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Monitoring of volatile vacuum species using remote optical emission spectroscopy

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May 2018

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Outline of the talk

- Explanation of the Remote Plasma Emission (RPEM) method
- Gas detection and quantification by RPEM
- Examples of data from ALD, Etching and solvent analysis
- Conclusions

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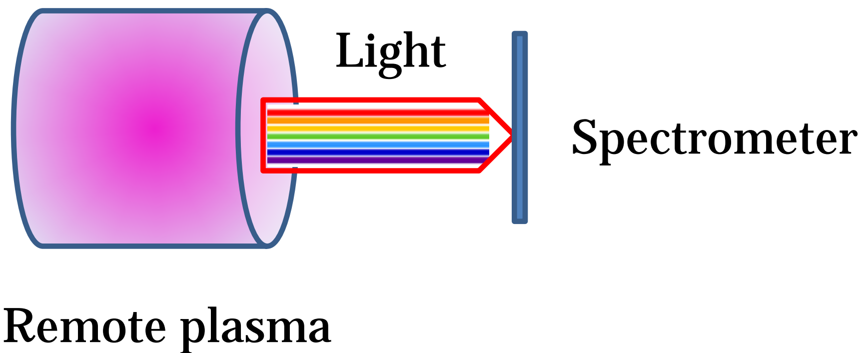
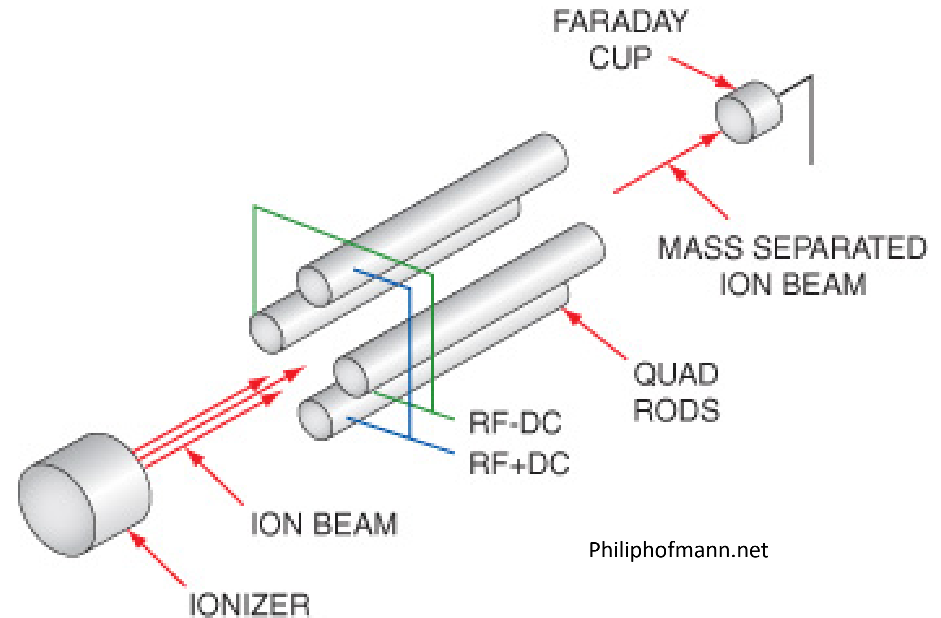
RPGA vs RGA

OPTIX – remote plasma gas analysis (RPGA)
Optical method



Low ppm detection

Quadrupole Residual Gas Analyzers (RGAs)



Low ppm detection

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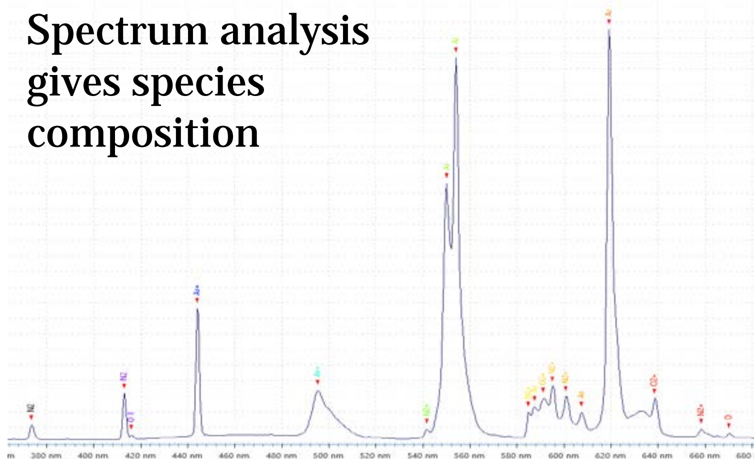
OPTIX Remote Plasma Gas Analysis RPGA

Vacuum process 0.5 to 10^{-6} mbar or with a rotary pump to support atmospheric sensing



Spectrum analysis gives species composition

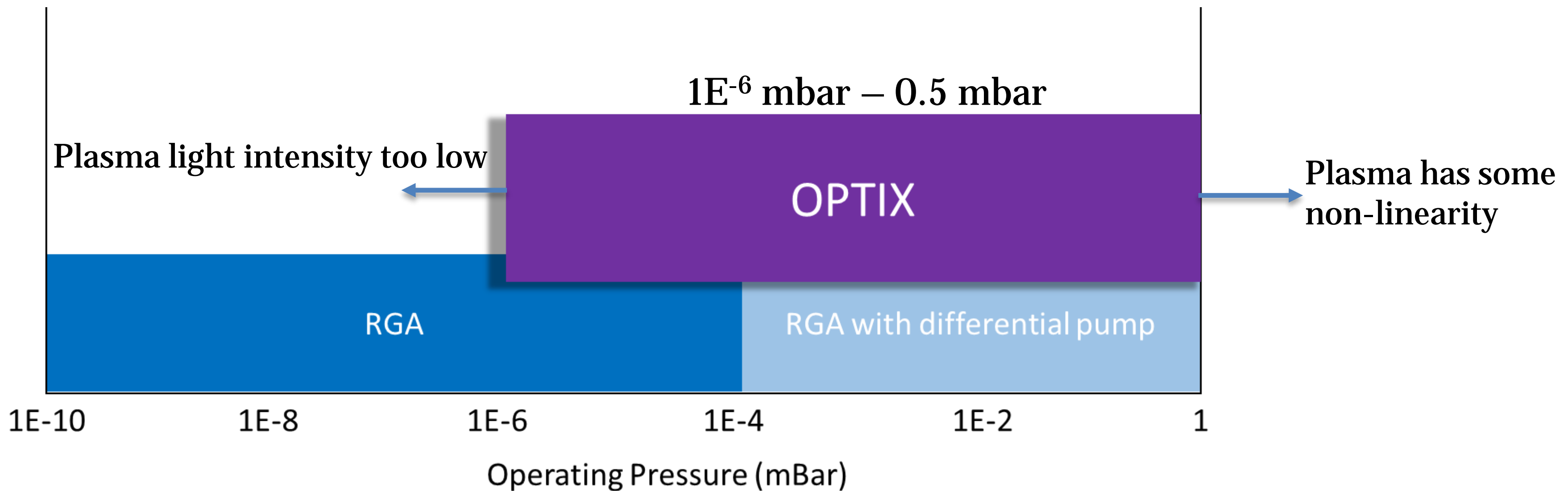
Wide pressure range remote plasma generator

