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ESTIMATION AND MEASUREMENT OF LEAK FLOW THROUGH SLITS/PRE-CRACKED PIPES

Reactor Safety Division

Introduction

The basic philosophy of Leak-Before-Break (LBB), as the name implies, is based on detection of Leak from a pipeline, carrying a high-energy liquid, Before it Breaks. The LBB methodology is now accepted as a justifiable approach in eliminating the traditional Double-Ended Guillotine Breaks (DEGB), in the design of high energy piping component where it is applicable. In the design of a nuclear reactor, sufficient safety features are incorporated, to limit the consequences of a DEGB. But the establishment of LBB concept has been helpful in the reduction of a large number of structures required for pipe whip restraints and jet shields. This is also beneficial in terms of cost reduction and radiation exposure. Hence, it is extremely important to detect and measure leak, from any high energy piping in a nuclear reactor.

LBB is ensured by demonstrating three levels of safety assessment against DEGB. Level 1 is inherent in the design philosophy of ASME Sec. III, which is normally followed in piping design. Ductile and tough materials are widely used in nuclear power plant piping, because of their high resistance to catastrophic rupture. The design is carried out with a well defined factor of safety on code-specified probability strength. The demonstration of Level 2 safety, consists of postulating a surface crack, which may go undetected during non destructive testing. It is then shown through fatigue analysis, that there is insignificant crack growth of this surface crack, during the entire reactor life. In Level 3 safety assessment, a through-wall leaking crack is postulated, at the location of maximum stress with worst material properties. The size (length) of the through-wall crack should be such that, the calculated leakage rate of fluid discharged from the crack under normal operating load, is equal to the immediate shutdown action limit. An elasto-plastic fracture mechanics assessment is performed, to demonstrate that adequate margins exist against the onset of unstable crack extension. The minimum margins which must be met are twice on the size (length) of crack and a square root of two on the extreme load, acting on the pipe, containing the through-wall crack.

Generally, it has been observed that a leak rate of 0.05 kg/s is easily detectable by suitable instrumentation. On this, applying the US Nuclear Regulatory Commission’s safety factor of 10, the reliable leak detection capability is considered to be 0.5 kg/s. A manual reactor shutdown is initiated on this flow value, to bring down the reactor power. The concept of LBB is now widely used to design the Primary Heat Transport (PHT) system piping, of a nuclear power plant. This approach has been used to design the 500 MWe Indian Pressurized Heavy Water Reactors (PHWRs) at Tarapur [1].
A systematic study on LBB has been initiated at the Reactor Safety Division to determine two important parameters: namely Crack Opening Area (COA) and critical leak flow rate through cracks/slits. A computer code named **Crack_Slit Flow Analysis (C_SFA)** has been developed with analytical models, to determine COA and leakage flow rate. The developed leakage flow rate model is validated [2] against published data [3,4,5] specific to Pressurized Water Reactors. In order to further validate the physical models for PHWRs and Advanced Heavy Water Reactor (AHWR), an experimental facility has been set up at Heat Power Laboratory, at the Mechanical Engineering Department, Jadavpur University, Kolkata, under the Board for Research in Nuclear Sciences (BRNS). Prof. S.K. Saha and Dr S. Das were the principal investigators of this facility.

The experimental facility consists of High Pressure and High Temperature (HPHT) loop, pressurized by nitrogen system and a test section, housing the slit/ pre-cracked pipe. The HPHT system is designed to operate for a pressure and temperature range of 70 to 90 bar and 200° C - 270° C respectively, having a maximum leakage flow rate of 0.8 kg/s. This condition simulates the reactor condition of existing PHWRs and the cold leg of proposed AHWR. Prediction of crack flow with physical models involves uncertainty in COA determination and consequently in critical mass flow rate prediction. To minimize the uncertainty level in the prediction, it was also decided to carry out experiment with pipes having slits with definite flow geometry.

The loop is well instrumented, to measure the required parameters, to validate the physical models developed at the Reactor Safety Division. The typical feature of this facility is that, during the course of the experiment, pressure and temperature of the system is maintained constant at the crack/slit upstream as expected for leakages, within the leak handling system capacity of a Nuclear Power Plant (NPP). Similar experiments have been conducted by the US Nuclear Regulatory Commission [3,4,5] for assessing the safety and integrity of high energy piping systems.

**Analytical modeling of code C_SFA**

Determination of COA and leakage flow rate through cracks/slits are the two aspects of this code, for which models are developed. Following is a brief description of these two models.

**Crack Opening Area (COA)**

For determination of crack opening area, several models e.g., Tada-Paris Model [6], Bertholome et al [7] model, Kastner et al [8] can be used. Tada-Paris model is the most widely used model for calculating COA. In this model, crack opening area depends on the load acting on the crack plane, material property e.g. Young’s Modulus of the piping material, pipe dimensions, crack size and finally on the crack orientation. For circumferential crack in tension and bending, crack opening areas are calculated as follows:

\[
A_t = \sigma_t \left( \frac{\pi R^2}{E} \right) I_t(\theta')
\]

\[
A_b = \sigma_b \left( \frac{\pi R^2}{E} \right) I_b(\theta')
\]

\[
A = A_t + A_b
\]

where \(I_t\) and \(I_b\) are integrals arising in energy method (castigliano’s theorem); \(\theta'\) is effective semi crack angle, \(A\), Crack Opening Area is the sum of areas due to tension and bending loads.
Semi-crack angle $\theta'$ is modified to effective crack angle ($\theta_{eff}'$) to take into account the small plasticity effect.

\[
\theta_{eff}' = \theta' + \frac{K'^2}{2\pi R \sigma_i^2}
\]  \hspace{1cm} (3).

Following assumptions are considered for Tada-Paris model:

a) The formula has been derived for $R/t$ ratio equal to 10.
b) Estimation formula is expected to yield slightly overestimated results for $R/t$ near 10.
c) For smaller $R/t$ ratio, the degree of overestimate would increase.

**Leak Flow Rate Estimation**

This model consists of two parts. The first part deals with pressure drop formulation in the flow path (cracks and slits) and the second part deals with models for critical mass flux estimation. For estimation of the flow rate, these two models work interactively through an iterative procedure.

