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Real-time contamination monitoring on mass standards stored in inert gas

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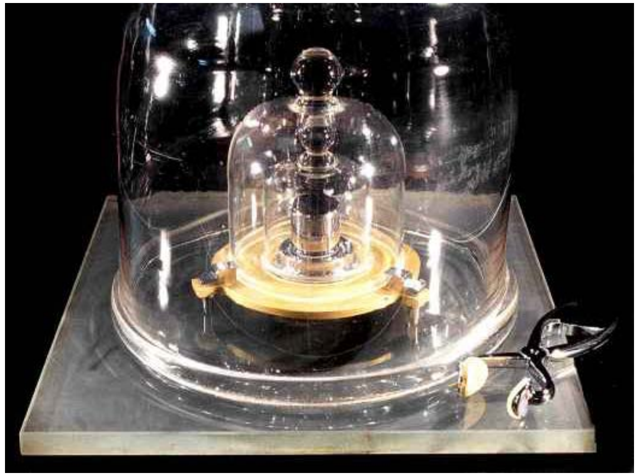
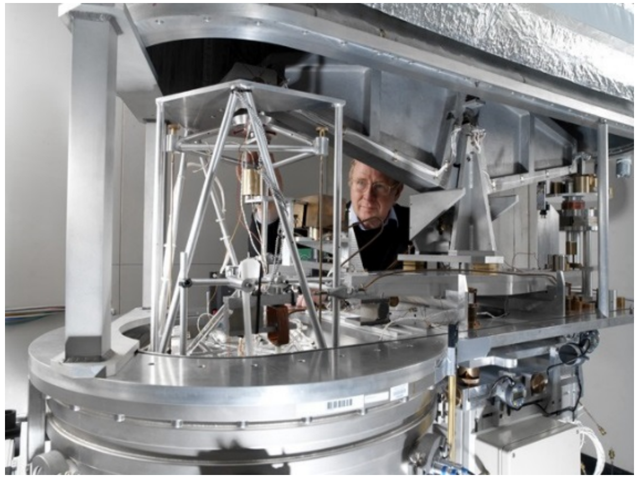
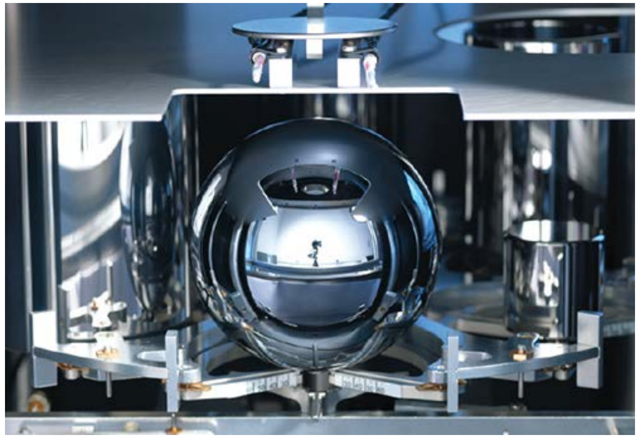
The new kilogram and the requirements for mass standards

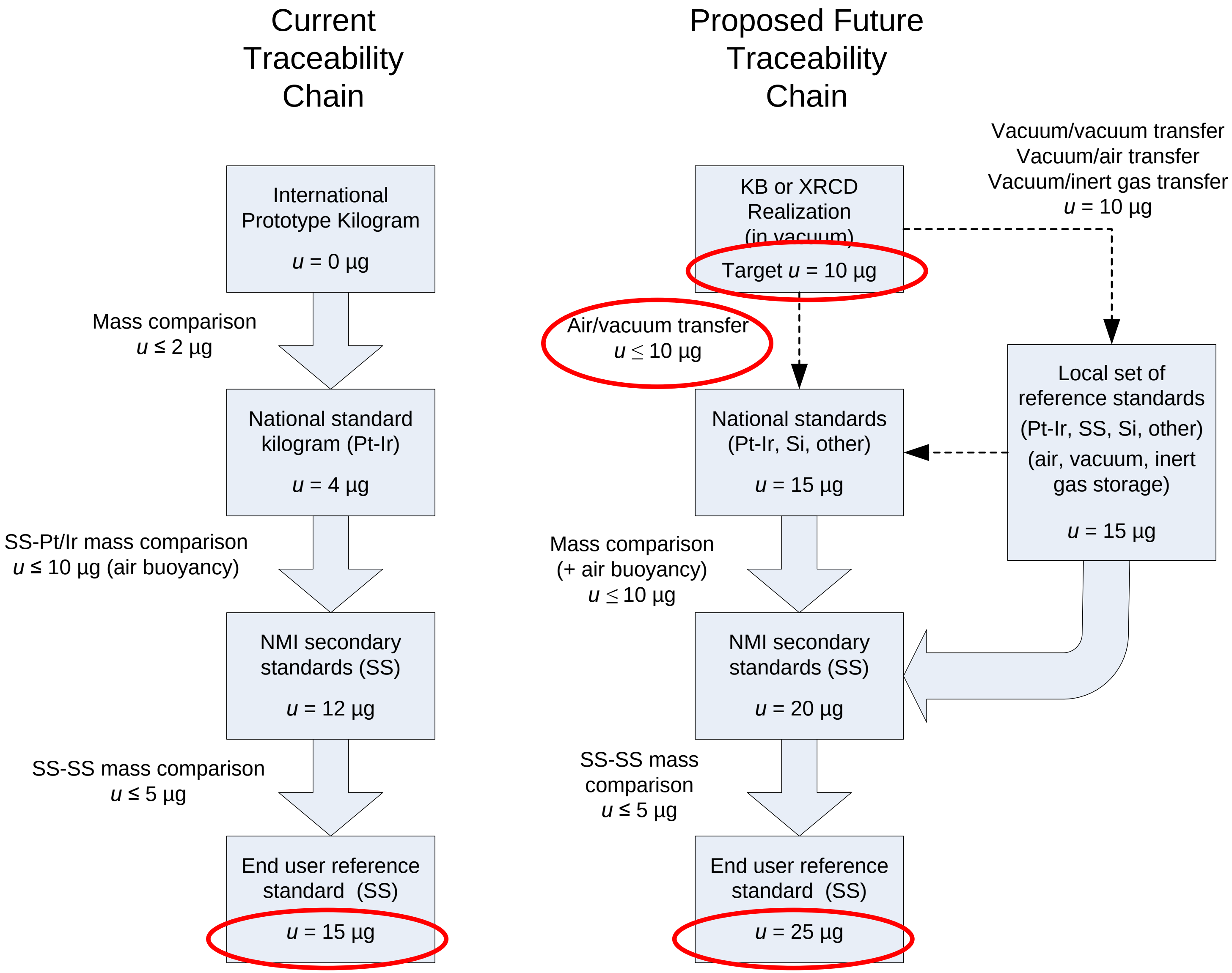
- In May 2019 the definition of the SI unit of mass will change from the mass of the IPK
- BUT current realisation experiments do not agree (as demonstrated by submissions to CODATA 2016 value for the Planck constant)
- SO until the realisations can be show to agree a consensus value will be used for the kilogram
- THUS the stability of mass artefacts will be even more crucial in maintaining the mass scale at an international and national level



