Developments of materials purification to produce calcium molybdate crystal for the AMoRE

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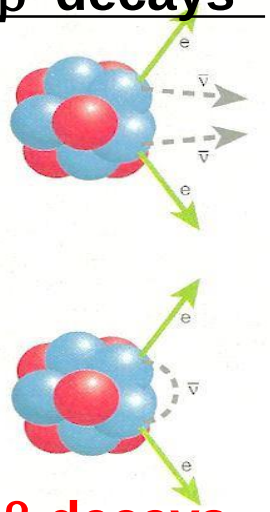
The AMoRE:

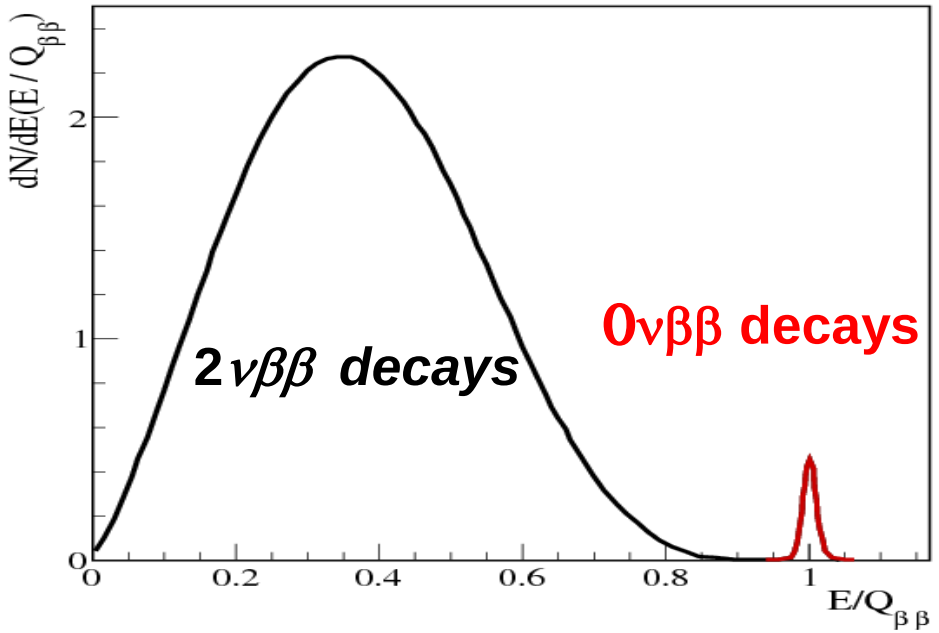
Advanced Mo-based Rare process Experiment

- **Search for 0νββ decays of ^{100}Mo**
  - **To answer if neutrino is a Dirac or Majorana particle**
- **The experimental signature for 0νββ decays**

2νββ decays



0νββ decays



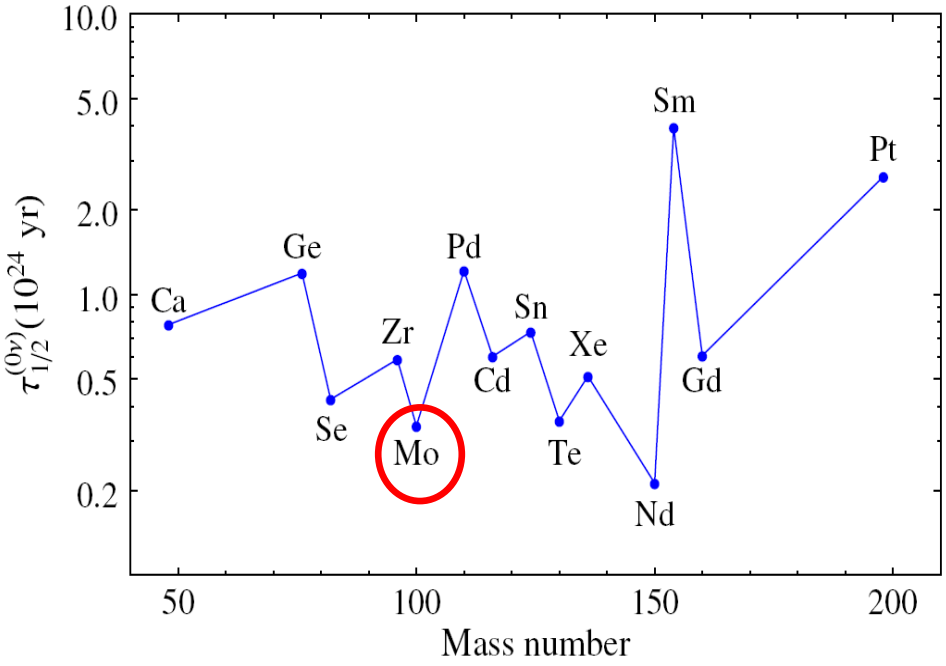
- **We uses $^{dep48}Ca^{100}MoO_4$ scintillating crystals**
  - **source=detector**

(See the talk "Status of the AMoRE…" by HongJoo Kim in the neutrino session.)

