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Technical Note

Development and Validation of a Methodology for Characterization of Sodium Aerosols in Cover Gas Region

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ABSTRACT

This paper describes an experimental methodology developed in an Aerosol Test Facility (ATF) for sampling and analysis of sodium aerosol (metal vapour) from the cover gas region of a fast rector, and details of the validation of the methodology in an experimental sodium loop. The methodology involves; (i) sampling of sodium aerosols by drawing them without exposure to the atmosphere, (ii) trapping them in paraffin oil medium, and (iii) analyzing the paraffin oil for the determination of mass concentration by the conductivity method and size distribution by using a Mastersizer. Validation of the methodology is carried out in a sodium loop called the SILVERINA facility. The aerosol size distribution is found to vary from 1 to 12 µm, with the Mass Median Diameter (MMD) around 4.0 µm (σ_g = 1.5), and the mass concentration is found to be ~9.50 g/m^3. The experimental results agree with the values found in the literature.

Keywords: Fast reactor; Cover gas region; Sodium aerosol; Size distribution; Mass concentration.

INTRODUCTION

Major energy resources that are being exploited for electricity generation at present are coal, natural gas, hydro, renewable energy resources and nuclear. In order to bridge the growing energy demand, the contribution from nuclear energy is inevitable and it can provide an environmentally clean, sustainable and secured energy (Bhoje, 2003). The development of nuclear reactor began in 1950, which resulted in commercial thermal fission reactors called first generation reactors. The thermal fission reactors can only use a very small fraction of the total energy available in Uranium and rest of the energy resource in U^{238} is not utilized. The fast reactor provides a method of converting the non fissile U^{238} into fissionable Pu^{239} isotopes i.e., allowing the possibility of breeding. (The breeding ratio is the ratio of the number of fission nuclei produced to the number consumed) (Raj and Rajan, 2006). The reactor design evolved further into generation IV type reactors, which addresses compact core, thermal and fast neutron spectra, and closed and open fuel cycles. The generation IV type reactor systems are (i) Very high temperature gas reactor (VHTR), (ii) Sodium cooled fast reactor (SFR), (iii) Super critical water-cooled reactor (SCWR), (iv) Gas cooled Fast Reactor (GFR) and (v) Molten salt reactor (MSR) (http://www.gen-4.org/PDFs/GIF_Overview.pdf & http://mragheb.com/NPRE%20402%20ME%20405%20Nuclear%20Power%20Engineering/Fourth%20Generation%20Reactor%20Concepts.pdf). Another development of generation IV reactor system is Integral Fast Reactor (IFR). The IFR is a closed nuclear power system that recycles its own waste, so that, the elements that are radioactive for tens of thousands of years are all consumed and converted into electricity and waste elements with short half-lives. So it essentially solves the nuclear waste problem (http://large.stanford.edu/courses/2013/ph241/waisberg1/docs/archambeau.pdf).

India's fast reactor program started with 40 MWt/13.2 MWe Fast Breeder Test Reactor (FBTR) which is presently in operation. As a logical follow-up of FBTR, a 500 MWe Prototype Fast Breeder Reactor (PFBR) is being constructed at Kalpakkam, INDIA. The PFBR is a pool type, liquid metal (sodium) cooled fast breeder reactor (LMFBR) (http://www.iaea.org/Publications/Magazines/Bulletin/Bull206/20604782938.pdf). It uses mixture of PuO_2-UO_2 as a fuel. The major components like the core, intermediate heat exchanger, primary coolant pumps lie submerged in a large pool of sodium and housed inside a Reactor Containment Building (RCB). The core of the reactor is compact which

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results in a very high power density. For efficient removal of heat, liquid sodium is used as a coolant. The advantage of using liquid sodium includes high thermal conductivity, high boiling point and low fast neutron absorption and scattering cross-section. Liquid sodium has low viscosity and high electrical conductivity allowing it to be easily pumped. Also, it is compatible with the structural materials. The sodium is made to run in two loops, the primary loops takes away the heat generated in the core of the reactor and the secondary loops takes the heat from the primary loop and transfers the heat in the boiler circuit for the generation of steam. Argon is used as cover gas which serves as a blanket for sodium above the reactor core in the reactor vessel.

