Trace Element Analysis of Geological, Biological & Environmental Materials By Neutron Activation Analysis: An Exposure

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Session 4

MIT- EAPS Neutron Activation Analysis Laboratory

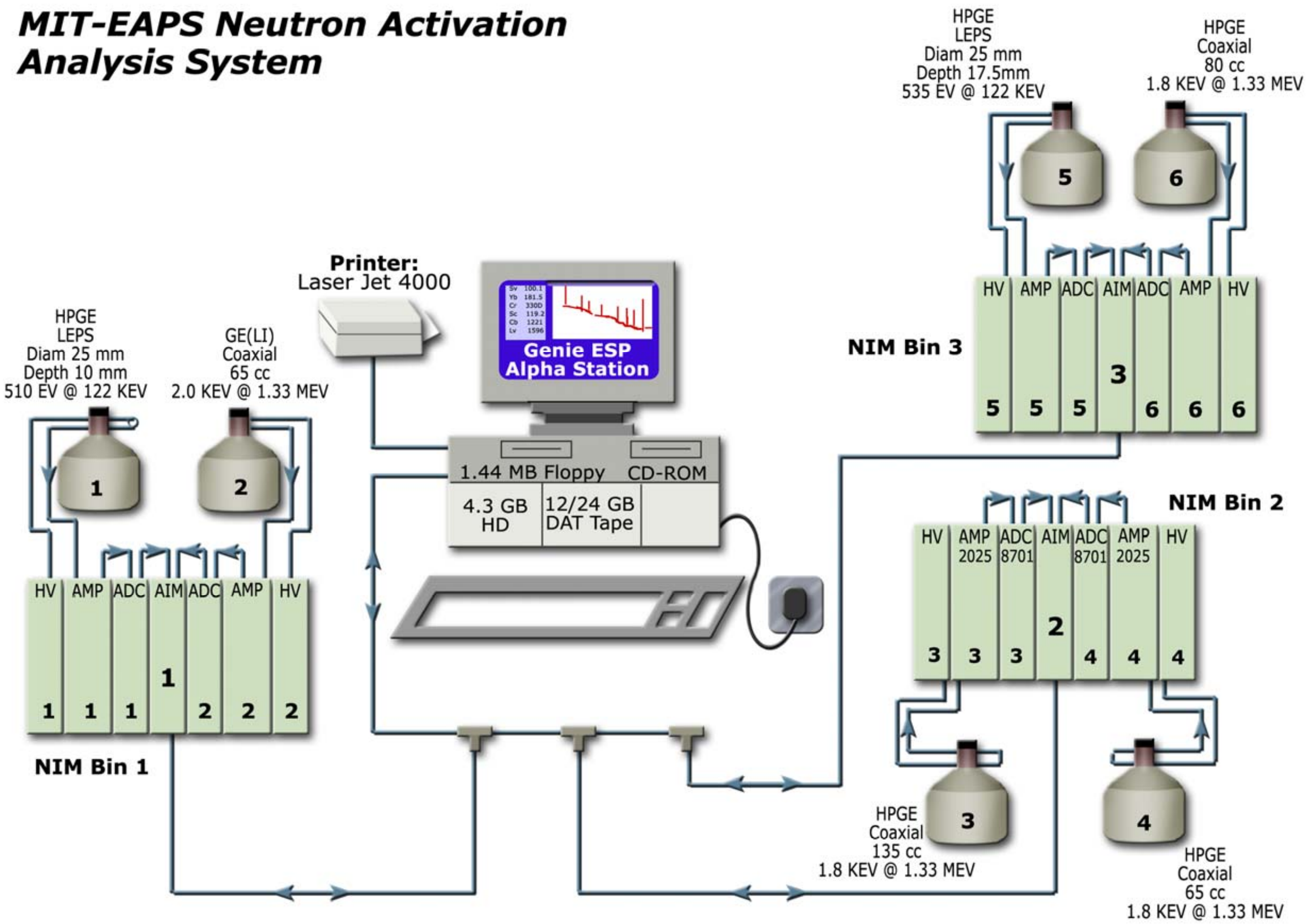
- **Hands on experience with gamma spectrometer**
- **Multi-element Trace Analysis by Neutron Activation Analysis –**
- **A Case Study of Environmental Samples**
- **12.091 Assignment Handout –**
  Due January 19, 2005
- **12.091 Course – Students' individual presentations Due January 19, 2005**