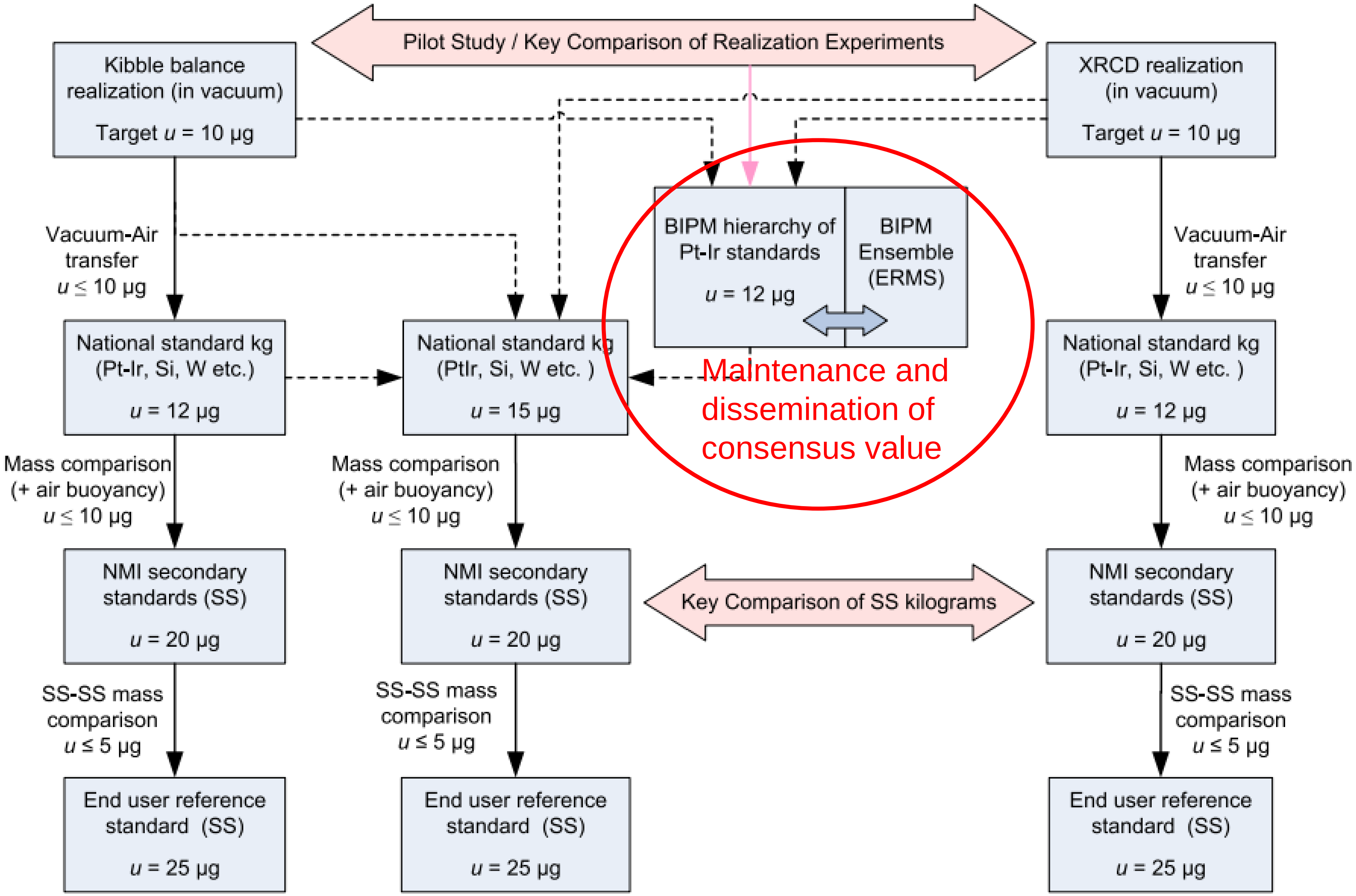




Old and new traceability (simple)

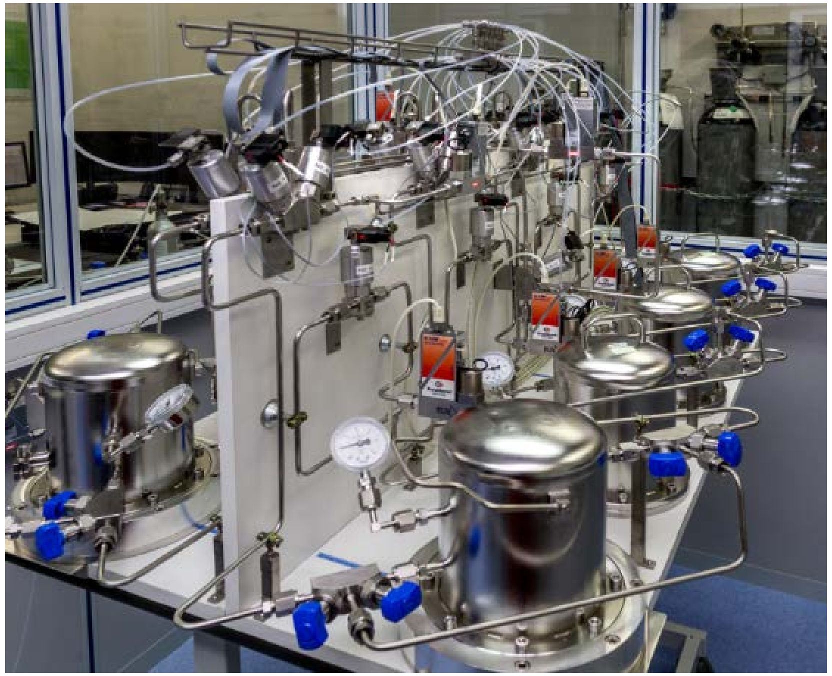


Old and new traceability (practical)



