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# Challenges in NORM analysis by gamma spectrometry: PT-2022 Sample 7 Spectrum Exercise of a spiked soil as a case study

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**and the TERC Proficiency Test Team**

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**External support:**

**Lucie Fiserova, Alexander Mairing, Henrik Persson**

**Andrey Bosko, Nikolaus Hermanspahn**



**ALMERA**



**IAEA**

International Atomic Energy Agency

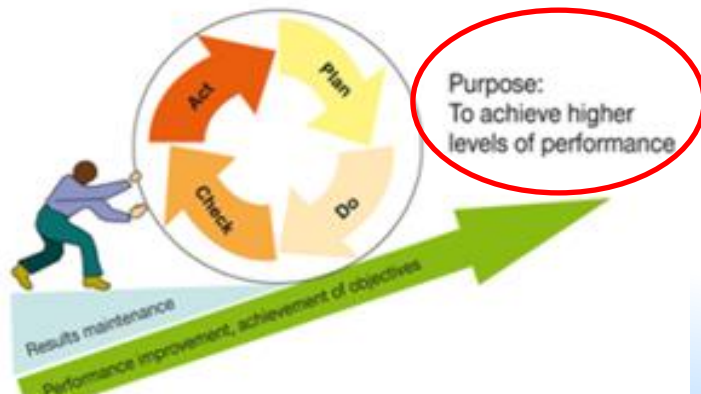
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# Presentation outline

- 1. Why:                    Scope**
  
- 2. What, how:    2.a Exercise overview**  
**2.b Reporting of results**  
**2.c Interactive review of results**

# 1. Why a Spectrum exercise

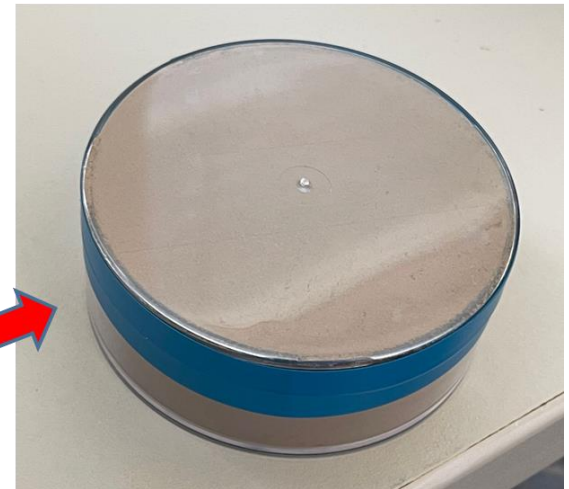
- **Easier logistics for organizers and participants**
- **No worries about dose rates or contamination of the laboratory**
- Practice analyzing external spectra and reporting results through official channels by a short deadline, as might be requested in an emergency situation
- **Focus on the actual analysis of the spectrum**
- Opportunity for Process Improvement (self assessment)



ISO/IEC 17025  
Management System  
Requirement

## 2.a What – PT2022 Spectrum exercise

- Spectrum obtained by measuring a spiked soil sample of undisclosed origin
- The soil has been
  - ✓ stored for more than 6 years, after spiking, into a plastic container which is not proven to be radon tight
  - ✓ prepared for measurement in a P105 geometry (105 cc)



## 2.a What – PT2022 Spectrum exercise

- Spectrum obtained by measuring the spiked soil sample on a BEGe5030 detector (BBE)

Equipment / Parameters (or model)	
<b>Detector Name</b>	PT2022 (BBE)
Detector Model	BE5030P
Detector Type	Broad energy, planar
High Voltage	+ 4000 V
Nominal Energy range	3-10000 keV
Relative efficiency	N/A
Cryostat	Low bkg. U-style
Endcap/Window	Al endcap Carbon-Epoxy window
<b>Lead shielding</b>	777-Canberra
Pb, mm	150
Cd, mm	2
Cu, mm	3
<b>Digital signal analyzer</b>	LYNX-MCA FW V1.1.83.1
<b>Software</b>	Genie 2000 v3.4
<b>Software</b>	ISOCS/LabSOCS v4.4





## 2.a What – PT2022 Spectrum exercise

- Spectrum obtained by measuring the spiked soil sample on a BEGe5030 detector (BBE)

**BBE is experimentally calibrated**  
for P105 geometry  
Energy range 46.5keV - 1836keV



## 2.a What – PT2022 Spectrum exercise

- Spectrum obtained by measuring the spiked soil sample on a BEGe5030 detector (BBE)

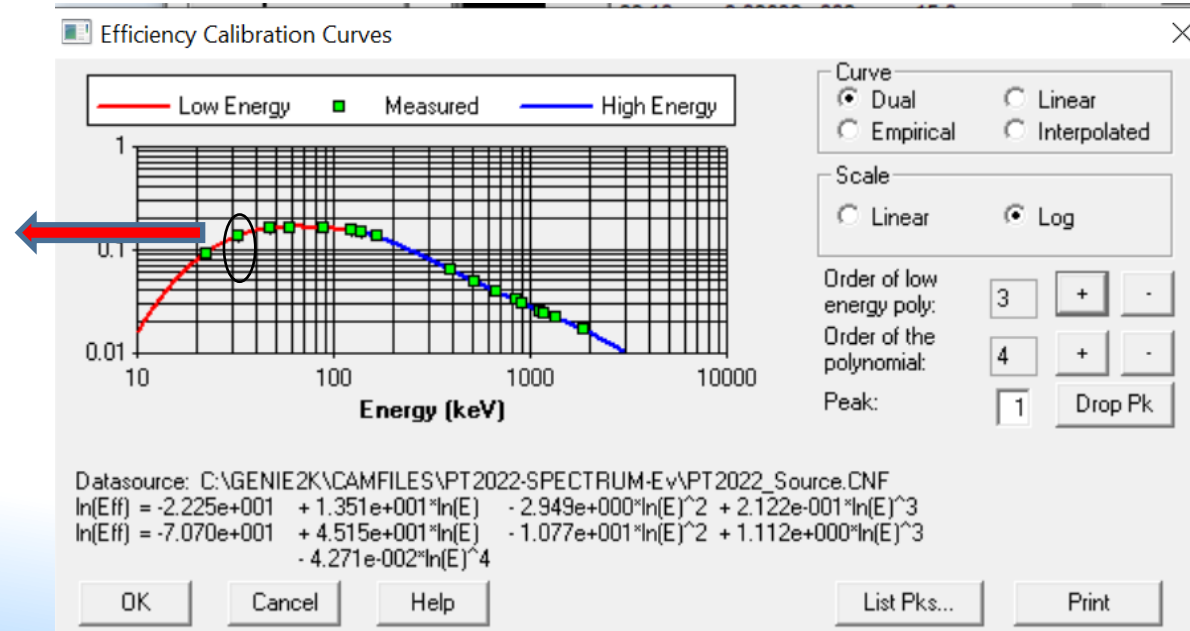
### BBE is LabSOCS characterized

- Original characterization from 45 keV
- “PT2022” detector characterization was reworked to allow efficiency outputs down to 10 keV

List Peaks

Pk/Index	X-Value (keV)	Calculated Efficiency
1	22.10	0.09095
2	32.06	0.13275
3	46.54	0.15783
4	59.53	0.16319

OK Help



## 2.a What – PT2022 Spectrum exercise

- Spectrum obtained by measuring the spiked soil sample on a BEGe5030 detector (BBE)

