DEPARTMENT OF COMMERCE * NATIONAL BUREAU OF STANDARDS *

NBS SPECIAL PUBLICATION 260-92

U.S. DEPARTMENT OF COMMERCE/National Bureau of Standards

Standard Reference Materials:

Calibrated Glass Standards for Fission Track Use

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Standard Reference Materials:

Calibrated Glass Standards for Fission Track Use (Supplement to NBS SP 260-49)

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PREFACE

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CALIBRATED GLASS STANDARDS FOR FISSION TRACK USE-SUPPLEMENT

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Two glasses of different uranium concentrations were prepared and reissued for certification by the National Bureau of Standards as standards for use as neutron monitors to aid fission track studies. These Standard Reference Materials (SRM's) and their uranium concentrations are: SRM 962a (37.4 ppm) and SRM 963a (0.823 ppm). These glass wafers were irradiated in the National Bureau of Standards Research Reactor and the neutron fluence was monitored using copper and gold foils, as well as an iron-cobalt foil.

Key Words: Fission tracks; flux monitors; glass standards; Standard Reference Material; thermal neutron irradiation; uranium.

I. INTRODUCTION

The fission track technique has become an accepted and widely used method for uranium analysis and radiometric dating in many laboratories. Therefore, the need exists for a primary standard to enable correlation of inter-laboratory results. In order to fulfill this need, the National Bureau of Standards (NBS), through the Office of Standard Reference Materials, has prepared and reissued for certification Fission Track Glass Standards, SRM's 962a and 963a,† (1,2). Although these are not the first standards prepared for fission track work (2,3,4), they are the first readily available fission track standards for monitoring neutron fluence during irradiations and for comparing track densities. Since the glasses have some inadequacies which will be discussed, they are not the "ideal" standards; they are, however, the best standards available at this time.

II. CHARACTERIZATION OF THE GLASS STANDARDS

The Fission Track Glass Standards, SRM numbers 962a and 963a are available at two different uranium concentration levels, 37.38 ppm and 0.823 ppm, respectively. These glass wafers, 12 mm in diameter and 2 mm thick, with a nominal base composition of 72% SiO_2, 12% CaO, 14% Na_2O, and 2% Al_2O_3, came from the same lot of material as the Trace Element in Glass SRM's 612 and 614 (5,6,7). This series of SRM's has been analyzed for homogeneity and was certified for approximately half of the more than 60 trace elements present at the four concentration levels, including uranium. However, in the preparation of these glasses, the uranium used for the dopant was depleted uranium, and each concentration has a different uranium-235 isotopic abundance (as seen in Table I). In determining uranium by the fission track technique, thorium and boron are two major elements that could cause possible interference since they emit charged particles that may be recorded in many external detectors. The concentrations of these two elements are given in Table II. In the certification of these glasses, the analytical competences employed by the Center for Analytical Chemistry were isotope dilution mass spectrometry, polarography, flame emission and atomic absorption spectrometry, spectrophotometry, and nuclear methods of analysis, which included both neutron activation analysis and nuclear track technique.

To further characterize these standards, the thermal stability of fission tracks in the glasses was also studied to determine the fading rate of the tracks when subjected to heat. This thermal stability was determined by the activation energy which is the amount of energy needed for track annealing. Reimer et al (8) found that the glasses had relatively low thermal stability. Extrapolation of track fading data indicates that tracks could begin to decrease at 50 °C after a time of only several months. Figure I illustrates this extrapolation for SRM 962. Table III lists the activation energies calculated from the track fading experiments that would produce a specific degree of track reduction. It is recommended that these glasses be stored below 20 °C when not being used.

III. CERTIFICATION AND PACKAGE DESCRIPTION

The glasses are certified for the total neutron fluence ($n.cm^{-2}$), in which they were irradiated. Copper and gold foils, as well as an iron-cobalt foil, were chosen to monitor the neutron fluence since many laboratories routinely use these foils.

The SRM package contains four unirradiated glass wafers and two separately irradiated wafers. The irradiations were performed in either of two positions of the NBS Research Reactor, each position providing different neutron energy spectra (9,10). Each package has two numbers designating the specific irradiated glasses enclosed. Figure II illustrates the configuration of the SRM package.

† Available from Office of Standard Reference Materials, National Bureau of Standards, Gaithersburg, MD 20899.

