# Source and implantation for HOLMES experiment

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on behalf of the HOLMES collaboration

**Determination of the effective electron (anti)-neutrino mass**
**ECT* 10-14 February 2020 Trento, Italy**




# Outline

- Holmium production
- Chemical purification

- Reduction/distillation
- Sputter target creation

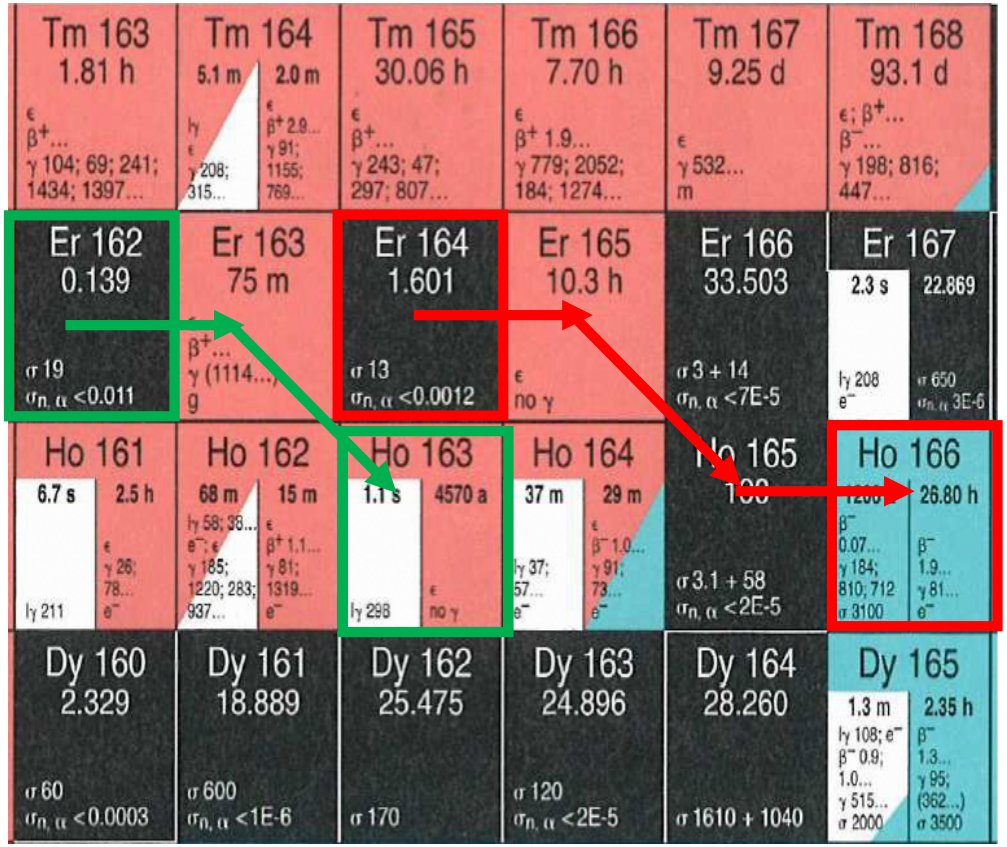
- Implantation system
- First tests, preliminary results (w/o Holmium)

# Holmium 163 production

$^{163}$Ho is produced by neutron irradiation of $Er_2O_3$ enriched (30%) in $^{162}$Er at the Institute Laue-Langevin (ILL, Grenoble, France).
Thermal neutron flux al ILL: $1.3 \times 10^{15}$ n/cm$^2$/s

$^{162}Er\ (n,\gamma)\ ^{163}Er \quad \sigma_{thermal} \approx 20b$

$^{163}Er \rightarrow\ ^{163}Ho + \nu_e \quad \tau_{1/2}^{EC} \approx 75min$




**Two types of contaminants:**

- **Other elements than Ho :** $^{170}$Tm and $^{171}$Tm, both beta emitters, are produced by irradiation of $^{168}$Er and $^{170}$Er. $^{159}$Dy could be produced from $^{158}$Dy impurities. Also the presence of stable elements like residual erbium (164, 166, etc...) or dysprosium (163, 164, etc...) are not suitable.

- **Holmium Isotopes:** residual of $^{165}$Ho, but in particular the irradiation of $^{165}$Ho present in the sample (as impurity or from irradiation of $^{164}$Er and all Dy isotopes) creates $^{166m}$Ho, $\beta^-$ with Q = 1.855 MeV, $\tau_{1/2}$ = 1200y.

The ratio between activity A($^{163}$Ho)/A($^{166m}$Ho) = 100 ~ 10000.

# Holmium chemical purification

Ho is separated by radiochemical separation with ion-exchange resins in hot-cell at PSI (efficiency > 80%).
The Ho is in oxide form ($Ho_2O_3$) in acid solution (PH<4) to avoid adhesion to the vial wall.

- *S. Heinitz et al., PLoS ONE 13(8): e0200910*

≈110 MBq of purified $^{163}$Ho available at Genova (≈250 kBq of $^{166m}$Ho)
The Er recovered from the purification procedure is available to produce other 80 MBq of $^{163}$Ho.

