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UNITED STATES ENVIRONMENTAL PROTECTION AGENCY

Radiochemistry Webinars

High Resolution Gamma Ray Spectrometry Analyses for Normal Operations and Radiological Incident Response

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Meet the Presenter… *Dr. Robert Litman*

Robert Litman, PhD, has been a researcher and practitioner of nuclear and radiochemical analysis for the past 44 years. He is well respected in the nuclear power industry as a specialist in radiochemistry, radiochemical instrumentation and plant systems corrosion. He has co-authored two chapters of MARLAP, and is currently one of a team of EMS consultants developing radiological laboratory guidance on radionuclide sample analyses in various matrices, radioactive sample screening, method validation, core radioanalytical laboratory operations, contamination, and rapid radioanalytical methods. He authored the Radionuclides section of the EPRI PWR Primary Water Chemistry Guidelines, and has been a significant contributor to the EPRI Primary-to-Secondary Leak Detection Guidelines. Dr. Litman has worked with the NRC in support of resolving GSI-191 issues (chemical effects following a loss of coolant accident) at current nuclear power plants and reviewed designs for addressing that safety issue for new nuclear power plants. His areas of technical expertise are gamma spectroscopy and radiochemical separations. Dr. Litman has been teaching courses in Radiochemistry and related special areas for the past 28 years.

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High Resolution Gamma Ray Spectrometry Analyses for Normal Operations and Radiological Incident Response

Robert Litman, PhD

EMS

National Analytical Management Program (NAMP)

TRAINING AND EDUCATION SUBCOMMITTEE

A Collaborative Effort

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Objective

- This webinar presents the major aspects of a new document for normal and emergency response operations:

  "High Resolution Gamma-Ray Spectrometry Analyses for Normal Operation and Radiological Incident Response"

- The objective of this webinar is to present the information provided in the guide and demonstrate the importance of software and radioactive decay laws when performing gamma-ray analysis

Is There a Need?

The incentive to develop this guide came from two significant observations:

- Most laboratory staffs have not had significant experience dealing with high activity concentrations in samples from a nuclear or radiological event
- An observation that many practitioners principally rely upon the software analysis of the gamma spectrum (even though some reported results are improbable)

Document Objectives

1. Describe the basic theoretical principles of gamma-ray spectrometry
2. Show how the interactions of gamma rays with the HPGe detector can yield artifacts that cannot be used to quantify radionuclides
3. Explain the radioactive equilibria and demonstrate how to calculate radionuclide concentrations when these equilibria are present
4. Provide examples of problems that can be encountered when analyzing specific matrices
5. Provide descriptions of the different software functions and how they are used in analyzing the gamma ray spectrum
6. Provide examples of analyses that were incorrectly performed by software based on preselected functions that were inappropriate for the type of sample analyzed, and how these problems can be avoided
7. Identify the different types of detection equations and how they differ in their determination of detection

Introductory Material in the Guide

- Modes of radioactive decay
- Review of the interactions of gamma rays with matter (in particular, with the detector)
- Identification of anomalous photopeaks
- ***Radioactive decay and parent-progeny relationships***
- Potential threat radionuclides from an IND, RDD, or another radiological event

Review Material

- Important concepts in gamma-ray analysis from different references
- Identification of potential threat radionuclides
- Establishing specific libraries
  - Different libraries for different samples/events
- Pictorial representation of gamma-ray interactions

Diagrams and Figures Unique to this Guide

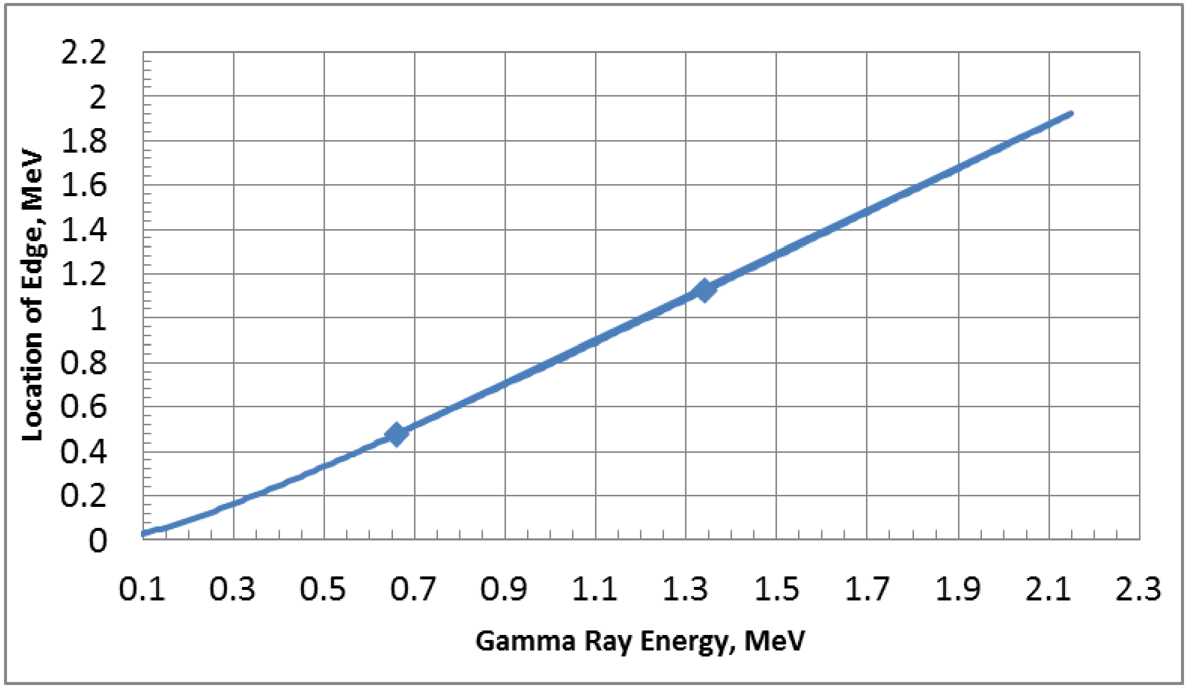
- The next few slides show examples of unique diagrams and figures that identify several different issues encountered in gamma-ray analysis

- The first one deals with the location of a Compton edge

Compton Edge Location

- The first equation identifies the minimum gamma ray energy from a Compton interaction