AMoRE Collaboration

Why we use ^{100}Mo for 0νββ search ?

- **High Q-value (ββ) of 3034.40 (12) keV.**
- **High natural abundance of 9.7%.**
- **Relatively short half life (0νββ) expected from theoretical calculation.**



Barea et al., Phy. Rev. Lett. 109, 042501 (2012)

Candidate	Q (MeV)	Abund. (%)
^{48}Ca	4.271	0.19
^{76}Ge	2.040	7.8
^{82}Se	2.995	8.7
^{100}Mo	3.034	9.7
^{116}Cd	2.802	7.5
^{124}Sn	2.228	5.8
^{130}Te	2.533	34.1
^{136}Xe	2.479	8.9
^{150}Nd	3.367	5.6

AMoRE Plan

- $^{dep48}Ca^{100}MoO_4$ crystals:
  - ^{100}Mo (> 95%) & $^{dep48}Ca$ (< 0.001%)

Internal backgrounds from crystals are dominated.

	Pilot	Phase I	Phase II
Mass	1.5 kg	5 kg	200 kg
Bkg [keV ·kg· year]$^{-1}$	10^{-2}	10^{-3}	10^{-4}
$T_{1/2}$ Sensitivity [years]	~10^{24}	~10^{25}	~5 x 10^{26}
$<m_{\beta\beta}>$ Sensitivity [meV]	300-900	100-300	15-40
Location	Y2L (700 m depth)		New deeper Lab.
Schedule	2016-2017	2017 - 2019	2020 - 2025

^{238}U & ^{232}Th: ~ μBq/kg in crystals for the Phase II

^{100}Mo enriched & ^{48}Ca depleted materials

- $^{100}MoO_3$ (^{100}Mo > 95%) powder, produced by the ECP, Russia
- $^{dep48}CaCO_3$ (^{dep48}Ca < 0.001%) powder produced by the ELECTROCHIMPRIBOR, Russia