**Pressure Drop Formulation**

Pressure drop estimation for crack and slit flow paths are essential to estimate the critical pressure, which in turn, determines the critical mass flux. Nature of roughness of the crack bed surface, depends on Crack Opening Displacement (COD). For large COD, mean flow path is straight and global roughness acts as surface roughness, as against global and local roughness accounted together, for small COD with zig-zag flow path. This is illustrated in Fig. 1.

Surface roughness parameter defines the peak-to-peak roughness of the crack face surface. This is used in the calculation of friction factor and pressure loss due to friction, for fluid flow through a crack in a pipe. Earlier, Surface Roughness ($S_r$) was assumed to be invariant with respect to Crack Opening Displacement (COD) and Crack Width (CW). However, appropriate surface roughness could be large (global) or small (local) depending on whether the COD or CW was large or small respectively. For cracks, flow path is complicated as compared to flow path of slits where roughness is due to wall friction only.

Thus, the total pressure drop, along the flow path through the crack, is the sum of the different pressure drop components as listed below:

\[
\Delta P_{tot} = \Delta P_e + \Delta P_f + \Delta P_{aph} + \Delta P_{aa} + \Delta P_K
\]  \hspace{1cm} (4)

where, $\Delta P_e$ the entrance loss is given by,

\[
\Delta P_e = \frac{G^2 V_{lp}}{2C_d^2}
\]  \hspace{1cm} (5)

$C_d$ is the contraction coefficient, for which Clarke [9] recommended the value of 0.65. Bean [10] has
recommended $C_d = 0.95$ for tight cracks. An average value of 0.85 for $C_d$ has been adopted for the model.

$\Delta P_f$ is the pressure drop due to friction given by,

$$\Delta P_f = 0.5 f G^2_c \left[ \frac{v}{D_h} + \left( \frac{L-L_f}{D_h} \right) \right] \left[ (1-\beta)v_{lo} + \beta (v_f + x(v_f-v_l)) \right] \ldots (6)$$

where friction factor $f$ is given by John et al [11],

$$f = \left( \frac{3.89 \log \frac{D_h}{S_r} - 0.866}{\sigma} \right)^2 \ldots (7)$$

Pressure drop accounted for change in acceleration from phase change is given by,

$$dP_{aph} = G_c^2 d \left[ (1-x)v_f + xv_g \right] \ldots (8)$$

This expression can be integrated from $Z = Z_t$ (ie. $x = 0$) to the critical condition at the throat ($x = x_c$) and is expressed as,

$$\Delta P_{aph} = G_c^2 \left[ v_{lo} + x_c (v_{ge} - v_{ke}) - v_{lo} \right] \ldots (9)$$

The acceleration pressure drop due to area change along the flow path is given by,

$$\Delta P_{aas} = \frac{1}{2} K_a G_c^2 v_{lo} \ldots (10)$$

where $K_a$ is the pressure loss coefficient due to change of flow area.

Finally, $\Delta P_K$ is the pressure drop due to bends, turns and protrusions in the flow path that occur in actual cracks. For slits this term becomes zero.

$$\Delta P_K = \frac{1}{2} K G_c^2 v_{lo} \ldots (11)$$

where $K$ is the loss coefficient for turns and bends [2].

The critical pressure $P_c$ is the difference between the initial stagnation pressure $P_o$ and total pressure drop $\Delta P_{tot}$.

$$P_c = P_o - \Delta P_{tot} \ldots (12)$$

For a given stagnation condition and crack geometry, the critical mass flow rate ($G_c$) is calculated by iterative solution of equation (4) and equation (12). The values of $G_c$ and $P_c$ are assumed to be correct when the relative errors between two successive iterations are of a less than a specified value.

**Critical Flow Models**

The critical mass flux ($G_c$) is estimated with various critical flow models namely Burnel Model, Homogeneous Non-equilibrium Models (HNMs) and Homogeneous Frozen Models (HFMs). Based on the thermal-hydraulic conditions, these models are used. Following is a brief discussion of these models:

**Burnel’s Model**

This model [12] takes into account the delay caused in nucleation which results in high flow rates through nozzles and orifices. The critical mass flux is given by,

$$G_c = \left[ 2 \rho_l (P_s - (1-c)P_{sat}) \right]^{0.5} \ldots (13)$$

where ‘c’ is given by Weisman and Tentner [13]

$$c = 1.0 - 0.8645/\exp(\sigma_{Psat}/6.286\sigma_{12.32 bar}) \ldots (14)$$

where all properties are based on upstream condition.

**Homogeneous Non equilibrium Model**

In Henry’s [13] homogeneous non-equilibrium model, high energy flow through cracks and slits, modeled as critical flow through a pipe, are characterized by the following attributes: a large $L/D_h$ ratio, a relatively small residence time and a large relative surface roughness. Henry [15, 16] had simplified his earlier model based on the following assumptions,
i. for homogeneous flow: $k = 1$,
ii. for isentropic flow: $s = \text{constant}$,
iii. Liquid is incompressible fluid: $v_l = v_{l0}$,
iv. Vapour is at equilibrium with pressure:

With these assumptions, the expression for critical mass flux reduces to

$$\frac{1}{G_c^2} = -\left[ x \frac{dv}{dP} + \left( v_g - v_{l0} \right) \frac{dx_E}{dP} \right]$$  \hspace{1cm} (15)

where,

$$N = \begin{cases} \alpha x_E & \text{if } x_E < 1/\alpha \\ 1 & \text{if } x_E > 1/\alpha \end{cases}$$

Where $\alpha = 20$, $x_E$ is the equilibrium quality given by:

$$x_E = \frac{s_E - s_{li}}{s_g - s_{li}}$$

**Homogenous Frozen Model**

Experiments have identified a sharp pressure gradient at the pipe exit for a large length-to-diameter ratio. Experiments have also shown that the flow gradually accelerates along the flow path, with rapid acceleration occurring at the exit, where the two-phase mixture was seen to be “froth flow”. As a result of rapid acceleration at the exit, the composition of the fluid just upstream of the exit remains “frozen”, while the flow undergoes further acceleration. In this model, the average velocity of the two phases is assumed to be equal and both the phases remain in thermodynamic equilibrium, up to the end of the crack, but phase change is frozen at the throat. Nathwani et al [17] have recommended the critical mass flux relation as,

$$G_c^2 v_e^2 = \frac{P_i^2}{\left( xv_g \right)_e}$$  \hspace{1cm} (16)