**BBE modelling parameters are available (“nOn”-Canberra users)**

### Detector PT2022 (BBE):

Crystal diameter	81.00	mm	End cap (housing) diameter	101.60	mm
Crystal length	31.50	mm	End cap (housing) thickness	1.50	mm
			End cap (housing) material	aluminium	
Bulletization (crystal rounding) radius	0.00	mm			
Top dead layer	0.00	mm	Window thickness	0.50	mm
Side dead layer	0.80	mm	Window-to-crystal gap	5.35	mm
			Window material	carbon	
Crystal hole (cavity) length	0.00	mm			
Crystal hole (cavity) diameter	0.00	mm			
Crystal material	germanium		Mount cup (holder) thickness	1.45	mm
			Mount cup (holder) material	copper	



# 2.a What – PT2022 Spectrum exercise

- the spiked soil sample measurement setup (BBE):



<b>Geometry</b>	<b>P105-26.5mm</b>
Container type	Cylinder
Container diameter	73.8 ± 0.05 (inner) or 77.8 ± 0.05 mm (outer)
Bottom thickness	1.5 ± 0.05 mm
Side thickness	2.0 ± 0.05 mm
Container material	Plexiglas
Container-endcap distance	Approximately <b>1 ± 0.2 mm</b> <b>1.1 ± 0.05 mm</b>
Sample holder present	Yes
Absorber present	No
Sample material	Tarjanian Soil*
Sample weight	145.32 ± 0.03 grams
Sample density (estimate)	between 1.28 and 1.29 g/cm <sup>3</sup>
Filling height	26.5 ± 0.05 mm

## Impact on PT2022 Sample:

On efficiency:

< 0.4%

On efficiency uncertainty:

≤ 1.5% Vs ≤ 0.4%

## 2.a How – PT2022 Spectrum exercise Package



- PT2022 Sample 7 package:
  - ✓ Overview of suggested and required steps
  - ✓ Supporting Files and Information



### Review Team 1:



*Lucie Fiserova, Alexander Muring, Henrik Persson*

- ✓ Tested and debugged the beta version of the PT2022 Spectrum Exercise Package
- ✓ Provided valuable inputs and recommendations for improvement
- ✓ Confirmed readiness of the final package, delivered in June 2022

## 2.b What – PT2022 Spectrum exercise Reporting

- PT2022 Sample 7 package :

**69** Laboratories from the ALMERA Network

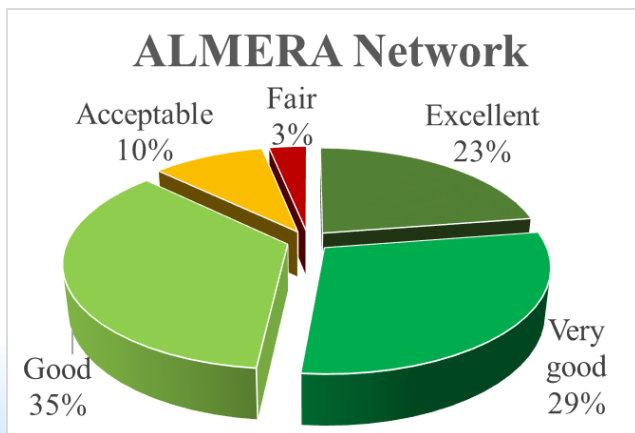
**128** Laboratories from the World-Wide Network

=

**197** Laboratories reported their results

AND

**89** Laboratories reported **their feedback**



**My feedback:**





## 2.b What – PT2022 Spectrum exercise Reporting

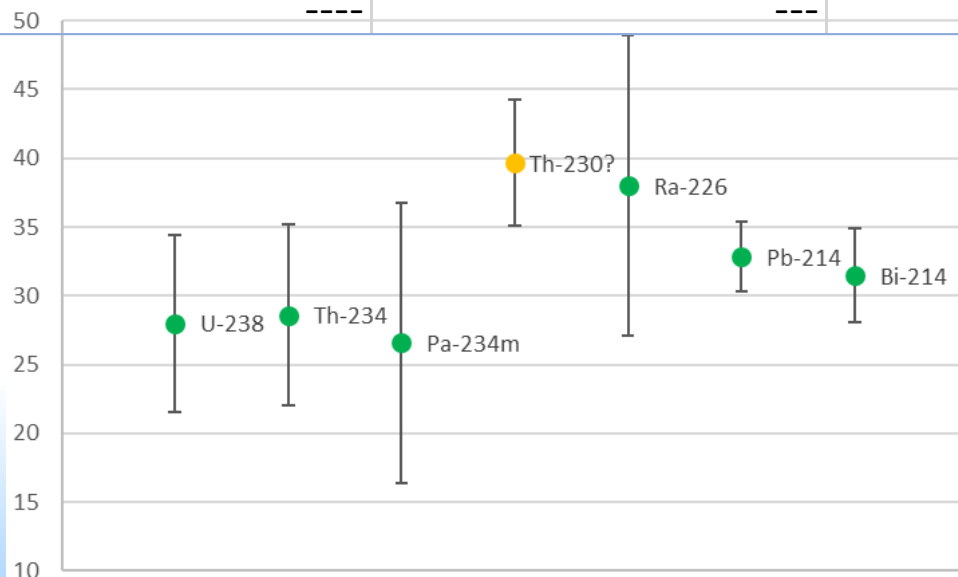
- Spiked radionuclides

Analyte	Rob. Avg. [Bq/kg]	Rob. Std. Dev. [Bq/kg]	# Reported Results
K-40	449.74	31.25	193
Co-60	123.96	8.12	175
Cs-134	39.8	3.85	175
Cs-137	83.2	4.79	175
Am-241	78.89	7.59	169
Ba-133	62.13	8.76	165
Pb-210	621.79	80.1	126
Sn-113	1.84	0.26	27
Mn-54	0.59	0.08	17
Cd-109	55.5	39.4	8
Eu-155	-----	-----	1
Po-210	-----	-----	1
Y-88	-----	-----	1

# 2.b What – PT2022 Spectrum exercise Reporting

- U-238 decay series

Analyte	Rob. Avg. [Bq/kg]	Rob. Std. Dev. [Bq/kg]	# Reported Results
U-238	28	6.45	16
Th-234	28.6	6.56	66
Pa-234m	26.57	10.19	35
Th-230? ?	39.65	4.58	4
Ra-226	38	10.91	146
Pb-214	32.86	2.5	94
Bi-214	31.49	3.39	100
U-234 ?	----	---	1



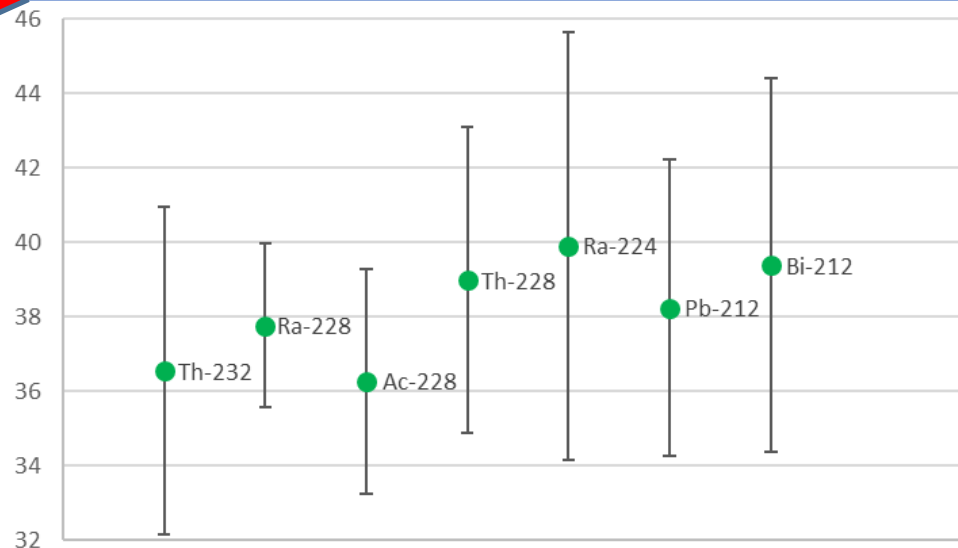


# 2.b What – PT2022 Spectrum exercise Reporting

- Th-232 decay series

Analyte	Rob. Avg. [Bq/kg]	Rob. Std. Dev. [Bq/kg]	# Reported Results
Th-232	36.55	4.41	11
Ra-228	37.77	2.2	29
Ac-228	36.27	3.03	118
Th-228	38.98	4.11	27
Ra-224	39.91	5.74	27
Pb-212	38.24	3.99	96
Bi-212	39.39	5.02	81
Tl-208	13.25	1.92	90

