

# Status of the **HOLMES** experiment to directly measure the electron neutrino mass with a calorimetric approach

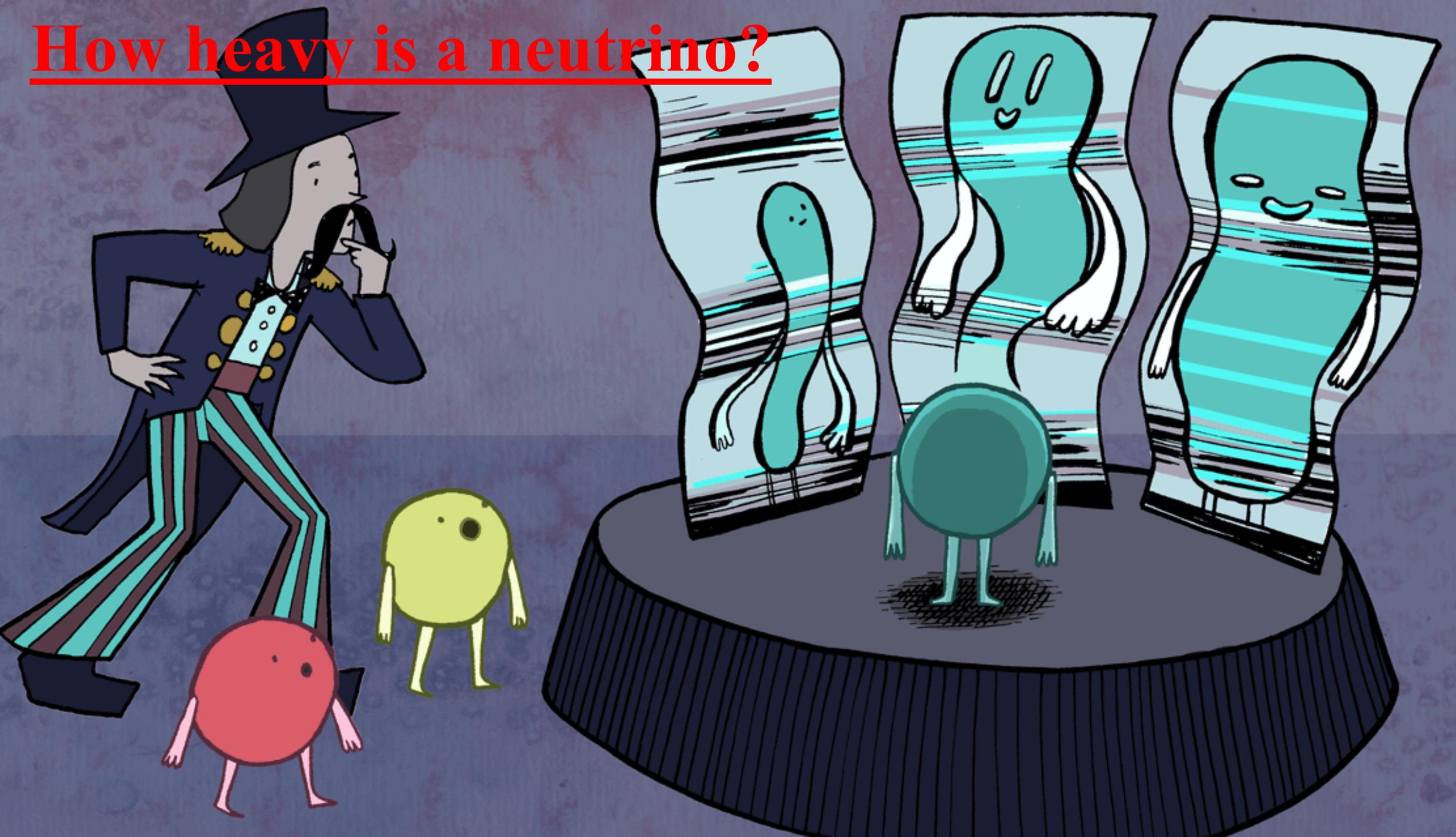
Giovanni Gallucci – INFN Genova  
On behalf of the HOLMES collaboration

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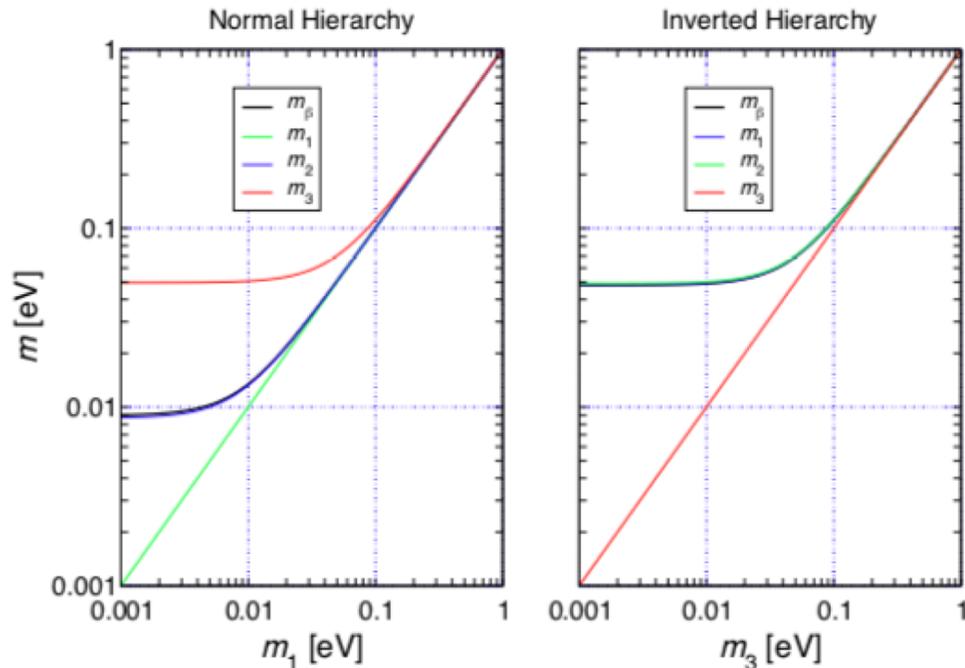


# How heavy is a neutrino?



# $\nu$ -mass

- Better understand the origin of fermion masses
- Extend the Standard Model
- Improve cosmological models ( $\nu$ -mass affects the large-scale structure and dynamics of the universe)
- Neutrino mass hierarchy
- etc...



From direct measurement :  $\lesssim 2 \text{ eV}$  (Mainz and Troitsk experiments)

From Double beta decay:  $\lesssim 0.5 \text{ eV}$  (only Majorana neutrino)

From cosmological and astrophysical data :  $\lesssim 0.2 - 1.3 \text{ eV}$  (model dependent)

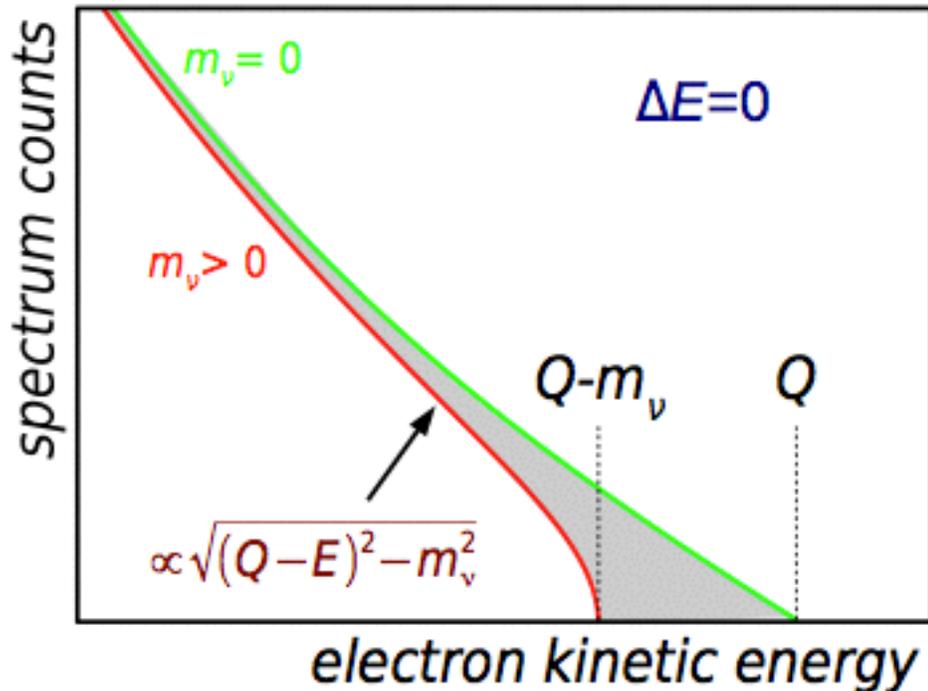
# Direct measurement (1)

Kinematics of weak decay with  $\nu$  emission:

- low Q nuclear  $\beta$  decays ( $^3\text{H}$ ,  $^{187}\text{Re}$ ,  $^{163}\text{Ho}$ ...)
- model independent: only E, p conservation
- $\nu$  mass appears as a distortion in the Kurie plot



$$N(E_\beta) \propto p_\beta E_\beta (Q - E_\beta) \sqrt{(Q - E_\beta)^2 - m_\nu^2} F(Z, E_\beta) S(E_\beta)$$



2 different approaches:

- **spectrometry**: source placed outside the detector (**KATRIN** approach)
- **calorimetry**: source embedded inside the detector (**ECHO**, **MARE**, **HOLMES** approach)  
 $\Rightarrow$  low T  $\mu$ -calorimeters

# Spectrometry vs Calorimetry

General requirements for a  $\nu$  mass experiment:

- High statistics near the end point
- low Q-value (stat  $\sim 1/Q^3$ )
- high activity/efficiency of the source
- Energy resolution order  $\sim eV$  or below (comparable with  $m_\nu$ )
- small systematic effects

## **Spectroscopy: external source**

- high statistics
- high energy resolution (below eV)
- systematics due to the source (energy loss)
- systematics due to decay to excited states
- background

## **Calorimetry: embedded source**

- no backscattering
- no energy loss in source
- no solid state excitation
- no atomic/molecular final state effects
- good energy resolution ( $\sim eV$ )
- limited statistics
- systematics due to pile-up
- background

# Electron capture (EC) in Holmium 163



${}^{163}\text{Ho}$  decay via EC from shell  $\geq M1$

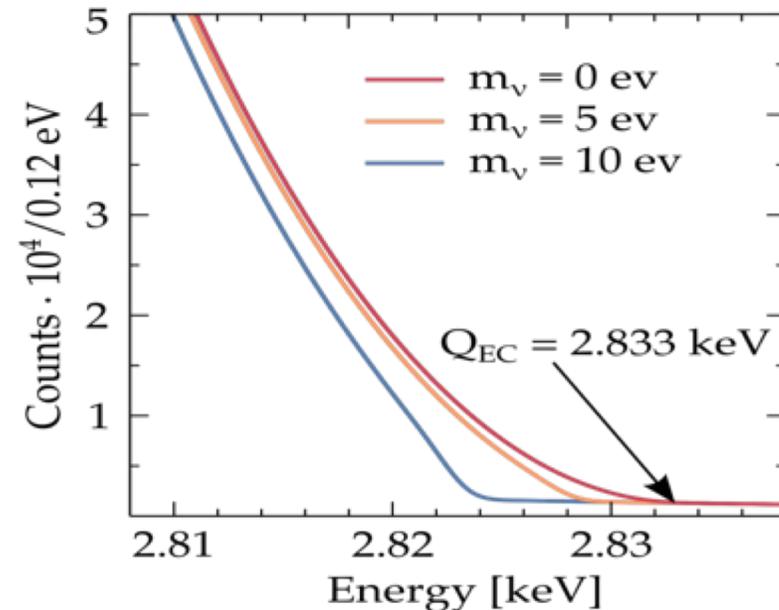
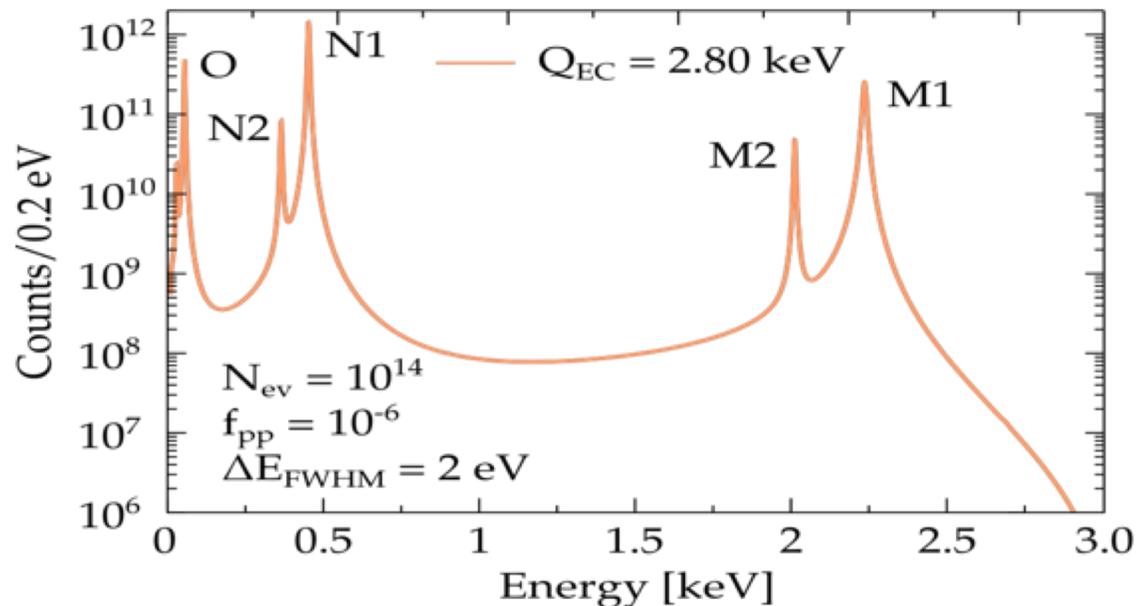
Proposed by A. De Rújula and M. Lusignoli,  
Phys. Lett. B 118 (1982) 429

