SAND2007-1491C

Soil Sampling and Analysis

(Using High Resolution Gamma Spectroscopy)

Presented at the 3rd RMCC Workshop Held at The Food and Environment Monitoring Centre

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Ministry of Regional Municipalities, Environment & Water Resources (MRMEWR), Muscat, Oman

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Presentation Overview

Part 1: Soil Sampling

- Determining Need for Sampling
- Identification of Sampling Locations
- Sample Size
- Sampling Frequency
- Sample Transfer, tracking, and chain of custody
- Sample Screening; prevent cross contamination
- Sample Preparation; homogeneous mix

Presentation Overview

Part 2: Sample Analysis Using Gamma Spectroscopy

- Components of a Basic Gamma Spectroscopy Setup
- Impact of Detector Size on Spectrum Features
- Impact of Surrounding Material/Shielding on Spectrum Features
- Combined Impacts of Detector Size and Surrounding Material/Shielding on Spectrum

Presentation Overview

Part 2: Sample Analysis Using Gamma Spectroscopy

- Impacts of Back Scatter of High Energy Gammas and Source Strength on Spectrum
- Detector Energy Calibration
- Detector Efficiency Calibration
- Analysis Software (routines)

Soil Sampling

Determining Need for Sampling

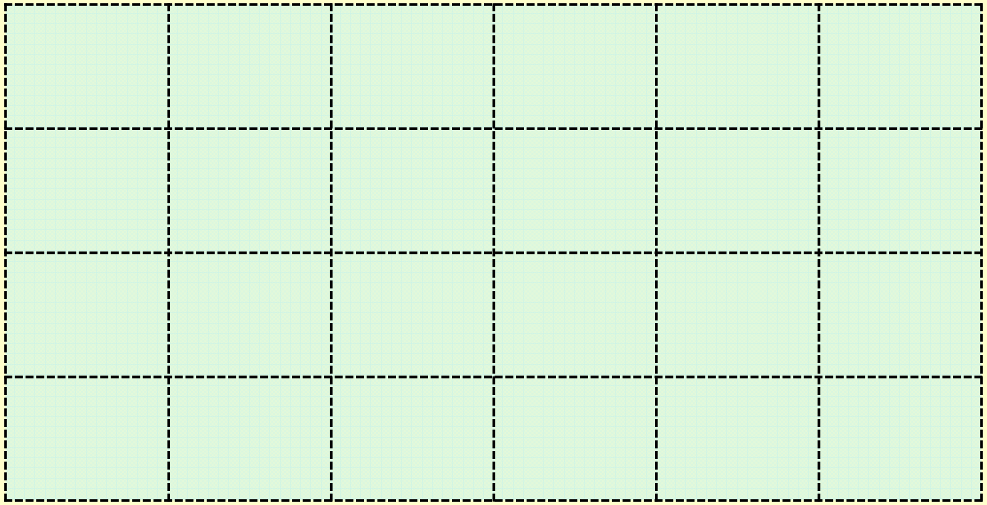
- Historical data or Knowledge Indicate Potential for Presence of Radiological Contamination in Soil
  - Mining Activity Sites, past or current
  - Radioactive Material Production or Processing, past or current
  - Nuclear Power Generation Site, past or current
  - Disposal or Storage Site, past or current
  - Regulatory Requirement
  - Others
- Establishing Background, Baseline
- Others

Soil Sampling

Identification of Sampling Locations

1. Perform a Survey

- Start with Setting up a Grid



Soil Sampling

Identification of Sampling Locations

1. Perform a Survey

- Surveying Technique: Depends on Area Size, Terrain, and Available Equipment

Soil Sampling

Identification of Sampling Locations

1. Perform a Survey
   - Manual (walk-over or Stationary) Survey
   - o Hand-held portable Gamma detectors, NaI(Tl) Gross Gamma
   - o Hand-held portable Gamma detectors, NaI(Tl) Single Channel Gamma
   - o Hand-held portable Gamma detectors, NaI(Tl) MCA
   - o Any of above using high resolution GE detector

Soil Sampling

Identification of Sampling Locations

1. Perform a Survey
   - Manual (Stationary) Survey

Soil Sampling

Identification of Sampling Locations

1. Perform a Survey

- Mobile (All-Terrain Vehicle - ATV)

Soil Sampling

Identification of Sampling Locations

1. Perform a Survey
   - Arial Survey

Note: Results could be season dependent: Soil Moisture Content

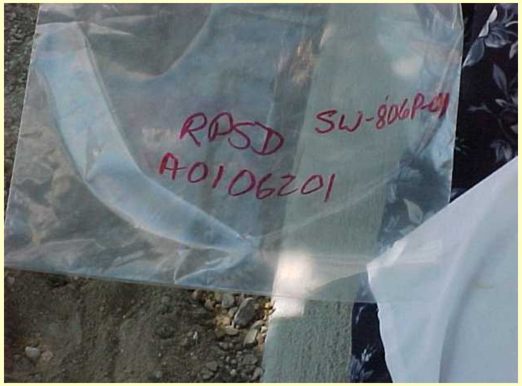
2. Random Samples Analyzed in Lab and Correlation with Survey Results is Developed

Soil Sampling

Sample Size

Sample Size Depends on:

- Analysis Technique
- Required Accuracy
- Distribution of Contaminant in Soil
- Can be Dictated by Regulation
- Others

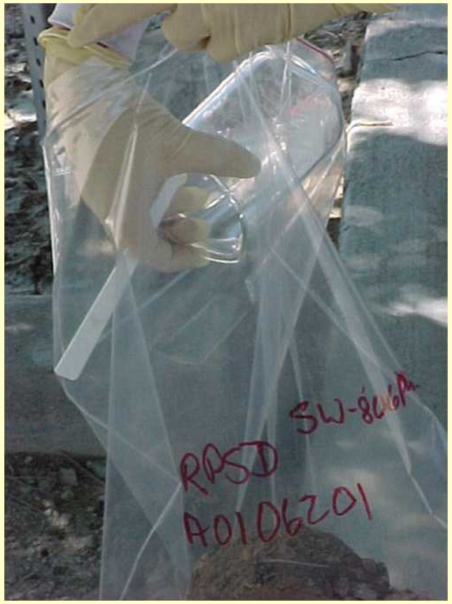


Soil Sampling

Frequency

Frequency: Depends, among other factors, on:

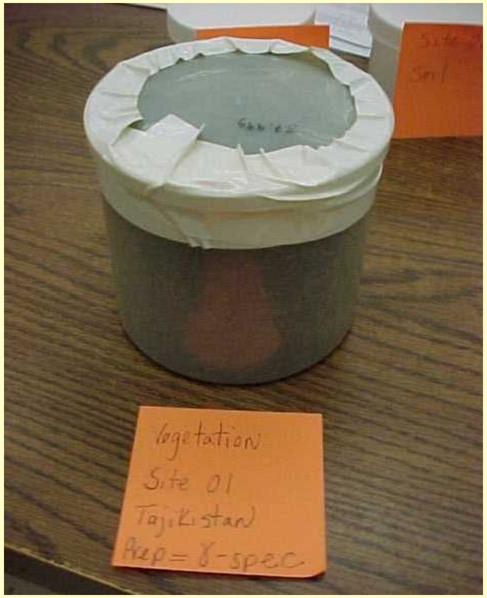
- Expected variation in concentration
- Impact of seasonal changes on concentration
- Regulatory Requirements
- Others



Soil Sampling

Sample transfer, tracking, and chain of custody

- Pack samples properly to avoid cross contamination and to protect the integrity of sample



Soil Sampling

Sample transfer, tracking, and chain of custody

- Log movement of sample to ensure it can be located during the different stages of transfer

Soil Sampling

Sample transfer, tracking, and chain of custody

Sample Transfer, tracking, and chain of custody

- Sample shipper should provide for acknowledgement of transfer by the deliverers and recipients

- Sample collection information (location, time and date, ID number, etc) shall accompany the sample during the transfer

Soil Sampling

Sample Screening, Prevent Cross Contamination

Screen sample; prevent cross contamination

- Samples shall be screened for activity level so that proper equipment are used for sample preparation, grinding and mixing

- Samples shall be sorted according to their activity levels so that the highly contaminated samples are no introduced into low level sample prep equipment and counting systems

Soil Sampling

Sample Preparation; Homogeneous Mix

Sample Preparation

- Homogeneous mixing of the sample prior to counting/analysis is a crucial requirement because:
  - *<u>Almost all</u>* calibration soil standards used for calibration of counting system are homogeneous;

