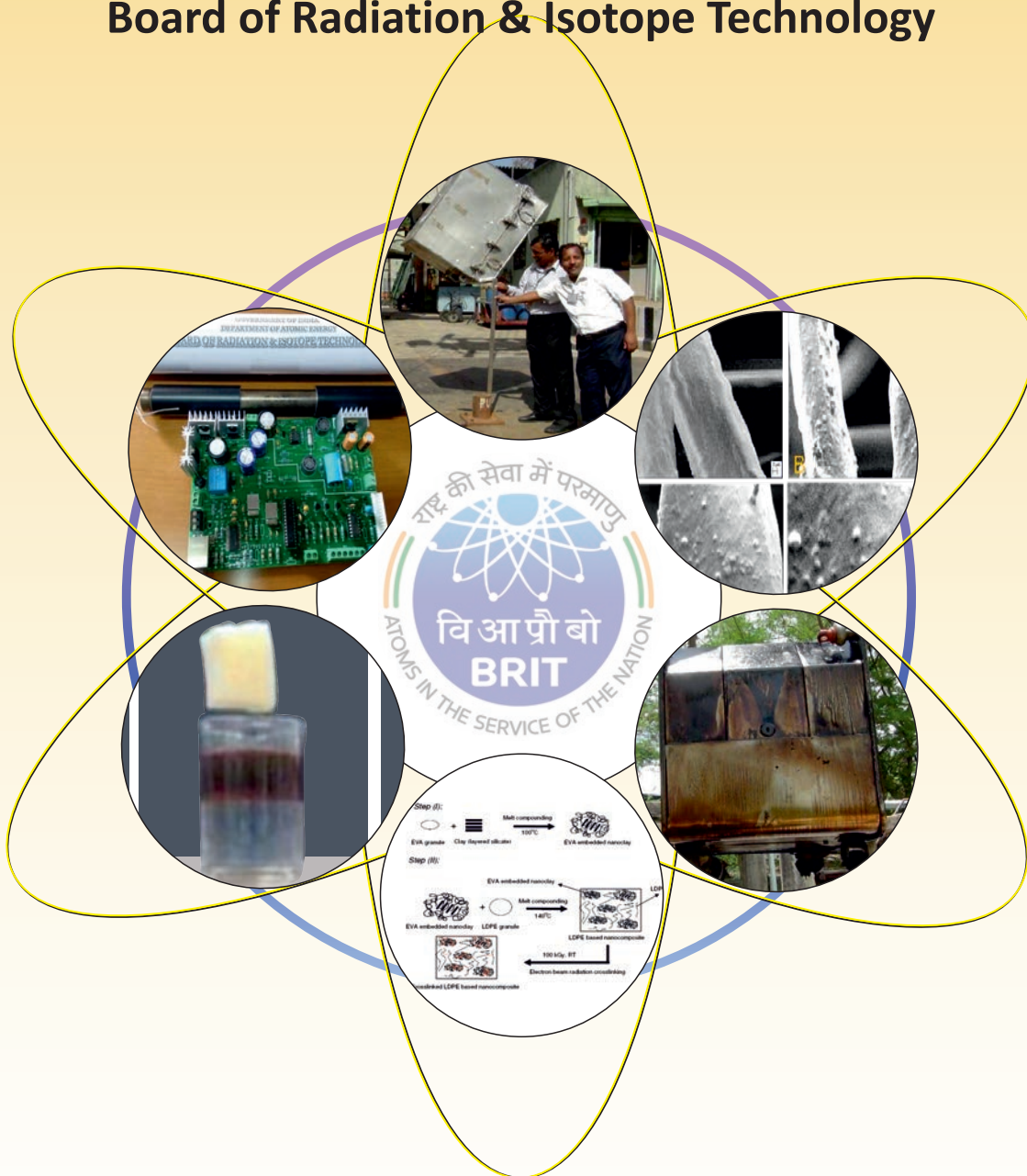




# BRIT Bulletin 2019

'Scientific Magazine'  
Board of Radiation & Isotope Technology



GOVERNMENT OF INDIA  
DEPARTMENT OF ATOMIC ENERGY  
BOARD OF RADIATION & ISOTOPE TECHNOLOGY



**It does not matter how slowly  
you go as long as you do not stop.**

*Confucius*



**A creative man is motivated  
by the desire to achieve,  
not by the desire to beat others.**

*Ayn Rand*





# *BRIT* *Bulletin 2019*

**'Scientific Magazine'**  
**Board of Radiation & Isotope Technology**





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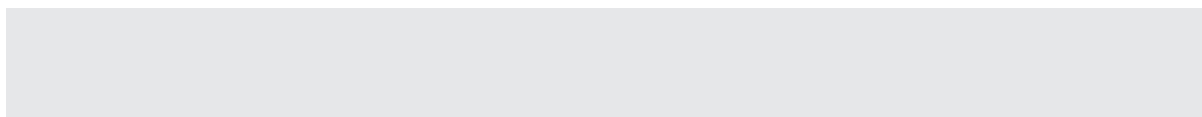
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# Development of an *In-vitro* Technique for Assessing the Biological Behavior of $^{99m}\text{Tc}$ -ECD

Soumen Das, Preethi Nair, Anupam Mathur, R. Vanaja, S. S. Sachdev

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## Abstract

Drug delivery in nuclear medicine using Tc-99m for brain perfusion imaging, is a big challenge as the agent not only need to be passing through the blood brain barrier (BBB), but also should be retained in the brain, while the brain image can be taken. Ethyl Cysteinate Dimer (ECD) is one such agent that can passively diffuse through the BBB. But this molecule gets trapped only in humans or primates and hence to study its efficacy, we need to have some alternative method to *in-vivo* testing. BRIT developed an *in-vitro* quality control method to study the efficacy of the ECD kit which can be routinely used at the production end before its supply to nuclear medicine centers around the country.

## Introduction

The process of passage of fluid through the circulatory system or lymphatic system to an organ or a tissue, usually referring to the delivery of blood to a capillary bed in tissue, is termed as perfusion. All animal tissues require an adequate blood supply to deliver oxygen and nutrients, remove carbon dioxide and metabolic waste and convey hormonal signaling. Hence perfusion imaging is an important aspect of detecting functional abnormalities in different organs.

In case of brain perfusion imaging the major challenge lies in drug delivery, i.e.,

to make the agent pass through the blood brain barrier (BBB), which is a term used to describe the unique restricting property seen in the microvasculature of the central nervous system (CNS). This is a highly selective semi-permeable border that results from the selectivity of the tight junctions between endothelial cells in CNS vessels [1]. This system allows the passage of water, some gases ( $\text{O}_2$ ,  $\text{CO}_2$ ) and lipid-soluble molecules (e.g. hormones) by passive diffusion, as well as some small polar molecules such as glucose and amino acids that are crucial to neural function through active transport mechanism using specific transport proteins while restricting the diffusion of solutes in

the blood (e.g. bacteria) and large or hydrophilic molecules into the cerebrospinal fluid (CSF). Thus two essential characteristics required of a brain perfusion imaging agent are the ability to surpass the BBB and significant retention via some trapping mechanism allowing images to be obtained providing useful clinical information. This necessitates the complex to be neutral and lipophilic so that it can diffuse through the endothelial cells of the brain's capillaries. The trapping mechanism is one which converts the complex into a more polar one via enzymatic or chemical pathway and thereby preventing its' movement out of the BBB [2-5].

Technetium-99m labeled ethyl cysteinate dimer ( $^{99m}\text{Tc-ECD}$ , Fig. 1), also known as  $^{99m}\text{Tc-Bicisate}$ , is one such complex that passively diffuses through the normal brain tissue in proportion to regional cerebral blood flow [6-11]. Consequently, on brain images, regions of normal brain that are adequately perfused demonstrate uptake of radioactivity, whereas brain lesions (such as infarcts) that have diminished or absence of blood flow; appear as areas of decreased radioactivity.

ECD can have four possible isomeric forms (D,D-, L,L-, L,D-, or D,L-). Among them, the L,L-and D,D- isomers demonstrate brain uptake, but only the L,L-isomer exhibits brain retention as only this isomer is recognized by the esterase enzyme and is metabolized into a polar species that is trapped in the human brain [12-14]. Brain retention is not only

stereospecific but also species-specific as the tracer localizes only in the brains of primates (monkeys and humans), following enzymatic hydrolysis of one of the ester groups to form a mono-ester, mono-acid metabolite which remains ionized at physiological pH and is therefore trapped in the brain [15-21].

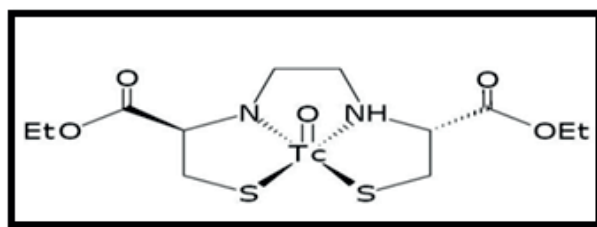


Fig. 1: Structure of  $^{99m}\text{Tc-ECD}$

Kit for the preparation of  $^{99m}\text{Tc-ECD}$  complex (TCK-42) is a regular product of BRIT which, unlike other kits, does not have bio-distribution study as a quality control testing parameter. This is essentially because it is not possible to do primate testing. Thus, the aim of the present work was to develop an *in-vitro* method that, instead of primate testing, would be able to ascertain the biological efficacy of  $^{99m}\text{Tc-ECD}$  prepared from the kit prior to injection in patients. Also, the results of this study could be used for the extension of the shelf-life of  $^{99m}\text{Tc-ECD}$  cold kit. Currently the shelf-life is four months but nuclear medicine physicians prefer to have kits with longer shelf life because the kit can then be used for a longer period of time and wastage of the kit can be prevented. At the manufacturer's end the extension of shelf life offers advantages such as the reduction in frequency of production and allied cost.

## Experimental

### Materials and methods

Pig liver esterase (PLE) enzyme was purchased from Sigma Aldrich, kit for the preparation of  $^{99m}\text{Tc}$ -ECD, ethylene dicysteine (EC) and ECD ligand were obtained in house. Sodium pertechnetate ( $\text{Na}^{99m}\text{TcO}_4$ ) was eluted with normal saline prior to use from a  $^{99}\text{Mo}$ - $^{99m}\text{Tc}$  column generator, supplied by BRIT. Analysis of  $^{99m}\text{Tc}$ -labelled preparations were carried out on a JASCO PU 2080 Plus dual pump HPLC system, Japan, with a JASCO 2075 Plus tunable absorption detector and Gina Star radiometric detector system, using a C18 reversed phase HiQ Sil ( $5\ \mu\text{m}$ ,  $4\times 250\ \text{mm}$ ) column.

### Radiolabelling

Radiolabelling of ECD kit was performed following a standardized procedure while  $^{99m}\text{Tc}$ -EC was prepared through a slightly modified procedure to achieve a neutral complex. This was done through two steps: first one involved the formation of  $^{99m}\text{Tc}$ -GHA and the second was a transchelation reaction involving EC at physiological pH.

### Enzymatic hydrolysis of $^{99m}\text{Tc}$ -ECD

$^{99m}\text{Tc}$ -ECD prepared from a lyophilised kit was incubated with pig liver esterase enzyme at  $37^\circ\text{C}$ . The reaction kinetics was monitored through HPLC after removal of excess enzyme by addition of ethanol followed by centrifugation and collection of the supernatant.

