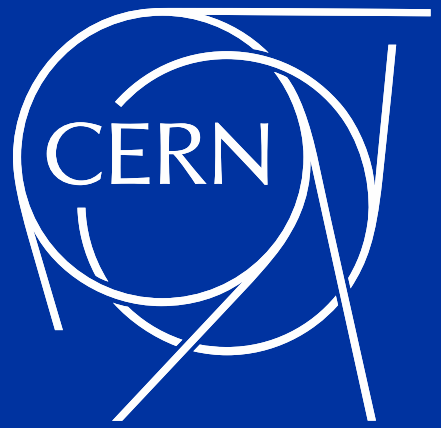


Vacuum characterization capabilities

Ivo Wevers

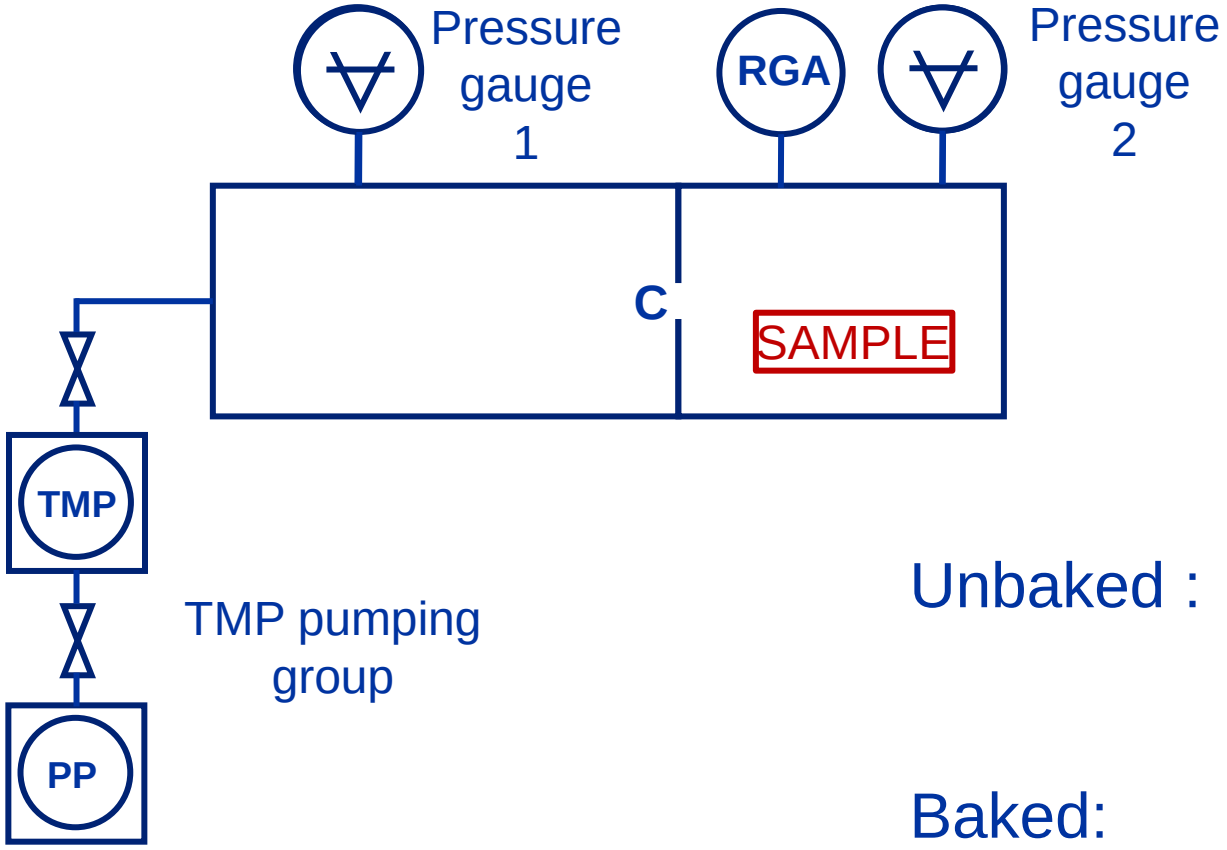
Physics Beyond Colliders workshop

April 2022

Content

- Outgassing:
  - ❖ Throughput method
  - ❖ Coupled method
- Thermal Desorption Spectroscopy - TDS
- Total Mass Loss
- Electron Stimulated Desorption - ESD
- Pumping speed
- Permeation

Outgassing measurement – throughput method



Sample & background

background

$$Q = \left(\frac{C\,(P_2 - P_1) - C\,(P_{2bg} - P_{1bg})}{A_{sample}} \right)$$

Q in mbar l s^{-1} cm^{-2}

Unbaked : outgassing of water – time dependent
Cold ionization gauges

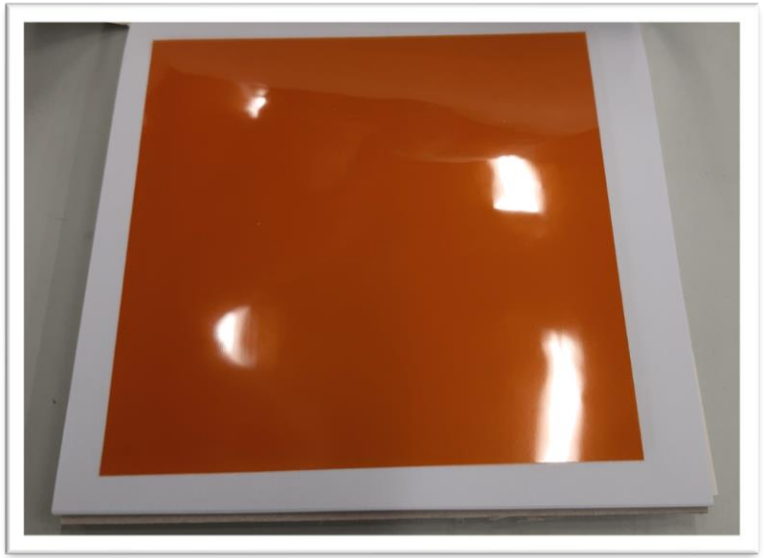
Baked: outgassing of (mainly) hydrogen
cold/hot ionization gauges

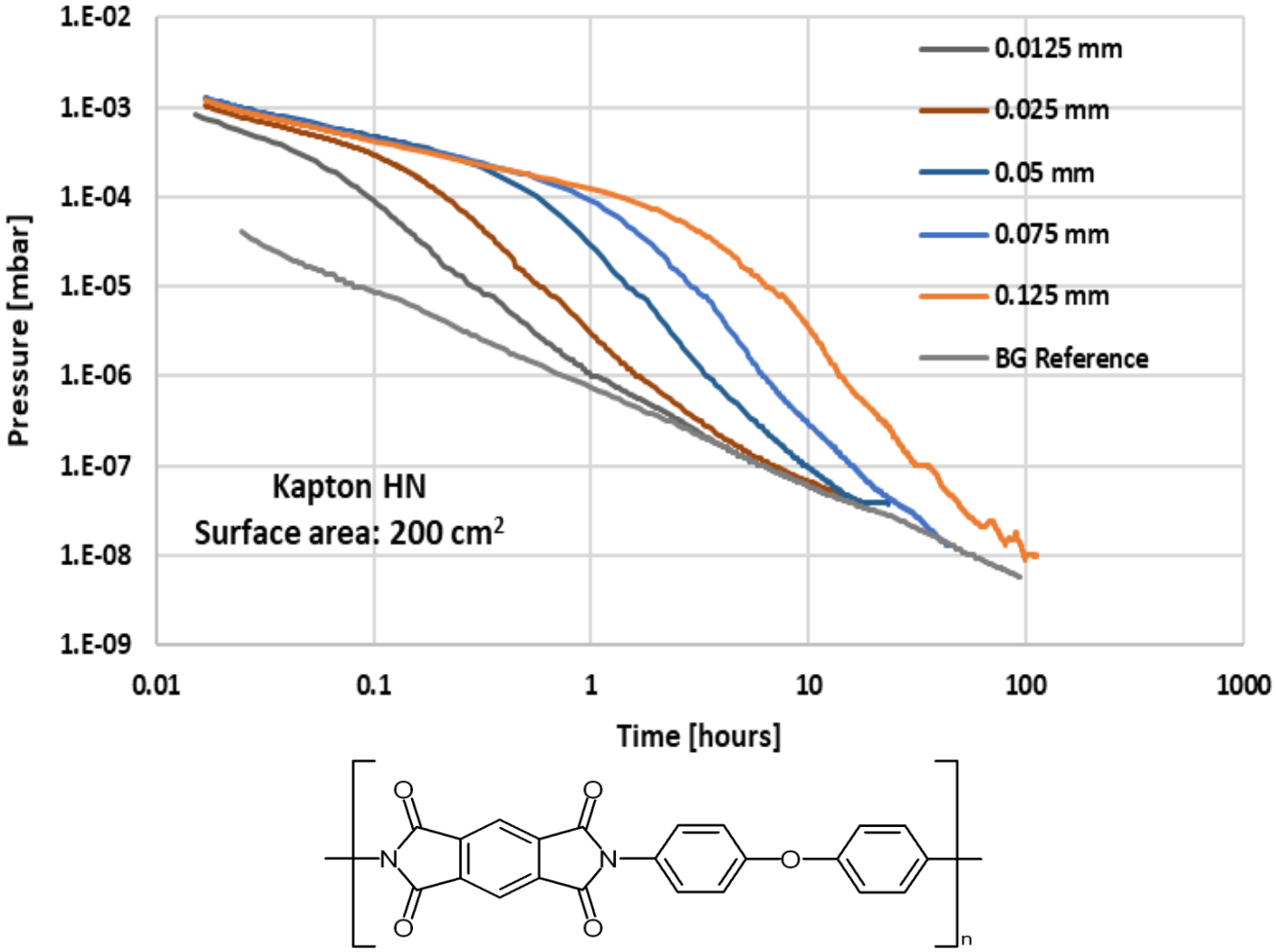
Outgassing measurement - throughput method

Kapton HN

Polyimide - Melting temperature: none
Expected water content: 0.96%

Samples: square sheets side 100 mm
Thicknesses: 0.0125-0.125 mm





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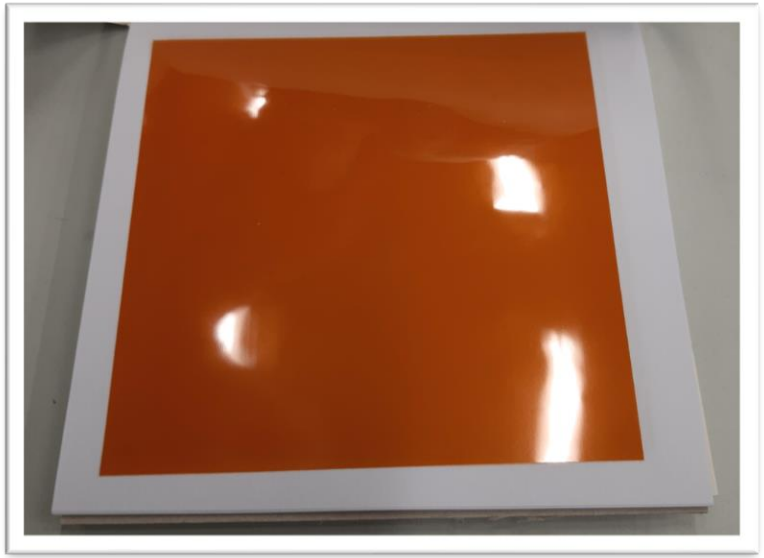
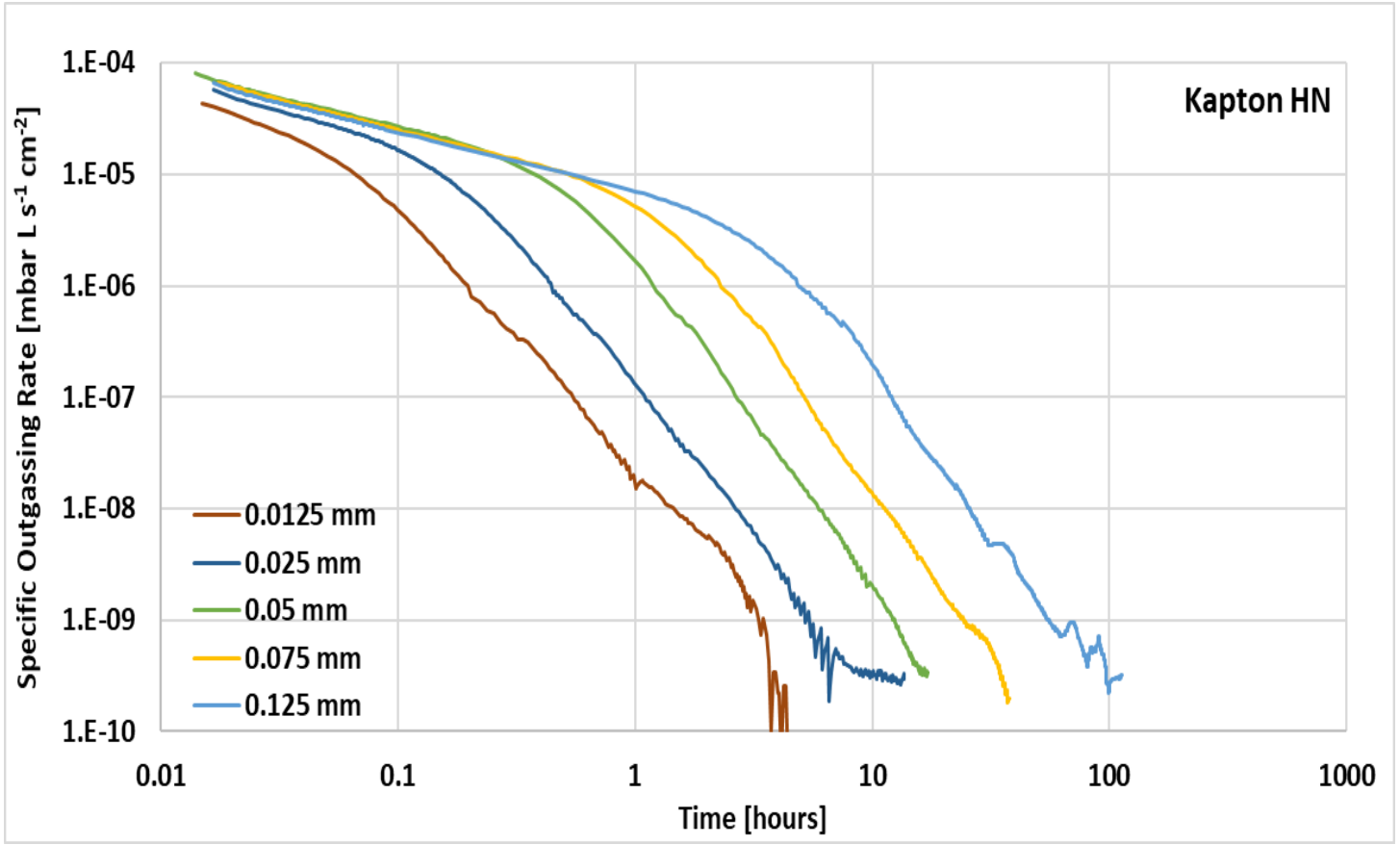
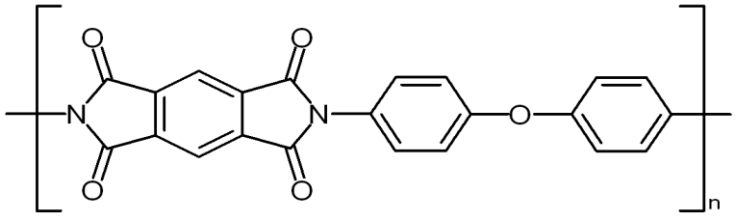
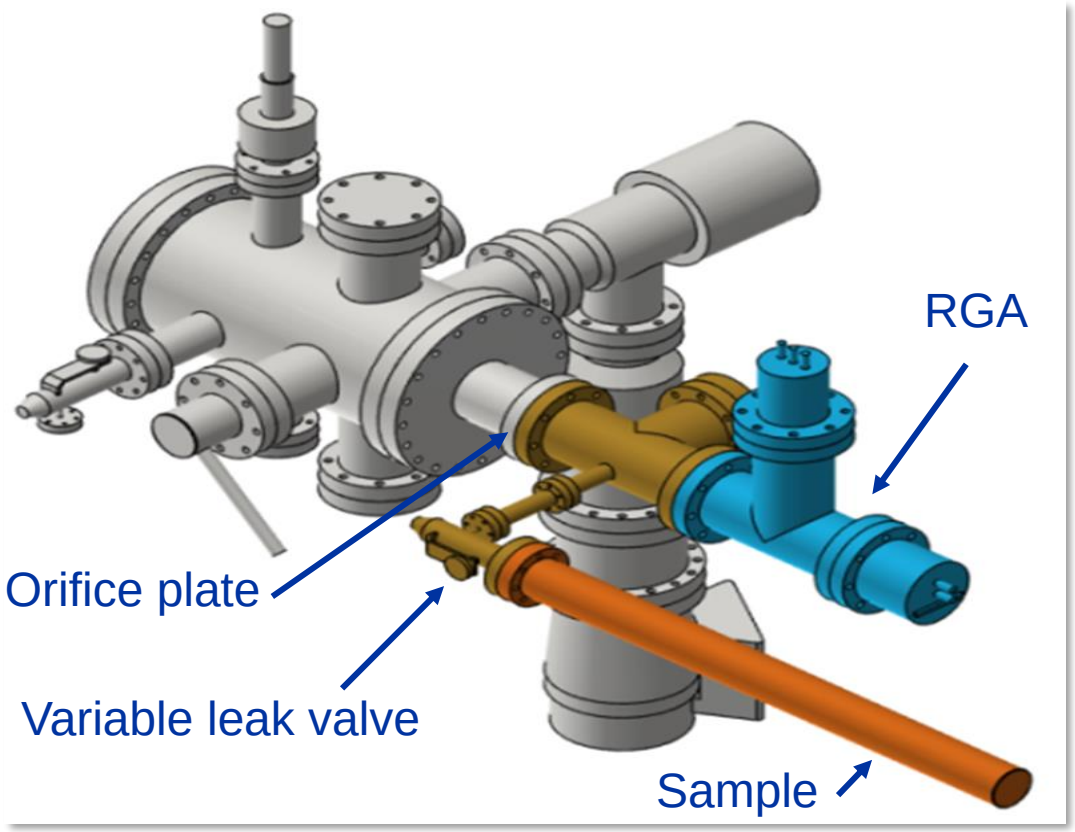