High intensity plasma

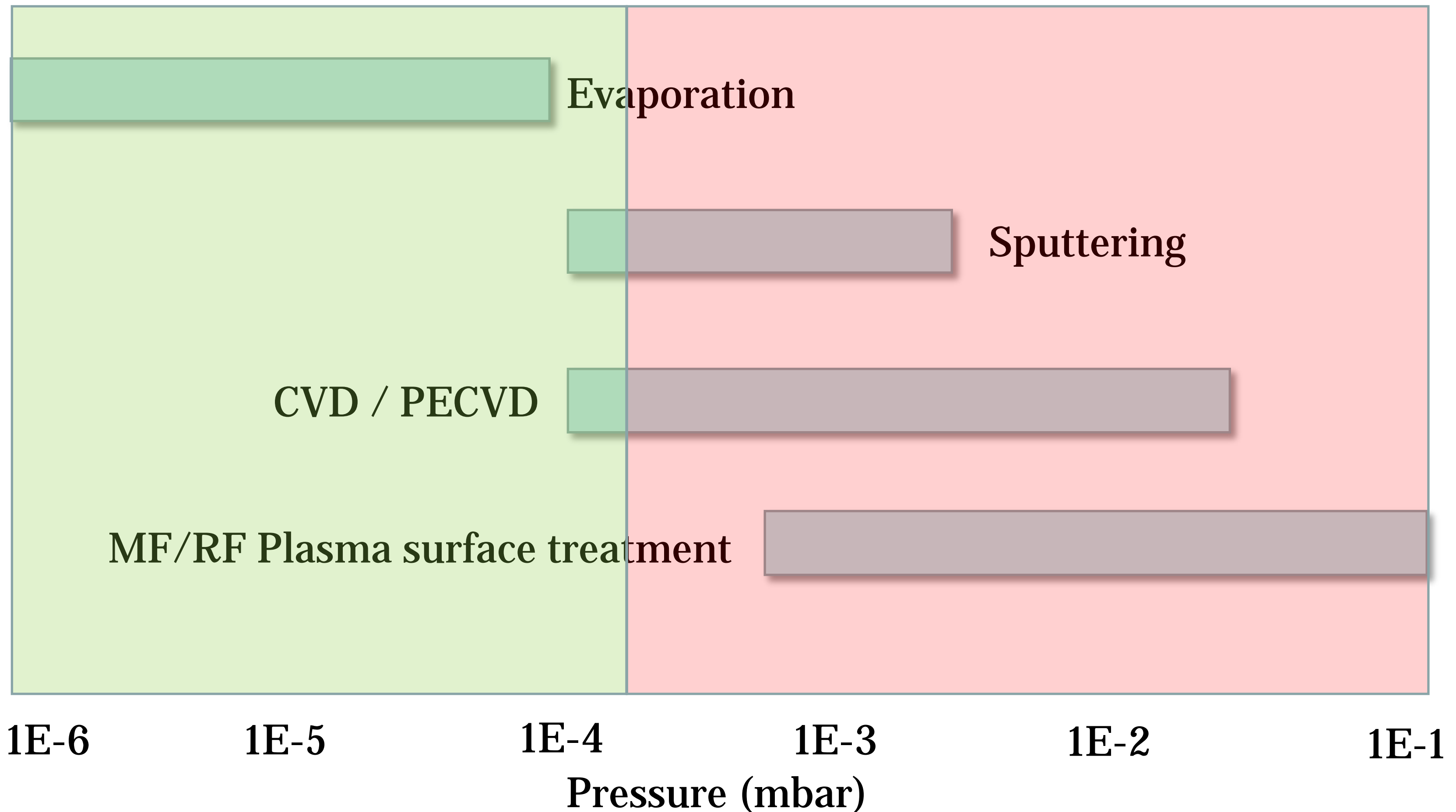
Wide range spectrometer 200-850nm

OPTIX operates in the typical plasma processing pressure range

Easy to use and wide operating range



OPTIX vacuum based applications



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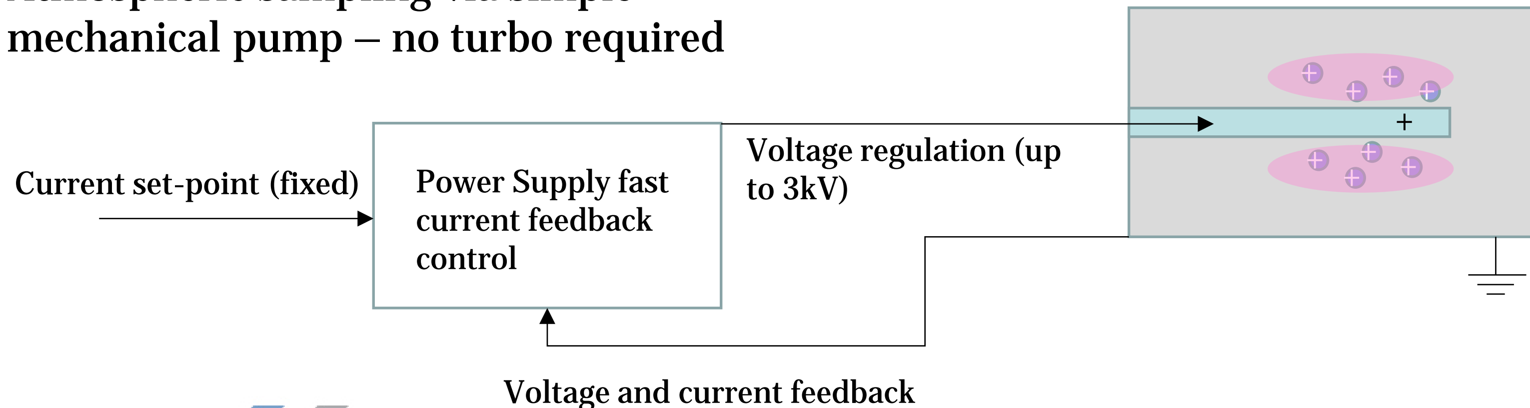
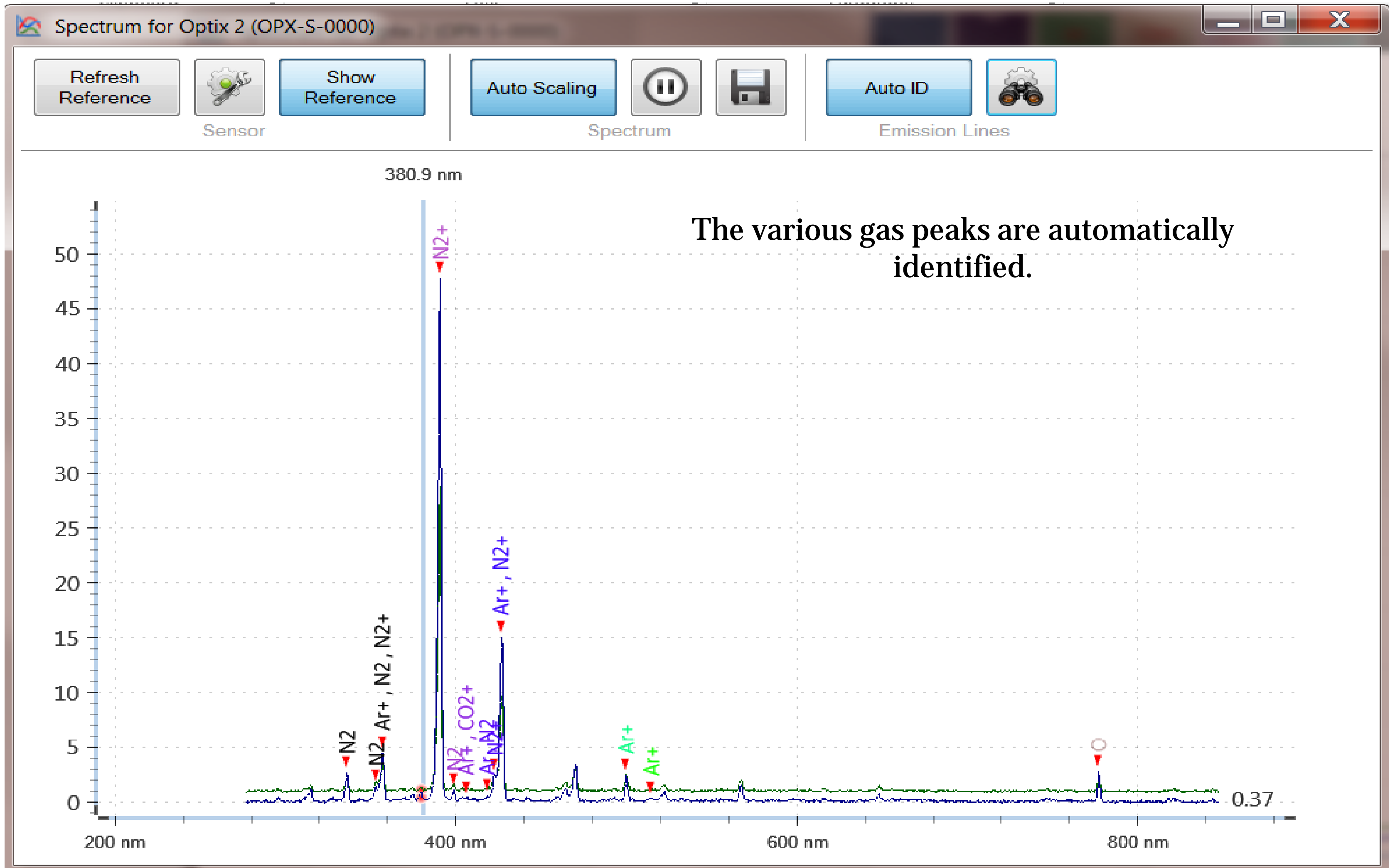
OPTIX Plasma Generation

Unlike RGA's OPTIX detector is separated from the chemicals by an optical window – more rugged – detector cannot contaminate

- Purpose designed and patented plasma generation source
- Very wide range of operation - Plasma present from 0.5 to 10^{-6} mbar
- Fast current feedback control
- Constant current = constant excitation source
- DC mode as standard for 95% of applications, Pulsed DC for highly contaminating atmospheres
- Atmospheric sampling via simple mechanical pump – no turbo required



Software Spectrum View - spectrum displays the constituent species of the plasma



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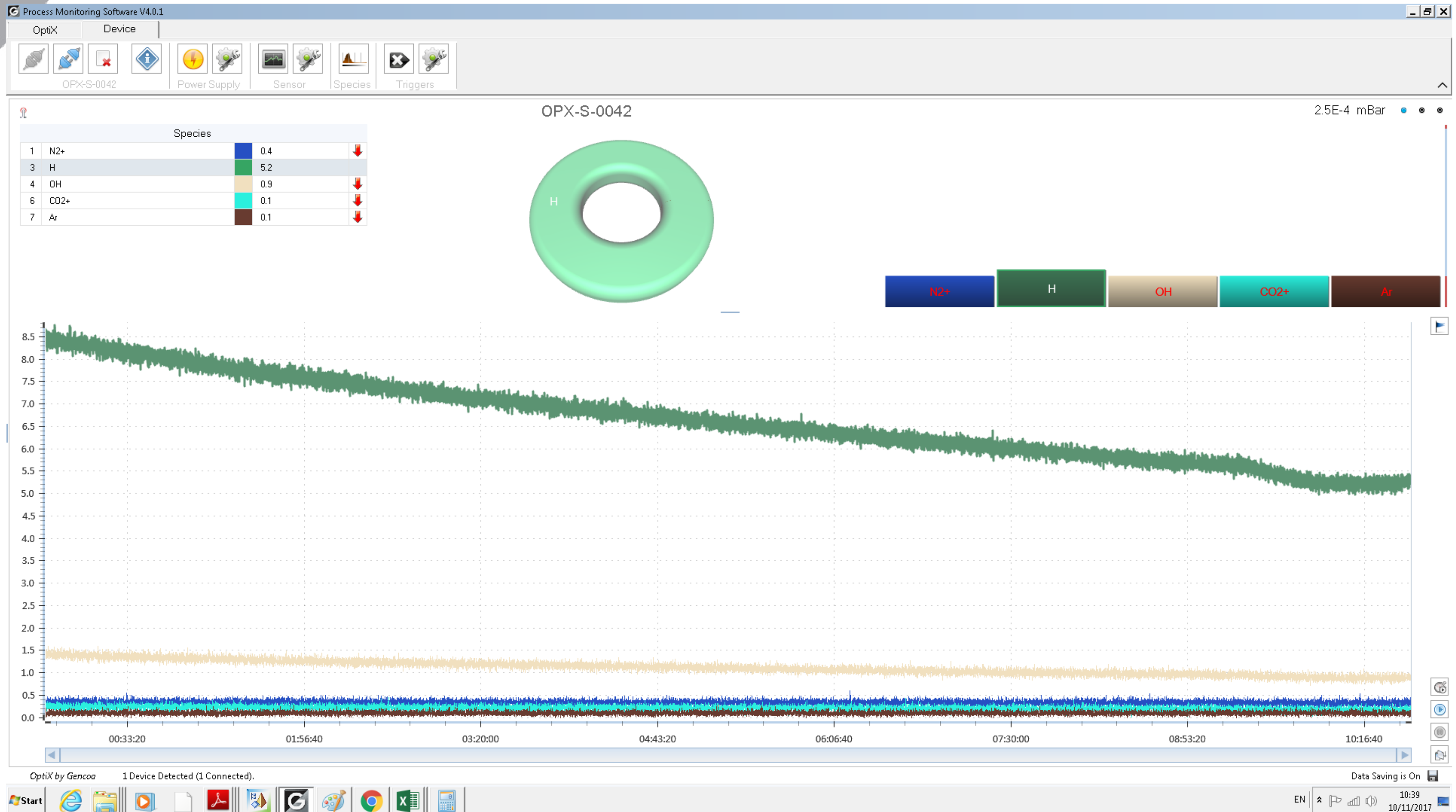
Software Gas Tracking View



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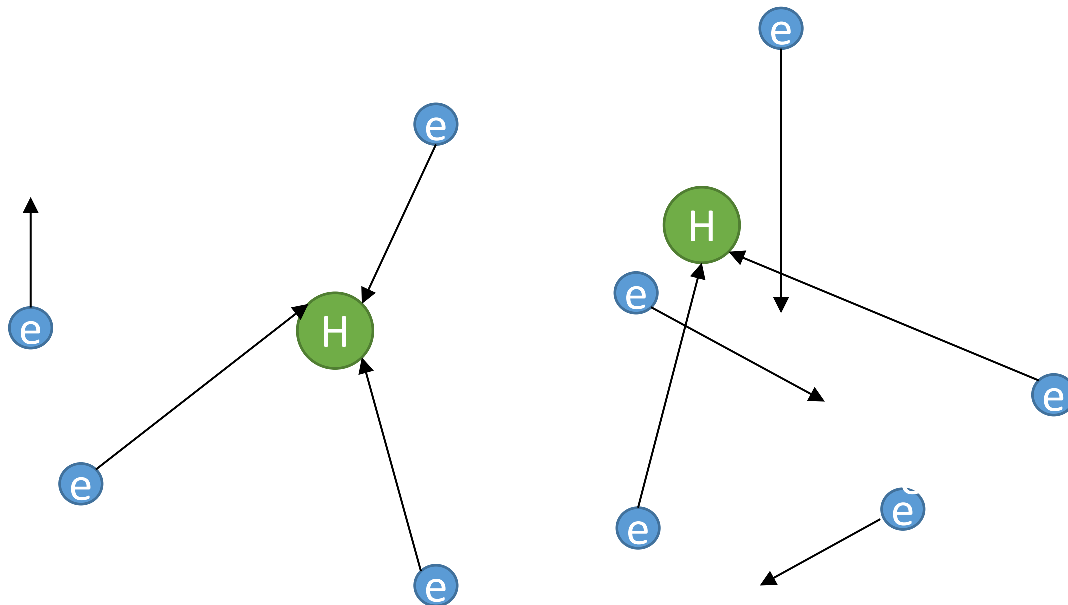
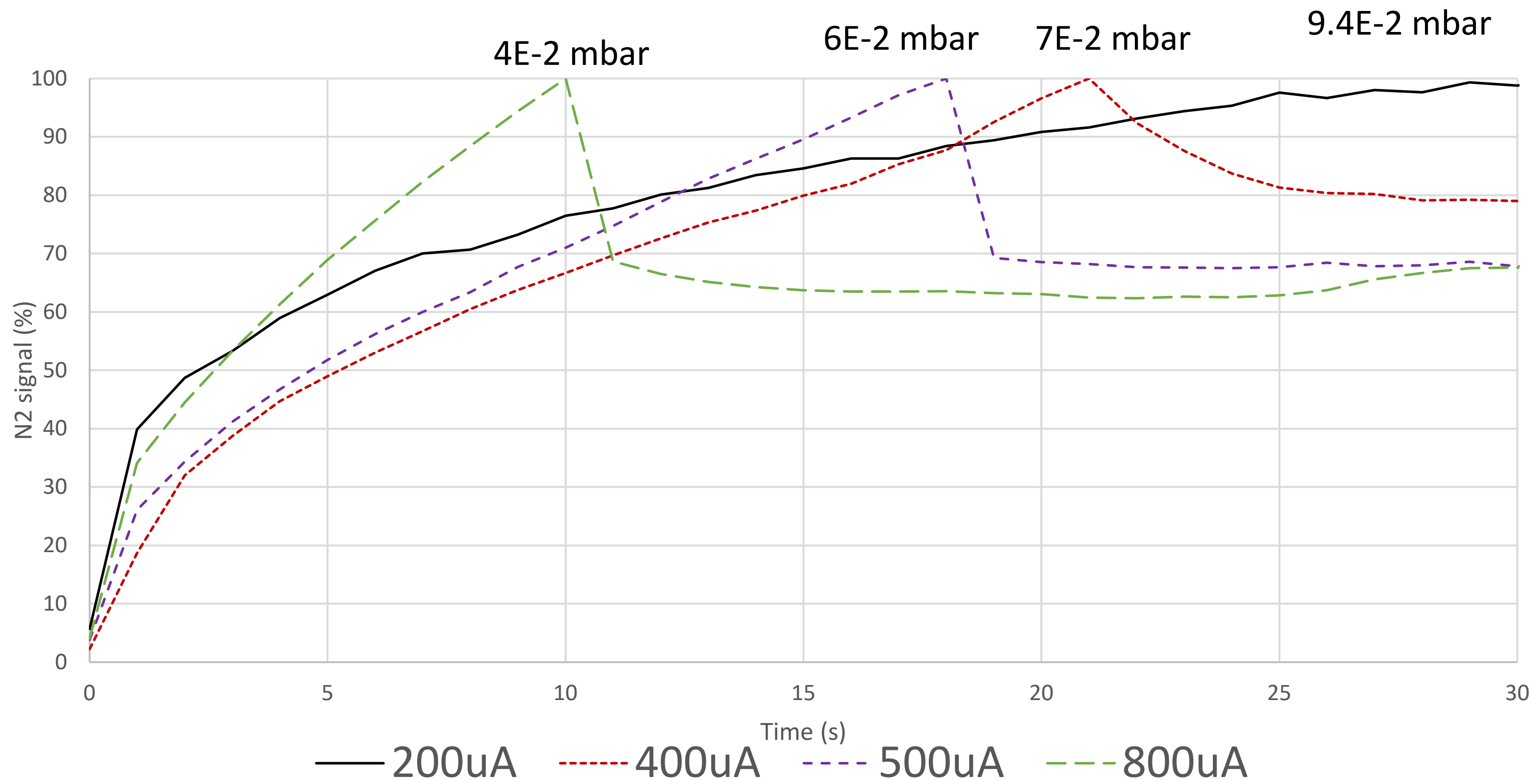
Quantification of gas levels using RPGA