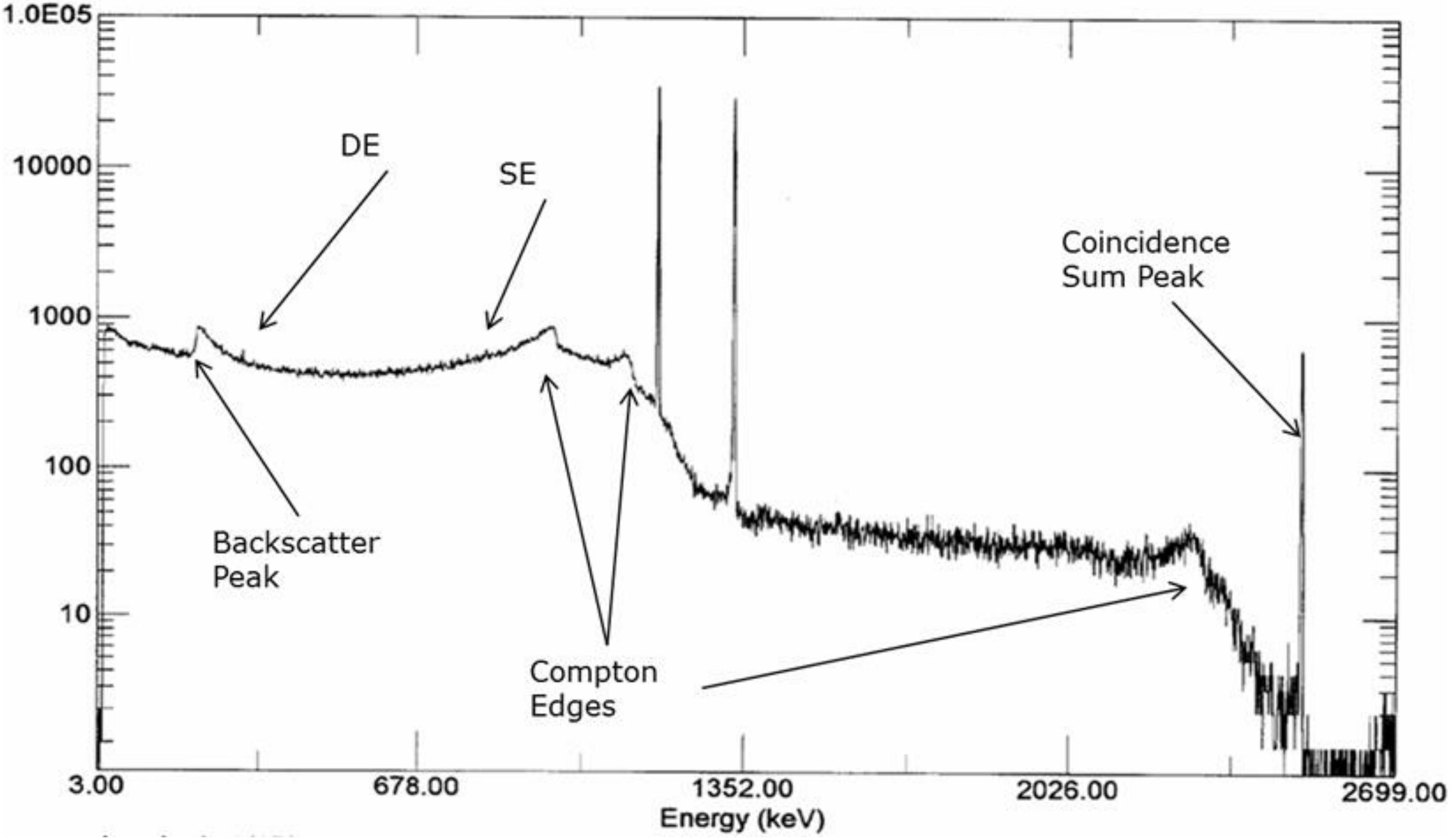
$$E_{\gamma}^{\min} = \frac{0.511 \times E_{\gamma}}{(2 \times E_{\gamma} + 0.511)}$$



- The next equation identifies the location of the Compton edge, $E_{e^-}^{\max}$

$$E_{e^-}^{\max} = E_{\gamma} - E_{\gamma}^{\min}$$

Co-60 Spectrum Showing Compton Edges



Importance of the Compton Edge

- All gamma rays have a Compton edge and distribution
- Creates a change in the gamma background that can hide low-intensity gamma rays
- Can cause broadening of gamma rays, yielding less accurate results (i.e., more uncertainty)

Decay During Counting (DDC) Correction

- A software feature that may be selected to correct for decay during counting
  - Important for long count times when radionuclides undergo “significant” decay during the count

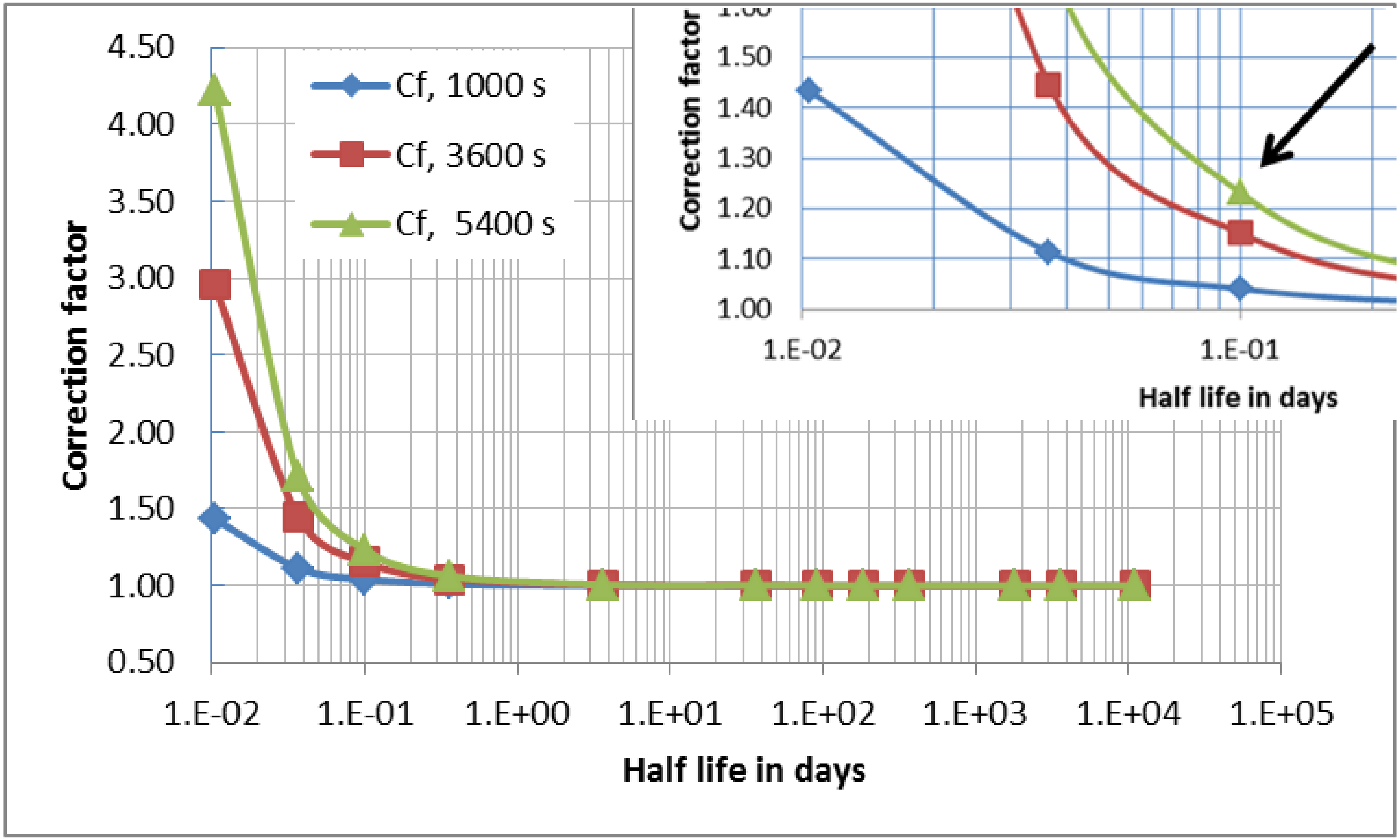
$$C_f = \frac{\lambda t_c}{(1 - e^{-\lambda t_c})}$$

Where:

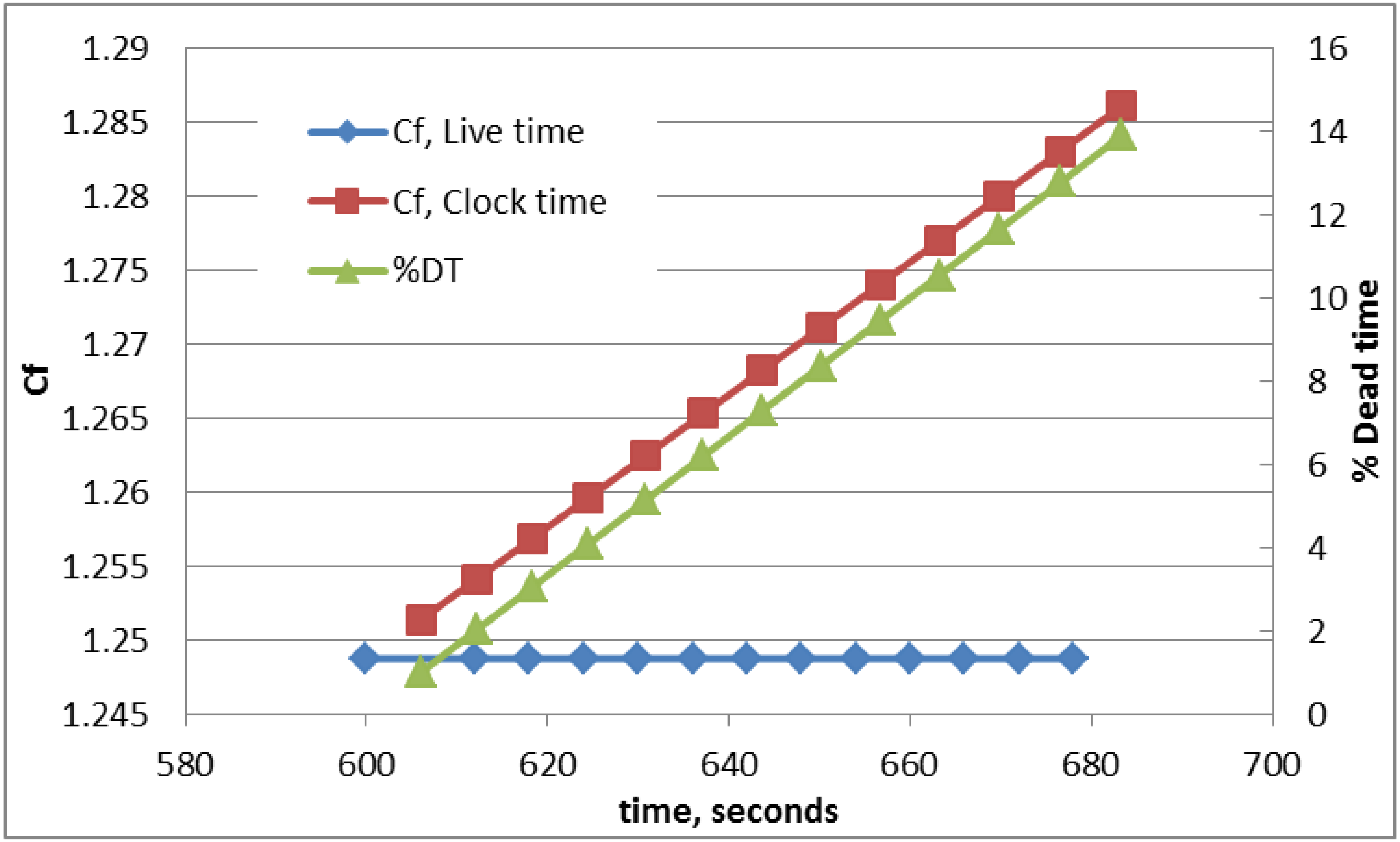
C_f is the correction factor (DDC, a dimensionless quantity)
λ is the decay constant for a particular radionuclide (s^{-1})
t_c is the live time of the analysis (s)

- What is "significant”?

Correction Factor for Decay during DDC; Zero Dead Time



DDC: Non-Zero Dead Time

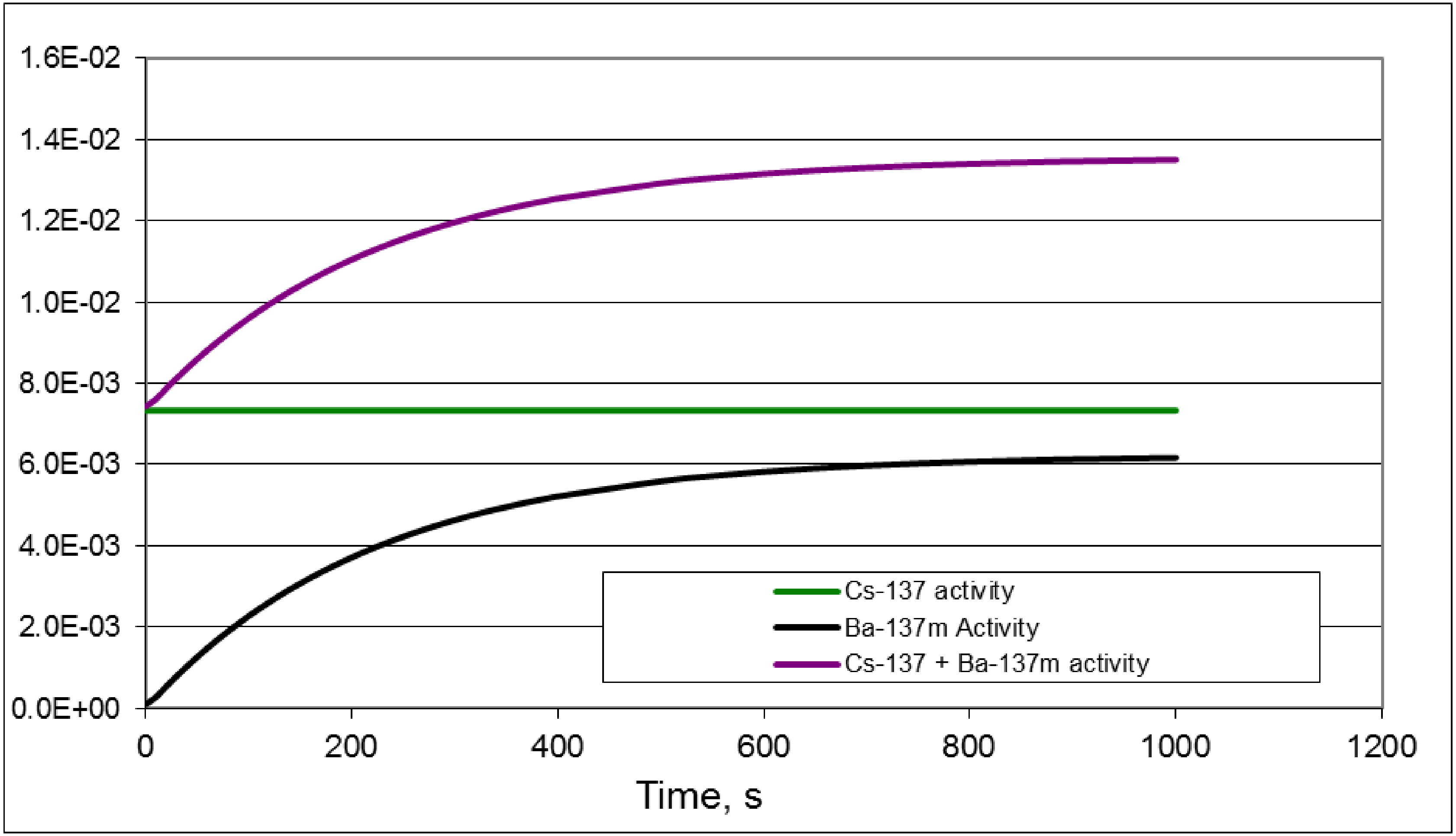


Correction Factor for DDC (assumed half-life of 900 s, live time is 600 s)

