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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

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PREFACE

Standard Reference Materials (SRM's) as defined by the National Bureau of Standards are "well-characterized materials, produced in quantity, that calibrate a measurement system to assure compatibility of measurement in the nation." SRM's are widely used as primary standards in many diverse fields in science, industry, and technology, both within the United States and throughout the world. In many industries traceability of their quality control process to the national measurement system is carried out through the mechanism and use of SRM's. For many of the nation's scientists and technologists it is therefore of more than passing interest to know the details of the measurements made at NBS in arriving at the certified values of the SRM's produced. An NBS series of papers, of which this publication is a member, called the NBS Special Publication - 260 Series is reserved for this purpose.

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Inquiries concerning the technical content of this paper should be directed to the author(s). Other questions concerned with the availability, delivery, price, and so forth will receive prompt attention from:

Office of Standard Reference Materials
National Bureau of Standards
Washington, D.C. 20234

J. Paul Cali, Chief
Office of Standard Reference Materials

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CALIBRATED GLASS STANDARDS FOR FISSION TRACK USE

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Four glasses of different uranium concentrations were prepared and certified by the National Bureau of Standards as standards for use as neutron monitors to aid fission track studies. These Standard Reference Materials (SRM) and their uranium concentrations are: SRM 941 (461 ppm), SRM 962 (37.4 ppm), SRM 963 (0.823 ppm), and SRM 964 (0.0721 ppm). These glass wafers were irradiated in the National Bureau of Standards Reactor and the neutron flux was monitored using copper and gold foils.

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 certified Fission Track Glass Standards SRM's 961, 962, 963, and 964† (1). Although these are not the first standards prepared for fission track work (2,3), they are the first readily available fission track standards for monitoring neutron fluxes during irradiations and for comparing track densities. Since the

† Available from Office of Standard Reference Materials, National Bureau of Standards, Washington,D.C. 20234.

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 961, 962, 963, and 964, are available at four different uranium concentration levels, 461.5 ppm, 37.38 ppm, 0.823 ppm and 0.0721 ppm, respectively. These glass wafers, 12 mm in diameter and 3-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 610 through 616 (4,5,6). This series of SRM's has been analyzed for homogeneity and was 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 Analytical Chemistry Division 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. Reimer et al (7) 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 neutron flux ($n \cdot cm^{-2} sec^{-1}$), in which they were irradiated. Copper and gold foils were chosen to monitor the neutron flux 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 Reactor, each position providing different neutron energy spectra (8,9). Each package has two numbers designating the specific irradiated glasses enclosed. Figure II illustrates the configuration of the SRM package.

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, 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, and gold, 0.0254-mm thick with average weight 0.031 g, were placed in every tenth rabbit. (See Figure III for sample configuration during irradiation.) Each glass concentration level was divided into two groups of 125 samples, and each sample was irradiated individually in one of two different pneumatic tubes of the NBS Reactor (NBSR). In the radiation 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 (9). The irradiation time used depended on the uranium concentration and uranium-235 isotopic abundance (see Table II). Due to the high uranium content of the 961 series of glasses, the irradiations were not performed at the usual 10 MW Reactor power but at a reduced power of 1 MW.

V. FLUX 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 was counted with a 60cc Ge(Li) solid state detector; the same procedure was followed for counting the activity of the 0.412-MeV gamma-ray of ^{198}Au. Each foil was counted 2 cm from the detector. The detectors had been previously calibrated with NBS SRM's chosen from the NBS radio-activity

series; therefore, absolute peak efficiencies were determined for the various γ-ray energies of copper and gold. 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:

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

A= activity in nuclear transformations per second at T_o (Time of zero decay, i.e., end 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) = 0.38; ^{198}Au(0.412 MeV) = 0.95 (10)

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

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

σ_{th} = Thermal neutron cross section; $^{63}Cu = 4.5 \times 10^{-24} cm^2$, $^{197}Au = 98.8 \times 10^{-24} cm^2$

N = Total number of target nuclei

λ = Disintegration constant of radionuclide (^{64}Au or ^{198}Au) material

t = Time duration of exposure to neutron flux

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 15 minutes at 20 °C, rinsed in water, and heated to dry and to volatilize any remaining HF. The irradiated glass wafers were etched in 16% HF for 75 seconds at 20 °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 480X. 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 flux determinations for glasses SRM 961, 962, 963, and 964 are reported in Tables IV, V, VI, and VII, respectively. The neutron fluence is determined by multiplying the flux by the irradiation time. There were no obvious trends or variations outside of those imposed by counting statistics that would require rejecting any of the irradiated material. However, a systematic effect was noticed for Glasses 963 and 964 which were irradiated over several days. No such errors were indicated in Glasses 961 and 962 which were all irradiated during the same day.

