

CHAPTER 1: Neutron Activation for the Majorana Collaboration

Section 1.1: Introduction

Neutron activated samples of polytetrafluoroethylene (PTFE) and fluorinated ethylene propylene (FEP) tubing were γ -assayed in the low background counting (LBC) lab at the Kimballton Underground Research Facility (KURF). Presented in this report are activities from detected isotopes, many of which are neutron activation products of transition metals.

Section 1.2: Sample Preparation

Thinly sliced PTFE samples and small sections of FEP tubing were activated at various times from September 30, 2015, to October 2, 2015, at the PULSTAR high flux reactor at North Carolina State University (<http://www.ne.ncsu.edu/nrp/pulstar.html>). Total time in the reactor over this period was 18 hours. Samples of varying masses as shown in Figure 1.1 were placed in plastic vials and packaged for shipping to Virginia Tech (VT). Ten vials containing 0.002-in thick PTFE samples were placed in a heat sealed plastic bag. This plastic bag was placed inside of a resealable plastic bag and labeled accordingly. Eight vials containing 0.005-in thick PTFE samples were placed in a heat sealed plastic bag along with the one vial containing FEP tubing. The samples arrived at VT on October 7, 2015. Environmental Health and Safety at VT received the samples and checked them for external contamination leakage. None was found and all samples demonstrated no more than 0.2 mrem/hr of activity externally. Samples were transported underground via the VT state-provided diesel truck to KURF on October 7, 2015.

Samples were shipped along with vials containing irradiated aqueous solutions with trace uranium and thorium concentrations which were used to calibrate the spectra from the

Sample Parameters		Irradiated	Flux	Flux	Corrected
<u>Index</u>	<u>Desc.</u>	<u>Mass (g)</u>	<u>Index</u>	<u>Factor</u>	<u>Std Mass (ug)</u>
1	Gasket 1 - Center	8.897	2Y	1.000	8897
2	Gasket 1 - Outside	11.476	2Y	1.000	11476
3	Gasket 2 - Outside	9.867	2Y	1.000	9867
4	Gasket 2 - Inside (with small piece outer)	13.053	2Y	1.000	13053
5	Gasket 3 - Outside	13.346	2X	0.930	12412
6	Gasket 3 - Inside	12.772	2X	0.930	11878
7	Gasket 4 - Outside	11.541	2X	0.930	10733
8	Gasket 4 - Inside	8.167	2X	0.930	7595
9	Gasket 5 - Outside	12.283	3Y	0.944	11595
10	Gasket 5 - Inside	8.664	3Y	0.944	8179
11	Gasket 6 - Outside	13.574	3Y	0.944	12814
12	Gasket 6 - Outside	12.724	3Y	0.944	12011
13	Gasket 6 - Inside	10.595	3X	0.879	9313
14	Gasket 6 - Inside	10.787	3X	0.879	9482
15	Gasket 7 - Outside	12.954	3X	0.879	11387
16	Gasket 7 - Outside	13.036	1Y	0.832	10846
17	Gasket 7 - Inside	9.828	1Y	0.832	8177
18	Gasket 7 - Inside	11.635	1Y	0.832	9680
20	FEP Shrink Tube	1.034	3X	0.879	909
Standard Parameters		Transfer	Flux	Flux	Corrected
<u>Index</u>	<u>Desc.</u>	<u>Loss</u>	<u>Index</u>	<u>Factor</u>	<u>Std Mass (ug)</u>
U-1	Uranium Standard (0.5 ug initial)	0.993	4X	0.678	0.496
U-2	Uranium Standard (0.5 ug initial)	0.956	4X	0.678	0.478
U-3	Uranium Standard (0.5 ug initial)	0.977	4X	0.678	0.489
U-4	Uranium Standard (0.5 ug initial)	0.985	4X	0.678	0.493
TH-2	Thorium Standard (0.5 ug initial)	0.765	4Y	0.710	0.383
TH-3	Thorium Standard (0.5 ug initial)	0.826	4Y	0.710	0.413
TH-4	Thorium Standard (0.5 ug initial)	0.745	4Y	0.710	0.372
TH-5	Thorium Standard (0.5 ug initial)	0.693	1X	0.780	0.346

Figure 1.1: Table of masses for neutron activated samples and standards. Information provided by Matthew Green at North Carolina State University.

samples and to find the ratio between absolute U/Th levels in the samples and measured γ -ray activities. The standards were packaged in sealed plastic bags as well as shown in Figure 1.2.