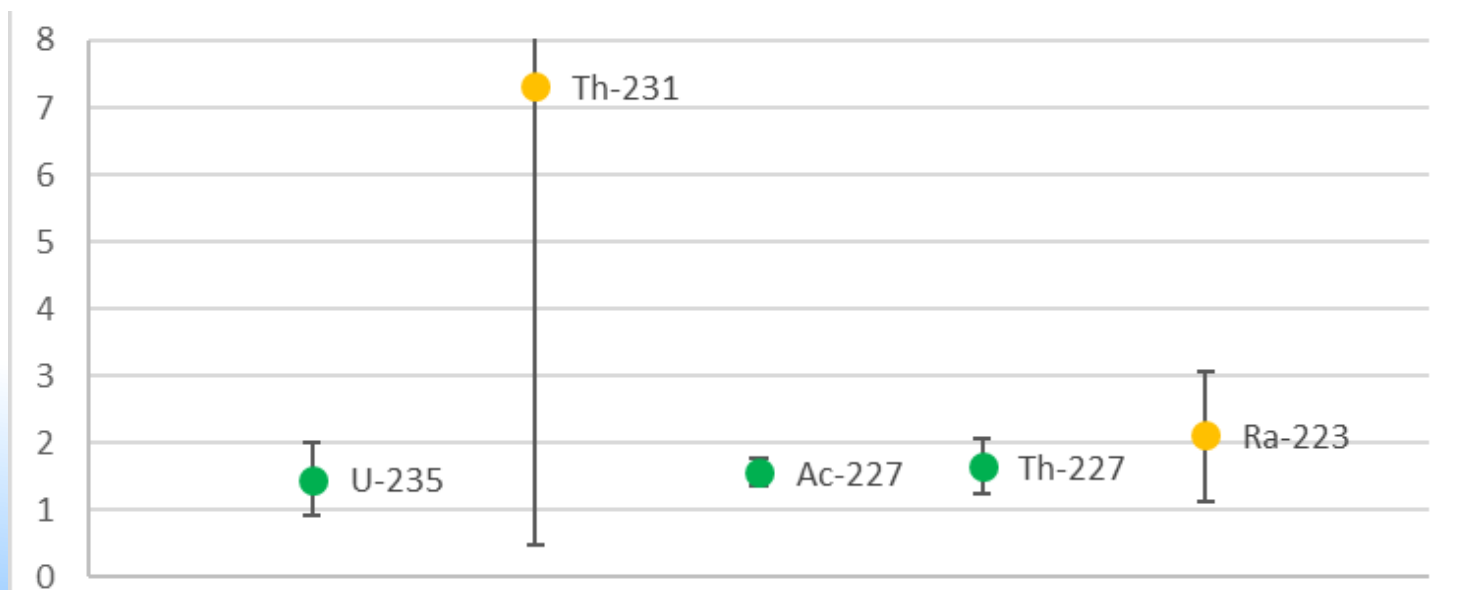
Tl-208: **36.83 Bq/kg**, (i.e. consistent) if 35.93% alpha-decay branch of Bi-212 is taken into account



# 2.b What – PT2022 Spectrum exercise Reporting

- U-235 decay series

Analyte	Rob. Avg. [Bq/kg]	Rob. Std. Dev. [Bq/kg]	# Reported Results
U-235	1.46	0.55	117
Th-231	7.3	6.82	5
Ac-227	1.56	0.21	5
Th-227	1.65	0.41	15
Ra-223 ?	2.1	0.97	2
Bi-211 ?	11.55 ?	1.85	1



## 2.c What – PT2022 Interactive review of results

- U-235, U-238, Th-232 decay series + false positives ?

### Review Team 2:

*Barbara Nadalut*

*Gamma Spectrometry Specialist at TERC - NAPC, IAEA*

*Andrey Bosko*

*Radiometry Team Leader at NML - SGAS, IAEA*

*Nikolaus Hermanspahn*

*PT Evaluation Team Leader, IMS/ED, CTBTO*



- ✓ Feedback on nuclide identification (false positives)
- ✓ Corroboration of PT results

### **CHALLENGE:**

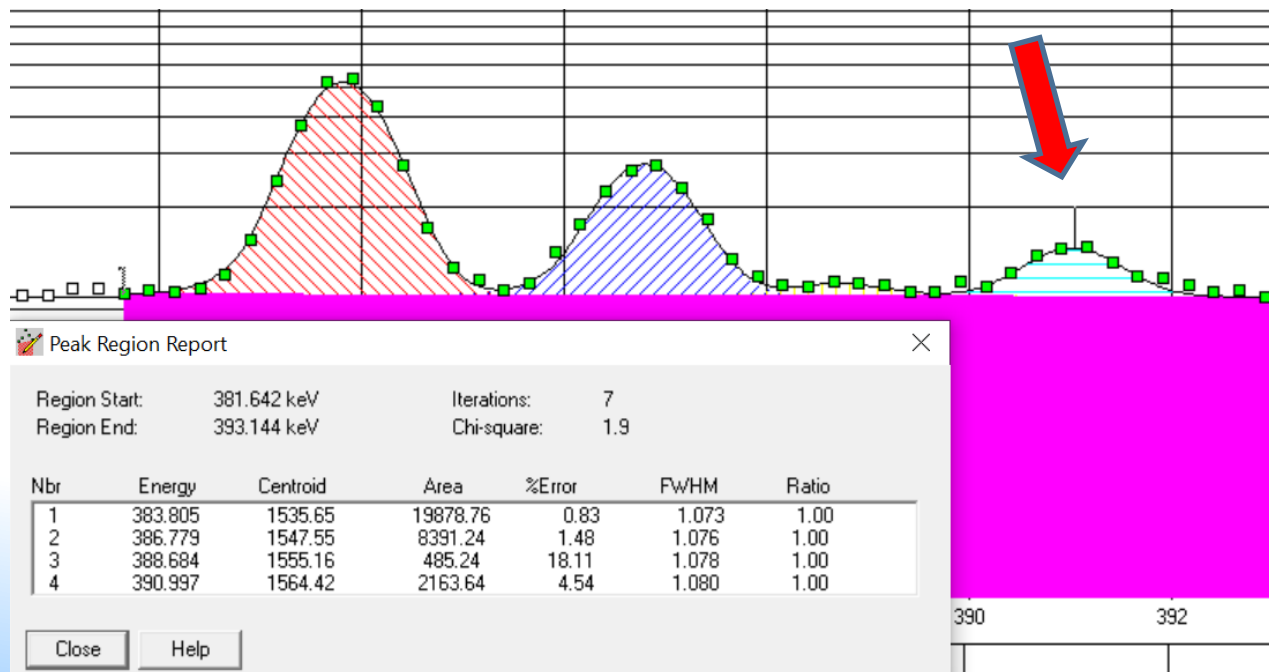
Only results are available, no additional information on methods – feedback is based on assumptions.

## 2.c What – PT2022 Interactive review of results

- Spiked radionuclides – False positives ?

