Transforming Leak Detection in Vacuum Coating Systems with Remote Plasma Optical Emission Spectroscopy (RPOES)

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Vacuum integrity and quality

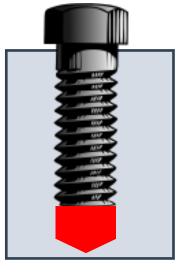
Ensuring the integrity of vacuum systems prior to vacuum deposition and surface treatment processes is paramount, as even minor leaks can compromise the quality of coatings and lead to costly production downtimes

Typical bottlenecks for vacuum quality

- **Leaks** – Physical holes, cracks in the chamber or welding, compromised seals or loose fittings
- **Virtual Leaks** – Trapped volumes inside components such as screw threads or O-ring groove spaces slowly releasing gas
- **Permeation** – materials slowly letting gas through such as gas lines

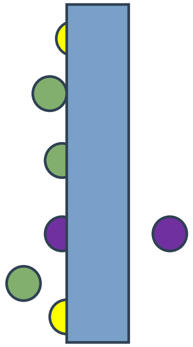
- **Water vapour** – moisture absorbed on vacuum surfaces, dependant upon exposure time and humidity levels
- **Organic outgassing** – materials releasing gas molecules over time due to contamination
- **Pumping issues** – oil backstreaming

Damaged O-ring



Trapped volume

Outgassing & permeation



Leak detection methods

- **Rate of rise** – unable to distinguish the cause of the leak. Leak rates dependant upon initial pump down time
- **Base pressure** – unable to distinguish the cause of the leak and can often take days to diagnose
- **Dedicated helium leak checkers** – Helium cost and supply chain issues. Filament degrades over time and is susceptible to contamination with regular maintenance and calibration required
- **Mass spectrometers (RGA)** – sampling of vacuum system typically involved. Complex systems with sensitive components. Often over sensitive and expensive for the specific requirement. Complex analysis required and often contaminants share the same mass

Leak flow (Q)

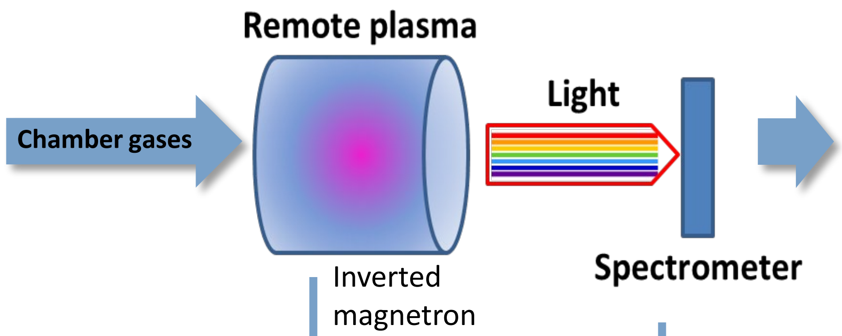
$$Q = \frac{\Delta P \cdot V}{\Delta t}$$

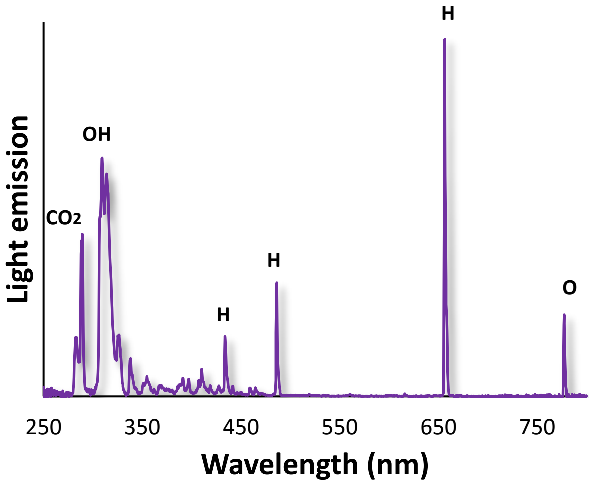
ΔP – Pressure change
V – Volume
Δt – time taken for pressure change