## HPLC

20  $\mu\text{L}$  test solutions were injected in the HPLC column and were eluted through a gradient mixture of water and acetonitrile (0 min 10% B to 30 min 90% B) at a flow rate of 1 mL/min.

## Results & Discussion

Cold kit for the formation of  $^{99m}\text{Tc}$ -Ethylene Cystienate Dimer (ECD) is a regular product of BRIT which is used for brain perfusion imaging after radiolabelling with  $^{99m}\text{Tc}$ . This radiolabelled complex is a neutral liophillic complex that is able to penetrate the BBB and there it undergoes enzymatic hydrolysis whereby one of the two ester groups get hydrolyzed by the esterase enzyme to form a comparatively polar hydrophillic species which is unable to revert back and thus gets trapped inside the brain which forms the basis for imaging. But this process occurs only inside the brain of primates. Non-primates do not show any such biological behavior as esterase enzyme is lacking in their brain. Thus, the primary aim of the present work was to develop an *in vitro* method which could substitute the primate testing, as the latter species are not available for biological evaluation of radio pharmaceuticals.

Bearing this in mind, an *in vitro* enzymatic reaction method was developed, which involved incubating the  $^{99m}\text{Tc}$ -ECD complex with pig liver esterase enzyme at  $37^\circ\text{C}$  and monitoring the reaction progress through HPLC. For this, characterization of  $^{99m}\text{Tc}$ -ECD and neutral charge bearing  $^{99m}\text{Tc}$ -EC (Fig. 2), the product

resulting from diester hydrolysis of  $^{99m}\text{Tc}$ -ECD, was done first following the said method. Peaks with retention times ( $R_t$ ) of 26 min and 8 min (Fig. 3 & 4) were obtained for  $^{99m}\text{Tc}$ -ECD and  $^{99m}\text{Tc}$ -EC respectively.

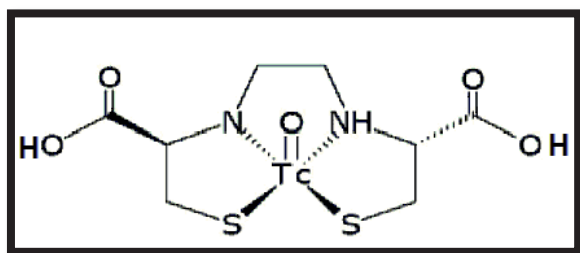


Fig. 2: Structure of  $^{99m}\text{Tc}$ -EC

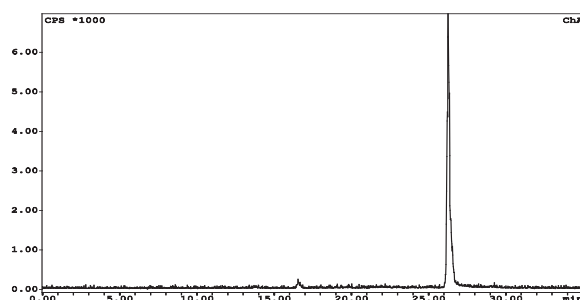


Fig. 3: HPLC spectrum of  $^{99m}\text{Tc}$ -ECD

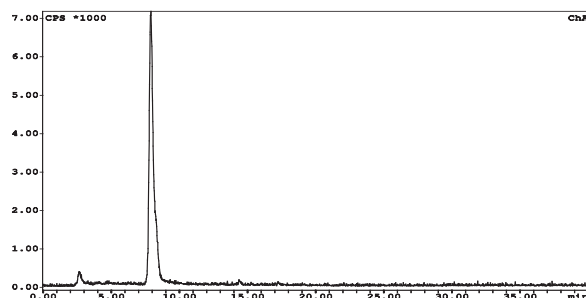


Fig. 4: HPLC spectrum of  $^{99m}\text{Tc}$ -EC formed at neutral pH

After enzymatic incubation an additional peak was observed at  $R_t \sim 17$  min (Fig. 5), appearing exactly midway between the peaks of  $^{99m}\text{Tc}$ -diester ( $^{99m}\text{Tc}$ -ECD) and  $^{99m}\text{Tc}$ -diacid ( $^{99m}\text{Tc}$ -EC) complex, indicating hydrolysis of one of the two ester groups and thus confirming the *in*

*vitro* recognition of the  $^{99m}\text{Tc}$ -ECD complex by the esterase enzyme as is the case *in vivo*.

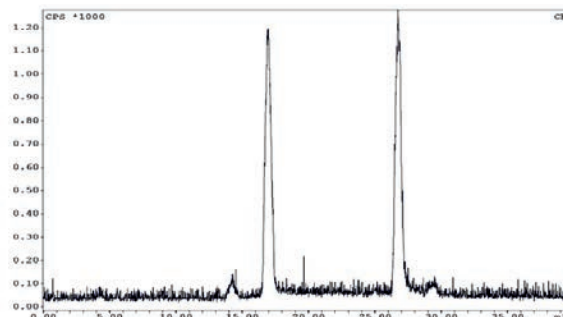


Fig. 5: Hydrolysis pattern of  $^{99m}\text{Tc}$ -ECD with PLE enzyme

## Conclusion

$^{99m}\text{Tc}$ -ECD complex is recognized by esterase enzyme *in vitro* and the progress of the hydrolysis reaction could be followed through HPLC. Thus this new *in vitro* enzymatic method could be used as a means of quality control testing of ECD kit before using the labeled product in patients.

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# Purification of Thymidine-methyl-T by High Performance Liquid Chromatography

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## Abstract

Tritium labelled thymidine is in high demand due to its exclusive use in DNA based biological research. To meet the continuous demand of users, purification of tritiated thymidine, using high performance liquid chromatography, is standardized. It has important advantages over the other conventional methods of purification such as thin layer chromatography, paper chromatography and column chromatography. The purification of tritiated thymidine from the radioactive impurities, using reverse phase separation technique, involves high precision and accuracy, while being rapid and cost effective as compared to other conventional methods.

## Introduction

Thymidine is a pyrimidine nucleoside that is composed of the pyrimidine base thymine attached to the sugar, deoxyribose. As a constituent of DNA, thymidine pairs with adenine in the DNA double helix. Radiolabelled thymidine containing the hydrogen  $\beta$ -emitting radionuclide, tritium ( $^3\text{H}$  or Hydrogen-3); is widely used in biological research to measure the growth of tissues and tumors [1, 2], both in cell culture and *in-vivo* studies. When cells are incubated with radiolabelled thymidine, they use the labelled thymidine to synthesize DNA, and gets incorporated into their DNA backbone. Hence, it can be used as a marker to measure the synthesis

of DNA. It is also used to measure T lymphocyte proliferation [3] *in-vitro*, as the amount of tritium labelled thymidine taken up is a general measure of the number of new lymphocytes that are produced. Standardization of cell-based immunologic monitoring for cellular immunity, to assess the ability of cell-based assays and to predict T-cell responses, thymidine incorporation studies using tritium labelled thymidine, is found to be very important.

To meet the regular demand of tritium labelled thymidine, it is prepared in bulk quantity and stored. These stocks are regularly checked for radiochemical purity before supply. During the storage of tritiated thymidine, the radioactive impuri-

ties are formed. If these impurities are not removed, it will seriously affect the researcher's studies. It is found that 8-10% radioactive impurities were formed within two months on storing of high specific activity tritiated thymidine solutions [Fig.2]. So, before the supply of compound, it should be devoid of these impurities. Although the separation and identification of thymidine has been achieved by paper chromatography [4, 5] and column electrophoresis, the methods described are not suitable for the resolution of impurities on milligram and microgram quantities. New technique like high performance liquid chromatography (HPLC) with different detectors is a leading analytical tool for identification, separation and quantification of thymidine.

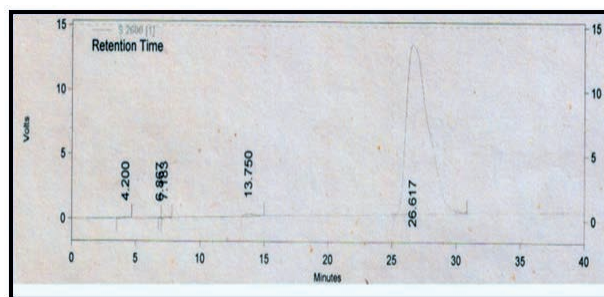
This paper presents a simple and sensitive reverse phase HPLC (RP-HPLC) method with UV/Visible detection for the purification of tritiated thymidine. The method is based on the reverse phase separation technique with high precision, accuracy as well as rapid and cost effective in comparison with the other conventional methods.

## Experimental

All the reagents used were of A.R. Grade. The starting material was procured from M/s. Sigma Co. USA. Silica gel coated plastic TLC plates were purchased from E. Merck. Hidex 300SL TDR liquid scintillation analyzer was used for radioactivity assay. The radiochemical purity of labeled product was determined by scanning the TLC sheets by means of a

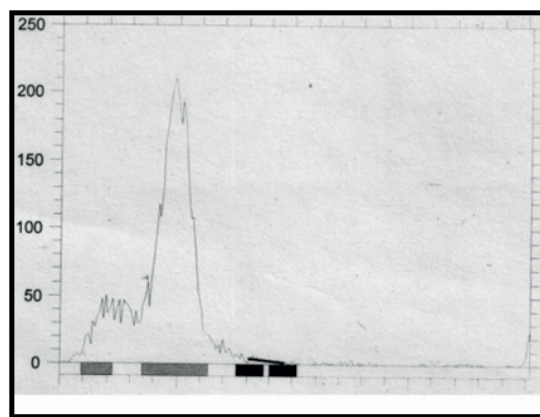
Bioscan make, radio TLC scanner. Knauer make semi-preparative HPLC with a UV detector (model 2600) and C-18 semi-preparative column (Length 250 mm, OD 10mm) were used for the purification.

Tritium labelled thymidine is prepared in-house. The solutions used for HPLC were degassed under vacuum and filtered through 0.45  $\mu$ m membrane filters prior to use. 20 microlitre of standard authentic solution of thymidine of concentration 20 microgram was injected to get the peak area as well as retention time [Fig. 1].



**Figure 1:** HPLC chromatogram of authentic Thymidine

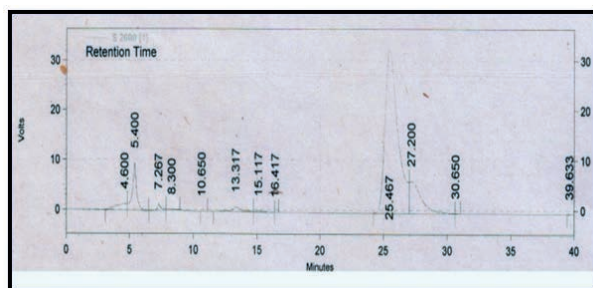
Impure tritium labelled thymidine (Fig. 2), which was found to be contributing ~8-10% radioactive impurities within two months on storage of high specific activity tritiated thymidine solution, was



**Figure 2:** Radiochromatogram of impure Tritiated Thymidine (on storage)

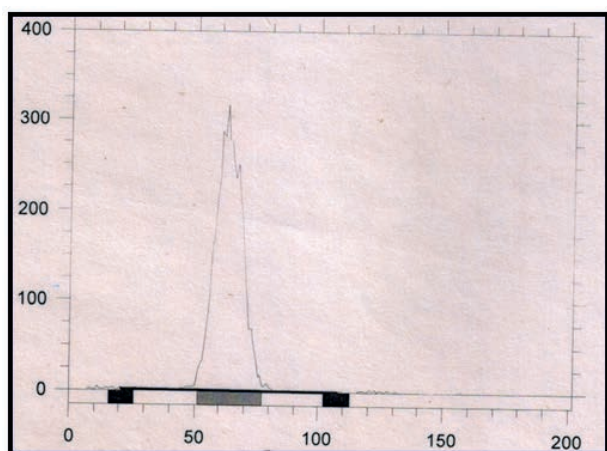
dissolved in water and 500 microlitre of this solution was used for injection at each time.

