



© 2023 Society of Vacuum Coaters all rights reserved, ISSN 0737-5921, ISBN 978-1-878068-43-9

Process Monitoring and Control by Remote Plasma Optical Emission Spectroscopy

Erik J. Cox, Dermot Monaghan, Joseph Brindley, Benoit Daniel, Victor Bellido-Gonzalez
Gencoa Ltd, Liverpool, United Kingdom

Monitoring of the vacuum is essential for the efficient operation of any vacuum process. In particular, residual gas analysis (RGA) can be performed with quadrupole mass spectrometers. RGA allows for detection and identification of individual species within the vacuum. This can result in higher process yields through faster troubleshooting, scrappage reduction through contamination detection, and/or a more controlled vacuum environment. The limiting factor for quadrupole RGAs is the pressure range over which they can operate. Above 1×10^{-4} mbar damage will occur to the sensor's filament. An alternative residual gas monitoring sensor that operates directly at pressures above 1×10^{-4} mbar has been built around plasma emission monitoring. A small "remote" plasma is generated inside a vacuum sensor. Consequently, species that are present within the vacuum will become excited in the sensor's plasma, emitting a spectrum of light, which can then be used to identify and monitor the emitting species, resulting in a robust, lower-cost, multi-purpose vacuum sensor. Importantly, this plasma, generated inside the sensor, has a sole function as a gas detector and does not affect the process itself. Presented are examples and findings from using this method for monitoring of plasma treatment and deposition processes and comparisons with an equivalent differentially pumped RGA.

<https://www.svc.org>

DOI: <https://doi.org/10.14332/svc23.proc.0020>



Process Monitoring and Control by Remote Plasma Optical Emission Spectroscopy

**Erik J Cox, Dermot Monaghan, Joseph Brindley, Benoit Daniel, Victor
Bellido-Gonzalez,**
Gencoa Ltd, Liverpool, Merseyside, UK





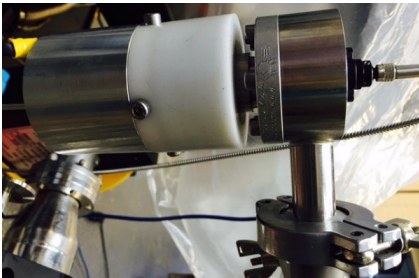
'Optix' – A remote plasma optical emission spectrometer



Optix history

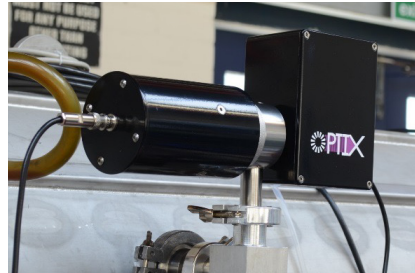
- Introduction to the market in Feb 2017
- Had to trailblaze as there were no serious similar products
- Over 400 global sales to date (US, Europe, China, Japan, Korea)
- Customers including Apple, Samsung, Huawei, CERN, GKN, Lawrence Livermore National Laboratory

Development (2015 – 2017)



2015

Market introduction (2017)



2017

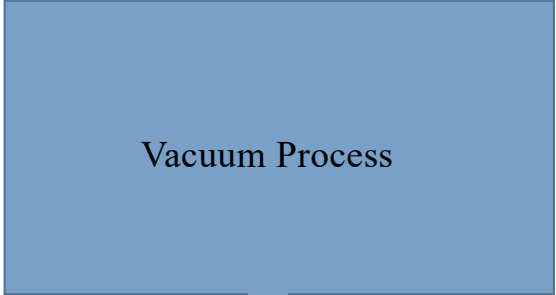
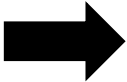
“Version 2.0” (2020)