Q - typically expressed in **mbar·l/s**

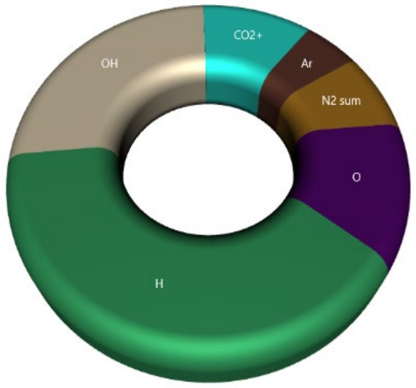
RPOES …

Remote Plasma Optical Emission Spectroscopy (RPOES) method



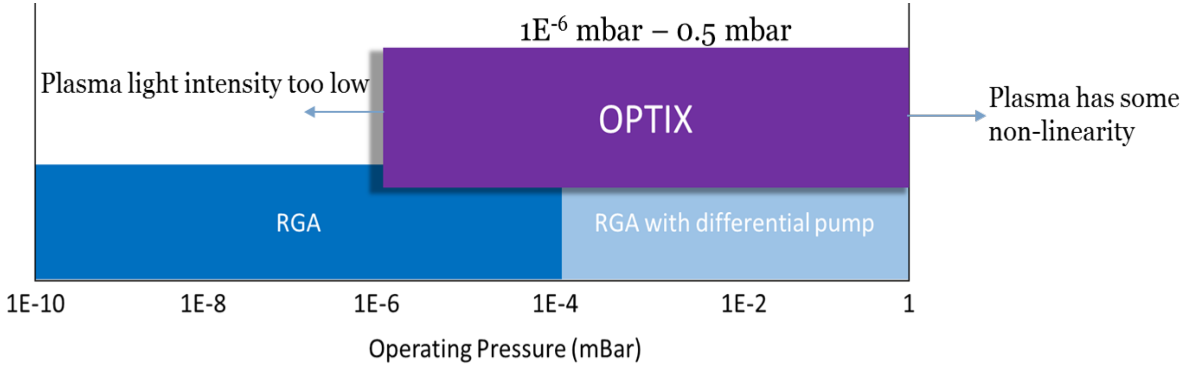


Gas partial pressures

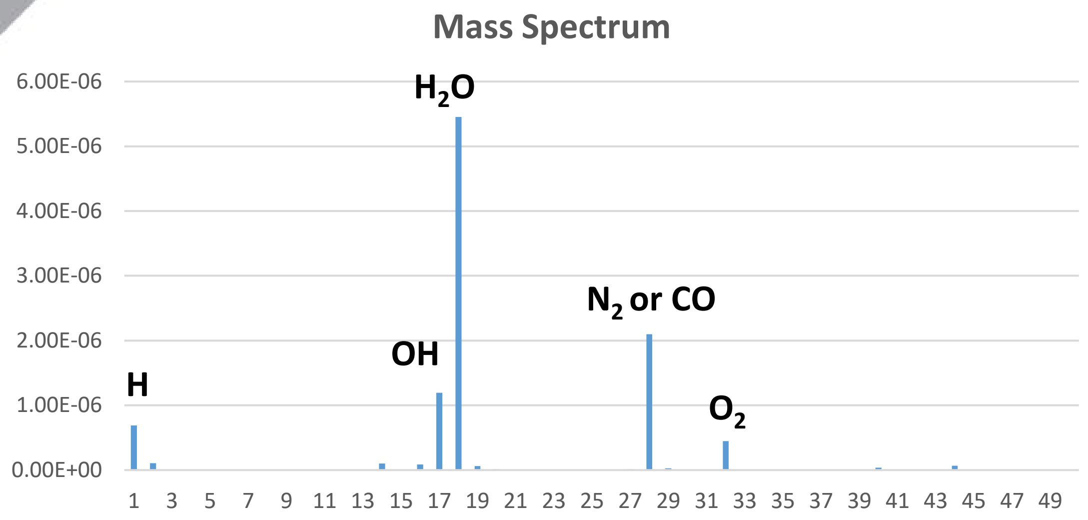


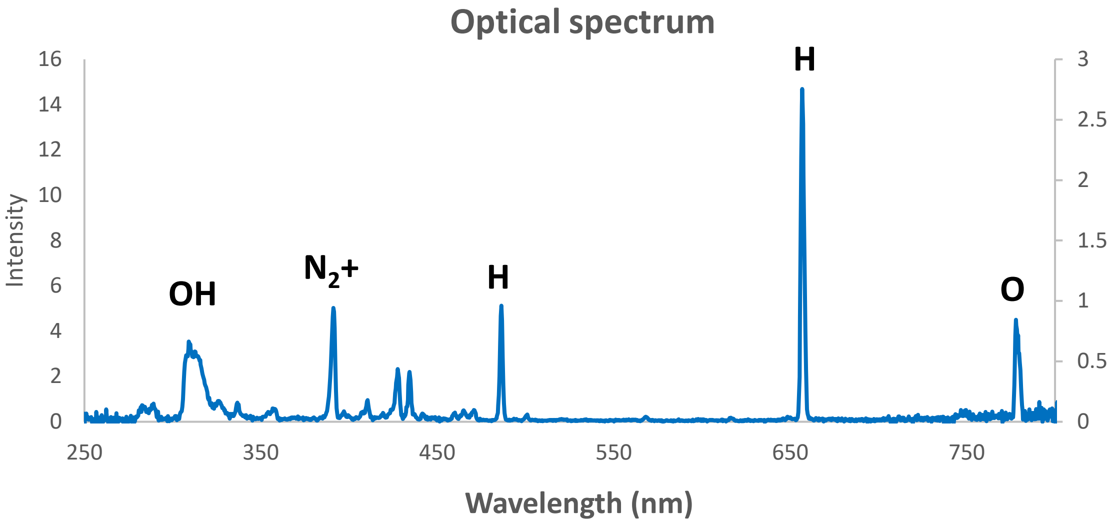
< 100 ms updated partial pressures