The present authors adopted the critical mass flux estimation formulation as given by Henry and Fauske [16] and suitably modified it as given,

$$G_c^2 v_e^2 = \frac{\gamma}{\gamma + 1} \left( xv_g P \right)_e$$  \hspace{1cm} (17)

**Experimental Validation Programme**

The main aim of this experimental facility [18] is to estimate leak rates through slits and pre-cracked pipes of various sizes, for the system pressure range of 70-90 bar and a maximum temperature of 270°C and to validate the C_SFA code. The generated data has also been used to assess the applicability of the critical flow models of safety analysis code RELAPS/mod3.2 [19] for slit geometry which are validated against critical flow for nozzles (Marviken’s Test) and large flow area (Edward Pipe Blowdown Experiments).

**Facility description**

The experimental facility (Fig. 2) consists of High Pressure and High Temperature (HPHT) loop. This loop consists of Buffer Chamber containing subcooled water at reactor pressure and temperature, Nitrogen system for maintaining the system pressure and thermic-fluid heating system to externally heat up the water in the Buffer Chamber (BC). The HPHT loop is properly insulated to avoid heat loss. The technical feasibility (design and operation) of the system was assessed with the help of the thermal-hydraulic computer code RELAPS/MOD3.2 against a slit leak flow rate of 0.8 kg/s, for which the design of the facility has been made. Inventory of nitrogen for holding the system pressure at 90 bar for 15 minutes against a flow rate of 0.8 kg/s, rating of thermic fluid heater, efficiency of external heating mode, sump capacity and concrete temperature of the sump for 15 minutes discharge, jacket volume to limit the pressure below 2 bar etc. are some key design features that were assessed. Operation (heating and pressurization) and experimentation were assessed with the code and a part of Standard Operating Procedure (SOP) was evolved through this assessment. Fig. 3 gives the nodalization of the simulation model and predicted system behaviour under a flow of 0.8 kg/s.
A brief description of the component/systems is given in the following paragraphs and Fig. 4 shows some of the components of this facility.

**Buffer chamber**

The Buffer Chamber (BC) is made of a seamless pipe (600 NB Sch. 100 and 5.5 m in height) of grade (ASTM-A106 Gr. B) with an average thickness of 42 mm. The volume of this vessel is 1.25m³. A helical path is provided on the outer surface of this vessel for thermic fluid flow. The buffer chamber water is heated by Thermic Fluid. A jacket on BC outer surface with helical path provides the flow path for the thermic fluid. Excessive heat loss is prevented by a thick layer of insulation applied to the outer surface of BC. Safety valves and rupture disk at the top of BC take care of any sudden overpressures.

**Nitrogen System**

Constant primary system stagnation pressure is maintained with the help of Nitrogen system containing forty eight N₂ cylinders at 140 bar pressure connected to a common manifold. The pressure in the BC is maintained with a pressure controller and a pressure control valve.

**Thermic Fluid Heating System (TFHS)**

The Thermic Fluid Heating System consists of a heating section, circulating pump and a blower. Thermic fluid (thermibol-59, boiling point 350°C) is heated by burning High Speed Diesel (HSD) in a burner of the heating section. The system is designed for 5 bar pressure and 350°C temperature.
Fig. 3 : Techno-feasibility study with RELAP5 simulation model
**Water Treatment Plant (WTP)**

An iron removing filter and water softener have been incorporated to ensure that water used for the experiments has the following specifications:

a) Iron \(< 0.20 \text{ ppm as Fe}\)

b) Hardness \(< 5.0 \text{ ppm as CaCO}_3\)

In order to avoid corrosion in CS pipe of HTPT loop, pH will be maintained within a range of 9.8 to 10.3.

**Test Section**

Slits are generated by EDM machine on 100mm NB sch 80 pipes. One such pipe is placed in the Test Section enclosed by a jacket of 1m³. A 100 mm NB pipe connects the jacket with a 5 ton capacity hotwell which acts as a sump.

**Instrumentation and Safety of the Facility**

The loop is instrumented with Pressure Transmitters (0-100 bar), RTDs (30°C to 350°C), Coriolis type mass flow meter (0.01 to 1.0 kg/sec) and level transmitter. A dedicated control panel has been designed and fabricated for the facility, which consists of a Machine Control Centre (MCC) for the pumps, blowers and pneumatically controlled valves, interlocks, displays, temperature, pressure and level-indicating controllers.
It also contains a Data Acquisition System and an alarm annunciation system amongst other features.

The design, fabrication and erection of all the above components/systems have been carried out as per ASME standards. Keeping in view of the safety considerations, a single level failure analysis has been carried out as part of the design cycle. As part of safe operation, a manual in the form of “Standard Operating Procedure” (SOP) has been prepared.

Operation

The different phases of operating procedure of the experimental loop can be summarized as follows:

Priming of the system

Water is filled up in the overhead tank from the sump, with the help of a makeup pump through the WTP. Later, water is filled up in BC and test section with the help of a reciprocating pump.

Pressurization and Heating

Cold Pressurization: The BC is pressurized for 2 bar and water is drained to establish the initial level in the BC. After attaining the desired operating level, the BC is further pressurized to 20 bar pressure.

Hot Pressurization: A pressure of 40 bar and temperature of 200°C is attained within 30 minutes by heating with TFHS. Further pressurization is done in the pressure range of 67-87 bar, with the help of pressure controller and pressure control valve (PCVN, Fig. 3). The test temperature range (220°C-270°C) is achieved, by heating from controlled TFHS and wall stored heat (a relatively slow process, 30 min.). As the temperature gradient between TFHS and BC decreases, the efficiency of TFHS goes down. Desired pressure range of 70-90 bar is achieved, during the process of attainment of desired temperature range. Fine-tuning of the pressure set point is carried out with feed and bleed of nitrogen.

