

## REFERENCE MATERIALS FOR CHEMICAL ANALYSIS OF URANIUM AND PLUTONIUM

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### Introduction

Optimum performance of a reactor depends on the quality of the fuel loaded into it. Accurate chemical analysis of uranium and plutonium in the nuclear fuels is an essential activity in the quality assurance programme. The analytical methods developed for chemical assay have to be regularly checked for their reliability by analysing certified chemical reference materials which are well characterised compounds of well-defined stoichiometry and high purity. Besides, these materials ensure uniformity in performance in different laboratories and can be used for routine checking of method performance and elimination of bias.

A reference material (RM), as defined in ISO guide 30 (1992), is a material or substance, one or more of whose property values are sufficiently homogeneous and well established to be used for calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials. Reference material, accompanied by a certificate, one or more of whose property values are certified by a procedure which establishes its traceability to an accurate realisation of the unit in which the property values are expressed, and for which each certified value is accompanied by an uncertainty at a stated level of confidence, is known as Certified Reference Material (CRM).

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Uranium and plutonium reference materials are produced to calibrate reagents used in titrimetric methods or to check instrumental techniques like electrochemical methods. High purity uranium metal,  $U_3O_8$  and sintered and depleted uranium dioxide pellets have been prepared as elemental reference standards for uranium. Uranium metal and  $U_3O_8$  are listed as reference materials for uranium as SRM 960 and SRM 950b respectively by National Bureau of Standards (now known as National Institute of Standards and Technology, NIST).

For plutonium, high-purity metal has long been accepted as an RM. It was formally available as a standard reference material from NIST, as SRM-949e. Although NIST no longer produces or supplies this material, it is available as a certified reference material (CRM) from New Brunswick Laboratory, as NBL-CRM 126. Plutonium metal is also available from Commissariat à l'Energie Atomique, France, as CRM-MP2. Another plutonium reference material is made available in the form of  $PuO_2$  powder as European Community certified nuclear reference material EC-NRM 210. Harwell Laboratory of the United Kingdom Atomic Energy Authority has also reported the availability of a Pu-Ga alloy reference material (UK Pu1 80990) containing  $(98.075 \pm 0.0017)$  wt % plutonium. Pu-Al and compounds  $Cs_2PuCl_6$ ,  $Pu(SO_4)_2 \cdot 4H_2O$  and its anhydrous salt have also been evaluated for their use as RMs.

Metal RMs are readily oxidised in air, are difficult to cut in massive form, and are hard to clean if they are in the form of turnings. For this reason, uranium and plutonium metal standards are stored and supplied in the glass ampoules sealed in inert atmosphere. Once the vial is opened and the surface is cleaned, the entire sample should be used. The oxides of uranium and plutonium undergo deterioration on long storage in the atmospheric conditions.  $U_3O_8$  should be heated to  $900^\circ C$  before use to achieve the stoichiometry and  $PuO_2$  must be calcined at  $1250^\circ C$  for two hours, before weighing. Besides, such high-fired materials are chemically inert and can be dissolved only with difficulty.

Though  $Pu(SO_4)_2 \cdot 4H_2O$  and its anhydrous salt have been evaluated as standard reference material (SRM) for plutonium, the corresponding sulphates of uranium do not satisfy the necessary requirements of a standard material. Hydrated uranium sulphate does not have a defined degree of hydration and hence not considered suitable as a standard assay material. Also, anhydrous  $U(SO_4)_2$  is found to be unstable at ambient conditions. Detailed physico-chemical evaluation of the stability, stoichiometry and purity of several alkali metal uranium (IV) and plutonium(IV) double sulphates done in Fuel Chemistry Division eventually resulted in the identification of  $Rb_2U(SO_4)_3$  and  $K_4Pu(SO_4)_4$  as suitable reference materials for uranium and plutonium respectively.

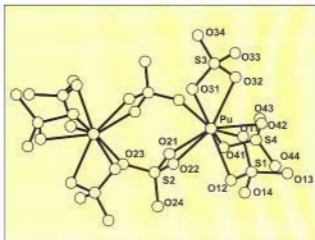


Fig. 1. Structure of  $[Pu_2(SO_4)_8]^{4-}$  anion in  $K_4Pu(SO_4)_4 \cdot 2H_2O$

Though uranium salt crystallises from solution as anhydrous salt,  $K_4Pu(SO_4)_4$  is prepared by heating  $K_4Pu(SO_4)_4 \cdot 2H_2O$  at  $250^\circ C$ . The structural study on  $K_4Pu(SO_4)_4 \cdot 2H_2O$  has shown that the compound can be better represented as  $K_8Pu_2(SO_4)_8 \cdot 4H_2O$  and that the water molecules are not coordinated to the plutonium atoms. The removal of the water molecules by heating at  $250^\circ C$ , therefore, does not affect the structure of the  $[Pu_2(SO_4)_8]^{4-}$  ion as shown in Fig. 1. This was confirmed from the infrared spectral studies which showed that the spectra of anhydrous and hydrated compounds are similar except for the presence of absorption peaks due to water molecules in the dehydrate.

## Preparation of RMs for Uranium and Plutonium

The preparation of RMs required a great deal of planning prior to undertaking actual activity in the project. A substantial part of the planning deals with the amounts of material needed, as well as with the design of homogeneity, stability and characterization studies. The design also includes the choice of appropriate measurement methods for these studies.