The mobile phase used was 10% methanol in water. The flow rate was maintained at 1.5 ml/minute, UV-detector was set at 264nm. The thymidine peak of standard sample was found at 25.6min and the same retention time was used for collecting tritiated thymidine peak separated on column [Fig. 3].



**Figure 3:** HPLC chromatogram of impure Tritiated Thymidine

The radiochemical purity of the purified product obtained was checked by TLC in two different solvent systems (a) Ethyl acetate: Formic acid: water (60:5:35 v/v/v) and (b) Butanol: Acetic acid: Water (4:1:5 v/v/v/) followed by radio TLC scanning. From 24mCi of crude



**Figure 4:** Radiochromatogram of purified Tritiated Thymidine

tritium labelled thymidine, 19.6mCi of pure product of 129.6  $\mu$ g was obtained with a radiochemical purity > 98% [Fig.4].

## Results and Discussion

The purification of tritiated thymidine using this method has advantages over the conventional methods such as thin layer chromatography, paper chromatography, column chromatography, etc. In the conventional methods of purification, the compound obtained after purification in a single run is never found to be sufficiently pure and required one or more steps of purifications. Many a times these methods necessitate overnight operations for completion. Therefore, during the course of purification there are chances of formation of radioactive impurities because of repeated exposure of compound to chemicals and air. Not only this, but it was also observed that these techniques are associated with incomplete recovery of purified product resulting in the loss of costly labelled compound and hence, the conventional methods are found to be un-economical.

The method adopted here for the purification of tritiated thymidine was found to be rapid, very efficient, completes within 30 minutes and can be applied on microgram to milligram level with complete recovery of the compound. The yield obtained is ~90% for microgram to milligram level, with the radiochemical purity > 98% and hence is the most suitable method for purification of tritiated thymidine.

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# Performance of COCAM-120: A Radiography Exposure Device under 9m Drop and Fire Test

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## Abstract

Gamma radiography devices are widely used for non-destructive testing (NDT) in industries. COCAM-120 is a Co-60 based radiography exposure device that can be used for non-destructive testing of steel with a thickness range of 40mm–200mm. The device uses multiple shielding material such as tungsten, depleted uranium (DU) and lead to make the device light and compact and to keep the radiation levels on the surface of the device within acceptable limit. Stainless steel AISI SS304L is used as a structural material. During transportation, the device can be put in an over pack made of SS 304L steel filled with indigenously developed Polyurethane Foam (PUF) which will not only protect the device from the damage under 9m drop test but also act as a thermal barrier during fire test.

The device has been designed to qualify it as a Type B (U) transportation package and meet the normal and accident conditions of transport as specified in regulations of International Atomic Energy Agency (IAEA) and Atomic Energy Regulatory Board (AERB). The cask along with the over pack was dropped from 9 m on unyielding target at Automotive Research Association of India (ARAI), Pune and successively it was dropped on a punch of diameter 150 mm and height 200 mm from a height of 1m. The deformed cask was then put in a furnace with temperature 800°C for 30 minutes. The cask maintained its structural integrity after these tests. The paper brings out the tests such as 9 m drop test, 1 m punch test and 800°C thermal test carried out on the exposure device. The tests for normal condition of transport and 15 m water immersion test are not in the scope of this paper.

**Keywords:** Type B (U), Structural integrity, PUF, NDT

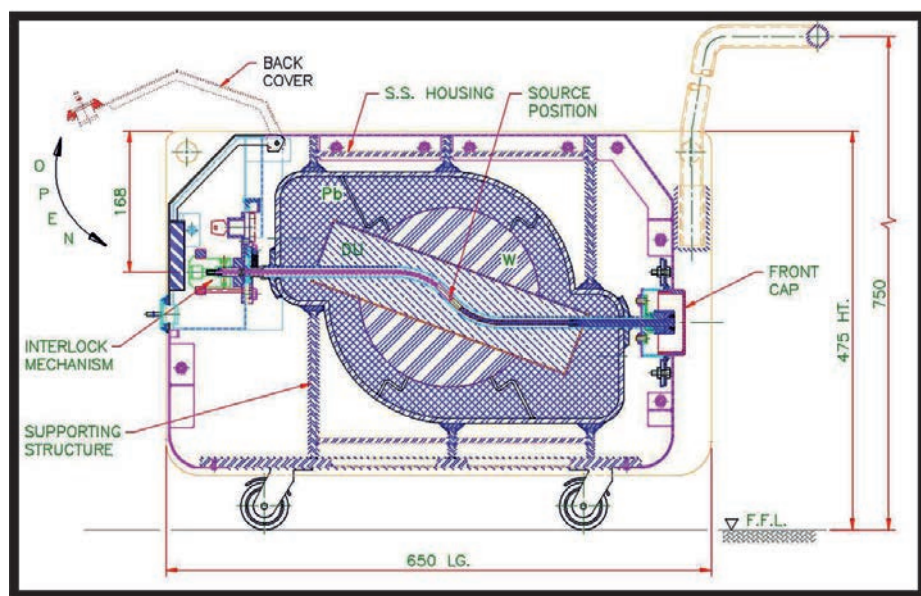
## Introduction

Radiography exposure devices are used in industries for non-destructive testing. Commonly used radioactive sources for radiography are Ir-192, Se-75, Co-60 etc. For steel, with a thickness range from 5 mm to 40 mm, Ir-192 or Se-75 is selectively used for radiography. When the thickness of steel is beyond 40 mm, Ir-192 and Se-75, both of which has lower energy, are not suitable, due to low penetration power. Co-60 having gamma energy of 1.17 MeV and 1.33 MeV is more suitable as a radiography source for thickness range for 40 mm – 200 mm. Some of the Co-60 based radiography exposure devices that are available in Indian market are ExertusVox 100, Sentinel 680-OP and 741- OP, Sentry 330 and 110 etc. Most of them are imported and expensive. Board of Radiation and Isotope Technology (BRIT) has developed an indigenous Co-60 based radiography device COCAM-120 that can carry 120 Ci of Co-60 and can be used for non-destructive testing. A sectional view of COCAM-120 radiography device is shown in Figure 1.

The device is designed using multiple shielding material such as lead, tungsten and depleted uranium to keep the radiation level within limit and optimize its weight against the material

availability and cost. These shielding materials are encased in a 6 mm stainless steel (AISI SS304L grade) enclosure. A zircaloy tube in the form of “S” shape passes through the center which facilitates the movement of the radiation source. The device is provided with an interlock mechanism to avoid any unintentional operations. The weight of the exposure device is 316 kg with its major dimensions as 650 mm (L) x 370 mm (W) x 475 mm (H).

The device can be put in an over pack and transported in public domain. The over pack weighs around 168 kg with a major dimension of 893 mm(L) x 618 mm (W) x 737 mm (H). The device along with the over pack is called the transportation package. The over pack is made of 3 mm of SS304L and filled with polyurethane foam to protect the transportation package under drop and fire test. The device along with its over pack has been designed as a Type B (U) package as per



**Figure 1:** Sectional view of COCAM -120 radiography device

regulations of IAEA and AERB [1] [2]. As per the regulation the device need to show its structural integrity and shielding integrity under the cumulative effect of 9 meter drop on unyielding target, 1 meter drop on a punch of diameter 150 mm and height 200 mm, and 800°C fire test for 30 minutes. The paper brings out the tests carried out to assess the package performance under drop, punch and fire test.

## Experimental

Experiments on the exposure device were carried out at Automotive Research Association of India (ARAI), Pune. The 9m drop facility consists of an unyielding target of size 3 m x 3 m size. The unyielding target is designed for testing of packages up to 10 ton weight. a quick release mechanism a attached to a over head crane to drop the package. The package was dropped in such an orientation that the edge of the over pack hits the unyielding target. The drop orientation was chosen based on numerical simulation



**Figure 2.** Overview of experimental set up for 9m drop test

[3][4] of various possible orientations such that the package sees the maximum damage. The punch test is carried out on the deformed package. The punch is made



**Figure 3.** Experimental set up for Punch test

of mild steel with a dimension of 150 mm diameter and 200 mm in length fixed rigidly to the unyielding target. The experimental set up for 9m drop and 1m punch test is shown in Figure 2 and Figure 3 respectively. All the tests were carried out with a dummy source inside the device.

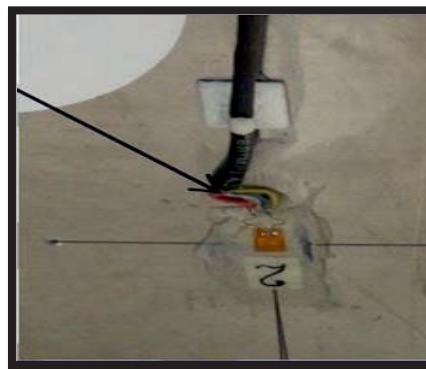
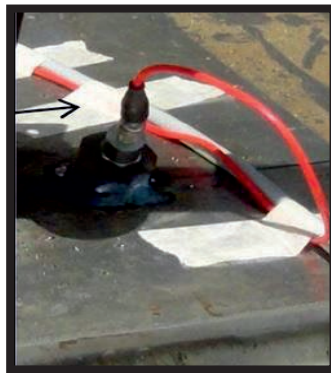
## Instrumentation

An accelerometer of model 3200B of Dytran Instrument Inc. with a range of 10,000 g was attached near the lifting position to find the deceleration of the specimen. Six numbers of uniaxial strain gauges of 350 ohm resistance were attached on each face of the exposure device. A 32 channel data acquisition unit (e-DAQ plus) with a sampling rate of 16 kHz for strain gauge and accelerometer was also attached for data acquisition. Two high speed cameras were placed perpendicular to each other to obtain the deformation at various time frames. The

drop orientation and instrumentations arrangements are shown in Figure 4 & Figure 5 respectively. On the deformed cask, thermocouples were mounted to study the temperature profile on the package. Ungrounded metal sheathed "K" type thermocouples (Chromel-Alumel) of 12 SWG was used for the temperature measurement of furnace wall and grounded "K" type thermocouples (Chromel-Alumel) of 22 SWG was used for specimen temperature measurement. An ARAI make 96 channel temperature logger for data acquisition was used for "K" type thermocouple. Six thermocouples, one at the centre of each face of the COCAM-120 over-pack were spot-welded on the outer surface. Additionally one plate thermocouple was mounted on the stand as shown in the Figure 6. After fixing the thermocouple the device was kept in a furnace with an average temperature of 800°C for 33 minutes as shown in Figure 7.



