EXPERIMENT 17

Neutron Activation Analysis (Slow Neutrons)

EQUIPMENT NEEDED FROM EG&G ORTEC

113 Scintillation Preamplifier
266 Photomultiplier Tube Base
Bin and Power Supply
556 High Voltage Power Supply
480 Pulser
575A Amplifier
905-3 2-in. x 2-in. or 905-4 3-in. x 3-in. NaL(Tl) Detector and PM Tube
ACE-2K MCA System including suitable IBM PC (other EG&G ORTEC MCAs may be used)
Oscilloscope
1- to 3-Ci Am-Be neutron source in howitzer
308 Neutron Howitzer and Activation Chamber
313 Activation Sample Set
317 Activation Sample Set
Model CD-17 Cadmium Shields
Model V-17 Vanadium Saturation Factor Kit
Model RE-17 Special Sample Set
ORC-17 Cable Set
Source Kit SK-1G (see Appendix)

Purpose

This experiment will demonstrate the principles of element identification using the technique of slow neutron activation.

Introduction

Neutron activation analysis is a very powerful technique for identifying many elements. Basically the technique is quite simple. A sample is irradiated by slow neutrons and becomes radioactive. By measuring the β^+s, γs, β^-s, and half-life of the resulting sample, the elemental constituents of the sample and their relative concentrations can be identified.

Industrial activation analysis is usually done with slow neutrons from a reactor, where the neutron flux can be as high as 5×10^{13} neutrons/cm^2/s, or with an accelerator with fast neutron fluxes of 10^{10} neutrons/cm^2/s. When activation analysis is compared with other instrumental analyses such as gravimetric, colorimetric, spectrographic, or mass spectroscopy, its sensitivity is usually shown to be better by a factor of 10 than that of other methods. Activation analysis is used extensively in such fields as geology, medicine, agriculture, electronics, metallurgy, criminology, and the petroleum industry.

The Neutron Source

This experiment is described for 1 Ci of Am-Be for the neutron source, with the source located in the center of a paraffin howitzer. The samples are irradiated at a point ~4 cm from the source by the neutrons that have been moderated by the paraffin between that point and the source. Any of the commonly found isotopic neutron sources can be used for this experiment.

Neutron Activation Equations

Assume that the sample has been activated in the howitzer. At the instant when the activation has been terminated, (t = 0), the activity of the sample is given by the following expression:

$$A_0 = \frac{\sigma m \eta \phi \alpha S}{w}, \qquad (1)$$

where

A_0 = the number of disintegrations per second of the element in the sample at t = 0 (when irradiation stops),
σ = cross section for the reaction, cm^2,
m = mass of the target element, grams,
η = Avogradro's number, 6.023×10^{23} molecules/mole,
ϕ = neutron flux, neutrons/cm^2/s,
α = fraction of the target isotope in the sample [e.g., with an ordinary copper sample producing the $^{63}Cu(n,\gamma)^{64}Cu$ reaction, $\alpha = 0.69$ since 69% of all natural copper is ^{63}Cu],
S = saturation factor, $1 - e^{-\lambda t}$, where $\lambda = 0.693/T_{1/2}$ and $T_{1/2}$ is the half-life for the reaction,
w = atomic weight of the element.

(Note: In the above definitions, t and $T_{1/2}$ must be in the same time units.)

Let us examine Eq. (1) in terms of our knowledge about a reaction. For example, if we were activating an aluminum sample, the following reaction would take place:

$$^{27}Al(n,\gamma)^{28}Al,\ T_{1/2} = 2.3 \text{ min} \qquad (2)$$

The cross section from ref. 13 is 0.21×10^{-24} cm^2. For our example then, we can determine everything in Eq. (1) except A_0 and ϕ. A_0 can be measured with a scintillation counter (the technique that is outlined), and ϕ will be determined in Experiment 17.1.

Table 17.1 is a list of common thermal neutron cross sections that is taken from ref. 11.

Table 17.1. Common Thermal Neutron Cross Sections.