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.

It is recommended that one of the non-irradiated wafers, or a portion of it, be included with the samples being irradiated. The choice of the wafer included should be based upon the total expected neutron exposure and the uranium concentration of the wafer so that the number of tracks produced will be in a range convenient for counting.

After irradiation, the wafer should be polished together with the SRM irradiated wafer to remove approximately 30 micrometers from the surface, which is greater than the fragment range, thus providing an internal surface. Low speed polishing should be used to keep heating to a minimum. Both glasses should then be etched and counted together. The unknown neutron fluence can be determined from the track densities and the known neutron exposure of the SRM irradiated wafer. The counting accuracy would be dependent on the number of tracks counted.

For future irradiations, it is not necessary to repolish and re-etch the SRM irradiated standard if the etching conditions of the newly irradiated wafer are identical to those of the previously etched SRM irradiated wafer. They need only to be counted together. It is suggested, however, that all the irradiated 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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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.

Table I

The Uranium Concentration ^{235}U Isotopic Abundance

	Total U Concentration ppm (by weight)	^{235}U Atom Percent	Reactor Location and Irradiation Time (sec.)	
SRM 961	461.5 ± 1.1	0.2376	RT-3	8
			RT-4	12
SRM 962	37.38 ± 0.08	0.2392	RT-3	8
			RT-4	12
SRM 963	0.823 ± 0.002	0.2792	RT-3	80
			RT-4	120
SRM 964	0.0721 ± 0.0013	0.616	RT-3	360
			RT-4	540

Table II

Concentration of Elements That May Cause Track Interferences

	Boron (ppm by weight)	Thorium (ppm by weight)
SRM 961	351	457.2 ± 1.2
SRM 962	32	37.79 ± 0.08
SRM 963	1.3	0.748 ± 0.006
SRM 964	0.20 ± 0.002	0.0252 ± 0.0007

Table III

Activation Energies for Track Fading
in the NBS Standard Glasses

Density Reduction ρ/ρ_0	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

SRM 961 Fission Track Glass Standard

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

[a] Standard deviations refer to individual metal foils.

[b] Irradiation was performed at a power of 10 megawatts;

[c] A 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.

Table V

SRM 962 Fission Track Glass Standard

NBS Reactor Position	Neutron Flux Mean Value and Standard Deviation[a] ($\times 10^{13}$ n·cm^{-2}·sec^{-1})[b]	Tolerance Intervals[c] (95%)	(99%)
	Cu Foil		
RT-4	1.29 ± 0.072	±0.16	±0.23
RT-3	5.29 ± 0.21	±0.49	±0.69
	Au Foil		
RT-4	1.43 ± 0.012	±0.03	±0.04
RT-3	5.93 ± 0.17	±0.40	±0.57

[a] Standard deviations refer to individual metal foils

[b] Irradiation was performed at a power of 10 megawatts; 8 seconds in RT-3, or 12 seconds in RT-4.

[c] A 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.

Table VI

SRM 963 Fission Track Glass Standard

Wafer Number Identification	NBS Reactor Position	Neutron Flux Mean Value and Standard Deviation[a] ($\times 10^{13}$ n·cm^{-2}·sec^{-1})[b]	Tolerance Intervals[c] (95%)	(99%)
		Cu Foil		
614001-614040	RT-4	1.26 ± 0.026	±0.06	±0.09
614041-614090	RT-4	1.24 ± 0.026	±0.06	±0.09
614091-614125	RT-4	1.26 ± 0.026	±0.06	±0.09
614126-614165	RT-3	5.38 ± 0.17	±0.42	±0.60
614166-614215	RT-3	5.10 ± 0.17	±0.42	±0.59
614216-614251	RT-3	5.14 ± 0.17	±0.42	±0.60
		Au Foil		
614001-614040	RT-4	1.41 ± 0.024	±0.06	±0.09
614041-614090	RT-4	1.36 ± 0.024	±0.06	±0.08
614091-614125	RT-4	1.44 ± 0.024	±0.06	±0.09
614126-614165	RT-3	6.03 ± 0.059	±0.15	±0.21
614166-614215	RT-3	5.97 ± 0.059	±0.15	±0.21
614216-614251	RT-3	6.13 ± 0.059	±0.15	±0.21

[a] Standard deviations refer to individual metal foils.

[b] Irradiation was performed at a power of 10 megawatts; 80 seconds in RT-3, or 120 seconds in RT-4.

