

Fig. 11a. *P. sinfensis*. Fig. 11b. Exine process. Fig. 12a. *Athalmia pinguis*. Fig. 12b. Process. Fig. 13a. *Fimbriaria angusta*. Fig. 13b. Process. Fig. 14a. *F. blumeana*. Fig. 14b. Exine. Fig. 15a. *F. reticulata*. Fig. 15b. LO-pattern. Fig. 16a. *Exormychea tuberculata*. Fig. 16b. Surface pattern. Fig. 17. *Cryptometrium himalayense*. Fig. 18a. *Conocephalum conicum*. Fig. 18b. Exine. Fig. 19a. *Reboulia hemispherica*. Fig. 19b. Exine. (All figures  $\times 450$ ; Fig. representing LO  $\times 1000$ .)

*F. angusta* (reticulate with papillae). *F. blumeana* (reticulate with large lumina). *F. reticulata* (punctate). Exine with papillae of different sizes and blunt ends is represented by *Plagiochasma articulatum*, *P. intermedium*, *Athalmia pinguis*, *Conocephalum conicum* and *Reboulia hemispherica*. Out of three species of *Plagiochasma* studied only *P. intermedium* bears a distinct triradiate mark.

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## GLASS MICROSLIDE AS A THERMAL NEUTRON FLUENCE METER

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#### ABSTRACT

Analysis of the spatial distribution of  $U^{235}$  induced fission tracks in ordinary glass microslides reveals that they can be used for integrated thermal neutron fluence measurement over many orders of magnitude. Fission events are induced in  $U^{235}$  atoms occurring as natural impurities in the glass slides by irradiation with thermal neutrons in Apsara nuclear reactor. The etching of fission tracks and the measurements of track density variation, both in area and depth of the samples, have indicated a uniform and equal distribution of uranium in the slides. The microslides have been calibrated against NBS SRM962 fission track standard glass and GE-Fisher glass. The uranium content in the microslides has been found to be 0.5 ppm. The value for inter-laboratory calibration constant B has been determined as  $1.53 \times 10^{11}$  neutrons per track.

#### INTRODUCTION

FLEISCHER *et al.*<sup>1</sup> have used earlier the sodalime GE-Fisher glass with a calibrated uranium content of 0.35 ppm as a thermal neutron fluence meter in F-T dating. F-T standard glasses were later prepared by many workers<sup>2-5</sup>. At present, various methods exist for the determination of neutron dose given to a material in a nuclear reactor<sup>6</sup> but fission track (F-T) glass fluence meter is one of the cheapest and most accurate. Though small quantities of these standard glasses can be purchased from US National Bureau of Standards yet to use them on a large scale and on routine basis, to study the spatial variation of thermal neutron fluence in a capsule, one needs large

quantities. Hence the necessity arises to calibrate a glass standard available in large quantities.

The basic criteria to be satisfied for dosimetric purposes are that the material (preferably glass) should have uranium as an impurity at a concentration level of 0.01 to 500 ppm and it should be uniformly distributed in the volume. A box of Blue Star Super Deluxe microslides manufactured by Polar Industrial Corporation, Bombay, was investigated for using it as a uranium standard glass fluence meter. These microslides are routinely used in optical microscopy for mounting samples before scanning. They are optically plane, highly transparent and are of uniform thickness.

Since these slides are often used in F-T laboratories for mounting thin sections of rocks for dating work, it is also essential to know the trace quantities of uranium and thorium present in these slides. If the U and Th contents are more than the average value found in minerals, then there are chances of errors in the estimation of uranium concentration of the sample and therefore these slides will prove unsuitable for mounting the mineral sample in F-T dating work. The microslides will prove to be a good thermal neutron fluence meter if the U content is low, i.e. at ppm level.

