



Enlighten Conference and
Vacuum Symposium

12th October 2017

The use of remote plasma emission spectroscopy as an
alternative method of gas sensing for direct monitoring of
vacuum processes

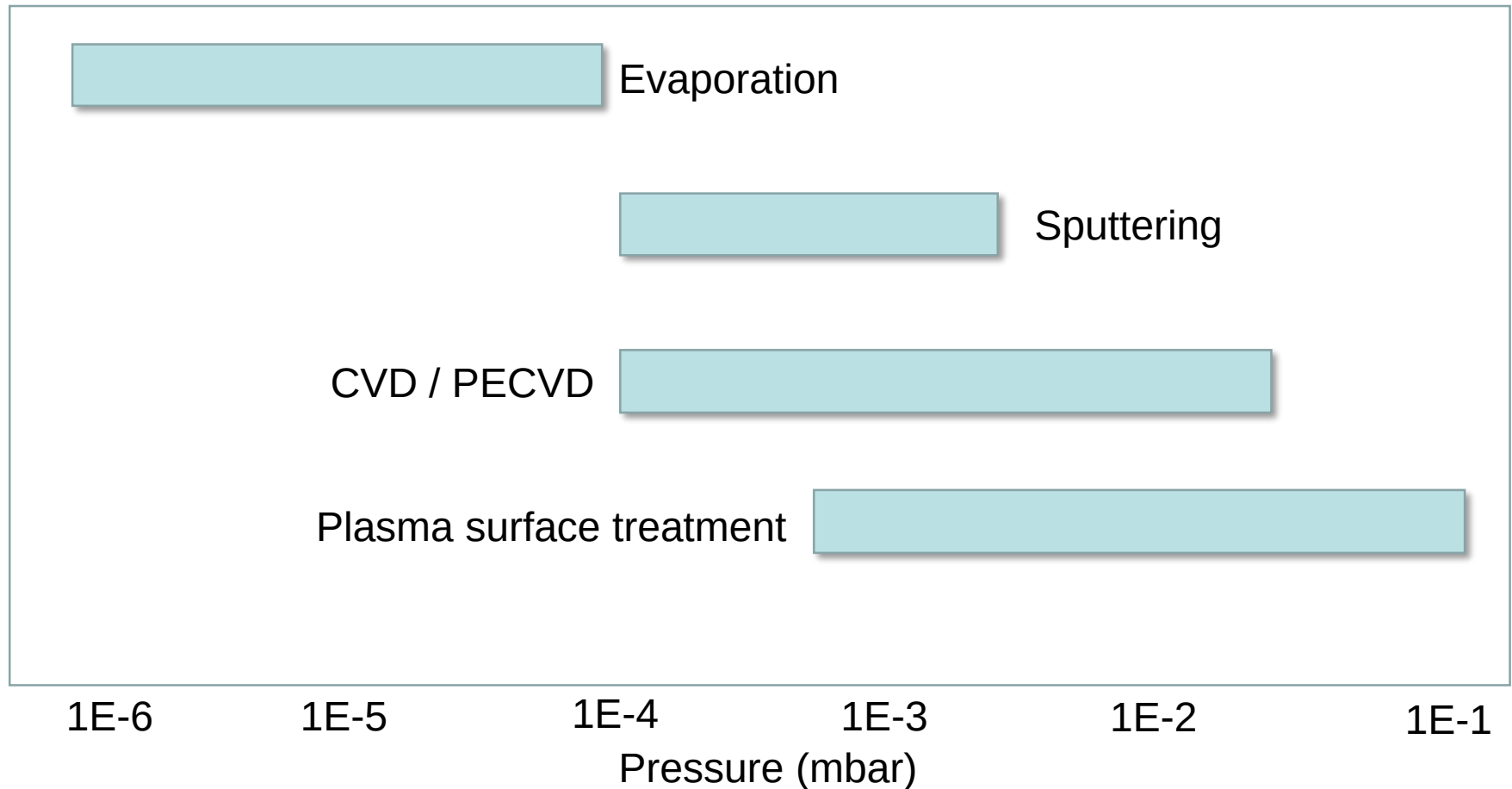
Joe Brindley, Benoit Daniel, Victor Bellido-Gonzalez

Genco Limited, Liverpool, UK

- Motivation for the remote plasma emission sensing technique
- Caveats and considerations when using the technique
- Field trials of the sensing technique
- Conclusions

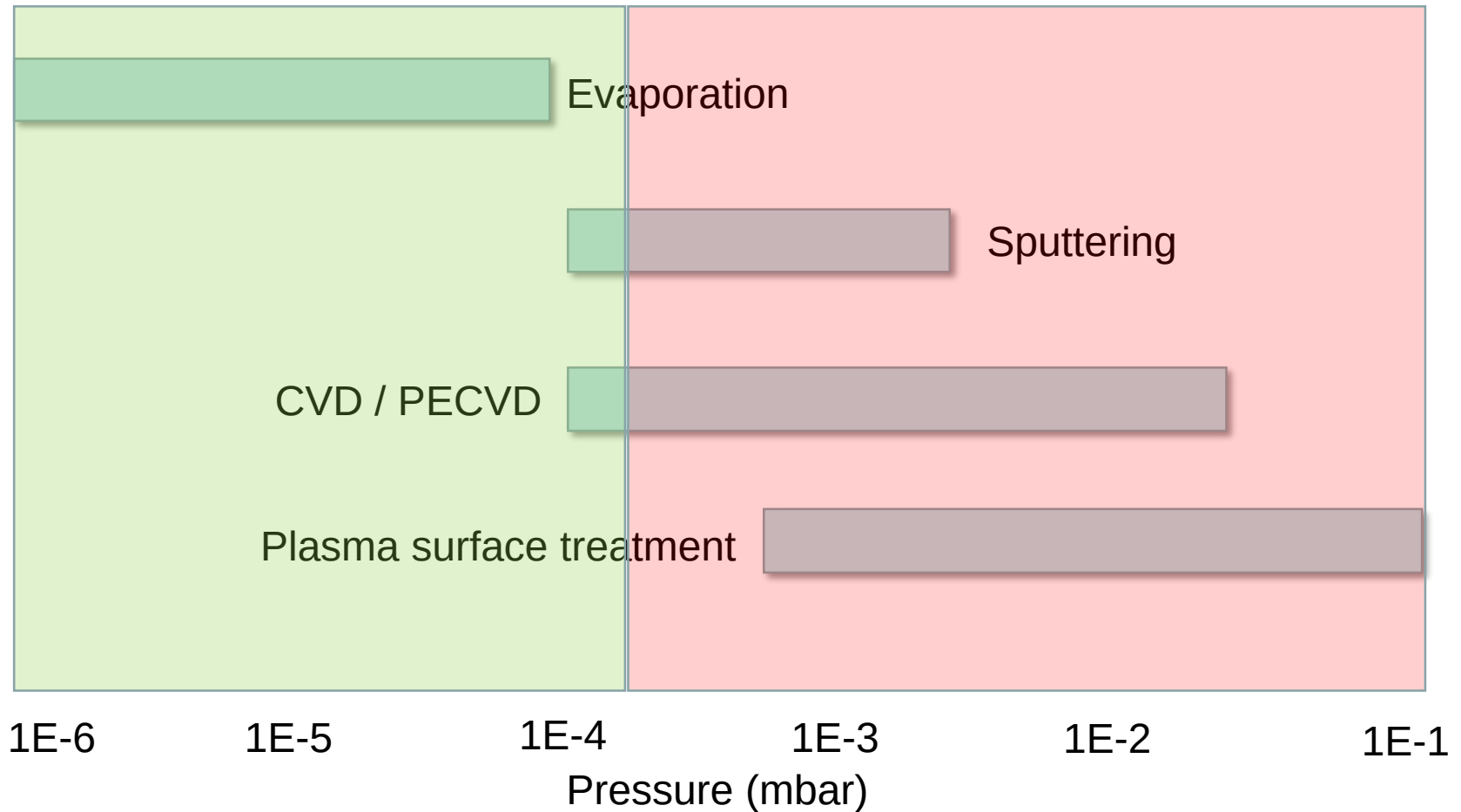
Motivation for the sensing technique

Typical vacuum deposition and surface treatment pressure ranges



Motivation for the sensing technique

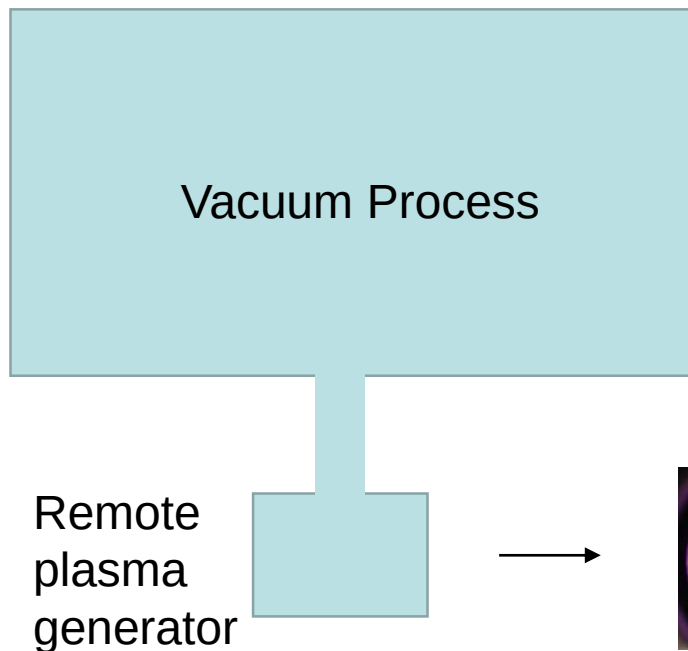
Typical vacuum deposition and surface treatment pressure ranges



Remote Plasma Emission Spectroscopy

- Original concept used by Mann in 1981(!) for leak detection

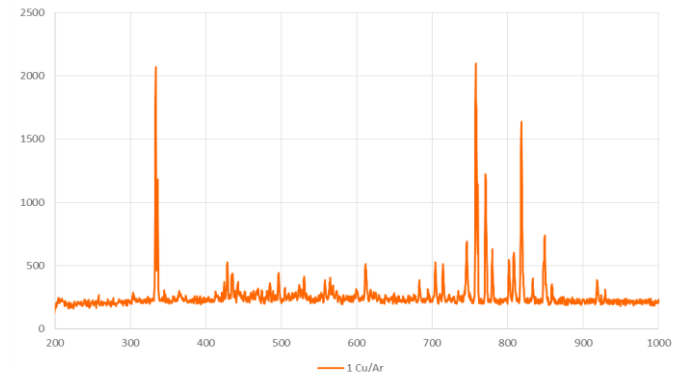
Spectrum analysis
gives species composition



