A tritium target for Ptolemy

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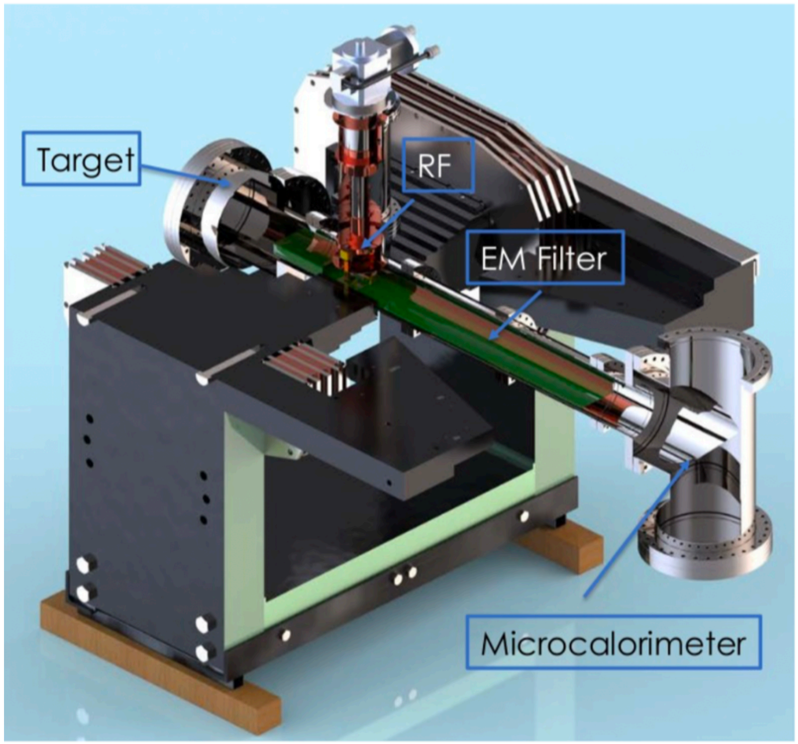
Ptolemy meeting in NYU
8th Nov 2023

A target for the Ptolemy demonstrator



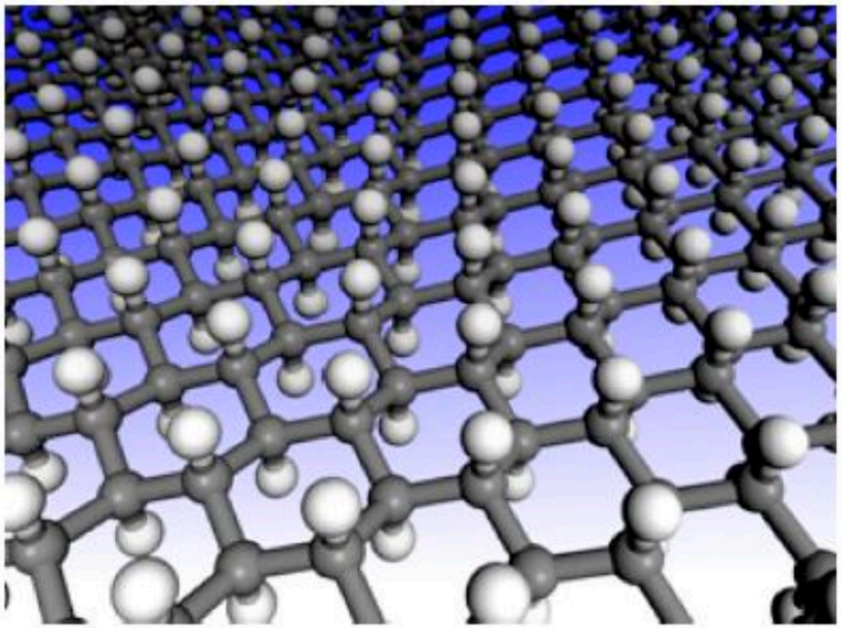
- Requirements
- **Atomic** tritium
- Sitting at well defined **position** in space
  (i.e. voltage difference to micro-cal accurately known)
- Easy to be handled
  - "Solid" tritium target
- **Thin** (reduce interaction with beta electron)
- **Stable** (tritium not released to the environment)