Gamma Spectrometer

The Major components of a gamma spectrometer are :

1. Germanium detector
2. Preamplifier & Amplifier
3. Analog to Digital Converter (ADC)
4. Multi Channel Analyzer (MCA)
5. Computer with data acquisition and reduction software
6. NIM Bin, High Voltage Unit and other peripherals

MIT-EAPS Neutron Activation Analysis System

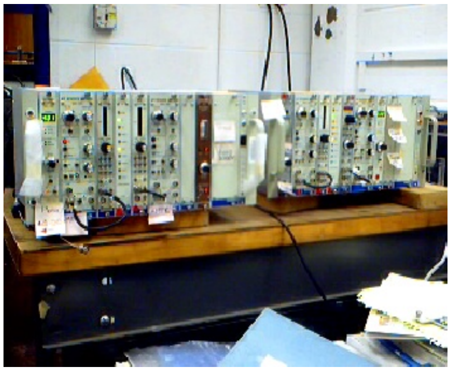


Germanium detector – vertical configuration - shielding

Germanium detector – vertical configuration - shielding

Germanium detector – horizontal configuration - shielding

NIM Bin with High Voltage, Amplifier and ADC units



Computer and Peripherals

Germanium Detector Performance

- Energy Resolution
- Full Energy Peak Efficiency
- Peak to Compton Ratio

1. Chapter IV : Instrumentation in neutron activation analysis,
P. Jagam and G. K. Muecke, pages 73-108,
Mineralogical Association of Canada
Short Course in Neutron Activation Analysis in the Geosciences,
Halifax May 1980, Ed: G. K. Muecke

Germanium Detector Performance …

- Energy Resolution:
  - Germanium crystals provide excellent resolution for gamma-ray energies. The average energy required to produce electron-hole pair in Ge is only 2.98 eV as compared to 40 eV for gas detectors and 300 eV for scintillation detectors. Thus the signal from a Ge detector is 10-100 times bigger, and hence the resolution is better. Gamma spectrometer system resolution depends on detector and Electronics, given by
  - System total resolution = SQRT ((Detector Resolution)^2 + (Electronics Resolution)^2) Conventionally, energy resolution of a Ge detector is quoted at 1.33 MeV of ^{60}Co for high energy detectors and 123 keV of ^{57}Co. The unit of energy resolution is keV or MeV, usually provided by the width of the gamma-ray peak at half of its height called the Full Width Half Maximum (FWHM)
  - Energy resolution varies with gamma-ray energies and also the size of the detector. Energy resolution decreases with increasing gamma-ray energy and size of the Ge crystal.

Typical gamma-ray energy vs. resolution of different types of Ge detectors information is given by
http://www.canberra.com/products/465.asp
http://www.ortec-online.com/pdf/biggerbetter.pdf

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Germanium Detector Performance …

- Full Energy Peak Efficiency

Usually the efficiency of a Ge detector is expressed in terms of relative efficiency, relative to the efficiency of 3" x 3" NaI(Tl) crystal; measuring the activity of ^{60}Co source placed at 25 cm from the NaI(Tl).

The efficiency of a detector depends on

type, size and geometry of the detecting medium,
and the energies of the gamma-rays incident.

