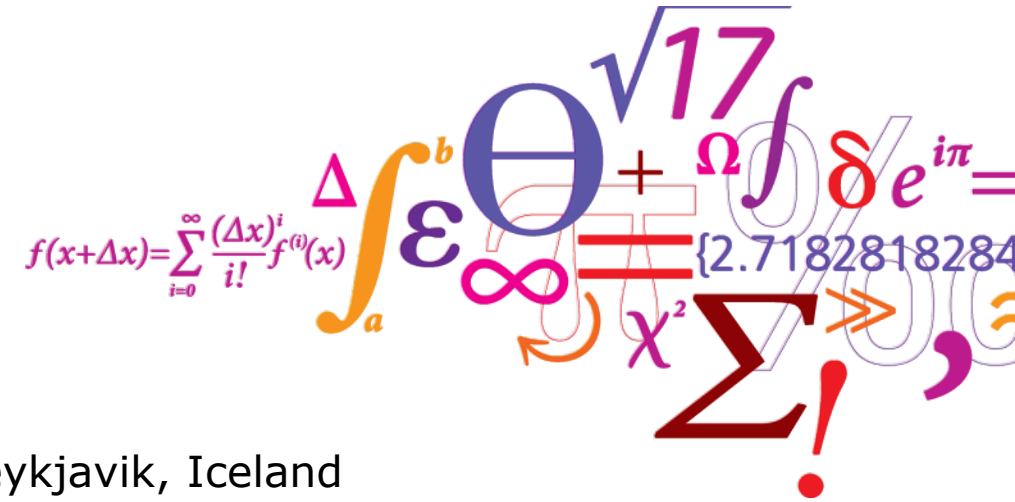
Recent check of calibration across detectors

Sven Nielsen



NKS GammaSpec 2018 Seminar, Reykjavik, Iceland

DTU Nutech
Center for Nuclear Technologies

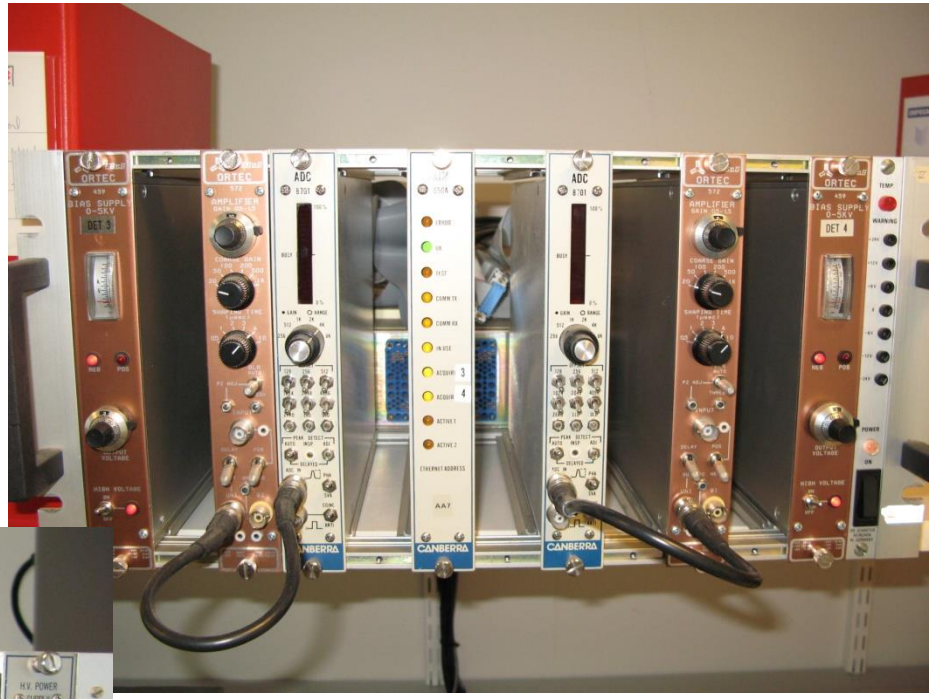
Calibration 2016

- 11 Ge detectors
- 7 standard sample containers, 3-5 fillings each
- About 250 calibration spectra
- AREVA calibration solution containing ^{241}Am, ^{109}Cd, ^{139}Ce, ^{57}Co, ^{60}Co, ^{51}Cr, ^{137}Cs, ^{54}Mn, ^{113}Sn, ^{85}Sr, ^{88}Y, ^{65}Zn (range 60 – 1836 keV)
- Using Genie2000 and EFFTRAN to generate certificate files and nuclide libraries free from coincidence summing effects
- Checking quality of calibration by analysing calibration solutions as samples and comparing results with reference values

Detector specifications

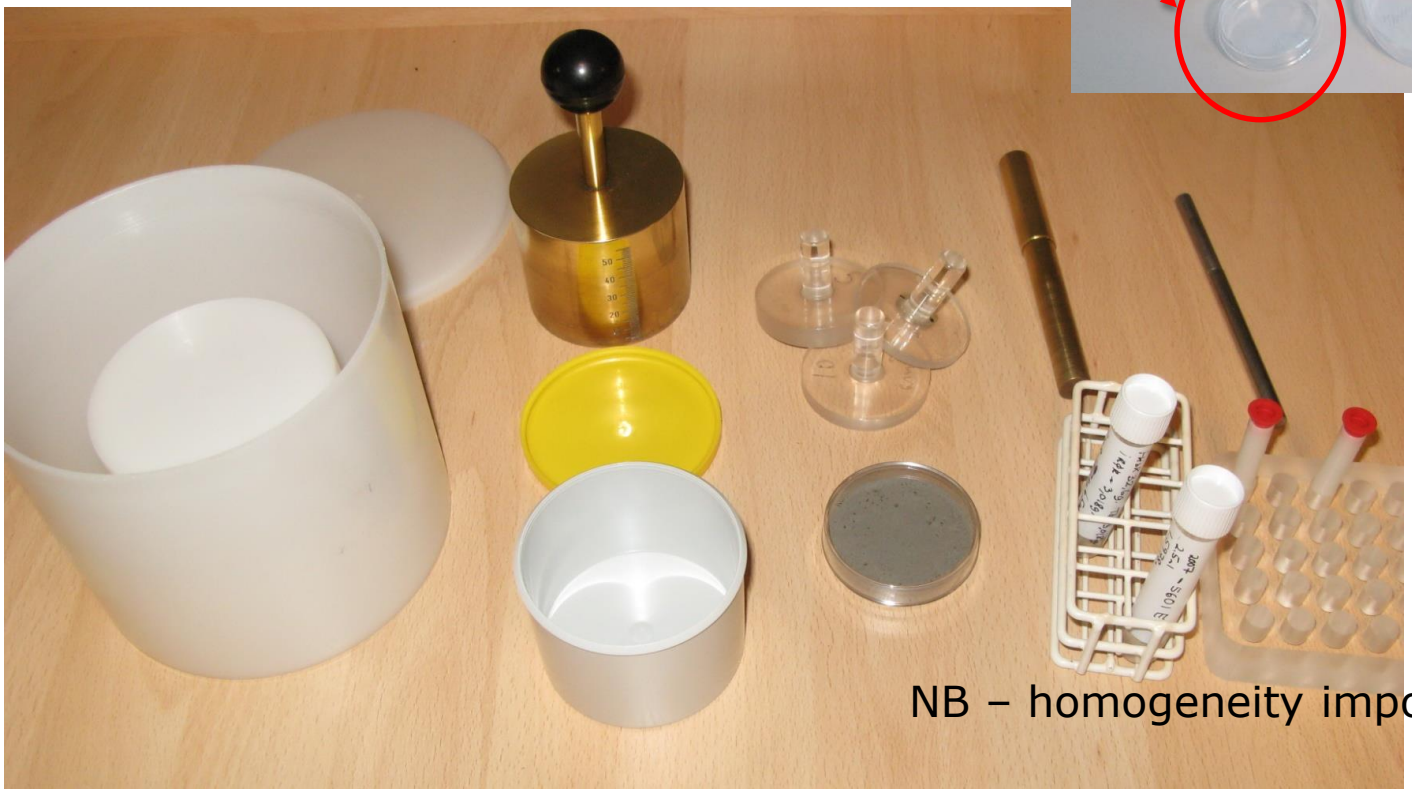
DTU ID	Producer	Year	Efficiency	Other
004	Ortec	1986	33%	n-type, 0.5 mm Be window
006	Canberra	1998	BE3830	n-type, 0.5 mm carbon epoxy window, low background
008	Canberra	2015	BE5030	n-type, 0.6 mm carbon epoxy window, low background, characterised
009	Canberra	2015	BE5030	n-type, 0.6 mm carbon epoxy window, low background, characterised
010	Canberra	2015	BE5030	n-type, 0.6 mm carbon epoxy window, low background, characterised
083	Canberra	1996	60%	p-type
414	Canberra	1979	25%	p-type, Ge(Li)
952	Ortec	1995	37%	n-type, 0.5 mm Be window, low background