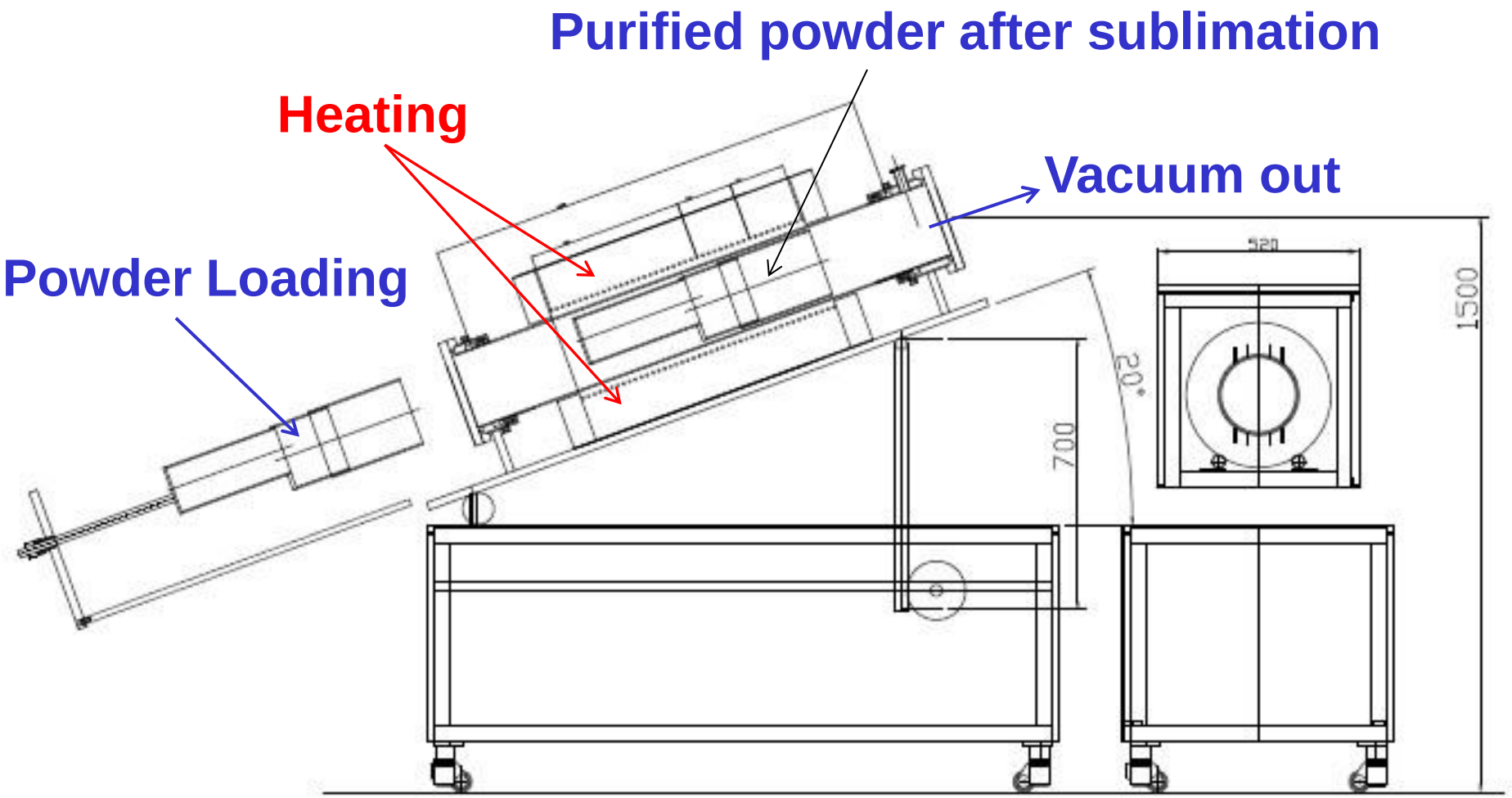
Impurities of powders in mBq/kg unit

	^{238}U	^{232}Th	^{226}Ra	^{228}Ac
MoO_3 powder	1.8	< 2.4	8.3	< 1
$CaCO_3$ powder	< 1.2	< 2.4	5.9	1

By purification of powders and crystal growing process:
- Reduction of impurities by a factor of 1,000
- High recovery efficiency (> at least 90%).
-> (100 by purification of powder) x (10 by crystal growing)

Purification of MoO_3 powder: Sublimation method (I)

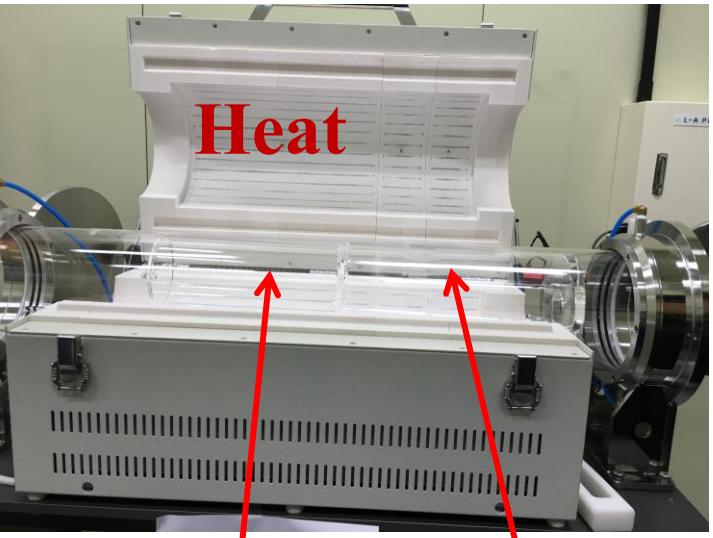
- **Schematic diagram for the equipment**

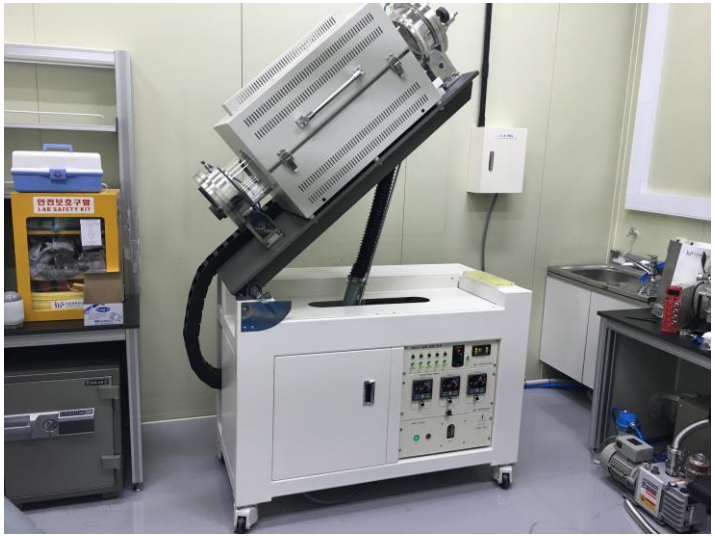


Purification of MoO_3 powder: Sublimation method (II)