	Reaction	σ(barns)
1.	$^{27}Al(n,\gamma)^{28}Al$	0.210 ± 0.020
2.	$^{51}V(n,\gamma)^{52}V$	5.00 ± 0.010
3.	$^{63}Cu(n,\gamma)^{64}Cu$	4.51 ± 0.23
4.	$^{23}Na(n,\gamma)^{24}Na$	0.536 ± 0.010
5.	$^{55}Mn(n,\gamma)^{56}Mn$	13.3 ± 0.2

After irradiation the sample is transferred immediately to the NaI(Tl) detector, and a spectrum is accumulated for a time, (t_i), long enough to get reasonable statistics under the photopeak. The time is usually at least one half-life. The true number of disintegrations, (N_d), that occurred during t_i can be determined from the following:

$$N_d = \frac{\Sigma_p - \Sigma_\beta}{G\epsilon_p f} , \qquad (3)$$

where

Σ_p = sum under the photopeak,

Σ_β = background for the same counting period under the photopeak,

G = $A/2\pi s^2$, where A = area of detector in cm^2, and s = distance from source to detector in cm,

ϵ_p = intrinsic peak efficiency for the gamma energy and the detector size used (Fig. 3.6, Experiment 3.6, or ref. 10 in Experiment 3),

f = decay fraction of the unknown activity, which is the fraction of the total disintegrations in which the measured gamma is emitted (refs. 7 and 10 and Table 3.2 in Experiment 3.6).

From the decay equation, N_0 can be calculated:

$$N_d = N_0 (1 - e^{-\lambda t}) , \qquad (4)$$

$$A_0 = \lambda N_0 , \qquad (5)$$

where t is the time for which the sample was counted.

Therefore we have reduced Eq. (1) to one unknown, ϕ, which is the number of neutrons/cm^2/s for our howitzer. The secondary purpose of this experiment is to find ϕ for the howitzer, using the $^{27}Al(n,\gamma)^{28}Al$ reaction. We will also use that value of ϕ to determine the cross section for the $^{51}V(n,\gamma)^{52}V$ reaction.

EXPERIMENT 17.1
Neutron Flux Determination

Procedure

1. Set up the electronics as shown in Fig. 17.1. Use the bipolar output from the 575A to furnish pulses into the MCA.

2. Calibrate the system for full scale on the MCA of ~2 MeV. Use the ^{137}Cs and ^{60}Co gamma sources from the source kit for the calibration. Draw the calibration line (as in Experiment 3 or use the MCA energy calibrate feature).

3. Place the aluminum sample in the howitzer and activate it for 5 min. Transfer it immediately to the scintillation counting position and count it for a clock time [t_i in the discussion preceding Eq. (3)] of 2 min.

EXERCISE

a. Read the data out of the MCA and determine E_γ and the sum under the photopeak.

4. Remove the sample and count the background for 2 min. From your data determine N_d of Eq. (3).

EXERCISES

b. Substitute the value for N_d into Eq. (4). Solve for N_0 and A_0 in Eqs. (4) and (5).

c. Solve Eq. (1) for ϕ using the accepted cross section for the $^{27}Al(n,\gamma)^{28}Al$ reaction as 0.21 x 10^{-24} cm^2.

5. Since the half-life for the reaction is 2.31 min, the sample activity will die out within ~20 min. After approximately this period of time, repeat the experiment and determine a second value for ϕ. If the work has been done carefully, the numbers should agree to within 5% of each other. Determine the average value of ϕ; this will be used in Experiment 17.2.

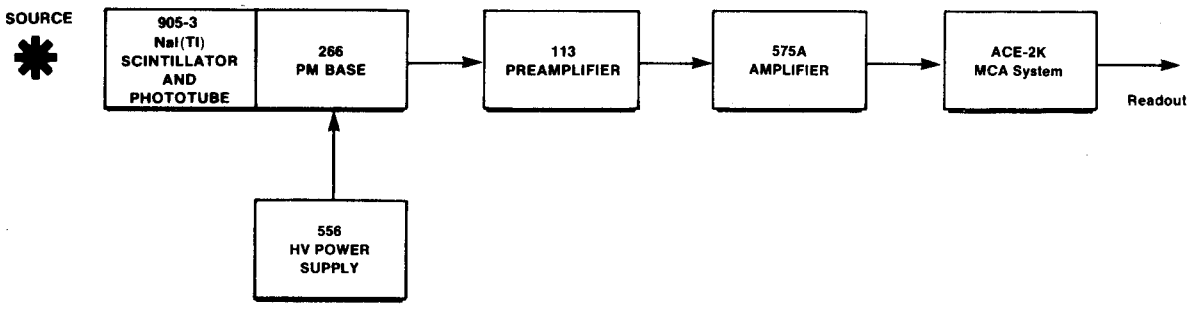

Fig. 17.1. Electronics Interconnections for Experiment 17.1.