Electronics



Sample Geometries

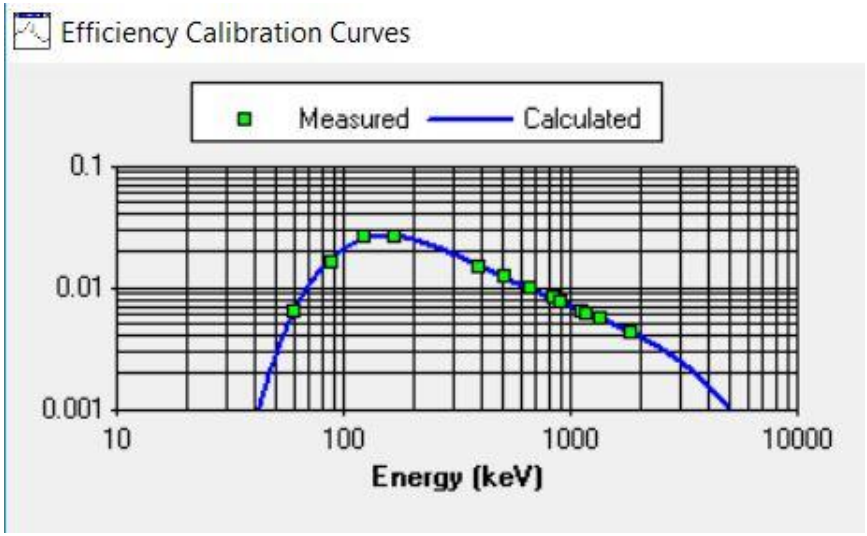
- 1-L Marinelli beaker (0.6-1 L)
- 210 mL cylindrical beaker (range 20-200 mL)
- 400 mL cylindrical beaker (range 20-390 mL), WH400
- 25 mL Petri dish (5, 10 and 15 mL), PET25
- 70 mL Petri dish (20-65 mL)
- 10 mL vial (range 1-8 mL)
- 2 mL vial (range 0.2-2 mL)



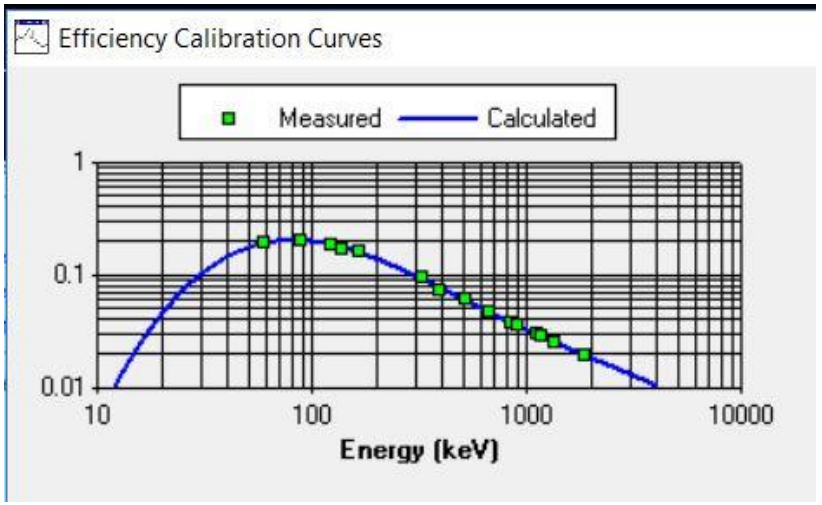
NB – homogeneity important

Detector Efficiency, Examples

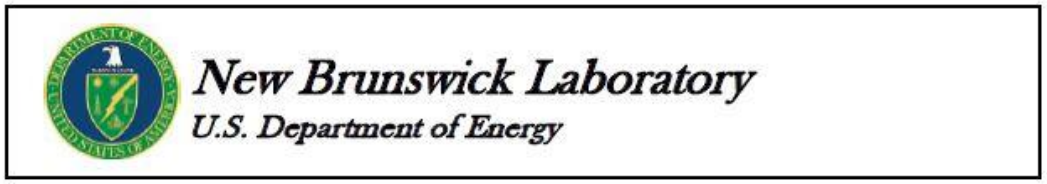
- Detector 414, p-type
- Petri dish 25 mL geometry



- Detector 008, n-type
- Petri dish 25 mL geometry



Sample - Certified Reference Material Uranium Standard, 0.05% U



New Brunswick Laboratory
U.S. Department of Energy

Certificate of Analysis
CRM 103-A
Pitchblende Ore – Silica Mixture
Uranium Standard

Uranium:	$0.0499_2 \pm 0.0007_8$ Wt.% ($\alpha = 0.05$, df = 24)
*Radium (calculated):	1.72×10^{-8} Wt.%

This Certified Reference Material (CRM) was prepared by milling and blending NBL CRM 6-A Pitchblende Ore (67.91 ± 0.05 Wt.% U_3O_8) with silica (99.9% SiO_2) to obtain a uniform mixture of desired uranium concentration. Characterization and certification analyses for uranium content were performed on ten (10) units selected from the packaged final product.