Steady-State Experimentation

After attaining the desired pressure and temperature in the buffer chamber, flow through crack/slit is established by opening the valve (PCV2, Fig. 3) connecting the BC and the test section. The steady state conditions of crack/slit upstream pressure and temperature are achieved, with the help of pressure and temperature controllers. Fig. 5 shows trends of a couple of parameters during the experimentation period, indicating a successful attainment of the steady state conditions, upstream to the crack/slit. The steady crack flow is measured for 10-15 minutes and for 3-5 minutes for slit flow. Influence of pressure variation with a constant subcooling and subcooling variation with a constant pressure on crack/slit flow is done, through a pre-planned pressurization and heating program, to avoid release of nitrogen to the environment as well as heat loss to the environment.

Shutdown of the System

Shutdown of the system is done, with the help of blowers and pumps of the TFHS, which cool the thermic fluid and which in turn cools the BC water to 200°C. The system is depressurized from 90 bar to 40 bar with the help of a bleed valve. The system is further cooled to 40°C and depressurization is continued up to 5 bar. BC water is drained through a blowdown valve and further flushing is accomplished with 2 bar nitrogen pressure. The vessel (BC) is isolated from the nitrogen loop and kept at 1.5 bar pressure. The whole process takes 6 hrs.

Experiments and Results

A number of experiments were conducted. The details of these experimental runs and related results are described below.

Experiments with Pre-cracked Pipes

Experiments have been conducted for the determination of critical leak rate, at high pressure and high temperature
Fig. 5: Parametric trends showing progression of the experimentation

subcooled water, through tight pre-cracked pipes using the above test facility. This experiment was conducted as part of commissioning procedure. Tight cracks of semi-crack angle of 22.5° were generated by high cycle fatigue loading at the Materials Science Division on a 100 mm NB, Sch. 80 5316 pipe. Experiment was conducted in the pressure range: 70 – 90 bar at a maximum temperature of 250 °C. A minimum
subcooling of 45 °C was maintained for all runs. A pressure higher than 80.0 bar only resulted in crack flow. The observed flow at 90.0 bar and 250 °C was found to be 0.023 kg/s against the predicted flow of 0.03 kg/s. Repeatability test yielded the same results.

**Experimentation with Pipes containing Slits**

Critical flow models for small openings involve a number of uncertain parameters and two major uncertain parameters: namely Crack Opening Area (COA) and the crack surface roughness which govern the prediction. Experimentation with well defined slit geometries eliminates this uncertainty and gives first hand confidence about the critical flow models. To test this idea, experimentation with three slit sizes were planned and conducted. Slits were generated with wire EDM technique at the Production Engineering Department, Jadavpur University, Kolkata, on 100 mm NB sch. 80 pipes.

**Overall Loss Coefficient Evaluation with Cold Run**

Several cold condition experiments were conducted and flow rates were compared with code C_SFA prediction and RELAP5 code. Table 1 furnishes the results along with Overall Loss Coefficient (K) for each condition. The K value for the slit geometry considers losses due to entry, flow path and exit. The expression for K is given below:

\[
K = \sqrt{\frac{G^2 v_{tr}}{2 \Delta P_{tot}}} \quad \ldots (18)
\]

Table 2 shows that the predicted values with C_SFA code and RELAP5 code are in agreement with experimental findings within a variation of 10%. The average loss coefficient calculated from the experimental data was found to be consistent and the average value was found to be 1.168. A typical characteristic for variation of flow rate with upstream pressure (Fig. 6), shows attainment of critical flow at upstream pressure of 60 bar.

**Leak Measurement with Hot Condition**

Tables 2-4 show the experimental results against the code predictions (C_SFA and RELAP5) for different slits. The tables show the variation of flow rates with pressure variation, with a constant

<table>
<thead>
<tr>
<th>Slit Upstream pressure (Bar)</th>
<th>Saturation temperature (°C)</th>
<th>Slit Upstream temperature (°C)</th>
<th>Subcooling (°C)</th>
<th>Loss Coefficient (K)</th>
<th>flow rate (kg/s)</th>
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<tbody>
<tr>
<td>11.4</td>
<td>185.65</td>
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<td>30.3</td>
<td>233.84</td>
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<td>248.64</td>
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<td>80.0</td>
<td>294.96</td>
<td>30.0</td>
<td>264.96</td>
<td>1.088</td>
<td>0.780</td>
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</table>

Table 1: Comparison of Experimental Results and Theoretical Prediction for Slit-2
[Slit Width = 0.038 cm, Slit Depth = 0.8 cm, Slit Length = 1.5 cm]
subcooling (within 2º C variation) and subcooling variation with a constant pressure (within 2 bar variation).

The tables show agreement between experimental data and C_SFA predictions within a variation of 10%. Among the various critical flow models in code C_SFA, the Burnel model is found to be most suitable for these validation tests. Prediction by RELAP5 is also found to be in agreement with experimental findings and it predicts a value higher than C_SFA code value by 5%.

The increase in flow with increase in subcooling at constant pressure and increase in flow with increase in pressure at constant subcooling confirm the critical flow theory as well as findings by other researchers like Amos and Schrock et al [4 and 5].

Table 2: Comparison of Experimental Results and Theoretical Prediction for Slit-1 [Slit Width =0.027cm, Slit Depth =0.8cm, Slit Length = 4.373cm]
Conclusions

1. The LBB code C_SFA validation exercise has been carried out with slits generating higher flow rate (>0.5 kg/s). The predicted values have matched fairly well with the measured ones. The same code has been validated with a limited number of experiments that have been performed with pre-cracked pipes.

2. The performance of the experimental set-up (attainment of test condition and steady-state experimentation) was found to be quite satisfactory.

Future Work

It is proposed to conduct some more experiments with the same slit width having lower slit lengths, to achieve lower flow rates (<0.5 kg/s). This experiment has been planned under a BRNS project. Extensive experimentation for crack flow (cracks proposed to be generated at SERC, Chennai) measurement has been planned under the 11th Plan project, in the same setup at Jadavpur University, Kolkata. As observed during the experimentation for measurement of flow through cracks, absence of bending moment causes very less opening of the crack. Provision
for applying bending moment on the pre-cracked pipe has been worked out and will be implemented under the above mentioned plan project.