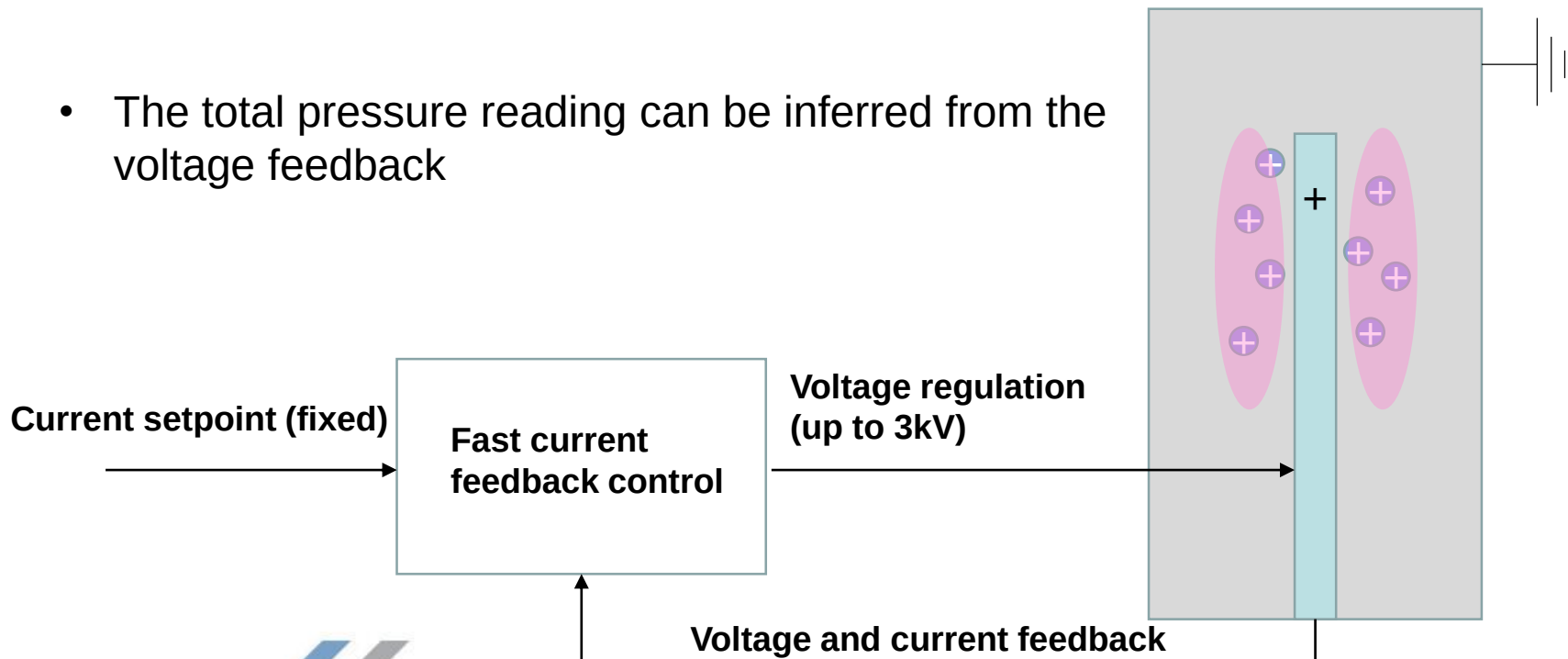
Plasma



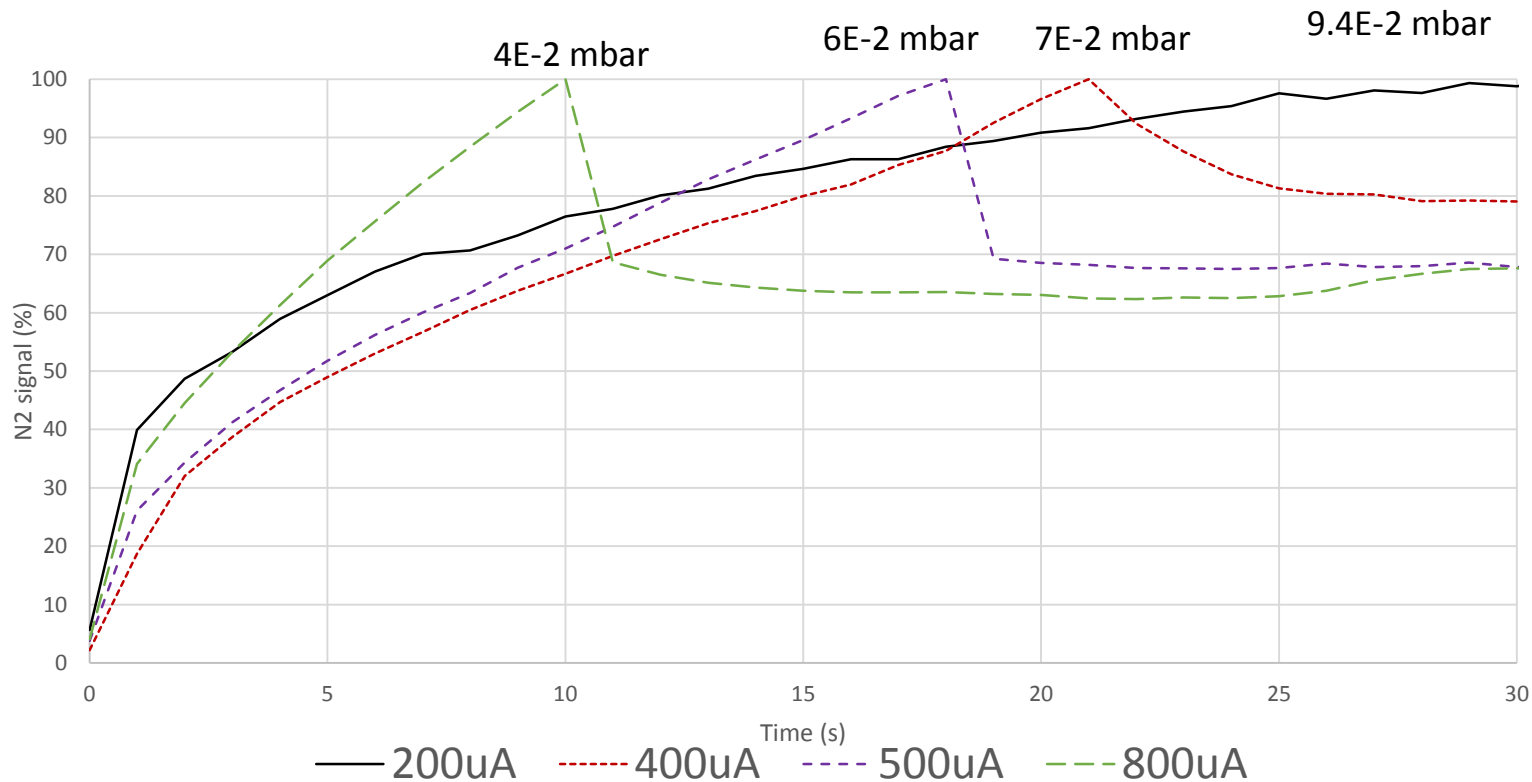
Miniature spectrometer



- Fast feedback control of the current allows for a stable plasma to be generated from **1E-6 mbar to 1 mbar**
- Overall species excitation is determine by the current setpoint.
- The total pressure reading can be inferred from the voltage feedback



- Higher currents give a superior signal to noise ratio but at the expense of upper operating pressure limit.

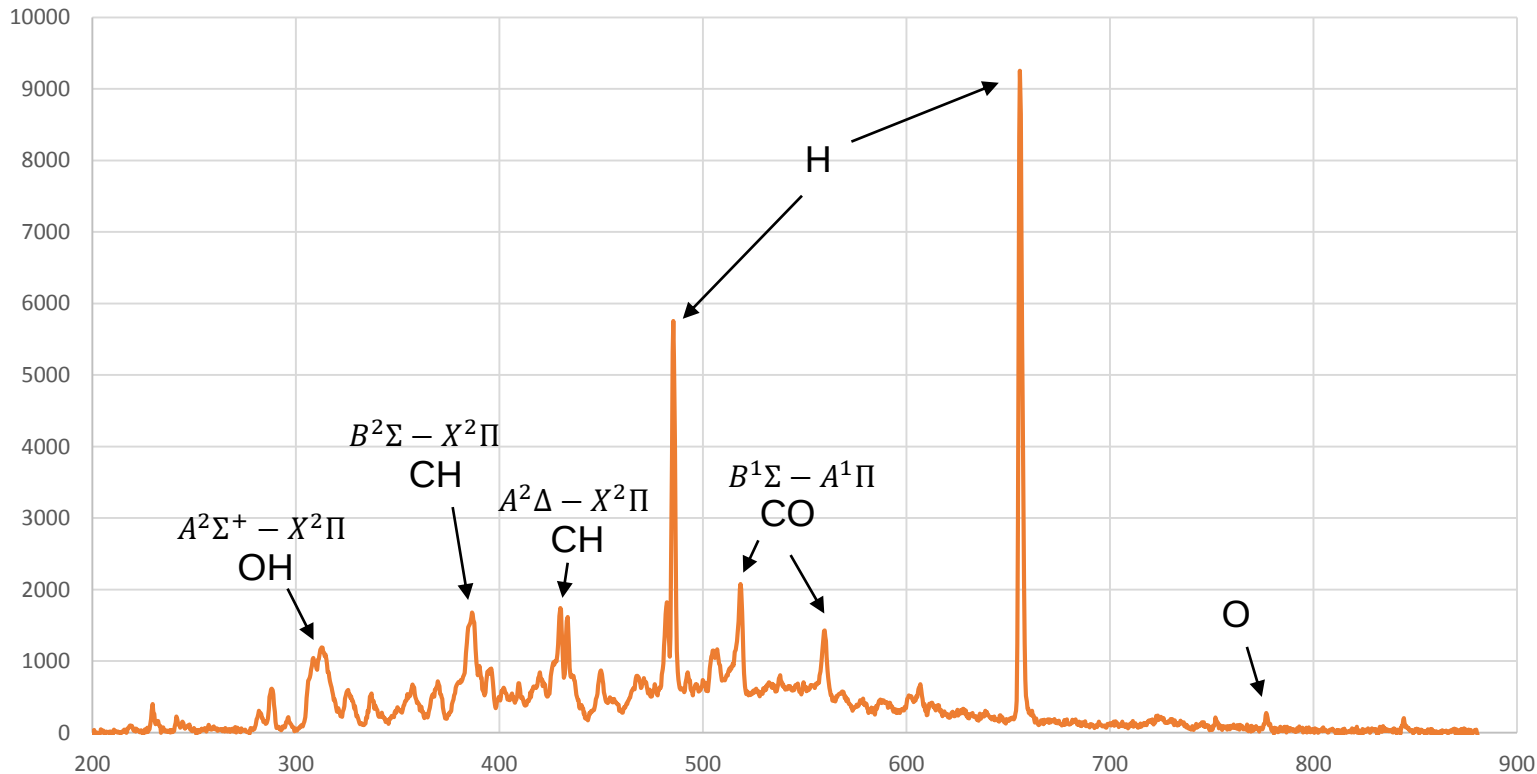


Caveats and considerations when using RPEM

- Disassociation

- Disassociation of molecules occur within the sensor's plasma
- Larger molecules are observed as fragments

