

INTERNSHIP REPORT

BY

NAME: HARIHARAN.R

PURSUING DEGREE: M.Sc.CHEMISTRY

DEMEED UNIVERSITY:SATHYABAMA



भारतसरकार
GOVERNMENT OF INDIA
परमाणु ऊर्जा विभाग
DEPARTMENT OF ATOMIC ENERGY



सत्यमेव जयते

Dr Rajini P Antony
Scientific Officer –D
WATER AND STEAM CHEMISTRY DIVISION
भाभा परमाणु अनुसंधान केंद्र सुविधाएँ
BHABHA ATOMIC RESEARCH CENTRE
FACILITIES
कलपक्कम **KALPAKKAM-603 102**
Phone : 044-27480500-23349
Email : rajiniantony@igcar.gov.in
raji.anna@gmail.com

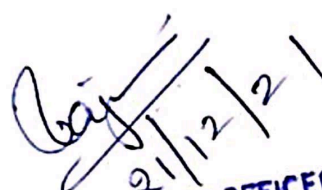
21-12-2021

To whomsoever it may concern

Mr Hariharan R, Student, MSc Chemistry, Sathyabama Institute of Science and Technology did his internship under my supervision from 6th December 2021 to 21st December 2021 at water and steam chemistry division, Bhabha Atomic Research center, Kalpakkam facilities. This letter is issued for whatever official purpose it may serve.

Yours

Dr Rajini


21/12/21
SCIENTIFIC OFFICER / ENGINEER - E
WATER & STEAM CHEMISTRY DIVISION
Government of India
B. A. R. C. Facilities
DAE, KALPAKKAM - 603 102

DECLARATION

I here declare that the internship report submission entitled “**SYNTHESIS AND EVALUTION OF LAYERED METAL SULFIDE FOR REMOVING METAL ION RELEVANT TO NUCLEAR INDUSTRY**” it has been submitted by me (HARIHARAN.R “40910002”) in the partial fulfilment for the award of degree of Master of Science in Chemistry, Deemed university “SATHYABAMA INSTITUTE OF SCIENCE AND TECHNOLOGY” the internship carried out by me during the period from **6th December 2021 to 21st December 2021** under the external guide of **Dr. Abdual Nishad** in Water and Steam Chemistry Division, Bhabha Atomic Research Center Facilities, Kalpakkam.

HARIHARAN.R

ACKNOWLEDGEMENTS

First my honourable thank to my guide “**Dr. Kavitha**” who should offer an chance to do my internship and project in “**Bhabha atomic reach center**”, My Second thanks proudly said to my external guide “**Dr. Abdual Nishad**” who is an employe in “**Bhabha atomic reach center**”, The above mentioned opinions are explored and declared my knowledge.

ABOUT COMPANY

Some history relavant to “**Bhabha atomic reach center**”. Dr. Homi Jehangir Bhabha conceived the nuclear program in india. He established the **Tata Institute of Fundamental Research (TIFR)** for carrying out nuclear science research in 1945. Dr. Bhabha established the Atomic energy Establishment , Trombay (AEET) in january 1954 for

multidisciplinary research program essential for the ambitious nuclear program of India. After mourning demise of Bhabha in 1966, AEET was renamed **Bhabha atomic reach center (BARC)**.

BARC is the mother of R&D institutions such as **Indira Gandhi Center for Atomic Research (IGCAR)**, **Raja Ramanna Center for Advanced Technology (RRCAT)**. **Variable Energy Cyclotron Center (VECC)**.

In BARC in it “ **Water Steam and Chemistry Division**”, My internship and project has been progressing.

My project topic is “**SYNTHESIS AND EVALUTION OF LAYERED METAL SULFIDE FOR REMOVING METAL ION RELEVANT TO NUCLEAR INDUSTRY**”.

Nuclear Reactor

Nuclear reactors are the heart of a nuclear power plant.

