



SATHYABAMA

INSTITUTE OF SCIENCE AND TECHNOLOGY
(DEEMED TO BE UNIVERSITY)

INTERSHIP REPORT
FOR
MASTERS IN CHEMISTRY

BY

THULASI RAMAN A
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**INTEGRATED RADIATION MONITORING FACILITY
(IRMF)**

**INDIRA GANDHI CENTRE FOR ATOMIC RESEARCH
(IGCAR)**

KALPAKKAM



INTERNSHIP REPORT BY:

THULASI RAMAN A

REG NO. : 42910024

INTERNSHIP COMPLETION CERTIFICATE

Mr. THULASI RAMAN A doing Second year M.Sc (Chemistry) in the Sathyabma

Institute of Technology has undergone an internship course for 12 days from 11/12/2023 to

22/12/2023 at the **INTEGRATED RADIATION** MONITORING FACILITY, ANUPURAM.

The student was sincere, regular and showed keen interest in LEARNING and Executing the assigned works.

The internship provided are

- Basics of radioactivity
- Radio analytical procedure for the quantification of radioactivity content in the urine samples of radiation workers
- Preparation of samples for Radiometric counting and efficiency calibration.
- Hands-on experience to use Alpha Spectrometer.
- Radiation safety aspects to work in radiological laboratories.

Dr. S.Chandrasekaran Dr.D.Ponraju

Head, Health and Industrial Safety Division

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IGCAR

INTEGRATED RADIATION MONITORING FACILITY-ANUPURAM

Estimation of Uranium (^{233}U) using Alpha Spectrometer

1. Kalpakkam Nuclear Complex consists of Reactors, Fuel Reprocessing Plants, Waste Management Plants and other Radiological R & D Laboratories. In these facilities safety is given top most priority. occupational workers are well trained in radiological and industrial safety aspects. However, radiological exposures to a very few personnel cannot be ruled out in extreme cases due to human error. Integrated Radiation Monitoring Facility located at Anupuram has been established to monitor internal exposures of any worker. Sophisticated advanced instruments and well established procedures are available for the measurement of radioactivity content in the body even if it is a trace quantity. Workers are periodically monitored by collecting the urine samples and subjected to chemical treatment and radiometric counting.
2. **we have learnt the chemical treatment procedures for the estimation of uranium - 233 in spiked urine samples.**
3. **we have learnt the procedure of electro deposition on a ss disk.**
4. **we have learnt the functioning of alpha counting system for the estimation of radioactivity in the given sample.**
5. **we have also learnt how to handle state of the art sophisticated alpha spectrometer to identify the alpha emitting radionuclides in the given samples.**

radioanalytical chemistry lab staff demonstrated and explained the chemical treatment procedures. we were instructed to prepare chemical reagents required for the work such as : 6.2n hcl, 0.2n H_2SO_4 , 1:1 ammonium oxalate, ammonium hydroxide independently. these were checked by their laboratory assistants and found to be in good agreement. wet oxidation, pre concentration, co precipitation, ion exchange column, electro deposition, alpha spectrometry are the steps involved in the quantification of radioactivity content in the samples.

Wet Oxidation :

The Urine Sample Collected Is Poured In A 2 L Beaker And The Volume Is Noted. Then 50 Ml HNO_3 And 10 Ml H_2O_2 Is Added In Sequence. This Content Is

Heated Continuously For About 6 To 7 Hours. This Step Is Completed When A Pale Yellow Colour Appears.

I Was Shown Wet Oxidation Procedure.

Co Precipitation:

The Above Sample Is Stirred On Adding 200 Mg Hydroxylamine Hydrochloride, 50 Ml - 90 Ml Ammonia With 0.5 Ml Calcium Carrier Using A Magnetic Stirrer For About 1 Hour And The Precipitate Is Allowed To Settle. After Half An Hour The Precipitate And Supernatant Is Separated And Supernatant Is Thrown Out. To The Precipitate 8m HNO_3 Is Added And Heated Until A White Colour Precipitate Is Formed. To This Precipitate 200 Mg Sodium Nitrate And 10 Ml Of 8m HNO_3 Is Added And Stirred For 30 Minutes. The Content Is Then Filtered.