#### EXPERIMENTAL PROCEDURE

A fresh box of microslides containing 72 slides (75 mm long, 25 mm wide and 1 mm thick) was selected for neutron irradiation. Specimen samples were taken from every fourth slide for study. In this way, small pieces (area =  $1.5 \text{ cm}^2$ ) of the 18 slides were broken from a corner of each slide. The samples were thoroughly cleaned and were packed in an aluminium capsule in the manner shown in Fig. 1. The capsule was irradiated with thermal neutrons to a fluence of  $10^{16} \text{ n cm}^{-2}$  using in IA-2 position of 1 MW Apsara reactor of BARC, Bombay, vide irradiation can No. F1-A 3009. The irradiation time was  $10^4 \text{ s}$  (calculated from the flux value of  $10^{12} \text{ n/cm}^2/\text{s}$  for IA-2 position); NBS SRM962 and GE-Fisher glasses were also packed at specific locations (Fig. 1) in the capsule, to check the homogeneity of the flux given to the finite volume of capsule and to calibrate the parameters of inter-laboratory constant R and elemental uranium concentration, C (eqn. 2). The etching conditions of track revelation were optimized by studying the variation of track density with successive etch time intervals on the irradiated wafer of NBS SRM 962 (RT-3) glass<sup>5</sup>. The results obtained are shown in Fig. 2. The optimum etching conditions have been found to be 20% HF at  $21^\circ \text{C}$  for 1 min. It has been found that optimum etching conditions for GE-Fisher and microscope glasses are the same as those for SRM962. All the three types of irradiated glasses (NBS SRM962, GE-Fisher and Blue Star microslides) were etched with these optimized etching conditions for further analysis.

The procedure for counting of the tracks was the same given by Fleischer *et al.*<sup>1</sup>. For all measurements the track counting was made under  $400\times$  magnification using an amplival (Carl Zeiss Jena make) microscope. Each measurement of the surface track density was followed by a grinding of at least  $15 \mu\text{m}$  thickness of the slide to remove all the surface tracks (confirmed by observations under microscope), polishing and re-etching. These repeated measurements of track density were carried out in each case for at