EXPERIMENT 17.2

Measurement of the Thermal Neutron Cross Section for the $^{51}V(n,\gamma)^{52}V$ Reaction

Procedure

1. Use the same electronics setup as for Experiment 17.1.

2. Activate the vanadium sample for 5 min. Transfer it to the scintillation counting station and count for $t_1 = 2$ min.

EXERCISE

Use the average value of ϕ from Experiment 17.1 and the procedure discussed above to solve for σ, the cross section for the $^{51}V(n,\gamma)^{52}V$ reaction. The accepted value is 4.9×10^{-24} cm^2. The measured gamma-ray energy for this reaction is 1.434 MeV. Do your results agree with this value?

EXPERIMENT 17.3

Determination of the Half-Life for the $^{27}Al(n,\gamma)^{28}Al$ Reaction

Procedure

1. Use the same electronics setup as for Experiment 17.1. Set the MCA Region of Interest, (ROI), so that it brackets the 1780-keV peak from the decay of ^{28}Al. See Fig. 17.2 for a typical spectrum from aluminum.

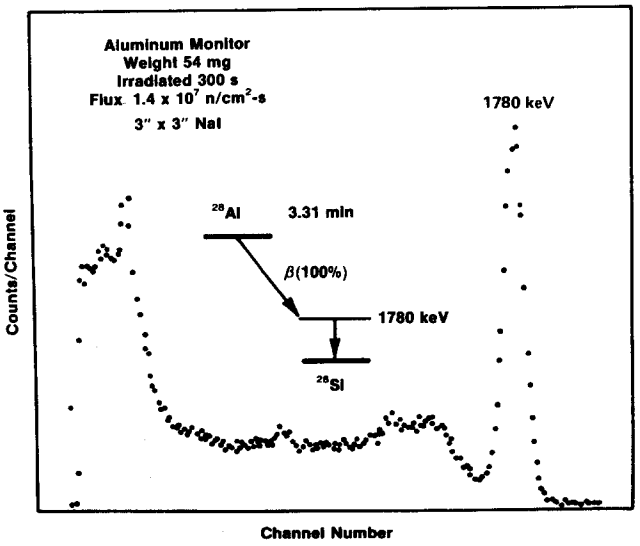

Fig. 17.2. Thermal Neutron Activation Spectrum of ^{28}Al.

2. Activate the aluminum sample (used in Experiment 17.1) for 5 min.

3. Transfer the sample and take 15-s counts every 45 s.

EXERCISE

Plot the data on semilog paper and determine $T_{1/2}$.

Note: Many other slow neutron reactions can be studied with the isotopic neutron source and the electronics of Experiment 17. Any of these can be made if the appropriate target materials are available. Reference 11 outlines many of these experiments. Table 17.2 (taken from ref. 11) is a rather com-

Table 17.2. Recommended Slow Neutron Reactions.

Element	Target Nuclide	Target Material	Product Nuclide	$T_{1/2}$	E_γ (MeV)
Aluminum	^{27}Al	Al, Al_2O_3	^{28}Al	2.30 m	1.78
Sodium	^{23}Na	Na_2CO_3	^{24}Na	15.0 h	2.75, 1.37
Vanadium	^{51}V	NH_4VO_3 or metal	^{52}V	3.77 m	1.43
Manganese	^{55}Mn	MnO_2	^{56}Mn	2.58 h	0.845, 1.81, 2
Cobalt	^{59}Co	CoO or foil	^{60m}Co	10.5 m	0.059
Copper	^{63}Cu	CuO or foil	^{64}Cu	12.9 h	0.511 $\gamma\pm$
Gallium	^{71}Ga	Ga_2O_3	^{72}Ga	14.3 h	0.63, 0.83
Germanium	^{74}Ge	Ge (metal)	^{75}Ge	82 m	0.26, 0.20
Arsenic	^{75}As	As_2O_3	^{76}As	26.5 h	0.56, 0.66, 1
Bromine	^{79}Br	NH_4Br	^{80}Br	18 m	0.62, 0.51 $\gamma\pm$
Indium	^{115}In	In (metal foil)	^{116m}In	54 m	0.40, 1.09, 1.27, 2.08
Tellurium	^{130}Te	Te	^{131}Te	25 m	0.15, 0.45
Iodine	^{127}I	NH_4I	^{128}I	25 m	0.46
Lanthanum	^{139}La	La_2O_3	^{140}La	40.2 h	0.48, 1.59
Tungsten	^{186}W	WO_3	^{187}W	24 h	0.480, 0.686, 0.134
Gold	^{197}Au	Au-Dowex-1 (gold foil)	^{198}Au	64.8 h	0.411