Calorimetric measurement of Dy atomic de-excitations

$Q \approx 2.83$  keV (recently measured with Penning trap)

Rate on the end point depends on  $(Q-M1)$ : the proximity to M1 resonance peak enhances the statistics at the end point

$\tau \approx 4570$  years  $\rightarrow 2 \times 10$  Ho nuclei  $\leftrightarrow$  1 Bq



# Electron capture (EC) in Holmium 163 (2)

Pile-up occurs when multiple events arrive within the temporal resolving time of the detector.

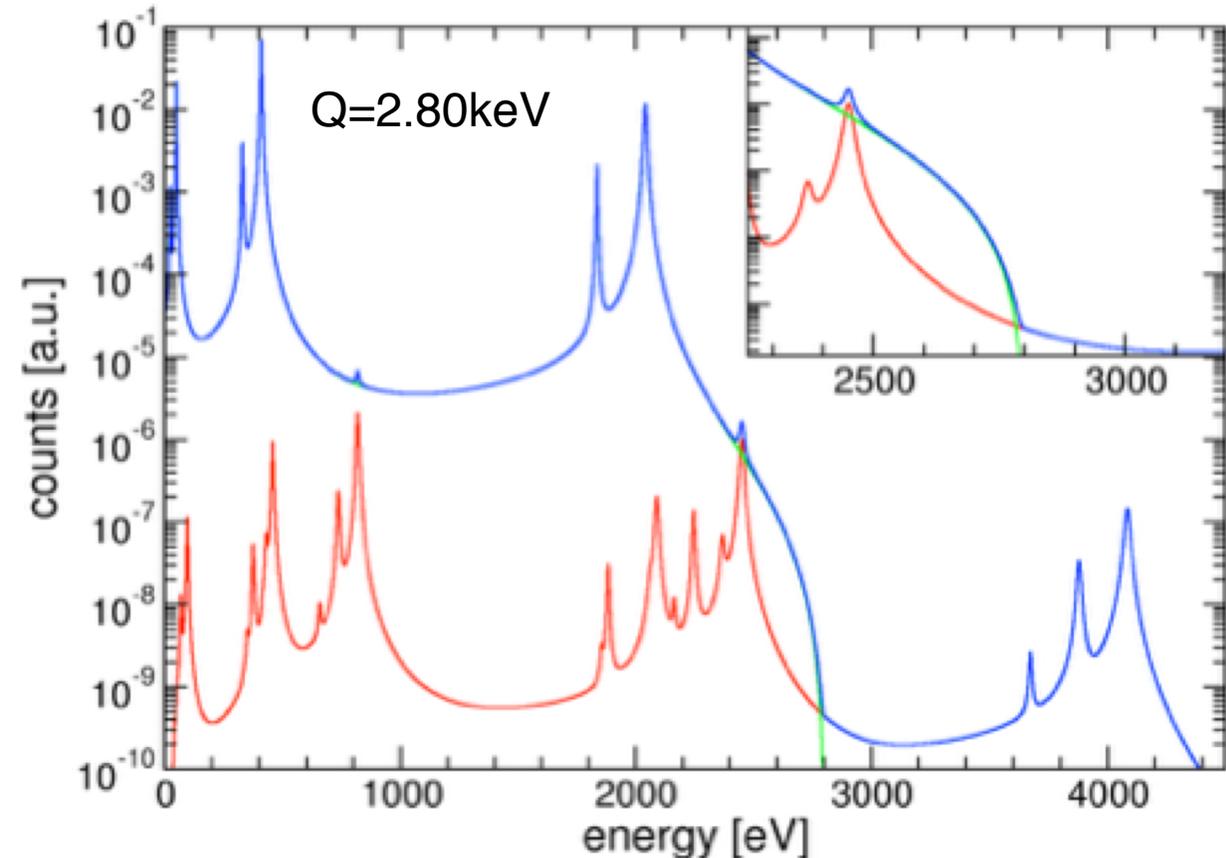
In a first approximation, the fraction of unresolved pile up is given by  $f_{pp} = \tau \times A(\text{EC})$

In calorimetric measurement detector speed is critical

$$N_{pp}(E) = f_{pp} N_{EC}(E) \otimes N_{EC}(E)$$

In order to reduce pile-up:

- trade-off between activity and statistic
- detector with fast signal rise time  $\tau_r$
- pile-up resolving algorithm



# Holmes experiment in a nutshell

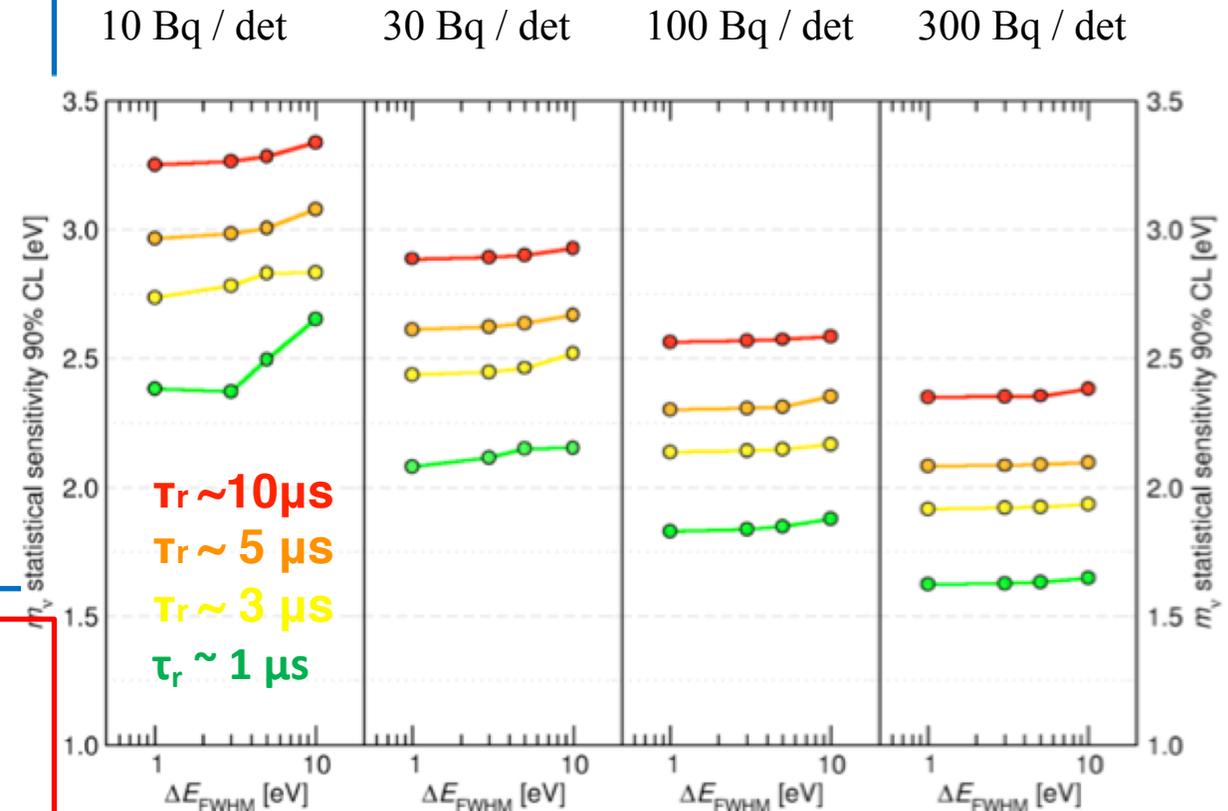
Direct neutrino mass measurement with statistical sensitivity around 1 eV.

Usage of Transition edge sensor (TES) based micro-calorimeters with  $^{163}\text{Ho}$  implanted and Au absorber:

- Energy resolution  $\Delta E \sim 1 \text{ eV}$ , time  $\Delta t \sim 1 \mu\text{s}$
- $6.5 \times 10^{13}$  nuclei/det,  $A(\text{EC}) \sim 300 \text{ Bq/det}$
- 1000 channels array:  $6.5 \times 10^{16}$  total nuclei ( $\approx 18 \mu\text{g}$ )
- $O(10^{13})$  events / year, data taking  $\sim 3$  years
- Pile up fraction  $f_{\text{pp}} \approx A \times \Delta t = 3 \times 10^{-4}$

Should prove the technique potential and scalability by:

- assessing EC spectral shape
- assessing systematic errors
- sensitivity on  $m_\nu \sim \text{eV}$



**exposure: 1000 det x 3 years**

# BACKGROUND

## External sources

- Environmental  $\gamma$  radiation
- $\gamma$ , X and  $\beta$  from close surroundings
- cosmic rays

Measured  $200 \times 200 \times 2 \mu\text{m}^3$  Au absorber (Holmes-like)

→  $\text{bkg}(1-10 \text{ KeV}) \approx 5 \times 10^{-3} \text{ count/eV/day/det}$

## Internal source

radionuclides (byproduct of Ho production)

$^{166\text{m}}\text{Ho}$ , ( $\beta^-$ ,  $Q = 5970 \text{ keV}$ ,  $t_{1/2} = 1200 \text{ y}$ )

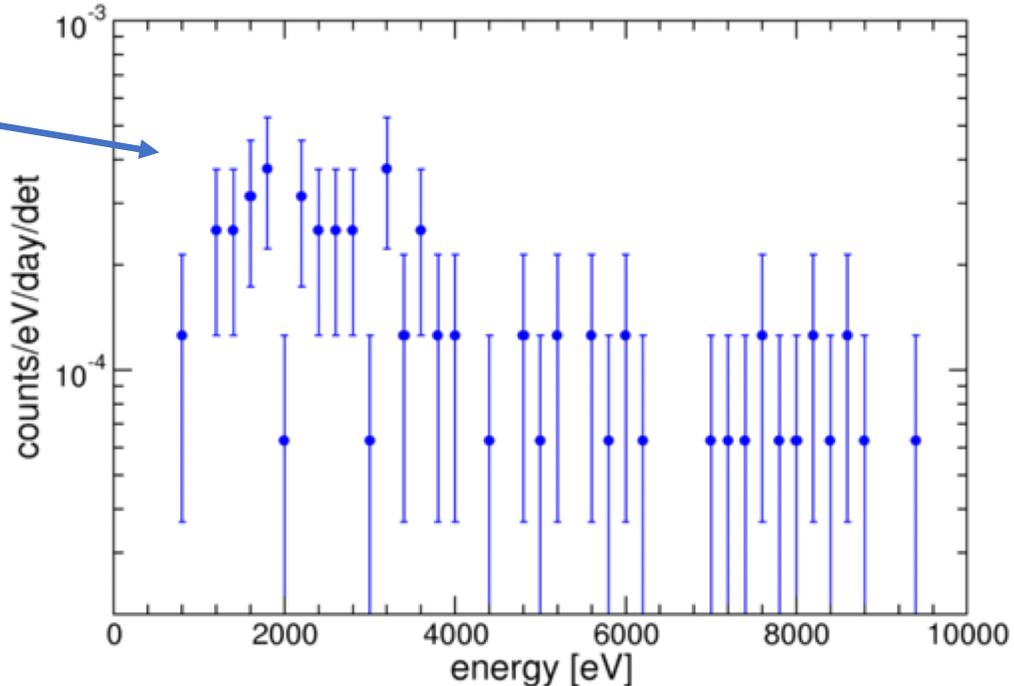
GEANT4 simulation for  $200 \times 200 \times 2 \text{ mm}^3$  Au absorber produce