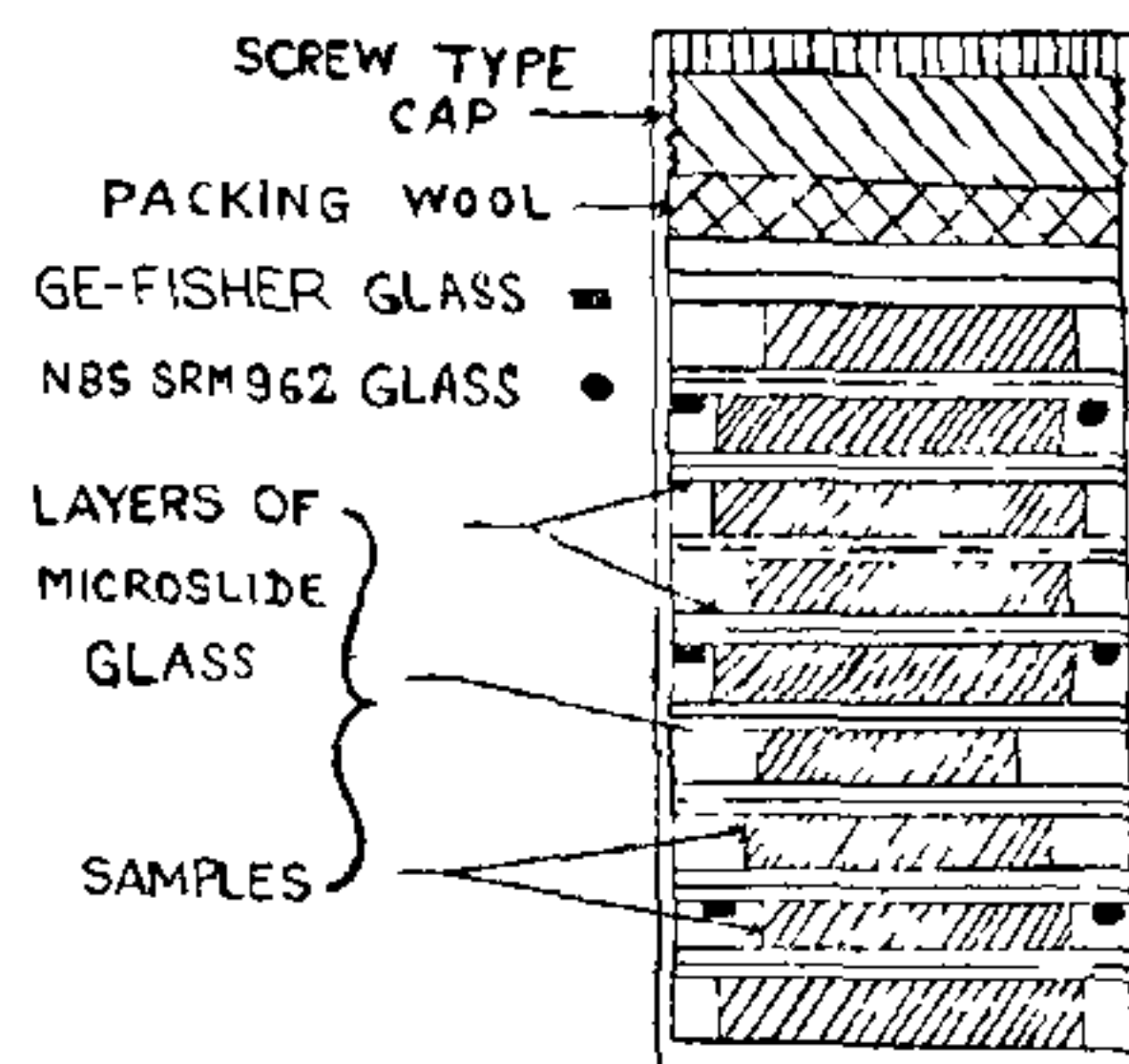


FIG. 1. Packing pattern of the microslides and other uranium standard glasses.