- The sensor results as displayed in the raw spectrum are **qualitative** due to the interaction of different gases within the vacuum
- Even quantities of a gas are equally likely to be collide with free electrons
- Gencoa have developed a mathematical treatment to accurately calculate gas partial pressure



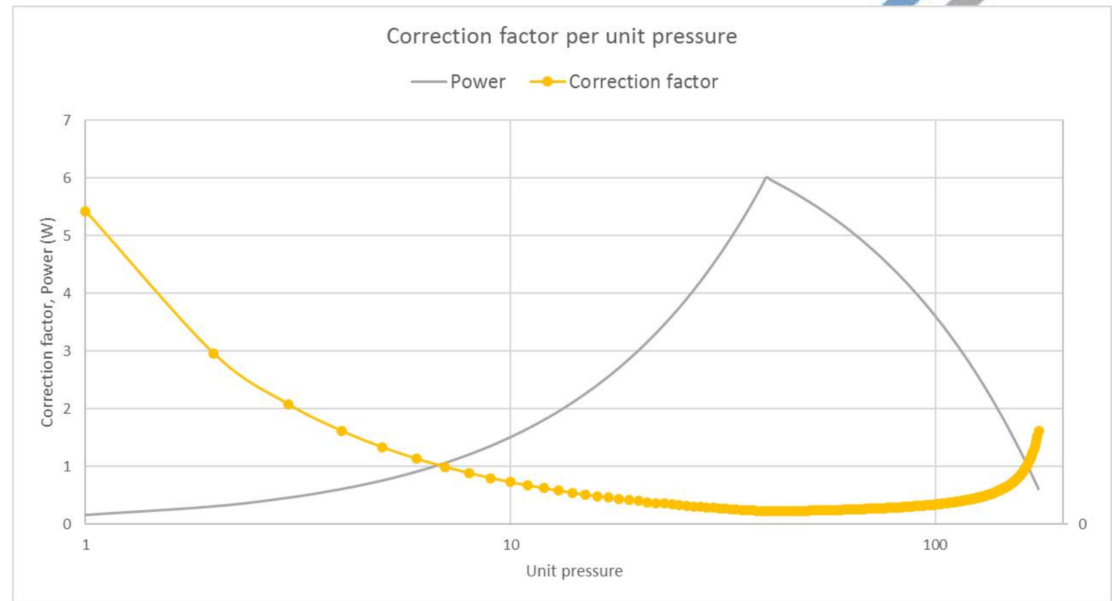
Quantification - Pressure limitations

- Higher currents give a superior signal to noise ratio but at the expense of upper operating pressure limit.
- Maximum linear operating range can be achieved with a lower current setpoint – OPTIX can select the current via the user interface.

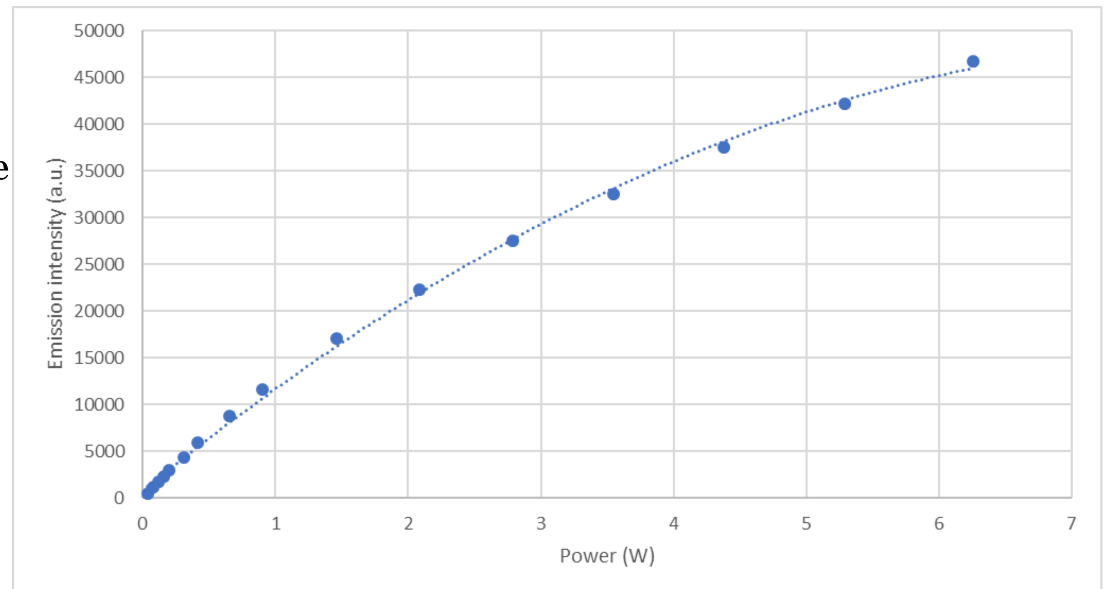


Quantification of gas partial pressures – Plasma Power correction

- The power delivered to the plasma generator will modify the emission intensities and hence distort the gas partial pressure measurement

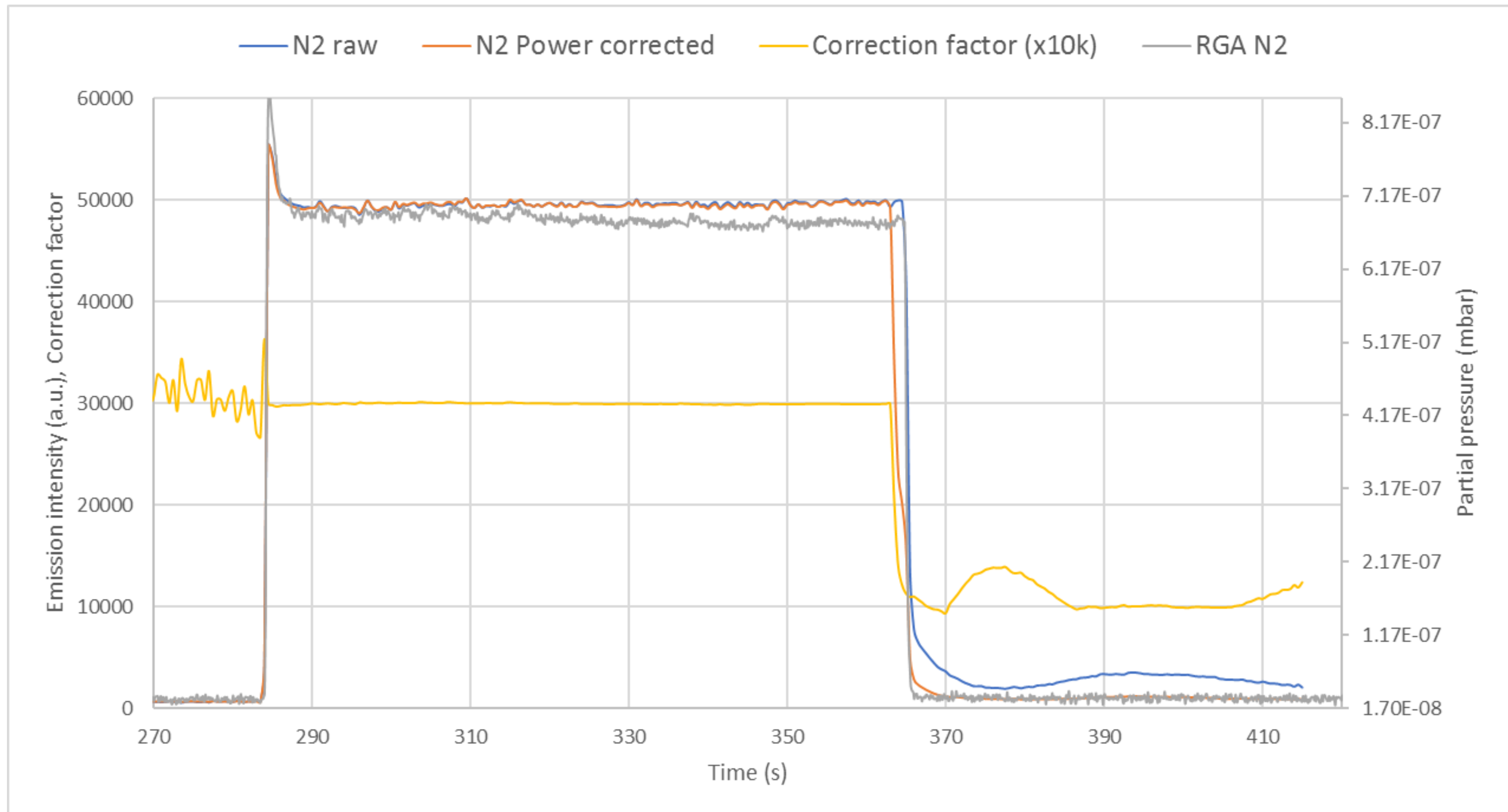


- A correction factor based on the measured power can be applied to the emission to remove this effect



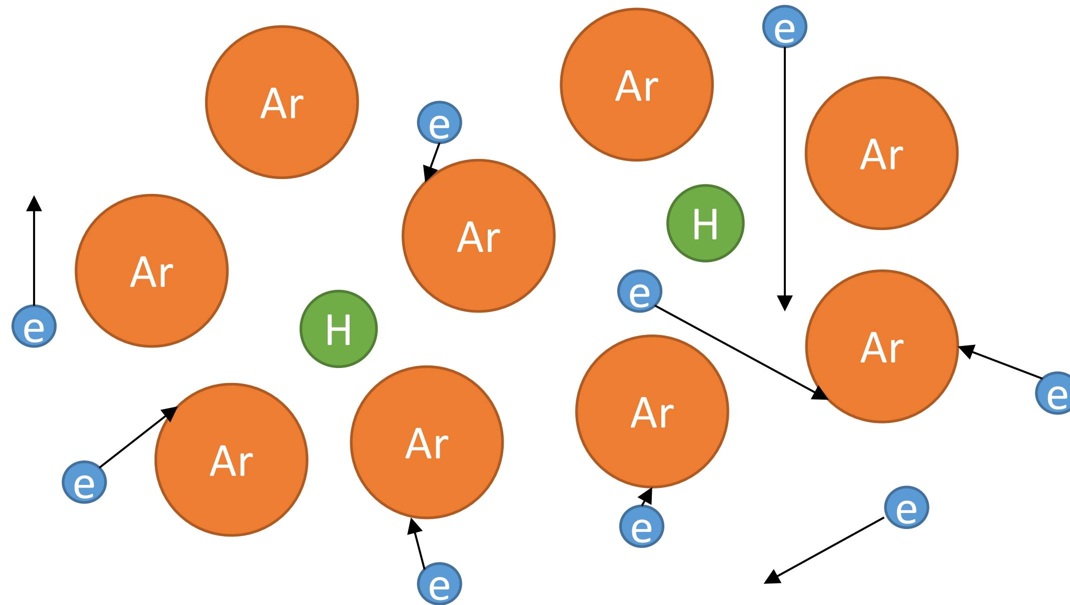
Quantification - Power correction

- The effect of the correction can be clearly seen when compared with a differentially pumped RGA



Quantification of gas levels using RPGA

- Introduction of a >> larger quantity of an additional gas will reduce the likelihood of electron impact on species of a << smaller quantity
- This will have an effect of supressing the emission of these species
- The OPTIX has a correction algorithm for the gas interaction effect to allow accurate quantification of the gas partial pressures



Quantification – Gas interaction

Experimental setup

- The most significant challenge for quantification of the sensor readings is the interactivity of gases
- Without correction the readings are **relative** not absolute
- i.e. increasing partial pressure of one gas will lead to a reduction in the readings of other gases.
- An experimental setup was constructed to investigate this effect and to demonstrate the correction method