In world wide more than 454 nuclear power rectors and 226 nuclear research reactors in operation

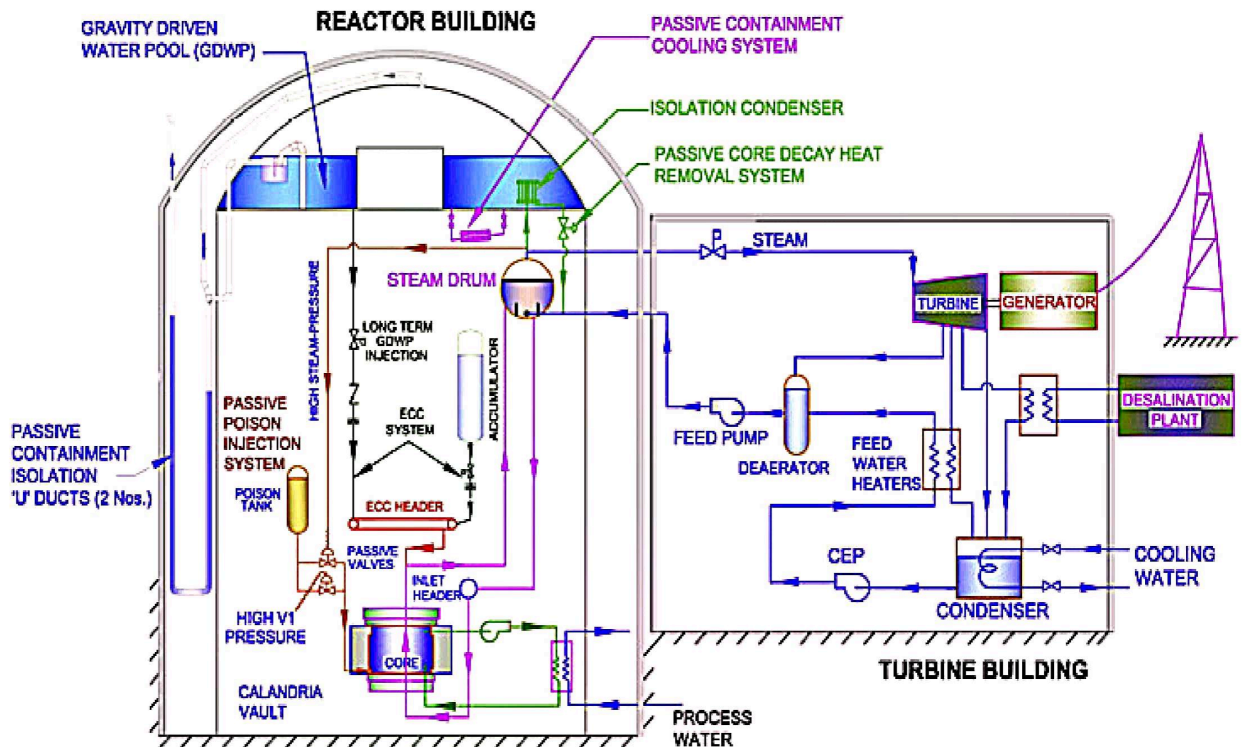
around the world including 94 nuclear reactor in united states these reports from **International Atomic Energy Agency (IAEA)**.

Type of nuclear reactor:

- ☒ Boiling Water Reactor (BWR)
- ☒ High Temperature Gas cooled Reactor (HTGR)
- ☒ Pressurized Water Reactor (PWR)
- ☒ Pressurized Heavy Water Reactor (PHWR)
- ☒ Advanced Gas cooled Reactor (AGR)
- ☒ Light Water Graphite Moderated Reactor (LWGR)
- ☒ Fast Neutron Reactor (FNR)
- ☒ Prototype Fast Breed Reactor (PFBR)

From this the most commonly used reactor in India is (PWR).The water in the core is heated by nuclear fission and then pumped into tubes inside a heat exchanger. Those tubes heat a separate water source to create steam. The steam then turns an electric generator to produce electricity.

But in IGCAR has an Pressurized Heavy Water Reactor (PHWR)



The Pressurized Water Reactor used an enriched uranium fuel it is responsible for a high power density within the reactor core. In PWR contain 100 tonnes of uranium refuelling is carried out by removing the above for safety purpose to avoid an accident. The PWR has a generating capacity of 1000mW. It efficiency is around 33%. PWR is typical most popular reactor in world around approximately 292. The most important commercial PWR was developed by westinghouse for ship

propulsion and later converted to power generation. The Russians developed their own model of PWR called VVER and units of this type continue to operate in Russia and former Soviet countries. France also developed a PWR which was based on the Westinghouse design but the design later diverged so that the French is now an independent design.

Some of the advantages are following: PWR reactors are very stable due to their tendency to produce less power as temperature elevates. This makes the reactor to operate from a stability standpoint. The primary loop in PWR turbine cycle and loop so the water in the secondary loop is not contaminated by radioactive materials.