Outgassing: Coupled-method measurements



All system components are baked to temperatures ranging from 200°C to 350°C

Sample and Variable leak valve are heated to requested temperature and duration

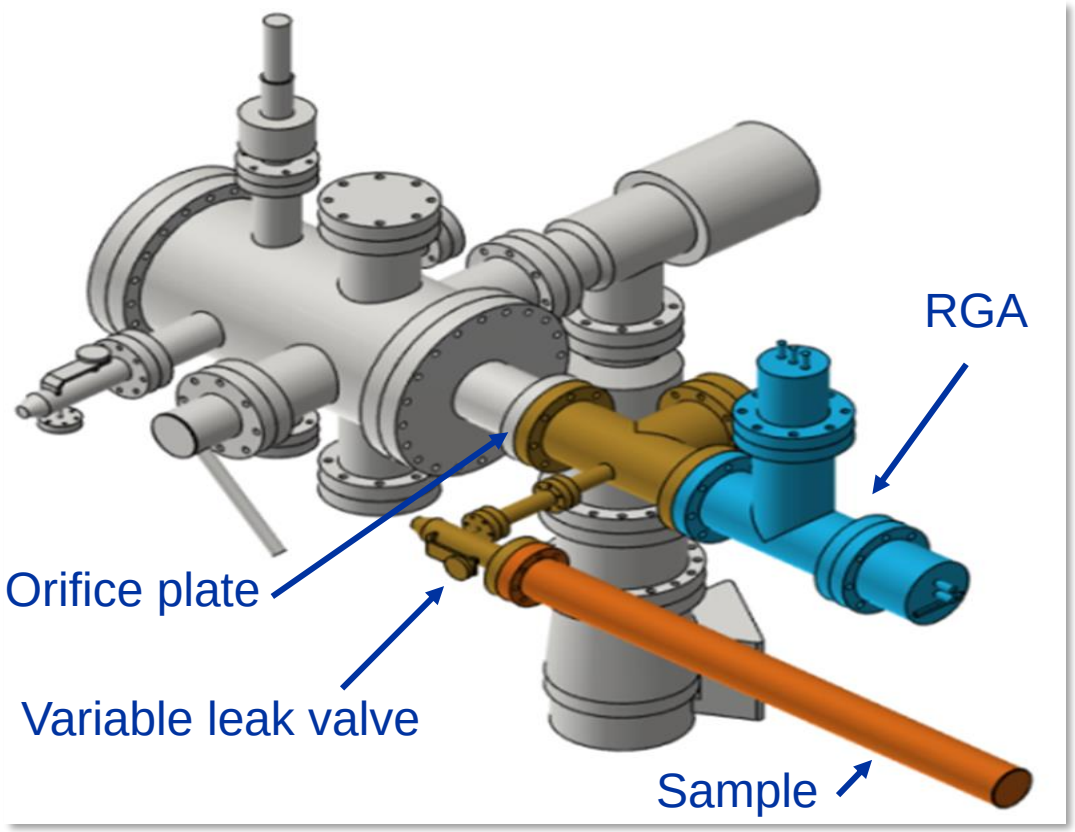
Prior to cooldown all instrumentation is degassed

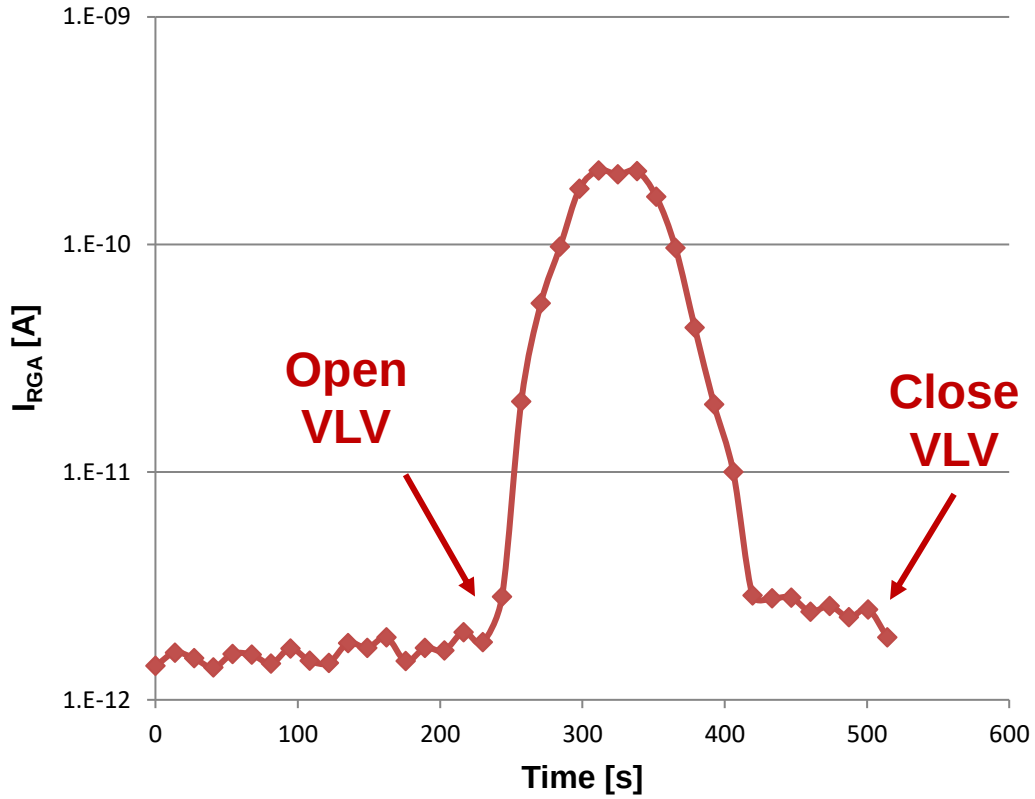
Variable leak valve body has been vacuum fired to minimize contribution

24 hours at room temperature before the start of the first measurement

RGA has been calibrated to an in-house calibrated hot ionization gauge

Outgassing: Coupled-method measurements





Outgassing: Coupled-method measurements

$$Q\ [mbarl/s] = \frac{S_c \times \int_{\Delta\tau} I_{RGA} \times \alpha_{RGA}\, d\tau}{\Delta t_a}$$

Δt_a: duration of the accumulation [s]

I_{RGA}: current recorded for the gas of interest [A]

S_c: conductance of the orifice for the gas of interest [l/s]

α_{RGA}: calibration factor for the gas of interest [A/mbar]

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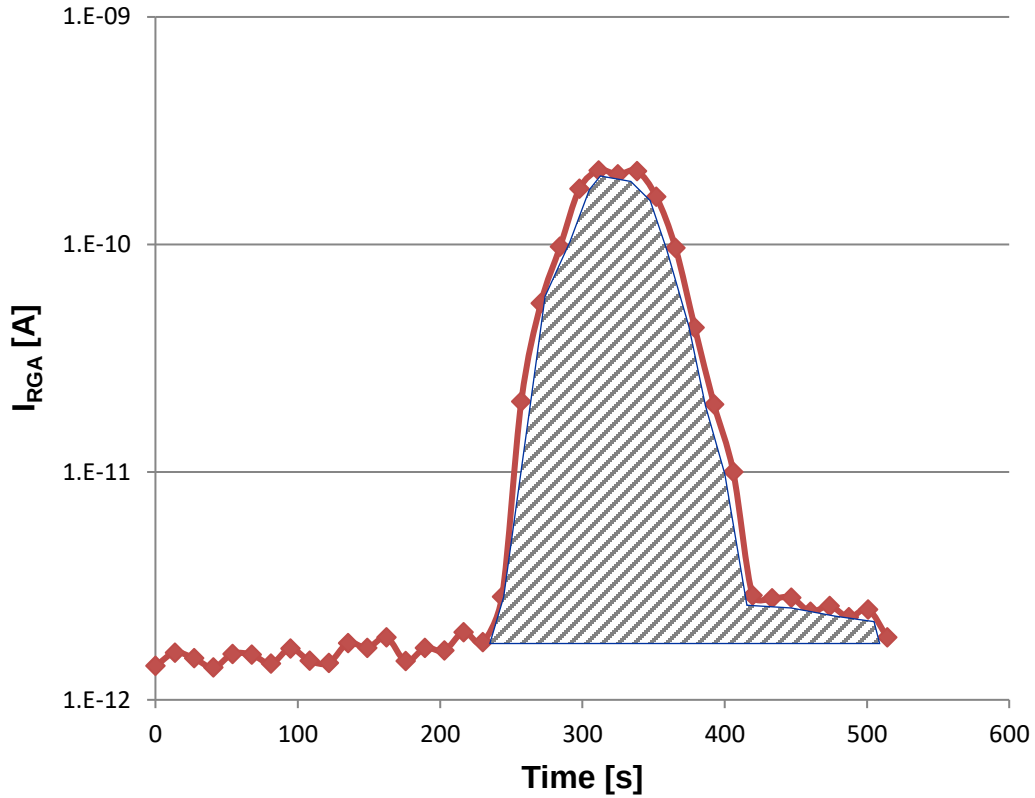

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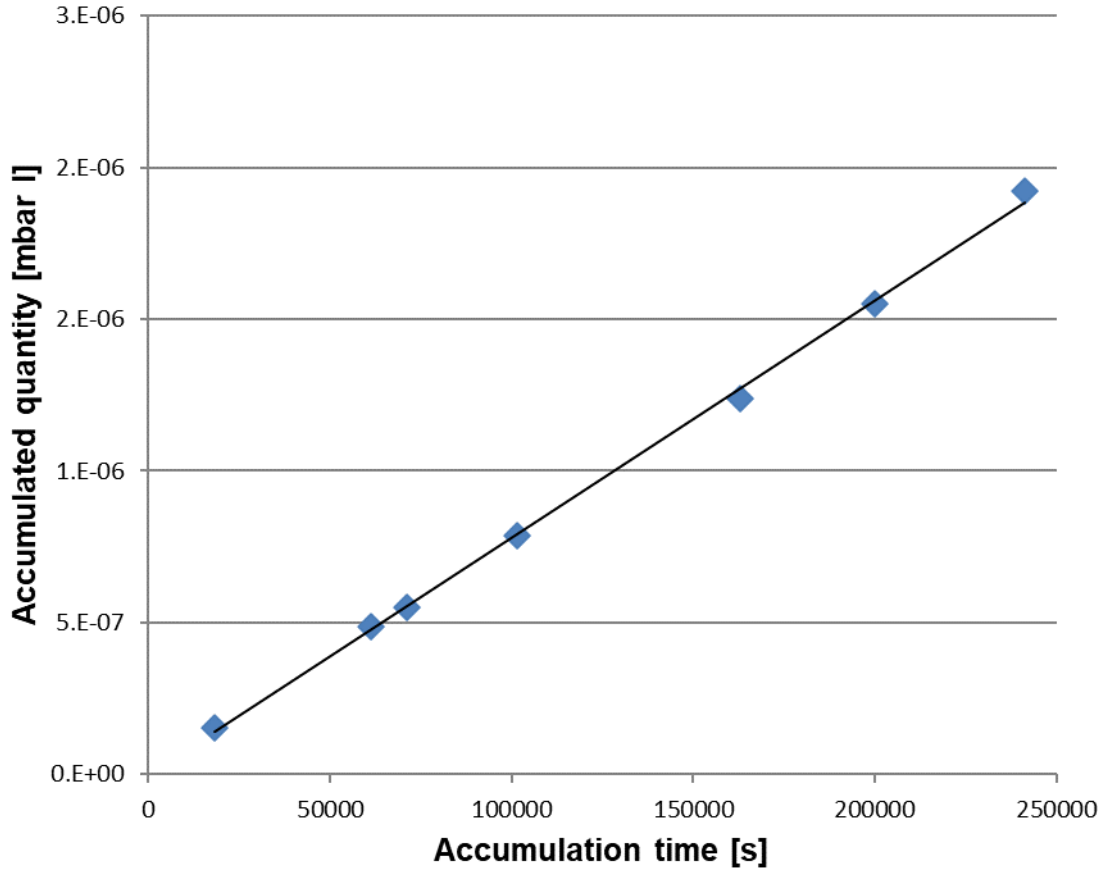
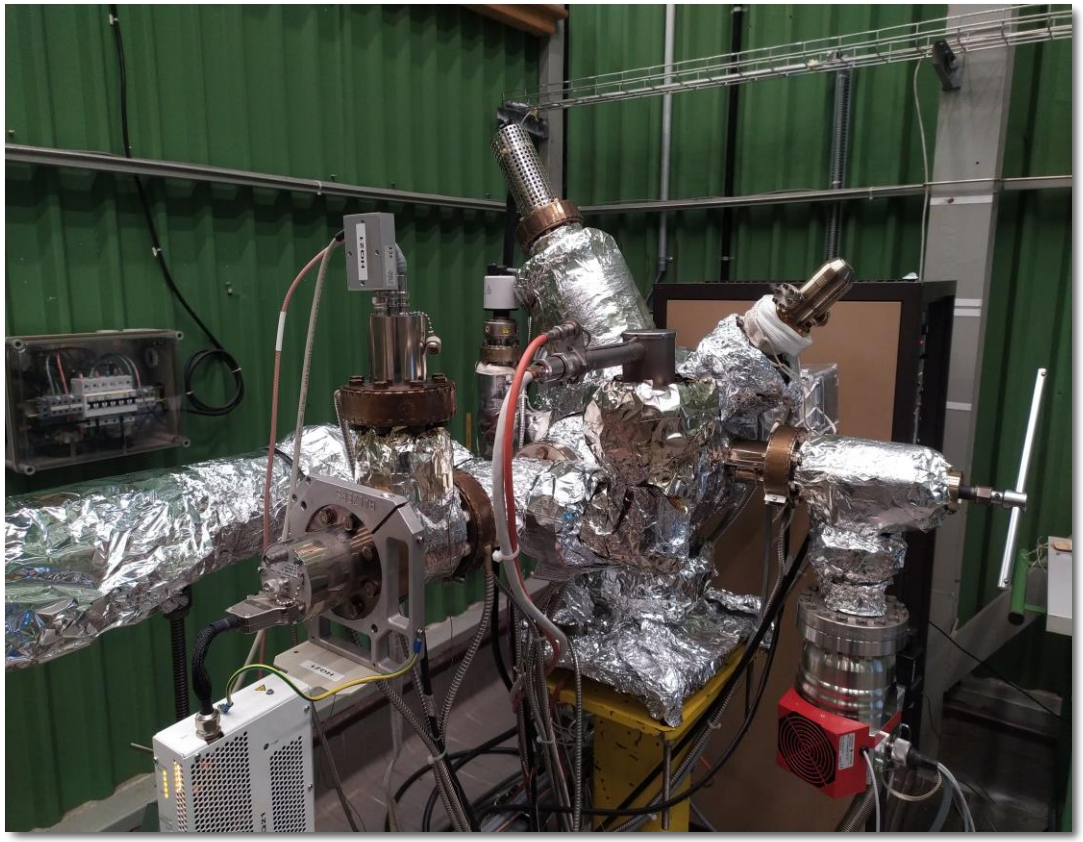
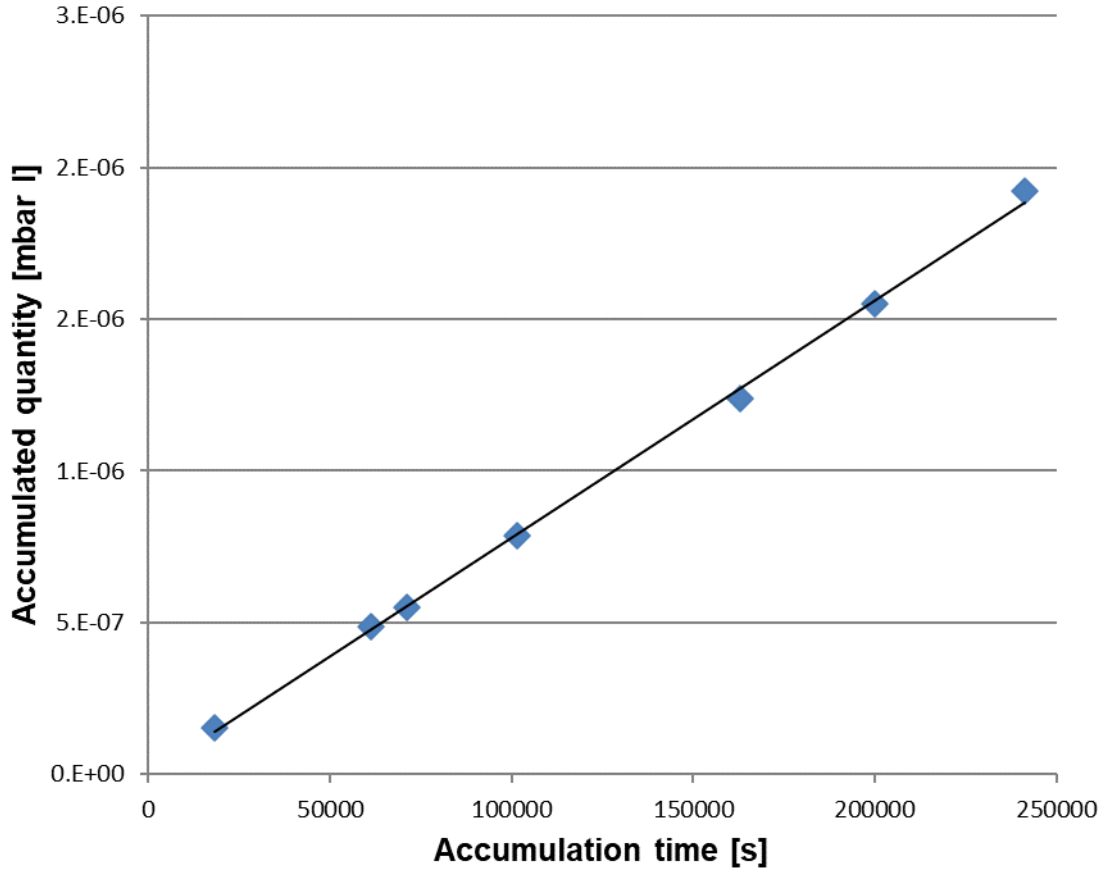