Measurement Range



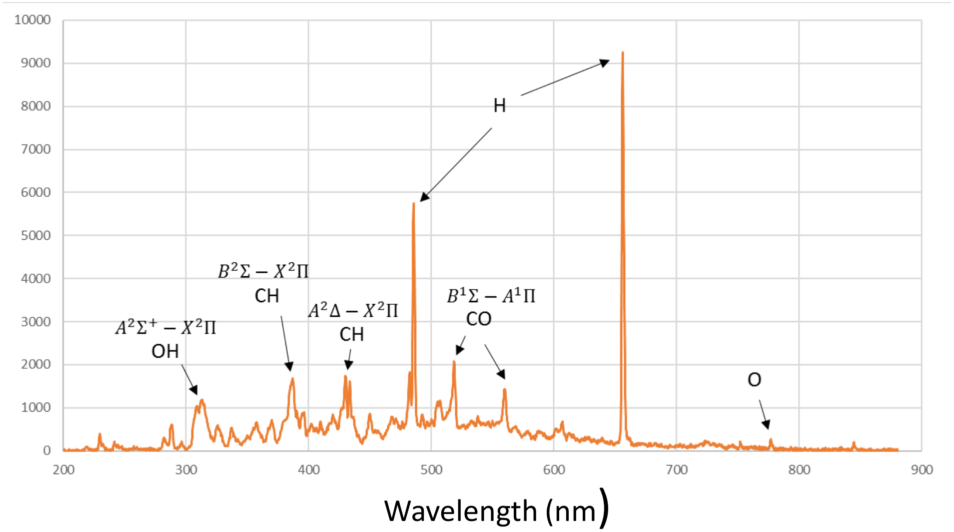
Mass vs Optical spectrum





- Remote plasma will break larger molecules into smaller components
- Prominent, defined peaks from component species - mass spec would require high mass detection as cracking low

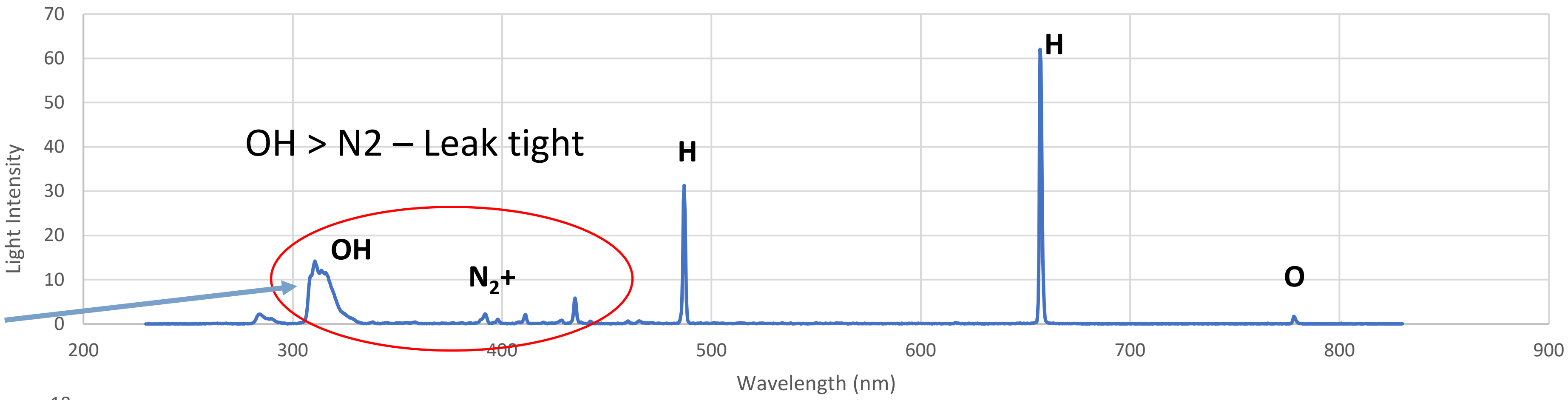
Isopropanol (CH3CHOHCH3)