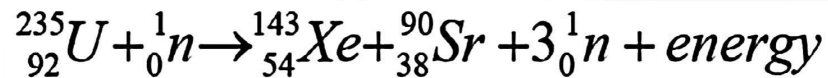
PHWR (Pressurized Heavy Water Reactor) is similar to PWR. But some of the very slight difference between PWR and PHWR. Pressurized Heavy Water Reactor commonly uses unenriched natural uranium as its fuel and in this we use heavy water (Deuterium Oxide, D_2O) as a coolant and moderator. The heavy water coolant (D_2O) is kept under pressure without boiling, allow it get heated at

high temperature. Typically it has seen in PWR. Since heavy water is cost wise more expensive compared with light water it enhance the greater yield of neutron allow the reactor to operate without fuel enrichment facilities and generally intensify the ability of the reactor to efficiently make use of alternate fuel cycle.

In PHWR has an pressurized coolant is pumped through a reactor core, being heated by the nuclear fuel assemblies in the core. Then steam generators has tube bundle the coolant is follow in it. After steam generator release the heat to a pool of lower pressure water. This pool of lower pressure water turns to steam which is used to drive the electrical turbo generator.

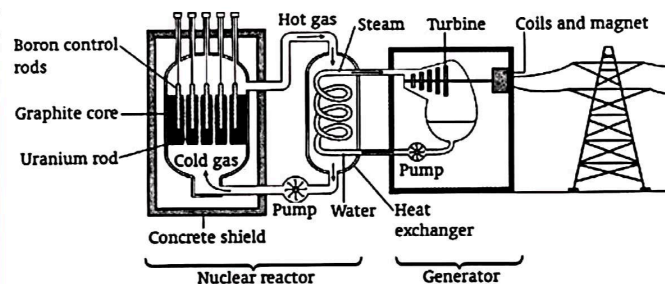
At nuclear fission from the uranium we get an fission product as

When a neutron hits a uranium-235 nucleus, causes it to split producing strontium-90, xenon-143 and three neutrons + energy.



Use of nuclear fission

Electricity can be generated from the energy released by fission reactions. A nuclear power station consists of a nuclear reactor and a generator.



This ${}_{38}\text{Sr}^{90}$ is found in spent fuel rods in nuclear reactors and is considered a waste product. Strontium-90 is radioactive form of Strontium. The half life of strontium is 29 years. Strontium found in nature is not radioactive and is sometime called stable strontium.

The people will be exposed through breathing, drinking or touching strontium-90. People are consuming grain, dairy products and drinking water but chemical form of strontium

does not dissolve in water easily, a small amount may remain in the lungs.

Human causes:

People eat food or drink water containing strontium it causes when enter into blood it flows to other parts of the body. It enters and leaves cells easily. In the body, strontium acts like very much calcium. A large portion of attaches to the surface of bones. In children, strontium may create the hard bone mineral it stored in the bones for many years. The harmful effects of strontium-90 are caused by high energy of radiation. Since radioactive strontium is taken up to the bone, the bone itself and nearby soft tissues may be damaged by radiation released over time. Bone marrow is the most important source of red blood cells, which are exhaust if the strontium-90 level is too high. Some cancer patients injected strontium-90 to destroyed the cancer tissue in the bone marrow. But some problem are facing from red blood cell counts include anemia, which causes excessive tiredness, blood that does not clot properly and

a decreased resistance to fight disease due to this disadvantage we should reduce the radioactivity of strontium-90.

We have a solution to decrease the radioactivity of $^{90}_{38}\text{Sr}$ is an essential to reducing the risk of human exposure to radiation and for the considerable cost savings and minimization of the storage space requirements for the nuclear waste. $^{90}_{38}\text{Sr}$ can be found in the reactor coolant system of light water at nuclear power plants and are present in both spent nuclear fuel and in the liquid HLW found in tank farms at DOE sites. The technologies for removing strontium such separation technologies developed for liquid wastes are; solvent extraction, fractional crystallization, inorganic ion exchange and organic ion exchange. The most liquid waste processing system using ion exchange media. There are two types of appropriate ion exchange media such as Organic ion exchange and inorganic ion exchange.

But Organic ion exchangers must be synthesized using multi-step organic synthesis

processes, and typically have lower exchange capacity compared with their inorganic counterparts. Inorganic ion-exchangers offer a superior chemical, thermal and radiation stability but it is not achievable with organic compound. However, inorganic ion exchange material can be produced using a straightforward process compared with organic ion exchange materials.

Some of the **disadvantage** for organic ion exchanger why we not use more, **lower exchange capacity compared with inorganic exchangers, both excessive swelling and peptide formation, lower radiation and thermal stabilites**. For the above reasons to take an consideration we using inorganic ion exchangers.