**Figure 4.** Overview of experimental set up for 9m drop test.



**Figure 5.** Location of strain gauge and accelerometer.



**Figure 6.** Location of thermocouple



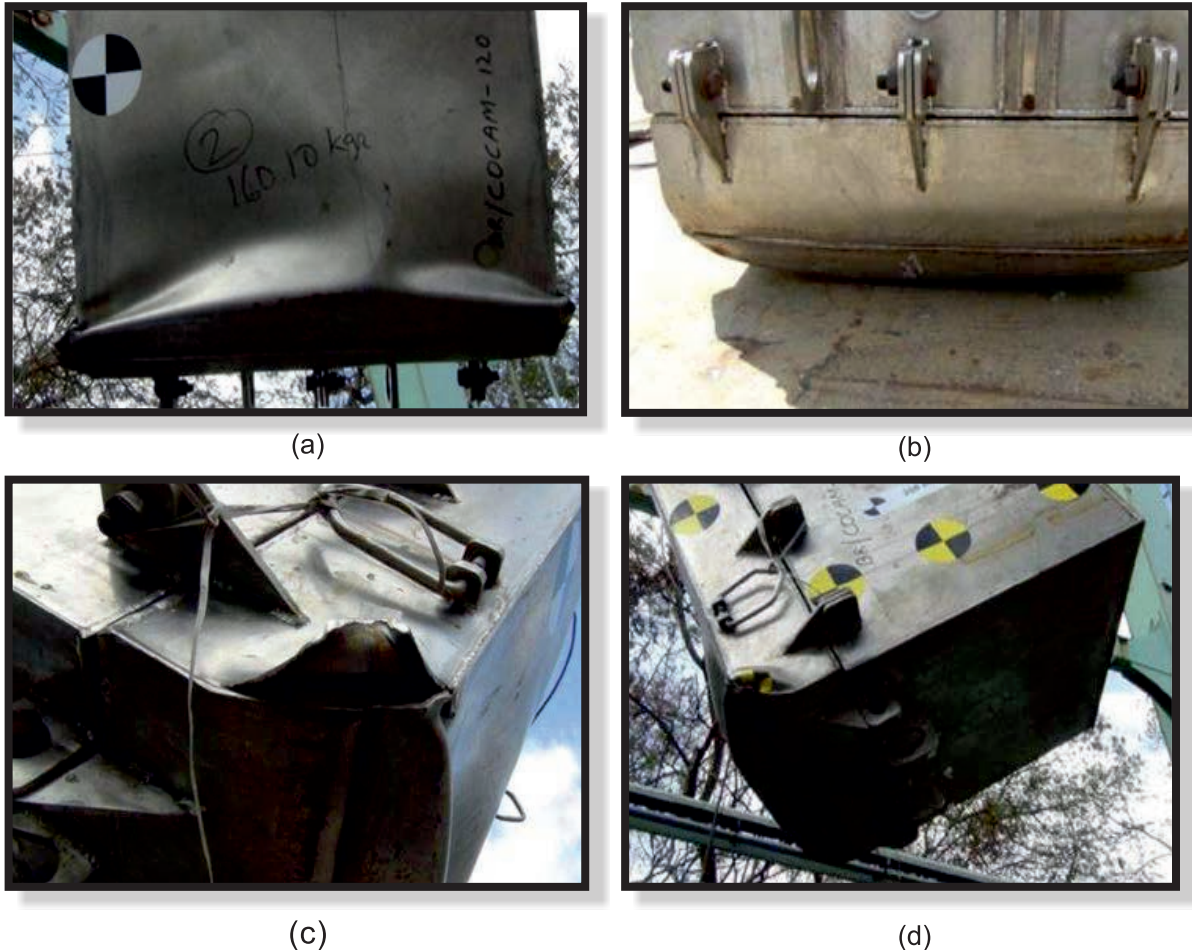
**Figure 7.** Overview of experimental set up for 800°C fire test in furnace

## Experimental Results

The gross deformation of the exposure device after the experimental drop test is shown in Figure 8. It can be observed that the steel shell of outer

cracked and the steel plate tore away exposing the PUF while the other corner of the impact edge tore and got punctured into the PUF (Figure 8d).

The deformed cask was dropped on a



**Figure 8:** Gross deformation of exposure device after 9m drop (a) bulging of top cover (b) Flattening of impact edge, (c) impact corner torn up, (d) another corner punctured into the PUF

enclosure that houses the polyurethane foam got deformed and bulged and is not uniform. This may be due to the fact that in experiment the corner edge was not perfectly in line with the target and undergone successive impact. The impact edge also got flattened (Figure 8b). It can be seen from the Figure 8(c) that at one corner of the impact edge, the weld

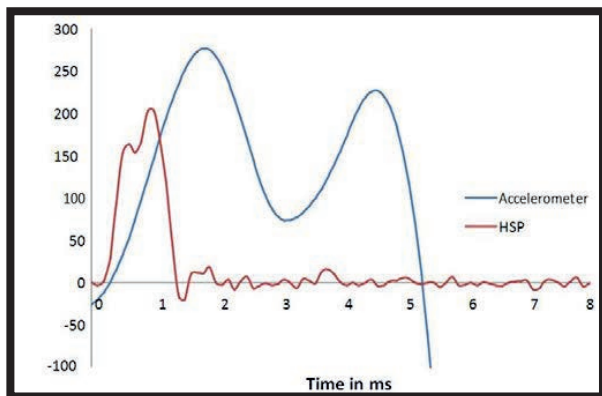
punch of diameter 150 mm and 200 mm length. The package was oriented in such a way that the package corner where the plate is torn hit the punch so that the package will have the maximum damage. The drop height was kept at 1m. The deformed cask after the punch test is shown in Figure 9. It can be seen that a depression in the shape of the punch is

observed at the corner without any crack. The PUF has been compressed.

is the limit of the strain gauge. This was expected as the location of strain gauge 2 was near to the impact side.

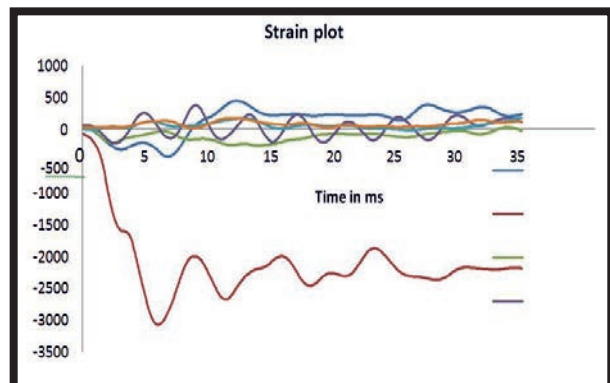


**Figure 9.** Overview of experimental set up for 800°C fire test in furnace



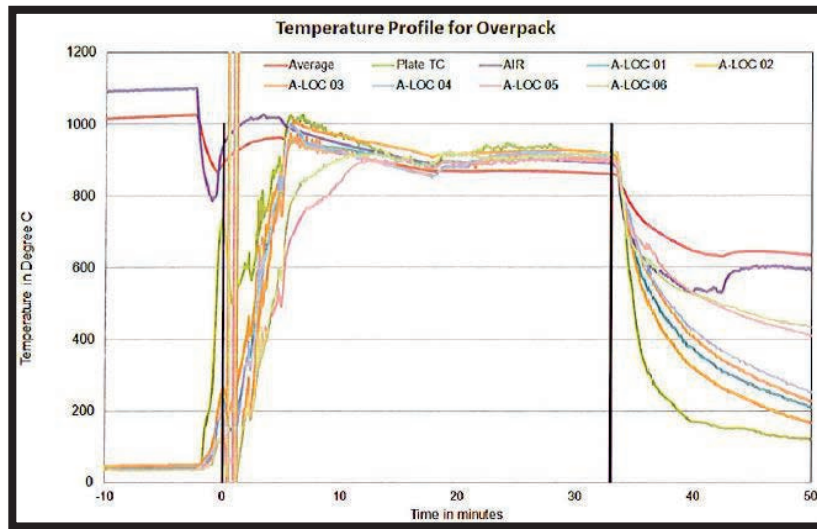
**Figure 10.** Acceleration plot obtained in accelerometer and high speed photography (HSP) under 9m drop test

The acceleration obtained in accelerometer and high speed photography is shown in Figure 10. The g loading observed in the accelerometer for corner drop was 276g whereas in the high speed photography 202 g was observed. The strain data of all the six strain gauges are shown in Figure 11. It can be seen that the strain for all strain gauges except strain gauge 2 are in the range of + 500 micro strains. The strain gauge 2 shows a reading greater than 3000 micro strain which



**Figure 11.** Strain plot obtained in various strain gauges

Cask along with the over pack was taken to the furnace for 800°C fire test for 30 minutes. Thermocouples at different locations of the furnace were attached to measure the temperature inside the furnace. Before taking the cask to the furnace it was ensured that the temperature in the furnace is above 800°C. The temperature profile on the package surface is shown in Figure 12. It can be seen that the temperature in the thermocouple remains above 800°C for more than 30 minutes. A



**Figure 12.** Temperature plot observed in the thermocouple during 800°C fire test in furnace



**Figure 13. (a)** COCAM with over pack after fire test



**Figure 13. (b)** COCAM Device after 800°C

maximum temperature of 1006°C is recorded on the thermocouple mounted on the over pack of the exposure device.

After the fire test the package was allowed to cool at ambient conditions. The condition of the package after fire test is shown in Figure 13 (a) & (b). The over pack

was opened and the device was checked for any structural deformation and lead melting. After proper inspection it was found that there is no lead melting and the shielding integrity of the exposure device is intact. Traces of burnt polyurethane foam were found inside the package.

## Conclusion

COCAM-120 exposure device has been tested for different hypothetical accident scenarios such as 9m drop on unyielding target, 1m drop on punch and 800°C fire tests at ARAI, Pune. The maximum acceleration observed in the accelerometer is 276 g. The strains observed in the outer enclosure are within + 500 micro strain except in one strain gauge which measure a strain greater than 3000 micro strain. There was no gross structural damage on the device. However, the outer enclosure was bulged and the corner welds cracked and opened up exposing PUF. In 1m punch test the depression of the punch was clearly visible without any crack. The PUF got burnt in the fire test at certain places where it was exposed. The outer enclosure was opened and checked for any lead leakage. No lead leakage was found. The COCAM-120 radiography exposure device maintained its structural and shielding integrity after the 9m drop, 1m punch test and 800°C fire test.

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# New thoughts of electron beam application in materials science with the main emphasis on surface and interface engineering of polymeric system

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## Abstract

Radiation technology based on gamma and electron beam (EB) radiation has become popular for couple of decades in the field of polymer science and technology. The gamma is being used mainly for grafting of some specific functional groups and EB is being used crosslinking and curing of single polymer mainly. Here, in our group at BRIT-BARC Vashi Complex, the EB has been used for different purposes such as grafting as well as crosslinking for interface and surface engineering of multicomponent polymeric materials. We have developed a biodegradable oil/water separator through micron scale surface roughness and surface energy tuning with the help of radiation. We have synthesized industrially important nanocomposites consisting of two components with different surface wettability through interface engineering. These nanocomposites include polypropylene/clay nanocomposites with uniform and reproducible properties, Nylon-6/clay nanocomposites with reduced water absorption and enhanced mechanical properties. By this technology we have also combined the impact of nanocomposite technology and radiation technology to develop easily recyclable LDPE based nanocomposite. EB technology is also used to engineer interface of organic (POE, Polyolefin elastomer) and inorganic (Silicon rubber) hybrid material to improve dispersion leading to improved mechanical and high temperature application polymeric systems. These are fewer representative examples of our last 7 years research. In materials sciences, EB assisted grafting and crosslinking have immense potential to develop desired materials for various applications.

