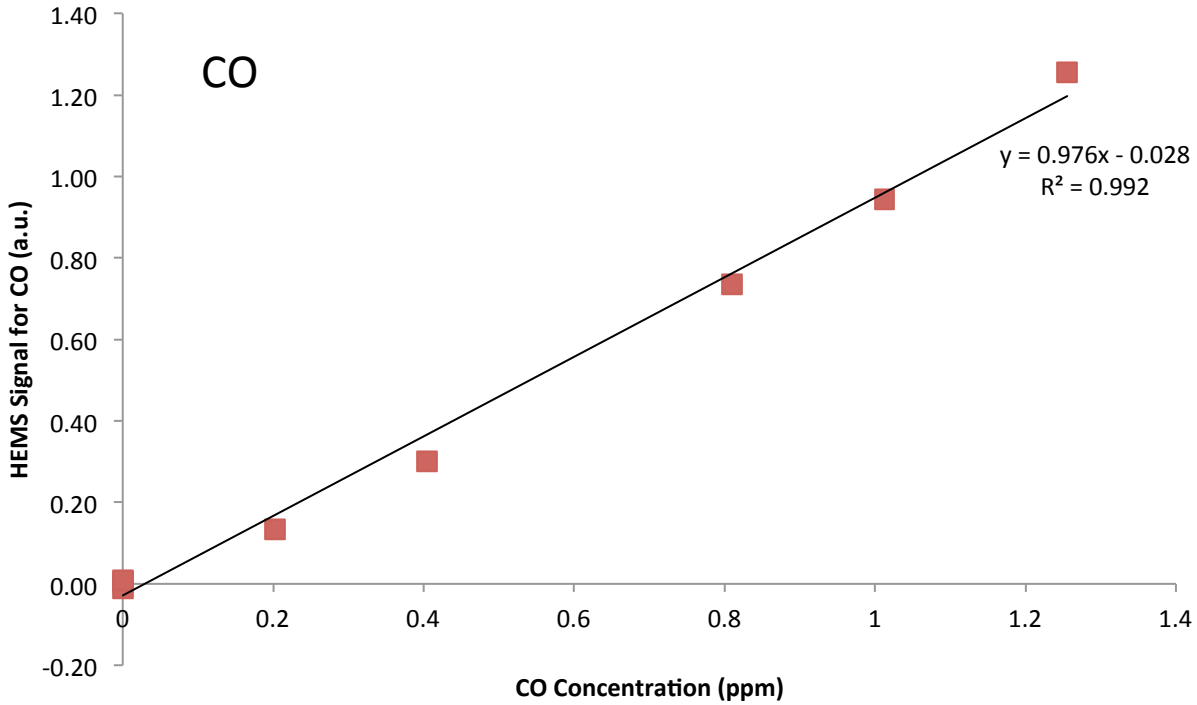
HEMS Analysis for CO in presence of 51 ppm of N_2



Demonstrated capability to separate species with same mass and do concentration measurement of each independently

Signal response : N_2 and CO_2

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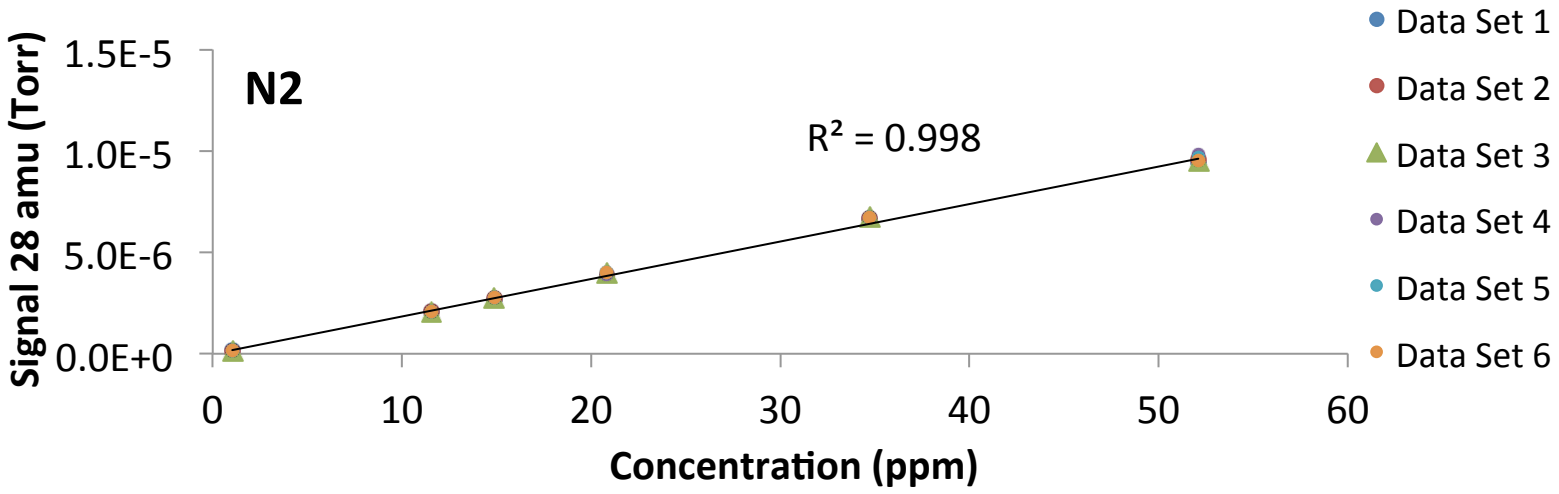

