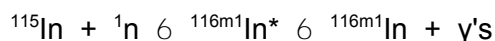


## Neutron Activation of Indium

### I. Introduction

Nuclear reactions often involve the nuclear capture of particles such as neutrons or protons, resulting in a very unstable "compound" nucleus which promptly ( $<10^{-12}$  sec) de-excites by emission of particles and/or gamma rays. The typical de-excitation energy associated with the absorption of a low-energy neutron is around 7-8 MeV, the nuclear binding energy for such a particle. Since neutrons are electrically neutral, there is no coulomb barrier to overcome, so neutron absorption can take place readily with neutrons of thermal energies ( $\sim 0.025$  eV). Neutron absorption followed by de-excitation by prompt  $\gamma$  emissions (see reaction below) will result in a product nucleus with same Z, but A+1. If this product is radioactive, one can then detect the radiation emitted in its decay.

In this experiment, the stable isotopes of indium will be exposed to neutron radiation and activated. The reaction of interest is:



This reaction can be written in shorthand notation as:  $^{115}\text{In}(n,\gamma)^{116\text{m}}\text{In}$ . The gamma radiation emitted during the decay of radioactive  $^{116\text{m}}\text{In}$  will be monitored.

The amount of radioactivity induced by any such neutron activation can be calculated by:

$$A_0 = N \phi \sigma S \quad (1)$$

where  $A_0$  is the induced activity in disintegrations per second (dps) at the end of the irradiation;

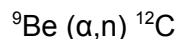
$N$  is the number of target nuclei, and can be calculated from mass of element in sample ( $m$ ), abundance of target isotope ( $a$ ), Avogadro's number ( $N_a$ ) and atomic weight ( $AW$ ) according to  $\frac{m \cdot a \cdot N_a}{AW}$ ;

$\phi$  is the neutron flux ( $\text{n}/\text{cm}^2\text{-sec}$ ) used to irradiate the sample;

$\sigma$  is the cross section of the neutron absorption reaction, in units of  $\text{cm}^2$ ;

$S$  is called the saturation term and accounts for decay of any induced activity during the irradiation;  $S$  equals  $1 - e^{-0.693t_i/t_{1/2}}$ , where  $t_i$  is the irradiation time and  $t_{1/2}$  is the half life of the product radionuclide.

In this experiment, the source of neutrons is a 5 Ci Pu-Be source.  $^{238}\text{Pu}$  emits alpha particles which are absorbed by  $^9\text{Be}$ , followed by the prompt emission of a neutron. This reaction can be summarized as:



The approximate neutron flux of this source is  $10^4$   $\text{n}/\text{cm}^2\text{-sec}$ . The neutrons emitted by this source have an average energy of 4.5 MeV and must be thermalized (slowed down)

before significant absorption by the indium isotopes will take place. Slowing down of the neutrons is achieved by use of a moderator, such as water or paraffin, which contains many hydrogen atoms, thus allowing for maximum energy transfer in collisions between neutron and moderator. The large container surrounding the neutron source contains much paraffin for this purpose, as well as other shielding material to protect against the  $\gamma$  radiation present in this source.

A 3-inch x 3-inch well-NaI(Tl) scintillation detector coupled to a Canberra InSpector and S-100 multichannel analyzer (MCA) card will be used to measure the gamma-ray activity of an indium standard and unknown following irradiation. Your instructor will demonstrate the operation of the gamma-ray spectrometry system. **You should note carefully the settings for the amplifier, high voltage supply, and multichannel analyzer, and avoid altering these settings unless instructed to do so.**

Usually neutron activation analysis is carried out using a nuclear reactor with a flux of about  $10^{12}$  n/cm<sup>2</sup>-sec as the neutron source. Because of the low neutron flux of the Pu-Be source being used for your irradiations, the only significant radioactivity induced in your samples is that of  $^{116m1}\text{In}$ , and the observed gamma-ray spectrum of your samples a short time after irradiation is due primarily to  $^{116m1}\text{In}$ . As it decays  $^{116m1}\text{In}$  emits several prominent gamma rays. Each gamma ray of a particular energy will give rise to a distinct photopeak in the NaI(Tl) gamma-ray spectrum, and, in principle, any of these photopeaks can be used for quantitative analysis. Usually the most prominent and/or clearly separated photopeak is chosen.

If a sample containing a known amount of In is irradiated and counted in exactly the same way as an unknown sample, then eq. (1) can be manipulated to show that a comparison of a particular  $^{116m1}\text{In}$  photopeak count rate for each sample enables one to determine the indium content in the unknown. That is,

$$\frac{R_{\text{sam}}}{R_{\text{std}}} = \frac{m_{\text{sam}}}{m_{\text{std}}} \quad (2)$$

where R represents the  $^{116m1}\text{In}$  photopeak count rate (in counts per second, cps) and m is the mass of In. This assumes that both samples are of similar size and composition.