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Figure 1.2: Neutron activation sample packaging

Upon arrival at the lab, a sample preparation station was arranged. A table was placed outside of the detector lab with a plastic sheet on top. Everything inside of the shipping container was treated as contaminated for safety purposes. At all times, nitrile gloves were worn when handling anything that was inside the original shipping container or came in contact with something inside the shipping container. Two Marinelli beakers were designated

for use in the detectors with the samples.

Section 1.3: Measurement

Measurements were made using the two detectors at the UNC low-background counting facility at KURF. The first detector, “MELISSA,” is a 1.1kg, 50% RE (relative efficiency compared to NaI) Canberra LB (low-background) detector. MELISSA is oriented vertically and is cooled using a dipstick cryostat. MELISSA’s shield consists of 15 cm of Doe-run lead and 2.54 cm OFHC (oxygen-free high conductivity) copper. At the time of sampling, however, the top 30% of the shield was unstacked due to previous detector maintenance. The sample cavity is 38 cm \times 38 cm \times 38 cm. The FWHM at 1.33 MeV is 1.70 keV, and the threshold is 20 keV. The other detector, “VT-1,” is a 0.956 kg, 35% RE ORTEC LLB Series detector in a J-type configuration. VT-1’s shield consists of a 10.1-cm ORTEC commercial lead shield and 0.3 cm of OFHC copper. The sample cavity is cylindrical with dimensions 41 cm (height) \times 28 cm (diameter). VT-1 has a FWHM of 1.80 keV at 1.33 MeV and the threshold is 20 keV. For more technical details regarding the facility and detector setup, see [?].

Each standard vial was placed in one of the detectors for 20 minutes at a time. For the first 20 minutes, the ^{238}U standard was placed in MELISSA and the ^{232}Th standard was placed in VT-1. For the second 20 minutes, the samples were each moved to the other detector. The spectrum for the ^{238}U standard in MELISSA is shown in Figure 1.3 along with a magnification of the ROI in Figure 1.4 Figures 1.5 and 1.6 show the same spectra for the ^{232}Th standard. After the standards were measured, the 0.002-in PTFE samples were placed in MELISSA and the FEP tubing samples were placed in VT-1 for assay. Emphasis was placed on these two samples at the request of the MAJORANA project engineer.

Once the standards were measured and the spectra were reviewed, the samples were placed in the detectors. The ten vials containing 0.002-in thick slices of PTFE were placed in a Marinelli beaker inside MELISSA. The samples were oriented as shown in Figure 1.7.