Radioactive Equilibria

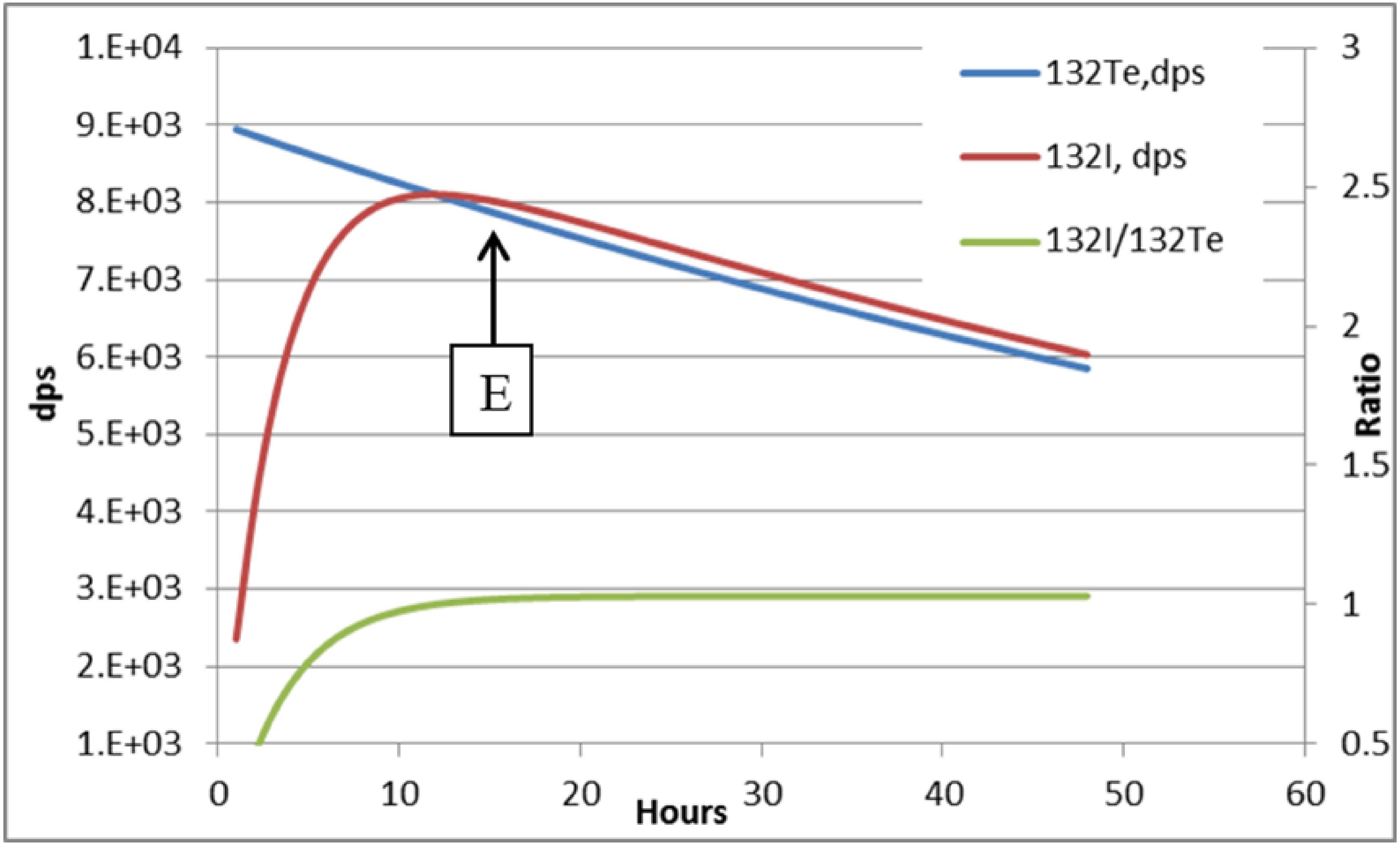
- There are three types of Radioactive Equilibria
- The next slides provide examples of some that may occur during a radiological event
- In each case, you may see an unexpected "feature"
- In cases of true equilibrium, the activity curves for parent and progeny will be parallel at some point