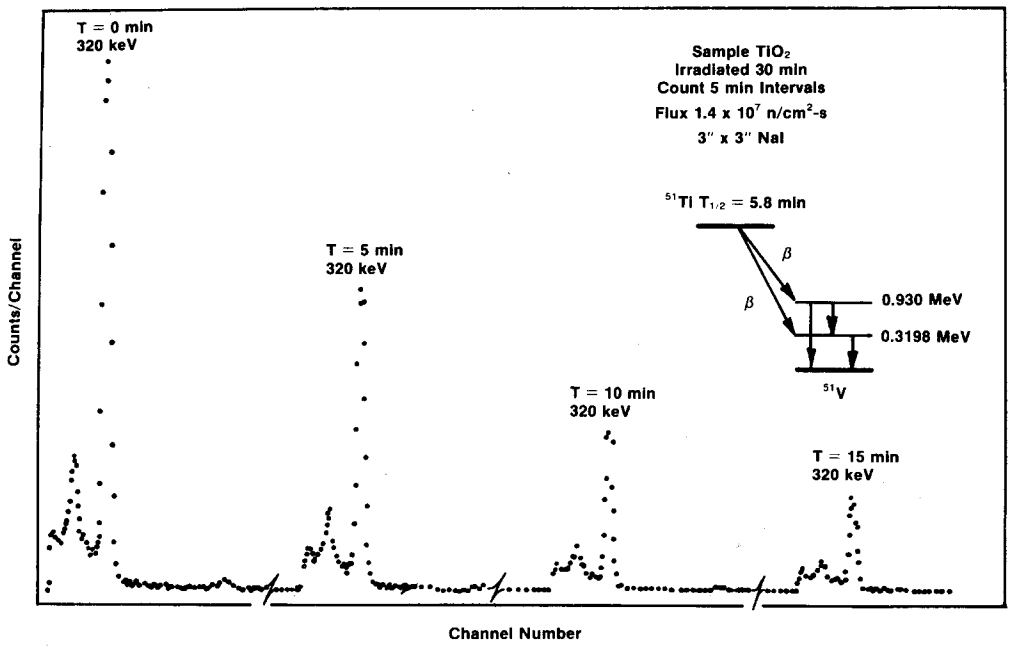

Fig. 17.3. Series of Four Pulse-Height Spectra for ^{51}Ti Showing the Fall-Off in Activity in Time.

plete listing of reactions that seem to work well for experiments by students. Figure 17.3 shows the fall-off in intensity of the 5.8-min ^{51}Ti activity displayed as four pulse-height spectra. If we were doing this experiment the ROI would be set to bracket the 320-keV photopeak. Table 17.2 shows a list of other recommended slow neutron reactions. Some of the resulting decay schemes are more complex than those we have studied in Experiments 17.1, 17.2, and 17.3. For example, Fig. 17.4 shows the decay of ^{187}W. Some of the gammas are listed in Table 17.2.

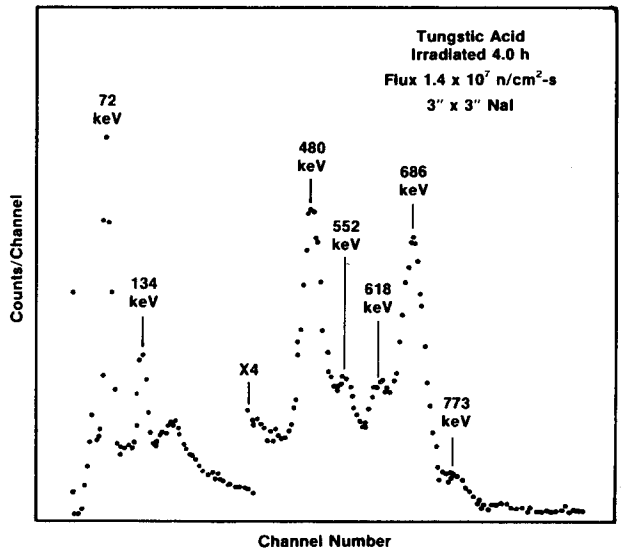

Fig. 17.4. Neutron Activation Spectrum of Tungsten.

EXPERIMENT 17.4

The Saturation Factor in Neutron Activation Analysis

From the definitions at the beginning of Experiment 17, the saturation factor is given by:

$$S = 1 - e^{-\lambda t}$$

From this expression it can be seen that the activity at any time is given by:

$$A_t = A_s\,(1 - e^{-\lambda t}), \qquad (6)$$

where A_s is the activity at saturation.

