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Abstract

Neutron Activation Analysis is a method of determining the composition of a sample wherein the sample is irradiated, then measured through gamma ray spectroscopy. This method can also be used to determine the mass of a particular substance within an unknown sample by comparing the gamma ray spectroscopy results to that of known results and applying a predetermined linear relationship between known sample mass (or molarity) and the primary peak area form the gamma ray spectroscopy results. This relationship is what our project set out to determine for differing concentrations in a standard volume of Cadmium Nitrate solution ($CdNO_3$). We found a linear relationship to within an acceptable standard deviation, based on Molarities ranging from .001 to 1 Molar.

Introduction

Neutron Activation Analysis (NAA) is a method of sample analysis by which the composition of samples and relative concentrations of different isotopes within the samples can be determined. It relies on the principle that certain elements become radioactive when exposed to a source of neutrons. These elements then emit gamma rays at specific energies according to the particular isotope emitting. The gamma rays emitted are then analyzed through a process called gamma ray spectroscopy. Each isotope that is able to emit Gamma radiation will have a signature spectrum containing peaks, or elevated readings of gamma rays at specific energies. By comparing these peaks to data from known sources of radiation, one can reasonably deduce the composition of a particular sample. Also, by analyzing the area of a peak and subtracting out the background, a relation can be made between the corrected peak area and the mass of a particular element within a sample. This procedure can be very useful when analyzing unknown samples, but it is not a very simple procedure to set up. Establishing the relation between peak area and sample mass depends greatly on particular equipment, neutron flux, composition of the sample, and a standardized process.

The original aim of the project was to use NAA to determine the amount of Cadmium (Cd) picked up by gel samples washed in a Cadmium Nitrate solution, using a standardized process devised by Bren Phillips. It became quickly apparent that, to verify the results, a standard mass to peak area relation would be needed for Cadmium. Using set volumes and differing concentrations of Cadmium Nitrate ($CdNO_3$), Bren and I set out to build such a relation.

Discussion

Neutron Activation Analysis (NAA)

NAA was discovered in 1936 by Hervesy and Levi as a method to determine the presence of rare earth elements in rock samples[1]. The method uses a neutron source, usually a reactor, to excite an atom to a particular isotope. That isotope then emits gamma rays at specific energies, which are detected by the process of gamma ray spectroscopy.

Procedure

The procedure for the analyzation of a sample is as follows:

Sample Preparation

(1) A solution is made at a certain molarity of $CdNO_3$. We started the molarity of the samples in factors of ten (.01M, .1M, 1M) then began to fill in the gaps. A 1mL sample is placed in a 2mL plastic vial.

(2) Then place the small vial in one of the small zip-lock bags.

(3) Open the old rabbit vials used for the last round of irradiations and extract the centering jigs. The vials and all of the contents are hot so do your work handling the vials and the jigs on the hot bench.

(4) Place the centering jigs into new rabbit vials. Be careful to not contaminate the outside of the new rabbit vial while placing the centering jig in the vial.

(5) Place the small vial in the centering jig inside the rabbit vial and make sure it is firmly seated.

(6) Adjust the extra plastic from the small bag the 2 ml vial is in to act as packing when the lid is placed on the rabbit vial

(7) Close the rabbit vial lid and heat seal.

(8) Repeat steps (1) through (7) for all 6 vials

(9) Clean the outside of the rabbit vials with alcohol

(10) Label the vials with CdNO3 and numbers starting with the next number after the last vial that was irradiated.

(11) Check the calibration on the Ge detector and recalibrate if necessary.

(12) Irradiate the vial for 30 seconds at 2 KW, but be sure the power history of the reactor is not such that it will affect the instrumentation.

(13) When you hear the Nitrogen discharge to shoot the sample back up start a stopwatch.

(14) Remove the sample from the glovebox and take the 1-foot dose reading and report to the operator as required.