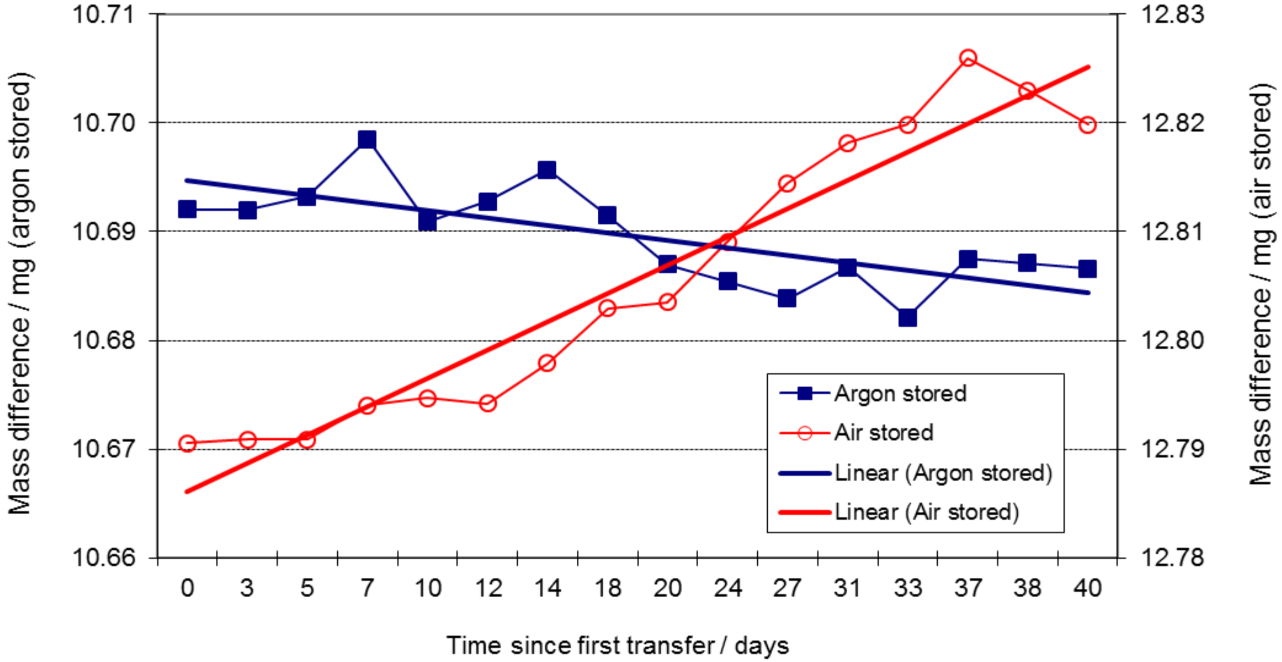
Storage of primary mass standards for long-term stability

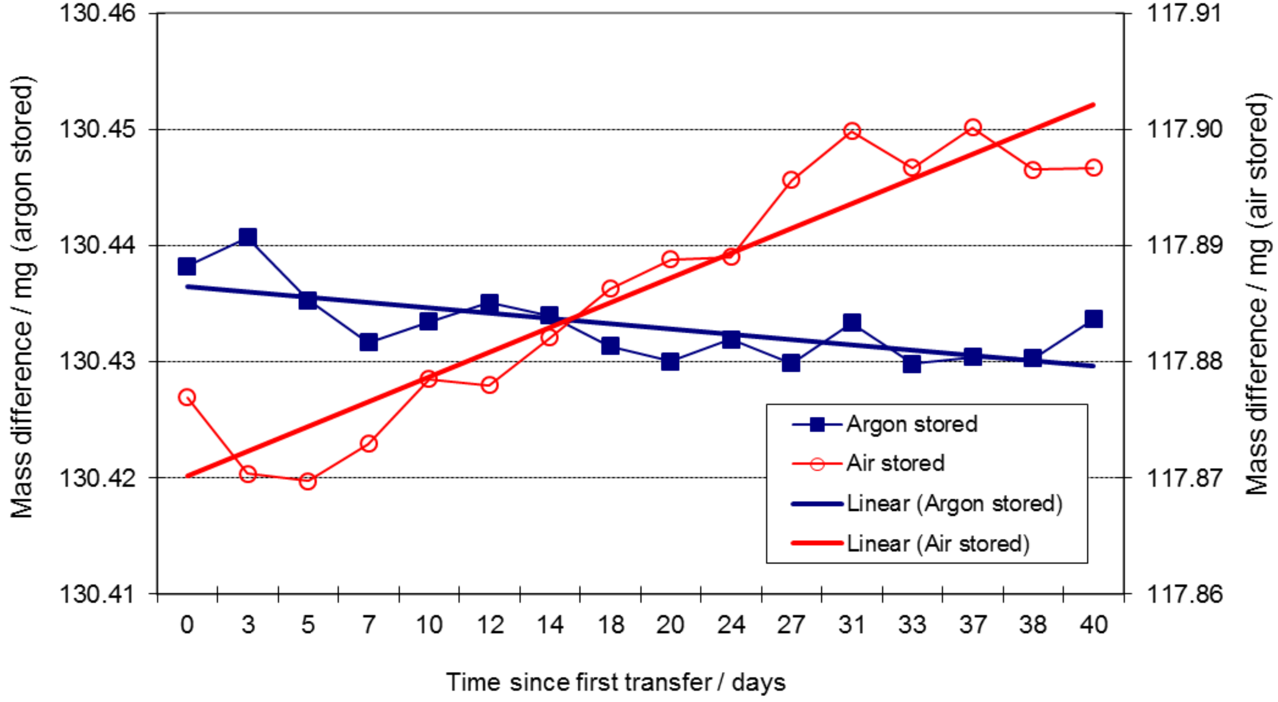
- The stability of primary standards is critical to the maintenance of the mass scale until the realisation experiments are reliable and agree
- The BIPM Platinum-Iridium working standards will “hold” the reference value for the ongoing Key Comparisons of realisation experiments
- NMIs will also see a greater requirement for the stable storage of mass standards



Previous work on inert gas storage

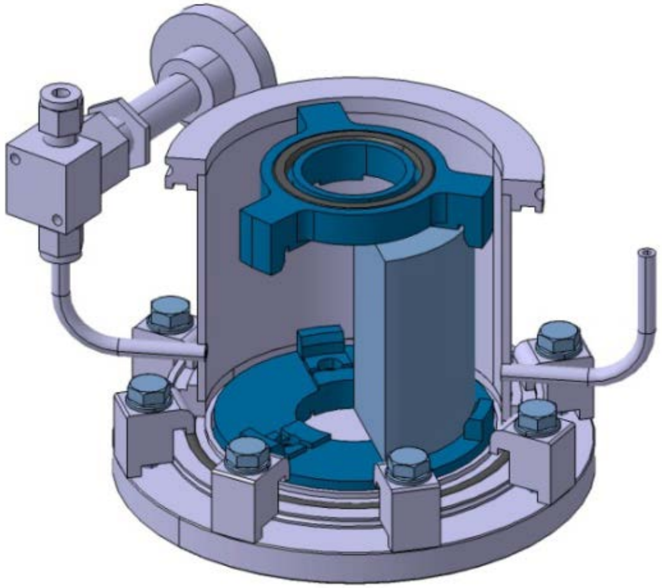
- Silicon and SS weights stored in air or inert gas. Mass measured in vacuum
- Artefacts stored in air show increase in mass due to surface contamination
- Artefacts in argon show slight decrease in mass
- Care with handling and transfer of artefacts (to balance) is critical in maintaining stability



