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## Process Monitoring, Control, and Leak Detection Using Remote Plasma Optical Emission Spectroscopy

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This talk will outline the principles of remote plasma optical emission spectroscopy and demonstrate its capabilities within various industry sectors. Gencoa's 'Optix' uses a remote plasma spectroscopy concept which generates a small plasma within the sensor head. A built-in spectrometer analyses the plasma, automatically interpreting the light spectrum to provide quantitative measurement of the presence and concentration of gas within the vacuum. The Optix spectral information and sophisticated back-end software creates a range of uses for a wide range of applications, including contaminating processes involving hydrocarbons, solvents and long-chain polymers. The Optix can be applied to many applications and processes including PVD, ALD, Leak detection, heat treatment, hot isostatic pressing and process gas analysis. These will be briefly discussed. The sensor has several distinct advantages over conventional quadrupole mass spectrometers which will also be considered, including operation over a wide pressure range – no requirement for differential pumping; No filaments and low maintenance; Direct monitoring of the vacuum with msec response time; Significantly less expensive than RGA and differential pump combination. Gencoa has utilised the technique to develop a dedicated leak detector which does not use helium. This is targeted at industry sectors where leak rates of  $1 \times 10^{-7}$  mbar.l/s (equivalent helium leak rate) are sufficient as a pass/fail criterion.

<https://www.svc.org>

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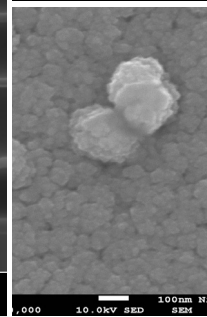
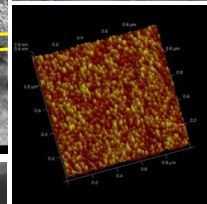
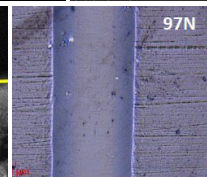
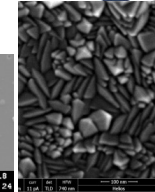
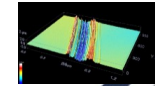
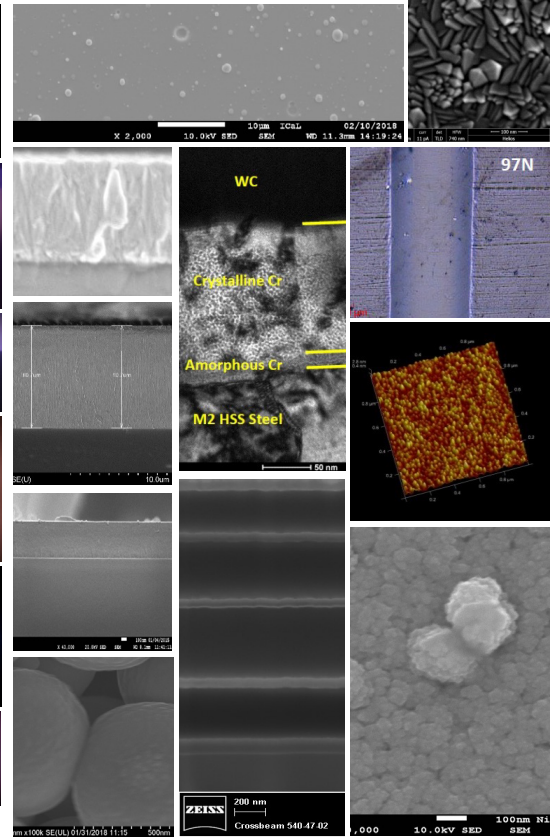
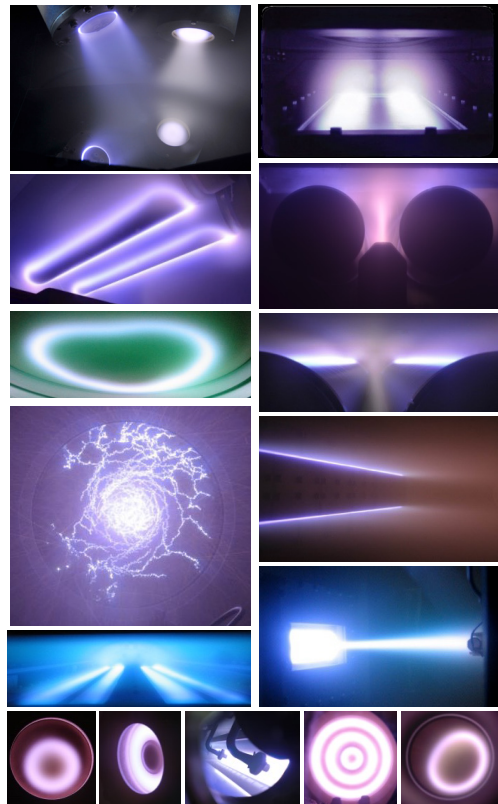
**Erik J Cox, Dermot Monaghan, Joseph Brindley, Benoit Daniel, Victor  
Bellido-Gonzalez,**  
Gencoa Ltd, Liverpool, Merseyside, UK





# 27 Years of Products and Technology from Gencoa

Rotatable & Planar Magnetron Sputter Cathodes • Retrofit magnetic packs • Plasma Treaters • Speedflo Reactive Gas Controllers • IM Ion Sources & power supplies • Arc MAX sources & power supplies • Active Anodes and Gas Delivery Bars • OPTIX Gas and Chemical Sensing • S and Se Sensor • PEC Pulsed Effusion Cell • V<sup>+</sup>DLC - Transparent DLC • IC Nano antimicrobial layer technology • Process implementation & tuning •





## 'Optix' – A remote plasma optical emission spectrometer

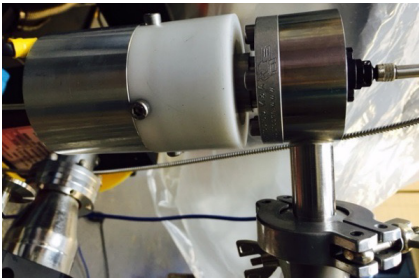


PTIX

## Optix history

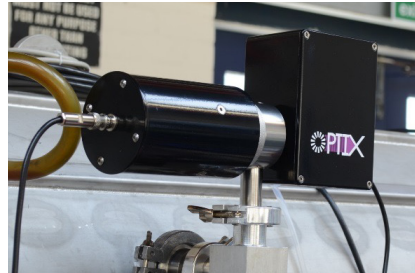
- Introduction to the market in Feb 2017
- Had to trailblaze as there were no serious similar products
- Over 400 global sales to date (US, Europe, China, Japan, Korea)
- Customers including Apple, Samsung, Huawei, CERN, GKN, Lawrence Livermore National Laboratory

### Development (2015 – 2017)



2015

### Market introduction (2017)



2017

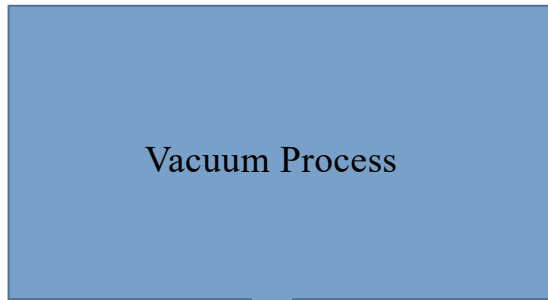
### “Version 2.0” (2020)