Soil Sampling

Sample Preparation; Homogeneous Mix

Sample Preparation

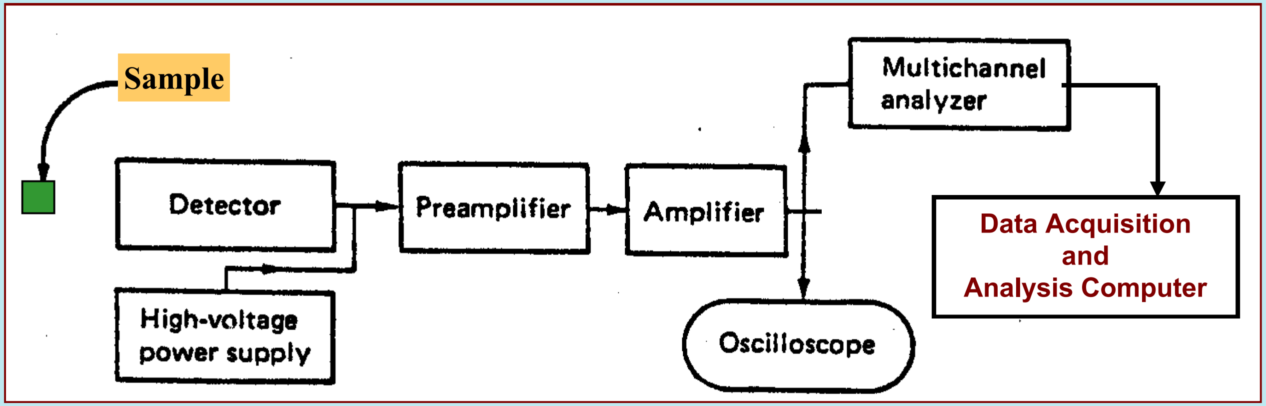
- Homogeneous mixing of the sample prior to counting/analysis is a crucial requirement because:
  - Calibration of counting systems for non-homogeneous sample are very expensive and results will have much larger errors.

Part 2

Sample Analysis Using Gamma Spectroscopy

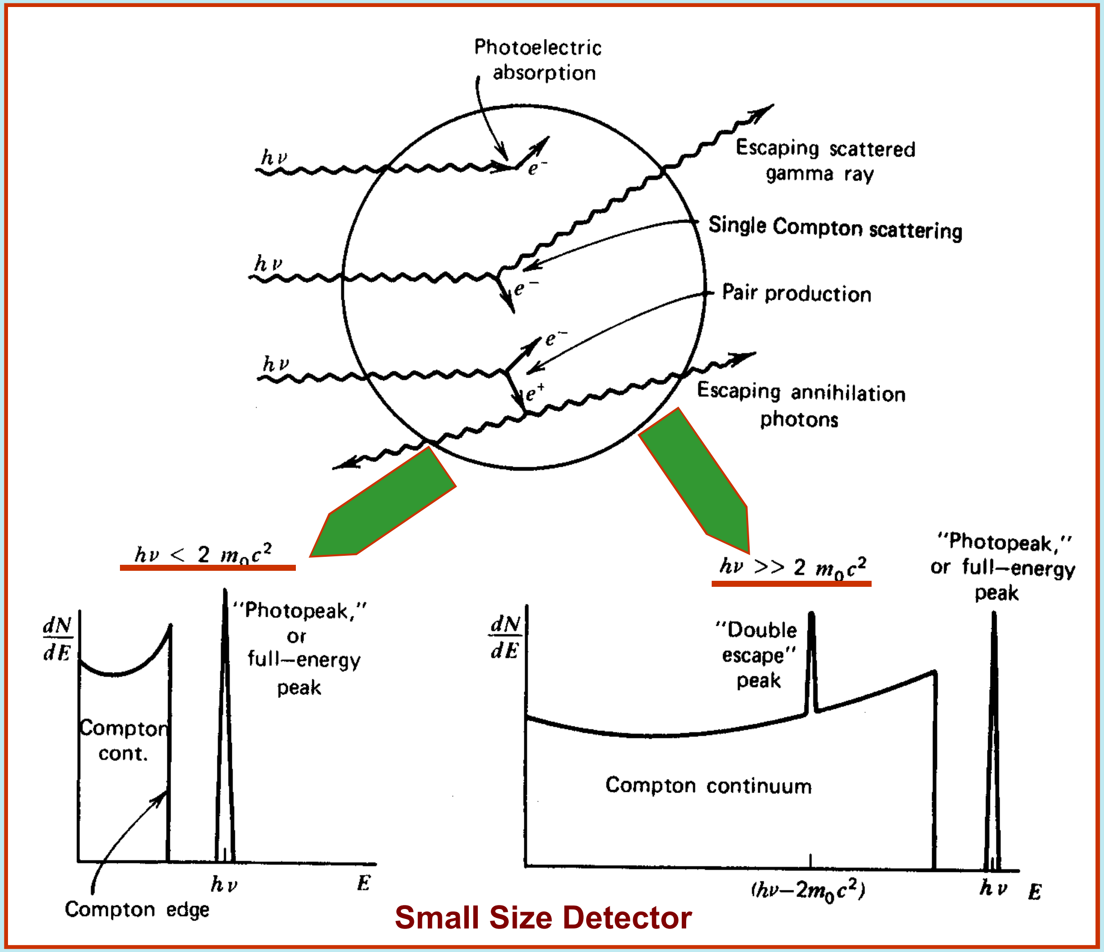
Sample Analysis Using Gamma Spectroscopy

Components of a Basic Gamma Spectroscopy Setup



Sample Analysis Using Gamma Spectroscopy

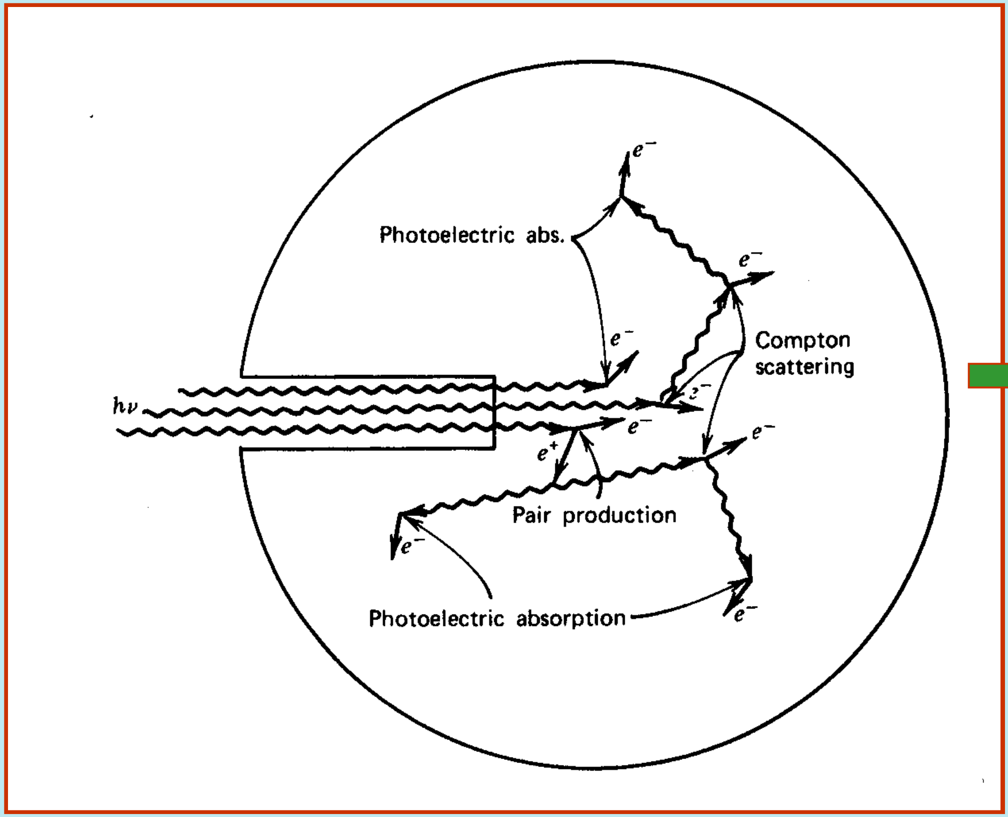
Impact of Detector Size on Spectrum Features