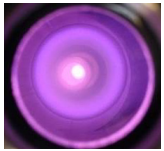
2020



RPEM principal



Remote plasma generator

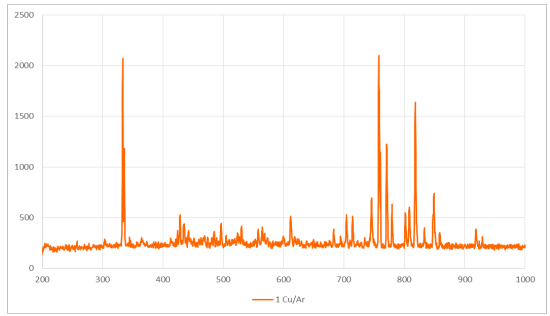


Plasma



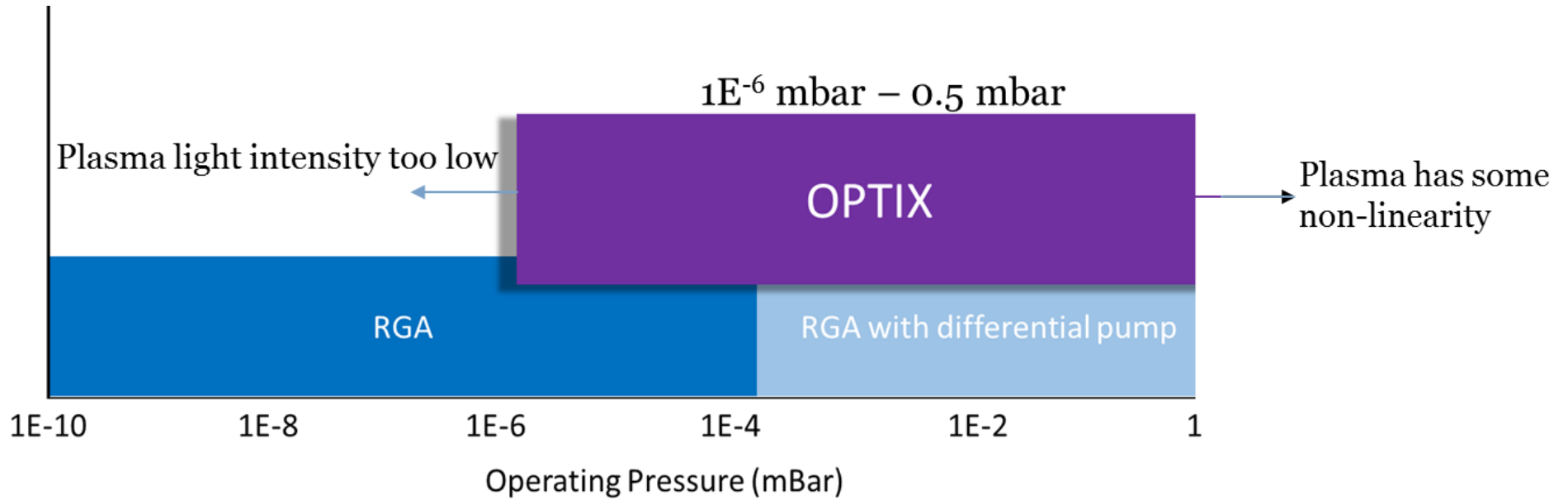
Miniature spectrometer

Spectrum analysis gives species composition and concentration

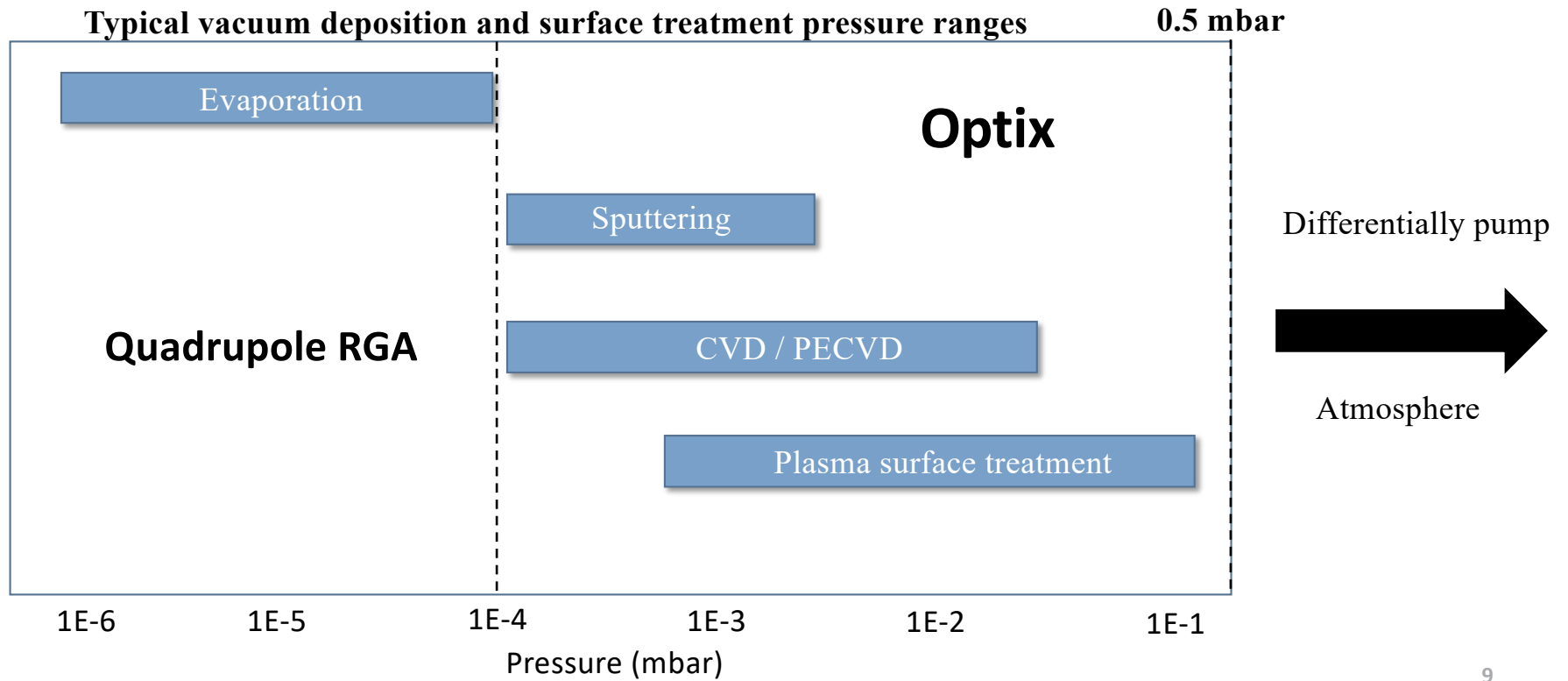


Optix pressure range

Pressure range