# Reduction/distillation

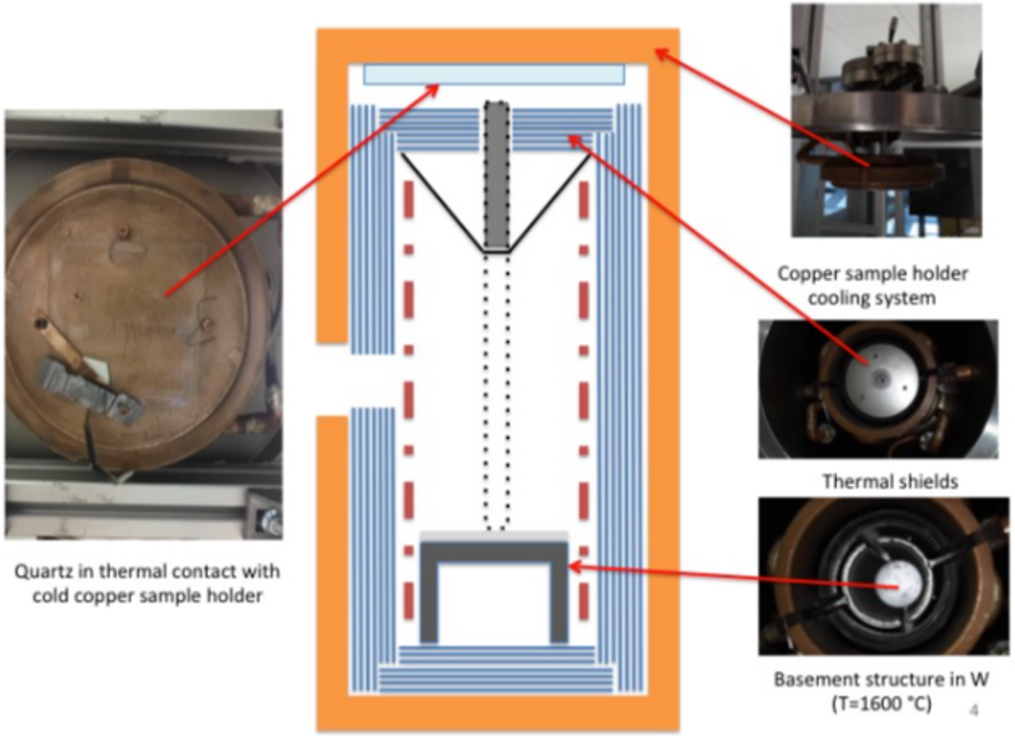
Holmium in the metallic form:

- Increase ionization and sputter efficiency;
- Reduce oxygen presence in plasma chamber;
- Metallic form for sputter ion target .

$$Ho_2O_3 + 2\mathrm{Y}(m) \rightarrow 2\mathrm{Ho}(m) + Y_2O_3$$

- Holmium Oxide powder is mixed with metallic Yttrium.
- The mixture is heat up to about 1600 ºC (Y melting point).
- When the Y wets the oxide the reaction starts.
- Metallic Ho has a high vapour pressure at this temperature and evaporated.
- Ho condensates on a cold substrate

# Evaporation chamber (1)



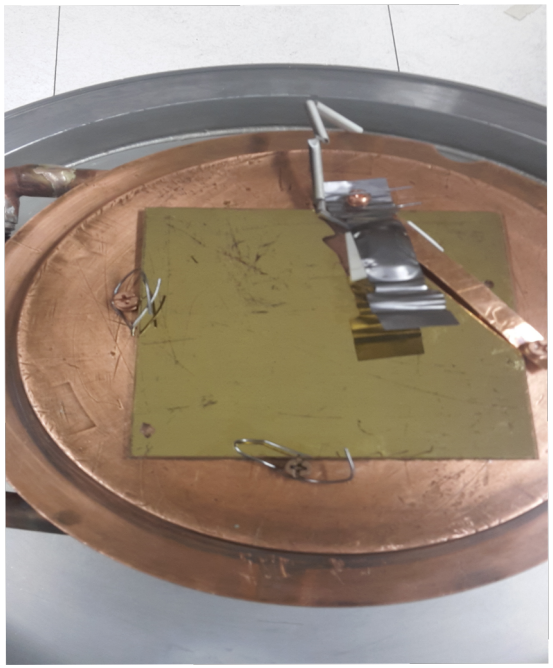

Dedicated evaporation chamber:

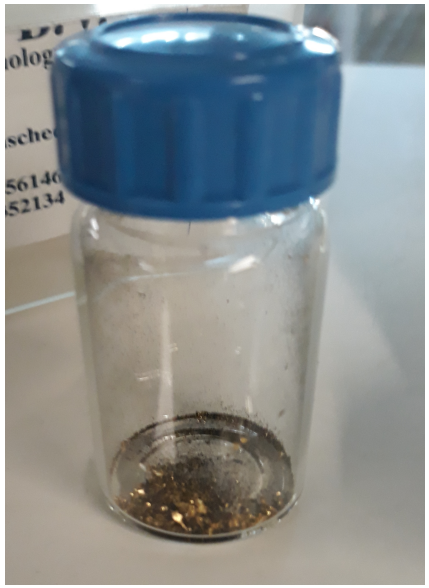
- The hot zone is thermally isolated by nine tungsten layers and closed in a water cooled box copper
- The upper part of the shield is holed allowing the evaporated Ho flows from crucible to a substrate.
- The whole system reach pressure as low as $10^{-8}$ mbar

The cap, where the collector substrate is fixed, is moveable using an automatic system.
The substrate is fixed in a closable structure to avoid dispersion, contamination, etc… and increase the collection efficiency.