In the normal operating condition of LMFBR or PFBR, evaporation of sodium vapor from the hot pool surface and subsequent condensation of vapors results in the formation of sodium aerosol within cover gas space (Ford *et al.*, 1993). (The space above the pool surface and below the roof slab of reactor vessel and it is filled with Argon). These aerosols will absorb and scatter the heat radiation coming out from the pool surface and modify the total heat transfer to the cooled roof structure and side wall. Further, the mass transfer occurs due to condensation of sodium vapor on the cooler surface like annular gaps, roof top, control plug, rotating plug and fuel handling machine resulting deposition of aerosols, which hinders the rotational movement of rotating plug, and operation of fuel handling machine (Robert *et al.*, 1995). The sodium aerosols are also affecting the operation of cover gas purification system and reduce its efficiency (flow through cold trap circuit). The aerosols with sufficient concentration reduce the visibility of cover gas region [Core inspection facility in RAPSODIE, France and Fast Breeder Test Reactor (FBTR), India]. To understand the above issues, it is necessary to know the spatial variation of aerosol concentration and size distribution in the cover gas region.

Studies on aerosol characteristics were carried out by various groups. It is observed form the literature, sodium aerosol size ranges from 1–16 µm and mass concentration ranges from 1–40 g/m^3 (Himeno and Takahashi, 1980; Himeno and Yamagishi, 1982; Frukawa *et al.*, 1984; Glockling *et al.*, 1991; Minges and Schutz, 1991; Yamamoto *et al.*, 1991; Ford *et al.*, 1993; Jackson *et al.*, 1993; Minges and Schutz, 1993; Newson *et al.*, 1993; Robert *et al.*, 1995). In general, the sodium aerosol mass concentration increases with the increasing of difference between roof to pool temperatures whereas, there is no definite correlation of aerosol size distribution with the variation of pool and roof temperatures. It is to be noted here that, there were several techniques used for the characterization of sodium aerosol size and mass distribution viz: Andersen Impactor, laser light based techniques, sodium ionisation detector, and SS mesh filter. But, it is important to adopt suitable method by drawing aerosols from the cover gas region and trap them in a suitable medium without exposing them to the atmosphere, to determine the characteristics of the sodium aerosols. Jackson *et al.* (1993), Minges and Schutz (1991, 1993) and Yamamoto *et al.* (1991) in their studies, used Andersen multistage Impactor and determined the mass-size distribution cumulatively over a period of time. By using stainless steel wire mesh filter (filtration technique), Himeno and Takahashi (1980), Himeno and Yamagishi (1982), Frukawa *et al.* (1984), Yamamoto *et al.* (1991) and Newson *et al.* (1993) determined the size distribution based on sieve size. In the above methods, measurements were conducted by keeping the sampling unit/system in the atmospheric condition. Glockling *et al.* (1991) used Malvern 2600C (based on Franhofer diffraction) and Dantec particle dynamic analyzer for characterizing sodium aerosols in the cover gas region, but the results obtained in both techniques were found different for the same experimental condition. Laser mist concentration meter and sintered stainless steel filter were used for sodium aerosol mass concentration measurements by Himeno and Takahashi (1980) and Himeno and Yamagishi (1982). In the above three investigations, the methodologies adopted in their measurements were not dealt in details. Roberts *et al.* (1995) used a technique to collect the sodium aerosol and measured the mass concentration by using atomic absorption spectroscopy and size distribution by using laser scattering technique. He observed that, the sodium aerosol characteristics in cover gas region are strongly dependent on the geometric dimension (diameter of the sodium pool surface, height of the argon cover gas and shape of roof top) of the cover gas system.

It is inferred from the literature that, sodium aerosol characteristics were studied in various experimental facilities but none of them in the reactor environment. It is important to note that in the reactor environment, sodium aerosols are immersed in the sea of bi-polar charges (generated by gamma radiation) and get charged upon interaction with ions. The aerosol properties (size and mass concentration, coagulation coefficient & deposition rate etc.) would get modified due to enhanced coagulation of charged sodium aerosols (Subramanian *et al.*, 2008; Kumar *et al.*, 2010; Subramanian *et al.*, 2012). Thus, it is important to characterize sodium aerosols in cover gas region with and without the presence of gamma radiation. In order to characterize the sodium aerosols, it is required to have in-situ sampling and suitable analysis technique. In this paper, methodology for characterization of sodium aerosols developed in Aerosol Test Facility (ATF) and validation of methodology in SILVERINA Loop are presented. The methodology includes, sampling technique and determination of concentration and size distribution of sodium aerosols. This paper also includes brief description of SILVERINA loop, sampling arrangements and the experimental results obtained in SILVERINA Loop.