$\text{bkg} \ 10^{-11} \text{ c/eV/day/det} / ^{166}\text{HoNucleus}$

If  $A(300\text{Bq})$  and requiring  $\text{bkg} < 0.1 \text{ count/eV/day/det}$

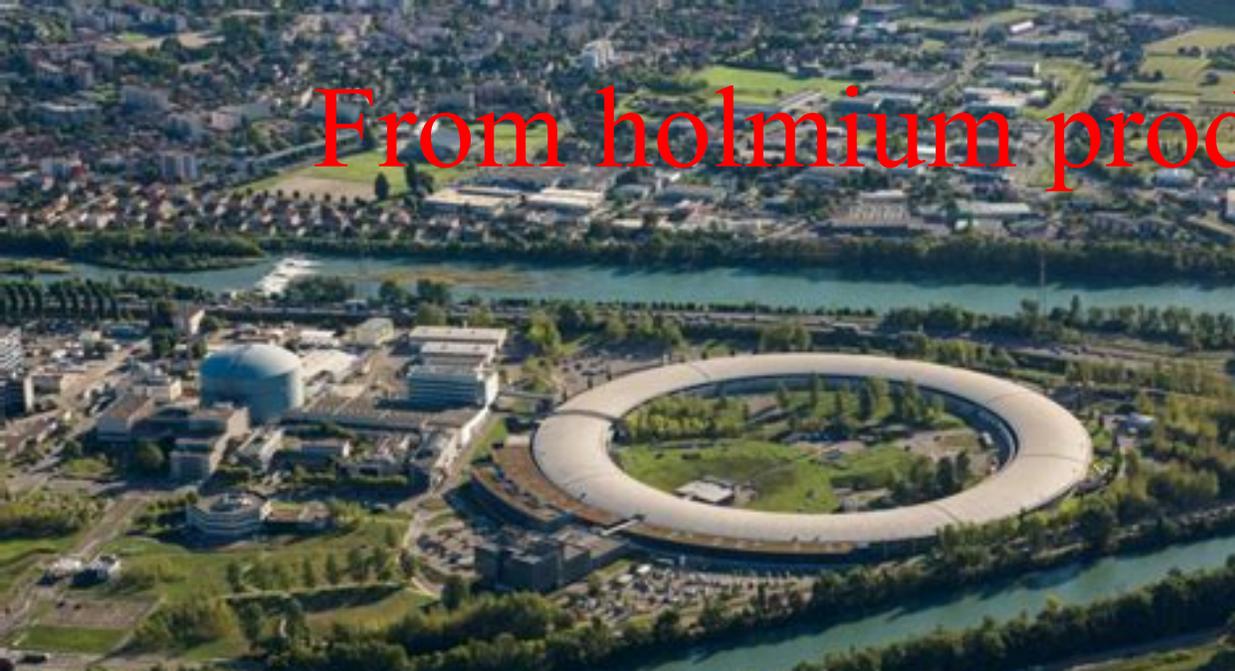
HOLMES baseline:  $^{163}\text{Ho}$  pile-up rate

$$\langle r_{pp} \rangle = A \cdot f_{pp} / 2Q = 300 \text{ Bq} \times 3 \cdot 10^{-4} / 2Q = 1.5 \text{ count/eV/day/det}$$



$$\begin{aligned} N(163)/N(166) &> 6000 \\ A(163)/A(166) &> 1500 \end{aligned}$$

# From holmium production to sputter target

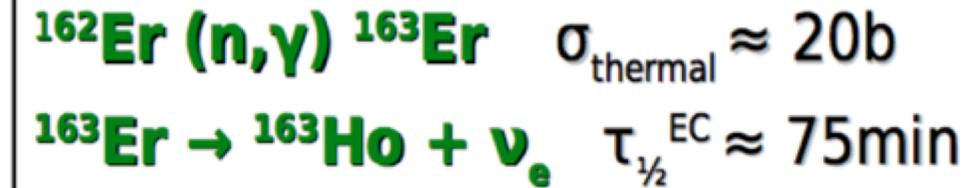


# 163-Holmium production

Tm 163 1.81 h	Tm 164 5.1 m 2.0 m	Tm 165 30.06 h	Tm 166 7.70 h	Tm 167 9.25 d	Tm 168 93.1 d
Er 162 0.139	Er 163 75 m	Er 164 1.601	Er 165 10.3 h	Er 166 33.503	Er 167 2.3 s 22.869
Ho 161 6.7 s 2.5 h	Ho 162 68 m 15 m	Ho 163 1.1 4570 a	Ho 164 37 m 29 m	Ho 165 100	Ho 166 1200 a 28.80 h
Dy 160	Dy 161	Dy 162 475	Dy 163 24.896	Dy 164 28.260	Dy 165 1.3 m 2.35 h



$^{163}\text{Ho}$  produced by neutron irradiation of  $\text{Er}_2\text{O}_3$  enriched (30%) in  $^{162}\text{Er}$  at the Institut Laue-Langevin (ILL, Grenoble, France). Thermal neutron flux at ILL:  $1.3 \times 10^{15}$  n/cm<sup>2</sup>/s



high yield (but not all cross sections are well known)

## Contaminants:

1. Other elements (residual Er, rare earth contaminants, decay product, etc...)
2. Holmium isotopes, in particular  $^{166\text{m}}\text{Ho}$

$$A(^{163}\text{Ho})/A(^{166\text{m}}\text{Ho}) = 100\text{-}1000$$

- 1) Chemical purification at PSI;
- 2) Isotopic separation using an implanter.

# Holmium chemical purification

Three different batches are produced:

- 1) 25 mg irradiated for 55 days (2014),  $A(163\text{Ho}) \approx 5\text{MBq}$  ( $A(166\text{mHo}) \approx 10\text{kBq}$ )
- 2) 150 mg irradiated for 50 days (2015),  $A(163\text{Ho}) \approx 38\text{MBq}$  ( $A(166\text{mHo}) \approx 37\text{kBq}$ )
- 3) 540 mg irradiated 50 days (2017),  $A(163\text{Ho}) \approx 130\text{MBq}$  ( $A(166\text{mHo}) \approx 180\text{kBq}$ ) (~500 detectors)

The Er/Ho mixture is subjected to a radiochemical separation with ion-exchange resins in hot-cell at PSI.

Efficiency > 80%



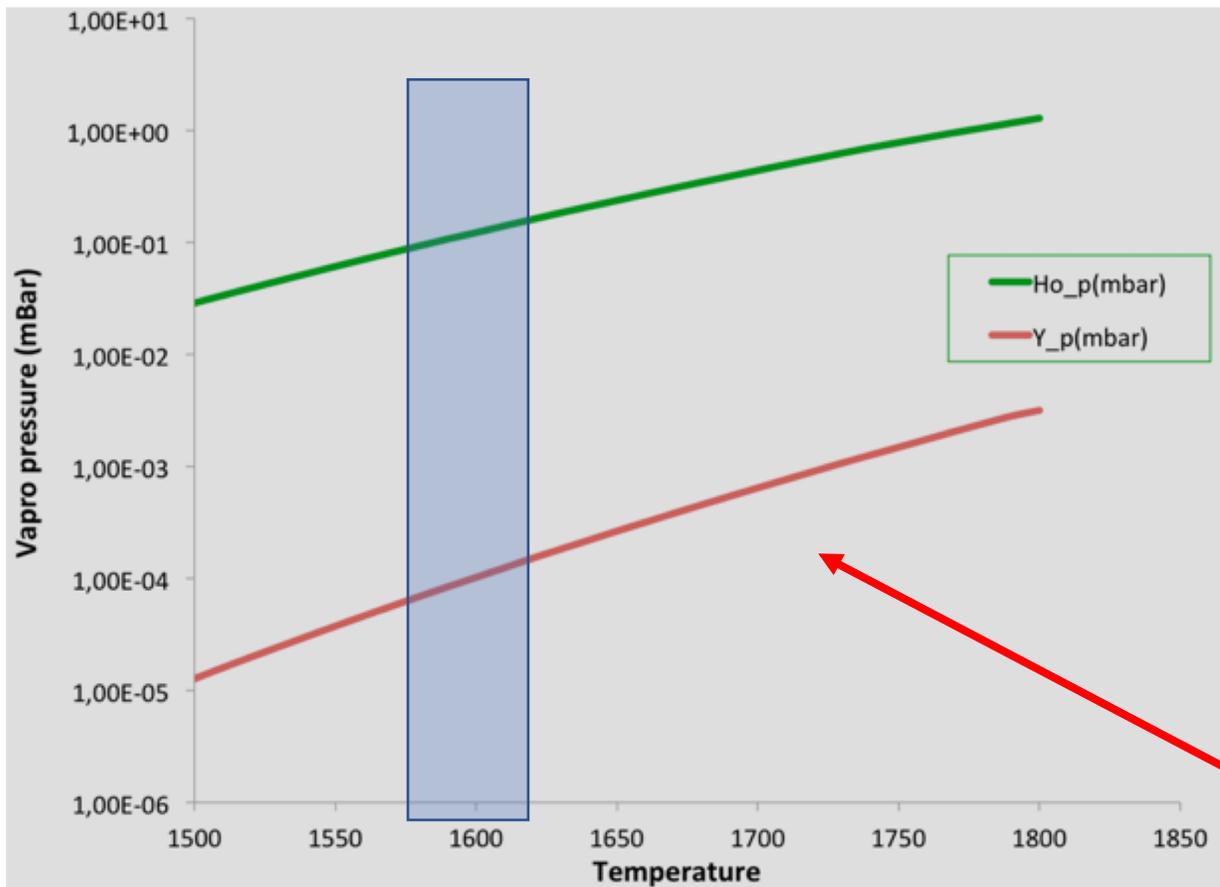
# Reduction and distillation process (1)

Only holmium in the metallic chemical form must be introduced:

Increase ionization efficiency in metallic form;

Metallic form for sputter ion target .

Metal	Melting Point (°C)	Oxyde	$\Delta G$ (kJ/mol)
Holmium	1460	Ho <sub>2</sub> O <sub>3</sub>	-1791.1
Yttrium	1526	Y <sub>2</sub> O <sub>3</sub>	-1816.2



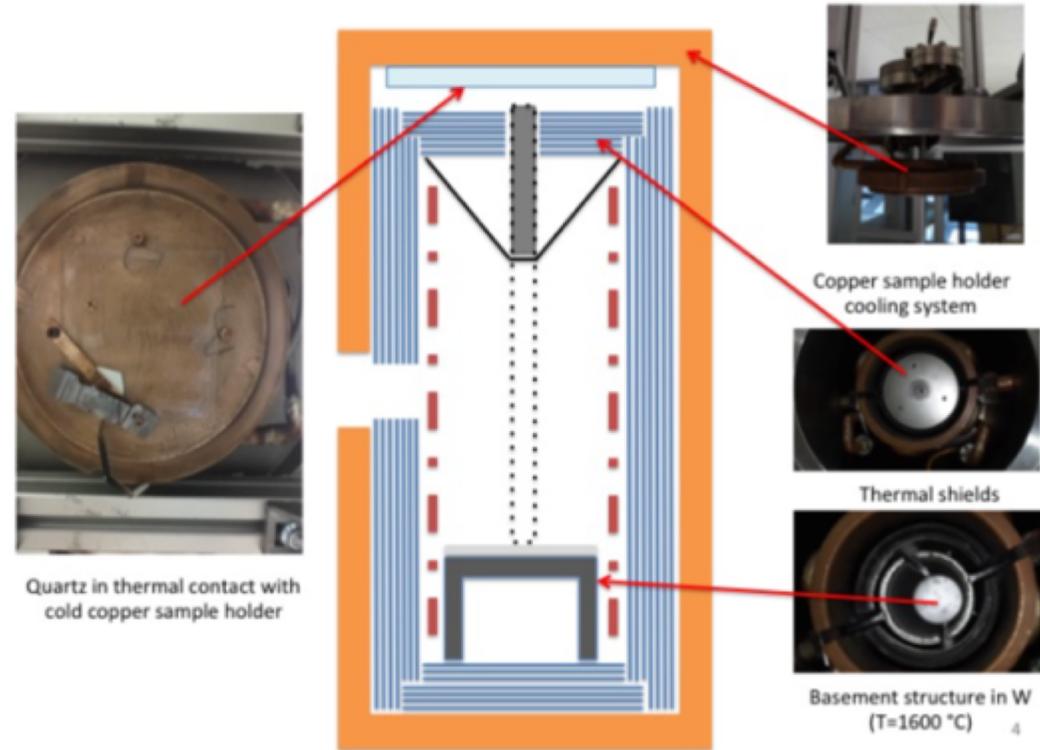
- Holmium Oxide powder is mixed with metallic Yttrium (lump).
- The mixture is heat up to about 1600 °C (Y melting point).
- When the Y wets the oxide the reaction starts.
- Metallic Ho has a high vapor pressure at this temperature and evaporated.
- Ho condensates on a cold substrate.