Gas input – Ar, N_2, O_2

OPTIX

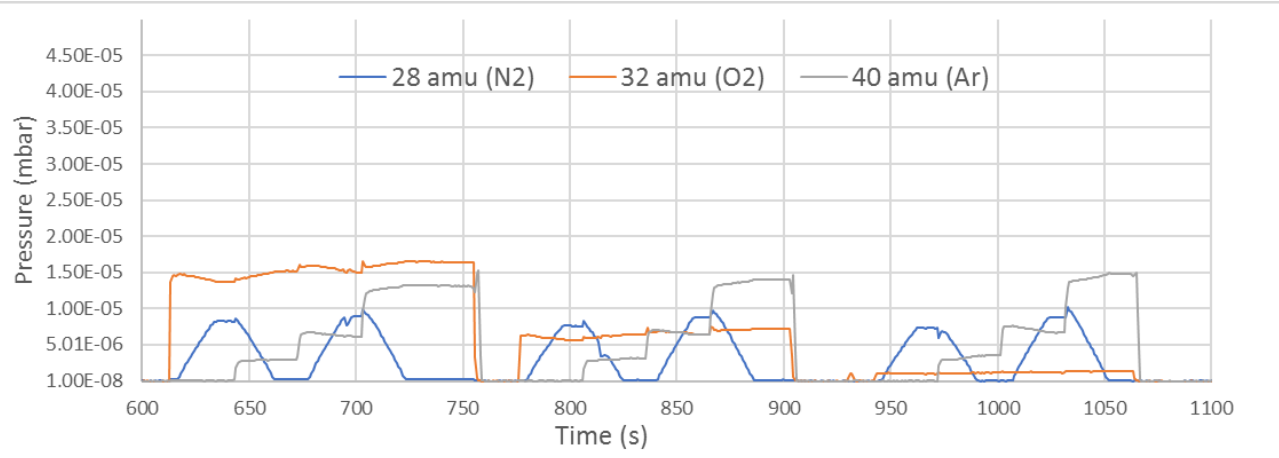
Diff. pumped side

High pressure side

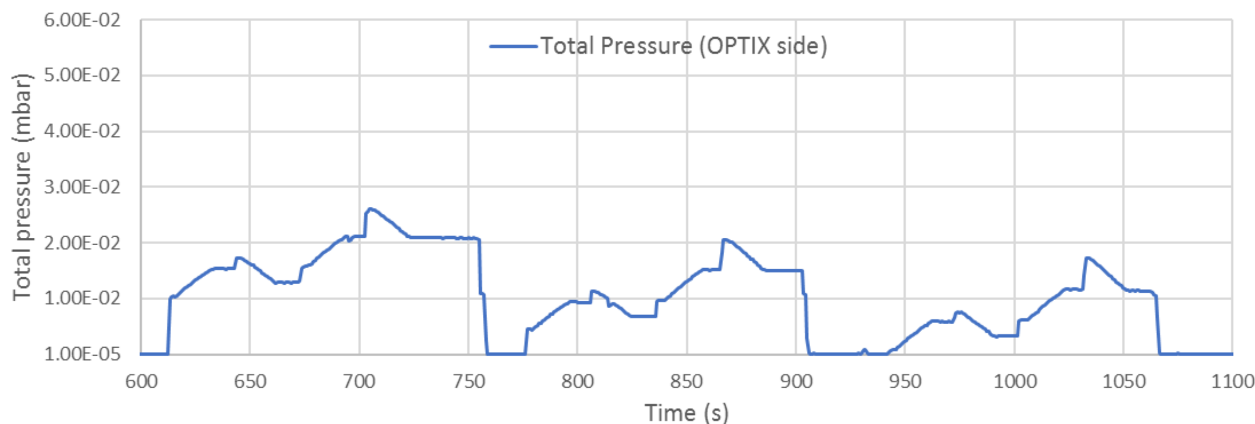
Quantification – Gas interaction

- Ar, N_2, and O_2 were mixed in varying quantities
- Total pressure variation was from 1E-5 to 2E-2 mbar on the high pressure side
- Differentially pumped side was kept below 1E-4 mbar

Diff. pumped RGA



Total pressure



Quantification – Gas interaction

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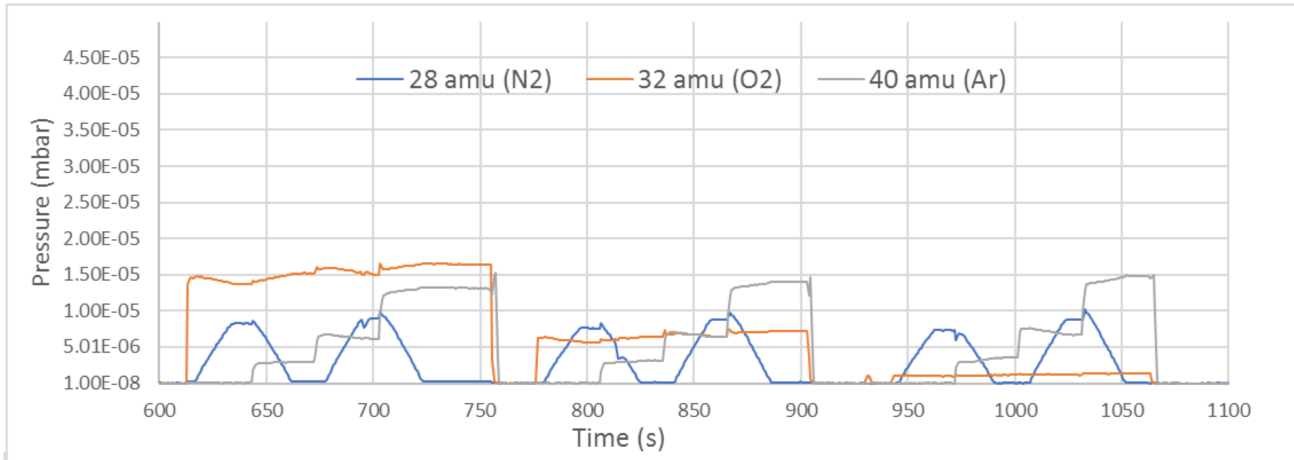
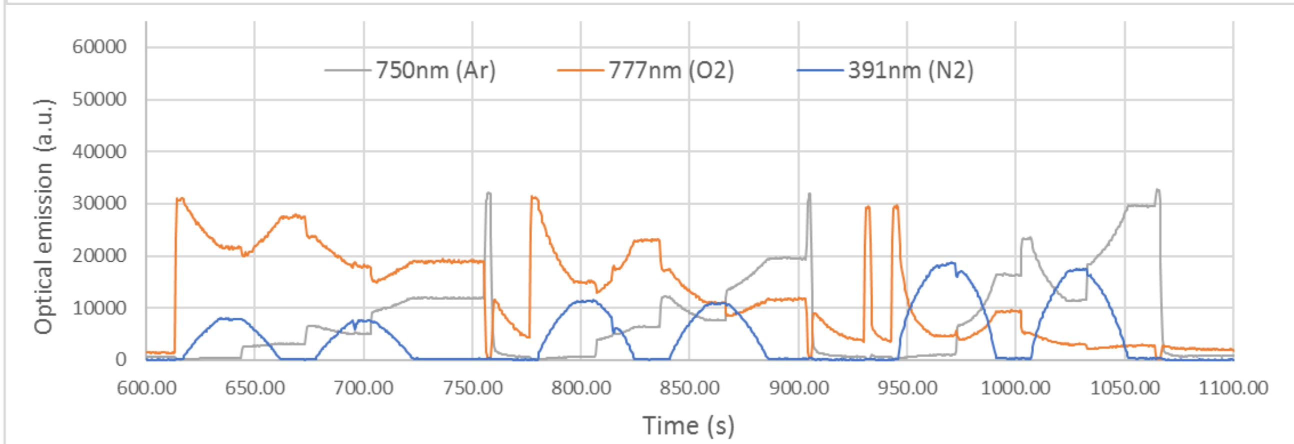
- Gas interaction effects can be clearly seen on the OPTIX readings resulting in different partial pressure measurements compared to the RGA

Diff. pumped RGA



OPTIX readings



Quantification – Accurate gas partial pressure measurements after the gas effect correction algorithm is used

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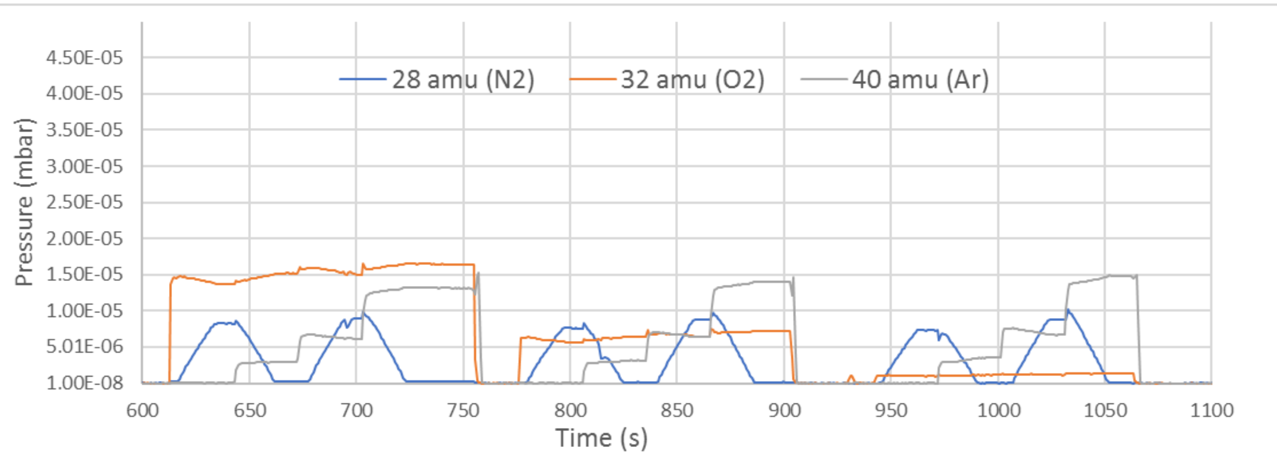
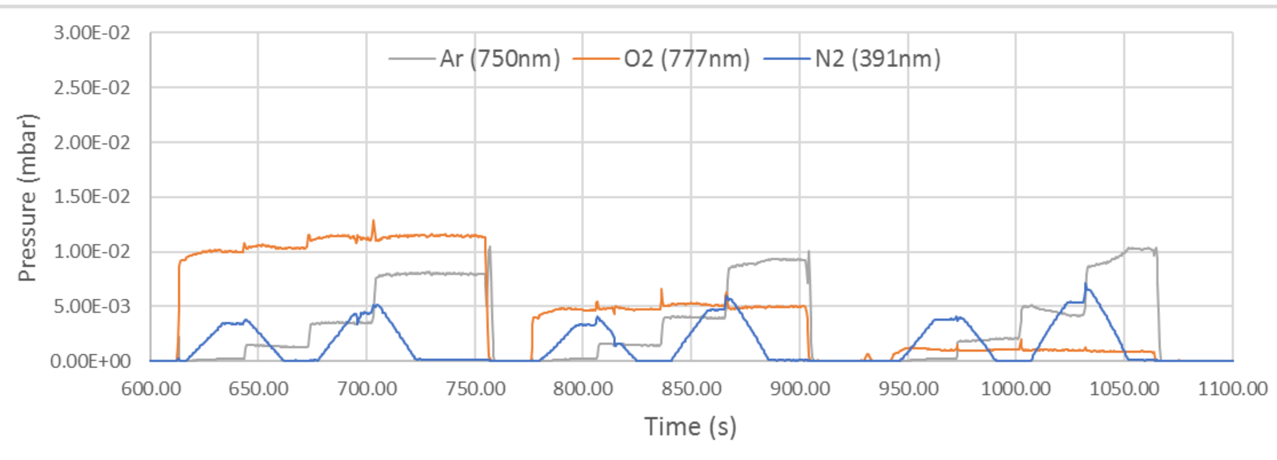
- An algorithm can be used to correct for the interaction effects
- Partial pressures can then be derived

Diff. pumped RGA

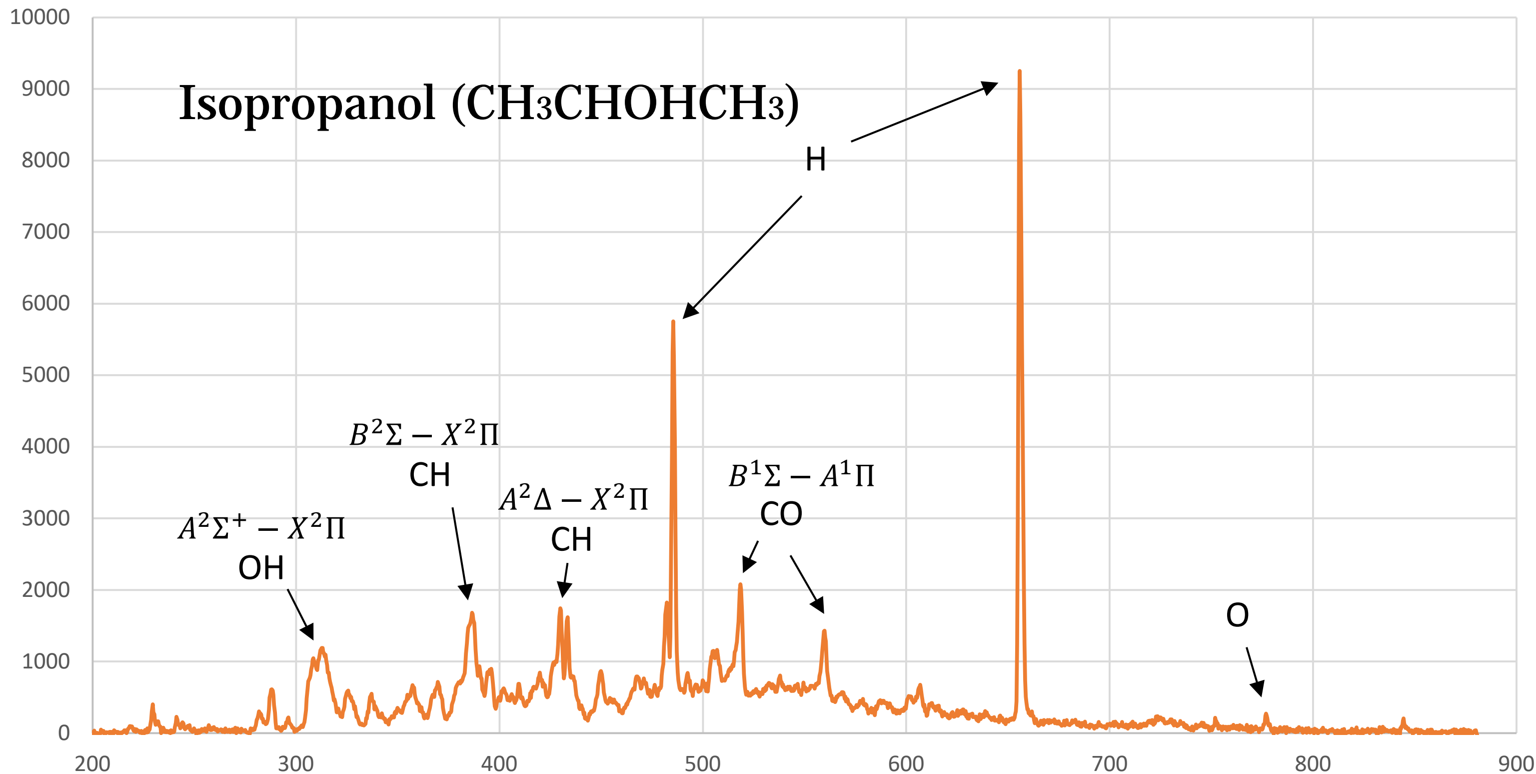


OPTIX readings



Which species can be observed?