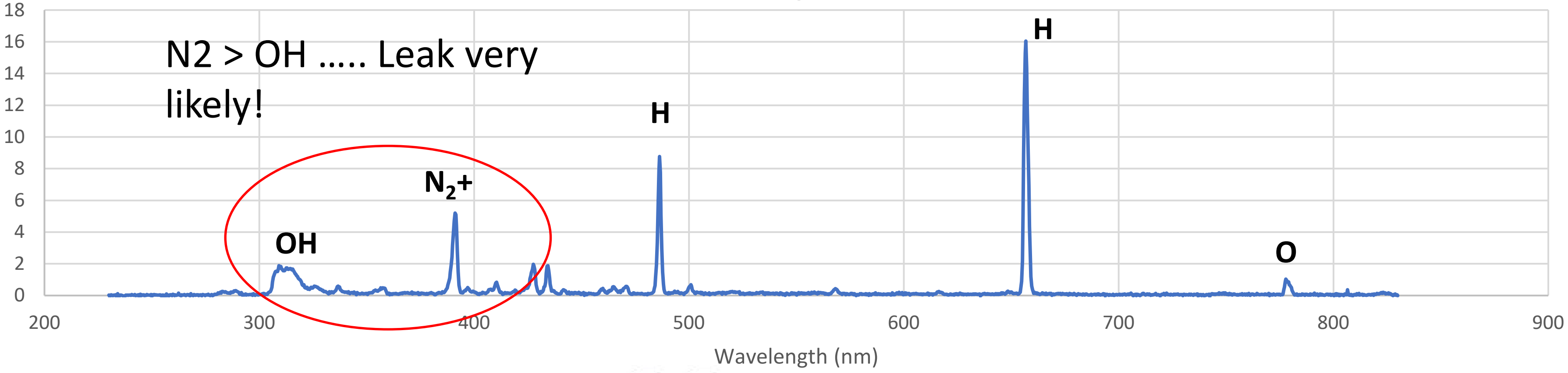


Application examples – Water and Air

- water vapour – OH and H emissions
- Example of "Clean" vacuum system
- Small amount of N_2 in relation to water vapour
- General rule that < 1E-2 mbar a leak tight system should not have significant N2 present

Residual OH



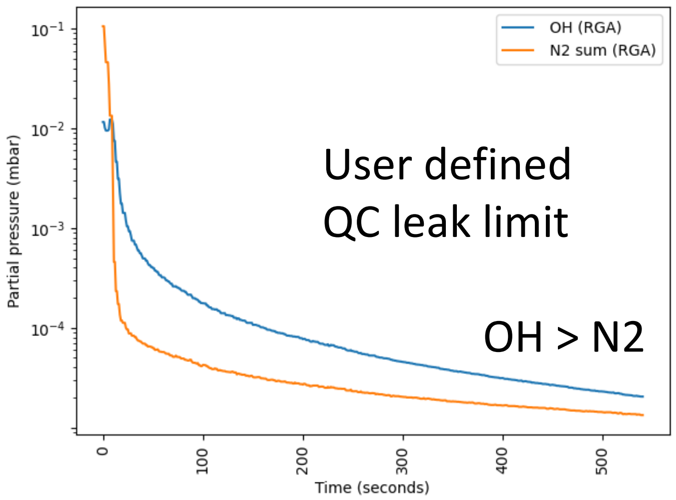


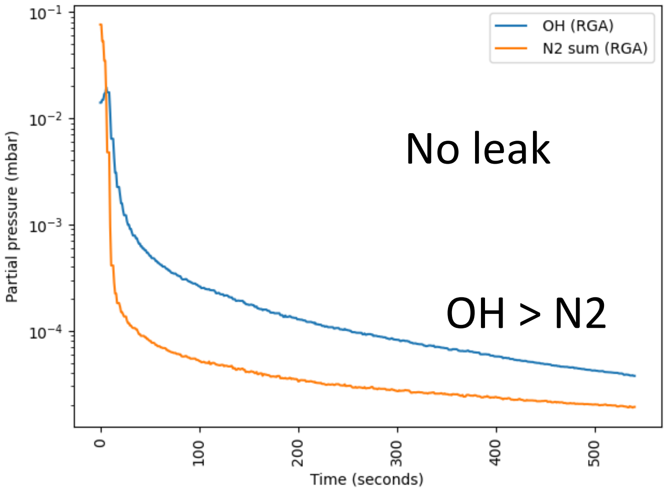
Application examples – Air leaks during evacuation