In this experiment we will verify Eq. (6) for the decay of ^{52}V (Fig. 17.5). Each of the vanadium samples in the Model V-17 Kit contains 2 g of NH_4VO_3. In the experiment we will irradiate these identical samples for different times, (t), and plot the corresponding ^{52}V activity as a function of irradiation time.

Procedure

1. Use the electronics shown in Fig. 17.1. Set the ROI of the MCA so that it brackets the 1434-keV gamma line (Fig. 17.5). This can be done by irradiating one of the samples for 5 min and counting it directly against the face of the NaI(Tl) detector.

2. Irradiate samples for 2, 4, 6, 10, 15, and 25 min. Note: All of the ports of the neutron howitzer may be used, since the flux density, ϕ, is the same in all ports.

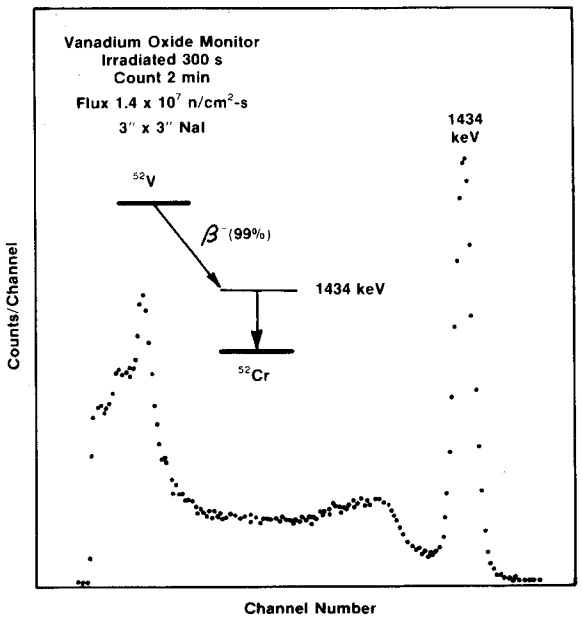

Fig. 17.5. Thermal Neutron Activation Spectrum of ^{52}V.

3. After irradiation, immediately transfer each sample to the counting station and count for exactly 100 s.

EXERCISE

Since the $T_{1/2}$ of the vanadium activity is 3.77 min, an irradiation time of 25 min will yield a value of A_s from Eq. (6). Therefore we can call the count under the ROI 25 min = A_s. For any other irradiation time, define the count under the ROI to be A_t.

On two-cycle graph paper make a plot of the experimental data A_t/A_s versus irradiation time. Plot the theoretical function, $1 - e^{-\lambda t}$ on the same graph.

EXPERIMENT 17.5

The Study of a Complex Sample with Two Half-Lives Present

Ordinary silver has two pronounced isotopes, ^{107}Ag (51.35%) and ^{109}Ag (48.65%). If we activate a piece of silver, the following activities are produced: $^{107}Ag(n,\gamma)^{108}Ag$ ($T_{1/2}$ = 2.3 min) and $^{109}Ag(n,\gamma)^{110}Ag$ ($T_{1/2}$ = 24 s). The purpose of this experiment is to determine these half lives by correcting the compound decay curve. ^{108}Ag has a 0.44-MeV gamma while ^{110}Ag has gammas of 0.66 and 0.94 MeV.

Procedure

1. Obtain two silver samples from the Activation Sample Set No. 317. Set up the electronics as shown in Fig. 17.1. Activate the first sample for 5 min and immediately place it against the face of the NaI(Tl) detector. Set the ROI of the analyzer so that it brackets the region from 0.44 MeV to 0.94 MeV. Under these conditions we are counting both half lives. This first sample is being irradiated so that we can set the ROI.

2. We are now ready to take data for the complex decay curve. Irradiate the second silver sample for a time of 10 min. Transfer the sample to the counting station and take 10-s counts every 30 s for 8 min. It is necessary that you record each ROI reading and erase quickly in preparation for the next reading.

EXERCISES

a. Plot the resulting data on a semilog scale. The straight line that represents the long-lived (2.3 min) component can easily be drawn on the curve by constructing a line through all points taken after 3.5 min. Determine the half life of the long-lived component.

b. Extrapolate the straight line for the 2.5-min activity back to zero time. Now subtract the straight line counts from this activity from all points from t = 0 to t = 3.5 min. Plot the resulting short-lived activity and determine the half life of the short-lived activity.

