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# Update of LAB, acrylic and PPO measurements at Bicocca

**JUNO Italia meeting – Università di Roma Tre**
**28-29th March 2023**

**Speaker: Nastasi Massimiliano**

# LAB radiopurity measurements

# Radiopurity LAB measurements

In the last months we have worked on a **measurement procedures** suitable to achieve the required sensitivity

**Juno baseline request for LAB:**
$^{238}U$, $^{232}Th$, $^{40}K$ $< 1 \cdot 10^{-15}$ g/g

In the latest test (before July 2022) we have achieved sensitivity at ppq level for $^{238}U$, $^{232}Th$ and $^{40}K$

*(JUNO-doc-8698-v1)*

After July 2022:

**Uranium and Thorium** → We studied the repeatibility of the method

**Potassium** → We studied chemical/radiochemical efficiency process

# Main steps for $^{238}U$ and $^{232}Th$ measurements

**Cleaning protocol (Pre-Irradiation)** → Any manipulation or treatment of the sample could introduce contaminations before irradiation

**Chemical/Radiochemical Treatments (Pre-Irradiation)** → Allows to **remove interferences** and **concentrate** the sample

**Sample irradiation** → Allows to transform long life nuclide $^{238}U/^{232}Th$ into the radioactive short life $^{239}Np/^{233}Pa$ nuclide. Sensitivity <1ppt

$$^{232}_{90}Th + \mathrm{n} \longrightarrow {}^{233}_{91}Pa \qquad {}^{238}_{92}U + \mathrm{n} \longrightarrow {}^{239}_{93}Np$$

**Radiochemical Treatments (Post-Irradiation)** → Remove **remaining** interferences

**γ measurements** → We developed a new detector suitable to **β – γ coincidence** measurements on liquid irradiated samples

# Blank measurements

In **september 2022** we have performed a new measurement on a blank sample

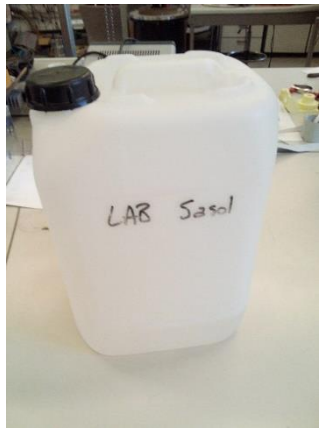
The blank went through all processing steps **just without LAB**

| Date | Blank | $^{238}$U[g/g] | $^{232}$Th[g/g] |
|---|---|---|---|
| March 2022 – T1 | Mass sample 228g | $(7,9\pm1,4)\cdot10^{-15}$ | $<4,4\cdot10^{-14}$ |
| **Sept 2022 – T2** | Mass sample 230g | **$(9,5\pm1,0)\cdot10^{-15}$** | **$<2,4\cdot10^{-14}$** |

limits @ 90% C.L.

Results achieved in September 2022 confirm the **reliability** of the measurements

Rescaling results for a LAB mass of 1kg we could achieve a sensitivity:
**$2\cdot10^{-15}$g/g** for **$^{238}$U - $5\cdot10^{-15}$g/g** for **$^{232}$Th**




# Preliminary measurement:

In **october 2022 p**rocedure has been tested on **SASOL LAB sample**

| **Sample** | **Mass[kg]** | **$^{238}$U [g/g]** | **$^{232}$Th[g/g]** |
|---|---|---|---|
| SASOL LAB | 1 | $(4.9\pm0.3)\cdot10^{-14}$ | $<4.6\cdot10^{-15}$ |

# Radiopurity LAB – $^{40}$K – July 2022

Sample and container cleaning **preparation**

↓

**LAB + STD reference has been irradiated**

↓

**γ measurements (HPGe)**

↓

**Calculation** of the **quantity of precursor** element

To study potassium contamination we irradiated a **Distilled LAB sample** (mass: **19g**)