Water signature = OH

QC vacuum chamber specification

OH > N2
N2 sufficiently low

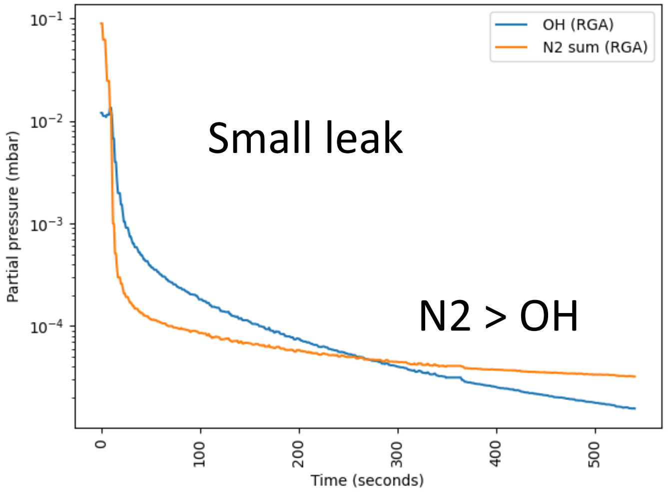


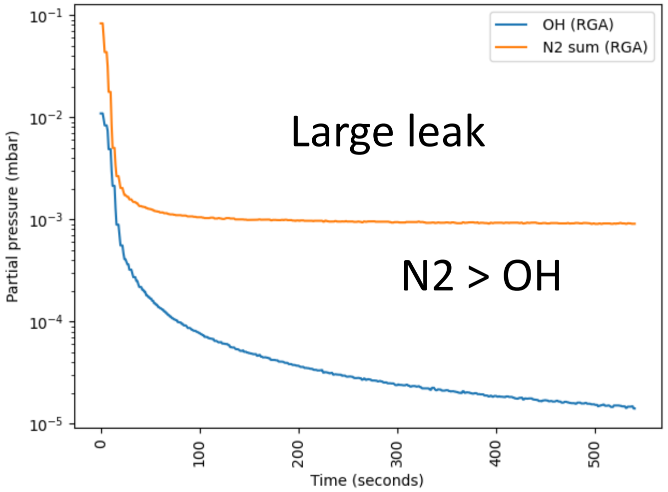


Passing partial pressure traces for user defined leak limit

OH (RGA)
N2 sum (RGA)