Ion Exchange Column:

The Ion Exchange Column Containing Dowex Resin Is Conditioned With 8m HNO_3 And HCl. The Sample Filtered From Coprecipitation Is Loaded In The Column Such That 5 Drops Per Minute Flows In To The Column. Then The Plutonium Is Collected By Loading 1.5n $\text{NH}_2\text{OH.HCl}$ And 1.2n HCl

Then We Were Taught How To Do Electrodeposition .

Electrodeposition:

I Did The Electro Deposition Process By Adding The Prepared Electrolyte (Hydrochloric Acid, Sulphuric Acid, Ammonium Oxalate And Ammonium Hydroxide) In Respective Quantities. The Electrolytes And The Sample Obtained After The Ionic Exchange Process Are Mixed Thoroughly Using A Glass Rod And The Contents Are Poured Into Electrodeposition Setup. The Current Is Set At 0.60 Amperes. The Current Must Be Constant Throughout The Process. The Sample Undergoes Electrolysis For 2 Hours And 45 Minutes After The Completion Of The Time The Set Up Is Turned Off And The

Planchette Is Taken Out Carefully And Placed In A Petri Dish And Heated For 10 Minutes And Cooled Down For 15 Minutes. The Planchet Is Taken For Alpha Counting.

I Carried Out Electrodeposition Process Under Their Supervision.

Alpha Counting:

I Placed The Planchet Inside The Chamber Of The Alpha Counter Using A Glass Slide. Then The Dc Voltage Of 50v And Vacuum Pressure Of 100 Torr Is Set As Configuration. And The Time For The Counting Process Is Set As 86,400 Seconds And The Sample Is Proceeded For Alpha Counting By Clicking *Run*. After The Completion Of Set Time The Configuration Is Turned Off In A Reverse Manner. The Screen Displays A Spectrum With X-Axis In Energy (Kev) Vs Y-Axis As Alpha Counts (Cps) The Sharp Peak At 4.8 Kev Energy Level Shows That The Radioactive Element Is Uranium 233 And Its Alpha Counts Are 237 And 259 Respectively. With These Alpha Counts The Activity Of The Sample Is Calculated Using The Following Formula.

$$\text{Activity} = \text{Cps (Measured)} - \text{Cps (Blank)} / \text{Efficiency}$$

We Were Taught The Basics Of Alpha Activity Estimation Using Alpha Counting System.

Outcome Of Internship:

1. **Learnt A Lot About The Basics Of Radioactivity, Monitoring Methods, Quantification, Safety Precautions Involved In Various Stages Of Sample Analysis.**
2. **Exposure To Handle Advanced Instruments.**

Acknowledgement:

We Sincerely Acknowledge Director Igcar , Director Hseg, Head Hisd, And The Officials Of Integrated Radiation Monitoring Facility At Anupuram For Providing A Very Good Opportunity For Us To Learn Entirely A New Topic.We Are Grateful To The Staffs Of Irmf For Their Valuable Guidance And Necessary Assistance. We Got A Good Inspiration To Take Up A Employment Carrier In Department Of Atomic Energy Which We Are Eagerly Looking For.

Alpha spectrometry is an effective tool for the identification of alpha emitting radio nuclei in any given sample. This technique is commonly used to obtain the uranium composition in urine samples. The uranium isotope measurements at low level activity concentration are of great interest in nuclear fields. A proper Uranium source for alpha spectrometer must be prepared by Electrodeposition method at constant current mode. A combination of these two techniques- electrodeposition followed by alpha spectrometry using planar detectors (PIPS) can produce a very good resolution up to 19.5 KeV at 26 mm STDD. Alpha source produced by electrodeposition consists of a thin uniform radioactive deposit onto a stainless steel cathode (Electrodeposition cell cathode).