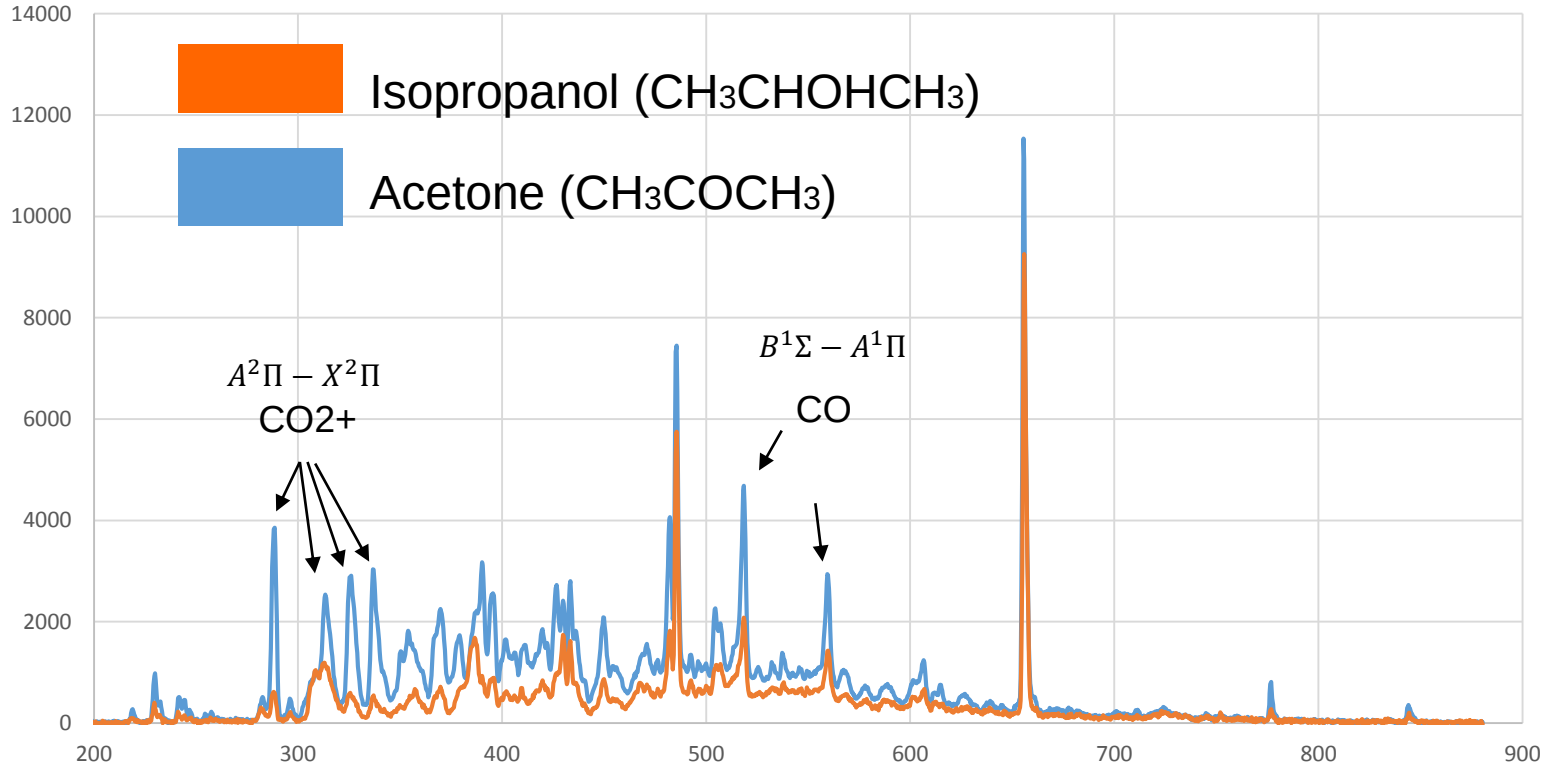
Isopropanol ($\text{CH}_3\text{CHOHCH}_3$)



Caveats and considerations when using RPEM

- Disassociation

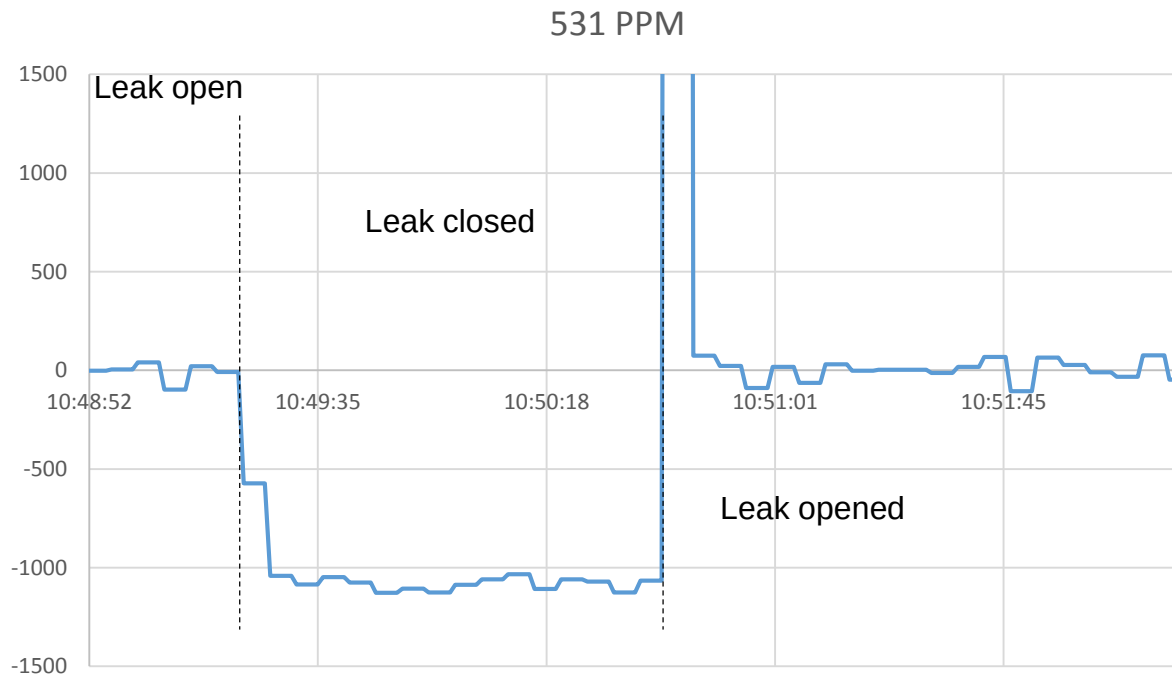
- Possibility of “Fingerprints” of the original molecule



Caveats and considerations when using RPEM

- Sensitivity

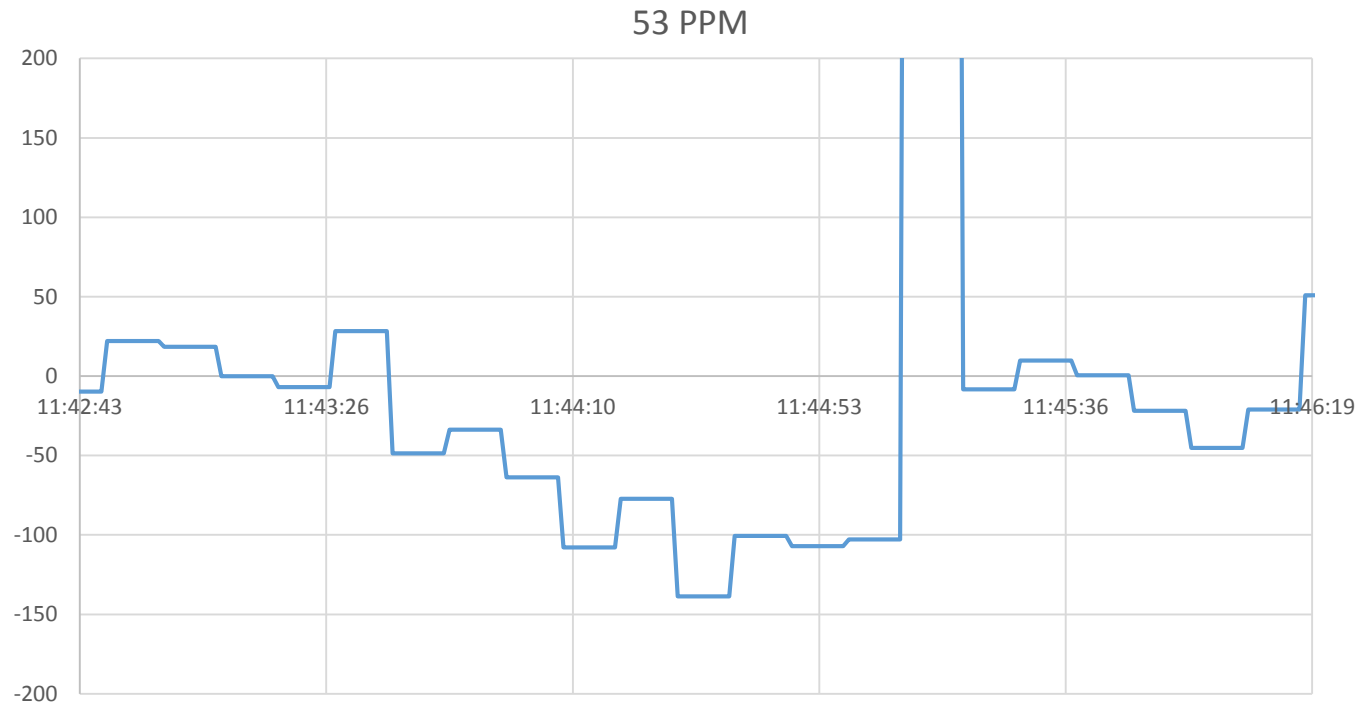
- Nitrogen (391.4 nm) was recorded and the change in signal level when the leak was opened and closed was observed.
- PPM levels were progressively reduced in order to find the PPM detection limit by increasing the argon flow