Falling QC vacuum chamber

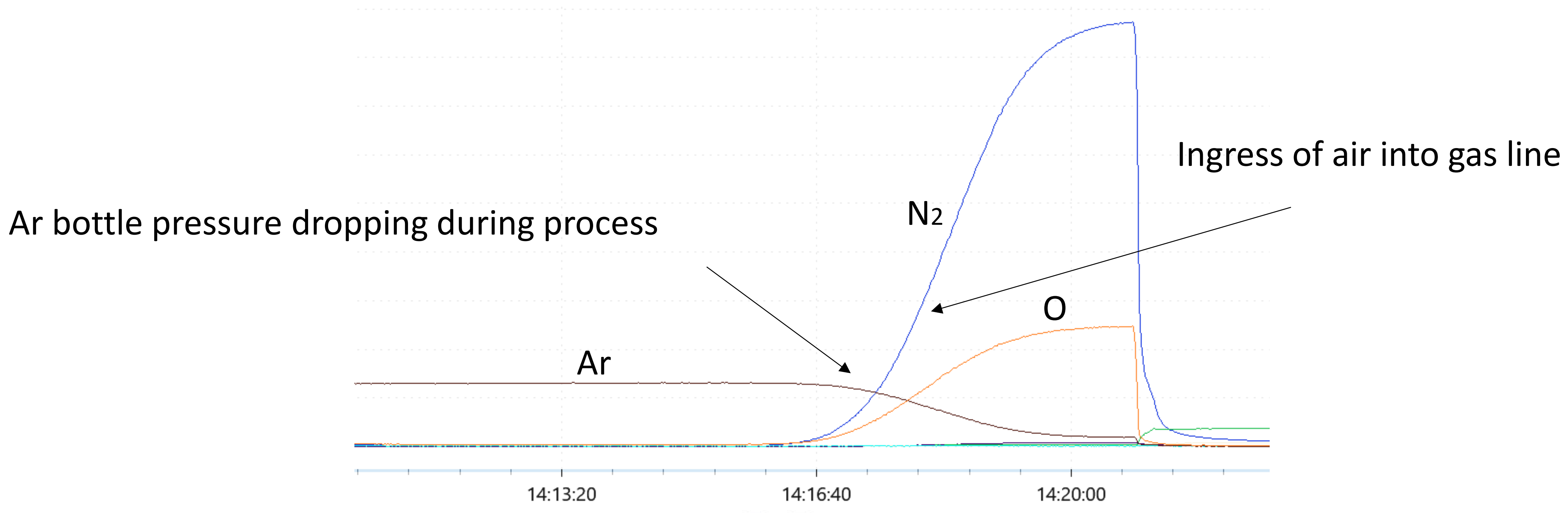




Very large N2 concentration – large leak

Application examples – Gas line check

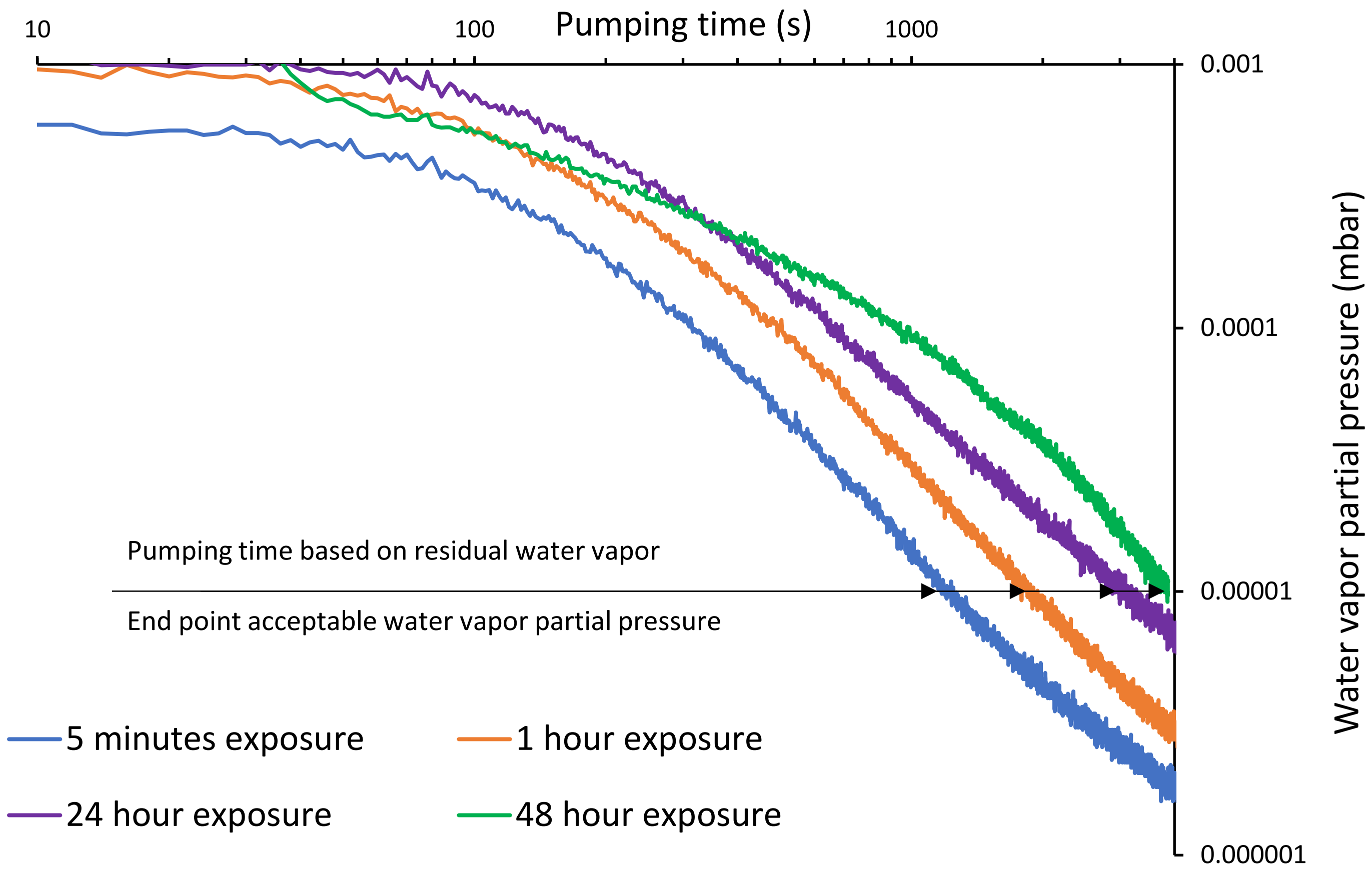
- Ar process gas line contaminated with air
- MFC feedback would have shown no problem, Pressure gauges would also not detect the problem
- **No system leak to detect** – in situ gas monitoring only way to see this