# Evaporation chamber (2)

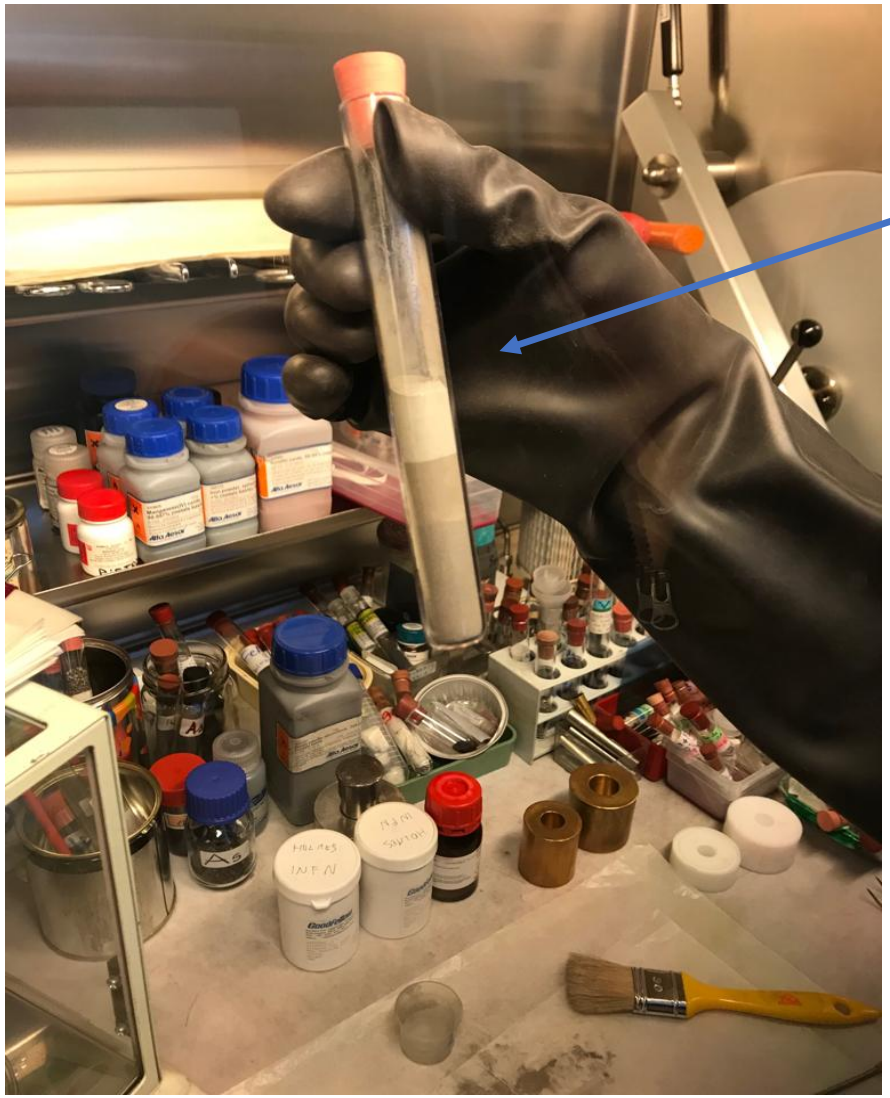
- The substrate used for the collection of the distilled Ho is made of quartz because of its high resistivity to thermal deformation.
- A thin layer of a low degassing thermal conducting paste is used to improve the thermal conductance between the quartz substrate and the cooled copper cap.
- A gold thin film has been deposited on quartz by thermal evaporation to easy remove Ho film from substrate. The high reflectivity of gold help to avoid excessive heating of substrate too.





Estimated Efficiency > 75%.

# Sputter target (1)



In collaboration with prof. Manfrinetti ( from Chemistry Department of Genova University) we decide to realize a sintered sputter target.

- Natural $^{165}$Ho(5%) is included in a metallic mixture of Ti(36%), Ni(41%), Sn(18%) fine grained powder (< 40 μm) in a copper support;
- Pressed at 350 bar/cm$^2$ and heated at 950 °C pressure $10^{-4}$ mbar for 2 days to improve the mechanical proprieties of the sintered. The crystallographic measurements and SEM-EDS analysis show a $Ti_2Ni_2Sn$ matrix with homogeneously distributed islands of HoNiSn.

After pressure

Final result

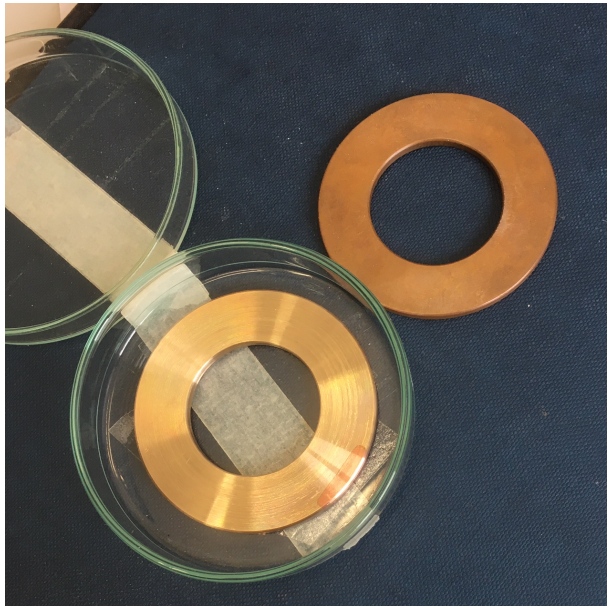
# Sputter target (2)