Optix pressure range



Maintenance of the plasma generator

Robustness

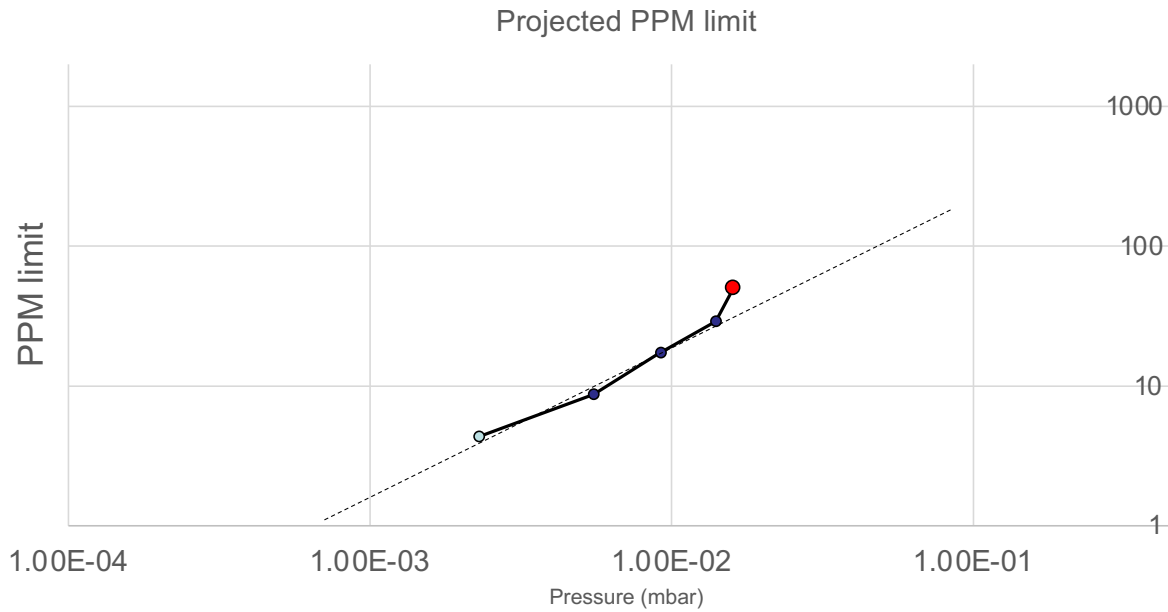
- **No filament**
- **Interlocked via internal pressure gauge**
- **Easily cleanable and replaceable electrodes**



Common questions from RGA users

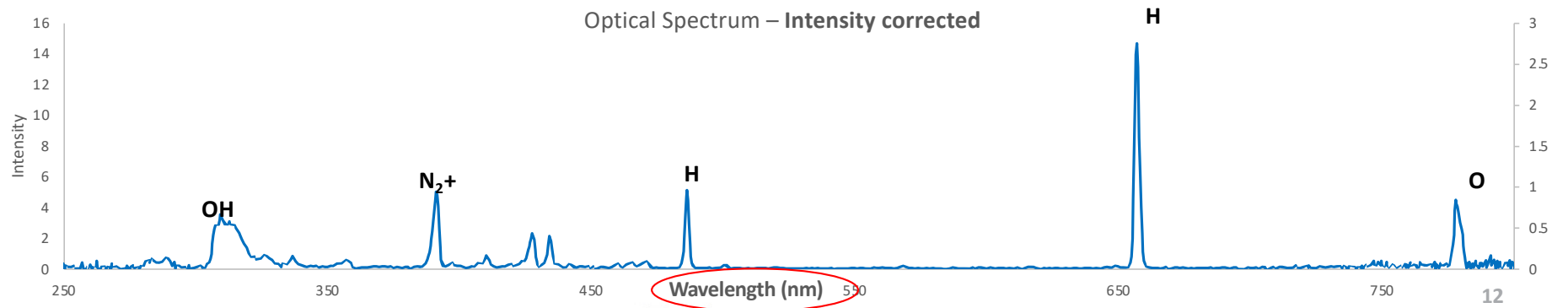
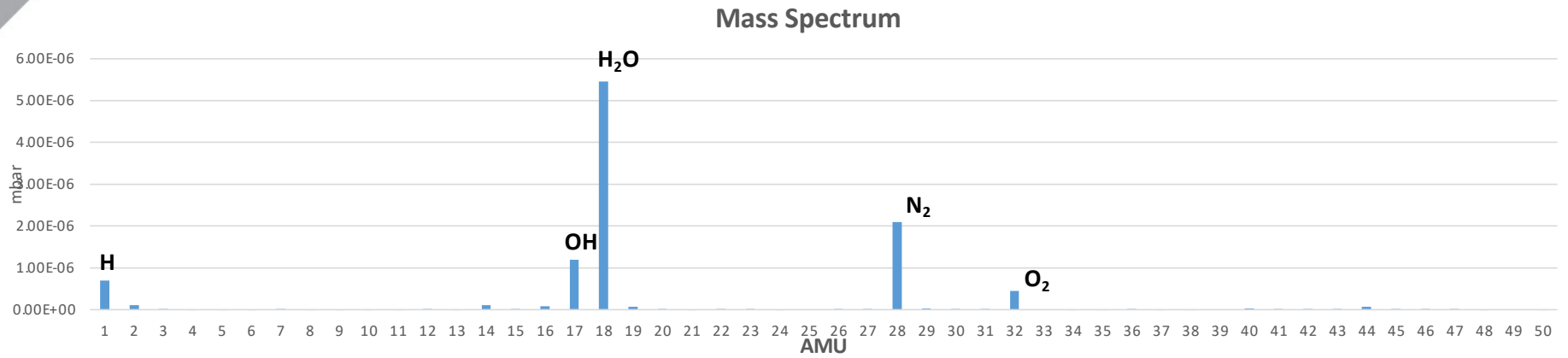
How sensitive is it?