Application examples – Water

The effect of atmospheric exposure on water vapor concentration

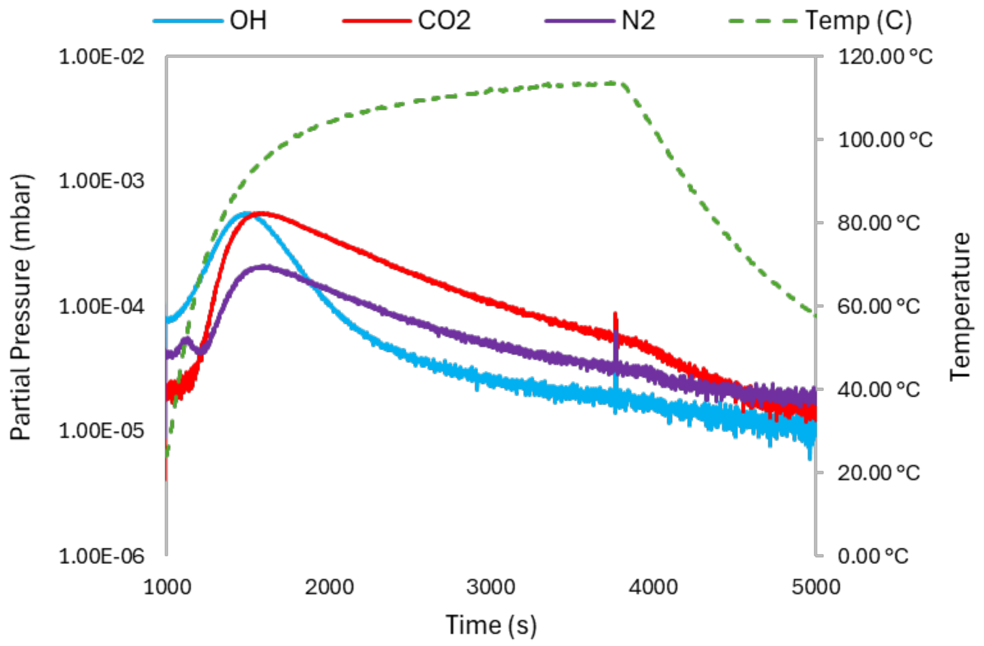
Chamber exposed to atmosphere for several different times



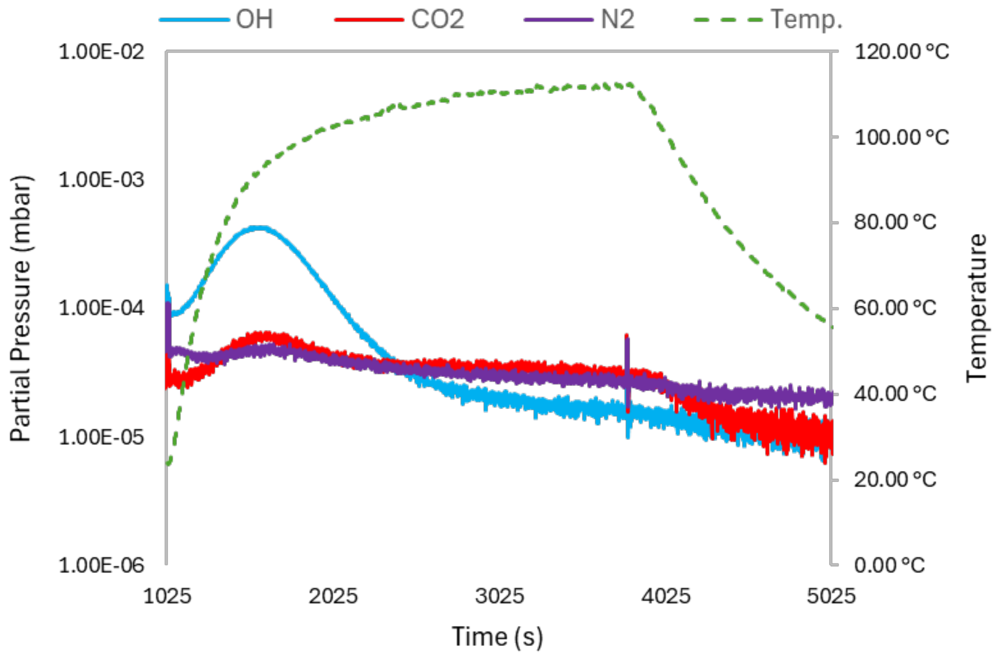
End point detection - reduces degassing cycle times significantly, while ensuring contaminant thresholds are met.

Case study – Organics outgassing
Barrier coatings

Polymer A – **No Barrier**



Polymer A – **SiN Barrier**



- Evolution of outgassing of polymers in vacuum with time – no barrier coating vs SiN (silicon nitride) barrier layer
- The barrier coating significantly reduces outgassing of CO_2 and N_2 ,providing a quantification of the effectiveness of the coating

RPOES as an alternative to conventional mass filter-based helium leak checkers?