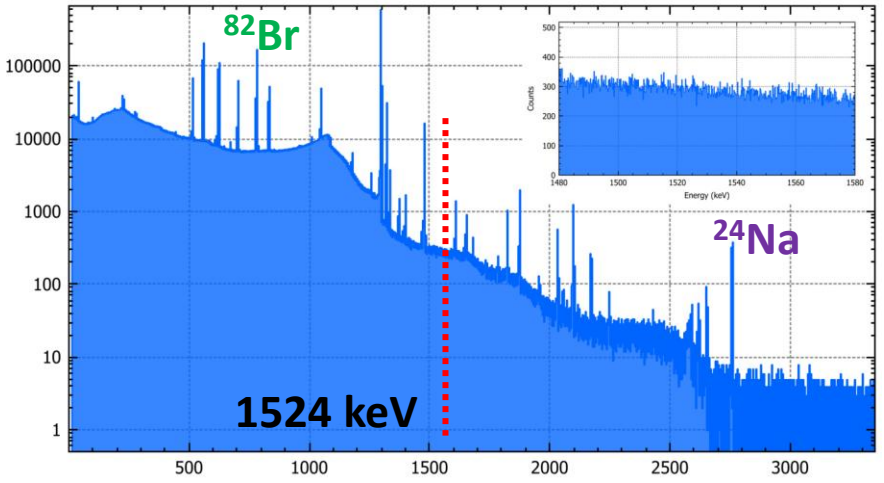
$$n + {}^{41}K \longrightarrow {}^{42}K \xrightarrow[12{,}36\,h]{\beta^-} {}^{42}Ca$$

Isotopic abbundace$K$:
$K^{39} \rightarrow \sim 93\%$
$K^{40} \rightarrow \sim 0{,}01\%$
$K^{41} \rightarrow \sim 7\%$

| ϒ-ray(keV) | BR(%) |
|---|---|
| 312.6 | 0.34 |
| 1524.6 | 17.64 |

| $^{40}$K [g/g] |
|---|
| **< 8,3·10$^{-15}$** |

limits @ 90% C.L.



# Potassium procedure

Sample and container cleaning **preparation**

↓

**LAB + STD reference has been irradiated**

↓

**Chemical/Radiochemical Treatments**

↓

**γ measurements (HPGe)**

↓

**Calculation** of the **quantity of precursor** element

In order to **remove interferences** and **concentrate** the sample we have introduced two steps

***Liq-Liq Extraction***

↓

***Gravimetric precipitation***

Allow to transfer the contamination of **K** from **LAB sample** into a liquid solution(**$NaNO_3$ + water**)

Precipitation reaction allow us to **separate** potassion and deposit it on a glass filter

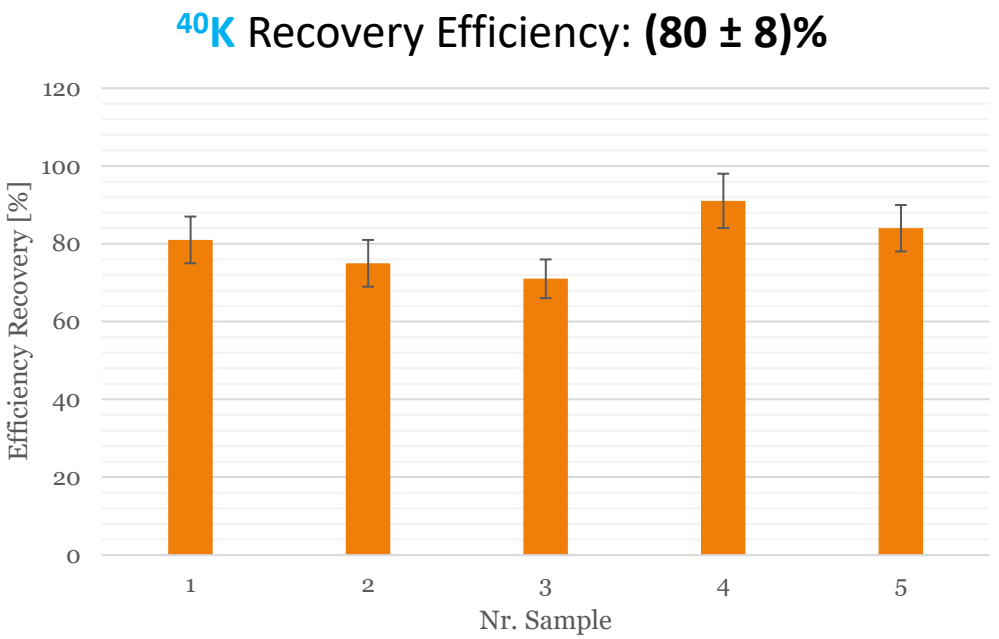
**Well detector:** $\varepsilon_{rel}$ 60%