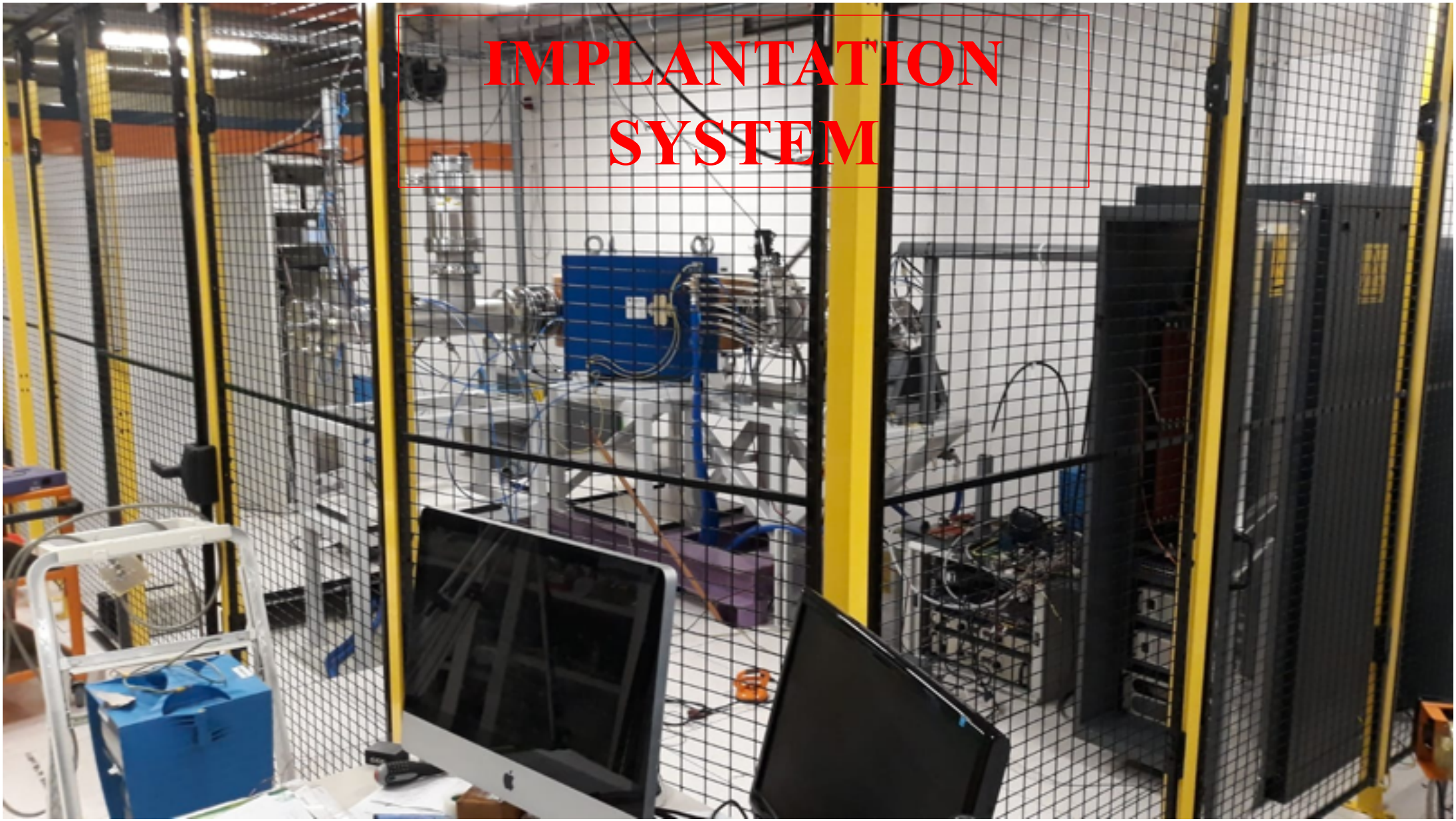
Dedicated glove box for storage (Argon atmosphere) and handling of powder and target.


## Some alternatives

In parallel we are developing some alternatives to do the sputter target:

- Gold coated copper target with Ho deposited in drops (holmium oxide or chloride.
- Ho drops deposited in metallic matrix.