Secular Equilibrium

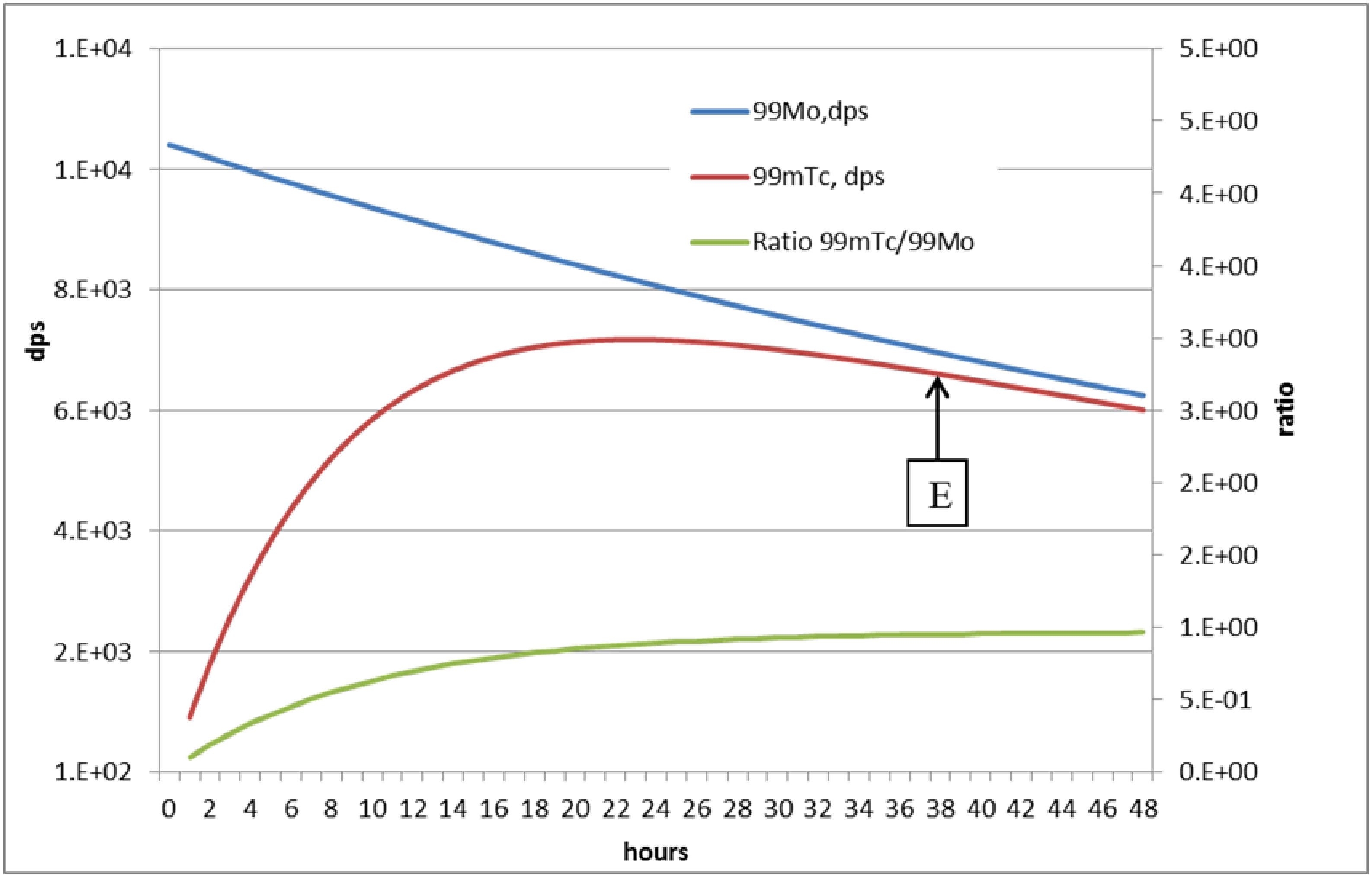


Note: The ^{137m}Ba activity takes into account the branching from ^{137}Cs to ^{137m}Ba, and the internal conversion for the 662 keV gamma ray of ^{137m}Ba

Transient Equilibrium

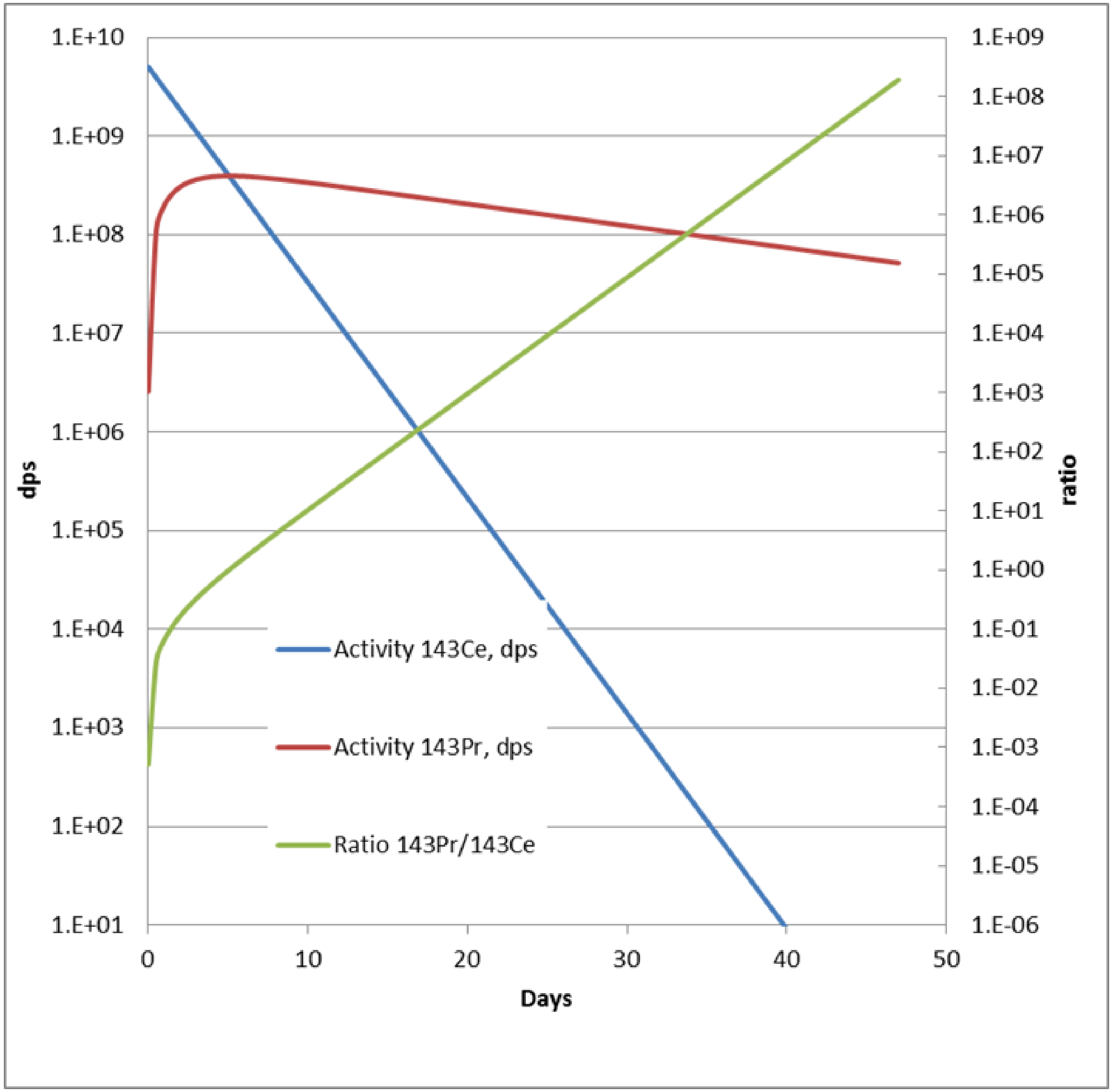


Transient Equilibrium (II)



Note: The ^{99m}Tc activity takes into account the branching from ^{99}Mo to ^{99m}Tc

No Equilibrium



Equilibrium Pairs and Time to Equilibrium

$$T_{\max activity} = \frac{(ln\lambda_p - ln\lambda_{pr})}{(\lambda_p - \lambda_{pr})}$$

Radionuclide Pair	**λ Parent (Days^{-1})**	**λ Progeny (Days^{-1})**	**Time to Peak Progeny Activity [3] (Days ^{1})**	**Type of Equilibrium**	**Decay Correction (post equilibrium)**	**Activity Ratio Progeny/Parent Post Equilibrium[1]**
Fission Products						
^{95}Zr/^{95}Nb	1.08×10^{-2}	1.98×10^{-2}	67.3	Transient	λ Parent + Equation	2.2
^{99}Mo/^{99m}Tc**[2]**	0.252	$2.77\text{x}10^{0}$	0.952	Transient	λ Parent	0.96
^{140}Ba/^{140}La	5.44×10^{-2}	4.13×10^{-1}	5.7	Transient	λ Parent	1.15
^{106}Ru/^{106}Rh	$1.87\text{x}10^{-3}$	$2.00\text{x}10^{+3}$	$2.8\text{x}10^{-3}$	Secular	λ Parent	1
^{132}Te/^{132}I	2.17×10^{-1}	$7.30\text{x}10^{0}$	0.5	Transient	λ Parent	1.03
^{131}I/^{131m}Xe	8.64×10^{-2}	5.82×10^{-2}	14	No	λ Progeny + Equation	N/A
^{137}Cs/^{137}Ba	$6.31\text{x}10^{-5}$	$3.91\text{x}10^{+2}$	$6.9\text{x}10^{-3}$	Secular	λ Parent	1
^{147}Nd/^{147}Pm	6.31×10^{-2}	7.23×10^{-4}	71.6	No	λ Progeny + Equation	N/A
^{143}Ce/^{143}Pr	5.03×10^{-1}	5.11×10^{-2}	5.1	No	λ Progeny	N/A
Naturally Occurring Radionuclides						
^{238}U/^{234}Th	$4.25\text{x}10^{-13}$	$2.88\text{x}10^{-2}$	155	Secular	λ Parent	1
^{228}Ra/^{228}Ac	$3.29\text{x}10^{-4}$	$2.58\text{x}10^{0}$	1.9	Secular	λ Parent	1
^{228}Ra/(^{228}Ac)/^{228}Th	$3.29\text{x}10^{-4}$	$9.92\text{x}10^{-4}$	4.6	Transient	λ Parent	1.4
^{226}Ra/^{222}Rn	$1.19\text{x}10^{-6}$	$1.81\text{x}10^{-1}$	27	Secular	λ Parent	1
^{214}Pb/^{214}Bi	$3.70\text{x}10^{+1}$	$5.01\text{x}10^{+1}$	0.15	Transient	λ Parent	3.8
^{212}Pb/^{212}Bi	$1.56\text{x}10^{0}$	$1.66\text{x}10^{1}$	0.25	Transient	λ Parent	1.1
^{210}Pb/^{210}Bi	$8.51\text{x}10^{-5}$	$1.38\text{x}10^{-1}$	53.5	Secular	λ Parent	1.0