# Preliminary results

| Nr.Sample | LAB - Mass[g] |
|---|---|
| 1 | 28 |
| 2 | 29 |
| 3 | 27 |
| 4 | 24 |
| 5 | 25 |

$^{24}Na - \in_{Removal}$ ~ 91%

$^{82}Br - \in_{Removal}$ ~ 99%



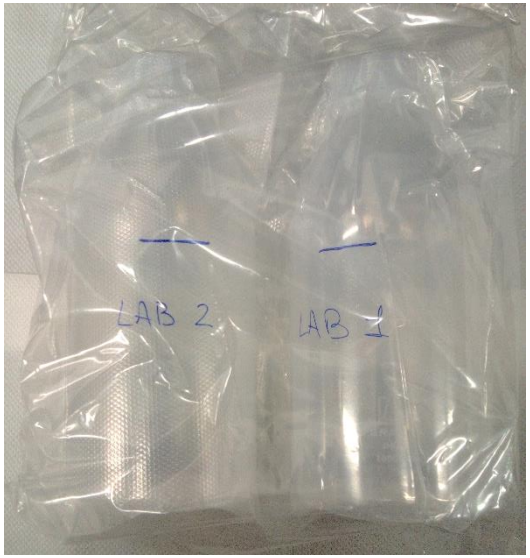

We are going to test the procedure on
**LAB samples**

It's crucial to **pay attention** at the **containers** in which LAB is stored

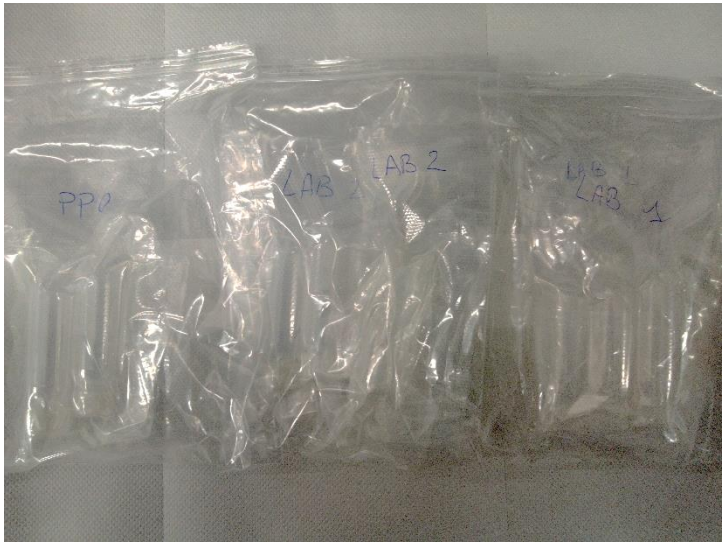
# Future plan:

We sent to China several containers in order to get LAB samples

**Containers** have been **conditioned** with nitric acid and packed in **vacuum bags**








We hope that we will receive some samples in next weeks

# Preliminary LAB measurements plan:

**Coming soon**

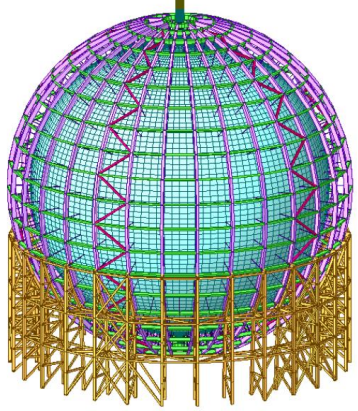
**Next delivery**

Raw LAB → Allumina → Distilled → Mixing System → Underground pipes → Water extraction → Steam Stripping → Filling tanks

