L15g Validation of urine analysis measurements

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Validation planning

Validation of indirect internal monitoring by urine analysis can be done by:

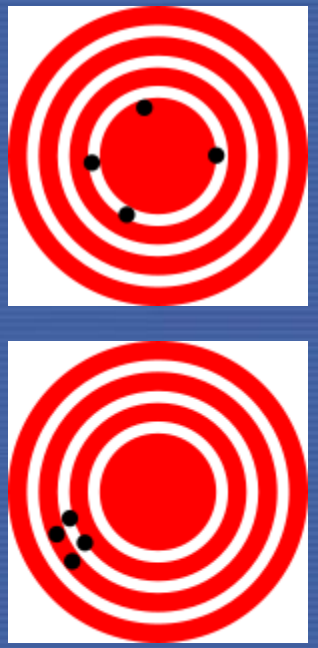
- Calibration of the measurement instrument (using standard reference materials)
- Performance Testing
- Systematic testing using controls and by
- Laboratory intercomparison

Performance criteria

The performance criteria are recommended by ANSI N13.30-1996.

For measurements at the Minimum Testing Level:

- **Accuracy – 25% to + 50%**
- **Precision < 40 %**



Accuracy

- Accuracy of one measurement may be defined as bias $\mathbf{B_i}$:
  - $B_i = \frac{(A_i - A)}{A}$

- Where
- $\mathbf{A_i}$ is the value of the i^{th} measurement
- **A** is the actual quantity in the test sample

Relative Bias

Use relative bias, which may be obtained at different activity levels.

Calculate from the relative bias B_{ri} as:

$$B_r = \bar{B}_i = \sum_{i=1}^{n} B_i / N$$

- Where
- **N ≥ 5** is the number of test samples measured by the lab.

Precision

- Precision is the relative dispersion of the values of B_{ri} from its mean B_r:

$$S_B = \sqrt{\frac{\sum_{i=1}^{N} (B_i - B_r)^2}{(N-1)}}$$

Minimum Testing Level (MTL)

- The MTL should preferably be five times, but may be ten times the Minimum Detectable Amount (MDA) as defined by

$$MDA = \frac{4.65\ sb + 3}{KTe^{-\lambda\Delta t}}$$

- Where
- **sb** is the standard devia t
- **K** is the calibration constant
- **T** is the counting time
- **Λ** is the radiological decay constant
- **Δt** is the elapsed time between an establish reference date and the analysis date.

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MTL examples

Measurement Category	Radionuclide	MTL (per liter or per sample)
BETA activity: average energy <100 keV	^{3}H	2 kBq
	^{14}C	2 kBq
	^{35}S	20 Bq
	^{228}Ra	0.9 Bq
BETA activity: average energy = or >100 keV	^{32}P	4 Bq
	$^{89/90}Sr$ or ^{90}Sr	4 Bq
ALPHA activity: Isotopic analysis	$^{228/230}Th$ or ^{232}Th	0.02 Bq
	$^{234/235}U$ or ^{238}U	0.02 Bq
	^{237}Np	0.01 Bq
	^{238}Pu or $^{239/240}Pu$	0.01 Bq
	^{241}Am	0.01 Bq
GAMMA (photon) activity	^{137}Cs	2 Bq
	^{60}Co	2 Bq
	^{125}I	0.4 kBq

Validation planning

- Definition of standards to be used for calibration and measurement conditions (measuring time)
- Calibrate the detector system using above defined standards and record the calibration results.
- Measure a blank sample (reagent blank for UAL)
- Determine the MDA for each radionuclide of concern.

Validation planning

- Set the limits values for the MTL based on the criteria 5 times MDA.
- Prepare a testing sample with activity value above the MTL for each radionuclide of concern.
- Define measuring time for sample measurements.
- Carry out minimum five repetitive measurements on these samples with repositioning the samples after each measurement and record the results.

Validation planning

- Calculate B_r and sb as indicated above for each measured radionuclide.
- Compare B_r and sb with the acceptance values stated in the table above.
- If the calculated values meet the performance criteria the method is accepted as validated.
- Prepare a report on the performed measurements, results, acceptance values and outcome of the validation process for documentation.