Caveats and considerations when using RPEM

- Sensitivity

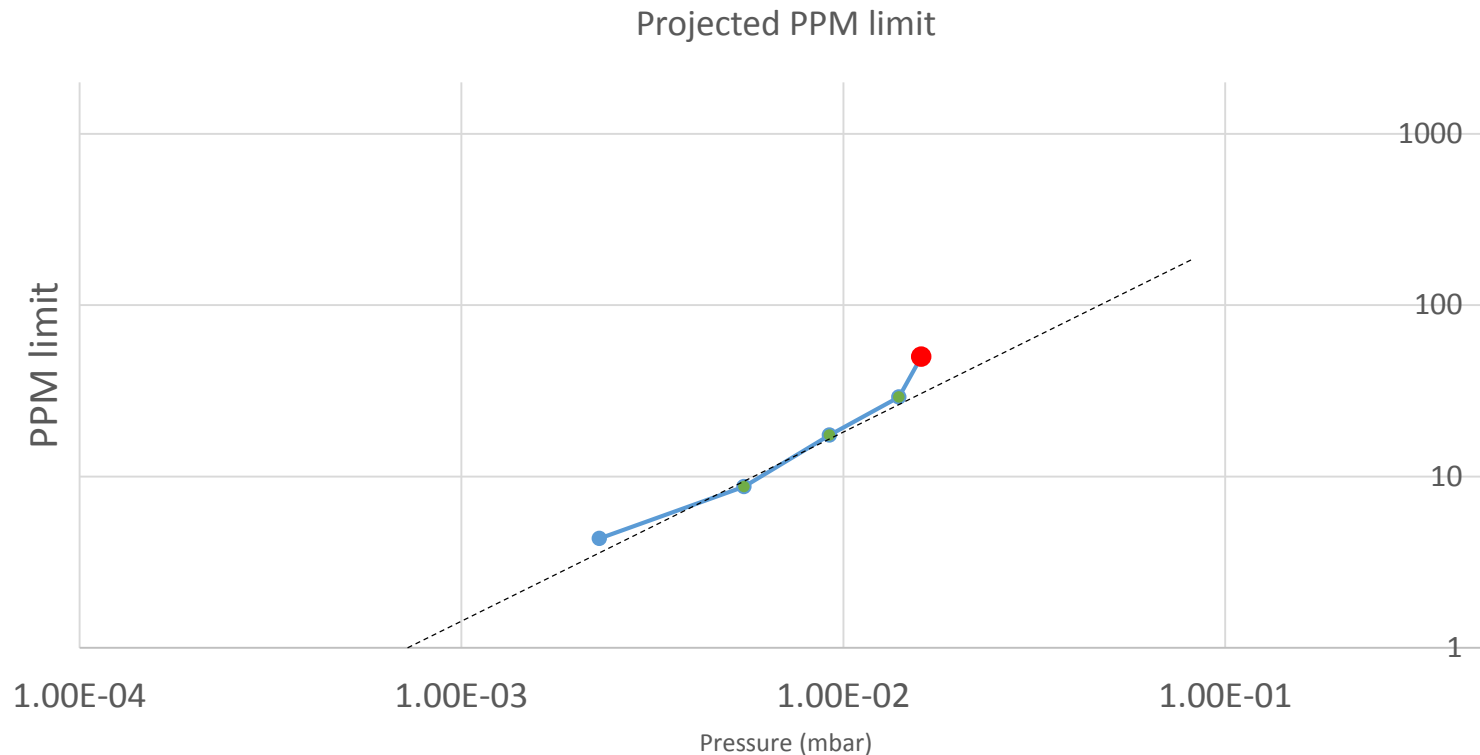
- The change in signal level below 50 PPM was greater than the noise floor average, therefore 50 PPM can be said to be the detection limit



Caveats and considerations when using RPEM

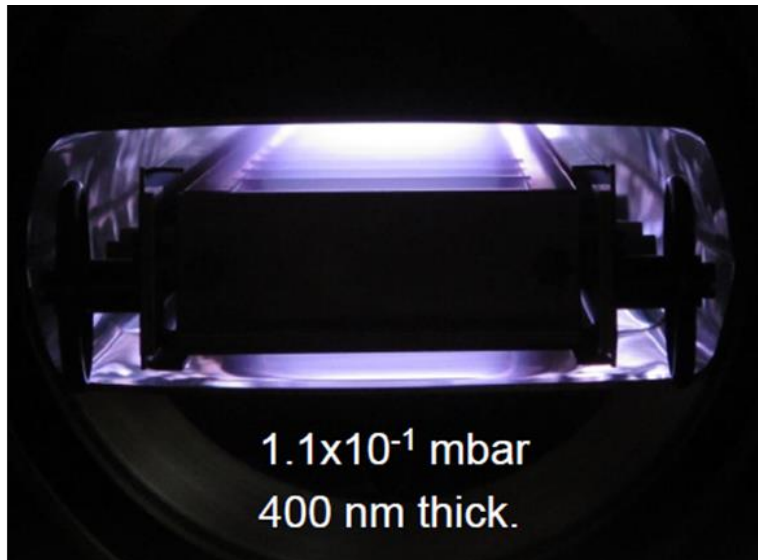
- Sensitivity

- The PPM limit at lower pressures may actually be significantly lower due to increased sensitivity at lower pressures.



Case study 2 – Outgassing measurement during carbon coating process

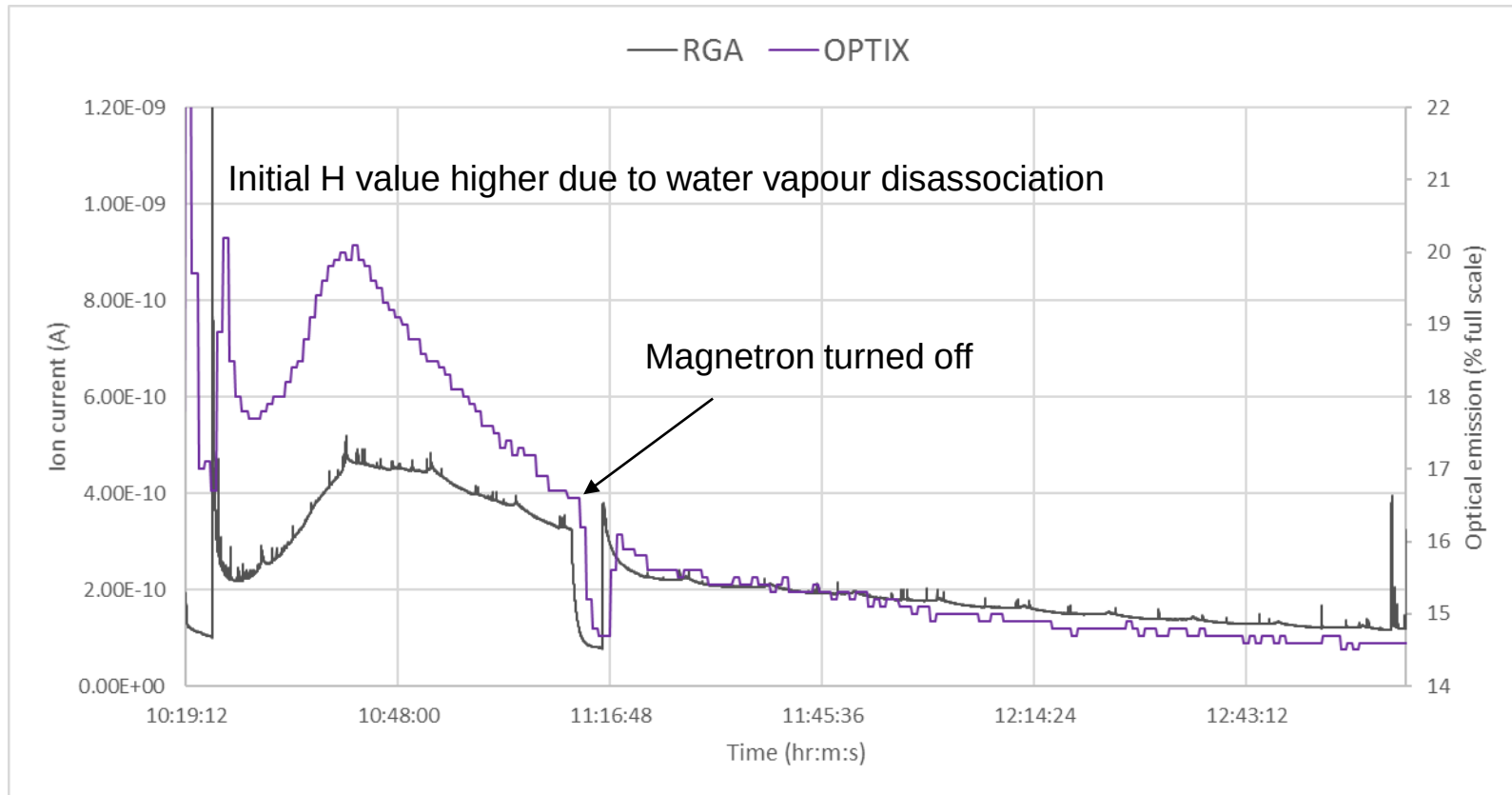
Courtesy of CERN Vacuum Surfaces and Coatings Group



- Carbon sputtered coating
- Deposited on particle accelerator inner surface to reduce secondary electron yield
- Deposition pressure of **1.1E-1 mbar**
- Performance of coating is sensitive to the presence of H outgassing from the magnetron
- Objective to monitor H outgassing during the deposition