IV. PREPARATION AND IRRADIATION PROCEDURES

All of the glasses were ground flat and polished on both sides, cleaned in dilute nitric acid solution (1 part HNO_3: 10 parts H_2O) for 2 minutes, rinsed in distilled water and in ethanol, and air dried. Each cleaned wafer was then sandwiched between two pre-cleaned numbered detectors, Lexan* polycarbonate and muscovite mica. The combination was placed in a polyethylene bag which was hermetically vacuum sealed to ensure intimate contact between detectors and wafers. Each package was then placed in individual irradiation rabbits.

Pre-weighed foil flux monitors of copper, 0.1270-mm thick with average weight 0.021 g; gold, 0.0254-mm thick with average weight 0.031 g; iron-cobalt, 0.0254-mm thick with average weight 0.0037 g, were placed in every tenth rabbit starting with the first rabbit. (See Figure III for sample configuration during irradition.) Each glass concentration level was divided into two groups of 128 samples, and each sample was irradiated individually in one of two different pneumatic tubes of the NBS Reactor (NBSR). In the irradiation positions used for this work, RT-3 has a cadmium ratio of 10.2 for gold foils and 65 for copper foils, while RT-4 has a cadmium ratio of 87 for gold and 536 for copper foils (10). The irradiation time used depended on the uranium concentration and uranium-235 isotopic abundance (see Table II). Due to the NBSR operating schedule the 962a series of glasses were not irradiated at the usual 10 MW Reactor power but at a reduced power or 8.5 MW.

V. FLUENCE DETERMINATION

After irradiation, the gross radioactivity was allowed to decay and the foil flux monitors were removed from the irradiation containers while external detectors and glasses were separated. The activity of the 0.511 MeV, ^{64}Cu positron and its 1.345 MeV gamma-ray was counted with a lithium drifted germanium detector; the same procedure was followed for counting the activity of the 0.412 MeV gamma-ray of ^{198}Au, the 1.099 and 1.292 MeV gamma-rays of Fe and the 1.173 and 1.332 MeV gamma-rays of ^{60}Co. Each foil was counted at various distances from the detector. The detectors had been previously calibrated with NBS SRM's chosen from the NBS radioactivity series; therefore, absolute peak efficiencies were determined for the various γ-ray energies of copper, gold, iron and cobalt. The copper foils were placed between aluminum discs 0.32-mm thick in order to ensure annihilation of all ^{64}Cu, 0.511-MeV positrons close to the foil. After counting all the foils that had been irradiated in the two reactor positions, the neutron flux for each foil was determined using nuclear and physical constants. The following equations were used to calculate the thermal neutron flux:

$$\text{Activity } A = \frac{C}{\Xi a} \qquad (1)$$

A= Activity in nuclear transformations per second at T_o (Time of zero decay, i.e., and of irradiation)

C= Counts per second at T_o

Ξ= Absolute counter efficiency for the appropriate energy gamma-ray

a= Gamma ray intensity; ^{64}Cu(0.511 MeV) = 0.382 and (1.345 MeV)= 0.0062;
^{198}Au(0.412 MeV) = 0.955 ^{59}Fe(1.099 MeV)= 0.565 and (1.292 MeV)
= 0.435; and ^{60}Co(1.173 MeV)= 0.999 and (1.332 MeV)= 0.998 (11)

$$\phi = \frac{A}{\sigma_{th} N(1-e^{-\lambda t})} \qquad (2)$$

ϕ = Thermal neutron flux ($n.cm^{-2}.sec^{-1}$)

σth = Thermal neutron cross section; ^{63}Cu = $4.5x10^{-24}cm^2$, ^{197}Au=9.8.8x$10^{-24}cm^2$, ^{58}Fe=1.14x$10^{-24}cm^2$ and ^{59}Co=18.2x$10^{-24}cm^2$ (Figure IV)

N = Total number of target nuclei

λ = Disintegration constant of radionuclide (^{64}Cu, ^{198}Au, ^{59}Fe or ^{60}Co material)

t = Time duration of exposure to neutron field

then the neutron fluence is

$$\Phi = \phi t \qquad (3)$$

The external detectors, previously separated from the glass wafers, were chemically etched to optically reveal the fission tracks. The polycarbonate detectors were etched in a 6.5N NaOH solution for 45 minutes at a constant temperature of 50 °C, then rinsed in distilled water and air dried.

The mica detectors were etched in 48% HF for 45 minutes at 25 °C, rinsed in water, and heated to dry and to volatilize any remaining HF. The irradiated glass wafers were etched in 24% HF for 20 seconds at 25 °C, rinsed with water for 15 minutes and allowed to air dry.