System calibration

- The calibration source contains the following isotopes:

- **^{234}U 4.8 MeV**
- **^{239}Pu 5.2 MeV**
- **^{241}Am 5.5 MeV**

- The control source contains the following spike isotopes:

- **^{237}Np 4.8 MeV**
- **^{243}Am 5.3 MeV**
- **^{244}Cm 5.8 MeV**

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System calibration control

Nuclide	A_{ai} [kBq]	A_i [kBq]	B_{ri}	B_r	S_B
^{241}Am	1	9.97E -01	-3.32E -03	-0.01	0.013
		9.92E -01	-8.19E -03		
		9.90E -01	-1.05E -02		
		9.79E -01	-2.12E -02		
		1.02E+00	1.55E -02		
^{244}Cm	1	1.09E+00	8.93E -02	0.09	0.012
		1.09E+00	9.31E -02		
		1.07E+00	6.61E -02		
		1.09E+00	8.97E -02		
		1.10E+00	9.86E -02		
^{239}Pu	1	1.23E+00	2.27E -01	0.22	0.009
		1.22E+00	2.23E -01		
		1.22E+00	2.16E -01		
		1.21 E+00	2.12E -01		
		1.23E+00	2.33E -01		

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System control with spike sample

Nuclide	A_{ai} [Bq]	A_i [Bq]	B_{ri}	B_r	S_B
Np -237	0.0334	0.0319	-0.04	-0.10	0.05
	0.0335	0.0314	-0.06		
	0.0336	0.0280	-0.17		
	0.0333	0.0291	-0.13		
	0.0333	0.0302	-0.09		
Am -243	0.0304	0.0294	-0.03	0.01	0.04
	0.0303	0.0316	0.04		
	0.0305	0.0298	-0.02		
	0.0305	0.0304	-0.003		
	0.0304	0.0323	0.06		
Cm -244	0.0294	0.0215	-0.27	-0.22	0.06
	0.0293	0.0 234	-0.20		
	0.0294	0.0209	-0.29		
	0.0293	0.0238	-0.19		
	0.0292	0.0250	-0.15		

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Example of validation data

MDA = minimum detectable amount

MTL = minimum testing level

Nuclide	Energy [keV]	MDA [Bq]	MTL [Bq]
TH-228	5423	2.77E-04	1.39E-03
TH-229	4845	2.88E-04	1.44E-03
TH-230	4688	2.36E-04	1.18E-03
TH-232	4010	2.36E-04	1.18E-03
U-232	5320	2.39E-04	1.19E-03
U-233	4825	2.40E-04	1.20E-03
U-234	4776	2.38E-04	1.19E-03
U-235	4395	2.79E-04	1.39E-03
PU-236	5768	1.93E-04	9.64E-04
NP-237	4788	3.02E-04	1.51E-03
PU-238	5499	2.47E-04	1.24E-03
U-238	4196	5.81E-05	2.91E-04
PU-239	5156	6.03E-05	3.01E-04
PU-240	5168	6.03E-05	3.02E-04
AM-241	5486	2.63E-04	1.32E-03
PU-242	4901	2.47E-04	1.24E-03
AM-243	5277	2.63E-04	1.32E-03
CM-244	5805	2.29E-04	1.14E-03

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Validation by intercomparison

- Regular intercomparisons each year organized by:

http://www.procorad.org/uk/index.html

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PROCORAD samples

Typically samples are supplied for

- Tritium (H3)
- Actinides (Pu238, Pu239, Am241, Th230, Cm244)
- Uranium (U234, U235, U238)
- Gamma-emitters (Ba133, Cs134, Cs137, I129, Eu152, Mn54, K40)
- Surprise sample with unknown isotope composition.