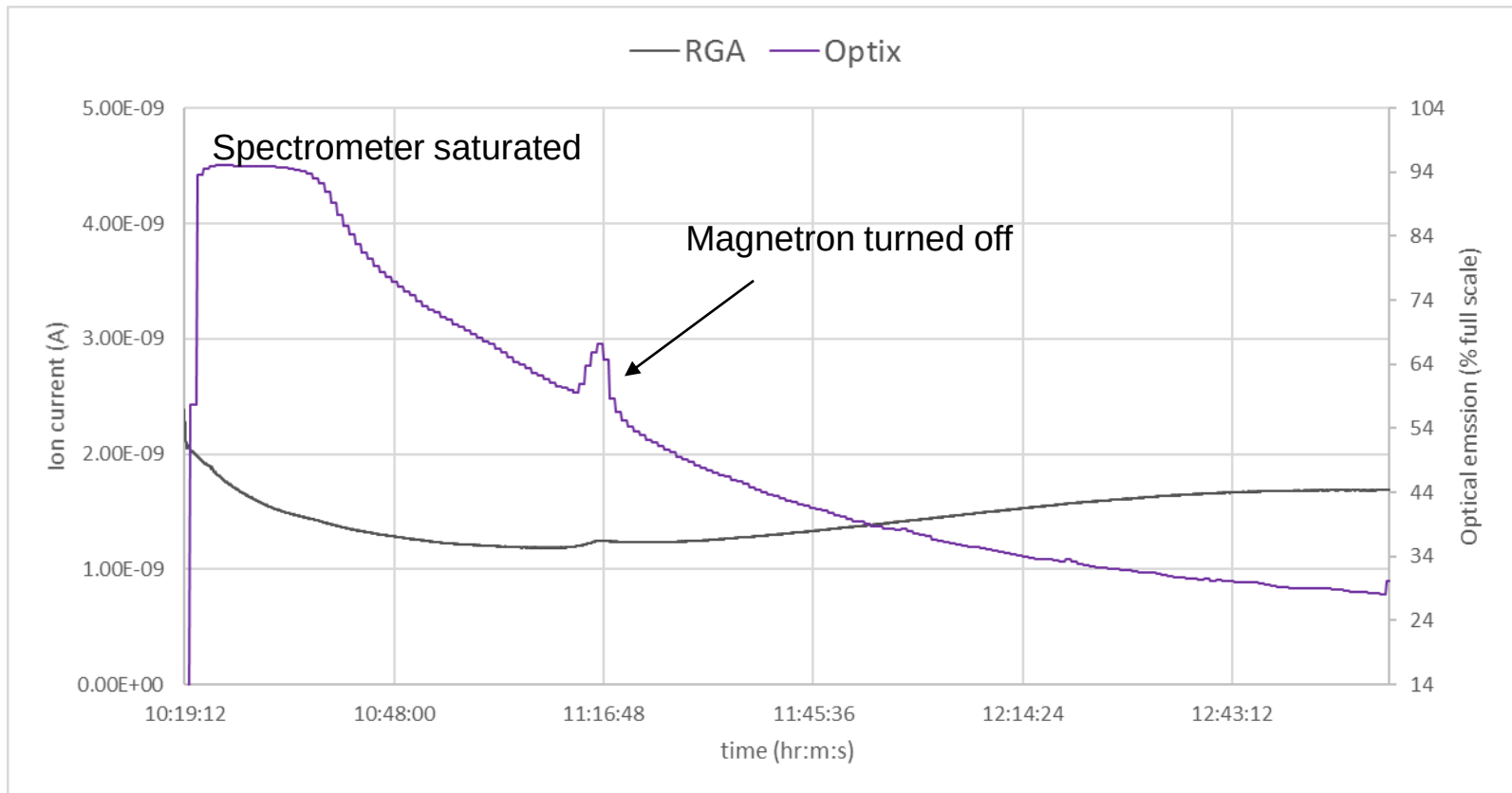
Case study 2 – Outgassing measurement during carbon coating process

Hydrogen - 656 nm, 2 AMU



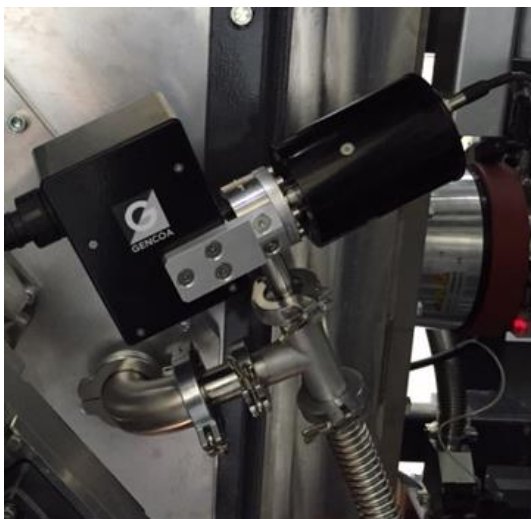
Case study 2 – Outgassing measurement during carbon coating process

Water vapour - 309.6 nm (OH), 18 AMU



Case study 3 - Characterising an AlOx magnetron sputter deposition on roll-to-roll web

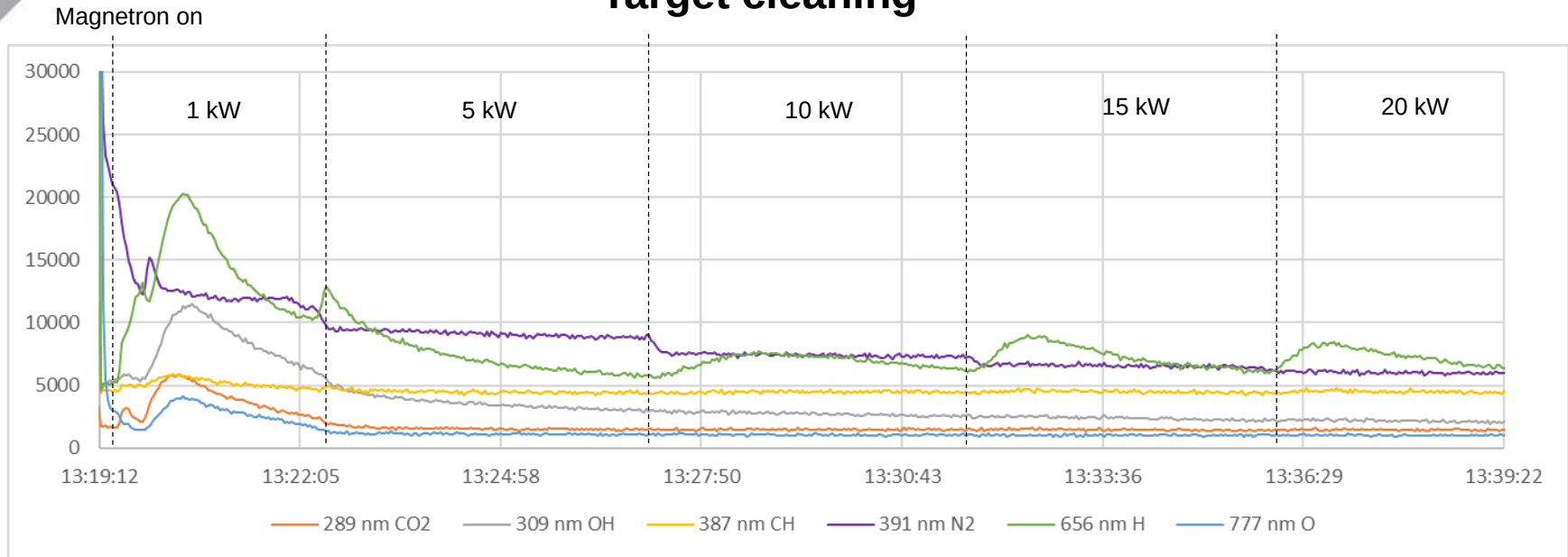
- Roll-to-roll deposition of reactively sputtered AlOx onto 125μm PET
- Optix sensor teed with a differentially pumped RGA



Courtesy of Emerson and Renwick

Case study 3 - Characterising an AlO_x magnetron sputter deposition on roll-to-roll web