2020



# RPEM principal



Remote plasma generator

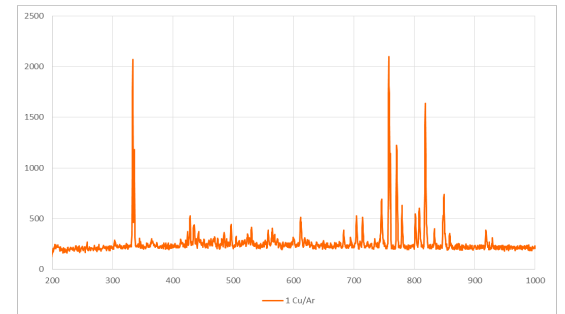


Plasma



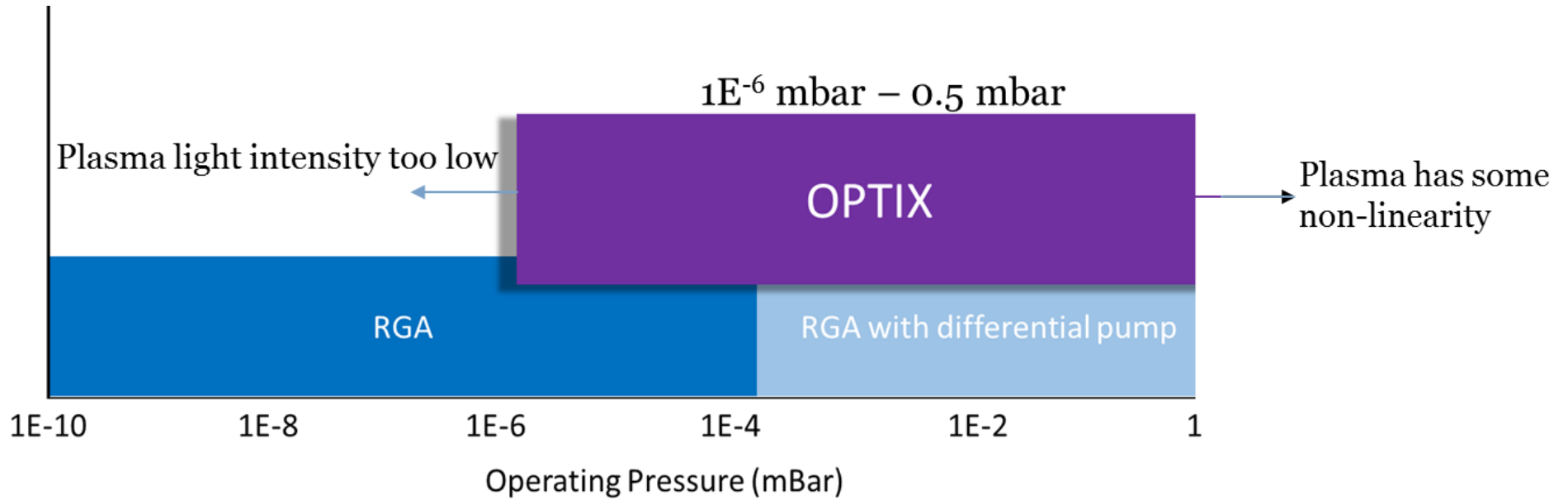
Miniature spectrometer

Spectrum analysis gives species composition and concentration

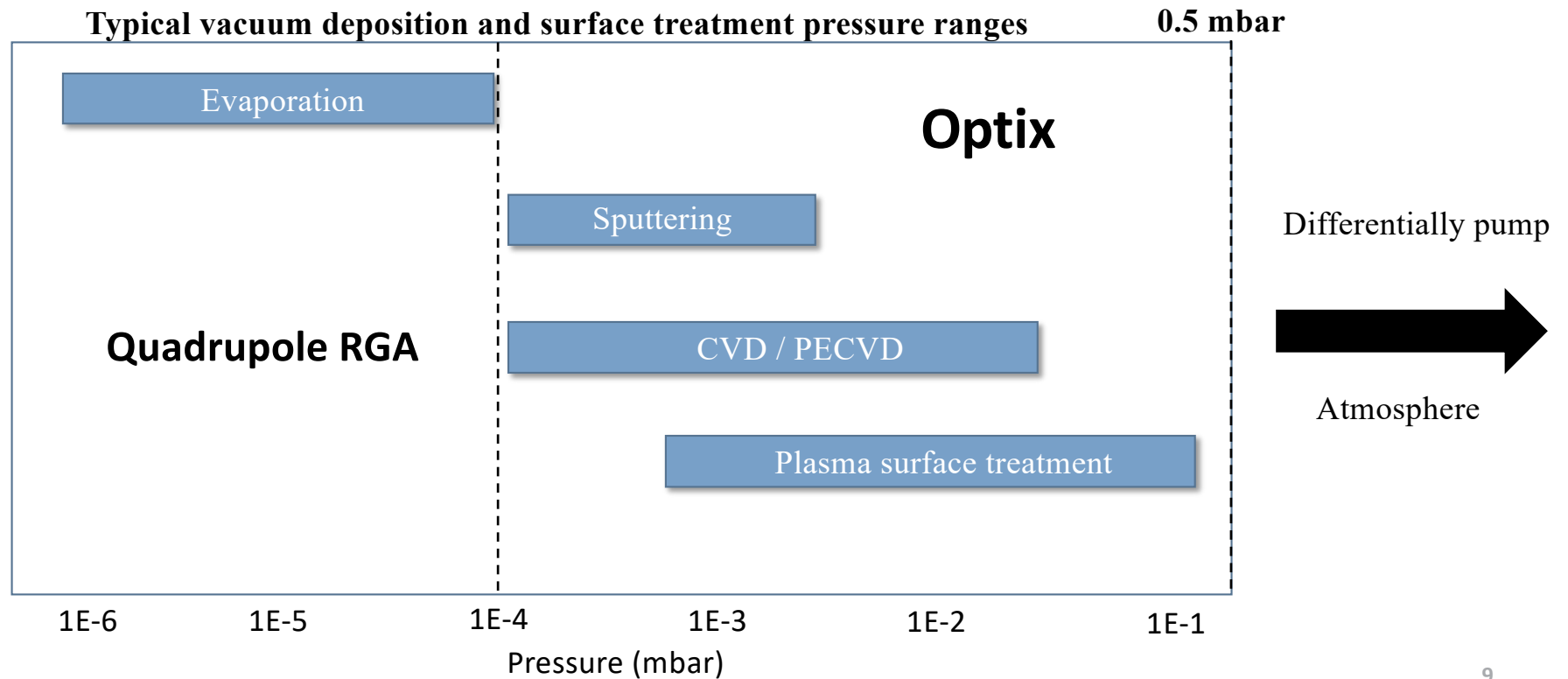


# Optix pressure range

## Pressure range



# Optix pressure range



## Maintenance of the plasma generator

### Robustness

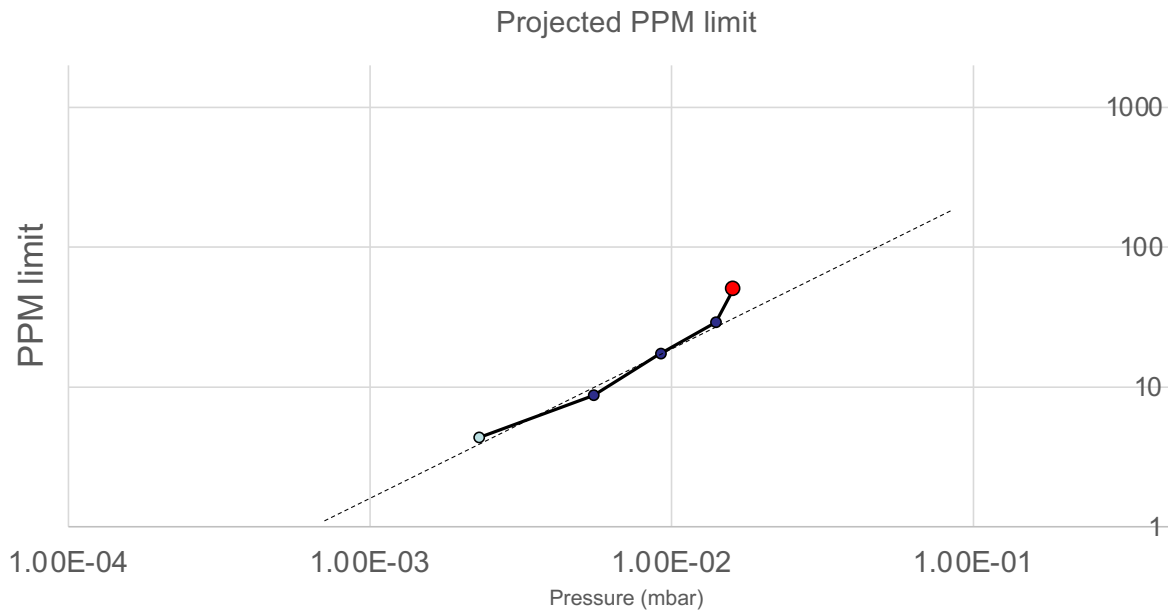