- Atomic emissions and molecular emissions
- Larger molecules are observed as fragments – due to disassociation in the sensor's plasma



Caveats and considerations when using Remote Plasma Gas Analysis - Disassociation

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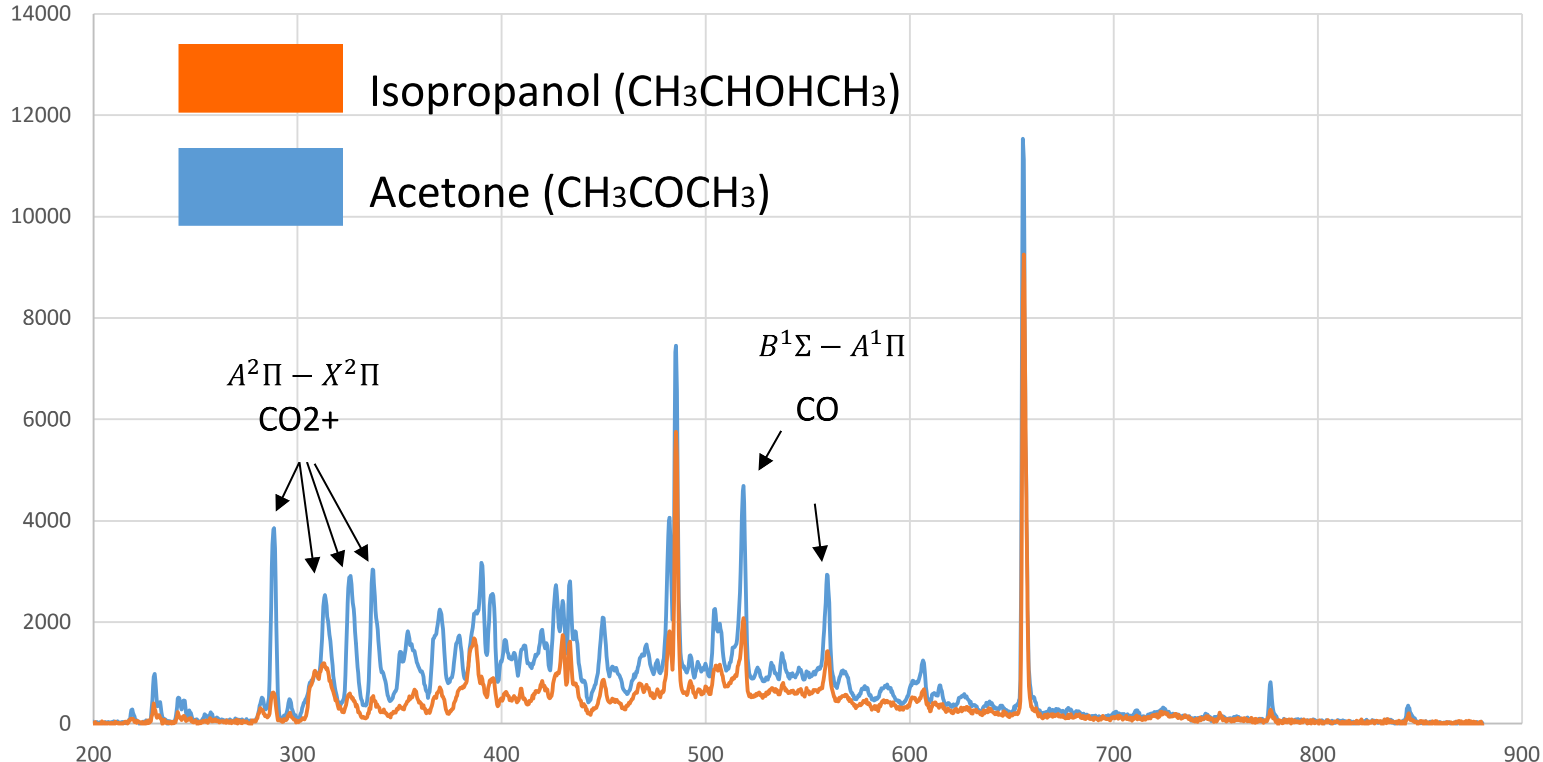
- “Fingerprints” of the original molecule

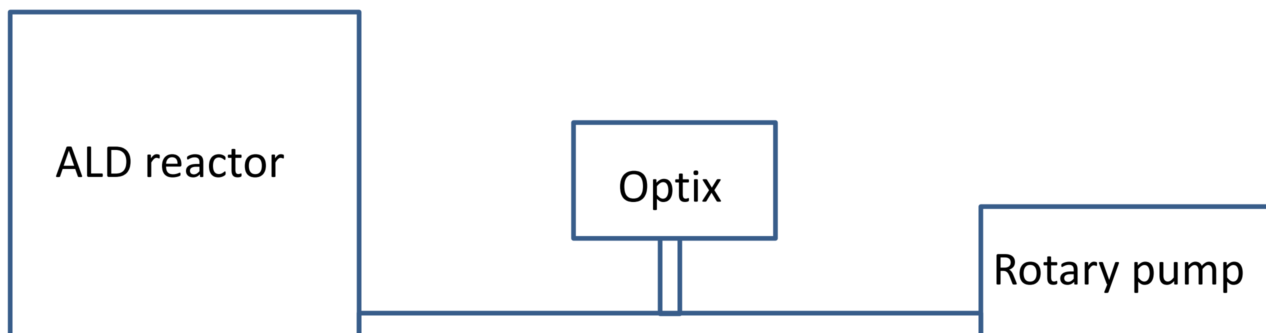


ALD monitoring experimental setup

UNIVERSITY OF LIVERPOOL

University of Liverpool

Dr. Richard Potter and Ben Peek



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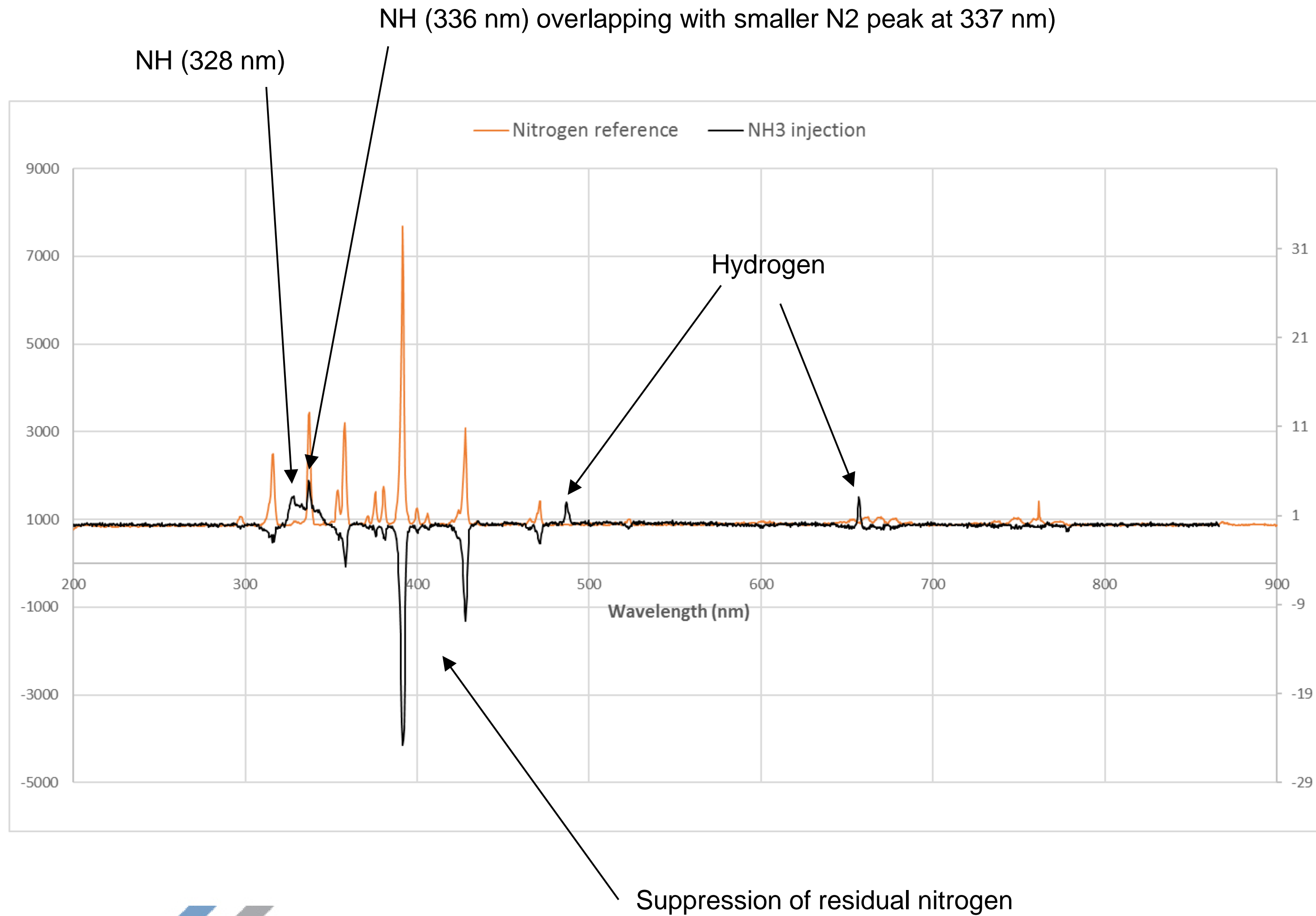
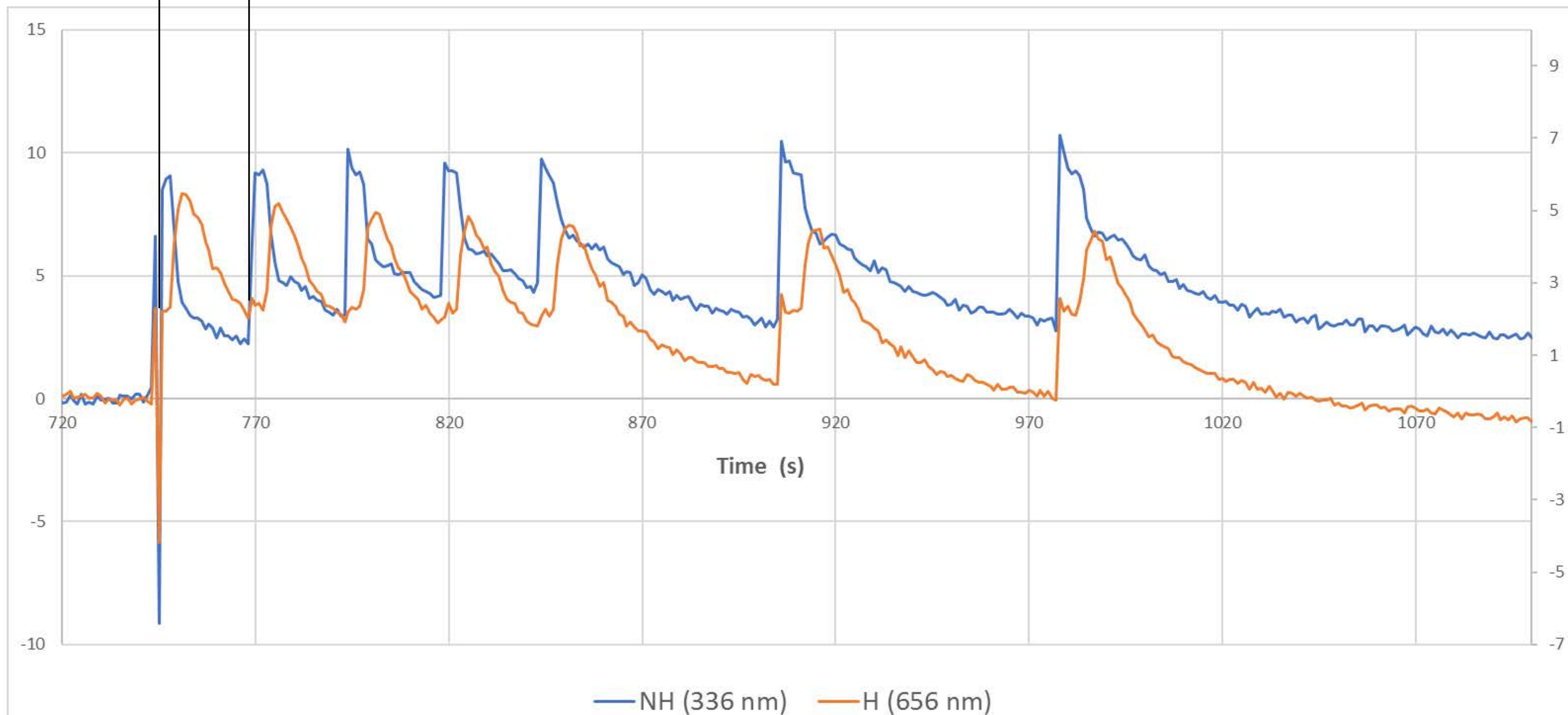
Precursor detection

