Preparing Thin Liquid Targets for Ion Beam Analysis

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Motivation

Ion Beam Analysis techniques such as PIXE, PIGE, and RBS are used to determine the trace elemental composition and concentrations of unknown samples. Liquid samples taken from the environment cannot be put directly into a particle accelerator thus, in order to test liquid samples by use of the 1.0 MV tandem electrostatic Pelletron particle accelerator the samples must be prepared and dried. Hence, there is a need for an accurate and reliable procedure to prepare liquid targets.
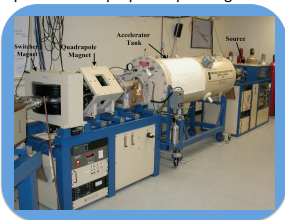

Figure 1: 1.0 MV Tandem Electrostatic Pelletron Particle Accelerator- Used to generate a beam of high energy particle (1.8MeV protons in energy).

Experiment

Union College's tandem electrostatic Pelletron particle accelerator, shown in Figure 1, was used to generate a *1.8-MeV* beam of protons that was steered to be incident onto a dried red wine target and $Q = 10$-μC of charge was collected. When the protons interact with elements in the target there is a probability, called the cross-section, that inner shell electrons will be ejected and that higher orbital electrons will de-excite to fill these vacancies accompanied by an emission of an x-ray photon. This is called Proton Induced X-ray Emission Spectroscopy (*PIXE*)[1] and a schematic is shown in Figure 2[1]. Each element has its own unique set of x-ray energies associated with electron transitions and this allows us to fingerprint each element. To fingerprint the elements we generally look for *K*-series x-rays, which are electron transitions from any orbital higher than the ground state to the ground state of the atom. The lower energy, higher probability $n = 2$ to $n = 1$ transition is termed a K_α transition, while the higher energy, lower probability $n = 3$ to $n = 1$ transition, is called a K_β.

PIXE Technique
Using Ion Beam from Accelerator

Electron Knocked Away

Incident Proton from Accelerator

Proton Out to Faraday Cup

Outer Electron Moves In to fill vacancy

Gives off X-Ray

Atom with two of its orbiting electrons shown

PIXE - Proton Induced X-ray Emission

Catch and measure the X-ray.
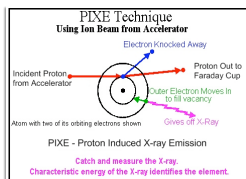Characteristic energy of the X-ray identifies the element.

Figure 2: Drawing showing the basics of the *PIXE* process[1]. Here the ground and first excited states of an arbitrary atom are shown. The inner shell electron is ejected by the proton and a higher orbital electron transitions from the $n = 2$ to the $n = 1$ state with an emission of an x-ray photon. This is called a K_α transition.

The X-ray spectra are then collected on a silicon drift detector and are displayed as intensity versus x-ray energy and are analyzed to determine the trace elemental composition of the target with a software program called GUPIX[4]. Using trace elemental standards, GUPIX[4] will be calibrated to give concentrations of selected trace elements in the red wine target. GUPIX[4] calculates the concentrations C_{target} from the x-ray yields of the sample and target, Y_{sample} and Y_{target}, the amount of charge collected, Q, the x-ray detector efficiency, e and an overall energy independent constant, H shown in the equation below.

$$C_{\text{target}} = \frac{Y_{\text{target}}}{Y_{\text{standard}} \times H \times Q \times \varepsilon \times t}$$

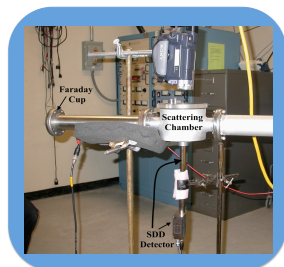Faraday Cup

Scattering Chamber

SDD Detector

Figure 3: Silicon Drift Detector

Experimental Procedure

We need to prepare a thin target from a liquid sample in order to perform a PIXE analysis to determine composition and concentration. From the literature we picked the three most common pre-concentration methods for preparing liquid samples and will compare these results with un-concentrated samples. We chose red wine as a liquid to work with because it has been suggested that there is Pb in wine and there is a wide elemental distribution.