**= Measurement <u>after</u> each step**

We will perform an **irradiation of 4 samples** for each step

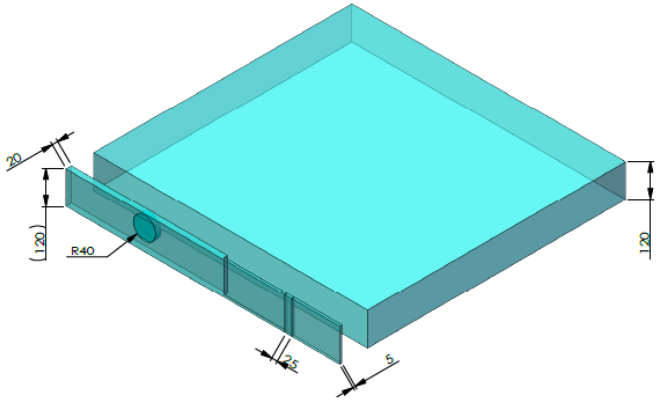
Live time: **~ 1 month**

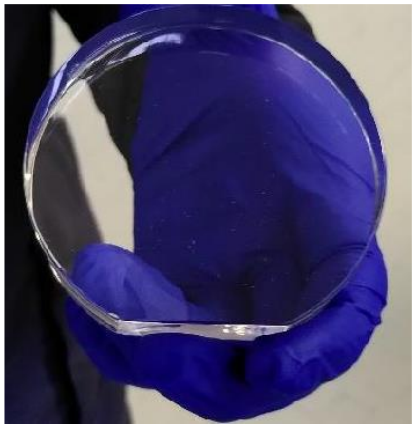


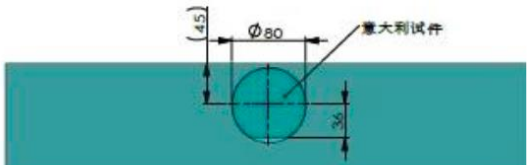
**Total Live Time: ~ 8 months** → It's crucial to define a measurements strategy

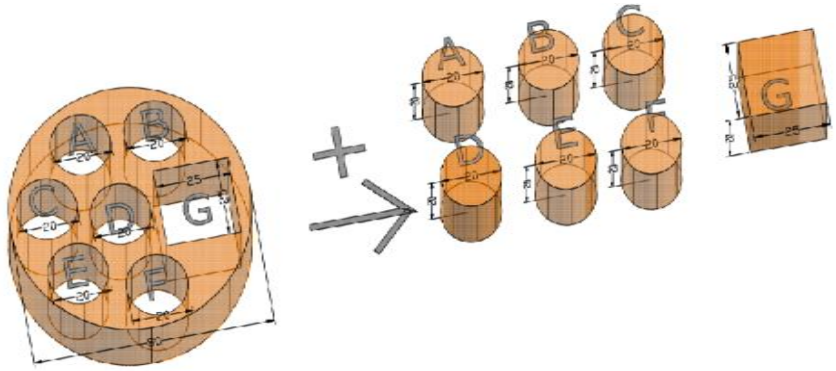
# Acrylic mass production quality control by NAA

# Acrylic: Radiopurity measurements






**Laser cutting of acrylic samples**

**Small cylinders: mass ~7g**

# Acrylic samples: delivery of July 2022

## Final surface cleaning procedure

Samples received: S0305, S0309, S0313, S0401, S0407, S0413

**Measured**: S0305 and S0401. → NAA on **October 26, 2022**

**3 cylinders** for each samples

| Sample | Mass [g] | $^{40}$K [$10^{-12}$ g/g] | $^{238}$U [$10^{-12}$ g/g] | $^{232}$Th [$10^{-12}$ g/g] |
|---|---|---|---|---|
| S0305 | 20.9 | 0.35±0.02 | <0.26 | **3.63±0.62** |
| S0401 | 21.2 | **2.83±0.07** | **3.98±0.42** | **14.2±1.4** |