The in-vitro Bioassay techniques at Integrated radiation monitoring Facility (IRMF) is used for the determination of radionuclides present in the excretion samples like urine and faeces. In IGCAR, the occupational workers from Compact Reprocessing Facility for Advanced Fuels in Lead Cell (CORAL), Demonstration Fuel Reprocessing Plants (DFRP) and Chemical Group (CG) are involved in the reprocessing of spent carbide fuels of Plutonium and Uranium. During the course of plant operation, there is a chance of radionuclides may become air borne and pose inhalation hazard to occupational worker incidentally or accidentally. Urine assay is the preferred method of estimating internally deposited Uranium. Routine monitoring of occupational workers at various nuclear facilities in IGCAR are being carried out by Bioassay Laboratory of HISD Division. The analysis of alpha emitters in urine samples is difficult because very low activity of radioisotopes is excreted in urine following the intake. An anion exchange method followed by alpha spectrometric measurement is used to estimate U present in urine is adopted which is being used for the analysis of U in urine samples. This procedure is chosen due to its ease of handling and consistently good recovery obtained with tracer. At this context, the isotopes are separated by co-precipitation and further purified by passing through anion exchange resin. Afterwards, it is deposited on stainless steel disc and this source is used for alpha spectrometric measurements.

In the present internship project work, designing and developing of electrodeposition cell for uniform plating of uranium isotope which is leak proof and simple in construction and operation has been reported. The present study was used to quantify the Uranium activity concentration present in the sample. The goal of the work was used to prepare uniform thin alpha source with good spectral properties and maximum percentage yield of the study. The

report discusses the estimation of Uranium (^{233}U) spiked activity present in the sample using alpha spectrometer.

Materials and Methods

2.1 Instrumentation

Alpha spectroscopic measurements of samples were done with an alpha spectrometer with Maestro basic alpha spectrometry software and a vacuum pump (Edward model RV8). Each chamber has a Passivated ion implanted planar silicon (PIPS) detector of surface area 450 mm^2 with $100\text{ }\mu\text{m}$ depth. The Maestro software was used for the alpha spectral analysis. The resolution of the detector is 19.5 KeV (FWHM) at source to detector distance of 26 mm which is equal to detector diameter. The counting efficiency of the PIPS detector at the closest distance is approximately 21% for all the eight chambers.