(15) Place the rabbit vial in the over pack with the centering sleeve inside.

(16) Place the over pack on the Ge detector by using the aluminum-centering jig and close the lid.

(17) Start a 10-minute live time count 2 minutes after the sample returned from the core.

(18) Save the spectrum and repeat steps (12) through (17) for all 6 samples

(19) Analyze the two peaks in the spectrum by calculating the net peak area using Equation 12.17 in Dr. Tsoulfanidis' *Measurement and Detection of Radiation* book[2]. This is also programmed in an Excel data sheet. This equation just takes a straight line from the background on each side of the peak and subtracts those counts. This step will be discussed in the next section.

(20) Make adjustments to the procedure to improve accuracy, such as decreasing the mass irradiated to increase power and irradiation time to prevent gamma flux from influencing instrumentation.

(21) Use the data collected to irradiate samples with unknown amounts of Cadmium in them and determine the amount.

Gamma Spectrum Analyzation

The method alluded to in Step 19 will be elaborated upon in this section. On the Gamma Ray Spectrum for Cadmium Nitrate, a peak will appear at 245 Kiloelectron-volts. This is the primary energy at which Cadmium emits gamma rays. There are other secondary energies that can be analyzed, but the peaks for these will be smaller, and therefore not as conducive to accurate results. For this study, only the peak at 245 keV will be analyzed.

To analyze the peak, the data must be separated into channels. Enough channels must be used on either side of the peak to include the background so that it may be subtracted from the original peak area. To determine the number of channels needed, the width of the peak at half of its maximum was determined. This number was defined to be э, and the uncorrected peak area was defined to be the summation of all the peaks within 3 times this number (3э), centered at the peak. An additional 1.5э on either side of this area determines the background. The average is taken over all of the background peaks, and that number times 3э is subtracted from the uncorrected peak area to yield the corrected peak area. (*figure 1*)[2]

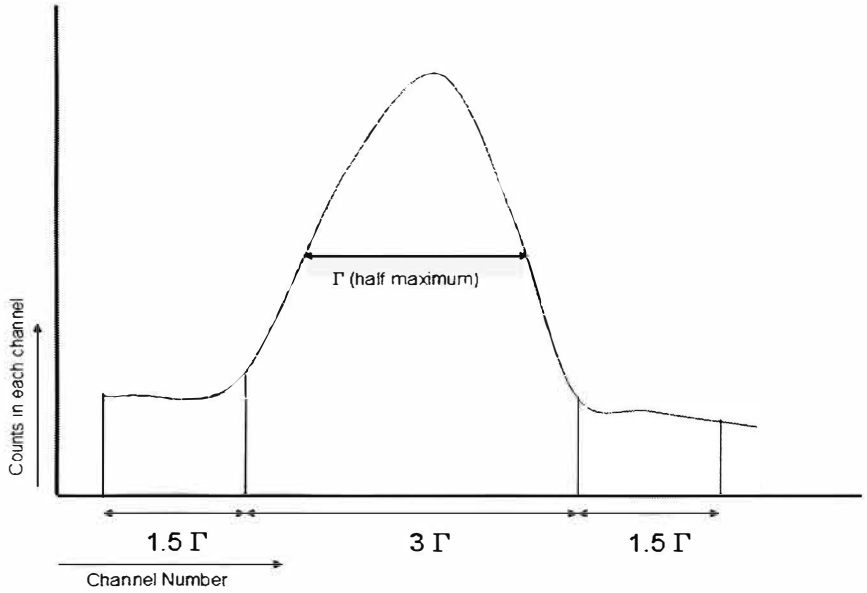

***figure 1*. Gamma Spectrum Analyzation**

Results

Several Samples were analyzed for each Molarity, and the average peak value was calculated for each. The standard deviations stayed below 4% for the most part. By graphing the average corrected peak areas for each molarity versus the molarity value, a linear relationship was discovered (*figure 2).* The data for the graph is shown in *table 1.*