Solution: **use tritium uptake on carbon nanostructure as graphene**
Known not to be perfect but a very good start for the demonstrator

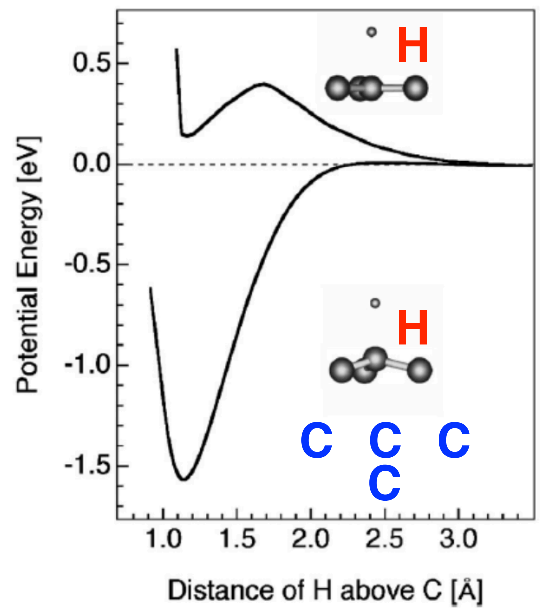
Hydrogen uptake on graphene

- Atomic H can 'pinch' the graphene **sp²** bonds towards
an **sp³** configuration

"chair" conformation

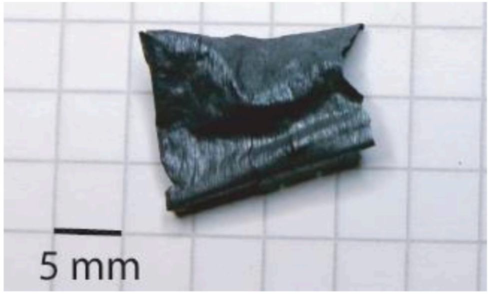


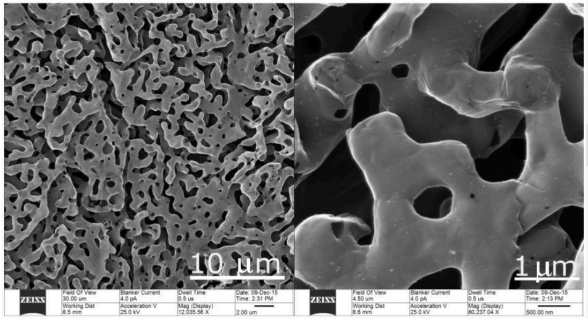
Sofo et alii, Phys. Rev. B **75**, 153401 (2007)

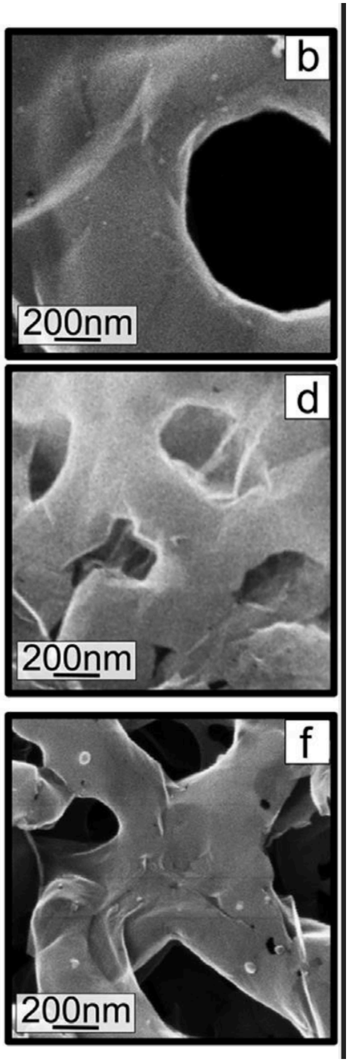


Formation of sp3 hybrid when H gets close to carbon atoms

The nano-porous graphene







- Grown out of a metal matrix then dissolved: **no substrate!**
  - Comes in the form of small flakes
- Self-standing single (or bi-) layer graphene
- Micrometer structure presents **curvatures** that helps the H chemisorption!