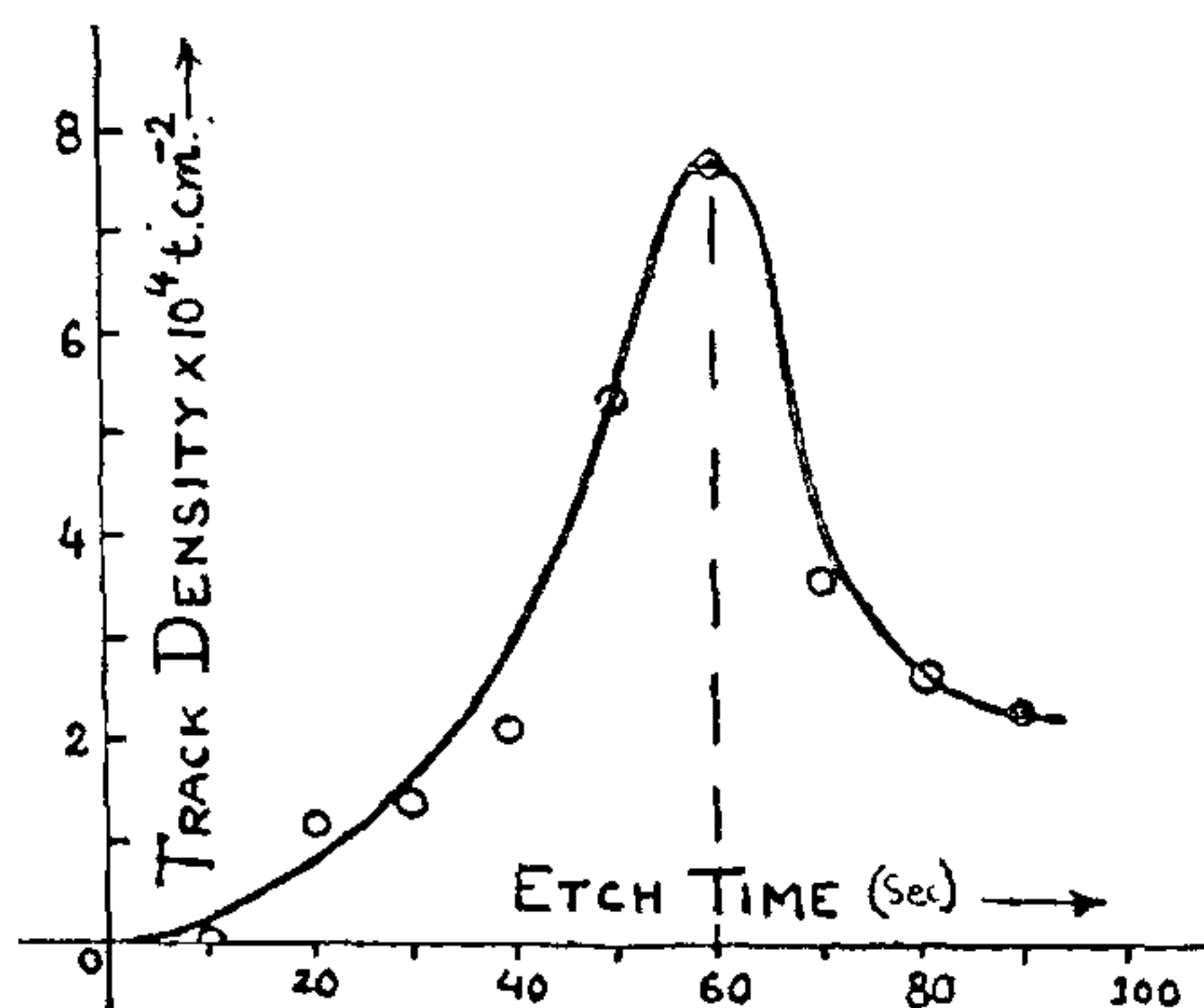


FIG. 2. Fission track density vs. etch time interval.

least 2 levels of depth to determine the volume distribution of U atoms in the microslides. The minimum ground off thickness is approximately  $60 \mu\text{m}$  in this process. Large number of tracks were counted in order to improve the counting statistics. The results thus obtained are summarized in Table I for track counts on microslides used in the present study. The track density measurements on NBS SRM962 and GE-Fisher glasses are given in Table II.

As seen in Table I, the average value of induced track density at different levels of depth in the thickness of these slides comes out to be  $7.67 \times 10^4$ ,  $7.63 \times 10^4$  and  $7.47 \times 10^4$  tracks  $\text{cm}^{-2}$  with an average of  $7.59 \times 10^4$  tracks  $\text{cm}^{-2}$ . Since the NBS SRM962 and GE-Fisher glasses were also irradiated along with the microslides, the thermal neutron dose given to them was same. The uranium standard glasses (NBS SRM962 and GE-Fisher) placed at three different places in the capsule (shown in Fig. 1) indicate that there was no flux gradient in the finite volume ( $18 \text{ mm dia} \times 38 \text{ mm}$ ) of the capsule.



TABLE I  
Track Analysis on Blue Star microscope glass slides

Microscope glass slide number	1st Level		2nd Level		3rd Level	
	Tracks/600 graticules	Track density $\times 10^4 \text{ t cm}^{-2}$	Tracks/600 graticules	Track density $\times 10^4 \text{ t cm}^{-2}$	Tracks/600 graticules	Track density $\times 10^4 \text{ t cm}^{-2}$
1.	2550	7.02	2431	6.69		
2.	2720	7.49	2667	7.34	2690	7.41
3.	2695	7.42	2684	7.39		
4.	2437	6.71	2598	7.15	2407	6.63
5.	3000	8.26	2870	7.90		
6.	2751	7.57	2752	7.58	2674	7.36
7.	2857	7.87	2814	7.75		
8.	2876	7.92	2883	7.94	2810	7.74
9.	2677	7.37	2524	6.95		
10.	2759	7.60	2890	7.96	2790	7.68
11.	2810	7.74	2954	8.13		
12.	2562	7.05	2718	7.48	2710	7.46
13.	3102	8.54	2871	7.90		
14.	2883	7.94	2770	7.63	2719	7.49
15.	2586	7.12	3006	8.28	2840	7.82
16.	3275	9.02	3057	8.42	2810	7.74
17.	2828	7.79	2910	8.01		
18.	2790	7.68	2490	6.85	2688	7.40
Average value	2787	7.67	2771	7.63	2714	7.47

TABLE II  
Track counts in NBS SRM962 and GE-Fisher uranium standard glasses

Position of glasses in the capsule	GE-Fisher		NBS SRM 962	
	Tracks/700 graticules	Track density $\times 10^4 \text{ t cm}^{-2}$	Tracks/300 graticules	Track density $\times 10^6 \text{ t cm}^{-2}$
I	2238	5.28	19469	1.07
II	2148	5.07	20015	1.10
III	2187	5.16	19842	1.09
Average value	2191	5.17	19775	1.09