[c] A 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.

Table VII

SRM 964 Fission Track Glass Standards

Wafer Number Identification	NBS Reactor Position	Neutron Flux Mean Value and Standard Deviation[a] ($\times 10^{12}$ n·cm^{-2}·sec^{-1})[b]	Tolerance Intervals[c] (95%)	(99%)
		Cu Foil		
616001-616027	RT-4	1.31 ± 0.057	±0.15	±0.21
616028-616060	RT-4	1.26 ± 0.057	±0.15	±0.21
616061-616090	RT-4	1.32 ± 0.033	±0.08	±0.12
616091-616125	RT-4	1.39 ± 0.033	±0.08	±0.12
616126-616163	RT-3	4.84 ± 0.20	±0.52	±0.76
616164-616185	RT-3	4.57 ± 0.20	±0.57	±0.84
616186-616195	RT-3	4.50 ± 0.20	±0.66	±0.96
616196-616215	RT-3	4.55 ± 0.20	±0.57	±0.84
616216-616250	RT-3	4.41 ± 0.20	±0.52	±0.76
		Au Foil		
616001-616027	RT-4	1.46 ± 0.033	±0.08	±0.12
616029-616060	RT-4	1.48 ± 0.033	±0.08	±0.12
616061-616090	RT-4	1.40 ± 0.057	±0.15	±0.21
616091-616125	RT-4	1.41 ± 0.057	±0.14	±0.21
616126-616163	RT-3	4.69 ± 0.10	±0.26	±0.37
616164-616185	RT-3	5.21 ± 0.10	±0.28	±0.41
616186-616195	RT-3	5.45 ± 0.10	±0.32	±0.47
616196-616215	RT-3	4.11 ± 0.10	±0.28	±0.41
616216-616250	RT-3	4.50 ± 0.10	±0.26	±0.37

[a] Standard deviations refer to individual metal foils.

[b] Irradiation was performed at a power of 10 megawatts; 360 seconds in RT-3, or 540 seconds in RT-4.

[c] A 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.

Figure I. Annealing studies of uranium fission tracks in the 50 ppm SRM 962.