Analyte	Rob. Avg. [Bq/kg]	Rob. Std. Dev.[Bq/kg]	# Reported Results
Sn-113	1.84	0.26	27

**Our assumption:** False identification due to the Ba-133 Gamma-X TCS ( $356.0129 + 34.987 = 391$  keV)

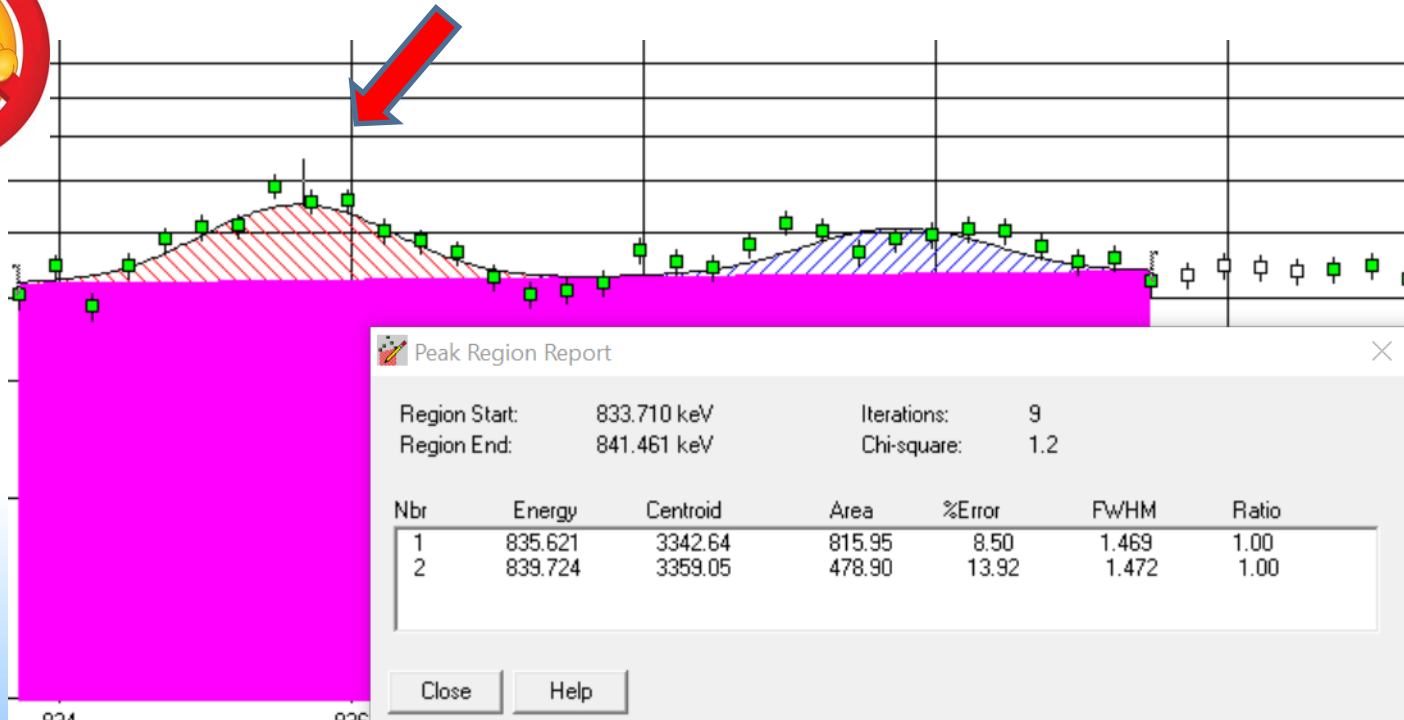


## 2.c What – PT2022 Interactive review of results

- Spiked radionuclides – False positives ?

Analyte	Rob. Avg. [Bq/kg]	Rob. Std. Dev.[Bq/kg]	# Reported Results
Mn-54	0.59	0.08	17

**Our assumption:** 834.848 keV peak is missing. Likely, false identification using the 835.71 keV line of Ac-228



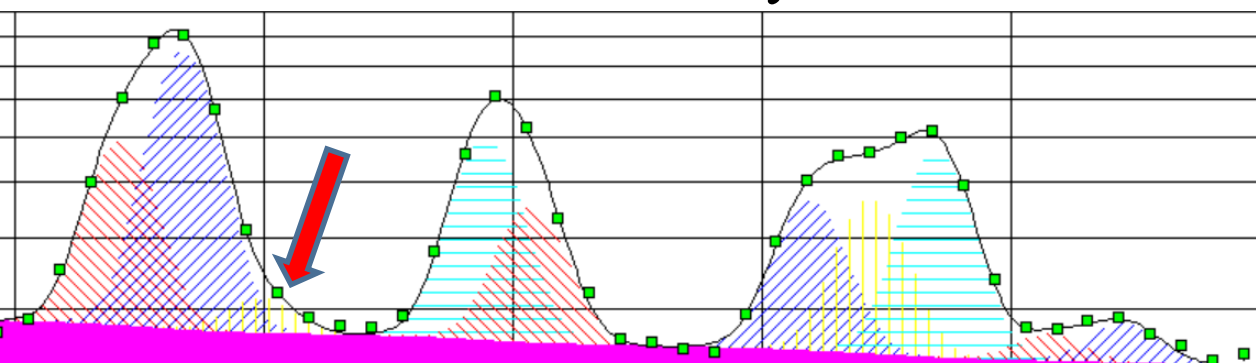


## 2.c What – PT2022 Interactive review of results

- Spiked radionuclides – False positives ?

Analyte	Rob. Avg. [Bq/kg]	Rob. Std. Dev.[Bq/kg]	# Reported Results
Cd-109	55.5	39.4	8

**Our assumption:** Likely, false identification using the 87.3 keV Bi X-rays. Nevertheless.....



**IF** peaks deconvolution is trusted, then Cd-109 activity based on 88keV peak is more than 10 times lower than the robust average:

$$4.5 \pm 1.0 \text{ Bk/kg}$$

Region Start:	85.847 keV	Iterations:	19			
Region End:	96.099 keV	Chi-square:	1.4			
Nbr	Energy	Centroid	Area	%Error	FWHM	Ratio
1	86.848	348.00	8631.75	4.91	0.636	1.00
2	87.343	349.98	15816.26	2.32	0.637	1.00
3	87.966	352.47	1144.66	19.97	0.638	1.00
4	89.774	359.70	9342.71	7.44	0.642	1.00

# 2.c What – PT2022 Interactive review of results

- Spiked radionuclides – False positives ?

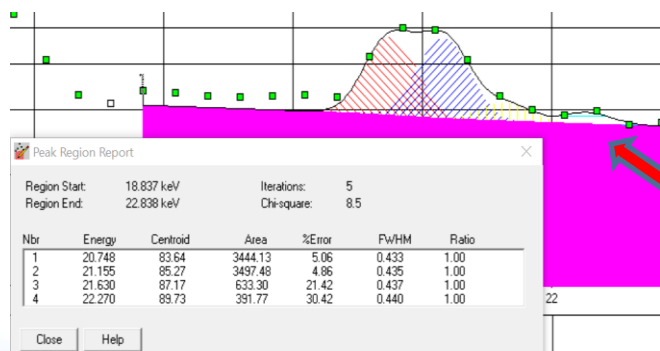
Analyte	Rob. Avg. [Bq/kg]	Rob. Std. Dev.[Bq/kg]	# Reported Results
Cd-109	55.5	39.4	8

**Our cross-check:** Corroboration to identification from Cd X-rays ?

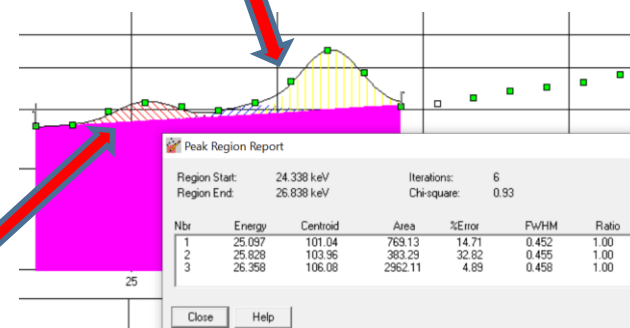
Germanium escape from Cs X-rays ?