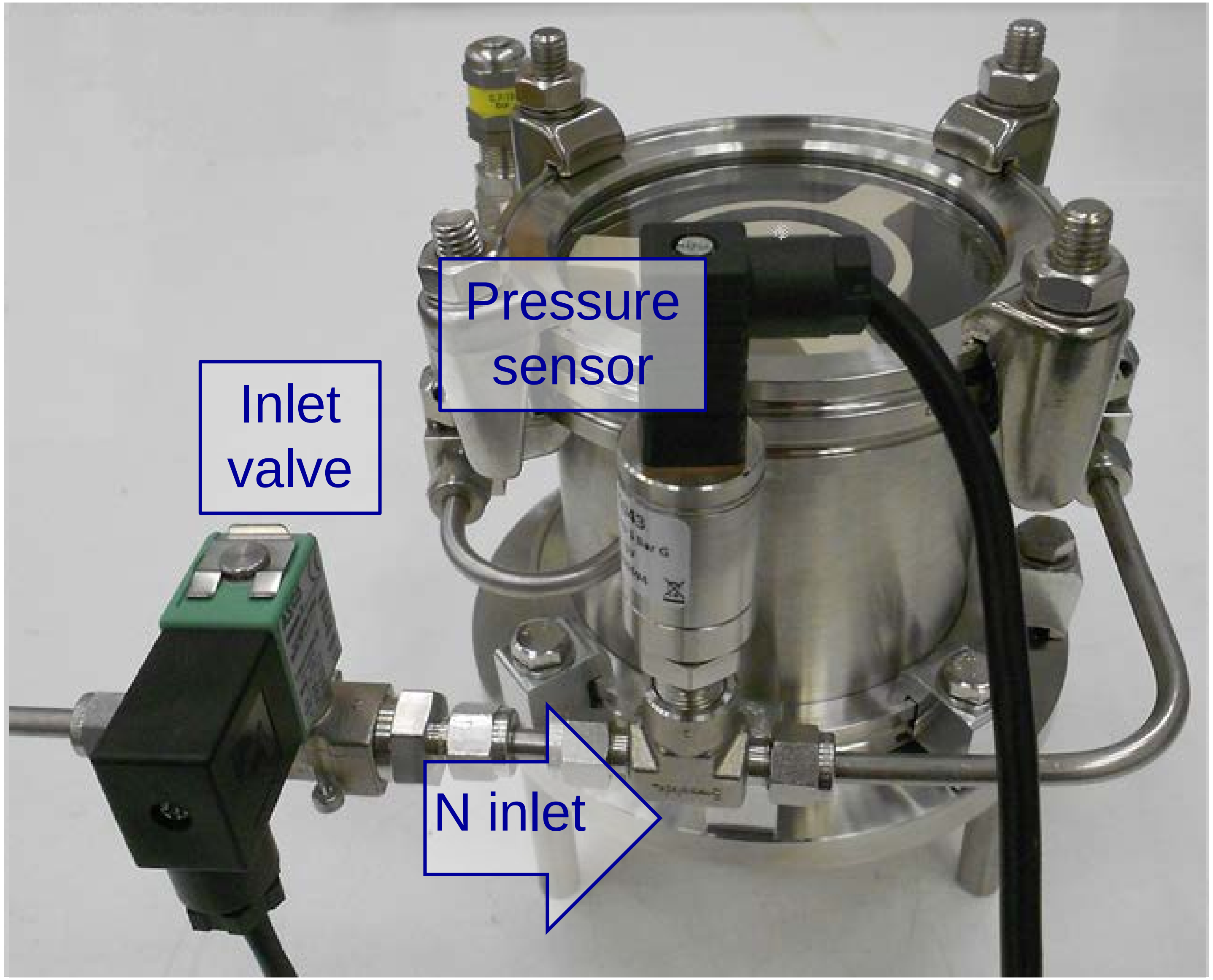


Mass storage enclosure

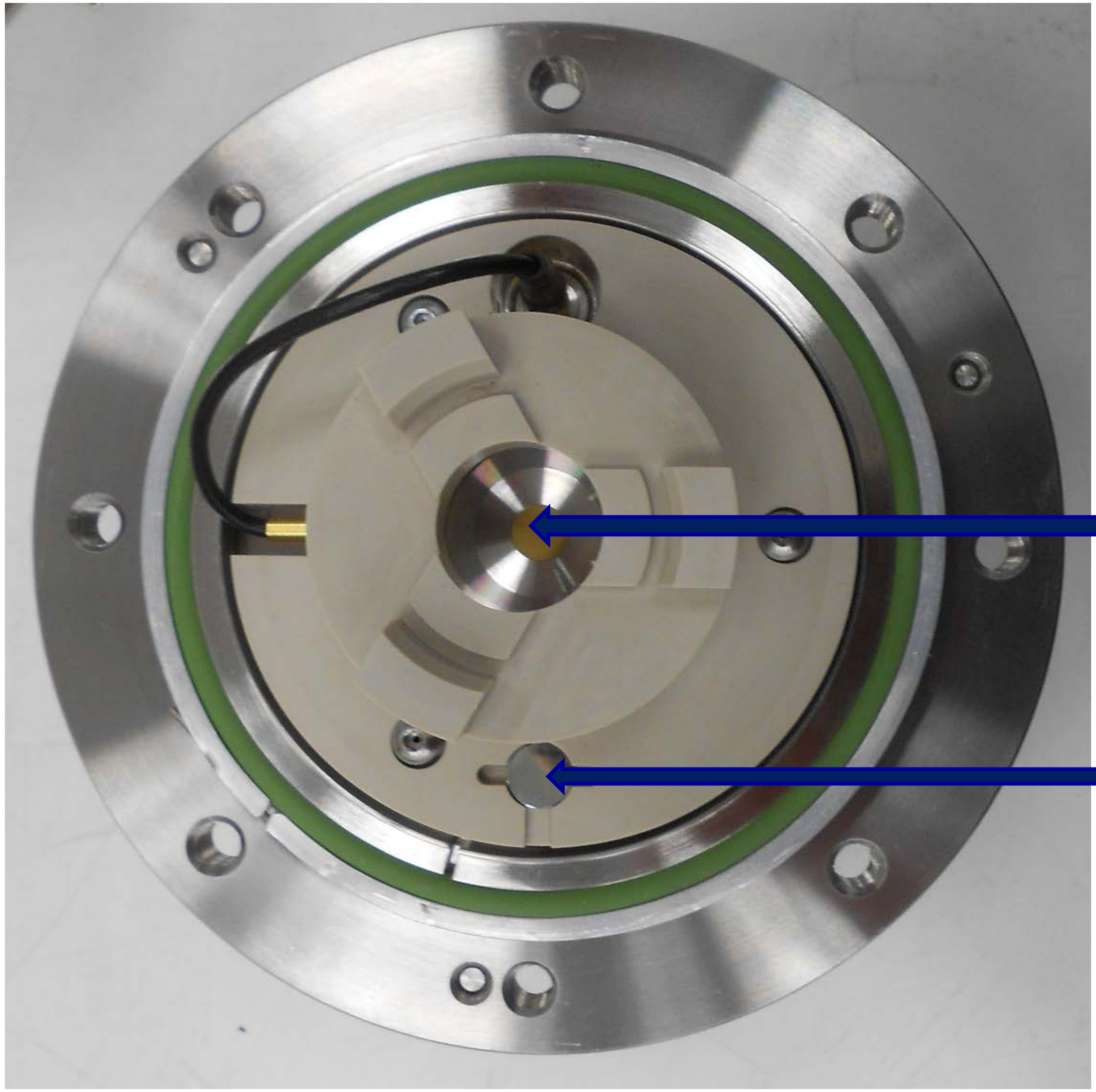
- Storage in nitrogen at positive pressure between 10% and 20% above atmosphere
- Pressure monitored via calibrated gauge
- Surface layers characterised by X-ray photoelectron spectroscopy (XPS) before and after storage
- Surface contamination monitored in real time using a quartz crystal microbalance (QCM)
- Computer control maintains and monitors positive pressure and records QCM output