Threat or Accident Radionuclides

- Many commercial uses for radionuclides:
  - Radioisotope thermoelectric generator (RTG)
    - ^{90}Sr, ^{238}Pu, ^{237}Np
  - Medical isotopes
    - ^{131}I, ^{103}Pd, ^{192}Ir
  - Well-logging devices
    - ^{60}Co, ^{124}Sb, ^{140}La
  - Radiography
    - ^{60}Co, ^{137}Cs, ^{75}Se, ^{241}Am

Alpha Emitters

Alpha Emitters							
Radionuclide	**Gamma Energy, keV**	**Gamma-ray Abundance [6]**	**Half-Life**	**Radionuclide**	**Gamma Energy, keV**	**Gamma-ray Abundance [6]**	**Half-Life**
Am-241	**59.5**	**0.359**	432.7 y	Ra-226	**186.2**	**0.0364**	$1.599x10^{3}$ y
Cm-242	**44.1**	**0.000035**	162.8 d	Th-228	**84.4**	**0.0122**	1.91 y
Cm-243	**277.6, 228.2**	**0.14, 0.106**	29.1 y	Th-230	**67.7**	**0.0038**	$7.56x10^{4}$ y
Cm-244	**42.8**	**0.0026**	18.1 y	Th-232	**63.8**	**0.000263**	$1.4x10^{10}$ y
Np-237	**86.5**	**0.124**	$2.14x10^{6}$ y	U-234	**53.2**	**0.000123**	$2.46x10^{5}$ y
Pu-238	**43.5**	**0.000392**	87.7 y	U-235	**185.7**	**0.570**	$7.04x10^{8}$ y
Pu-239	**51.6**	**0.000272**	$2.41x10^{4}$ y	U-238	**49.6**	**0.00064**	$4.47x10^{9}$ y
Pu-240	**45.2**	**0.000447**	$6.56x10^{3}$ y	U-Nat	**185.7 (^{235}U)**	**0.570**	$4.47x10^{9}$ y

Beta Emitters

Beta Emitters

Radionuclide	Gamma Energy, keV	Gamma-ray Abundance	Half-Life	Radionuclide	Gamma Energy, keV	Gamma-ray Abundance [6]	Half-Life
Ac-227/ Th-227	236	0.129	21.7 y/18.7 d	Ba-140/La-140	537/1596	0.2439, 0.9540	12.8 d/1.68 d
Bi-212	727	0.0667	60.6 min	Mo-99/Tc-99m	740, 141	0.1226, 0.89	2.75 d/6.01 h
Bi-214	609	0.455	19.9 min	Pd-103	39.7	0.00683	17.0 d
Co-57	122, 136	0.856, 0.1068	271.8 d	Pb-210	46.5	0.0425	22.3 y
Co-60	1173, 1332	0.9985, 0.9998	5.271 y	Pb-212	239	0.436	10.6 h
Cs-137/ Ba-137m	662	0.899	30.0 y	Pb-214	352	0.356	27 min
I-125	35.5	0.0668	59.4 d	Pu-241/Am-241	59[5]	0.359	14.3 y
I-129	39.6	0.0751	1.57x107 y	Ra-228/ Ac-228	911 (Ac)	0.258	5.76 y/6.15 h
I-131	364	0.815	8.01 d	Ru-106/ Rh-106	511.9, 622	0.204, 0.0993	1.02 y / 299 s
Ir-192	317	0.8286	73.8 d	Se-75	265, 136	0.589, 0.585	119.8 d

Software Functions

- What they do
- Why we should select or not select some of them
- What we need to know about them

Software Functions

- Peak Search Sensitivity
- Peak Cutoff Uncertainty
- Energy Comparison
- Half-life Period Exceeded
- Key Line Designation
- Abundance or Fraction Limit
- Weighted Mean Average
- Compton and Peak Background Subtract
- Decay Correction
- Detection Equations

Examples of some of these are provided on the next few slides

Energy Comparison

- Library lists energy values for the gamma rays
- Software identifies a peak, determines energy, then compares the "found" to the "listed" energies
- The delta may be in terms of keV or multiples of the FWHM
- User selects the allowable delta for a positive ID (recommendation)
  - High activity samples – small delta
  - Low activity samples – large delta

Half-Life Period Exceeded

- Time period between the time of sampling and the start time of analysis exceeds a predetermined number of half-lives (based on the specific radionuclide half-life)

$$HL_{ratio} = \frac{\Delta T}{t_{1/2}}$$

- Example: a sample is analyzed after one week. The radionuclide half-life = 2 hours. The radionuclide would have gone through:

  1 week x (168 hour/week)/(2 hours/half-life) = 89 half-lives

- Its original activity would have been *decreased* by a factor of 2^{89}, or $6.2\text{x}10^{26}$
- Very low probability that radionuclide will be present: identity rejected
- Generally speaking, most preset functions will default to a value of about 8 to 12 for half-lives passed, representing a decrease in activity of 256 to 4,096

Beware of Parent-progeny relationships!

Key Line

- Usually at least one gamma ray has a significant abundance and is interference free – typically designated the “key line”

- If the key line for a radionuclide is *not* found, software will not identify the radionuclide as being present

Note: The key line and abundance (or fraction) limit are tests of radionuclide presence that are redundant and should not be used together

Key Line? – Oops!