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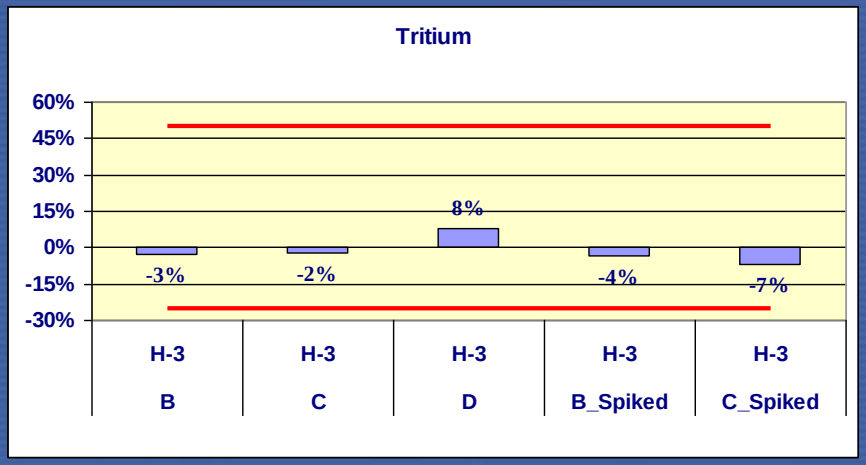
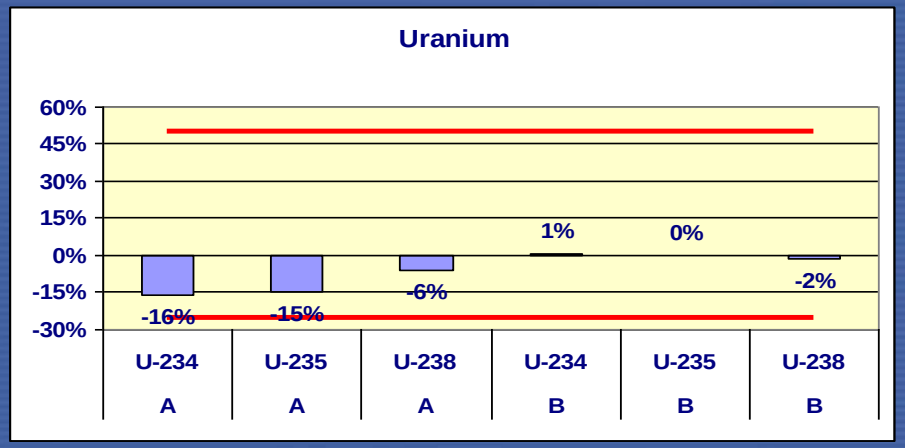
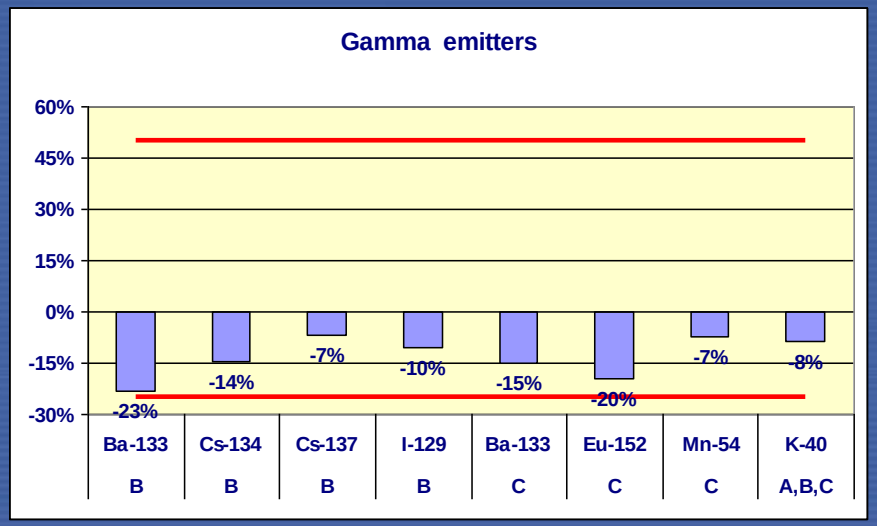
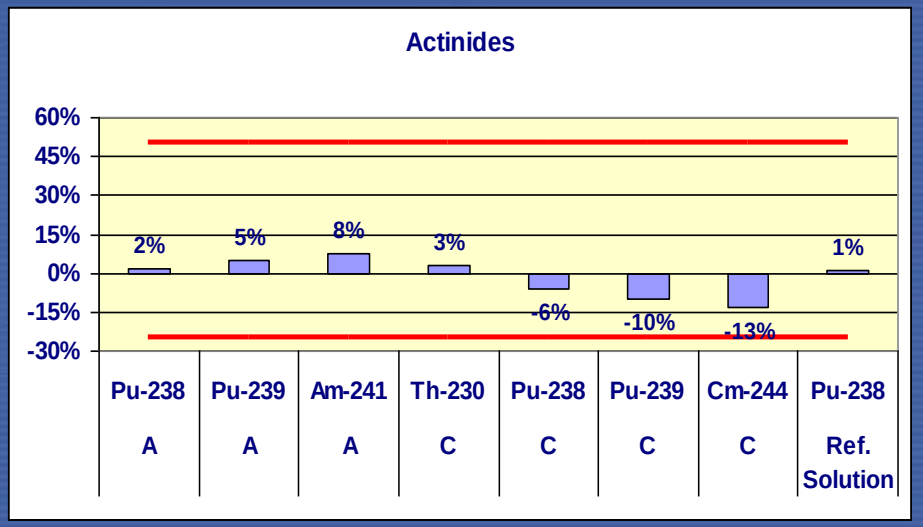
Intercomparison results









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