Storage enclosure assembly



Storage enclosure – monitoring and surface analysis



QCM Sensor

XPS surface sample

Quartz crystal microbalance

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- Quartz crystal coated with (e.g.) aluminium or gold are resonated
- Frequency of resonance depends on (added) mass of overlayers
- Sauerbrey equation relates frequency change to mass change

$$\Delta f = -\frac{2f_0^2}{A\sqrt{\rho_q \mu_q}}\Delta m$$

f_0 – Resonant frequency (Hz)

Δf – Frequency change (Hz)

Δm – Mass change (g)

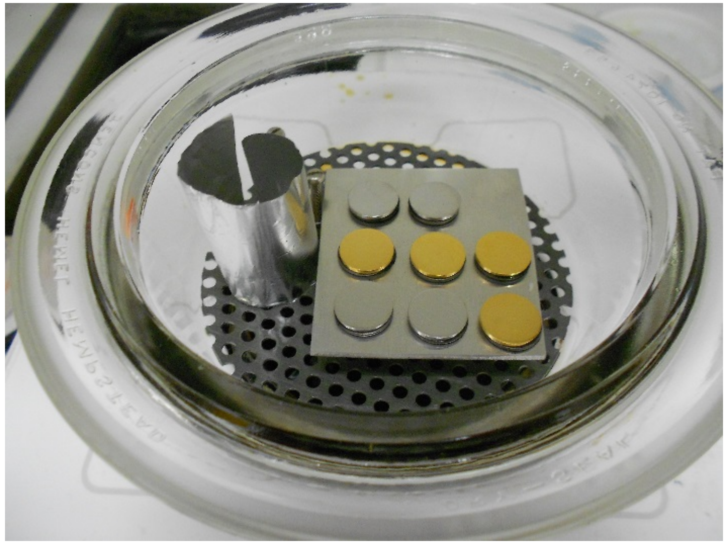
A – Piezoelectrically active crystal area (Area between electrodes, cm^2)

ρ_q – Density of quartz (ρ_q = 2.648 g/cm^3)

μ_q – Shear modulus of quartz for AT-cut crystal (μ_q = 2.947x10^{11} g·cm^{-1}·s^{-2})

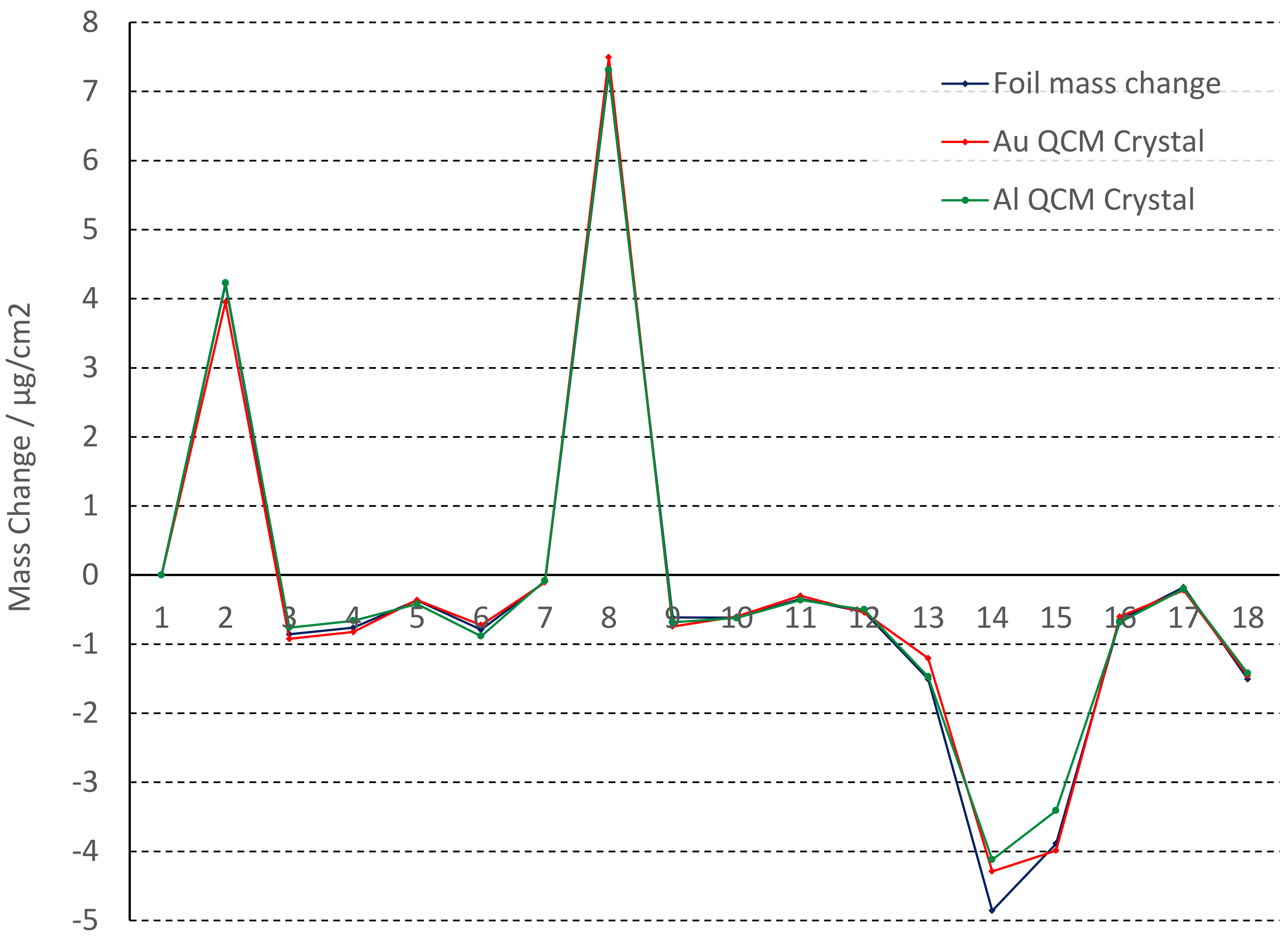
QCM calibration

- QCM resolution 1 Hz (approximately equivalent to 0.05 μg/cm^2)
- Check how well the Sauerbrey equation holds for non-bonded overlayer (e.g. H-C contamination)
- Artificial contamination of foil, crystals and surface samples with oil (vapour) at 80 °C
- Measurements of;
  - QCM frequency change
  - Change in mass (foil)
  - Change on overlayer thickness (XPS)
  - Overlayer composition (XPS)



Comparison of mass changes

- Foil mass change measured gravimetrically
- Change in resonant frequency of QCM crystals (with nominally the same contamination) measured
- Good agreement between QCMs frequency change and (gravimetric) foil contamination