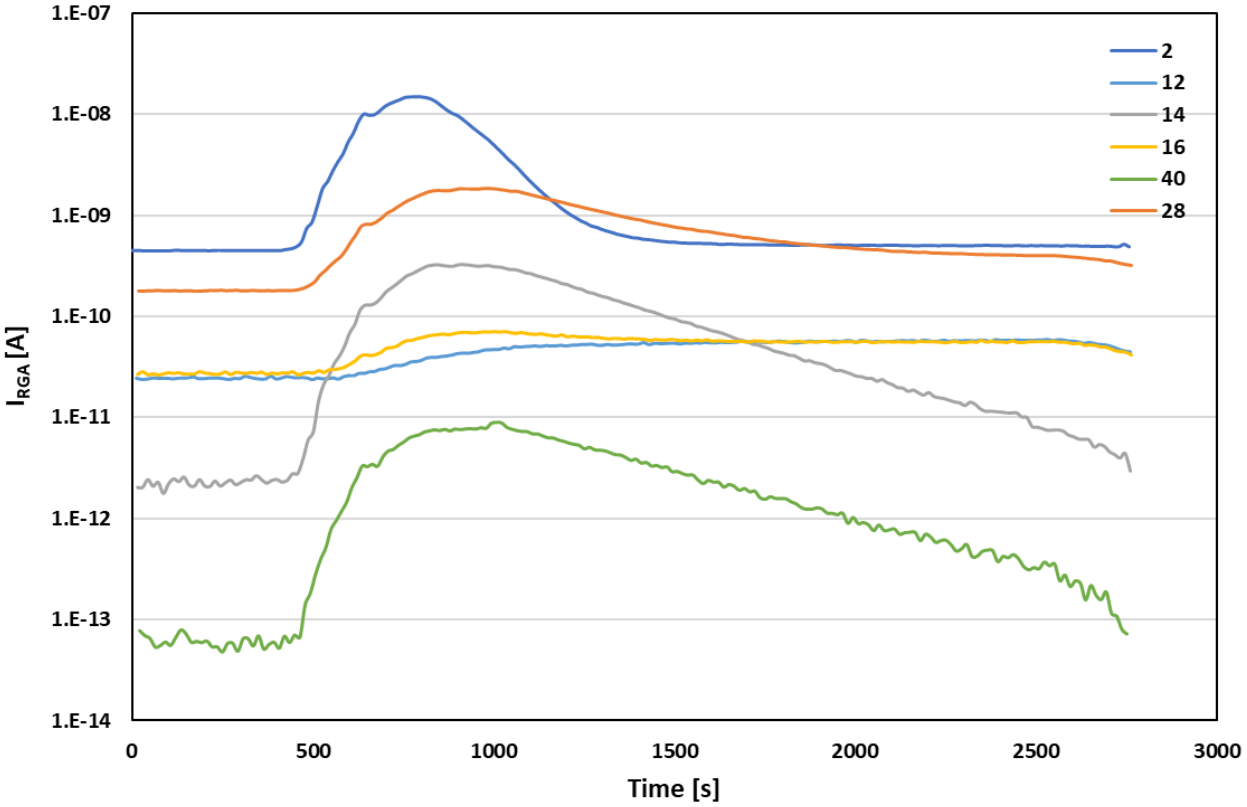
Outgassing: Coupled-method measurements

For a description of the method, see p. 11 of https://arxiv.org/ftp/arxiv/papers/2006/2006.07124.pdf





Outgassing: Coupled-method measurements



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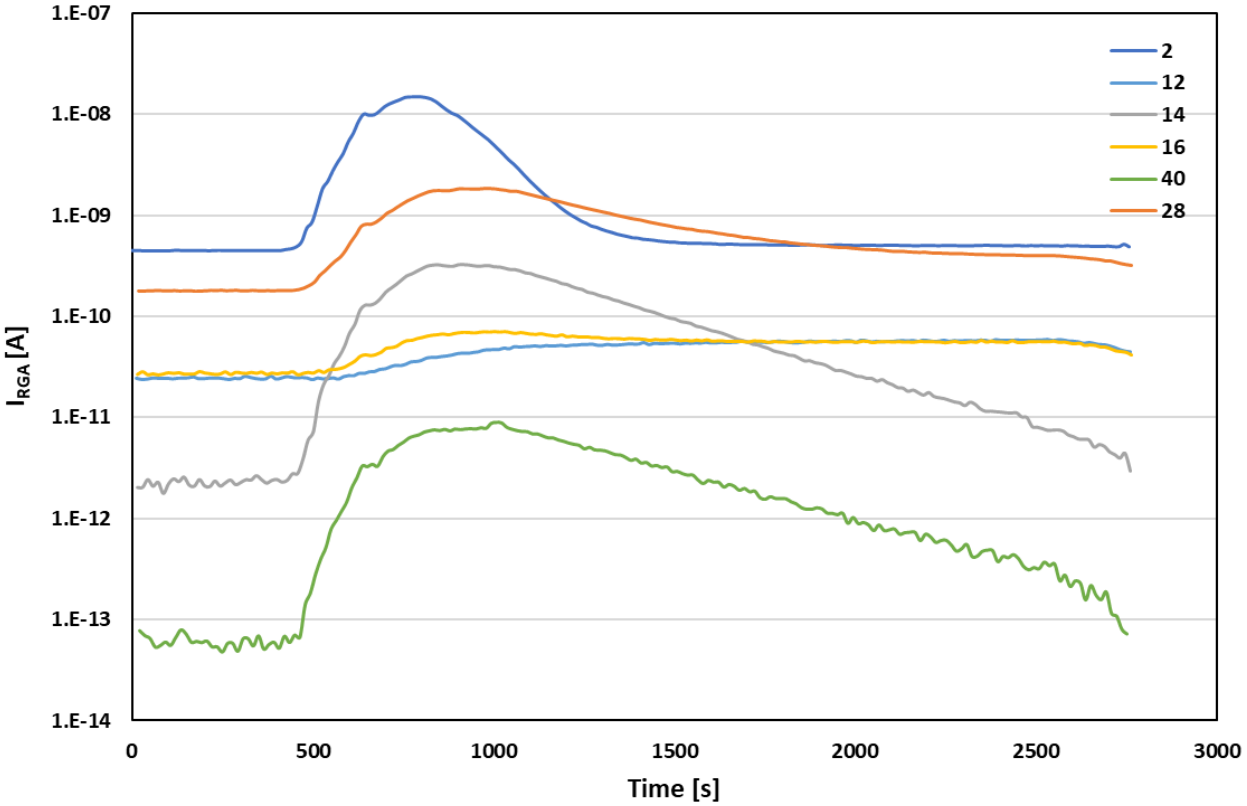
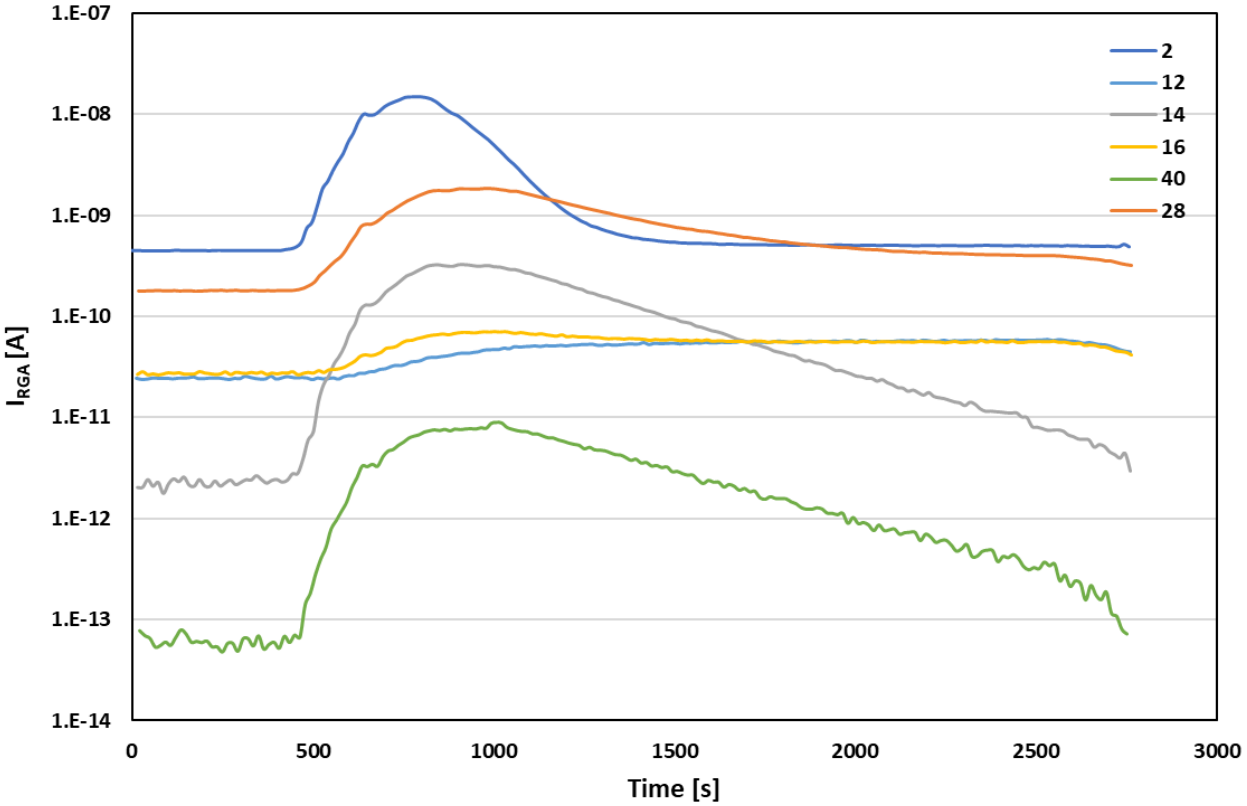


Mass 12, 16 & 28 do not show a similar behavior not CO

Outgassing: Coupled-method measurements



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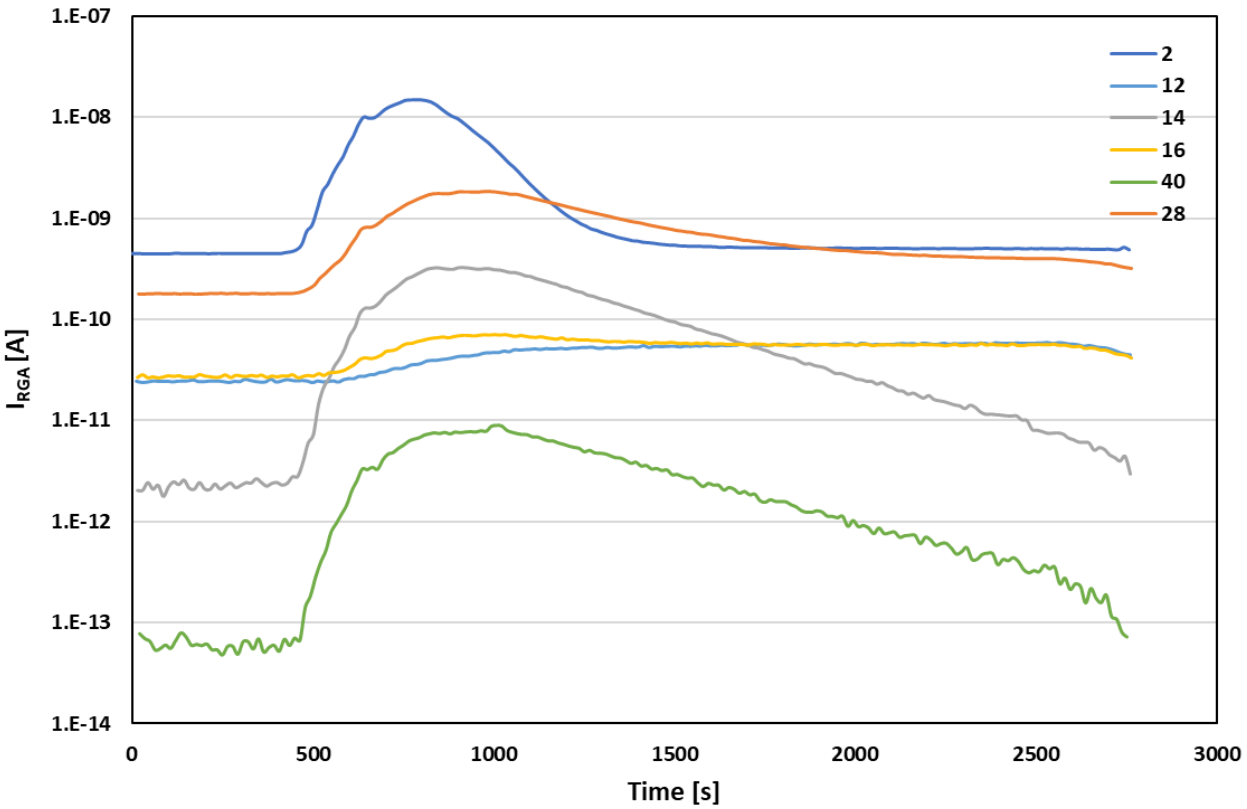
Mass 14 & 28 show a similar behavior — N_2