Nonenclature

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<th>Symbol</th>
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<td>Area</td>
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<tr>
<td>E</td>
<td>Young’s modulus</td>
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<td>G</td>
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References

PRODUCTION OF STERILE [F-18] NAF FOR SKELETAL PET IMAGING

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Introduction

Indications for a radionuclide bone scan include detection of bone-metastasis of cancers, osteomyelitis, avascular necrosis, trauma, metabolic disorders and arthritic diseases. In the mid 1970s, skeletal X-ray was replaced by bone scan, using 99mTc – phosphate (99mTc- PYP) and 99mTc-phosphonate SPECT radiopharmaceuticals (99mTc-MDP, 99mTc- HMDP, 99mTc- HEDP etc.), because of its 95% sensitivity in detecting metastatic bone diseases. On an average bone lesions could be detected six months earlier than with X-ray examination, which has a sensitivity of only 78%. The reason for the latter lies in the fact, that radionuclide scan demonstrates osseous remodeling, which precedes structural changes seen on the X-ray images [1, 2]. In the last decade, the compact Medical Cyclotron has become an economic source for the production of short-lived PET radionuclides, which can be rapidly converted to PET radiopharmaceuticals using an automated chemistry synthesizer. In conjunction with highly sophisticated PET scanners, imaging of various diseases is possible with exquisite sensitivity. Using [F-18] NaF, bone physiology has been studied through PET imaging by several groups. This is now a reality in India too, with the availability of [F-18] NaF. CT-PET “fusion” imaging has made PET imaging even more effective. The advantages of bone scanning with [F-18] are manifold [3]. [F-18] NaF is a natural bone-seeking agent and a patient has to wait for a shorter time after injection, in comparison to 99mTc - phosphates. It provides better target to non-target ratio and provides better count rate as uptake of [F-18] NaF is almost twice than that of 99mTc-MDP. [F-18] NaF is very effective in detecting both lytic and sclerotic metastases. The PET imaging technique makes F-18 Fluoride scans much sharper with high S/N ratio than 99mTc-MDP scans, making detection of metastasis easier. Though it may appear that there is no processing required to make [F-18] NaF, since 18F- comes from the cyclotron, through the irradiation of [O-18] water with proton, it should be noted that the 18F- from the cyclotron target has to be “cleaned up” of all target body and target-window-foil related radionuclides, formed during proton bombardment and metallic impurities and the final product made suitable as an injectable, fulfilling pharmacopoeia requirement. We have achieved this by using the shielded C-11 module by suitable modification of the chemistry process and the program “time list”. We carried out all the necessary quality control steps and applied for RPC clearance before regular supply of [F-18] NaF to various hospitals. The present article briefly describes the production procedure of [F-18] Fluoride, the QC results, bio-distribution studies through PET/CT imaging of rabbit and evaluation in human volunteers.
Materials and Reagent

H$_2^{18}$O of 95% enrichment was procured from ROTEM Industries. $^{18}$F Separation Cartridge in HCO$_3^-$ was obtained from Chromafix (Cat. No. Chromafix 45-PS-HCO$_3^-$), Germany. Sterile, pyrogen-free and pharmaceutical grade isotonic saline solution was obtained from ABX Advanced Biochemical Compounds, Germany. Sterile, non-pyrogenic, hypodermic single use 0.2-$\mu$m syringe filter was procured locally. Sterile, pyrogen-free, evacuated vials of capacity 10 ml (Pyrovac®) were procured from ACLLA AG; Germany and was used for multiple dispensing of the sterile [F-18]. Normal, healthy rabbits were used for bio-distribution studies. They were 6-9 months old, weighing - 2.6 kg and maintained in the divisional animal house. PET-CT imaging studies was done in a GE PET-CT machine at the Bio-Imaging Unit, Tata Memorial Hospital.

Protocol of [F-18] NaF Production

The production of [F-18] NaF is done as shown in the General Purpose Fluorination Module, with the help of menu-driven NINA (GE Health Care) based software programme, developed indigenously by us. (The schematic of General Purpose Fluorination Module is given in Fig.1).The production of [F-18] NaF is done in four steps, apart from Cyclotron irradiation for the production of the radionuclide. Enriched [O-18]-H$_2$O is irradiated with protons, in the high-yield silver target of the cyclotron, to produce

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**Fig. 1 : Graphics of general purpose fluorination module**
$^{18}$F through the nuclear reaction, $^{16}$O (p, n) $^{18}$F. The time of irradiation as well as the proton beam current used, will depend on the amount of radioactive $^{18}$F required. After irradiation, the irradiated [O-18] water is transferred to the chemistry module under He gas pressure through tubings. The [F-18]-F is trapped on the Chromafix 45-PS-HCO$_3$ mentioned earlier by anion exchange and the target water is recovered. The [F-18]-F is eluted from the column, with 2ml sterile, endotoxin free physiological saline. The [F-18]-F is eluted as [F-18] NaF solution. Finally, the product is dispensed into sterile, pyrogen-free vials, through a 0.2 µ filter in a class-100 area, in an automated dispensing unit (Fig. 2). The vials are rapidly autoclaved at 134ºC for four minutes, also built-in inside the dispenser. Once the F-18 is received from the cyclotron into the synthesizer, the production of sterile, injection grade [F-18] NaF, hardly takes about 15 minutes.

**Quality Control Checks for Product**

QC checks are performed on the [F-18] NaF to ensure that it fulfills pharmacopoeia requirements. The visual appearance of the product for its clarity, absence of any colour and any suspended particulate matter are the first checks. Secondly, the pH should be in the range 4.5 - 8.5. This is followed by tests for the radiochemical purity through thin layer chromatography technique using silica-coated TLC plates and 95:5 acetonitrile : water mixture, as the mobile phase. The $R_f$ value corresponding to [F-18]-NaF is 0.0-0.12 in this mobile phase and the total counts in this area should be >95% of the total counts as per pharmacopoeia (Fig. 3). The radionuclide purity is assessed from its $T_{1/2}$, and it is considered to be F-18 if found to be 110±5 minutes. Since the [F-18] NaF is to be used intravenously in patients, checks for biological contamination viz. sterility and endotoxin tests are carried out on the samples (post radioactive decay) of each batch according to standard procedure as used for [F-18] FDG. This is done because of the short $T_{1/2}$ of $^{18}$F, that does not permit these tests to be carried out prior to release. The procedure used for production is validated by testing the product from several batches. The confidence from such validation enables us to practice “parametric release” of the product with >99% confidence that they will pass sterility and endotoxin tests. All these QC tests are in keeping with the US and European
Pharmacopoeias [5, 6].