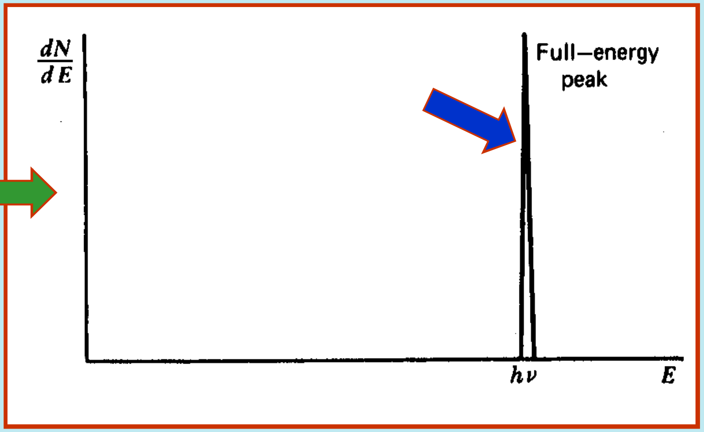


Small Size Detector

Sample Analysis Using Gamma Spectroscopy

Impact of Detector Size on Spectrum Features

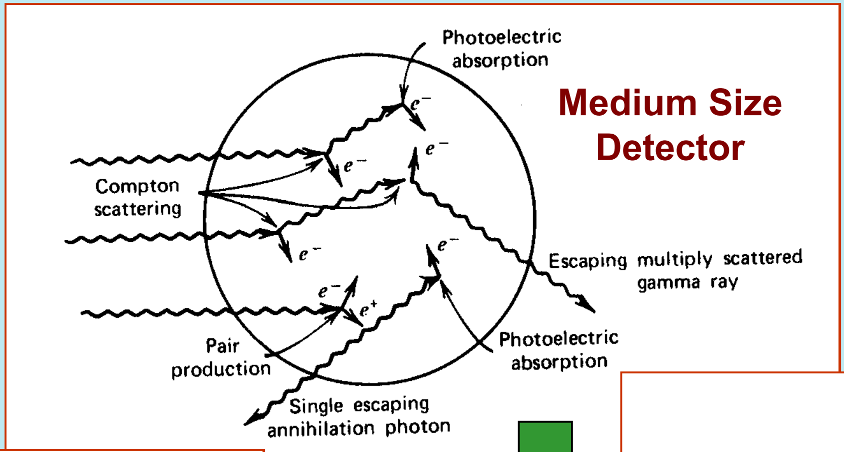




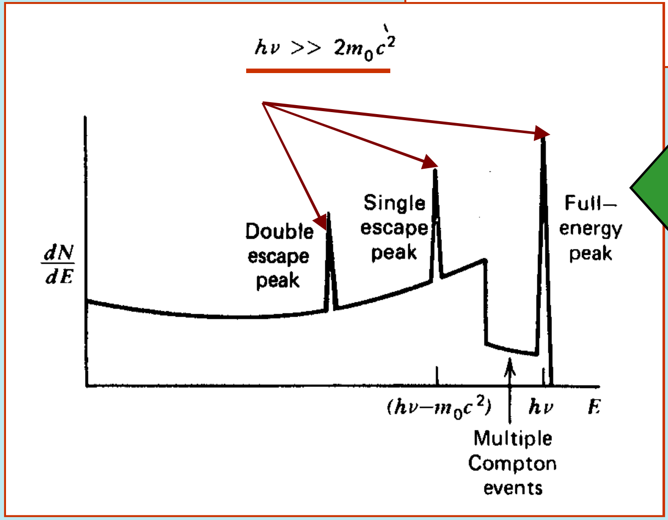
Large Size Detector

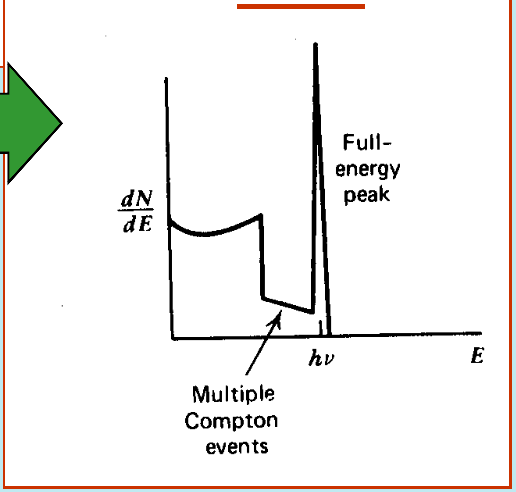
Sample Analysis Using Gamma Spectroscopy

Impact of Detector Size on Spectrum Features



Medium Size Detector



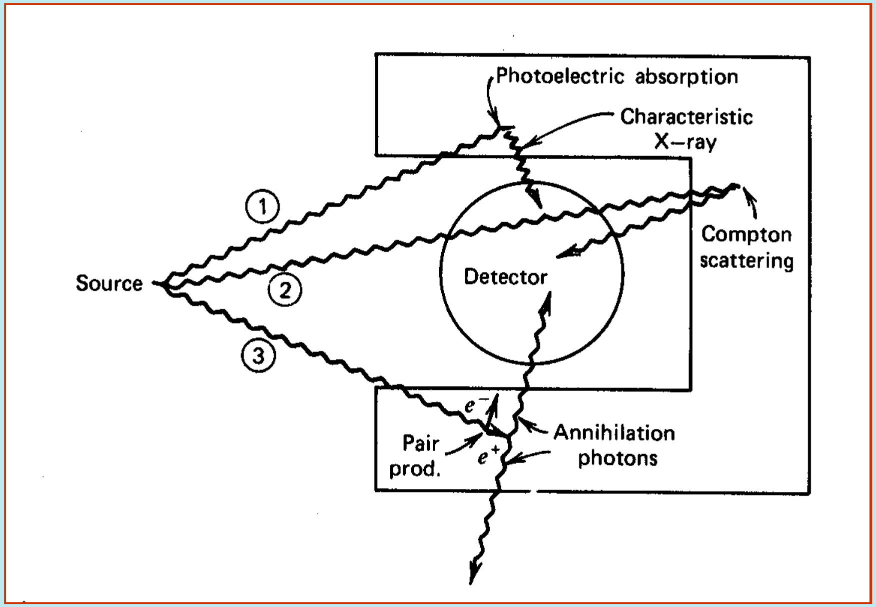


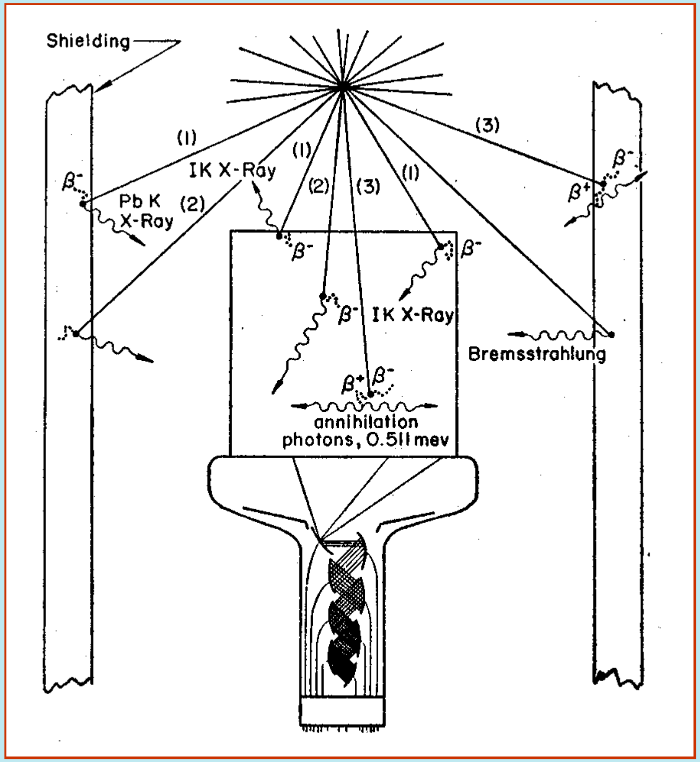
Sample Analysis Using Gamma Spectroscopy

Impact of Surrounding Material/Shielding on spectrum features

Primary Interactions That Affect the Response Curves of NaI(Tl) Scintillation or Ge Detector.