- The sensitivity of Optix is reduced as pressure increases above 1E-4mbar
- Gas dependent – smaller molecules are generally less sensitive



Common questions from RGA users

Mass vs Optical spectrum

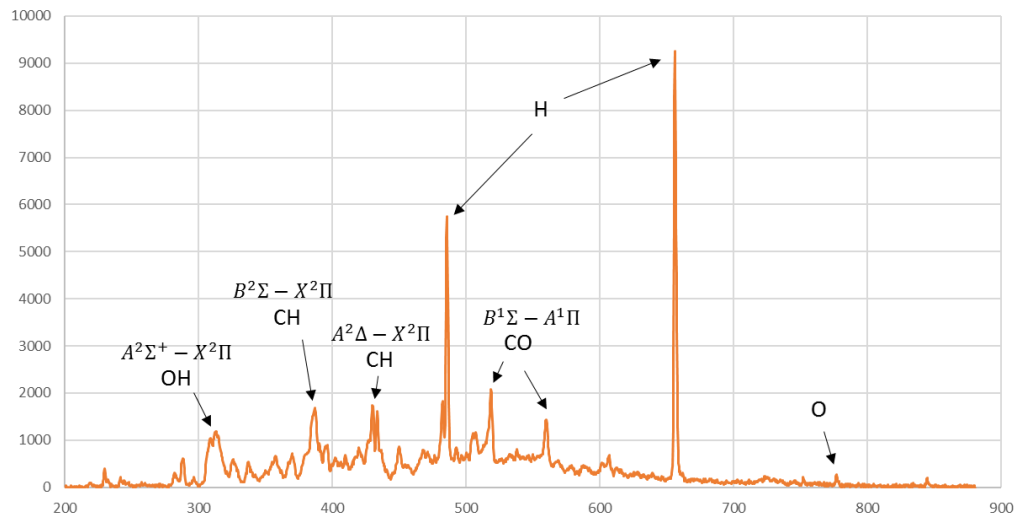


Common questions from RGA users

Equivalent AMU range

- The plasma inside the Optix will break larger molecules into smaller components
- Prominent, defined peaks from component species

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



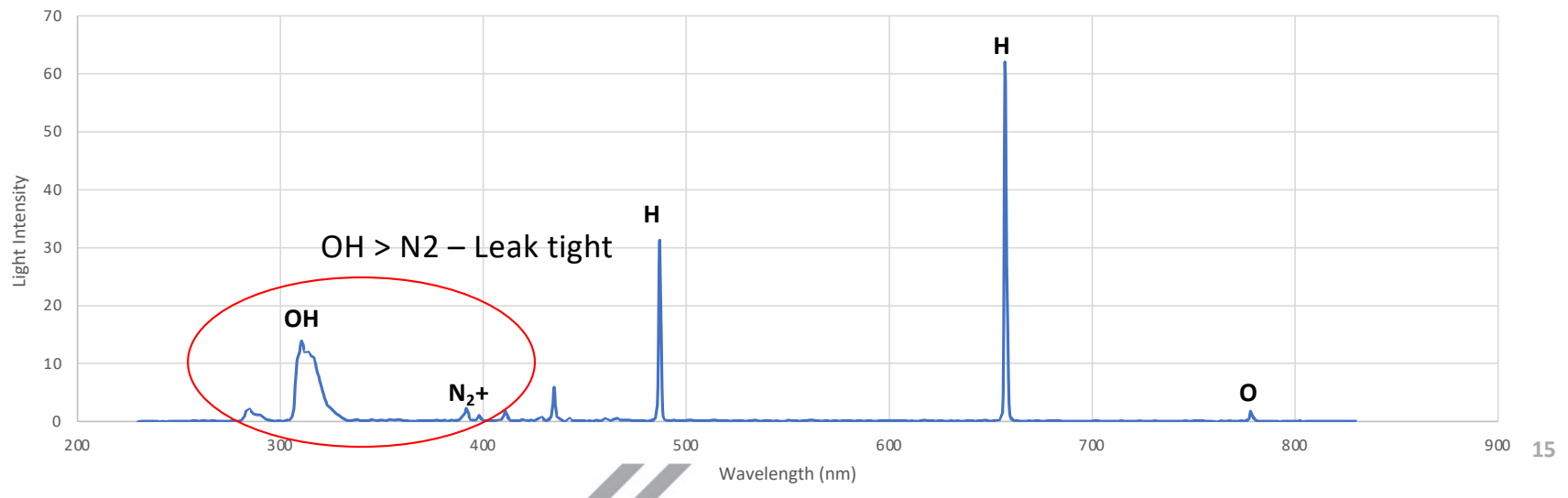
- Can directly observe up to tri-atomic molecules
 - c. 50 AMU
- Can indirectly observe very large molecules via fragmentation
 - c. 100s AMU