Bio Distribution and Patient Studies

About 3-4 mCi of [F-18] NaF in a volume of 1 ml was administered to rabbits through IV injections. After a one hour wait, images were taken on PET/CT machine. PET/CT images of a few patients with osteochondroma problem, osteoporotic changes etc. were studied using ~10mCi of [F-18] NaF. Clearance from TMH Ethics Committee was taken for this purpose.

Results

The product data from twelve trial batches are given in Table I. The QC data including sterility and endotoxin test results are given in Table 2. Figs. 4 & 5
are the PET/CT images of injected [F-18] NaF rabbit. Figs. 6, 7 are the PET/CT images of the patient having osteoporotic changes in the vertebra. Finally Figs. 8 and 9 are the images from a patient with osteochondroma.

Table 1: Consolidated Processing and Product data of [F-18]-NaF Injection in saline

<table>
<thead>
<tr>
<th>Sr. No</th>
<th>Batch No</th>
<th>Final Product Details</th>
<th>RAC (mCi/ml)</th>
<th>Total Volume (ml)</th>
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<tbody>
<tr>
<td>1</td>
<td>Trial-20</td>
<td></td>
<td>11.5</td>
<td>6</td>
</tr>
<tr>
<td>2</td>
<td>Trial-21</td>
<td></td>
<td>14.0</td>
<td>6</td>
</tr>
<tr>
<td>3</td>
<td>Trial-22</td>
<td></td>
<td>15.0</td>
<td>6</td>
</tr>
<tr>
<td>4</td>
<td>Trial-23</td>
<td></td>
<td>22.0</td>
<td>6</td>
</tr>
<tr>
<td>5</td>
<td>Trial-24</td>
<td></td>
<td>4.0</td>
<td>6</td>
</tr>
<tr>
<td>6</td>
<td>Trial-25</td>
<td></td>
<td>5.0</td>
<td>6</td>
</tr>
<tr>
<td>7</td>
<td>Trial-26</td>
<td></td>
<td>10.0</td>
<td>6</td>
</tr>
<tr>
<td>8</td>
<td>Trial-27</td>
<td></td>
<td>3.0</td>
<td>6</td>
</tr>
<tr>
<td>9</td>
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<td></td>
<td>4.0</td>
<td>6</td>
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</tr>
<tr>
<td>12</td>
<td>Trial-31</td>
<td></td>
<td>8.4</td>
<td>6</td>
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</table>

Processing as per standard protocol (Annex.-C), *Radioactive concentration

Table 2: Consolidated QC Evaluation Data of [F-18]-NaF in saline injection

<table>
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<tr>
<th>Sr. No</th>
<th>Batch No</th>
<th>Final Product Details</th>
<th>RCP</th>
<th>RNP</th>
<th>pH</th>
<th>Test of sterility</th>
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<td>Trial-20</td>
<td></td>
<td>&gt; 95%</td>
<td>113.5</td>
<td>7.0</td>
<td>Passes</td>
<td>Passes</td>
</tr>
<tr>
<td>2</td>
<td>Trial-21</td>
<td></td>
<td>&gt; 95%</td>
<td>108.6</td>
<td>6.0</td>
<td>Passes</td>
<td>Passes</td>
</tr>
<tr>
<td>3</td>
<td>Trial-22</td>
<td></td>
<td>&gt; 95%</td>
<td>111.9</td>
<td>7.5</td>
<td>Passes</td>
<td>Passes</td>
</tr>
<tr>
<td>4</td>
<td>Trial-23</td>
<td></td>
<td>&gt; 95%</td>
<td>110.9</td>
<td>7.0</td>
<td>Passes</td>
<td>Passes</td>
</tr>
<tr>
<td>5</td>
<td>Trial-24</td>
<td></td>
<td>&gt; 95%</td>
<td>115</td>
<td>6.5-7.0</td>
<td>Passes</td>
<td>Passes</td>
</tr>
<tr>
<td>6</td>
<td>Trial-25</td>
<td></td>
<td>&gt; 95%</td>
<td>108</td>
<td>7.0</td>
<td>Passes</td>
<td>Passes</td>
</tr>
<tr>
<td>7</td>
<td>Trial-26</td>
<td></td>
<td>&gt; 95%</td>
<td>108.5</td>
<td>7.0</td>
<td>Passes</td>
<td>Passes</td>
</tr>
<tr>
<td>8</td>
<td>Trial-27</td>
<td></td>
<td>&gt; 95%</td>
<td>105.5</td>
<td>7.0</td>
<td>Passes</td>
<td>Passes</td>
</tr>
<tr>
<td>9</td>
<td>Trial-28</td>
<td></td>
<td>&gt; 95%</td>
<td>105.5</td>
<td>6.5-7.0</td>
<td>Passes</td>
<td>Passes</td>
</tr>
<tr>
<td>10</td>
<td>Trial-29</td>
<td></td>
<td>&gt; 95%</td>
<td>110.4</td>
<td>7.0</td>
<td>Passes</td>
<td>Passes</td>
</tr>
<tr>
<td>11</td>
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<td></td>
<td>&gt; 95%</td>
<td>102.0</td>
<td>7.0</td>
<td>Passes</td>
<td>Passes</td>
</tr>
<tr>
<td>12</td>
<td>Trial-31</td>
<td></td>
<td>&gt; 95%</td>
<td>109.1</td>
<td>7.0</td>
<td>Passes</td>
<td>Passes</td>
</tr>
</tbody>
</table>
Fig. 5: PET/CT Images of [F-18] Fluoride scan of rabbit, sagittal

Fig. 6: PET/CT Coronal Images of [F-18] Fluoride Scan of a patient showing changes in vertebra due to osteoporosis.

Fig. 7: PET/CT sagittal images of [F-18] Fluoride scan of a patient showing changes in vertebra due to osteoporosis.