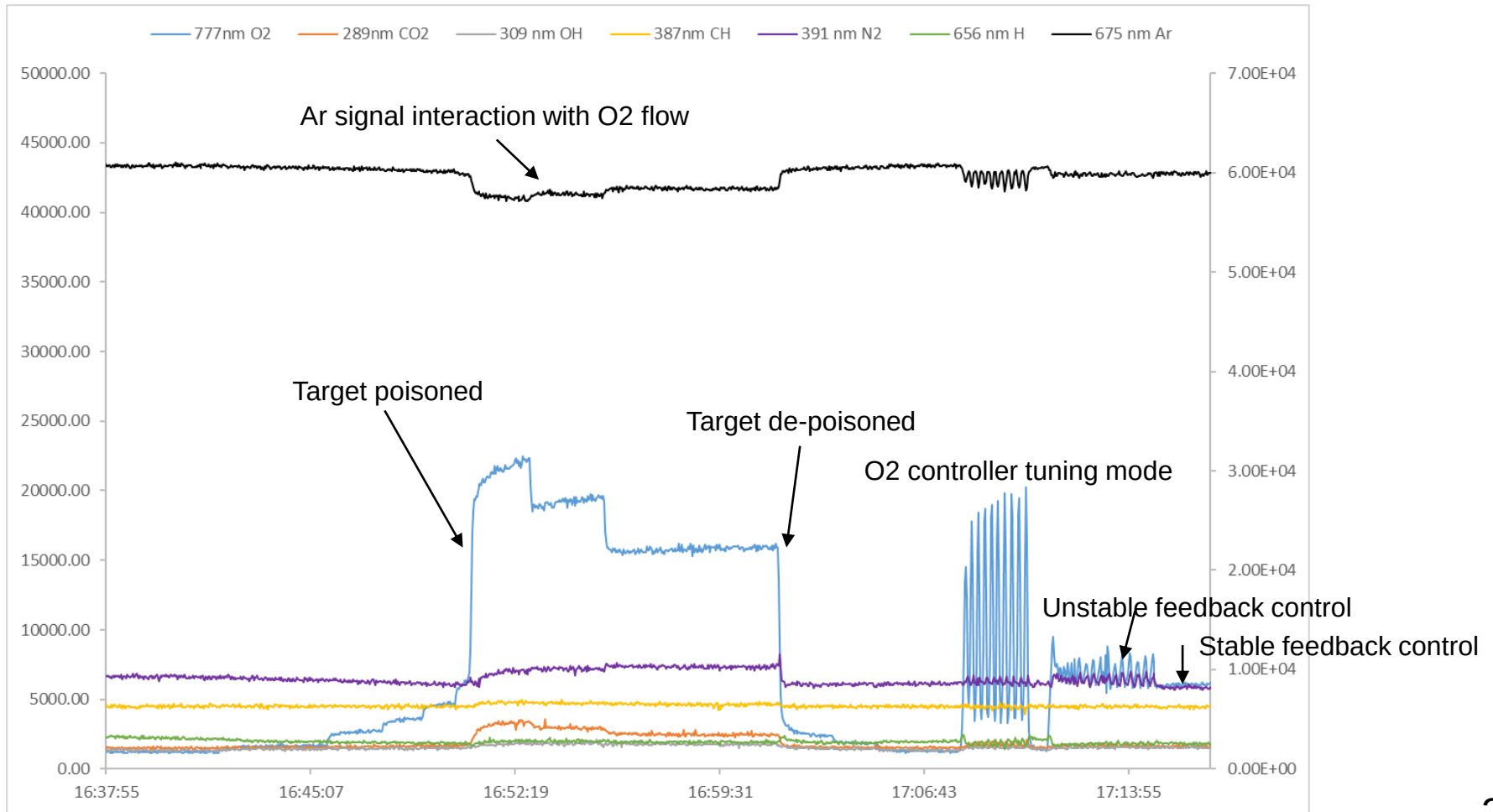
Target cleaning



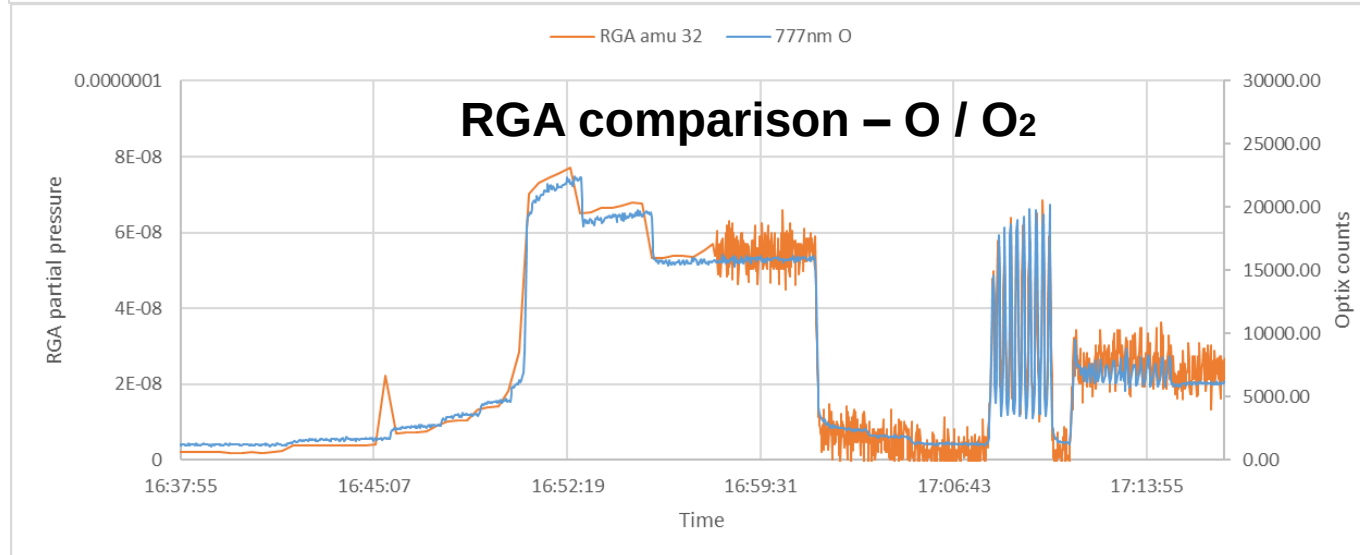
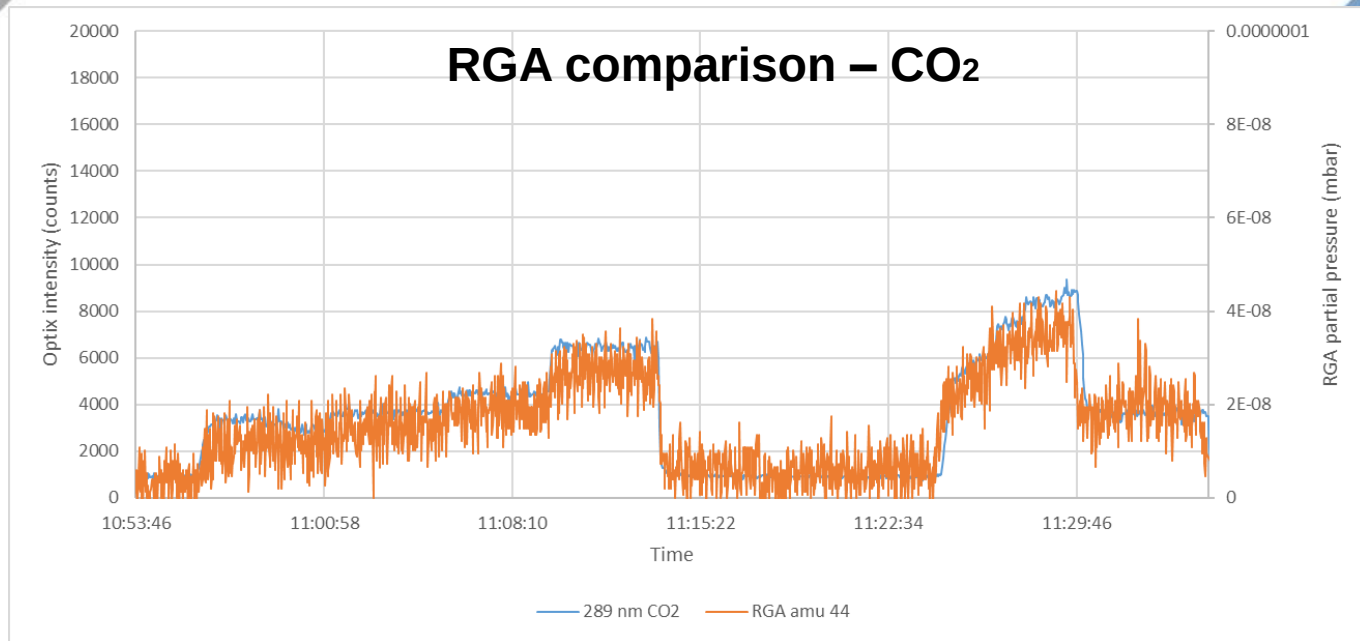
- Very large H outgassing – taking significant time to reach steady state
- Other species also observed initially outgassing – OH, CO₂, O
- Subsequent power increases cause increased H outgassing and additional settling time
- Consumption of N₂ also observed – small chamber leak

Case study 3 - Characterising an AlOx magnetron sputter deposition on roll-to-roll web

Reactive sputter characterisation

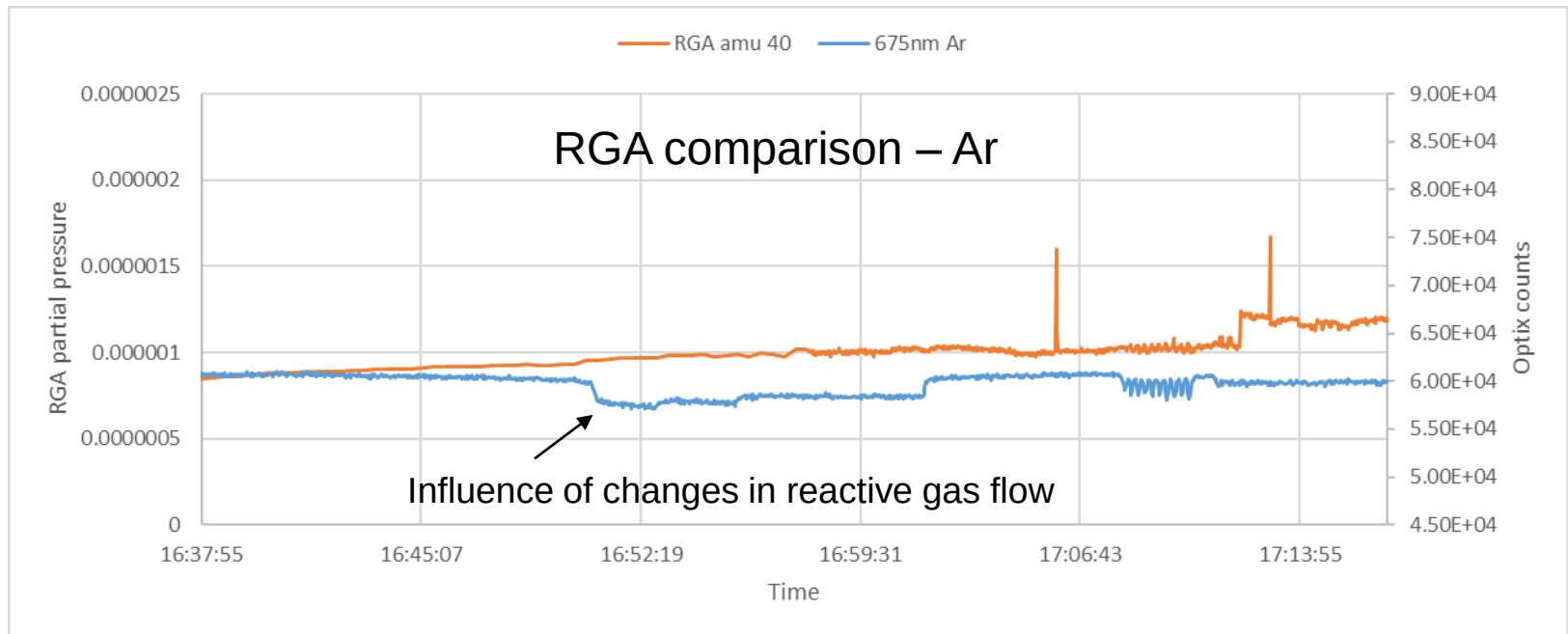


Case study 3 - Characterising an AlOx magnetron sputter deposition on roll-to-roll web