NH_3



Atomic layer deposition precursor monitoring

NH3



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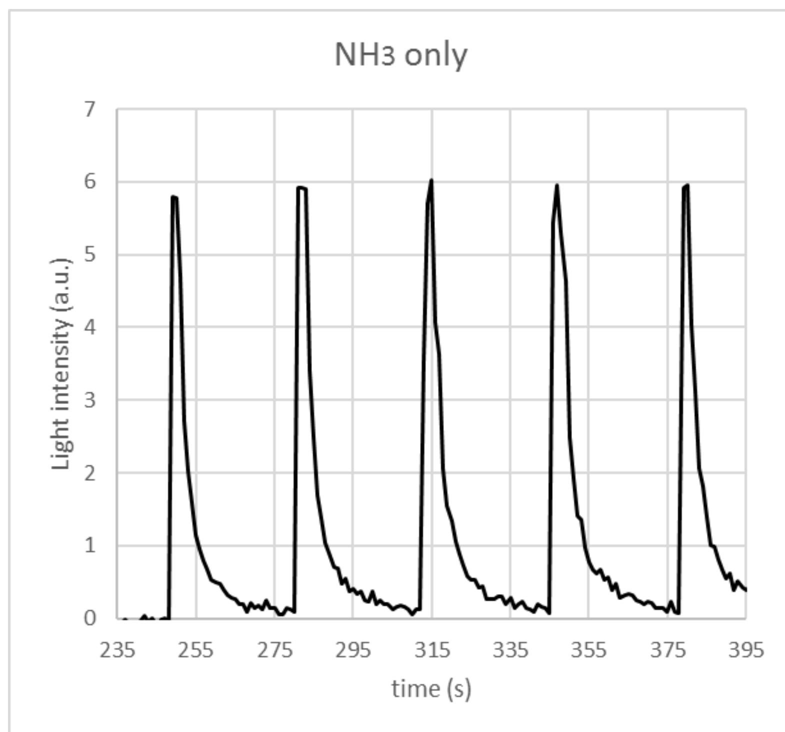
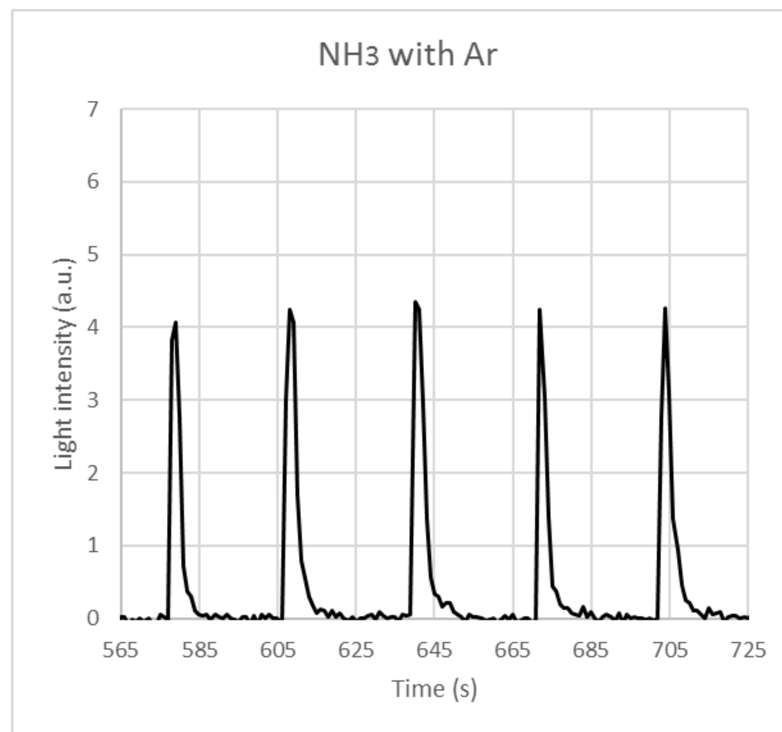
Precursor detection

NH_3

NH (328 nm)





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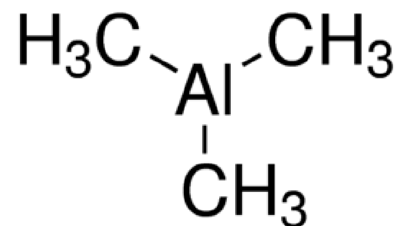
Precursor detection

TMA



CH (431 nm)

H (656 nm)

OH (283 nm)

OH (310 nm)

4
2
0
-2
-4
-6
-8
-10

200 300 400 500 600 700 800 900

Wavelength (nm)

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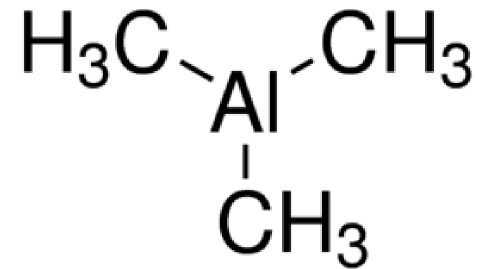
Atomic layer deposition precursor monitoring



TMA

Injection

TMA Ar chamber injection (1sec int, 900uA DC), with heating

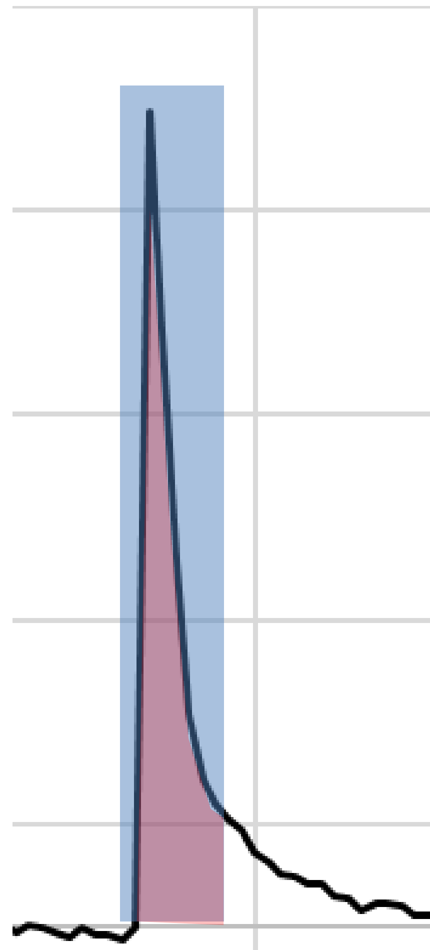
10
5
0
-5
-10
-15
-20
-25
-30

350 370 390 410 430 450 470 490 510 530 550

H (656.9nm) OH (309.5nm) CH (387.8nm) CH 431 (432.1nm)

Deposition cycle monitoring

Synchronisation of the CCD capture with the ALD pulse



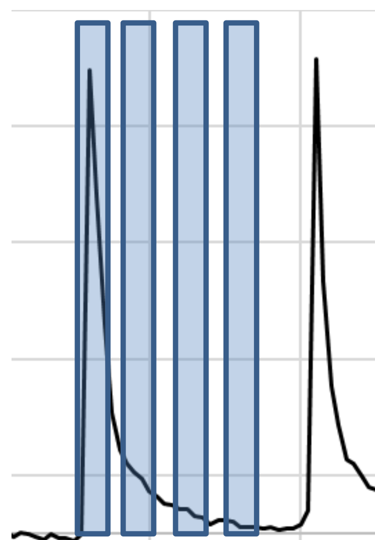
Deposition cycle monitoring

Synchronisation of the CCD capture with the ALD pulse

- A series of CCD spectrum captures is synchronised with each precursor injection

Intercept ALD valve opening signal

Trigger series of integrations



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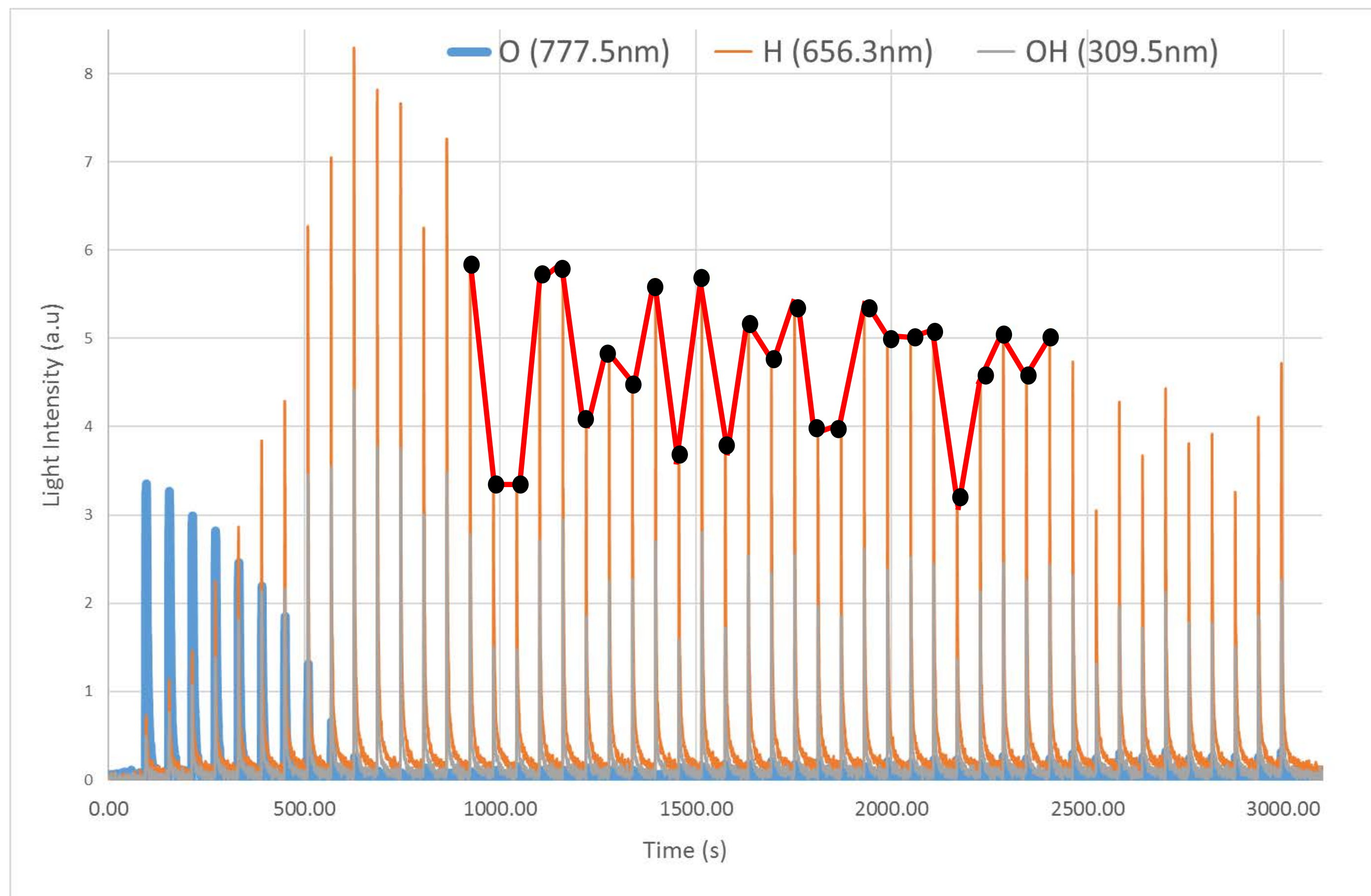
Deposition cycle monitoring

Synchronisation of the CCD capture with the ALD pulse

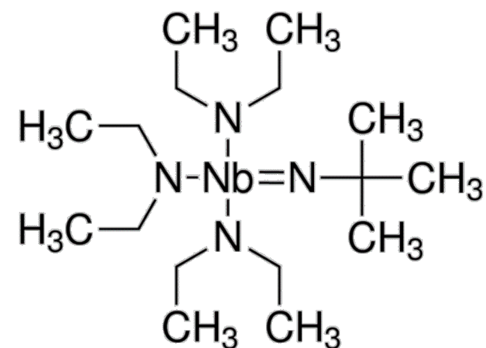
- The H maxima of each precursor pulse was recorded



Application Example Atomic layer deposition precursor monitoring Deposition of NbN via PEALD