METHODOLOGY

The methodology for characterization of sodium aerosols is developed in Aerosol Test Facility (ATF) (Baskaran *et al.*, 2007; Misra *et al.*, 2012). The methodology involves: (i) aerosols are drawn from the region above sodium pool and allowed to get trapped in liquid paraffin oil medium without exposing them to atmosphere, (ii) determination of mass concentration by using conductivity measurement technique and (iii) determination of size distribution by using Malvern Mastersizer.

Sampling Technique

A schematic diagram of sodium aerosol sampling system in ATF is shown in Fig. 1. About 5 g of sodium is taken in a crucible and heated in the sodium combustion cell under Argon environment. The temperature is raised upto 500°C and maintained, which results in formation of sodium vapour above the pool surface. The sodium vapour along with argon is made to bubble through a gas washing bottle filled with liquid paraffin oil (250 mL) kept at room temperature, at a flow rate of 2 L/min for about 10 minutes. Condensation of sodium vapor and solidification of aerosol particle would occur as the vapour passes through the paraffin oil while argon escapes out (Singh *et al.*, 2011). The second bottle is used to trap the sodium aerosols if any, escaped from the first bottle. Line heaters are used to prevent solidification of aerosols in the sampling lines.

Characterization of Sodium Aerosols

The measurement of sodium aerosol size distribution is carried out by using Mastersizer (M/s Malvern Instruments, UK). The Mastersizer uses the principle of ensemble diffraction technique. The Mastersizer measures the volume-size distribution of particles in the laden volume of liquid medium from 0.05–900 µm. Sodium aerosol trapped in the liquid parafin is made to circulate in the liquid flow cell and the volume-size distribution and its Mass Median Diameter (MMD) of the sodium aerosols are determined. The volume size distribution of sodium vapor is given in Fig. 2. It is observed from the Fig. 2, that the particles size range is from 5.68–12.20 µm with MMD of 7.72 µm.

The methodology adopted for determination of sodium aerosol mass concentration is explained as follows: The mass of sodium aerosols trapped in the liquid paraffin oil is measured by transferring them into a water medium (to become NaOH) and then measure the conductivity of the NaOH solution. The conductivity measurement of NaOH solution is carried out by using conductometer (M/s Metrohm, 856 conductivity module, Switzerland). The calibration of the

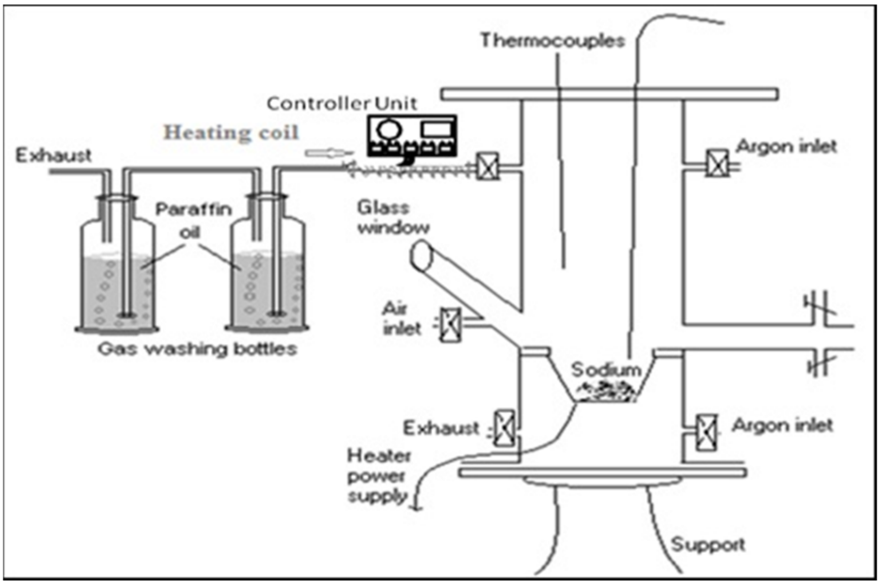

Fig. 1. A schematic diagram of sodium aerosols sampling system in ATF.