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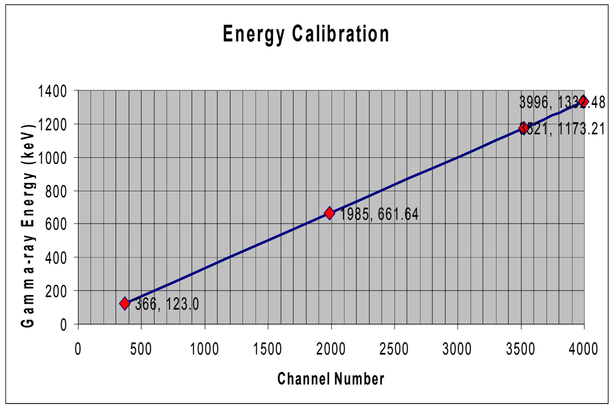
Germanium Detector Performance …

- Peak to Compton Ratio

The compton background due to high energy gamma radiation effects the low-energy gamma-ray measurements. The photo peak/Compton ratio, which is provided to indicate the detector performance at low-level peaks, increases with increasing efficiency and resolution of the detector. Compton suppression electronics are available for detector performance improvement.

Energy Calibration

Source	Gamma-ray Energy keV	Channel Number
^{57}Co	123.0	366
^{137}Cs	661.64	1985
^{60}Co	1173.21	3521
	1332.48	3996



Gamma Spectrometer - Amplifier

Amplifier:

The amplifier of the gamma spectrometer is very important and performs sophisticated tasks like

1)Pulse shaping
2)Time constant
3)Base line restoration
4)Pole-Zero cancellation
5)Pile-up detection

1. Chapter IV : Instrumentation in neutron activation analysis,
P. Jagam and G. K. Muecke, pages 73-108,
Mineralogical Association of Canada
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Gamma Spectrometer – Analog to Digital Converter (ADC) Multi Channel Analyzer (MCA)

Electronic pulses can be categorized as mainly of two types – 1)linear and 2)logic.
Linear pulses retain information of parameter of interest such as Gamma-ray energy in the amplitude of the signal.
Logic pulses retain information about timing.
The analog to digital converter (ADC) is a device that converts pulse height which is the analog equivalent of gamma-ray energy to channel number which is the digital equivalent of the energy.
Multi-channel analyzer is a device that is capable of distinguishing pulse height of the incoming signal in finer divisions of 0 to 8K or 16K, etc, speeding up the recording time of the gamma-ray spectrum.
Dead time of the system is the time during which the system is busy.
The higher the incident gamma-ray radiation on the Ge detector, the higher the dead time. Dead time is inversely proportional to the distance of the radioactive Source from the detector.

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12.091 Assignment

1) In a house in Cambridge, the water from the faucet suddenly started showing some particulate matter, which is suspected to be copper from a pipe. It was brought to the MIT Reactor for analysis. You are asked to calculate the activity that would be produced by thermal neutron activation, if 1 gram of copper is irradiated in the reactor flux of 4 x 10^{12} n.cm^{-2}.sec^{-1} for 2 hours.

i)On removal what is the activity of each copper isotope?

ii)What will be the activity of each isotope 1 hour after the removal from the reactor?

The answer should contain: the activity equation, the parameters and values used, and the activity calculated.

Suggested Use:

1) The Chart of Nuclide Handout,

2) Table of Nuclides Appendix D, p 606 -650,
Gamma-ray sources Appendix E 651-660,
Nuclear and Radiochemistry by
G. Friedlander, J. Kennedy, E. S. Macias, J. M. Miller

2)An entrepreneur wants to know whether a particular area of interest has Molybdenum and Antimony. So what are the radioisotopes that can be used for the thermal neutron activation analysis.

Provide all the relevant information of the X (n,γ)Y reaction, identify the parent and daughter nuclei, the activation cross section, the half-life of the daughter product, and the predominant gamma-ray energy for identification.

Suggested Use:

Table of Nuclides Appendix D, p 606 -650,
Gamma-ray sources Appendix E 651-660,
Nuclear and Radiochemistry by
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12.091 Assignment …

3)An unknown sample powder was found in an envelope. It was brought to the reactor for analysis. The gamma spectrum revealed significant gamma-ray peaks of energy 320 KeV, 1368 keV and 2754 keV. Identify the content of the powder.
Suggested use:
Appendix 5, Table 2
Neutron Activation Analysis
By D. De Soete, R. Gijbels, J. Hoste

12.091 Assignment …

4)The weights of empty vial, empty vial + sample powder were taken 6 times.
Write the formula for the propagation of errors, calculate the error in the weight of the sample powder. Interpret the results.