## Introduction

It has been couple of decades that radiation technology is being utilized to synthesize and modify single phase polymeric materials. The radiations which are being used mainly are gamma and electron beam (EB) radiation. The high energy gamma has been used mainly for grafting purpose, i.e. attaching an unlike functional group/polymer chains to a polymer chain by covalent bonding. Those functional groups attract some elements, ions, heavy, toxic metals and molecules (e.g. dyes) and/or remove them [1]. The polarity of surface is also altered by this technique, or special functional group is attached onto surface, which is the key for being used for biological, medical and some other applications [2]. EB is used mostly for curing and cross linking of polymers to form 3D network. Due to incorporation of cross linking, mechanical properties, thermal properties, crystallinity, chemical resistance, weather resistance etc. are improved. For some systems, EB has been used also for grafting following pre-irradiated technique. Thus, for last few decades many materials have been developed and even commercialized. Most of the polymers are single phase, though few are reported to be binary or ternary, but those are subjected to intra and inter phase cross linking only by electron beam radiation.

In our work, we have utilized radiation technology for designing surface and interface of multi component polymeric system, which includes polymer blends, polymer composites and polymer nano

composites. Strong and high quantity interface is the main key for making a material with more than one constituent. For this, some interaction between polymer-polymer for blends, polymer-filler for composites and polymer-nano filler for nano composites, is invariably required. The interaction depends on several factors, such as, interfacial energy, surface energy of constituent phases, surface polarities etc., which are related with processing behaviour, i.e., rheological performance of polymers and fillers. We have designed the interface and surface of polymers and fillers to generate required distribution, dispersion and interfacial strength. We have developed many successful blends, composites and nano composite materials for various applications using these techniques.

Moreover, we have been successfully applying this radiation technology for molecular scale surface and bulk engineering, which renders a fresh surface with different topography and surface energy. By radiation assisted molecular scale engineering we have bio mimicked some super-hydrophobic natural entities to develop super-hydrophobic super absorbent, which separates oil/water from mixtures. In this article, glimpse of our last seven years research is provided. Our developed materials can be used for industrial, environmental and social benefits.

## Experimental

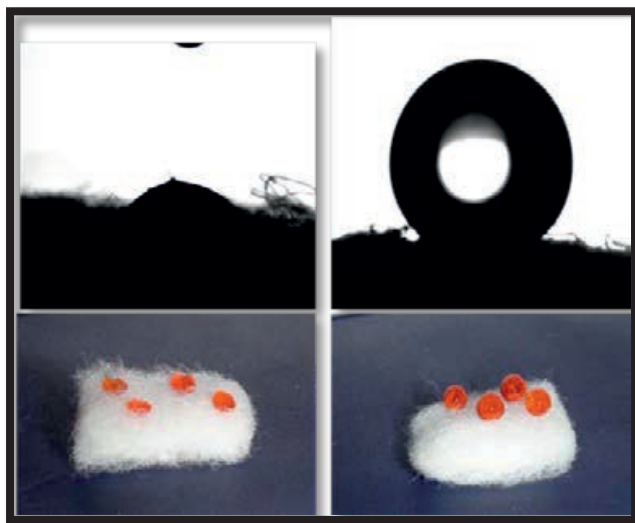
**Materials:** Used materials were IDM (Isodecyl Methacrylate), Methacrylic acid

(MAA), Acrylic acid (AA). The used polymers were elastomer, thermoplastic and inorganic polymers. The reinforcing agent mainly was clay nano particles.

For sample preparation traditional polymer processing instrument were used.

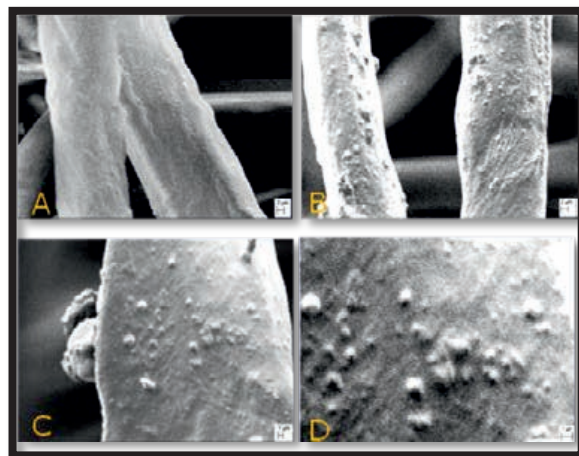
***i) Bio-mimicry through EB assisted grafting leading to oil/water separator***

A biodegradable multifunctional superabsorbent is developed through molecular scale engineering (topography and surface energy) by electron beam radiation assisted grafting of special molecules. The submicron scale roughness of the surface along with surface energy of super hydrophobic natural entity such as lotus leaf, leg of water strider, petal of red rose etc. is mimicked on the cellulose fibre with the help of EB radiation (Fig. 1a and 1b).



**Fig 1a:** Images of contact angles and beading of water and oil

This material turns into a super hydro



**Fig 1b: SEM:** Micro roughness of cotton fibre

phobic and super oleophilic superabsorbent (Fig.1a). This separates oil and low surface energy toxic liquids from aqueous media by 30-40 gm/gm ratio (Fig. 1c). This can work at high temperature, in acidic, alkaline and neutral media. It can separate oily substance from immiscible as well as emulsion mixtures. The material can be used for multiple times and oil can be removed from absorbent by squeezing or centrifuge [3].

***ii) Polyolefin/clay nano composite development using compatibilizer prepared by EB assisted grafting***

Low density polyethylene (LDPE) is widely used nowadays for various applications, which include packaging, cable insulating and sheathing materials etc. because of its good mechanical properties, resistance to chemicals, good processability, low cost, reusability, high backbone strength and resistivity, resistance to shock etc. Despite this large number of applications, it has some drawbacks, such as a low modulus, low tensile strength, low impact strength, low

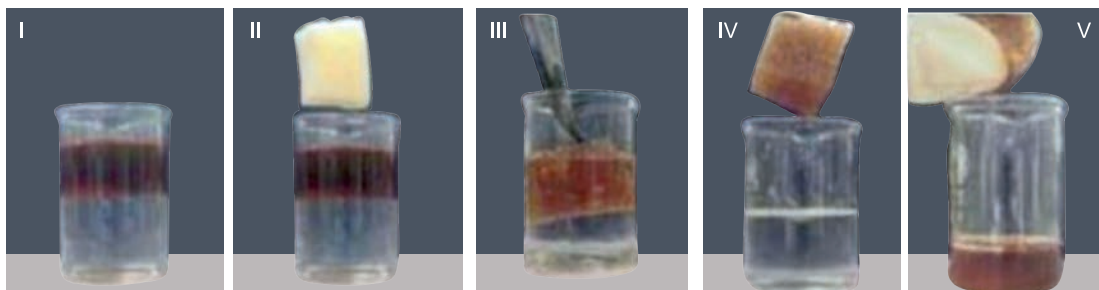


Fig 1c: Separation of oil from oil/water mixture

flammability, low thermal stability etc. On the other hand, nano hybrid formation from polymers and inorganic clays (layered silicate) is reported to increase the mechanical properties, dynamic mechanical properties, barrier properties, thermal properties, crystallinity and insulation, and allow zero smoke and halogen free flammability etc. But the formation of LDPE (low density polyethylene)/clay nano composites with uniform and reproducible properties is a persisting challenge for more than a decade. People have tried many ways to design the interface between polyolefin/clay composites to improve uniformity, degree of dispersion and strength of interface. But the results are not satisfactory as the clays are hydrophilic and polyolefins are hydrophobic in nature. We have designed the interface to overcome these challenges with the help of radiation grafting.

A polar group, methacrylic acid (MAA) was grafted onto LDPE (low density polyethylene) by gamma radiation to increase the compatibility of LDPE with organically modified clay by reducing the hydrophobicity of LDPE. The clay, Cloisite 20A, was melt-mixed with g-LDPE (grafted LDPE) to synthesize a nanohybrid and the mixes were subsequently compression moulded to make a sheet, which

was used for characterization (Fig. 2a). The interlayer distance ( $d$  values) change in wide angle X-ray spectroscopy (WAXS) confirmed nano hybrid formation, which was later directly seen by TEM (Fig. 2b). Young's modulus of nano hybrid was increased considerably by a small amount of clay incorporation (by 50% and 85% for 2 and 8 wt% clay) keeping the elongation-at-break unchanged (Fig. 2c).

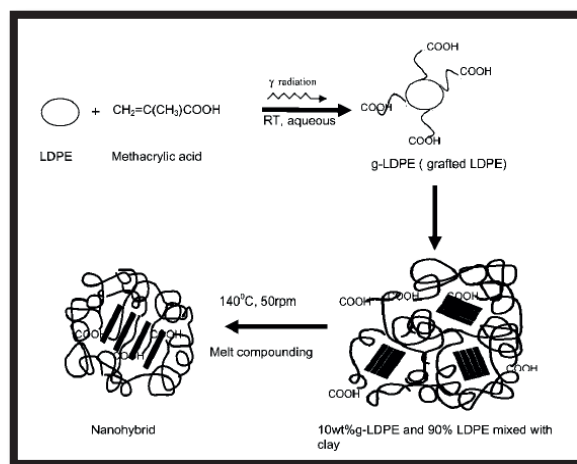


Fig. 2a: Schematic diagram of Nano hybrid synthesis

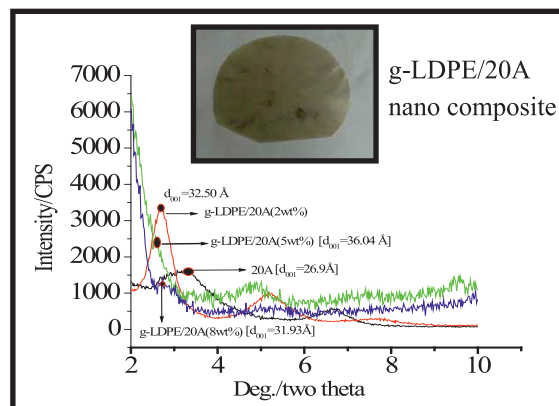


Fig. 2b: XRD of g-LDPE/20A nano composites

Thus, by this rapid, clean, user friendly and well controlled technique, pure and high-performance nano hybrids with uniform reproducible properties were developed successfully [4].