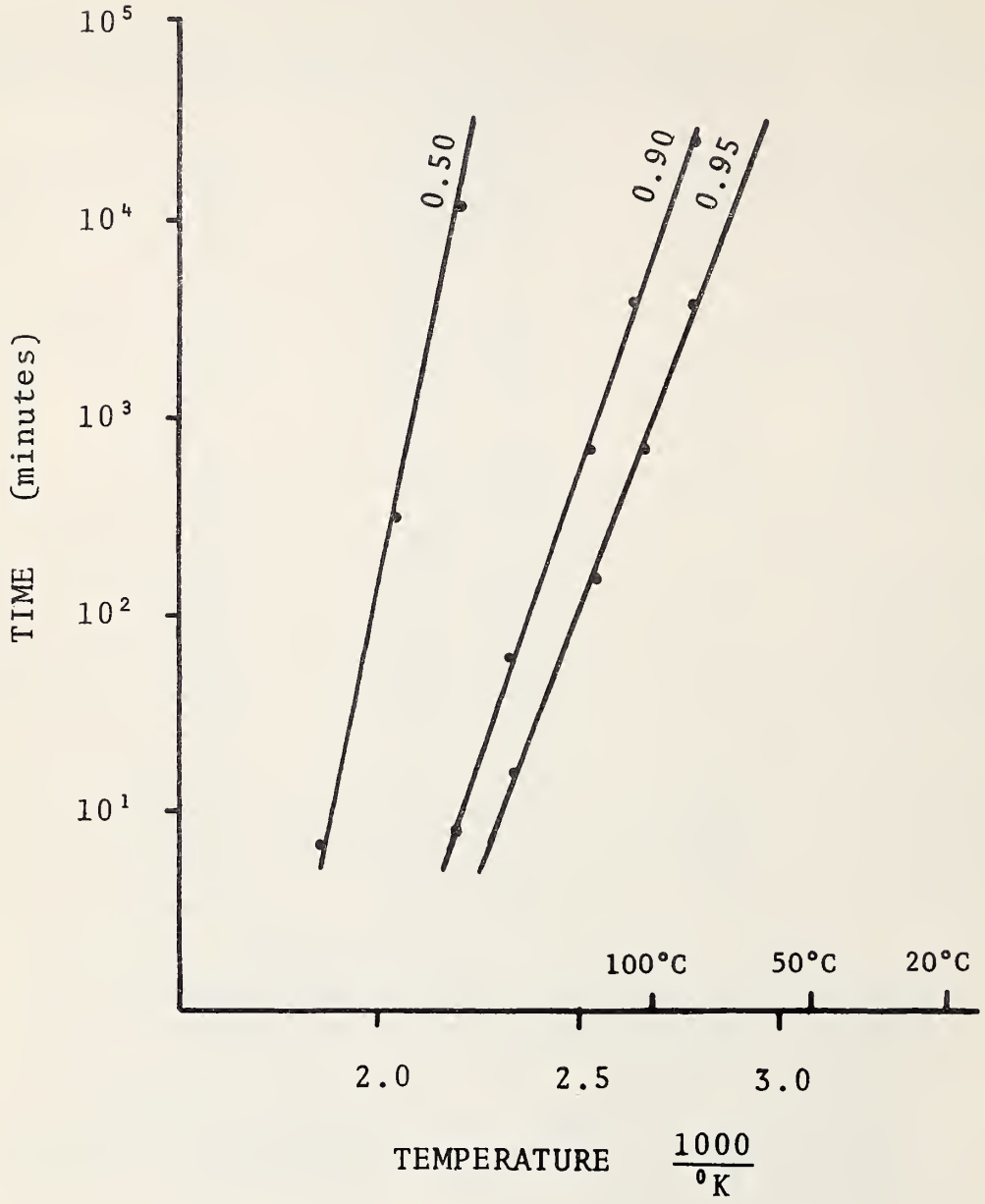


Figure II. Position of RT-3 and RT-4 irradiated glasses.