Mass 40 (Argon) also in line with N_2 — **AIR**

Outgassing: Coupled-method measurements



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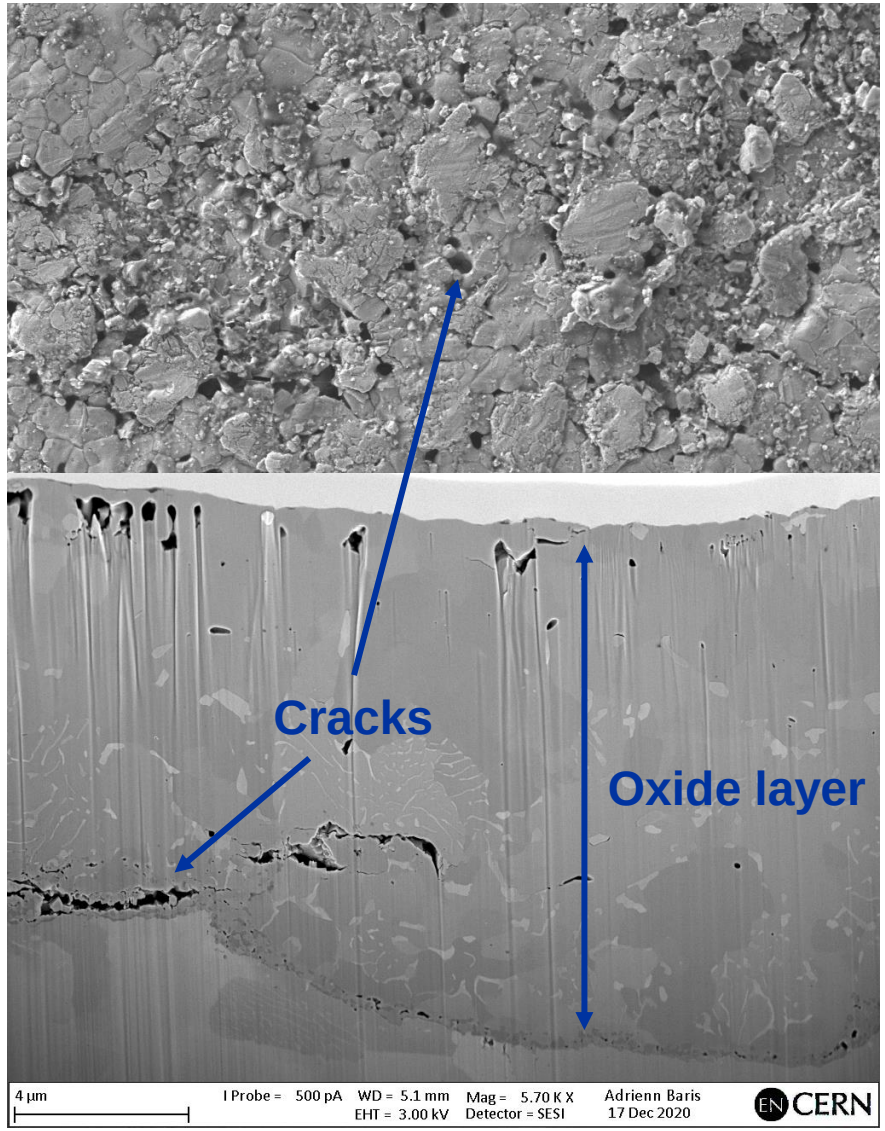
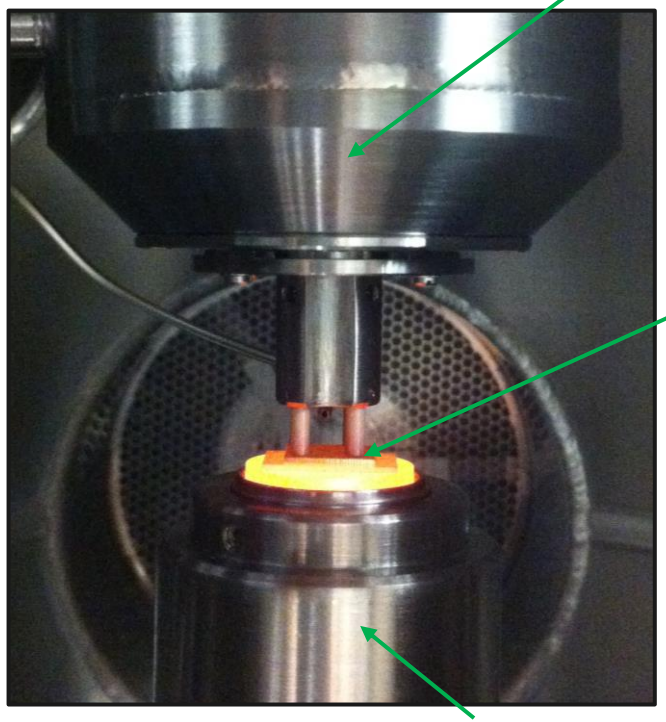
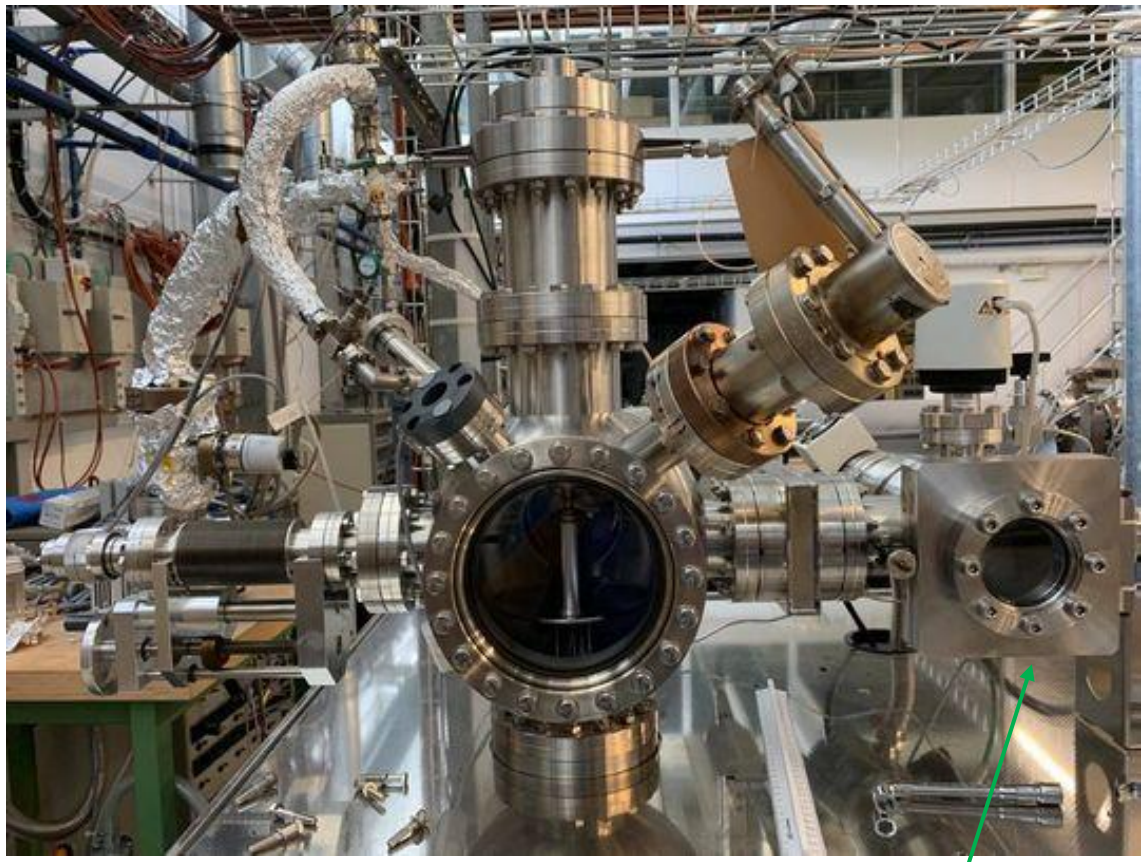


Thermal desorption spectroscopy

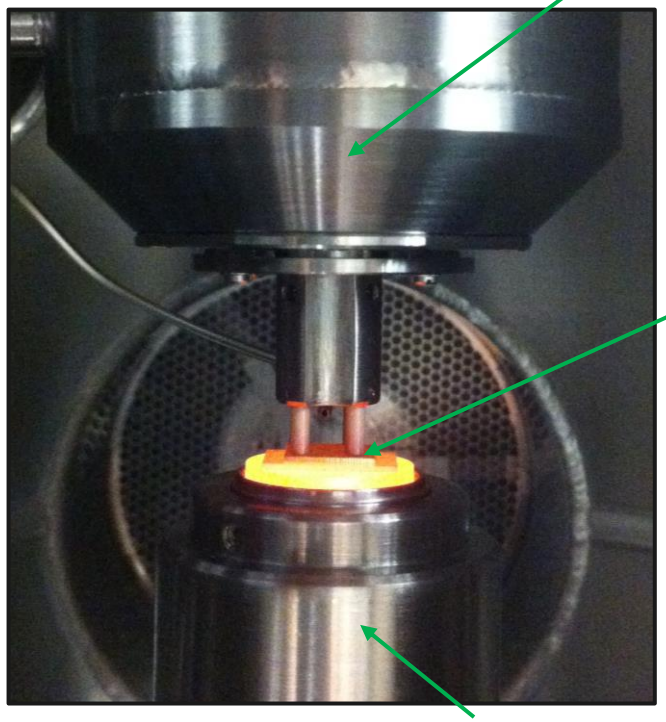


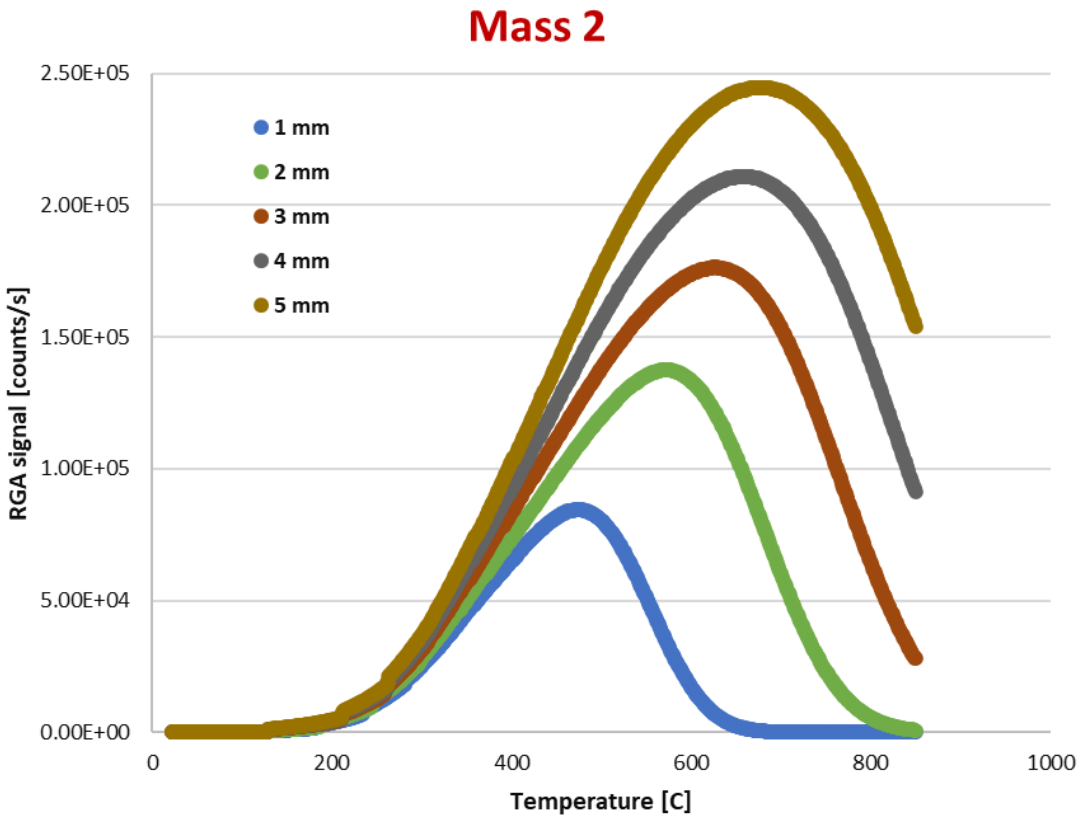


Heating rate: 5K/min

Dimensions of sample: 10mm x 10mm x 1mm

Thermal desorption spectroscopy





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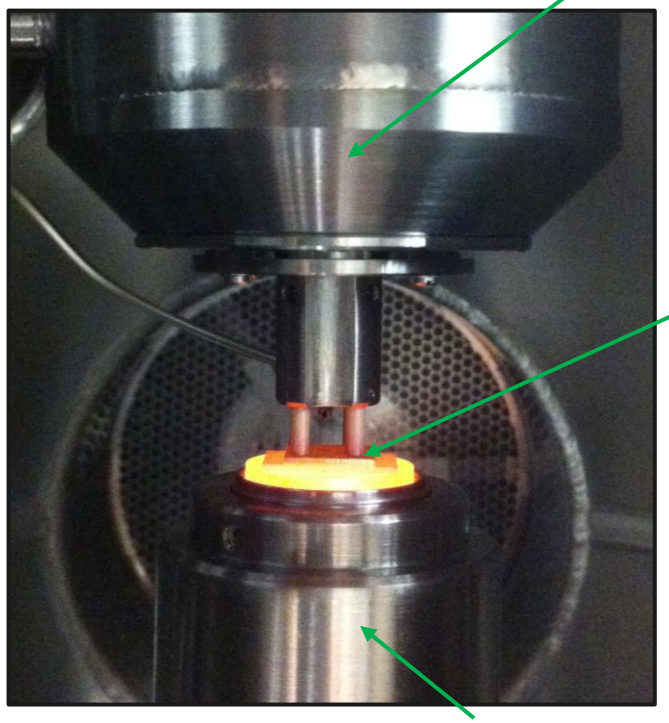

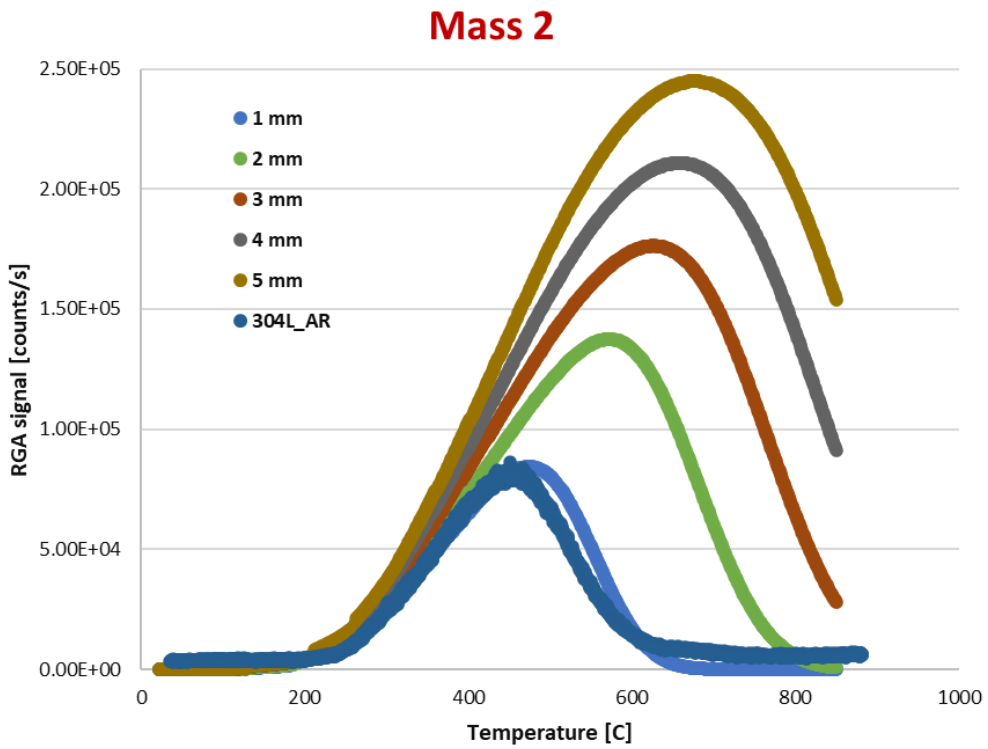

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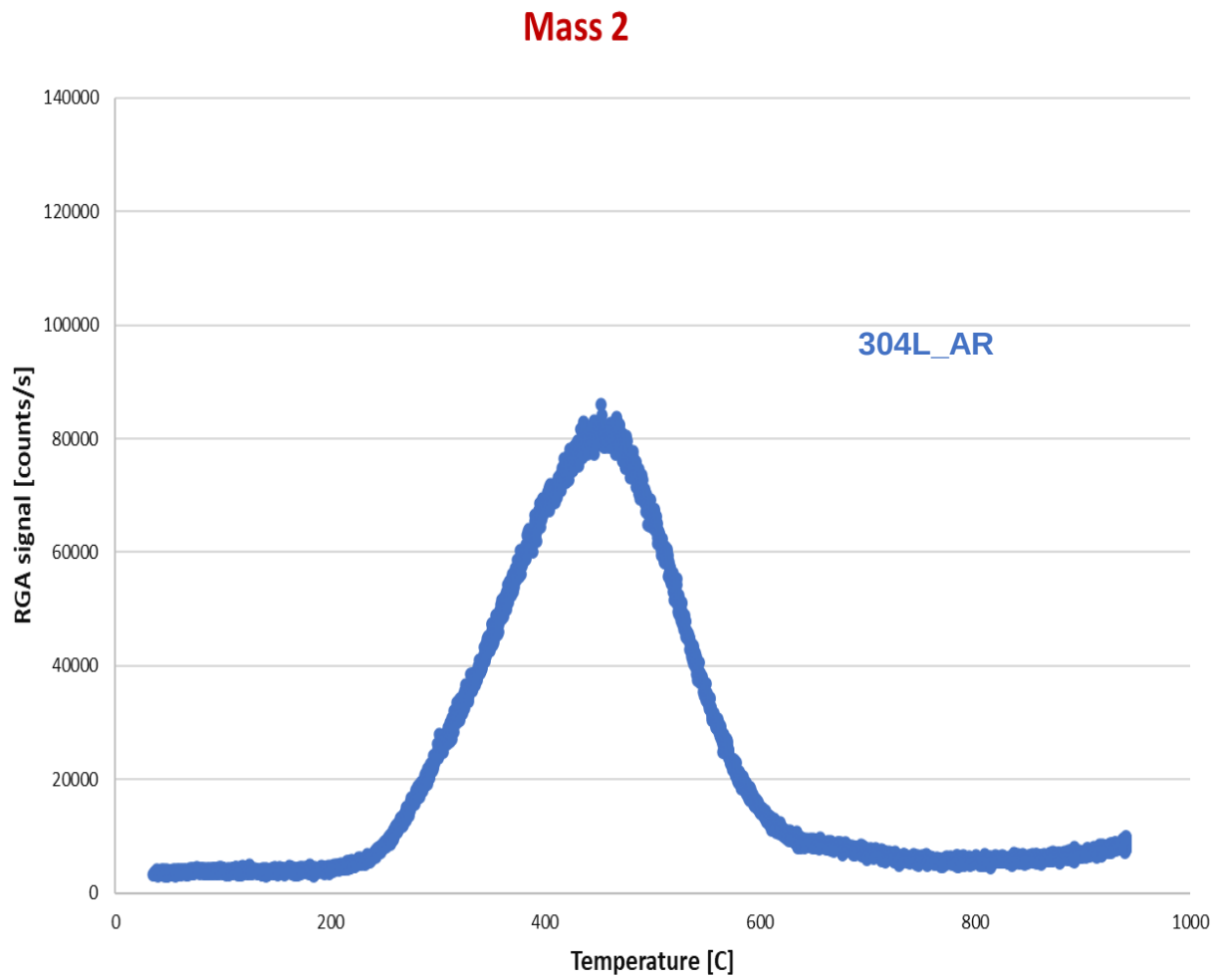
TDS measurement