(1) Photoelectric Interaction
(2) Compton Interaction
(3) Pair Production

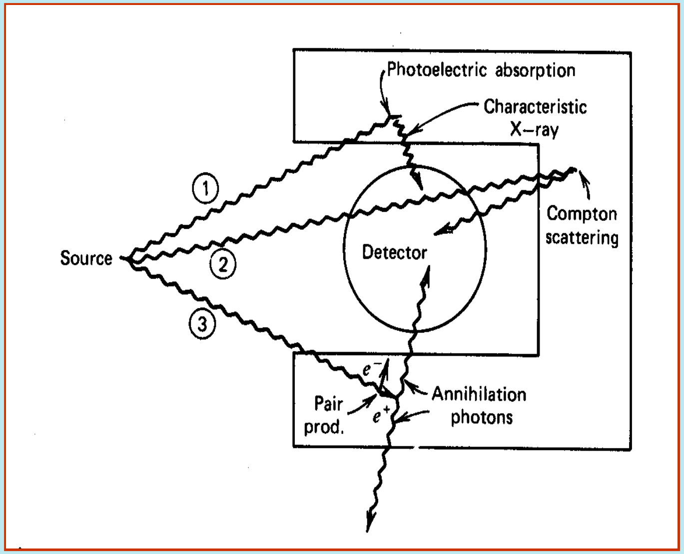


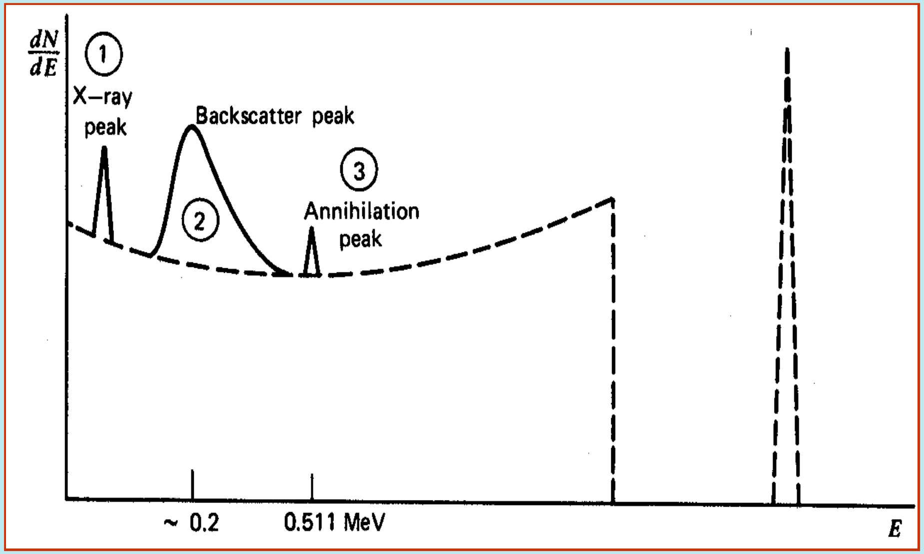


Sample Analysis Using Gamma Spectroscopy

Impact of Surrounding Material/Shielding on spectrum features

An Example of Impact on a NaI(Tl) Spectrum. The Impact on a High Resolution Detector Spectrum Will Be Similar.



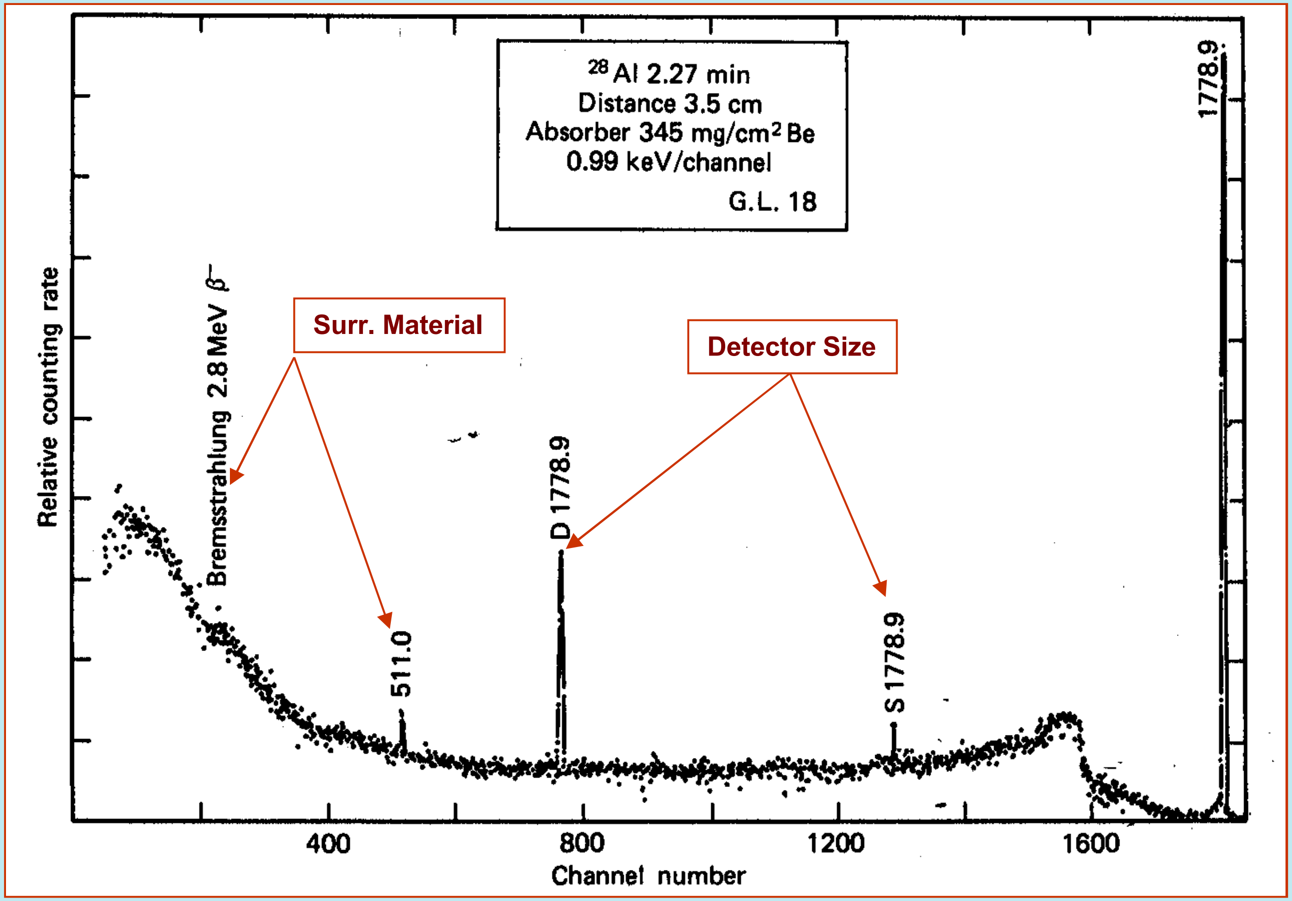


(1) Photoelectric Interaction; (2) Compton Interaction; (3) Pair Production

In addition to the expected spectrum (dashed line), effects of surrounding appear as peaks in the spectrum.