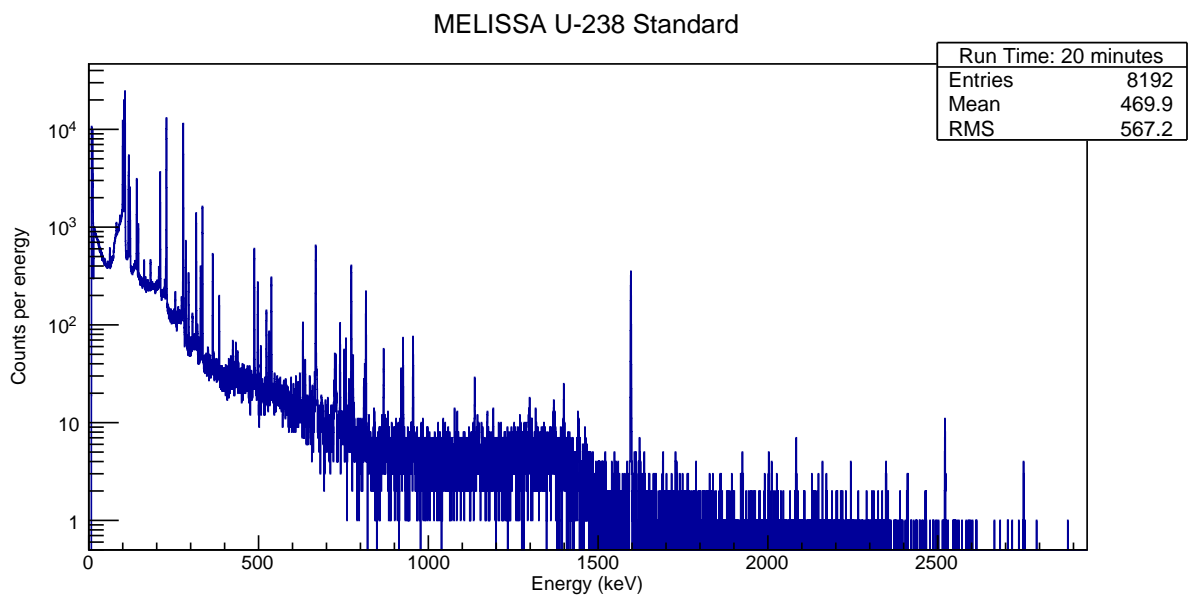


Figure 1.3: ^{238}U standard spectrum in MELISSA

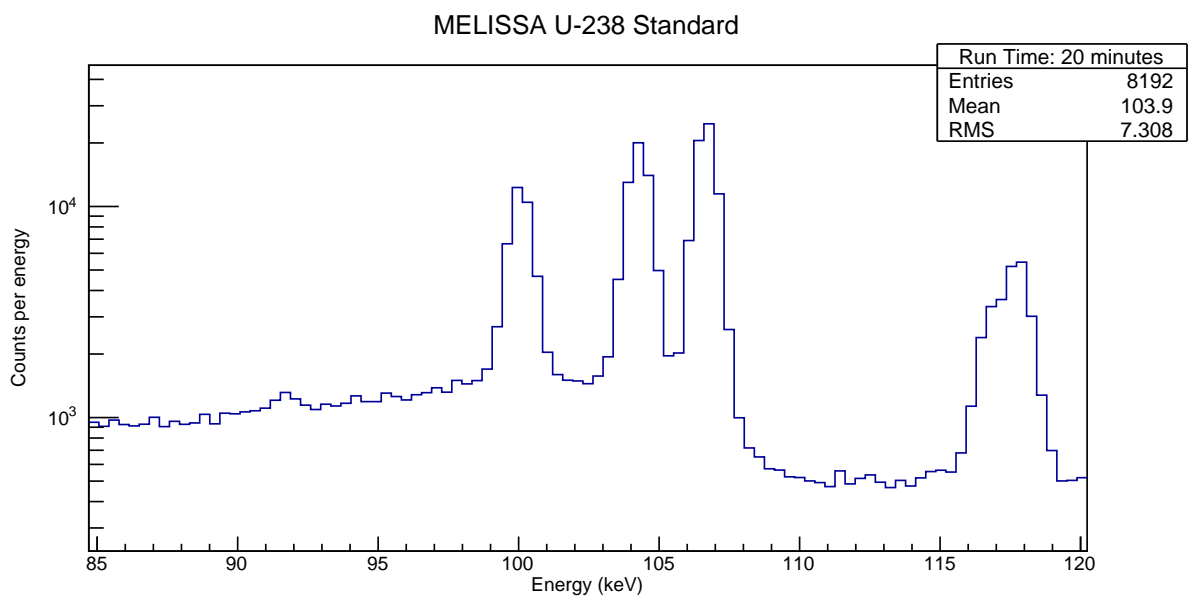


Figure 1.4: ^{238}U standard spectrum in MELISSA showing peak at 106 keV

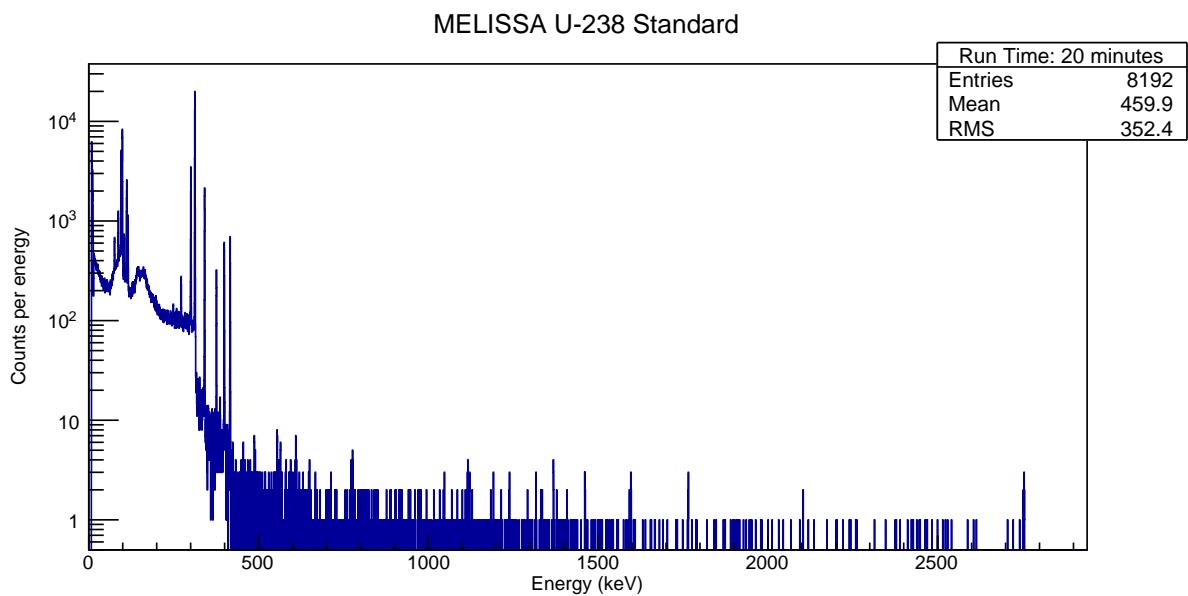


Figure 1.5: ^{238}Th standard spectrum in MELISSA