Optix vs RGA summary

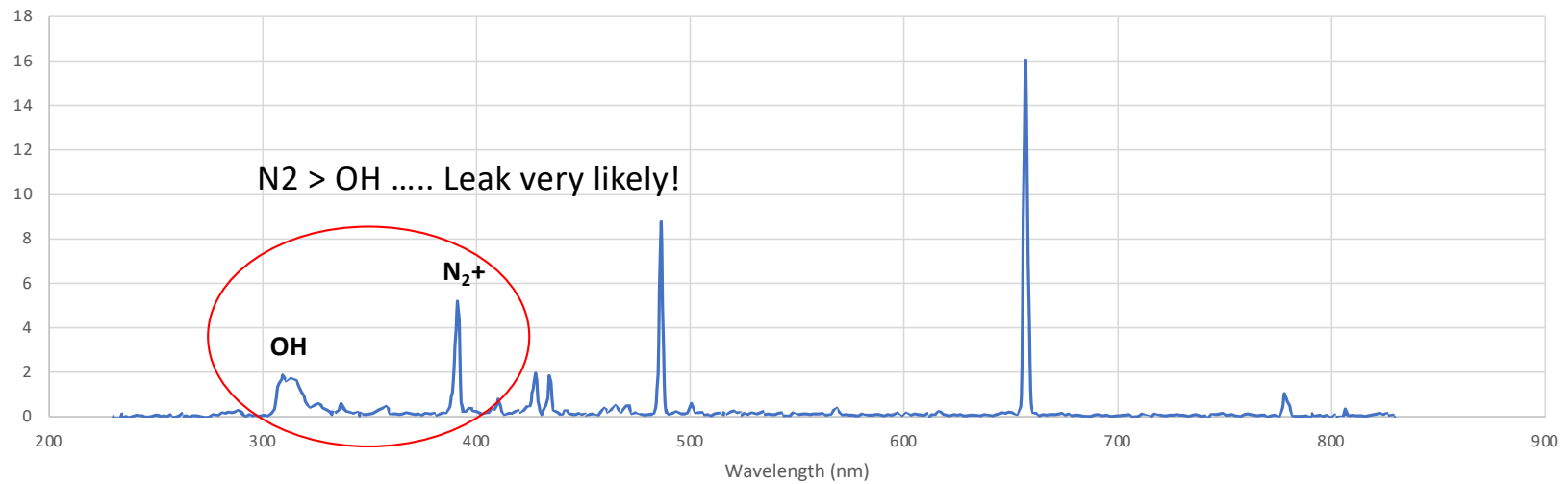
OPTIX – remote plasma gas analysis (RPGA) Optical method	Quadrupole Residual Gas Analyzers (RGAs)
Robust– detector separated from chemicals by optical window	Detector in contact with chemicals – easy to contaminate, hard to clean
No filaments –simple electrode geometry	Filaments and ionizers are consumables
Operates 0.5 to 10^{-7} mbar	Only operates reliably down from 10^{-4} mbar
Direct chamber monitoring – no need for differential pumping unless atmospheric sampling	Higher than 10^{-4} mbar pressure needs differential pumping – loss of sensitivity
FAST – ‘speed of light’, 10-50 msec response	Typically 0.5 to several seconds range
Tolerates volatiles in the vacuum – hydrocarbons, solvents, long chain polymers	Only small amounts of contamination before sensor failure
Wide range of useful software applications available – gas tracking, leak detection, pump-down monitoring, water tracker, end-point detection, multi-mode process tracking	Typically gas tracking & leak detection
Sensor degassing mode – avoid false reading	Yes, but degas can affect filament lifetime

