Participation in the IAEA Worldwide Open Proficiency Tests for Nuclear and Related Analytical Techniques Laboratories

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University of Maryland Radiation Facilities

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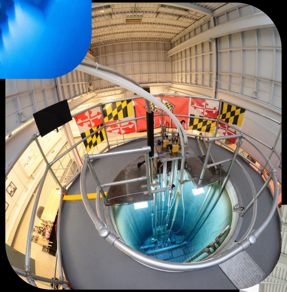
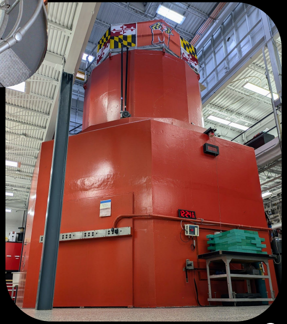
FEARLESSLY FORWARD

Maryland University Training Reactor

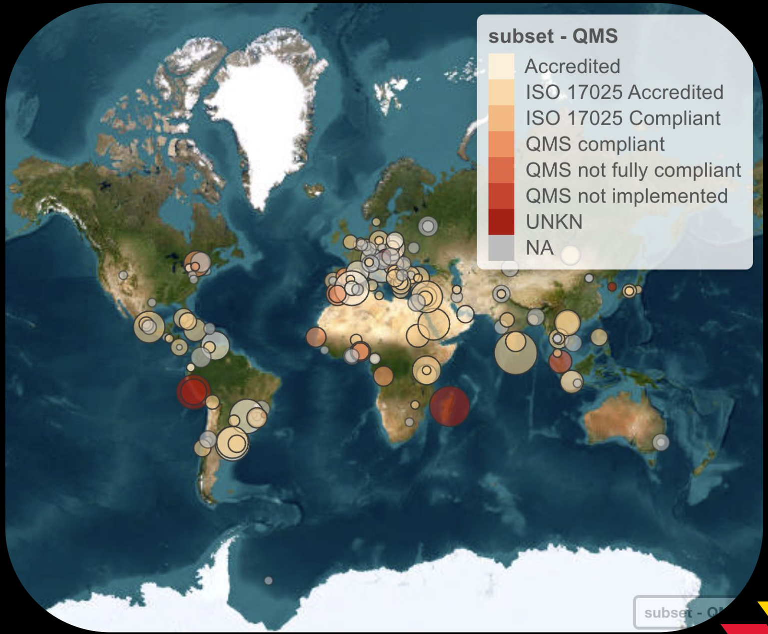
- 250 kW TRIGA Conversion Reactor
    - Built in 1960, converted to TRIGA in 1974
- Activities Include:
    - Neutron Activation Analysis
    - University Lab Classes and Training
    - Outreach Activities
    - Neutron Detector Testing
    - Neutron Imaging
    - Isotope Production

IAEA Worldwide Open Proficiency Tests for Nuclear and Related Analytical Techniques Laboratories (PTNATIAEA)

- Annual interlaboratory comparison test for elemental analysis
  - Since 2002
  - Free to participate in
  - 3 months to complete analysis

- 2023 had 98 participants in 57 countries
  - NAA
  - XRF
  - PIXE/PIGE
  - AAS
  - ICP-MS/OES



http://www.pt-nsil.com/

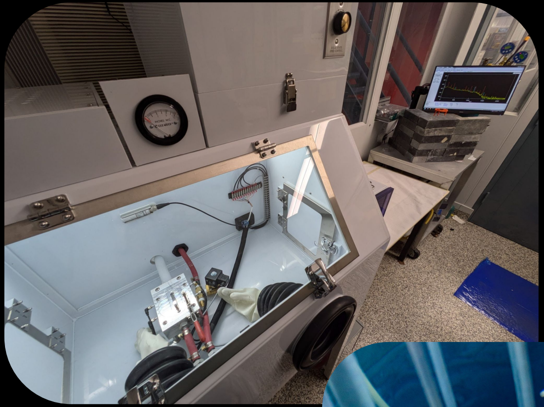
PTNATIAEA/21 Samples

- Soil sample with elevated mass fractions of elements
  - Siliceous Sample
- Plant material sample
  - Sterilized plant-derived cellulose powder
- Samples were provided dried, powdered, and homogenized

MUTR Neutron Activation Analysis Facilities

- NAA is performed using the MUTR Rabbit System
  - PLC Control System
  - CO_2 driven, positive pressure
  - Rabbits 2.15” long, 0.75” in diameter
  - In-core terminus surrounded by graphite
  - Thermal neutron flux of about $2*10^{12}$ $n/cm^2/s$

- 3 High Purity Germanium Detectors
  - 1 located adjacent to Rabbit receiving station for counting of short lived samples
  - 2 in another lab
    - 1 LabSOCS characterized detector