- **MoO_3 has the transition from the solid to the gas phase around 700 ^oC.**

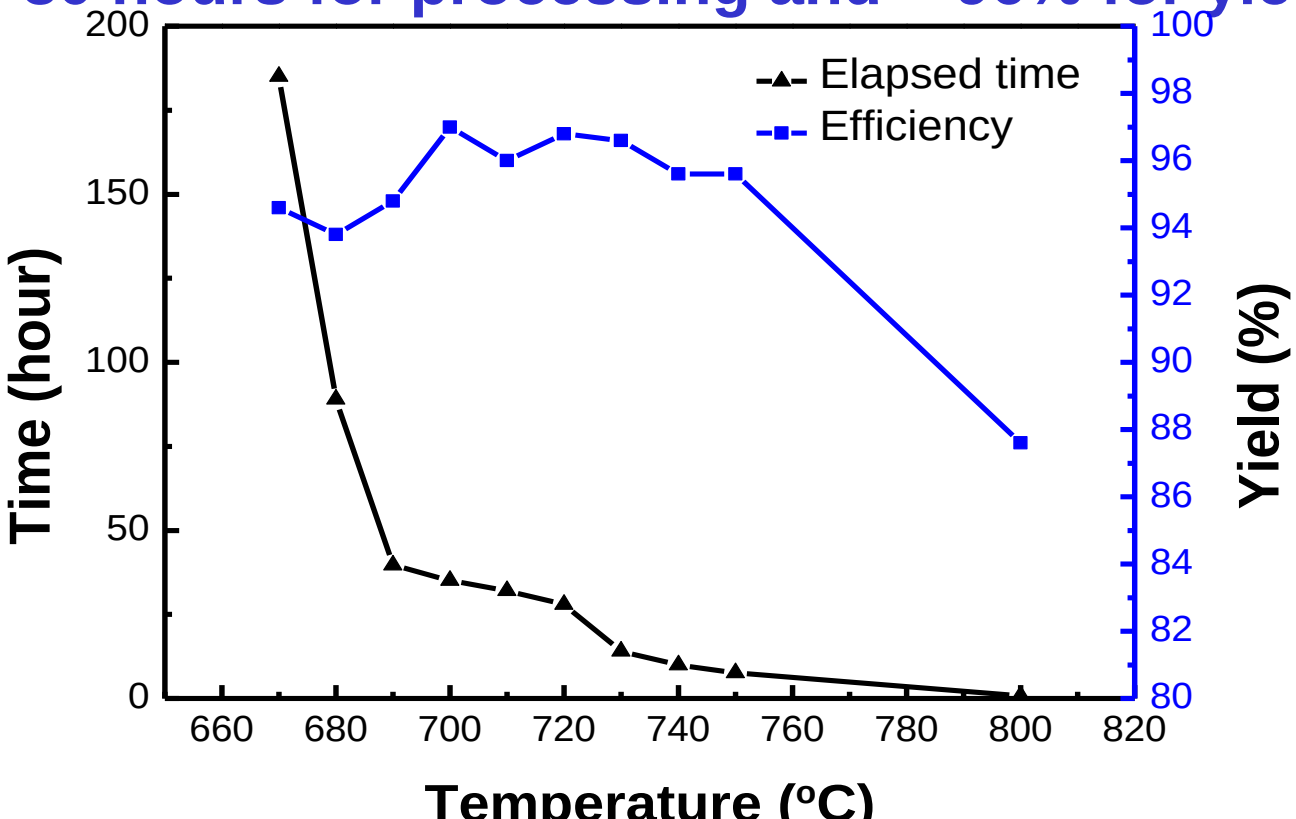
  -> Some impurities, U/Th, are still in the solid phases.





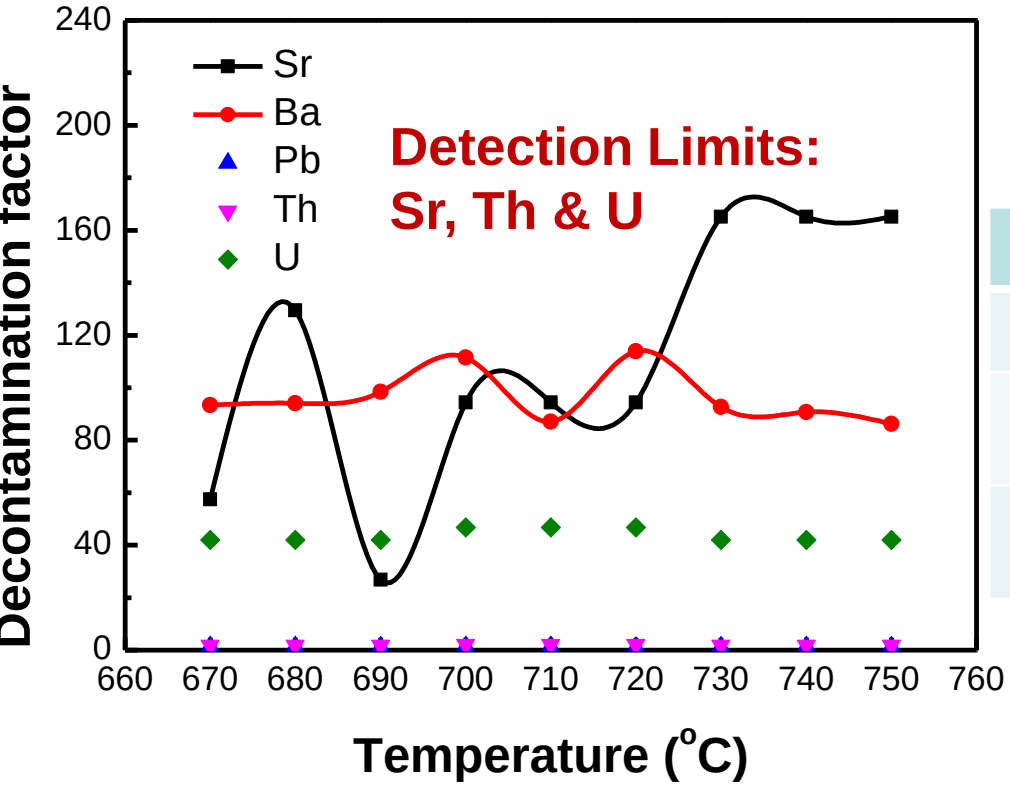
Purification of MoO_3 powder: Sublimation method (III)