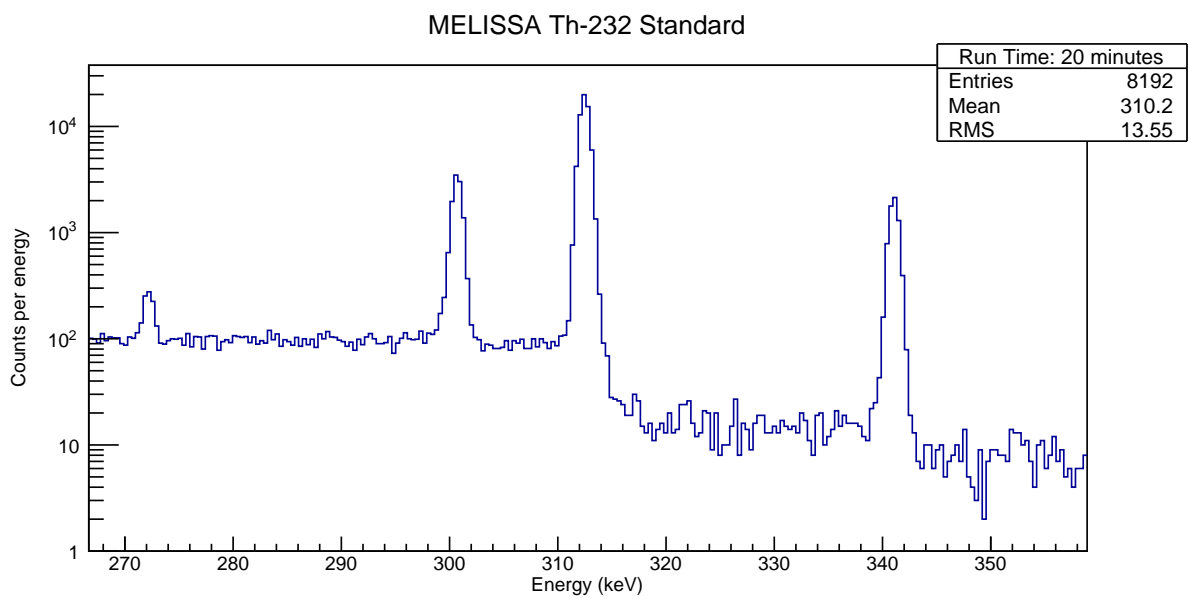


Figure 1.6: ^{238}Th standard spectrum in MELISSA showing peak at 312 keV

The vial containing the FEP tubing was placed in a Marinelli beaker inside of VT-1. Both detectors were sealed and the data collection was allowed to run for 30 hours. All analysis was based on this 30-hour period.