Germanium escape from Cs X-rays ?



Energy (keV)	Intensity (%)	Type	Origin*
22.16317	55.1	X <sub>Kα1</sub>	Ag
(-)	(5)		
21.9906	29.21	X <sub>Kα2</sub>	Ag
(-)	(30)		
25.0002	15.25	X <sub>Kβ1</sub>	Ag
(-)	(20)		
3.191	10.37	X <sub>L</sub>	Ag
(-)	(27)		
88.0336	3.66	γ	Ag-109
(10)	(5)		
25.4843	2.65	X <sub>Kβ2</sub>	Ag
(-)	(10)		



?Cd-109?

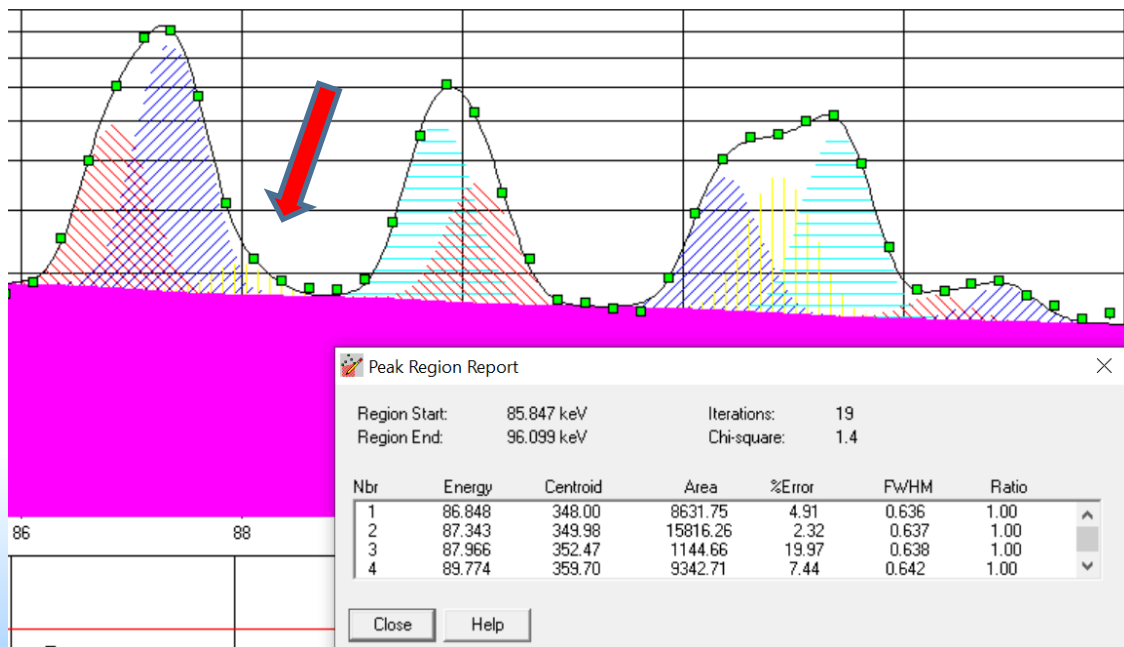


# 2.c What – PT2022 Interactive review of results

- Spiked radionuclides – False positives ?

Analyte	Rob. Avg. [Bq/kg]	Rob. Std. Dev.[Bq/kg]	# Reported Results
Cd-109	55.5	39.4	8

**Question:** What is the Limit of Detection for Cd-109 in this spectrum?



### ALMERA

Labcode	Rep. Value	Rep. Unc.
1	109.7	8.4
28	65.1	2.1

### World Wide

Labcode	Rep. Value	Rep. Unc.
29	4.7	0.89
32	63.8	1.4
76	57.2	2.50
83	62.1	1.8
175	6.9	1.0
299	74.5	11.3

?Cd-109?

**NO WAY !**

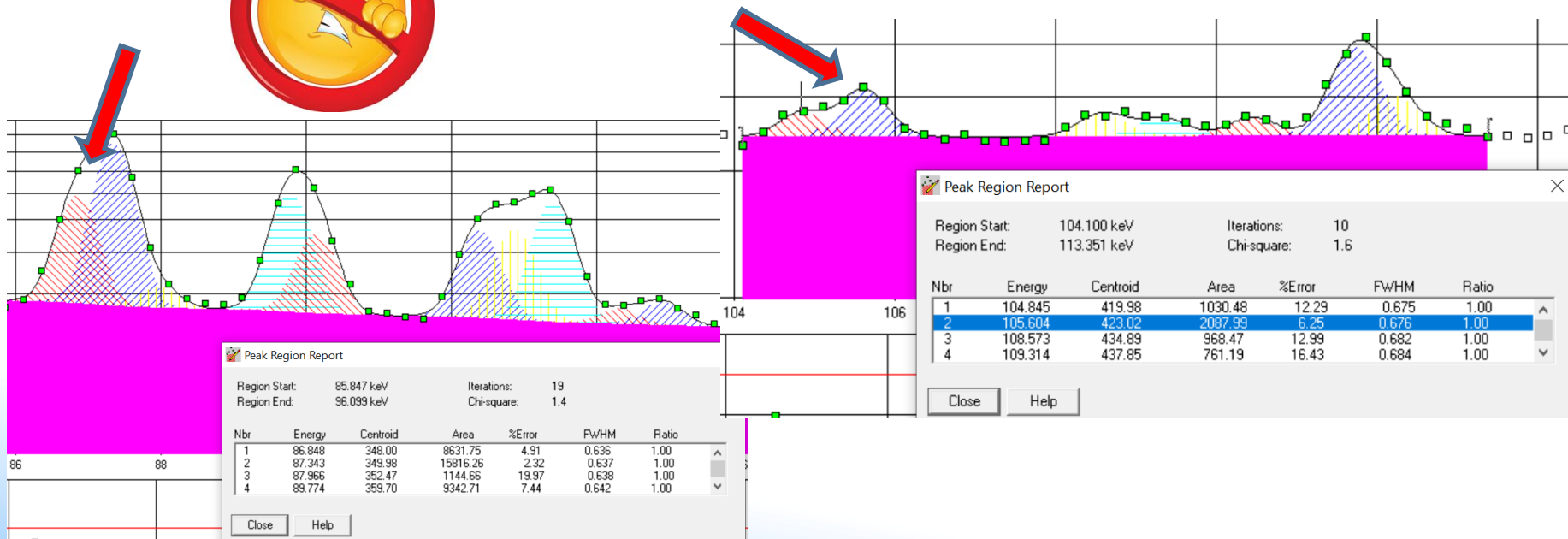


# 2.c What – PT2022 Interactive review of results

- Spiked radionuclides – False positives ?