- **Processing time and yield from 670 ºC to 800 ºC with 50 g of natural MoO_3 powder.**
  - **Around 700 ºC:**
    ~ 30 hours for processing and ~ 96% for yield



Purification of MoO_3 powder: Sublimation method (IV)

- **Decontamination factor from 670 °C to 750 °C.**
  - **DF=(initial impurity)/(final impurity)**



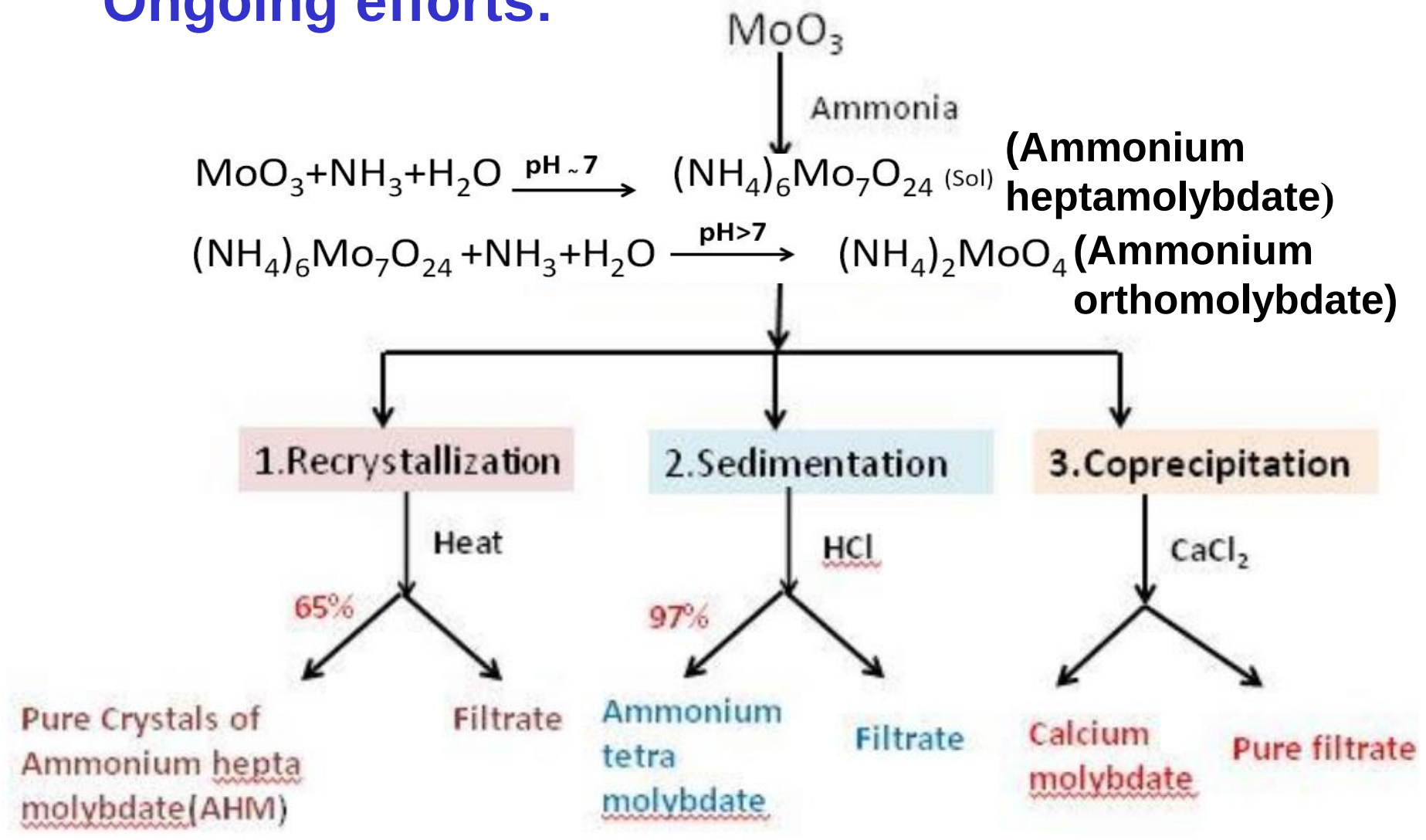
ICP-MS results at 720 °C (ppt unit)

	Sr	Ba	Th	U
Initial	6,605	1.37M	224	4,205
final	<70	0.012M	<100	<90
Df	>94	113	> 2	> 46

HPGe measurements to check ^{226}Ra & ^{228}Ac are ongoing.