Method #1- following the example of Ghorai[5]
➢Two 40mL beakers were acid washed using nitric acid.
➢10mL of each wine (Merlot and Cabernet) were measured into each beaker and the wines were slowly evaporated at 70º-80ºC for approximately 6 – 7 minutes to concentrate the samples to approximately 5mL.
➢8 µL of each wine were pipetted onto a 12 µm Mylar backing and the samples were dried under vacuum. [Time: 19hrs 40min]

Method #2- following Kennedy[3]
➢10mL of each wine (Merlot and Cabernet) will be measured into acid washed beakers and the wines will be heated approximately 6 – 7 minutes to concentrate the samples to approximately 5mL and the wine will be pipetted onto two Nucleopore filter papers (25 mm diameter –pore size and 10 microns thick)
➢The filter papers will be dried for approximately 20 minutes under a heat lamp.

Method #3- following Henrici[2]
➢For this method we will prepare each sample of wine by freeze drying. After freeze drying, the samples will be prepared as thin targets which can be run in the accelerator.

Sample Preparation

Homemade Cabernet and Merlot red wines from Lodi, California were prepared according to Ghorai[5] and Kennedy[3] and then a PIXE analysis was performed using GUPIX[4].
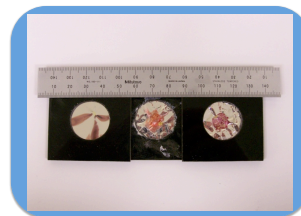

Figure 4:The first slide is a sample of plain 12 µm Mylar backing. The following two are the two wine samples which were dried under vacuum following Ghorai[5].
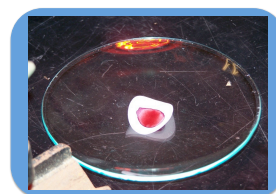

Figure 5: The above image is a sample of 5mL of red wine which was placed onto an 80µm thick Nucleopore filter paper and dried under a heat lamp following Kennedy[3].

Preliminary Results

Figure 6:The counts per channel are higher for the concentrated than un-concentrated Merlot. Thus, to produce the most definitive and accurate results clearly the liquid targets should be concentrated. This spectrum is for 3µC of charge collected.
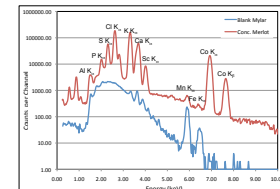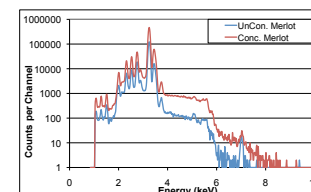
Element	Concentration (ng/cm²)
Al	5500
Si	450
P	1700
S	5600
Cl	18400
K	17800
Ca	7500
Mn	132
Co	18800

Figure 4: PIXE Spectrum of Concentrated Merlot dried onto a 12 micron Mylar backing and overlaid with the spectrum from a non-deposited Mylar Backing. A total charge of 10 µC was collected.
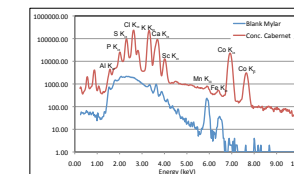
Element	Concentration (ng/cm²)
Al	1300
Si	660
P	3100
S	9600
Cl	23200
K	25300
Ca	11200
Mn	105
Fe	145
Co	20300

Figure 5: PIXE Spectrum of Concentrated Cabernet dried onto a 12 micron Mylar backing and overlaid with the spectrum from a non-deposited Mylar Backing. A total charge of 10 µC was collected.

The preceding results are merely for Method #1 of the chosen preconcentration methods. It is still necessary to prepare the liquid targets following Method#2 and Method#3 to check for reproducibility and accuracy.

References

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3. Kennedy, J.V. Trompetter, W.J. Barry, B. Markwitz, A. NIM B, 258, 435-439.
4. GUPIX, University of Guelph. http://pixe.physics.uoguelph.ca/gupix/main/
5. Trace Elemental Composition and Acidity of Central-Alabama Rain. Ghorai, S.K. Tekyi-Mensah, O. Sims, J.F. Williams, J.R. Alford, W.L. NIM B, 63, 139-142.
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