RMs for uranium and plutonium were prepared in three different lots from high-purity materials in acid-leached quartz wares by adding respective alkali sulphates to the solutions of uranium(IV) and plutonium (IV) in sulphuric acid medium. Further purification of the compound was achieved by recrystallising twice the materials from 1.0 M  $H_2SO_4$ . The crystallised compounds had the molecular composition as  $Rb_2U(SO_4)_3$  for the uranium salt and  $K_4Pu(SO_4)_4 \cdot 2H_2O$  for the plutonium salt. The crystals were washed several times with absolute alcohol and dried in a current of air.  $K_4Pu(SO_4)_4 \cdot 2H_2O$  was dehydrated at  $250^\circ C$  to obtain anhydrous  $K_4Pu(SO_4)_4$ . In this way,  $Rb_2U(SO_4)_3$  and  $K_4Pu(SO_4)_4$  were prepared in three different lots and stored in glass bottles in a desiccator.

## Characterization of RMs

Characterization of the finally produced reference materials is one of the most important activities before their use in chemical analysis. Final characterization of  $Rb_2U(SO_4)_3$  and  $K_4Pu(SO_4)_4$  was carried by using following techniques:

X-ray diffraction

- Thermogravimetry
- UV-visible spectroscopy
- Infrared spectrometry
- Emission spectroscopy
- Redox titrimetry
- Gravimetry

Characterization and evaluation of the RMs of uranium and plutonium was done for:

- Stability
- Stoichiometry and purity
- Homogeneity
- Solubility

## Stability

The stability studies of RMs were performed on samples stored in a desiccator over a period of two years under repeatable conditions similar to those needed for homogeneity studies. Thermogravimetric patterns showed that both the RMs are stable upto  $500^\circ C$ . TG and DTA patterns of  $Rb_2U(SO_4)_3$  are shown in Fig. 2. The materials stored in vials showed no mass change and the uranium and plutonium content determined in  $Rb_2U(SO_4)_3$  and  $K_4Pu(SO_4)_4$  respectively by redox titrimetric methods at regular intervals showed no significant differences at all confidence levels.

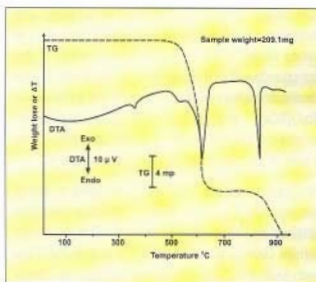


Fig. 2 TG and DTA curves of  $Rb_2U(SO_4)_3$

## Stoichiometry and purity

Valence state of uranium and plutonium in  $Rb_2U(SO_4)_3$  and  $K_4Pu(SO_4)_4$  was established as +4 by dissolving the sample in  $H_3PO_4$  and recording their UV-visible spectra. The IR spectra showed the absence of water of hydration in  $Rb_2U(SO_4)_3$  and

**Table 1 : Results of all the five analysts expressed in uranium % in  $Rb_2U(SO_4)_3$**

	Protentiometry			Amperometry			Coulometry		
	D1	D2	D3	D1	D2	D3	D1	D2	D3
Analyst A1	34.163	34.174	34.182	34.200	34.193	34.224	34.259	34.201	34.135
	34.161	34.163	34.177	34.214	34.193	34.082	34.190	34.254	34.233
	34.129	34.146	34.129	34.199	34.172	34.199	34.172	34.085	34.139
Analyst A2	34.185	34.240	34.185	34.198	34.215	34.255	34.206	34.157	34.208
	34.243	34.173	34.180	34.198	34.186	34.250	34.195	34.197	34.208
	34.147	34.164	34.220	34.185	34.180	34.242	34.207	34.203	34.227
Analyst A3	34.178	34.169	34.220	34.180	34.137	34.176	34.160	34.147	34.182
	34.223	34.160	34.177	34.183	34.149	34.190	34.171	34.164	34.170
	34.178	34.178	34.203	34.182	34.170	34.168	34.132	34.179	34.133
Analyst A4	34.178	34.330	34.186	34.026	34.091	34.227	34.147	34.163	34.164
	34.161	34.141	34.203	34.056	34.137	34.110	34.180	34.135	34.180
	34.174	34.142	34.157	34.022	34.141	34.156	34.158	34.151	34.153
Analyst A5	34.139	34.088	34.176	34.111	34.231	34.257	34.178	34.184	34.226
	34.114	34.102	34.083	34.145	34.140	34.178	34.191	34.241	34.163
	34.121	34.126	34.163	34.143	34.166	34.179	34.154	34.210	34.235

D1 = Dissolution 1, D2 = Dissolution 2, D3 = Dissolution 3

$K_4Pu(SO_4)_4$ . Uranium content in  $Rb_2U(SO_4)_3$  and plutonium content in  $K_4Pu(SO_4)_4$  agreed well with the values expected values for the stoichiometry of the compounds within the precision of the method. The chemical analysis of sulphate and for rubidium in  $Rb_2U(SO_4)_3$  and potassium in  $K_4Pu(SO_4)_4$  further established the stoichiometry of the compounds. The results of chemical analysis are given in Table 1.

The purity of the products was determined by taking into account the concentration of trace metallics obtained by spectrometric methods. The total trace metallic constituents in  $Rb_2U(SO_4)_3$  and  $K_4Pu(SO_4)_4$  was less than 300 ppm of the sample. X-ray diffraction patterns of all the three lots were identical, and showed the absence of any other phase.

### Homogeneity

Homogeneity testing is of prime importance for certification and use of RMs. In practice, a material is accepted to be homogeneous with respect to a given characteristic if the difference between the value of

this characteristic from one part (or unit) to another cannot be detected experimentally. A determination of analyte homogeneity in RMs was performed prior to assignment of metal content in RMs by repetitive determination of uranium content  $Rb_2U(SO_4)_3$  and plutonium content in  $K_4Pu(SO_4)_4$  by electrometric methods to confirm that the variations between bottles were neither statistically nor practically significant compared to the certified uncertainty ( $\pm 0.1\%$ ) so that an appropriate result about the level of homogeneity is obtained.