Demonstration of graphene hydrogenation

- Several attempts in the last years within Ptolemy
  - (Cold) plasma, low energy ions, **<0.2 eV atoms**

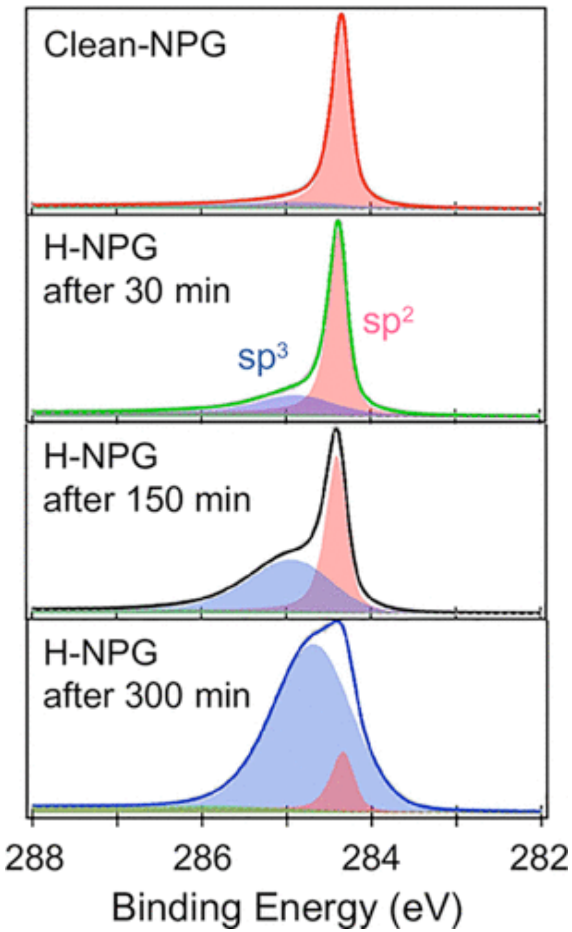
- Abdelnabi, M. M. S.et al. **Towards free-standing graphane: atomic hydrogen and deuterium bonding to nano-porous graphene**. Nanotechnology 2021, 32, 035707.
- Abdelnabi, M. M. S. et al. . **Deuterium Adsorption on Free-Standing Graphene.** Nanomaterials 2021, 11, 130.
- Zhao, F. et al. **High hydrogen coverage on graphene via low temperature plasma with applied magnetic field.** Carbon 2021, 177, 244–251.

A key point it to avoid introducing too many defect to graphene during hydrogenation: low kinetic energy preferable

Absence of substrate is crucial: C to hydrogen bond is not affected by graphene interaction with substrate.

X-ray spectroscopy (XPS) from C electrons

Nano Lett. 2022, 22, 7, 2971–2977



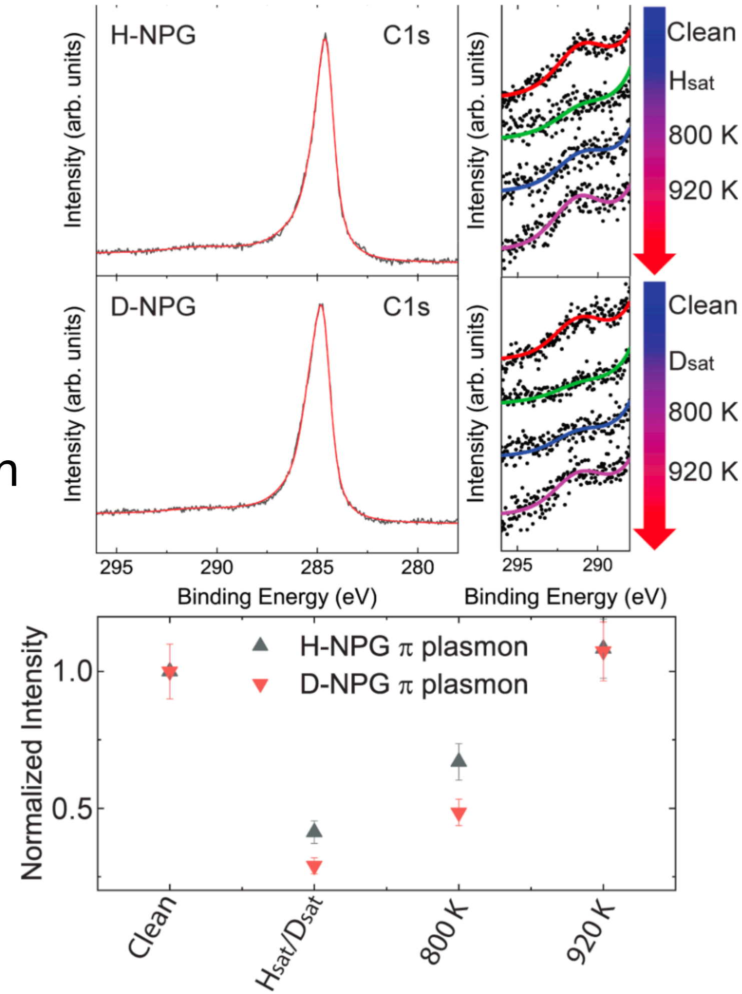
- In vacuum procedure
- Crack hydrogen molecule by heating the gas at T > 2000K: H atoms diffuse over the NPG
- Shine X-ray on the sample and detect the emitted photoelectron
- From the ph.e. kinetic energy accurately measure the **atomic binding energy.**
- **Interpret sp3 component as due to hydrogen C-H bond**