Analyte	Rob. Avg. [Bq/kg]	Rob. Std. Dev.[Bq/kg]	# Reported Results
Eu-155	-----	-----	1

**Our assumption:** Likely, false identification using the 105.6 keV line of Ac-228 and/or the 87.3 keV Bi X-rays



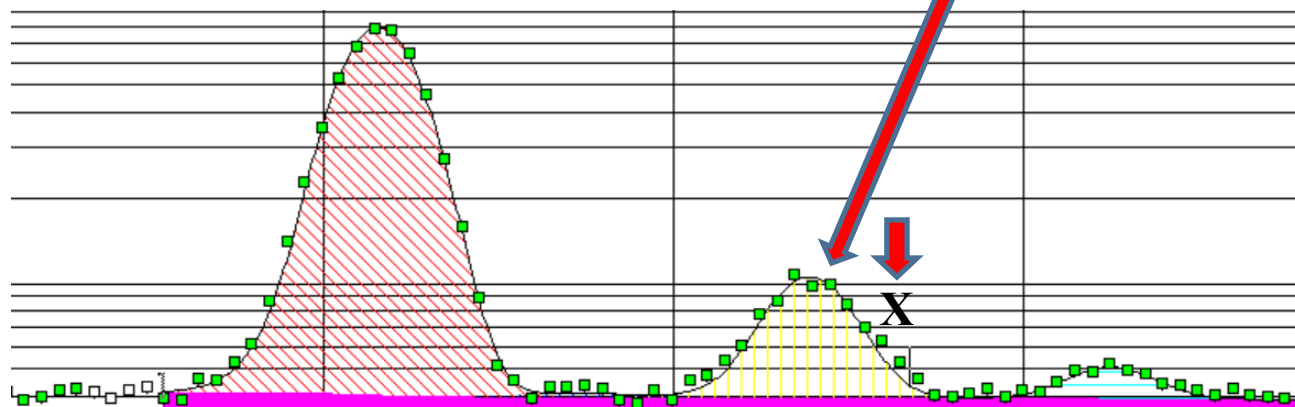
# 2.c What – PT2022 Interactive review of results

- Spiked radionuclides – False positives ?

Analyte	Rob. Avg. [Bq/kg]	Rob. Std. Dev.[Bq/kg]	# Reported Results
Po-210	-----	-----	1

Our assumptions:

Likely, false identification using the 801.95 keV line of Cs-134.



Peak Region Report

Region Start:	792.704 keV	Iterations:	32
Region End:	808.957 keV	Chi-square:	2.3

Nbr	Energy	Centroid	Area	%Error	FWHM	Ratio
1	795.769	3183.26	47956.05	0.45	1.439	1.00
2	798.642	3194.75	99.92	61.78	1.441	1.00
3	801.922	3207.86	4214.54	2.09	1.444	1.00
4	806.217	3225.04	741.02	9.11	1.447	1.00

Close Help

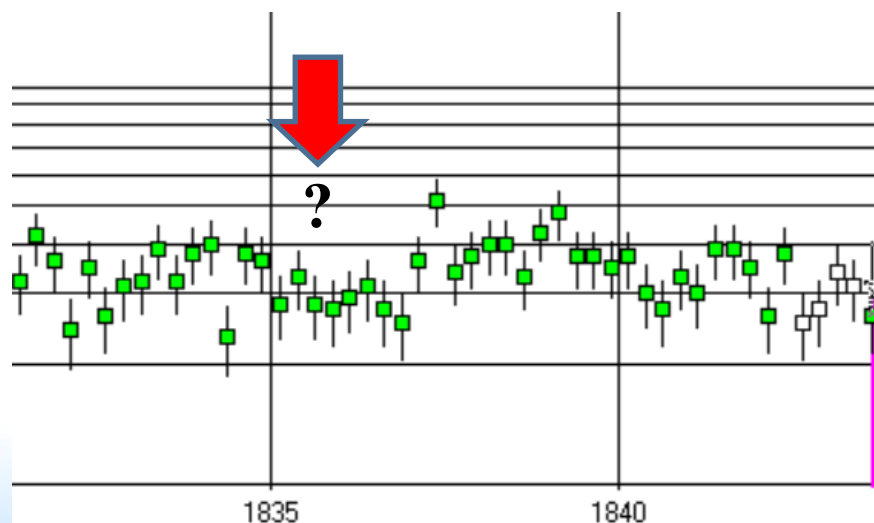
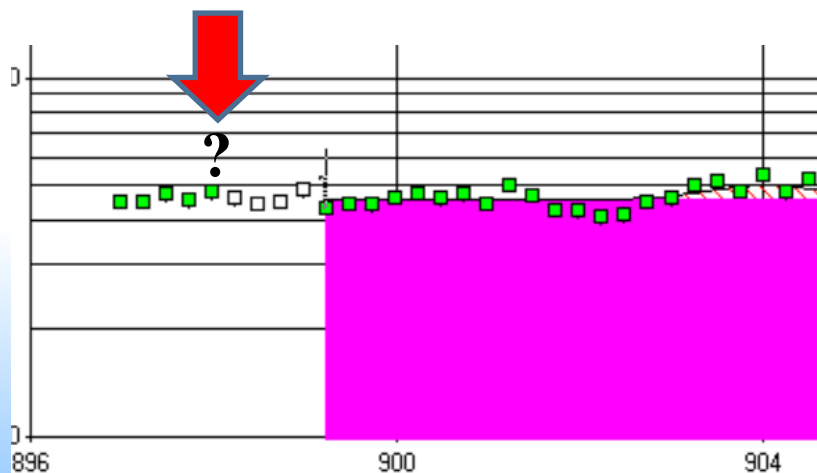


## 2.c What – PT2022 Interactive review of results

- Spiked radionuclides – False positives ?

Analyte	Rob. Avg. [Bq/kg]	Rob. Std. Dev.[Bq/kg]	# Reported Results
Y-88	-----	-----	1

**No assumptions:** Peaks not present, not clear how it was identified.....

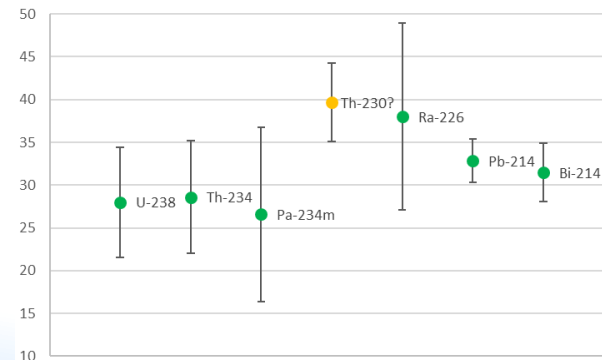
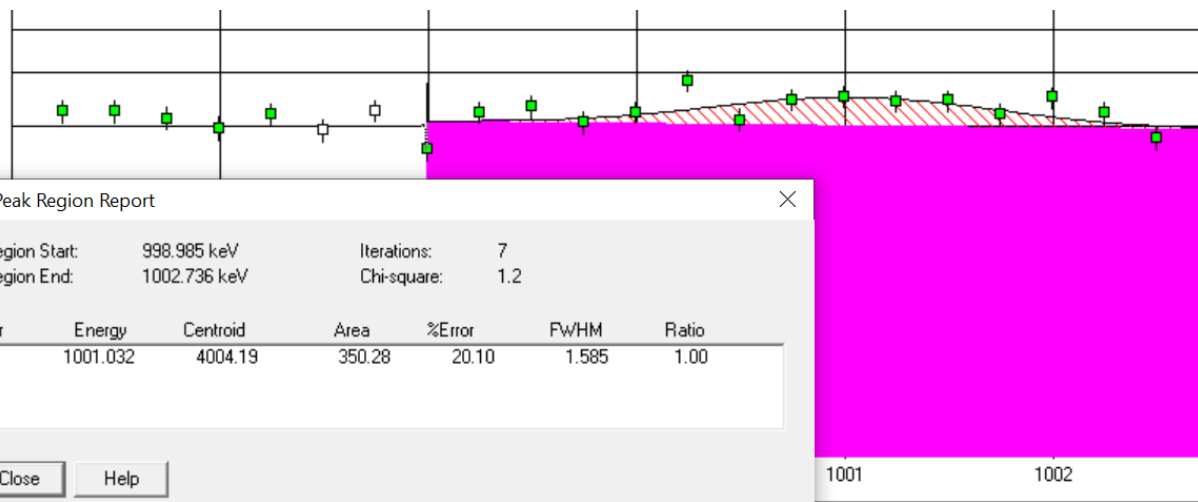