Oxide (Y and residual Ho) have a negligible vapor pressure.

The Y vapor pressure is three order of magnitude lower than Ho one at working point.

**We use a dedicated evaporation chamber to the whole process.**

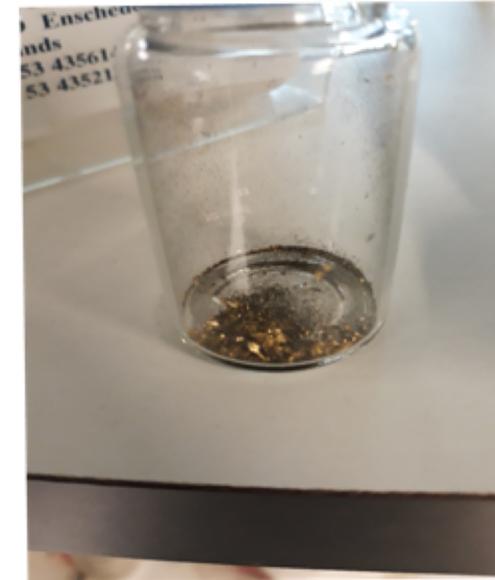
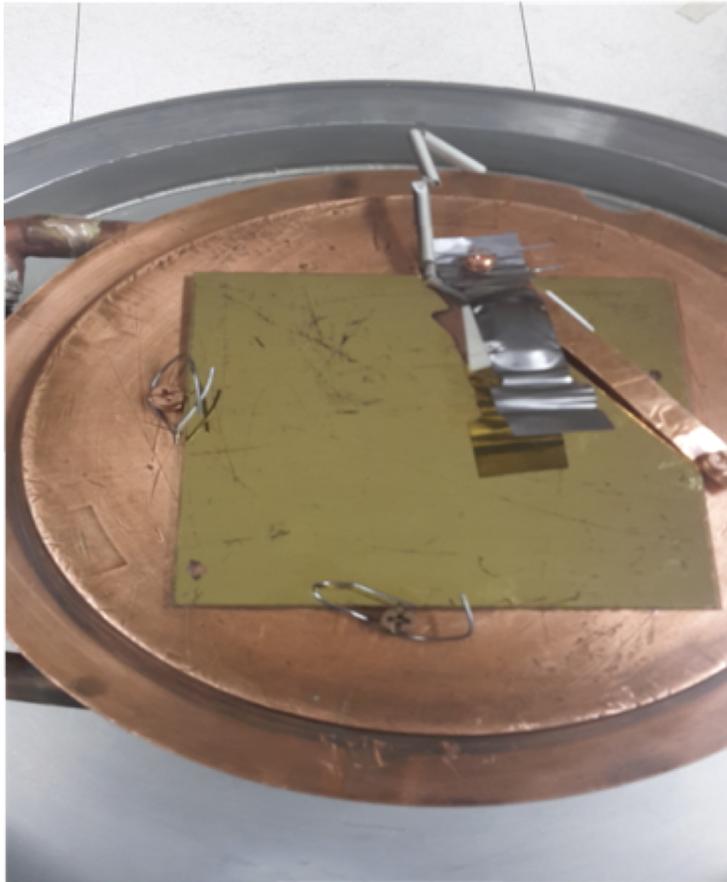
# Reduction and distillation process (2)



The hot zone is thermally isolated by nine tungsten layer. The external copper box is water cooled. The upper part of the shield is holed allowing the evaporated Ho flows from the crucible to a substrate fixed on the top of the copper box. The whole system is set in a vacuum chamber which could reach pressures as low as  $10^{-8}$  mBar.

# Reduction/Distillation Process (3)

- 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.



**Ho + Au powder**  
Efficiency  $\geq 70\%$ .

Next steps  
Increase the collection  
efficiency

# Sputter target

The sputter ion source of the implanter needs a metallic cathode.  
We need turn the holmium powder into a metallic disk.



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

- Natural  $^{165}\text{Ho}$ (5%) is included in a metallic mixture of Ti(36%), Ni(41%), Sn(18%) fine grained powder ( $< 40 \mu\text{m}$ ) in a copper support;
- Pressed at  $350 \text{ bar/cm}^2$  and heated at  $950 \text{ }^\circ\text{C}$  pressure  $10^{-4} \text{ mbar}$  for 2 days to improve the mechanical proprieties of the sintered.



Next steps  
Produce more test targets  
Produce a target with  $^{163}\text{Ho}$



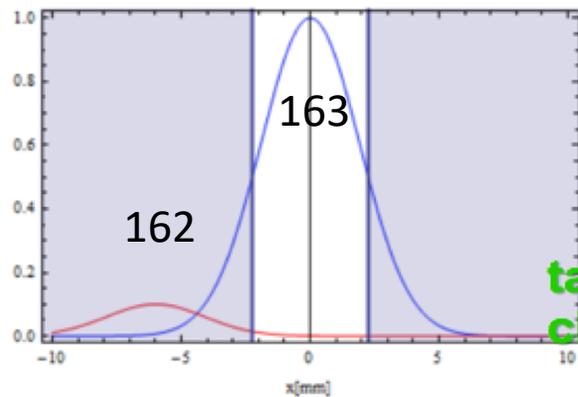
# Mass separation and ion implantation

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

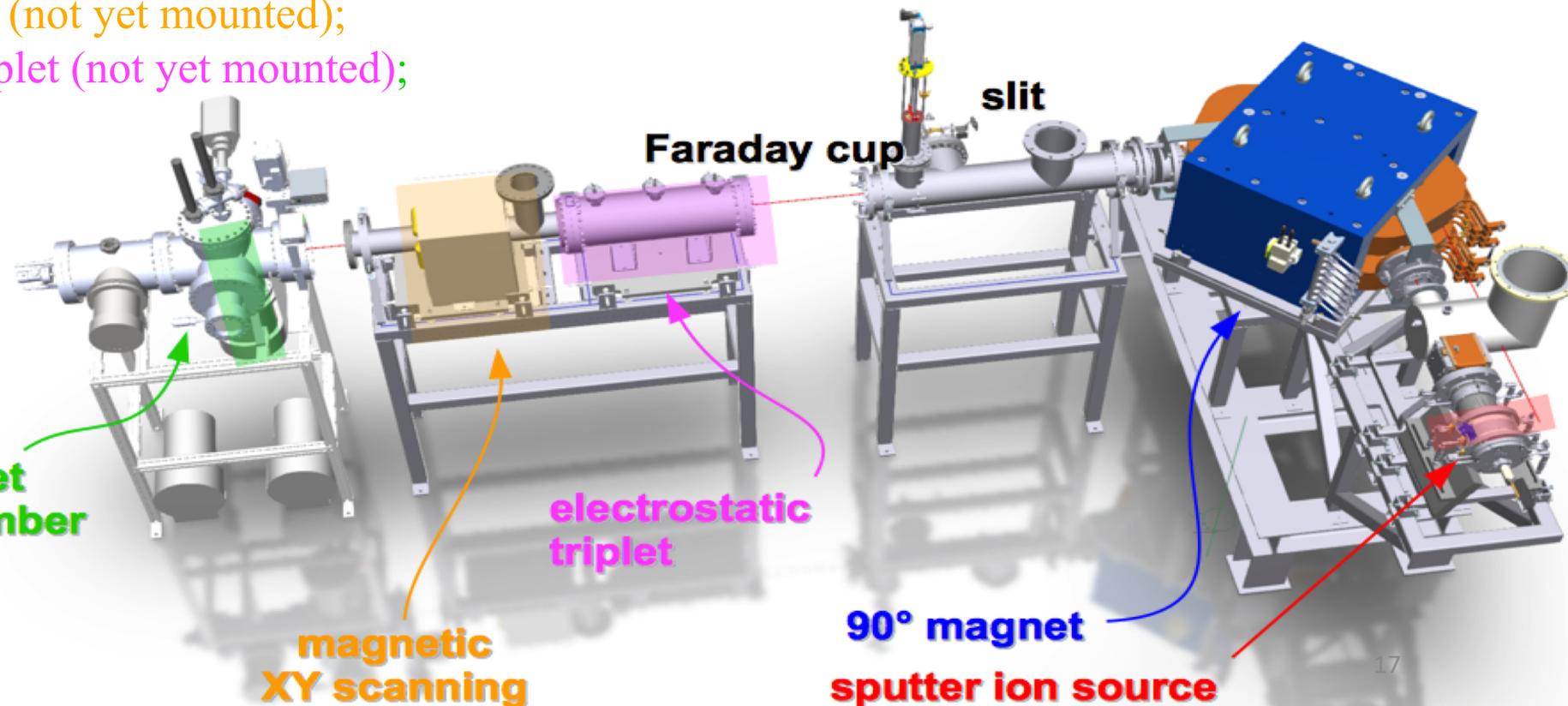
The ion implanter consists of six main components:

1. an argon sputter ion source;
2. an acceleration section to reach the beam energy of 50 KeV ( $\sim 50$  nm implantation depth)
3. a magnetic/electrostatic mass analyzer with magnetic field until 1.1 Tesla  $^{163}\text{Ho}/^{166\text{m}}\text{Ho}$  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.