## II. Equipment

- A. NaI(Tl) scintillation detector
- B. Canberra InSpector, S-100 multichannel analyzer (MCA) card Genie-PC software
- C. 5 Ci Pu-Be neutron source
- D. 12x75mm test tube
- E. half-dram polyethylene vial (obtain from instructor)
- F. razor blade (common locker)
- G. soldering iron (obtain from instructor)

## III. Reagents (obtain indium metal and unknown indium sample from instructor)

- A. Hydrochloric acid, 12.0 M
- B. Nitric acid, 15.7 M

## IV. Procedure

## A. Before coming to lab

1. Calculate the  $^{116m}\text{In}$  activity, in dps, to be expected at the end of a 24-hour irradiation of 100 mg of indium in a thermal neutron flux of  $10^4$  n/cm<sup>2</sup>-sec. The (n, $\gamma$ ) cross section for  $^{116m}\text{In}$  formation is 71 barns (1 barn =  $10^{-24}$  cm<sup>2</sup>). The isotopic abundance of  $^{115}\text{In}$  is 95.7% and the half life of the product nuclide is 54.2 minutes.
2. Calculate the percent of In atoms in your 100-mg sample that are present as radioactive  $^{116m}\text{In}$  atoms at the end of the 24-hour irradiation above. Remember that radioactive decay follows first-order kinetics and that the decay rate A (or  $A_0$  for the end of the irradiation) is related to the number of radioactive nuclei  $N^*$  by

$$A = \lambda N^* \quad (3)$$

where  $\lambda$  equals  $\frac{\ln 2}{t_{1/2}}$ .

## B. Unknown and Standard Preparation

The unknown will be provided to you in a half-dram polyethylene vial as a 1.00 mL sample of an aqueous solution containing indium.

Standard Indium Solution

1. Accurately weigh a 0.1-gram sample of indium metal to the nearest 0.1 mg.
2. Transfer the indium to a 12x75 mm test tube.
3. Using a Pasteur pipet, carefully add exactly 6 drops of 12.0 M HCl to the test tube followed by exactly 4 drops of 15.7 M HNO<sub>3</sub>. Do this operation in a fume hood to avoid NO<sub>2</sub> fumes in the lab.
4. After the evolution of gases has slowed, add 2 additional drops of 12.0 M HCl and 4 additional drops of 15.7 M HNO<sub>3</sub>.
5. Heat in a small water bath until all metal has dissolved. This may take about 30 minutes.

- Using a Pasteur pipet, transfer the solution from the test tube to a plastic half-dram vial. Using a second pipet, add 8 drops of water to the test tube, washing the test tube walls as you add the water. Using your previous pipet, transfer this wash water to the plastic vial. Add a second 8 drops of wash water to the test tube and transfer this to the plastic vial as well, using the original pipet.
- Compare the contents of your vial to a reference vial containing exactly 1.0 mL of water. If necessary, carefully add water dropwise to make your solution volume 1.0 mL.
- Close your vial, cut off the hinge and top tab with a razor blade and heat seal your vial with a soldering iron.
- Give your labeled standard to your instructor to have it, along with your unknown, irradiated in the Pu-Be neutron source for your next lab period.

### C. Energy Calibration of the Spectrometer

- In order to identify the energy associated with each photopeak in the gamma-ray spectra of your samples, a calibration of the counting system must be made for the particular high voltage, amplifier and ADC gain settings used. This calibration consists of counting sealed gamma standards (counting geometry is not important) and locating the center of each photopeak of known energy.
- The calibration sources and their gamma-ray energies are:

<u>Source</u>	<u>Energy (keV)</u>
$^{22}\text{Na}$	511, 1275
$^{60}\text{Co}$	1173, 1332
$^{137}\text{Cs}$	662
$^{54}\text{Mn}$	835

For each of the available gamma sources place the source by the detector and count until sufficient counts are collected to enable a definite determination of the photopeak center. All counting should be performed so that the MCA dead time is less than 2%. Adjust the location of the source to ensure this. Note that you may count more than one source at a time, provided that their photopeaks don't overlap significantly. After counting, record the peak center channel number (to the nearest half channel) of each photopeak. Return each standard to the storage case after it has been counted.

- On a linear scale, plot gamma-ray energy vs channel number. These data should form a straight line plot (but may not go through 0,0). Fit your data with a linear least-squares line and use the equation for this line to determine the energies of the photopeaks observed during the counting of your samples.

#### D. Counting the Standard and Unknown Solutions

1. Upon receipt of your activated samples, monitor the radioactivity level of the samples and record this in the Log Book. Before counting, wipe each sample with a moistened Kimwipe. Count each sample with the NaI(Tl) detector for 1000 seconds live time; **count the unknown first**. Use the identical counting geometry for both unknown and In standard; each sample should rest on the bottom of the detector well. When each counting is completed, save the data in a file on the computer hard disk. When both samples have been counted, print out the analyzer data. Since one sample sits decaying on the lab bench while the other is being counted, a correction for this decay must be made. **Start recording the time on a timer when the first sample count is started and stop the timer at the beginning of the second counting.** This recorded time is "t" in eq. (6) below, and is used for the decay correction.