- **No filament**
- **Interlocked via internal pressure gauge**
- **Easily cleanable and replaceable electrodes**



# Common questions from RGA users

## How sensitive is it?

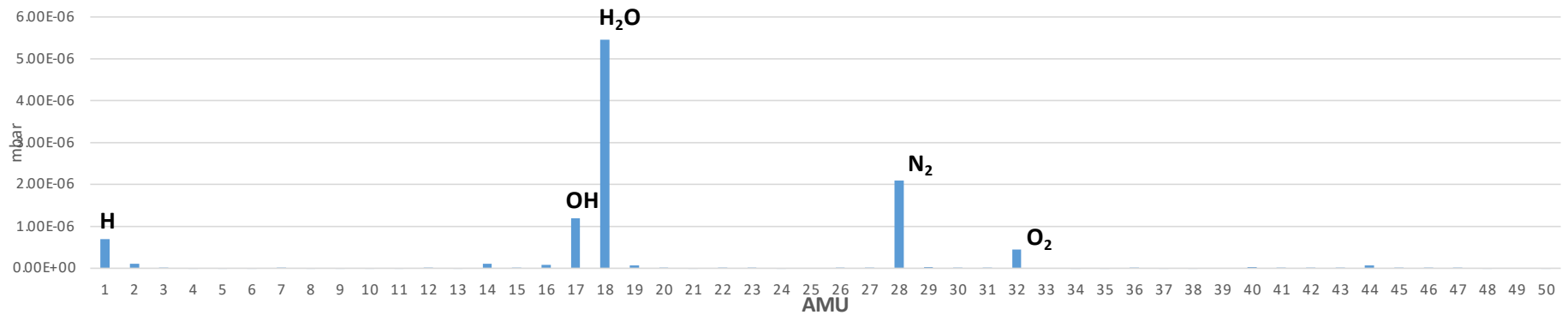
- The sensitivity of Optix is reduced as pressure increases above 1E-4mbar
- Gas dependent – smaller molecules are generally less sensitive