**Noble gases inside acrylic**

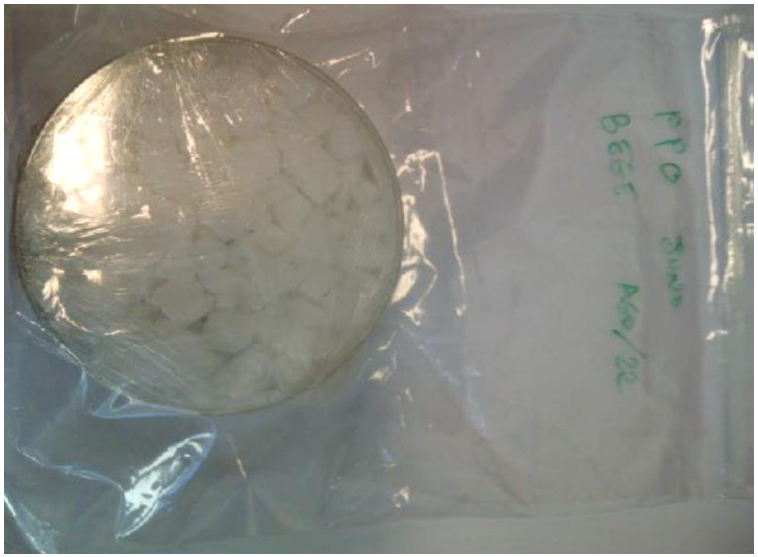
| Sample | Mass [g] | Argon [$10^{-6}$ g/g] | Kripton [$10^{-9}$ g/g] |
|---|---|---|---|
| S0305 | 20.9 | 1.74±0.39 | 1.93±0.44 |
| S0401 | 21.2 | 1.76±0.40 | 1.66±0.38 |

Gases are probably incorporated during panel production

# PPO:
# high purity final batch

# HPGe measurement

**Received on July 21, 2022**



- Mass 0.083 kg
- Diameter 90 mm, height 20 mm
- **Measuring time: 932 h**

| | **Activity [Bq/kg]** |
|---|---|
| **$^{232}$Th** | |
| $^{228}$Ac | <0,04 |
| $^{208}$Tl | <0,03 |
| **$^{238}$U** | |
| $^{226}$Ra | <0,18 |
| $^{214}$Bi | <0,04 |
| **$^{235}$U** | <0,01 |
| **$^{40}$K** | <0,3 |
| **$^{60}$Co** | <0,01 |
| **$^{137}$Cs** | <0,01 |

CL 90%

# PPO HP: NAA measurements

## Irradiation of November 29, 2022

After NAA irradiation

| Sample | Mass [g] | $^{40}$K [$10^{-12}$ g/g] | $^{238}$U [$10^{-12}$ g/g] | $^{232}$Th [$10^{-12}$ g/g] |
|---|---|---|---|---|
| **PPO HP** | 44.8 | 0.33±0.01 | <0.5 | **2.9±0.4** |

We detected also the presence of **Ar** and **Kr** gases in the PPO sample

| Sample | Mass [g] | Argon [$10^{-6}$ g/g] | Kripton [$10^{-9}$ g/g] |
|---|---|---|---|
| **PPO HP** | 44.8 | 2.19±0.46 | 0.40±0.07 |

## Irradiation of February 16, 2023

Sample - from the same batch of previous measurement - **washed with MilliQ water** before irradiation to remove eventual dust (no washing was performed on the sample irradiated in November 2022)

| Sample | Mass [g] | $^{40}$K [$10^{-12}$ g/g] | $^{238}$U [$10^{-12}$ g/g] | $^{232}$Th [$10^{-12}$ g/g] |
|---|---|---|---|---|
| **PPO HP** | 30.1 | 0.24 ± 0.01 | < 0.5 | **< 0.7** |

**Next measurements**: 1 bis-MSB + 2 PPO samples from China