Response of QCM (with Al coated crystal) to mass change

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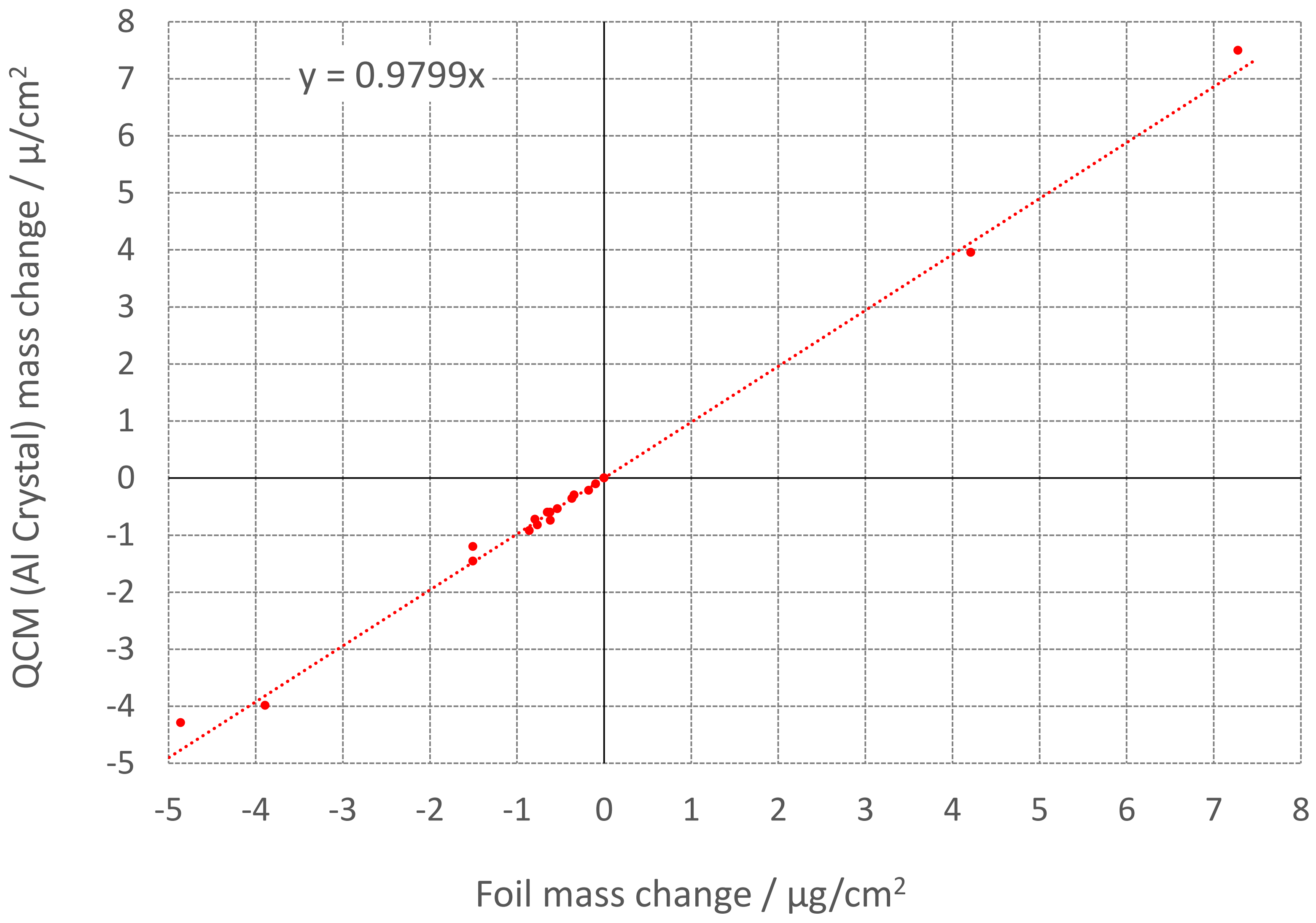
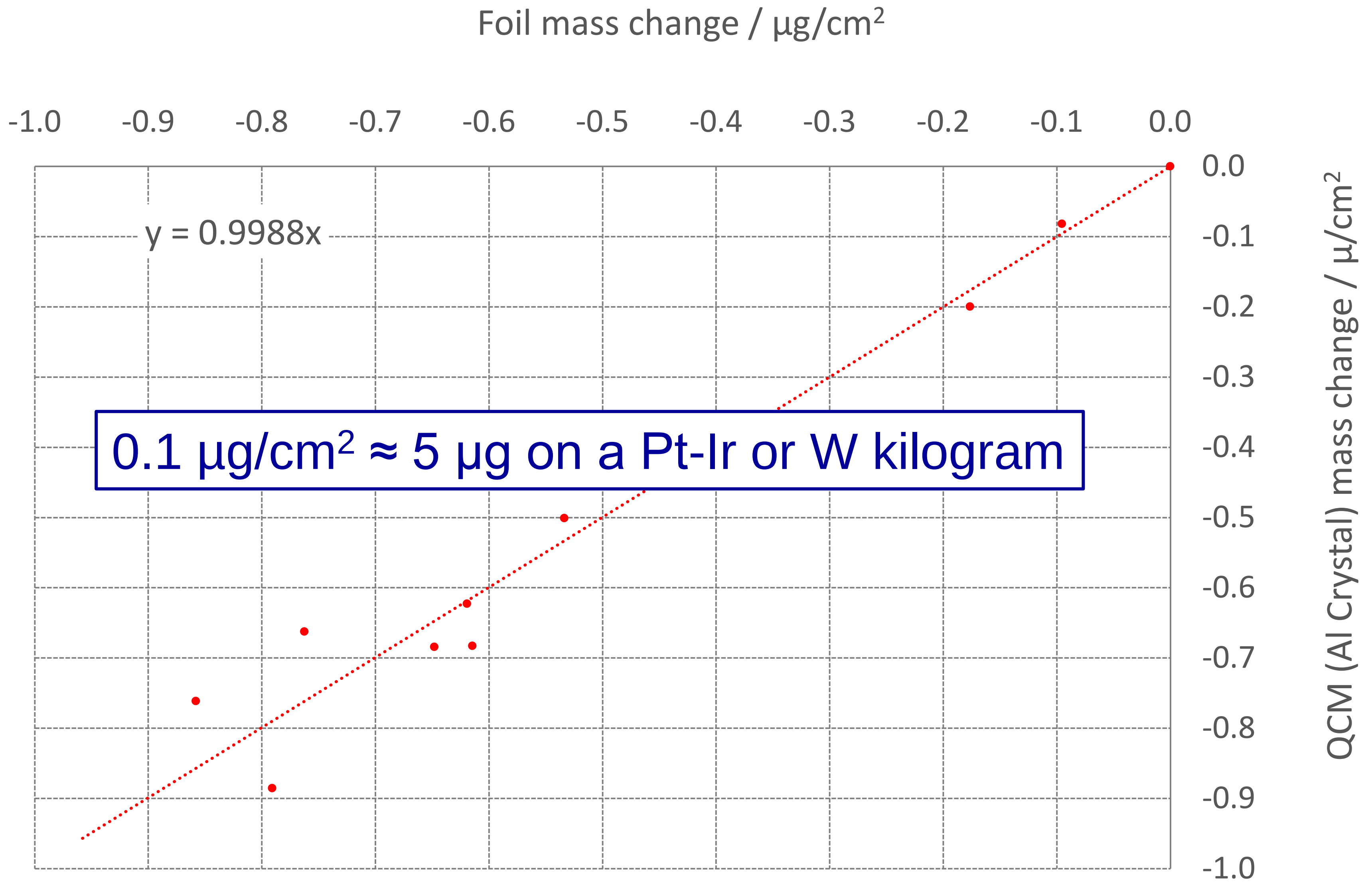
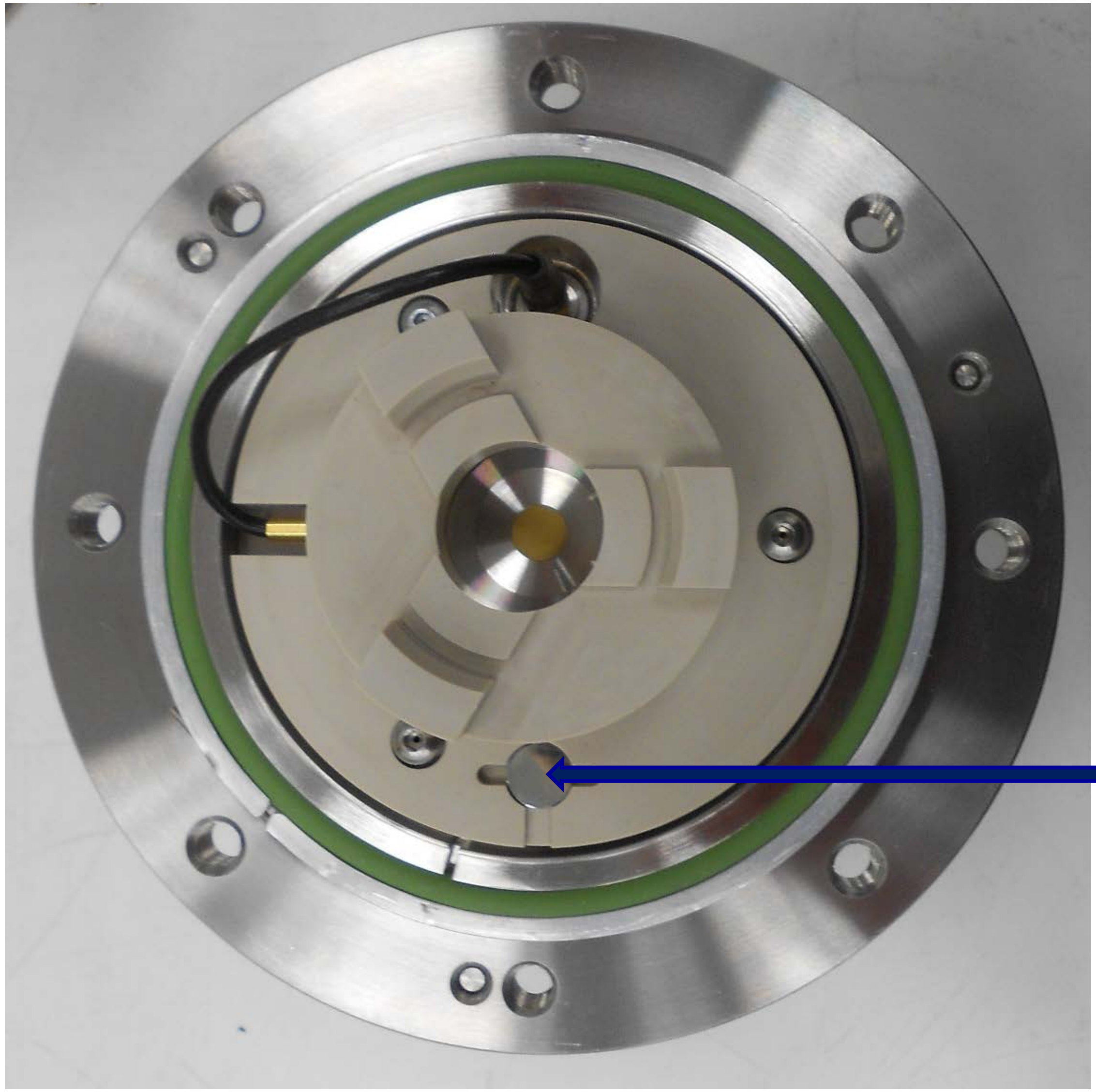