Radionuclide	Half Life	Energy, keV	Abundance, %	Alternate Key Line?	Abundance, %
^{110m}Ag	249 days	657	95.6	884	75.0
$^{97}Nb^{*}$	1.2 hours	657	98.2	---	---
^{134}I	52.5 minutes	847	96	884	65.1
^{56}Mn	2.57 hours	847	98.85	1810	26.9

*The precursor of ^{97}Nb is ^{97}Zr ($t_{1/2}$ = 16.7 hours) gamma ray at 743 keV is 97 %

Abundance Limit

- Each gamma ray emitted by a radionuclide has an abundance
- This is the frequency that a gamma ray is emitted per decay
- The abundance limit entered by the user is compared to the ratio of the abundance of the gamma rays found for a particular radionuclide to the sum of all gamma rays listed in the library for that radionuclide
- If the calculated ratio does not exceed the user-entered preset abundance limit, gamma rays are moved to an unidentified or rejected lines report

Weighted Mean Average

- Two types of found gamma rays
  - Weighted by abundances
  - Weighted by uncertainty

- In both cases
  - Review the range of values for the gamma rays used for analysis

Equations for Weighted Mean Value

- Uncertainty Based

$$C_{avg} = \frac{\sum_{i=1}^{n}(C_i/\sigma_{C_i^2})}{\sum_{i=1}^{N} \frac{1}{\sigma_{C_i^2}}}$$

- Abundance Based

$$C_{avg} = \frac{\sum_{i=1}^{n} C_i \times I_{C_i}}{\sum_{i=1}^{n} I_{C_i}}$$

Detection Equations

- Many different terms are used for “detection”
  - MDA, MDC, LLD, L_c
- Each term has a different equation
- Each equation can have different degrees of confidence associated with it
- Some software packages have as many as 8 different options
- The next slide shows an example of four different calculations

Detection Equation Calculations

			Activity at Beginning of Count Interval, pCi/L			
	Bg, cps	**Fractional Efficiency**	**Lc**	**MDA**	**LLD**	**MDC**
14400 sec	0.01	0.01	6.2	10.5	12.3	12.9
(4 Hours)	0.05	0.01	13.8	23.5	27.6	28.1
	0.1	0.01	19.5	33.3	39.0	39.5
	1	0.11	5.6	9.6	11.2	11.2
	10	0.21	9.3	15.8	18.6	18.6
	100	0.31	19.9	33.9	39.8	39.7
3600 sec	0.01	0.01	12.3	21.0	24.7	27.0
(1 Hour)	0.05	0.01	27.6	47.0	55.2	57.5
	0.1	0.01	39.0	66.5	78.0	80.3
	1	0.11	11.2	19.1	22.4	22.6
	10	0.21	18.6	31.7	37.1	37.2
	100	0.31	39.8	67.9	79.6	79.5

Data Verification and Validation

- Who performs each function?
- Is it the same for the vendor and the client?
- What does each function entail?
- Is the process different for emergency response versus normal operations?

Checklist (Partial) for Gamma Spectrometry Data Verification

Sample Matrix_______	Sample Date/time_________ __	Sample ID_________
Geometry__________ Library	Detector_______	Count date/time_____
• Are all of the above inputs identified correctly on the report?	__	__
• Are all identified radionuclides included based on half-life?	__	__
• Have appropriate members of decay chains been identified?	__	__
• Are proper half-lives used for radionuclides in parent-progeny relationships?	__	__
• Are all the FWHM used to calculate activity concentrations at the approximate value for the gamma-ray energy?	__	__
• Are all identified radionuclides expected or probable?		
• …	__	__
• …	__	__
• Any “N” requires a description and resolution	__	__

Checklist (Partial) for Gamma Spectrometry Data Validation

Project: ___________	Client: ______________
Project QA Document:_________	Analytical Laboratory Used: _____________
Are the following satisfactory:	
Sample COC?	Y___ N___
Sample Preservation?	Y___ N___
Sample holding time?	Y___ N___
For any "N" provide explanation: ____ ____	
All verification report inputs satisfactory ?	Y___ N___
If "N" provide explanation: ____ ____	
All QC analyses Satisfactory?	Y___ N___
For any "N" provide explanation: ____ ____	
Have all software preset functions been optimized based on the client requirements and sample history to identify the radionuclides present?	Y___ N___
Client Requirements Met?	Y___ N___
Sensitivity Factor:	
Half-life ratio:	
Energy Difference:	
Abundance factor:	
Key line:	
Weighted Mean:	
Have all unknown gamma-ray lines with a cps uncertainty less than 50 % been identified?___	Y___ N___
List all unidentified gamma rays: ____ ____	

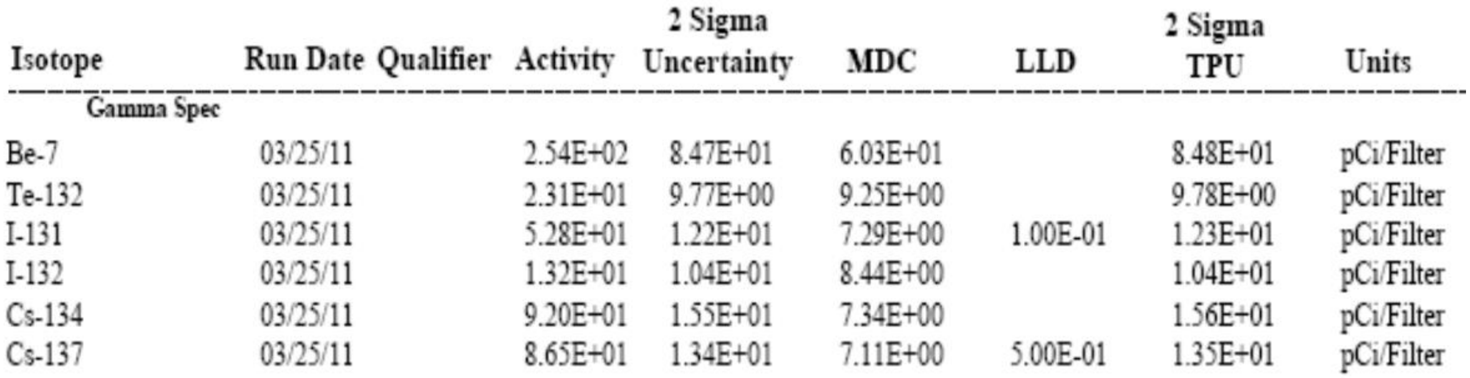
Examples – Attachment II

- Examples provided are with the gracious consent of the originating organization (notations are anonymous)
- Each organization has made adjustments to its methods, based on feedback
- Just a few of the examples are shown here

Results from the Irradiated Uranium PT

Laboratory		Activity Concentration, pCi/L		Measured Ratio/Theoretical (progeny/parent)	Activity Concentration, pCi/L		Measured Ratio/Theoretical (progeny/parent)
		^{140}La	^{140}Ba		^{99m}Tc	^{99}Mo	
1	Activity[1] at the start of the counting interval	1980	1879	1.05/ 1.13	---	---	---
	Corrected for decay back to time of collection	207,000	3457	59/1.00	$1.0\text{x}10^{26}$	$5.03\text{x}10^{7}$	$2\text{x}10^{22}$/0.96
2	Activity[1] at the start of the counting interval	---	---	---	---	---	---
	Corrected for decay back to time of collection	$2.49\text{x}10^{6}$	$8.97\text{x}10^{3}$	$2.78\text{x}10^{2}$/1.00	$4.17\text{x}10^{19}$	$2.59\text{x}10^{3}$	$1.6\text{x}10^{16}$/0.96