Sample Analysis Using Gamma Spectroscopy

Combined Impacts of Detector Size and Surrounding Material/Shielding



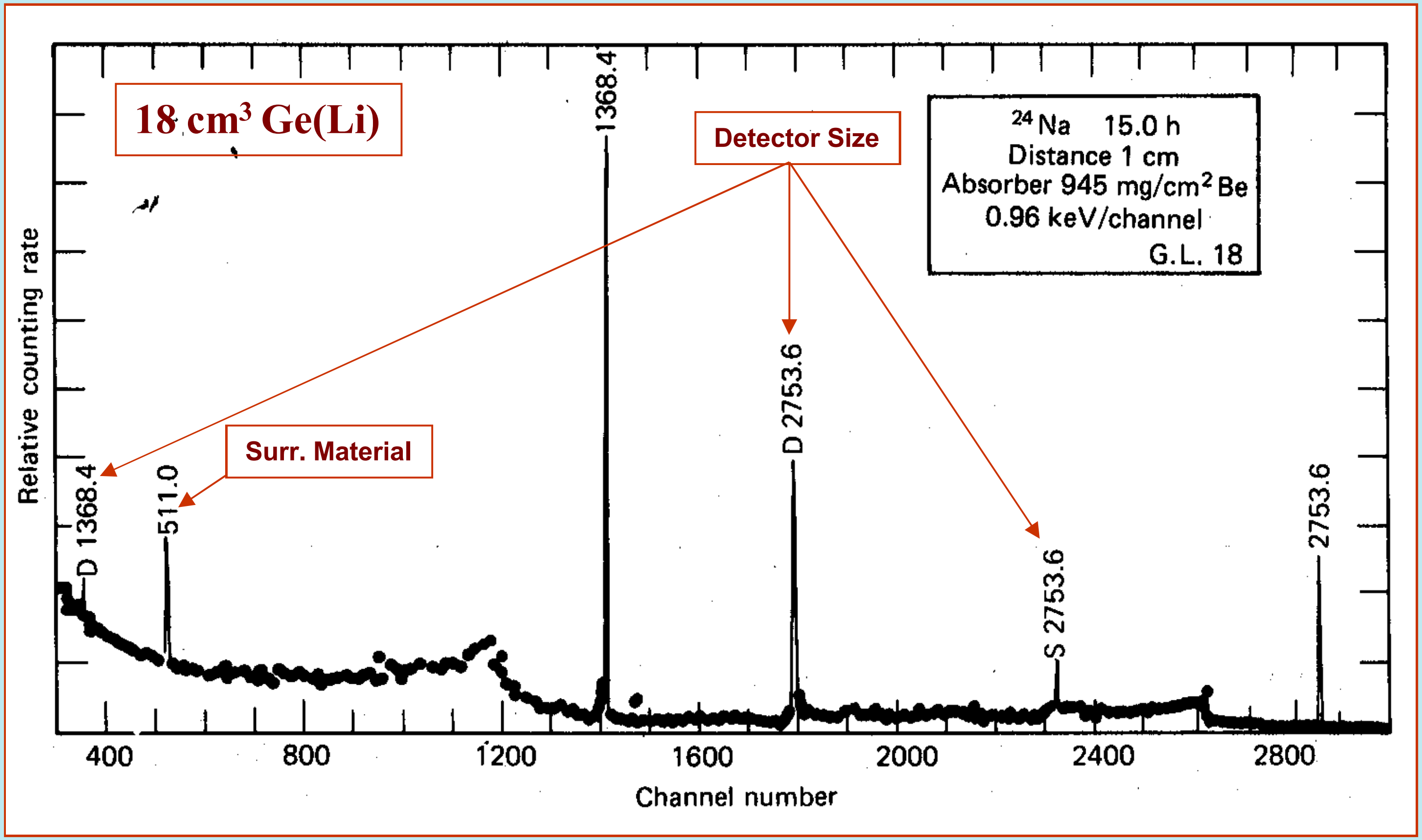
Surr. Material

Detector Size

Example 1

Sample Analysis Using Gamma Spectroscopy

Combined Impacts of Detector Size and Surrounding Material/Shielding



Example 2

Sample Analysis Using Gamma Spectroscopy

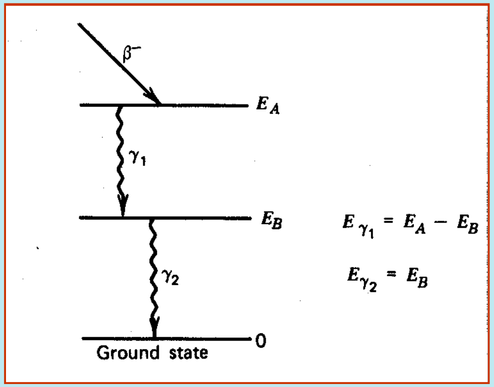
Impacts of Back Scatter of High Energy Gammas and Source Strength on Spectrum

When Gamma Energy >> $m_0c^2/2$, and $\theta = \pi$:
A backscatter peak in the vicinity of 200 – 250 KeV.

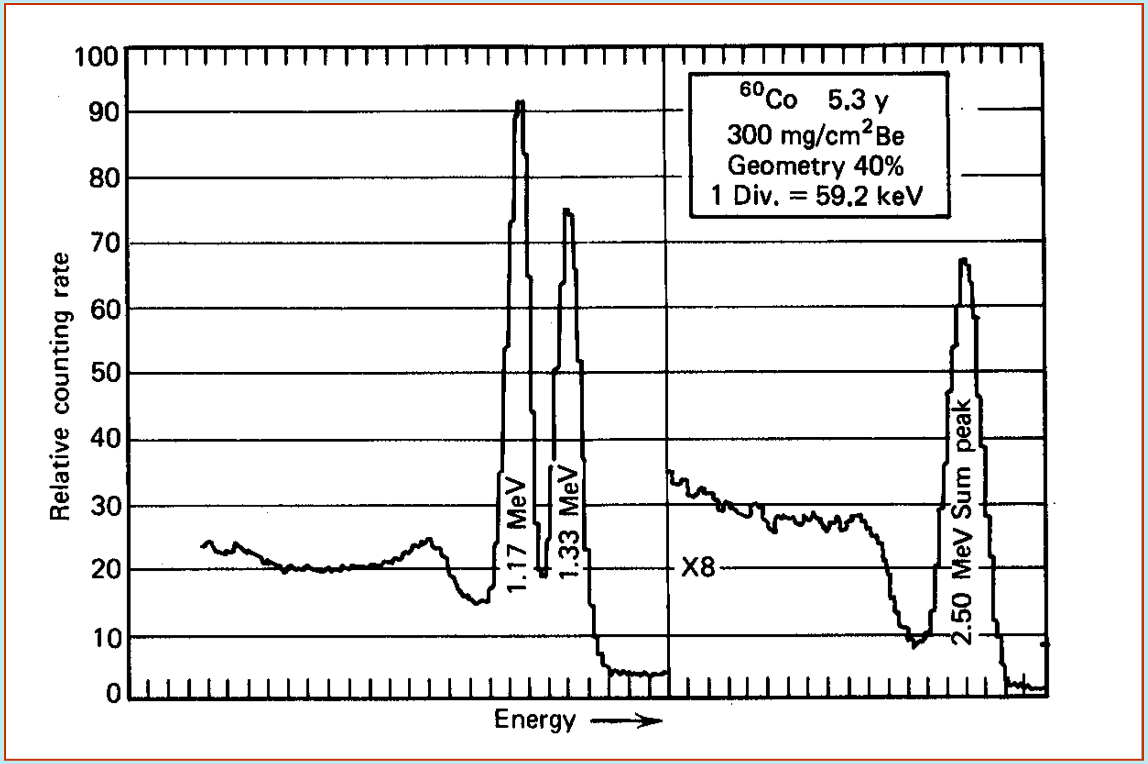
Source Strength/Cascade Gamma Emission => Sum Peak
At Very High count rates, when counting " multiple cascade emitters", there is potential for summation of energies from coincident detection of two or more gamma rays in the detector. This will result in the recording of gamma peaks with energies equivalent to the sum of the energies. In environmental samples of low activity the " multiple cascade emitters" are the main contributors to this effect.

Sample Analysis Using Gamma Spectroscopy

Impacts of High Source Strength on Spectrum – Sum Peak



Simplified nuclear decay scheme that can lead to summation effect in the gamma spectra. The intermediate state (at E_B) must be short lived compared to the time-resolution of the detector.

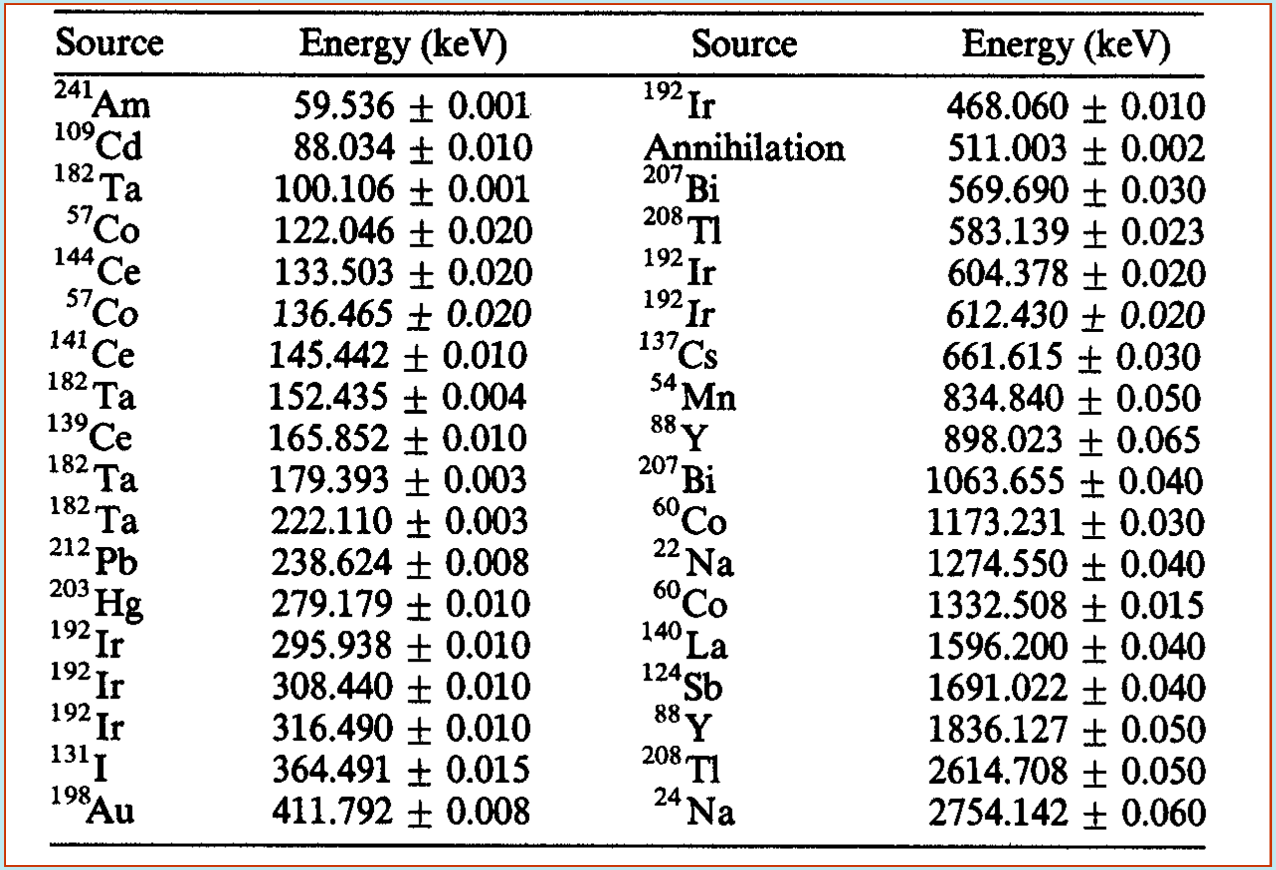


Experimentally measured spectra from ^{60}Co. The sum peak appears at 2500 KeV (sum of 1170 KeV and 1330 KeV gamma rays).