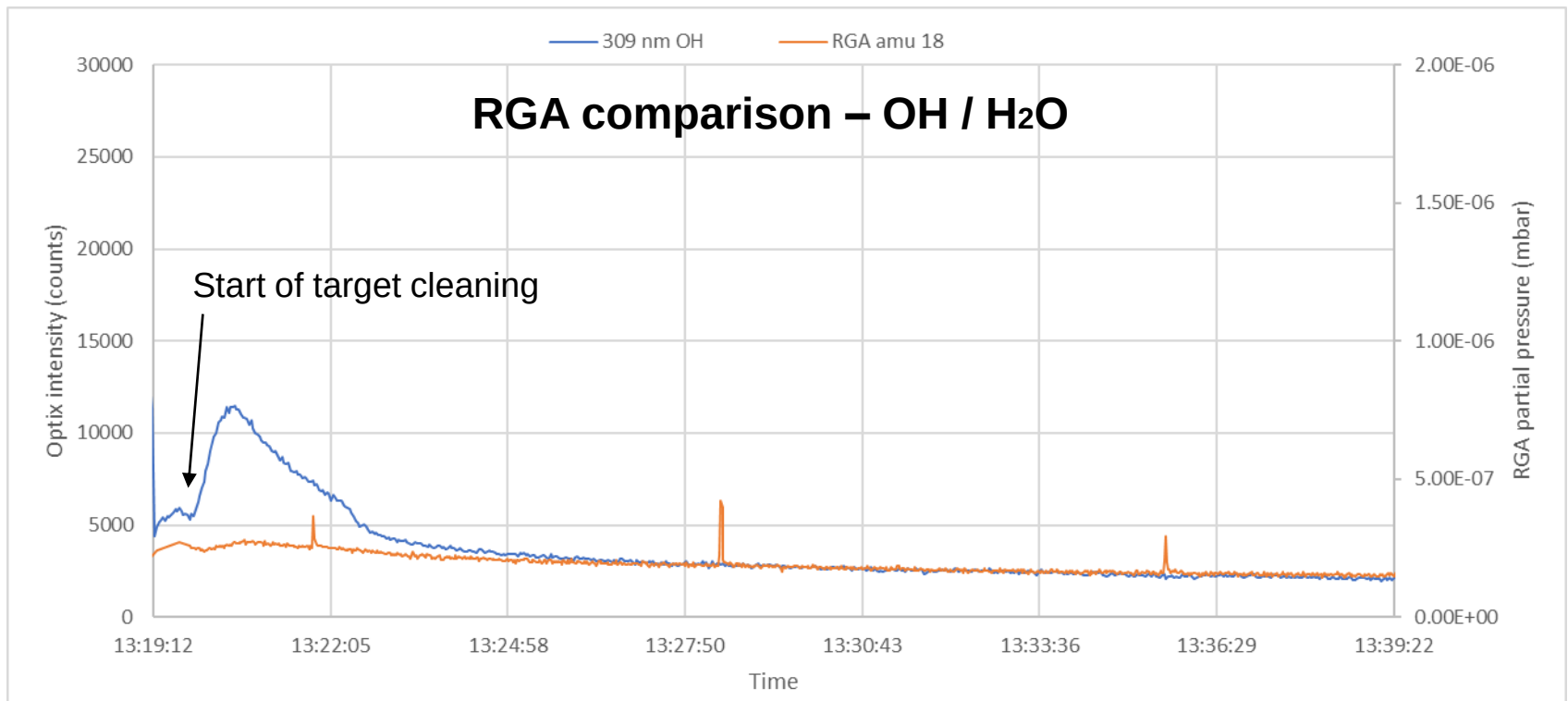


Case study 3 - Characterising an AlOx magnetron sputter deposition on roll-to-roll web

- There are discrepancies between 675 nm (Ar) and amu 40 (Ar)
- Gradually increasing RGA signal is spurious as Ar flow is constant
- Variations in the Optix Ar signal are due to interaction with the O₂ process gas

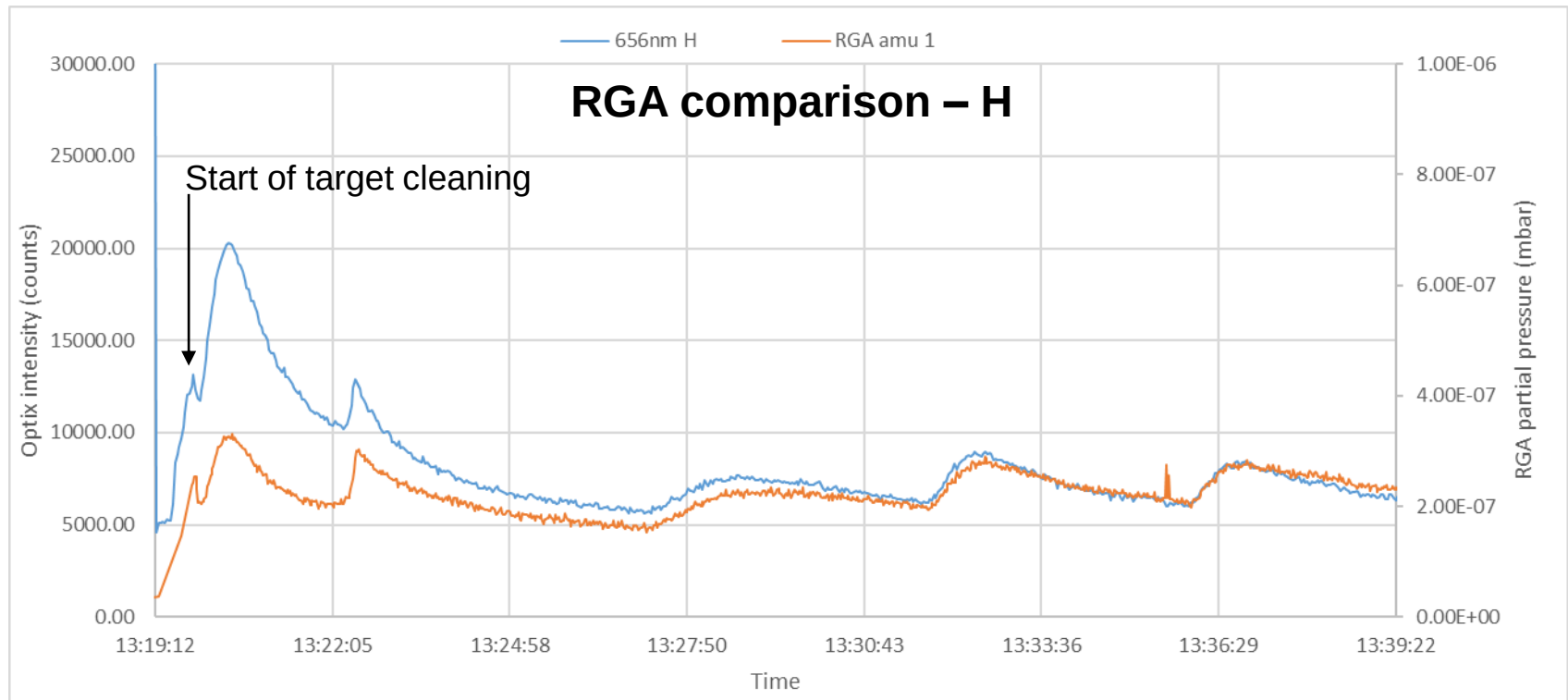


Case study 3 - Characterising an AlOx magnetron sputter deposition on roll-to-roll web



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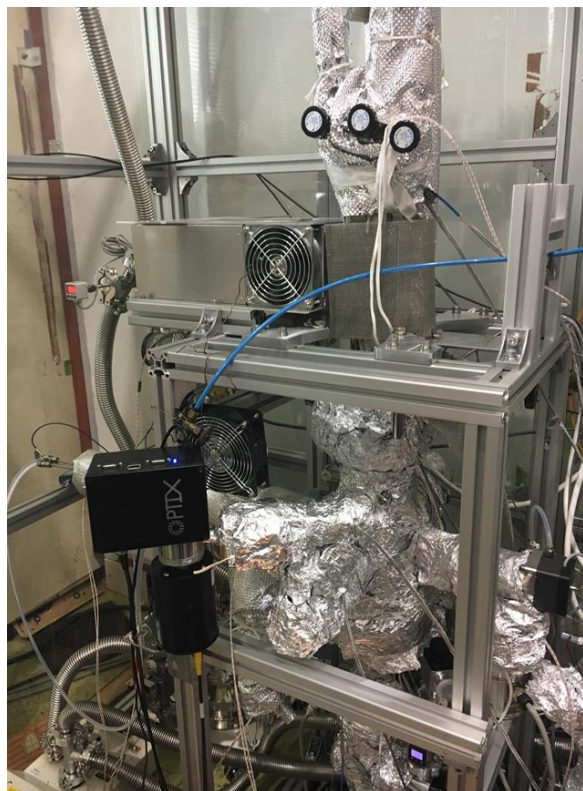
- 656 nm (H) and amu 1 (H) are generally a good match.
- The difference at the start of the trace can be attributed to water vapour disassociating inside the Optix sensor into H, increasing the H reading.