Sample Preparation

- 15 replicates of each sample were prepared
  - Weighed into heat sealed polyethylene vials
  - Around 40 mg each for plant, 100 mg for soil
    - No special moisture control precautions
- 4 replicates of standards prepared with by same method as samples
  - NIST SRM 2710a: Montana I Soil
  - NIST SRM 1537a: Tomato Leaves
- 5 mm diameter, 0.1 mm thick iron foils, were cut to act as flux monitors

Sample Preparation

- Samples and standards for long irradiations were stacked with flux monitors between each vial
  - Held in place with aluminum foil
  - All 12 sample/standard vials were placed into 1 rabbit
    - 1 rabbit for plant, 1 for soil

- Samples and standard for short irradiations were each activated separately
  - No flux monitors used

Sample Irradiations

- Soil samples irradiated either 1 minute or 1 hour with the reactor at 100 kW
- Plant samples irradiated either 5 minutes or 2 hours with the reactor at 100 kW
- Control Rod positions maintained as consistent as possible
  - Power variations around 1-2%
  - Pool temperature increased 4 ℃/hr

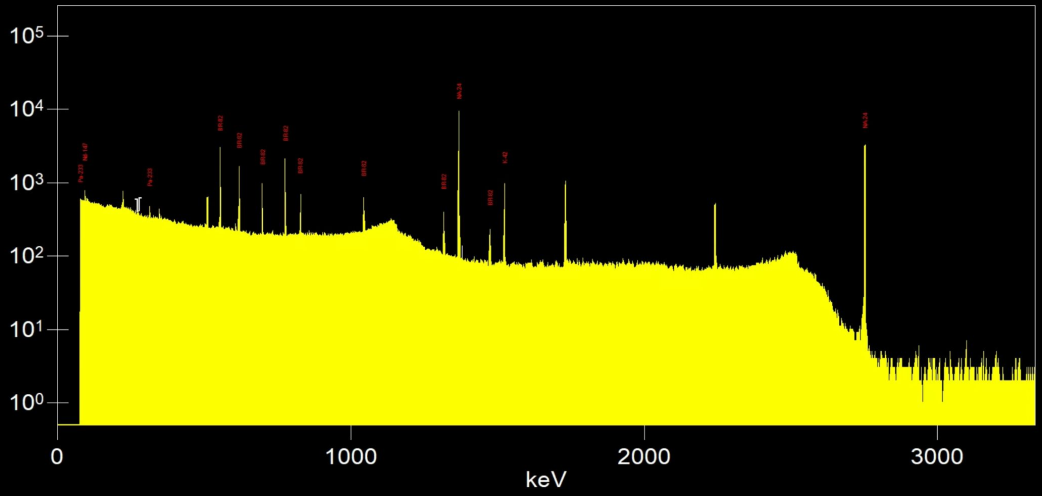
Sample Counting

- Samples and standards were allowed to decay for 3-4 minutes following short irradiations
    - 5 minute counts
    - Dead times around 2% for plant samples, 35% for soil samples

- Samples and standards were allowed to decay for 3-5 days following long irradiations
    - 1 hour counts
    - Dead times around 1-2%
    - Flux monitors counted for 1 hour

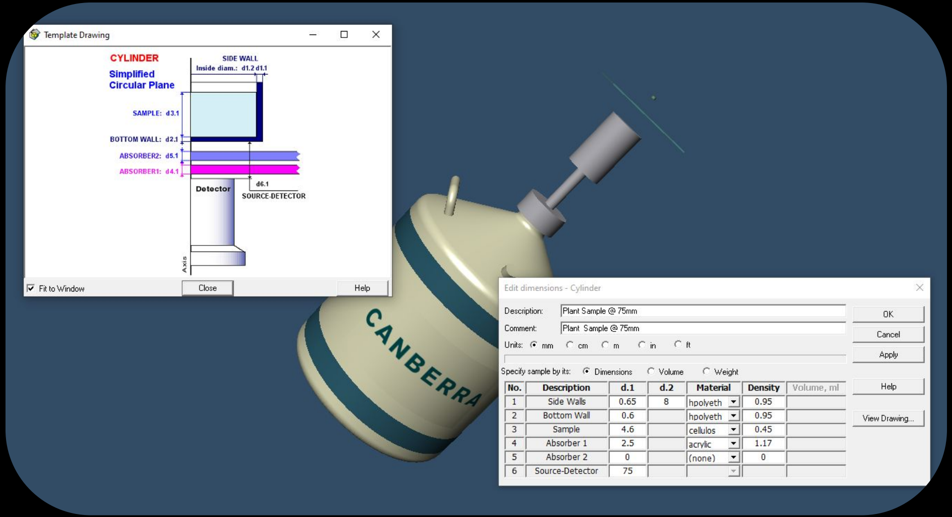
Data Analysis

- Elemental analysis was performed primarily by the comparator method
  - Peak areas were determined using Genie 3.4
  - Peaks were manually assigned to isotopes
  - Count rates were decay corrected to the end of the activation
  - Relative count rates were compared to standards to determine the amount of element present
  - Flux correction was applied based on relative count rates of sample and standard flux monitors