All the three lots were found to be homogeneous and lot to lot variations were found to be insignificant. Subsequently, all the three lots were mixed into a single lot.

### Solubility

The solubility data on  $Rb_2U(SO_4)_3$  and  $K_4Pu(SO_4)_4$  was obtained in HCl,  $H_2SO_4$  and  $HNO_3$  by equilibrating the compounds in different concentrations of three acids at room temperature and in boiling



water bath.  $Rb_2U(SO_4)_3$  and  $K_4Pu(SO_4)_4$  are soluble in all acids and the maximum solubility was observed in 2 to 3 M nitric acid.

## Assignment of Value to Uranium and Plutonium Content

The certification or assignment of concentration data which approaches as closely as possible the 'true value' together with uncertainty limits, is one of the most important requirements for the use of RMs. Two broad types of approaches were used for the assignment of uranium content in  $Rb_2U(SO_4)_3$  and plutonium content in  $K_4Pu(SO_4)_4$ . The statistical approach relies on in-depth application of statistical calculations to a body of analytical results obtained from diverse exercises.

A necessary condition for certification of constituent concentrations is that determination must be made by at least two independent, complimentary, valid and reliable methods based on more than one principle of measurement to avoid systematic errors associated with any particular method or technique. When the results from two, three or more reference methods agree, one can have a high degree of assurance that they are likely to be accurate and to have more than one analyst/laboratory involved to avoid possible specific biases. The approach required development of a statistical plan for sampling and measurement, selection of reliability methodology of known demonstrable accuracy, maintenance of statistical control of measurement process, and quality assessment of the data by concurrent measurement of suitable RMs. These measurements require that the systematic and random errors of the procedures used to determine the particular constituent be sufficiently well known to state the concentration of that measured within a required uncertainty level.

For assigning the value to uranium content in  $Rb_2U(SO_4)_3$  and plutonium content in  $K_4Pu(SO_4)_4$ , uranium and plutonium were determined by three different redox titrimetric methods by trained and experienced analysts. All the three methods developed in the Division have the precision of the

same order ( $\pm 0.1\%$ ). The following methods were used for the determination of uranium and plutonium in the weighed aliquots containing 15 to 20 mg of uranium or plutonium.

Method I: Redox titrimetric method using potentiometric method as end point detection technique.

Method II: Redox titrimetric method using amperometric method as end point detection technique.

Method III: Coulometric method.

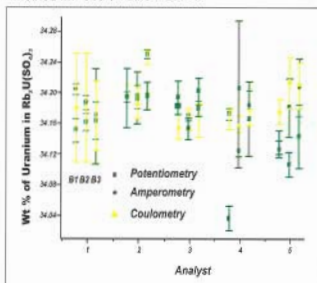


Fig.3. Weight % of U in  $Rb_2U(SO_4)_3$  in three different batches determined by five different analysts employing three different techniques

An experimental design based on a Randomised Block Design (RBD) was prepared to assign a value to metal content in RMs. Effects of factors such as method, inhomogeneity and lot were studied. Since all the three lots were found to be homogeneous, all the three lots were mixed into a single lot. For the combined lot, three dissolutions were carried out by dissolving about one gram of the sample from three different places. Five analysts were involved for carrying out the analysis by three methods mentioned above. Each analyst analysed three aliquots from each dissolution and by each method. The results of analysis on  $Rb_2U(SO_4)_3$  expressed as percent uranium are given in Table 1. The statistical variation of the data are shown in Fig. 3. The data were analysed using RBD designs with methods and

Table 2 : ANOVA for the chemically analysed data

Source of Error	Sum of Squares(SS)	Degree of Freedom	Mean Sum of Squares	F	Tab	Remarks
Method	0.002154	2	0.001077	0.624	1.98	NS**
Analyst	0.034724	4	0.008681	5.032	3.44	S*
Error	0.220816	128	0.001725 <sup>#</sup>			
Total	0.257964	134				

\*=Significant, \*\*=not significant, #=value of  $S_0^2$

analysts as factors of RBD. The analysis of variance (ANOVA) table for the chemically analysed data, given in Table 2, shows that method-effect is insignificant but analyst-effect is significant. Using the value of critical difference (cd), the results of the analysts whose values are significantly higher or lower were excluded while assigning a value to the uranium content in  $Rb_2U(SO_4)_3$ .

The data analysis showed that the mean of A2 differs significantly from those of others and hence results of analysts A2 were not considered for assigning the value to uranium content in  $Rb_2U(SO_4)_3$  material. Based on the data of analysts A1, A3, A4 and A5, a value was assigned to uranium content. A value of

$[34.167 \pm 0.042(1\sigma)]$  % was assigned to the uranium content in  $Rb_2U(SO_4)_3$ . Similarly, the value of  $[30.687 \pm 0.009(1\sigma)]$  % was assigned to plutonium content in  $K_4Pu(SO_4)_4$ . These values are in close agreement with the theoretical values of 34.152% and 30.665% for uranium content in  $Rb_2U(SO_4)_3$  and plutonium content in  $K_4Pu(SO_4)_4$ , respectively.