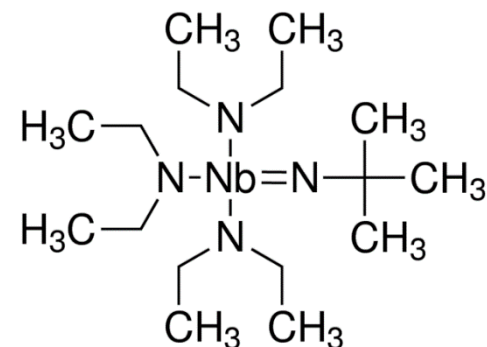
Deposition of NbN via PEALD

High Energy Accelerator Research Organization (KEK), Tsukuba, Japan

Image and data courtesy of S. Kato



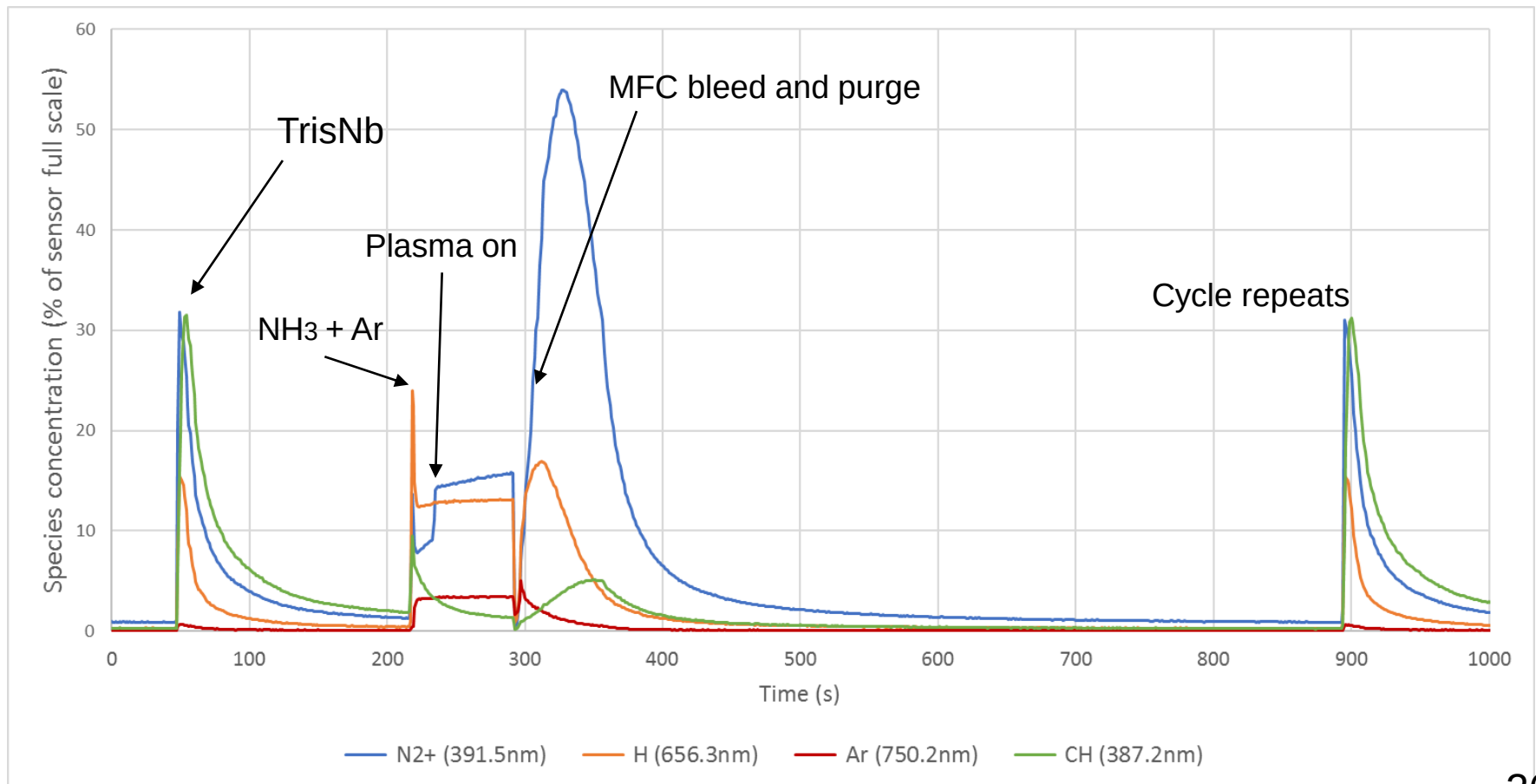
- Detection of TrisNb via CH, N and H



- Detection of NH₃ via N and H

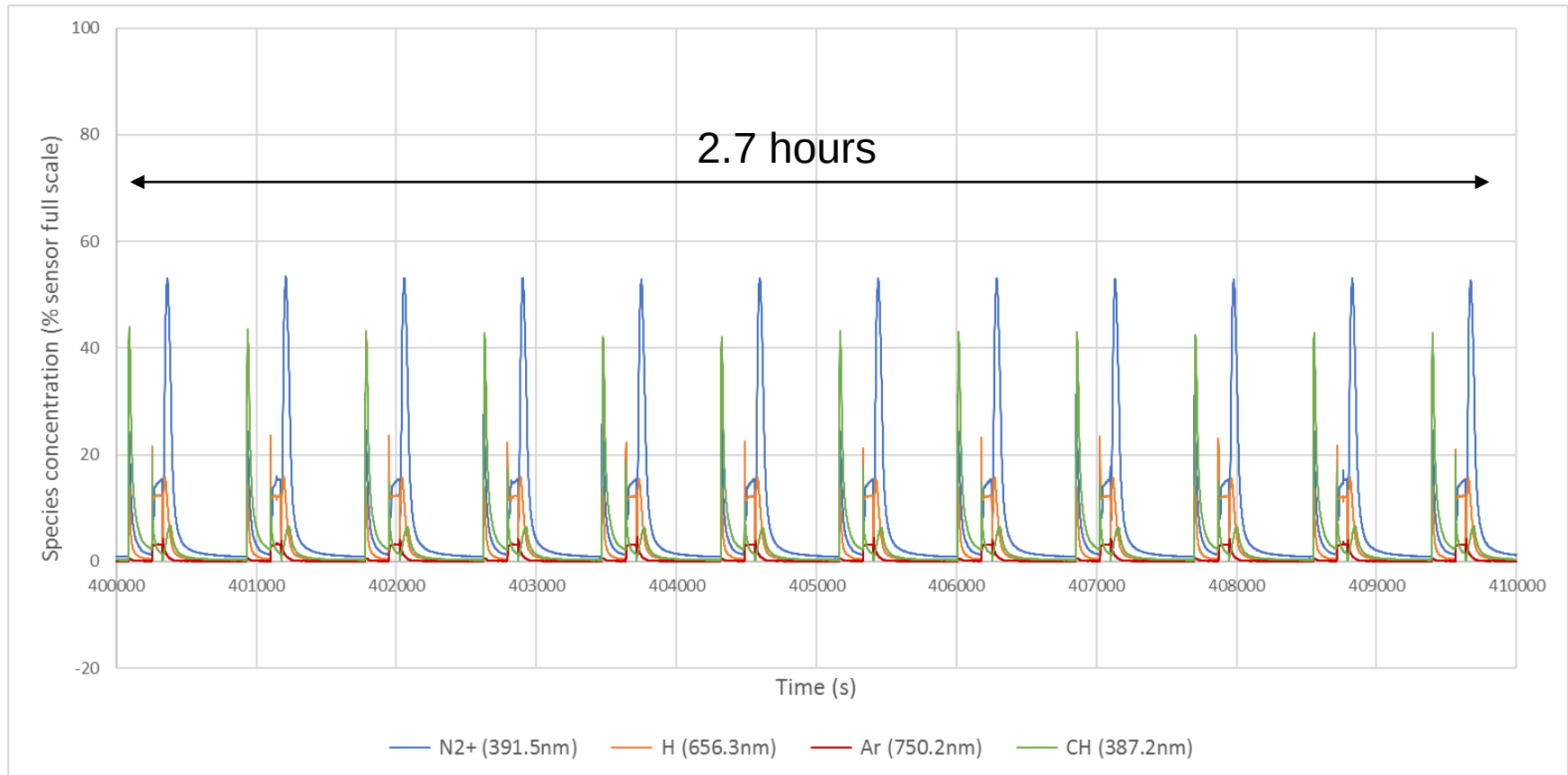
ALD Monitoring – Full process

NbN deposition step



ALD Monitoring – Full process

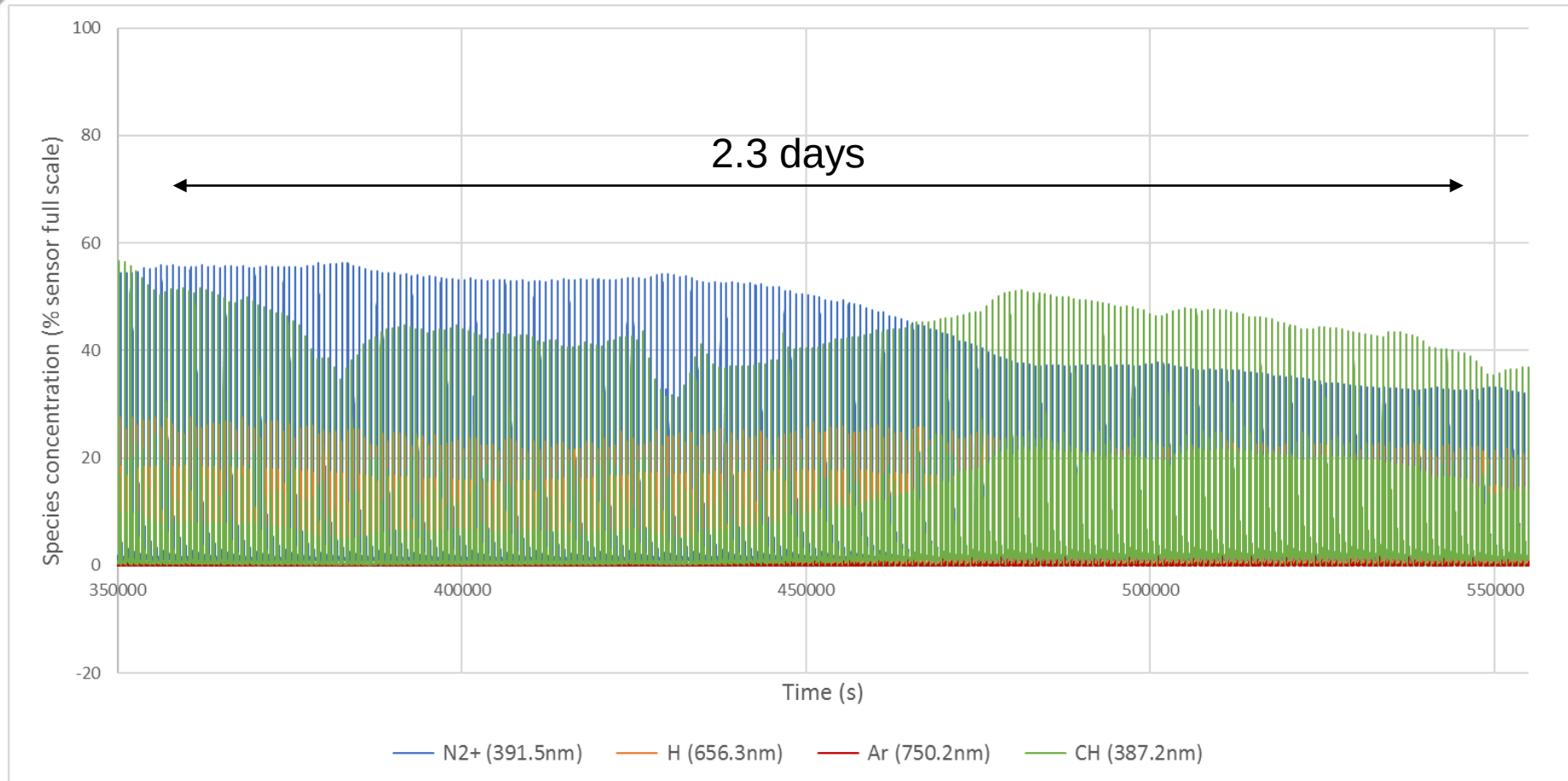
Deposition of NbN via PEALD



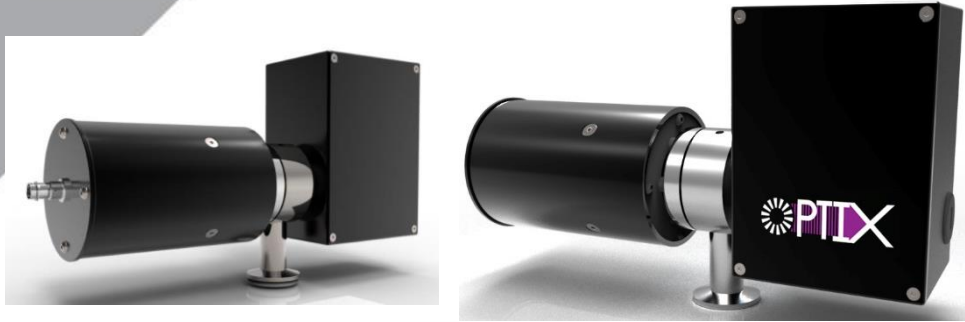
ALD Monitoring – Full process

Deposition of NbN via PEALD

- Sensor is robust of the full 2+ day deposition cycle



- **Remote plasma emission monitoring can be used to provide “RGA-like” capability directly at higher process pressures – results are qualitatively comparable**
- **Essential information on process contamination and performance can be extracted – limitations of the sensing technique have to be considered**
- **Potentially enhanced sensitivity to water vapor over a differentially pumped RGA**
- **Robustness demonstrated with contaminating processes**



Thank you for your attention!

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