- Close to 100% uptake (every C atom has a H atom)
- Energy gap emergence: **graphAne** (semi-conductor)

Thermal stability

- H on graphene with a metallic substrate (e.g. Pt(111) was previously found to be **unstable** with **temperature**
  - 300 °C enough to desorb H
- We demonstrates **H (or D) on NPG stable** up to more than **600 °C**

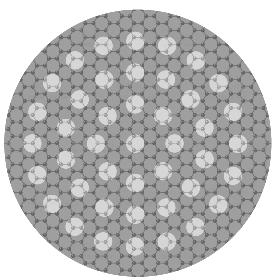
π-plasmon fingerprint of sp2 reduction



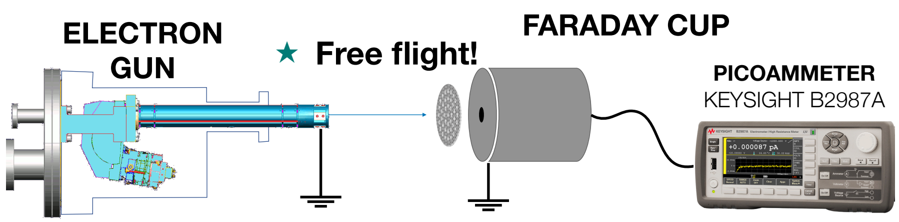
Electron transparency

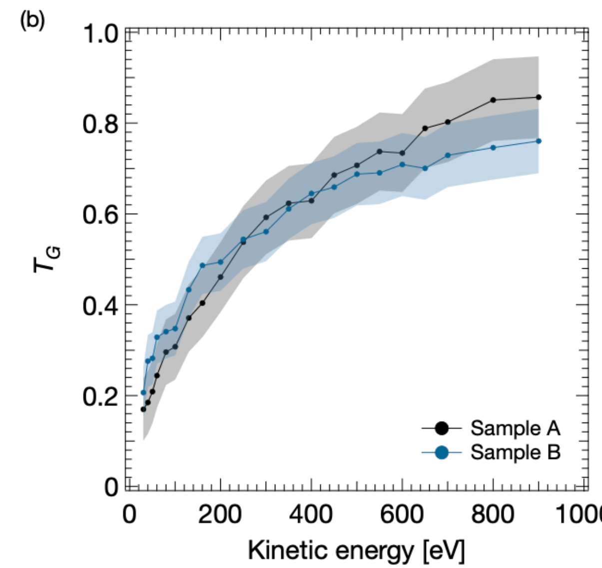
- Novel method based on current measurement through suspended graphene.
- Currently at E < 1 keV but can be extended

Apponi et al., Carbon (2023), https://doi.org/10.1016/j.carbon.2023.118502



Monolayer graphene on TEM grid





- Gained experience with manipulation and characterisation of single layer graphene (XPS, SEM microscopy, UV spectroscopy) for hydrogenation