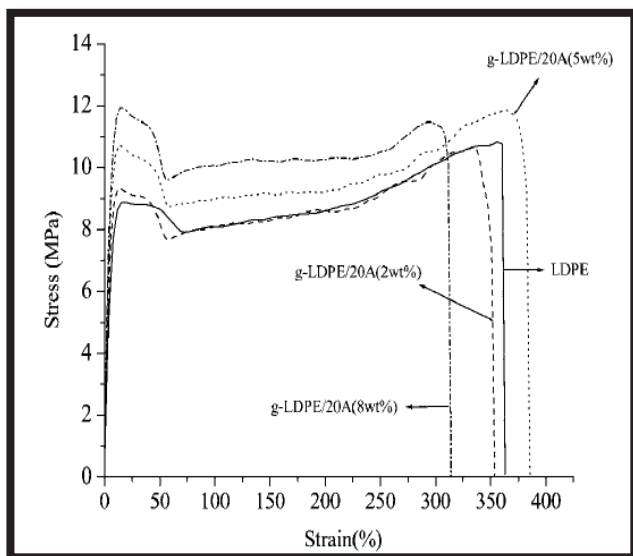


Fig 2c: Tensile properties of g-LDPE/20A nano composites

### iii) EB cross linked Nylon/clay nano composites with high mechanical performance and reduced water absorption property

Nylons or polyamides are high performance, widely used materials. They are semi crystalline thermoplastics with attractive physical and mechanical properties, which make them important in many end-use industrial applications. However, one common property of all polyamides is that they are hygroscopic, which makes their applications limited. They absorb moisture or liquid water at substantially higher levels than most other engineering plastics. Water has a plasticizing effect. Water molecules get into intermolecular spaces and as a consequence, intermolecular force is reduced. As a result, processing

(compounding, moulding, welding, etc.) and end-use performance (mechanical, dimensional, surface appearance, etc.) are seriously affected as water acts as a plasticizer. Thus, our goal was to develop a nylon based material with reduced water absorption capacity and uncompromised mechanical properties.

We have developed Nylon6/clay nano composites with reduced water absorption capacity and improved properties derived from nano building block formation (i.e. nano composites) (Fig. 3). Three dimensional EB cross linked network are introduced, which does not hamper the nano scale dispersion of clay here. Thus, both nano technology and radiation technology are used simultaneously to develop nylon based nano composites with improved industrially pertinent properties [5].

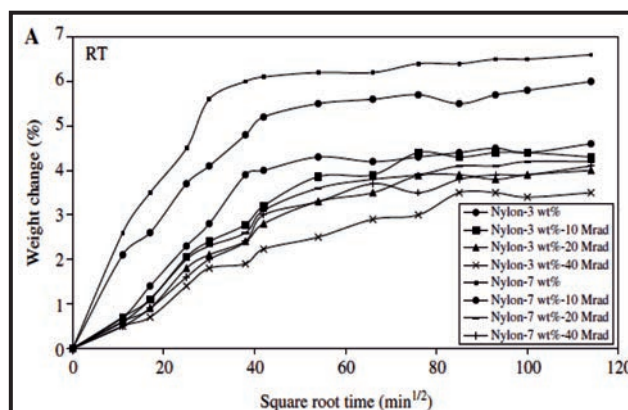
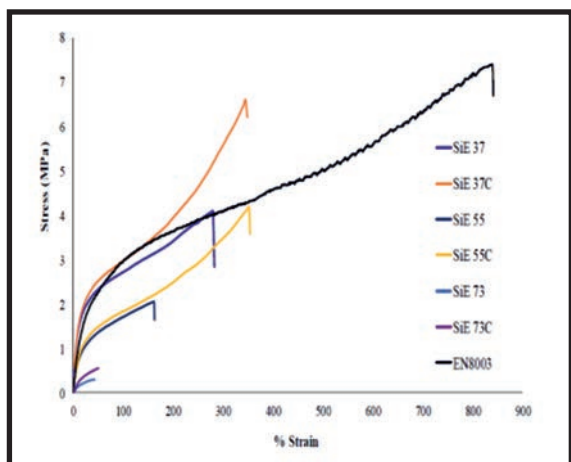


Fig. 3: Water absorption properties of Nylon6/20A nanocomposites

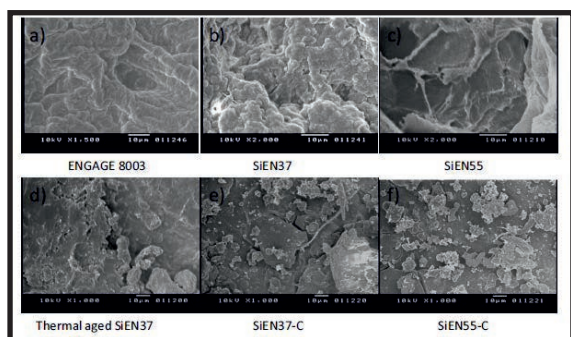
### iv) Polymer blends by radiation assisted grafting for high temperature application

Next, silicone rubber can withstand high temperature but its processibility is very poor. On the other hand, POE

(polyolefin elastomer) is a polymer, which has excellent thermal stability and processability. So, those two polymers can be blended. For high temperature applications we have developed some silicone/ENGAGE based blends. ENGAGE is relatively new polyolefin elastomer (POE) invented by Du Pont. We have developed some grafted ENGAGE and mixed it with virgin ENGAGE and silicone rubber. The presence of polar ENGAGE enhances the dispersion of silicone phase dramatically, and this is responsible for high temperature withstanding (Fig. 4a). As a result the thermal aging properties of the blends are

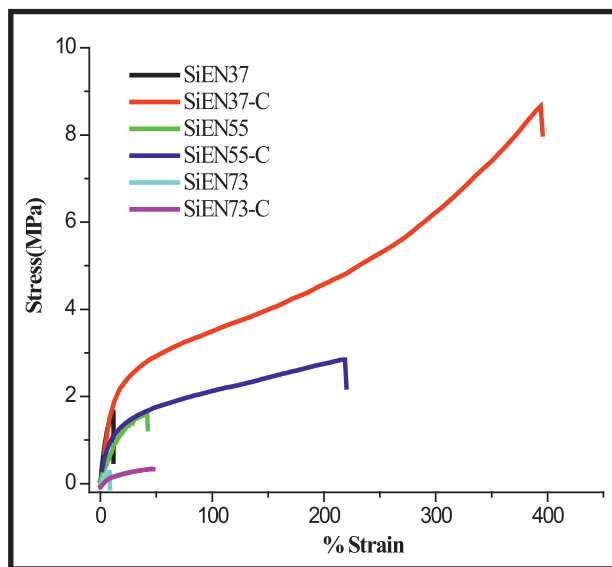


**Fig. 4a:** Surface morphology of Silicone/Engage 30/70 (SiE37) and Silicone/Engage 50/50 (SiE55) in presence and absence of compatibilizer (MAA grafted ENGAGE)



**Fig 4b:** Mechanical properties Silicone / Engage blends with and without compatibilizer (MAA grafted ENGAGE)

increased remarkably [Fig. 4b, Fig. 4c]. This blend has potential for being applied in high temperature field as it shows excellent mechanical properties and thermal properties.



**Fig 4c:** Mechanical properties Silicone/Engage blends with and without compatibilizer (MAA grafted ENGAGE) after 8 weeks aging

#### ***v) High temperature and high radiation resistant polymer blends compatibilized by radiation treated constituent elastomer***

Some high temperature as well high radiation resistant polymer blends are developed in our laboratory. We have used ethylene-propylene-diene elastomer (EPDM) and Silicone rubber and blended them with a surfactant, which was developed by radiation assisted grafting. EPDM was grafted by methacrylic acid (MAA) and 10wt% of that grafted EPDM was used to compatibilize EPDM and silicone rubber. The developed blend showed satisfactory high temperature and high energy radiation resistance.

**vi) An electron beam radiation cross linked recyclable LDPE/'EVA-embedded nano clay' nano composites**

We have developed some polyolefin/clay based nano composites [Fig. 5]. In that nano composites we have utilized both nanotechnology and EB technology simultaneously and consequently some properties are improved in such a degree, which is not possible only by either one individually. Interestingly, the morphology of nano composites (nano scale dispersion) is not disturbed. Due to the combined contribution of nano composites formation and EB cross linking, the degree of the required degree of EB cross linking is not so high. That is the reason why recycling of those composites will not be so complicated [6].

Similarly, we have developed some high radiation resistant blends of EVA/EPDM, HDPE/EPDM, LDPE/EPDM etc. [7-10]. We have also designed the interface of silicone rubber/EPDM blends

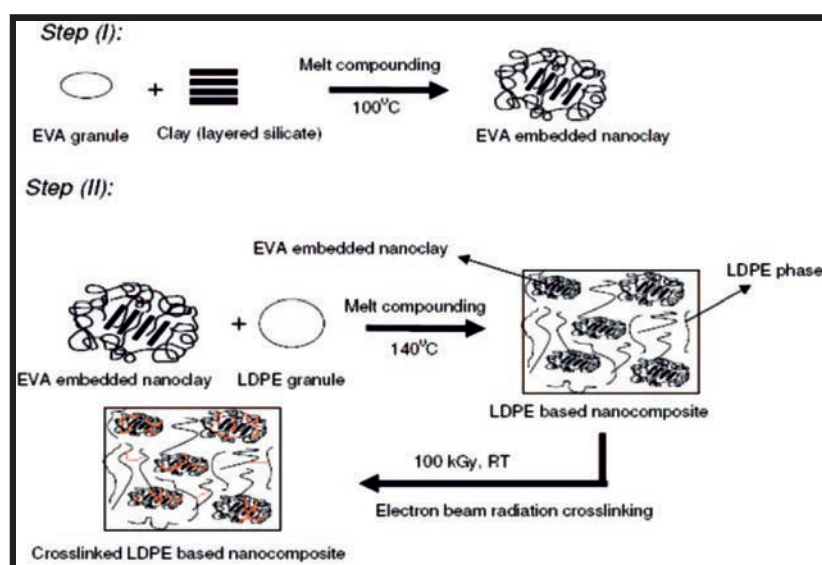
by EB assisted grafting and obtained considerable degree of property improvement.

## Conclusion

Radiation technology possesses a remarkable potential for molecular scale surface and interface engineering. Only two keys, grafting and cross linking can change a material surface wettability (super hydrophobicity, super oleophilicity etc.), surface topography (micro roughness), surface energy, compatibility between two or more polymers, polymer and nanoparticle, and nature of interface (strength, thickness and quantity). Hence, radiation technology not being confined into single polymer system must be used in multi component polymeric system as well.

## Acknowledgements

We acknowledge the tireless effort of Mr. Rabindra Patkari and Mr. Vittal



**Fig. 5:** Schematic diagram of LDPE/EVA/20A nanocomposite synthesis. EVA: poly (ethylene vinyl acetate); LDPE: low-density polyethylene.

Shrimangle for all kinds of help for our R & D in EBPS.

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# Development of Prototype Zone Radiation Monitor (ZRM-Mark 2) for Civilian areas having Ethernet connectivity

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## Introduction

Use of Zone Radiation Monitor is to detect gamma radiation dose rate above a certain threshold values to facilitate the experts in mitigating the source causes and protecting personnel from over exposure. We know that for general public the exposure limit is 1mSv and for occupational radiation worker is 50mSv annually. Essentially, the concept of ALARA (As Low as Reasonable Achievable) should be followed. For measuring this exposure, Radiation monitoring instruments are required and one such instrument, i.e. Zone Radiation Monitor (ZRM) has been developed to alert and avert the unnecessary exposure to people. A radiation monitor is the first line of defense against unnecessary radiation exposure. Safety and precaution shall be prime concern of any Radiation industry. To make sure we must use instruments in the field and ensure safety of workers as well as general population.