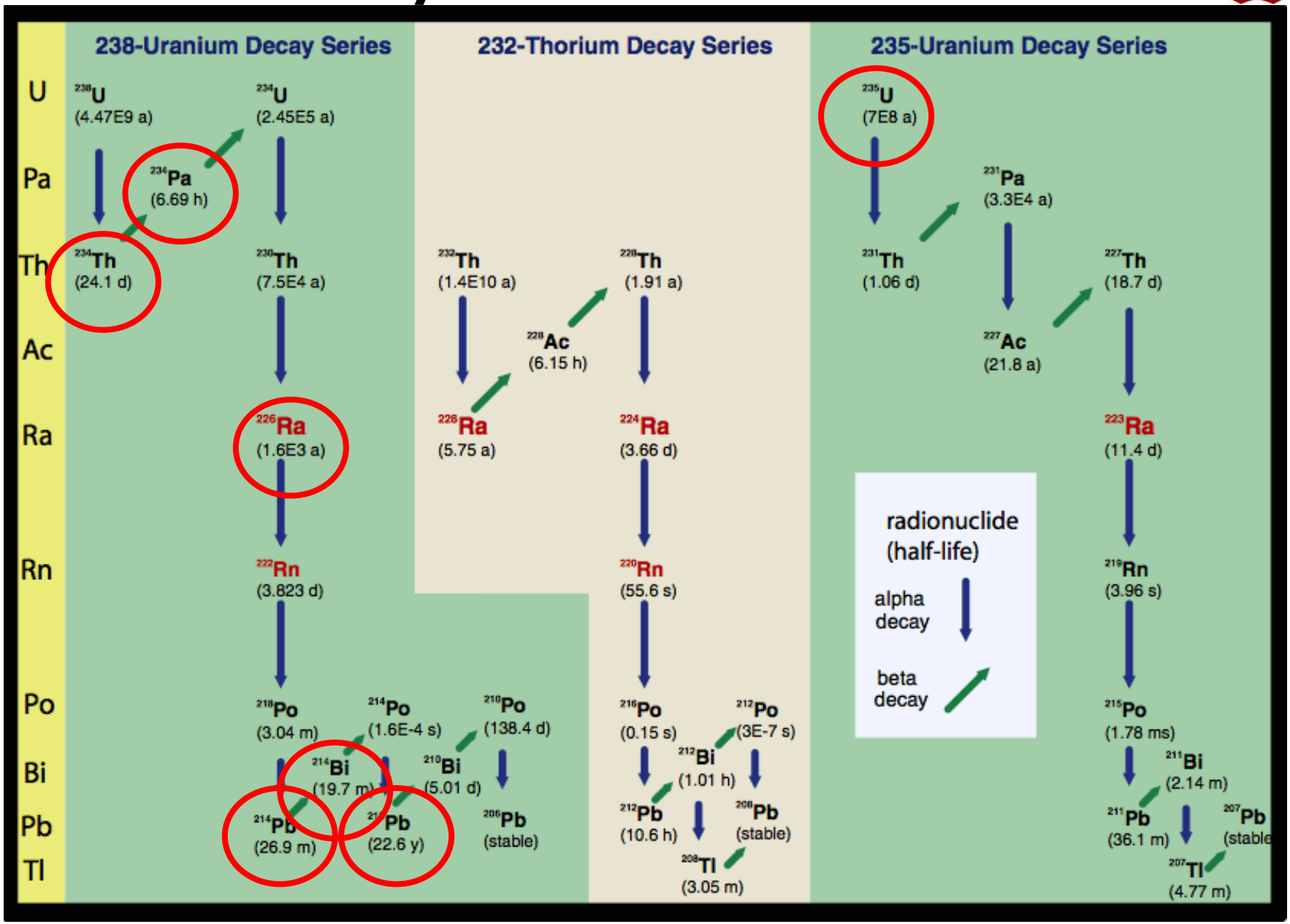
The certified value listed above is expressed in terms of 95% confidence limits, defined as as $\bar{x} \pm \sigma \cdot t$, where $\bar{x}$ is the pooled mean of the measurement data, σ is the pooled standard deviation of the mean, and t is the Student's t value for the indicated degrees of freedom (df) and at the 5% significance level (α).

REFERENCE METHODS OF ANALYSIS: Spectrophotometry verified with NBL Uranium Oxide (U_3O_8) and Fluorimetry verified with NBL CRM 112-A Uranium Metal.

*Calculation is based on the radium/uranium ratio of 3.44×10^{-7} g Ra/g U for NBL CRM 6-A Pitchblende Ore.

- Reference material analysed in two geometries
  - PET25, petri dish, 25 mL volume, 36 g sample mass
  - WH400, cylindrical beaker, 400 mL total volume, 185 g sample mass
- Plastic containers used without sealing against escape of radon and no time allowed for build up of equilibrium
- Samples prepared and gamma analysed in August 2018