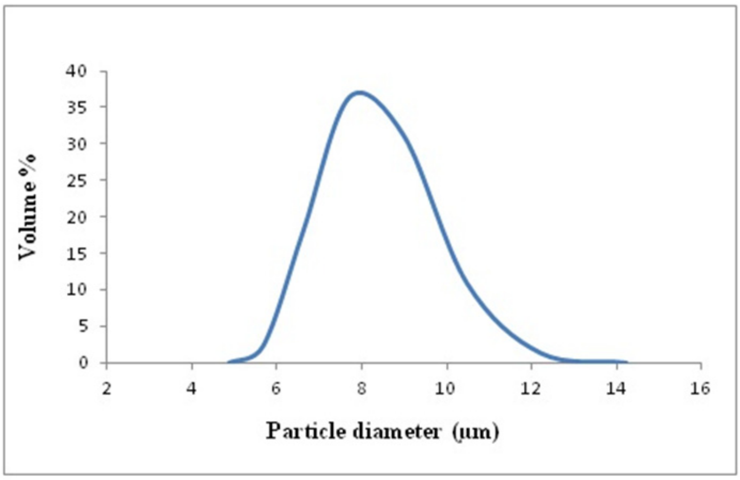

Fig. 2. Volume – size distribution of sodium vapor.

conductometer is verified before each set of measurements with standard KCl reference solution supplied with the instrument. Sodium mass concentration is estimated by using a pre-established calibration graph between conductivity versus concentration of NaOH (http://myweb.wit.edu/sandinic/Research/conductivity%20v%20concentration.pdf, May 2013) and it is shown in Fig. 3.

The experimental procedure is as follows: 200 mL of liquid paraffin oil (trapped with sodium aerosols) is mixed with same amount of water (1:1 ratio) in a separating funnel and vigorously shook for few hours to transfer the sodium into the water medium (to become NaOH). Out of 200 mL solution, 100 mL solution is taken for the conductivity measurement. The conductivity of NaOH is determined after subtracting the background conductivity of DM water (1 µS/cm). From the calibration graph, the trapped sodium mass in the paraffin oil is determined and the sodium aerosol mass concentration in the combustion cell is calculated by taking into account of flow rate and sampling time. The sodium aerosols mass concentration in the combustion cell is found to be around 0.173 g/m^3. The measured value is verified with the standard procedure. For that, the remaining solution is subjected to Pulsating Conductometric Titration (Subramanian *et al.*, 2009) method. An aliquot of the sample is titrated against HCl reactant, added in small steps and simultaneously measuring the change in conductivity. Conductivity variation as a function of the volume of HCl is obtained. It is found that, the sodium aerosols mass concentration in combustion cell is 0.18 g/m^3, and the error is found to be nearly 4.0% from the conductivity measurement (Kumar *et al.*, 2012).

VALIDATION OF CHARACTERIZATION TECHNIQUE IN SILVERINA LOOP

After developing methodology for characterization of sodium aerosols in ATF, the experiments are conducted in SILVERINA Loop for validating the methodology. The details of SILVERINA sodium loop is described in Chandramouli *et al.* (2006). SILVERINA sodium loop was constructed and commissioned at Engineering Hall-I, FRTG, IGCAR to conduct various experiments related to Prototype Fast Breeder Reactor (PFBR) and general Sodium Technology. The loop consists of three cylindrical test pots namely Test Pot-1(TP-1), Test Pot-2 (TP-2) and Test Pot-3(TP-3). This is a dynamic sodium loop with an electromagnetic pump, cold trap, plugging indicator, sodium sampler, heater vessel, interconnecting pipe lines, bellows sealed valves, flow meters and cover gas circuit. Sodium is filled in to the loop from a storage tank (capacity of 1300 kg). The sodium aerosol characterization experiments are carried out in TP-1. The height and internal diameter of the TP-1 are 2235 and 750 mm respectively and the height of the cover gas region is 820 mm. The total volume and quantity of sodium holdup in the TP-1 are 1.04 and 0.512 m^3 respectively. The top flange of TP-1 is provided with three nozzles for sodium level indication (low, middle and high levels) and a spare nozzle. The internal diameter of spare nozzle is 60.3 mm. The features of SILVERINA Loop are as follows: (i) the loop consists of a sodium tank in which liquid sodium could be maintained at various temperatures from 200–550°C and it simulates the reactor pool condition, (ii) the region above the sodium pool is filled with cover gas (Argon) and the cover gas height equals to that of FBTR, IGCAR.