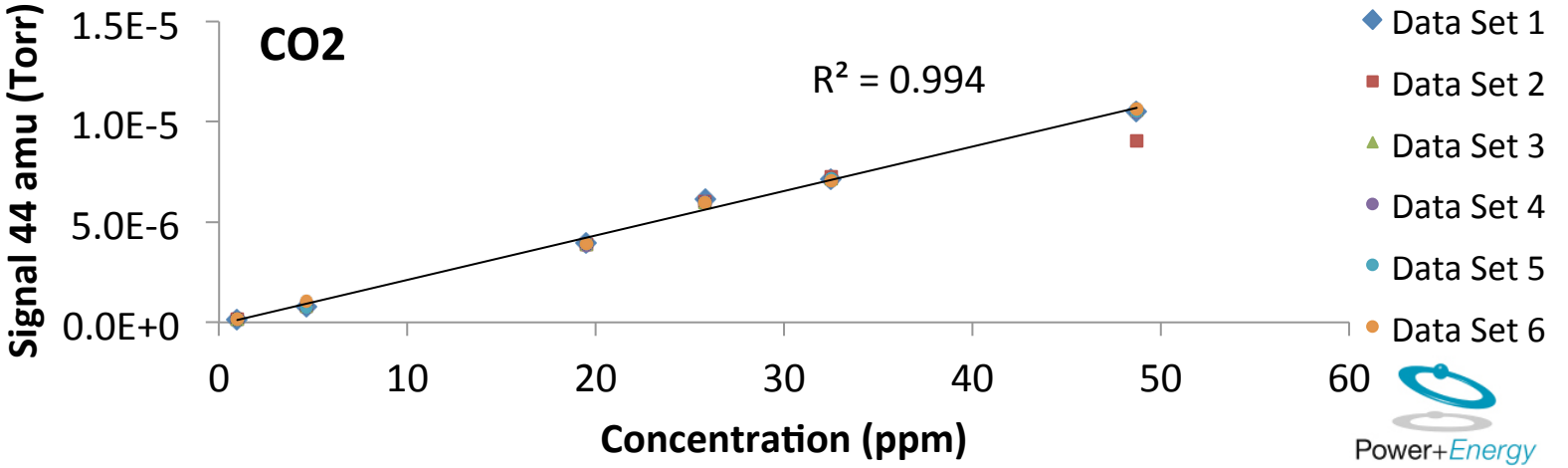

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HEMS: Summary of Analytical Results

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Analyte	Acceptable limit from the standard SAE-J2719 (ppm)	Range of concentrations analyzed (ppm)	R^2 of calibration	RMSEC (*)	RSD (*)
N_2	100	1-50	0.998	2.8%	1.8%
CO_2	2	1-50	0.994	10.6%	4.8%
CO	0.2	0.2-1.2	0.992	4.7%	**
CH_4	2 (for THC)	1-50	0.992	8.7%	3.1%

- * Values are reported at 1σ
- ** Only one data set collected
- H_2S and NH_3 analysis in progress

Current State of HEMS Technology

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- HEMS is new, novel analytical system, continuously improving
- Prototypes in customer hands, improvement opportunities identified
- Commercial device deliveries, includes improvements, in August 2014
- P&E participating in an industry wide round robin test run by NPL-UK
- P&E willing to lend HEMS-M to potential users to evaluate and test its performance in exchange for data and learnings
- Would like to partner with stakeholders, users and potential joint R&D groups to ensure interface issues with all are addressed
- Cost of species specific contamination detection with current device:
  - Assuming 3 year depreciation, 30 cars/day ~$2.00 /vehicle; 100 cars/day ~$0.60/vehicle, 300 cars/day ~$0.20/vehicle
  - See pathway to reduce this by 80% longer term

Outstanding Opportunities

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- **Cost of species specific contamination can be reduced up to 80%:**
  - **Wireless communication of analyte data to FCEV's memory**
  - **Potential to build data base of contaminants specific to source of H_2 and FCEV**
  - **Identify those that are critical, and those that are not depending on H_2 source**
  - **Future HEMS generation to focus on data driven specific targets**
- **Significantly reduce cost of species specific analysis**
  - **Use Hydrogen Elimination as a front end to concentrate species, but add different, lower cost analyzer for the species, such as MEMS or electrochemical for specific analytes, or….**
  - **Stretch potential to cut cost of analysis by ~ 80%**
- **Just measuring total contaminant level left in separator and alarm only if it exceeds a predetermined amount**
  - **Potential to reduce cost of analysis to less than $0.10/vehicle**
- **Looking to partner to explore these options**

Conclusions

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- The results demonstrate HEMS' analytical figures of merit to enable validating compliance with hydrogen purity standard SAE-J2719 and ISO 14687 for the analytes selected during the tests
- Demonstration of H2S and NH3 analyte performance in progress
- No fundamental issues identified, feasibility demonstrated
- HEMS at dispenser doing real time analysis (~8-10 minutes)
- Opportunity to substantially reduce cost of analysis by reducing cost of analyzer
- Reliable, low cost, automated analyzer at dispenser is available

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Thanks for Listening and Asking Questions