Purification of MoO_3 powder: Chemical methods

Ongoing efforts:



Purification of CaO_3 powder: Column Chromatography (I)

- **Starting process:**
  $CaCO_3 + HNO_3 \rightarrow Ca(NO_3)_2 + CO_2 + H_2O$
- **Affinity of MDM column (based Mn):**
  Sr < Ba < Ra

Purification of CaO_3 powder: Column Chromatography (II)

Synthesizing $CaMoO_4$ powder (I)

- **Usual process to grow CMO crystal:**
  - **Mix $CaCO_3$ and MoO_3 powders with 1:1 ratio and apply heat**

- **Recently we develop a new method:**
  - **In $CaCO_3$ and MoO_3 powders purification:**

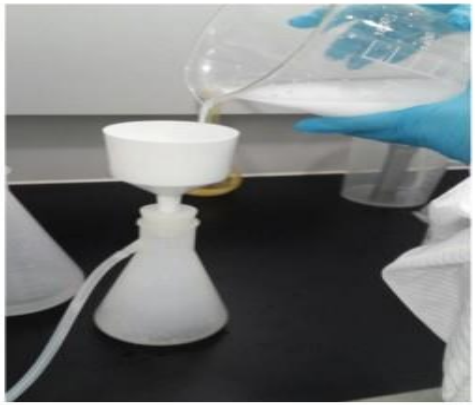
$$CaCO_3 + HNO_3 \rightarrow Ca(NO_3)_2 + CO_2 + H_2O$$

$$MoO3 + NH3 \rightarrow (NH_4)_2MoO_4 + \text{etc.}$$

Synthesizing $CaMoO_4$ powder (II)

- **Mix together,**
  Purified $Ca(NO_3)_2$ sol.+ Purified $(NH_4)_2MoO_4$ sol.
  -> $CaMoO_4 \downarrow$ + $2NH_4NO_3$ (Ammonium nitrate solution)