U and Th decay series

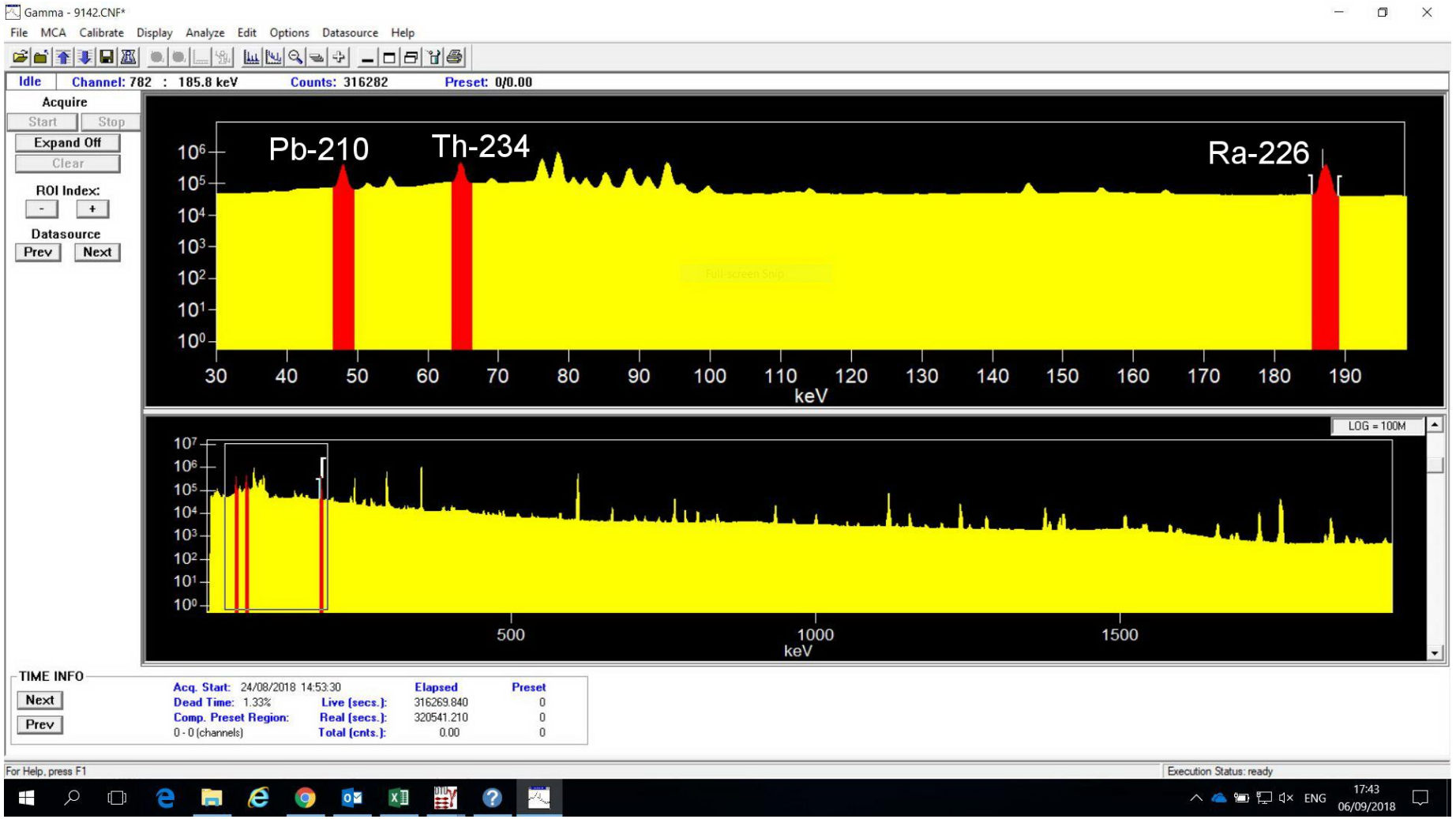


Spectrum analysis

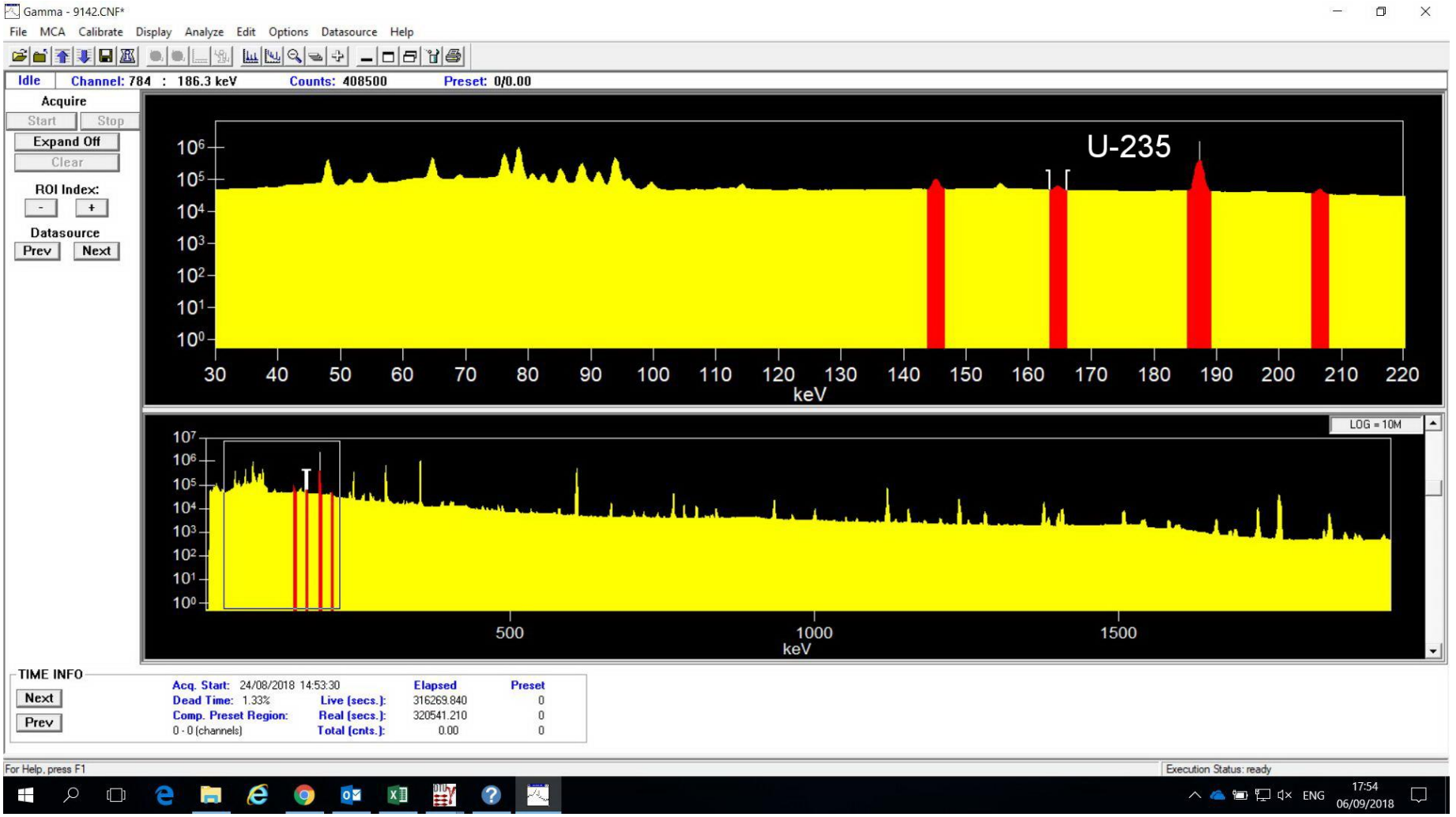
- Analyses of sample spectra based on calibration of detectors in 2016 and use of Genie2000 and EFFTRAN
- EFFTRAN used for 1) transfer of detector efficiency from liquid calibration solution and geometry to sample matrix and geometry and 2) corrections for true coincidence losses
- Genie2000 feature for interference correction used to obtain activity concentrations for Ra-226 and U-235 from 186-keV peak
- Analyses carried out with GenTran (Nikola Markovic), software for control and coordination of user input with Genie2000 and EFFTRAN calculation procedures
- Analytical uncertainties include components from counting statistics and calibration combined with a random component of 5%

- Practical point
  - Non-linear channel/energy relations (<100 keV) were found for detectors connected to a Multiport MCA requiring special procedure for identification and quantification of Pb-210