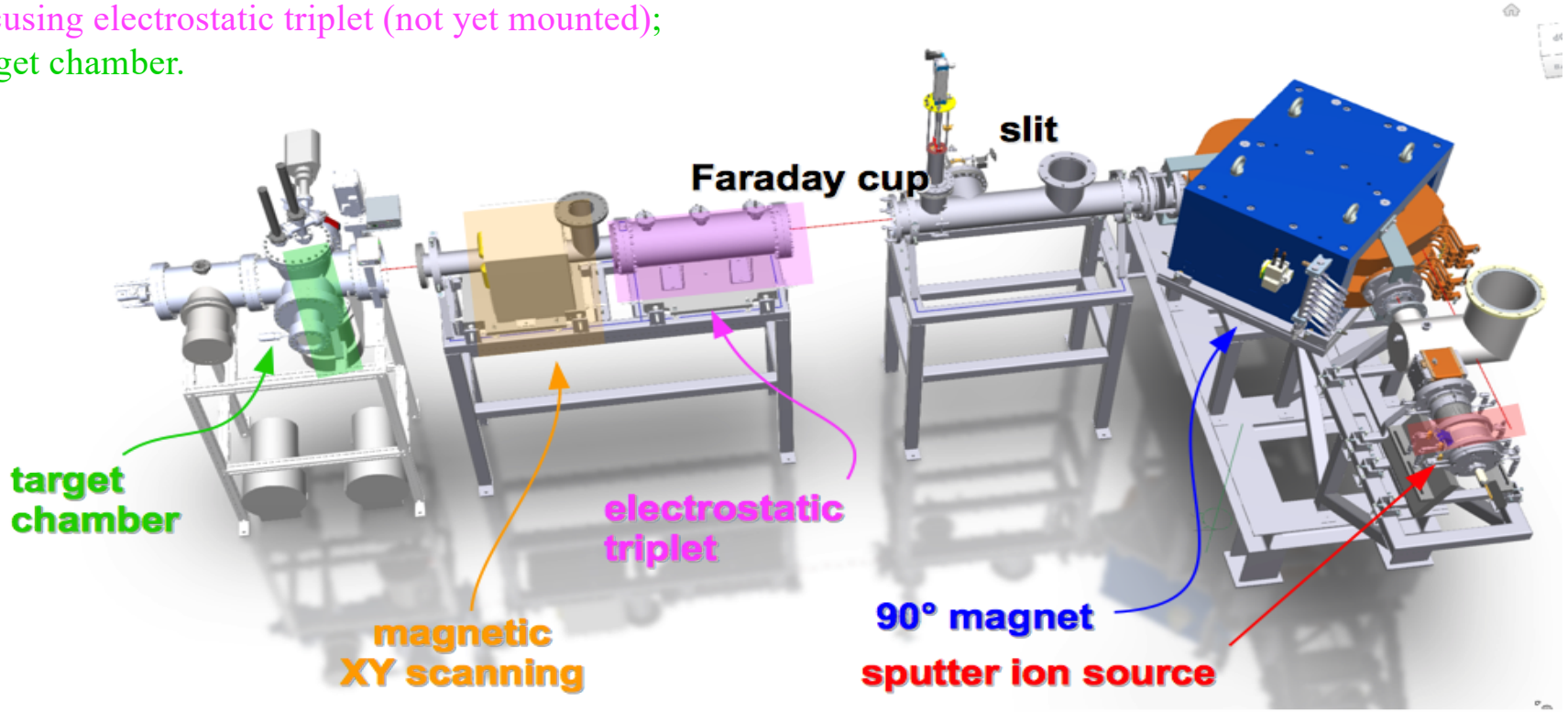

IMPLANTATION
SYSTEM

# Implantation system

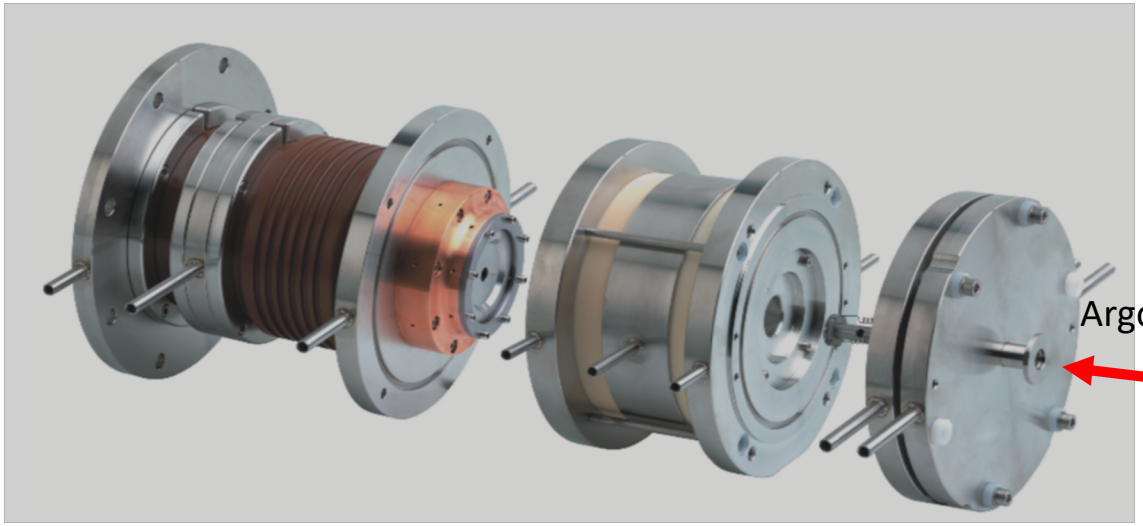
A dedicated ion implanter will be used to remove contamination of holmium isotopes different from $^{163}Ho$ as well as other impurities.

The ion implanter consists of six main components:

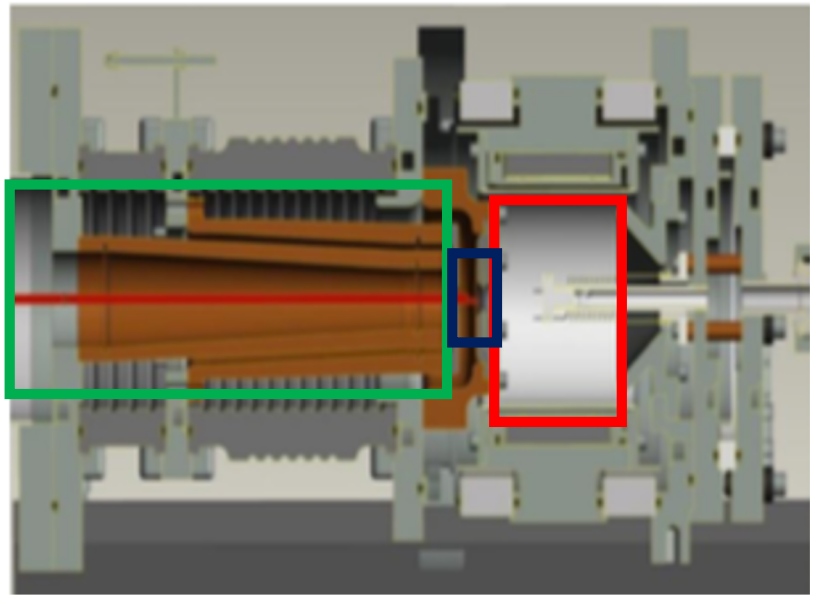
1. an argon sputter ion source;
2. an acceleration section to reach the beam energy of 50 KeV (~50 nm implantation depth)
3. a magnetic/electrostatic mass analyzer with magnetic field until 1.1 Tesla $^{163}Ho/^{166m}Ho$ separation better than $10^5$
4. a magnetic scanning stage (not yet mounted);
5. a focusing electrostatic triplet (not yet mounted);
6. a target chamber.