Precipitate of CaMoO4

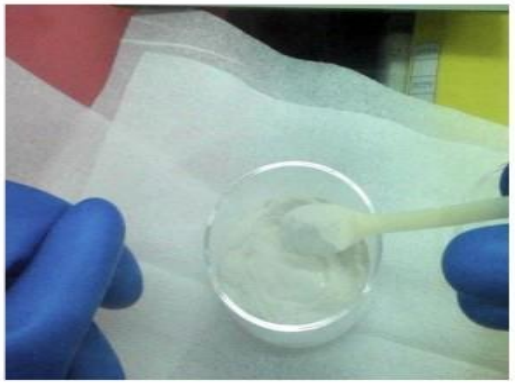


Vacuum filtration

CaMoO4 powder

CaMoO4 powder before heating

Calcination

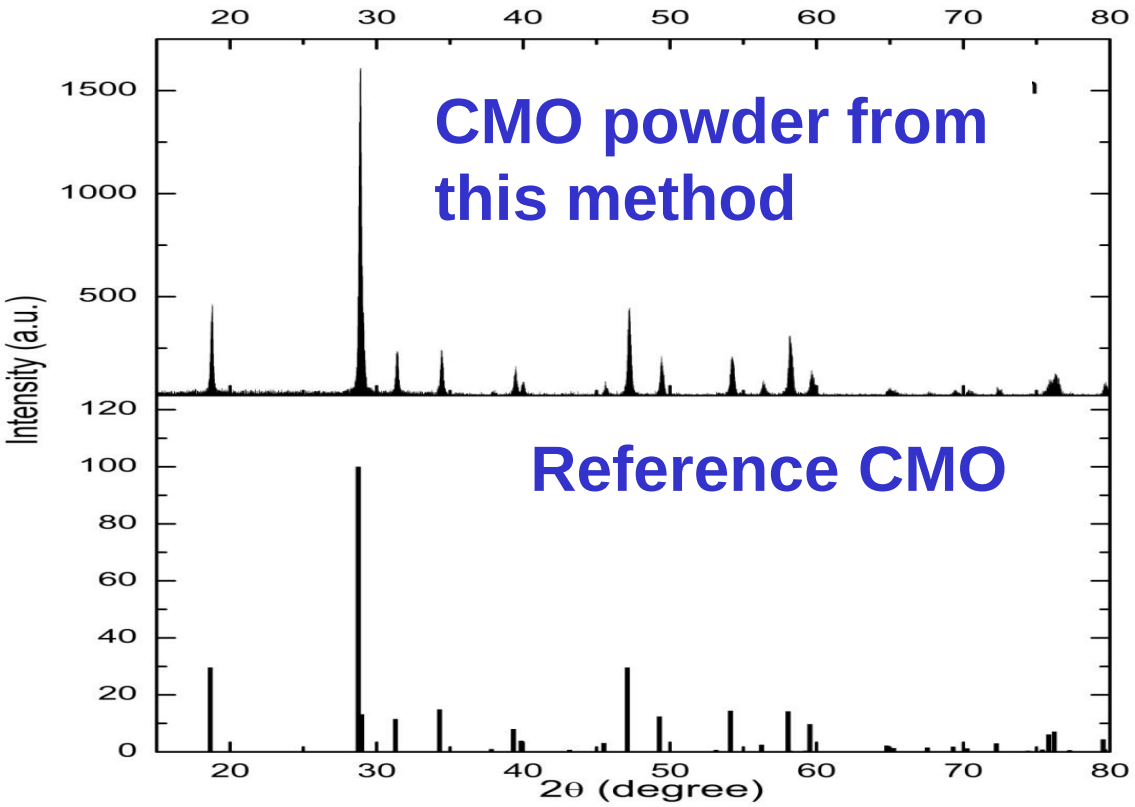


CaMoO4 powder

800° for ~4 hr or drying up in the vacuum dryer.

Synthesizing $CaMoO_4$ powder (III)

- **X-ray diffraction analysis**



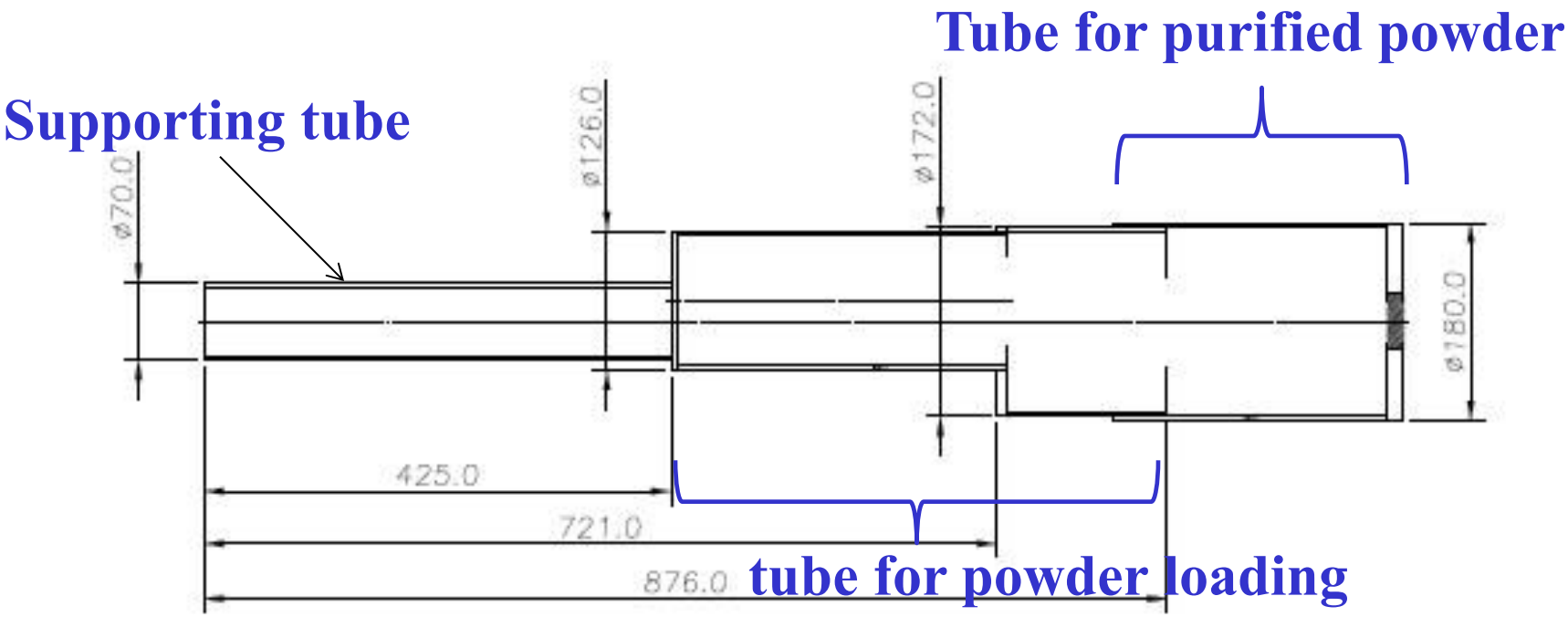
- **We are planning to grow CMO crystal with this method**

Summary

- We have studied the sublimation method for MoO_3 powder purification:
  - DF > 46 for ^{238}U, DF > 2 for ^{232}Th
  - ^{226}Ra reduction check with HPGe will be done.
- Chemical purification methods for MoO_3 and $CaCO_3$ powders are under studies.
- We have developed the new method to make CMO powder.
- A new detection technique is necessary to lower current detection limits.