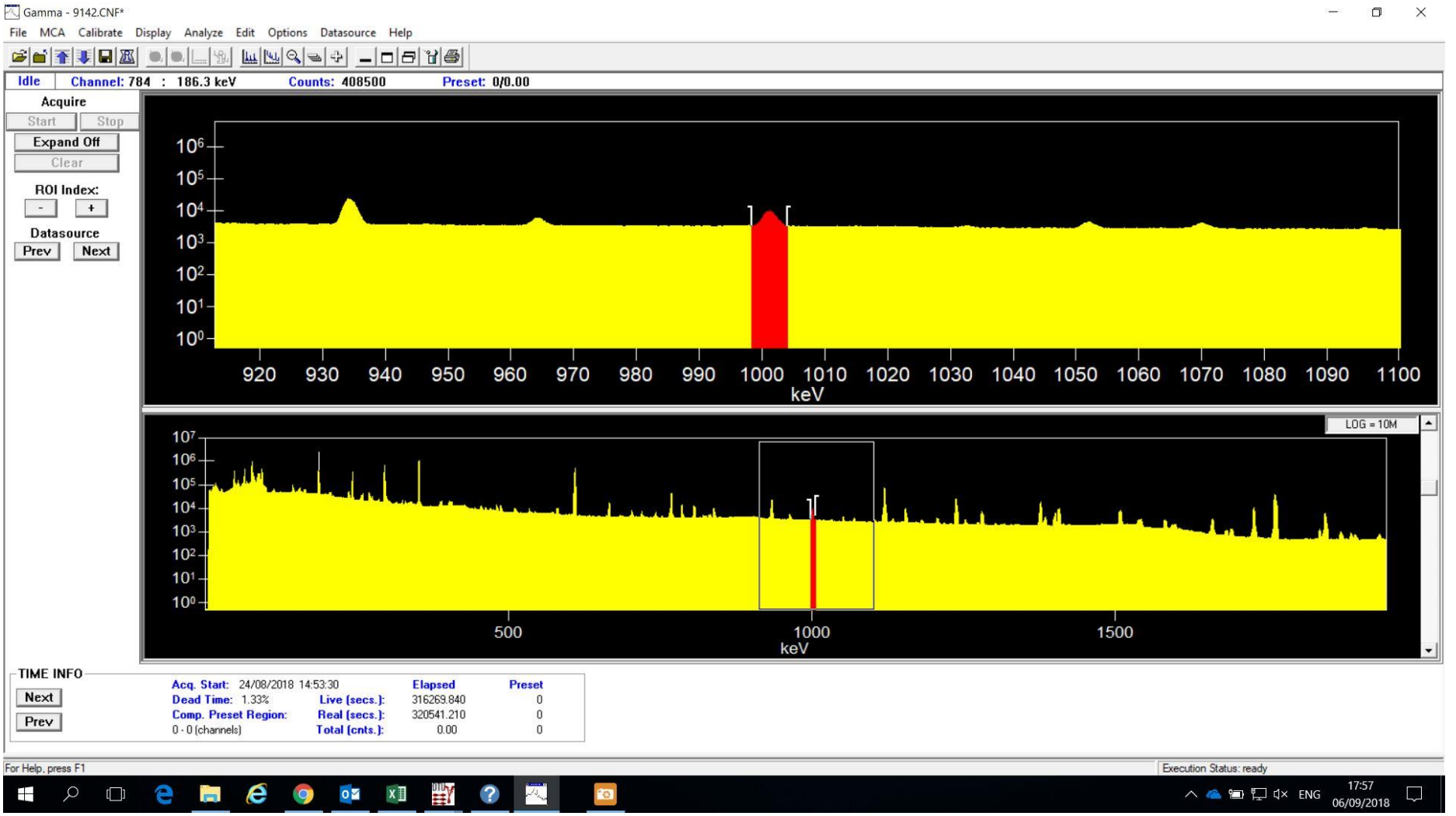
Identification of Pb-210, Th-234 and Ra-226