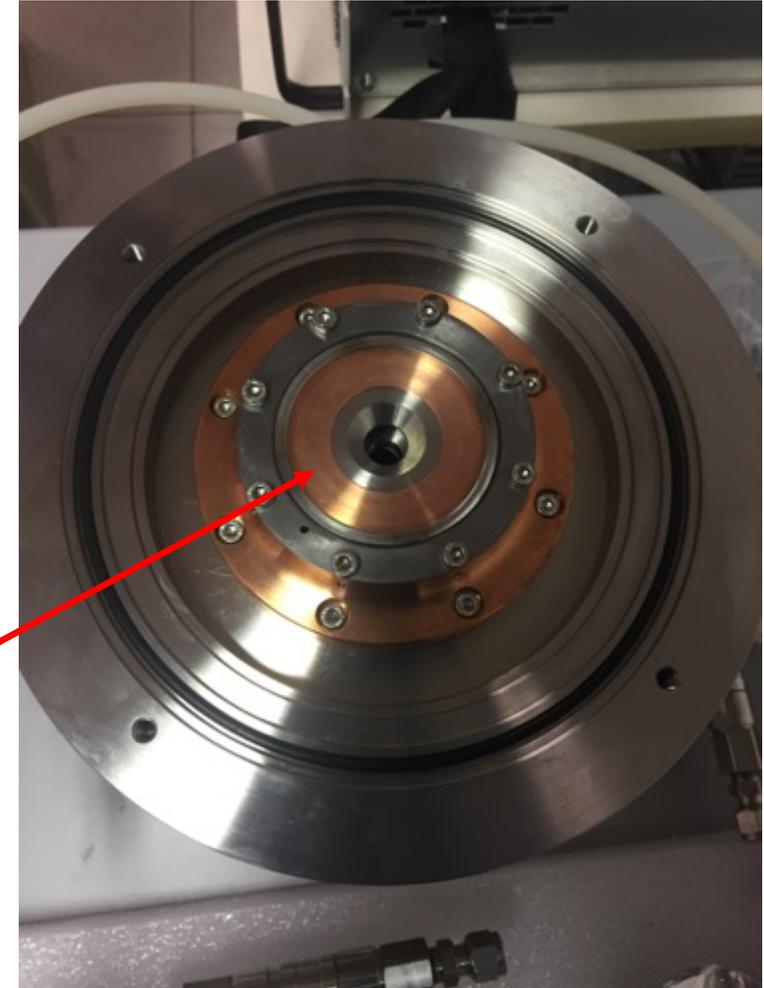
4mm FWHM at the slits



target chamber



# First implantation test



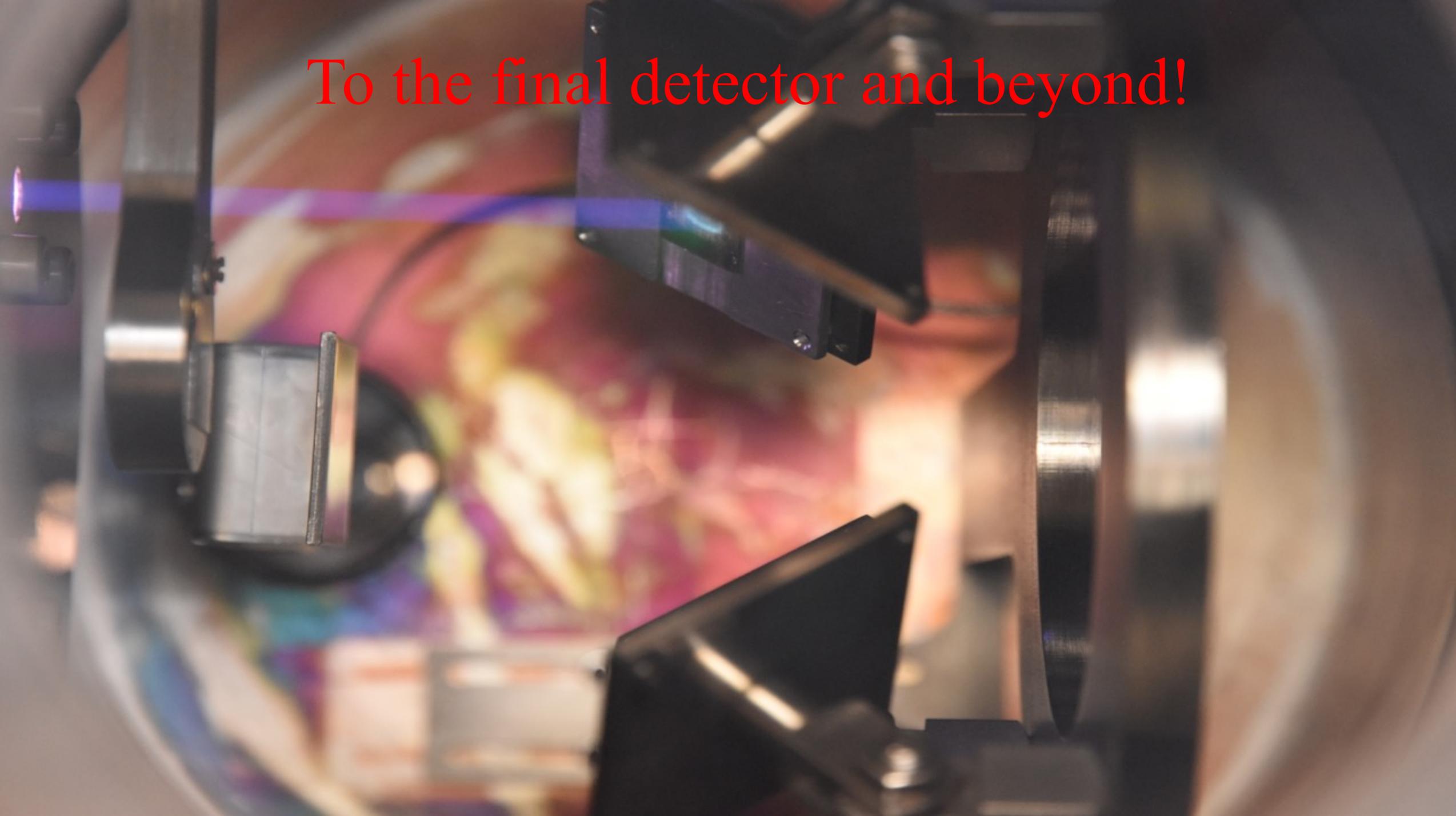
test in progress at INFN Genova

- no focusing
  - sputter target made in Cu
- measured current of  $100 \mu\text{A}$  (preliminary)**

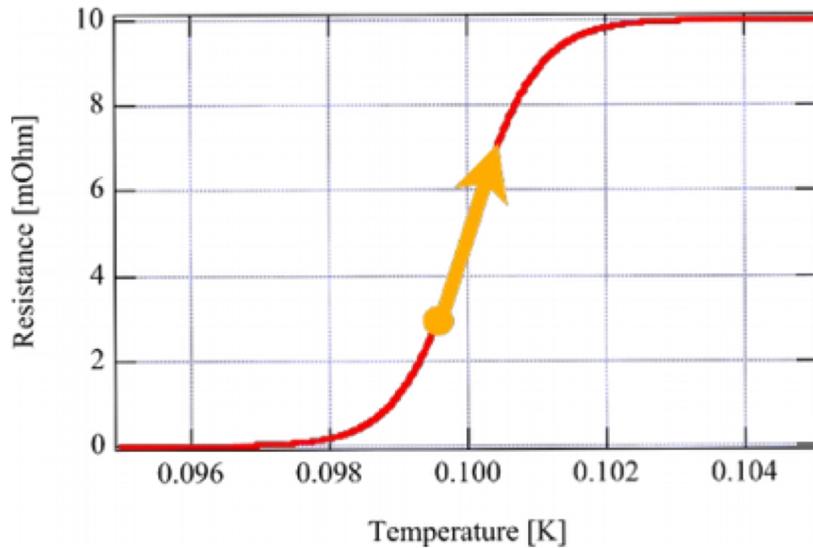
## Next steps

- Natural holmium ( $^{165}\text{Ho}$ )
- Test with  $^{163}\text{Ho}$

To the final detector and beyond!



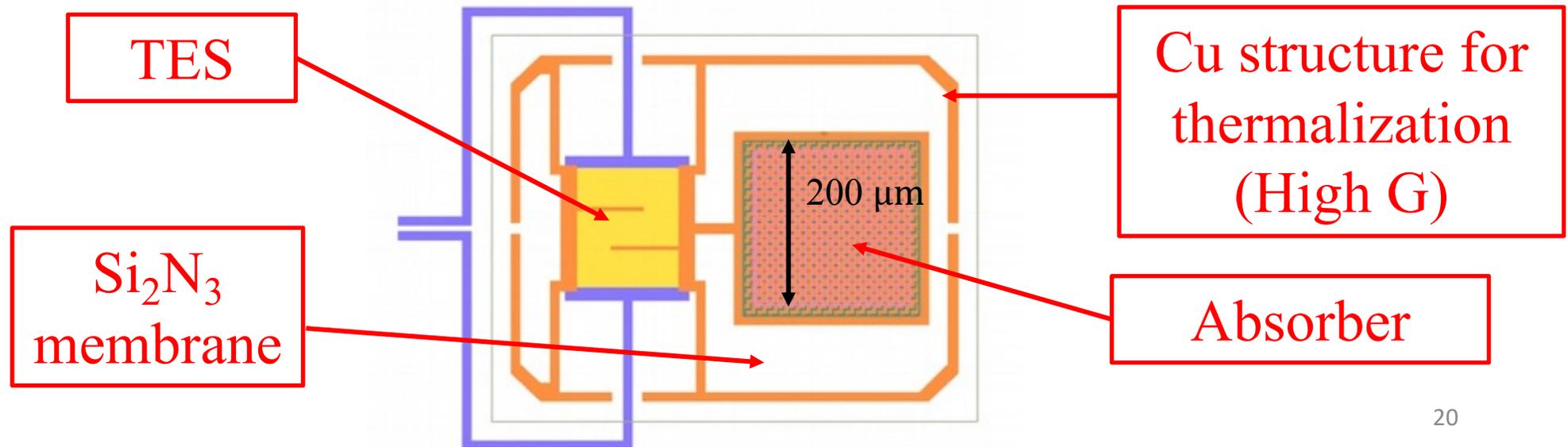
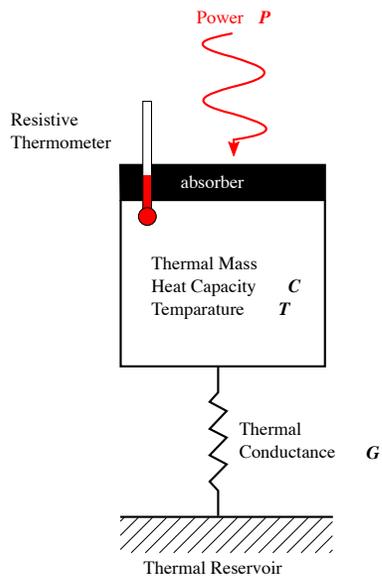
# Detector design and test (1)



## Transition Edge Sensors Superconductive Detectors (TES)

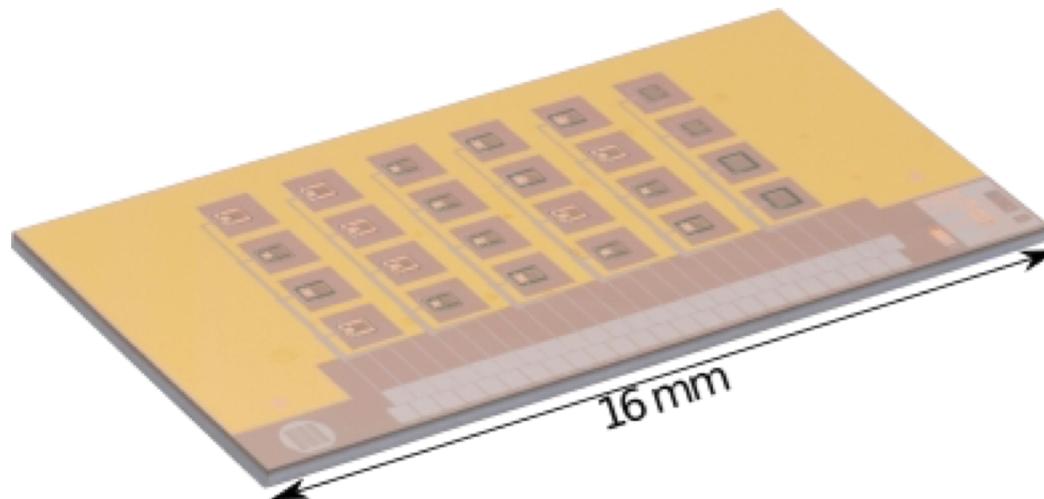
- Molybdenum/copper ( $T_c \sim 100$  mK)
- Very steep R vs T dependency in transition region;
- Gold absorber with  $^{163}\text{Ho}$  inside coupled to TES thermometer ;
- Ho sandwiched between two 1 mm thick gold layers for a total electron containment
- Fast detectors to reduce pile-up
  - tunable rise time  $\sim L/R$
  - decay time dependent on detector characteristics  $C/G$