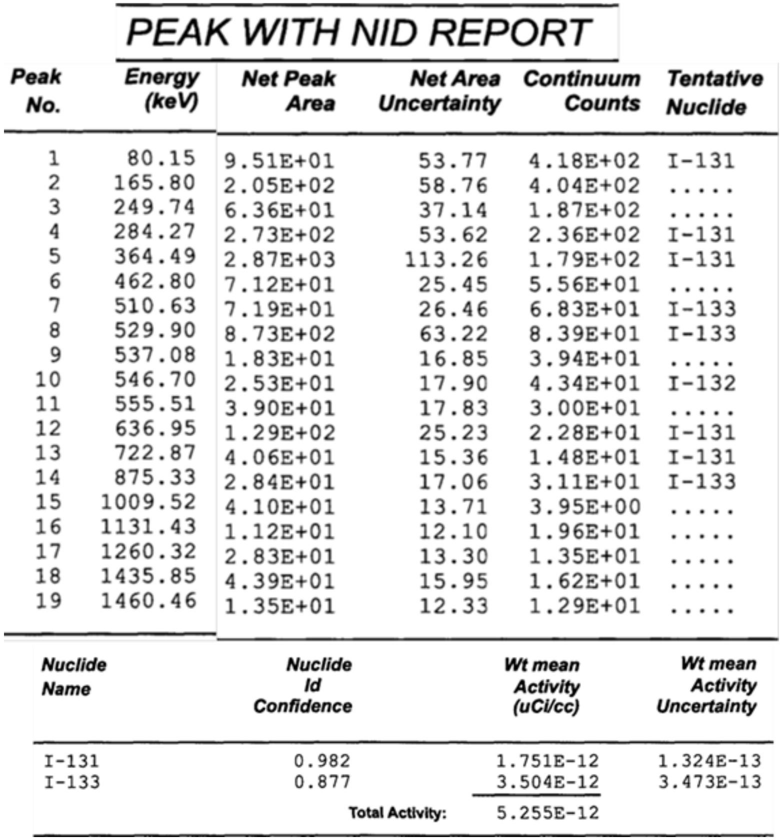
Incorrect Preservation of Samples and Its Effect on Analysis - Dry Deposition Samples Following Fukushima Event



Isotope	Run Date	Qualifier	Activity	2 Sigma Uncertainty	MDC	LLD	2 Sigma TPU	Units
Gamma Spec								
Be-7	03/25/11		2.54E+02	8.47E+01	6.03E+01		8.48E+01	pCi/Filter
Te-132	03/25/11		2.31E+01	9.77E+00	9.25E+00		9.78E+00	pCi/Filter
I-131	03/25/11		5.28E+01	1.22E+01	7.29E+00	1.00E-01	1.23E+01	pCi/Filter
I-132	03/25/11		1.32E+01	1.04E+01	8.44E+00		1.04E+01	pCi/Filter
Cs-134	03/25/11		9.20E+01	1.55E+01	7.34E+00		1.56E+01	pCi/Filter
Cs-137	03/25/11		8.65E+01	1.34E+01	7.11E+00	5.00E-01	1.35E+01	pCi/Filter

- Dry deposition samples taken on a “sticky” pad
- Shipped in a Zip-Loc™ bag
- Time between the end of sampling and start of analysis = ~3 days
- The ^{132}Te/^{132}I should be in ratio of 1/1.03

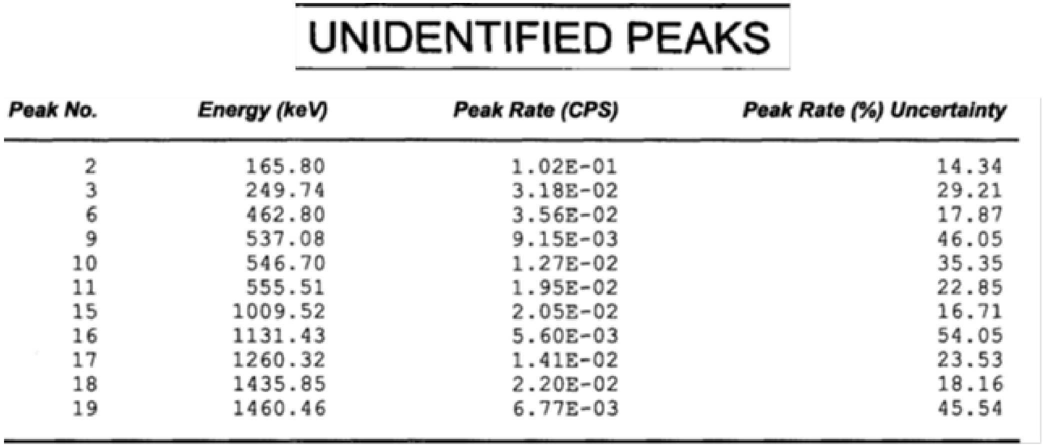
Unidentified Gamma Rays



PEAK WITH NID REPORT

Peak No.	Energy (keV)	Net Peak Area	Net Area Uncertainty	Continuum Counts	Tentative Nuclide
1	80.15	9.51E+01	53.77	4.18E+02	I-131
2	165.80	2.05E+02	58.76	4.04E+02	.....
3	249.74	6.36E+01	37.14	1.87E+02	.....
4	284.27	2.73E+02	53.62	2.36E+02	I-131
5	364.49	2.87E+03	113.26	1.79E+02	I-131
6	462.80	7.12E+01	25.45	5.56E+01	.....
7	510.63	7.19E+01	26.46	6.83E+01	I-133
8	529.90	8.73E+02	63.22	8.39E+01	I-133
9	537.08	1.83E+01	16.85	3.94E+01	.....
10	546.70	2.53E+01	17.90	4.34E+01	I-132
11	555.51	3.90E+01	17.83	3.00E+01	.....
12	636.95	1.29E+02	25.23	2.28E+01	I-131
13	722.87	4.06E+01	15.36	1.48E+01	I-131
14	875.33	2.84E+01	17.06	3.11E+01	I-133
15	1009.52	4.10E+01	13.71	3.95E+00	.....
16	1131.43	1.12E+01	12.10	1.96E+01	.....
17	1260.32	2.83E+01	13.30	1.35E+01	.....
18	1435.85	4.39E+01	15.95	1.62E+01	.....
19	1460.46	1.35E+01	12.33	1.29E+01	.....

Nuclide Name	Nuclide Id Confidence	Wt mean Activity (uCi/cc)	Wt mean Activity Uncertainty
I-131	0.982	1.751E-12	1.324E-13
I-133	0.877	3.504E-12	3.473E-13
	Total Activity:	5.255E-12	



UNIDENTIFIED PEAKS

Peak No.	Energy (keV)	Peak Rate (CPS)	Peak Rate (%) Uncertainty
2	165.80	1.02E-01	14.34
3	249.74	3.18E-02	29.21
6	462.80	3.56E-02	17.87
9	537.08	9.15E-03	46.05
10	546.70	1.27E-02	35.35
11	555.51	1.95E-02	22.85
15	1009.52	2.05E-02	16.71
16	1131.43	5.60E-03	54.05
17	1260.32	1.41E-02	23.53
18	1435.85	2.20E-02	18.16
19	1460.46	6.77E-03	45.54

- One week collection time, decay corrected to mid-point of week
- Unidentified peaks belong to ^{135}I (6.6 h), ^{138}Cs (32.2 min) and ^{139}Ba (83 min) were not in selected library
- Half-life ratio function was set to 12
- Delay between counting and sampling midpoint was 3.6 days

Summary

- Knowing the basics of gamma ray interactions and detection is important
- There is a lot that goes on behind the scenes in gamma spectrometry
  - There are many software functions to select: know which ones you need to use and what they do!
- Sample preservation is important in gamma spectrometry too!
- There is no Silver Bullet
  - Knowledge and vigilance are the keys to accurate reporting

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Questions and Comments are Welcomed!

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