After etching, both of the external detectors were counted with the aid of an image analyzing microscope under the pre-established conditions of either a minimum of at least 1,000 tracks or 50 random fields of view. The random fields of view were of an area of $1.74 \times 10^{-4} cm^2$ at a magnification of 50X. An example of the tracks densities obtained from the counting of the external detectors and glass wafers are given in Table IV. The same counting criteria were employed for the glasses. Counting data was also collected for each series of metal foils, the two external detectors and both sides of the glasses, and a statistical evaluation was performed for trends or variations in the neutron flux.

The fluence determinations for glasses SRM 962a and 963a are reported in Tables V and VI, respectively. The neutron fluence was determined by multiplying the flux by the irradiation time. There were no obvious trends or variations outside of those imposed by irradiated material.

VI. RECOMMENDED USE OF STANDARD REFERENCE MATERIALS

The SRM's were prepared and certified in a manner to minimize restriction of their specific use. The selection of a reactor facility, the etching conditions, and the counting criteria are all the choice of the individual laboratory.

In order to determine an unknown neutron fluence with this set of glasses, the following procedures are suggested.

1. Determine whether the laboratory will be using glass, mica, or polycarbonate as its primary counting detector medium.
2. Place a piece, or pieces, of unirradiated SRM 962a or SRM 963a with, or without, an external detector (mica or polycarbonate) in the irradiation container with samples.
3. After irradiation, polish all pieces of glass, including RT-3 and RT-4 glasses (SRM 962a or SRM 963a irradiated glasses) to reveal an internal surface. At least 30 μm should be removed. Simultaneously etch all pieces of glass, rinse well, and count. The unknown neutron fluence can then be calculated from the resulting track densities. Repeat the polishing and etching of RT-3 and RT-4 glasses each time a set of samples are irradiated and counted.
4. Etch the external detectors which were over the RT-3 and RT-4 SRM glasses during irradiation. (Note these detectors can only be etched once). At the same time, etch the external detector used in step 2 when unirradiated pieces of SRM 962a or SRM 963a were prepared for irradiation. Then count both sets of detectors and determine the neutron fluence from the track densities.

The users of this material are cautioned that the track densities shown in Table IV are approximate. This is because the observed track density, particularly in glass, is dependent upon etching conditions, magnification, and microscope illumination. It is also suggested that all of the irradiated SRM 962a and SRM 963a glasses be stored below 20 °C to reduce the chance of track annealing.

The two SRM irradiated glasses allow an additional option. There was a difference in the thermal to fast neutron ratio for the irradiation of these wafers, as indicated by the cadmium ratios for gold and copper foil. The wafer selected for the counting comparisons should most closely approximate the neutron energy conditions of the reactor used. The specific glass used as a standard should always be mentioned by its NBS-SRM number in publication.

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Disclaimer

Certain commercial equipment, instruments, or materials are identified in this paper in order to adequately specify the experimental procedure. In no case does such identification imply recommendation or endorsement by the National Bureau of Standards, nor does it imply that the material or equipment identified is necessarily the best available for the purpose.

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Table I

The Uranium Concentration ^{235}U Isotopic Abundance

	Total U Concentration ppm (by weight)	^{235}U Atom Percent	Reactor Location and Irradiation Time (s)	
SRM 962a	37.38 ± 0.08	0.2392	RT-3	9.2
			RT-4	34.5
SRM 963a	0.823 ± 0.002	0.2792	RT-3	80
			RT-4	300

Table II

Concentration of Elements That May Cause Track Interferences

	Boron (ppm by weight)	Thorium (ppm by weight)
SRM 962a	32	37.79 ± 0.08
SRM 963a	1.3	0.748 ± 0.006

Table III

Activation Energies for Track Fading in the NBS Standard Glasses (8)

Density Reduction ϱ/ϱ_o	SRM 964	SRM 963	SRM 962	SRM 961
0.95	0.8 eV	0.7 eV	1.2 eV	0.8 eV
0.90	1.0	0.8	1.3	1.0
0.80	1.3	0.9	1.5	1.2
0.70	1.5	1.1	1.7	1.4
0.60	1.7	1.2	1.8	1.6
0.50	1.8	1.5	2.0	1.7

Table IV

Approximate Track Densities

SRM 962a

	Glass[1]	Mica[2]	Polycarbonate[3]
RT-3	10 x 10^4 t·cm^{-2}	9 x 10^4 t·cm^{-2}	9 x 10^4 t·cm^{-2}
RT-4	9 x 10^4 t·cm^{-2}	8 x 10^4 t·cm^{-2}	8 x 10^4 t·cm^{-2}