Inorganic ion exchangers:

These material can be easily synthesized using readily available compounds using either a solid-state or hydrothermal synthesis techniques. But we used hydrothermal synthesis techniques for good yield compared with solid state synthesis.

The potentially elutable materials based upon layered metal sulfide with ion exchange interlayer cation has been developed. For example of such polycrystalline material are KMS-1 or $K_{2x}Mn_xSn_{3x}S_6$ has been shown to be an exceptionally efficient Sr-ion remover over a wide range pH.

Experimental part using Hydrothermal synthesis:

The determined amount of for an example; Sn (15mmol, 1.780g), Mn (7.5mmol, 0.412g), S (45mmol, 1.443g), K_2CO_3 (7.5mmol, 1.036g) these all are weighted and transferred into the mortar pestle for milling at the same time take some amount of ultra pure water (UPW) through that passed an argon gas for removal of oxygen. Then milled

powder compound will be transferred into clean teflon (PTFE) pour some argonated water into the teflon then closed tightly while using teflon tap. After teflon contain should be kept into the stainless steel autoclave. The autoclave must be checked sealed tightly then placed into the electrical heating thermostatic blast (cylindrical furnace) set with determined temperature and time at 200°C for 96hrs. After 4 days take an teflon contain then cooled over the room temperature it reach room temperature open the teflon poured the slurry material into separate beaker to make sure about completely transfer using distilled water (DMW). Then wash several times with distilled water at same time centrifugate then after washed with distilled water then washed with acetone and diethyle ether for single wash with centrifugation. After completion of washing we will place the sample into vacuum air oven for dried at determined temperature 70°C for over night. Finally we get brown polycrystalline product. Then few milligram of sample should be taken into 10ml beaker in that some 3ml of aqua regia will be added and at the same time made up multi std. Solution

(all element in one compound) at different ppm in 50ml SMF. Then after dissolved in aqua regia it is made up into 50ml SMF. Then it should be placed for ICPAES analysis.

ICPAES:

Inductively coupled plasma atomic emission spectroscopy (ICPAES), it is also referred to as inductively coupled plasma optical emission spectrometry. It is a spectral method used to determine very precisely the element composition of samples it can also be used to quantify the elemental concentration with the sample.



Principle:

Inductively coupled plasma atomic emission spectroscopy (ICP-AES) is also referred to as inductively coupled plasma optical emission spectrometry (ICP-OES) it is an analytical technique used to determine the amount of element present in the sample. The atoms or ions can absorb energy to transmit electrons from the higher level either from excited levels and excited levels to ground level. In ICPAES, the source of energy is heat from an argon plasma that operates at 10,000 kelvin. A excited

atom releasing light at a specific wavelength as they shift to the lower energy level.

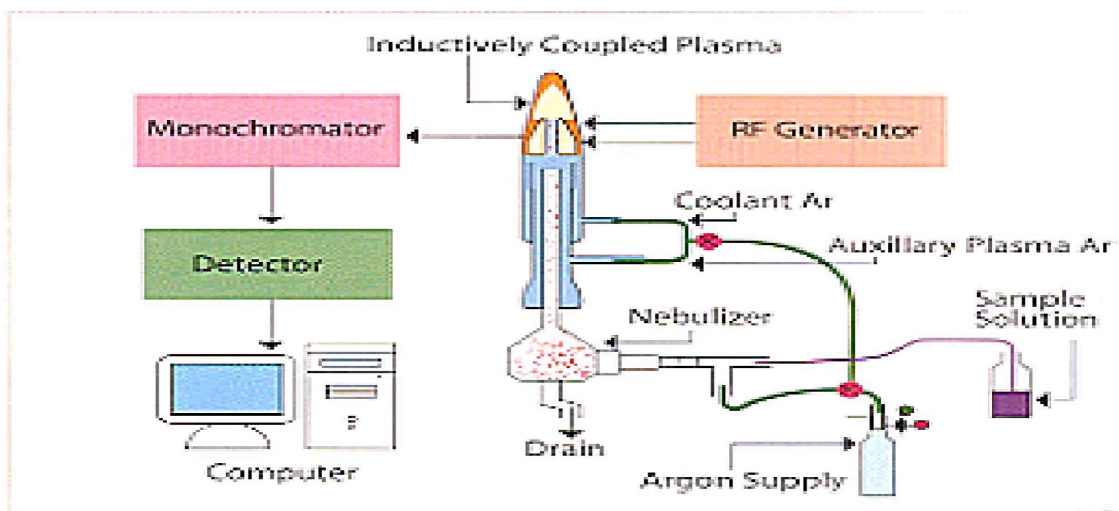
Atom and energy level picture

When electron it emits a very specific wavelength due to the electron returns back from higher energy level to lower energy level (ground state). The qualitative analysis was done by wavelength of the emitted radiation, and then quantitative analysis was done by the intensity of the emitted radiation. At each wavelength the amount of light can be emitted it is directly proportional the number of atoms and ions transition. The Beer Lambert law describes the relationship between light intensity and concentration of the element. The hands to say intensity of emitted light will be proportional to the concentration of the element present in the sample.