(The following officers of Fuel Chemistry Division have participated in this work:

*Preparation and characterization:* Dr R.R. Khandekar and Dr K.Krishnan; *Chemical analysis:* Ms N.Mirashi and Ms A.U. Bhanu, Mr P.R.Nair and Dr U.M.Kasar; *Statistical analysis:* Mr M.B.Yadav)

## A SIMPLE FIELD METHOD FOR THE ESTIMATION OF FLUORIDE IN GROUND WATERS FOR COMMON MAN'S USE

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Fluoride is considered as an essential element for human beings. In potable waters, a fluoride concentration of 1  $\mu\text{g/ml}$  is necessary to prevent tooth decay. However, at higher concentrations (>2  $\mu\text{g/ml}$ ), it has adverse effects such as causing fluorosis. Fluoride toxicity is prevalent in various parts of Andhra Pradesh, Madhya Pradesh and Gujarat. Due to natural as well as anthropogenic causes, the levels of fluoride in ground waters can

exceed the permissible levels. These levels may also vary from well to well and from season to season. Hence, periodic monitoring of potable water samples is necessary. If the water supply is centralised, government agencies can check the quality of the water in well- equipped laboratories. In our country, most of the people use their own water sources. In such cases, checking the fluoride content at every water source is practically

impossible by government laboratories. In order to monitor fluoride, there is a need for fast, simple and cost effective method, which can be easily adapted by common man. Visual colorimetric methods would be the most suited for such applications. The most commonly used visual method is based on bleaching of Zirconium-Alizarin complex. This method requires one hour for colour development and moreover, in this method, change in colour with fluoride concentration is gradual and hence a series of standards need to be prepared for quantification. This requires a skilled and experienced person.

A method for the estimation of fluoride based on the bleaching of Zirconium-Xylenol orange complex has been developed at BARC. Zirconium forms several complexes with xylenol orange. In the present method, the metal to ligand ratio was optimised. Acidity and dye concentration were optimised such that distinct colours are produced at three different levels (deficient, normal and toxic) of fluoride. Zirconium ions, which get polymerized at a lower acidity, affect colour development. De-polymerization of zirconium ions has been achieved using specific treatment, which helps in instantaneous colour development. Stability of the reagent solution prepared has been tested for a period of six months. Purity of the dye is also very important in getting colour contrast and stability.

This simplifies the procedure that any common man can so easily adapt it for field-testing.

Final reagent solution of zirconium-xylenol orange complex was prepared by mixing required amount of dye with De-polymerized zirconium solution. At the time of analysis, 1 ml of reagent solution is diluted with 4 ml of water sample (1: 4). The resultant colour of the solution if pink indicates fluoride deficient (<1 ppm), brown indicates safe level of fluoride (~ 1 ppm) and yellow indicates toxic level (> 2 ppm). A colour chart (Fig.1) can also be used as reference. Using this procedure, analysis of fluoride can be carried out within a minute. The method has been found to be very simple, fast, and cost effective and can be adapted by any common man. Thus, the common man can conclude himself whether the water is suitable for drinking or not, once the test reagent is made available to him. If the fluoride content is >2 ppm, it can also be quantified with a minor modification in the procedure. One ml of the resultant yellow solution is to be taken in a graduated test tube and diluted with another reagent solution, which is prepared by mixing 4 parts of distilled water and one part of reagent solution, till it turns brown. The volume (ml), at which yellow colour of the solution turns brown (~ 1 ppm colour), equals the concentration of fluoride in ppm.

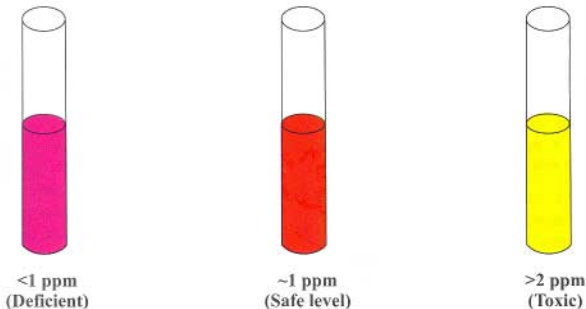


Fig.1 Colour chart for three different fluoride concentrations

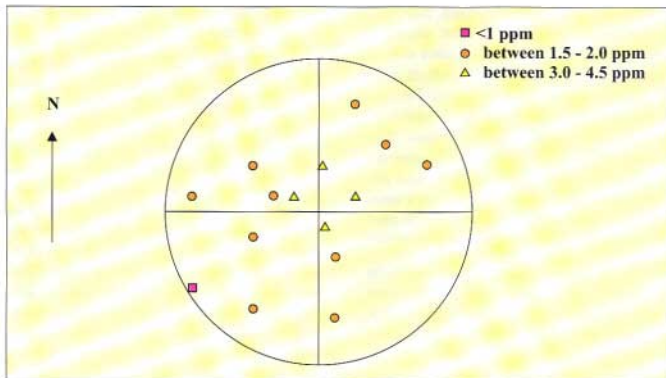


Fig.2 Variation of the fluoride concentration in ground water in Nagarjuna colony (Hyderabad) within a radius of 100metres with Sai Sindhu Grammar School as origin.

The method has been applied for the analysis of water samples (~200) from different places of Hyderabad and some fluoride affected areas of Nalgonda district, AP. Fluoride content was found to be in the range of 1 to 11 ppm in the fluoride affected areas. Variations of fluoride content with respect to various parameters such as distance, depth and season were studied. With respect to change in distance, larger variations in fluoride concentration have been observed even in shorter distances (Fig.2). With respect to depth variation, no specific trend was observed. As a part of seasonal study, water samples collected from selected borewells were analysed in the month of June and October. It was observed that there was no significant variation of fluoride content.

Variation in fluoride content with respect to the distance seems to be very attractive for getting suitable water source for use in fluoride affected areas. Since this method is fast and very simple, it can be applied to locate any water spot(s) with safe fluoride levels, in fluoride affected areas, so that people can draw water from that source. Recently,

the fluoride-affected villages of Marriguda Mandal in Nalgonda district of Andhra Pradesh were surveyed. It has been observed that each village is having 2 to 4 drinking water sources. All the water sources of some selected villages were tested for fluoride content on field. Out of 12 fluoride-affected villages, 11 villages were found to have at least 1 or 2 water sources with near to safe level of fluoride content (a few are shown in Fig.3a, b, c, d). The results of the analysis were made available to the villagers. The samples were cross-checked using the Ion Selective Electrode at our laboratory and the results were in good agreement within a variation of  $\pm 0.5$  ppm.