# 2.c What – PT2022 Interactive review of results

- U-238 decay series

Analyte	Rob. Avg. [Bq/kg]	Rob. Std. Dev. [Bq/kg]	# Reported Results
Pa-234m	26.57	10.19	35

Small peak around the 1001 keV peak, which may be quantified, but with very large uncertainty



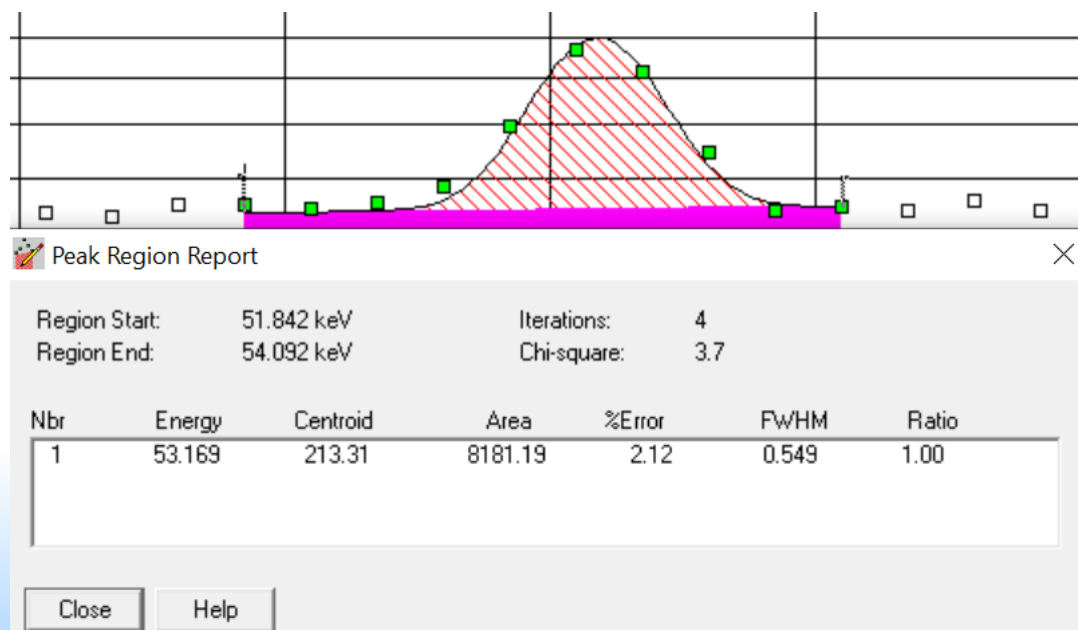
## 2.c What – PT2022 Interactive review of results

- U-238 decay series

Analyte	Rob. Avg. [Bq/kg]	Rob. Std. Dev. [Bq/kg]	# Reported Results
U-234	----	---	1

The strongest U-234 peak at 53.2 keV is very weak, and would not have been seen on such background.

Likely a false identification on the 53.2 keV line (Ba-133? Pb-214?)



# 2.c What – PT2022 Interactive review of results

- U-238 decay series

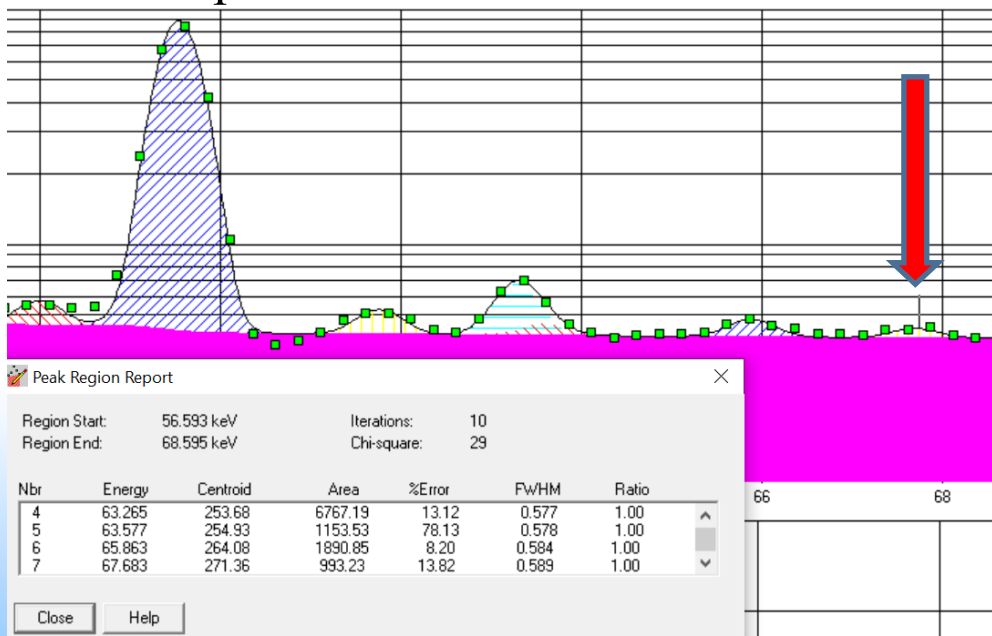
Analyte	Rob. Avg. [Bq/kg]	Rob. Std. Dev. [Bq/kg]	# Reported Results
Th-230?	39.65	4.58	4

Likely using a very weak 67.67 keV line in the highly complicated region?

This result is ~30% higher than one would expect from NU in nature.

Possible additional contribution from the Bi X-ray Ge escape peak in this region?

Other possible reasons?



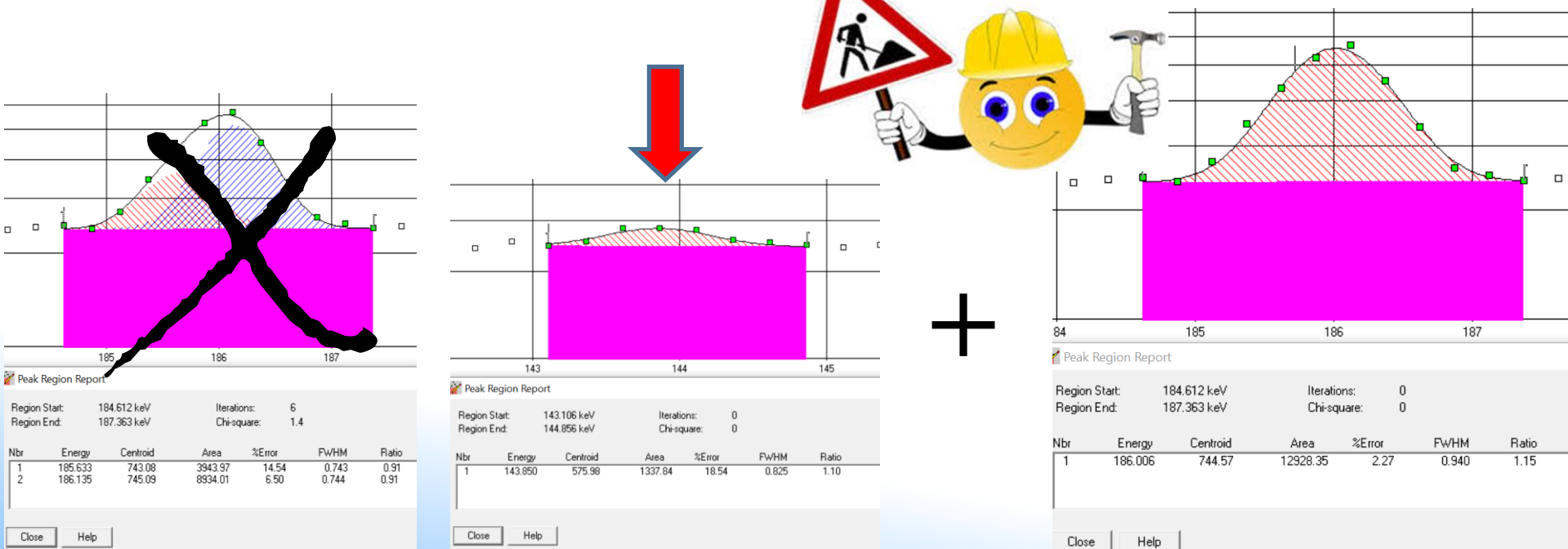
# 2.c What – PT2022 Interactive review of results