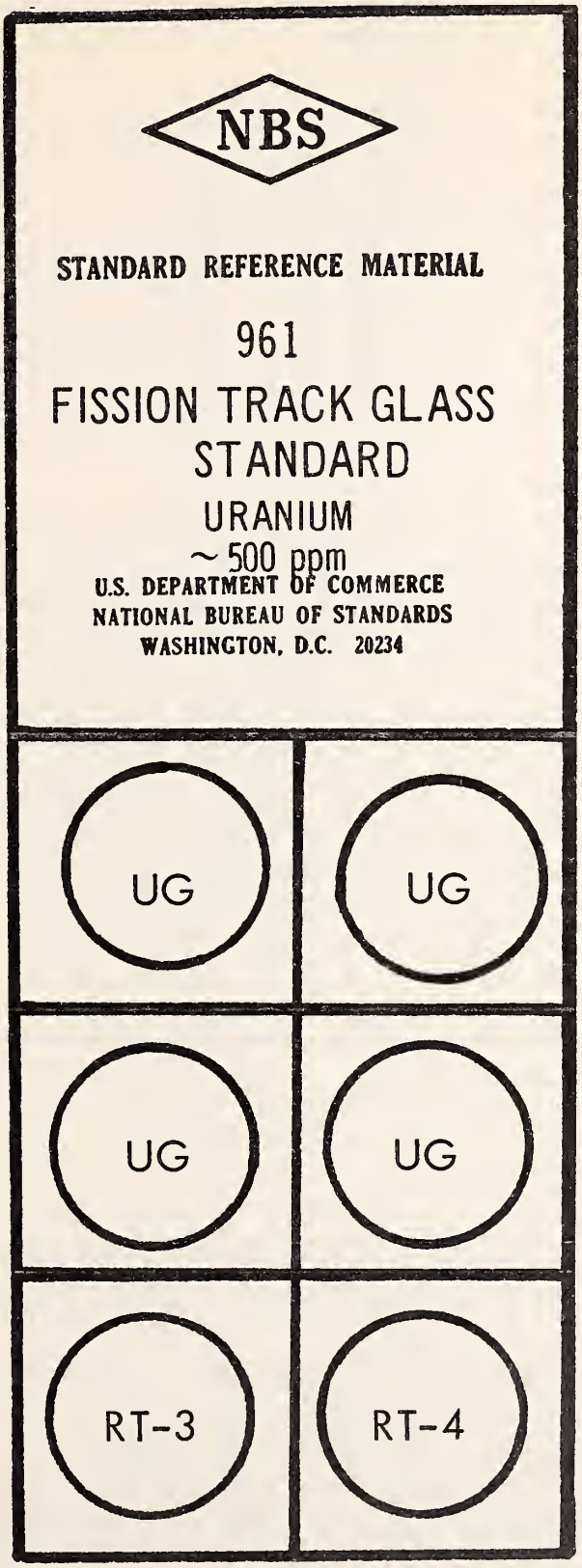
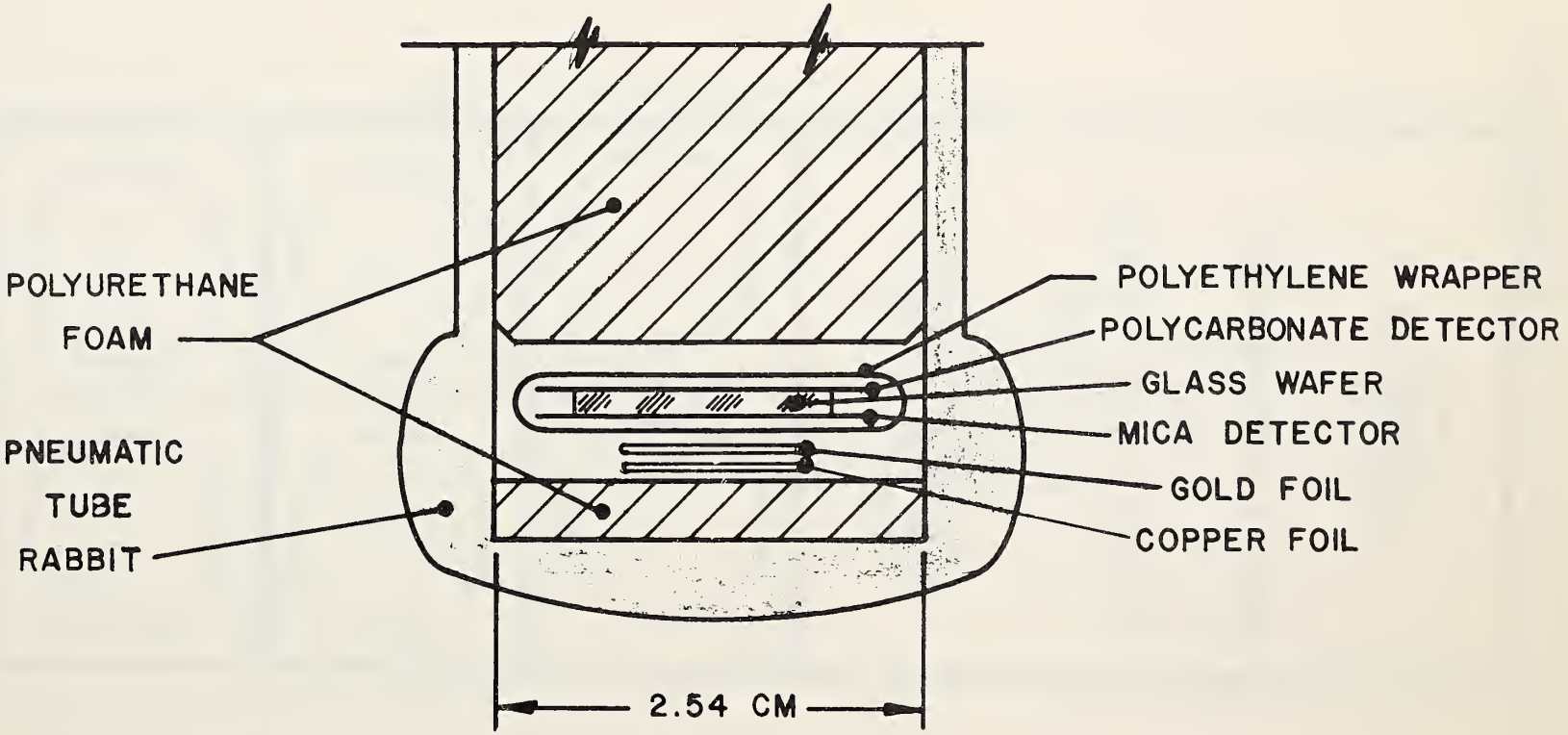


Figure III. Arrangement of the glass, metal foils and detectors during neutro irradiation.



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

Four glasses of different uranium concentrations were prepared and certified by the National Bureau of Standards as standards for use as neutron monitors to aid fission track studies. These Standard Reference Materials (SRM) and their uranium concentrations are: SRM 941 (461 ppm), SRM 962 (37.4 ppm), SRM 963 (0.823 ppm), and SRM 964 (0.0721 ppm). These glass wafers were irradiated in the National Bureau of Standards Reactor and the neutron flux was monitored using copper and gold foils.

17. KEY WORDS *(six to twelve entries; alphabetical order; capitalize only the first letter of the first key word unless a proper name; separated by semicolons)* Fission tracks; flux monitors; glass standards; Standard Reference Material; thermal neutron irradiation; uranium.

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