

National Bureau of Standards Certificate

Standard Reference Material 961 Fission Track Glass Standard (Wafers, 12 mm diameter and 3 mm thick)

B. S. Carpenter

This Standard Reference Material was produced and certified to aid fission track laboratories for interlaboratory comparisons of data and in monitoring neutron flux for irradiations. Uranium fission was induced by neutron irradiation of selected wafers, in separate positions of the NBS Research Reactor, at two different neutron energies.

The wafers comprising this SRM are from the same lot as SRM 610, Trace Elements in Glass. SRM 610 is certified for about half of the more than 60 trace elements including uranium at an elemental concentration = 461.5 ± 1.1 ppm, with isotopic abundance $^{235}\text{U} = 0.2376$ atom percent. The uncertainty of the uranium concentration represents approximately a 95% tolerance interval.

NBS Reactor Position	Neutron Flux Mean Value and Standard Deviation ^a ($\times 10^{12} \text{n} \cdot \text{cm}^{-2} \cdot \text{sec}^{-1}$) ^b	Tolerance Intervals ^c	
		(95%)	(99%)
	<u>Cu Foil</u>		
RT-4	1.31 \pm 0.028	\pm 0.06	\pm 0.09
RT-3	4.56 \pm 0.086	\pm 0.20	\pm 0.28
	<u>Au Foil</u>		
RT-4	1.46 \pm 0.042	\pm 0.09	\pm 0.13
RT-3	5.43 \pm 0.17	\pm 0.39	\pm 0.55

^aStandard deviations refer to individual metal foils.

^bIrradiation was performed at a reduced power of 1 megawatt; 8 seconds in RT-3, or 12 seconds in RT-4.

^cA 95 percent tolerance interval is estimated to include the measurement of approximately 95 percent of all individual wafers of the population of wafers. Thus, the probability is approximately 95 percent that any individual wafer measurement will lie inside the 95 percent tolerance interval. A similar definition holds for 99 percent tolerance interval.

The statistical evaluations were performed by J. Mandel.

Irradiations were performed by G. M. Reimer, P. D. LaFleur, and B. S. Carpenter and preparations by L. Hernandez, J. A. Hormuth, R. M. Young, G. M. Reimer, and B. S. Carpenter.

Track counting was performed by G. M. Reimer and B. S. Carpenter.

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by J. Paul Cali and R. E. Michaelis.

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J. Paul Cali, Chief
 Office of Standard Reference Materials

The SRM package contains four unirradiated glass wafers and two irradiated wafers. The wafer irradiated in position RT-3 is located on the right; on the left is the wafer from RT-4; on the reverse side of the package are numbers corresponding to the irradiation position and respective mica and polycarbonate detectors⁽¹⁾.

Track densities obtained under NBS counting criteria and etching conditions were $\sim 4 \times 10^4 \text{T} \cdot \text{cm}^{-2}$ for RT-4 and $\sim 9 \times 10^4 \text{T} \cdot \text{cm}^{-2}$ for RT-3.

The neutron flux values on this certificate are provided as relative values for the use by the analyst in making nuclear track measurements.

Supplemental Information

Preparation of Wafers for Irradiation

Preparation of this material included grinding and polishing. The glasses were then cleaned with alcohol and given a mild surface cleaning (not etched) in dilute (1:10) nitric acid. Following cleaning, each wafer was placed between numbered pieces of Lexan polycarbonate and muscovite mica and the combination placed in a polyethylene bag that was hermetically vacuum sealed. The sealed material was placed in individual containers for irradiation and every tenth one contained a copper and gold metal foil flux monitor. The material was irradiated in two pneumatic tubes of the NBS reactor. At the proximal terminal position, RT-3 has a cadmium ratio of 10.2 for gold and 65 for copper. Position RT-4 has a cadmium ratio of 87 for gold and 536 for copper. In addition, the mica, polycarbonate and glass were etched ⁽²⁾ and counted. The results from the track counting data further support the consistency of the certified values for the neutron flux.

Homogeneity

Considerable care and effort were devoted to the manufacturing of SRM 610 to ensure homogeneity. To date no element has proven to be heterogeneous outside the limits of error quoted on the Certificate of Analysis.

Recommendations for Use

It is recommended that the irradiated material be polished, removing about 30 micrometers of the glass to reveal an internal surface. The heat buildup during polishing should be kept to a minimum. The surface should be wiped with alcohol and cleaned in dilute (1:10) nitric acid. The irradiated wafers should be etched under the individual laboratory's own etching conditions. One of the nonirradiated wafers, or a piece of a wafer, should be sent to the laboratory's own reactor facilities, irradiated, then polished and etched as described above.

Counting⁽³⁾ of the three wafers [2 NBS irradiated and certified and 1 irradiated by the laboratory] should be done at the same time with the laboratory using its own counting criteria. By comparing track densities of the three wafers, the laboratory should be able to obtain the neutron flux of the reactor used. After the materials have been irradiated and are not in use they should be stored below 20 °C to prevent annealing of the tracks.

NOTE: Elements that may cause possible track interferences are: boron ~ 350 ppm and thorium 457.2 ± 1.2 ppm by weight.

Footnotes

- (1) The corresponding detectors will be retained at NBS for checking purposes should any questions arise concerning a particular sample.
- (2) Etching conditions used were: (a) Polycarbonate 45 minutes at 50 °C in 6.5N NaOH, (b) Mica 15 minutes at 20 °C in 48 percent HF, (c) Glass 75 seconds at 20 °C in 16 percent HF.
- (3) At least 1000 tracks should be counted.