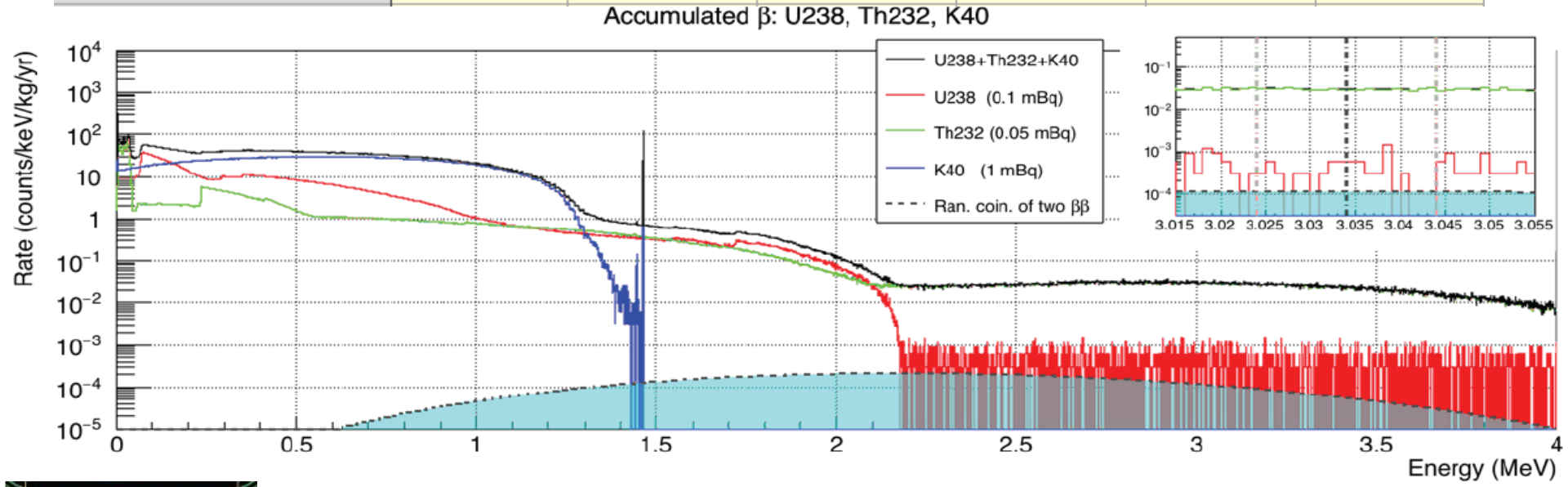
Sublimation Equipment (II)

Quartz tubes for powder loading & purification



Internal background simulation for AMoRE-I

		Pb210	U238	Th232	K40	U235
Concentration		10 mBq/kg	0.1 mBq/kg	50 μBq/kg	1 mBq/kg	1 mBq/kg

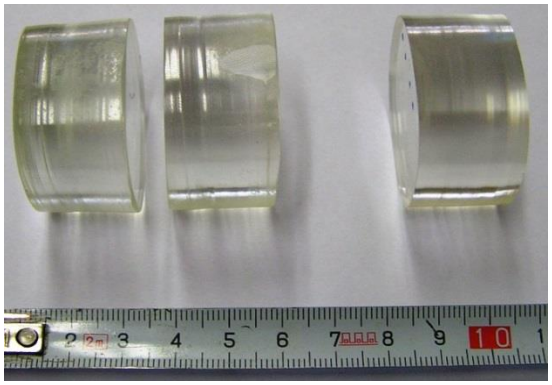


- ^{208}Tl with α-tagging : 0.0018 DBU.
- Random coincidence of 2νββ of ^{100}Mo: 1.2x10^{-4} DBU.

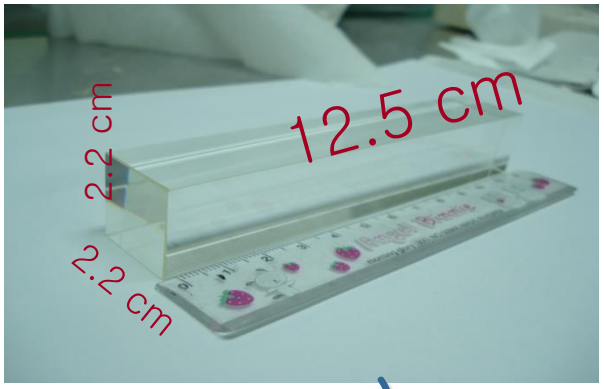
-> Goal of 0.002 for AMoRE-I can be achieved

$CaMoO_4$ crystal development

Korea(2003)



Ukraine-CARAT(2006)



Russia(2006)

30x30x200mm

$^{40}Ca^{100}MoO_4$ Crystals for AMoRE-pilot


SB28 (196 g)


SB29 (390 g)


S35 (256 g)


SS68 (350 g)


SE1 (354 g)

Total mass : 1.546 kg

- **All crystals for AMoRE-pilot are in the cryostat.**

Low background Crystal growing facility

- **Main goal**
  - **$CaMoO_4$ crystal growing R&D for AMoRE-200**
  - **Other DB or DM crystal R&D**
- **Deep purification of $CaCo_3$ and MoO_3 powders (<50 μBq/kg for U,Th chain)**
- **Crystal growing equipment:**
  1 Czochalski, 2 Kyropoulous, 1 Bridgman crystal growing machine.

Czochalski machine

The 1st CMO crystal by us.