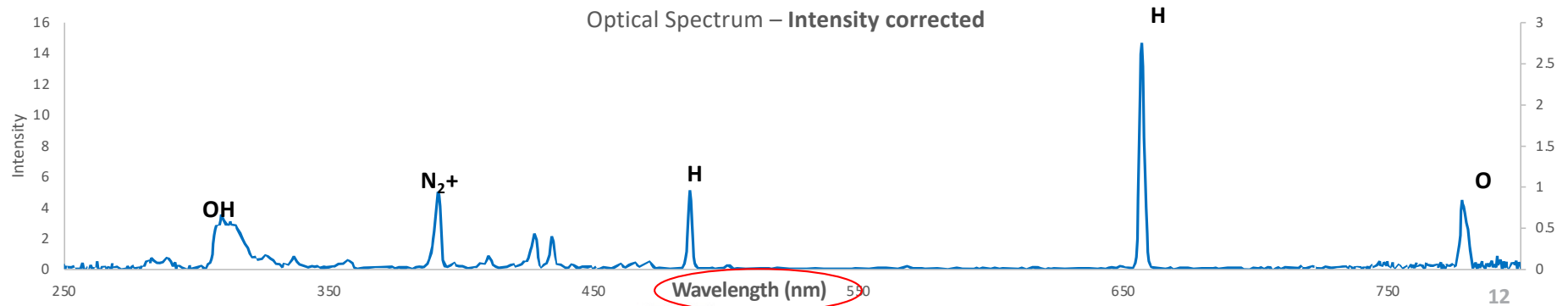
# Common questions from RGA users

## Mass vs Optical spectrum

Mass Spectrum



Optical Spectrum – Intensity corrected

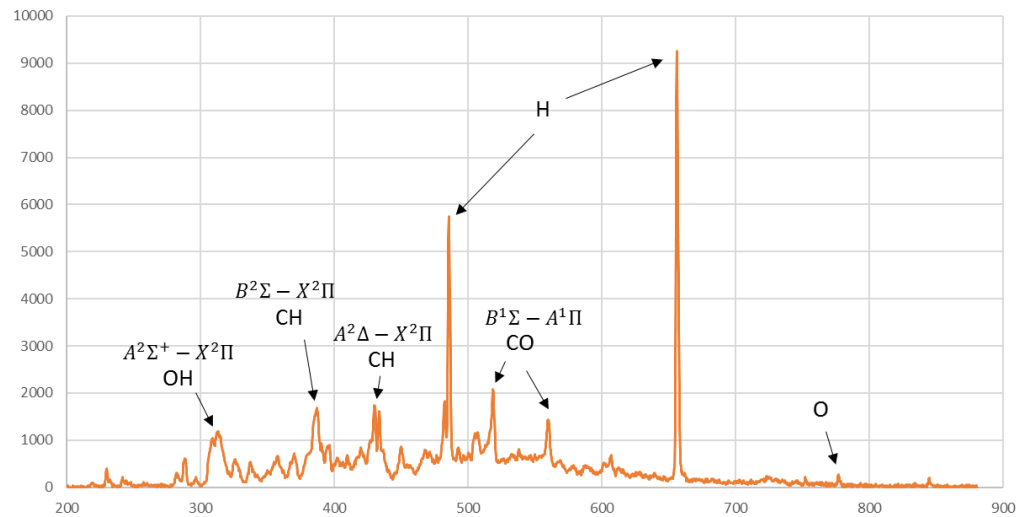


# Common questions from RGA users

## Equivalent AMU range

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

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



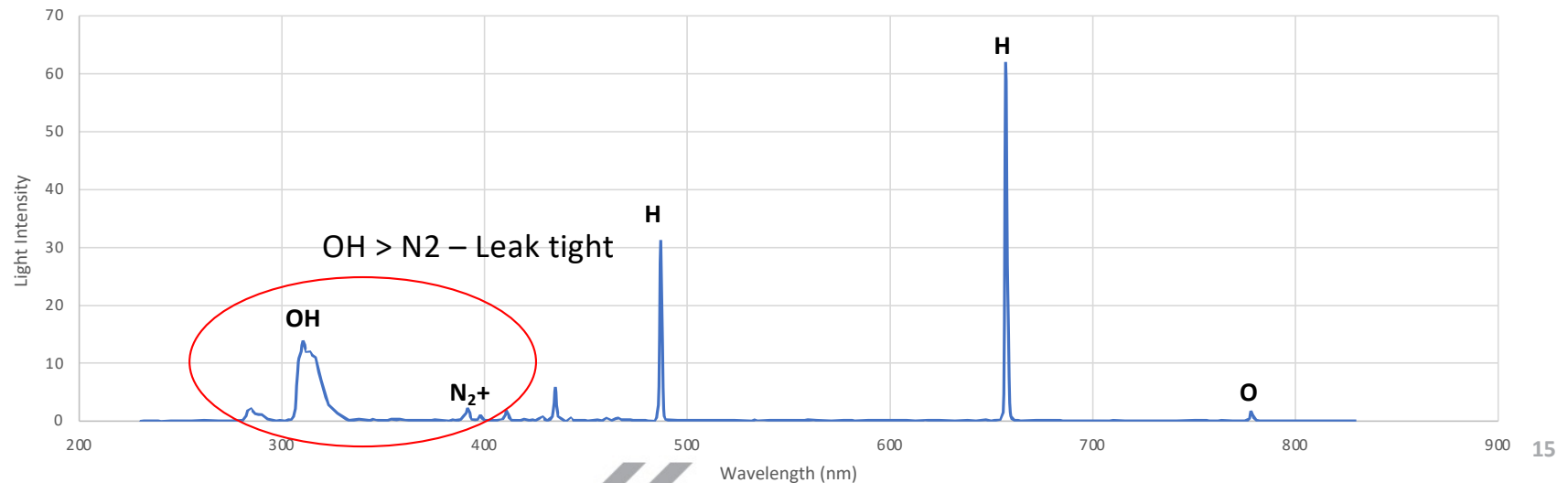
- Can directly observe up to tri-atomic molecules
  - c. 50 AMU
- Can indirectly observe very large molecules via fragmentation
  - c. 100s AMU

## Optix vs RGA summary

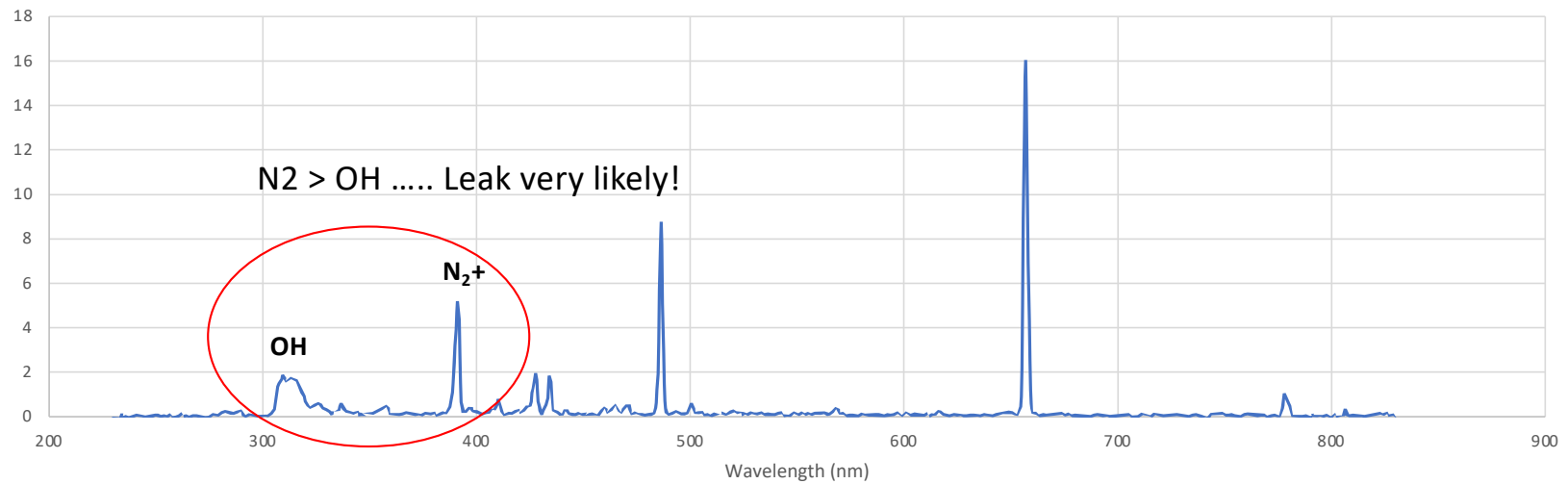
OPTIX – remote plasma gas analysis (RPGA) Optical method	Quadrupole Residual Gas Analyzers (RGAs)
Robust– detector separated from chemicals by optical window	Detector in contact with chemicals – easy to contaminate, hard to clean
No filaments –simple electrode geometry	Filaments and ionizers are consumables
Operates 0.5 to 10 <sup>-7</sup> mbar	Only operates reliably down from 10 <sup>-4</sup> mbar
Direct chamber monitoring – no need for differential pumping unless atmospheric sampling	Higher than 10 <sup>-4</sup> 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 N<sub>2</sub> in relation to water vapour
- General rule that < 1E-2 mbar a leak tight system should not have significant N<sub>2</sub> present