ALD user, Japan

- Detection of TrisNb via CH, N and H



- Detection of NH3 via N and H

OPTIX Atomic layer deposition precursor monitoring

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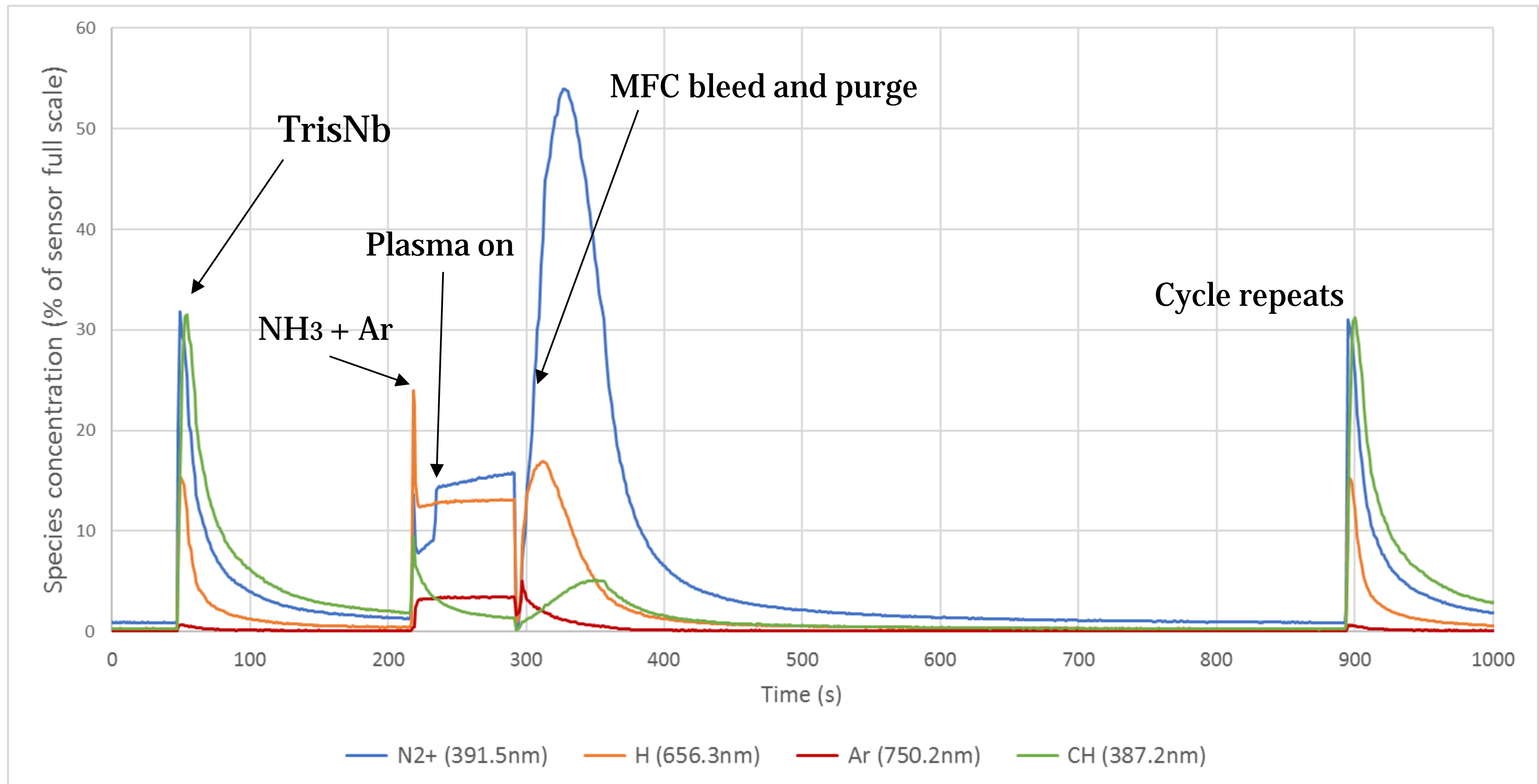
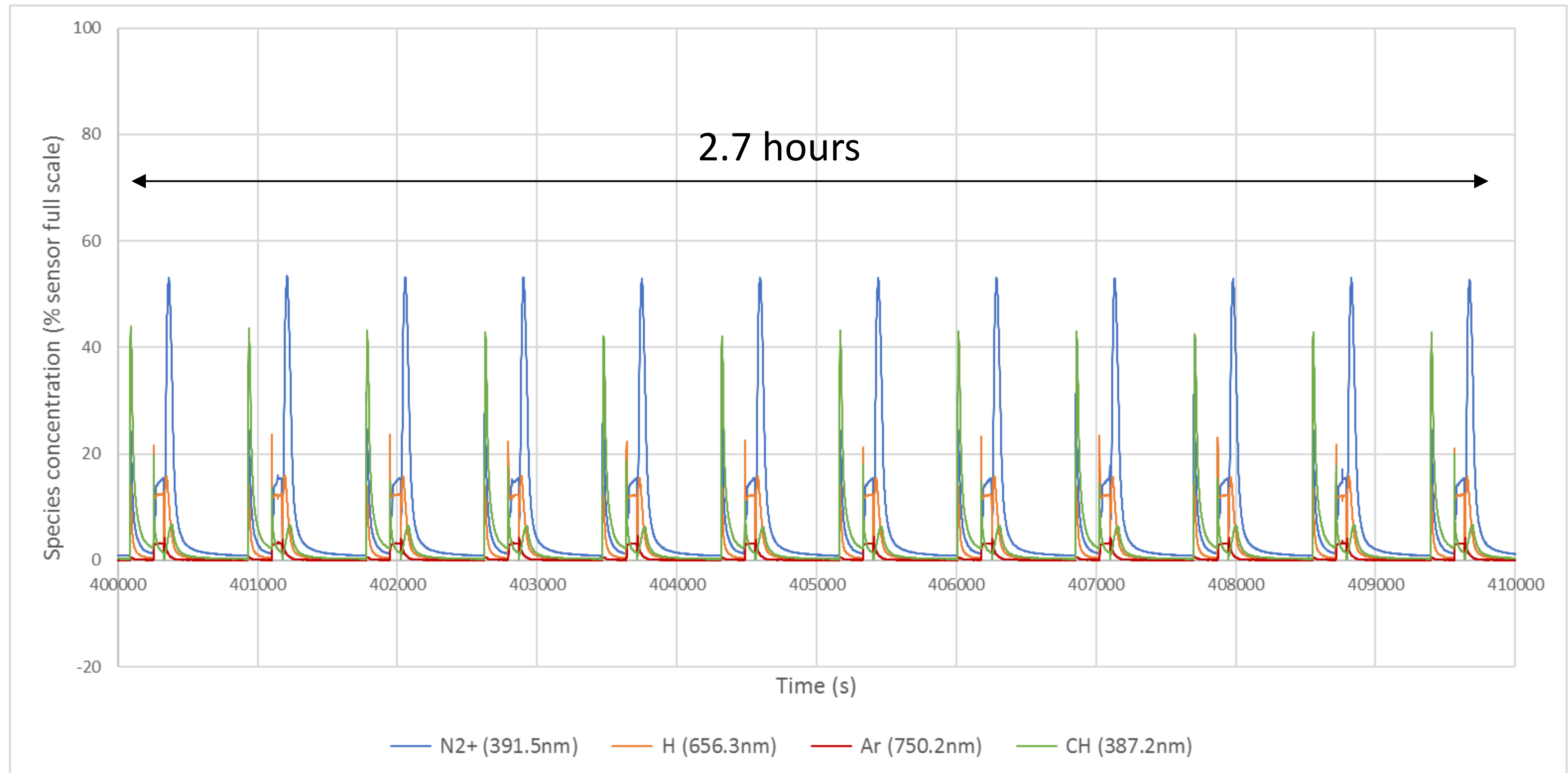
NbN deposition step



ALD Monitoring – Full process for 2.7 hours

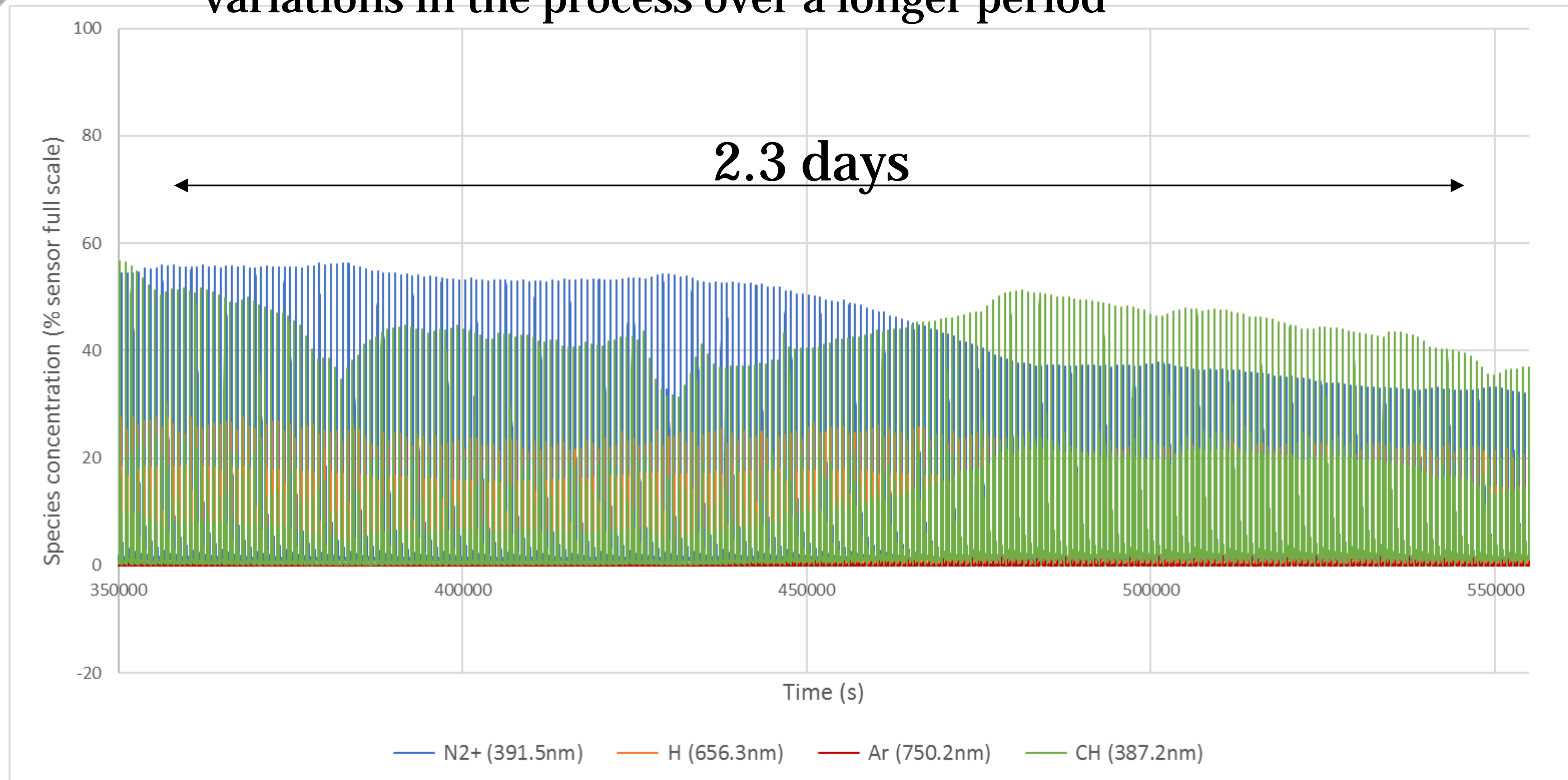
Deposition of NbN via PEALD



Atomic layer deposition precursor monitoring

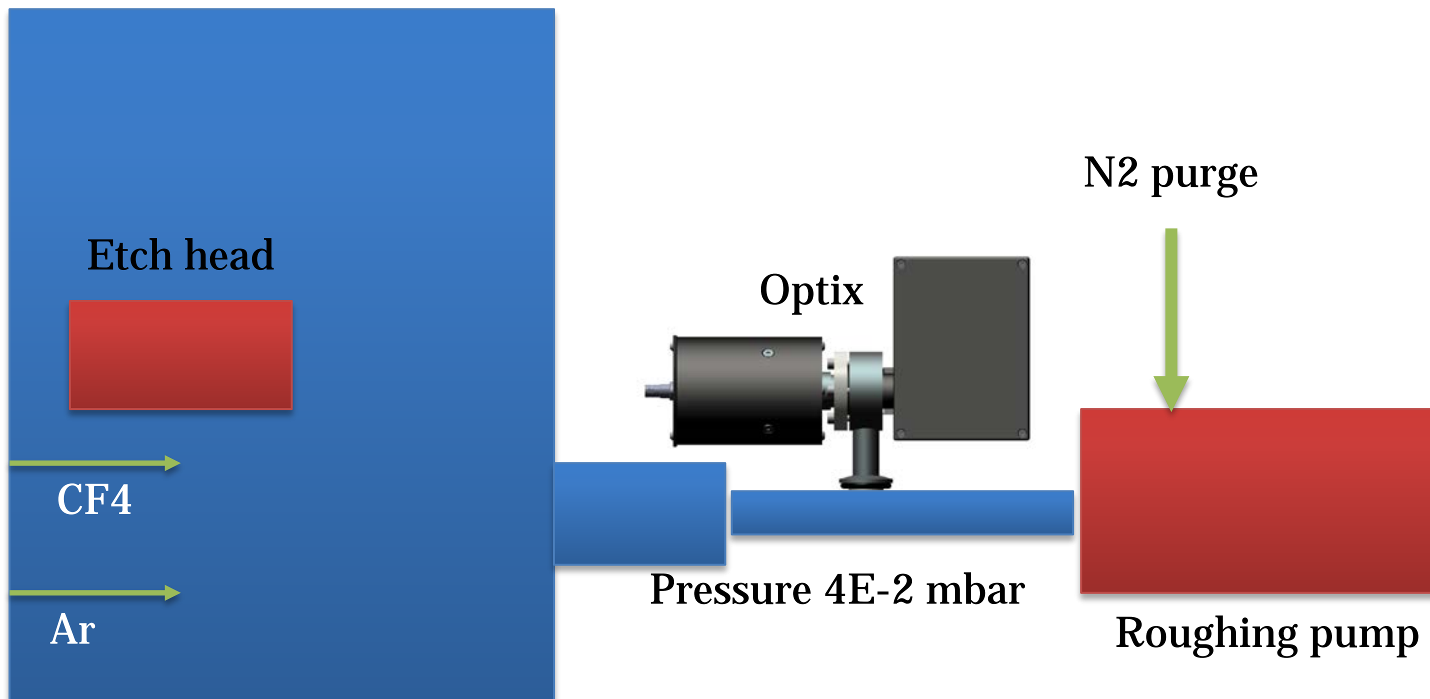
Deposition of NbN via PEALD

- Sensor is robust of the full 2+ day deposition cycle and displays variations in the process over a longer period