Tritium on graphene

Hydrogen (and deuterium) share the **same chemistry** with tritium

still interesting in future to do spectroscopy
on graphene-tritium system (C-tritium bond)

Port the graphene hydrogenation technique
to **tritium storage on graphene**
(and other carbon nanostructure in future)

Start with **NPG** since it is self-standing and
proved to allow large uptake

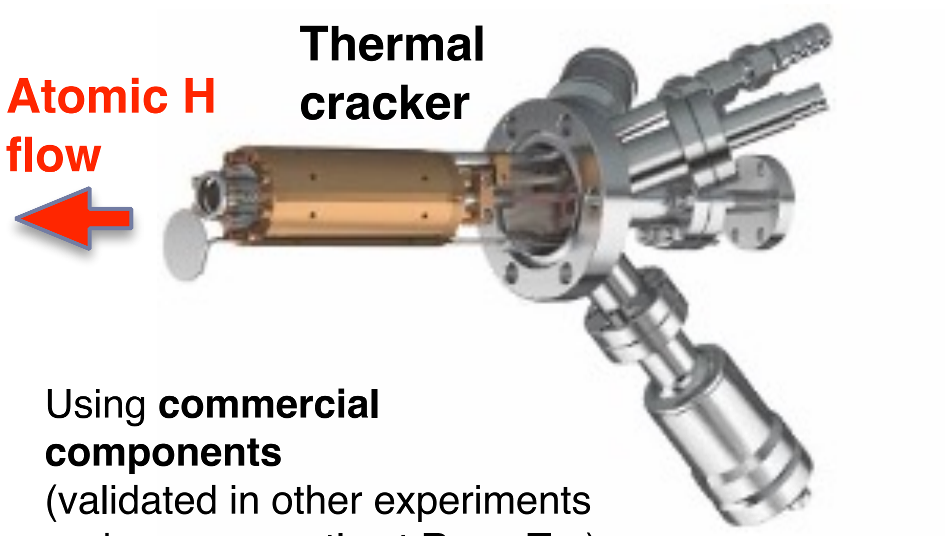
Assuming a mass density of NPG ~ 1/100 graphite with 100% T uptake
15 µg tritium can be stored in NPG flake equivalent to 4 GBq activity

Goals

- Have a **< 1 GBq solid atomic tritium** target
  - Less troubles with radio safety regulations
- Use carbon nanostructure as support
  - Well defined **position** in the apparatus, well defined **potential**
- Demonstrate the solid target is **stable** (i.e. no tritium release) at **room temperature**
  - To be certified according to radio-protection standards
- **Measure**
  - Radioactivity **activity**
  - band gap, resistivity
- First **beta spectrum** measurement
  - With solid state sensors, relatively poor energy resolution ~100 eV
  - With electron analyser (as in XPS) with retarding potential to select the end point of the spectrum

Plans for graphene target production

- Based on the work of C.Mariani et al. on NPG hydrogenation
  - Use **thermal cracking** (2400 K) of hydrogen molecule
    - Atomic thermal hydrogen flowing onto the sample



Using **commercial components**
(validated in other experiments and more recently at RomaTre)