Experiments have been carried out in SILVERINA loop by designing and fabricating a sampling system. The integrated view of aerosol sampling system with TP-1 is shown in Fig. 4. The aerosol sampling system (Fig. 4) consists of (a) Aerosol sampling tube, (b) Aerosol sampling bottle, (c) Line heaters and controller and (d) Aerosol flow controlling device. The sampling tubes are inserted in the spare nozzle with suitable arrangement.

(a) *Aerosol Sampling Tube*: The sampling tube is divided into two parts. One part of the sampling tube is inside the TP-1 and other part is outside. Both the parts are connected with a flange. The sampling tube which goes inside the TP-1 has been fabricated with three different lengths (300 mm, 500 mm and 700 mm) to enable the sampling at three different heights. Before starting the experiment the required length tube is attached with the flange and inserted into the TP-1. The aerosol sampling tube is made of SS316 with

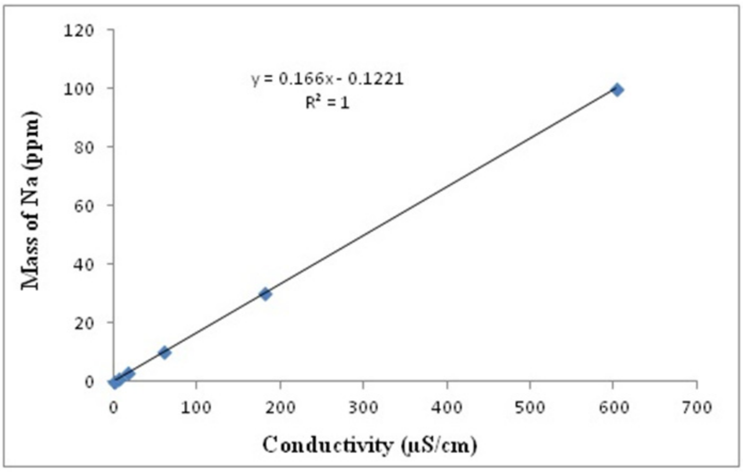

Fig. 3. Sodium mass vs Conductivity.