# Sputter Ion source



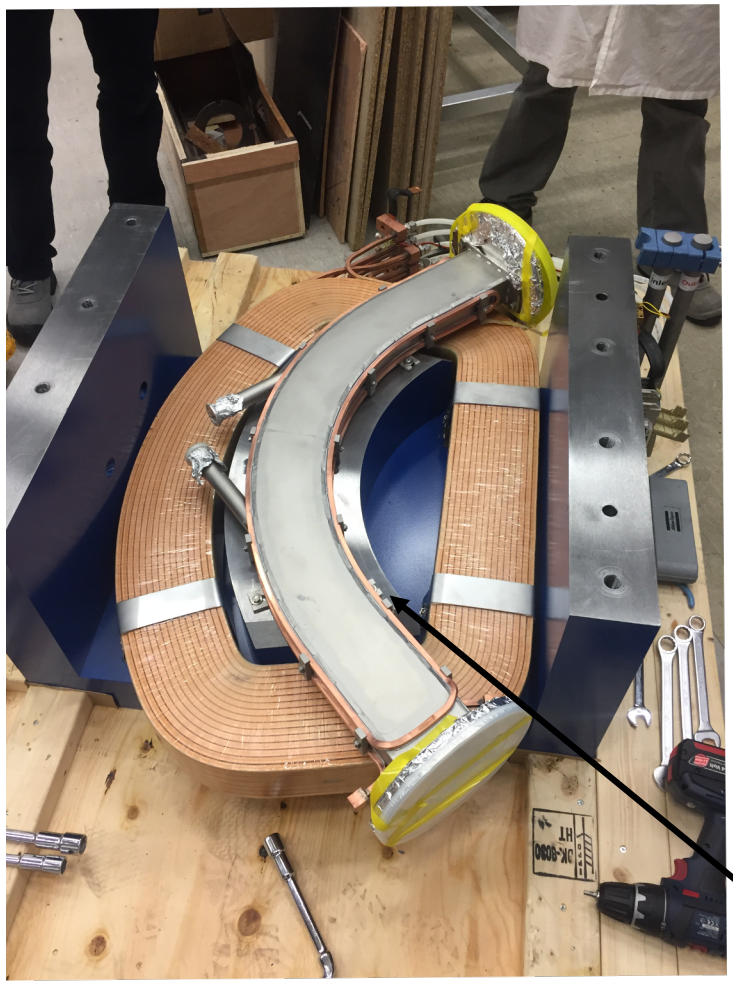

- Argon gas to sputter material
- Electron produced by thermoionic effect from tantalum filaments
- Plasma chamber with tunable potential and magnetic field
- Sputter target with sputter potential until 600 V
- Extraction potential 20-50kV

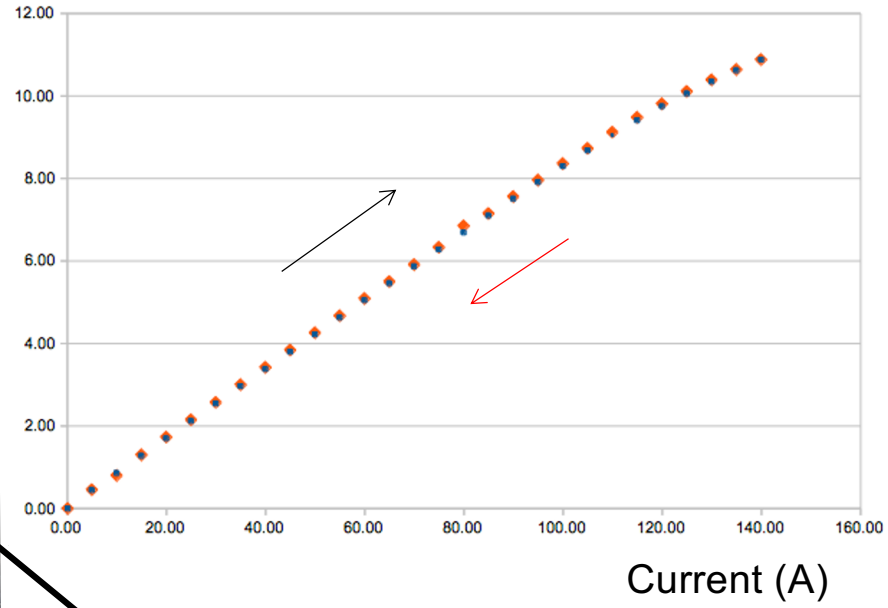


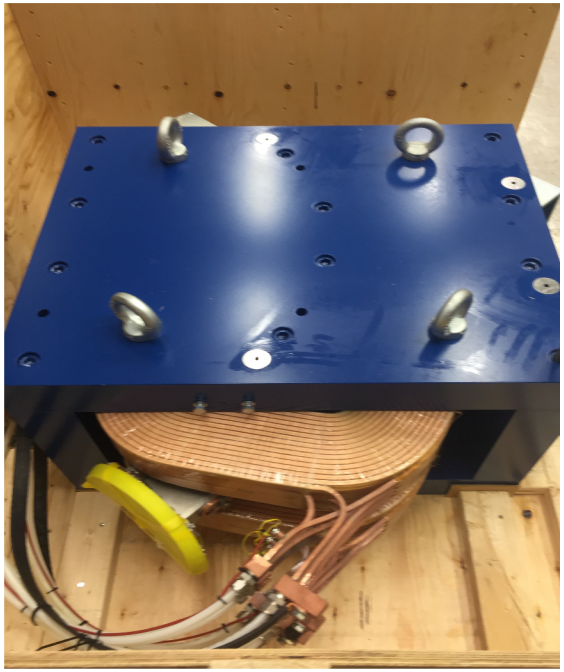
High Ion current (≈ 1 mA)
20-50 kV of extraction potential, 10-50 nm of implantation depth

# Magnet

- Induction until 1.1 Tesla
- Bending radius 46.1 cm
- Deflection Angle 90 degree