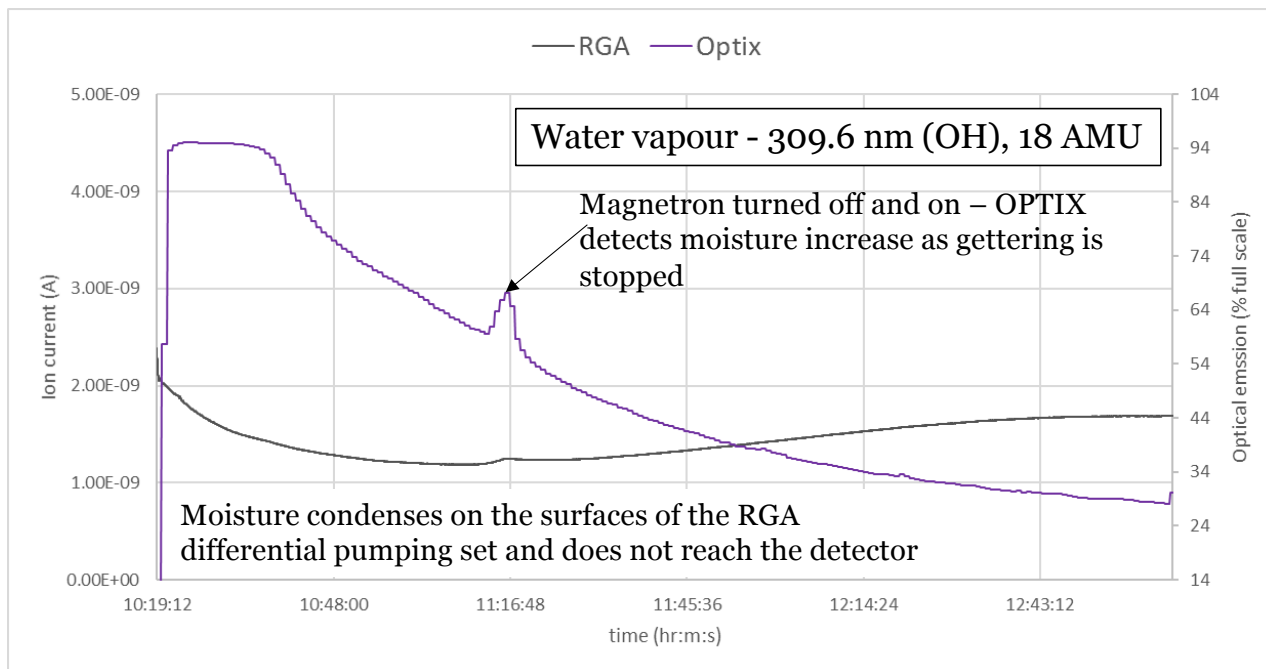
# Application examples – Water and Air



## Application examples – Water

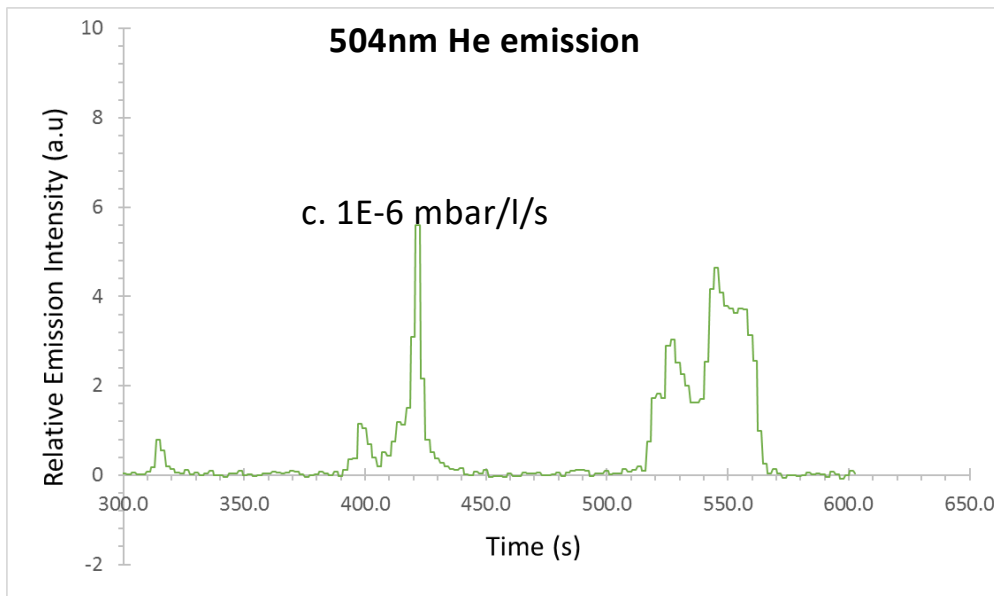
### Direct sensing

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

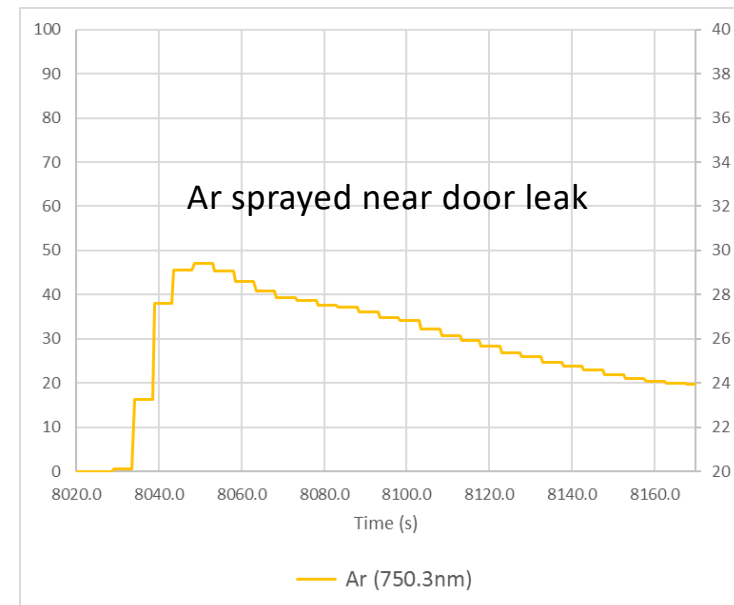


# Application examples – He leak detection

- Possible to localise air leaks by monitoring He emission



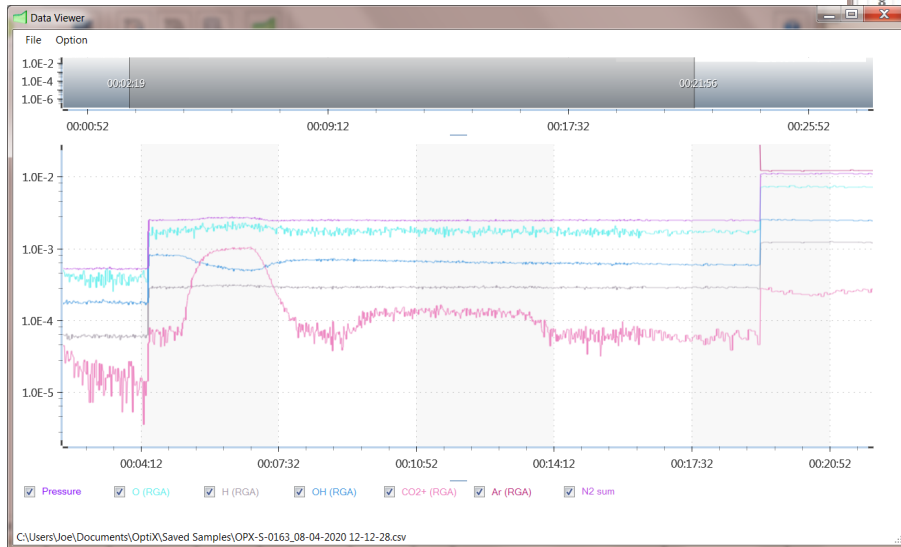
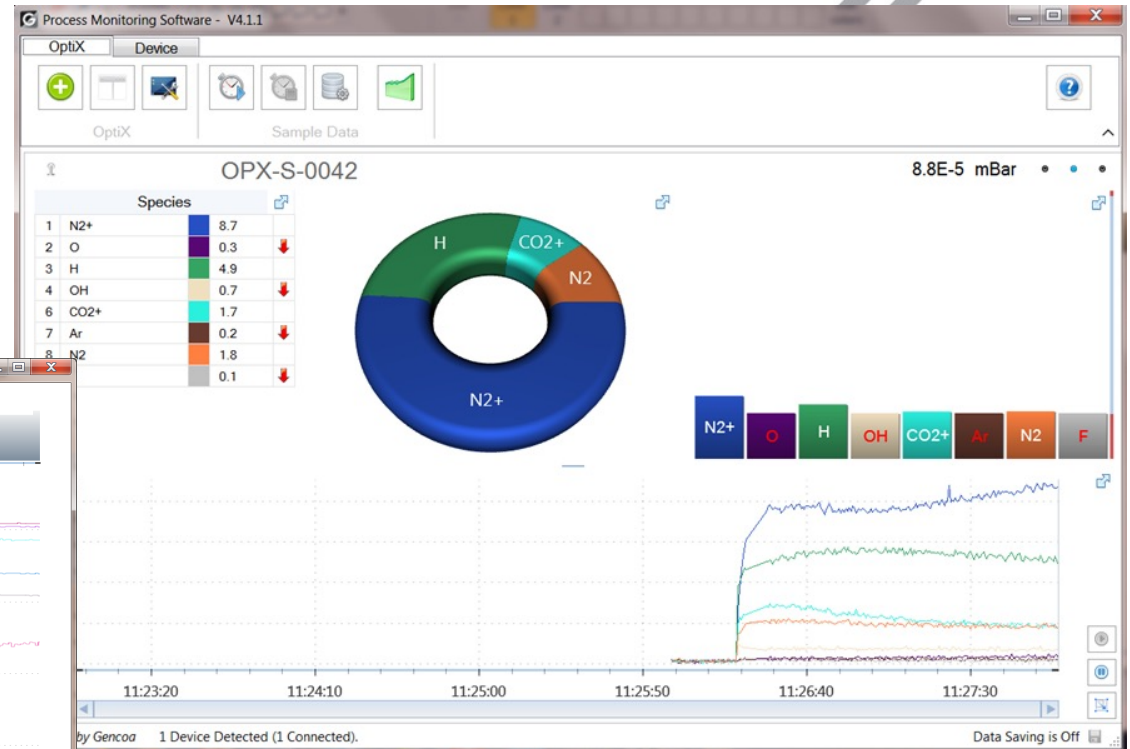
## Why not use Argon?