Response of QCM (with Al coated crystal) to mass change



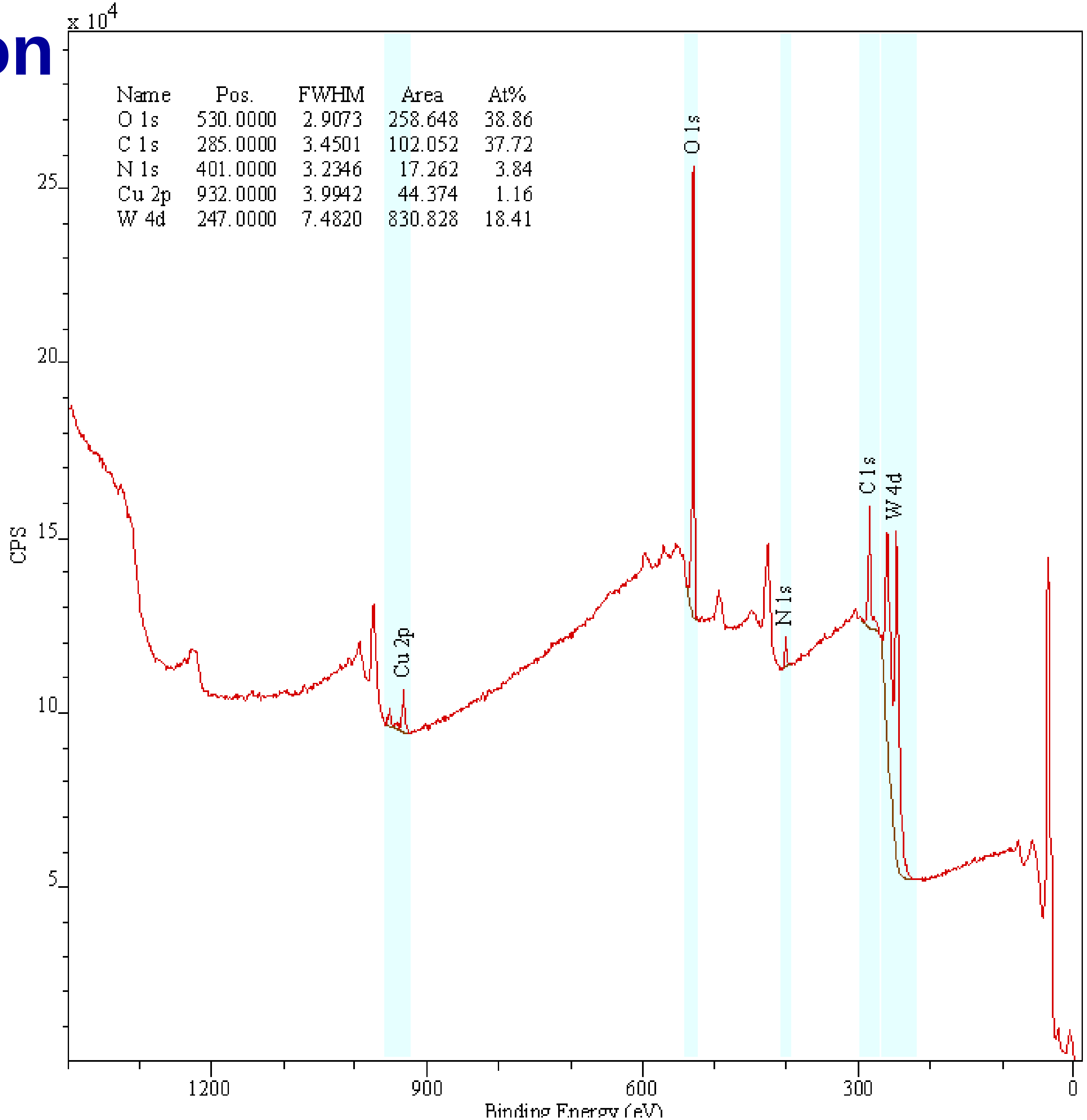
Storage enclosure – monitoring and surface analysis