This reagent can also be used to check the saturation level of fluoride removal kits (defluorination units) by analysing the water samples from its outlet for fluoride level at regular intervals. No interference was found from cations and anions at those levels at which normally they are present in ground waters.

The present method can also be used for quantitative determination of fluoride concentration



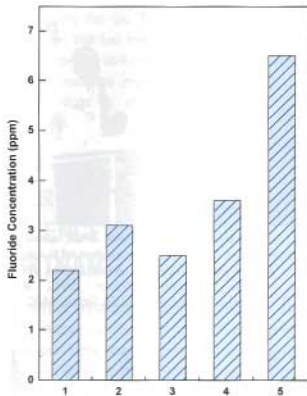


Fig.3a Different water sources in Marriguda of Nalgonda District

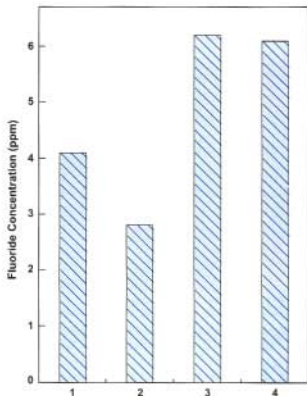


Fig.3b Different water sources in Bhodagonda of Nalgonda District

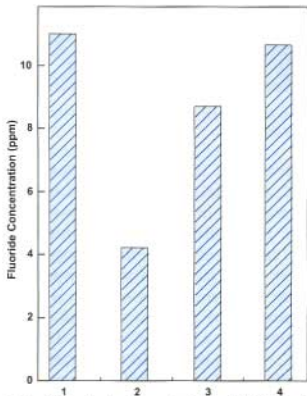


Fig.3c Different water sources in Rajahpet II of Nalgonda District

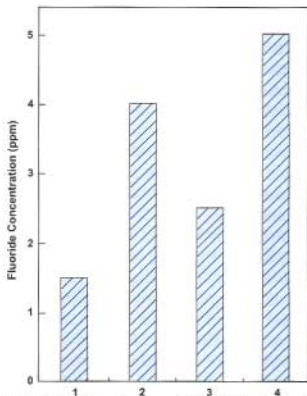


Fig.3d Different water sources in Vasavi Shiva Nagar in Hyderabad

by measuring absorbance at absorption maxima (550 nm) using spectrophotometer. This method is linear between 0.04 and 1.00 ppm of fluoride concentration. Sensitivity of this method has been computed as  $0.0066\mu\text{g}/\text{ml}$  for 1% absorbance with 1cm optical path length.

**Cost per analysis:** In the present method, the reagent cost per analysis is ~50 paise where as the commercially available kits could cost as high as Rs.250/- for each estimation which requires a portable spectrophotometer also.

## FUELLING MACHINE HEADS DELIVERED TO NPCIL

A function was held at Hall No. 3 at BARC for handing over two Fuelling Machine heads of 220 MWe PHWR, one for Kaiga and another for RAPS 3&4, on October 27, 2001. The machines were handed over by Mr B. Bhattacharjee, Director, BARC, to Mr A. Sanatkumar, Sr. Executive Director, NPCIL, after calibration, pre-commissioning and acceptance testing. These were the 32<sup>nd</sup> & 33<sup>rd</sup> machines which have been tested at Fuelling Machine Testing facility at Hall No. 3 and Integral Thermal facility at Hall-7. With the handing over of these machines, the present programme of fuelling machine testing of 220 MWe PHWR has been completed. Initially, Mr R.G. Agrawal, Head, RTD, recounted a brief history of this activity and also mentioned the utility of this programme for improvement in performance of fuel handling system, handling of incidences at different power reactors and development of tools for saving man rem during inspection and maintenance of coolant channels at sites. He made special mention of the technique for axial creep measurement, BARCIS & Wet Scraping. Mr Agrawal also mentioned the contribution made by different Divisions of BARC and NPCIL in this programme.



Mr A. Sanatkumar, Sr. Executive Director, NPCIL, speaking during the function. Others on the dias are (from left to right) : Mr R.G. Agrawal, Head, RTD, Mr A.K. Anand, Director, RPG, BARC, Mr B. Bhattacharjee, Director, BARC, and Mr G. Govindarajan, Director, A&M and E&I Groups, BARC.



Fuelling Machine being unloaded from service cart on way to Project stores, NPCIL.

Mr Govindarajan, Director, A&M and E&I Groups, BARC, assured full co-operation to NPCIL in future programmes of 220 MWe & 500 MWe PHWR. Mr B. Bhattacharjee, Director, BARC, mentioned about the complexity of the fuel handling system not only in size, but also the precision engineering required for proper working of the system. He also complimented the staff who contributed to this programme. Mr A.K. Anand, Director, TC&IRG, BARC, recounted his memories of working at Hall-3 and told the audience that PHWR programme was initiated in Hall-3. Mr Sanatkumar thanked Director, BARC, for his presence at the function. He also recounted his past days of working at Hall-3 where he

joined from the the BARC training school. He mentioned that fuel handling programme has been quite successful as it has been fully indigenised, and told the audience present that they should make similar efforts to make 500 MWe programme successful.

## BARC SETS UP NUCLEAR SCIENCE CENTRE IN VIETNAM

As part of continuing collaboration between India and Vietnam in the field of nuclear sciences, "Vietnam- India Nuclear Science Centre" (VINSC) has been set up at Dalat, Vietnam, by BARC.

VINSC was inaugurated by Mr B. Bhattacharjee, Director, BARC, and Vietnam's Vice Minister of Science, Technology and Environment, Dr Hoang Van Huay, on January 10, 2002.