Sample Analysis Using Gamma Spectroscopy

Energy Calibration

Reference Isotopes / Reference Standards for Energy Calibration



Source	Energy (keV)	Source	Energy (keV)
^{241}Am	59.536 ± 0.001	^{192}Ir	468.060 ± 0.010
^{109}Cd	88.034 ± 0.010	Annihilation	511.003 ± 0.002
^{182}Ta	100.106 ± 0.001	^{207}Bi	569.690 ± 0.030
^{57}Co	122.046 ± 0.020	^{208}Tl	583.139 ± 0.023
^{144}Ce	133.503 ± 0.020	^{192}Ir	604.378 ± 0.020
^{57}Co	*136.465 ± 0.020*	^{192}Ir	*612.430 ± 0.020*
^{141}Ce	145.442 ± 0.010	^{137}Cs	661.615 ± 0.030
^{182}Ta	152.435 ± 0.004	^{54}Mn	834.840 ± 0.050
^{139}Ce	165.852 ± 0.010	^{88}Y	898.023 ± 0.065
^{182}Ta	179.393 ± 0.003	^{207}Bi	1063.655 ± 0.040
^{182}Ta	222.110 ± 0.003	^{60}Co	1173.231 ± 0.030
^{212}Pb	238.624 ± 0.008	^{22}Na	1274.550 ± 0.040
^{203}Hg	279.179 ± 0.010	^{60}Co	1332.508 ± 0.015
^{192}Ir	295.938 ± 0.010	^{140}La	1596.200 ± 0.040
^{192}Ir	308.440 ± 0.010	^{124}Sb	1691.022 ± 0.040
^{192}Ir	316.490 ± 0.010	^{88}Y	1836.127 ± 0.050
^{131}I	364.491 ± 0.015	^{208}Tl	2614.708 ± 0.050
^{198}Au	411.792 ± 0.008	^{24}Na	2754.142 ± 0.060

Sample Analysis Using Gamma Spectroscopy

Energy Calibration

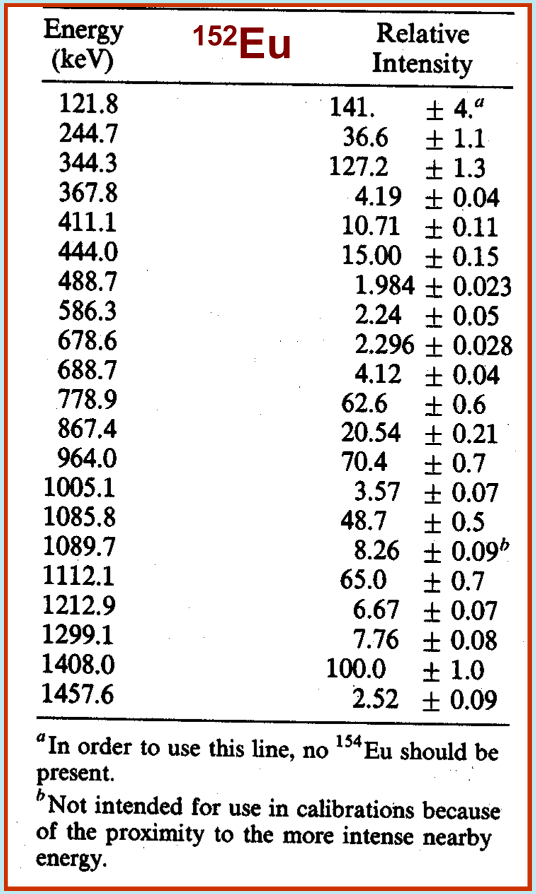
When a mixed standard or a standard with multiple gamma energies is used, a least square fitting is applied to drive the calibration function. This is the approach used by all commercially available gamma spectroscopy software.

Clearly, the "geometry configuration" of calibration standard shall match that of the sample.

Sample Analysis Using Gamma Spectroscopy

Efficiency Calibration

Reference Isotopes / Reference Standards for Efficiency Calibration

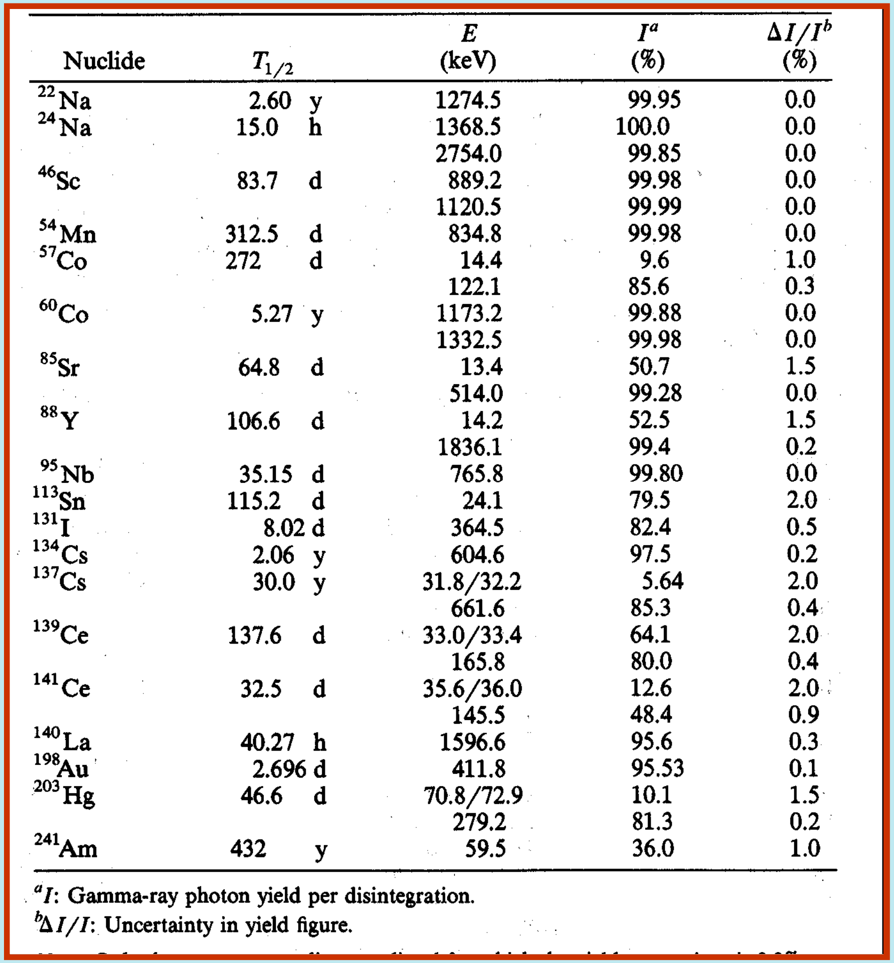


^{152}Eu

Energy (keV)	Relative Intensity
121.8	141. ± 4.[a]
244.7	36.6 ± 1.1
344.3	127.2 ± 1.3
367.8	4.19 ± 0.04
411.1	10.71 ± 0.11
444.0	15.00 ± 0.15
488.7	1.984 ± 0.023
586.3	2.24 ± 0.05
678.6	2.296 ± 0.028
688.7	4.12 ± 0.04
778.9	62.6 ± 0.6
867.4	20.54 ± 0.21
964.0	70.4 ± 0.7
1005.1	3.57 ± 0.07
1085.8	48.7 ± 0.5
1089.7	8.26 ± 0.09[b]
1112.1	65.0 ± 0.7
1212.9	6.67 ± 0.07
1299.1	7.76 ± 0.08
1408.0	100.0 ± 1.0
1457.6	2.52 ± 0.09

[a] In order to use this line, no ^{154}Eu should be present.

[b] Not intended for use in calibrations because of the proximity to the more intense nearby energy.



Nuclide	$T_{1/2}$		E (keV)	I^a (%)	$\Delta I/I^b$ (%)
^{22}Na	2.60	y	1274.5	99.95	0.0
^{24}Na	15.0	h	1368.5	100.0	0.0
			2754.0	99.85	0.0
^{46}Sc	83.7	d	889.2	99.98	0.0
			1120.5	99.99	0.0
^{54}Mn	312.5	d	834.8	99.98	0.0
^{57}Co	272	d	14.4	9.6	1.0
			122.1	85.6	0.3
^{60}Co	5.27	y	1173.2	99.88	0.0
			1332.5	99.98	0.0
^{85}Sr	64.8	d	13.4	50.7	1.5
			514.0	99.28	0.0
^{88}Y	106.6	d	14.2	52.5	1.5
			1836.1	99.4	0.2
^{95}Nb	35.15	d	765.8	99.80	0.0
^{113}Sn	115.2	d	24.1	79.5	2.0
^{131}I	8.02	d	364.5	82.4	0.5
^{134}Cs	2.06	y	604.6	97.5	0.2
^{137}Cs	30.0	y	31.8/32.2	5.64	2.0
			661.6	85.3	0.4
^{139}Ce	137.6	d	33.0/33.4	64.1	2.0
			165.8	80.0	0.4
^{141}Ce	32.5	d	35.6/36.0	12.6	2.0
			145.5	48.4	0.9
^{140}La	40.27	h	1596.6	95.6	0.3
^{198}Au	2.696	d	411.8	95.53	0.1
^{203}Hg	46.6	d	70.8/72.9	10.1	1.5
			279.2	81.3	0.2
^{241}Am	432	y	59.5	36.0	1.0

[a] I: Gamma-ray photon yield per disintegration.