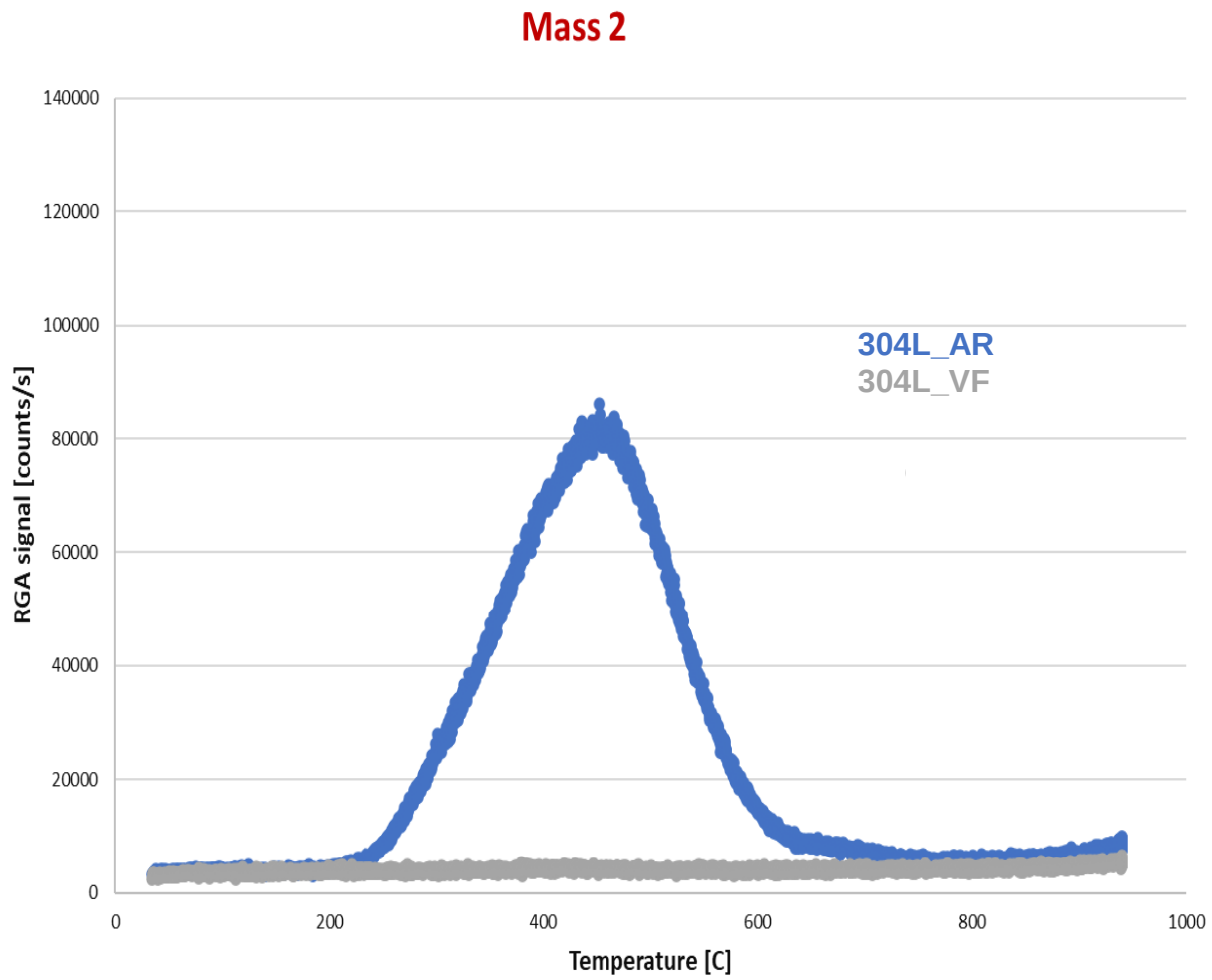


304L_AR

62.0 ppm (atomic)

AR : As Received
VF : Vacuum Fired (950C/2h)
aSi:H CVD coating

TDS measurement



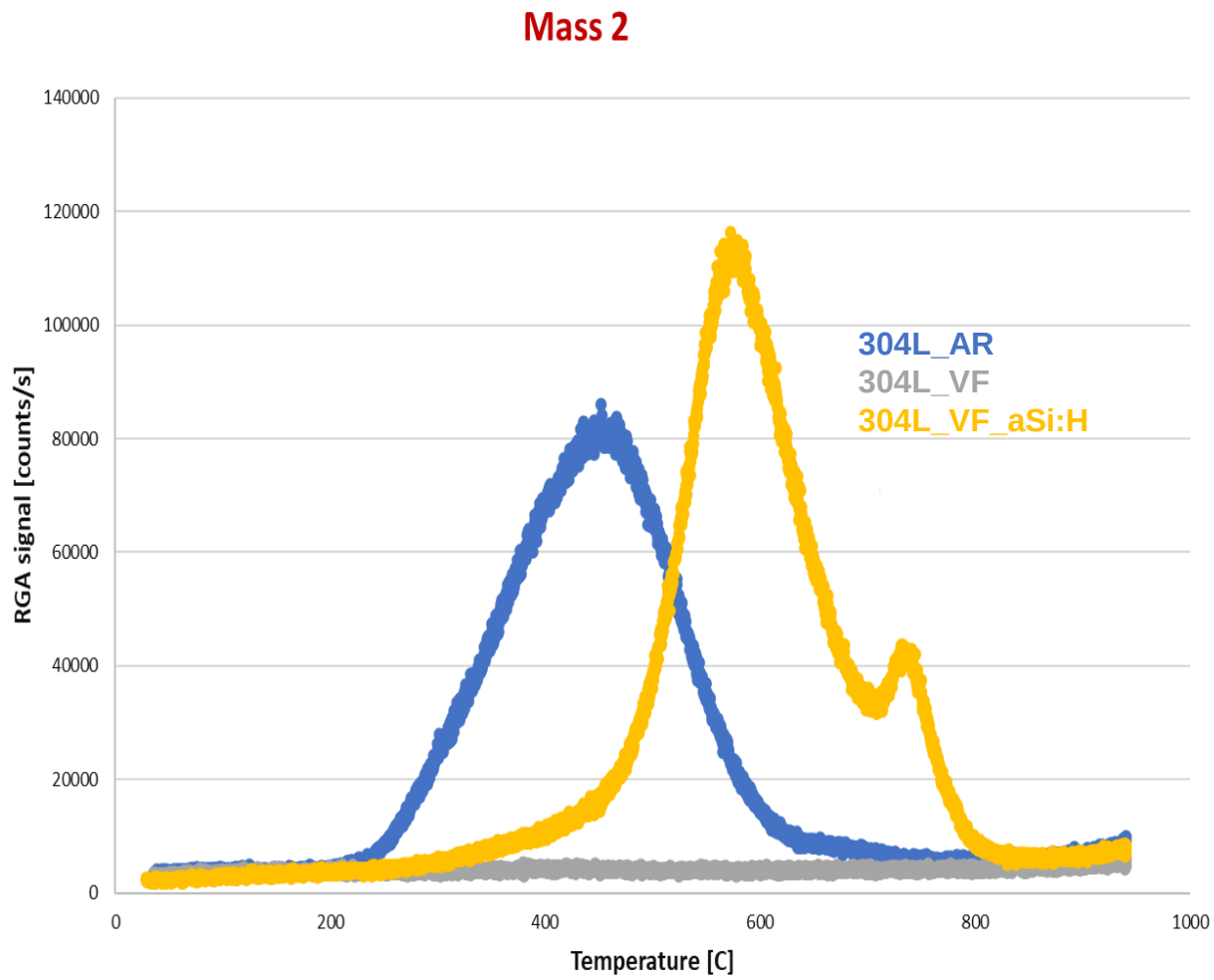
304L_AR — 62.0 ppm (atomic)

⇩

304L_VF — 1.4 ppm (atomic)

AR : As Received
VF : Vacuum Fired (950C/2h)
aSi:H CVD coating

TDS measurement



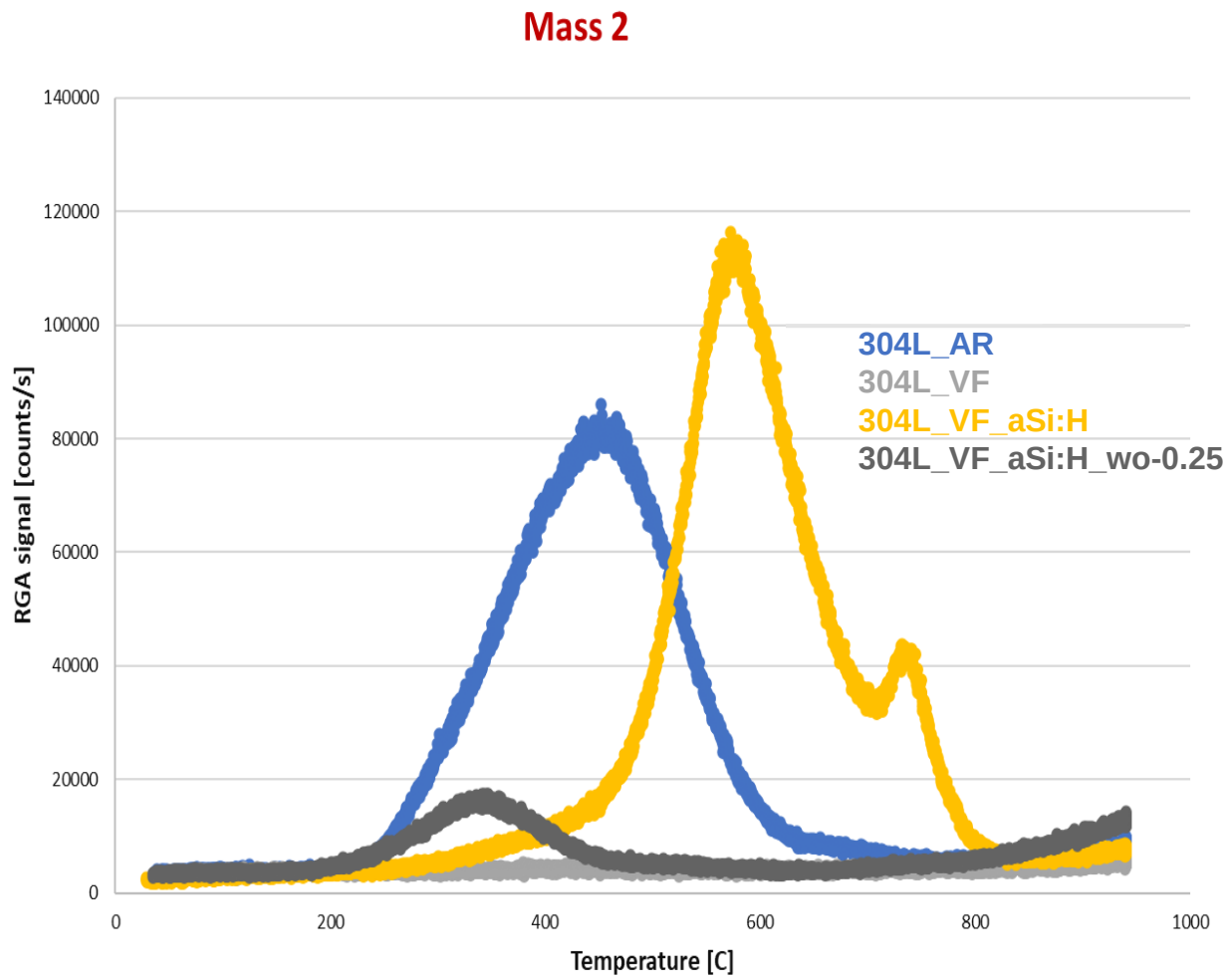
304L_AR → 62.0 ppm (atomic)

304L_VF → 1.4 ppm (atomic)

304L_VF_aSi:H → 81.5 ppm (atomic)

AR : As Received
VF : Vacuum Fired (950C/2h)
aSi:H CVD coating

TDS measurement



304L_AR → 62.0 ppm (atomic)

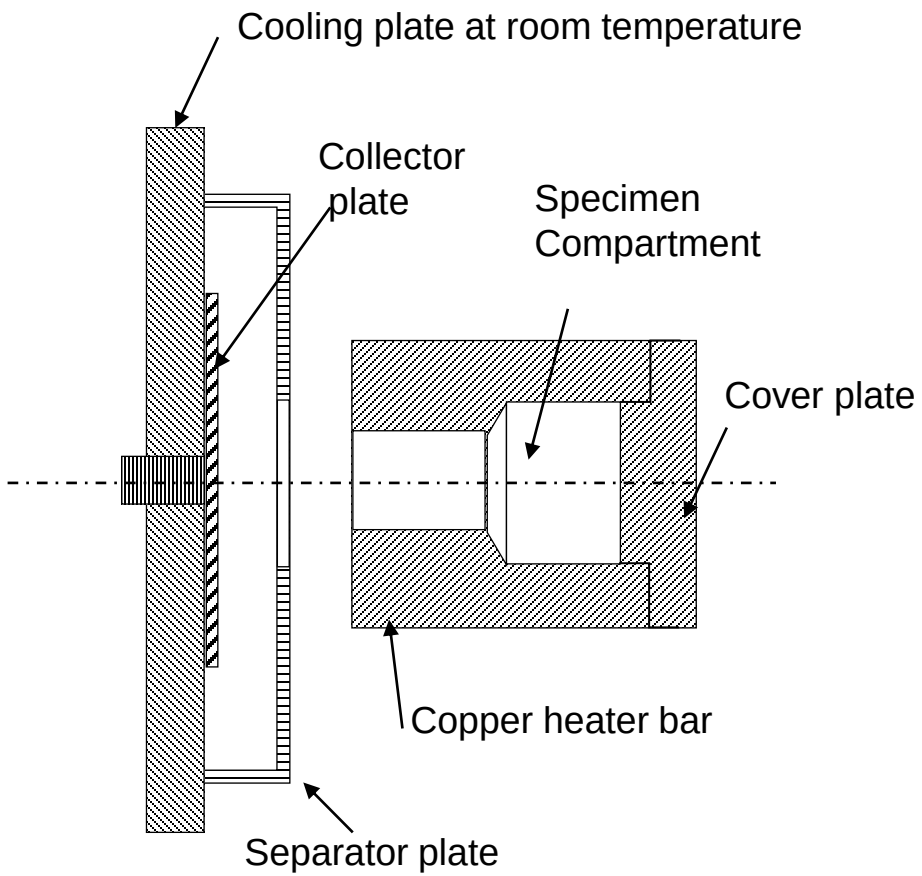
304L_VF → 1.4 ppm (atomic)

304L_VF_aSi:H → 81.5 ppm (atomic)

304L_VF_ aSi:H _wo-0.25 → 20.4 ppm (atomic)

AR : As Received
VF : Vacuum Fired (950C/2h)
aSi:H CVD coating

Total Mass Loss



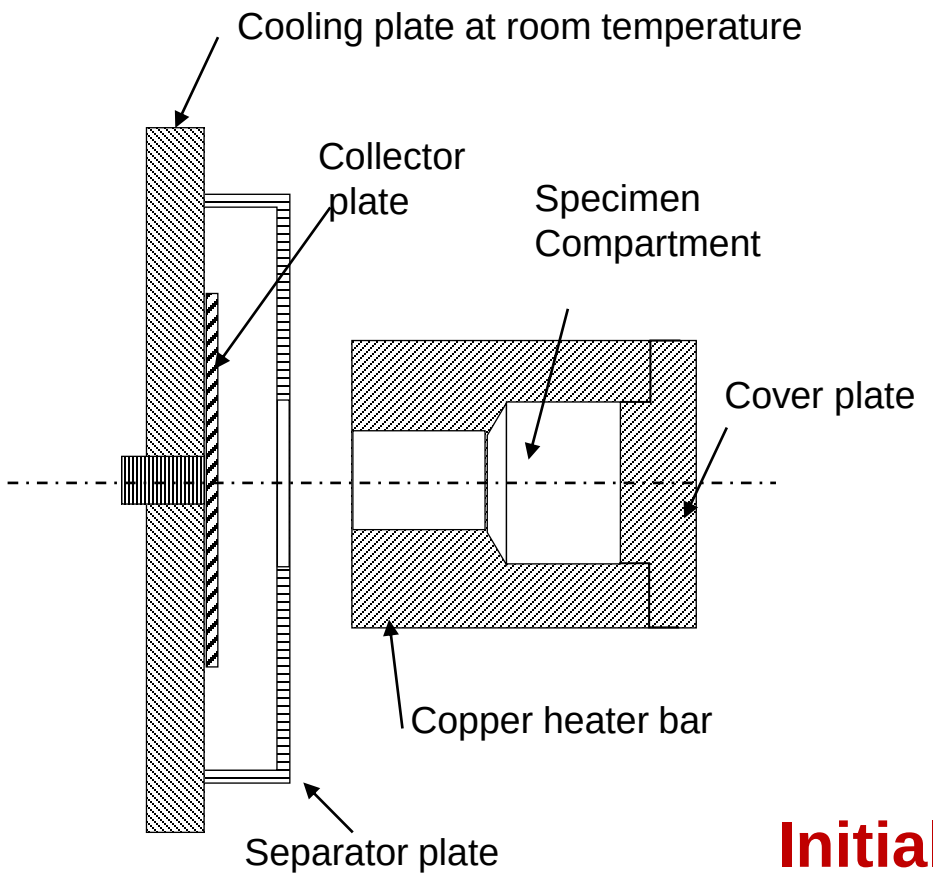
- Polymer specimen is conditionned in a 50% RH environment for 24 hours
- Weight of specimen and collector plate is measured
- Under vacuum the specimen is heated up to 125°C for 24 hours
- Weight of specimen and collector plate is measured

$$TML[\%] = \frac{S_{init} - S_{final}}{S_{init}} \text{ x } 100$$

$$CVCM[\%] = \frac{C_{init} - C_{final}}{S_{init}} \text{ x } 100$$

ASTM E595-93 : Standard Test Method for Total Mass Loss and Collected Volatile Condensable Materials from Outgassing in a Vacuum Environment

Total Mass Loss



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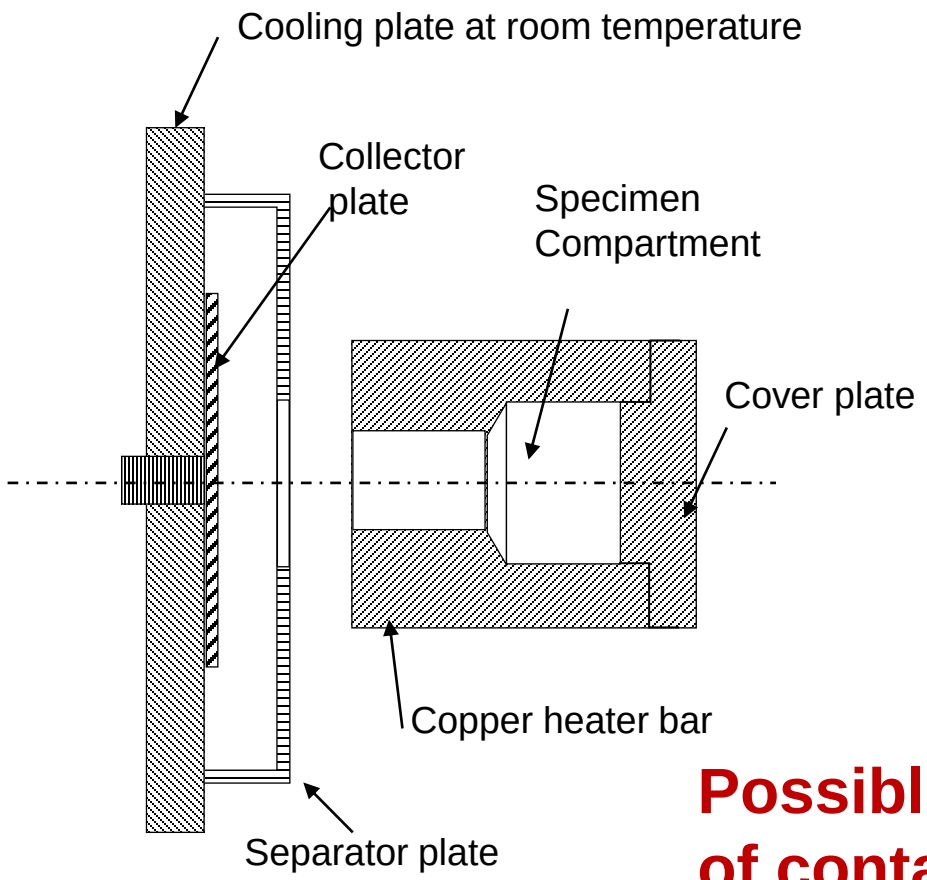
Initial concentration

$$CVCM[\%] = \frac{C_{init} - C_{final}}{S_{init}} \text{ x } 100$$

ASTM E595-93 : Standard Test Method for Total Mass Loss and Collected Volatile Condensable Materials from Outgassing in a Vacuum Environment