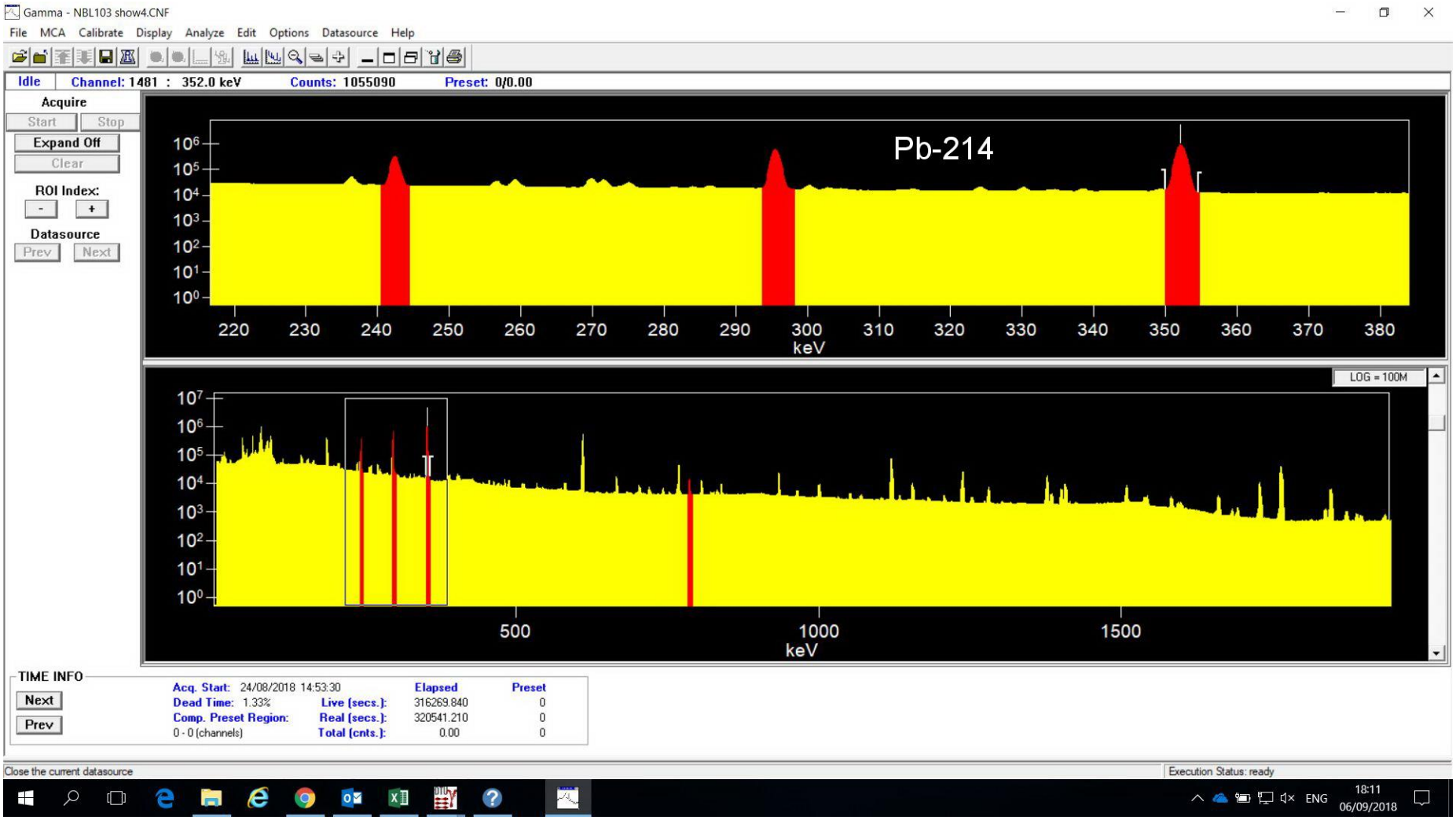
Identification of U-235



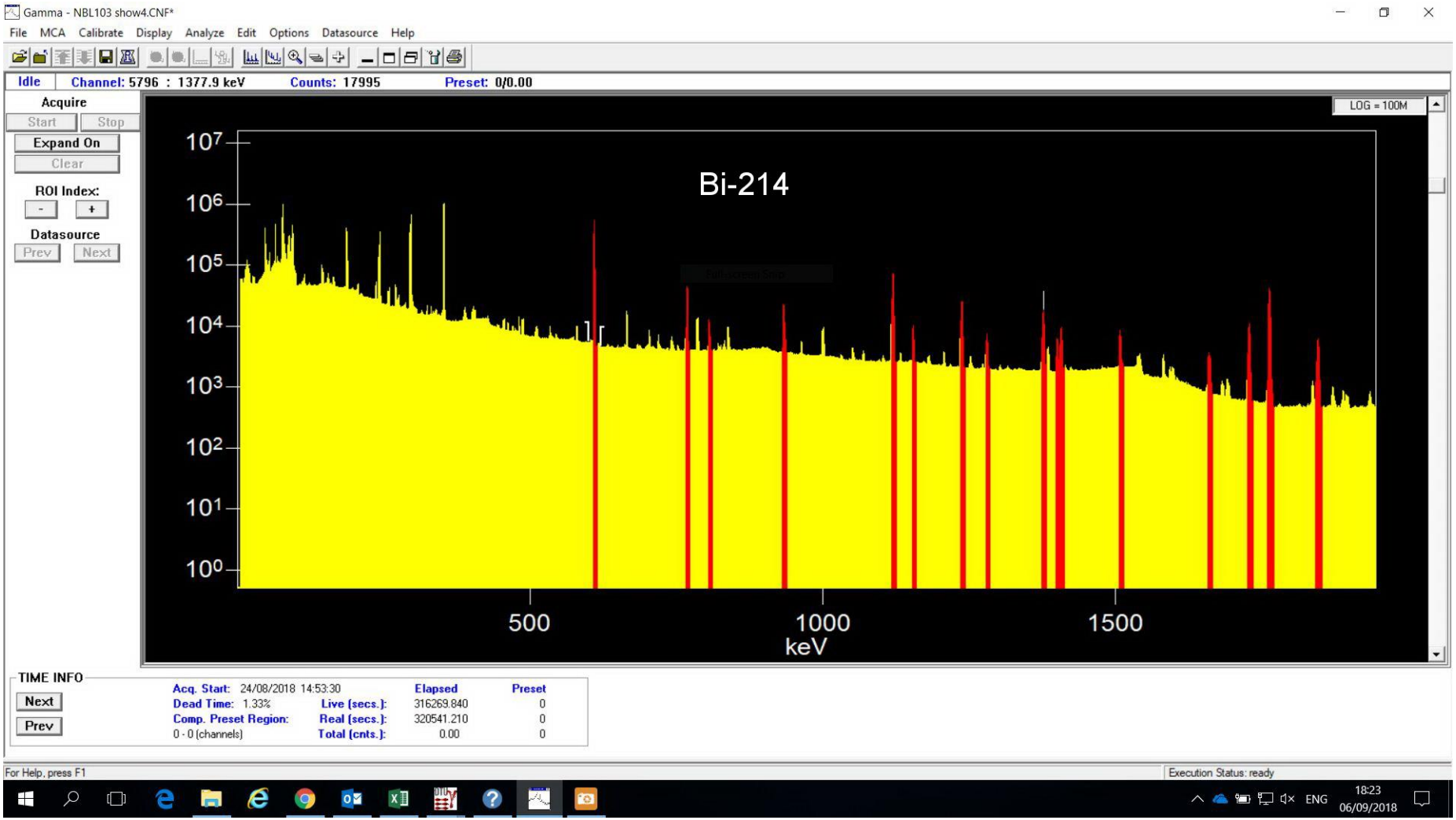
Identification of Pa-234m



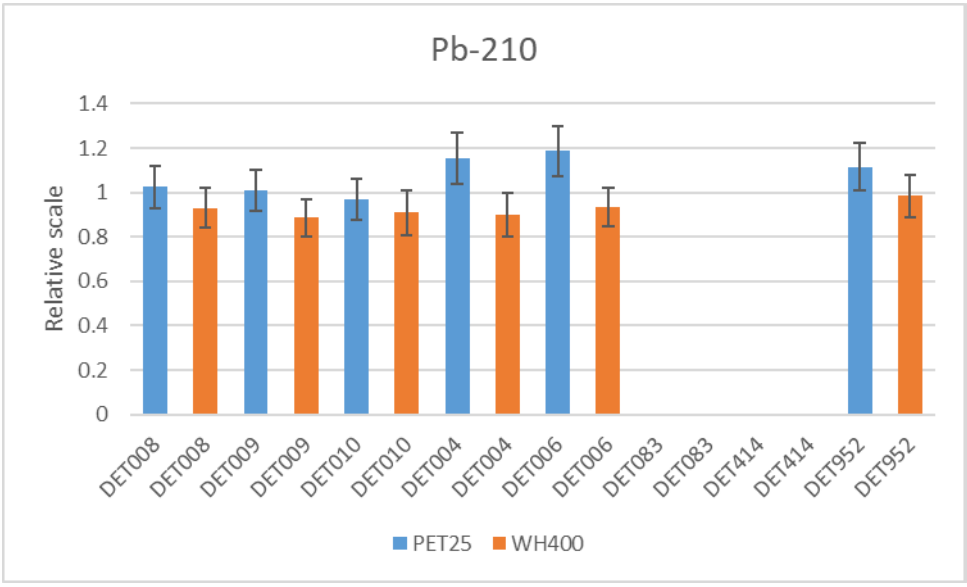
Identification of Pb-214

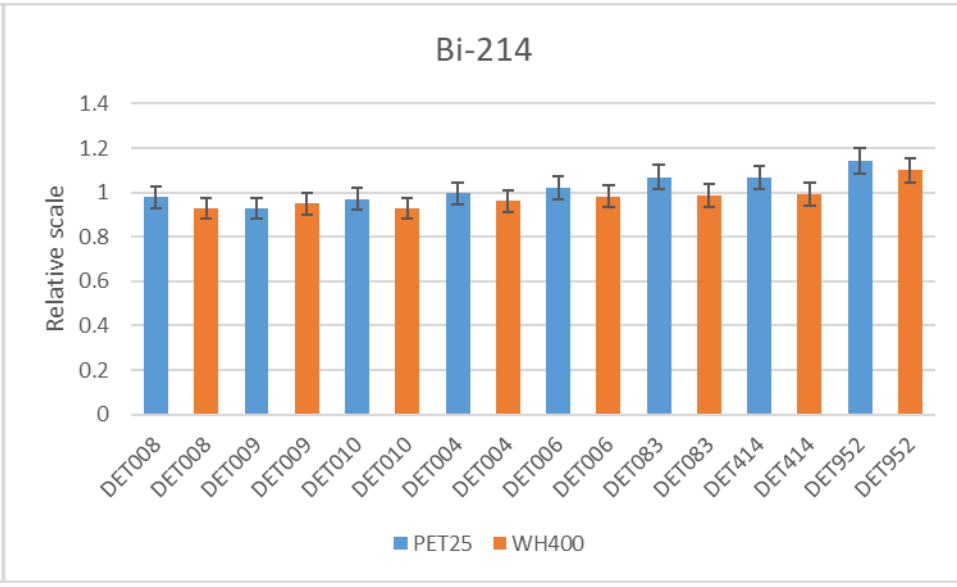


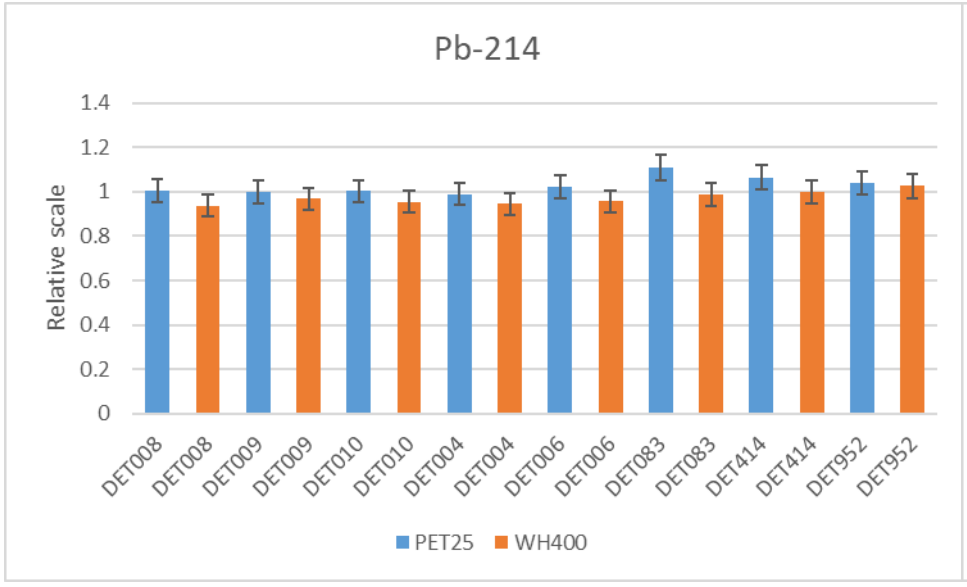
Identification of Bi-214

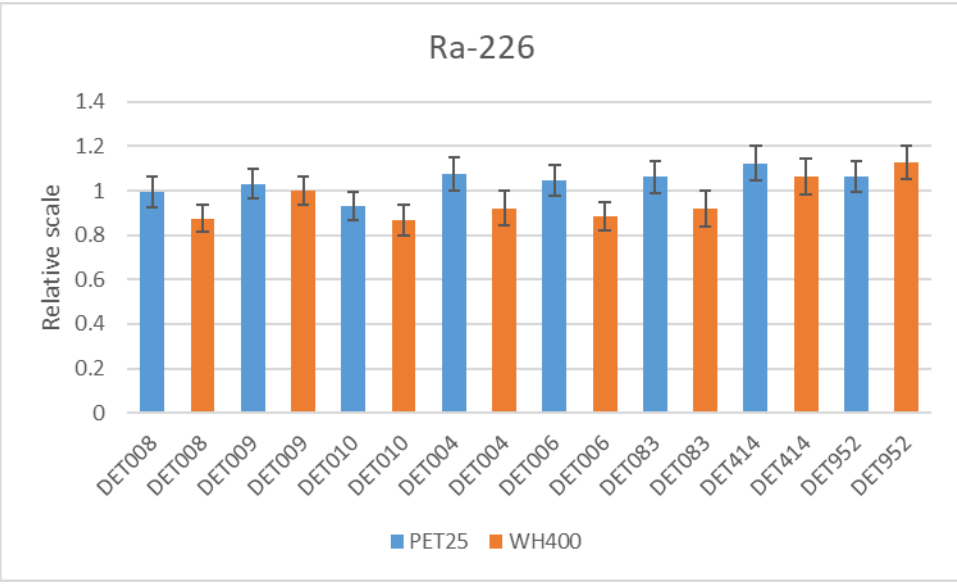


Results normalised to average values

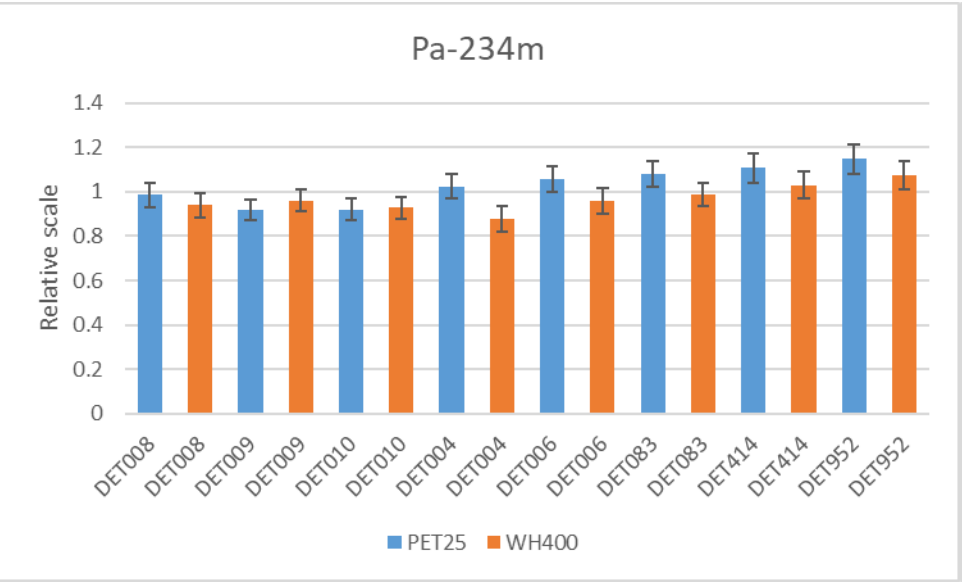


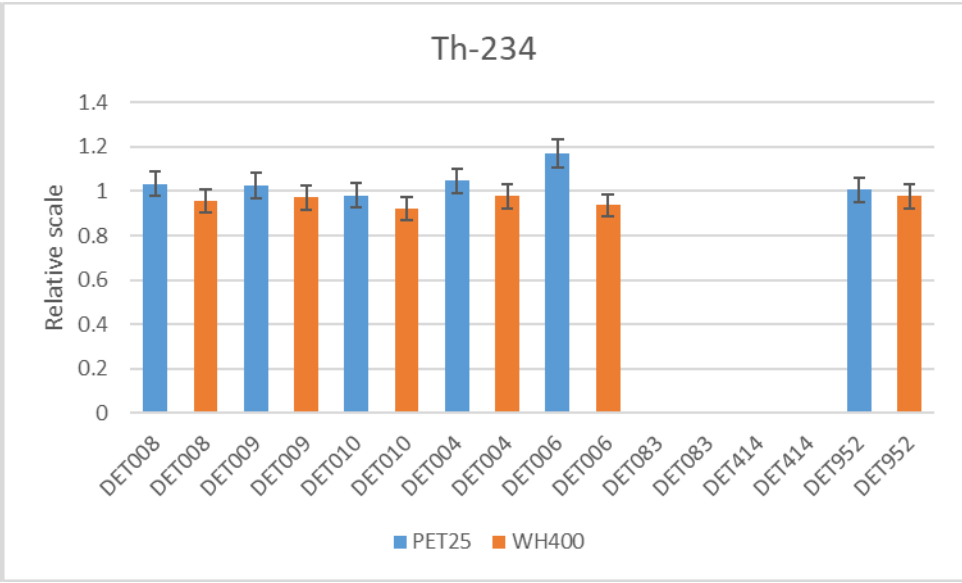


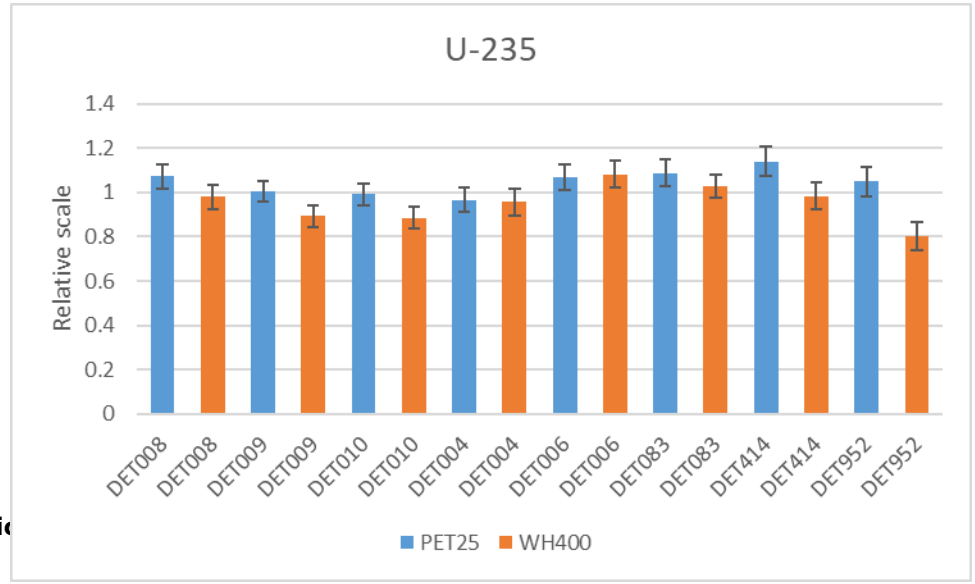




Results normalised to average values







Comparing results normalised to average values

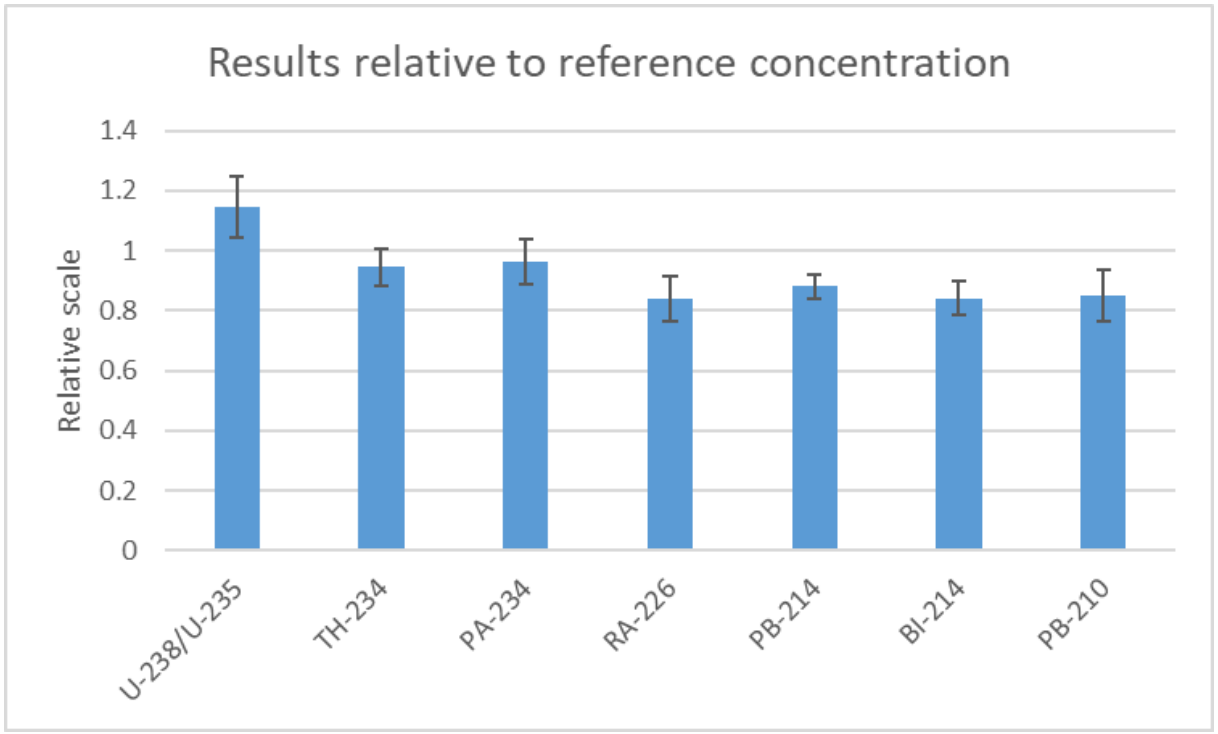
- Testing differences between detectors and geometries
- Using normalised results with analytical uncertainties
- Table shows average results across detectors