Application examples – Water and Air

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



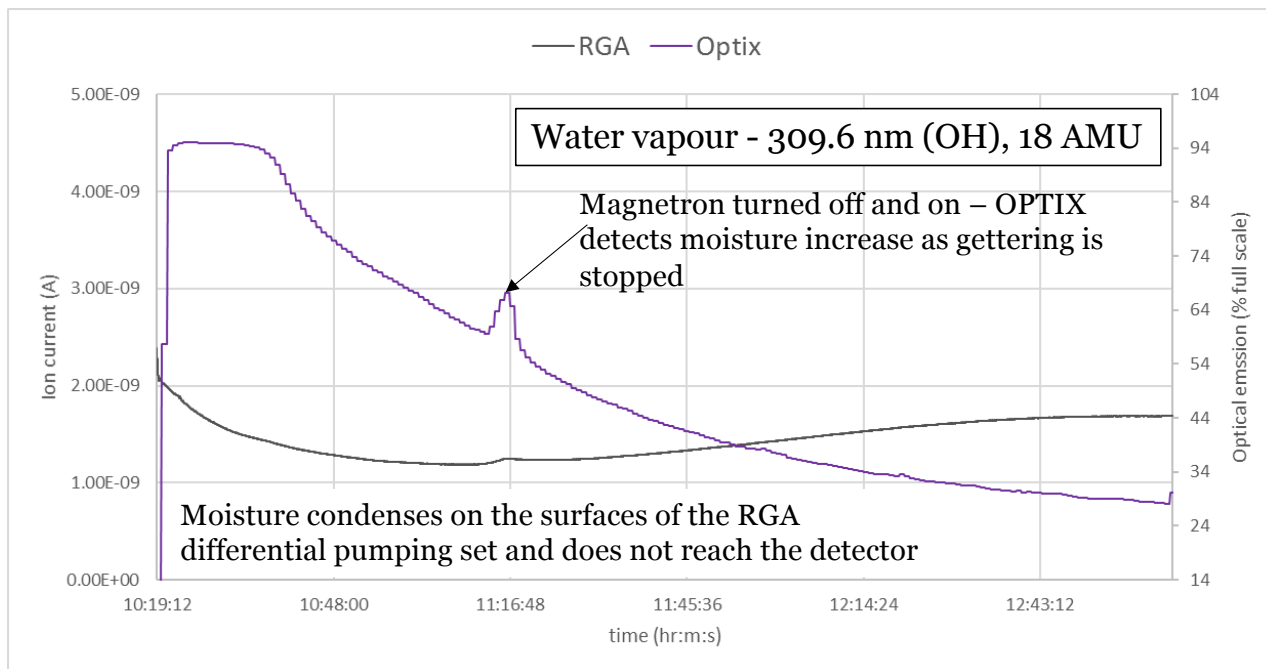
Application examples – Water and Air



Application examples – Water

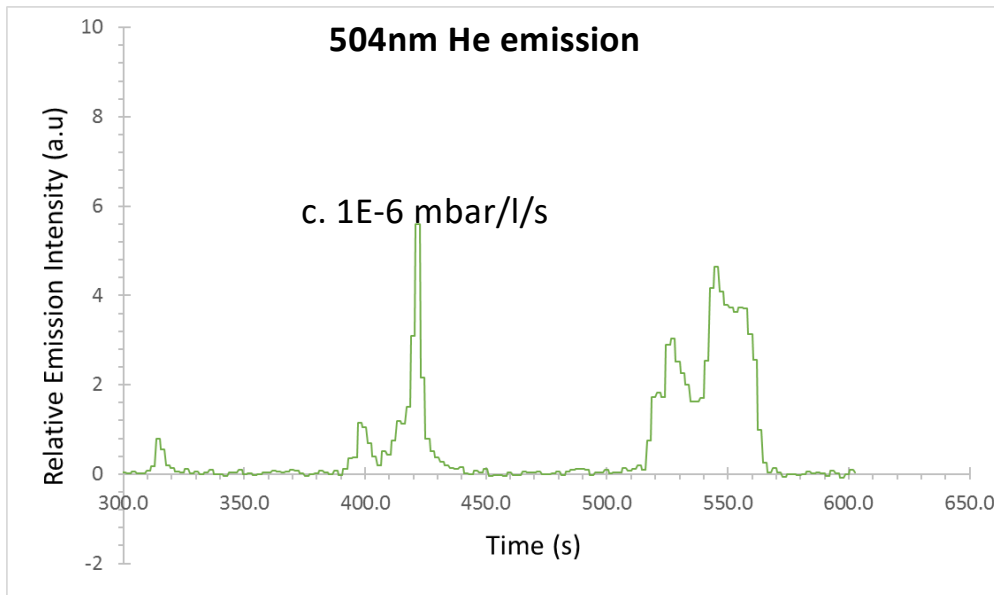
Direct sensing

- Optix can directly sense the vacuum: **Pressure 0.1 mbar**
- **Enhanced sensitivity for condensable species**

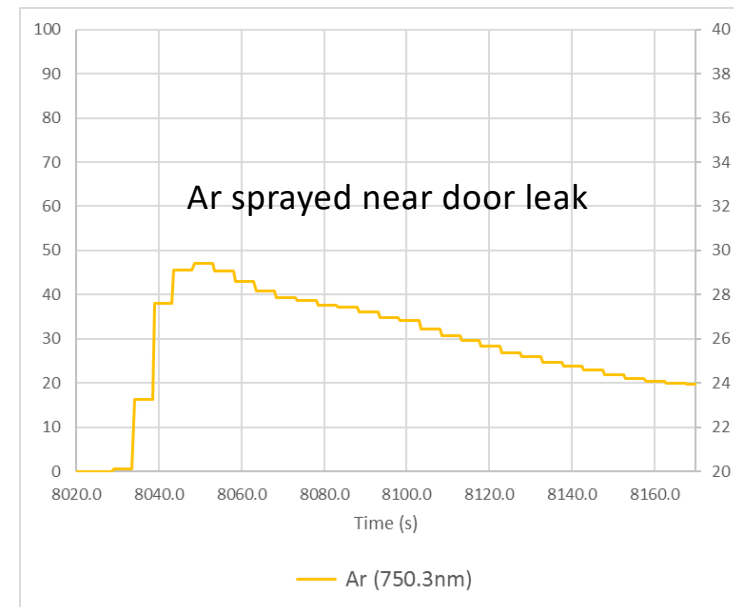


Application examples – He leak detection

- Possible to localize air leaks by monitoring He emission



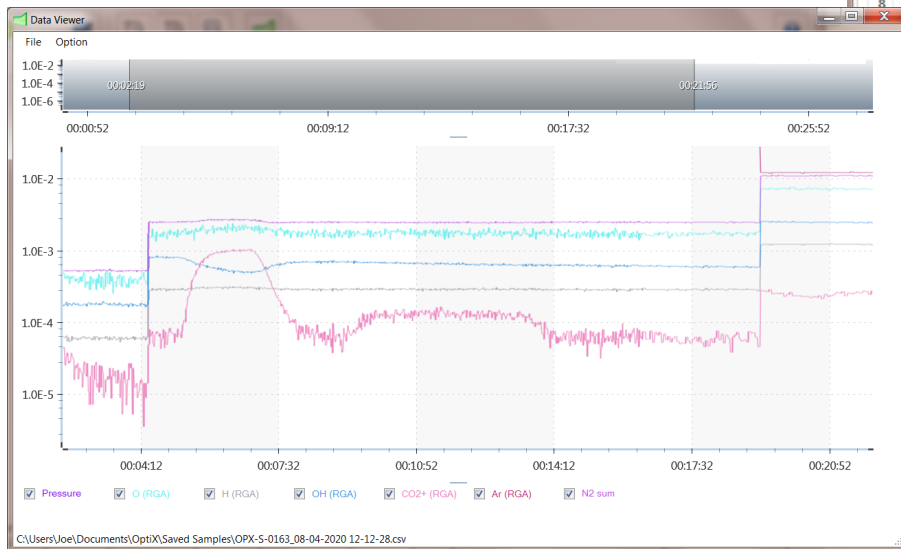
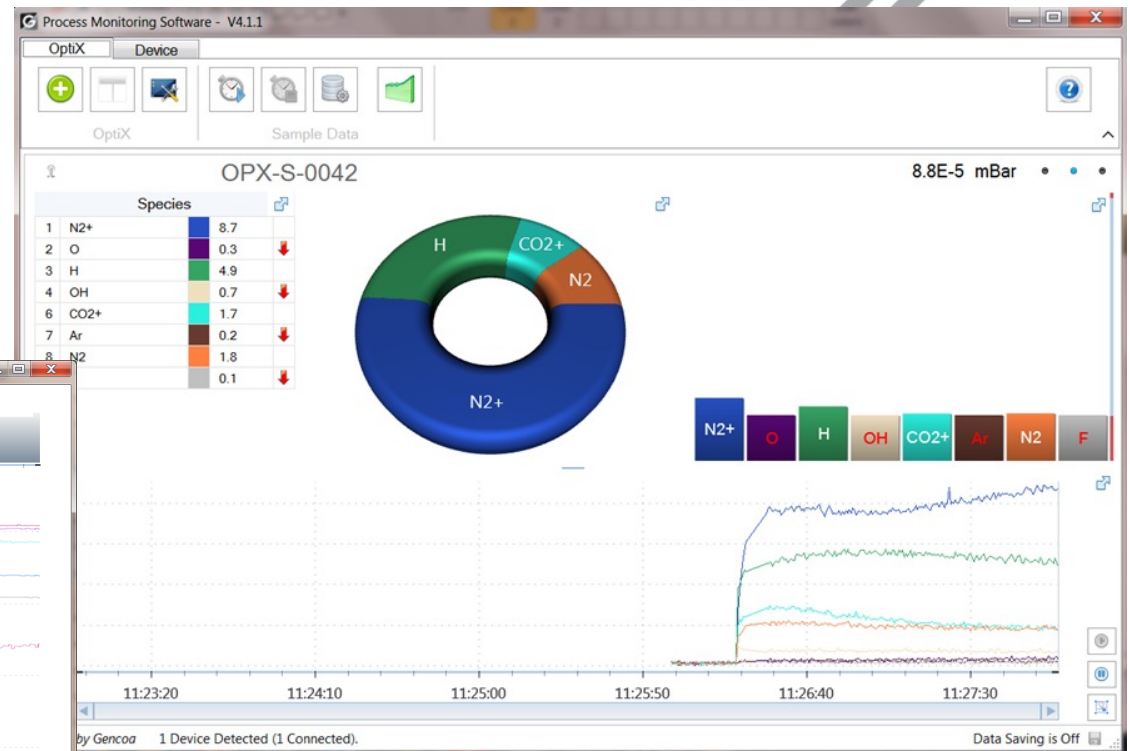
Why not use Argon?