Section 1.4: Analysis

The number of counts in ROI for the standards are found by subtracting the background from the peak in the ROI. The net peak area is found by subtracting the peak area of a background peak from the corresponding standard peak. The background in this case was found by fitting a linear function to a region extending $\pm 3\sigma_{res}$ from the centroid of the peak in the standard.

The activities of a specific peaks in the standards are then given by:

$$A_{\gamma s} = \frac{N}{\epsilon_{\gamma} m t} \quad (1.1)$$

where N is the net peak area at the energy observed, ϵ_{γ} is the estimated peak efficiency, t is the assay live time, and m is the mass of the standard. This activity was then used to find the initial activity for the isotopes of interest with:

$$A_{os} = A_{\gamma s} e^{\lambda \bar{t}} \quad (1.2)$$

where A_{os} is the initial activation activity, $A_{\gamma s}$ is the net peak area of the irradiated standards at known energies, \bar{t} is the average time since activation, and λ is the decay constant for that isotope.

The same energies were then observed from samples' spectra. The ROI was defined as the same $\pm 3\sigma_{res}$ regions used to analyze the standards. If no statistically significant peak is

present, an upper limit can be placed on the activity in a sample. The signal in an energy bin is then required to be less than or equal to the upper limit, 90% C.L. - 1.65σ (σ here is just the square root of the recorded counts). We can convert this limit on the counts to a limit on the mass fraction of the isotope using the measured activities in the activated standards. This is given by:

$$MassFractionLimit = 1.65 \frac{\sqrt{A_p}}{tm\epsilon_\gamma} \frac{1}{A_{os}} e^{\lambda \bar{t}} \quad (1.3)$$

where A_p is the activity in the ROI for the sample, t is the assay live time of the sample, m is the mass of the sample, ϵ_γ is the estimated efficiency, A_{os} is the initial activity of the standard, \bar{t} is the average time since activation, and λ is the decay constant. This mass fraction is the activity limit of the calculated original activity of the sample in part per trillion (ppt).

Section 1.5: Results

A list of γ -active isotopes found in the spectra is given for both the 0.002-in thick PTFE and the FEP tubing. All isotopes found were products of neutron activation. The isotopes of interest for this assay were products of the neutron activation of ^{238}U and ^{232}Th impurities in the samples, specifically the peaks at 106 keV for ^{239}Np and 311 keV for ^{233}Pa respectively. No statistically significant peaks were found at either energy. Calculated initial activity limits of ^{238}U and ^{232}Th in the 0.002-in PTFE samples were 5.2 ppt and 3.5 ppt respectively. The same limits in the FEP tubing sample were 9.4 ppt and 6.3 ppt respectively. A list of additional isotopes identified in the samples is shown in Table 1.1.

Isotope	Half-life	Lines observed (keV)
⁵¹ C	28 days	320
⁸² Br	35 hours	554, 620, 698, 764, 776, 827, 1043, 1316, 1474
¹¹⁰ Ag	250 days	657, 707, 884, 937, 1383
⁶⁰ Co	1925 days	1173, 1333
⁵⁴ Mn	312 days	835
⁵⁸ Co	71 days	810
⁵⁹ Fe	44 days	1098, 1291
⁶⁵ Zn	244 days	1115
²² Na	3 years	1273
²⁴ Na	15 hours	1368, 2751
¹²⁴ Sb	60 days	603, 1690
⁴⁰ K	1.25×10 ⁹ years	1460
¹²³ Sn	129 days	159

Table 1.1: List of additional isotopes detected in the samples