#### V. Treatment of Data

- A. Locate by channel number each peak in your gamma-ray spectra. Calculate the number of net photopeak counts (NPPC) in the 417-keV (largest)  $^{116m1}\text{In}$  photopeak for each sample according to

$$\text{NPPC} = \text{GPPC} - \text{BD} \quad (4)$$

where GPPC (the gross photopeak counts) equals the sum of the counts in all channels of the photopeak, and BD represents the counts of a linear baseline (background) under the photopeak. The data shown below illustrate how to calculate these various quantities (your instructor will demonstrate how to print out photopeak data). First, decide where the photopeak begins and ends by noting the peak channel with the maximum counts and then moving left and right until the counts start to level off. In the example shown, the peak starts at channel 143 and ends at channel 171, as noted by the brackets. If you are not sure of the exact beginning or ending channel, it is better to include an extra channel or two in the peak rather than have too few channels chosen. The sum of all the counts in channels 143-171 is GPPC.

The background under the photopeak is calculated according to

$$BD = \frac{N}{10}(C_L + C_R) \quad (5)$$

where  $C_L$  and  $C_R$  are the sum of the counts of five channels of the baseline to the left and right of the peak, respectively, and  $N$  is the number of channels in the photopeak. In the example below,  $C_L$  is gotten by adding the counts in channels 138-142,  $C_R$  is gotten by adding the counts in channels 172-176, and  $N$  equals 29. Note that if five channels of baseline are not taken, then the denominator (10) in eq. (5) must be modified. The calculated net photopeak counts for the example peak below are 21,526. (**Verify this.**) The photopeak count rate is obtained by dividing the net photopeak counts by the counting time (equal to 1000 sec for this experiment).

Sample photopeak data:

Channel #	Data							
130	110	99	112	91	86	103	101	88
138	( 99	94	82	90	83 )	[ 127	132	173
146	227	340	468	632	928	1168	1456	1738
154	1973	2113	2146	2069	1846	1636	1228	897
162	629	439	244	145	103	35	34	18
170	21	23 ]	( 7	14	17	10	8 )	7
178	12	9	8					

- B. Since your samples are not counted simultaneously, you must correct for the decay that occurs while waiting to count the second sample. The decay correction is given by:

$$R_0 = \frac{R_t}{e^{-(\ln 2) \frac{t}{t_{1/2}}}} \quad (6)$$

where  $R_0$  is the decay-corrected photopeak count rate for the second sample counted;  
 $R_t$  is the measured (uncorrected) photopeak count rate for the second sample;  
 $t$  is the elapsed time between the start of count of the first sample and the start of count of the second sample;  
 $t_{1/2}$  is the half life of the  $^{116m1}\text{In}$  activity.

- C. Using decay-corrected counting data, calculate the amount of indium (in grams) in your unknown according to eq. (2) and report this value along with your unknown number.

## VI. Questions

1. Using the results of your energy calibration, determine the energy corresponding to each observed photopeak for either your standard or unknown, and tabulate these results. Indicate which of these peaks result from  $^{116\text{m1}}\text{In}$  activity. (A comprehensive list of gamma-ray energies emitted by  $^{116\text{m1}}\text{In}$  can be found online at: <http://ie.lbl.gov/toi/nucSearch.asp>. Just enter the appropriate mass number and element symbol, then SEARCH.)
2. Why is each sample cleaned with a moistened Kimwipe before counting?
3. Why is it not necessary to use the same counting geometry for each of the standards used for the energy calibration?

## Lab Cautions for Neutron Activation of Indium

1. **IN ORDER TO MINIMIZE CONTAMINATION DURING THIS EXPERIMENT, WEAR RUBBER GLOVES WHILE HANDLING YOUR IRRADIATED SAMPLES AND CLEANING THEM AT THE DESIGNATED WORK AREA. REMOVE THE GLOVES BEFORE USING THE COMPUTER-INTERFACED DETECTOR.**
2. **When you first receive your irradiated samples, thoroughly wipe off the outside surface of each with a moistened Kimwipe. Samples are conveniently placed by and removed from the detector with forceps.**
3. **Dispose of all gloves, Kimwipes and irradiation vial sealing tape used in this experiment in the Radioactive Waste container. The irradiation vials, along with the irradiated samples, should not be disposed of in this fashion. Leave your irradiated samples at the work area for this experiment. DO NOT REMOVE THE TAPE WITH THE UNKNOWN NUMBER FROM THE UNKNOWN VIAL.**
4. **DO NOT EXIT THE GENIE-PC SOFTWARE WHEN YOU ARE FINISHED.**