Molarity of CdNO3	Average Corrected Peak Area (245 keV)
0.001	643.5
0.01	736.8
0.1	7117.4
0.25	18563.6
0.5	32336.9
1	54106.67

Table 1. Avg. Peak areas for Molarities of $CdNO_3$

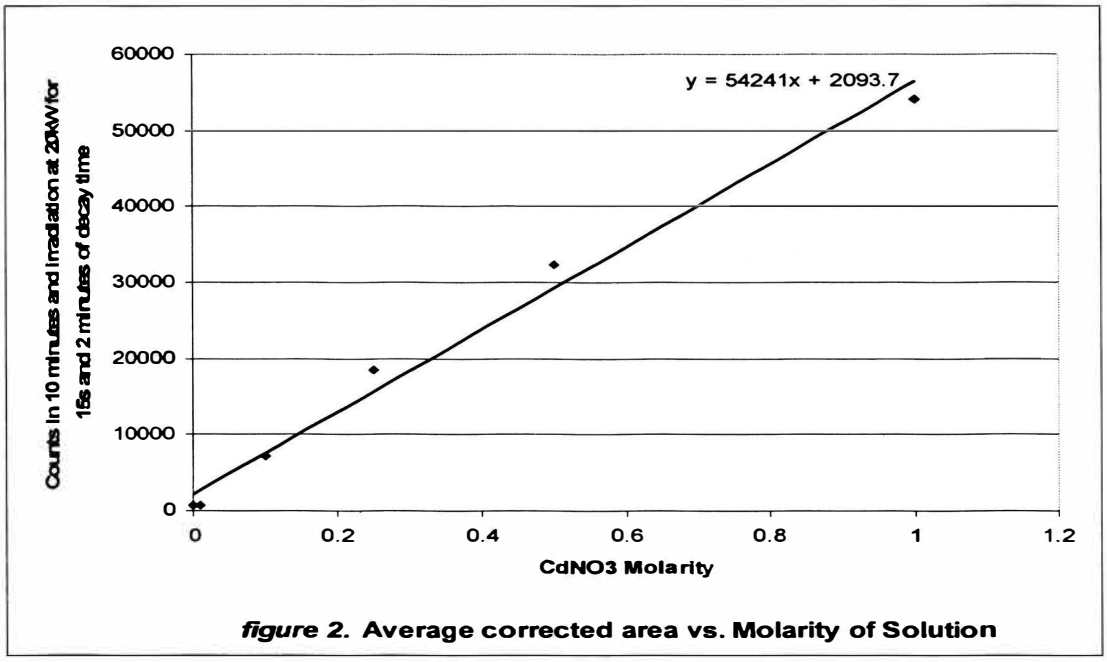

figure 2. **Average corrected area vs. Molarity of Solution**

The linear relationship inferred from our results is represented by the trendline on the graphed results in *figure 2*. The formula for this line is (approximately):

$$y = 54200x + 2100 \qquad \{1\}$$

More experimentation would be needed to reduce the uncertainty of this relationship for practical use, but the results are encouraging.

Conclusion

In conclusion we have shown a linear relationship between the molarity of Cadmium Nitrate and the peak areas of the Gamma Spectrum produced after an exposure time of 15 seconds, standardized, and a decay time of 2 minutes. This relationship will be useful when analyzing samples containing an unknown mass of Cadmium Nitrate. Unfortunately, preliminary experimentation has shown that additional substances irradiated with the $CdNO_3$, such as a silica aerogels severely alters the gamma spectrum results. Future experimentation will be needed to address this concern.

Acknowledgements

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References

[1] Michael Glascock, An Overview of Neutron Activation Analysis, http://www.missouri.edu/~glascock/naa_over.htm

[2] Nicholas Tsoulfanidis, Measurement and Detection of Radiation, Second Edition, 1995, Taylor and Francis