Total Mass Loss



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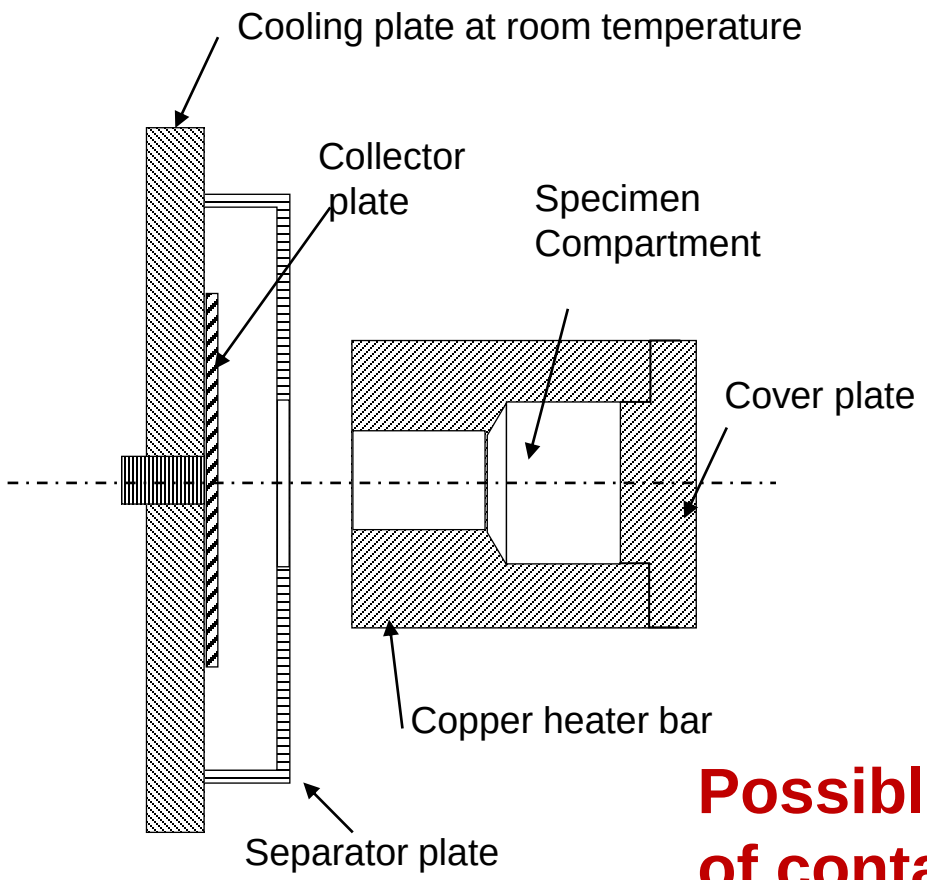
$$TML[\%] = \frac{S_{init} - S_{final}}{S_{init}} \text{ x } 100$$

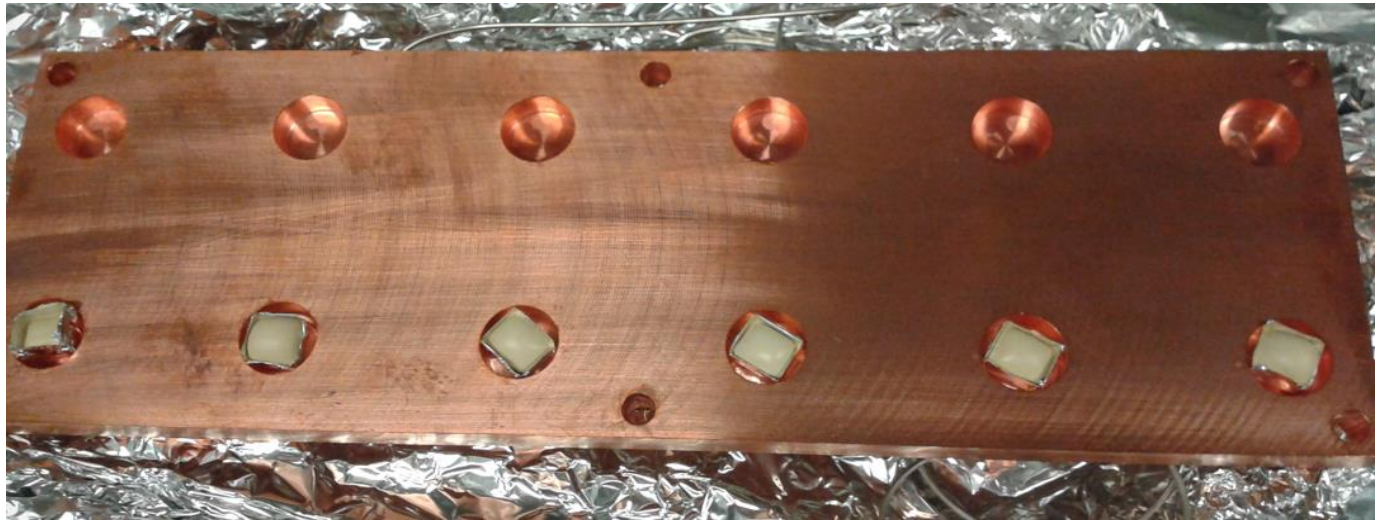
Possible source of contamination

$$CVCM[\%] = \frac{C_{init} - C_{final}}{S_{init}} \text{ x } 100$$

ASTM E595-93 : Standard Test Method for Total Mass Loss and Collected Volatile Condensable Materials from Outgassing in a Vacuum Environment

Total Mass Loss





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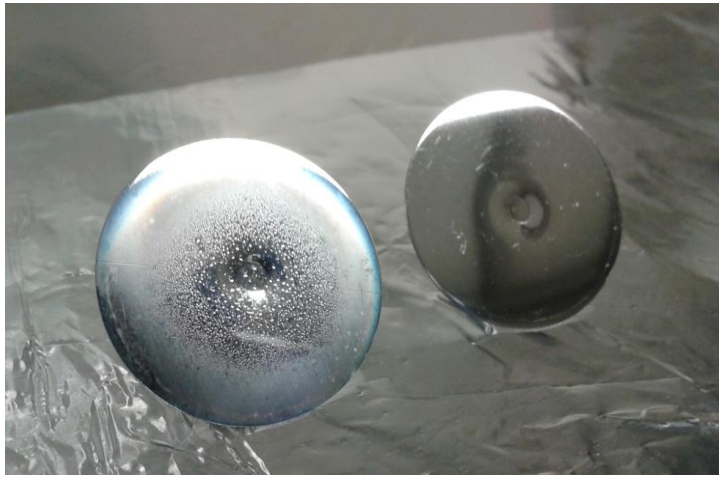

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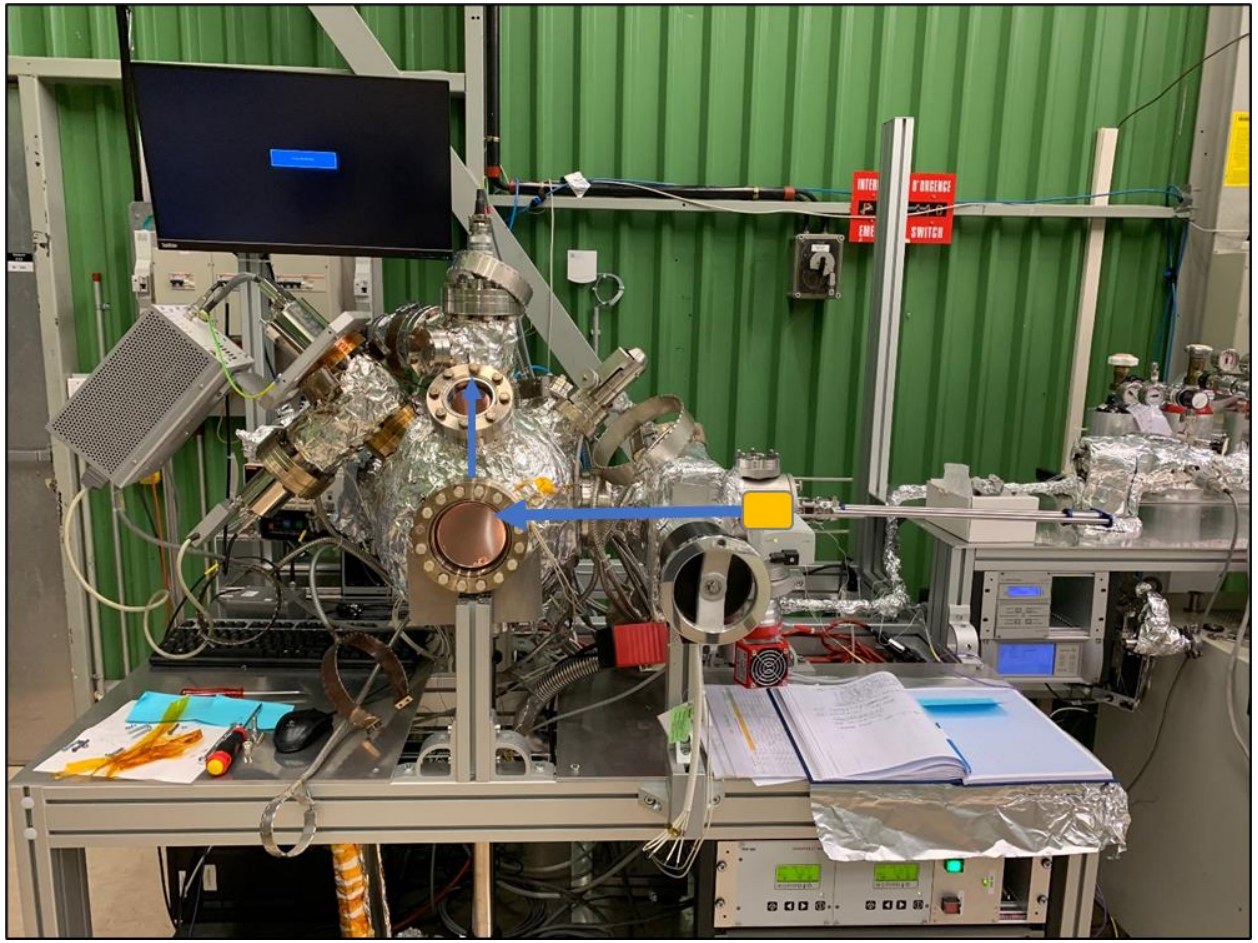
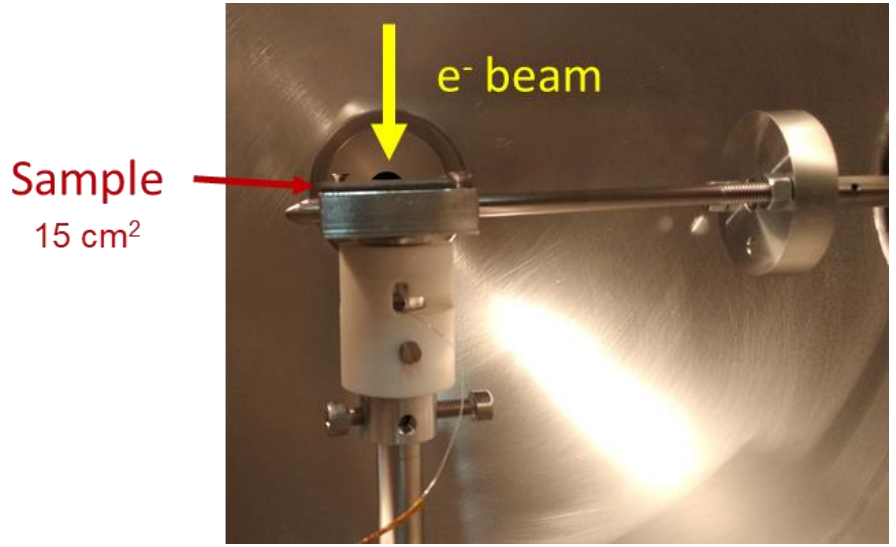
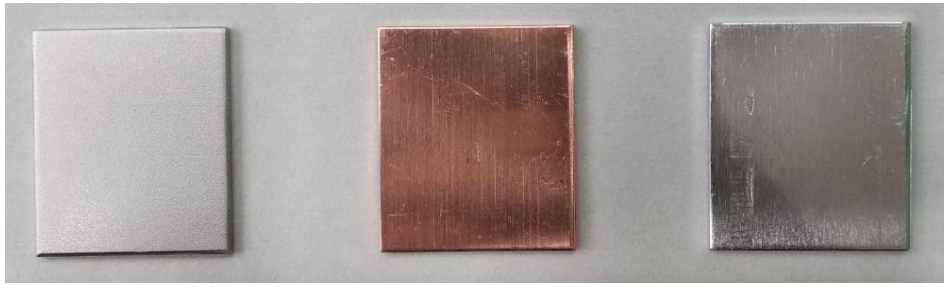
Electron Stimulated Desorption