The relation used for calculating thermal neutron fluence,  $\phi$ , given to the sample is

$$\phi = B\rho_t \quad (1)$$

This relation<sup>1</sup> has been obtained from the total number of fission events induced in a volume of unit area and depth  $2R$ ,  $R$  being the range of fission fragment in the material. On chemical etching, etch-

pits are formed wherever the fission fragments have crossed the surface.  $\rho_t$  refers to measured track density in tracks/cm<sup>2</sup> due to neutron induced fission events and  $B$  is a constant given by

$$B = \frac{Mf}{I N_0 R C d \sigma_f \cos^2 \theta} \quad (2)$$

where  $M$  is the mass number for uranium;  $f$  is the production factor equal to 2 for uranium,  $I$  is the isotopic abundance of the fissioning nuclide ( $\text{U}^{235}$ );  $N_0$  is Avogadro's number;  $R$  is the range of the fission fragment,  $10 \mu\text{m}$  for glass;  $C$  is the concentration of uranium in the glass;  $d$  is the density of the matrix material;  $\sigma_f$  is the thermal neutron fission cross section for  $\text{U}^{235}$  atoms, and  $\cos^2 \theta$  is an etching efficiency factor which is taken to be 1. The values of  $B$  for different glasses determined by different workers are given in Table III. Using the known value of  $B$  for GE-Fisher glass ( $2.26 \times 10^{11}$  neutron/track), the total thermal neutron fluence given to the capsule has been determined to be  $1.16 \times 10^{16} \text{ n/mc}^2$ .

The experimental results obtained on the microslides (Table I) clearly show that the uranium present in them is homogeneously distributed and is of equal concentration in all the slides. This uniform distribution of uranium is not only observed on a parti-

TABLE III  
Characteristic features of various uranium standard glasses used at present in fission track work

Glass fluence meter	Uranium content ppm	$U^{235}/U^{238} \times 10^{-3}$	Value of B neutron/track
Blue Star Microslide	$0.5 \pm 0.01$	7.25	$(1.53 \pm 0.005) \times 10^{11}$ (127184)*
NBS SRM962	$37.38 \pm 0.08^5$	2.392 <sup>5</sup>	$(1.06 \pm 0.005) \times 10^{10}$ (59325)
NBS SRM 962	$37.38 \pm 0.08^5$	2.392 <sup>5</sup>	$5.12 \times 10^9$
GE-Fisher	$0.35 \pm 0.02^1$	7.25 <sup>1</sup>	$2.26 \times 10^{11}{}^1$
Corning U2	$43 \pm 1^2$	0.036 <sup>2</sup>	$(6.41 \pm 0.06) \times 10^9$ (10000)
Kleeman KS43	$0.32 \pm 0.03^8$	Not known	$(1.96 \pm 0.02) \times 10^{11}{}^7$ (11000)

\* Brackets show total number of tracks counted.

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cular surface but in the whole volume of the glass slides.

The concentration of uranium present in the microslides can be determined from the following relation

$$C = 2Mp_4/No d\phi\sigma_f IR \quad (3)$$

where  $\phi$  has earlier been calculated using eqn. (1) and by comparison with standards (NBS SRM962 and GE-Fisher glass). The uranium concentration of microslides has been calculated to be 0.5 ppm.

#### CONCLUSIONS

The box of microscope slides used in the present study has been found to have a uranium impurity at concentration level of 0.5 ppm. The track density measurements in the volume indicate that the uranium is very uniformly distributed in the whole volume of the slides. This characteristic along with the low concentration of uranium present makes these microslides ideal as a neutron fluence meter for all F-T dating work. Since track densities of  $10^2$  to  $10^7$  tracks/cm<sup>2</sup> can be measured with ease, the microslides used in the present study (containing 0.5 ppm U) are useful for monitoring thermal neutron fluences ranging from  $10^{12}$  to  $10^{18}$  neutron/cm<sup>2</sup>. In 1A-2 position (flux  $10^{12}$  n/cm<sup>2</sup>/s) of the Apsara reactor, this will mean that the microslides can be used as thermal neutron monitors for irradiation times ranging from a few seconds to about 10 days. The value for the interlaboratory calibration constant, B for the microslides has been determined to be  $1.53 \times 10^{11}$  neutron/track. The present study indicates that each F-T

dating laboratory may be able to standardize their own neutron fluence meter glass standards using microslides since the box of microscope slides used here has not been chosen under any specific criteria. Specimen sample of the microslides used in the present study is available to any laboratory wishing to use the same as a thermal neutron fluence meter.

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