Fig. 8: PET/CT Coronal images of [F-18] Fluoride scan of a patient with osteochondroma.
Discussion

\(^{18}\)NaF is a positron-emitting radiopharmaceutical, used in skeletal PET imaging, for the diagnosis of a variety of disorders, importantly bone metastasis. The direct use of the \(^{18}\)F from the cyclotron target, following irradiation of \(^{18}\)O water is undesirable due to the presence of contaminating long-lived radionuclides, originating from the target body, foil, etc. In our method, the \(^{18}\)F is trapped in an anion exchange column and eluted as \(^{18}\)NaF using sterile saline. The \(^{18}\)NaF produced was analyzed using high purity germanium semiconductor detector in a multi-channel analyzer for a sufficiently long period of 6 to 7 hours and we did not find any other radioactivity peak, than the one at 511 keV, thereby ruling out the presence of any contaminating radionuclides. Since the production of \(^{18}\)NaF is a simple anion exchange procedure, the yield is almost one hundred percent. The radioactive concentration (Table I) is relatively low as these batches are trial production batches. However, the same technique can be used to produce 500-1000 mCi (18.5-37 GBq), since the \(\text{HCO}_3^–\) anion exchanger is capable of trapping 2-3 Ci (74-100 GBq) of \(^{18}\)F and the entire activity can be eluted out.
in 2ml isotonic saline. The pH of the product is in the neutral range of 6.5-7.0 as expected. $^{18}$F Fluoride ion is incorporated into the bone tissue by ion exchange with hydroxyl ion [8, 9]. It quickly passes from the plasma into the hydration shell surrounding each bone crystal and is taken up in bone in proportion to the blood flow and bone metabolic activity [10, 11]. This allows visualization of osseous lesions since skeletal uptake of $^{18}$F Fluoride is altered in the areas of abnormal osteogenesis. From pharmacokinetics point of view, about 50% of the $^{18}$F NaF intravenously administered, is rapidly taken up by the skeleton, where it remains for the entire period of its radioactive decay. The remainder is distributed into the extracellular fluid and is rapidly eliminated in urine within a few hours [12]. $^{18}$F NaF normally accumulates in the skeleton symmetrically with greater deposition in the axial skeleton and in the bones around joints, than in the appendicular skeleton and in the shafts of long bones. Increased deposition occurs around fracture sites and in bones affected by osteomyelitis, fibrous dysplasia, spondylitis tuberculosis, Paget’s disease, hyperostosis frontalis interna, myositis ossificans, tumors and in rapidly growing epiphyses. $^{18}$F F- PET images of the skeletal system are remarkable for the resolution and sensitivity (Figure 4 and 5). In the patient studies, the location of osteochondroma is clearly defined by the PET/CT images. In the second patient, the changes in the vertebra due to osteoporosis is clearly evident. It may be concluded that $^{18}$F Fluoride is an excellent skeletal imaging PET agent and with the availability of PET/CT it will play a very crucial role in the field of skeletal imaging and bone related problems.

References

10. The clinical use of sodium fluoride F 18 in bone photo scanning, JAMA 204(11), 1968 Jun; 116-22.
SESTEC-2006, sponsored by DAE-BRNS, was held at Mumbai, from September 29 – October 1, 2006. In his inaugural address, Dr R.A. Mashelkar, Director-General, CSIR, emphasized the role of separation science & technology in meeting the increasing energy demands in the 21st century. He stressed the need for recovering Uranium from sea water, for the production/separation of H₂ and urged upon separation scientists and technologists to develop methodologies, to meet new challenges. Dr Kakodkar, Chairman, AEC, mentioned that the recovery of Uranium from lean ores at Jaduguda has helped India’s atomic energy programme. He reiterated the need to recover uranium even from secondary sources to meet energy demands.

Dr S. Banerjee, Director, BARC, urged the delegates to open new avenues through interactions/co-operative research efforts, to meet the challenges of the nuclear industry. Dr V.K. Manchanda, Convener, SESTEC-2006 and Head, Radiochemistry Division, BARC, briefed the delegates about the scope of the symposium.

A total of about a hundred contributed papers and 19 invited lectures formed the core of the symposium programme. The contributed papers were grouped into nine sections: synthesis of reagents/solvents, ion exchange separation, process development, novel separations, membrane-based separations, waste water treatment, radiochemical separations and nuclear fuel cycle. Dr Mashelkar released the volumes of the proceedings of SESTEC-2006, which included both invited lectures and extended abstracts of all the contributed papers.

A special session was arranged during the symposium to highlight the activities of the Heavy Water Board in the field of separation science & technology.

In the concluding session, chaired by Dr V. Venugopal, Director, RC&I Group, BARC, several new areas for further R&D in separation science & technology, were identified.
The technology of the “Particle Aerodynamic Size Separator (PASS)” has been developed by the Environmental Assessment Division, BARC. The Particle Aerodynamic Size Separator (PASS) is an import substitute product, for aerodynamic particle sizing, based on the principle of inertial impaction and it separates particles according to their aerodynamic diameters, in the range of 0.53-10 µm, in seven size class intervals. This instrument will be of help to several national laboratories and universities for carrying out studies on atmospheric pollution and aerosol characterization.

The instrument is useful in occupational monitoring of radioactive and non-radioactive environments, arising in the context of materials processing, metal cutting, powder handling etc. It is useful for air quality regulators as well as for researchers engaged in various aspects of air pollution and aerosol research. It can also be used by pharmaceutical industries for characterizing the lung aerosol delivery systems, such as nebulizers and metered dose inhalers.

The know-how of “Particle Aerodynamic Size Separator (PASS)” was transferred to M/s Para Electronics (India) Pvt. Ltd., Mumbai (Maharashtra) on April 12, 2007.

The Technology Transfer and Collaboration Division coordinated all activities related to the transfer of this technology, such as preparation of the technical brochure, the technology transfer document, the advertisement of the technology, technology transfer agreement preparation and signing of the agreement in collaboration with the Environmental Assessment Division.
abhaya. Scientists honoured

BARC scientists honoured

P. V. Bhagwat, Head, Pelletron Accelerator Facility, is an accelerator technologist. His current fields of interest are room temperature RFQ Linac, accelerator based applications like AMS and production of Track Etched Membranes. He is heading the team working on design and development of the alternate injector for superconducting linear accelerator.