Indigenously designed and manufactured laboratory equipment were supplied and installed at VINSC, Dalat. The lab equipment are now in use in Vietnam and are listed below :

1. GM Counting System
2. Alpha Counting System
3. Radiation Survey Meters ( $\gamma$ , x-ray,  $\beta$ )
4. Gamma Ray Spectrometer
5. Gamma Ray Spectroscopy System
6. BF<sub>3</sub> Neutron Counting System
7. X-ray Counting System
8. Digital Pocket Dosimeters
9. QF Direct Reading Dosimeters
10. Atomic Absorption Spectrometer
11. Gas Chromatograph
12. Electronic Analytical Balance
13. Electronic Semi - Micro Balance
14. Radioactive Sources
15. Image Processing System (Trinocular-based)
16. Refrigerated Centrifuge
17. Oven

18. 100 MHz Digitizer PC Add-on Card

19. IC Testers



*Mr B. Bhattacharjee, Director, BARC, giving the inaugural speech at Vietnam - India Nuclear Science Centre, Dalat, at Vietnam*

A large number of Divisions from BARC, viz., Electronics Division, Computer Division, Radio-chemistry Division, Technical Physics & Prototype Engineering Division, Radiation Safety Systems Division, Radiological Physics & Advisory Division, Solid State Physics Division, Cell Biology Division, Human Resource Development Division, and Board of Radiation & Isotope Technology were involved in various activities including equipment specifications, selection and inspection for procurement from Indian industry, such as M/s Nucleonix Systems Pvt. Ltd., M/s Plasto Craft Industries, M/s Shital Scientific Industries, M/s Toshniwal Instruments (India) Ltd., M/s Pulsecho Systems, M/s Mechantronics Equipments (I) Pvt Ltd., and M/s Netal Chromatographs.

Many of the radiation related equipments were designed, fabricated and tested in BARC in different Divisions such as ED, RSSD, TPPED, SSPD and Computer Division. Technology Transfer & Collaboration Division coordinated the complete activity in BARC with outside industries, DAE and Vietnam. Accounts Division, Directorate of Purchase & Stores, and Central Stores Unit executed very promptly all jobs related to financial sanctions, procurement, export worthy packing and air freighting the equipment to Vietnam.

A team of five experts from BARC consisting of Mr A.M. Patankar, Head, TT&CD, Dr A.G. Page and Dr T.K. Thulsidas of RCD, Mr Y.V. Chaudhari, Computer Division and Ms Voleti Madhavi, Electronics Division, installed all these equipments at VINSO, Dalat, and trained Vietnamese scientists/engineers in the operation, use and maintenance.

Setting up a centre of this kind outside the country, with all indigenous equipment, was a unique experience for BARC engineers/scientists. Every agency and individual involved in this endeavour made best efforts in planning and executing the assigned jobs in time, in such a multidisciplinary and multiorganisational time-bound activity. The setting up of such a state-of-the-art centre as VINSO with complete indigenous know-how and technology demonstrates the synergetic strength of BARC in the field of nuclear science and technology.

## IAEA/RCA REGIONAL WORKSHOP

An IAEA/RCA regional training course on 'Process Diagnostics Using Radioisotope Techniques in Petroleum/Chemical Industry' was conducted by Isotope Applications Division, BARC, in association with the International Atomic Energy Agency (IAEA), Vienna, during December 3 - 14, 2001 at Hotel Parle International, Vile Parle (East), Mumbai. Thirteen foreign participants and 5 Indian participants attended the workshop. The local participants were from Engineers (India) Ltd, Gurgaon, Mangalore University, Mangalore, and Vasantdada Sugar Institute, Pune. The course was inaugurated by Mr. A.K. Anand, former Director, RPG and TC & IRG, BARC & former RCA National Representative, DAE.

Dr S.V. Navada Head, Isotope Hydrology Section of Isotope Applications Division, BARC, gave the welcome address. Dr. N. Ramamoorthy, Associate

Director, Isotope Group, BARC, and Dr K. Raghuraman, RCA National Representative, also gave addresses. Course Director Mr. P. Sree Ramakrishnan of Isotope Applications Division, BARC, proposed the vote of thanks. Mr. Philip Berne from France and Dr J. Thu from Australia were IAEA experts for the programme. Dr. Jovan Thereska, Technical Officer, IAEA, was present during the programme.



The participants of the IAEA/RCA Regional Workshop along with the senior staff of BARC

The course dealt with various topics on radiotracer and sealed source applications for process diagnostics in petroleum/chemical industry. Faculty for the course was drawn from BARC, BRIT, UDCT Mumbai, NCL Pune, ONGC, etc. Use of software for validating radiotracer experiment was demonstrated by the experts. Field demonstrations were conducted at Reliance Industries Ltd, Patalganga, and Borosil Glass works, Mumbai. Experiments were conducted in the laboratories of IAD, BARC. The participants also attended the NAARRI International Conference (NIC - 2001) at Hotel Oberoi Towers during December 12 - 14, 2001. The programme concluded on December 14, 2001 with distribution of certificates to the participants.



## TRAINING COURSE IN 'BASIC RADIATION PROTECTION'

The Radiation Safety Systems Division, BARC, conducted a three-day training course (January 2-4, 2002) in basic radiation protection for the benefit of the staff members working in the Nuclear Recycle Group at Trombay, Tarapur and Kalpakkam. The course was conducted at the Waste Immobilisation Plant (WIP) Conference Room, Trombay.