[b] $\Delta I/I$: Uncertainty in yield figure.

Sample Analysis Using Gamma Spectroscopy

Efficiency Calibration

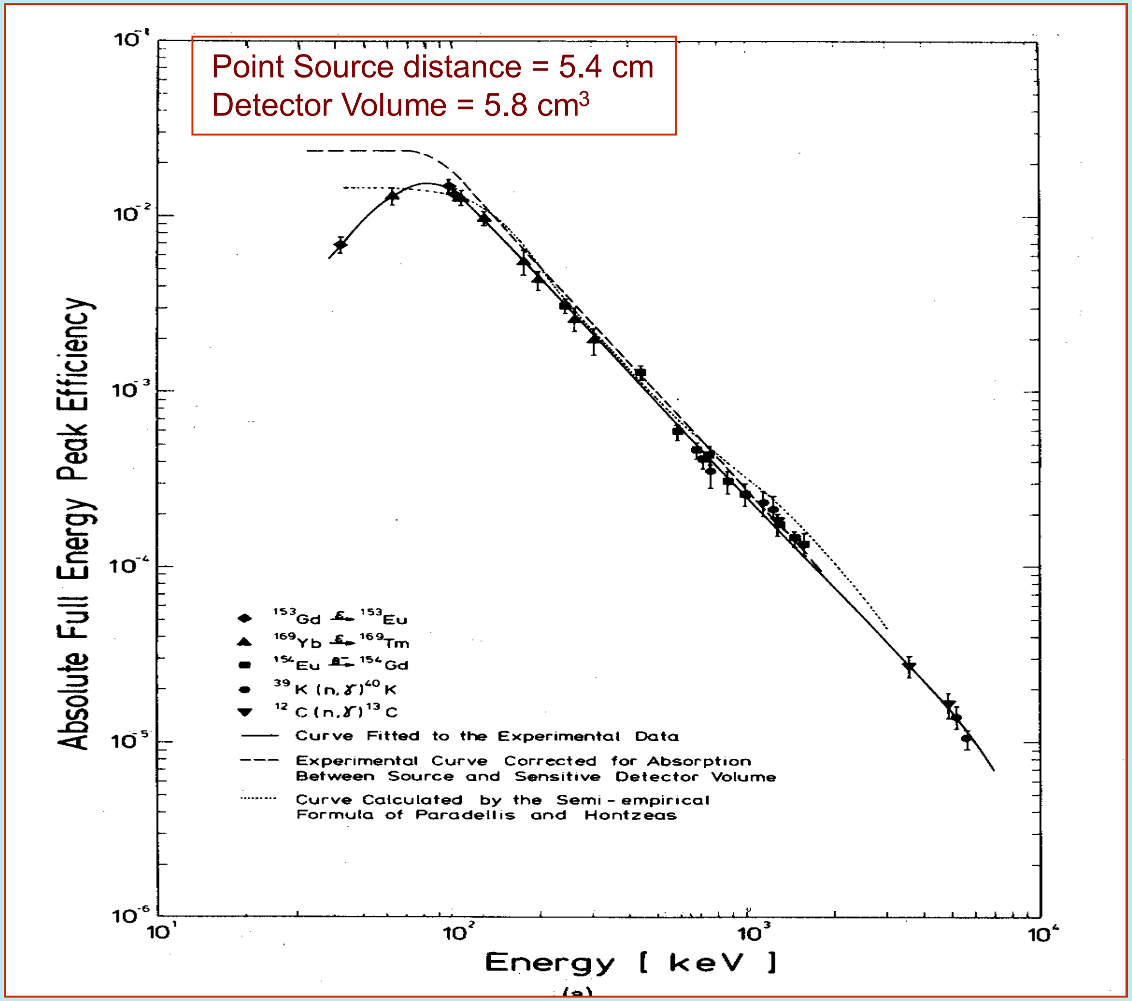
- Efficiency calibration shall be done to match the geometry and media / matrix of samples to be analyzed.

- Typically, there are several efficiency calibration files for each sample type (air filters, soil samples, water samples, etc) to match the sample size and geometry.

- The efficiency calibration shall include correction for self-absorption and any other attenuations due to the sample holders and surrounding material.

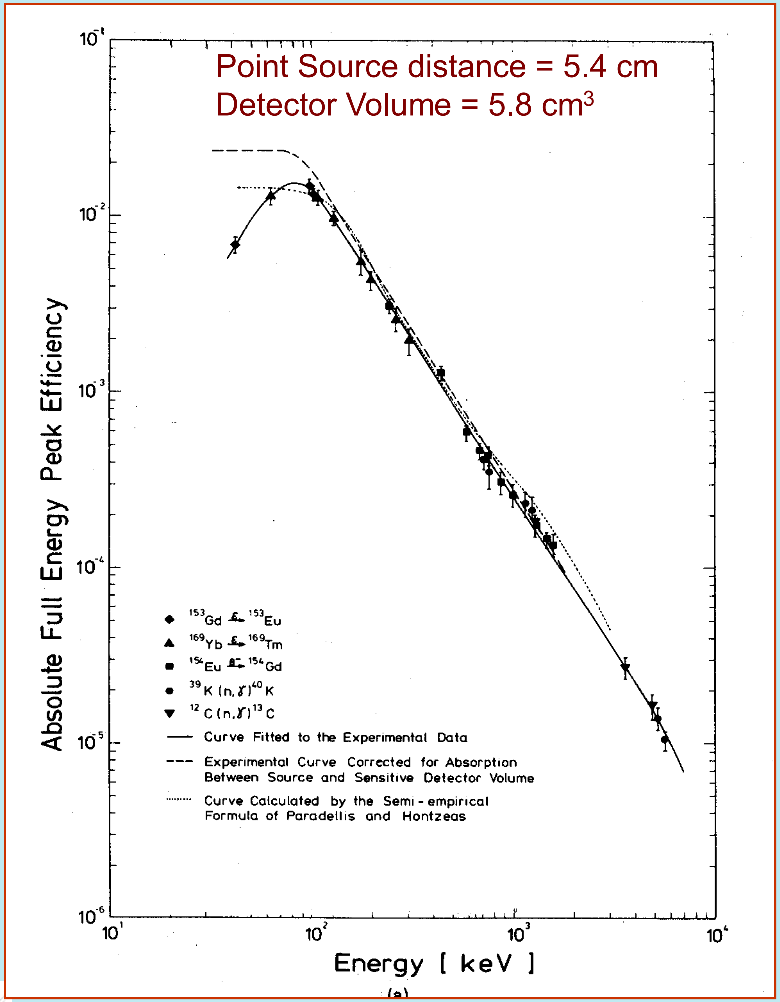
Sample Analysis Using Gamma Spectroscopy

Efficiency Ge(Li)