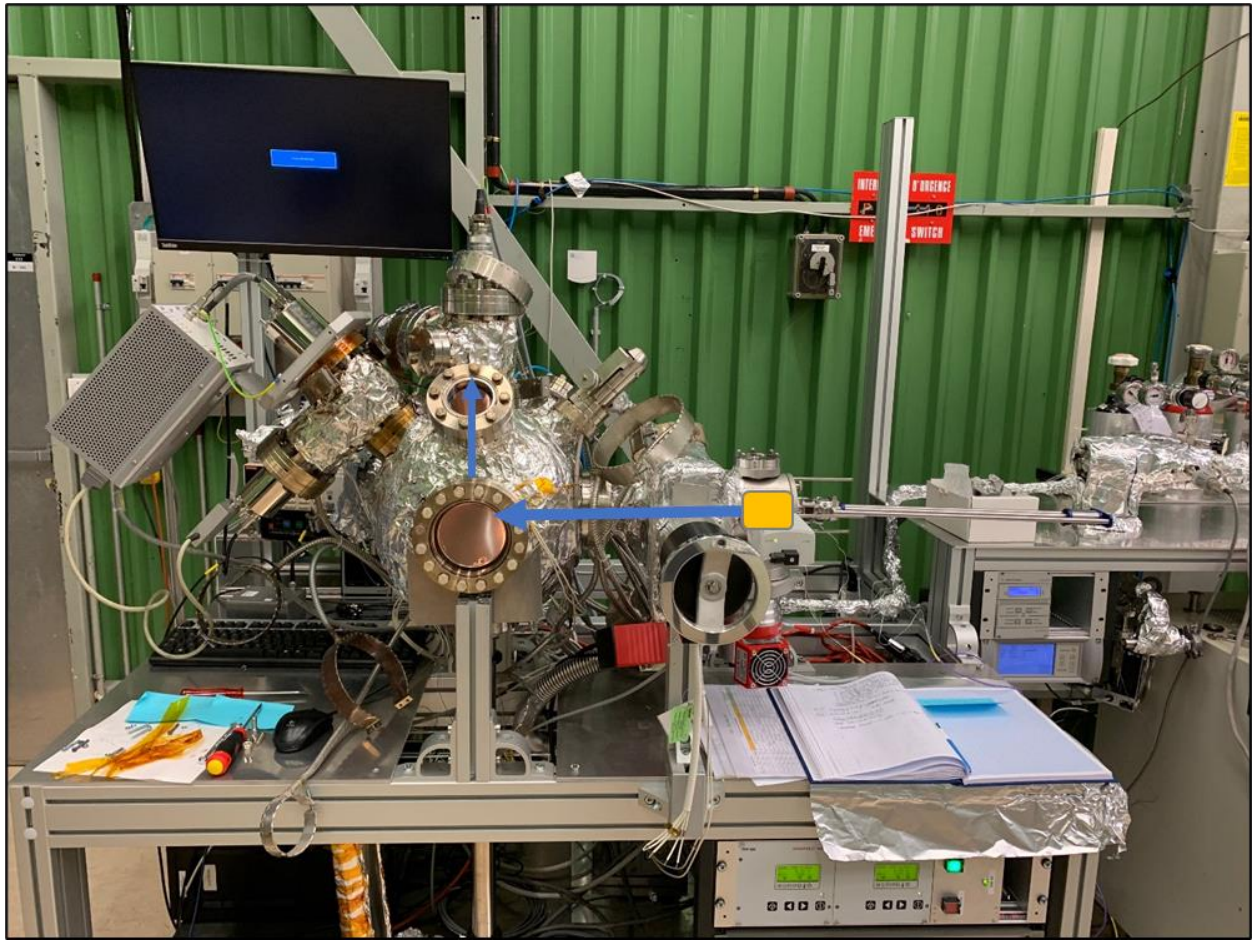


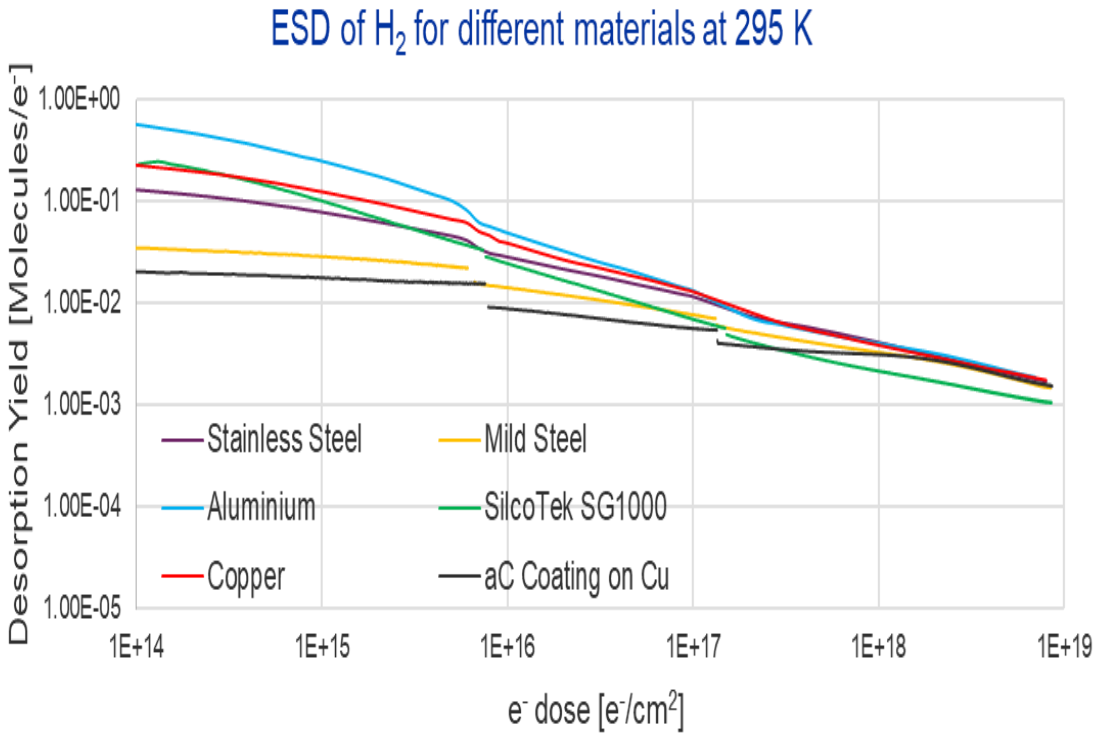




316 LN Cu-OFE Al

Electron Stimulated Desorption

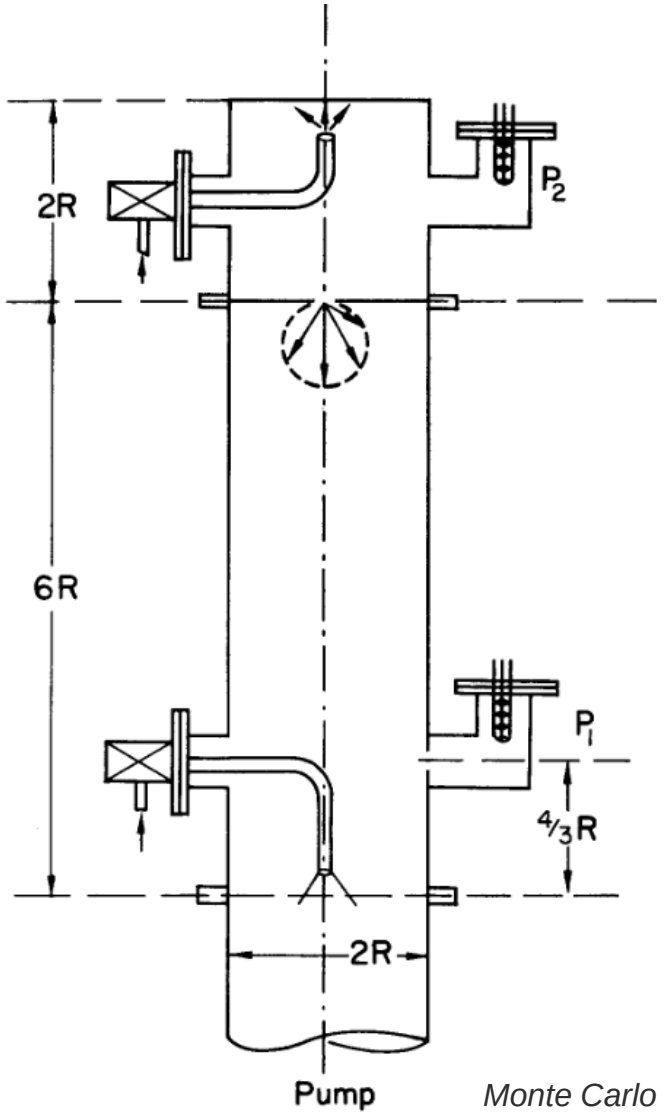




$$Desorption\ yield = \frac{molecules\ of\ desorbed\ gas}{incident\ electrons}$$

Dependent on: Energy – material – gas - dose

Pumping speed: Fischer-Mommsen dome

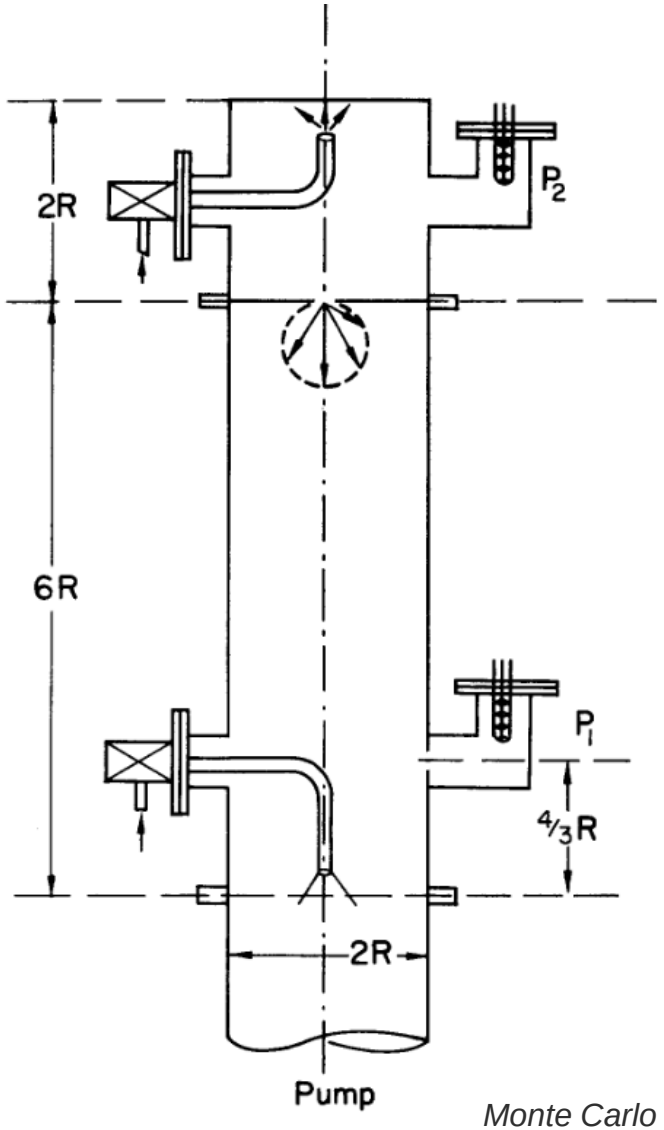


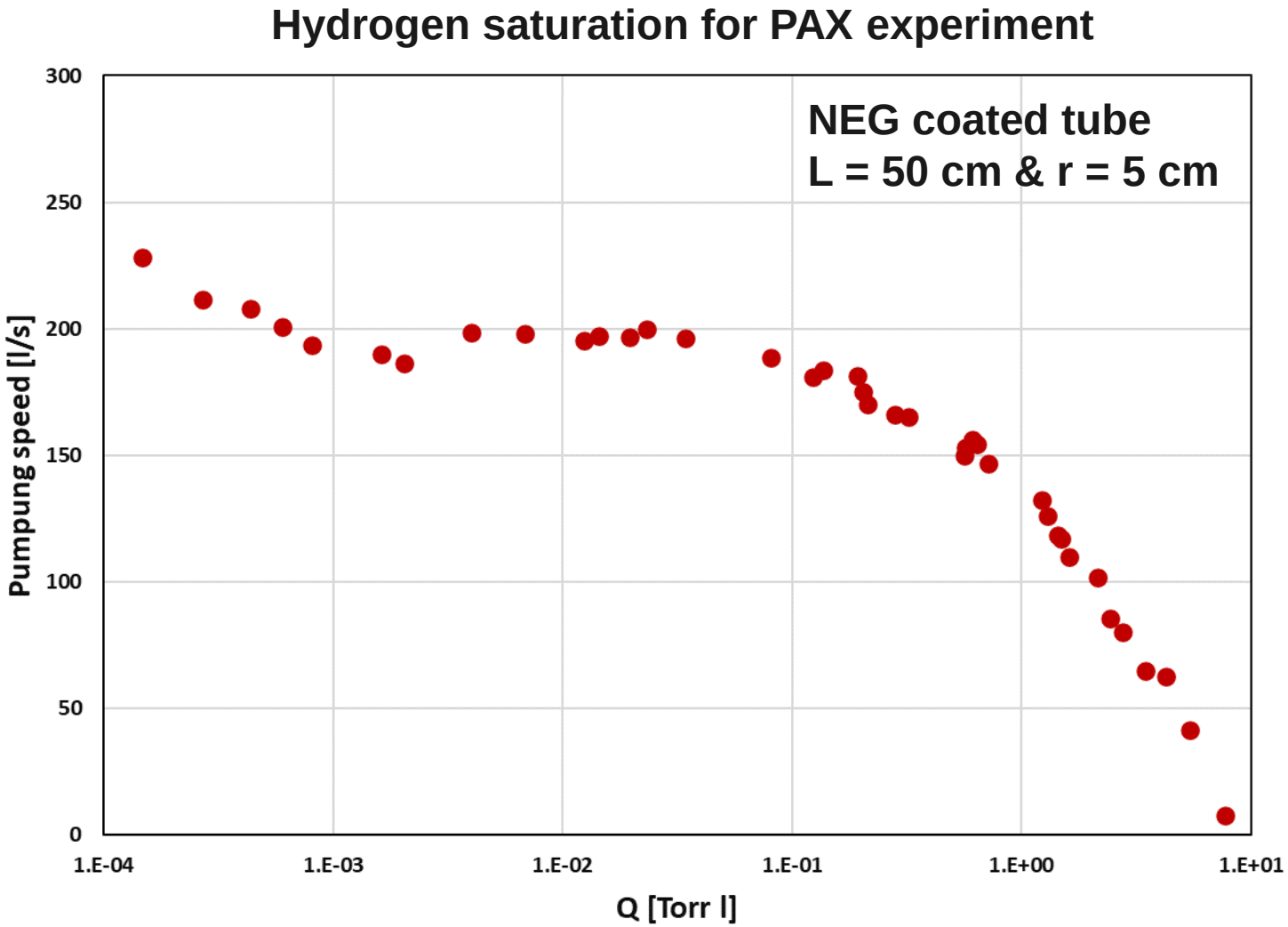
- Specific dimensions and gauge locations must be respected
- Orifice with conductance C separating 2 chambers

$$S = C\left(\frac{P_2}{P_1} - 1\right)$$

Monte Carlo computations on molecular flow in pumping speed test domes E. Fischer, H Mommsen - Vacuum June 1967 p 309-315

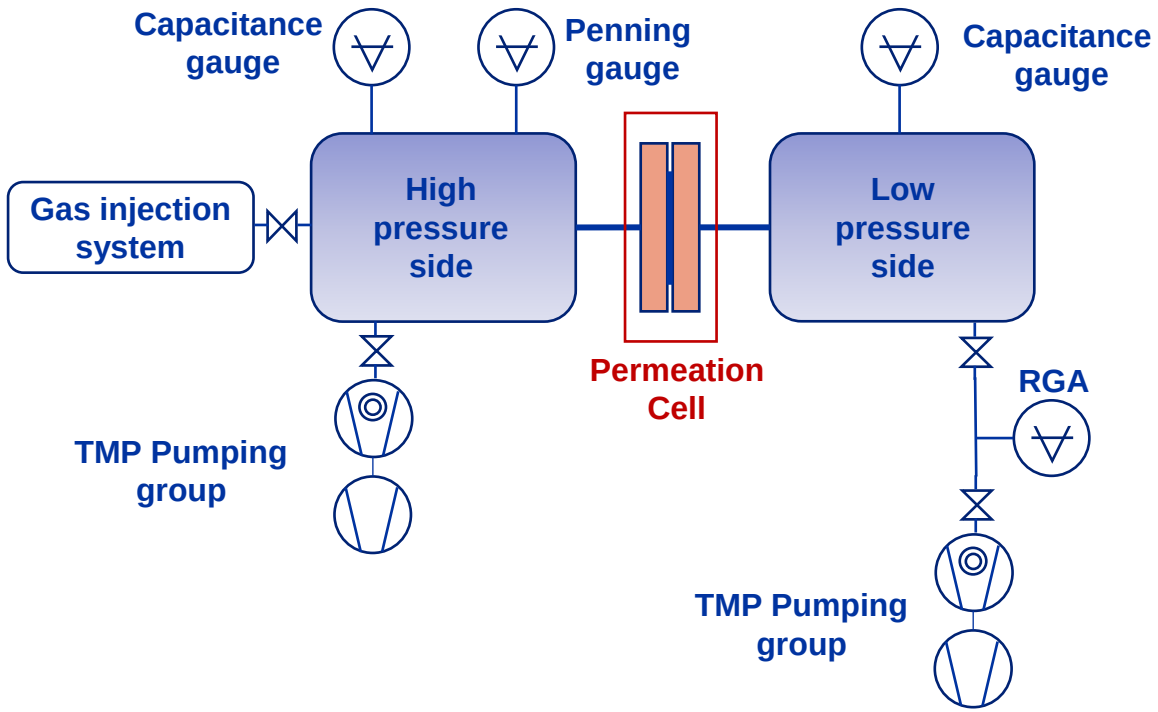
Pumping speed: Fischer-Mommsen dome





Monte Carlo computations on molecular flow in pumping speed test domes E. Fischer, H Mommsen - Vacuum June 1967 p 309-315

Permeation



- Permeation is a combination of dissolution and diffusion
  - ❖ Dissolution – Henry's Law