Funded and procured by Princeton University

↓

Being **commissioned** in Roma

↓

Shipped to lab equipped to manage tritium

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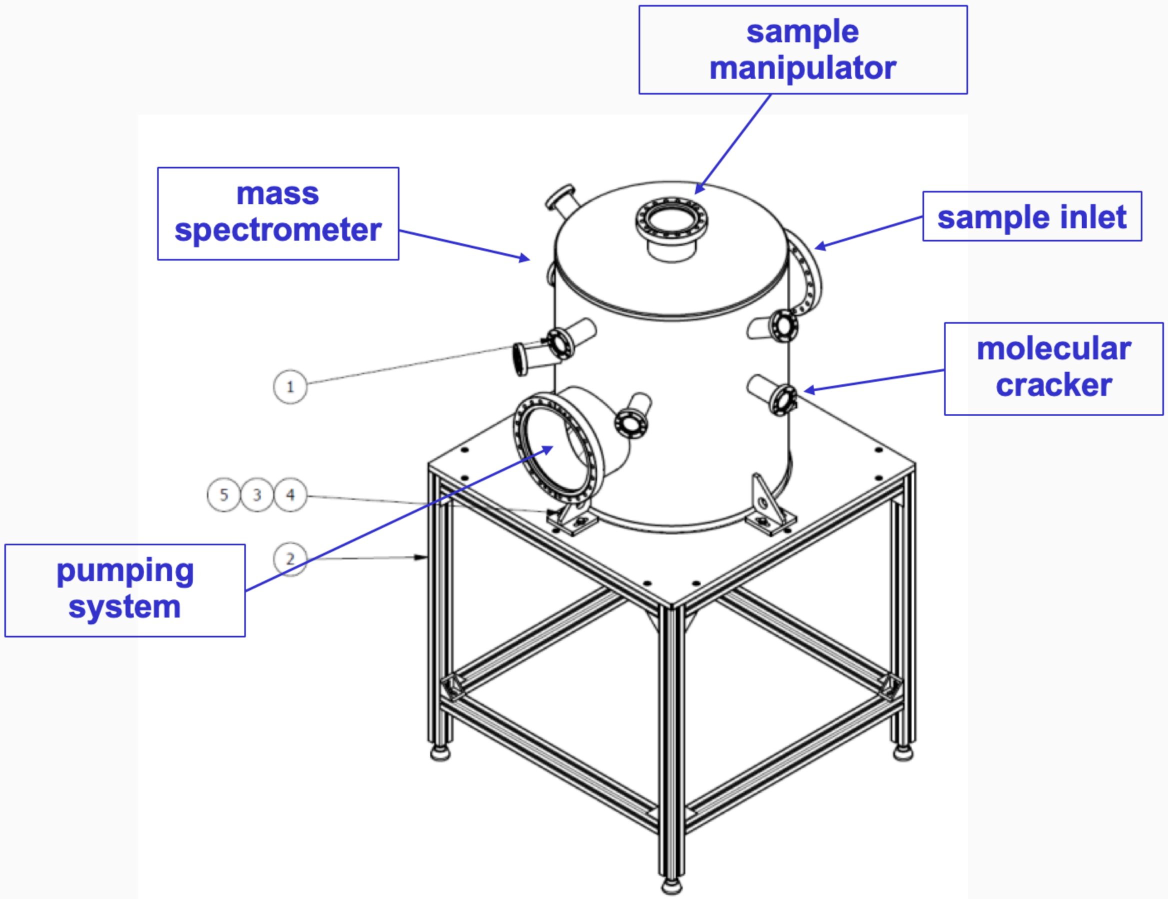
Tritiated samples back to Roma for radio-safety and beta spectrum measurements