# Included software

## Ease of use

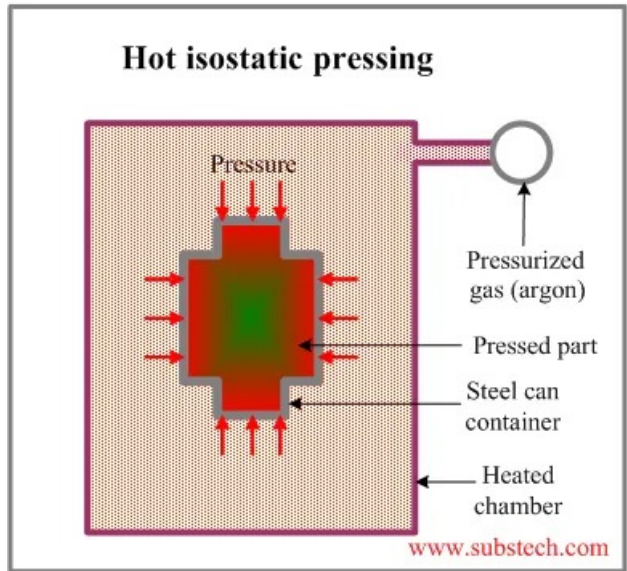
- Optix software is simple and easy to use
- Powerful analysis options (spectrum auto ID)
- Flexible options for integrating with PLCs



# 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 continuously saved

Any problem with end product can potentially be traced

End users likely to require

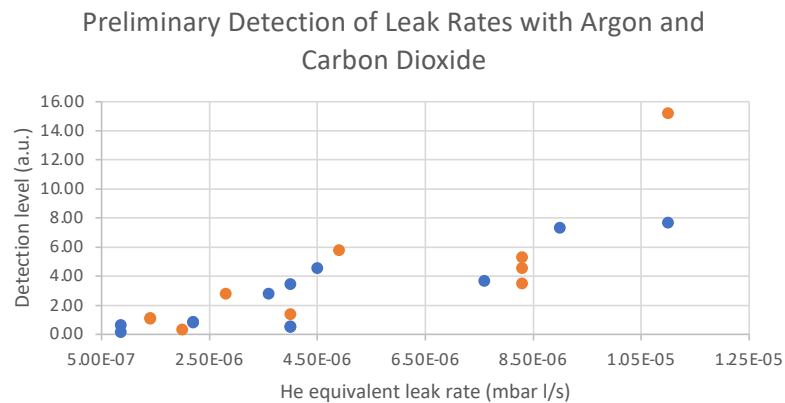


# Optix as a dedicated leak checker

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)

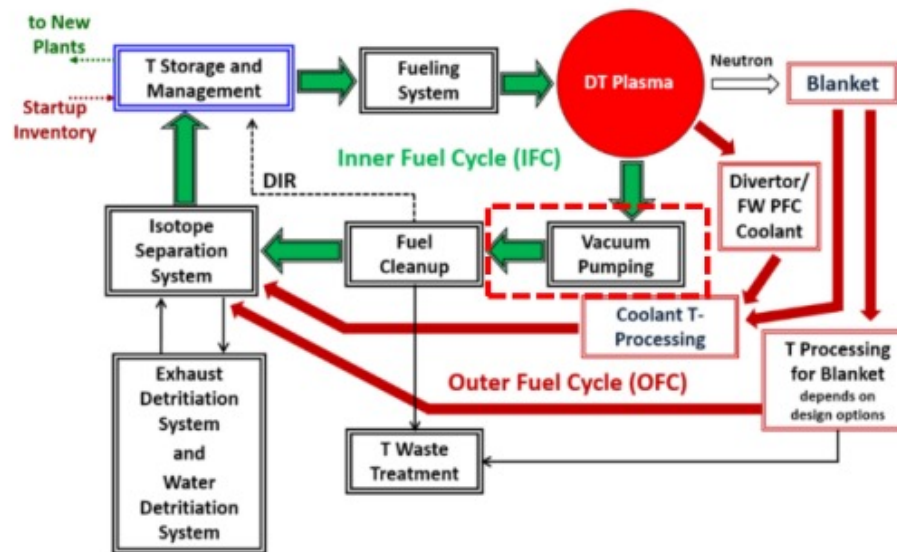


Preliminary detection of leak rates with argon and carbon dioxide. Scroll pump only with vacuum of  $\sim 1.5\text{E-}3\text{mbar}$  in sensor limiting sensitivity.

## An Alternative to Helium

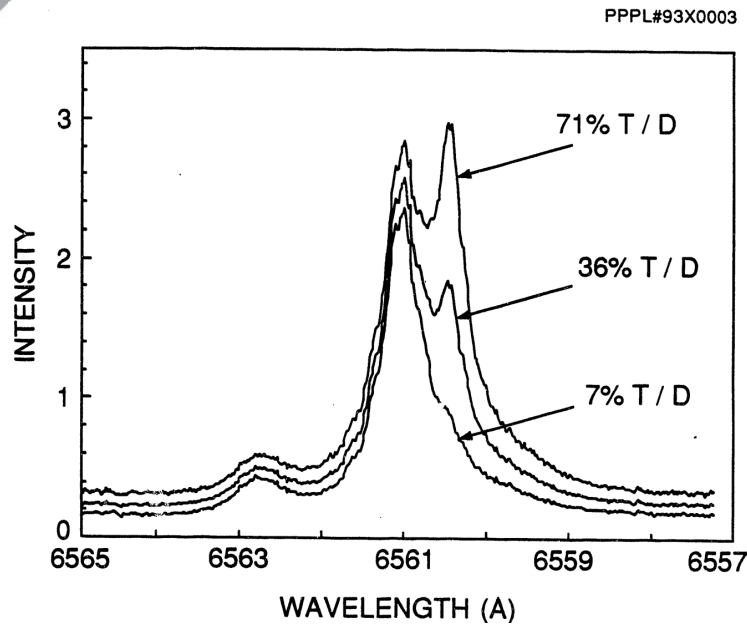
- Steps to improve sensitivity
  - Turbo molecular pumping of sensor to remove argon / carbon dioxide background more effectively
  - Signal processing / optical filtering
  - Alternative sensor technologies
- Target specifications
  - Detection levels equivalent to  $<5E-9$  mbar l/s helium
  - Fast 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