Weights (in grams) of the empty vial, weighed separately for 6 times:
1.14470, 1.14475,1.14472, 1.14476, 1.14478, 1.14475
Weights (in grams) of the empty vial, weighed separately for 6 times:
1.14470, 1.14475,1.14472, 1.14476, 1.14478, 1.14475
Weights (in grams) of the vial + sample powder, weighed separately for 6 times:
1.35041, 1.35040, 1.35029, 1.35018, 1.35026, 1.35035

5) Arsenic is determined in river sediment samples.
The abundance of As in the standard is 145 ppm.
The gamma-ray energy of 76As is 559 keV.
The gamma peak areas of the sample and standard are respectively, 32699, and 1533496 for the same counting times.
The delays from the end of irradiation for the sample and the standard counting are 5.953 d, and 4.252 d.
The weights of the sample and standard are 0.38476 g and 0.41669 g.
Calculate the abundance in the sample.
Estimate the propagation of errors. You may use the weighing error from problem 4 above.

12.091 Assignment …

6)In the MIT-EAPS INAA Laboratory, an internal standard has been analyzed 10 times. Nd is one of the Rare Earth Elements. Its measured abundance values (in ppm) are

24.0 ± 0.7, 23.7 ± 0.7, 24.0 ± 0.5, 24.3 ± 0.9, 23.7 ± 1.0, 24.3 ± 1.0, 24.0 ± 0.7, 23.8 ± 0.6, 24.0 ± 0.7, 24.7 ± 0.9.

The reference value of this standard is 24.7 ± 0.3.
Write the formulae and calculate the precision and accuracy of this measurement.
Express the precision and accuracy in percentage.

12.091 Assignment …

7)Our department chairperson came to know that an equipment grant would be available soon. So a memo was sent to our gamma-spectroscopy group asking the importance of gamma spectrometer.

Write the usefulness of a gamma spectrometer.

Describe the components of a gamma spectrometer.

Look at the latest products. To look at the website http:// www.canberra.com and look under product category and do a write up - one or two lines of each product you want to select.

12.091 Assignment …

8)The Department of Agriculture came to know that some fruit trees in Florida got contaminated. So they want to send some dry leaves for analysis of arsenic. Suggest a suitable standard.

Suggested use:
http://www.nist.gov
Look under
NIST Products and Services

Using the online Catalog (click on)

Advanced Search (click on)

Enter Keyword
(suggested keywords: agriculture, leaves)

View Certificate of Report and note down the information

Suggest your choice of the standard.

12.091 Assignment …

9)The MIT Libraries has asked you for some suggestions for new books on neutron activation analysis. What book titles can you suggest, which they do not already own?
Suggestions:
http://libraries.mit.edu/vera
type Books in Print in the search box
the next window opens
type keyword in the Quick search box.
Suggested keywords:
Neutron activation analysis
Nuclear analytical
Environmental geochemistry ...
Any keyword of your interest or field
You may search the Title in the Barton Search box to see whether that book already exists in the MIT library.
Barton, the Libraries online catalog.

12.091 Assignment …

10)Now that you are familiar with trace element analysis of materials by neutron activation analysis, briefly describe its application by giving one example.

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Activity Equation

A = number of decays per second (Activity) dps

N = number of atoms of the target isotope

$= \frac{m}{W} \times q \times 6.023 \times 10^{23}$

m = mass of the element in the irradiated sample g

θ **= isotopic abundance**

w = Atomic weight of the element

λ **= decay constant = $0.693/t_{1/2}$**

$t_{1/2}$ = Half-life of the isotope

ϕ **= neutron flux** $n.cm^{-2}.sec^{-1}$

σ **= activation cross-section** 10^{-24} cm^2

t_{irr} = irradiation time sec

Activity Equation …

$A = N \sigma \phi [1 - \exp(-\lambda t_{irr})]$

After a delay of time t_d

$A = N \sigma \phi [1 - \exp(-\lambda t_{irr})]\exp(-\lambda t_d)$

For a counting time of t_c

$A = N \sigma \phi [1 - \exp(-\lambda t_{irr})]\exp(-\lambda t_d) \; [1 - \exp(-\lambda t_c)]$