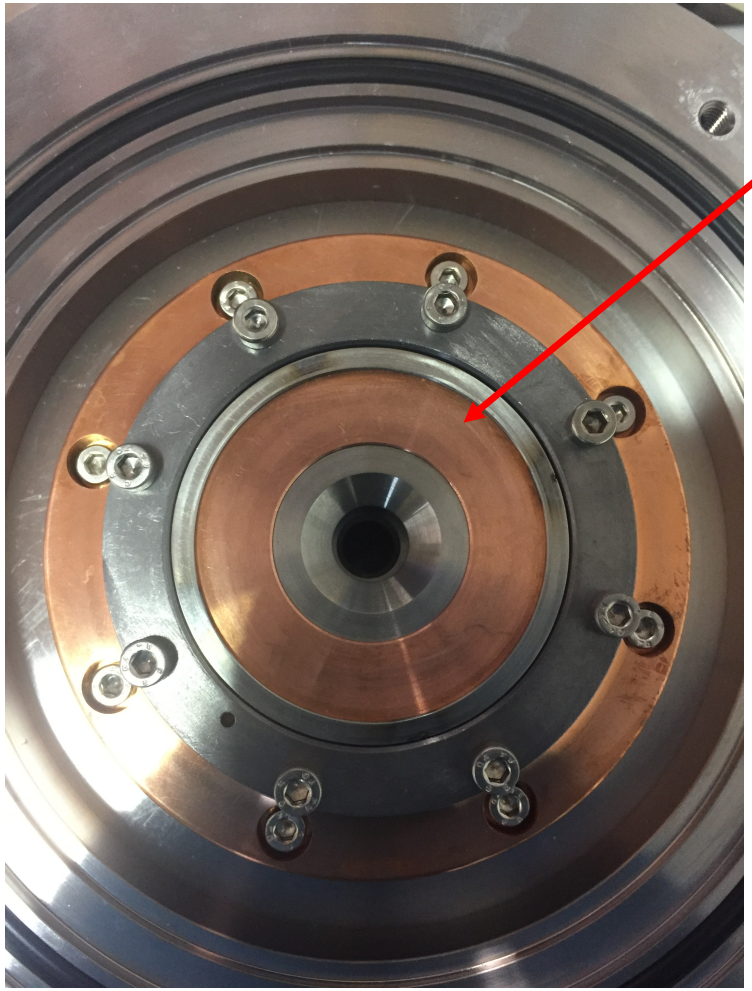


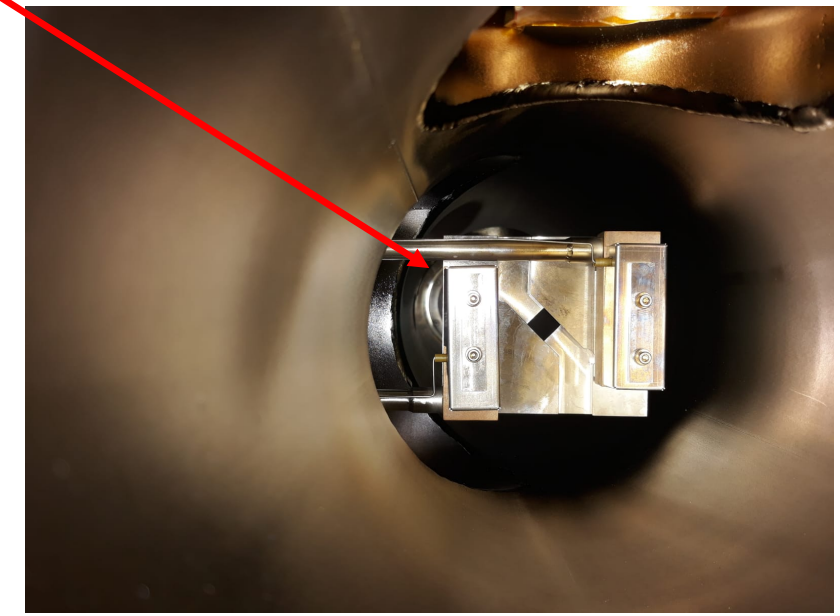
Negligible hysteresis effects.

A gauss probe is mounted inside the magnet near the wall of the vacuum chamber to have a real time field measurement.

# First tests (1)



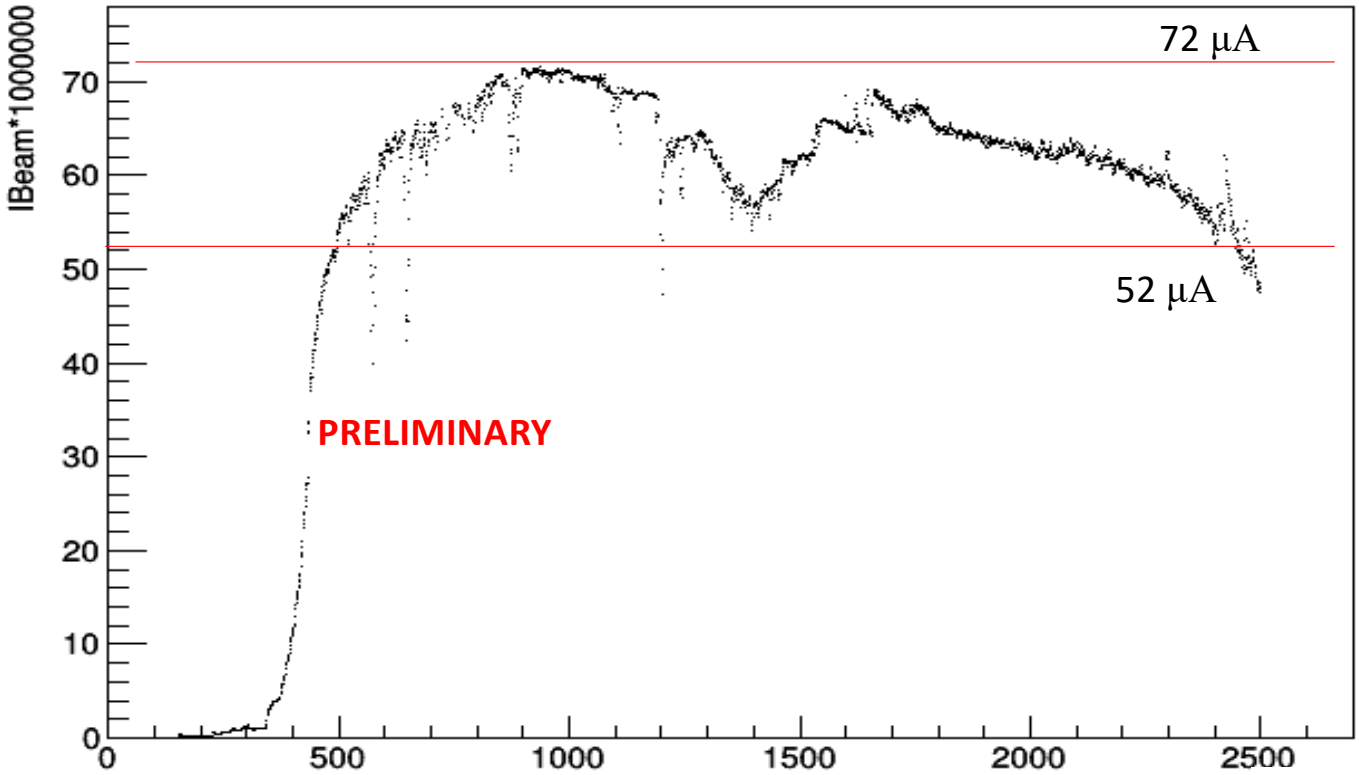
- Copper dummy target;
- Extraction potential ≅ 20kV , shield potential -7 kV
- Chamber Vacuum $10^{-7;}$
- Slits fixed to a 1 cm