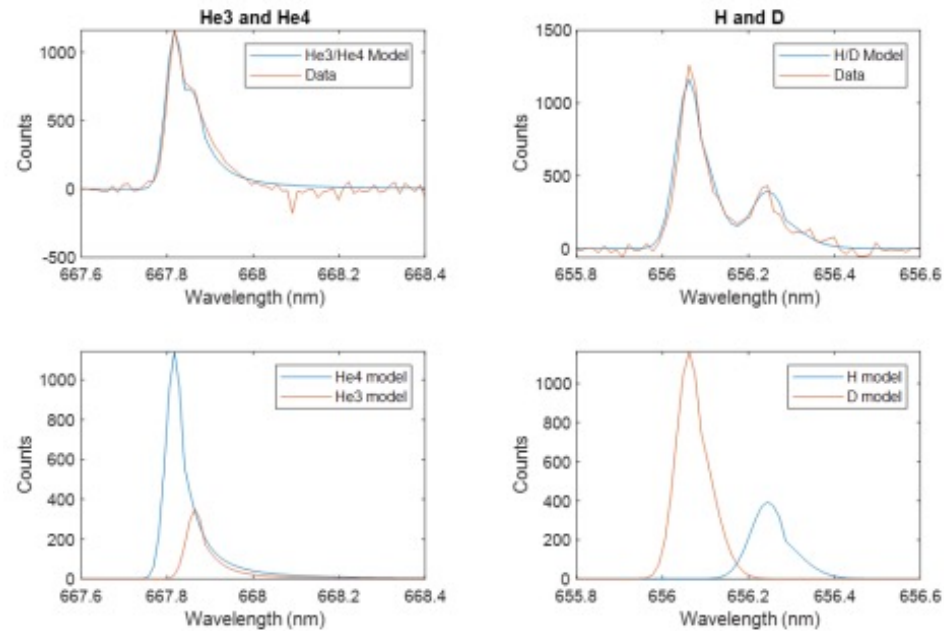


➤ *T/D emission peaks (5)*

- 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

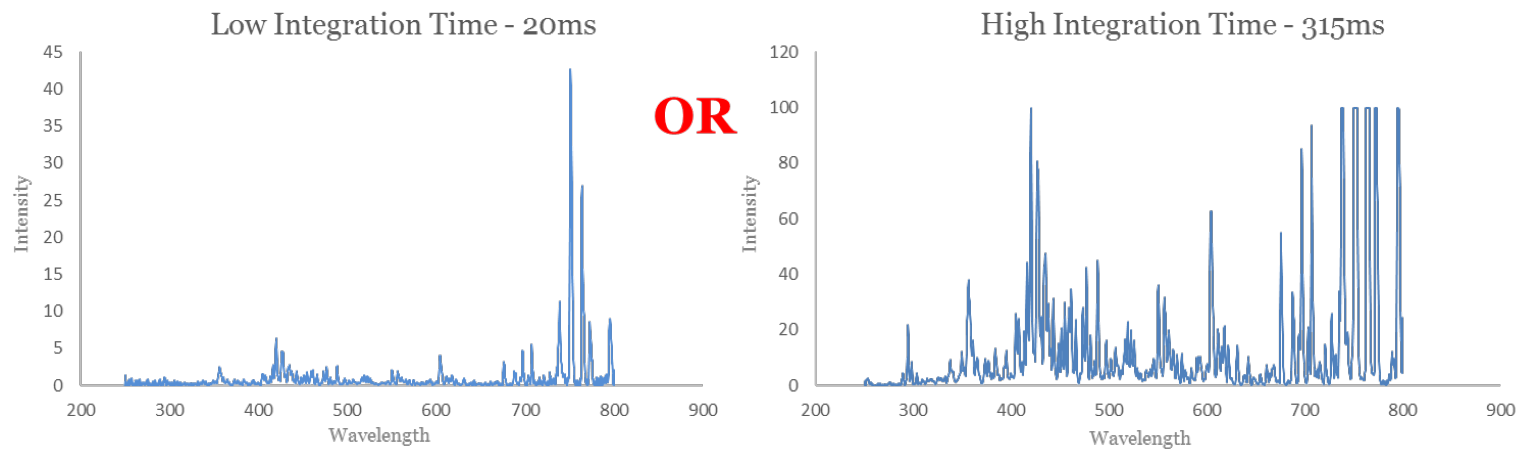
# Optix for Fusion Applications



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

# Method to increase sensitivity of data from plasma spectral information

- Aim: address the lack of dynamic range\*
- Integration time is proportional to the sensitivity
- Unable to measure very large signals and very small signals simultaneously



\*Dynamic range is the ratio between the maximum and minimum signal intensities that a spectrometer can detect.

# HDR Mode

## A New Feature of OPTIX

In order to obtain the best data we use HDR (High Dynamic Range) mode.

- Use a mixture of integration times, ranging from small to high.
- Obtain information from the spectra with different integration times.
- Exploit the linear relationship between the signal and the integration time to calculate the peak height beyond the saturation point.

Similar to HDR photography where a photo is created by capturing multiple images with different exposures at the same time.



Image: [https://vmi.tv/blog/learn-help/hdr\\_reality\\_and\\_monitoring-a\\_dops\\_perspective/](https://vmi.tv/blog/learn-help/hdr_reality_and_monitoring-a_dops_perspective/)

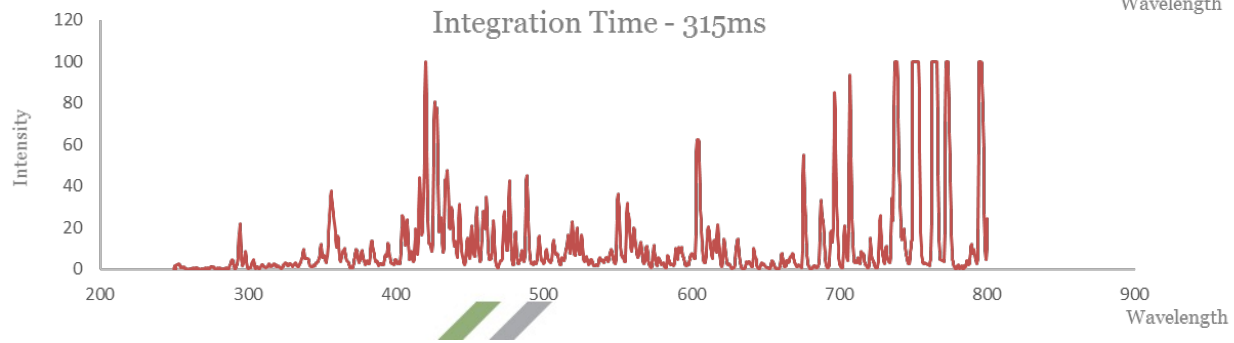
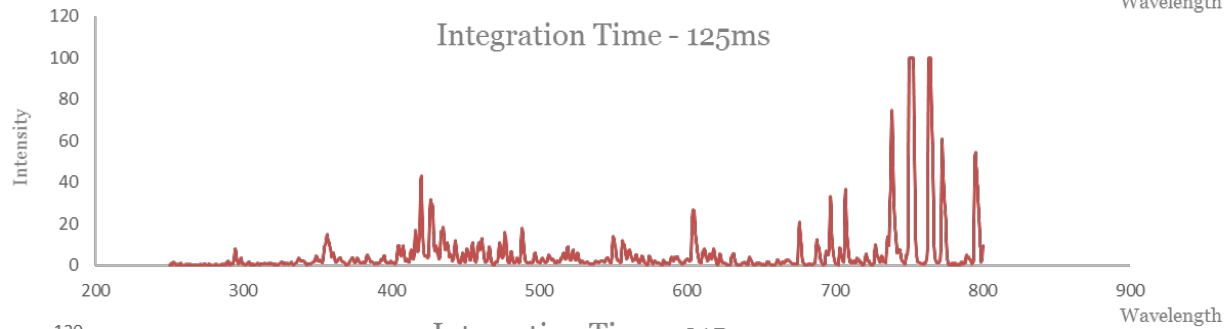
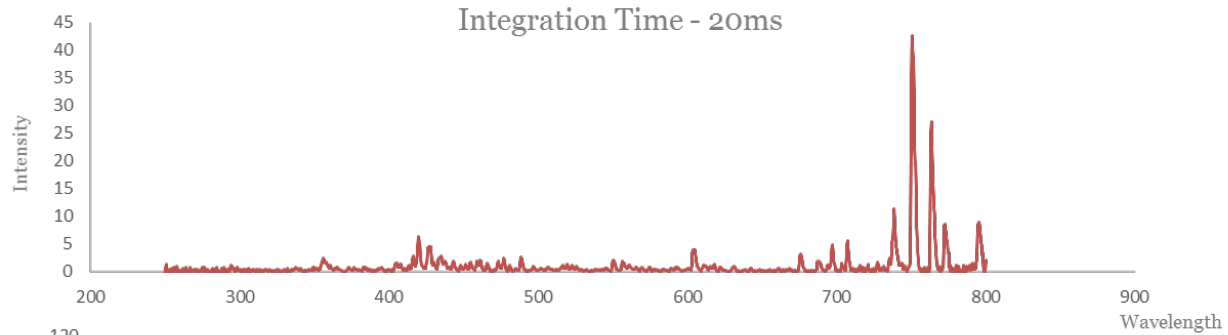
# HDR Mode