Activity Calculation - An Example

$A = N_1 \lambda$
$N_1 = A/\lambda$
Curie = Ci = 3.7×10^{10} dps
Example:
A cobalt wire 1 foot long and 1/16"th inch in diameter is left in the reactor core for two years.
The flux is 10^{12} n.cm^{-2} .sec^{-1}. What will be its activity (μc) upon removal? What will be the activity after 2 years?
Answer:
^{59}Co (n, g) produces two isotopes namely, ^{60m}Co (half life 10.5 m) and ^{60}Co (half life 5.24 y) with activation cross sections 18 and 19 barns, but the isomer decays to the long lived activity, hence its effective cross-section will be the sum (37 barns).

Activity Calculation - An Example …

λ_1 **= 0.693/ $t_{1/2}$** = 3.97 hr^{-1}

λ_2 **= 0.693/ $t_{1/2}$** = 1.505 x 10^{-5} hr^{-1}

V = volume

ρ= density

Activity Calculation - An Example …

Number of target nuclei = $V\rho N_{A\varpi} / A$
= $(0.785 \times (1/16)^2 \times 12 \times (2.54)^3 \times 8.71 \times 6.023 \times 10^{23} \ / \ 58.94)$
= 0.541×10^{23}
Number of ^{60m}Co (with half life 10.5 m) nuclei = N_1
= $(18\times10^{-24} \times 0.541 \times 10^{23} \times 10^6 \times 3600) \times 1/3.97$
= 8.83×10^8
Number of ^{60}Co (with half life 5.24 y) nuclei = N_2
= $37\times10^{-24} \times 0.541 \times 10^{23} \times 10^6 \times 3600 \times$
$(1 - \exp(-0.693\times2 / 5.24) / (1.505 \times 10^{-5})$
= 11.13×10^{13}

Activity Calculation - An Example …

The activity on removal will be

$A_0 = N_1\lambda_1 + N_2\lambda_2$

$= (8.83 \times 10^8 \times 3.97 /(3600 \times 3.7\times10^{10}))+ (11.13 \times 10^{13} \times 1.505 \times 10^{-5}/ (3600 \times 3.7\times10^{10}))$

$= 26.32+ 12.58 = 38.90\ \mu c$

The activity after two years will be

$= A_0 (\exp - \lambda_2\, t_d)$

= 38.90 x 0.767 = 29.89

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References

1. Chapter IV : Instrumentation in neutron activation analysis,
P. Jagam and G. K. Muecke, pages 73-108,
Mineralogical Association of Canada
Short Course in Neutron Activation Analysis in the Geosciences,
Halifax May 1980, Ed: G. K. Muecke

2. Chapter V : Instrumental neutron activation analysis of rocks and minerals,
I. L. Gibson and P. Jagam, pages 109-131,
Mineralogical Association of Canada
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Halifax May 1980, Ed: G. K. Muecke

References …

For routine geologic sample analysis:

4.Utilization of neutron activation analysis in the study of geologic materials,
P. Ila and F. A. Frey,
Atomkernenergie Kern-technik supplement 44 (1984) 710-716.

For dead time corrections during routine geologic sample analyses:

5. Determination of Wyttenbach correction factors in neutron activation system dedicated to trace element analysis of geologic samples,
P. Ila,
Journal of Radioanalytical and Nuclear Chemistry, 122 (1988) 103-113.

References

6. For typical gamma-ray energy vs. resolution, and gamma-ray energy vs. efficiency of different types of Ge detectors:
http://www.canberra.com/products/465.asp
http://www.ortec-online.com/pdf/biggerbetter.pdf

Session 4 End