- U-238 decay series: Ra-226 Vs U-235

Analyte	Rob. Avg. [Bq/kg]	Rob. Std. Dev.[Bq/kg]	# Reported Results
Ra-226	38	10.91	146

Likely using 143.8 keV line for U-235 quantification, and manually subtracting corresponding peak area from 185.7-186.2 keV doublet?

Other options?



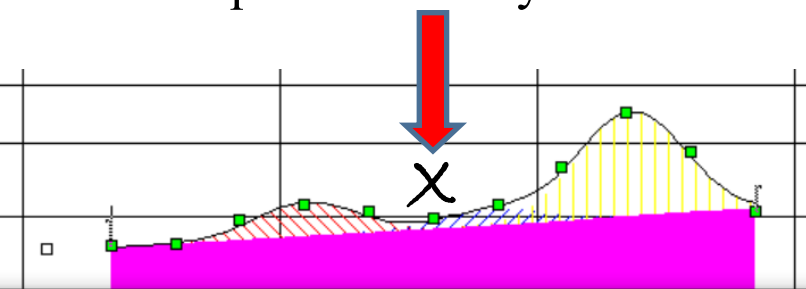
# 2.c What – PT2022 Interactive review of results

- U-235 decay series:

Analyte	Rob. Avg. [Bq/kg]	Rob. Std. Dev.[Bq/kg]	# Reported Results
Th-231	7.3	6.82	5

The most intense 25.64 keV peak is substantially missing; identification/quantification due to Th-228 (84.2-84.4 keV).

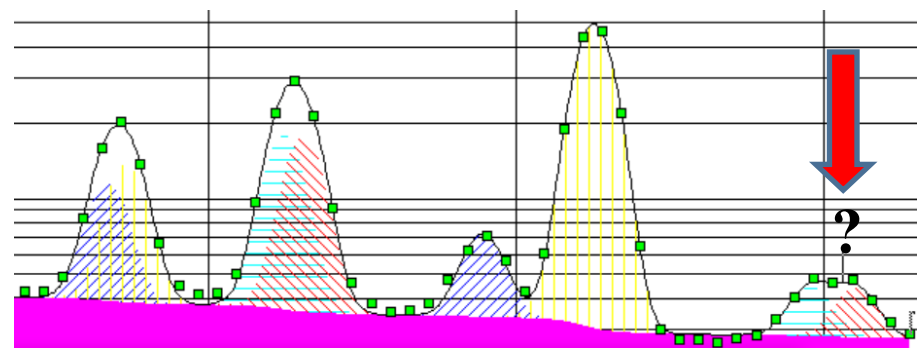
Insignificant estimated contribution to the overall 84.2 keV peak area, from expected activity



Peak Region Report

Region Start: 24.338 keV      Iterations: 6  
 Region End: 26.838 keV      Chi-square: 0.93

Nbr	Energy	Centroid	Area	%Error	FWHM	Ratio
1	25.097	101.04	769.13	14.71	0.452	1.00
2	25.828	103.96	383.29	32.82	0.455	1.00
3	26.358	106.08	2962.11	4.89	0.458	1.00



Peak Region Report

Region Start: 71.845 keV      Iterations: 8  
 Region End: 85.097 keV      Chi-square: 8.2

Nbr	Energy	Centroid	Area	%Error	FWHM	Ratio
6	79.547	318.80	10589.26	1.52	0.619	1.00
7	80.990	324.57	126939.09	0.30	0.622	1.00
8	83.810	335.85	4616.96	4.38	0.629	1.00
9	84.388	338.16	4465.87	4.51	0.630	1.00

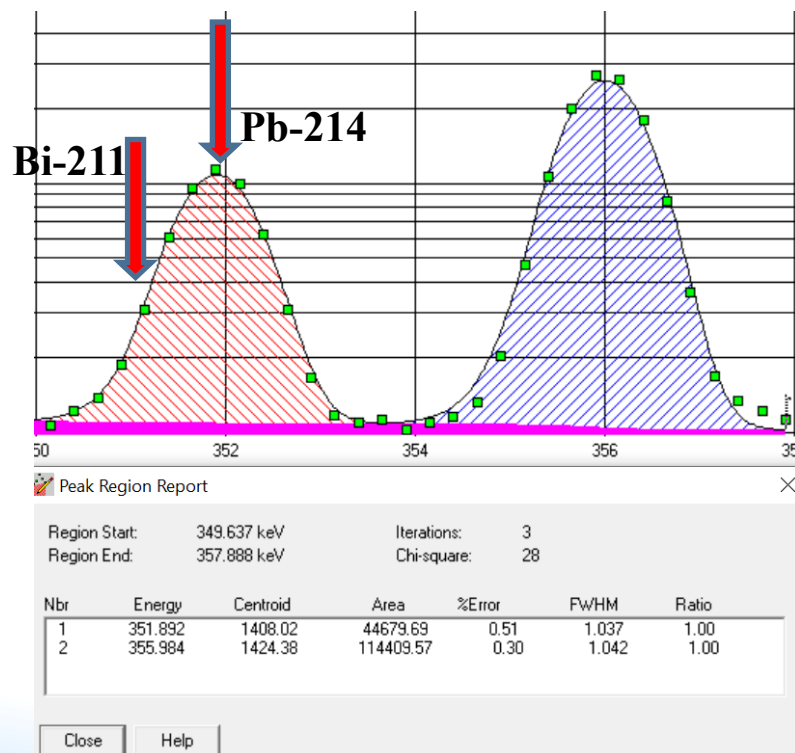


## 2.c What – PT2022 Interactive review of results

- U-235 decay series:

Analyte	Rob. Avg. [Bq/kg]	Rob. Std. Dev. [Bq/kg]	# Reported Results
Bi-211	11.55	1.85	1

Bi-211 has Significant interference for the only gamma-peak at 351.07 keV. The interfering peak (351.9 keV from Pb-214) is more than 50 times stronger than the expected peak of Bi-211 (assuming it is in equilibrium with U-235), so no interference correction is possible



## 2.c What Next ?

- Any comments, interactions or contributions from Laboratory experts are very welcome
- If interested, please contact the Proficiency Test Team at the contact email address:

[Proficiency-Tests.Contact-Point@iaea.org](mailto:Proficiency-Tests.Contact-Point@iaea.org)

BUT: please be a bit patient.....

During Q4/2023 the NAPC-TERC Proficiency Test team will be very busy with

**Proficiency Test 2023**

**data review and evaluation work**

(deadline for submission of PT2023 results is end of September).





**IAEA**

International Atomic Energy Agency  
*Atoms for Peace and Development*

*Any questions?*

*Thank you!*