UHV chamber



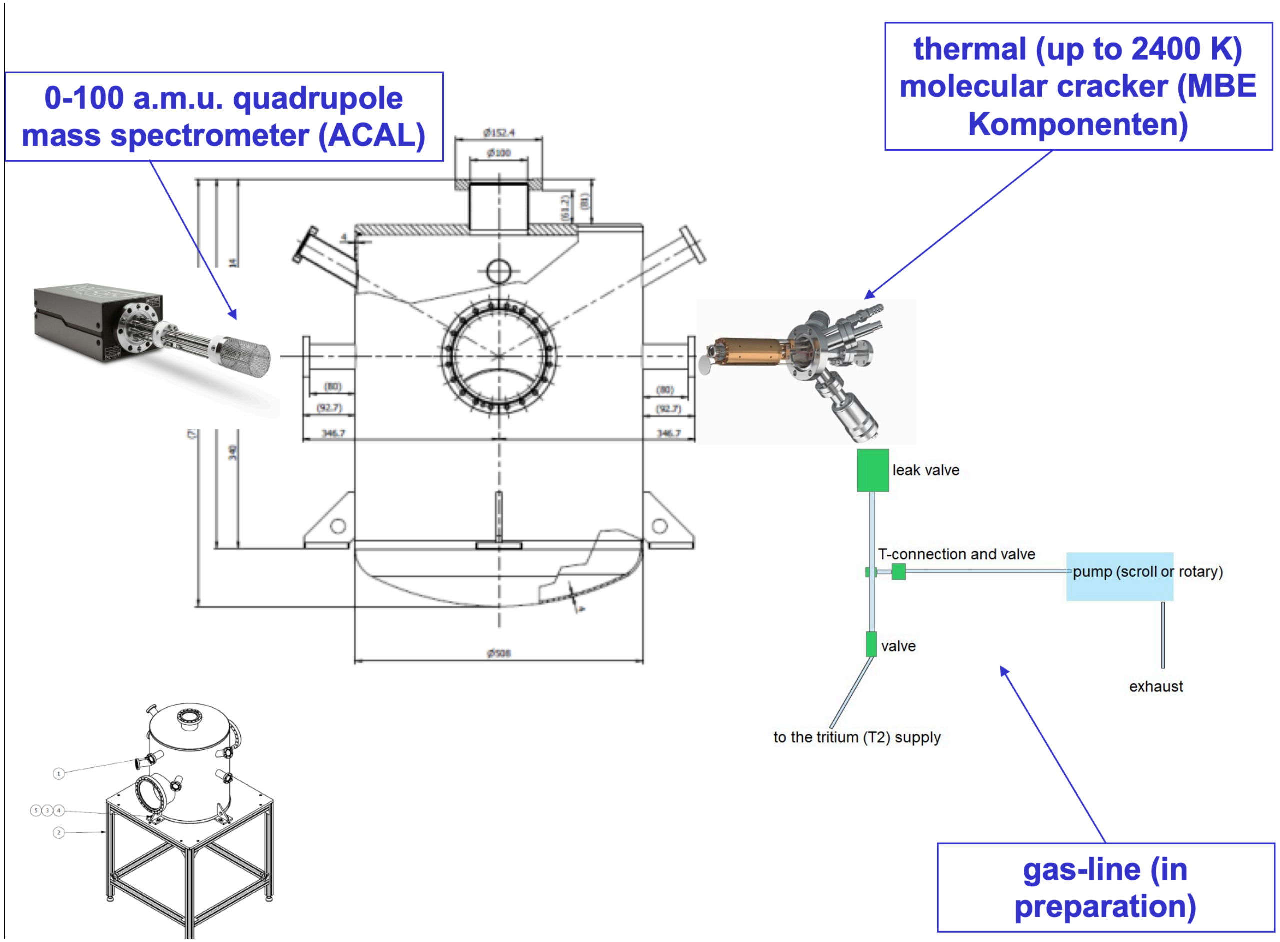
Other parts being procured separately

Custom UHV chamber

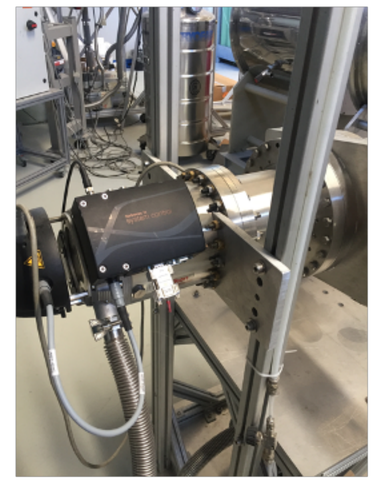
Designed in collaboration to with SAER_RIAL (Parma, Italy)

To be **shipped** to next week to Roma

Components of the chamber



- Pump system at LNGS



- Being moved to Roma

Schedule

- Serious delays due to procurements
(availability of companies, stainless steel market, …)
- Brand new lab equipped in Sapienza-INFN for flammable gases, available for testing the chamber (since **Sep 2023**)
- Without waiting for the manipulator,
we will do a **commissioning** using NPG substrates with **hydrogen starting Dec 2023**
  - Some standard test (XPS) in our lab's in Roma
- Ready for tritium by **Apr 2024**.