SRM 963a

	Glass[1]	Mica[2]	Polycarbonate[3]
RT-3	2.5 x 10^4 t·cm^{-2}	2.2 x 10^4 t·cm^{-2}	2.2 x 10^4 t·cm^{-2}
RT-4	2.4 x 10^4 t·cm^{-2}	2.2 x 10^4 t·cm^{-2}	2.1 x 10^4 t·cm^{-2}

1) Polished internal surface etched for 20 seconds in 24% HF at 25° C, counted at 500X.
2) External (2π) surface etched for 45 minutes in 48% HF at 25° C, counted at 500X.
3) External (2π) surface etched for 45 minutes in 6.5 N NaOH at 50° C, counted at 500X.

Table V

SRM 962a Fission Track Glass Standard Certified Values

NBS Reactor Position	Neutron Fluence Mean Value and Standard Deviation ($\times 10^{14}$ n·cm^{-2})[a, b]	Tolerance Interval[c]
	Cu Foil	
RT-4	3.87 ± 0.07	± 0.29
RT-3	4.37 ± 0.09	± 0.35
	Au Foil	
RT-4	4.17 ± 0.08	± 0.33
RT-3	4.75 ± 0.05	± 0.19
	"For Information Purposes Only"[d]	
	Fe Foil	
RT-4	3.89 ± 0.08	± 0.31
RT-3	4.25 ± 0.16	± 0.64
	Co Foil	
RT-4	3.79 ± 0.12	± 0.48
RT-3	4.49 ± 0.19	± 0.75

[a]Standard Deviations refer to individual metal foils.

[b]Irradiations were performed at a power of 8.5 megawatts; 9.2 seconds in RT-3, or 34.5 seconds in RT-4.

[c]Tolerance intervals for 95% of the SRM population at the 95% confidence level.

[d]These results show greater imprecision.

Table VI

SRM 963a Fission Track Glass Standard Certified Values

NBS Reactor Position	Neutron Fluence Mean Values and Standard Deviation ($\times 10^{14}$ $n \cdot cm^{-2}$)[a, b]	Tolerance Interval[c]
	Cu Foil	
RT-4	39.5 ± 0.1	± 0.5
RT-3	41.2 ± 0.7	± 2.6
	Au Foil	
RT-4	43.0 ± 0.7	± 2.9
RT-3	45.8 ± 0.4	± 1.5
	"For Information Purposes Only"[d]	
	Fe Foil	
RT-4	40.2 ± 1.3	± 5.3
RT-3	40.7 ± 1.1	± 4.5
	Co Foil	
RT-4	41.2 ± 1.7	± 6.7
RT-3	45.7 ± 2.2	± 8.7

[a]Standard Deviations refer to individual metal foils.

[b]Irradiations were performed at a power of 10.0 megawatts; 80 seconds in RT-3, or 300 seconds in RT-4.

[c]Tolerance intervals for 95% of the SRM population at the 95% confidence level.

[d]These results show greater imprecision.

Figure I. Annealing Studies Indicating the Fraction of Uranium Fission Tracks Reduced as a Function of Time and Temperature in the 50 ppm SRM 962