# FIRST TESTS (2)

Running the source for around 2 hours.
Filament current 75 A
Sputter current 0.6 A (sputter potential 300 V)
Discharge current 15 A (potential 80 V)

Current (μA)




Time (a.u.)

Integrated current measured at a Faraday cup.

Max current ≅ 70 μA

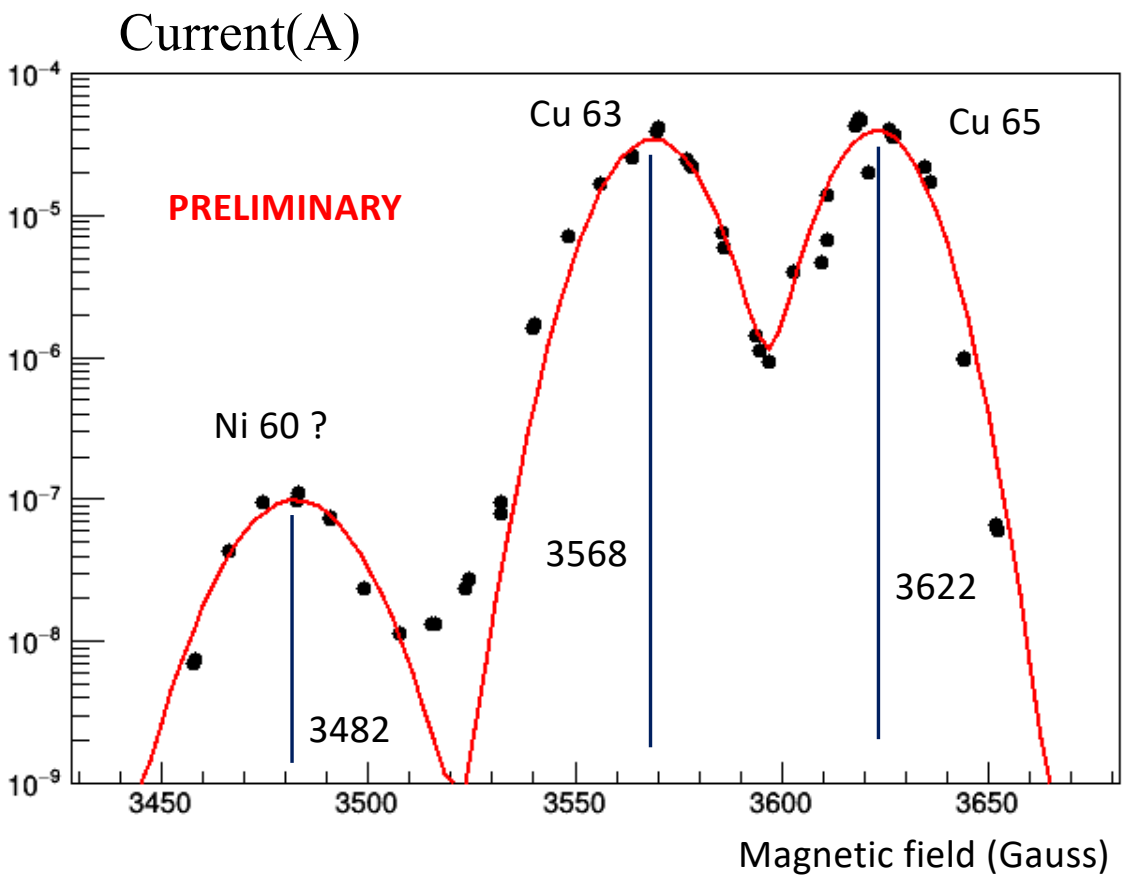
# First tests (30

Scan of the beam moving the magnetic field by step of 8 gaus s(100 mA)

1) Absolute Magnetic field $^{60}$Ni 3482 (3440)
$^{63}$Cu 3568 (3525)
$^{65}$Cu 3622 (3580)
Field difference $^{60}$Ni – $^{63}$Cu 86 (85)
$^{63}$Cu – $^{65}$Cu 54 (55)
Gauss probe not well calibrated (offset of ≅ 40 gauss)?
Source tilted ( ≅ 0.75 degree) ?

2) Rough estimation of beam spot using the $^{65}$Cu

FWHM ≅ 2 mm (better than expected).



# Conclusions

The production and chemical purification are finished and
the main $^{163}$Ho (110 MBq) sample is stored in the Genova's bunker now.
A good amount of Er has been recovered during the chemical purification and coulde be irradiated in the future to produce 80 MBq of $^{163}$Ho

The procedure to distillate holmium is tested. A metallic sintered sputter target has been obtained using Ti,Ni,Sn and Ho and a first target with natural holmium is ready. We are developing some backup solution.

The implantation system is ready in Genova. A dummy test with a copper target is done and the results are promising. The next step is a test with natural holmium.
First implantation with radioactive holmium is expected during this year.