The current state

Helium – Expensive (~ double in cost over the last decade) and general supply chain issues

Mass spectrometer – highly sensitive (below 10^{-11} mbar·l/s) sensitive to contamination, maintenance required, and separate pumping required

An alternative

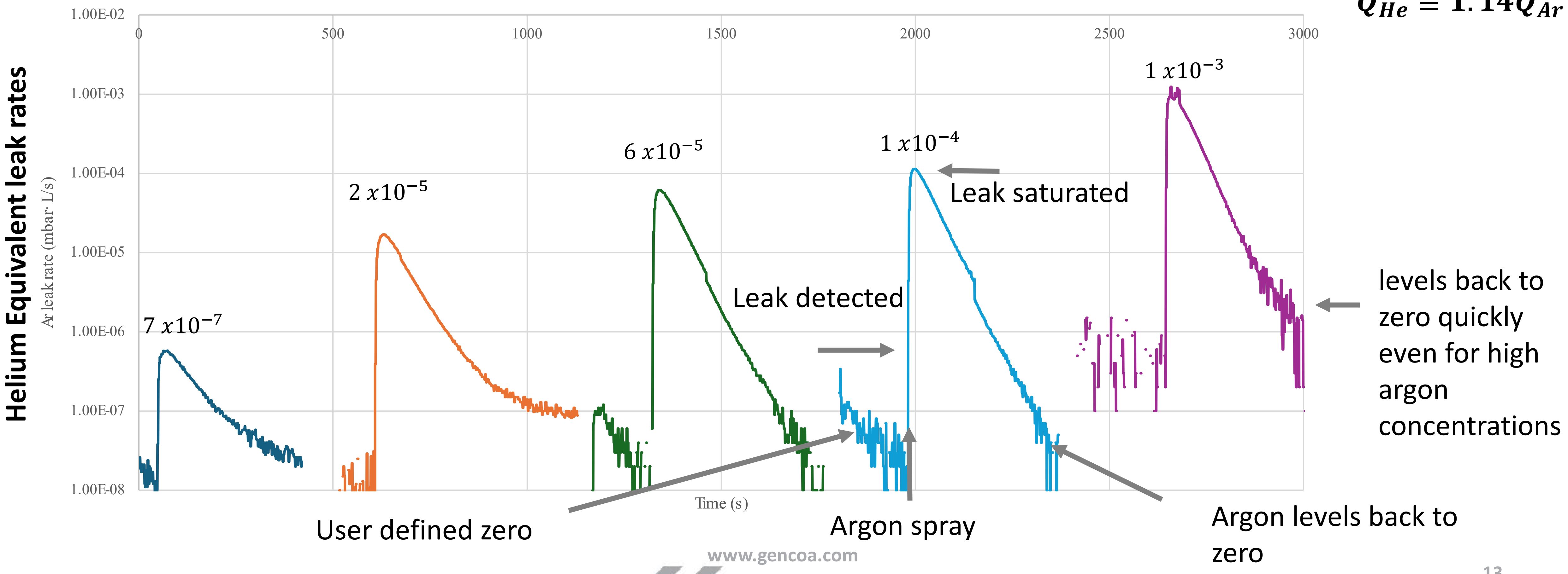
Argon – relatively cheaper and widely available

RPOES – Operates from 0.5 mbar, detect leaks in the 10^{-3} to 10^{-8} mbar·l/s range (helium equivalent). Often no leak check gas required

Argon spray experiments using RPOES

- Dosing valve attached to the vacuum chamber used to create leaks
- Calibrated (reservoir) leak used to convert Ar signal to leak rates
- Argon sprayed at the opening
- Signal recorded for various leaks.

Viscous flow:
$Q_{He} \cong 1.14 Q_{Ar}$



Advantages of RPOES vs conventional methods for leak checking

- Lower cost - no need for pump / sampling system
- High speed – direct contact to the vacuum and ‘speed of light’ delivery to spectrometer
- Reduced maintenance – No complex filaments
- Increased portability – small, compact device which can be hand carried
- True gas concentrations – not sampled concentrations - faster signals and identification of the leaking gas (can measure virtual leaks)

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Summary

Remote Plasma Optical Emission Spectroscopy (RPOES):

- Powerful tool for quantifying vacuum integrity in real-time.
- Noninvasive
- Wide pressure range 0.5 mbar down to 10^{-6} mbar
- rapid results with data collection speeds under 100 milliseconds.
- Can be used as a standalone leak detection solution without the need for helium to leak rates as low as 10^{-8} mbar·l/s (work still in process)

Thank you for your attention!
For further discussion, please visit us at our exhibition booth 1011