*The faculty and the participants of the training course in 'Basic Radiation Protection'*

Mr M.L. Joshi, Head, Radiation Hazards Control Section, welcomed the participants and informed that it was the second such training course after the one conducted at the Radiological Laboratories. Dr M.C. Abani, Head, RSSD, BARC, in his opening remarks, said that as BARC now has its own regulatory function, there is a greater responsibility on the staff to maintain the high standards of safety at BARC. He also said that the course has been specially designed keeping in view the safety requirements of the plants in the Nuclear Recycle Group. Mr V.P. Kansra, Director, Nuclear Recycle Group, BARC, in his inaugural address, said that the training provided through this course would be very useful in increasing the safety of the plants in the Group.

Thirty-six participants from Back-End Technology Development Division, Plutonium Plant, Waste

Management Facility, Trombay, Power Reactor Fuel Reprocessing Plant, Tarapur, Waste Management Facility, Tarapur, Kalpakkam Fuel Reprocessing Project and Centralised Waste Management Facility, Kalpakkam, attended the course. The faculty for the course was drawn from the Health, Safety & Environment Group, BARC. The course consisted of 12 lectures covering various subjects, namely, dose calculation, radiation biology, health physics instruments, dose and contamination control, industrial hygiene & safety, environmental monitoring, radiation emergency handling, internal dosimetry, plant-specific H.P. procedures, transportation of radioactive materials, unusual incidents in reprocessing plants, etc. The course was organized by Mr M.L. Joshi, Head, RHC, RSSD, and Dr K.L. Narasimharao, RSSD, with the active help and co-operation of all the health physics units at WIP and PP. Mr D.D. Bajpai, Head, BETDD, Mr N.K. Bansal, Head, Waste Management Division, and Mr Kanvar Raj, Plant Superintendent, WMF, Trombay, gave invited lectures on nuclear fuel reprocessing, management overview, and waste management respectively. Dr A. Ramonujam, Head, Fuel Reprocessing Division, BARC, in his concluding remarks, highlighted the several problems faced by the Nuclear Recycle Group such as criticality, containment of radioactivity and off-gas treatment. He appreciated the efforts put in conducting the training course and also said that such training would go a long way in increasing the safety awareness of the staff and subsequent safe operation of the plants.

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## COURSE IN ELECTRONICS MAINTENANCE & SERVICES

As a part of its Manpower Development Programme, Centre for Equipment Electronics & Management under the Nuclear Fuels Group at BARC organized a one-week course in Electronics Maintenance & Services during December 10-14,

2001. This course was specifically developed and conducted by CEEM/NFG for a group of 20 junior officers and scientific assistants associated with electronics maintenance activities at various Divisions of the Nuclear Fuels, Bio-Medical and Materials Groups of BARC.

Mr D.S.C. Purushotham, Director, NFG, BARC, inaugurated the course, and Mr Umesh Chandra, Associate Director, A&M Group, BARC, gave his views as the Chief Guest. During the inauguration, Mr Jagmohan Singh, Head, CEEM, emphasized the need for training of very scarce electronics manpower in the three Groups. The course contents included the necessary exposure to diverse topics in electronics and initiated the participants into some of the specialized areas of electronics services, into which the equipment and instruments at BARC are being categorised.

The faculty mainly consisted of the CEEM Core Group Leaders and their senior colleagues in the Nuclear Fuels Group and two senior engineers from CWS and RRMD. Two faculty members from CSIO, Chandigarh, has also been invited to share their experience in trouble-shooting and maintenance of optical and medical instruments.

Mr Purushotham, Director, NFG, BARC, awarded the certificates to the participants of the course. A feedback session was also conducted to gather information about any similar course(s) elsewhere and to invite comments/suggestions regarding the course contents.

## DAE SYMPOSIUM ON NUCLEAR PHYSICS

The 46<sup>th</sup> Nuclear Physics Symposium, sponsored by the Board of Research in Nuclear Sciences (BRNS) of DAE, was held at the Saha Institute of Nuclear Physics (SINP), Kolkata, during December 26-30, 2001. The symposium was inaugurated by Prof. S.N. Ghoshal, who did the seminal work on the

experimental verification of the compound nucleus model half a century ago. Prof. Bikash Sinha, Director, SINP and VECC, Kolkata, presided over the inaugural function. Dr S. Kailas, Chairman, Organising Committee, welcomed the participants and other dignitaries, and the Convener, Dr. Arun K. Jain, in his introductory remarks, focused on the theme of the symposium. The Local Convener, Prof. Subimal Sen of SINP, proposed the vote of thanks.



*Inauguration function of the 46<sup>th</sup> Nuclear Physics Symposium. On the dias (from left to right) are : Prof. S. Sen, SINP, Prof. S.N. Ghoshal, Prof. B.C. Sinha, Director, SINP & VECC, Dr S. Kailas, Head, Nuclear Physics Division, BARC, and Dr A.K. Jain, Convener of the symposium*

There were about 280 participants from various universities and research institutions all over the country. In all, 25 invited talks and 230 research papers were presented, besides 4 theses presentations. The first detailed results from the experiments performed at BARC-TIFR Pelletron accelerator facility, based on National Gamma Detector Array, were discussed. This is a collaborative effort involving DAE Institutions and Universities. These experiments revealed the unusual behaviour of nuclei subjected to high rotation and temperature.

This symposium took stock of the world-scenario in the field of Radioactive (Rare) Ion Beam (RIB) physics and compared this with the ongoing efforts in India. A panel discussion on the National RIB Facility to carry out competitive research in this field brought out various view-points and highlighted the existing efforts in this direction.

Nuclear matter under extreme conditions of high temperature and high density, perhaps the way universe existed at the time of its creation, in the form of quark-gluon plasma (QGP) is another frontier area of nuclear studies. Indian scientists have been part of the international effort using the accelerator facilities in Europe and USA. Some of the talks featured the experimental programs underway related to large detector setups ALICE, STAR and PHENIX.

The weakly interacting evasive particle, Neutrino, has many interesting aspects which are connected to nuclear research. Some of these were discussed in this symposium especially the national proposal for its detection using a huge deep underground detector setup.

