Determination of Manganese and Aluminum in Tea via Instrumental Neutron Activation Analysis (INAA)

Objectives: This experiment will acquaint you with the method of thermal-neutron activation analysis and the quantitative determination of trace element contents.

References: Ehmann & Vance: pp. 253-279.

A high-purity germanium semiconductor detector, coupled to a 4096-channel multichannel analyzer (MCA), will be used to measure the gamma-ray activity of a tea sample, and Mn and Al standards, following irradiation in the UC Irvine nuclear reactor. Your instructor will demonstrate the operation of the gamma-ray spectrometry system. You should note the settings of all controls on the amplifier, ADC, and data handling modules and avoid altering these settings unless instructed to do so.

It has been found that dry tea leaves contain about 200-1200 µg Mn per gram of tea. Aluminum is present in a comparable amount. If a sample of tea leaves is irradiated for a short time (1 minute in this experiment) in the neutron flux of a typical research reactor, the major radionuclides produced are ^{56}Mn ($t_{½}$ = 2.576 h) and ^{28}Al ($t_{½}$ = 2.25 m). Other radionuclides are also generated during this irradiation (e.g., ^{38}C1, ^{24}Na), but their induced activity levels are all quite low, so that the observed gamma-ray spectrum for such a sample a short time after irradiation is due primarily to ^{56}Mn and ^{28}Al. The photopeaks of the 847-keV gamma ray of ^{56}Mn and the 1779-keV gamma ray of ^{28}Al will be used in this analysis.

If a sample containing a known amount of manganese, for example, is irradiated and counted in exactly the same way as a sample of tea leaves, then a comparison of the ^{56}Mn photopeak count rate for each sample enables one to determine the manganese concentration in a known weight of tea, since

$$\frac{R_{std}}{R_{sam}} = \frac{m_{std}}{m_{sam}} \qquad \text{(VII.1)}$$

where R represents the ^{56}Mn photopeak count rate and m the mass of Mn (see reference for derivation of this relationship). This also assumes that both samples are of similar size and composition. Eq. VII.1 can also be applied to an Al standard and the tea sample.

<u>BEFORE</u> COMING TO LAB: Calculate how much ^{56}Mn and ^{28}Al activity (in µCi) you would expect to get in a 1-minute irradiation of a standard containing 5 µg each of Mn and Al in a thermal-neutron flux of 10^{12} n/cm^2-s. Recall that the induced activity at the end of the irradiation is given by A_O(dps) = $N\varphi\sigma[1-\exp(-0.693t_i/t_{½})]$.

PROCEDURE

A. Sample and Standard Preparation

Accurately weigh about 50 mg of your tea sample into a half-dram polyethylene vial, snap it shut, cut off the top hinge with a razor blade and heat-seal the cap rim. Label your sample with a black felt-tip pen.

Prepare a Mn standard solution having a concentration of about 25 μg Mn per mL (you need to know this exactly). Accurately weigh out the necessary manganese as the metal, dissolve it in a minimum amount of dilute HNO_3 and dilute to volume with deionized water. Pipet 200 μL of this solution into another polyvial, heat-seal and label as before. Prepare an Al standard in a similar way, except dissolve the Al metal in a minimum amount of KOH solution and dilute with deionized water to a concentration of 20 μg Al per mL. You may also prepare a blank (polyvial only or polyvial + 200 μL deionized water) in a similar fashion.

B. Sample Irradiation

All of these samples will be taken to the UCI reactor facility and irradiated. Each sample will be irradiated for 1 minute in a thermal-neutron flux of about 10^{12} n/cm^2-s using the pneumatic transfer tube irradiation position. Upon receipt of your activated samples, monitor the radiation level with a portable survey meter and count each sample with the HPGe detector for approximately 2 minutes. You must be sure to use the identical counting geometry for both tea leaves and standards. Check to see that all of the standard solution is at the bottom of the vial. Also allow for the same delay between removal of your irradiated sample from the reactor and start of the count. When all counting is completed print out the analyzer data.

C. Analysis of Data

The analyzer software will report the location, energy and area of each peak in the spectrum. The number of net photopeak counts (NPPC) in each peak is calculated according to:

$$NPPC = GPPC - BD \qquad \text{(VII.2)}$$

and

$$BD = \frac{n}{2}\left[\frac{BD_L}{Ch_L} + \frac{BD_R}{Ch_R}\right] \qquad \text{(VII.3)}$$

where GPPC equals the gross counts in the photopeak and BD represents the background counts under the photopeak, obtained by finding the average background counts per channel on each side of the photopeak, averaging these values and multiplying by the number of channels (n) in the photopeak (BD_L and BD_R represent the total number of counts in the channels Ch_L and Ch_R to the left and right of the photopeak, respectively). If one chooses $Ch_L = Ch_R = n/2$, then $BD = BD_L + BD_R$.

The standard deviation of each NPPC value based on the counting statistics is given by:

$$\sigma_{NPPC} = \left(\sigma^2_{GPPC} + \sigma^2_{BD}\right)^{1/2} = \left(GPPC + \sigma^2_{BD}\right)^{1/2} \qquad \text{(VII.4)}$$

where

$$\sigma_{BD} = \frac{n}{2}\left[\left(\frac{\sqrt{BD_L}}{Ch_L}\right)^2 + \left(\frac{\sqrt{BD_R}}{Ch_R}\right)^2\right]^{1/2} \qquad \text{(VII.5)}$$

For $Ch_L = Ch_R = n/2$, $\sigma_{BD} = (BD_L + BD_R)^{1/2}$.

First, identify the source of each peak in your tea spectrum. Then use the peak areas for the 847-keV ^{56}Mn peaks and the 1779-keV ^{28}Al peaks in your sample and standards spectra, along with eq. VII.1, to calculate the amount of Mn and Al in your tea sample. Report the Mn and Al concentrations in μg/g sample. Also calculate the uncertainty of each concentration value based on counting statistics alone.