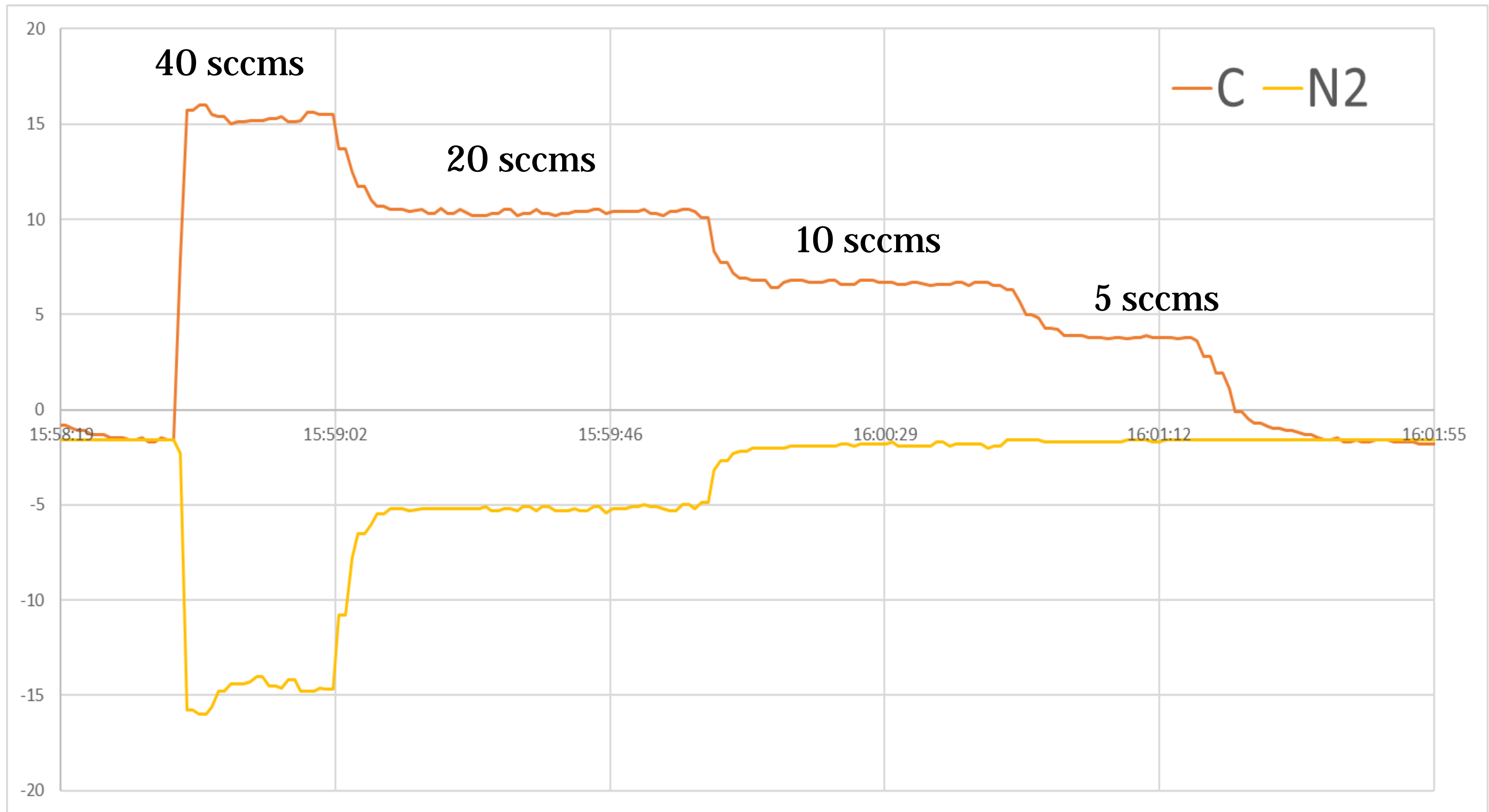


Application Example - Characterising a reactive ion etch process

Detection of reactive ion etching effluent in the process backing line



CF4 detection (no Ar background)



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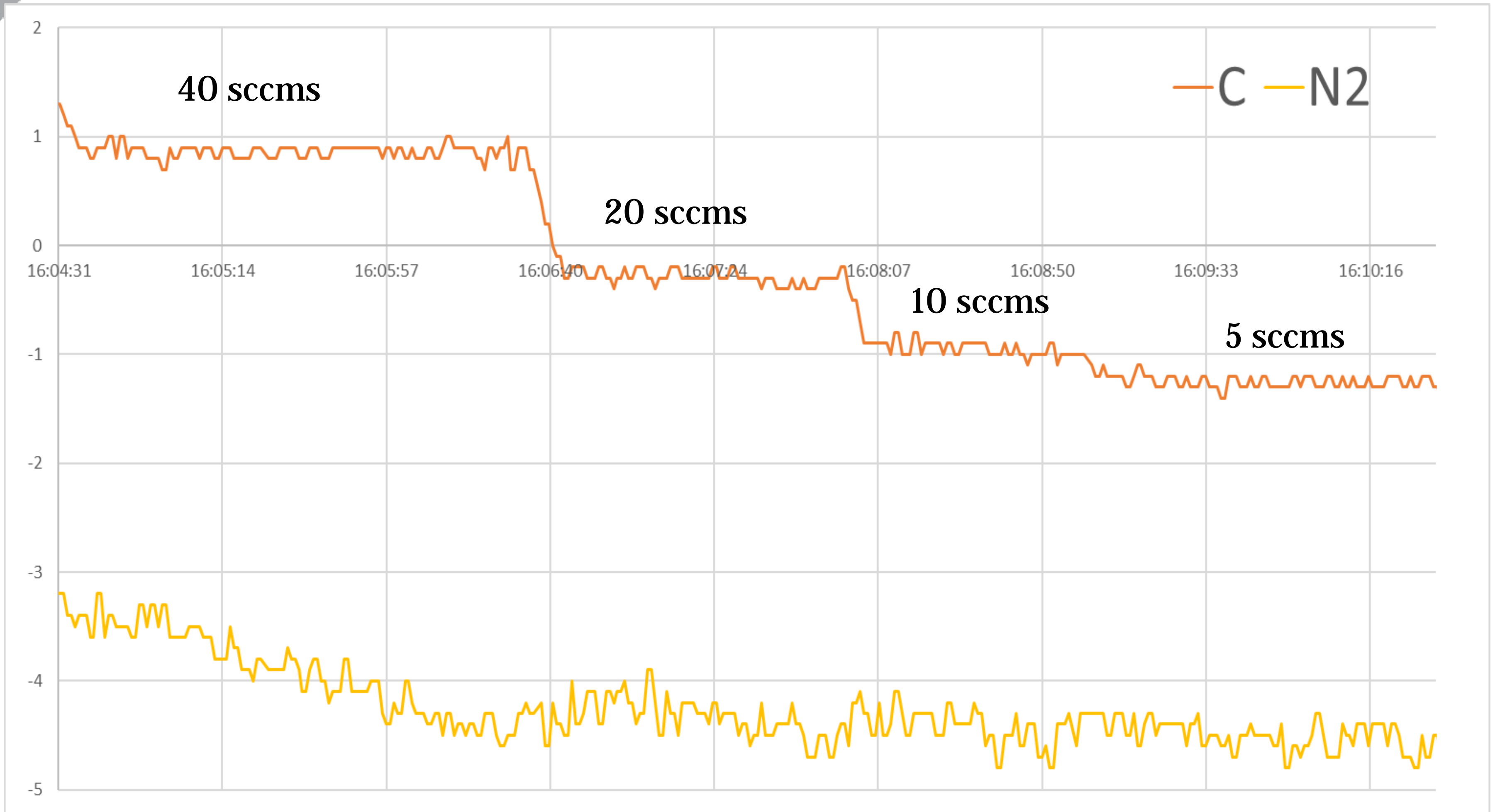
CF4 detection (Ar background)



Sensing from atmosphere

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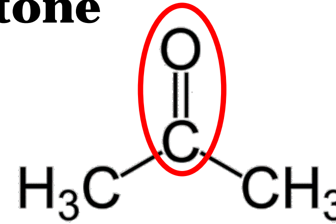
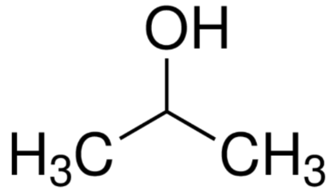
Acetone has a higher CO reading due to the presence of a CO bond

Acetone



IPA

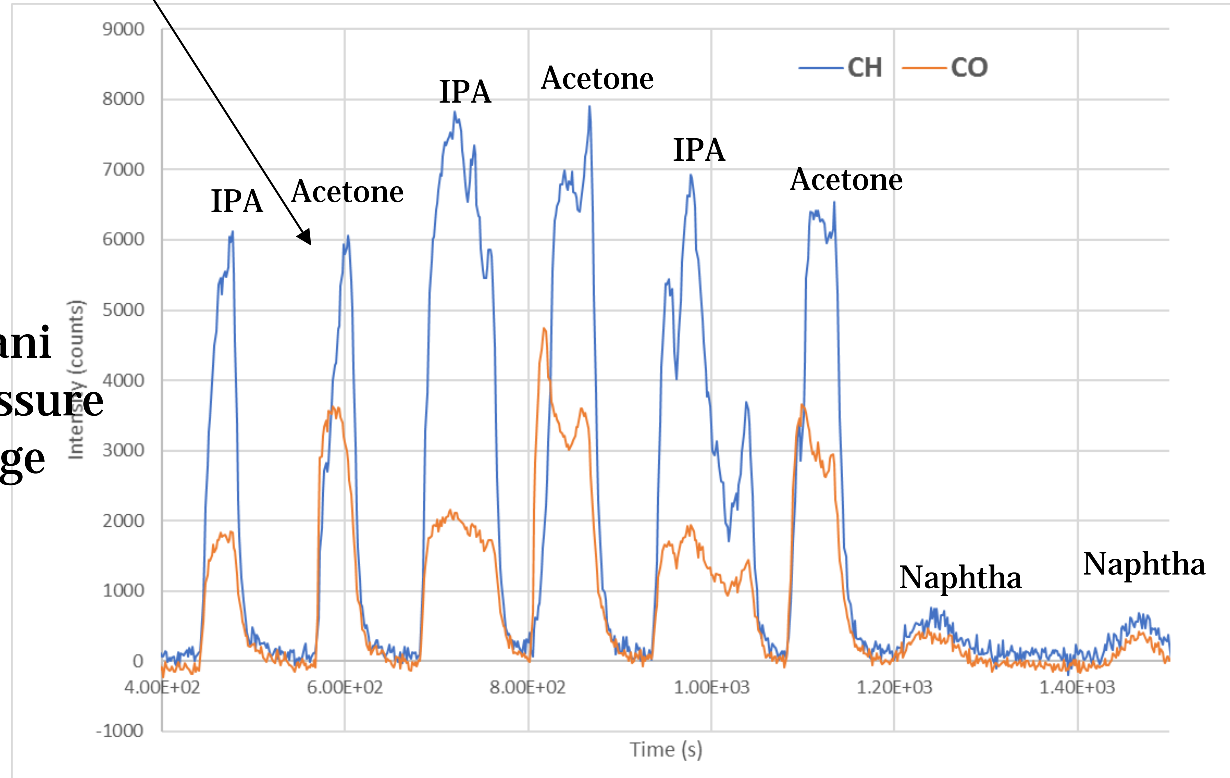


Optix

Pirani pressure gauge

Needle valve

Rotary pump



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Summary

- Remote PEM combined with spectroscopy can perform “RGA-like” functions
- Can use this method directly at higher process pressures – no need to differentially pump unless atmospheric sensing
- The detector is separated from the vacuum environment hence not affected by hostile chemistry present in the vacuum
- OPTIX is hence less sensitive to contamination than RGA’s, can be used for ’dirty’ hydrocarbon environments as well as etch, CVD and ALD type processes.
- This sensing technique is highly robust – plasma generator will not contaminate or stop functioning