TES design, production and preliminary test is done @NIST

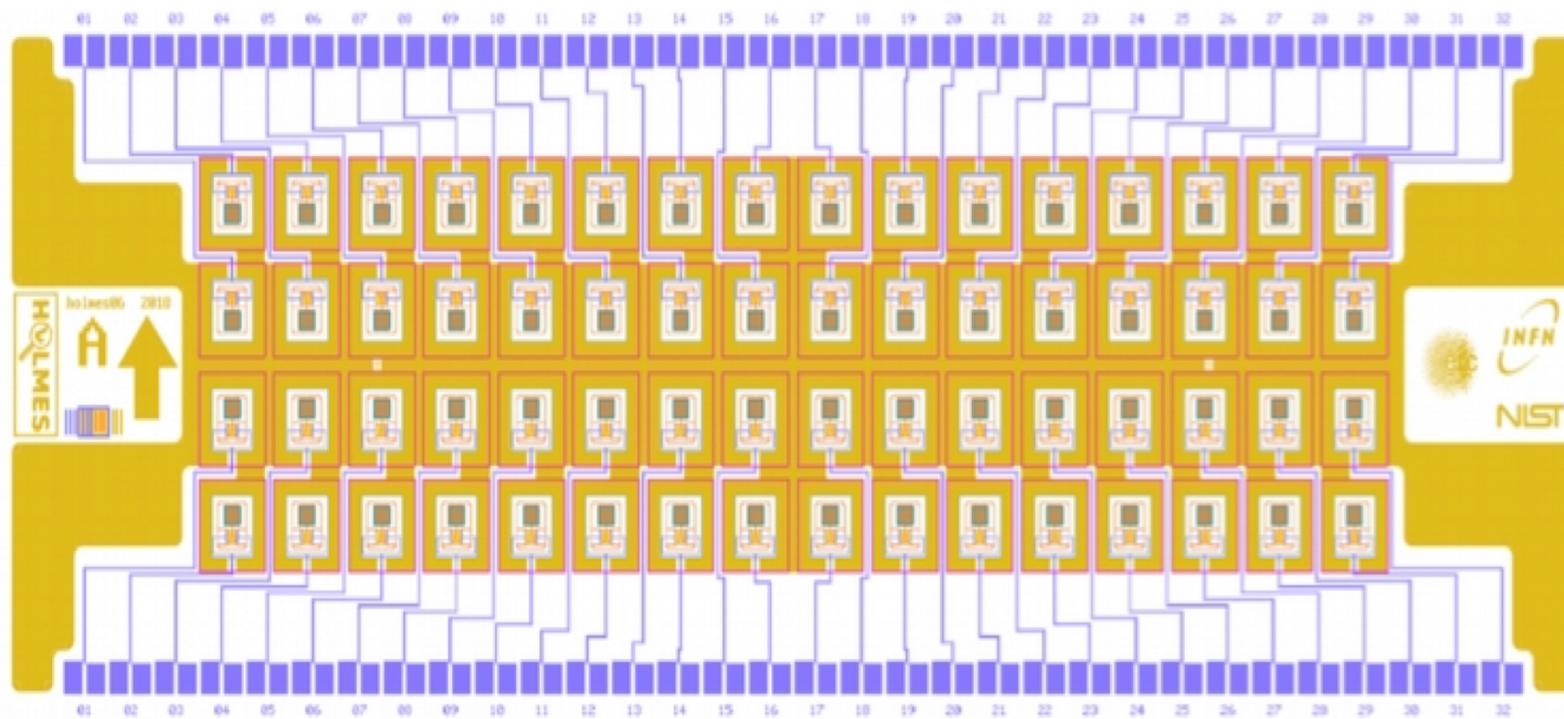


# Detector design and test (2)

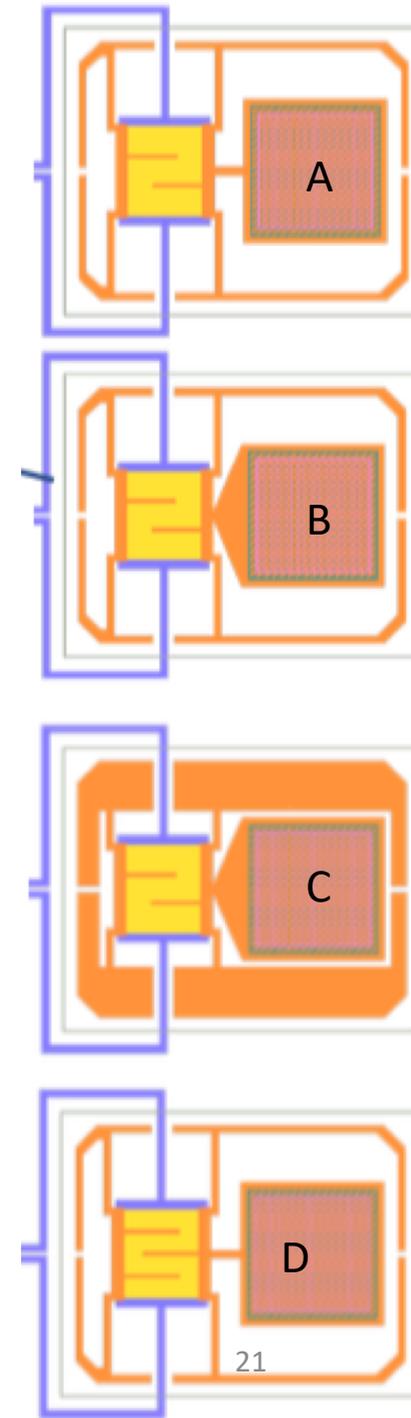
Four different designs to be tested  
Different thermal conductance  $G$   
Different TES intrinsic parameters



4 x 16 linear array



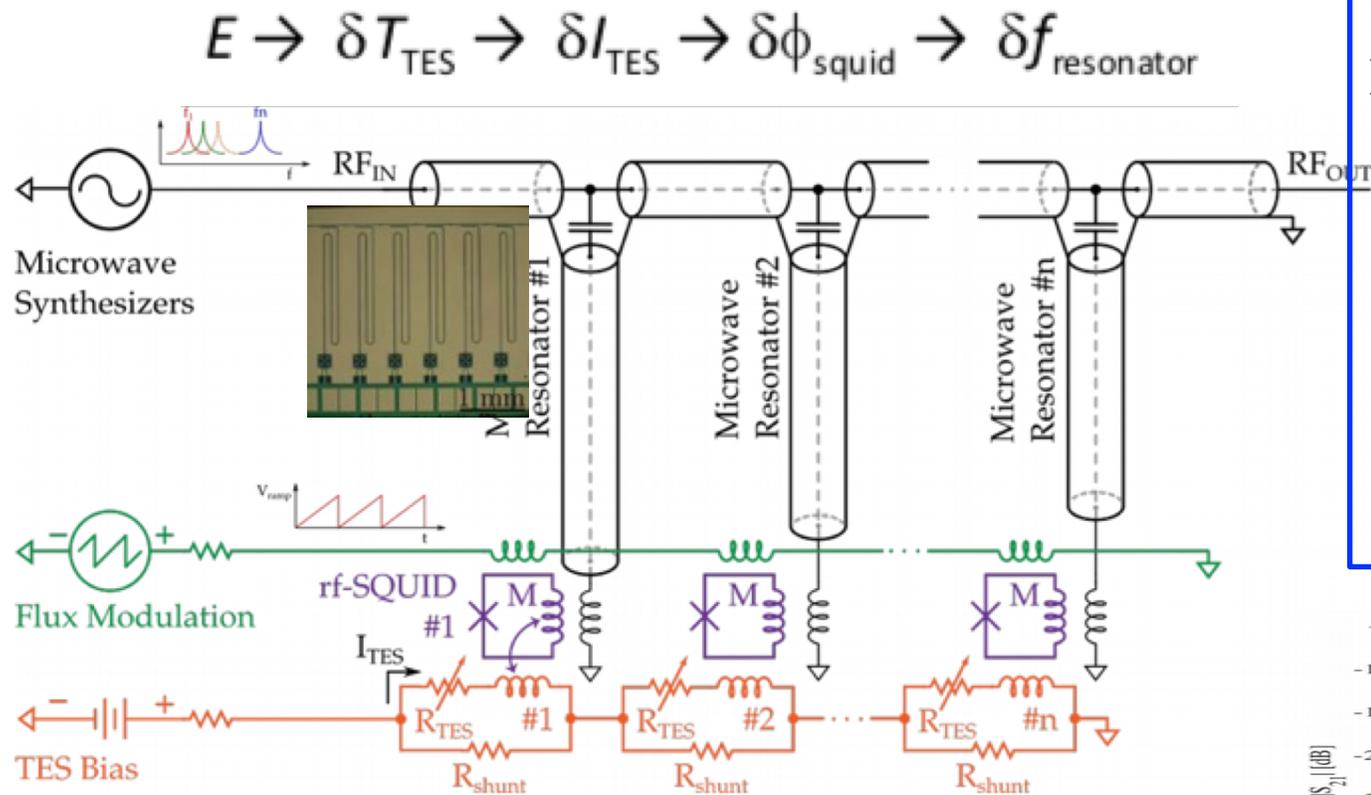
calculated  $^{163}\text{Ho}$   
beam FWHM



# Microwave multiplexing readout

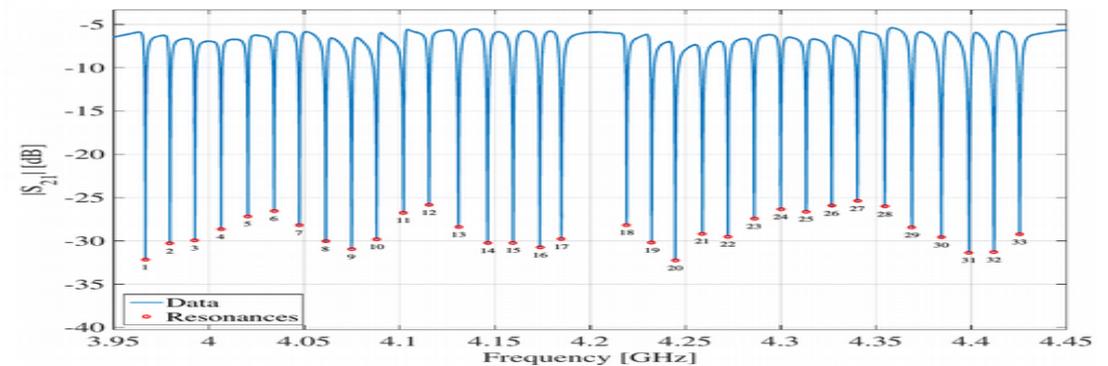
- Each TES is coupled to a RF-Squid
- Each RF-Squid is coupled to a common ramp
- Each RF-Squid is coupled to a resonant circuit (frequency multiplexing)

A comb of signals probe the resonators at their characteristic resonant frequency



Software Defined Radio with the open system ROACH2 (Casper collaboration)

- ADC BW 550 MHz
- real time pulse reconstruction
- Multiplexing factor proportional to the target ris
- time:  $n_{\text{TES}} = 3.4 \times 10^{-6} \times \tau_{\text{rise}}$
- 33 resonances in 500 MHz
- width 2 MHz
- separation 14 MHz



# Detector design and test (3)

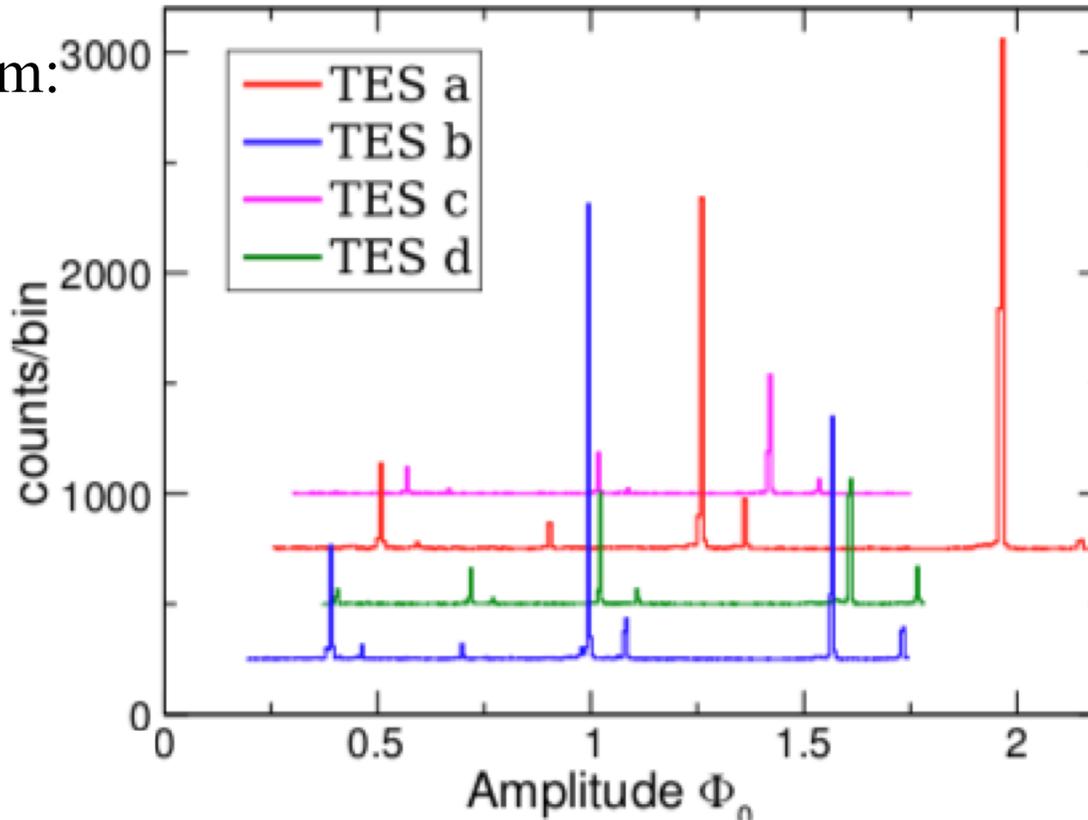
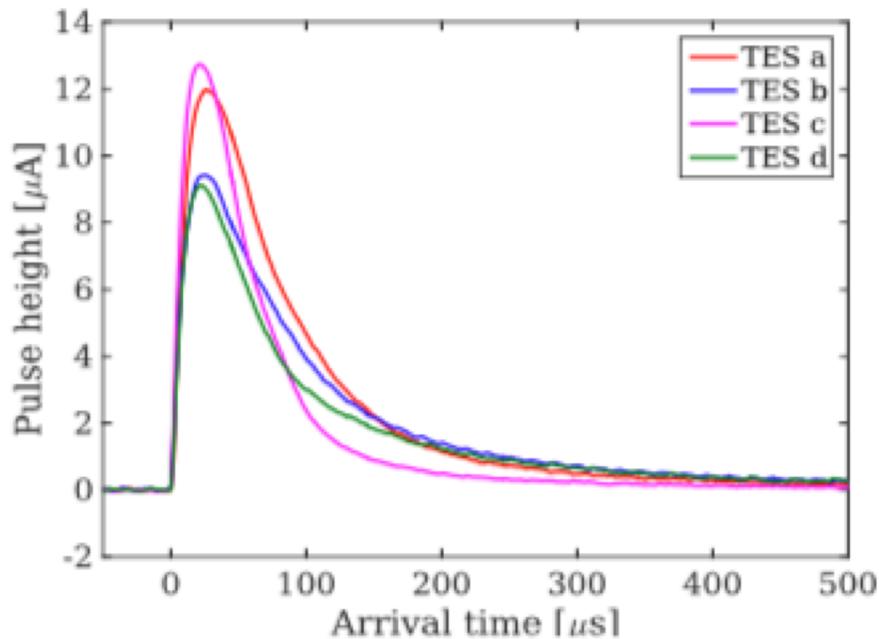
We tested the four different designs (non implanted detectors) to define heat capacity, thermal link geometry and TES design with:

$^{55}\text{Fe}$  (5.9 keV) + fluorescence from:

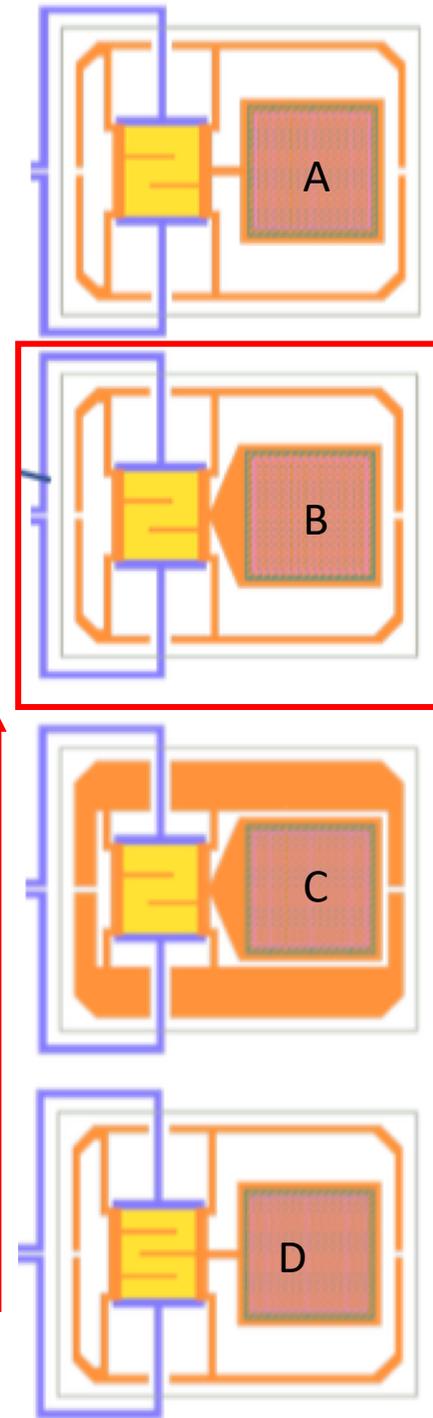
Ca – 3.7 keV

Cl – 2.6 keV

Al – 1.5 keV



Design B has the best performances



# Detector design and test (4)

We tested also the linearity.

Detectors have good linearity over a wide (0 keV to 6 keV) energy range.

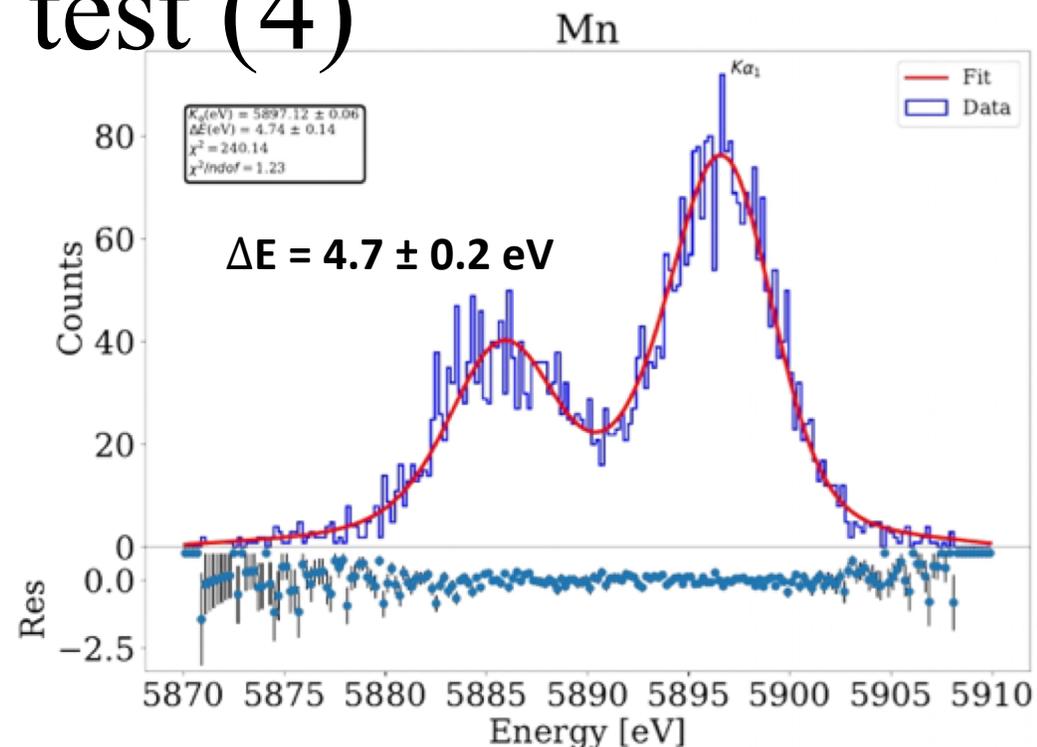
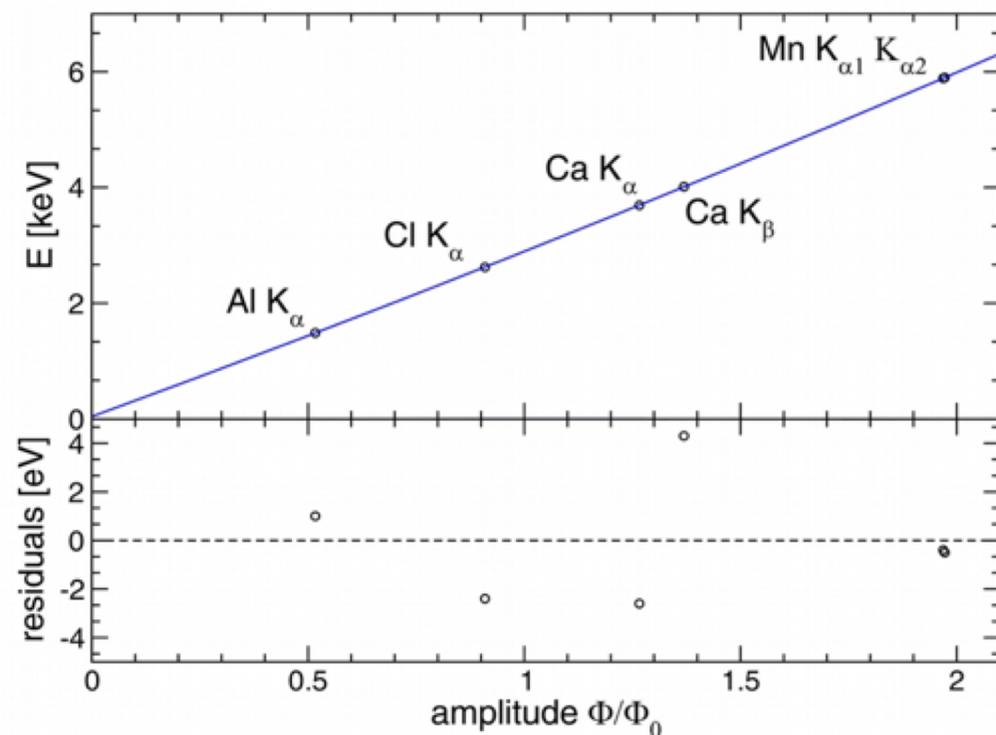
$\Delta E = 4.7 \text{ eV}$

$\tau_{\text{rise}} \sim 10 \mu\text{s}$

$\tau_{\text{decay}} \sim 190 \mu\text{s}$

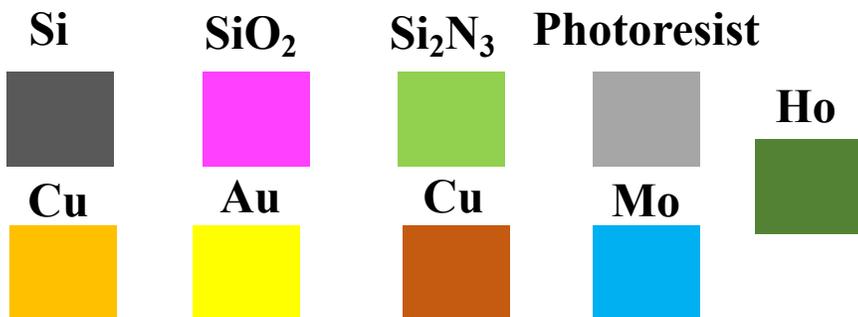
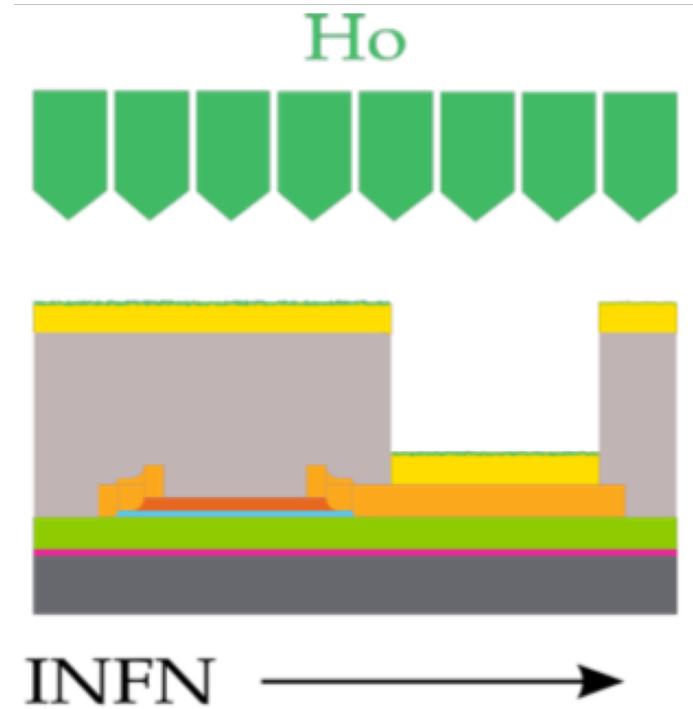
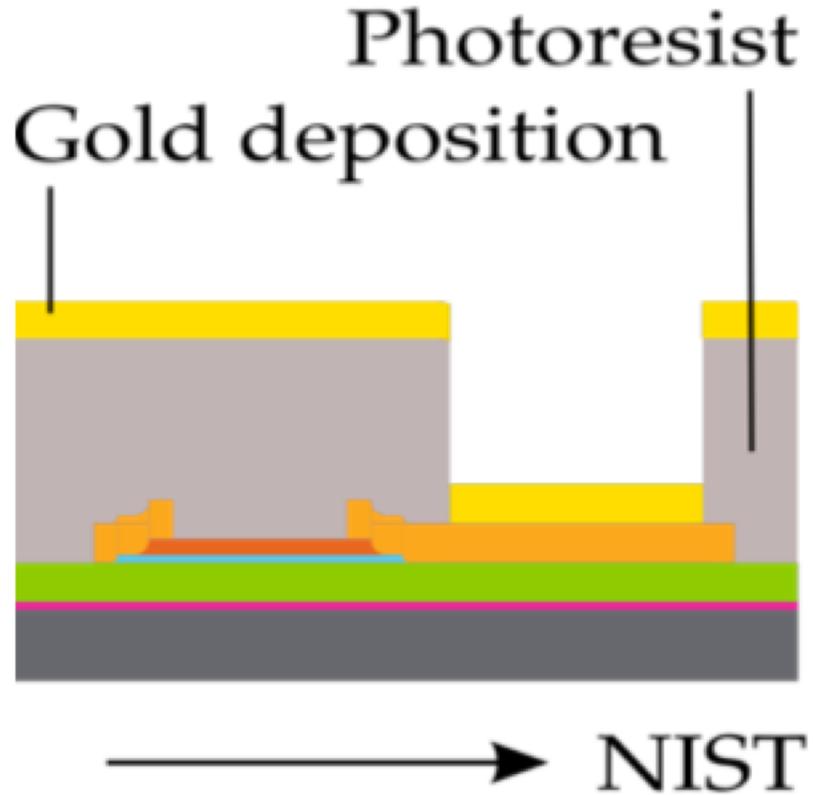
Calibration function:

$$E [\text{keV}] = 0.11927\phi_0^2 + 2.7345 \phi_0 + 0.041166$$



E [keV]	$\Delta E$ [eV]
1.49	$4.3 \pm 0.3$
2.62	$4.5 \pm 0.3$
3.69	$4.6 \pm 0.3$

# Detector fabrication (1)



<sup>163</sup>Ho concentration in absorbers saturate because <sup>163</sup>Ho sputters off Au from absorber. Maximum implantable activity ~ 3-4 Bq!!!

# Detector fabrication (2)

Four sputter ion sources coupled with the deposition chamber.

Effect compensated by Au co-evaporation.

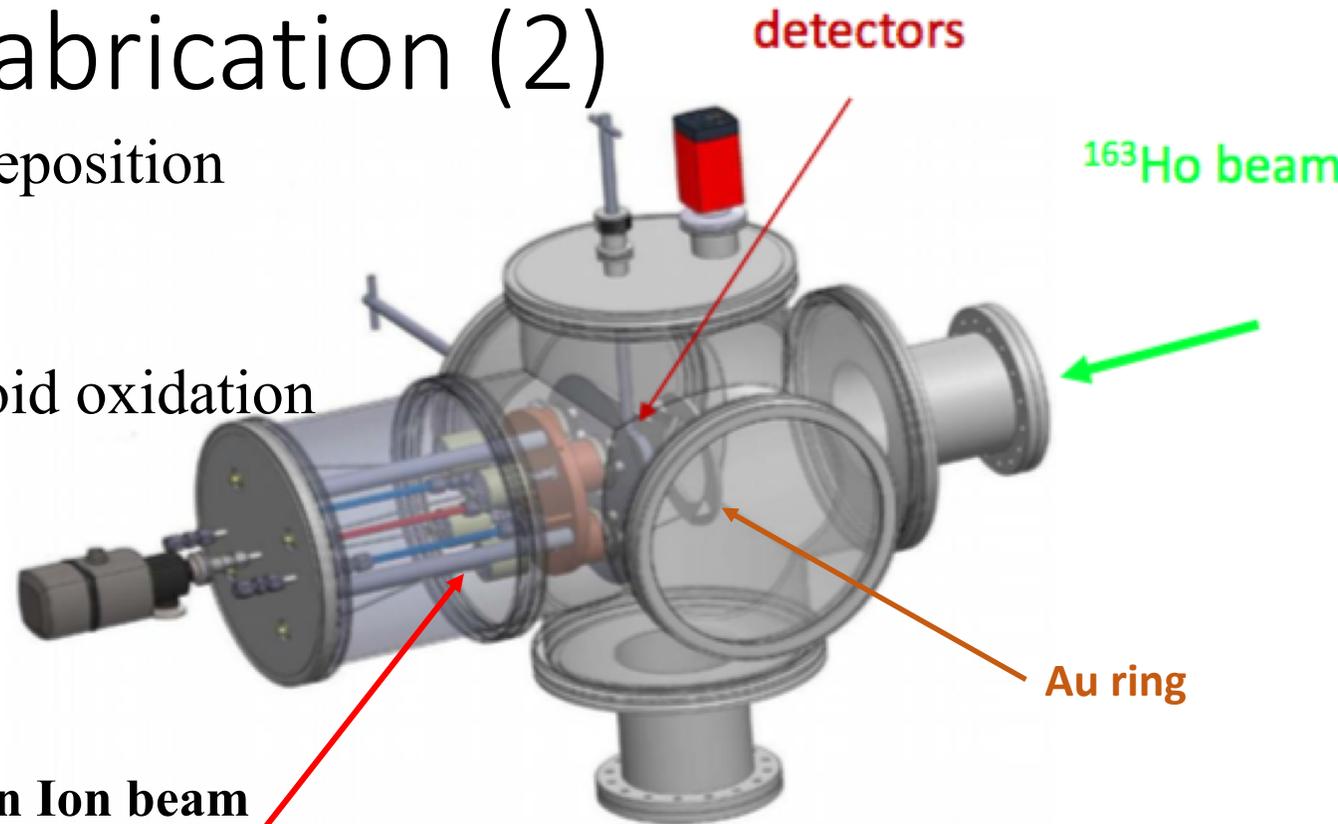
Final 1  $\mu\text{m}$  Au layer deposited in situ to avoid oxidation

**deposition rate (with 4 sputter sources)**

**$\approx 50\text{nm}/\text{hour}$**

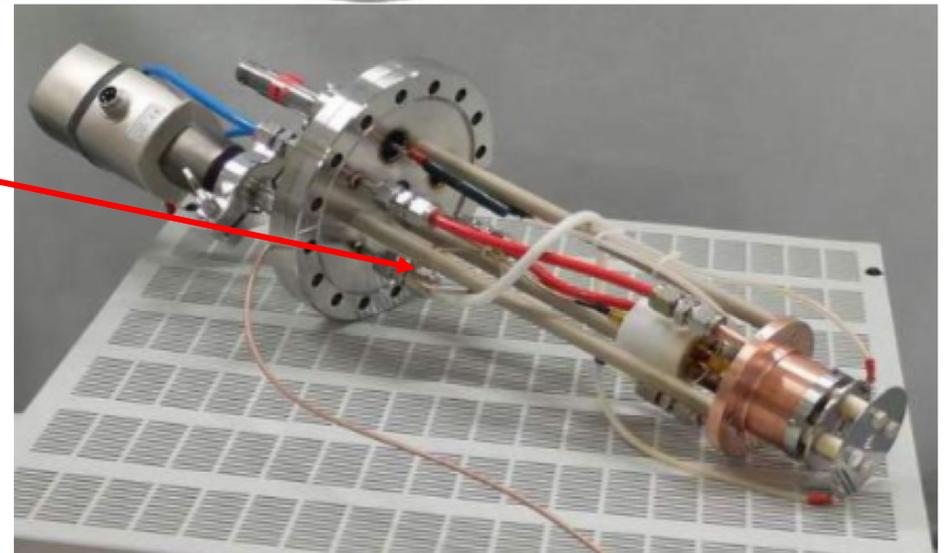
**$\approx 20$  hours to deposit 1  $\mu\text{m}$**

**High Uniformity**

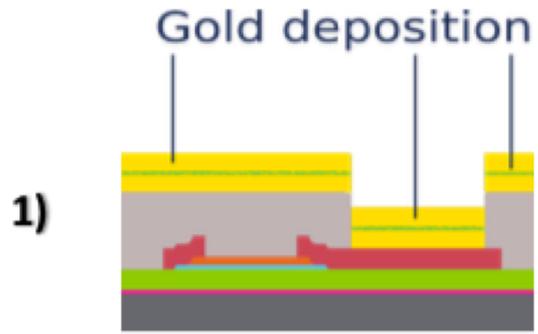


**Argon Ion beam**  
microwave sources  
for sputtering

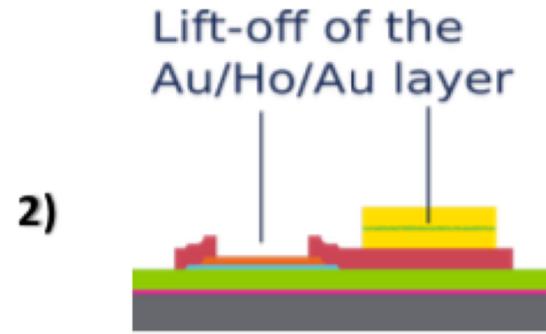
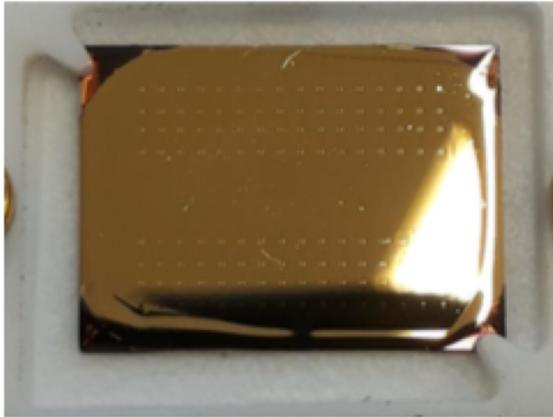
**Next step**  
Try to improve  
the deposition  
rate



# Detector fabrication (3)



1)



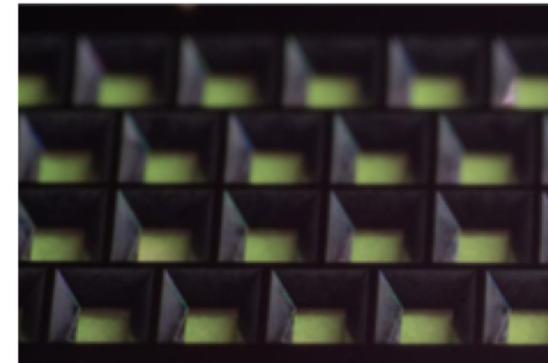
2)

Using Acetone

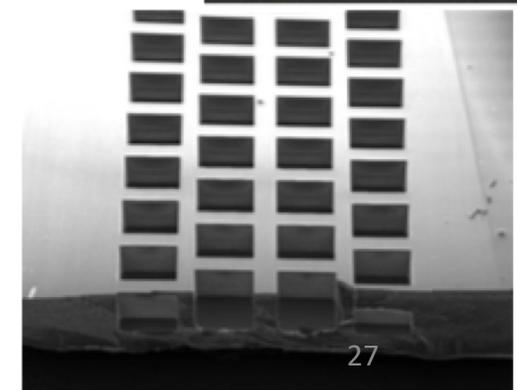


3)

KOH...



... vs DRIE



# Summary

The measurement of neutrino mass is still one of the most compelling issues in modern particle physics. Both calorimetric and spectrometric experiments are on going and could give a final answer.

HOLMES is a calorimetric experiment with  $^{163}\text{Ho}$  that aims to reach the statistical sensitivity around 1 eV.

Three batches of  $^{163}\text{Ho}$  are purified and ready to be moved in Genova.

The procedure to distillate holmium is tested. Some refinements are needed.

The procedure to fabricate sputter target is tested.

The installation of the implanter for isotopic separation is mainly finished

Every part has been individually tested. Integration and first tests in progress.

HOLMES detector production procedure is defined and the firsts (not implanted) detectors are being characterized.

Readout is on test and is almost ready.

With 32 pixels for 1 month  $\rightarrow$  m sensitivity  $\approx$  10 eV

BACKUP

# Cryostat

Detctor box is coupled to the Mixing Chamber of a  $^3\text{He}/^4\text{He}$  dilution refrigerator