The IPA award for the best thesis presentation was given to Dr L.M. Pant of NPD, BARC. In the poster presentation category, the presentation of S. Ray, N.S. Pattabiraman and S.S. Ghugre of IUC, Kolkata was adjudged for the First prize. The poster presentations of K.K. Pant et al. of CAT, Indore and R. Husain, et al. of AMU, Aligarh, were given the Second and Third prizes respectively.

The academic sessions were marked by lively discussions. The invited talks, oral presentations, the poster sessions and the poster discussions were all well-attended and appreciated by the participants. An absorbing evening lecture, "Are we alone in the Universe?", was delivered by Dr Saroj Ghosh, former Director, Science City, Kolkata. A delightful performance of CHHOU Dance by artists of Royal Chhou Academy was another evening attraction.

The symposium proceedings were comprehensively summarized by Prof. I.M. Govil of Panjab University and Dr D.K. Srivastava of VECC, Kolkata.

In the concluding session, the Symposium Secretary, Dr D.C. Biswas, thanked the participants and local volunteers for making the symposium a grand success.

## BARC SCIENTISTS HONOURED



- Dr J.P. Mittal, Distinguished Scientist and the Director of Chemistry & Isotope Group, BARC, added one more feather in his cap when he was conferred the prestigious Professor N.R. Dhar Memorial

Medal Lecture Award on January 19, 2002 at the National Academy of Sciences, Allahabad. The Award was given to him for his noteworthy contributions in the field of Radiation Chemistry, Photochemistry, Laser Induced Chemistry, Chemical Dynamics and Physical Chemistry. The Award carries a medal, a citation and Rs. 10,000/- in cash. Dr Mittal delivered a lecture, "Excitement in Radiation Research", which was highly appreciated.

The Award is given once in two years. Some of the earlier recipients of the Award are Prof. R.C. Mehrotra, Prof. C.N.R. Rao and Prof. M.M. Sharma.



- Mr D.N. Badodkar of Division of Remote Handling & Robotics, BARC, was conferred the 'BARC Technical Excellence Award' for the year 2000, for his contribution to the design and development of

Special Purpose Drives and Reactivity Control Mechanisms. The award was presented to him at the Founder's Day Function on October 30, 2001.

Mr Badodkar is heading the 'Control Mechanism Section' at DRHR. He is responsible for the design and development of Reactivity Control Mechanisms for TAPP-3&4. Shut-off Rod Drive Mechanism (Prototype-II) has been successfully tested for more than 5000 full drops and 3000 cycles of on-line tests on full-scale test station at BARC. The design drawings and specifications have been handed over to NPCIL for production of mechanisms for reactor use, under an MoU signed between BARC and

NPCIL. Mr Badodkar is also responsible for the design and development of Reactivity Control Mechanisms for AHWR and 'Critical Facility'.



- Dr V. Venugopal, Head, Fuel Chemistry Division, BARC, has been awarded the 'Indian Association of Solid State Chemists and Allied Scientists Silver Medal 2001' for his

outstanding contribution in the field of solid state chemistry. This award was given to him at the National Conference and Symposium on 'Solid State Chemistry and Allied Areas', organised jointly by Indian Association of Solid State Chemists and Allied Scientists (ISCAS) and Indian Institute of Technology (IIT, Kanpur) during December 6-8, 2001 at the Department of Chemistry, IIT, Kanpur. ISCAS is a professional body registered with Government of Jammu & Kashmir and has been conducting biannual symposia on solid state chemistry.



- Dr Sudhir Thattey of Laser & Plasma Technology Division, BARC, and his wife Ms Nandini Thattey, were jointly given the State Award for Literary Excellence by the Government of

Maharashtra, for their book 'Nobel Nagaritil Nawal Swapne 1999'. The award comprises a cash prize of Rs 5,000/- and a citation.

'Nobel Nagaritil Nawal Swapne' is an annual series of books, giving stories about discoveries that are honoured with Nobel prizes for Physics, Chemistry, and Physiology or Medicine. The first book in the series was published in 1996 in Marathi. The Government of Maharashtra honoured it with the State Award for Literature. The authors wrote the English version of the book in 1997, which was released by Dr Anil Kakodkar, Chairman, Atomic Energy Commission & Secretary, Department of Atomic Energy (the then Director, BARC).



- Jagadguru Sankaracharya Sastrothama Award was conferred on Dr P.K. Padmanabhan of Analytical Chemistry Division, BARC, on December 23, 2001 at Shanmukhananda Hall, Mumbai by His Holiness Jayendra Saraswathi Swamiji of Kanchi. This award was conferred on him for his outstanding contributions in the field of science for the last forty years.



- Dr Satyajeet Chaudhury of Fuel Chemistry Division, BARC, was awarded the Prof. R.C. Tripathy Award for his work on 'Synthesis and Characterisation of Organometallic Compounds', by

Orissa Chemical Society during its fifteenth annual conference held at Department of Chemistry, B.J.B. Autonomous College, Bhubaneswar, during December 22-23, 2001.



- Mr M. Padmanabhan of Division of Remote Handling and Robotics, BARC, has been awarded the S.N. Seshadri Memorial Instrumentation Award of Indian Physics

Association for the year 1999, for his contribution to the indigenous development of Single Crystal X-ray Diffractometer. The award was given to him during the Annual General Body of Indian Physics Association held at Saha Institute of Nuclear Physics, Kolkata, on December 30, 2001.

Mr Padmanabhan is working on the design and development of High Temperature X-ray Diffractometer, Texture Goniometer, Stress Goniometer and High Resolution Powder Diffractometer. He is also working on the development of advanced tools and techniques for in-service inspection of coolant channels.

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