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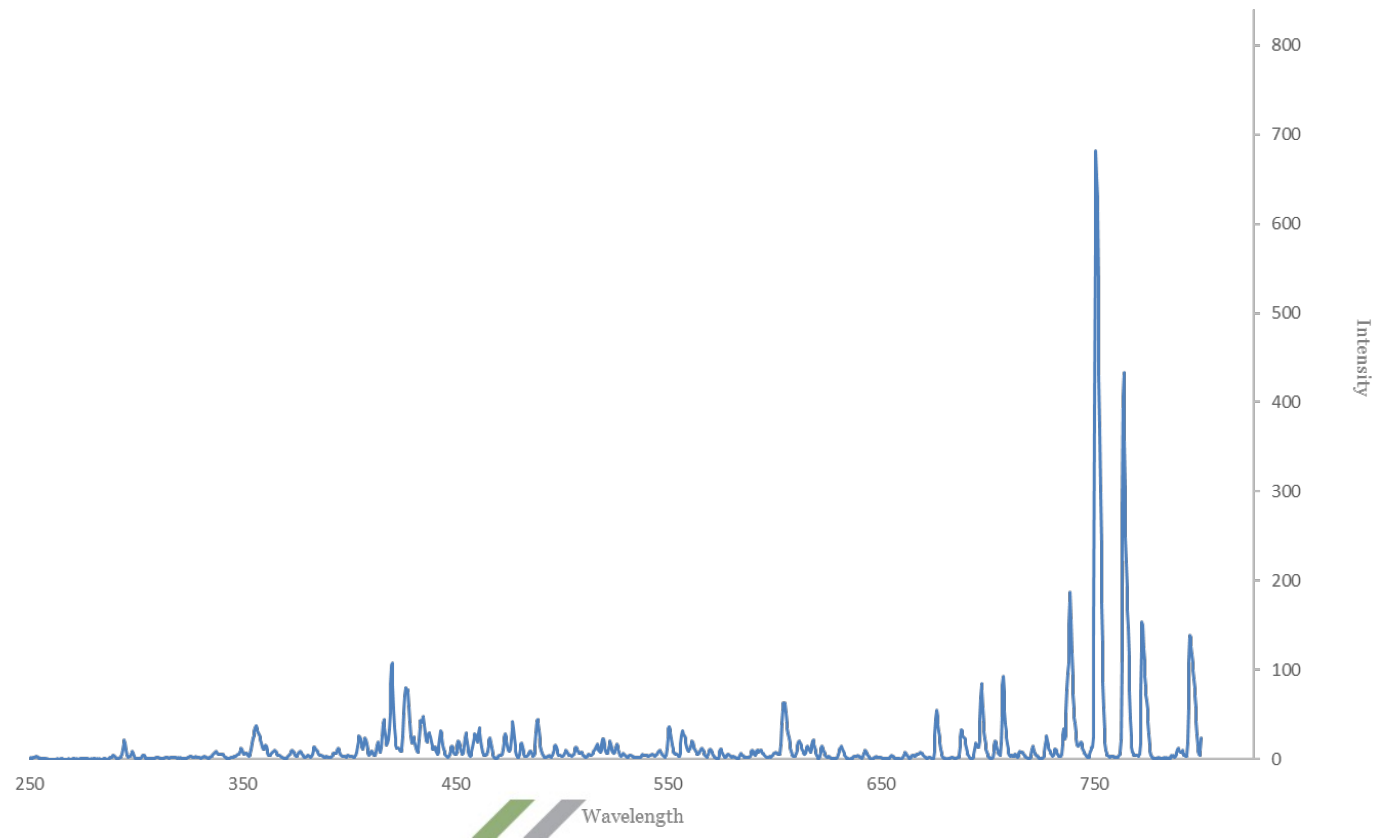
# HDR Mode





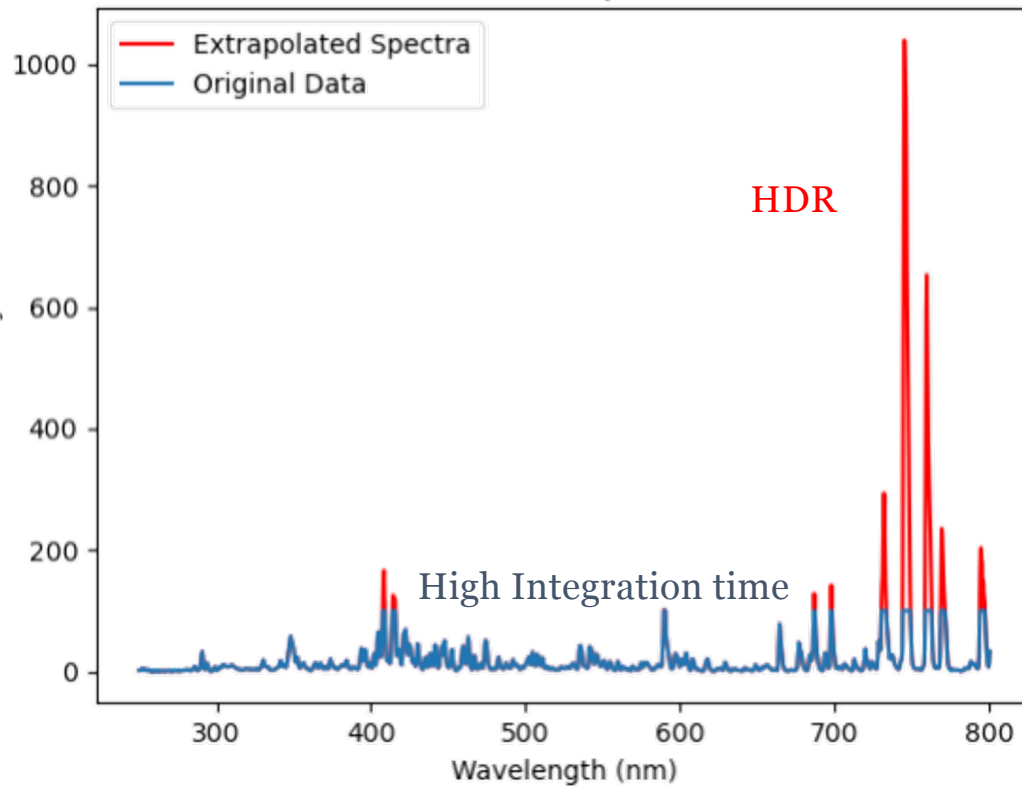
# HDR Spectrum

The HDR spectrum is a combination of the spectra taken at multiple integration times.



# HDR Mode vs High Integration

Artificial Spectra | t=500.0ms



# HDR Mode vs Low Integration