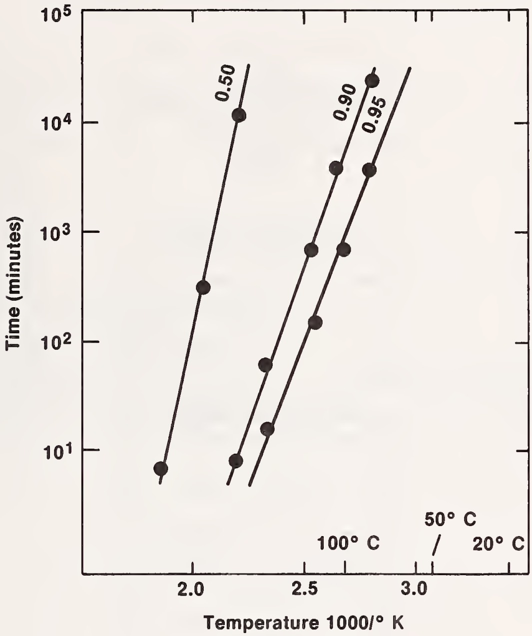


Figure II. Position of RT-3 and RT-4 Irradiated Glasses

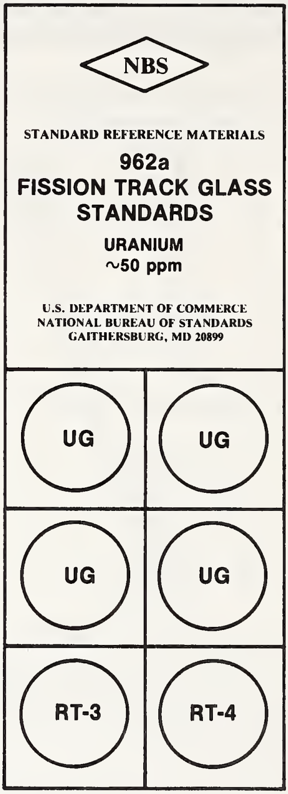


Figure III. Arrangement of the Glass, Metal Foils and Detectors During Neutron Irradiations

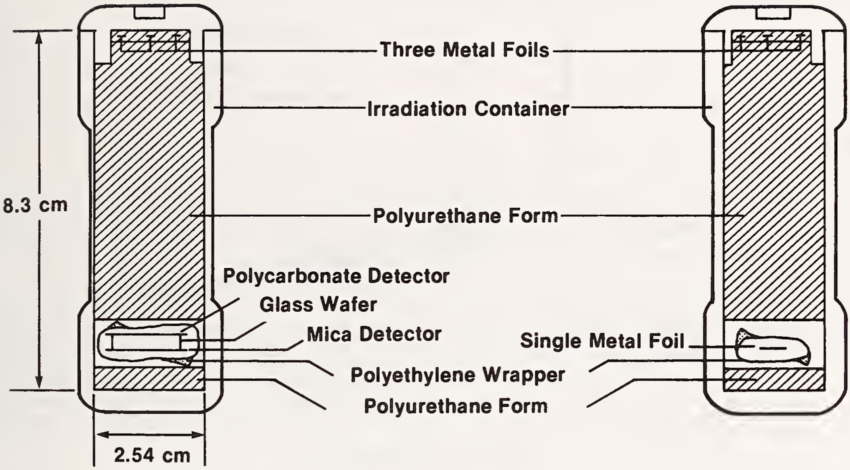
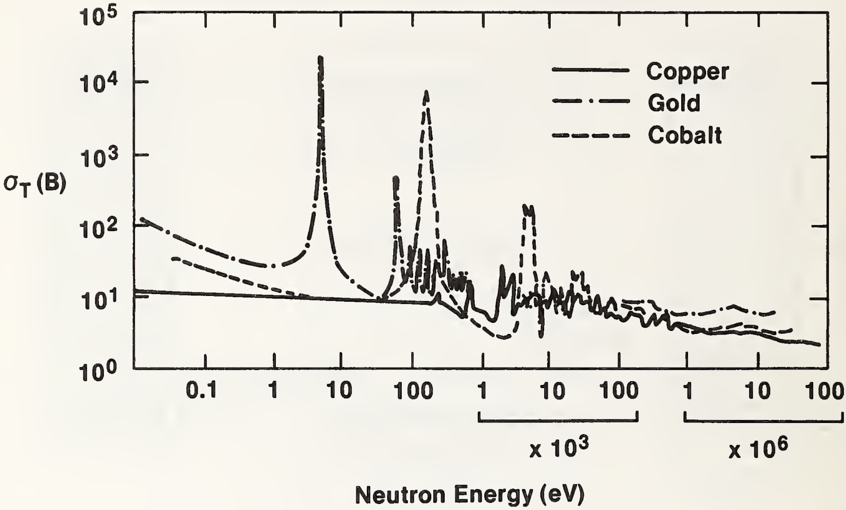


Figure IV. Neutron Cross Sections of Copper, Gold and Cobalt as a Function of Neutron Energy



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11. ABSTRACT *(A 200-word or less factual summary of most significant information. If document includes a significant bibliography or literature survey, mention it here)*

Two glasses of different uranium concentrations were prepared and reissued for certification by the National Bureau of Standards as standards for use as neutron monitors to aid fission track studies. These Standard Reference Materials (SRM's) and their uranium concentrations are: SRM 962a (37.4 ppm) and SRM 963a (0.823 ppm). These glass wafers were irradiated in the National Bureau of Standards Research Reactor and the neutron fluence was monitored using copper and gold foils, as well as an iron-cobalt foil.

12. KEY WORDS *(Six to twelve entries; alphabetical order; capitalize only proper names; and separate key words by semicolons)*

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