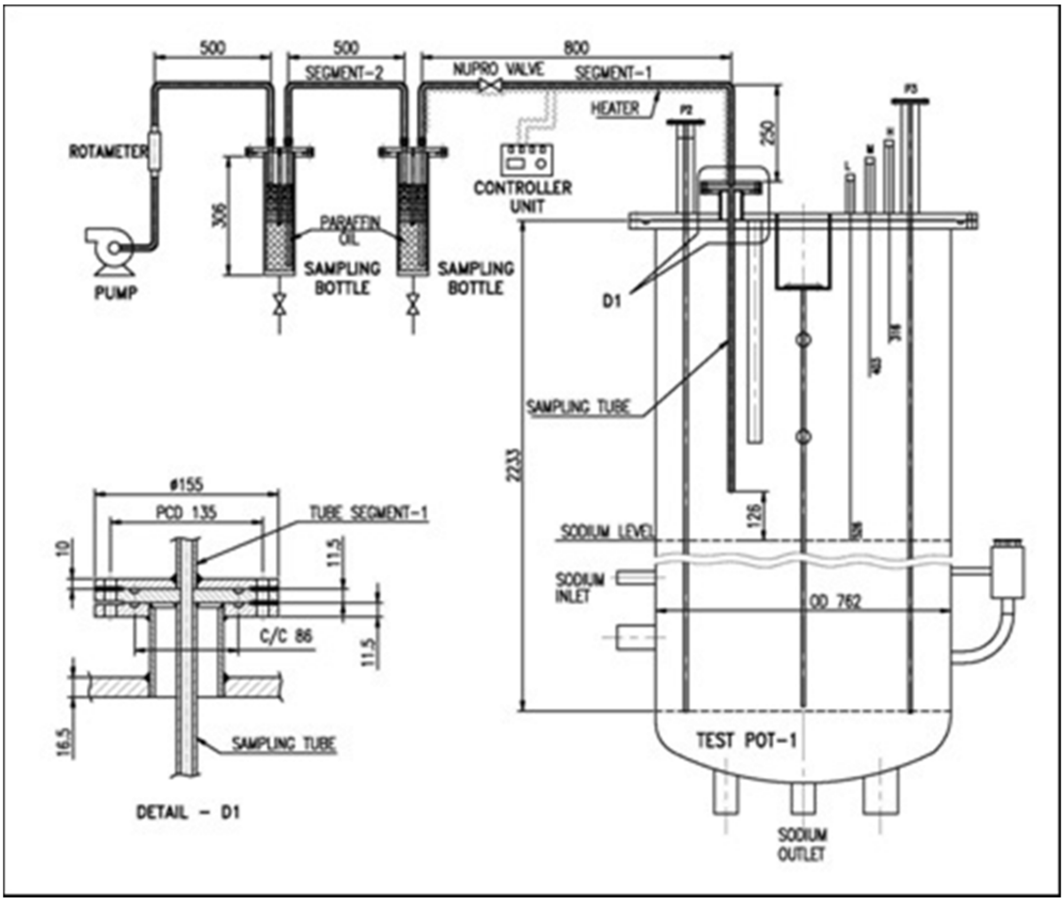

Fig. 4. The integrated view of aerosol sampling system with TP-1.

internal diameter of 10 mm (1.5 mm wall thickness), to avoid wall deposition by impaction for a flow rate of 2 L/min. The maximum particle diameter that can be collected without bias due to particle settling is 20 µm (Hinds, 1982).

(b) *Aerosol Sampling Bottle:* The schematic diagram of the sampling bottle is shown in Fig. 5. The capacity of the sampling bottle is 1.0 liter (height: 306 mm; diameter: 80 mm) and made of Perspex. The bottle is closed on top side with Perspex flange arrangement. The flange is provided with two opening for the insertion of inlet and outlet tubes. The inlet tube inserted upto bottom and it is connected to sampling tube by means of KF coupling and ball valve/gate valve. The paraffin oil will be drained by using a tube and needle valve arrangement at the bottom.

The sampling bottle is filled with paraffin oil (~700 mL) and maintained at ambient temperature. The sodium aerosol laden cover gas is made to pass through the bottle, thus condensation of sodium vapour and solidification of aerosol particle would occur as the cover gas enters into the paraffin oil. The residence time of the cover gas is increased due to baffle arrangement, which ensures that all sodium aerosols get trapped from the cover gas before it goes out of the bottle. The cover gas coming out from the sampling bottle – 1 is made to pass through sampling bottle – 2 so as to remove aerosols escaped, if any, from the 1st bottle. The sample bottles are removed from the loop and paraffin oil is drained for the analysis.

(c) *Line Heaters and Controller:* To prevent sodium vapour condensation and aerosol solidification within the sampling tube during experimental measurements, the wall temperature of the sampling tube is maintained at 110°C. The heating coils are wound on the sampling tube along with insulation and the current through the heating coils is controlled by usin g PID (proportional-integral-derivative**)** controller. The temperature along the sampling tube is monitored by 2 thermocouples. In order to have a redundancy in heating, a spare heater is also provided. The heater controller set-up consists of a panel board mounted with one PID controller, temperature display units, 3 pole rotary switch for double heaters, allied PCBs etc.

(d) *Aerosol Flow Control Device:* The cover gas is drawn from the TP-1 and made to bubble through the paraffin bottle at a specified flow rate using a rotameter. The flow rate is metered in such a way that (i) it takes into account of positive pressure of the cover gas inside the TP-1 and (ii) sufficient flow up to the second bottle. It is optimized that, for the pressure in the cover gas region 0.35 kg/cm^2 and flow rate of 2 L/min, sufficient flow up to the second bottle is achieved.

The pressure holdup test of sampling tube and gate valve has been carried out by pressurizing TP-1 up to 0.5 kg/cm^2. The functioning of surface heaters, PID temperature controller and thermocouples has been checked by heating the heater up to 110°C. The photograph of the sodium