Included software

Ease of use

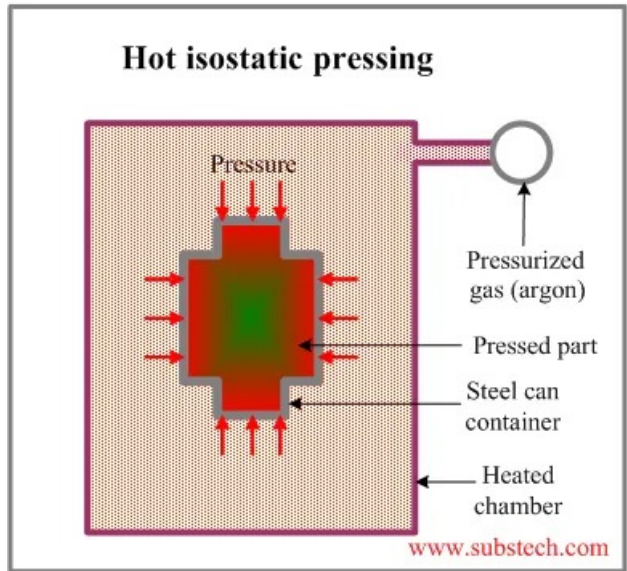
- Optix software is simple and easy to use
- Powerful analysis options (spectrum auto ID)
- Flexible options for integrating with PLCs



Versatility



Optix for Hot Isostatic Pressing



Degassing HIP Canisters

- Degassing is critical and time consuming
- End point is poorly defined
- Accumulated time?
- Pressure?

Optimisation of the HIP degassing process

- Continuous monitoring with Optix allows for endpoint to be determined
 - Significant reduction in degas times



Traceability

Optix data is continuously saved
Any problem with end product can potentially be traced
End users likely to require this capability

Pharmaceutical Drying

- Develop Optix as a single species detector
- Optical filter for H₂O
- Continuous monitoring of drying process
- Real-time data analysis allows for end point definition and automation



Benefits

- Consistent product quality
- Increased efficiency
- Reduced waste
- Improved safety



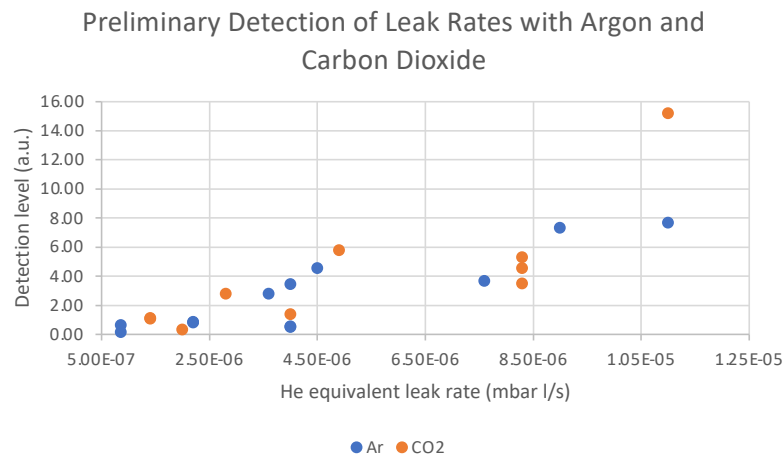


Optix as a dedicated leak checker

Helium Shortage 4.0 has been experienced since the beginning of 2022, resulting in supply allocations from suppliers and sharply higher prices for those who use helium in their business.

An Alternative to Helium

- Leak detection using plasma emission spectroscopy
- Lower cost, more robust, simple maintenance (compared to helium mass spec. leak checkers)