Point Source distance = 5.4 cm
Detector Volume = 5.8 cm^3

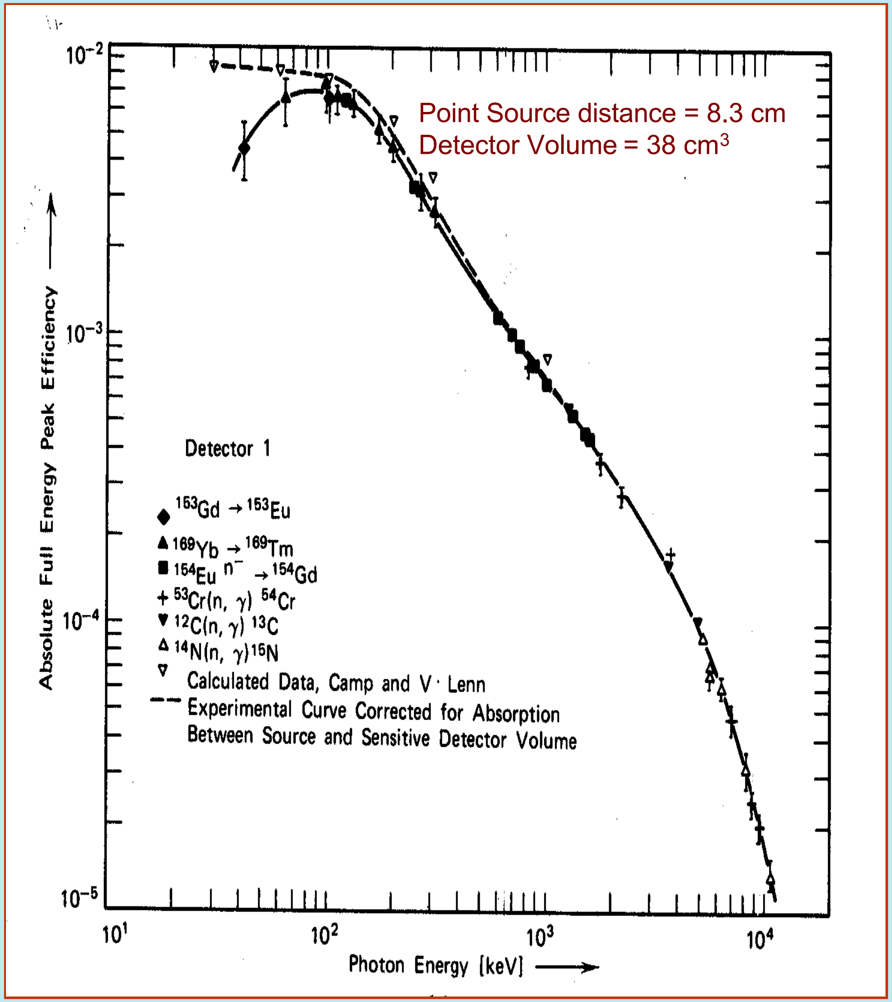
Sample Analysis Using Gamma Spectroscopy

Efficiency Ge(Li) vs Distance



Point Source distance = 5.4 cm
Detector Volume = 5.8 cm^3

- ◆ $^{153}Gd \xrightarrow{\varepsilon} {}^{153}Eu$
- ▲ $^{169}Yb \xrightarrow{\varepsilon} {}^{169}Tm$
- ■ $^{154}Eu \xrightarrow{\beta^-} {}^{154}Gd$
- ● $^{39}K\,(n,\gamma)\,^{40}K$
- ▼ $^{12}C\,(n,\gamma)\,^{13}C$
- —— Curve Fitted to the Experimental Data
- - - - Experimental Curve Corrected for Absorption Between Source and Sensitive Detector Volume
- ········ Curve Calculated by the Semi-empirical Formula of Paradellis and Hontzeas



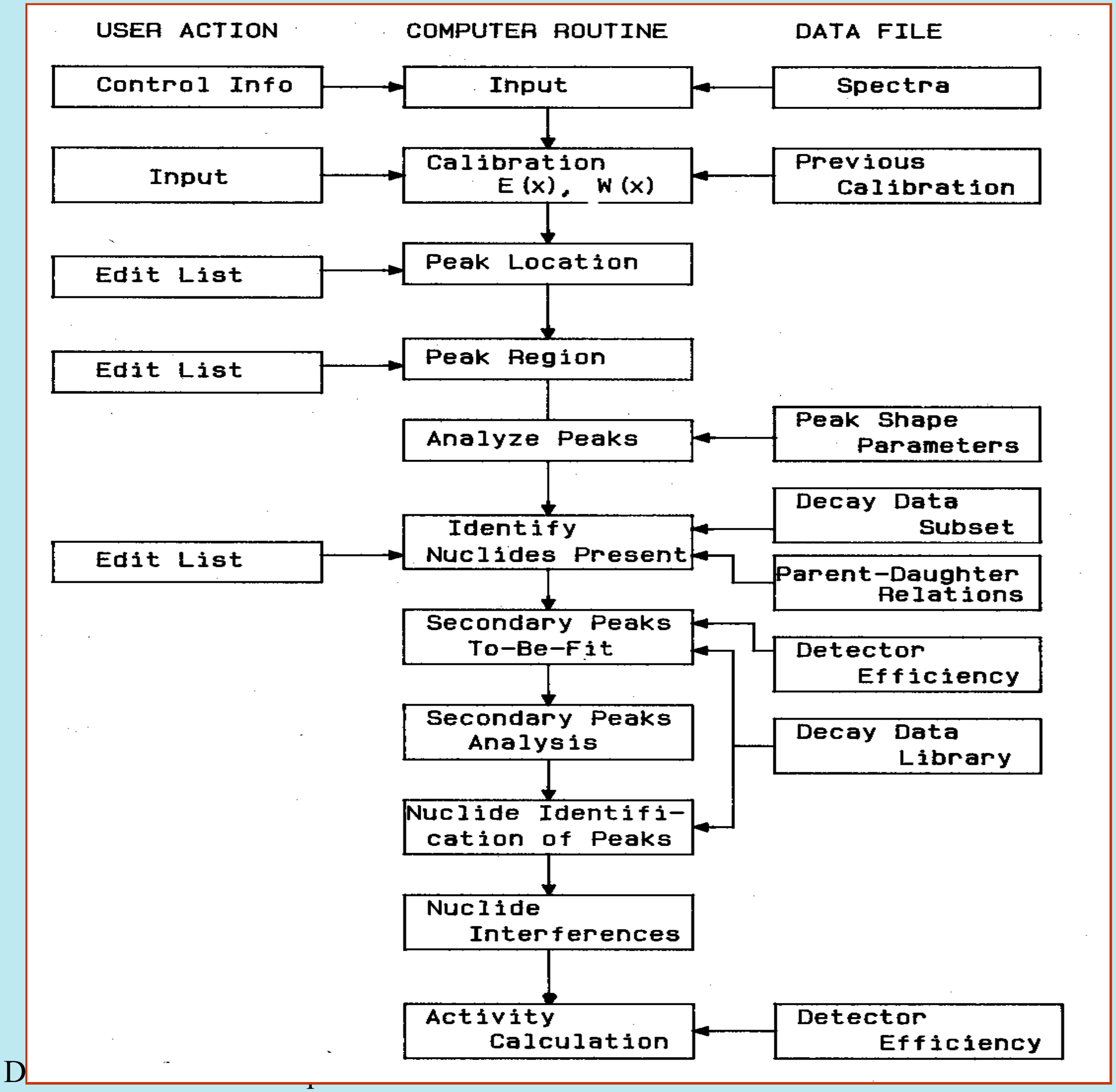
Point Source distance = 8.3 cm
Detector Volume = 38 cm^3

Detector 1

- ◆ $^{153}Gd \rightarrow {}^{153}Eu$
- ▲ $^{169}Yb \rightarrow {}^{169}Tm$
- ■ $^{154}Eu \; n^- \rightarrow {}^{154}Gd$
- + $^{53}Cr(n,\gamma)\,^{54}Cr$
- ▼ $^{12}C(n,\gamma)\,^{13}C$
- △ $^{14}N(n,\gamma)\,^{15}N$
- ▽ Calculated Data, Camp and V · Lenn
- — — Experimental Curve Corrected for Absorption Between Source and Sensitive Detector Volume

Sample Analysis Using Gamma Spectroscopy

Analysis Software



Reporting: General

Procedures must be established for management of all records associated with receipt, preparation, analyses, and disposal of samples and their derivatives:

1. These include the creation, distribution, use, maintenance, and disposition of the records.

2. The procedures are designed to comply with all applicable rules and regulations, such as local and state regulations, and the institution’s procedures.

3. They provide for the systematic control of information (from creation to disposition) generated by any of the laboratory’s information systems or media: paper, microfilm, or electronic.

Reporting: Gamma Spectroscopy Data (1)

The Gamma Spectroscopy Analysis report should, as a minimum, include:

- **Sample information:**
  - **Description, Identification number (code), Collection Date and Time, Location of Collection**
- **Analysis information:**
  - **Date, time, and location (lab name) of analysis**
  - **Detector name, Identification number(code)**
  - **Calibration files (energy, efficiency, geometry), QC files (daily / weekly check files)**
  - **Minimum Detection Levels, MDL, (units must be consistent with the reported results for the sample), Decision Level, DL, for each nuclide analyzed**
  - **Volume, weight, or quantity of the sample**
  - **Counting time**

Reporting: Gamma Spectroscopy Data (2)

The Gamma Spectroscopy Analysis report should, as a minimum, include:

- Analysis Results:
  - Calculated concentration or activity values in the units requested by the customer, Estimates of the uncertainty in the calculated concentration or activity
  - Identify the results that are below the MDA or DL
- Analyst and QA Review:
  - Name and signature of the analyst (dated)
  - Name and Signature of the QA reviewer (dated)
- Others
  - Other information requested by the customer per contract agreement

References

1. Radiochemistry Web Site: http://www.radiochemistry.org/periodictable/index.shtml
2. “Radiation Detection and Measurement”, Third Edition, G. Knoll.
3. “Gamma- and X-ray Spectroscopy with Semiconductor Detectors”, K. Debertin and R. G. Helmer.
4. ASNI N42.23-1996, “Measurements and Associated Instrumentation Quality Assurance for Radioassay Laboratories.”