## Development of Prototype Zone Radiation Monitor for Industries (ZRM-Mark 1)

In BRIT, we were successful in developing prototype Zone Radiation Monitor for Industries, which was made specifically for Radiation Industries. It had radiation detection, alarm generation and industrial analog and digital communication modules for interfacing with Programmable Logic Controller (PLC) and Supervisory Control and Data Acquisition (SCADA). Monitoring of gamma radiation using this instrument would help to detect radiation in the vicinity and in turn raise alarm in case radiation goes above a threshold level. This would in turn save industrial personnel from unnecessary exposure to radiation as well as alert them to take steps for mitigation of radiation source in that zone. The communication system modules will send radiation data to a centralized PLC/SCADA based control system and hence can be used for data

logging. The onboard module includes GM tube (LND7121 that works in the range of 450V to 600V HVDC) based counting circuit, Industrial 4-20mA current loop and RS-485 MODBUS based communication for industries. The calibration of this newly designed instrument was done for a range of 0mR/hr to 100mR/hr which depicted that within this range of measurement, the accuracy was found to be within 10% of actual reading. The details of these are mentioned in Chapter 3 of BRIT Annual Report 2017-18 [1].

### Development of Prototype Zone Radiation Monitor for Civilian area (ZRM-Mark 2)

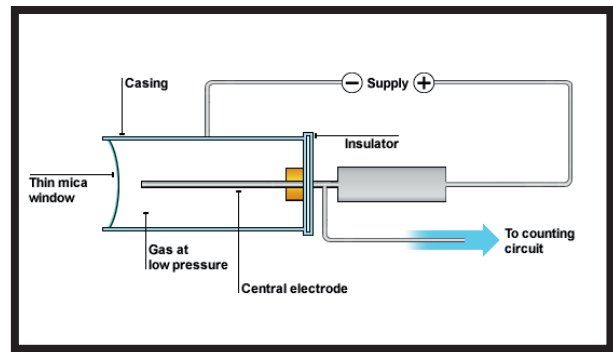
Further requirement which was conceived by BRIT was towards the development of **Zone Radiation Monitor (ZRM-Mark 2)** and this instrument would be useful for measuring dose rate in the vicinity of **Civilian area**, where it can be mounted to measure the radiation exposure for common man.

ZRM-Mark 2 was developed at BRIT which denoted radiation dose rate in terms of either mR/hr or  $\mu\text{Sv/hr}$  and would generate sound (alarm) and flash light, if radiation dose rate is high and beyond permissible limits for various zones for which the threshold would be set. It will also communicate the Radiation dose rate data and other instrument parameters through **TCP/IP Ethernet**. This particular communication protocol was chosen as

there is Ethernet connectivity almost anywhere now a days, namely, offices, ports, laboratories etc. Thus the instrument that was needed should be very rugged and was made from well known Geiger-Muller tube (GM Tube) [4]. Apart from that, High Voltage DC source for biasing the GM tube, Pulse shaper and counter circuit using microcontroller, readout display and alarm generation are all put into operation.

**Ethernet communication module has been implemented in the same instrument.**

GM tube is a gaseous ionization detector which is used to detect nuclear radiation by using Townsend Avalanche effect. The tube contains a gas mixture at low pressure of about 0.1 atm. The chamber contains two electrodes as shown in Figure 1.



**Fig. 1.a.** GM Tube internal structure;

The walls of the tube are either metal or have their inside surface coated with a conductor to form the cathode, while the anode is a wire in the center of the chamber.

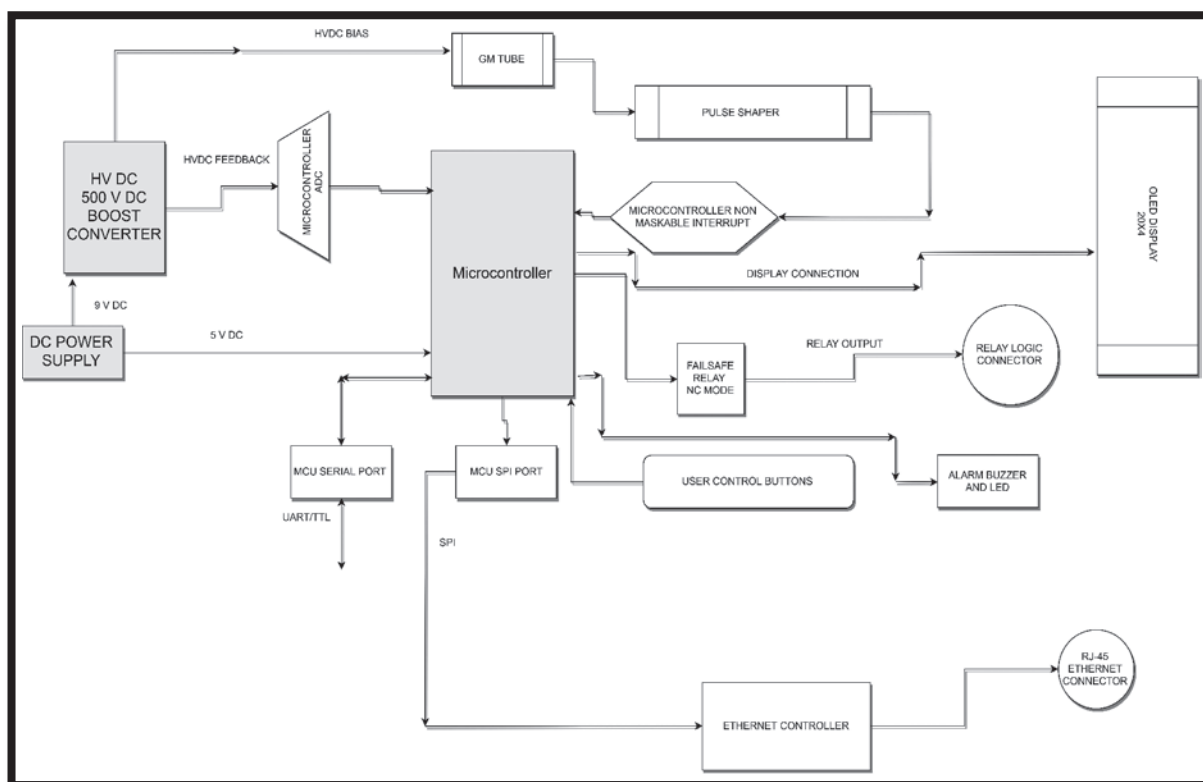


**Fig. 1.b.** GM tube with Energy Compensating shield, and Plastic Housing for the GM Tube for Environmental and Shock Protection

When ionizing radiation strikes the tube, some molecules of the gas are ionized, either directly by the incident radiation or indirectly by means of secondary electrons produced in the walls of the tube. This creates positively charged ions and electrons, known as ion pairs, in the filled gas. The strong electric field created by the tube's electrodes accelerates the positive ions towards the

cathode and the electrons towards the anode. Close to the anode in the "avalanche region" the electrons gain sufficient energy to ionize additional gas molecules and create a large number of electron avalanches which spread along the anode and effectively throughout the avalanche region. This is the "gas multiplication" effect which gives the tube its key characteristic of being able to produce a significant output pulse from a single ionizing event.

This short but intense pulse of current can be measured as a count event in the form of a voltage pulse developed across an external electrical resistor. This can be in the order of volts, thus making further electronic processing simple. Figure 2 represents block diagram of in-house



**Fig. 2** Block Diagram of the unit fabricated

fabricated unit.

The GM Tube used is **LND7807** which has the sensitivity of 150cps/mR/h that works with DC bias and ranges between 450V to 600V HVDC. The HVDC circuit has been constructed using a DC-DC boost converter that converts 9V DC to 500V DC and is a clean DC Voltage. The pulse output obtained when radiation is incident on GM tube can be seen in Fig. 3.



Fig. 3 The oscilloscope readout of GM tube pulses



Fig. 4 The output of Signal shaper

The pulses thus obtained are processed and converted into Logic level compatible pulses using pulse shaper circuit and this is shown in Figure 4. It can be read by Microcontroller based Counter assembly as CPM or CPS, as well as dead time compensation for GM tube [2].

The Microcontroller contains calibration data that converts the respective counts in terms of mR/hr or  $\mu\text{Sv/hr}$  [3]. The calibration has been done at BRIT Calibration laboratory at Vashi Complex. There is a preset Alarm radiation threshold in the system, and if the incident radiation crosses this limit, the Logic relay is activated for safety interlocking, while the Alarm LED starts to blink and Alarm buzzer starts to sound. The entire data can be seen on Instrument local display that consists of a very high contrast OLED display screen. Further Ethernet communication capability over TCP/IP communicates system data and parameters to a central logging server running on MySQL database where all the data is logged and can be monitored periodically, say after 15 seconds time gap. User can access the data and visualize the same in graphs that are updated continuously.

The present status of the development of the circuit board is shown in Figure 5.a. Use of OLED display is shown



Fig. 5.a Prototype Printed circuit board

in Figure 5.b. Use of OLED as an alternative to LCD display has been done in order

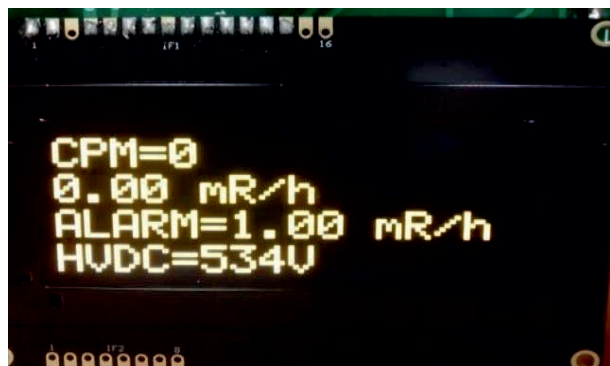


Fig. 5.b Instrument Screen readout

to provide very high contrast display for easy readability from all angles and at a given minimum distance.

Much iteration in design has been done to make HVDC power supply efficient for very high dose rate. Also, proper component placement modifications have been implemented in order to reduce the interference noise of DC to DC boost module.

The Calibration graph has been plotted between CPS and Dose rate. It is found to be almost linear for a range of 0-10mR/hr. The current Alarm threshold is visible as well as HVDC biasing voltage for the GM tube is visible. Alarms acknowledge and reset buttons connection has been provided on board. Application for visualization of data and configuration of the instrument online by Ethernet is being developed. Complete hardware unit is shown in Figure 6.



Fig. 6. Complete Hardware for the Instrument showing GM Tube, Tube Housing, PCB and Ethernet Connection

The Calibration was done for a range of 0mR/hr to 10mR/hr and residual errors were adjusted within the software as shown in Figure 7. Instrument prototype, thus developed, was found to be accurate within 5% of Actual reading which is acceptable. Figure 8 shows the scheme of mounting the instrument in the field.