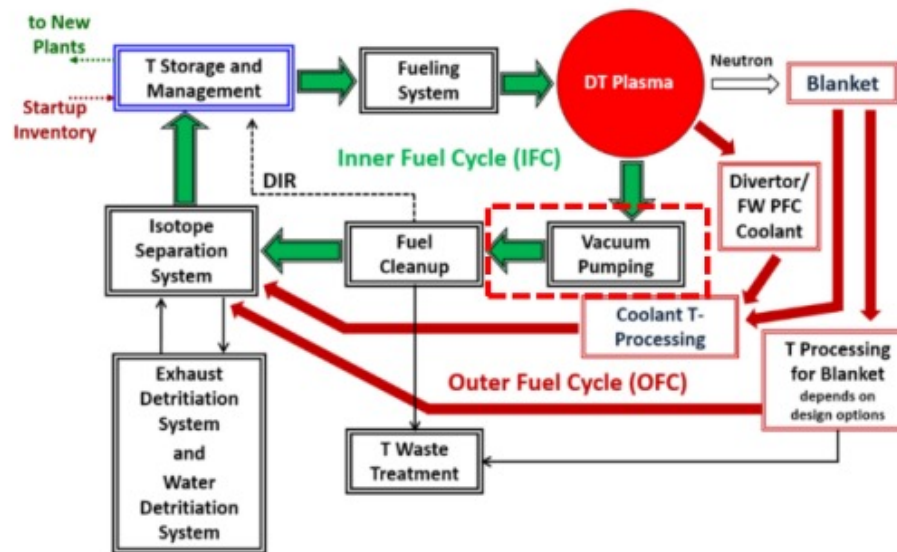


Preliminary detection of leak rates with argon and carbon dioxide. Scroll pump only with vacuum of $\sim 1.5E-3$ mbar in sensor limiting sensitivity.

An Alternative to Helium

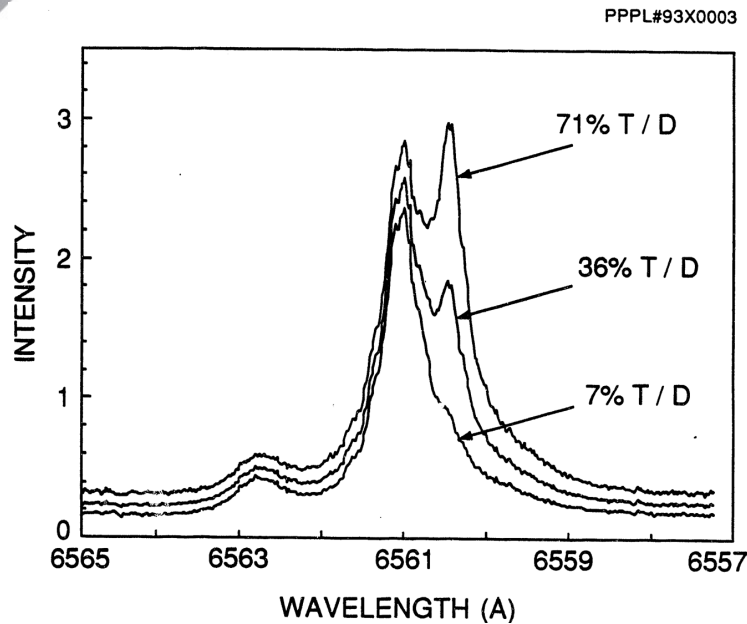
- Steps to improve sensitivity
 - Turbo molecular pumping of sensor to remove argon / carbon dioxide background more effectively
 - Signal processing / optical filtering
 - Alternative sensor technologies
- Target specifications
 - Detection levels equivalent to $<5E-9$ mbar l/s helium
 - Fast component cycle times (<3 minutes)
 - Simple to operate and robust for all industries

Optix for Fusion Applications



Fuelling will be accomplished using injection of cryogenic D-T pellets and will be closed loop: tritium in the plasma exhaust will be combined with tritium from the breeding blanket to feed the pellet system. Therefore, real-time, remote monitoring of the isotopic content of exhaust gases is essential for control of the process.

Optix for Fusion Applications

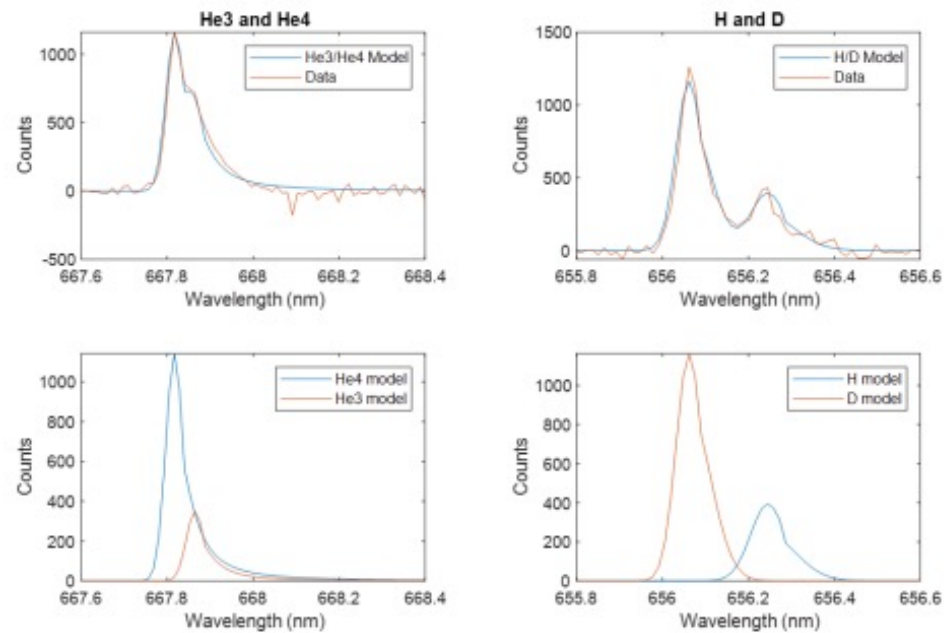


➤ *T/D emission peaks (5)*

- H and He isotopes **do not** have a unique AMU
- H and He isotopes **do have unique light emissions!**
- ✓ The alternative approach we propose is **remote plasma emission monitoring**

Isotope	4He	3He	H	D	T	Ne
Mass	4	3	1	2	3	20
Expected wavelength (nm)	667.815	667.865	656.28	656.11	656.04	667.828

Optix for Fusion Applications



3: User Interface Developed by Gencoa for real time isotopic measurements of $^3\text{He}/^4\text{He}$ and H/D



Thanks for your attention

Any Questions?