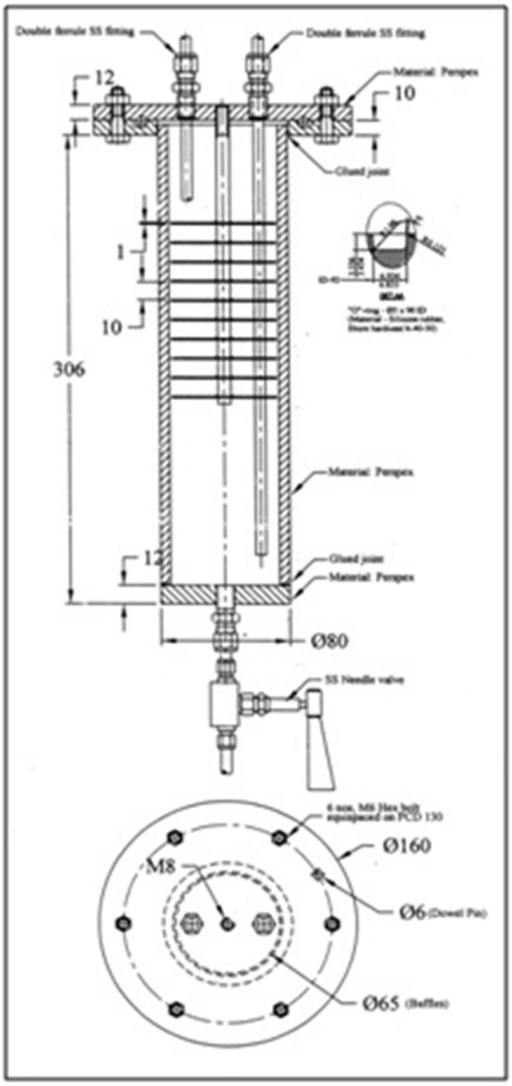

Fig. 5. Schematic diagram of the sampling bottle.

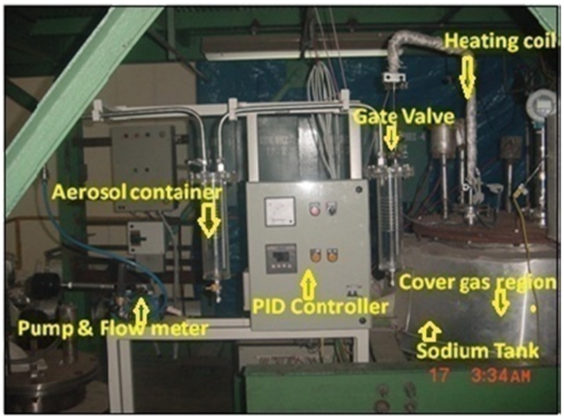

Fig. 6. Photograph of the sampling arrangement.

aerosol sampling system in SILVERINA loop is shown in Fig. 6. The sampling time and flow rate are kept such that the mass of sodium aerosol in the bottle is optimum for measurement of size distribution and mass concentration. Test runs showed that up to a trapped sodium aerosol mass of 50 mg in the bottle, the coagulation of aerosols in the sampling bottle was not noticed before analyzing with Mastersizer (within 20 minutes).

The sampling tube is inserted at the middle level of cover gas region (415 mm from the top). Sodium cover gas height and pressure were kept as 820 mm and 0.35 kg/cm^2 respectively. The sodium pool temperature was maintained at 550°C. Cover gas is made to bubble through liquid paraffin oil at a flow rate of 2 L/min for 15minutes. After sampling, the paraffin oil is analyzed using Mastersizer and the aerosol size distribution is found to vary from 2 to 12 µm with MMD around 4.0 µm (σ_g = 1.5) (Fig. 7). The mass of trapped sodium aerosol in the bottle was estimated and then the mass concentration of sodium aerosol in cover gas region of SILVERINA loop is determined to be 9.50 g/m^3. The mass collected in the second bottle was negligible (< 1% of the first bottle).

SUMMARY

Experimental methodology for characterization sodium aerosols in cover gas region is developed and evaluated. The experimental results agree with the values found in the literature. The design of sampling system in SILVERINA Loop is found very useful and similar one is being fabricated by taking into account of appropriate dimension of reactor vessel of FBTR, for next phase of experiments.

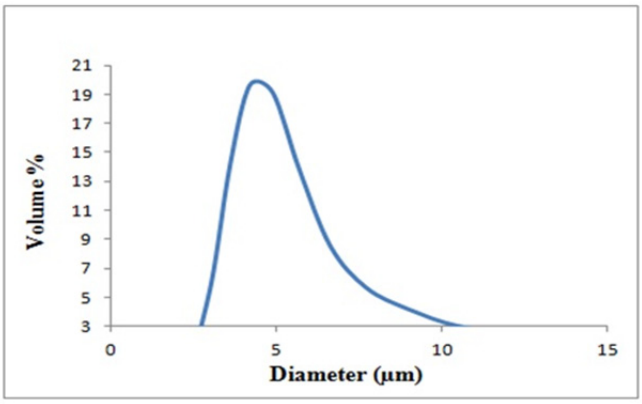

Fig. 7. Sodium aerosol size distribution in SILVERINA Loop.

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