

National Bureau of Standards

Certificate

Standard Reference Material 962a

Fission Track Glass

(Wafers, 12mm diameter and 2mm thick)

This Standard Reference Material (SRM) was produced and certified for use in uranium fission track analysis and for monitoring neutron fluences. The SRM is made up of: four unirradiated glass wafers, two irradiated glass wafers, and muscovite mica and polycarbonate detectors.

The initial preparation and certification of this SRM are described in NBS Special Publication 260-49, "Calibrated Glass Standards for Fission Track Use." The uranium fission in the irradiated wafers was induced by neutron irradiation in the RT-4 and RT-3 positions of the pneumatic tube facilities of the NBS Research Reactor, at two different neutron energies and fluences.

The two irradiated wafers and corresponding irradiation positions are identified on the reverse side of the SRM package.

The neutron fluence values on this Certificate, to be used in nuclear track measurements, are relative values.

The wafers comprising this SRM are from the same bulk material as SRM 612, Trace Elements in Glass. SRM 612 has a certified uranium content of 37.38 ± 0.08 ppm, and a ²³⁵-U isotopic abundance of 0.2392 atom percent. The uncertainty of the uranium content represents a 95% tolerance interval.

<u>NBS Research Reactor Irradiation Position</u>	<u>Neutron Fluence</u>	
	<u>Neutron Fluence Value^{a,b} ($\times 10^{14}$ n·cm⁻²)</u>	<u>Tolerance Interval^c</u>
	<u>Copper Foil</u>	
RT-4	3.87 ± 0.07	± 0.29
RT-3	4.37 ± 0.09	± 0.35
	<u>Gold Foil</u>	
RT-4	4.17 ± 0.08	± 0.33
RT-3	4.75 ± 0.05	± 0.19

(a) Standard Deviation (1 sigma) of individual metal foils.

(b) Neutron fluence based on the operation of the NBS Research Reactor at a power of 8.5 megawatts and irradiation times of 9.2 seconds in RT-3 and 34.5 seconds in RT-4 positions.

(c) The 95% tolerance interval is estimated to include the measurements of approximately 95% of all individual wafers of all units of SRM 962a. Thus, the probability is approximately 95% that any individual wafer measurement will lie within the 95% tolerance interval.

The statistical evaluation of the certification data was performed by R.C. Paule, statistical consultant to the National Measurement Laboratory.

Sample preparation and irradiations were performed in the Inorganic Analytical Research Division by T. Mitlehner and B.S. Carpenter.

The fission track counting was performed by B.S. Carpenter, and C.W. Naeser, N. Naeser, M. Miyachi and R. Zimmermann of the U.S. Geological Survey, Denver, Colorado.

The technical and support aspects involved in the preparation, certification, and issuance of this SRM were coordinated through the Office of Standard Reference Materials by T.E. Gills.

Supplemental Information

Preparation of Wafers

Preparation of SRM 962a included grinding and polishing the glasses from the same base material used for SRM 612. The glasses were then cleaned with alcohol and given a mild surface cleaning (not etched) in dilute 1:10 nitric acid.

Approximately thirty percent of the cleaned wafers were randomly selected for irradiation. Each of the selected wafers was placed between numbered pieces of polycarbonate and muscovite mica detectors and the combination placed in a polyethylene bag and hermetically vacuum sealed.

The sealed wafers were placed in individual irradiation containers with copper, gold, and iron-cobalt metal foils. The wafers were then irradiated using two of the pneumatic tube facilities (RT-3 and RT-4 positions) of the NBS Research Reactor. The cadmium ratio in the RT-3, proximal terminal position, is 10.2 for gold and 65 for copper. The cadmium ratio in the RT-4, proximal terminal position, is 87 for gold and 536 for copper. After irradiation, the mica and polycarbonate detectors and the irradiated glasses were individually etched in sets and counted. The etching conditions were: (a) Polycarbonate, 45 minutes at 50 °C in 6.5N NaOH. (b) Mica, 45 minutes at 25 °C in 48 percent HF, and (c) Glasses, 20 seconds at 25 °C in 24 percent HF. The results from the track counting data support the consistency of the certified values for the neutron fluence.

<u>NBS Research Reactor Irradiation Position</u>	<u>Neutron Fluence Value^{a,b} ($\times 10^{14}$ n·cm⁻²)</u>	<u>Tolerance Interval^c</u>
<u>Iron Foil</u>		
RT-4	3.89 ± 0.08	± 0.31
RT-3	4.25 ± 0.16	± 0.64
<u>Cobalt Foil</u>		
RT-4	3.79 ± 0.12	± 0.48
RT-3	4.49 ± 0.19	± 0.75

a,b,c - Refer to footnotes on page 1.

Note: The neutron fluence values for iron and cobalt are not certified but are provided for information only.

Homogeneity

Considerable care and effort were devoted to the production of SRM 612 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

In order to determine an unknown neutron fluence with SRM 962a, the following procedures are suggested:

1. Determine whether the glasses or the external detector (mica or polycarbonate) will be used as the primary counting medium.
2. Affix the selected detector to one of the nonirradiated wafers and irradiate in the laboratory's own reactor facility.
3. After irradiation, polish the glass wafer to reveal an internal surface. At least 30 micrometers of the glass wafer should be removed. Sequentially, etch the wafer, rinse well, and count the fission tracks. (At least 1000 tracks should be counted.) The unknown neutron fluence can then be calculated from the resulting track densities. Repeat the polishing and etching of the NBS irradiated glasses from RT-3 and RT-4 positions each time a glass is irradiated and counted.
4. Etch the external detectors which were affixed to the glasses (irradiated at the RT-3 and RT-4 positions) supplied in the SRM package. (This should be done only once.) Also, etch the detectors that were affixed to the wafer during irradiation. Count the fission tracks and determine the neutron fluence from the track densities.

The users of this material are cautioned that the track densities given below are approximate. This is because the observed track density, particularly in the glass wafer, is dependent upon etching condition, magnification, and microscope illumination.

After irradiation, all materials should be stored below 20 °C to prevent annealing of the tracks.

Approximate Track Densities ($\times 10^4$ Tracks \cdot cm $^{-2}$)

<u>NBS Research Reactor Irradiation Position</u>	<u>Glass</u>	<u>Mica</u>	<u>Polycarbonate</u>
RT-4	9.0	8.0	8.0
RT-3	10.0	9.0	9.0

NOTE: Elements and concentrations that may cause possible track interferences are: boron, 32 ppm and thorium, 37.79 ppm.