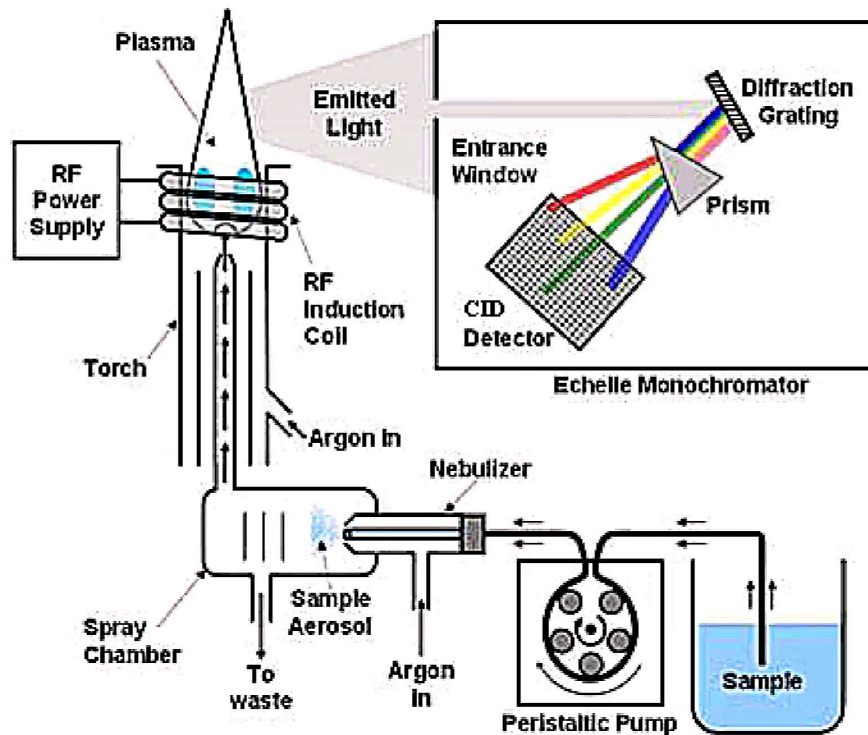
INSTRUMENTATION:

ICPAES is an emission spectrophotometric technique, exploiting the fact that excited electron emit energy at a given wavelength as they return to ground state after excitation by high temperature Argon plasma. It means excited levels are occupied by thermal energy we observe radiative de-excitation between

levels of superior energy to levels of lower energy. First the sample should be passing by fine tube in ICPAES then it goes through a nebulizer (droplets size from 1 to 100 micrometer) as spraying fine mist (aerosol) then mist in cyclonic nebulisation spray chamber it combines with argon gas which also passes through a nebulizer then it goes to the upper part of plasma it needs to get external energy through an electric field in order to ionize the gas which will transmit part of the energy to the sample it started by seeding electron through a spark.



Then emitted light source is enter into the monochromator. It is an detectors. It preformed as practical resolution,speed & spectral range.



Then we get result from the signal while conected through the pc as the result of calibration curve.

Advantages of Monochromator:

- ☒ It improved analysis precision and accuracy.
- ☒ Corrects for viscosity effects and changes in plasma conditions

- ☒ Monitors signal at the same time as the analyte elements.

Advantage of ICPAES

- ☒ Multi element analysis
- ☒ More than 70 elements can be analyzed
- ☒ High dynamic range
- ☒ Low running cost
- ☒ High speed technique

Drawbacks ICPAES:

- ☒ Usually requires sample in a liquid form
- ☒ Sample preparation can be long
- ☒ Limits of detection sometimes too high
- ☒ Spectral interferences.

Conclusion:

Layered metal sulfide such as KMS-1 are highly specific, high capacity and potentially elutable inorganic ion exchangers suitable for the removal of strontium from liquid radioactive wastes. Further testing and verification studies will involve the development of further

modified media for other isotopes and applications. Producing these ion specific media in a cost effective manner will be demonstrated in the next phase of development.

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