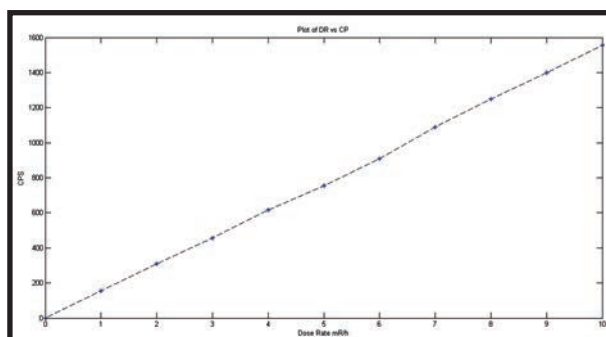


Fig. 7. The plot of Dose rate vs CPS (Calibration performed in BRIT Calibration lab)

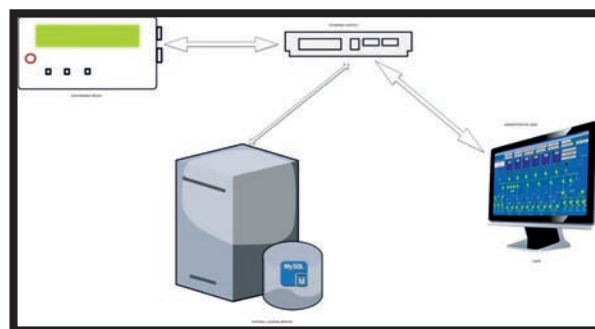


Fig. 8. Scheme of mounting the instrument in field.

### Observation

1. The accuracy of the instrument was initially  $\pm 5\%$ , which is expected to degrade once the instrument ages.
2. To mitigate this the instrument needs to be recalibrated over a period of 6 months to 1 year, subject to the accuracy if it is better than acceptable limit.
3. Higher sensitivity GM tube may be used in case of areas where high resolution of Radiation detection is required. Like LND7807 with 150 cps/mR/h has been used in this instrument.

### Conclusion

As this instrument is Civilian in nature, the mounting and interfacing is compatible with Ethernet Network. The Instrument has local display/alarm buzzer and light as well as TCP/IP Ethernet communication. The provision for a Central data logging server running MySQL database and Data Logging application in PHP pulls the data from the instrument automatically after given time delay. The time delay can be varied as per user requirement. The network parameters and instrument parameters can be modified using web browser. The instrument has capability of pushing the data over database irrespective of sampling time in case when radiation dose rate is higher than threshold. The central server shall have graph plotting

application and report generation of any particular instrument. The entire unit was fabricated by soldering components on General purpose printed circuit board and enclosing the same in a general purpose polymer box. The control button panel and display was fixed on the top cover. The instrument is being tested in the laboratories for its use.

### Future scope

- Construction of suitable IP rated instrument enclosure for the instrument developed.
- Testing of the instrument for various mechanical and electrical parameters.
- Implementing data storage and Real time clock on board of instrument.

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# Scientific Highlights for the Calendar Year 2018

✚ Export orders for various Products of BRIT that were executed during the Year 2018 are as follows:

- One Gamma Chamber-5000 (GC 5000) unit was exported to NEAD-Technology Applications & Development Company Ltd., Vietnam.

- One single supply of Cobalt-60 Irradiator source of 1000kCi (1MCi) strength was exported to REVISS Services, United Kingdom. This is the biggest order executed in the history of BRIT.

- One each of Co-60 Teletherapy Sources (CTS) was exported to Nigeria and Sri Lanka.

- Various Tc-99m Cold Kits for the preparation of Tc-99m Radiopharmaceutical Injections were exported to Molecular Supplies SpA, Santiago, Chile.

✚ BRIT signed seven MoU's for setting up Gamma Radiation Processing Plants for disinfestations, shelf-life extension for food products and sterilization applications of healthcare products.

✚  $^{177}\text{Lu}$ -EDTMP is a therapeutic radio pharmaceutical injection which is used for relieving that arises due to spread of cancer to the bones. This product was launched by BRIT during this period.

✚ Another therapeutic radio pharmaceutical injection, based on I-131, namely,  $^{131}\text{I}$ -Lipiodol,

for the treatment of Hepatocellular Carcinoma was launched by BRIT in collaboration with RPhD, BARC.

✚ AERB approval was obtained for handling Ga-68 radioisotope at BRIT and further BRIT was successful for the launch of Ga-68 based ready-to-use PET radiopharmaceuticals such as  $^{68}\text{Ga}$ -DOTA-TATE for the diagnosis of neuroendocrine tumors (NET).

✚ For the first time trouble shooting using angular gamma scanning was carried out at a Mumbai refinery for huge catalyst loss from the Fluidised Catalyst Cracking Unit (FCCU).

✚ Mo-99 in organic form was successfully used as an industrial radiotracer to identify the leaky heat exchanger in a refinery.


✚ More than 14000 Tritium Filled Sources (TFS) of various sizes, shapes and tritium content were supplied to defence establishments and used for illumination of various types of gadgets and instruments.


✚ Radiation Processing Plant Facility at Vashi Complex was certified for ISO 9001:2015 (Upgraded from 9001:2008 to 9001:2015). Surveillance audits for ISO 22000:2005 (Food Safety Management Systems) and ISO 9001:2008 were also carried out by certifying agency and found in full compliance with Standard requirement.

✚ Production of Fluorine-18 isotope for the preparation of Fluorodeoxyglucose

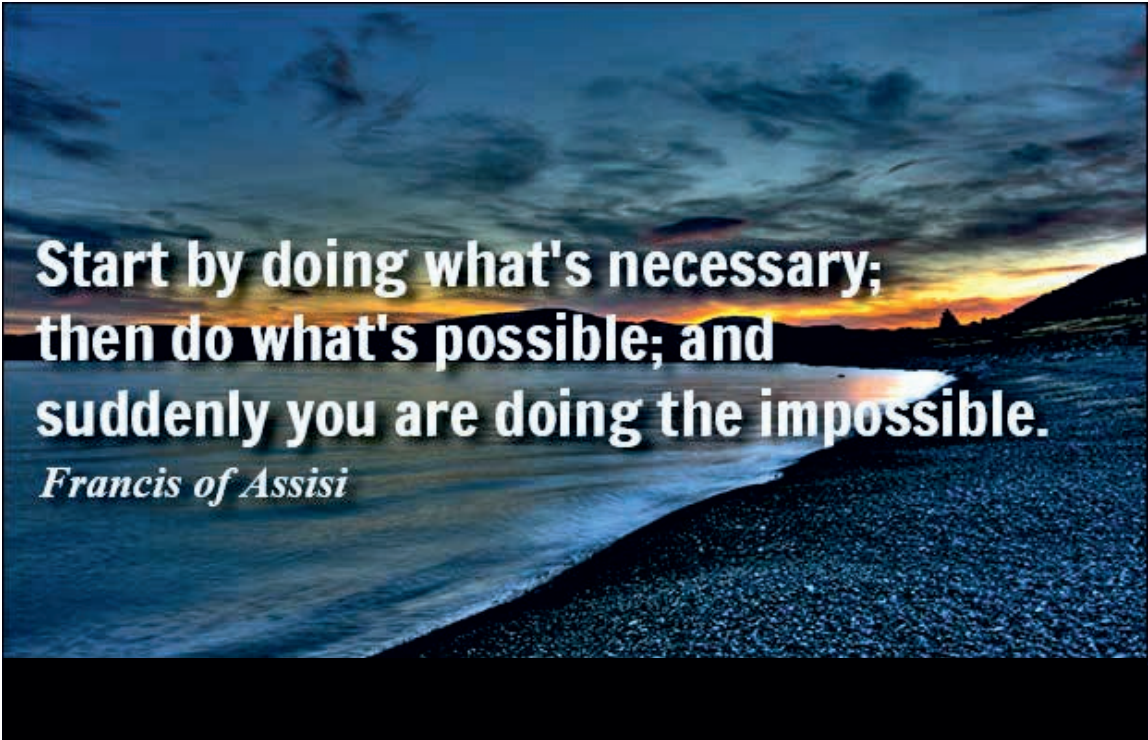
## Scientific Events

(FDG), a radioisotope for PET imaging, was carried out, after the start of operation of Medical Cyclotron Facility, Cyclone-30 at VECC, Kolkata. Regular production will start after commissioning of supporting systems and regulatory clearances.

 Towards achieving GMP compliance, <sup>131</sup>I-mIBG Production Facility has been installed and cold commissioning was completed. Trial cold runs for pharmaceutical validation are presently being undertaken.

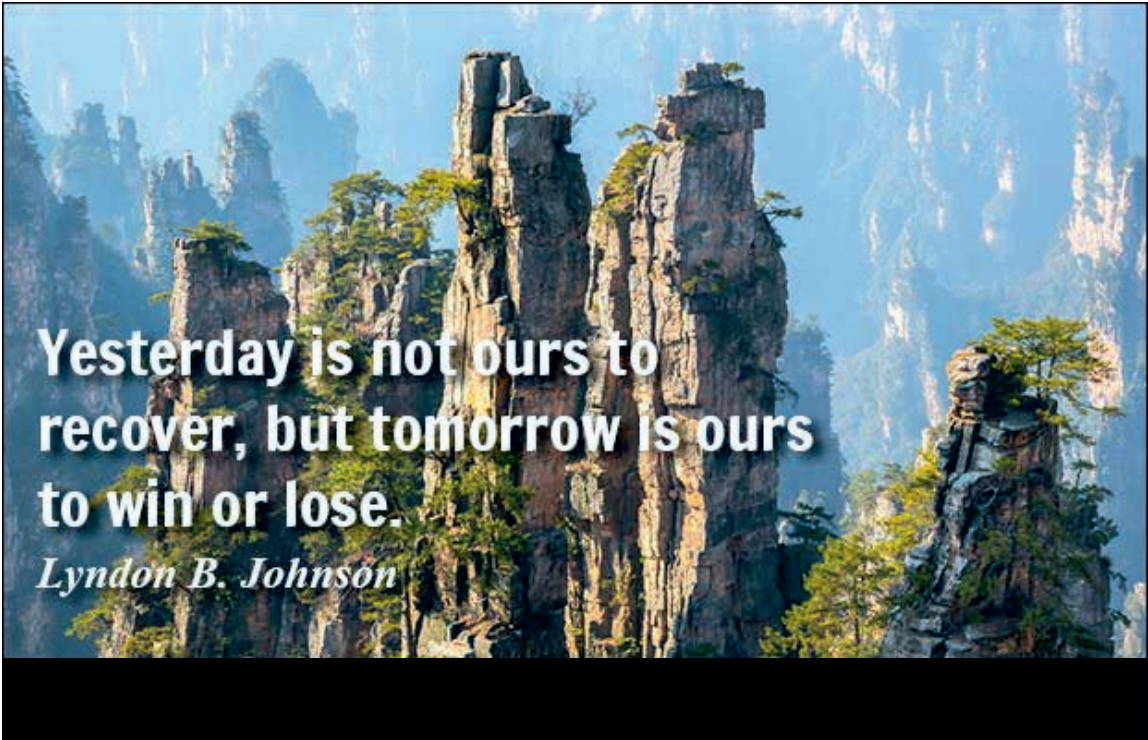
 Facility for the preparation of cold kits for Tc-99m radiopharmaceuticals is already certified for its compliance to Good Manufacturing Practice (GMP).





**Start by doing what's necessary;  
then do what's possible; and  
suddenly you are doing the impossible.**

*Francis of Assisi*



**Yesterday is not ours to  
recover, but tomorrow is ours  
to win or lose.**

*Lyndon B. Johnson*

# Swachhata

## Green & Clean BRIT



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