XPS surface sample

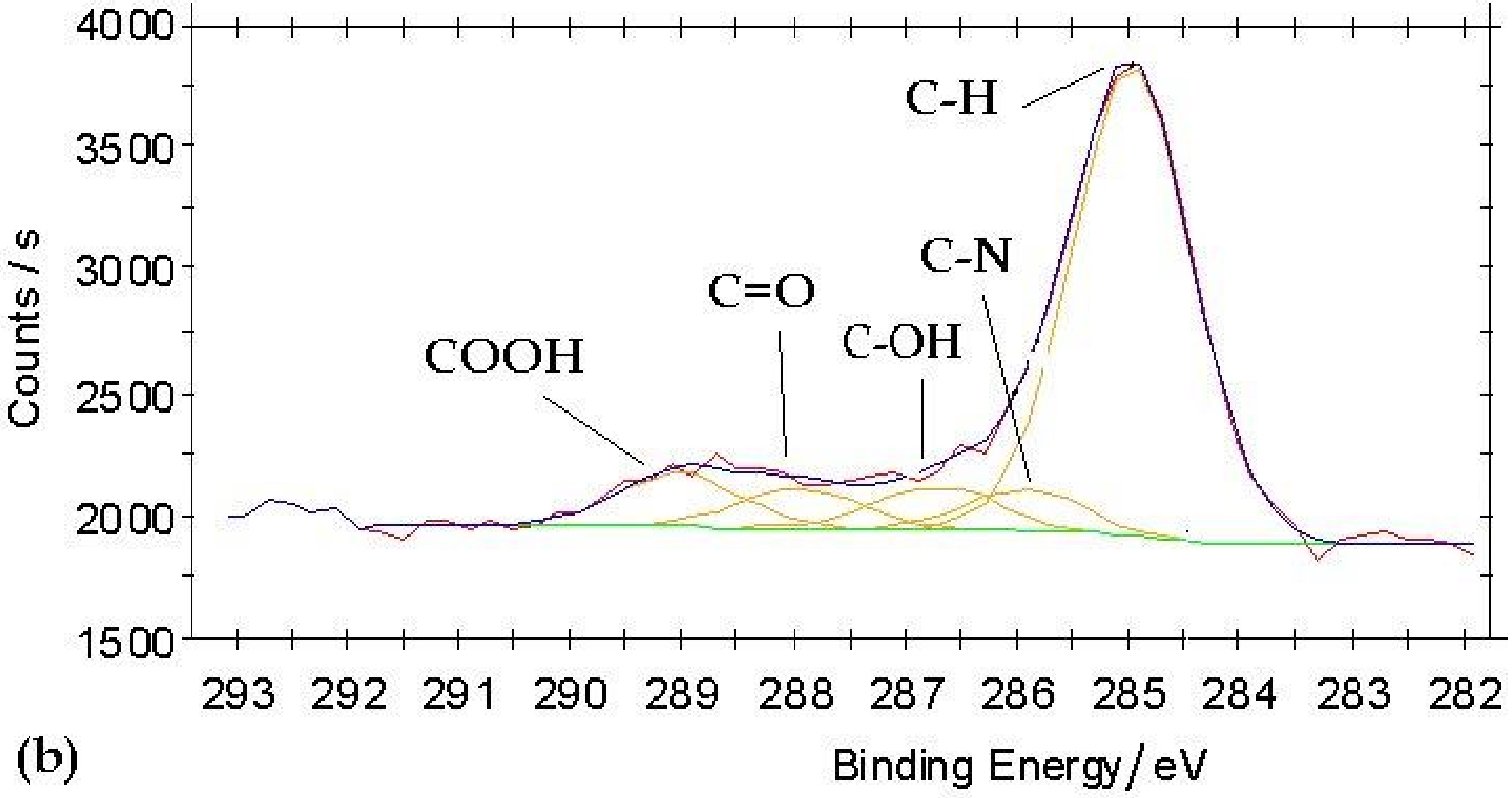
XPS surface characterisation

- Survey scan identifies the elements present (based on their binding energies)
- Scan for tungsten (W) sample
- Shows carbon and oxygen peaks (expected) but also copper (from polishing?)



XPS surface characterisation

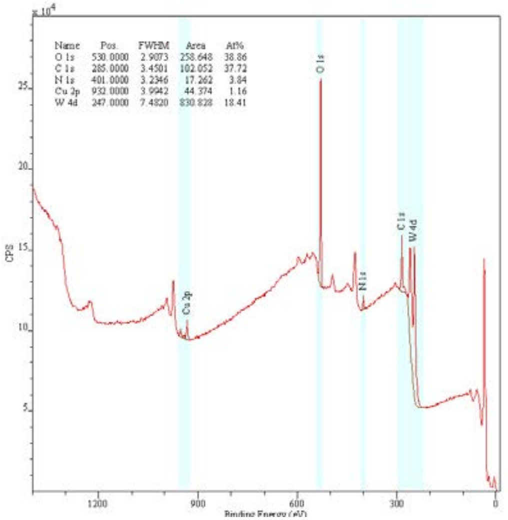
- Narrow scan gives more information – here details of the carbonaceous overlayer
- Pre- and post-storage measurements will show changes in surface chemistry (what sort of contamination has been accreted)



(b)

Summary

- Careful storage of mass standards is critical for;
  - Stability of national standards at NMIs
  - Maintenance of the mass scale between key comparisons of realization experiments
  - Continuity of access to the mass scale disseminated from (individual) realizations
- Storage in inert gas has been shown to improve the medium to long-term stability
- A relatively simple apparatus for storage of mass standards in inter gas has been developed
- Real time monitoring of surface contamination by QCM (Sauerbrey equation applied)
- XPS can be used pre- and post-storage to validate QCM measurements and characterise surface accretion



Thank you for your attention

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