Experiment XX. Activation analysis as a tracer technique

In this experiment, we shall explore another tracer technique, the use of "induced tracers". Rather than injecting a radiotracer into a system under study and following its dispersal, we shall transmute some of the atoms of a material into radioactive atoms and follow their dispersal. We shall use neutron activation as a means of inducing the radioactivity and as a sidelight, we will determine the trace element content of the system under study using the techniques of activation analysis.

The basic principle of tracer work is that the tracer must behave exactly the same as the material being traced. This means the tracer must generally be in the same chemical, physical and biological state as the material being traced. In ordinary tracer work, the fulfillment of this goal may require extensive chemical manipulations before the actual tracing takes place to insure the tracer and material being traced are in the same oxidation state, have the same speciation, etc. In this experiment, we shall carefully choose the method of irradiation of the sample to minimize any disruption of the chemical and physical state of the material.

The model systems to be studied are commercial vitamin preparations. These pills contain trace metals at the ppm-ppt levels. By neutron activation, we will transmute many of the minerals present into radioactive species. The following table shows the expected trace element levels in a typical vitamin pill (which has a mass of 1.5 g). Also shown are the expected levels of radioactivity induced in the pills after a seven hour irradiation in the thermal column of the Oregon State University TRIGA reactor. (Irradiation in the thermal column is chosen to maximize the flux of thermal neutrons and to minimize the flux of fast neutrons. Fast neutron reactions can cause the activated atoms to be displaced, chemical bonds to be broken, or oxidants to be created in the material being irradiated. The activation products from such reactions might be expected to show different chemical behavior than the unirradiated atoms. Irradiation with pure thermal neutrons minimizes these effects.)

Element	Pill Content (g)	Activation Product	EOB Activity (Bq)	One week activity (Bq)
Ca	0.200	Ca-49	1.8e06	0
P	0.048	P-32	8.0e05	5.7e05
I	0.00015	I-128	1.5e06	0
Mg	0.100	Mg-27	3.2e06	0
Zn	0.015	Zn-65	1.5e04	1.5e04
Se	0.000002	Se-75	64.2	62
Cu	0.002	Cu-64	6.6e06	698
Mn	0.002	Mn-56	8.6e07	0
Cr	0.00015	Cr-51	3150	2649
Mo	0.000075	Mo-99	364	63
Cl	0.072	Cl-38	3.8e07	0
K	0.080	K-42	1.3e07	1056
Ni	0.000005	Ni-65	198	0
V	0.00001	V-52	2.0e05	0
Fe	0.018	Fe-59	1086	974

As one can see from examining the table, the activity levels of the irradiated pill are quite high, due primarily to the presence of ^{38}Cl and ^{56}Mn. We shall let the irradiated pills decay for one week prior to initiating the experiment to reduce the activity levels used in the experiment. We shall count the irradiated sample using gamma ray spectroscopy and compare the activities observed with a natural standard, orchard leaves, with known trace element content, which was irradiated under the same conditions as the pills. From the ratio of the activities of the pills and the orchard leaves sample, we will determine the absolute trace element content of the vitamin pills. As an example of the type of experiment one might do with the induced tracers, we will measure the dissolution rate of the pills in a simulated gastric fluid. Further investigations of the uptake of the induced tracers in animal systems are possible.

Purpose of the Experiment

(a) To determine the trace element content of commercial vitamin pills.
(b) To simulate possible release and uptake studies using the induced tracers by a simple determination of the rate of dissolution of the pills and their mineral contents.

Experimental Procedures

1. Weigh 4 pills of each of ten commercial vitamin preparations. Package the pills for irradiation in the thermal column of the OSTR.
2. Weigh and prepare for irradiation a sample of the NIST standard Orchard Leaves. The sample size should be similar to the size of the pills to insure similar geometries in irradiation and counting.
3. Irradiate the sample in the thermal column of the OSTR for seven hours in a neutron flux of 3.5×10^{11} neutrons/cm^2-sec. Store the samples for 10 days prior to beginning the experiment.
4. Select two of the irradiated pills for each vitamin preparation for assay by gamma ray spectroscopy. Count the pills with a Ge gamma ray spectrometer for one hour each to determine the activities present. Position the pills relative to the detector to insure the sample deadtime is 10% or less. Count the Orchard leaves sample in the same geometry as the pill. For each gamma ray line of interest, calculate the abundance of the associated nuclide using the equation

   $$\text{Amt-pill} = \text{Amt-OL} \times (\text{Act.-pill/Act-Ol})$$

   where the activities refer to measurements taken in the same geometry and corrected for decay. The trace element content of the Orchard Leaves standard can be obtained from your instructor.
5. Take the remaining two pills from each preparation for the simulated tracer study. Place 50 mL of simulated gastric fluid in each of four 150 ml beakers (use pH 1.3 HCl). Prepare ~15 2-dram polyvials, labelling them appropriately (see below). Practice using a 5 mL pipet to withdraw samples of liquid from the beakers, making sure the solution volume in the beakers remains 50 mL. Place an irradiated pill in beakers 1 and 3. Note the time. Begin stirring the beakers at 50-100 rpm. At intervals of 5,15,25,35,45,and 55 minutes after beginning, withdraw a 5 mL sample from beaker 1, pipetting it into a clean polyvial and replacing it with 5 mL from

beaker 2. Do the same for beaker 3 (using beaker 4 for the makeup solution) at intervals of 10,20,30,40,50, and 60 minutes after beginning.

6. Count the samples for one hour using a gamma ray spectrometer for their ^{65}Zn content (or the content of other trace elements if you use a Ge detector). Plot the amount of tracer released as a function of time for each preparation, taking an appropriate average of the samples for each dissolution.

Report

1. Report the trace element content of each preparation as given on the label of the bottle and as determined by activation analysis. Comment on any discrepancies.
2. Report the fraction of each trace element that is released in the dissolution experiment and the time dependence of the release for each vitamin preparation. Comment on any unusual features of the data.