EXPERIMENT 17.6

Thermal Neutron Shielding

Introduction

In Experiment No. 3, we showed that the attenuation of gammas through a lead absorber is given by the following expression:

$$I = I_0 e^{-\mu x} \qquad (7)$$

(See Eq. (8), Experiment 3.) A similar expression can be written for the attenuation of neutrons through an absorbing foil.

The equation takes the following form:

$$I = I_0 e^{-N\sigma x}, \qquad (8)$$

where
N = number of atoms/cm^3 of the absorber,
σ = thermal neutron cross secition (in cm^2),
x = the absorber thickness (in cm).

In this experiment we will measure the reduction of the thermal neutron flux, ϕ, by placing a thin sheet of cadmium around the sample to be irradiated.

Procedure

1. Set up the electronics as shown in Fig. 17.1. Take two identical aluminum samples from the Activation Sample Set No. 317. Wrap one of the samples with a cadmium foil

Table 17.3. Recommended Activation Parameters for the Elements in Sample Kit RE-17.

Element	Reaction	σ_{barns}	$T_{1/2}$	Measured γ (keV)	Activation Time	Counting Time (s)
Indium	$^{115}In(n,\gamma)^{116}In$	210	54.2 min	1270	108 min	600
Copper	$^{65}Cu(n,\gamma)^{66}Cu$	2.10	5.1 min	1040	10 min	200
Germanium	$^{74}Ge(n,\gamma)^{75}Ge$	0.60	82 min	266	82 min	600
Tungsten	$^{184}W(n,\gamma)^{185}W$	2.0	1.7 min	171	6 min	100
Titanium	$^{50}Ti(n,\gamma)^{51}Ti$	0.140	5.8 min	322	12 min	200
Manganese	$^{55}Mn(n,\gamma)^{56}Mn$	13.3	2.58 h	845	4 h	1000

from the Cadmium Foil Set No. CD-17. The samples are now ready to irradiate.

2. Calibrate the MCA for 2 MeV full scale. Place the unshielded aluminum sample in the howitzer and irradiate for 8 min. By using the $^{27}Al(n,\gamma)^{28}Al$ cross section, (0.219 barns), and the techniques in Experiment 17.1 determine the flux, ϕ, in the howitzer. Define this flux to be ϕ_1.

3. Repeat procedure **2** exactly for the aluminum sample that is wrapped with the cadmium foil. Determine the new value of flux and label it ϕ_2.

EXERCISE

From Eq. (8) it can be seen

$$\frac{\phi_2}{\phi_1} = e^{-N\sigma x} \qquad (9)$$

The density of cadmium is 8.65 g/cm^2, and its atomic weight is 112.4. This gives N = 4.651 x 10^{22} atoms/cm^3.

The value, x, of the cadmium foil is written on the foil. From Eq. (9) the thermal absorption cross section for cadmium can be determined. How does your value compare to the accepted value of 2500 barns? The effective shielding parameter for cadmium is given in Eq. (9) by ϕ_2/ϕ_1.

EXPERIMENT 17.7

The Measurement of Thermal Neutron Activation Cross Sections of Elements with High Sensitivity Ratios

Introduction

A "Table of Relative Sensitivities of Elements to Thermal Neutron Activation" is given in the Appendix. In this experiment we will use the value of flux, ϕ, for the howitzer as determined by Experiment 17.1. From this value of ϕ we will calculate the cross section, σ, for several selected elements from the Special Sample Set RE-17.

Procedure

1. Set up the electronics as shown in Fig. 17.1. Calibrate the MCA for 2 MeV as in Experiment 17.1.

2. Place the indium sample in the howitzer and irradiate for two half lives ($T_{1/2}$ = 54.2 min). Transfer it to the NaI counting station and count for 600 s.

EXERCISE

Sum under the 1270-keV peak and determine σ from Eqs. (1) through (5). How does your value compare with the accepted value of 210 barns (Table 17.3)?

3. Repeat for several of the other samples from the Special Sample Kit RE-17. In each case, the sample should be irradiated for at least two half lives. The counting time should be adjusted so that reasonable statistics are obtained under the peak of interest. Table 17.3 shows some recommended parameters for the samples in the RE-17 Kit.

References

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NOTE OF CREDIT: Some parts of this experiment were taken from ref. 11 by G. I. Gleason. This reference contains 18 excellent experiments that can be done in Activation Analysis.