Dr. A. K. Gupta is an accelerator physicist specializing in ion sources and ion beam technology. His current fields of interest are atomic cluster physics, isotopic ratio measurements, alternate ECR injector system and radioactive ion beam development at BARC-TIFR Pelletron Accelerator Facility.

Mr. N. Mehrotra is an accelerator physicist. He does beam dynamics, electromagnetic design of accelerating structures and ion sources. His current field of interest is design and development of heavy ion RFQ Linac for the injector system to superconducting Linac at Pelletron Accelerator Facility.

A paper entitled “Beam Dynamics of the Heavy Ion RFQ with Prebuncher for the Alternate Injection at PAF” by N. Mehrotra, P. V. Bhagwat, A. K. Gupta, P. Surendran, J. A. Gore, R. K. Choudhury and S. Kailas of Nuclear Physics Division, BARC and M. B. Kurup of Tata Institute of Fundamental Research, Mumbai was awarded the oral presentation prize at the BARC Golden Jubilee DAE-BRNS Indian Particle Accelerator Conference (InPAC-2006), held at Bhabha Atomic Research Centre and Tata Institute of Fundamental Research from November 1-4, 2006. Mr. N. Mehrotra had presented the paper.

Mr. P. V. Bhagwat, Head, Pelletron Accelerator Facility.
Mr. P. Surendran is an accelerator physicist working at BARC-TIFR Pelletron Accelerator Facility. He was on deputation at CERN, Geneva for magnetic measurements of superconducting magnets for LHC, under Indo-CERN collaboration. He is actively involved in AMS and beam dynamics for new injector systems.

Mr. J. A. Gore is associated with electronics group at BARC-TIFR Pelletron Accelerator Facility. He was on deputation at CERN, Geneva for magnetic measurements of superconducting magnets for LHC, under Indo-CERN collaboration. He is presently involved in development of beam diagnostic devices for high current proton accelerator and computer-based control system for accelerators.

Dr. R. K. Choudhury is currently Head, Nuclear Physics Division. His research work is in frontier areas of Nuclear Physics and accelerator-based applications. He has also contributed to accelerator technology developments. During his tenure as Director, IOP, Bhubaneshwar, he successfully completed the commissioning of the AMS program for radiocarbon dating with the 3MV tandem accelerator.

Mr. S. Kailas is currently Associate Director (N), Physics Group. He is a fellow of Indian Academy of Science. His current areas of interest are basic research in frontier area of Nuclear Physics and accelerator-based applications such as accelerator driven sub-critical system, accelerator mass spectrometry, track etch membrane etc. He is also leading a team for heavy ion based alternate injector for the Pelletron Accelerator.

Dr. M. B. Kurup is currently Professor of Physics, Arka Vijnana, Banpur. He has been working in the area of nuclear astrophysics, spectroscopy and hadron physics for more than 35 years. He has more than 120 research publications.
Prof M. B. Kurup is currently chairman of BARC-TIFR Pelletron Accelerator Facility Committee. He is a pioneer in the indigenous development of lead-based superconducting linear accelerator technology at TIFR. His current fields of interest are ion-atom collisions, ion-beam modification of materials and accelerator-based applications.

Sri Sanjay Panwar, an esteemed scientist at BARC, is a pioneer in the field of nuclear reactor safety. He is the current head of the BARC-TIFR Pelletron Accelerator Facility Committee. He is known for his contributions to the development of lead-based superconducting linear accelerator technology at TIFR. His current research focuses on ion-atom collisions, ion-beam modification of materials, and accelerator-based applications.

After graduating from the 37th Batch of BARC Training School, Mr. Sanjay Panwar, joined RED in 1994. He has developed Integrated garter spring repositioning system and Coolant Channel Replacement Machine for pressure tubes of Indian PHWRs. He has also developed controls for a number of inspection systems. He has also developed in-situ Property measurement System for estimation of mechanical property of pressure tube of Indian PHWRs. Presently he is working on the development of tool delivery system for coolant channels and other life assessment tools for Zr-2.5%Nb pressure tubes of Indian PHWRs.
Indian Pressurized Heavy Water Reactors (PHWRs). Presently he is responsible for the development of technologies required for life management of Zr-2.5%Nb pressure tubes of Indian PHWRs.

After graduating from the 17th batch of BARC Training School, Mr B.B. Rupani, joined Dhruba project in 1974 and after successful completion of Drhuvra project, he was transferred to Reactor Engineering Division (RED) in 1985. Presently he is heading Reactor Coolant Channel Section of RED. He has developed various innovative inspection and rehabilitation tools and techniques for life management of coolant channels of Indian Pressurized Heavy Water Reactors (PHWRs). Presently he is responsible for the development of coolant channels of Advanced Heavy Water Reactor (AHWR) and the development of technologies required for Zr-2.5%Nb pressure tubes of Indian PHWRs.

After graduating from the 25th Batch of BARC Training School, Mr B.S.V.G. Sharma, joined RED in 1987. He has developed Integrated garter spring repositioning system and Coolant Channel Replacement Machine for pressure tubes of Indian PHWRs. Presently he is working on the development of tool delivery system for coolant channels and other life assessment tools for Zr-2.5%Nb pressure tubes of Indian PHWRs.

Shri B.S.V.G. Sharma, who completed his PhD in Nuclear Engineering from the Indian Institute of Technology, Roorkee, has been associated with BARC since 1987. He has made significant contributions towards the development of technologies for the life management of PHWRs. His work has been instrumental in improving the reliability and efficiency of these reactors.

Ms Deepti Appukuttan, a Ph.D. student (DAE-SRF) from the Molecular Biology Division at BARC, was awarded the 1st prize in the Ph.D. category for her paper entitled "Engineering of Deinococcus radiodurans R1 for bioprecipitation of uranium from dilute nuclear waste," at Dr Dhala's Felicitation Fund (DFF) Fifteenth Annual Research Paper Presentation Competition, held at Mumbai on March 3, 2007. The award carries a cash prize of Rs. 1000/- and a certificate.
IN THIS ISSUE

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