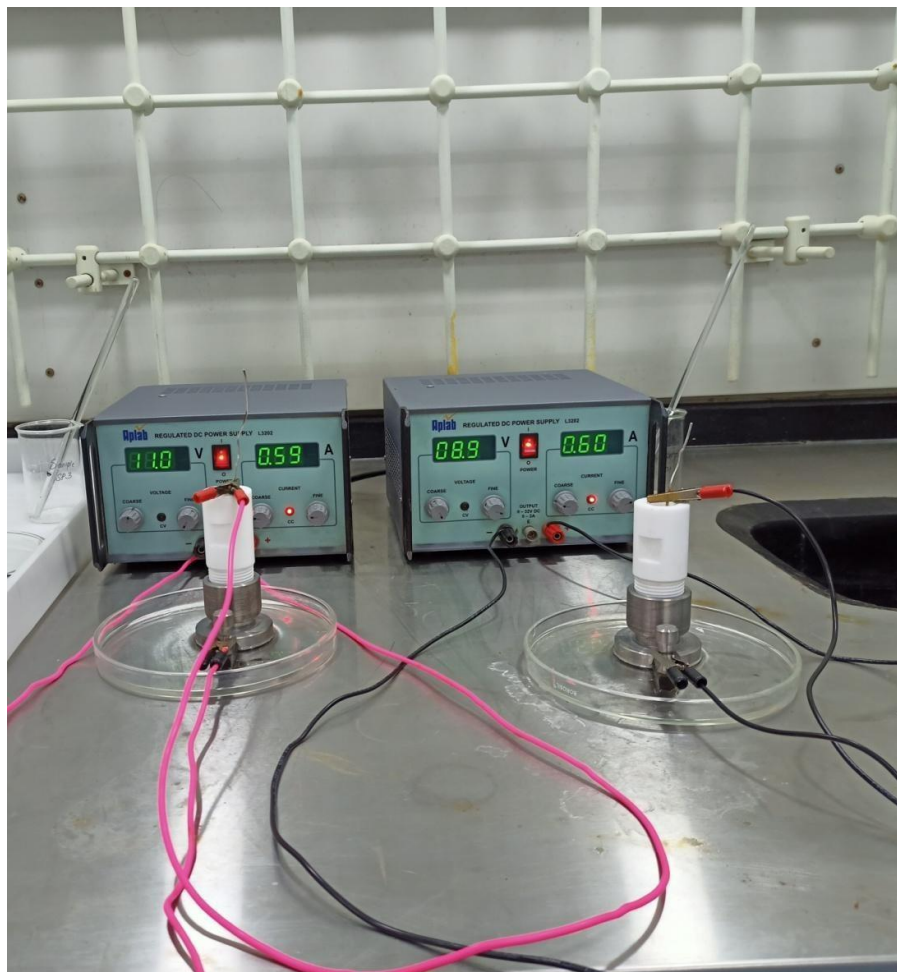


FIG 1:ALPHA COUNTING INSTRUMENTATION

2.2 Electro deposition device

An Electro deposition cell made of Teflon was used for the uniform deposition of Pu activity in urine. During the process, Plutonium isotopes were electrochemically deposited on the cathode (stainless

steel disc, thickness: 1 mm; diameter: 25 mm), which was placed at the bottom of the cell. The platinum electrode was used as anode. The effective area of the electro deposition on the disc was 2.14 cm². The DC Regulated Power Supply (Aplab Model: 3005) was used as an external electrical source during the reaction. The schematic representation of Electrolytic Cell is depicted below.



3. Samples

Two spiked samples (Code No: SP-1 and SP-2) of each 1000 ml were spiked with known activity of ²³³U in mBq level to estimate recovery factor of each sample.

4. Tracer

²³³U tracer obtained from the National Physical Laboratory (NPL) was purified to remove its daughter product ²²⁸Th. Purified activity was estimated by alpha spectrometry and stock solution of tracer having known activity (48.76±1.8mBq/ml) was prepared.

5. Chemicals

All chemicals were of analytical reagent grade and used without further purification.

3 Experimental

A Known activity of ^{233}U were spiked in clean dry 150 ml beaker and evaporated to dryness the candidates were asked to estimate activity of electro deposition method using alpha spectrometry. Blank experiments are also conducted to determine the background activity in the same ROI.

1. Electrodeposition of Uranium samples

- Leak test of the cell was conducted by putting 10 ml of distilled water into it and keep for half an hour. If leak is not found, pour out the water and preserve for the cell for electrolytic deposition.
- Add 0.5 ml of 6.2N Hydrochloric acid solution and 1.4 ml of 0.2N Sulfuric acid solution (Electrolytes) to the beaker.
- Warm up the solution and transfer the electrolytes effectively to the cell.
- Wash the beaker thrice with each time 3 ml of 5% Ammonium oxalate solution by rubbing thoroughly the sides of the beaker with glass rod.
- Transfer all the washings effectively to the cell.
- Keep Spiral Platinum electrode inside the cell at 0.5 mm distance above the S.S.planchette.
- Connect the DC power supply to cell by **Stainless steel body as cathode and platinum electrode as anode.**
- Start the electro deposition reaction at a voltage of **6V** and a constant current of **0.6 amps.**
- Continue the reaction for 2 hours 45 minutes.
- Add 0.5 ml of 1:1 NH_4OH solution to the cell before stopping the current.
- Continue the reaction for 1 minute and switch off the DC power supply.
- Allow the cell to cool down for half an hour.
- Dismantle the parts of electrolytic cell slowly.
- Remove the planchette carefully from the cell.
- Wash the planchette with distilled water then with 99 % alcohol.
- Heat the planchette for 10 minute at a temperature of 40°C .
- Allow the planchette to cool down for 1 hour

- Label the planchette with same identification number and preserved it for alpha counting.