ENEA INMRI collaboration

- Sapienza-ENEA **framework agreement** already in place, a specific memorandum being prepared (to be approved by early 2024)

- Next year (starting mid 2024) they can
  - measure the **activity**
    - Very standard techniques, important for us to understand the **actual deposited mass of tritium**
  - demonstrate the tritiated graphene is not releasing tritium
    - Again using commercial tritium sensors

- Possible extension of the collaboration to the use of **TES** for **metrology** of beta spectra

Conclusion

- A general goal for the collaboration is to have a atomic **tritium sample** at our disposal
  - This would enable us to do **measurement of the beta** spectrum in different forms (albeit preliminary)
- Goal is to have a sample that can be easily handled
- Project of having a UHV chamber is **now at a critical point**
  - Slow up to now, due to difficulties in parts procurement
  - Now parts are almost all at Sapienza
- We will first start with **NPG as a substrate**
  but in future we will also do planar graphene and CNT
  - Currently, planar graphene and CNT hydrogenation being studies at RomaTre and Sapienza

- Back up slides

UHV custom chamber ready



UHV custom chamber ready 2

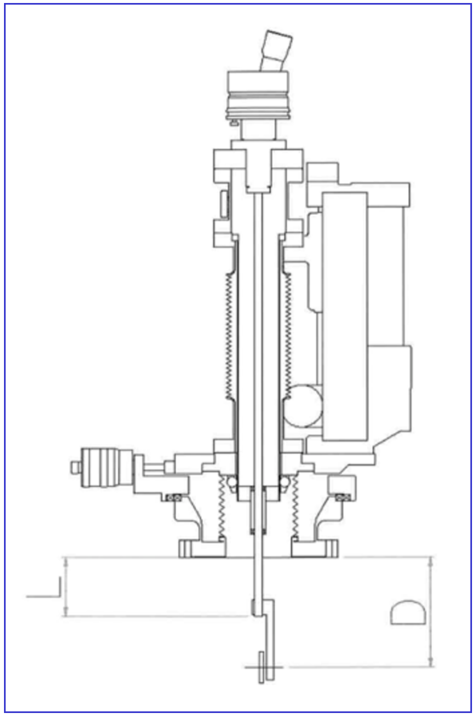
Parts produced

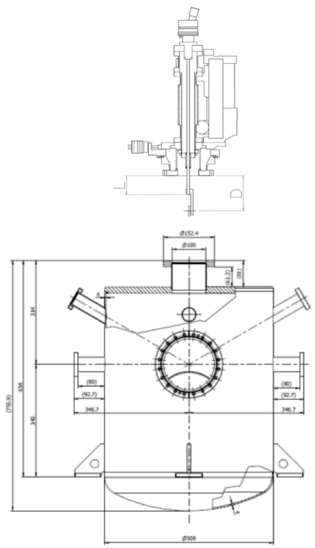
- Delivered to Roma Sapienza

Mass spectrometer

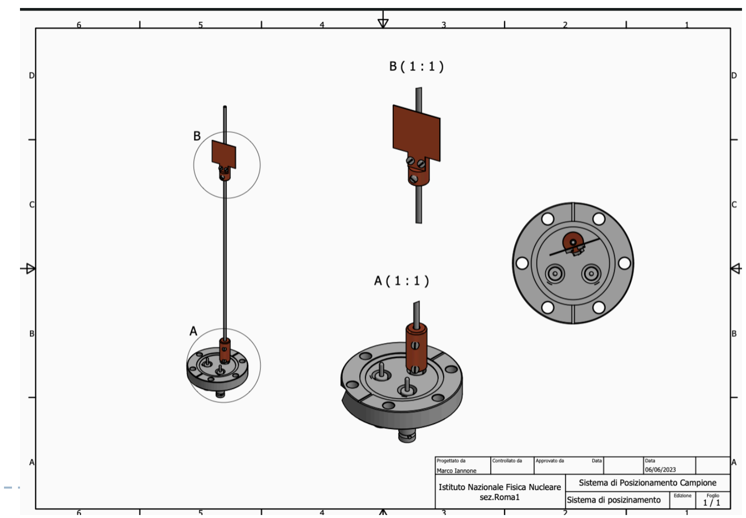
Therma cracker

Manipulator for the samples





- **VG HPT**
  - X - Y range +/-1.25 cm
  - Z range +/-5.0 cm
  - Heating system with e-bombardment (1300 K for sample annealing)
  - Standard sample holder



- Long procurement time (end of Feb)
- Currently a **static holder** has been designed and built in Roma INFN (with a heating system)