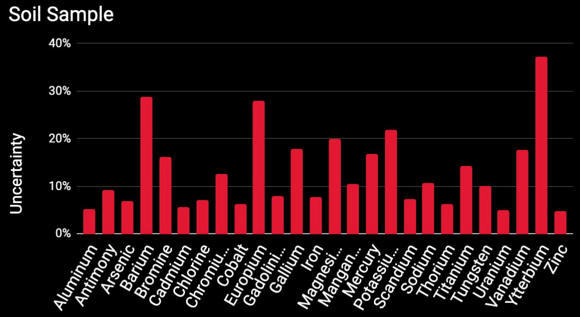
Data Analysis

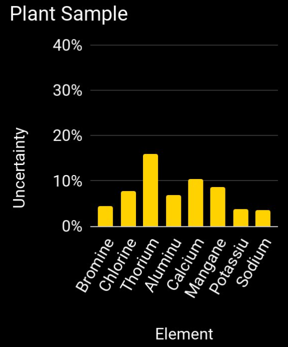
- In cases where element was present in the sample but not the standard the absolute method was used
  - Activity of the isotope was determined using LabSOCS for efficiency calibrations
  - Local flux was calculated from the adjacent flux monitors
  - Flux and activity used to calculate amount of isotope present



Uncertainty

- Factors considered in uncertainty
  - Standard concentrations
  - Mass
  - Flux
  - Peak Area
- Average combined uncertainties around 10%
  - Primarily due to peak area uncertainties
    - More targeted analysis usually around 3-5% uncertainty
- Did not consider
  - Irradiation Time
  - Decay Time
  - Self shielding
  - Cross sections





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Results

- Identified 26 elements in soil sample and 8 elements in plant sample
  - Overall results: 43 elements reported for soil, 24 for plant sample
  - Participant Code 274

- 17 elements in soil sample and 6 elements in plant sample were measured statistically correct

- Results using relative NAA method were good, results using absolute NAA were poor
  - 5/12 for soil sample
  - 0/4 for plant sample

Element	IAEA Value (PPM)	IAEA Uncertainty (PPM)	Average Concentration (PPM)	Averge Uncertainty (PPM)
Aluminum	86000	20000	78030	4000
Antimony	104	15	110	10
Arsenic	277	50	280	19
Barium	860	130	870	290
~~Barium~~	~~860~~	~~130~~	~~796~~	~~229~~
Bromine	2.7	1.9	1.1	0.2
Cadmium	328	60	223	13
Chlorine	14400	7000	1,939	138
Chromium	257	80	788	99
Cobalt	290	40	220	14
Europium	1.04	0.18	0.24	0.07
Gadolinium	3.7	1.4	0.51	0.04
Gallium	18	9	6	1
Iron	14900	4000	13000	1000
Magnesium	4400	2000	10000	2000
Manganese	730	300	960	100
Mercury	12.8	5	5.4	0.9
Potassium	6000	1800	6420	1400
Scandium	10.7	1.1	11	1
Sodium	2230	500	940	100
Thorium	11.3	2	16	1
Titanium	5900	2000	4900	700
Tungsten	4.6	1	2.0	0.2
Uranium	3.54	0.8	4.0	0.2
Vanadium	185	50	170	30
Ytterbium	1.92	0.5	0.4	0.2
Zinc	620	140	850	40

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Deficiencies Identified

- Poorly characterized epithermal flux
  - Likely the main source of error in the absolute NAA measurements
- High dead times
  - Lost counts on short lived isotopes lead to underestimating their activity
- Limited neutron flux
  - Uncertainties driven primarily by statistical uncertainty of peaks
- Limited time
  - Only 2 irradiations were performed
  - Samples were only counted a single time
    - No sample changer
- Lack of cooling
  - Pool temperatures increased throughout irradiations varying flux
- Small sample sizes
  - Below the recommended amounts of samples and standards were used

Improvements Considered

- Implementing k_0 NAA technique
  - Reduce need to select appropriate standards
- Better characterizing neutron flux in the Rabbit
  - Planned to be completed once additional fuel has been added to the core
    - Additional fuel should also increase flux by around a factor of 2.5
- Implementing loss free counting
  - Reduce the effects of dead time discrepancies
- Continued participation in PTNATIAEA
  - IAEA-TECDOC-2055 - New guidance for NAA with short lived nuclides

Questions?