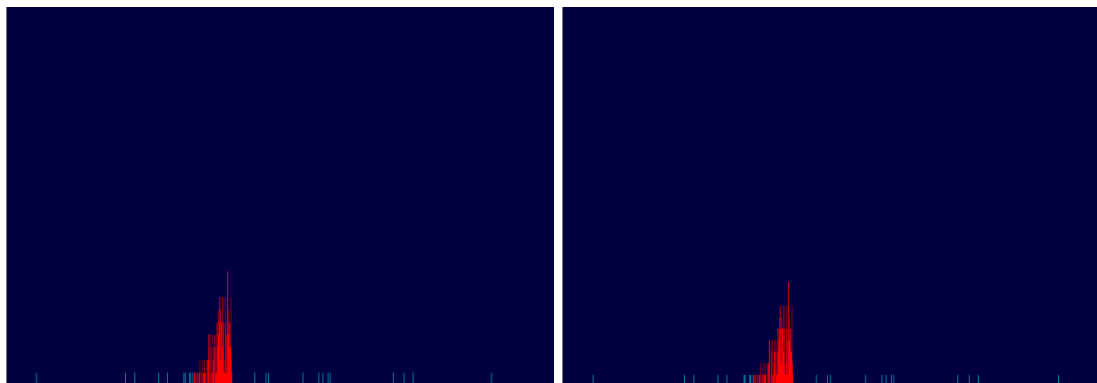
3.2 Estimation of Uranium activity concentration using Alph spectrometry

The electroplated plutonium samples were counted using Passivated Ion implanted Planar Silicon (PIPS) detector.

1. Place the mixed Electroplated planchette containing the unknown activity on the 1st rack (6 mm STDD).
2. Open the MAESTRO MCA application software. Go to the MCB properties and select parameters like preset time, amplifier, ADC and vacuum state of the ALPHA ENSEMBLE.
3. Activate the vacuum pump by selecting the pump mode from the pull down menu of MCB properties. Wait for the vacuum gauge to read < 100 mTorr.
4. After the desired vacuum pressure is obtained, put on the bias voltage and wait for at least 10 minutes (for warming up of electronic components).
5. Stabilize all the parameters before starting the energy calibration.
6. Acquire spectrum in such a way that counts in the peak channel exceeds 1000 counts. For mixed alpha source live time preset of 300 seconds is selected.
7. After acquiring spectrum, select the region of interest (ROI) in the energy range of 4620 to 4896 KeV.
8. Note down the Gross are counts in the marked ROI and save the spectrum for the further studies.

SPECTRUM 1

SPECTRUM 2



A blank experiment also conducted to estimate the background counts of samples in the alpha spectrometric estimation of actinides.

4 Result and Discussion

The present work is used for the estimation of the alpha activity concentration of Uranium present in the spiked sample (Code No: SP1 & SP2).

Table 1 : The activity present in the spiked samples

Sample Code	Alpha Chamber	Average Gross counts in the ROI (C1)	Average Background counts in the ROI (C2)	Calculated Activity (mBq)
SP 1	309 Input 1 (D1)	259	1	14.21(+,-)0.88mBq
SP2	309 input 2 (D2)	237	1	13.00(+,-)0.84mBq

Figures 3 & 4 shows typical alpha spectra of spiked sample with known activity of ^{233}U tracer solution. ^{233}U peak marked in the Region of Interest (ROI) of the alpha spectrum is 4620 - 4896 KeV.

4.1 Estimation of Activity concentration of ^{233}U in spiked samples

The activity of recovered ^{233}U tracer is given by the following formulae

The activity obtained for ^{233}U is . The counts were taken from Region of Interest (ROI) of the alpha spectrum of ^{233}U (4620 - 4896 keV). The Samples are counted for 86400 seconds.

References:

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