Isotope	Geometry PET25	Geometry WH400	Chi2-test
U-235	1.04 ± 0.02	0.94 ± 0.02	Significant
Th-234	1.04 ± 0.02	0.96 ± 0.02	Not significant
Pa-234m	1.01 ± 0.02	0.97 ± 0.02	Not significant
Ra-226	1.04 ± 0.03	0.95 ± 0.03	Not significant
Pb-214	1.03 ± 0.02	0.97 ± 0.02	Not significant
Bi-214	1.01 ± 0.02	0.97 ± 0.02	Not significant
Pb-210	1.06 ± 0.04	0.92 ± 0.04	Not significant

- Differences between detectors and geometries not significant ($p<0.01$) except for U-235

Comparing results with reference concentration

- Reference concentration of 0.05% Uranium corresponds to 6140 Bq/kg U-238 and similar for daughter products in case of equilibrium



- Average results across detectors and geometries shown with standard deviations as error bars

Conclusions

- The reliability of gamma spectrometric results from eight Ge detectors has been tested from measurements of a certified reference material available in the lab (Uranium standard)
- The quality of the calibration in 2016 is found acceptable based on comparisons of analytical results which show general agreement across eight detectors and two geometries
- However, the comparisons do indicate a small systematic bias between geometries
- Results for U-235 and Ra-226 overpredict/underpredict reference concentrations with 15% due to insufficient interference corrections
- Results for Pb-214, Bi-214 and Pb-210 are about 15% lower than the reference concentration due to escape of Rn-222 from samples
- The quality of this comparison would improve from using radon-tight sealing of samples and allow time for ingrowth of decay products prior to analyses