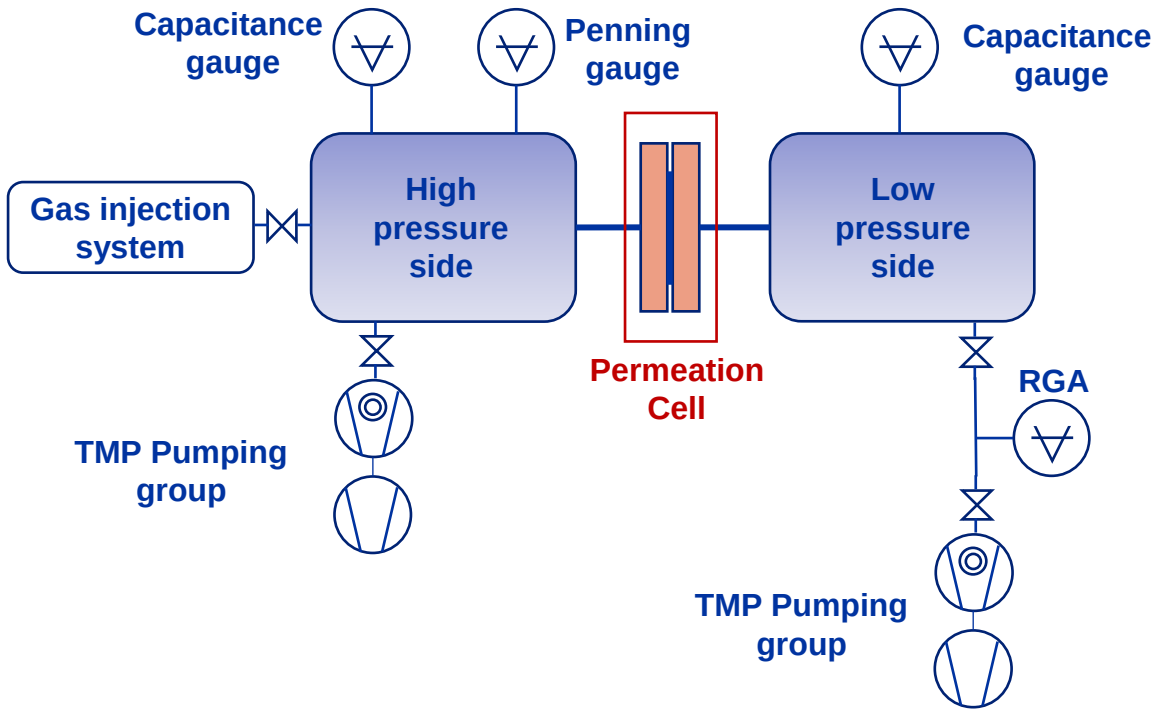
$$C = sP^n$$

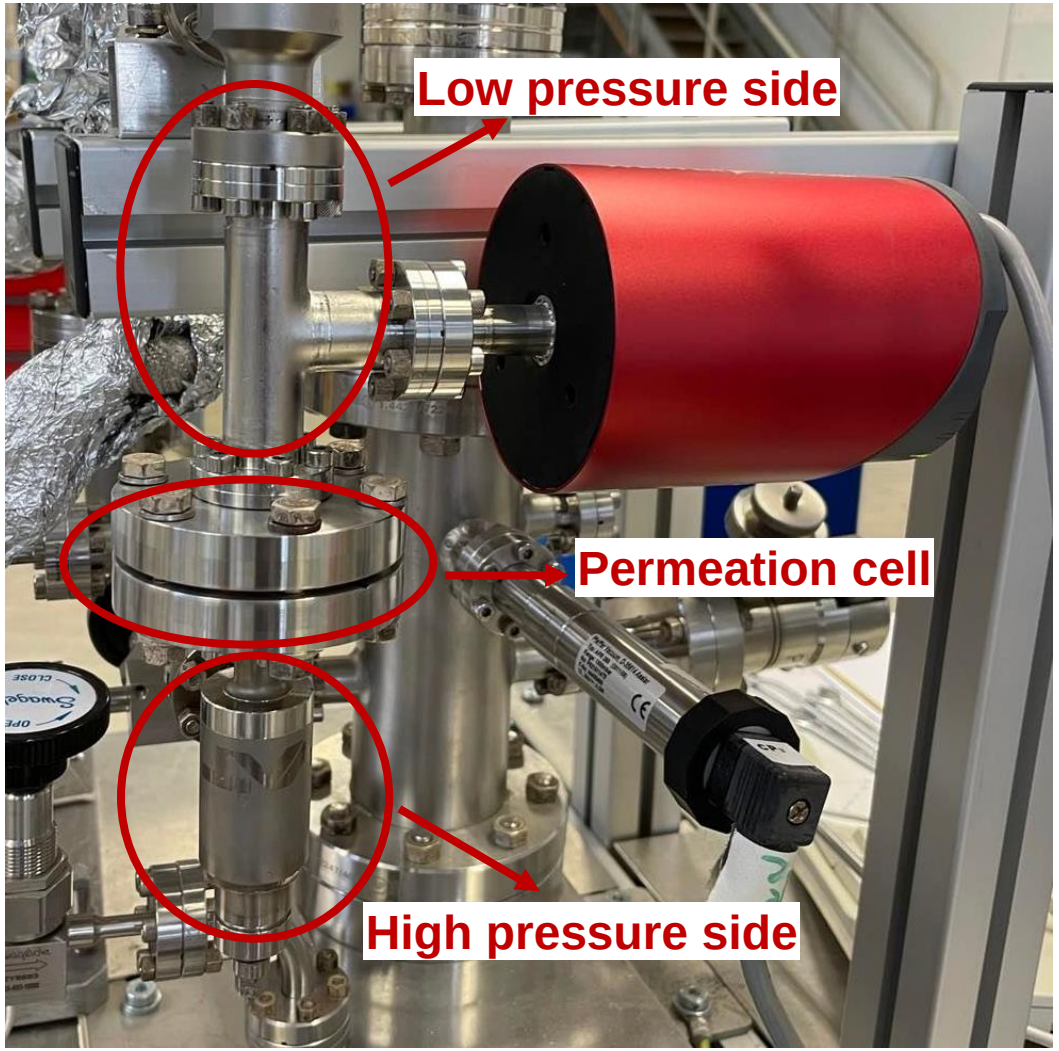
  - ❖ Diffusion – Fick's first Law

$$Q = -D\frac{dc}{dx}$$

- Dynamic mode
- Static mode

Permeation

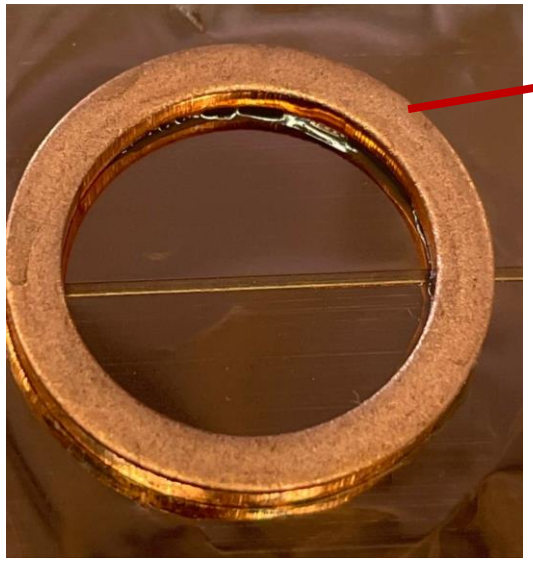

Capacitance gauge
Penning gauge
Capacitance gauge
Gas injection system
High pressure side
Low pressure side
Permeation Cell
RGA
TMP Pumping group
TMP Pumping group


Low pressure side
Permeation cell
High pressure side

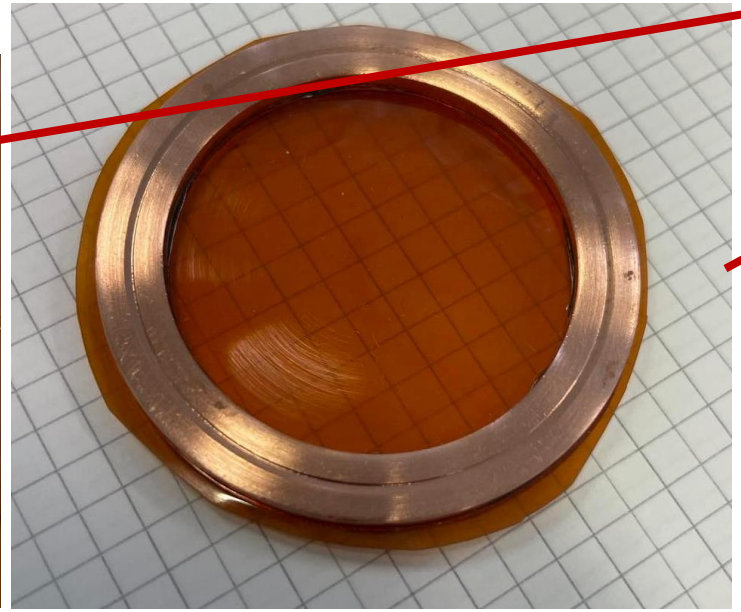
Permeation

Polymer glued in between 2 copper gaskets

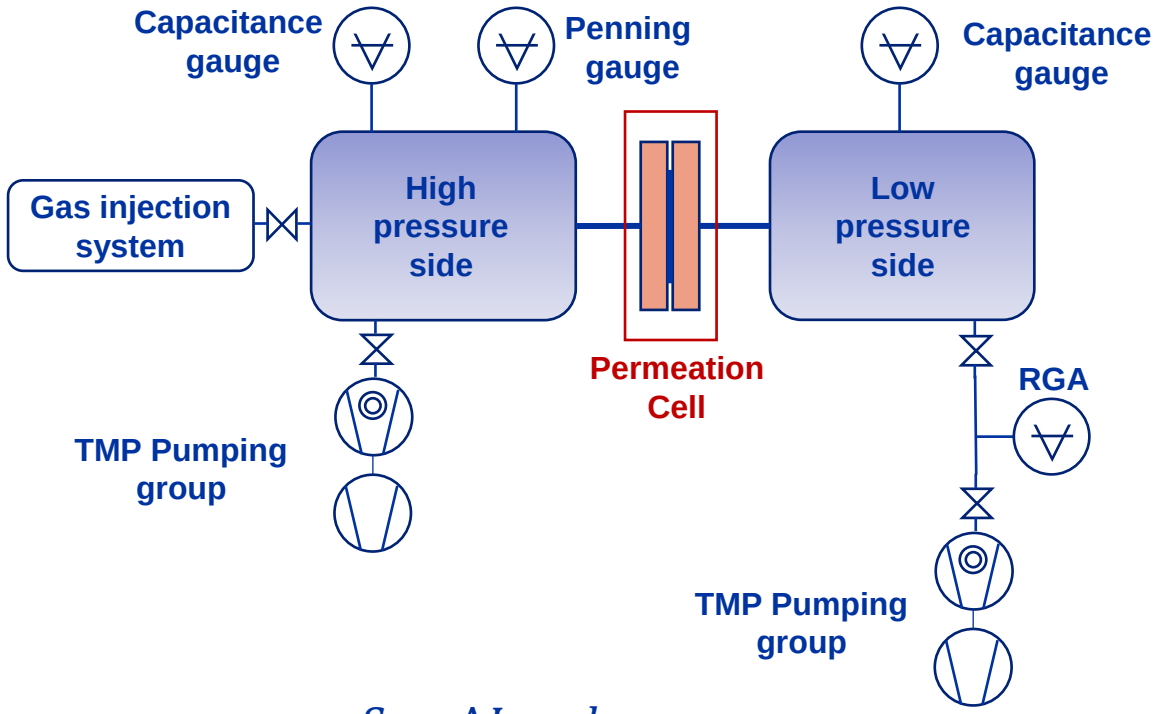
Kapton copper coated
DN16CF gaskets



Kapton
DN40CF gaskets

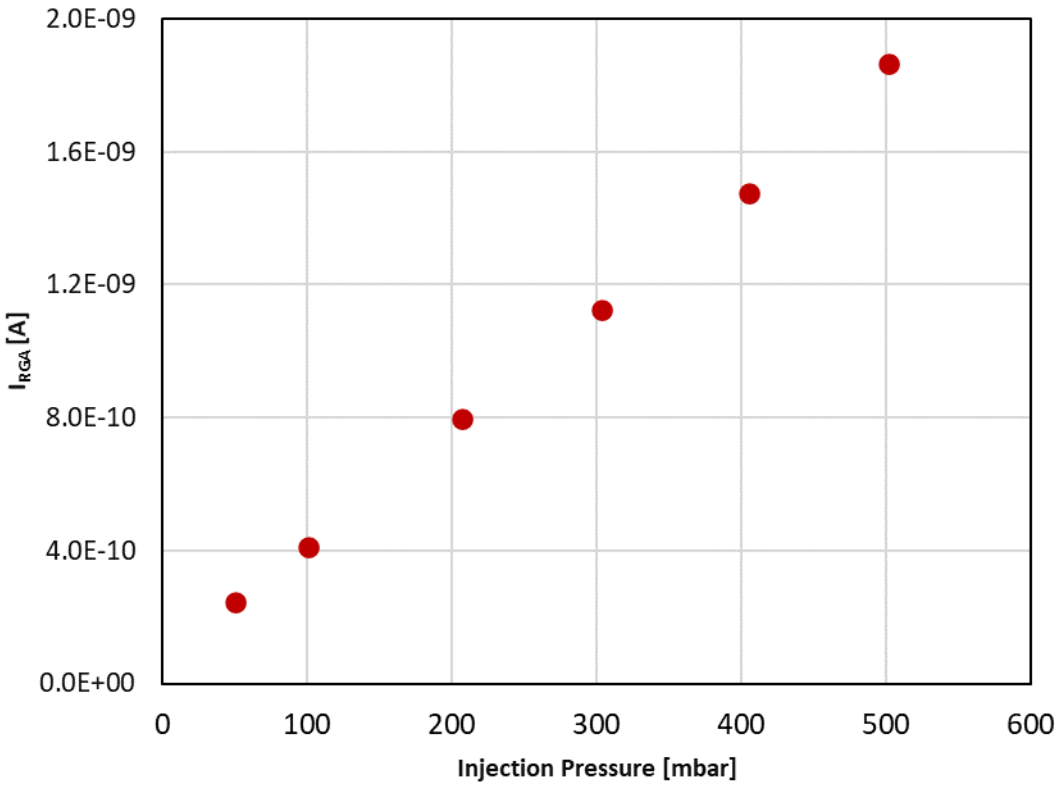


Permeation



$$Permeation = \frac{S_{eff}\ \Delta I_{RGA}\ l}{\alpha_{RGA}\ A\ P_{inj}}$$

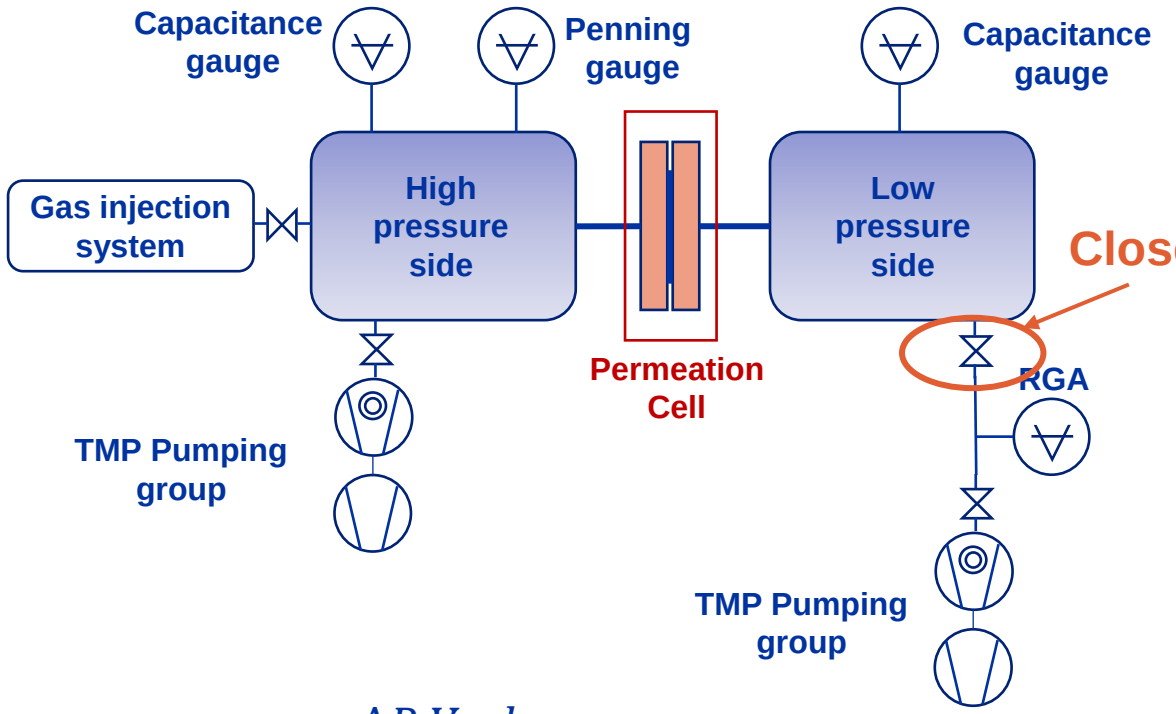
- **Dynamic mode**

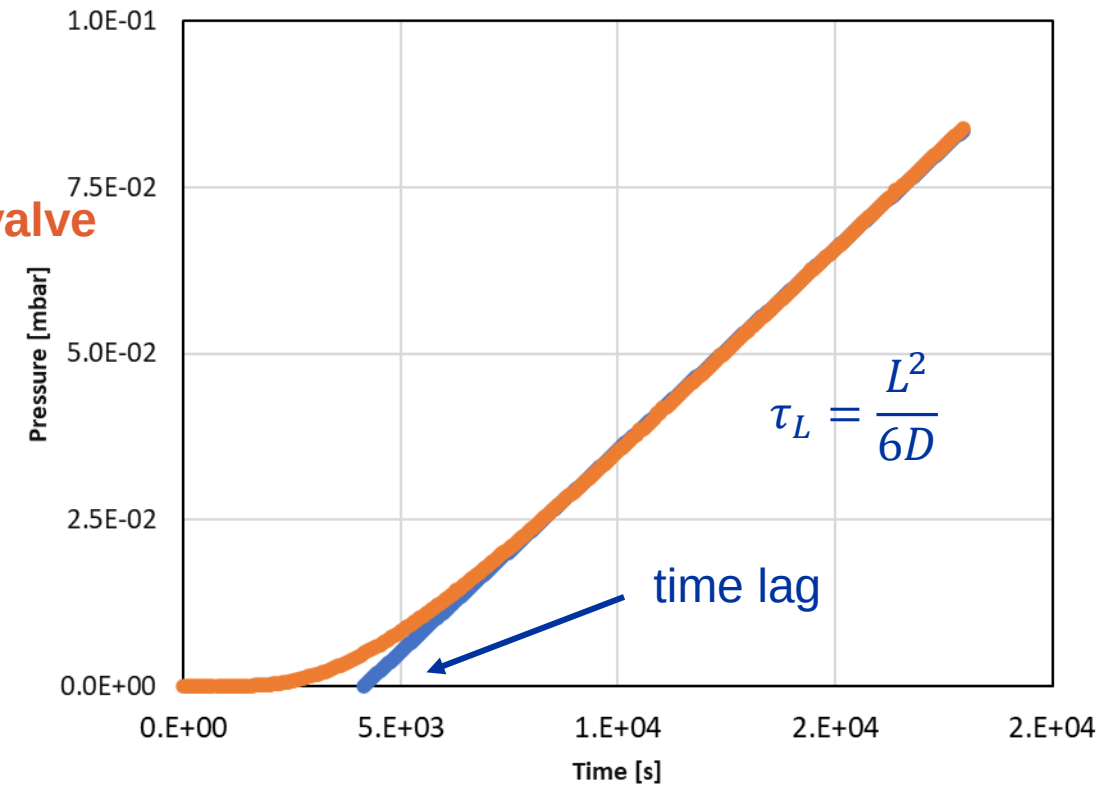


With: S_{eff} pumping speed - ΔI_{RGA} RGA signal for the injected gas - l sample thickness
α_{RGA} Calibration factor – A sample area - P_{inj} injection pressure

Permeation

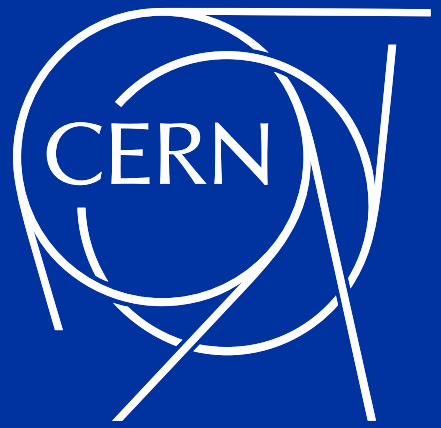
- **Static mode**





$$Permeation = \frac{\Delta P\ V_{lp}\ l}{\Delta t\ A\ P_{inj}}$$

With: $\Delta P/\ \Delta t$ slope at steady state - V_{lp} Volume low pressure side - l sample thickness
A sample area - P_{inj} injection pressure



Thank you for your attention